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AFFIDAVIT OF TRANSLATION

I, Alan F. Siegrist, of CROSSLINGUAL, LLC, hereby declare that:

1. I am fluent in Japanese and English.
2. I am an active member of the American Translators Association and a Certified Translator of Japanese to English.
3. The English translation attached to this declaration is an accurate and correct translation of the following document, attached hereto:
Sawa Declaration_2.18.2016

I declare that the foregoing is true and correct to the best of my knowledge.

Executed on February 19, 2016

Alan F. Siegrist, CT
 CROSSLINGUAL, LLC
 ATA Member No. 31889
 Certification #63788



Verify at www.ata.net.org/verify

A notary public or other officer completing this certificate verifies only the identity of the individual who signed the documents to which this certificate are attached, and not the truthfulness, accuracy, or validity of that document.

State of California, County of Contra Costa
 On February 19, 2016 before me, Daniel Isaac Baum, Notary Public
 personally appeared Alan F. Siegrist who proved to me on the basis of satisfactory evidence to be the person whose name is subscribed to the within Instrument and acknowledged to me that he executed the same in his authorized capacity, and that by his signature on the Instrument the person, or the entity upon behalf of which the person acted, executed the instrument.

I certify under PENALTY OF PERJURY under the laws of the State of California that the foregoing paragraph is true and correct.

Witness my hand and official seal.

Signature (Seal)



UNITED STATES PATENT AND TRADEMARK OFFICE

BEFORE THE PATENT TRIAL AND APPEAL BOARD

LUPIN LTD. and LUPIN PHARMACEUTICALS INC..

Petitioner,

v.

SENJU PHARMACEUTICAL CO., LTD.

Patent Owner.

Cases IPR2015-01099, IPR2015-01097, IPR2015-01105 & IPR2015-01100

Patents 8,669,290, 8,754,131, 8,871,813 & 8,927,606

DECLARATION OF SHIROU SAWA

I, Shirou Sawa, under penalty of perjury, declare as follows:

I. INTRODUCTION

1. I am of legal age and otherwise competent to make this declaration.
2. I am the first named inventor on U.S. Patent Nos. 8,669,290 (“the ’290 patent”), 8,754,131 (“the ’131 patent”), 8,871,813 (“the ’813 patent”) and 8,927,606 (“the ’606 patent”). I have been asked to submit a declaration attesting to how the data provided below, including data disclosed in the specification of the ’290 patent, the ’131 patent, the ’813 patent and the ’606 patent, were generated.

II. EDUCATION AND WORK EXPERIENCE

3. I graduated from the University of Tokushima, Tokushima, Japan in 1988, with a bachelor’s degree from the Department of Chemical Engineering, in the Faculty of Engineering. I received a master’s degree in engineering, with chemical engineering specialization, from the University of Tokushima, Tokushima, Japan in 1990.

4. I am currently employed by Senju Pharmaceutical, Co., Ltd. (“Senju”) and have been with Senju since 1990. I have worked at Senju as a researcher in the ophthalmic formulations area from 1990 to the present.

III. DATA, INCLUDING DATA IN THE ’290, ’131, ’813 and ’606 PATENTS

5. I was involved in formulating and testing bromfenac sodium (sodium 2-amino-3-(4-bromobenzoyl)-phenylacetate) formulations, including the formulations of the Experimental Examples reported in the '290, '131, '813 and '606 patents. As such, I have first-hand knowledge of how the formulations reported below were made and tested.

6. The laboratory notebooks in Appendices A, B and C describe the tested formulations and the experimental test results obtained with them.

A. Stability data, including data from Table 1 of the '290, '131, '813 and '606 patents

7. I prepared and tested the stability of the bromfenac sodium formulations disclosed in Table 1 of the '290, '131, '813 and '606 patents. The table below contains bromfenac sodium formulations from Table 1 of the patents, as well as additional formulations with varying amounts of tyloxapol. (Appendix A.) As the table indicates, formulations A-20, A-21 and A-27 correspond to Comparison Examples 1, A-02 and A-03, respectively, from Table 1 of the '290, '131, '813 and '606 patents.

8. As reflected in the laboratory notebook of Appendix A, the stability of these bromfenac sodium formulations was tested after adjusting the pH of the formulations to 7. Using these formulations, accelerated stability tests were conducted for various lengths of time and elevated temperatures, including for four weeks at 60 °C. The percent of bromfenac sodium remaining was measured using

a High Performance Liquid Chromatography (“HPLC”) method under the following conditions:

- Ultraviolet absorbance spectroscopy at 266 nm;
- Column: Capcelpak column;
- Column temperature: 25 °C;
- Mobile phase: 1.98 g of ammonium dihydrogen phosphate was dissolved in 750 mL of water, the pH was adjusted to 7.3 by adding phosphoric acid, and 250 mL of acetonitrile was added;
- Flow rate: Adjusted so that the elution time of bromfenac sodium becomes 18 minutes; and
- Injection volume of sample: 10 µL

9. The percent of bromfenac sodium remaining in each formulation after four weeks at 60 °C is tabulated below. The percent of bromfenac sodium remaining was adjusted to take into account the amount of water evaporation from the formulation.

Formulation code	A-20	A-21	A-27	A-28	A-29
Designated code in Table 1 of the '290, '131, '813 and '606 patents	Comparison Example 1	A-02	A-03	N/A	N/A
Bromfenac sodium hydrate ¹	0.1 g	0.1 g	0.1 g	0.1 g	0.1 g
Boric acid	1.5 g	1.5 g	1.6 g	1.6 g	1.6 g
Benzalkonium chloride	0.005 g	0.005 g	0.005 g	0.005 g	0.005 g
Polysorbate 80	0.17 g	-	-	-	-
Tyloxapol	-	0.15 g	0.02 g	0.05 g	0.1 g
Sodium hydroxide	q.s.	q.s.	q.s.	q.s.	q.s.
Distilled water	q.s.	q.s.	q.s.	q.s.	q.s.
Total amount	100 mL	100 mL	100 mL	100 mL	100 mL
pH	7	7	7	7	7
60 °C - 4 weeks	51.27%	73.81%	89.64%	85.96%	82.01%

B. Stability data, including data disclosed in Table 2 of the '290, '131, '813 and '606 patents

10. I prepared and tested the stability of the following bromfenac sodium formulations, including those disclosed in Table 2 of the '290, '131, '813 and '606 patents. As indicated in the following table, formulations A-01, A-02 and A-03 correspond to formulations A-04, A-05 and A-06, respectively, from Table 2 of the '290, '131, '813 and '606 patents. Stability tests on these formulations were carried out for various lengths of time and elevated temperatures, including four weeks and 60 °C, adjusted to a pH of about 8.15, as indicated. The percent of bromfenac sodium remaining in each formulation after four weeks at 60 °C was

¹ In the appended laboratory notebooks, I used the shorthand "bromfenac sodium" to refer to "bromfenac sodium hydrate."

measured using the HPLC method described above, which was adjusted to take into account the amount of water evaporation. The data are tabulated below.

(Appendix C.)

Formulation code	A-01	A-02	A-03
Designated code in Table 2 of the '290, '131, '813 and '606 patents	A-04	A-05	A-06
Bromfenac sodium hydrate	0.1 g	0.1 g	0.1 g
Boric acid	1.1 g	1.1 g	1.1 g
Borax	1.1 g	1.1 g	1.1 g
Benzalkonium chloride	0.005 g	0.005 g	0.005 g
Tyloxapol	0.02 g	0.05 g	0.03 g
Polyvinylpyrrolidone	2.0 g	2.0 g	2.0 g
Disodium edetate	0.02 g	0.02 g	0.02 g
Sodium hydroxide	q.s	q.s	q.s.
Distilled water	q.s.	q.s.	q.s.
Total amount	100 mL	100 mL	100 mL
pH	8.15	8.15	8.15
60 °C - 4 weeks	92.57%	90.93%	91.97%

C. Stability data for bromfenac sodium formulations containing polysorbate 80 and bromfenac sodium formulations containing tyloxapol

11. I prepared and tested the following bromfenac sodium formulations, containing the components and amounts as indicated below. Stability tests on these formulations, adjusted to a pH of about 8.2 to 8.3, were carried out for various lengths of time and elevated temperatures, including for four weeks and 60 °C, as indicated. The percent of bromfenac sodium remaining in each formulation after four weeks at 60 °C was measured using the HPLC method

described above, which was adjusted to take into account the amount of water evaporation. The data are tabulated below. (Appendix C.)

Formulation code	BF (PE) ² (Bronuck)	A-01 (PE)	A-03 (PE)
Bromfenac sodium hydrate	0.1 g	0.1 g	0.1 g
Boric acid	1.1 g	1.1 g	1.1 g
Borax	1.1 g	1.1 g	1.1 g
Benzalkonium chloride	0.005 g	0.005 g	0.005 g
Polysorbate 80	0.15 g	-	-
Tyloxapol	-	0.02 g	0.03 g
Polyvinylpyrrolidone	2.0 g	2.0 g	2.0 g
Disodium edetate	0.02 g	0.02 g	0.02 g
Sodium sulfite	0.2 g	-	-
Sodium hydroxide	q.s	q.s	q.s.
Distilled water	q.s.	q.s.	q.s.
Total amount	100 mL	100 mL	100 mL
pH	8.20	8.20	8.27
60 °C - 4 weeks	91.45%	93.61%	95.07%

D. Preservative efficacy of bromfenac sodium formulations

12. I was involved with testing the preservative efficacy of bromfenac sodium formulations as part of projects P2002B116 and P2002B131 at Senju. I formulated a bromfenac sodium formulation containing polysorbate 80 (identical to Bronuck) and bromfenac sodium formulations containing tyloxapol as shown in the following table. (Appendices B & C.) Formulations A-01 and A-02 in this table are the bromfenac sodium formulations containing tyloxapol and correspond

² "PE" in these designations signifies that the stability test was conducted using polyethylene containers.

to formulations A-04 and A-05, respectively, from Tables 2, 3-1 and 3-2 of the '290, '131, '813 and '606 patents:

Formulation Code	Bronuck	A-01	A-02
Designated code in Tables 2, 3-1 and 3-2 of the '290, '131, '813 and '606 patents	N/A	A-04 & Table 3-1	A-05 & Table 3-2
Bromfenac sodium hydrate	0.1 g	0.1 g	0.1 g
Boric acid	1.1 g	1.1 g	1.1 g
Borax	1.1 g	1.1 g	1.1 g
Benzalkonium chloride	0.005 g	0.005 g	0.005 g
Polysorbate 80	0.15 g	-	-
Tyloxapol	-	0.02 g	0.05 g
Polyvinylpyrrolidone	2.0 g	2.0 g	2.0 g
Disodium edetate	0.02 g	0.02 g	0.02 g
Sodium sulfite	0.2 g	-	-
Sodium hydroxide	q.s.	q.s.	q.s.
Distilled water	q.s.	q.s.	q.s.
Total Amount	100 mL ³	100 mL	100 mL
pH	8.3	8.19	8.20

13. The preservative efficacy of the bromfenac sodium formulation containing polysorbate 80 (identical to Bronuck) was tested as part of project P2002B116 at Senju. (Appendix B.) The preservative efficacy of the two bromfenac sodium formulations containing tyloxapol (formulations A-04 and A-05 from Tables 2, 3-1 and 3-2 of the '290, '131, '813 and '606 patents) was tested as part of project P2002B131 at Senju. (Appendix C.) The preservative efficacy tests

³ In Appendix B, the concentration of each component is disclosed as amount of component per 1 mL of the formulation. The value is converted to the amount of each component per 100 mL of the formulation.

were conducted at Senju using the European Pharmacopoeia (“EP”) standards, and I reviewed the results obtained from this testing. (Appendices B & C.)

14. According to the EP standards, the tested formulations were distributed in sterilized test tubes with stoppers. For inoculation of the organisms, suspensions containing 10^8 colony forming units (“CFU”)/mL were prepared for each bacterial species, and suspensions containing 10^7 CFU/mL were prepared for each fungal species. Each test formulation was inoculated in a separate test tube with a final concentration of 10^6 CFU/mL of bacteria and 10^5 CFU/mL of fungi. The test tubes were stored at 20 to 25 °C after inoculation. Samples were taken from each test tube after 6 hours, 24 hours, 1 week, 2 weeks, 3 weeks and 4 weeks. From each sample, 0.5 mL of the sample was diluted with 4.5 mL of sterilized isotonic sodium chloride solution. Ten-fold dilution was performed 1 to 3 times, and 1 mL of the diluted solution was placed onto a plate, and 15 to 20 mL of a culture medium was added to each plate. With respect to bacteria cultures, soybean-casein digest agar medium (SCD agar medium) containing inactivators (0.1% lecithin, 0.7% polysorbate 80) was used. With respect to fungi cultures, sabouraud’s glucose medium containing inactivators (0.1% lecithin, 0.7% polysorbate 80) was used. Cultures were kept under the following conditions, and the number of microorganisms was counted.

	Microorganism	Culture Condition
Bacteria	<i>Staphylococcus aureus</i> ATCC 6538	30–35 °C
	<i>Escherichia coli</i> ATCC 8739	
	<i>Pseudomonas aeruginosa</i> ATCC 9027	
Fungi	<i>Candida albicans</i> ATCC 10231	20–25 °C
	<i>Aspergillus niger</i> ATCC 16404	

15. The results obtained from these preservative efficacy tests are as follows (Appendices B & C):

Bronuck	Inoculum Count	Cell count (CFU/mL)					
		6 hours after inoculation	24 hours after inoculation	7 days after inoculation	14 days after inoculation	21 days after inoculation	28 days after inoculation
<i>S. aureus</i>	4.3×10^6	4.0×10^6	3.1×10^6	7.0×10^5	0	0	0
<i>E. coli</i>	6.9×10^6	2.5×10^3	0	0	0	0	0
<i>P. aeruginosa</i>	1.1×10^7	0	0	0	0	0	0
<i>C. albicans</i>	3.2×10^5	-	-	4.7×10^3	0	0	0
<i>A. niger</i>	1.1×10^5	-	-	6.0×10^3	3.0×10^3	0	0

Table 3-1	Cell count (CFU/mL)						
A-01 (A-04 in specification)	Inoculum Count	6 hours after inoculation	24 hours after inoculation	7 days after inoculation	14 days after inoculation	21 days after inoculation	28 days after inoculation
<i>S. aureus</i>	2.1×10^6	3.0×10^1	0	0	0	0	0
<i>E. coli</i>	6.5×10^6	0	0	0	0	0	0
<i>P. aeruginosa</i>	5.8×10^6	0	0	0	0	0	0
<i>C. albicans</i>	3.2×10^5	-	-	0	0	0	0
<i>A. niger</i>	1.8×10^5	-	-	0	0	0	0

Table 3-2	Cell count (CFU/mL)						
A-02 (A-04 in specification)	Inoculum Count	6 hours after inoculation	24 hours after inoculation	7 days after inoculation	14 days after inoculation	21 days after inoculation	28 days after inoculation
<i>S. aureus</i>	2.1×10^6	1.7×10^5	2.0×10^1	0	0	0	0
<i>E. coli</i>	6.5×10^6	0	0	0	0	0	0
<i>P. aeruginosa</i>	5.8×10^6	0	0	0	0	0	0
<i>C. albicans</i>	3.2×10^5	-	-	0	0	0	0
<i>A. niger</i>	1.8×10^5	-	-	0	0	0	0

16. The EP-A and EP-B standards used to compare the obtained results, at the time of testing, are the following:

European Pharmacopoeia Standards		
A standard	<p>Viabie cell counts of bacteria (<i>S. aureus</i>, <i>E. coli</i> & <i>P. aeruginosa</i>) 6 hours, 24 hours and 28 days after inoculation decrease to not more than 1/100, not more than 1/1000, and undetectable respectively.</p>	<p>Viabie cell counts of fungi (<i>C. albicans</i> & <i>A. niger</i>) 7 days after inoculation decreases to not more than 1/100, and the cell count levels off or decreases thereafter.</p>
B standard	<p>Viabie cell counts of bacteria (<i>S. aureus</i>, <i>E. coli</i> & <i>P. aeruginosa</i>) 24 hours and 7 days after inoculation decrease to not more than 1/10 and not more than 1/1000, and the cell count levels off or decreases thereafter.</p>	<p>Viabie cell counts of fungi (<i>C. albicans</i> & <i>A. niger</i>) 14 days after inoculation decreases to not more than 1/10, and the cell count levels off or decreases thereafter.</p>

17. Based on the EP-A and EP-B standards, the bromfenac sodium formulation containing polysorbate 80 did not pass either EP standard. The bromfenac sodium formulation containing tyloxapol and designated A-01 (corresponding to A-04 from the '290, '131, '813 and '606 patents) passed both standards. The bromfenac sodium formulation containing tyloxapol and designated A-02 (corresponding to A-05 from the '290, '131, '813 and '606 patents) passed the EP-B standard but not the EP-A standard.

E. The data in the specification and data disclosed in this declaration

18. Based on my involvement with the formulations of Tables 1, 2 and 3 of the '290, '131, '813 and '606 patents, I have personal knowledge about how the

test data disclosed in the '290, '131, '813 and '606 patents were generated. Based on my involvement with the remaining formulations and test data (beyond those disclosed in Tables 1, 2 and 3 of the '290, '131, '813 and '606 patents) provided in this declaration, including in Appendices A, B and C, I have personal knowledge about how such information was generated.

19. I hereby declare under penalty of perjury under the laws of the United States of America that the foregoing is true and correct, and that all statements made of my own knowledge are true and that all statements made on information and belief are believed to be true.

Date: 02/18/16

Shirou Sawa [signature]
Shirou Sawa

米国特許庁

特許庁審判部

LUPIN LTD. 及び
LUPIN PHARMACEUTICALS INC.
請求人

v.

千寿製薬株式会社
特許権者

事件 IPR2015-01099, IPR2015-01097, IPR2015-01105 & IPR2015-01100
特許 8,669,290, 8,754,131, 8,871,813 & 8,927,606

澤嗣郎の宣言書

1

私、澤嗣郎は、偽証罪の制裁の下に、以下のとおり宣言いたします。

I. 序文

1. 私は、成人しており、他にもこの宣言書を作成する能力に欠けることはありません。

2. 私は、米国特許番号 8,669,290 (「290 号特許」)、8,754,131 (「131 号特許」)、8,871,813 (「813 号特許」) 及び 8,927,606 (「606 号特許」) の筆頭発明者です。私は、290 号特許、131 号特許、813 号特許及び 606 号特許の明細書に記載されているデータを含む下記のデータがどのようにして生成されたかを証言する宣言書を提出するように依頼されました。

II. 経歴及び資格

3. 私は、日本国徳島県に所在する徳島大学を 1988 年に工学部化学工学科を卒業しました。私は、工学修士号化学工学専攻を日本国徳島県に所在する徳島大学から 1990 年に取得しました。

4. 私は、現在、千寿製薬株式会社(「千寿」)に雇用されており、1990 年から千寿に勤務しています。私は、千寿にて 1990 年から現在まで眼科用製剤に関わる研究者として働いています。

III. 290 号、131 号、813 号及び 606 号特許のデータを含むデータ

5. 私は、290号、131号、813号及び606号特許に報告されたブロムフェナクナトリウム(2-アミノ-3-(4-ブロモベンゾイル)フェニル酢酸ナトリウム)の製剤の、処方の実験例を含めた製剤化及び試験に関わりました。そのため、私は、下記に報告された製剤がどのようにして作られ、試験されたかの直接的な知識を持っています。

6. 添付書類 A、B 及び C の実験ノートが、試験された製剤及びそれによって得られた試験結果を記載しています。

A. 290号、131号、813号及び606号特許の第1表のデータを含む安定性
データ

7. 私は、290号、131号、813号及び606号特許の第1表に開示されたブロムフェナクナトリウム製剤の準備し、安定性を試験しました。下記の表は、特許の第1表のブロムフェナクナトリウム製剤だけでなく、他に異なった量のチロキサポールを含む製剤を含んでいます(添付書類 A)。表が示すように、製剤 A-20、A-21 及び A-27 は 290号、131号、813号及び606号特許の第1表の比較例 1、A-02 及び A-03 にそれぞれ対応しています。

8. 添付書類 A の実験ノートに反映されているように、これらブロムフェナクナトリウム製剤の安定性は製剤を pH7 に調整した後試験されました。これら製剤を使い、60°C で四週間実施されたものを含む、様々な時間、温度上昇下

での、加速安定性試験を実施した。残存ブロムフェナクナトリウムのパーセントは、下記の条件下に、高性能液体クロマトグラフィー(「HPLC」)法を用いて測定されました。

- 紫外吸光分光光度計で波長 266 nm
- カラム: Capcelpak カラム
- カラム温度: 25 °C
- 移動相: リン酸2水素アンモニウム 1.98 gを水 750 mLに溶解し、pH をリン酸を加えて 7.3 に調整した後、250 mLのアセトニトリルを加えた
- 流速: ブロムフェナクナトリウムの溶出時間が 18 分となるように調整
- 試料の注入量: 10 μ L

9. 各ブロムフェナクナトリウム製剤の 60°Cで四週間後に残っているブロムフェナクナトリウムのパーセントを下記表に記しました。この残存ブロムフェナクナトリウムのパーセントは水分蒸発量を補正したものであります。:

製剤コード	A-20	A-21	A-27	A-28	A-29
290号、131号、813号及び606号特許の第1表に表記のコード	比較例1	A-02	A-03	無	無
ブロムフェナクナトリウム水和物 ¹	0.1 g	0.1 g	0.1 g	0.1 g	0.1 g
ホウ酸	1.5 g	1.5 g	1.6 g	1.6 g	1.6 g
塩化ベンザルコニウム	0.005 g	0.005 g	0.005 g	0.005 g	0.005 g
ポリソルベート 80	0.17 g	-	-	-	-
チロキサポール	-	0.15 g	0.02 g	0.05 g	0.1 g
水酸化ナトリウム	適量	適量	適量	適量	適量
蒸留水	適量	適量	適量	適量	適量
全量	100 mL	100 mL	100 mL	100 mL	100 mL
pH	7	7	7	7	7
60 °C - 4 週	51.27%	73.81%	89.64%	85.96%	82.01%

B. 290号、131号、813号及び606号特許の第2表に開示されたデータを含む安定性データ

10. 私は、290号、131号、813号及び606号特許及び第2表に開示されたものを含む、下記のブロムフェナクナトリウム製剤を準備し、安定性を試験しました。下記表が示すように、製剤 A-01、A-02 及び A-03 は 290号、131号、813号及び606号特許の第2表の A-04、A-05 及び A-06 にそれぞれ対応しています。これらの製剤の安定性試験は、記載のとおり、おおよそ pH 8.15 に調

¹ 添付されている実験ノートには、私は「ブロムフェナクナトリウム水和物」のことを「ブロムフェナクナトリウム」と省略表現を用いた。

整し、60 °C 四週間実施されたものを含む、様々な時間、温度上昇下で行われました。60 °C で四週間実施後各製剤に残っているブロムフェナクナトリウムのパーセントは、上記 HPLC 法を用いて測定され、水分蒸発量を補正しました。データを下記表に記しました。(添付書類 C。)

製剤コード	A-01	A-02	A-03
290 号、131 号、813 号及び 606 号特許の第 2 表に表記 のコード	A-04	A-05	A-06
ブロムフェナクナトリウム 水和物	0.1 g	0.1 g	0.1 g
ホウ酸	1.1 g	1.1 g	1.1 g
ホウ砂	1.1 g	1.1 g	1.1 g
塩化ベンザルコニウム	0.005 g	0.005 g	0.005 g
チロキサポール	0.02 g	0.05 g	0.03 g
ポリビニルピロリドン	2.0 g	2.0 g	2.0 g
エデト酸ナトリウム	0.02 g	0.02 g	0.02 g
水酸化ナトリウム	適量	適量	適量
蒸留水	適量	適量	適量
全量	100 mL	100 mL	100 mL
pH	8.15	8.15	8.15
60 °C - 4 週	92.57%	90.93%	91.97%

C. ポリソルベート 80 を含むブロムフェナクナトリウム製剤及びチロキサポールを含むブロムフェナクナトリウム製剤の安定性データ

11. 私は、下記のとおり組成物、量を含む、以下のブロムフェナクナトリウム製剤を準備し、試験しました。これらのおおよそ pH 8.2 から 8.3 付近に調整した製剤の安定性試験は、記載のとおり、60 °C 四週間を含む、様々な時間、

温度上昇下で行われました。60 °C で四週間実施後各製剤に残っているブロムフェナクナトリウムのパーセントは、上記 HPLC 法を用いて測定され、水分蒸発量を補正しました。データを下記表に記しました。(添付書類 C。)

製剤コード	BF (PE) ² (ブロナック)	A-01 (PE)	A-03 (PE)
ブロムフェナクナトリウム水和物	0.1 g	0.1 g	0.1 g
ホウ酸	1.1 g	1.1 g	1.1 g
ホウ砂	1.1 g	1.1 g	1.1 g
塩化ベンザルコニウム	0.005 g	0.005 g	0.005 g
ポリソルベート 80	0.15 g	-	-
チロキサポール	-	0.02 g	0.03 g
ポリビニルピロリドン	2.0 g	2.0 g	2.0 g
エデト酸ナトリウム	0.02 g	0.02 g	0.02 g
亜硫酸ナトリウム	0.2 g	-	-
水酸化ナトリウム	適量	適量	適量
蒸留水	適量	適量	適量
全量	100 mL	100 mL	100 mL
pH	8.20	8.20	8.27
60 °C - 4 週	91.45%	93.61%	95.07%

D. ブロムフェナクナトリウム製剤の保存効力

12. 私は千寿で、プロジェクト P2002B116 及び P2002B131 の一環としてブロムフェナクナトリウム製剤の保存効力の試験に関わりました。私は、ポリソルベート 80 を含むブロムフェナクナトリウム製剤(ブロナックと同等)及びチロキ

² これら表記「PE」は安定性試験がポリエチレン容器を使って行われたことを意味します。

サポールを含むブロムフェナクナトリウム製剤を下記表に示されたように製剤化しました。(添付書類 B 及び C。)この表の製剤 A-01 及び A-02 は、チロキサポールを含むブロムフェナクナトリウム製剤であり、290 号、131 号、813 号及び 606 号特許第 2 表、3-1 と 3-2 表の A-04 及び A-05 にそれぞれ対応しています。

製剤コード	プロナック	A-01	A-02
290 号、131 号、813 号 及び 606 号特許の第 2、3-1 と 3-2 表に表記の コード	無	A-04 & 第 3-1 表	A-05 & 第 3-2 表
ブロムフェナクナトリ ウム水和物	0.1 g	0.1 g	0.1 g
ホウ酸	1.1 g	1.1 g	1.1 g
ホウ砂	1.1 g	1.1 g	1.1 g
塩化ベンザルコニウム	0.005 g	0.005 g	0.005 g
ポリソルベート 80	0.15 g	-	-
チロキサポール		0.02 g	0.05 g
ポリビニルピロリドン	2.0 g	2.0 g	2.0 g
エデト酸ナトリウム	0.02 g	0.02 g	0.02 g
亜硫酸ナトリウム	0.2 g	-	-
水酸化ナトリウム	適量	適量	適量
蒸留水	適量	適量	適量
全量	100 mL ³	100 mL	100 mL
pH	8.3	8.19	8.20

³ 添付書類 B では、各組成物の濃度を製剤 1 ミリリットルあたりの組成物の量で表記しています。この値を製剤 100 ミリリットル当たりの組成物の量に変換しています。

13. 試験コード P2002B116 の実験の一環でポリソルベート 80 を含むブ
ロムフェナクナトリウム製剤(ブロナックと同等)の保存効力の試験が千寿で実施
されました。(添付書類 B。)私は、試験コード P2002B131 の実験の一環でチロ
キサポールを含む2つのブロムフェナクナトリウム製剤(290号、131号、813号
及び606号特許の第2表、第3-1表と第3-2表の製剤 A-04と A-05)の保存効
力の試験実施を行わせました。(添付書類 C。)これらの製剤の保存効力試験
は、ヨーロッパ薬局方(「EP」)の基準を使用して千寿で行われ、私は、この試験
から得られた結果を検討しました。(添付書類 B 及び C。)

14. EP の基準によると、試験される製剤はストッパーのついた滅菌され
た試験管に分注されます。微生物の接種にあたり、各細菌種は 10^8 コロニー
形成単位(CFU)/mL の懸濁液、各真菌種は 10^7 CFU/mL の懸濁液が準備され
ました。各製剤を別々の試験管に、最終濃度が細菌は 10^6 CFU/mL、真菌は
 10^5 CFU/mL となるよう、それぞれ接種されました。接種後、試験管は 20 から
25 °C で保存されました。各試験管から、6 時間、24 時間、1 週間、2 週間、3 週
間及び 4 週間後にサンプルが採取されました。各サンプルから 0.5 mL のサンプ
ルが 4.5 mL の滅菌生理食塩液で希釈されました。10 倍希釈が 1 から 3 回行
われ、そして希釈溶液 1 mL がプレートに置かれました、更に 15 から 20 mL の
培地が各プレートに加えられました。細菌培養に関しては、不活化剤(0.1%レ

シチン、0.7%ポリソルベート 80)を含むソイビーン・カゼイン・ダイジェスト寒天培地(SCD 寒天培地)が使用されました。真菌培養に関しては、不活化剤(0.1%レシチン、0.7%ポリソルベート 80)を含むサブローブドウ糖培地が使用されました。培地は下記条件で保管されました、そして微生物の数が数えられました。

微生物		培養条件
細菌	<i>Staphylococcus aureus</i> ATCC 6538	30 - 35 °C
	<i>Escherichia coli</i> ATCC 8739	
	<i>Pseudomonas aeruginosa</i> ATCC 9027	
真菌	<i>Candida albicans</i> ATCC 10231	20 - 25 °C
	<i>Aspergillus niger</i> ATCC 16404	

15. これらの保存効力試験から得られた結果は下記のとおりです (添付書類 B 及び C):

プロナック	細胞数 (CFU/mL)						
	接種数	接種 6 時間後	接種 24 時間後	接種 7 日後	接種 14 日後	接種 21 日後	接種 28 日後
<i>S. aureus</i>	4.3 x 10 ⁶	4.0 x 10 ⁶	3.1 x 10 ⁶	7.0 x 10 ⁵	0	0	0
<i>E. coli</i>	6.9 x 10 ⁶	2.5 x 10 ³	0	0	0	0	0
<i>P. aeruginosa</i>	1.1 x 10 ⁷	0	0	0	0	0	0
<i>C. albicans</i>	3.2 x 10 ⁵	-	-	4.7 x 10 ³	0	0	0
<i>A. niger</i>	1.1 x 10 ⁵	-	-	6.0 x 10 ³	3.0 x 10 ³	0	0

表 3-1	細胞数 (CFU/mL)						
A-01 (明細書で A-04)	接種数	接種 6 時間後	接種 24 時間後	接種 7 日後	接種 14 日後	接種 21 日後	接種 28 日後
<i>S. aureus</i>	2.1×10^6	3.0×10^1	0	0	0	0	0
<i>E. coli</i>	6.5×10^6	0	0	0	0	0	0
<i>P. aeruginosa</i>	5.8×10^6	0	0	0	0	0	0
<i>C. albicans</i>	3.2×10^5	-	-	0	0	0	0
<i>A. niger</i>	1.8×10^5	-	-	0	0	0	0

表 3-2	細胞数 (CFU/mL)						
A-02 (明細書で A-04)	接種数	接種 6 時間後	接種 24 時間後	接種 7 日後	接種 14 日後	接種 21 日後	接種 28 日後
<i>S. aureus</i>	2.1×10^6	1.7×10^5	2.0×10^1	0	0	0	0
<i>E. coli</i>	6.5×10^6	0	0	0	0	0	0
<i>P. aeruginosa</i>	5.8×10^6	0	0	0	0	0	0
<i>C. albicans</i>	3.2×10^5	-	-	0	0	0	0
<i>A. niger</i>	1.8×10^5	-	-	0	0	0	0

16. 得られた結果を比較するために当時用いられた EP -A 及び EP- B 基準は以下のとおりでした。

ヨーロッパ薬局方基準		
基準 A	細菌(<i>S. aureus</i> , <i>E. coli</i> & <i>P. aeruginosa</i>)の生菌数が接種から6時間、24時間、及び28日後にそれぞれ1/100以下、1/1000以下、または検出不可能まで減少。	真菌(<i>C. albicans</i> & <i>A. niger</i>)の生菌数が接種から7日後、1/100以下に減少し、かつ、以後菌数が安定あるいは減少。
基準 B	細菌(<i>S. aureus</i> , <i>E. coli</i> & <i>P. aeruginosa</i>)の生菌数が接種から24時間、7日後にそれぞれ1/10以下、または1/1000以下に減少、かつ、以後菌数が安定あるいは減少。	真菌(<i>C. albicans</i> & <i>A. niger</i>)の生菌数が接種から14日後に1/10以下に減少し、かつ、以後菌数が安定あるいは減少。

17. EP-A 及び EP-B 基準に基づく、ポリソルベート 80 を含むブロムフェナクナトリウム製剤はどちらの EP 基準にも適合しませんでした。チロキサポールを含むブロムフェナクナトリウム製剤即ち A-01 と表記された製剤 (290 号、131 号、813 号及び 606 号特許の A-04 に該当) は両基準に適合しました。チロキサポールを含むブロムフェナクナトリウム製剤即ち A-02 と表記された製剤 (290 号、131 号、813 号及び 606 号特許の A-05 に該当) は EP-B 基準に適合しましたが、EP-A 基準に適合しませんでした。

E. 明細書のデータ及び本宣言書に開示されたデータ

18. 290号、131号、813号及び606号特許の第1、第2及び第3表の製剤への私の関与に基づき、私は、特許に記載された試験データがどのようにして生成されたかについて個人的な知識を有しています。添付書類A、B及びCを含めた本宣言書に示された製剤や試験データ(290号、131号、813号及び606号特許の第1、第2、及び第3表に記載されている製剤のみならず)への私の関与に基づき、私は、どのようにしてそのような情報が生成されたかの個人的な知識を有しています。

19. 私は、ここに、アメリカ合衆国の偽証罪の制裁の下に、上に述べたことが真実で正確であり、私自身の知識に基づきなされた供述は全て真実であり、情報と信念に基づきなされた供述は全て真実であると信じられることを宣言します。

日付: 16. 02. 18

澤 嗣郎

澤 嗣郎

APPENDIX A

CERTIFICATION OF TRANSLATION

The undersigned, Ryan Malcho, whose address is 2192 Queen St. E, No. 64, declares and states as follows:
Toronto, Canada

I am well acquainted with the English and Japanese languages; I have in the past translated numerous Japanese documents of legal and/or technical content into English.

I have been requested to translate into English the attached Japanese documents titled:

- Exhibit A_P2000B177 data relied on.pdf
- Exhibit B_P2002B116 data relied on.pdf
- Exhibit C_P2002B131 data relied on.pdf


To copies of these Japanese documents I therefore attach the English translations and my Certification of Translation.

I hereby certify that the English translations of the attached documents titled

- Exhibit A_P2000B177 data relied on.pdf
- Exhibit B_P2002B116 data relied on.pdf
- Exhibit C_P2002B131 data relied on.pdf

are, to the best of my knowledge and ability, accurate translations.

And I declare further that all statements made herein of my own knowledge are true, that all statements made on information and belief are believed to be true, and that false statements and the like are punishable by fine and imprisonment, or both, under Section 1001 of Title 18 of the United States Code.


Ryan Malcho

Nov 16, 2015
Date

Test Protocol

Name of test: Study of the Formulation of Bronuck Ophthalmic Solution at pH 7

Test code: P2000B177

Test system: None

Development code: AHR10282B

Test start date: 7 December 2000

Scheduled start date of test operations: 7 December 2000

Scheduled end date of test operations: 15 March 2000 [sic]

Scheduled test end date: 30 March 2001

Test facility: Kobe Creative Center, Senju Pharmaceutical Co., Ltd.

1-5-4 Murotani, Nishi-ku, Kobe-shi

(Division of work duties)

Study director: Shirou Sawa

Study personnel:

Test substance: Bromfenac sodium

Purpose: Bromfenac sodium is less soluble and unstable in the low pH range, so the pH (midpoint of the standard) of Bronuck Ophthalmic Solution is set to 8.3. The pH of tears is generally said to be around 7 to 7.4, and since the pH of Bronuck Ophthalmic Solution is believed to be near the upper limit used in ophthalmic solutions, a formulation at a lower pH is desired. Bromfenac sodium has an acetic acid group in its molecules, so its solubility increases at a pH of 6.5 and higher. Since the dissolution of Bronuck Ophthalmic Solution mostly occurs in association with the acetic acid group, control of the acetic acid group for solubilization and stabilization is believed to be important. Although the addition of counterions to control the acetic acid group has been considered, bromfenac sodium forms insoluble complexes due to the addition of quaternary ammonium salt and becomes cloudy. Thus, the purpose of this test is to use water-soluble aminosugars to study the solubilization and stabilization of bromfenac sodium, even if complexes are formed.

Test method:

1) Solubilization study

Add an excessive amount of bromfenac sodium to acetic acid (pH 3~6), phosphoric acid (pH 5~7), or boric acid (pH 7~9) buffer solution, add 0.1 to 1.0% of N-methylglucamine or glucosamine hydrochloride, and adjust the pH with hydrochloric acid. Filter this solution, and measure the concentration of bromfenac sodium in the filtrate by HPLC.

2) Stabilization study

Add N-methylglucamine or glucosamine hydrochloride to the following bromfenac sodium ophthalmic solutions, and adjust the pH to 7. In experiment 1), the N-methylglucamine or glucosamine hydrochloride has a pH of 6.5 and an amount in which 0.1% bromfenac sodium is dissolved. Fill this solution into colorless polypropylene containers, and store at 70°C, 60°C, and 40°C at 75% RH and at 25°C at 60% RH. Perform tests on the bromfenac sodium content, pH, external appearance, and foreign insoluble matter of the solution over time. Also, freeze and thaw the solution 10 times, and observe its external appearance and foreign insoluble matter.

	Rp-01	Rp-02	Rp-03	Rp-04
Bromfenac sodium	0.1g	0.1g	0.1g	0.1g
Boric acid	1.1g	—	1.1g	—
Borax	1.1g	—	1.1g	—
Sodium chloride	—	0.85g	—	0.85g
Sodium dihydrogen phosphate crystals	—	0.1g	—	0.1g
Polysorbate 80	0.15g	0.15g	0.15g	0.15g
Polysorbate 80	0.005g	0.005g	0.005g	0.005g
Benzalkonium chloride	—	—	2.0g	2.0g
Polyvinylpyrrolidone	—	—	0.2g	0.2g
Sodium bisulfite	—	—	0.02g	0.02g
Sodium edetate	q.s.	q.s.	q.s.	q.s.
N-methylglucamine	q.s.	q.s.	q.s.	q.s.
Glucosamine hydrochloride	q.s.	q.s.	q.s.	q.s.
Hydrochloric acid	q.s.	q.s.	q.s.	q.s.
Purified water				
Total amount	100mL	100mL	100mL	100mL
pH	7.0	7.0	7.0	7.0

3) HPLC conditions

Detector: Ultraviolet absorption photometer (wavelength: 266nm)

Column: Capcellpak AG-120

Column temperature: A constant temperature around 25°C

Mobile phase: Dissolve 1.98g of ammonium dihydrogen phosphate into 750mL of water, add phosphoric acid to adjust the pH to 7.3, and then mix in 250mL of acetonitrile.

Flow rate: Adjust so that the elution time of bromfenac sodium is about 18 minutes.

Sample injection amount: 10µL

Signature of study director

7 December 2000

Shirou Sawa [seal]

Signature of application development GM

8 December 2000

Akira Ohtori [seal]

Bronuck Ophthalmic Solution Stability Test
 Lot No. 01K131

Test code: P2000B177
 Tester: Shirou Sawa
 Test date: 02 April 2001

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Water Collect(%)	Initial	present	
STD	1	S2-01	2328333						
STD	2	S2-22	2321811						
STD	mean		2324072	1.0005					
A-26	60°C-2W	S2-02	2032330	0.8749	87.48	83.94	4.02	8.4304	8.2383
A-27	60°C-2W	S2-03	2214721	0.9534	96.82	93.03	3.72	8.4298	8.2521
A-28	60°C-2W	S2-04	2158136	0.9282	93.58	90.24	3.55	8.5538	8.3800
A-29	60°C-2W	S2-05	2217340	0.9546	92.39	89.22	3.43	8.5249	8.3580
A-30	60°C-2W	S2-06	2142178	0.9222	90.88	87.84	3.34	8.5115	8.3490
A-31	60°C-2W	S2-07	2041719	0.8789	86.55	83.53	3.49	8.5492	8.3785
A-32	60°C-2W	S2-08	1930369	0.8310	78.98	72.11	6.70	8.4060	8.2309
A-33	60°C-2W	S2-09	2070437	0.8913	88.93	85.67	3.67	8.4759	8.2989
A-34	60°C-2W	S2-10	2010017	0.8653	86.25	82.90	3.88	8.4139	8.2293
A-35	60°C-2W	S2-11	1990944	0.8571	84.78	81.68	3.67	8.5592	8.3792
A-26	50°C-4W	S2-12	1918768	0.8280	82.57	80.04	3.08	8.4391	8.2927
A-27	50°C-4W	S2-13	2178249	0.9389	94.94	91.82	3.50	8.4117	8.2453
A-28	50°C-4W	S2-14	2105338	0.9083	91.35	88.33	3.31	8.5416	8.3789
A-29	50°C-4W	S2-15	2137432	0.9202	89.06	86.05	3.38	8.4608	8.2981
A-30	50°C-4W	S2-16	2043998	0.8789	88.72	83.74	3.44	8.4297	8.2654
A-31	50°C-4W	S2-17	1933544	0.8324	81.97	79.44	3.09	8.5842	8.4128
A-32	50°C-4W	S2-18	1692208	0.7285	69.24	66.88	3.41	8.3947	8.2328
A-33	50°C-4W	S2-19	1942783	0.8384	83.45	80.85	3.12	8.4713	8.3210
A-34	50°C-4W	S2-20	1922369	0.8276	82.50	79.98	3.08	8.4168	8.2699
A-35	50°C-4W	S2-21	1927309	0.8297	82.08	79.45	3.20	8.5848	8.4286

*Re-edited in order to document the data necessary for calculation.
 Shirou Sawa, 6 May 2005*

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2000B177	Test date	02 April 2001		
Test item					Tester	Shirou Sawa	
Lot No. 01K131							
STD AHR10282B 0.02001g + MP → 20mL				2-Apr-2001		10:24:01	
Above solution 2mL + MP → 20mL				001:		0.02001 g	
					Turbidity	Foreign insoluble matter	Color
A26	60°C-2W	7.05	8.4304	8.2383	+	±	Dark yellow
A27		7.10	8.4298	8.2521	±	+ (r)	Yellow
A28		7.08	8.5538	8.3800	+	+ (r)	↓
A29		7.06	8.5249	8.3580	+	+ (r)	↓
A30		7.09	8.5115	8.3490	+	+ (r)	↓
A31		7.07	8.5492	8.3785	+	+ (r)	↓
A32		7.09	8.4060	8.2309	+	++ (r)	Dark yellow
A33		7.07	8.4759	8.2989	+	+ (r)	↓
A34		7.11	8.4139	8.2292	+	+ (r)	↓
A35		7.08	8.5592	8.3792	+	+ (r)	↓
A26	50°C-4W	7.06	8.4391	8.2927	+	±	↓
A27		7.11	8.4117	8.2453	+	++ (r)	Yellow
A28		7.10	8.5416	8.3799	+	+ (r)	↓
A29		7.08	8.4606	8.2981	+	+ (r)	↓
A30		7.09	8.4297	8.2654	+	+ (r)	↓
A31		7.06	8.5642	8.4126	+	+ (r)	↓
A32		7.10	8.3947	8.2328	++	++ (r)	Dark yellow
A33		7.11	8.4713	8.3210	+	+	Yellow
A34		7.12	8.4168	8.2699	+	++ (r)	Dark yellow
A35		7.14	8.5846	8.4266	+	+ (r)	Dark yellow

4/02 17:49
NO. 92 PH 7.05
24.1°C

4/02 17:51
NO. 93 PH 7.10
24.0°C

4/02 17:53
NO. 94 PH 7.08
24.5°C

4/02 17:55
NO. 95 PH 7.25
24.1°C

4/02 17:58
NO. 96 PH 7.03
24.1°C

4/02 17:59
NO. 97 PH 7.37
24.0°C

4/02 18:00
NO. 98 PH 7.37
23.9°C

4/02 18:01
NO. 99 PH 7.09
25.9°C

4/02 18:02
NO. 1 PH 7.07
23.9°C

4/02 18:04
NO. 2 PH 7.11
23.9°C

4/02 18:06
NO. 3 PH 7.09
23.6°C

4/02 18:07
NO. 4 PH 7.06
23.9°C

4/02 18:12
NO. 5 PH 7.11
24.2°C

4/02 18:14
NO. 6 PH 7.18
23.9°C

4/02 18:15
NO. 7 PH 7.08
23.8°C

4/02 18:16
NO. 8 PH 7.09
23.6°C

4/02 18:17
NO. 9 PH 7.05
23.5°C

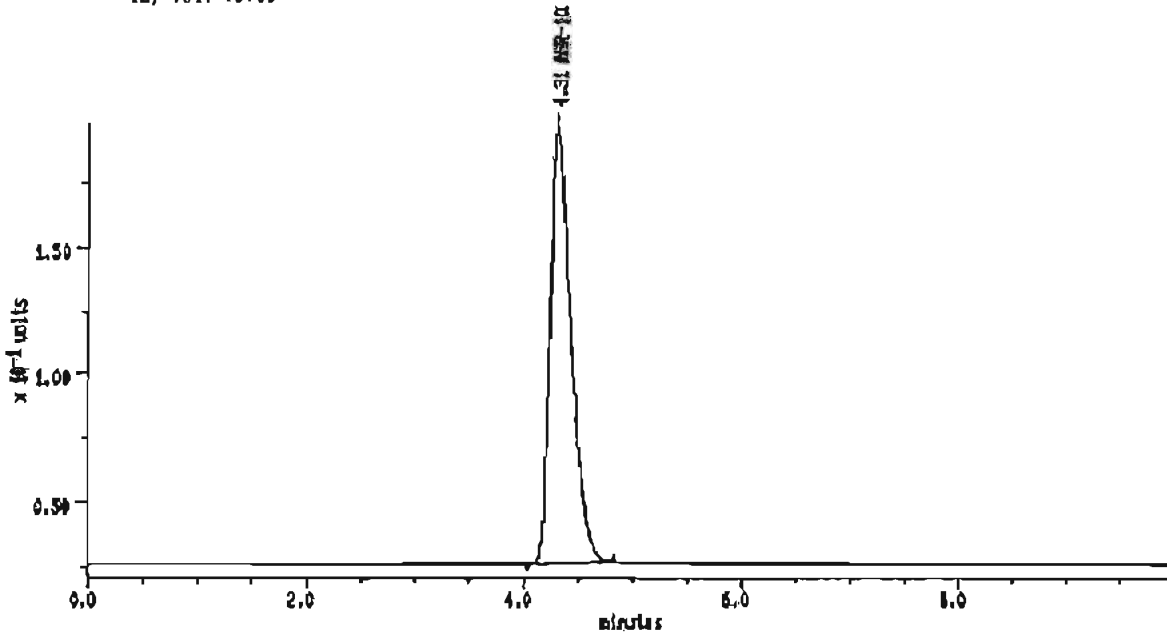
4/02 18:19
NO. 10 PH 7.10
23.7°C

4/02 18:21
NO. 11 PH 7.12
23.7°C

4/02 18:23
NO. 12 PH 7.11
23.7°C

4/02 18:26
NO. 13 PH 7.14
24.2°C

Sample: STD1 Channel: detector 1 Filename: 82-01 Chart Speed: Full Size
 Acquired: 02-APR-1981 20:31 Method: B:VAHRYIKI3V00-2R Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 20:4147

SAMPLE1: STD1

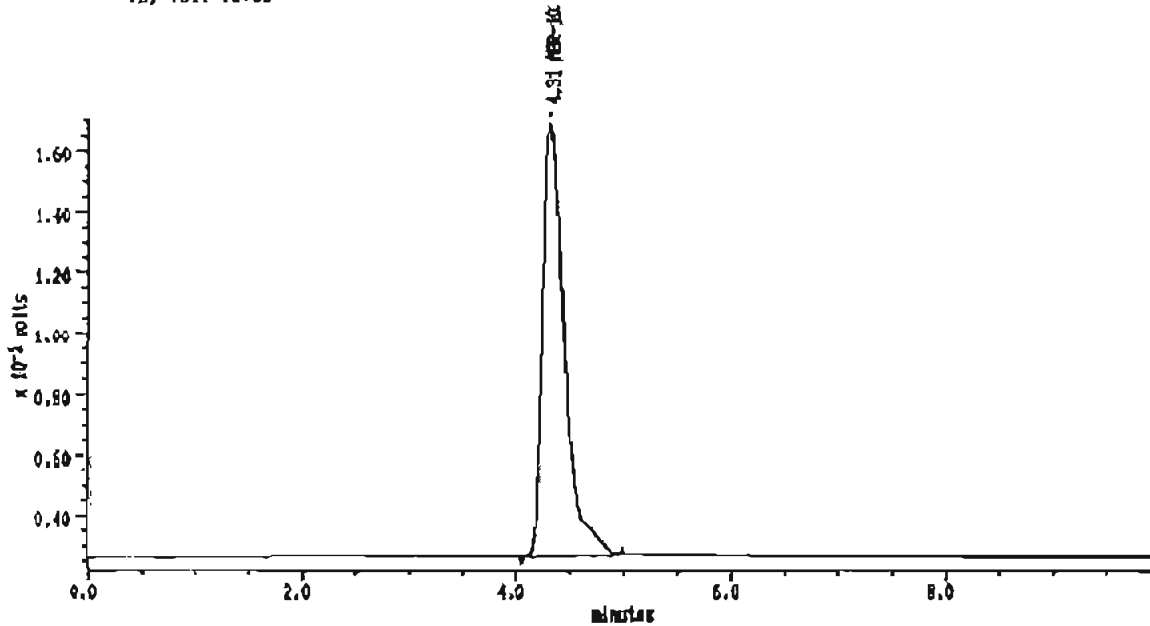
#1 In Method: **AHR-10282B**
 Acquired: 2-APR-2001 20:31
 Rate: 2.0 points/min
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: 82-01
 Injct: 20
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.306	SB	2320332	170546	100.00	AHR-10282B
TOTAL			2320332	170546		

Sample: A28 80°C-20 Channel: detector 1 Filename: S2-02 Chart Speed: Full Size
 Acquired: 02-APR-10 20:42 Method: B:VAHRNIK19Y00-20 Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 20:13:06

SAMPLE: A28 80°C-20

#2 In Method: AHR-10282B

Acquired: 2-APR-2001 20:42

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-02

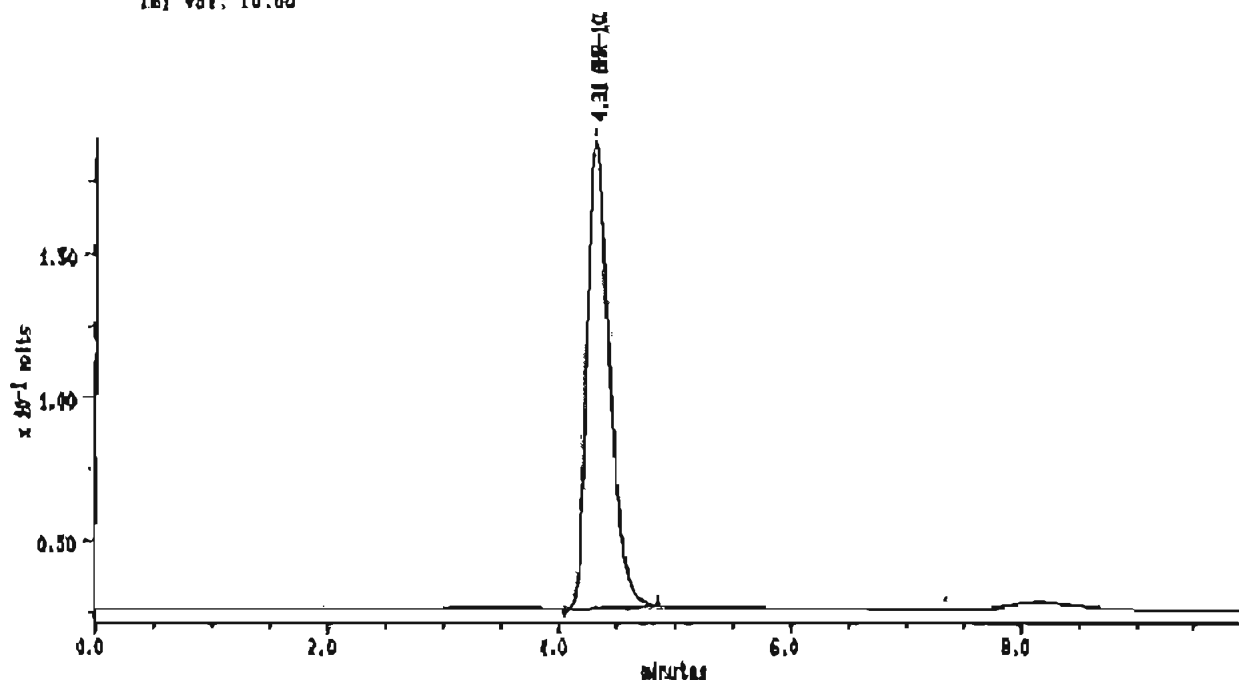
Index: 27

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time minutes	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	BO	2032330	142218	100.00	AHR-10282B
TOTAL			2032330	142218		

Sampler: A27 80°C-2W Channel: detector 1 Filenamer: 92-03 Chart Speed: Full Size
 Acquired: 02-APR-01 20:53 Method: BIVASIRV/K13V66-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 21:04:19

SAMPLE: A27 80°C-2W

#3 in Method: AHR-10282B

Acquired: 2-APR-2001 20:53

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filenamer: 92-03

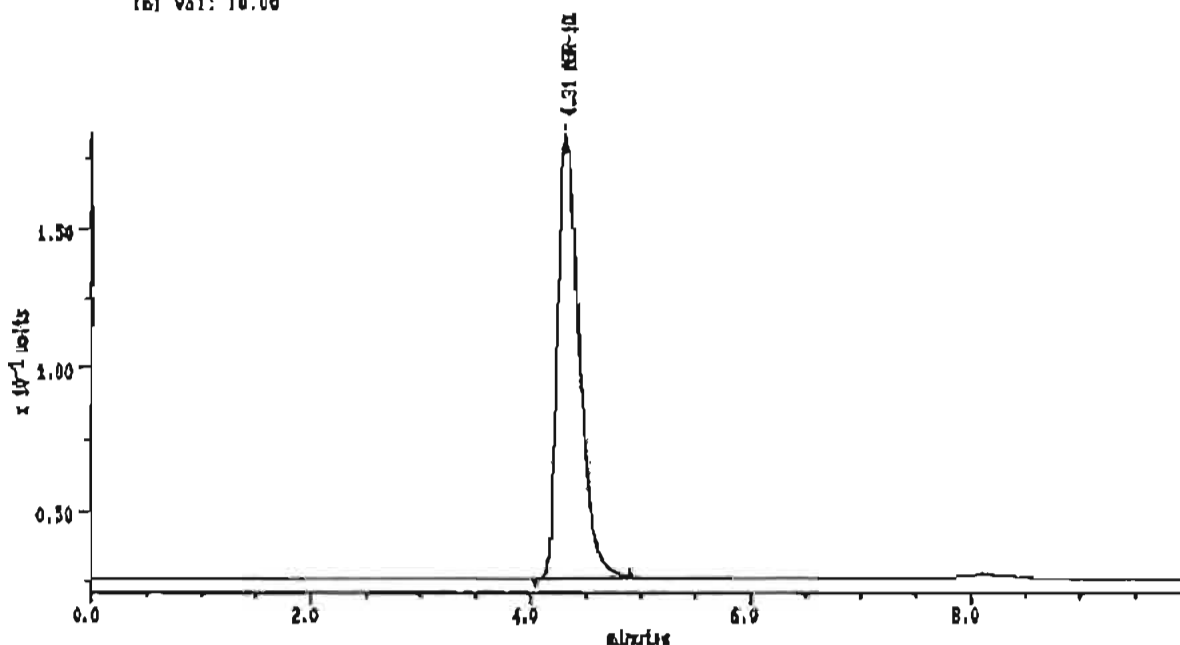
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	BB	2214721	101618	100.00	AHR-10282B
TOTAL			2214721	101618		

Sample: A28 80°C-2M Channel: detector 1 Filename: 52-04 Chart Speed: Full Size
 Acquired: 02-APR-101 21:05 Method: B:VAHRV1K13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 21:16:39

SAMPLE: A28 80°C-2M

#1 in Method: AHR-10282B

Acquired: 2-APR-2001 21:05

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: 52-04

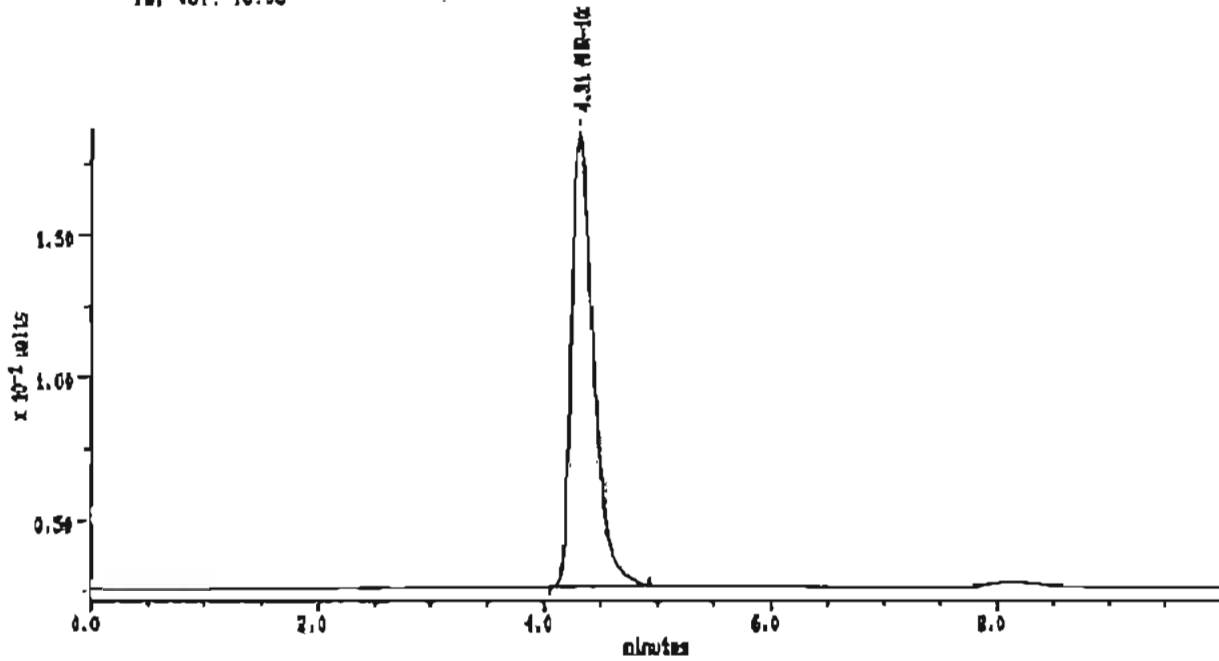
Index: 29

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	BD	2180136	180277	100.00	AHR-10282B
TOTAL			2180136	180277		

Sample: A28 80°C-2W Channel: detector 1 Filename: 52-05 Chart Speed: Full Size
 Acquired: 02-APR-01 21:10 Method: 8:VARIK10Y60-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 21:28:02

SAMPLE: A28 80°C-2W

#6 In Method: AHR-10282B

Acquired: 2-APR-2001 21:10

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: 52-05

Index: 30

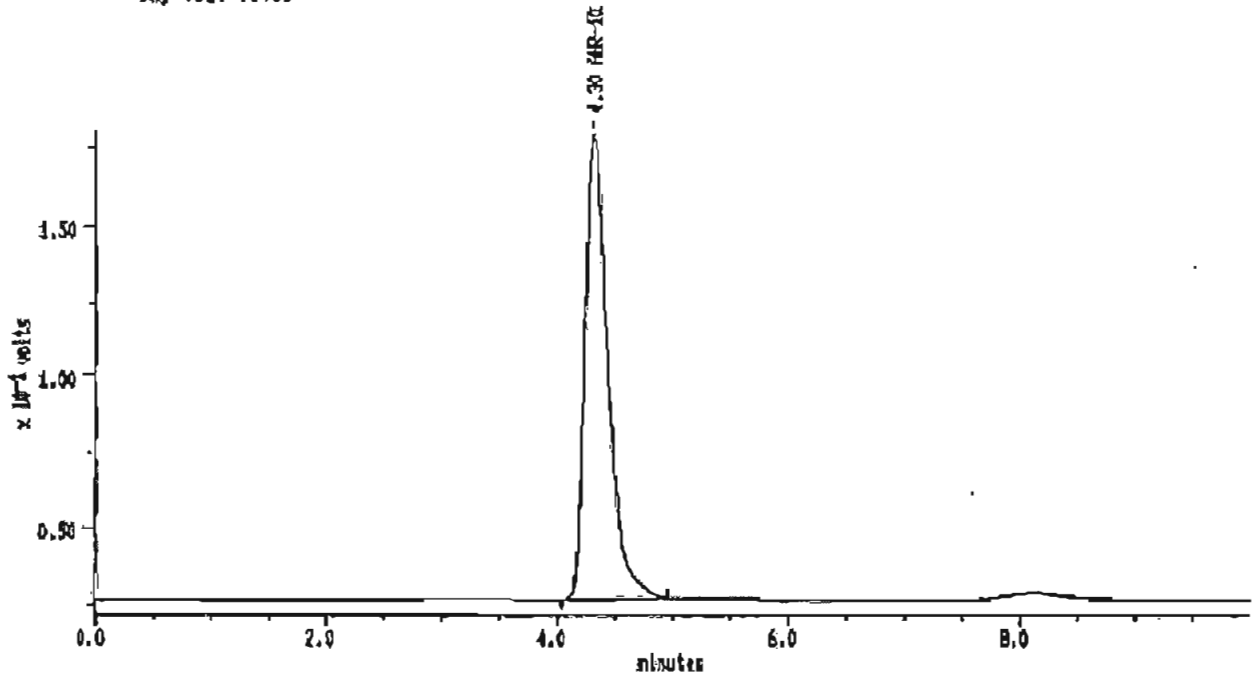
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	SS	2217340	169516	100.00	AHR-10282B
TOTAL			2217340	169516		

7

Sample: A20 B0-C-2W Channel: detector 1 Filename: S2-06 Chart Speed: Full Size
 Acquired: 01-APR-10 21:27 Method: B:VAFRYIK13V8D-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 21:38:10

SAMPLE: A20 B0-C-2W

#B In Method: AHR-10282B

Acquired: 2-APR-2001 21:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UMEM

Instrument: Instrument 1

Filename: S2-06

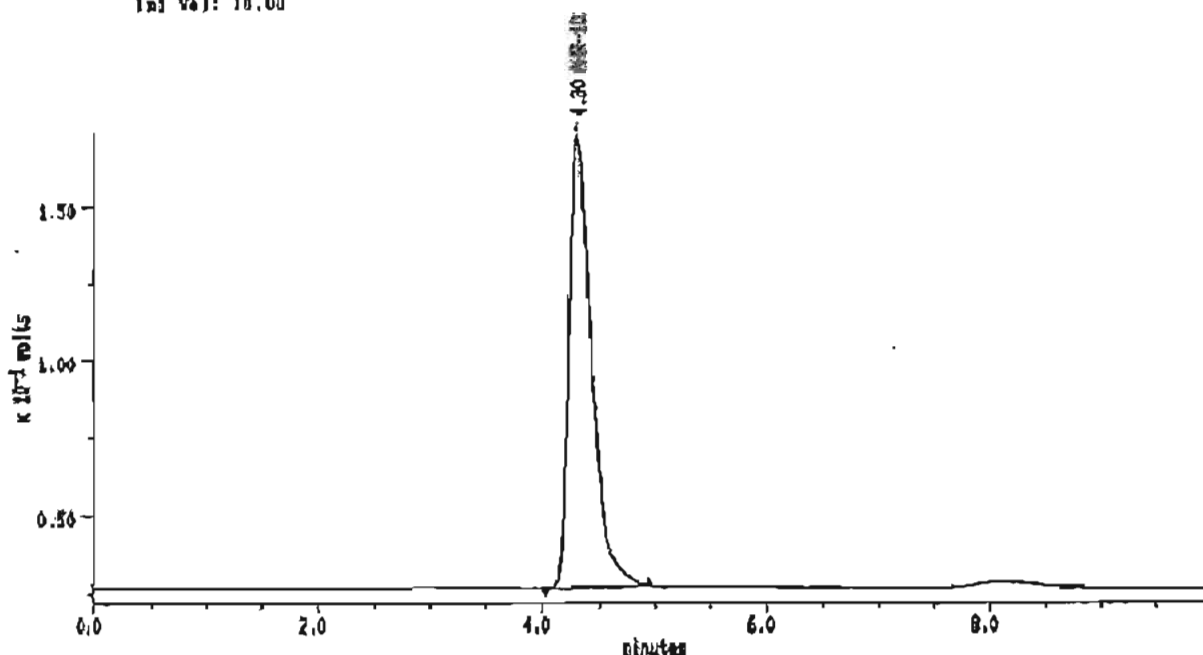
Index: 31

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	2142170	162031	100.00	AHR-10282B
TOTAL			2142170	162031		

Sample: A31 80°C-2W Channel: detector 1 Filename: 52-07 Chart Speed: Full Size
 Acquired: 02-APR-2001 21:38 Method: B:TAJIRVIK13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 21:49:28

SAMPLE: A31 80°C-2W

#7 In Method: AHR-10282B

Acquired: 2-APR-2001 21:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

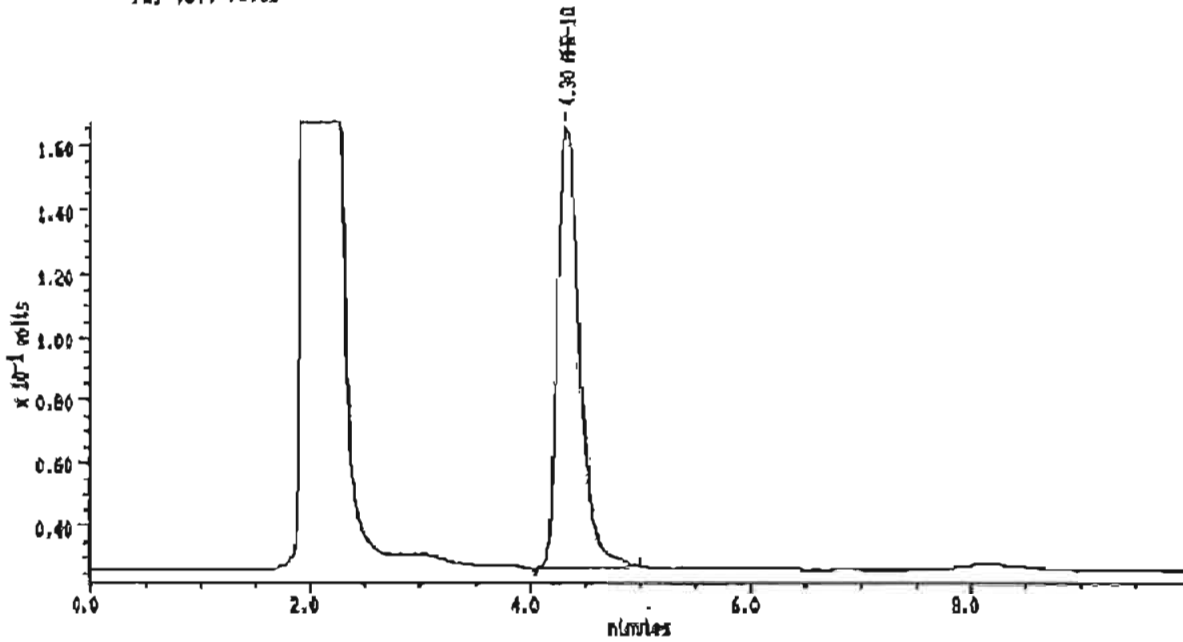
Filename: 52-07

Index: 32

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BP	2041718	148531	100.00	AHR-10282B
TOTAL			2041718	148531		



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 22:08:49

SAMPLE: A32 80°C-2W

File Method: AHR-10282B

Acquired: 2-APR-2001 21:50

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: 52-08

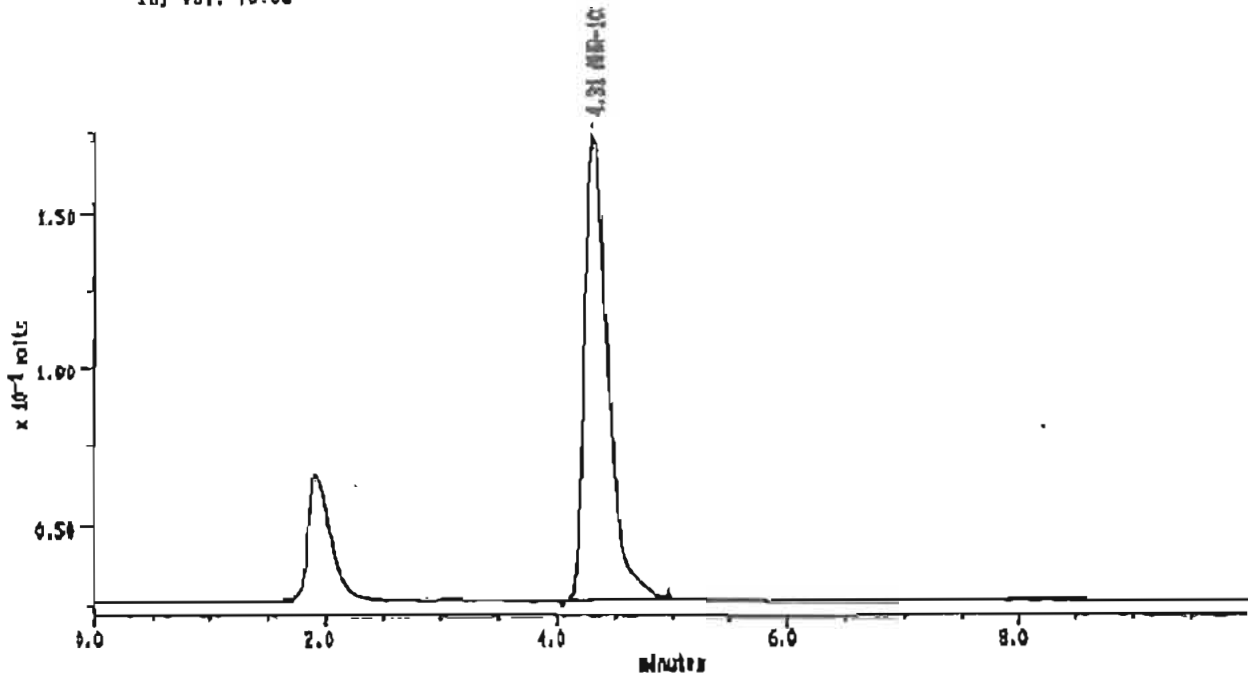
Index: 33

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	1930389	133435	100.00	AHR-10282B
TOTAL			1930389	133435		

Sample: A33 80°C-2W Channel: detector 1 Filename: S2-08 Chart Speed: Full Size
 Acquired: 02-APR-10 22:01 Method: 8:VAHRY\K10Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 22:11:59

SAMPLE: A33 80°C-2W

#9 In Method: AHR-10282B

Acquired: 2-APR-2001 22:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: [instrument 1]

Filename: S2-08

Index: 34

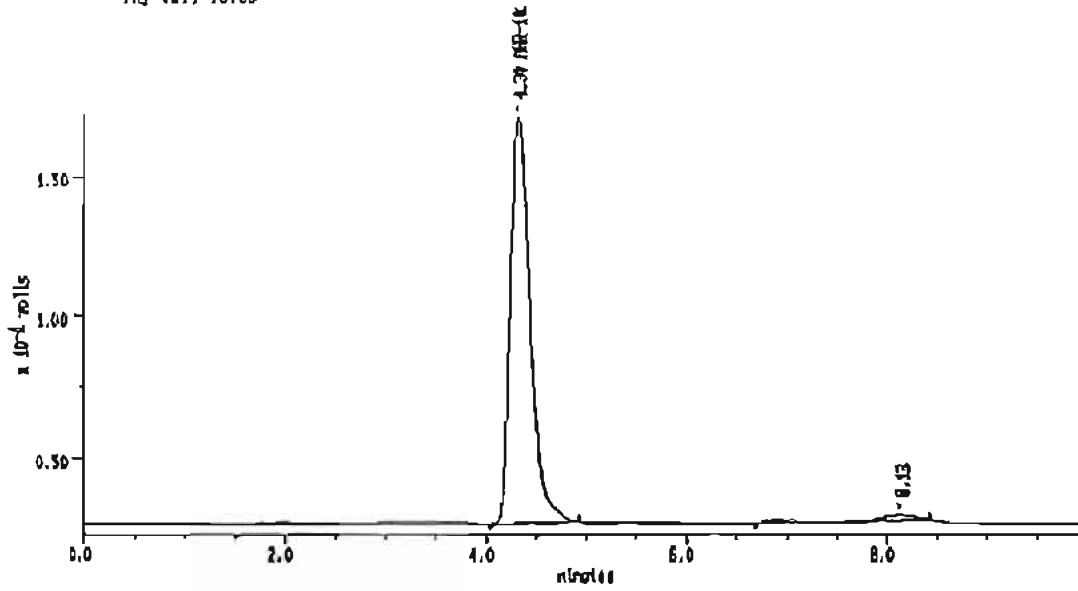
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	B0	2070437	147500	100.00	AHR-10282B
TOTAL			2070437	147500		

11

Sample: A34 60°C-2V Channel: detector 1 Filename: S2-10 Chart Speed: Full Size
 Acquired: 02-APR-2001 22:12 Method: B:VAIRYIK13780-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 828 Custom Report

Printed: 2-APR-2001 22:23:10

SAMPLE: A34 60°C-2V

#10 In Method: AHR-10282B

Acquired: 2-APR-2001 22:12

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNID

Instrument: Instrument 1

Filename: S2-10

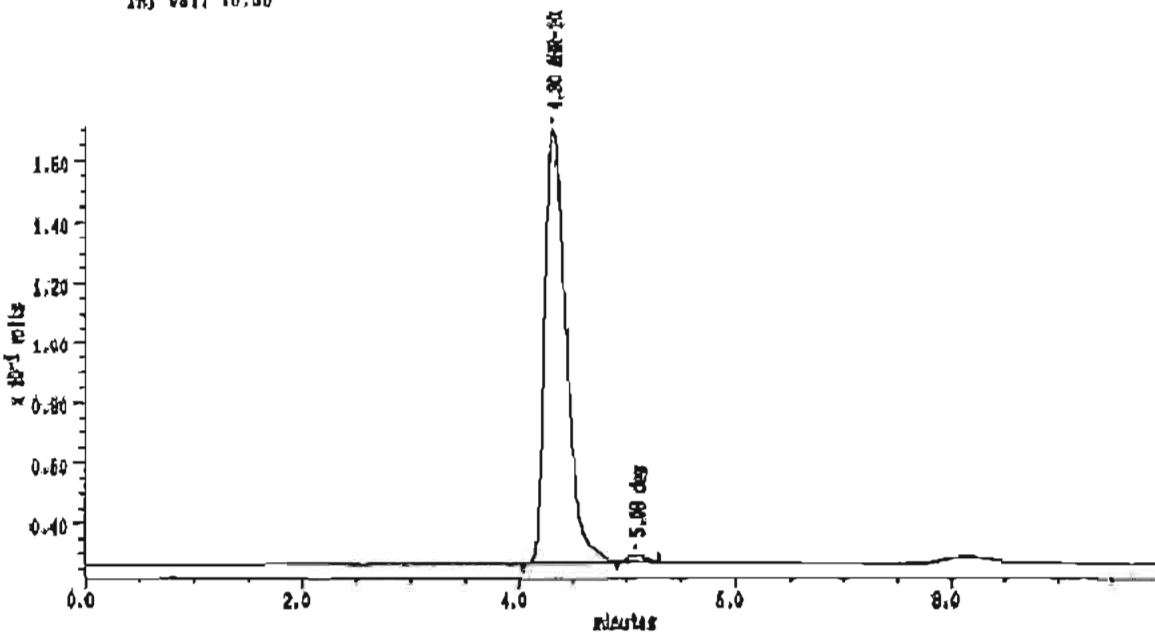
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	SB	2010017	145053	100.00	AHR-10282B
TOTAL			2010017	145053		

Sample: A36 60°C-24 Channel: detector 1 Filename: S2-11 Chart Speed: Full Size
 Acquired: 02-APR-01 22:24 Method: B:WARRVIX1BY60-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 22:34:52

SAMPLE: A36 60°C-24

#11 in Method: AHR-10282B

Acquired: 2-APR-2001 22:24

Rate: 2.0 ml/min/deg

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: S2-11

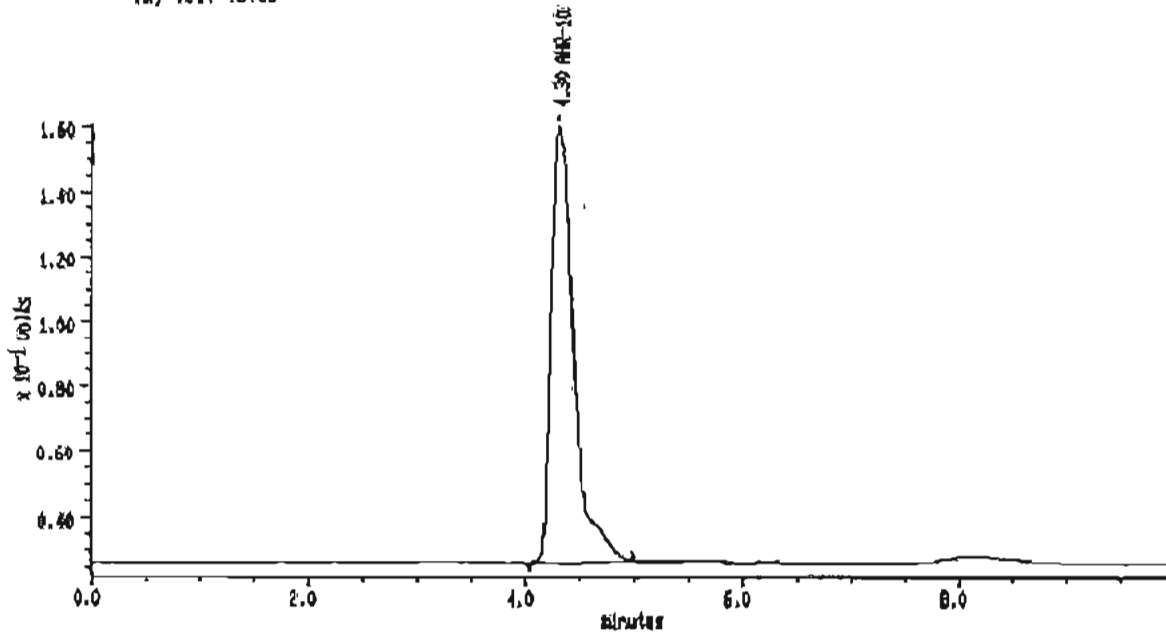
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	1890944	148218	98.91	AHR-10282B
2	5.076	SS	21896	1879	1.09	666
TOTAL			2012840	148080		

Sample: A26 50°C-4W Channel: detector 1 Filename: S2-12 Chart Speed: Full Size
 Acquired: 02-APR-01 22:26 Method: R:\AHR\WIKI\SY80-26 Operator: S.S
 Inj Volt: 10.00



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MAXIMA 828 Custom Report

Printed: 2-APR-2001 22:45:40

SAMPLE: A26 50°C-4W

#12 In Method: AHR-10282B

Acquired: 2-APR-2001 22:26

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: S2-12

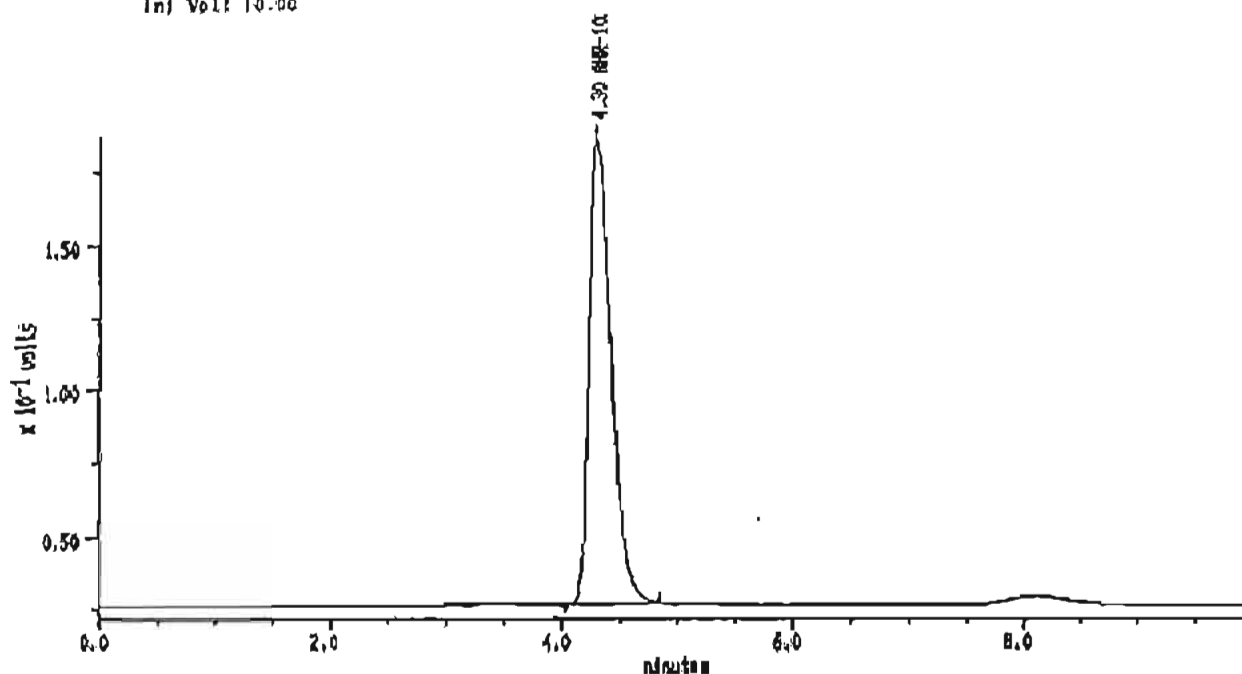
Index: 37

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	SS	1918700	133306	100.00	AHR-10282B
TOTAL			1918700	133306		

Sample: A27 50°C-4W Channel: detector 1 Filename: S2-13 Chart Speed: Full Size
 Acquired: 02-APR-01 22:46 Method: B:VAHRTIK13780-2M Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 22:07:08

SAMPLE: A27 50°C-4W

File Method: AHR-10282B

Acquired: 2-APR-2001 22:40

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNIX

Instrument: Instrument 1

Filename: S2-13

Index: 38

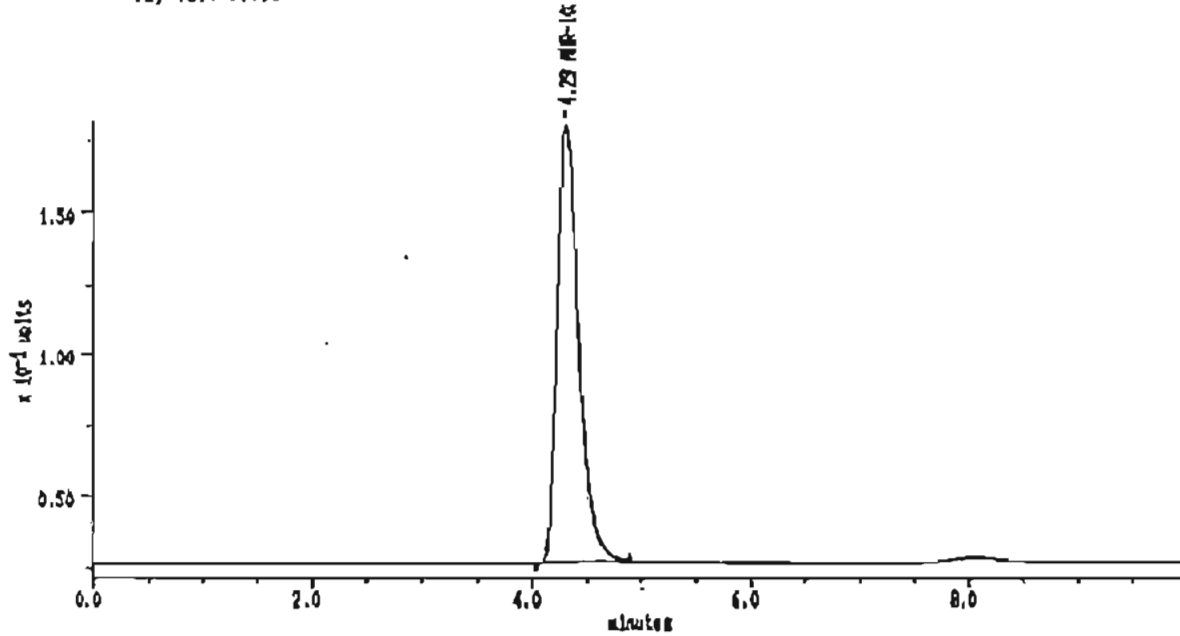
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	219249	159480	100.00	AHR-10282B
TOTAL			219249	159480		

15

Sample: A2B 50°C-4W Channel: detector 1 Filename: S2-14 Chart Speed: Full Size
 Acquired: 02-APR-10 22:57 Method: BINARY13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 23:08:23

SAMPLE: A2B 50°C-4W

File Method: AHR-10282B

Acquired: 2-APR-2001 22:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-14

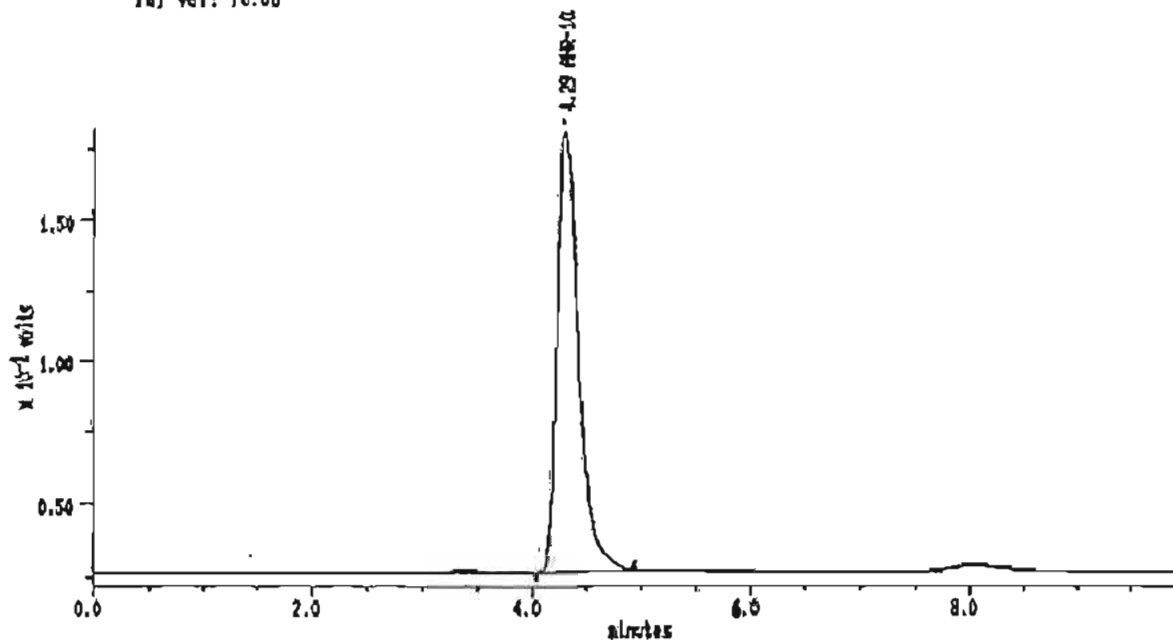
Injct: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	SB	2105398	163988	100.00	AHR-10282B
TOTAL			2105398	163988		

Sample: A29 50°C-4W Channel: detector 1 Filenama: S2-16 Chart Speed: Full Size
 Acquired: 02-APR-2001 23:09 Method: 8:YAMRVIK13Y80-2V Operator: S.S.
 Inj Vol: 10.00



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MAXIMA 828 Custom Report

Printed: 2-APR-2001 23:18:39

SAMPLE: A29 50°C-4W

File Method: **AHR-10282B**

Acquired: 2-APR-2001 23:09

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S.

Type: LMKH

Instrument: Instrument 1

Filenam: S2-16

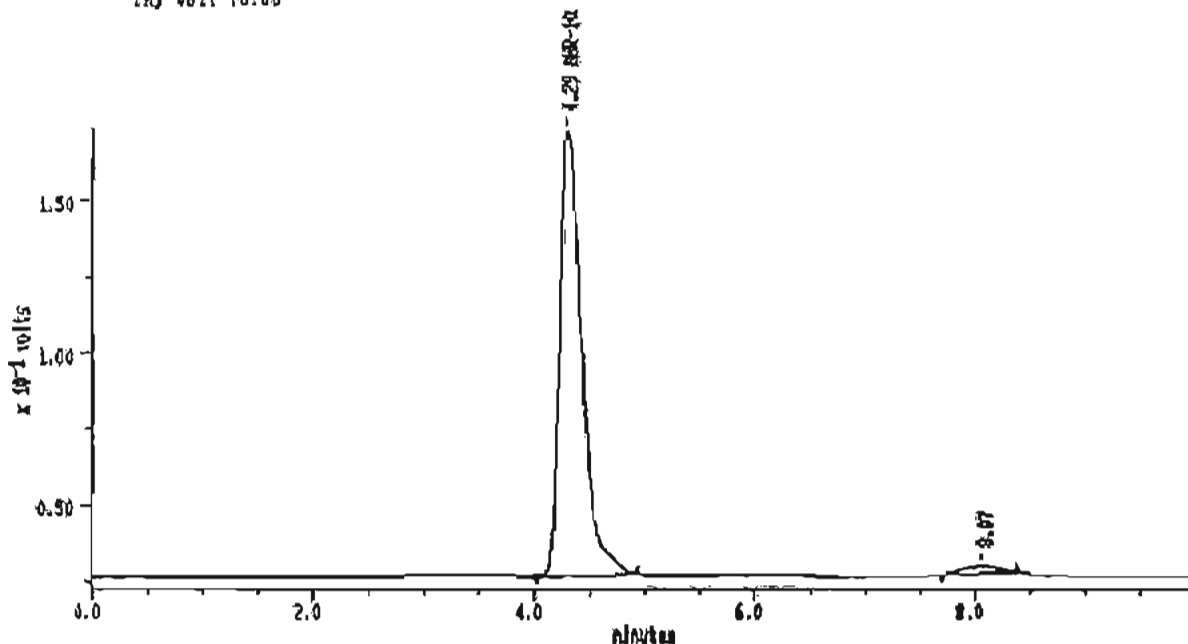
Index: 40

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BB	2137422	164351	100.00	AHR-10282B
TOTAL			2137432	164351		

Sample: A30 50°C-4W Channel: detector 1 Filename: 52-10 Chart Speed: Full Size
 Acquired: 02-APR-101 23:20 Method: B:7AURVIX13V60-2W Operator: S.S
 [n] Volt: 10.00



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MAXIMA 825 Custom Report

Printed: 3-APR-2001 23:58:50

SAMPLE: A30 50°C-4W

#18 in Method: AHR-10282B

Acquired: 3-APR-2001 23:20

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: QMOR

Instrument: Instrument 1

Filename: 52-10

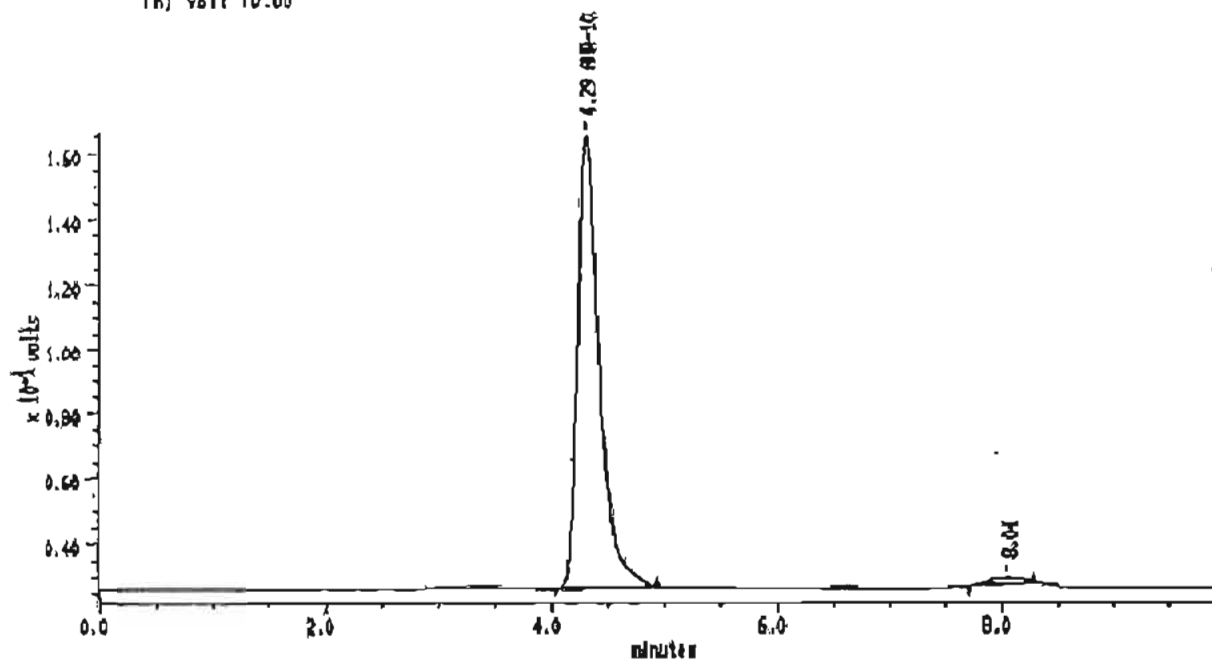
Index: 41

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.282	SD	204388	145010	100.00	AHR-10282B
TOTAL			204388	145010		

Sample: A31 50°C-4W Channel: detector 1 Filename: S2-17 Chart Speed: Full Size
 Acquired: 02-APR-10 23:31 Method: B:YANRVIRI3760-2W Operator: S.S
 Inj Volt: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 23:42:13

SAMPLE: A31 50°C-4W

#17 In Method: AHR-10282B

Acquired: 2-APR-2001 23:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: S2-17

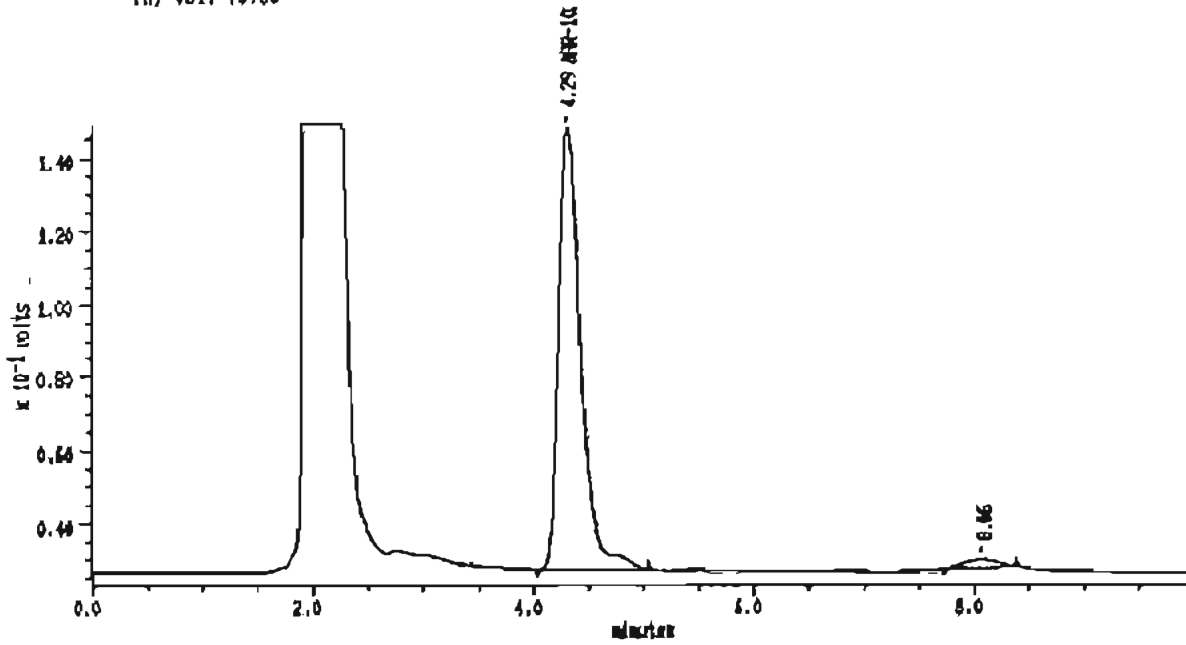
Index: 42

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.292	DB	1933544	138348	100.00	AHR-10282B
TOTAL			1933544	138348		

Sample: A32 50°C-4W Channel: detector 1 File Name: S2-18 Chart Speed: Full Size
 Acquired: 02-APR-10 23:43 Method: B:VAMPY\K13Y80-2# Operator: S.B
 (n) Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 2-APR-2001 23:52:00

SAMPLE: A32 50°C-4W

#10 in Method: AHR-10282B

Acquired: 2-APR-2001 23:43

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.B

Type: UNKN

Instrument: Instrument 1

File Name: S2-18

Index: 43

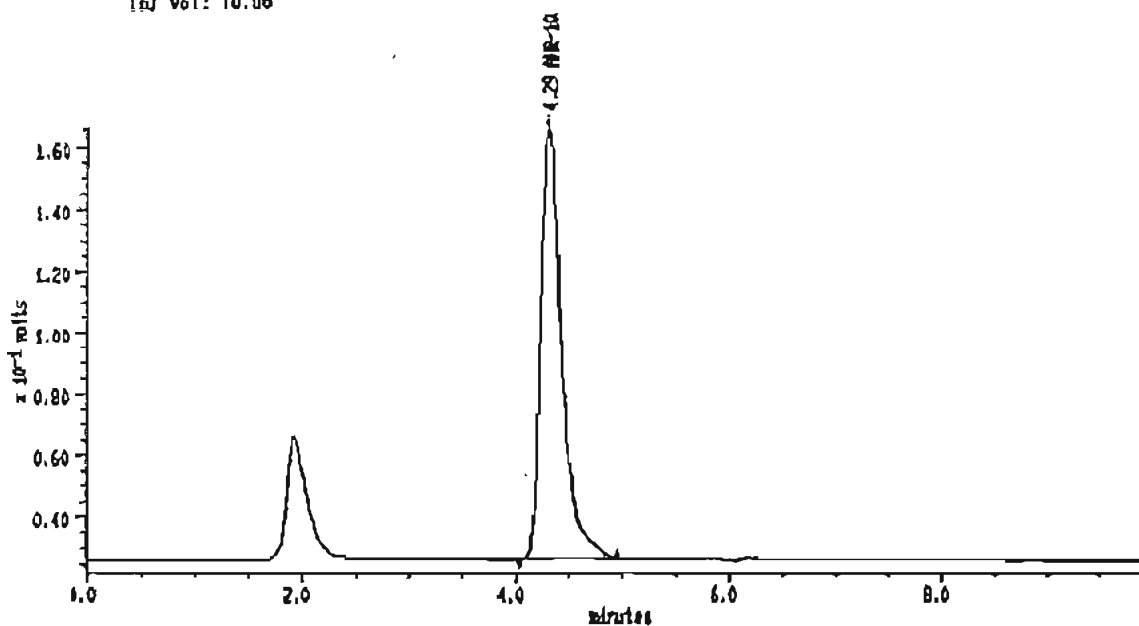
Injection Volume: 10.0

Amount: 0.000

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BB	1692208	120875	100.00	AHR-10282B
TOTAL			1692208	120875		

Sample: A37 50°C-40 Channel: detector 1 Filename: 52-19 Chart Speed: Full Size
 Acquired: 02-APR-01 23:54 Method: B:VAMRTX13Y60-20 Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 3-APR-2001 8:04:07

SAMPLE: A37 50°C-40

#19 In Method: AHR-10282B

Acquired: 2-APR-2001 23:54

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UVM

Instrument: Instrument 1

Filename: 52-19

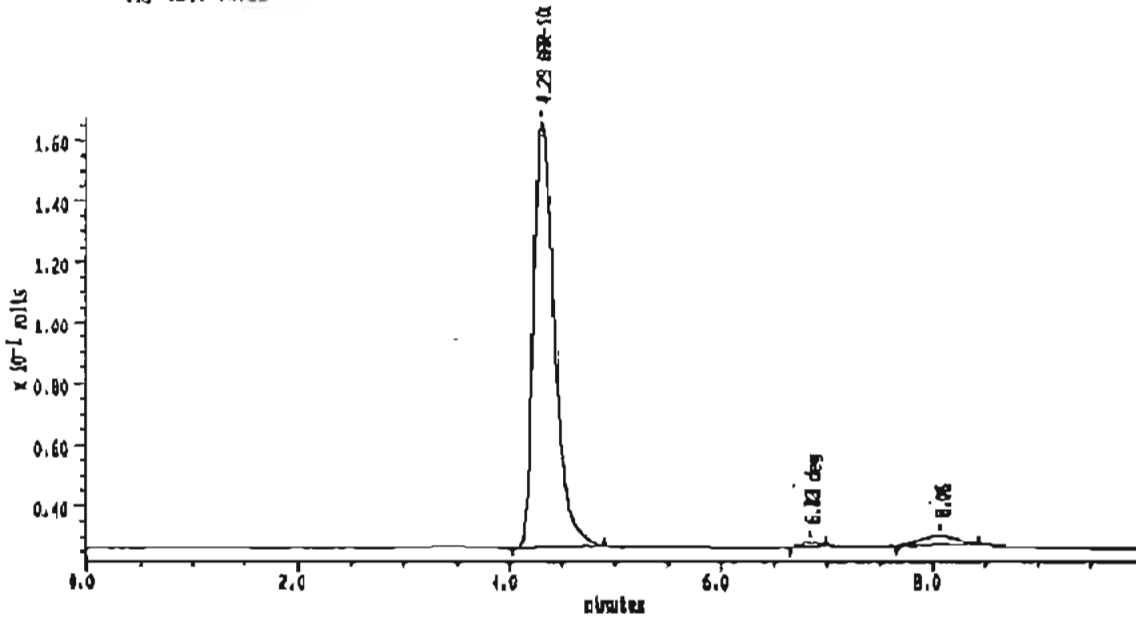
Index: 44

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.282	BB	1842703	156303	100.00	AHR-10282B
TOTAL			1842703	156303		

Sample: A34 50°C-4M Channel: detector 1 Filename: S2-20 Chart Speed: Full Size
 Acquired: 03-APR-2001 0:05 Method: B:VAHRVYIKI3Y4D-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (d) 1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 3-APR-2001 0:16:04

SAMPLE: A34 50°C-4M

M20 In Method: AHR-10282B

Acquired: 3-APR-2001 0:05

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKW

Instrument: Instrument 1

Filename: S2-20

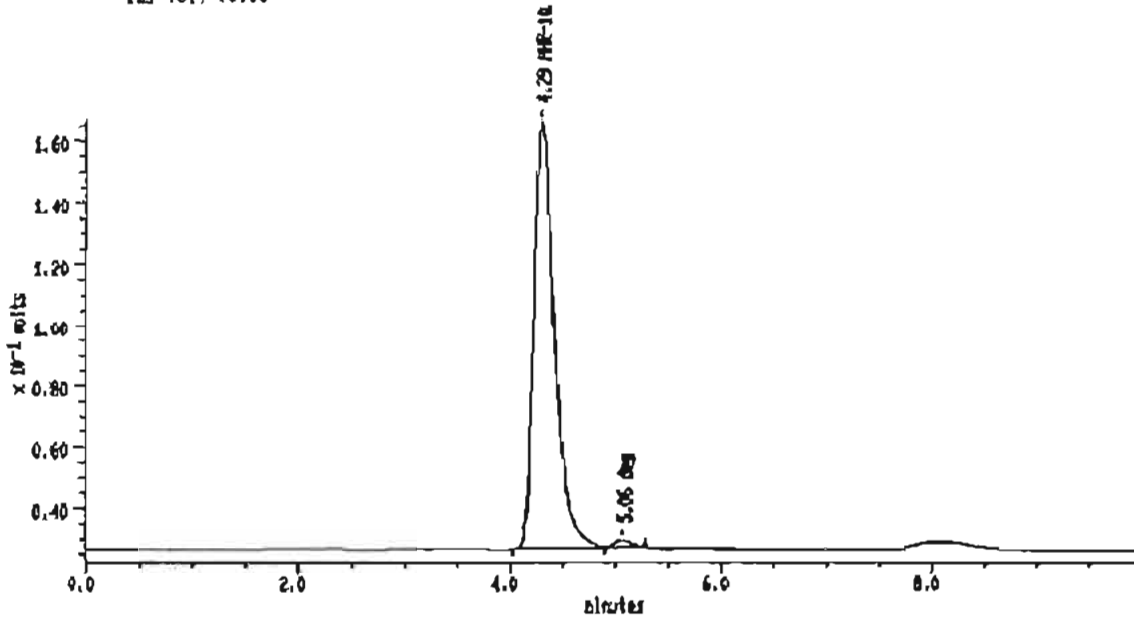
Index: 45

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.297	BB	1922389	139165	99.18	AHR-10282B
2	8.833	BB	16669	1272	0.82	40g
TOTAL			1939058	140457		

Sample: A25 50°C-4W Channel: detector 1 Filtration: S2-21 Chart Speed: Full Size
 Acquired: 03-APR-101 0:10 Method: B:VAHRYIXI760-2E Operator: S.S
 [n] Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 3-APR-2001 0:27:21

SAMPLE: A25 50°C-4W

Method: AHR-10282B

Acquired: 3-APR-2001 0:10

Rate: 2.0 points/min

Duration: 10.000 minutes

Operator: S.S

Type: LONON

Instrument: Instrument 1

Filtration: S2-21

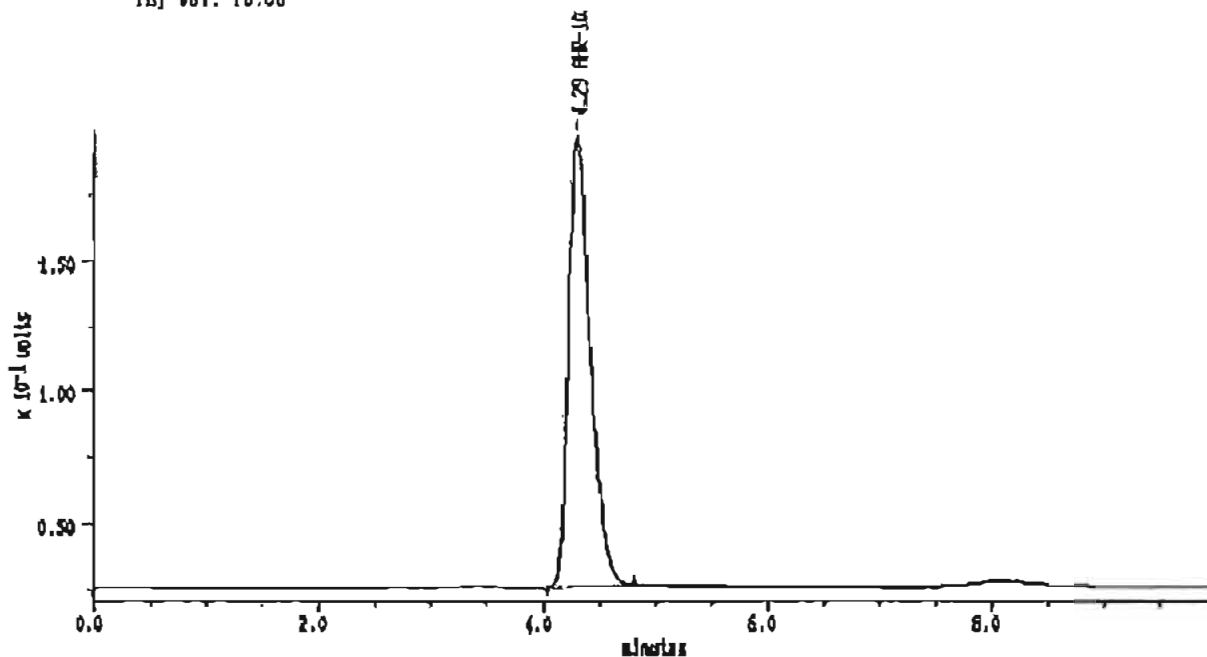
Index: 46

Injection Volume: 10.0

DETECTOR: detector 1

PKR	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BB	1927309	198800	99.83	AHR-10282B
2	5.058	SS	22848	1940	1.17	deg
TOTAL			1965157	140835		

Sample: ST02 Channel: detector 1 Pilename: S2-22 Chart Speed: Full Size
 Acquired: 03-APR-10 0:28 Method: BIVAKRVIK13V80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 3-APR-2001 0:38:59

SAMPLE: ST02

#22 In Method: AHR-10282B

Acquired: 3-APR-2001 0:28

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pilename: S2-22

Index: 47

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.282	BB	2321811	170012	100.00	AHR-10282B
TOTAL			2321811	170012		

4/02 17:49 NO.92 PH 7.05 24.1°C	4/02 18:07 NO. 4 PH 7.06 23.9°C
4/02 17:51 NO.93 PH 7.10 24.3°C	4/02 18:12 NO. 8 PH 7.11 24.2°C
4/02 17:53 NO.94 PH 7.08 24.3°C	4/02 18:14 NO. 6 PH 7.10 23.9°C
4/02 17:55 NO.95 PH 7.06 24.1°C	4/02 18:15 NO. 7 PH 7.08 23.8°C
4/02 17:56 NO.96 PH 7.09 24.1°C	4/02 18:16 NO. 3 PH 7.09 23.6°C
4/02 17:59 NO.97 PH 7.07 24.0°C	4/02 18:17 NO. 9 PH 7.05 23.6°C
4/02 18:00 NO.98 PH 7.07 23.9°C	4/02 18:19 NO.10 PH 7.10 23.7°C
4/02 18:01 NO.99 PH 7.09 23.9°C	4/02 18:21 NO.11 PH 7.12 23.7°C
4/02 18:02 NO. 1 PH 7.07 23.9°C	4/02 18:23 NO.12 PH 7.11 23.7°C
4/02 18:04 NO. 2 PH 7.11 23.9°C	4/02 18:26 NO.13 PH 7.14 24.2°C
4/02 18:06 NO. 3 PH 7.08 23.6°C	

P2000B177 Lot No. 01K131 60°C-2W

This raw data is a duplicated copy as it could suffer the possibility of deterioration over time.

Therefore, there is no difference between this copy and the original copy.

2005.05.06 Shirou Sawa

Bronuck Ophthalmic Solution Stability Test
 Lot No 01K131

Test code: P2000B177
 Tester: Shirou Sawa
 Test date: 23 March 2001

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Water Collec(%)	Water	Initial	present
STD	1	V2-01	2280068						
STD	2	V2-22	2318949						
STD	mean		2305019	0.9975					
A-26	70°C-2W	V2-02	2083024	0.8928	89.24	82.40	7.87	8.4916	8.1202
A-27	70°C-2W	V2-03	2247015	0.9724	98.54	90.59	7.97	8.3951	8.0171
A-28	70°C-2W	V2-04	2213332	0.9578	95.54	89.01	7.80	8.5470	8.1854
A-29	70°C-2W	V2-05	2240698	0.9697	93.85	84.93	9.50	8.4982	8.0358
A-30	70°C-2W	V2-06	2175407	0.9414	92.78	83.08	10.48	8.4388	7.9380
A-31	70°C-2W	V2-07	2034107	0.8803	86.69	79.82	7.92	8.4972	8.1193
A-32	70°C-2W	V2-08	1828465	0.7039	66.90	61.18	8.55	8.4194	8.0117
A-33	70°C-2W	V2-09	2080989	0.9005	89.84	82.51	8.18	8.4396	8.0487
A-34	70°C-2W	V2-10	2003119	0.8889	86.41	79.60	7.88	8.4808	8.1003
A-35	70°C-2W	V2-11	1915502	0.8289	82.00	75.89	7.45	8.5033	8.1417
A-26	80°C-4W	V2-12	1854181	0.8024	80.21	74.86	6.67	8.4743	8.1527
A-27	80°C-4W	V2-13	2184535	0.9497	96.24	89.84	8.88	8.4358	8.1076
A-28	80°C-4W	V2-14	2111436	0.9137	92.10	85.96	8.87	8.5133	8.1889
A-29	80°C-4W	V2-15	2112402	0.9141	88.47	82.01	7.30	8.4580	8.1051
A-30	80°C-4W	V2-16	2040435	0.8830	87.02	80.71	7.26	8.4431	8.0958
A-31	80°C-4W	V2-17	1089091	0.8092	79.68	74.12	6.98	8.6017	8.2604
A-32	80°C-4W	V2-18	1243872	0.5383	51.16	47.37	7.41	8.4342	8.0797
A-33	80°C-4W	V2-19	1805780	0.8247	82.28	78.82	6.83	8.4998	8.1784
A-34	80°C-4W	V2-20	1860887	0.8010	79.84	74.04	7.26	8.4355	8.0883
A-35	80°C-4W	V2-21	1813088	0.7846	77.62	72.33	6.81	8.6422	8.3022

*Re-edited in order to document the data necessary for calculation.
 Shirou Sawa, 6 May 2005*

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B		Test code	P2000B177		Test date	23 March 2001	
Test item						Tester	Shirou Sawa	
Lot No. 01K131								
STD 0.01995g + MP → 20mL								
Above solution 2mL + MP → 20mL								
<i>PH</i>					<i>Turbidity</i>	<i>Foreign matter</i>	<i>Color</i>	
A26	70°C-2W	6.89	8.4915	8.1202	+	+	Dark yellow	
A27		7.00	8.3951	8.0171	+	+	Yellow	
A28		7.00	8.5470	8.1654	+	+	Yellow	
A29		6.96	8.4962	8.0358	+	+(r)	Dark yellow	
A30		7.00	8.4388	7.9380	+	+(r)	↓	
A31		6.93	8.4972	8.1133	+	+(r)	↓	
A32		7.00	8.4194	8.0117	+	+	↓	
A33		6.99	8.4396	8.0487	+	+(r)	↓	
A34		6.95	8.4808	8.1003	+	+(r)	Brownish yellow	
A35		7.03	8.5033	8.1417	+	+(r)	↓	
A26	60°C-4W	7.00	8.4743	8.1527	+	+	Dark yellow	
A27		6.99	8.4358	8.1075	+	+(r)	Yellow	
A28		6.96	8.5133	8.1889	+	+(r)	Yellow	
A29		6.94	8.4560	8.1051	+	+(r)	Dark yellow	
A30		6.96	8.4431	8.0958	+	+(r)	↓	
A31		6.93	8.6017	8.2564	+	+(r)	Yellow	
A32		7.01	8.4342	8.0797	++	+(r)	Dark yellow	
A33		7.00	8.4998	8.1784	++	+(r)	↓	
A34		7.02	8.4355	8.0883	+	+(r)	↓	
A35		7.04	8.6422	8.3022	+	+(r)	↓	

3/23 17:07
NO.65 PH 6.89
25.2°C

3/23 17:08
NO.66 PH 7.00
25.6°C

3/23 17:09
NO.67 PH 7.90
25.7°C

3/23 17:10
NO.68 PH 6.95
25.7°C

3/23 17:11
NO.69 PH 7.00
25.7°C

3/23 17:12
NO.70 PH 6.93
25.7°C

3/23 17:14
NO.71 PH 7.03
25.6°C

3/23 17:16
NO.72 PH 6.99
25.6°C

3/23 17:17
NO.73 PH 6.95
25.6°C

3/23 17:18
NO.74 PH 7.03
25.7°C

3/23 17:20
NO.75 PH 7.02
25.8°C

3/23 17:22
NO.76 PH 6.99
25.9°C

3/23 17:23
NO.77 PH 6.96
26.0°C

3/23 17:24
NO.78 PH 6.94
25.8°C

3/23 17:24
NO.79 PH 6.95
25.8°C

3/23 17:25
NO.80 PH 6.95
25.8°C

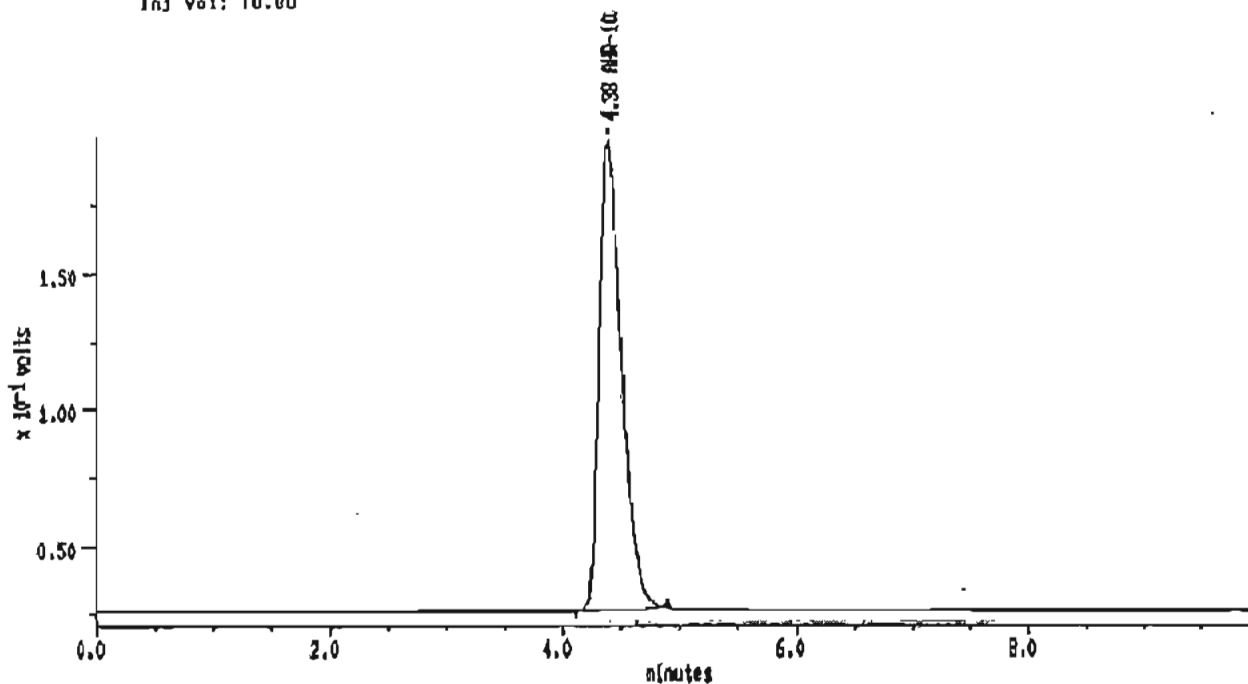
3/23 17:28
NO.81 PH 7.01
25.9°C

3/23 17:29
NO.82 PH 7.00
25.9°C

3/23 17:31
NO.83 PH 7.02
26.0°C

3/23 17:31
NO.84 PH 7.04
25.8°C

Sample: STD1 Channel: detector 1 Filename: V2-01 Chart Speed: Full Size
 Acquired: 23-MAR-2001 21:48 Method: B:VAHRY(K133V70-2W) Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:08:21

SAMPLE: STD1

#1 in Method: AHR-10282B

Acquired: 23-MAR-2001 21:48

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-01

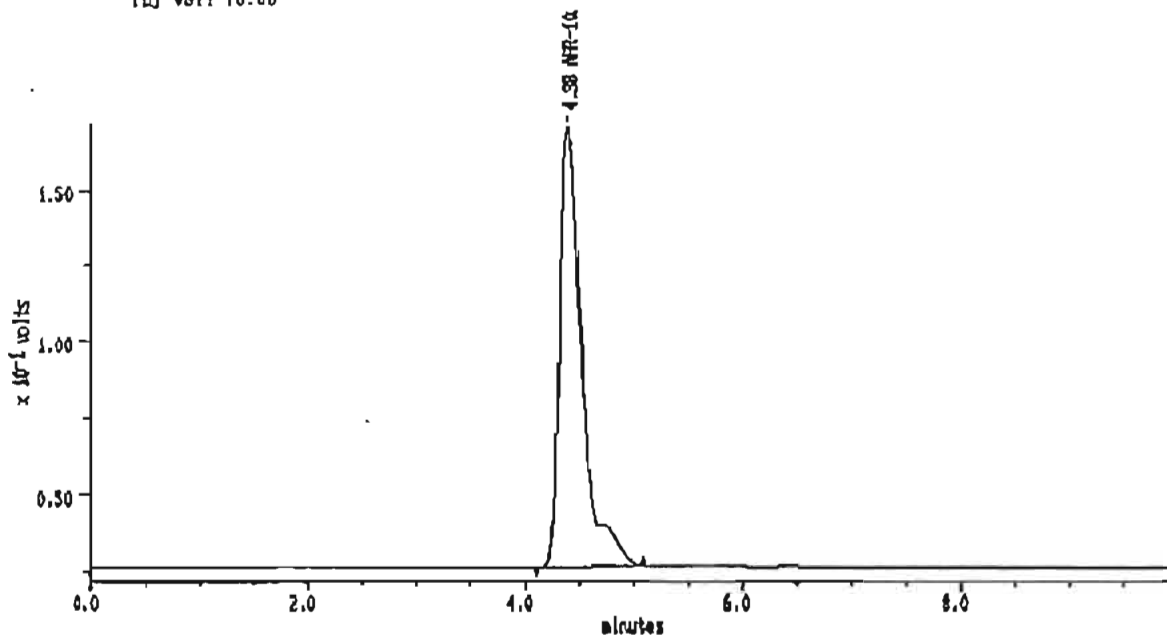
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.380	BB	2280088	171089	100.00	AHR-10282B
TOTAL			2280088	171089		

Sample: A28 70°C-2W Channel: detector 1 Filename: V2-02 Chart Speed: Full Size
 Acquired: 23-MAR-2001 21:59 Method: B:VAHRVVKI3Y7Q-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1998 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:08:51

SAMPLE: A28 70°C-2W

#2 In Method: AHR-10282B

Acquired: 23-MAR-2001 21:59

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNXX

Instrument: Instrument 1

Filename: V2-02

Index: 27

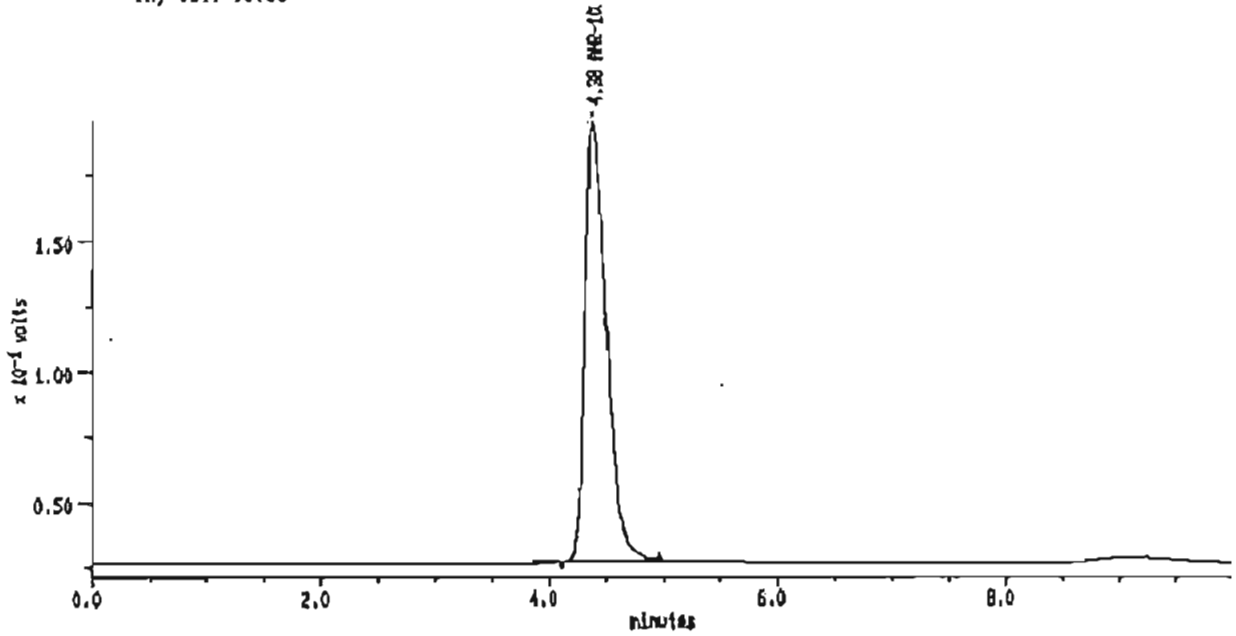
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.380	BB	2083024	143788	100.00	AHR-10282B
TOTAL			2083024	143788		

4

Sample: A21 70°C-2W Channel: detector 1 Filename: V2-03 Chart Speed: Full Size
 Acquired: 23-MAR-2001 22:11 Method: B:VAHRVYIK12V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:09:21

SAMPLE: A21 70°C-2W

#3 In Method: AHR-10282B

Acquired: 23-MAR-2001 22:11

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-03

Index: 28

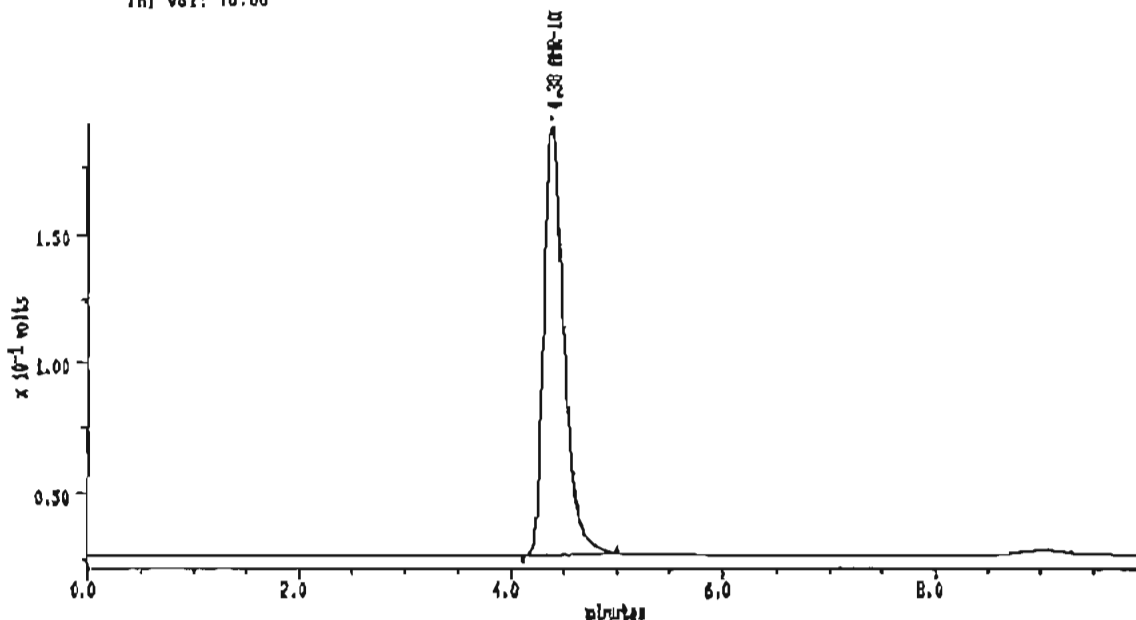
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.375	BB	2247015	167844	100.00	AHR-10282B
TOTAL			2247015	167844		

5

Sample: A28 70°C-2W Channel: detector 1 Filename: V2-04 Chart Speed: Full Size
 Acquired: 23-MAR-2001 22:22 Method: B:VAHRTIKI3Y10-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 24-MAR-2001 11:08:52

SAMPLE: A28 70°C-2W

#4 In Method: AHR-10282B

Acquired: 23-MAR-2001 22:22

Rate: 2.0 points/sec

Duration: 10,000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-04

Index: 29

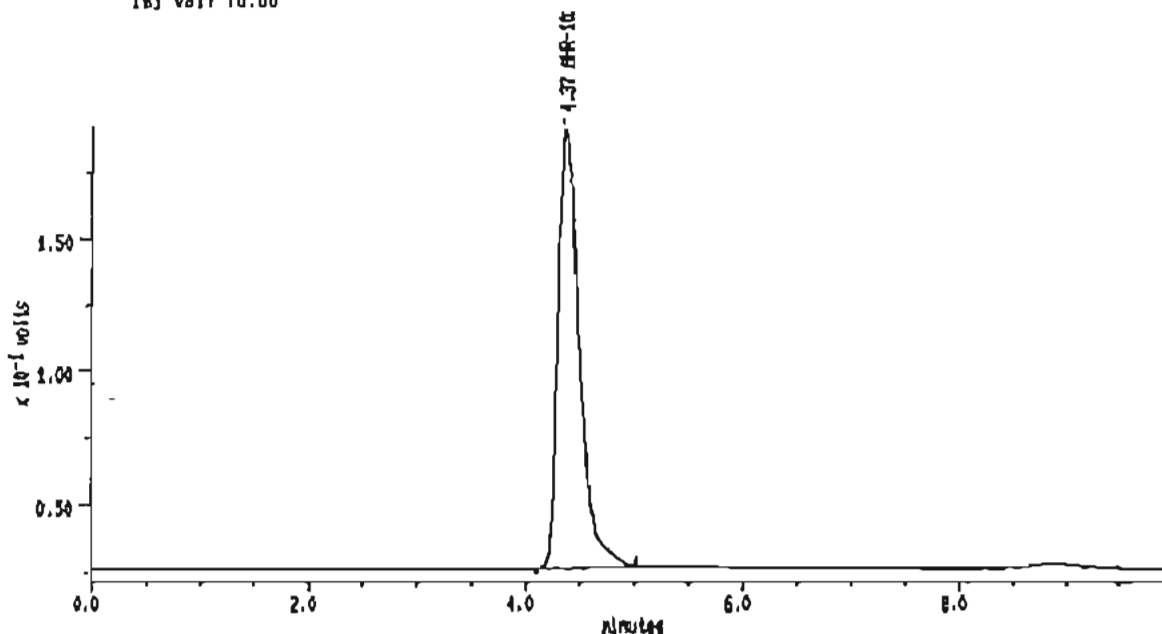
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.376	BB	2213332	163942	100.00	AHR-10282B
TOTAL			2213332	163942		

6

Sample: A20 70°C-2W Channel: detector 1 Filename: V2-06 Chart Speed: Full Size
 Acquired: 20-MAR-10 22:33 Method: B:YAJRYIK13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:10:23

SAMPLE: A20 70°C-2W

#5 in Method: AHR-10282B

Acquired: 20-MAR-2001 22:33

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: LINK

Instrument: Instrument 1

Filename: V2-06

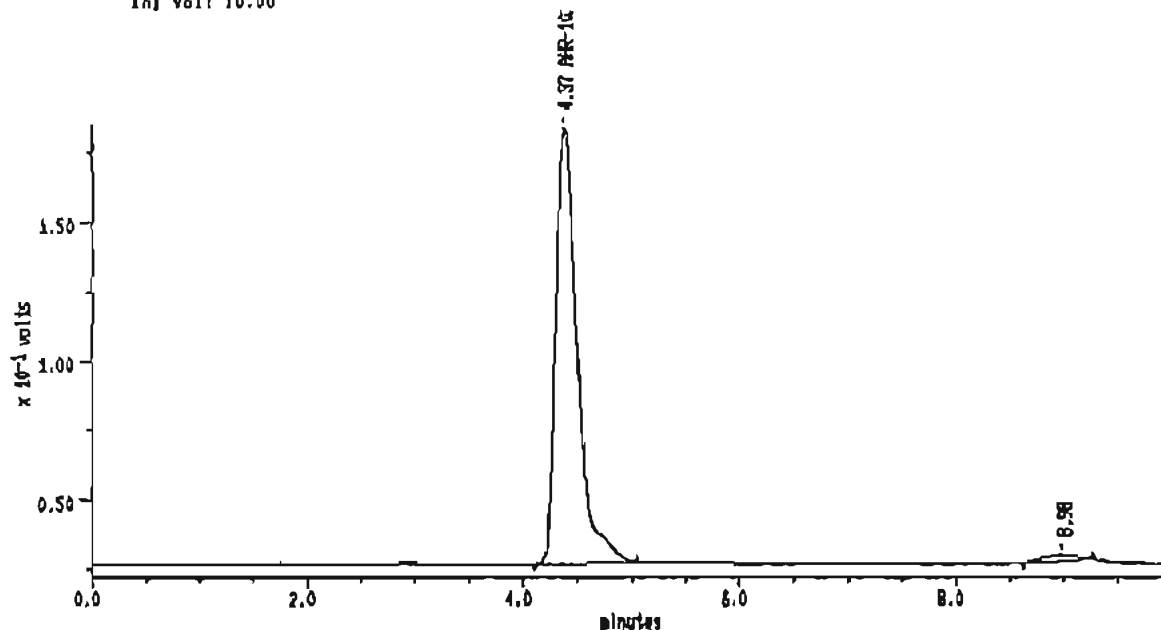
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.367	B0	2240098	104307	100.00	AHR-10282B
TOTAL			2240098	104307		

Sample: A30 70°C-2W Channel: detector 1 Filename: V2-00 Chart Speed: Full Size
 Acquired: 23-MAR-10 22:46 Method: D:YANRVIXI12V70-20 Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:10:54

SAMPLE: A30 70°C-2W

#B 16 Method: AHR-10282B

Acquired: 23-MAR-2001 22:46

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: V2-00

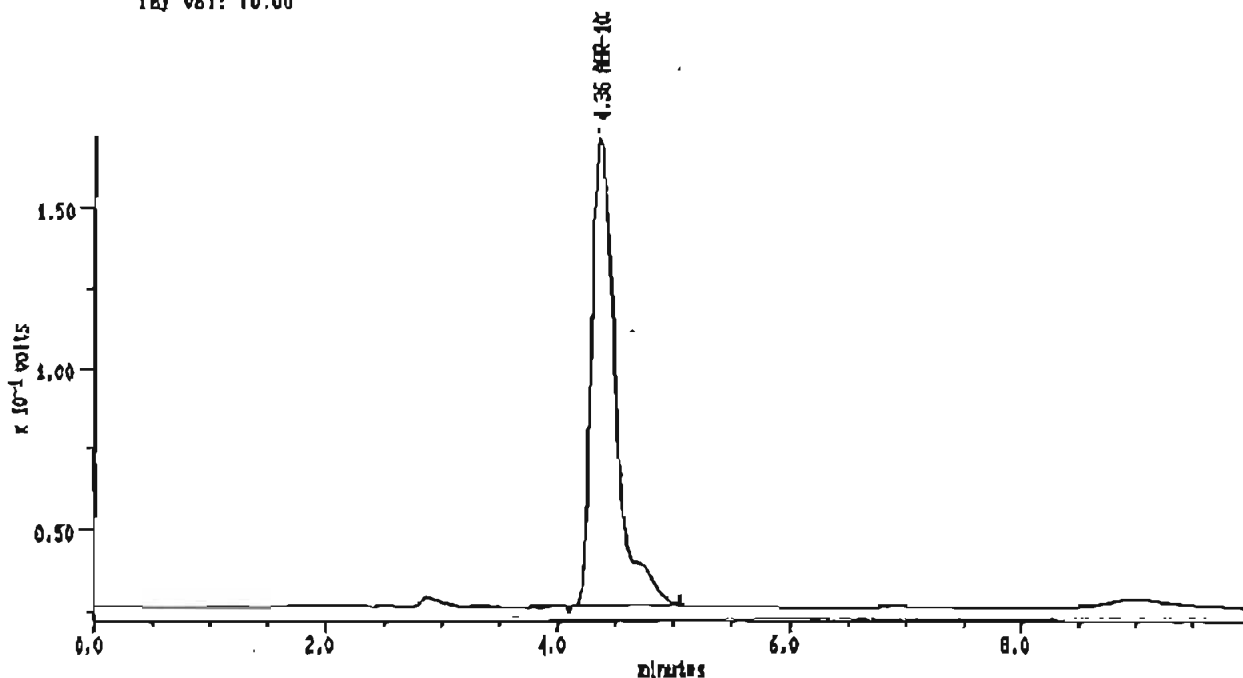
Index: 51

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.367	BB	2176407	168906	100.00	AHR-10282B
TOTAL			2176407	168906		

Sample: A31 70°C-2W Channel: detector 1 Filename: V2-07 Chart Speed: Full Size
 Acquired: 23-MAR-10J 22:58 Method: D:\AUM\YIK13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:11:24

SAMPLE: A31 70°C-2W

#1 in Method: AHR-10282B

Acquired: 23-MAR-2001 22:58

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: V2-07

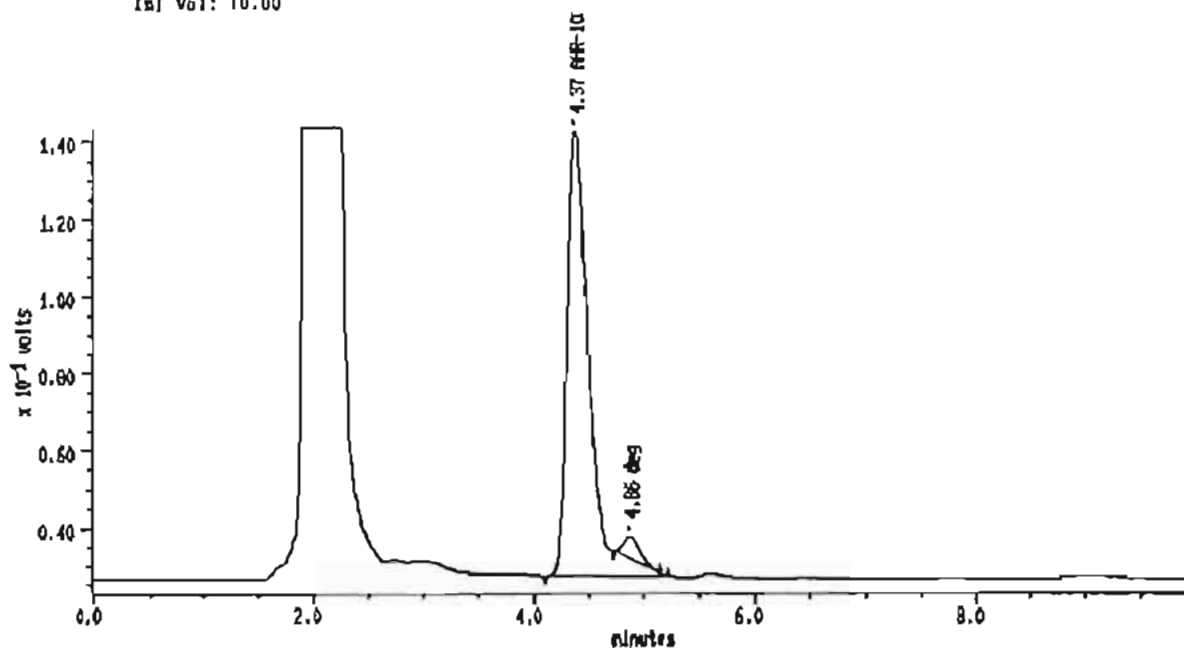
Index: 32

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.368	BB	2034107	143809	100.00	AHR-10282B
TOTAL			2034107	143809		

Sample: A32 70°C-2W Channel: detector 1 Filename: V2-08 Chart Speed: Full Size
 Acquired: 23-MAR-2001 23:07 Method: 8:YAHURYIKISV70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:11:55

SAMPLE: A32 70°C-2W

#8 In Method: AHR-10282B

Acquired: 23-MAR-2001 23:07

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-08

Index: 33

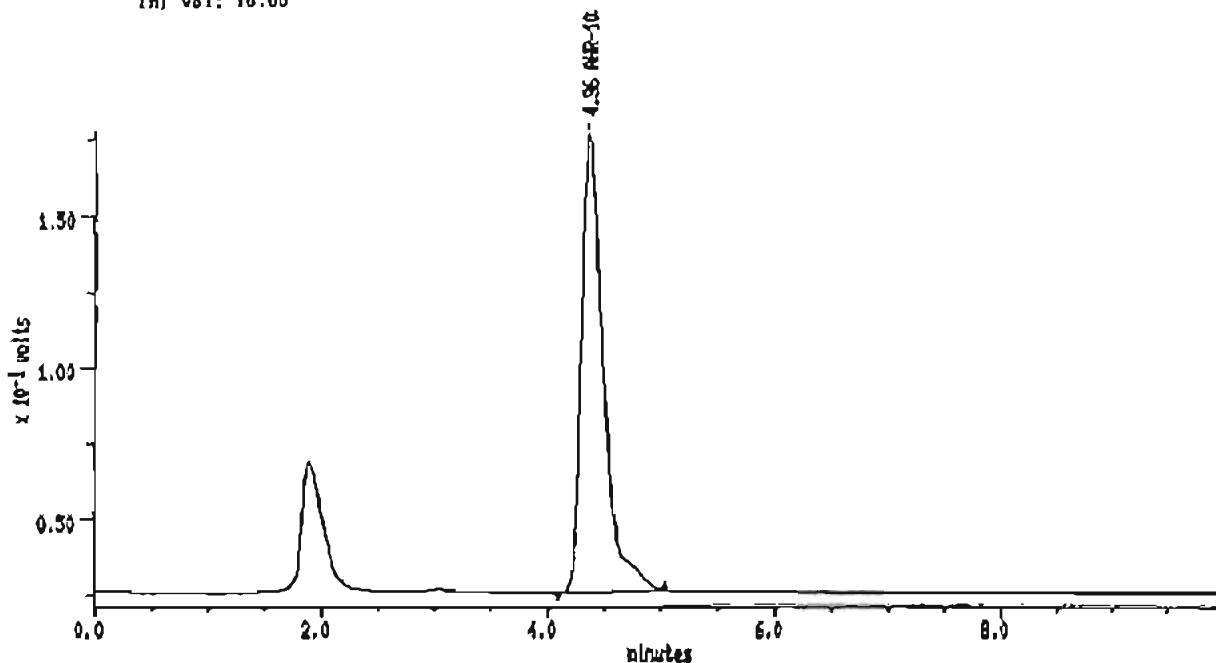
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.367	BB	1020406	114940	96.28	AHR-10282B
2	4.658	SS	83129	5460	3.74	deg
TOTAL			1089594	120390		

10

Sample: A33 70°C-2W Channel: detector 1 Filament: VZ-09 Chart Speed: Full Scan
 Acquired: 23-MAR-2001 23:19 Method: B:VAMRV\K13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:12:28

SAMPLE: A33 70°C-2W

#9 In Method: AHR-10282B

Acquired: 23-MAR-2001 23:19

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UMRN

Instrument: Instrument 1

Filament: VZ-09

Index: 34

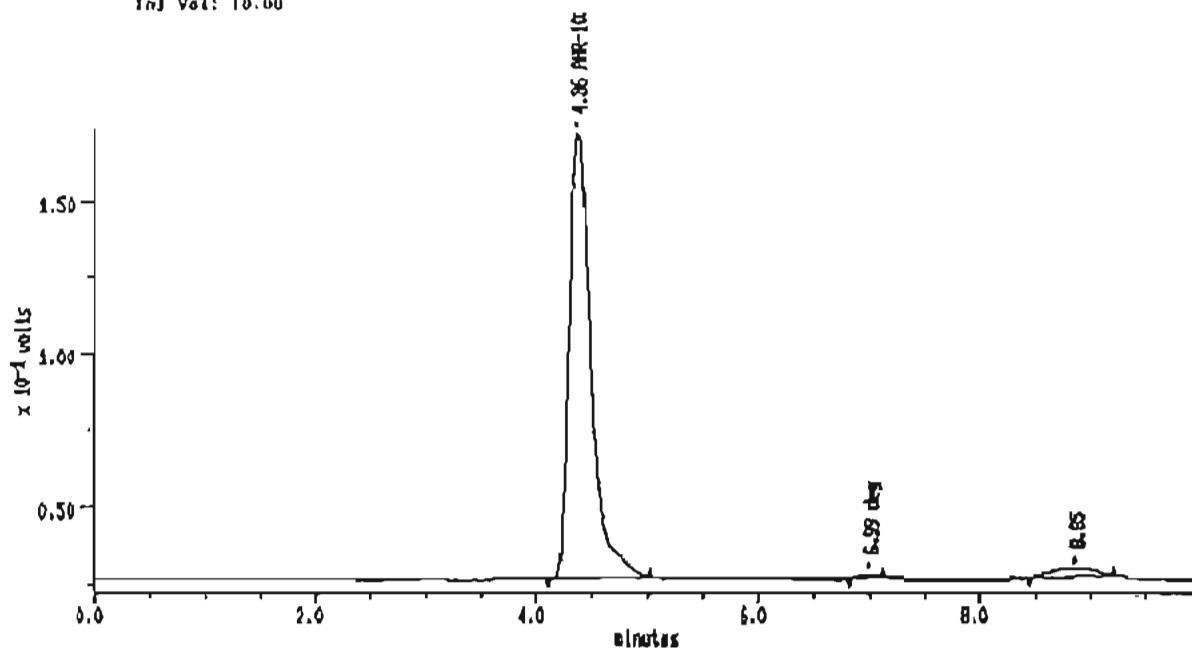
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.368	BB	2080909	149995	100.00	AHR-10282B
TOTAL			2080909	149995		

11

Sample: A34 70°C-2# Channel: detector 1 Filename: V2-10 Chart Speed: Full Size
 Acquired: 23-MAR-01 23:30 Method: B:YAHRY1K13V70-2# Operator: S.5
 Inj Vol: 10.00



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MAXIMA 826 Custom Report

Printed: 26-MAR-2001 12:12:58

SAMPLE: A34 70°C-2#

#10 In Method: AHR-10282B

Acquired: 23-MAR-2001 23:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.5

Type: UHPLC

Instrument: Instrument 1

Filename: V2-10

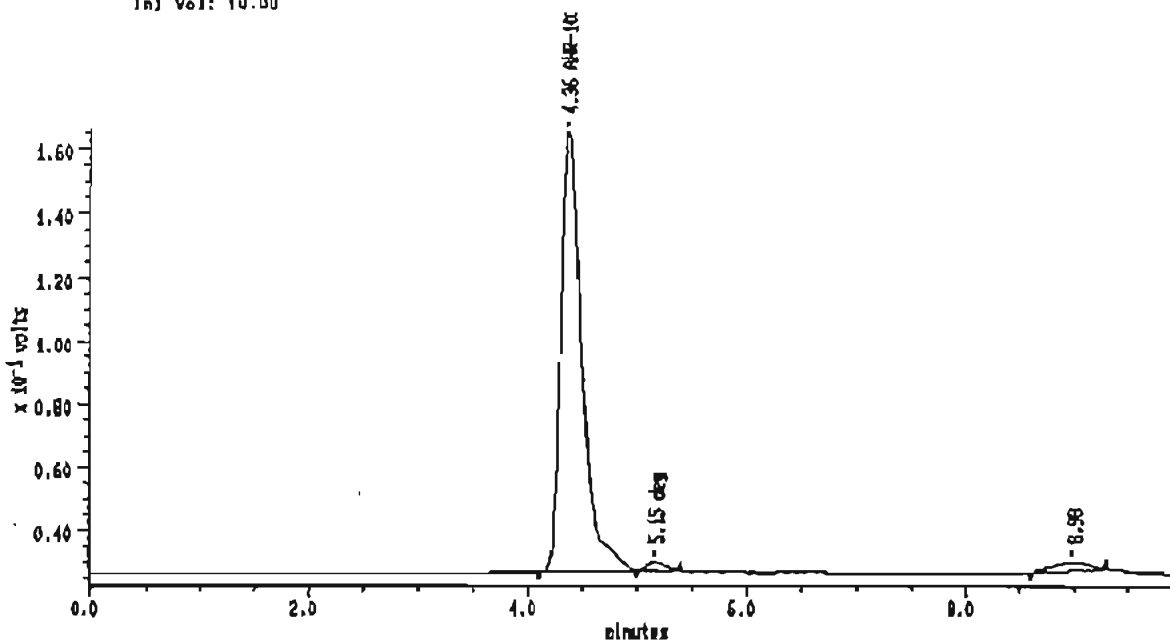
Index: 35

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.368	BB	2003119	145778	99.40	AHR-10282B
2	8.992	BB	12177	1039	0.60	dog
TOTAL			2015296	146816		

Sample: A35 70°C-2W Channel: detector 1 Filename: V2-11 Chart Speed: Full Size
 Acquired: 23-MAR-2001 23:41 Method: B:YAHRYIK13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:13:30

SAMPLE: A35 70°C-2W

File In Method: AHR-10282B

Acquired: 23-MAR-2001 23:41

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-11

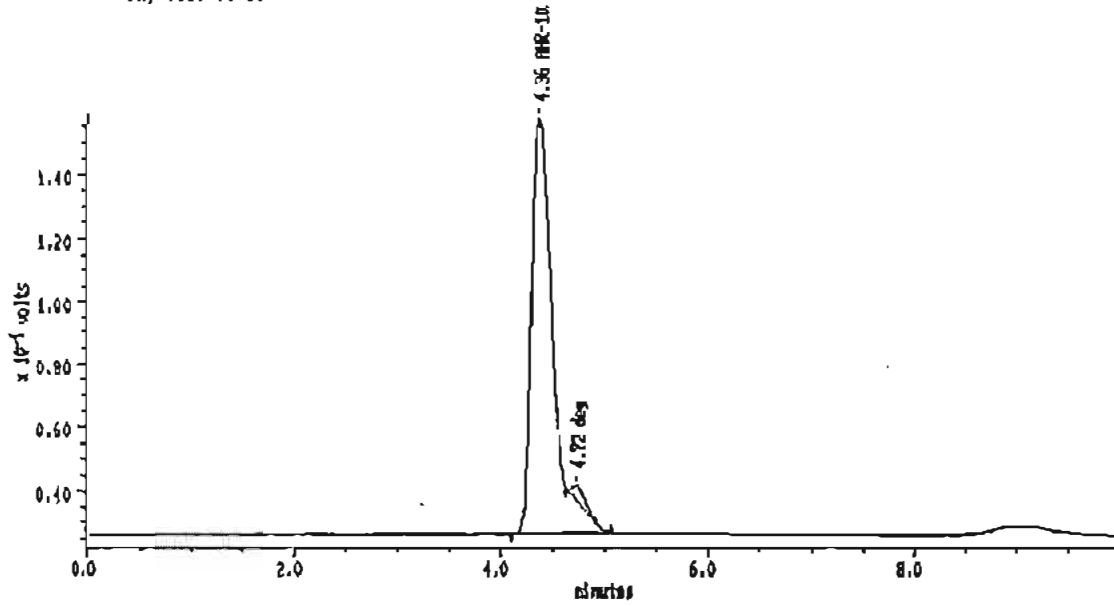
Index: 36

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.358	SB	1918307	138408	88.84	AHR-10282B
2	5.150	SS	20468	2224	1.36	deg
TOTAL			1941957	140832		

Sample: A28 80°C-4W Channel: detector 1 Filename: V2-12 Chart Speed: Full Size
 Acquired: 23-MAR-2001 23:52 Method: D:\MSRV\HR13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:39:07

SAMPLE: A28 80°C-4W

#12 In Method: AHR-10282B

Acquired: 23-MAR-2001 23:52

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-12

Index: 37

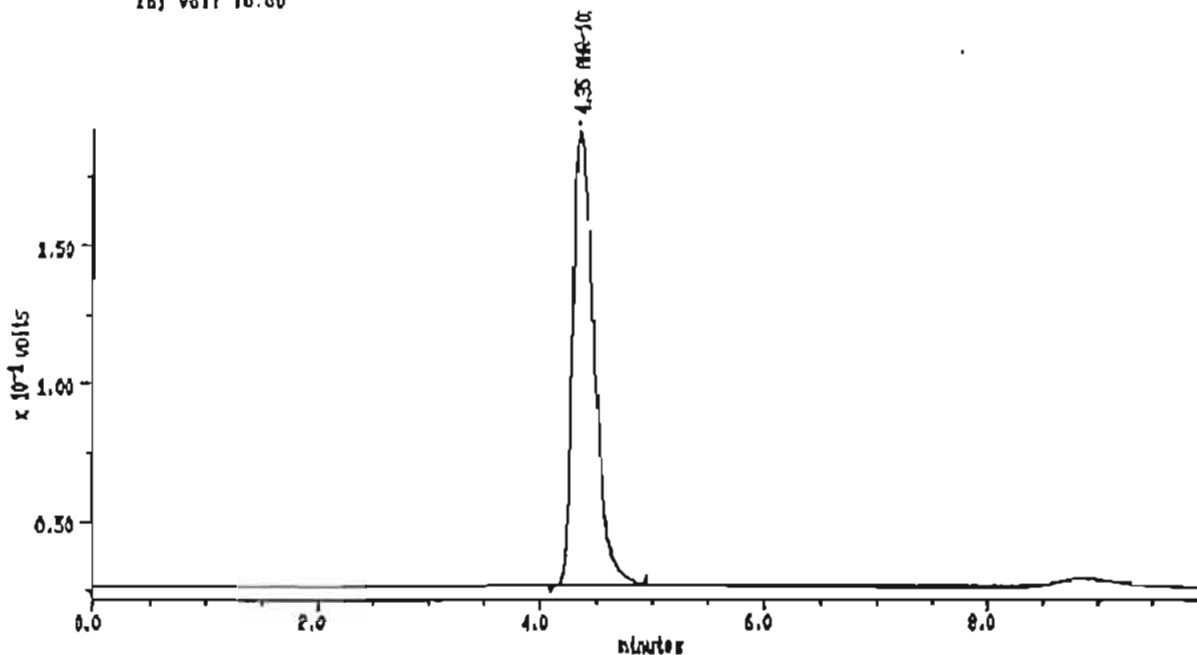
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.360	BB	1864181	130629	99.02	AHR-10282B
2	4.717	SS	37483	4280	1.98	deg
TOTAL			1891664	134779		

14

Sample: A27 80°C-4W Channel: detector 1 Filename: V2-13 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:04 Method: B:VAIRY(K13V70-2W) Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:14:23

SAMPLE: A27 80°C-4W

#13 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:04

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-13

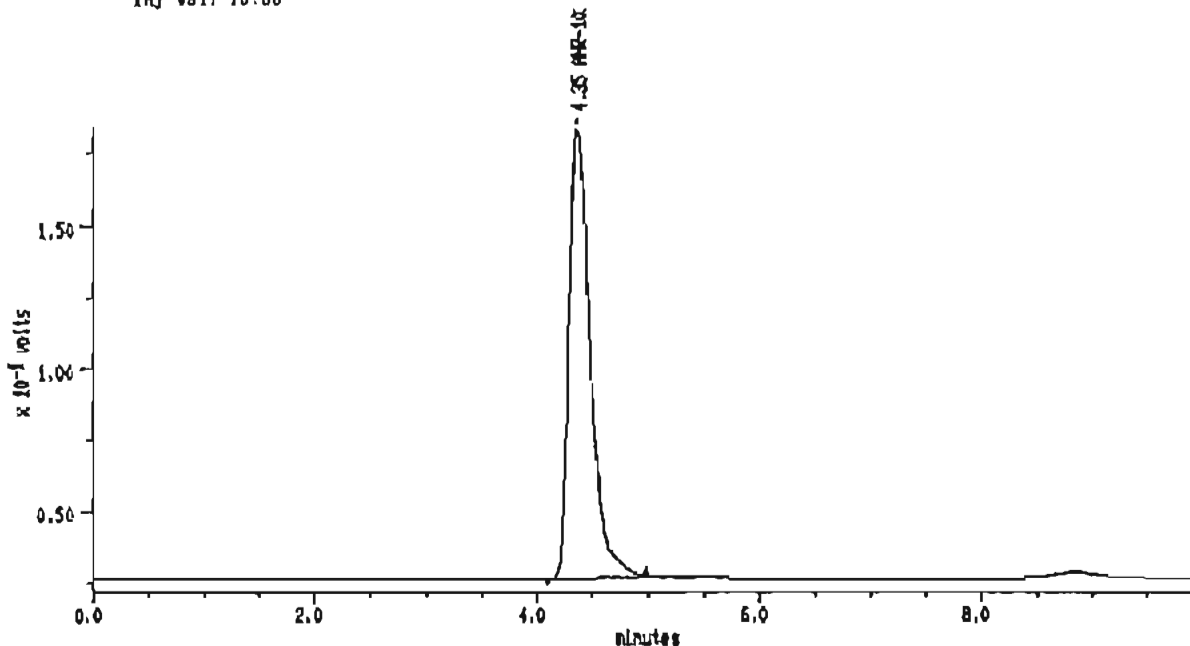
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	8B	2194636	184193	100.00	AHR-10282B
TOTAL			2194636	184193		

Sample: A28 60°C-4W Channel: detector 1 Filename: V2-14 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:16 Method: D:\AHRV\K13Y70-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (c)1998 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:16:03

SAMPLE: A28 60°C-4W

#14 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:16

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-14

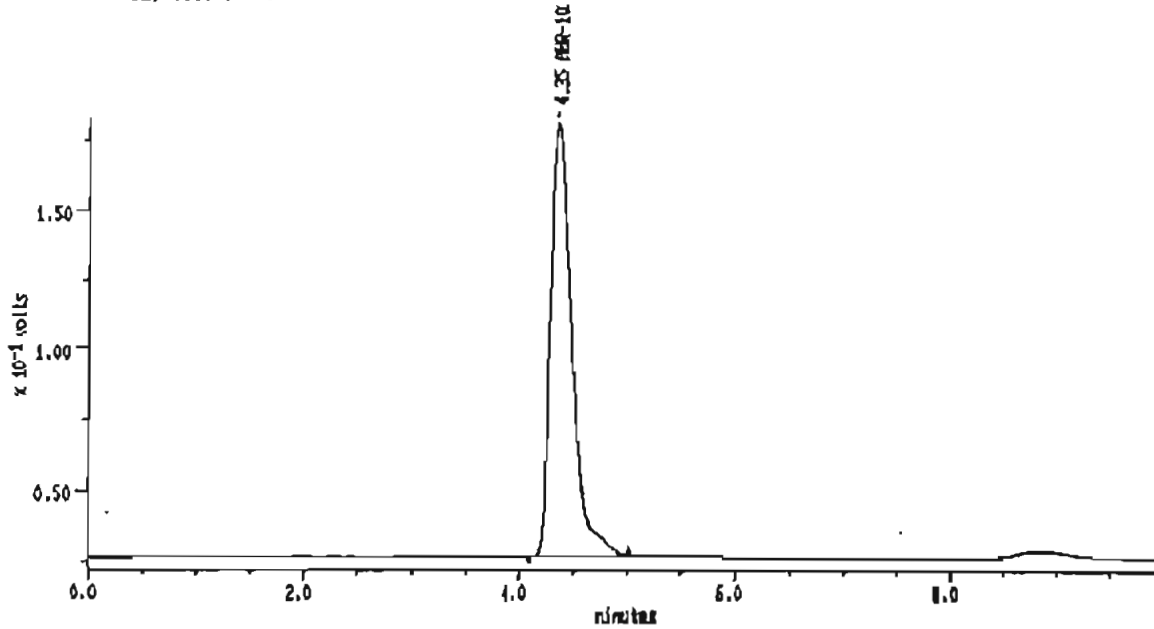
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	BB	2111436	156232	100.00	AHR-10282B
TOTAL			2111436	156232		

Sample: A28 80°C-4W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 24-MAR-101 0:27 Method: B:YAHRRWIKI3V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 26-MAR-2001 12:15:34

SAMPLE; A28 80°C-4W

#16 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-18

Index: 40

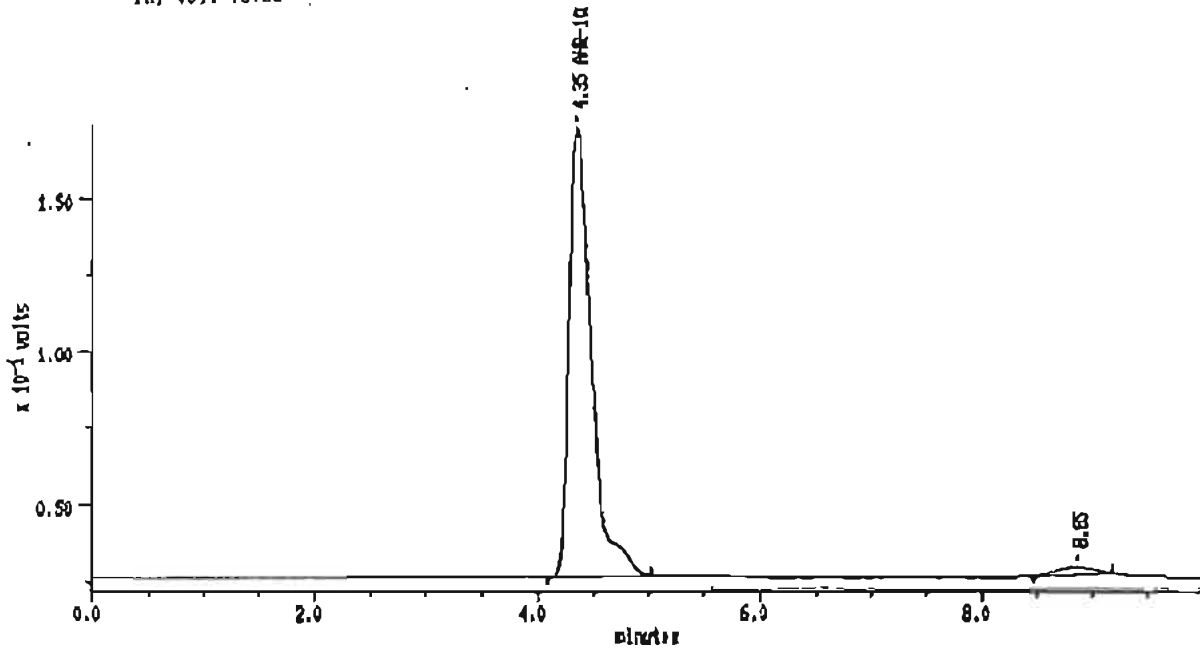
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.360	BB	2112402	164392	100.00	AHR-10282B
TOTAL			2112402	164392		

17

Sample: A30 80°C-4W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:38 Method: B:\AHR\YIK13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:18:08

SAMPLE: A30 80°C-4W

#16 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNEX

Instrument: Instrument 1

Filename: V2-18

Index: 41

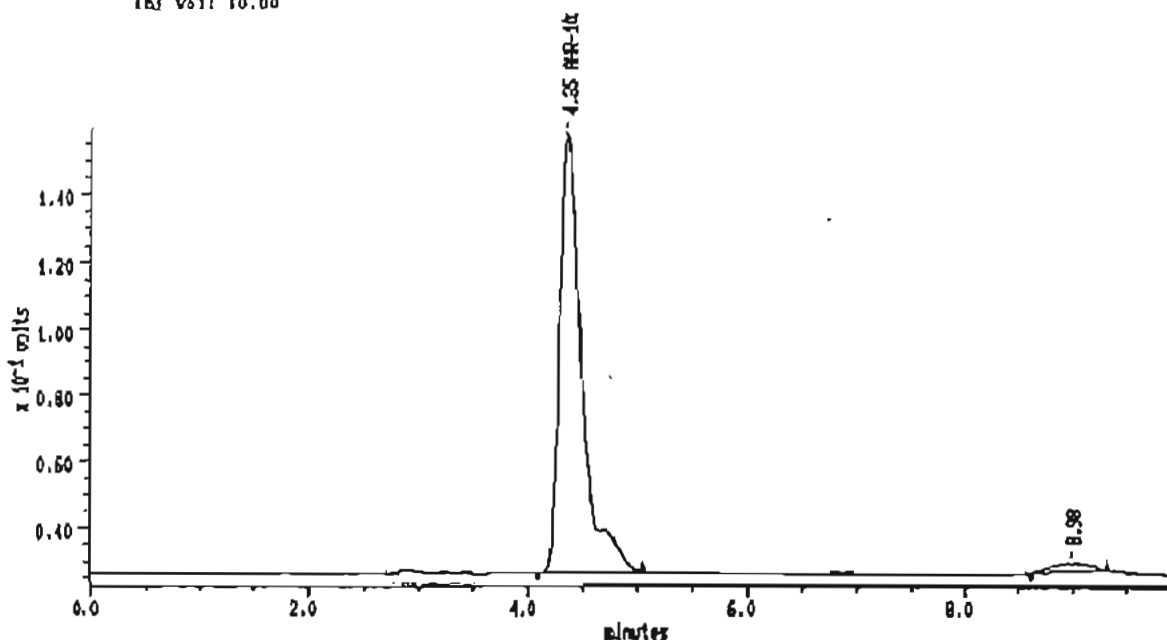
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	B0	2040435	148306	100.00	AHR-10282B
TOTAL			2040435	148306		

18

Sample: AD1 80°C-4W Channel: detector 1 Filament: V2-17 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:49 Method: 8:YAIRYIK12V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:18:37

SAMPLE: AD1 80°C-4W

017 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:49

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filament: V2-17

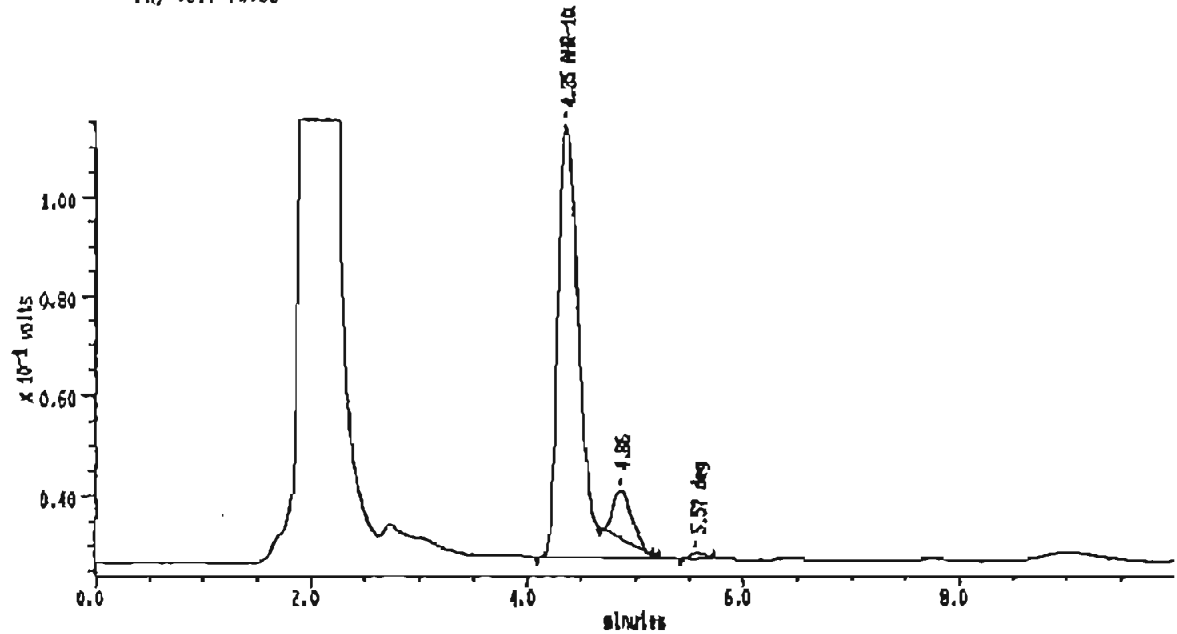
Index: 42

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	DB	1888891	131403	100.00	AHR-10282B
TOTAL			1888891	131403		

Sample: A32 80°C-4W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 24-MAR-2001 1:01 Method: B:VAHRV1K13V10-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

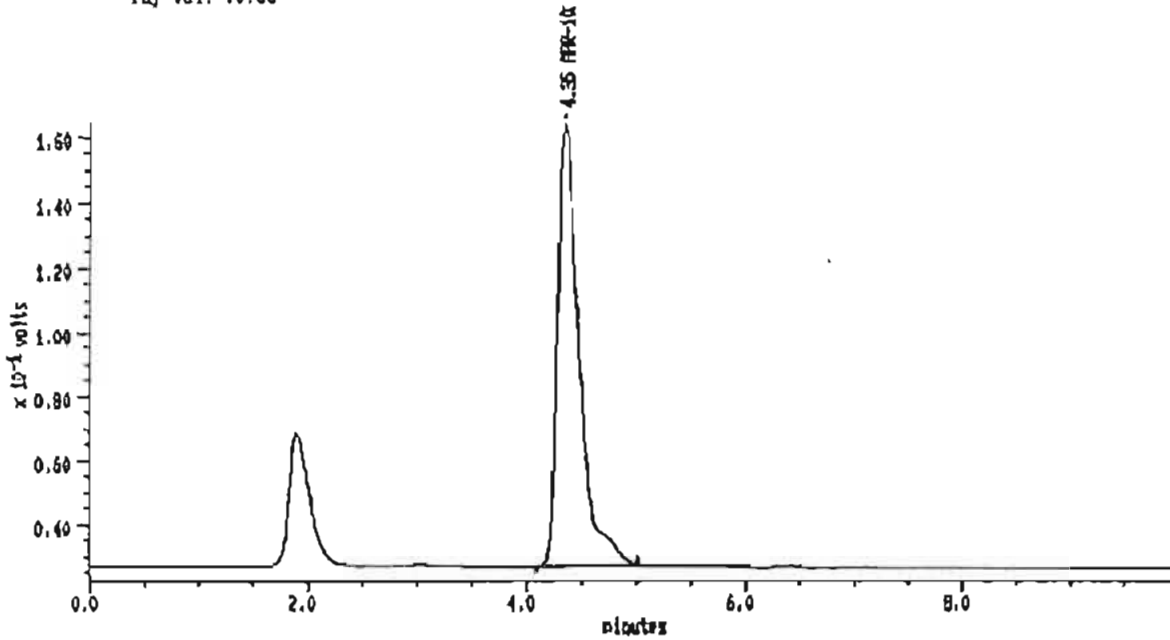
Printed: 26-MAR-2001 12:17:08

SAMPLE: A32 80°C-4W	Type: UNKN
#18 In Method: AHR-10282B	Instrument: Instrument 1
Acquired: 24-MAR-2001 1:01	Filename: V2-18
Rate: 2.0 points/sec	Index: 43
Duration: 10.000 minutes	Injection Volume: 10.0
Operator: S.S	Amount: 0.000

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	BB	1243872	88988	98.98	AHR-10282B
3	5.587	BB	13118	1180	1.04	deg
TOTAL			1288990	89172		

Sample: A33 80°C-4# Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 24-MAR-2001 1:12 Method: D:\VAHRVIX\10Y70-2# Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:17:38

SAMPLE: A33 80°C-4#

#19 (n Method: AHR-10282B

Acquired: 24-MAR-2001 1:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-18

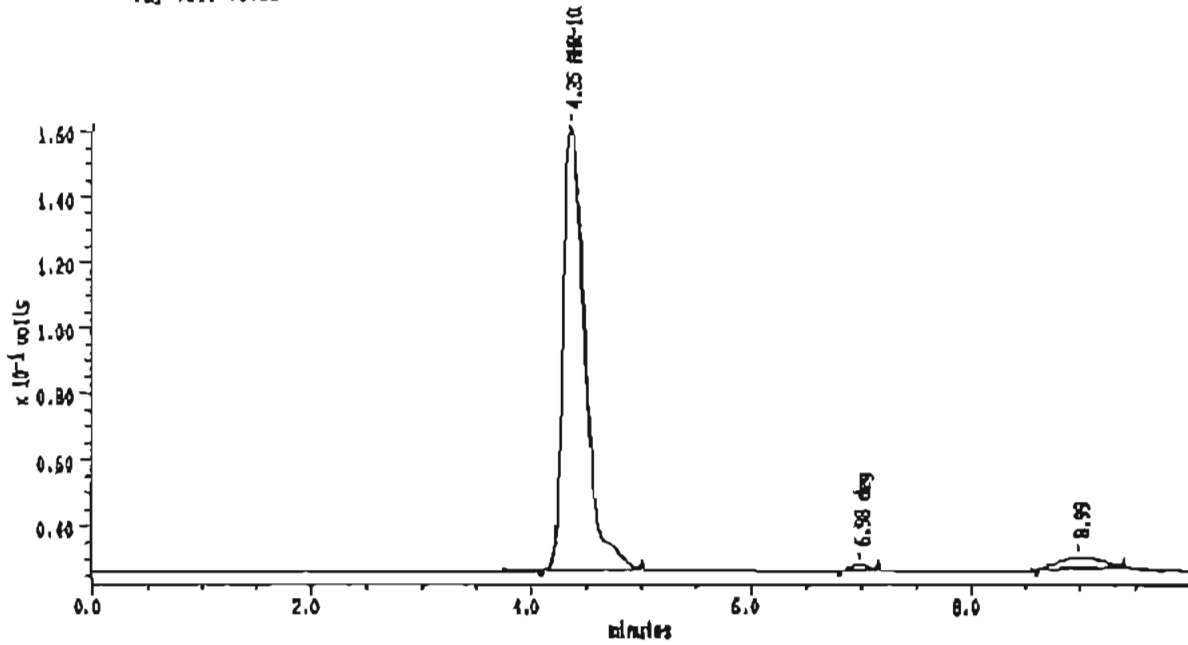
Index: 44

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	DD	1806780	138520	100.00	AHR-10282B
TOTAL			1806780	138520		

Sample: A34 80°C-4W Channel: detector 1 Filename: V2-20 Chart Speed: Full Size
 Acquired: 24-MAR-2001 1:23 Method: D:YAHRIK13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:18:11

SAMPLE: A34 80°C-4W

#20 In Method: AHR-10282B

Acquired: 24-MAR-2001 1:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-20

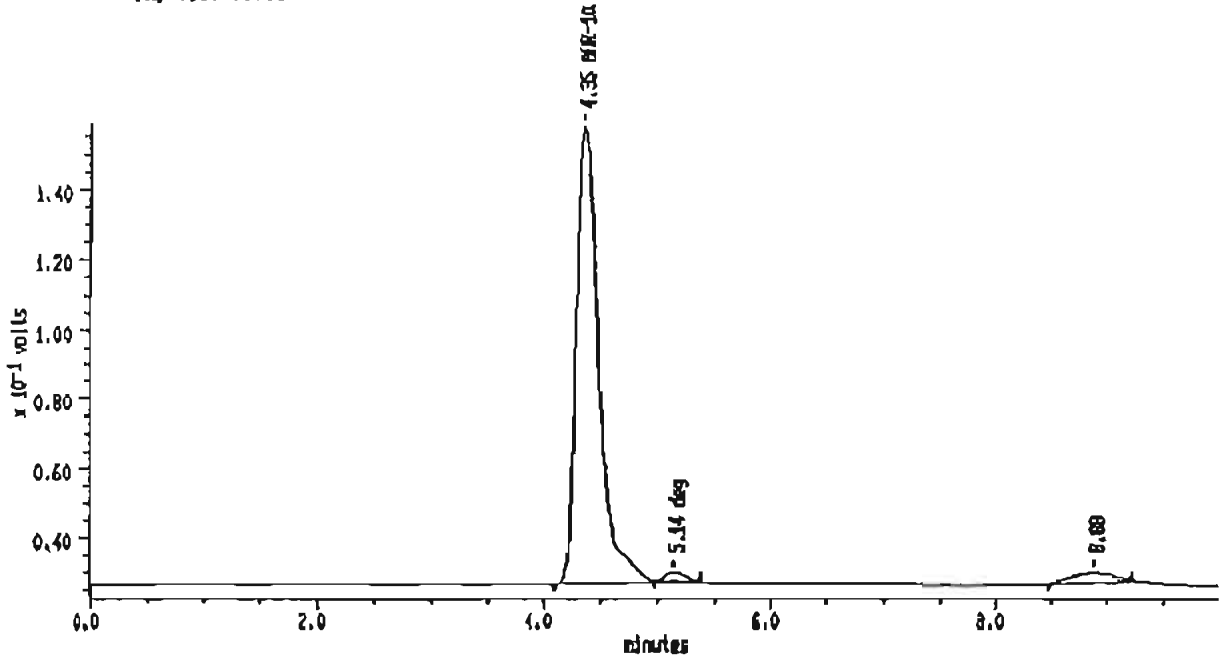
Index: 46

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	BB	1860867	134741	99.02	AHR-10282B
2	8.975	BB	16307	1393	0.88	deg
TOTAL			1889174	138138		

Sample: A35 60°C-4W Channel: detector 1 Pilename: V2-21 Chart Speed: Full Size
 Acquired: 24-MAR-2001 1:35 Method: B:VAHRVIKI0070-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 28-MAR-2001 12:18:49

SAMPLE: A35 60°C-4W

#21 In Method: AHR-10282B

Acquired: 24-MAR-2001 1:36

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pilename: V2-21

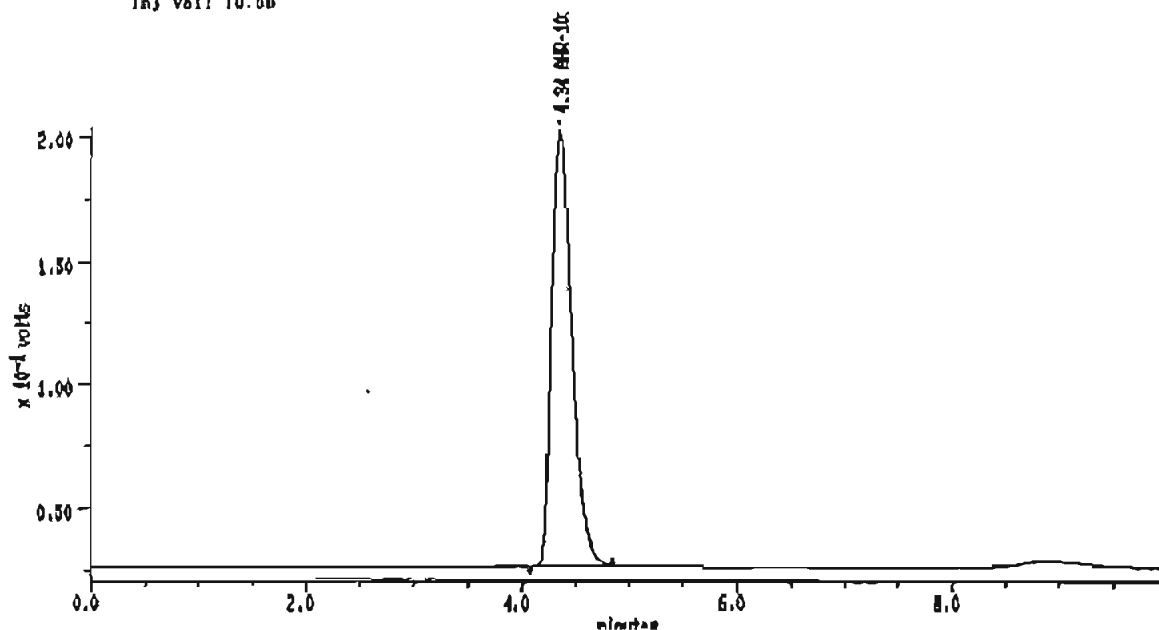
Index: 48

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	BB	1813088	130724	98.53	AHR-10282B
2	5.142	SS	27101	2288	1.47	deg
TOTAL			1840188	133010		

Sample: STD2 Channel: detector 1 Filename: V2-22 Chart Speed: Full Size
 Acquired: 24-MAR-01 1:48 Method: B:YABRVIKISV70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 26-MAR-2001 12:19:14

SAMPLE: STD2

#22 In Method: AHR-10282B

Acquired: 24-MAR-2001 1:48

Rate: 2.0 points/sec

Duration: 16.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-22

Index: 10

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.342	BB	2319988	176137	100.00	AHR-10282B
TOTAL			2319988	176137		

24

Bronuck Ophthalmic Solution Stability Test
 Lot No. 01K131

Test code: P2000B177
 Tester: Shirou Sawa
 Test date: 20 February 2001

	ID	Chromato No	Peak Area	Cono. (mg/mL)	Remaining (%)	Water Collec(%)	Initial	present	
STD	1	INI-01	2286918						
STD	2	INI-32	2278153						
STD	mean		2281536	0.9950					
A-26	Initial	INI-02	2293965	1.0004	100				
A-27	Initial	INI-03	2282847	0.9888	100				
A-28	Initial	INI-04	2274923	0.9921	100				
A-29	Initial	INI-05	2369117	1.0332	100				
A-30	Initial	INI-06	2326638	1.0147	100				
A-31	Initial	INI-07	2328545	1.0155	100				
A-32	Initial	INI-08	2412438	1.0521	100				
A-33	Initial	INI-09	2298387	1.0023	100				
A-34	Initial	INI-10	2300355	1.0032	100				
A-35	Initial	INI-11	2317836	1.0108	100				
A-26	70°C-1W	INI-12	2132144	0.9298	92.94	89.48	3.71	8.3864	8.1916
A-27	70°C-1W	INI-13	2253984	0.9830	99.81	95.88	3.74	8.3824	8.2055
A-28	70°C-1W	INI-14	2243981	0.9786	98.84	95.01	3.68	8.4773	8.2997
A-29	70°C-1W	INI-15	2312544	1.0085	97.61	94.30	3.39	8.4742	8.3106
A-30	70°C-1W	INI-16	2258811	0.9850	97.07	93.45	3.73	8.5162	8.3347
A-31	70°C-1W	INI-17	2195956	0.9577	94.31	91.14	3.36	8.5820	8.4165
A-32	70°C-1W	INI-18	2197422	0.9583	91.08	87.70	3.71	8.4924	8.3126
A-33	70°C-1W	INI-19	2186825	0.9537	95.15	91.55	3.78	8.4307	8.2501
A-34	70°C-1W	INI-20	2165761	0.9445	94.16	90.90	3.45	8.4785	8.3119
A-35	70°C-1W	INI-21	2115392	0.9225	91.28	87.80	3.79	8.5490	8.3635
A-26	60°C-1W	INI-22	2117458	0.9234	92.30	90.67	1.77	8.3539	8.2705
A-27	60°C-1W	INI-23	2250178	0.9813	99.44	97.70	1.75	8.4678	8.3837
A-28	60°C-1W	INI-24	2211902	0.9646	97.23	95.47	1.81	8.4976	8.4099
A-29	60°C-1W	INI-25	2290389	0.9989	96.88	95.15	1.58	8.5264	8.4495
A-30	60°C-1W	INI-26	2227585	0.9715	95.74	94.19	1.82	8.4971	8.4186
A-31	60°C-1W	INI-27	2170690	0.9487	93.23	91.58	1.77	8.5383	8.4500
A-32	60°C-1W	INI-28	2243853	0.9786	93.01	91.35	1.79	8.4164	8.3310
A-33	60°C-1W	INI-29	2193124	0.9564	95.42	93.75	1.75	8.4580	8.3718
A-34	60°C-1W	INI-30	2153242	0.9390	93.60	92.08	1.62	8.4995	8.4210
A-35	60°C-1W	INI-31	2116180	0.9229	91.30	89.65	1.81	8.6081	8.5163

*Re-edited in order to document the data necessary for calculation.
 Shirou Sawa, 6 May 2005*

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2000B177	Test date	20 February 200001		
Test item					Tester	Shirou Sawa	
STD 0.01990g AHR10282B/ 20mL x 2mL/20mLMP				- 20-Feb-2001 . 14:30:25			
				001: ... + 0:01990g			
					Turbidity	Foreign matter	Color
A0126	Initial	7.05			± -	-	Dark yellow
A0227		7.10			-	-	↓
A28		7.10			-	-	↓
A29		7.08			-	-	↓
A30		7.09			-	-	↓
A31		7.06			-	-	↓
A32		7.05			-	-	↓
A33		7.08			-	-	↓
A34		7.07			-	-	↓
A35		7.12			-	-	↓
A26	70°C- 2W1W *1	7.02	8.3664	8.1916	±	+	Dark yellow
A27		7.05	8.3824	8.2055	+ (r)	-	Yellow
A28		7.03	8.4773	8.2997	+	+	Yellow
A29		7.03	8.4742	8.3106	±	+	Yellow
A30		7.02	8.5162	8.3347	±	+	Yellow
A31		7.01	8.5820	8.4165	±	+	Yellow
A32		7.04	8.4924	8.3126	+ (r)	±	Dark yellow
A33		7.10	8.4307	8.2501	±	+	Yellow
A34		7.10	8.4785	8.3119	±	+	Yellow
A35		7.10	8.5490	8.3635	±	+	Yellow
A26	60°C- 2W1W *1	7.02	8.3539	8.2705	±	+	Dark yellow
A27		7.08	8.4678	8.3837	+	±	Yellow
A28		7.04	8.4976	8.4099	±	±	Yellow
A29		7.09	8.5264	8.4495	-	±	Yellow
A30		7.08	8.4971	8.4186	-	+	Yellow
A31		7.05	8.5363	8.4500	±	+	Yellow
A32		7.05	8.4164	8.3310	+ (r)	±	Dark yellow
A33		7.06	8.4560	8.3718	±	+	Yellow
A34		7.06	8.4995	8.4210	±	+	Yellow
A35		7.10	8.6061	8.5163	±	+	Yellow

*1 Correction of error
28 April 2003 Sawa

2/20 16:27
NO.21 PH 7.85
24.1°C

2/20 16:28
NO.22 PH 7.10
24.3°C

2/20 16:30
NO.23 PH 7.13
24.3°C

2/20 16:32
NO.24 PH 7.00
24.4°C

2/20 16:33
NO.25 PH 7.09
24.5°C

2/20 16:34
NO.26 PH 7.06
24.3°C

2/20 16:35
NO.27 PH 7.05
24.3°C

2/20 16:36
NO.28 PH 7.86
24.4°C

2/20 16:36
NO.29 PH 7.07
24.5°C

2/20 16:38
NO.30 PH 7.12
24.4°C

2/20 16:39
NO.31 PH 7.02
24.1°C

2/20 16:40
NO.32 PH 7.05
24.5°C

2/20 16:41
NO.33 PH 7.03
24.6°C

2/20 16:42
NO.34 PH 7.03
24.5°C

2/20 16:43
NO.35 PH 7.02
24.6°C

2/20 16:44
NO.36 PH 7.01
24.6°C

2/20 16:45
NO.37 PH 7.04
24.5°C

2/20 16:46
NO.38 PH 7.10
24.6°C

2/20 16:48
NO.39 PH 7.10
24.5°C

2/20 16:49
NO.40 PH 7.10
24.4°C

2/20 16:50
NO.41 PH 7.05
24.6°C

2/20 16:51
NO.42 PH 7.01
24.6°C

2/20 16:52
NO.43 PH 7.04
24.6°C

2/20 16:53
NO.44 PH 7.09
24.6°C

2/20 16:53
NO.45 PH 7.08
24.5°C

2/20 16:54
NO.46 PH 7.05
24.7°C

2/20 16:55
NO.47 PH 7.03
24.5°C

2/20 16:56
NO.48 PH 7.06
24.6°C

2/20 16:56
NO.49 PH 7.06
24.4°C

2/20 16:57
NO.50 PH 7.10
24.5°C

Preparation Record B (drug solution) Form 7 (15 November 2000)

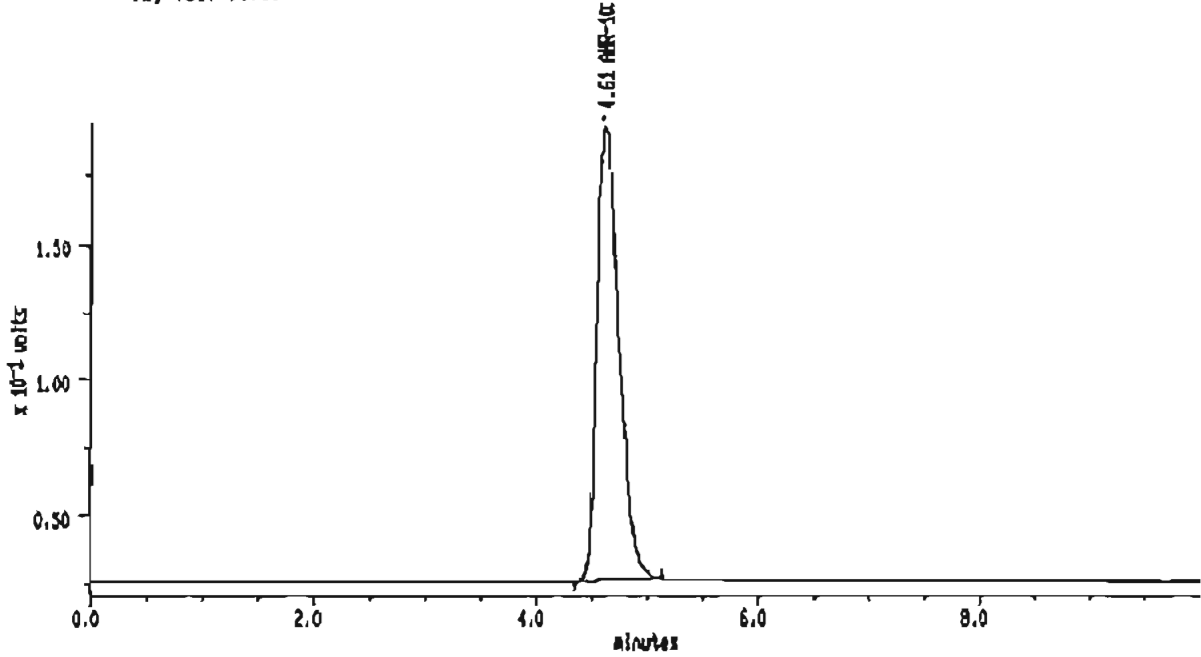
Test substance	Test code		Lot No.		Preparation date		Tester		Shiron Sawa		
Test item											
Amount manufactured		5mL colorless ampoules ×		5mL colorless PP ×		5mL brown PP ×		×		×	
Formulation No.	26		27		28		29		30		
Ingredients and amounts	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Manufacturer Lot No.
Ingredient	mL		mL		mL						
<i>Bronfenac sodium</i>	0.1	0.101	0.1	0.102	0.1	0.101	0.1	0.100	0.1	0.100	
<i>Boric acid</i>	1.6	1.601	1.6		1.6	1.603	1.6	1.598	1.6	1.599	
<i>Benzalkonium chloride</i>			0.005		0.005		0.005		0.005		
<i>Polysorbate 80</i>											
<i>Tylaxapol</i>	0.15	0.150	0.02	0.01944	0.05	0.05446	0.1	0.10024	0.15	0.15108	
<i>Methylglucamine</i>									0.1	0.101	

NO. 7		
NO. 13	1:27	2
NO. 14		
NO. 15	1:30	
NO. 16		
NO. 17		
NO. 18		
NO. 19		
NO. 20		
NO. 21		
NO. 22		
NO. 23		
NO. 24		
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NO. 95		
NO. 96		
NO. 97		
NO. 98		
NO. 99		
NO. 100		

Preparation Record B (drug solution) Form 7 (15 November 2000)

Test substance		Test code		Lot No.		Preparation date	MMDDYY	Tester	Shirou Sawa		
Test item											
Amount manufactured		5mL colorless ampoules ×		5mL colorless PP ×		5mL brown PP ×		×		×	
Formulation No.	31		32		33		34		35		
Ingredients and amounts	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Manufacturer Lot No.
Ingredient	mL		mL		mL						
<i>Bromfenac sodium</i>	0.1	0.102	0.1	0.102	0.1	0.100	0.1	0.100	0.1	0.101	
<i>Boric acid</i>	1.6	1.596	1.6	1.610	1.6	1.606	1.6	1.596	1.6	1.598	
<i>Benzalkonium chloride</i>	0.005		0.005		0.005		0.005		0.005		
<i>Tyloxapol</i>	0.15	0.14984	0.15	0.14980	0.15	0.15152	0.15	0.14995	0.15	0.14961	
<i>Methylglucamine</i>	1.0	0.999									
<i>Sorbic acid</i>			0.3	0.302							
<i>Dipotassium glycyrrhizate</i>					0.1	0.100					
<i>Epsilon-Aminocaproic acid</i>							0.5	0.499			
<i>Aminoethylsulfonic Acid</i>									0.5	0.502	

Sample: STD1 Channel: detector 1 Filename: INI-01 Chart Speed: Full Size
 Acquired: 20-FEB-01 18:27 Method: B:VAHRVIX13YINITIAL Operator: S.S
 Inj Vol: 10.00



MAXIMA is 1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 20-FEB-2001 18:37:33

SAMPLE: STD1

#4 in Method: AHR-10282B

Acquired: 20-FEB-2001 18:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-01

Index: 1

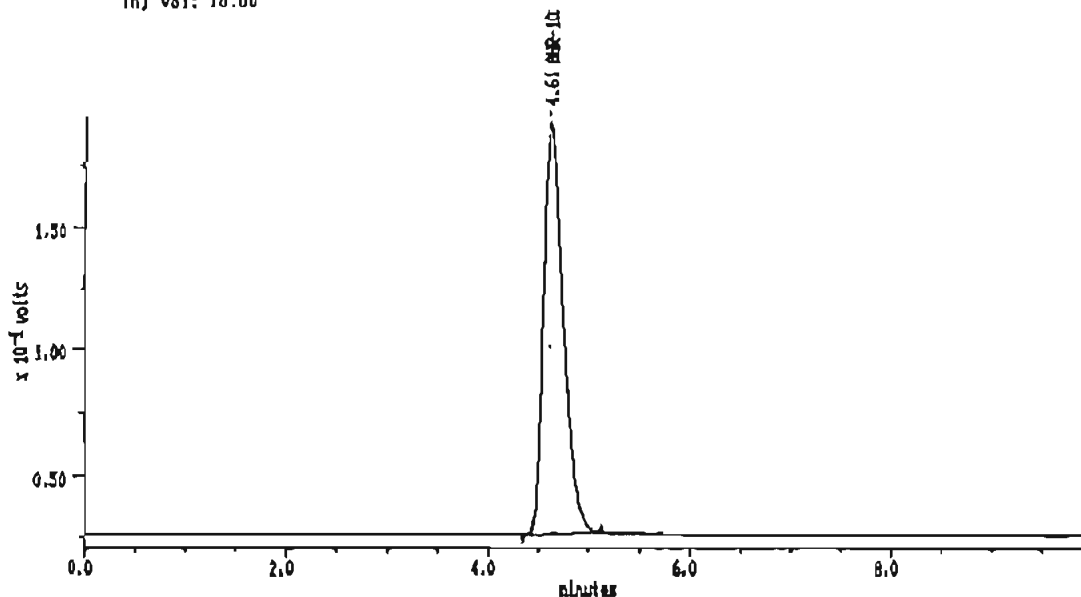
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.608	BB	2288918	185263	100.00	AHR-10282B
TOTAL			2288918	185263		

5

Sample: A28 INITIAL Channel: detector 1 Filename: IN1-02 Chart Speed: Full Size
 Acquired: 20-FEB-2001 18:38 Method: D:\AIRVIX\19\INITIAL Operator: S.S
 Inj Vol: 10.00



MAXIMA (c)1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 20-FEB-2001 18:48:46

SAMPLE: A28 INITIAL

#5 in Method: **AHR-10282B**

Acquired: 20-FEB-2001 18:38

Rate: 2.0 points/seo

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-02

Index: 2

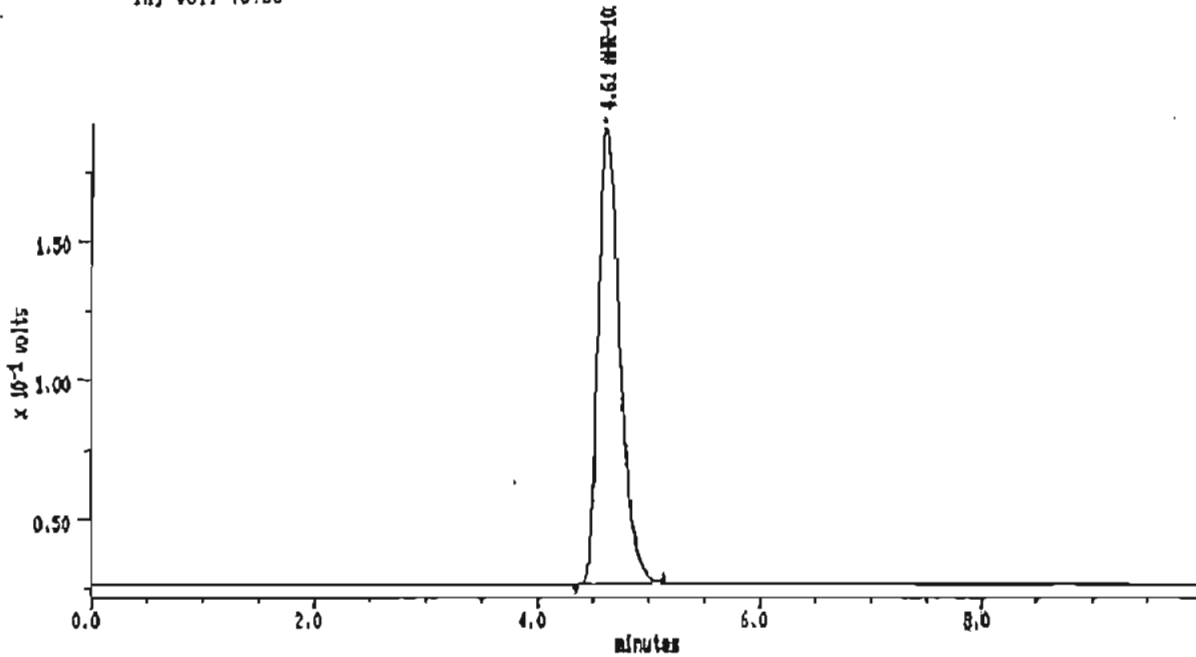
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.808	BB	2293985	185920	100.00	AHR-10282B
TOTAL			2293985	185920		

6

Sample: A27 INITIAL Channel: detector 1 Filename: INI-03 Chart Speed: Full Size
 Acquired: 20-FEB-2001 18:48 Method: 8:YAHROW\K13WINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 18:00:03

SAMPLE: A27 INITIAL

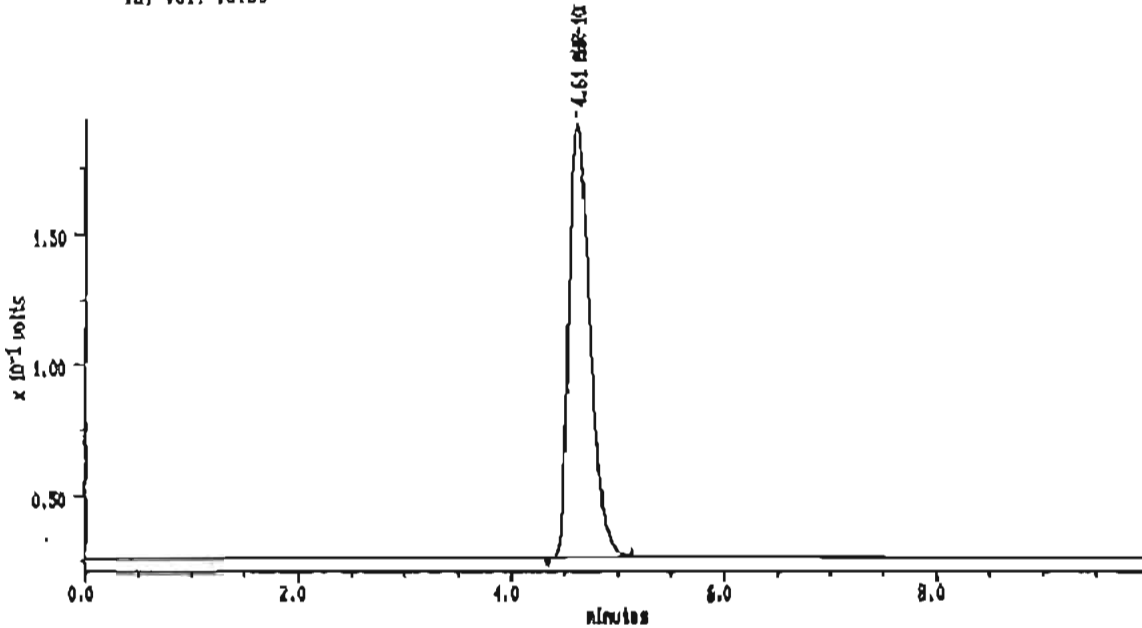
#8 In Method: AHR-10282B
 Acquired: 20-FEB-2001 18:48
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: INI-03
 Index: 3
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.608	BB	2262847	183883	100.00	AHR-10282B
TOTAL			2262847	183883		

Sample: A28 INITIAL Channel: detector 1 Filename: INI-04 Chart Speed: Full Size
 Acquired: 20-FEB-01 10:00 Method: DIVAJRYIKISYINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 19:11:20

SAMPLE: A28 INITIAL

#7 in Method: AHR-10282B

Acquired: 20-FEB-2001 19:00

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URMW

Instrument: Instrument 1

Filename: INI-04

Index: 4

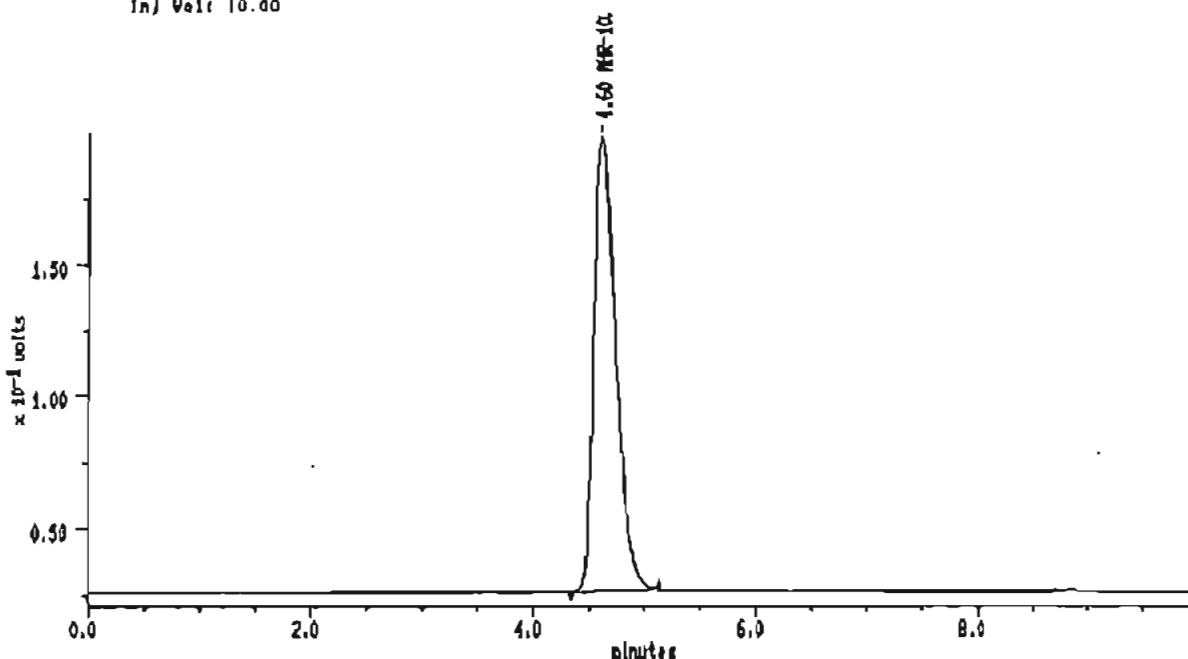
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.808	BB	2274923	184712	100.00	AHR-10282B
TOTAL			2274923	184712		

8

Sample: A29 INITIAL Channel: detector 1 Filename: INI-05 Chart Speed: Full Size
 Acquired: 20-FEB-2001 19:12 Method: B:VAHRV/R13V/INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 19:22:37

SAMPLE: A29 INITIAL

#8 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-05

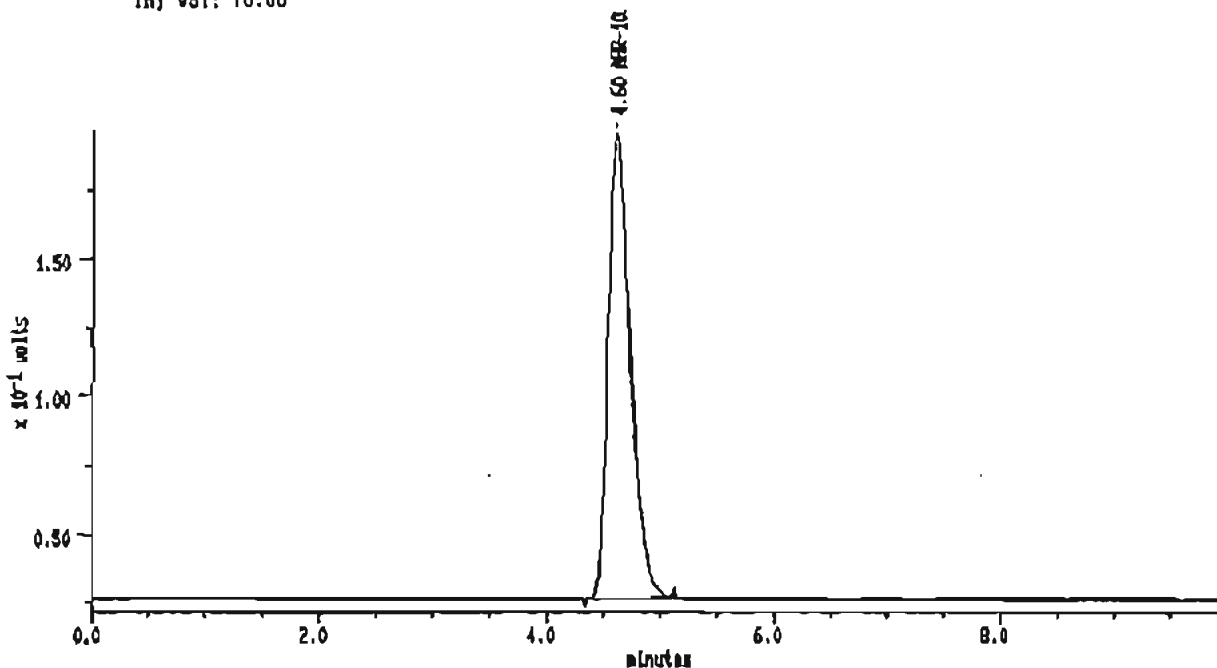
Index: 5

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.800	88	2389117	171584	100.00	AHR-10282B
TOTAL			2389117	171584		

Sample: A30 INITIAL Channel: detector 1 Filename: INI-08 Chart Speed: Full Size
 Acquired: 20-FEB-10 18:23 Method: B:YAIRVIX13VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 18:33:54

SAMPLE: A30 INITIAL

as In Method: AHR-10282B

Acquired: 20-FEB-2001 18:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-08

Index: 8

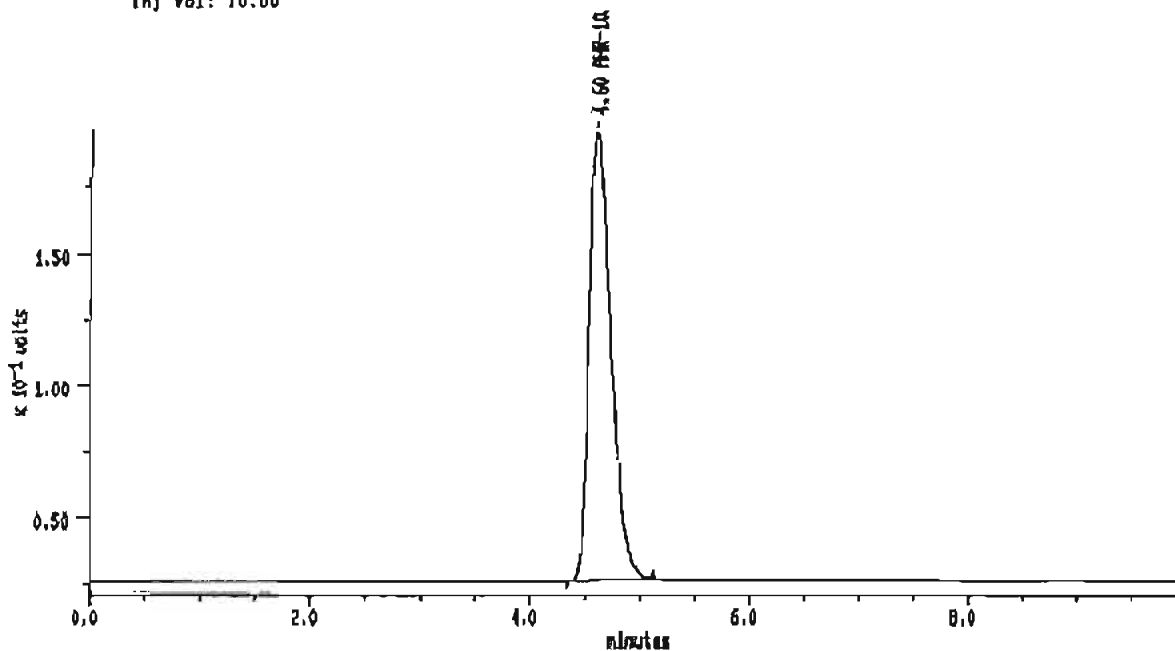
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2328838	188694	100.00	AHR-10282B
TOTAL			2328838	188694		

10

Sample: A31 INITIAL Channel: detector 1 Filename: INI-07 Chart Speed: Full Size
 Acquired: 20-FEB-2001 19:34 Method: D:YAHRYJK13YINITIAL Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 18:45:11

SAMPLE: A31 INITIAL

#10 (n Method: **AHR-10282B**)

Acquired: 20-FEB-2001 19:34

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-07

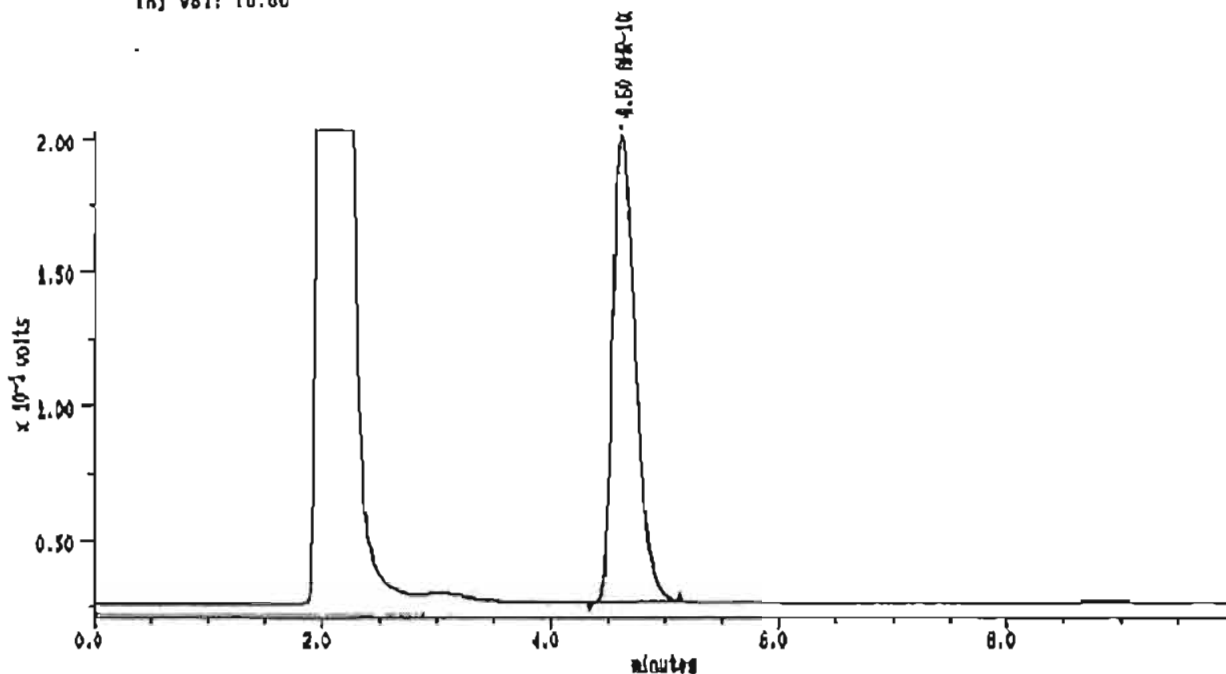
Index: 7

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2328548	188266	100.00	AHR-10282B
TOTAL			2328548	188266		

Sample: A32 INITIAL Channel: detector 1 Filename: INI-00 Chart Speed: Full Size
 Acquired: 20-FEB-01 19:45 Method: B:YAHRYIKI3VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 18:58:20

SAMPLE: A32 INITIAL

#1 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:45

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-00

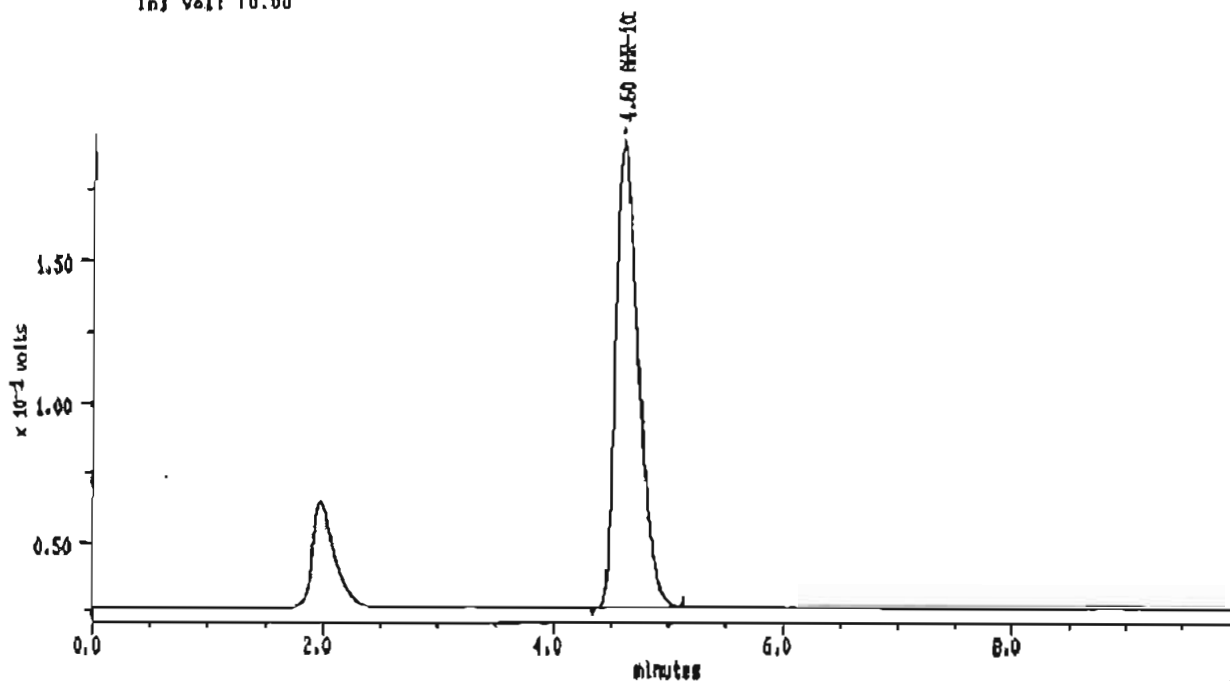
Index: 8

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.800	B0	2412438	174162	100.00	AHR-10282B
TOTAL			2412438	174162		

Sample: A33 INITIAL Channel: detector 1 Filename: INI-09 Chart Speed: Full Size
 Acquired: 20-FEB-2001 19:57 Method: B:\AHRV\K13\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 20:07:45

SAMPLE: A33 INITIAL

#12 (n Method: AHR-10282B

Acquired: 20-FEB-2001 19:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-09

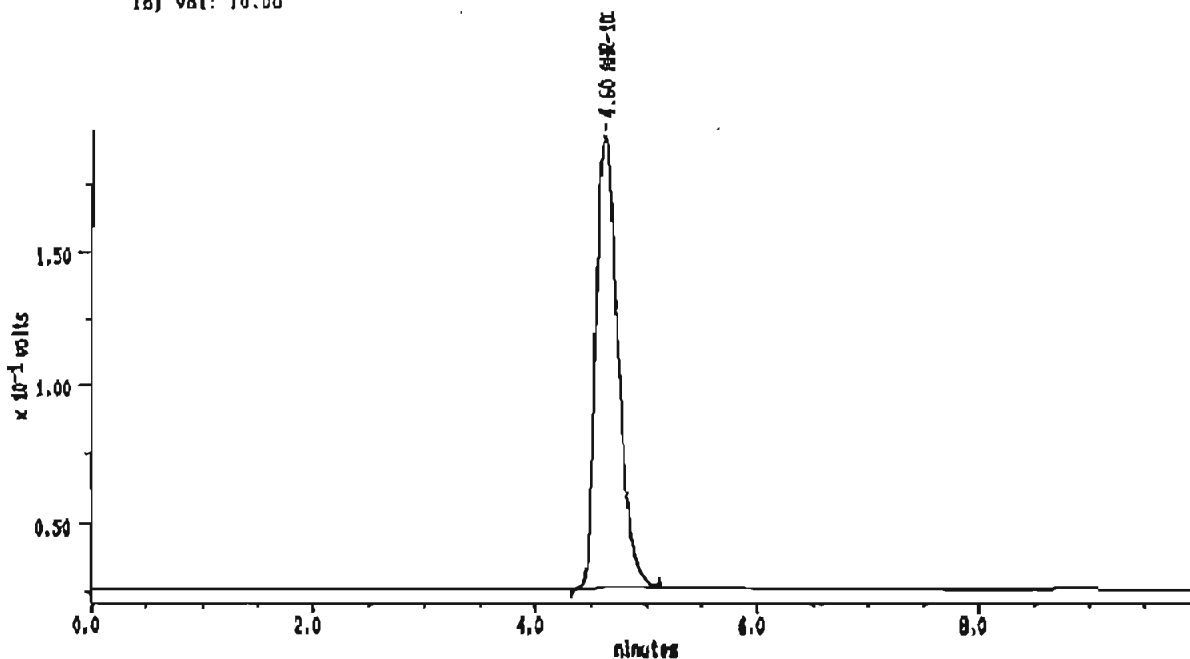
Index: 9

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2298397	166127	100.00	AHR-10282B
TOTAL			2298397	166127		

Sample: A33 INITIAL Channel: detector 1 Filename: INI-10 Chart Speed: Full Size
 Acquired: 20-FEB-2001 20:09 Method: B:VARIYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 20:19:02

SAMPLE: A33 INITIAL

#13 In Method: AHR-10282B

Acquired: 20-FEB-2001 20:08

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-10

Index: 10

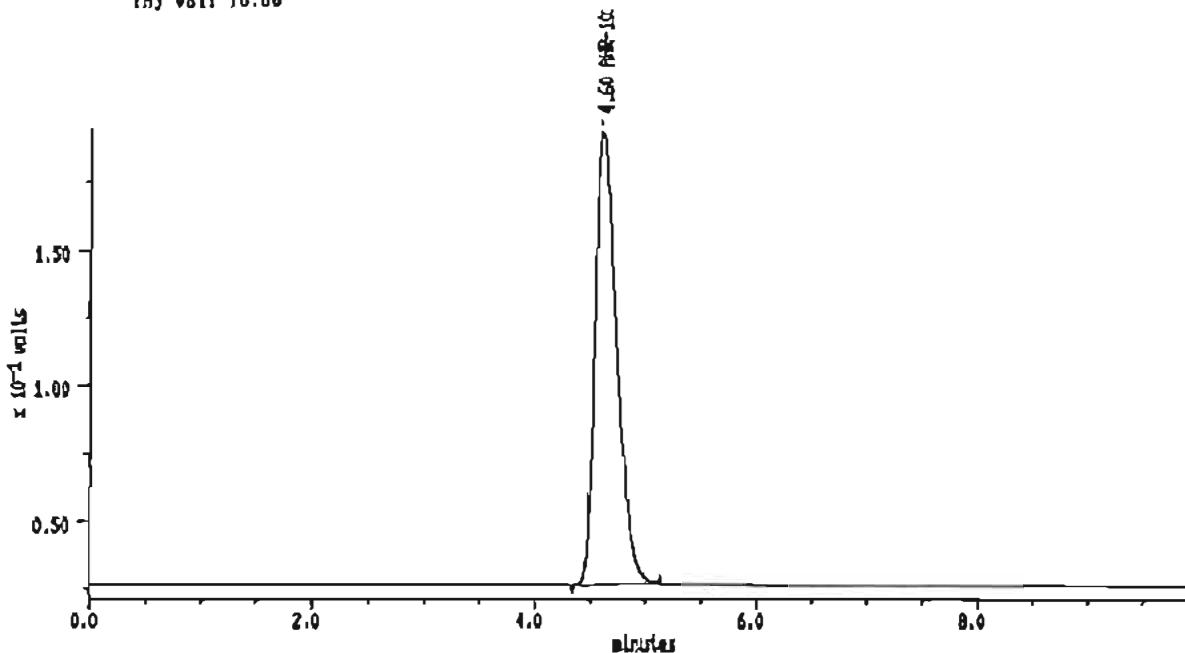
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2300356	168135	100.00	AHR-10282B
TOTAL			2300356	168135		

14

Sample: A36 INITIAL Channel: detector 1 Filename: INI-11 Chart Speed: Full Size
 Acquired: 20-FEB-2001 20:19 Method: D:VAHRV1K13V(INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 20:30:19

SAMPLE: A36 INITIAL

#14 In Method: AHR-10282B

Acquired: 20-FEB-2001 20:19

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URMN

Instrument: Instrument 1

Filename: INI-11

Index: 11

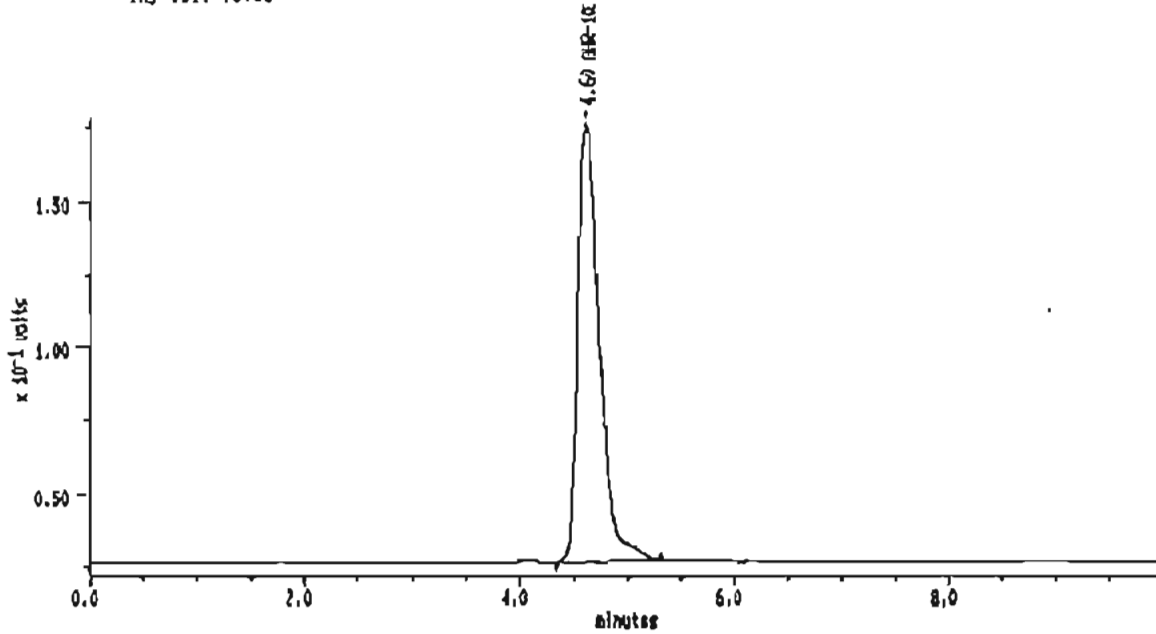
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	00	2317830	187378	100.00	AHR-10282B
TOTAL			2317830	187378		

15

Sample: A28 70°C-1W Channel: detector 1 Filename: INI-12 Chart Speed: Full Size
 Acquired: 20-FEB-10 20:31 Method: B:VAHRV(K13Y)N1TIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 20:41:38

SAMPLE: A28 70°C-1W

015 In Method: AHR-10282B

Acquired: 20-FEB-2001 20:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UHKN

Instrument: Instrument 1

Filename: INI-12

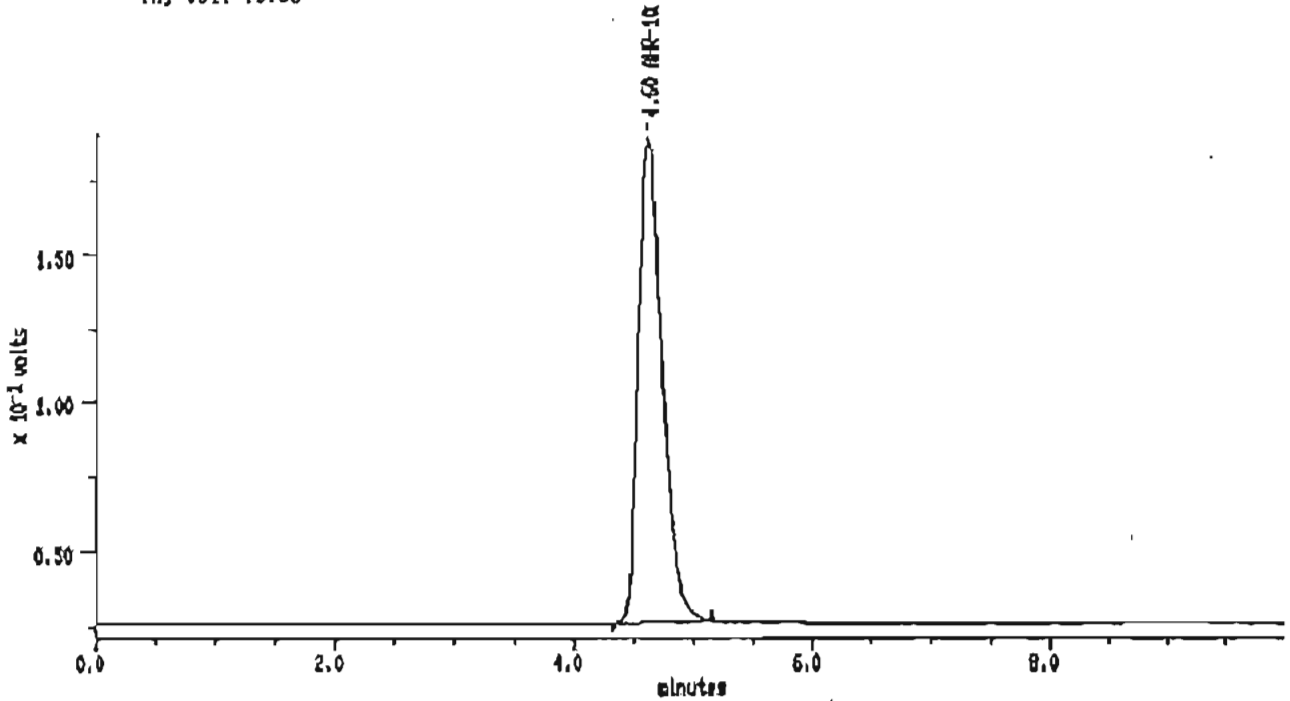
Index: 12

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2132144	140809	100.00	AHR-10282B
TOTAL			2132144	140809		

Sample: A27 70°C-1W Channel: detector 1 Filename: INI-13 Chart Speed: Full Size
 Acquired: 20-FEB-2001 20:42 Method: B:VAHRV)X13Y(INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 20:42:53

SAMPLE: A27 70°C-1W

818 In Method: AHR-10282B

Acquired: 20-FEB-2001 20:42

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-13

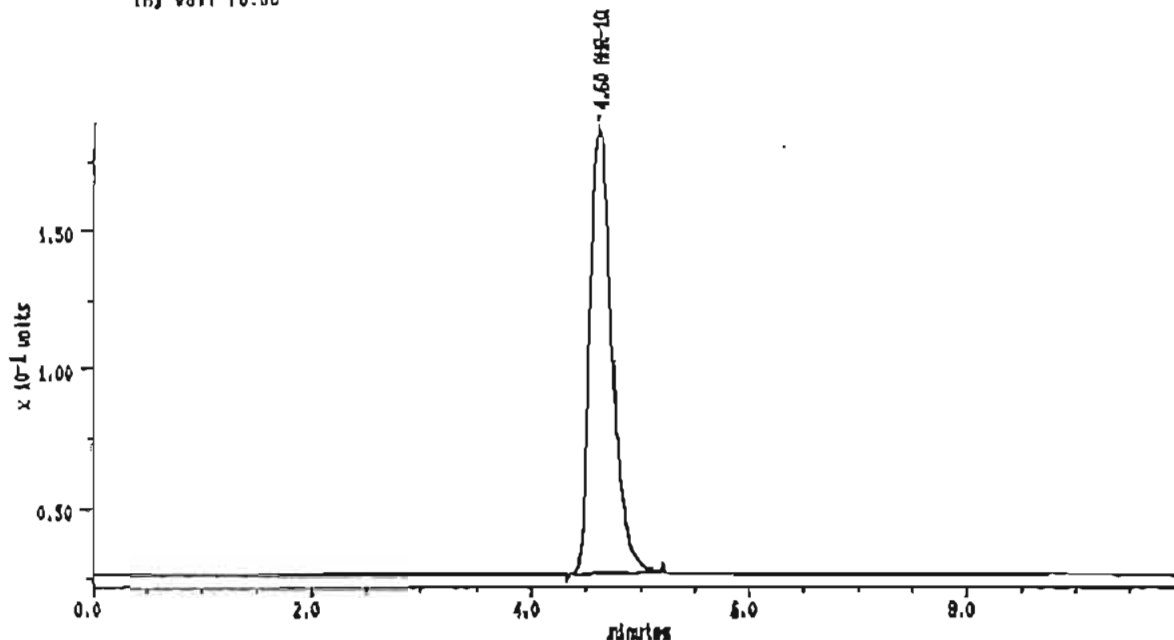
Index: 13

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	SS	2253984	162371	100.00	AHR-10282B
TOTAL			2253984	162371		

Sample: A28 70°C-14 Channel: detector 1 Filename: IM1-14 Chart Speed: Full Size
 Acquired: 20-FEB-2001 20:53 Method: B:\MIRYIK\13\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 21:04:11

SAMPLE: A28 70°C-14

#17 In Method: AHR-10282B

Acquired: 20-FEB-2001 20:53

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IM1-14

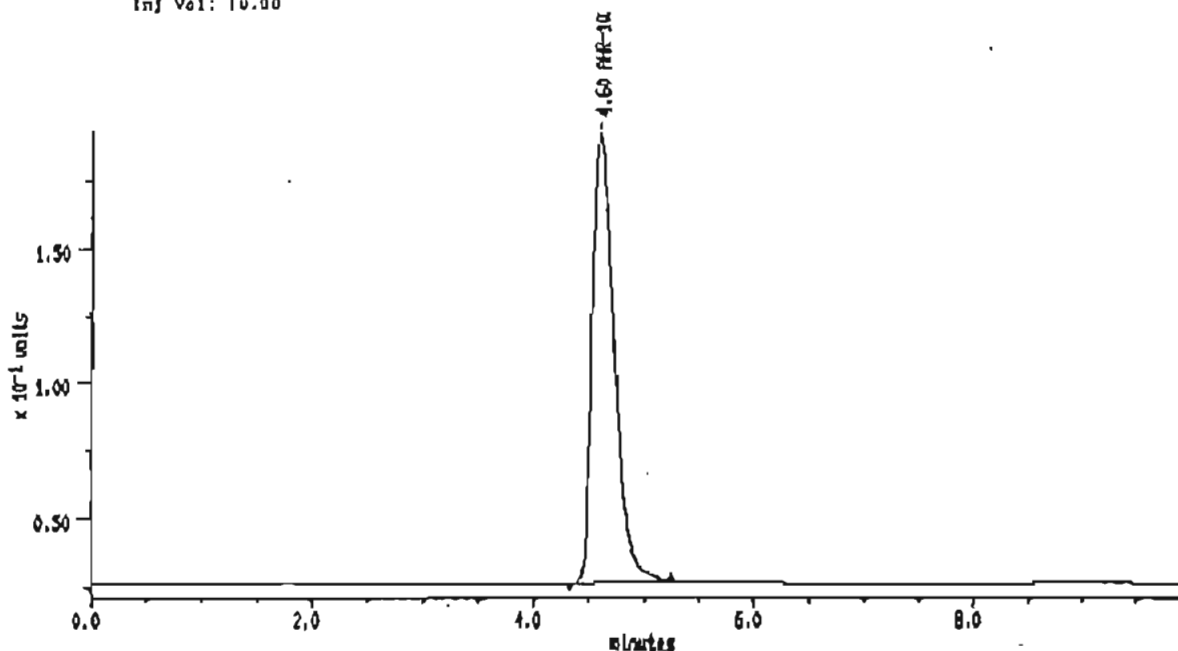
Index: 14

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.680	DB	2243981	188867	100.00	AHR-10282B
TOTAL			2243981	188867		

Sample: A29 70°C-1W Channel: detector 1 Filename: INI-16 Chart Speed: Full Size
 Acquired: 20-FEB-2001 21:04 Method: B:VAHRV1X13VINI1AL Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 21:16:28

SAMPLE: A29 70°C-1W

File In Method: AHR-10282B

Acquired: 20-FEB-2001 21:04

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

(Instrument: Instrument 1)

Filename: INI-16

Index: 16

Injection Volume: 10.0

DETECTOR: detector 1

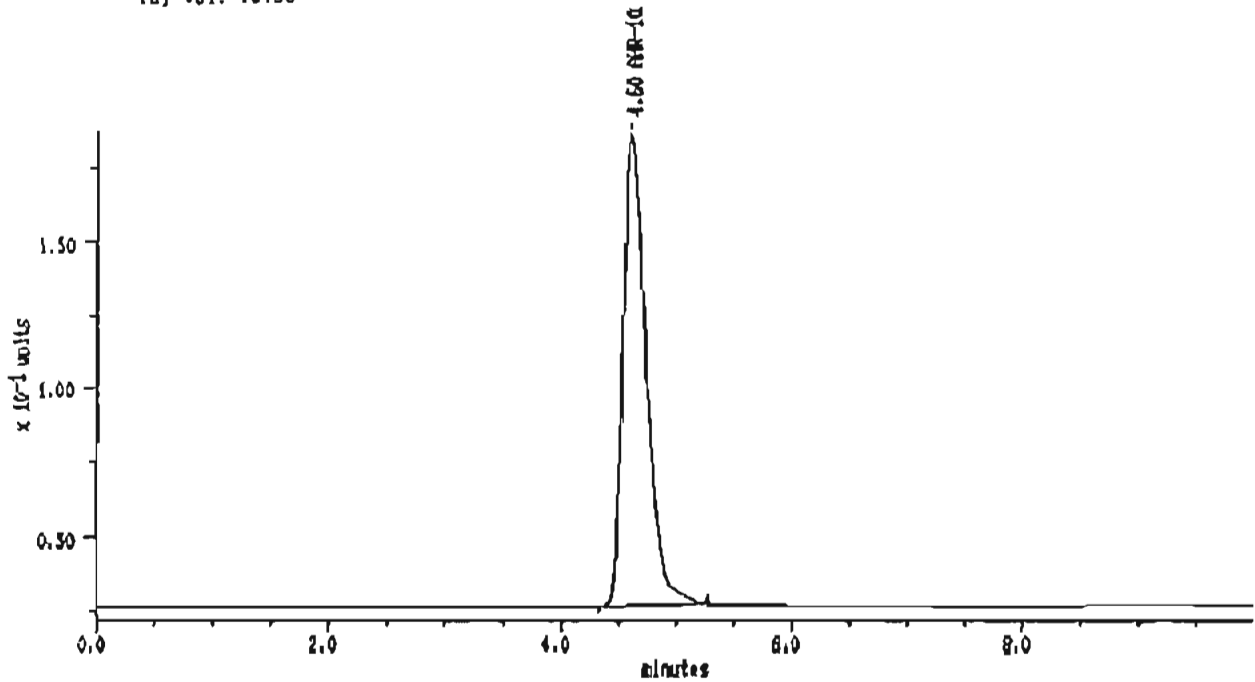
PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2312644	184798	100.00	AHR-10282B

TOTAL

2312644 184798

19

Sample: A30 70°C-1W Channel: detector 1 Filename: INT-18 Chart Speed: Full Size
 Acquired: 20-FEB-10 21:10 Method: D:\AHR\K13\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 21:28:42

SAMPLE: A30 70°C-1W

File Method: AHR-10282B

Acquired: 20-FEB-2001 21:10

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INT-18

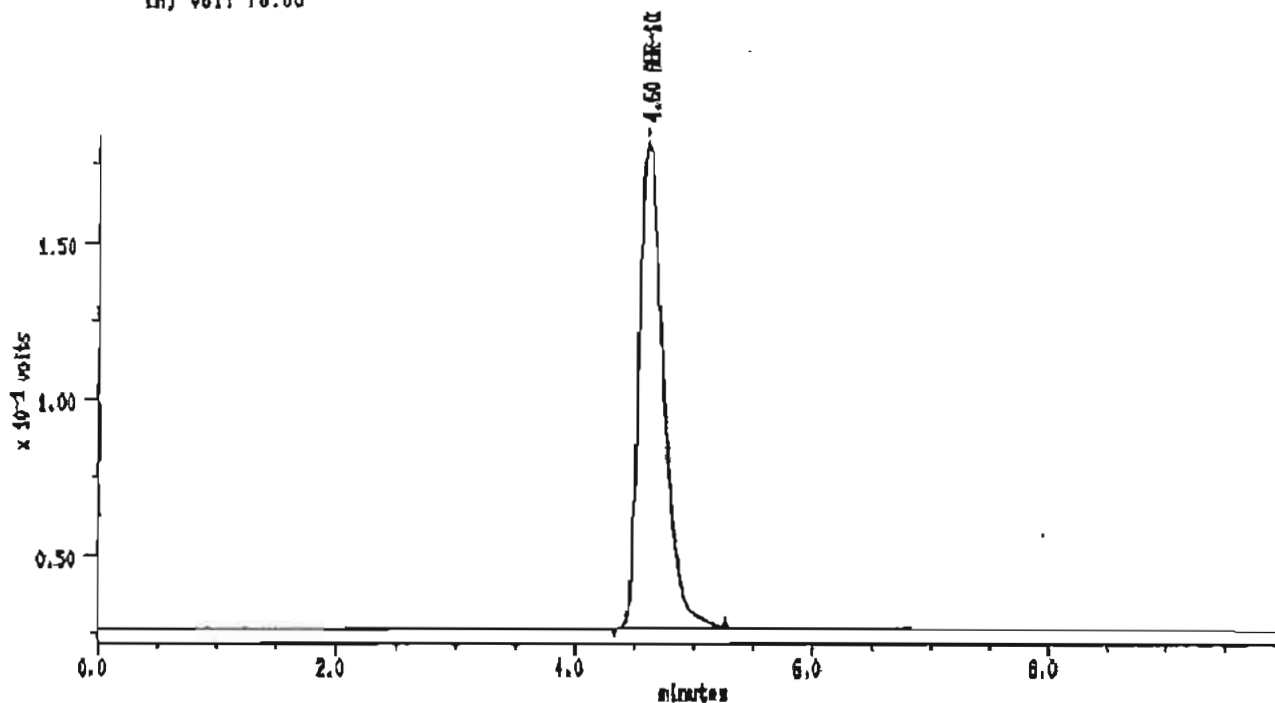
Index: 18

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	OB	2258811	180085	100.00	AHR-10282B
TOTAL			2258811	180085		

Sample: A91 70°C-1W Channel: detector 1 Filename: INI-17 Chart Speed: Full Size
 Acquired: 20-FEB-10 21:27 Method: B:VAHRYIKASVINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 21:38:00

SAMPLE: A91 70°C-1W

#20 In Method: AHR-10282B

Acquired: 20-FEB-2001 21:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UHPLC

Instrument: Instrument 1

Filename: INI-17

Index: 17

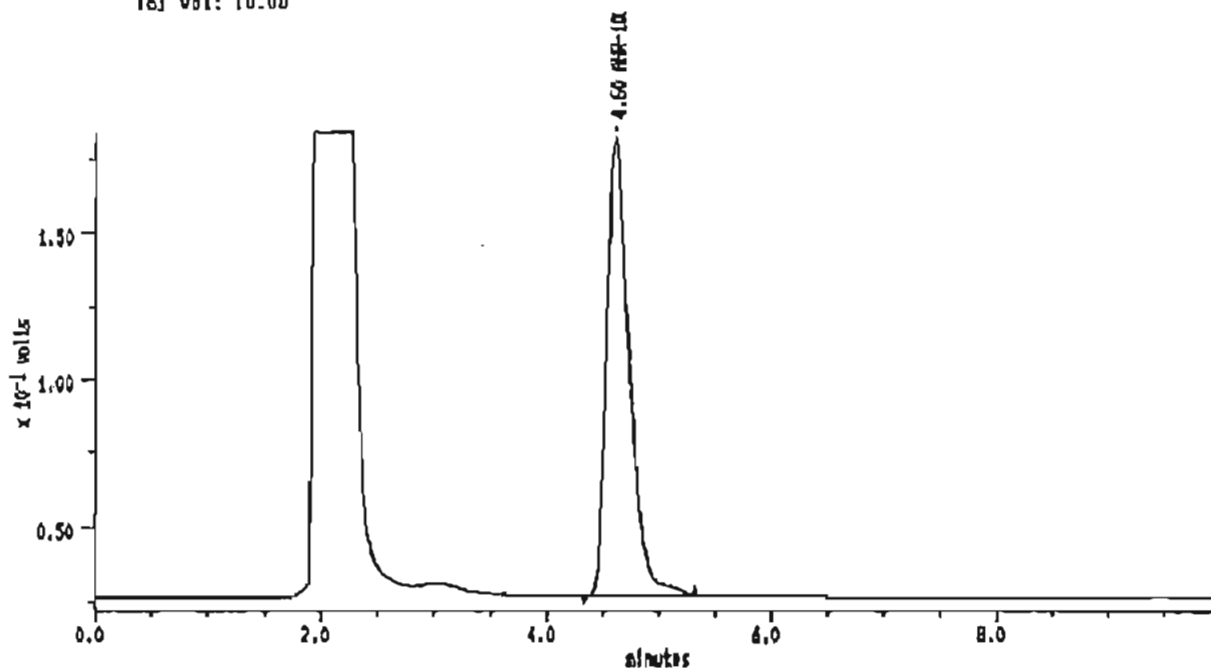
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.800	SD	2196968	166706	100.00	AHR-10202B
TOTAL			2196968	166706		

21

Sample: A32 70°C-1W Channel: detector 1 Filename: IRI-18 Chart Speed: Full Size
 Acquired: 20-FEB-2001 21:38 Method: B:YASRVIKI3VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 21:49:17

SAMPLE: A32 70°C-1W

#21 In Method: AHR-10282B

Acquired: 20-FEB-2001 21:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IRI-18

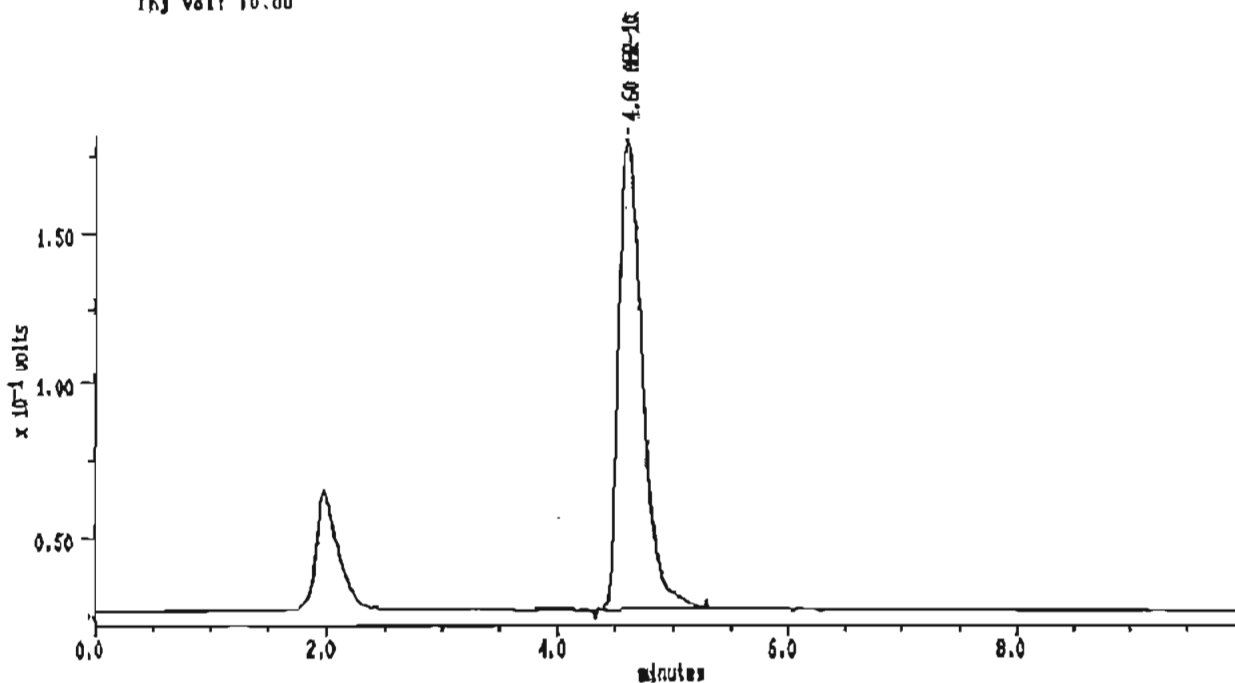
Index: 18

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2197422	156218	100.00	AHR-10282B
TOTAL			2197422	156218		

Sample: A33 70°C-1W Channel: detector 1 Filename: JMI-19 Chart Speed: Full Size
 Acquired: 20-FEB-01 21:50 Method: B:VAURVIRIKI3VINITIAL Operator: S.S
 Inj Volt: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 22:00:33

SAMPLE: A33 70°C-1W

#12 in Method: AHR-10282B

Acquired: 20-FEB-2001 21:50

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: JMI-19

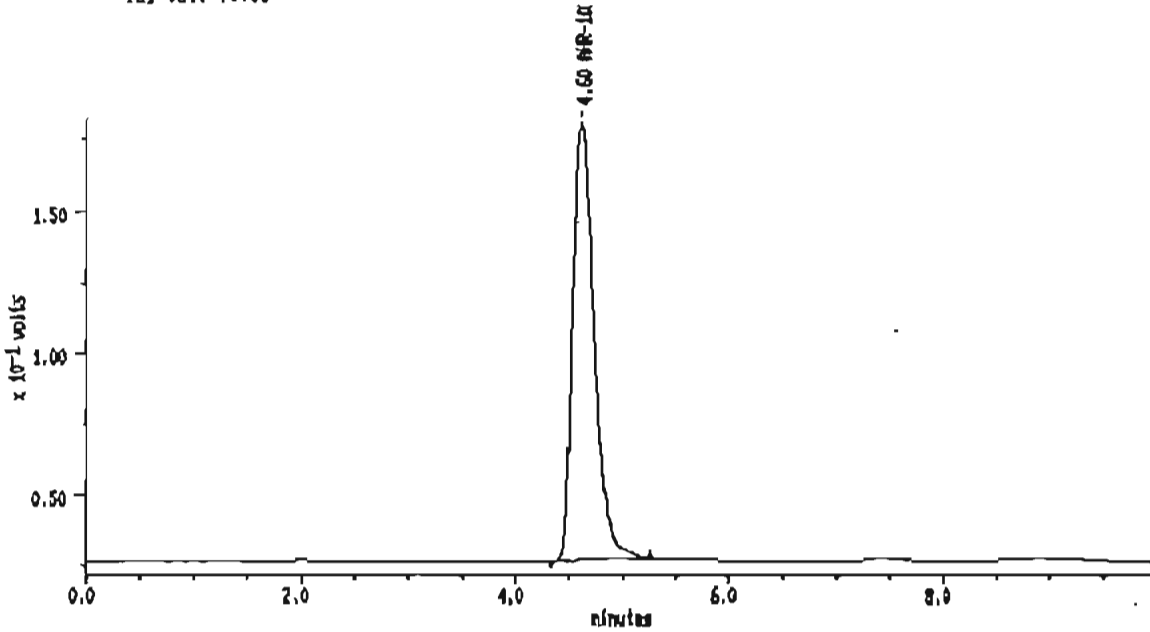
Index: 19

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2188928	163754	100.00	AHR-10282B
TOTAL			2188928	163754		

Sample: A34 70°C-1W Channel: detector 1 File Name: IN1-20 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:01 Method: B:VAIRVIXI3V\NITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 22:11:51

SAMPLE: A34 70°C-1W

#23 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: CNRM

Instrument: Instrument 1

File Name: IN1-20

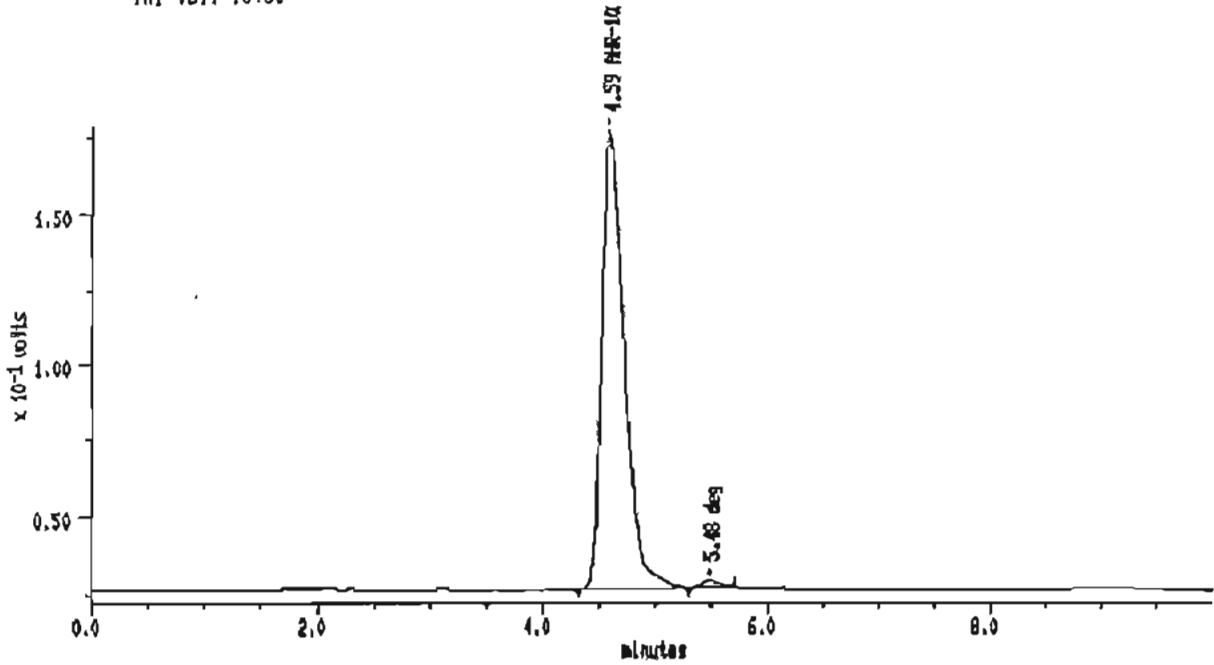
Inj Vol: 10.0

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.800	BB	2185791	154873	100.00	AHR-10282B
TOTAL			2185791	154873		

Sample: A06 70°C-1W Channel: detector 1 Filename: IN1-21 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:12 Method: B:VAHRVIM13YINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 22:23:07

SAMPLE: A06 70°C-1W

#24 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: GSKN

Instrument: Instrument 1

Filename: IN1-21

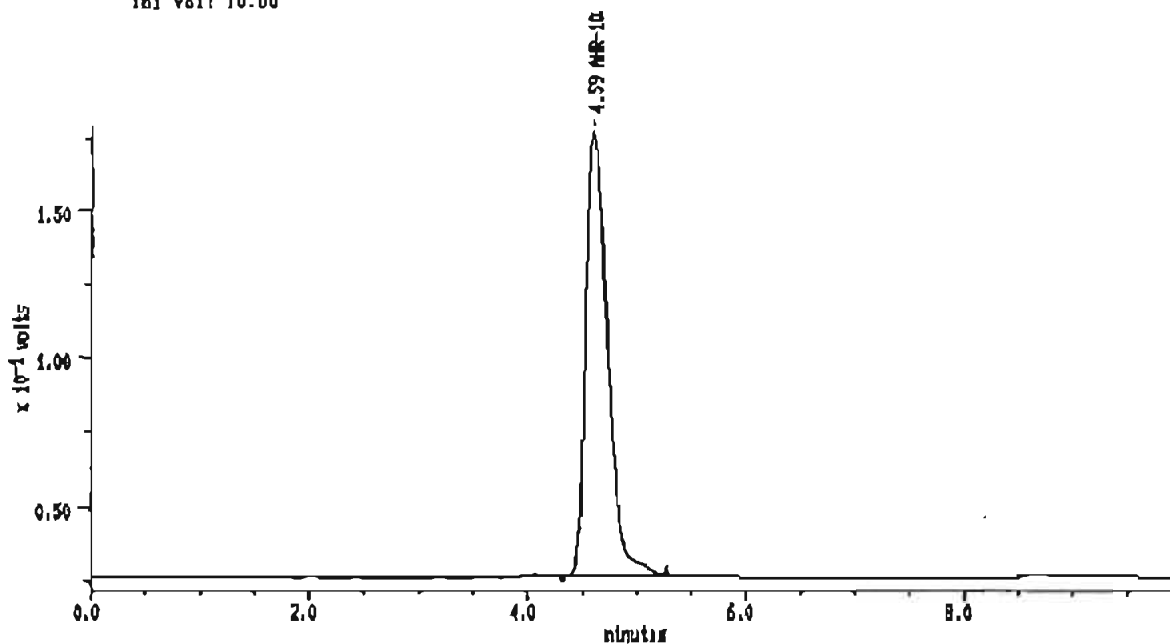
Index: 21

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.692	BB	2115392	150851	98.88	AHR-10282B
2	5.483	SS	23841	1953	1.12	deg
TOTAL			2139232	152804		

Sample: A28 80°C-1W Channel: detector 1 Filename: IN1-22 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:23 Method: 8:VAHRYXKJ3WINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 22:34:26

SAMPLE: A28 80°C-1W

#26 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: IN1-22

Index: 32

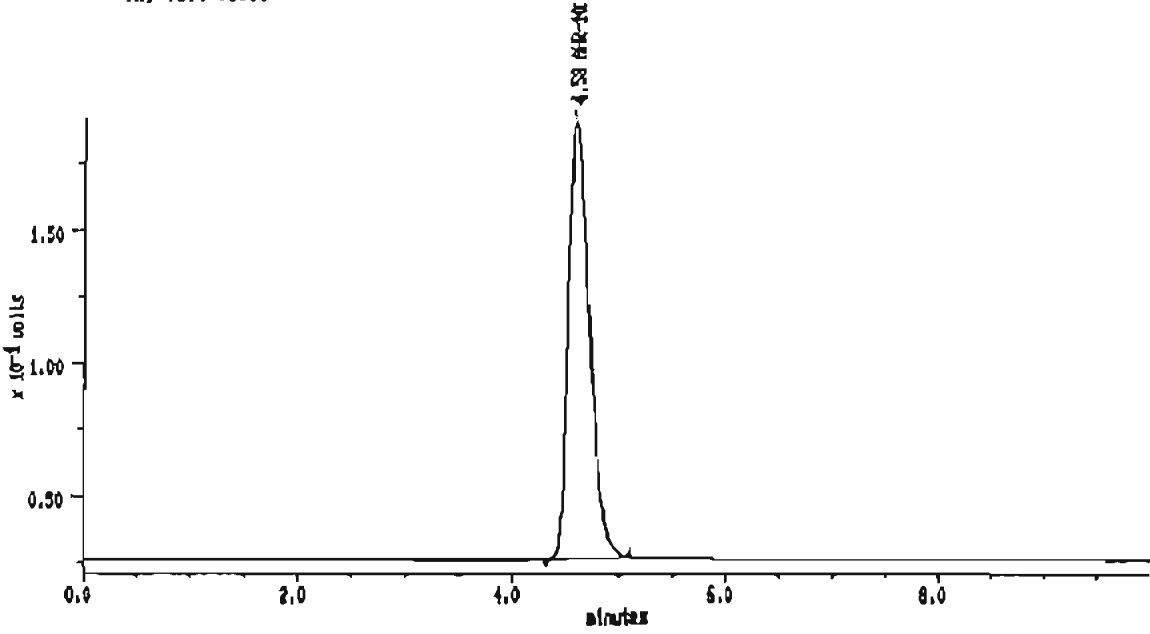
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.592	BB	2117458	150742	100.00	AHR-10282B
TOTAL			2117458	150742		

26

Sample: A27 80°C-1W Channel: detector 1 Filename: INI-23 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:36 Method: B:YAHYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 22:45:41

SAMPLE: A27 80°C-1W

#28 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:36

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-23

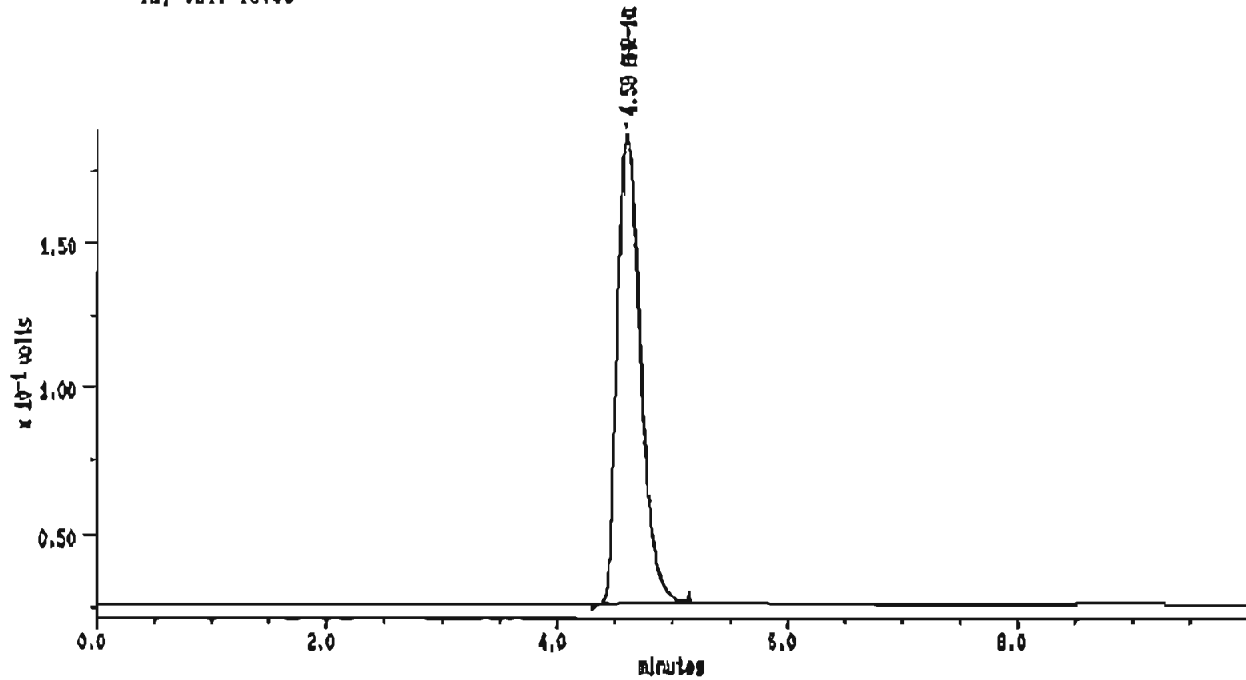
Index: 23

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	BB	2260178	163888	100.00	AHR-10282B
TOTAL			2260178	163888		

Sample: A28 80°C-1W Channel: detector 1 Filename: JN1-24 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:48 Method: B:YAKRYIK13WINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 21:58:58

SAMPLE: A28 80°C-1W

#27 in Method: AHR-10282B

Acquired: 20-FEB-2001 22:48

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: JN1-24

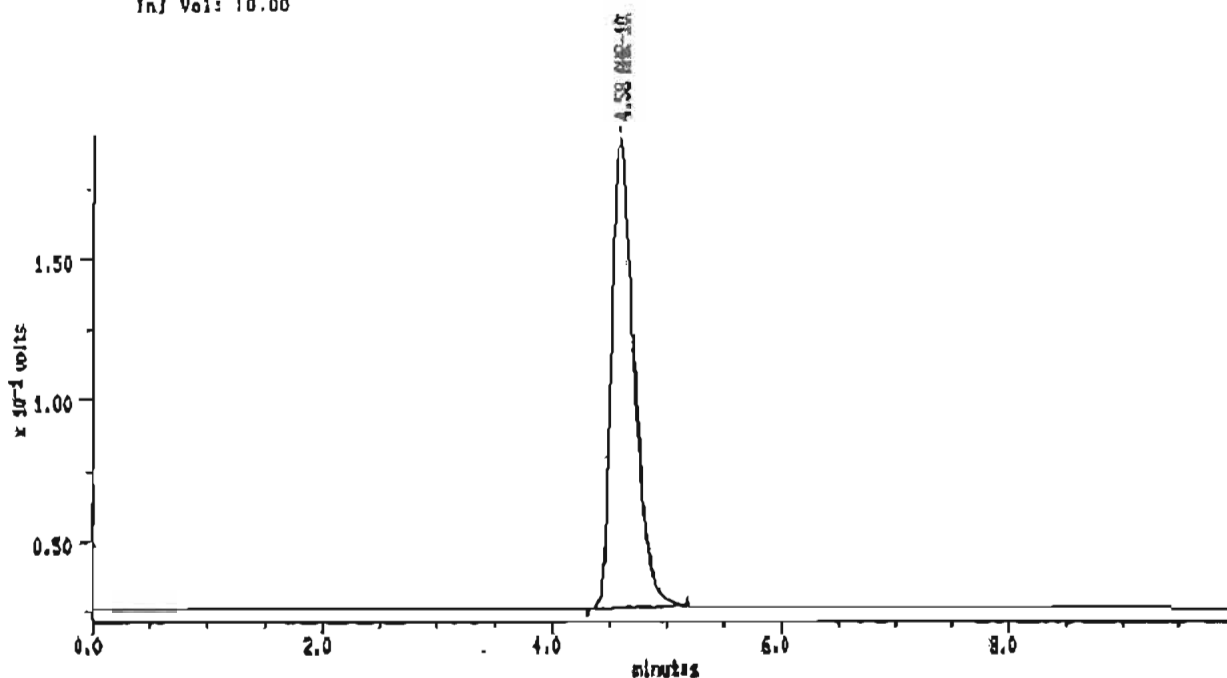
Index: 24

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	SB	2211802	180632	100.00	AHR-10282B
TOTAL			2211802	180632		

Sample: A29 80°C-1W Channel: detector 1 Filename: INI-25 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:57 Method: B:VAURYIKI3V1N1TIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 23:08:18

SAMPLE: A29 80°C-1W

#28 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: LMKR

Instrument: Instrument 1

Filename: INI-25

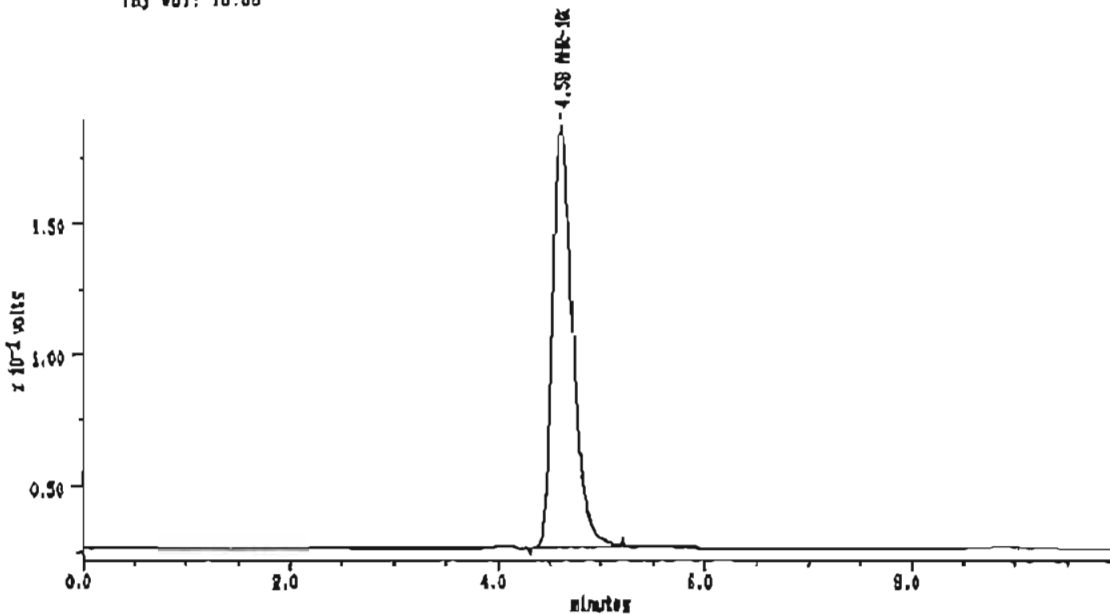
Index: 25

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	BB	2290389	165438	100.00	AHR-10282B
TOTAL			2290389	165438		

Sample: A30 80°C-1W Channel: detector 1 Filenama: INI-26 Chart Speed: Full Size
 Acquired: 20-FEB-2001 23:09 Method: B:VAHRYIXI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 24:19:32

SAMPLE: A30 80°C-1W

#29 in Method: AHR-10282B

Acquired: 20-FEB-2001 23:09

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URMH

Instrument: Instrument 1

Filenama: INI-26

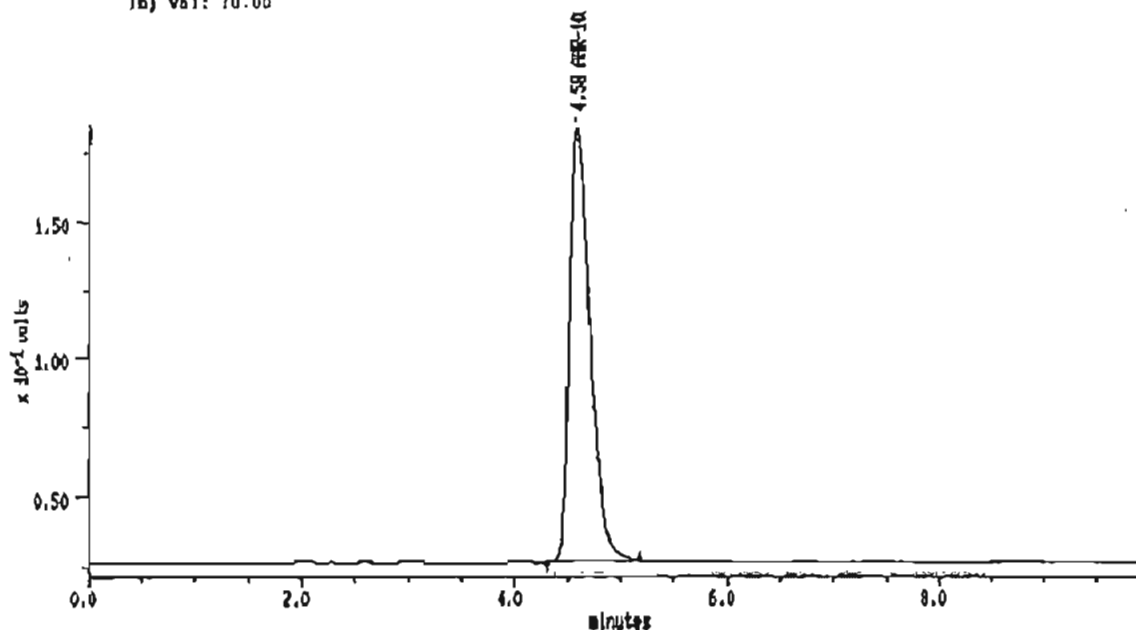
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.683	SB	2227685	180837	100.00	AHR-10282B
TOTAL			2227685	180837		

Sample: A3) 80°C-1W Channel: detector 1 Filename: IN1-27 Chart Speed: Full Size
 Acquired: 20-FEB-10) 23:20 Method: B:VAHRVIX13VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 23:30:48

SAMPLE: A3) 80°C-1W

#30 In Method: AHR-10282B

Acquired: 20-FEB-2001 23:20

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-27

Index: 27

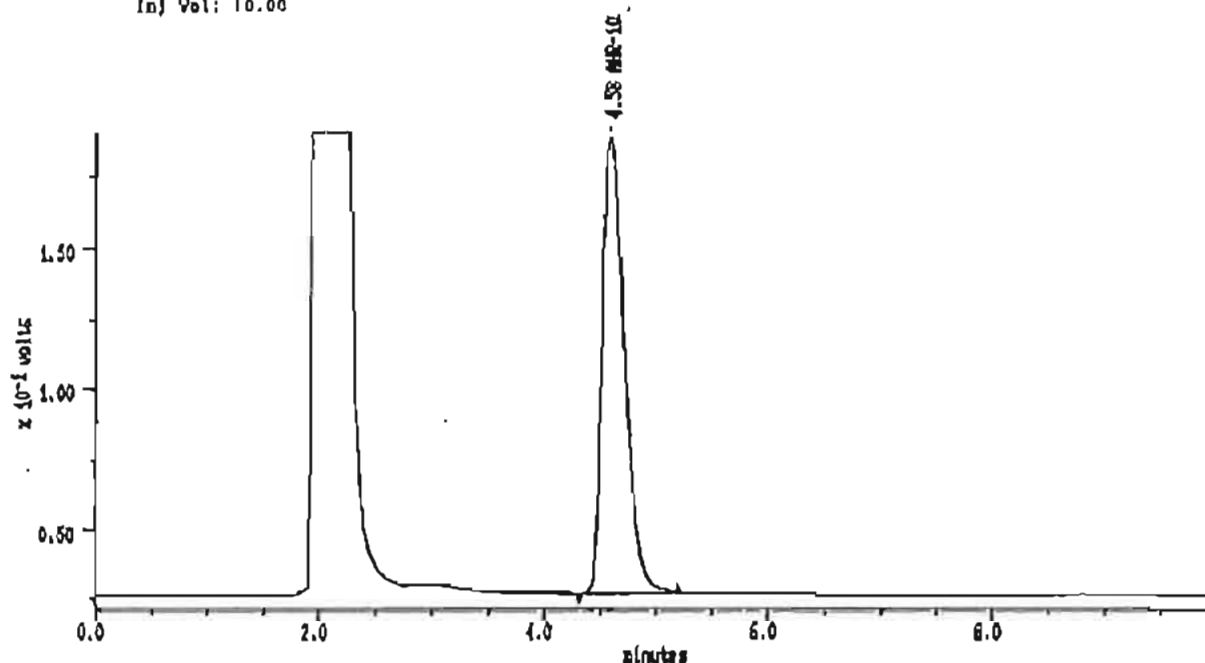
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.576	BS	2170690	168770	100.00	AHR-10282B
TOTAL			2170690	168770		

31

Sample: A32 60°C-1W Channel: detector 1 Filename: INI-28 Chart Speed: Full Size
 Acquired: 20-FEB-101 23:31 Method: B:YAHRYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 23:42:13

SAMPLE: A32 60°C-1W

#31 In Method: AHR - 10282B

Acquired: 20-FEB-2001 23:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-28

Index: 28

Injection Volume: 10.0

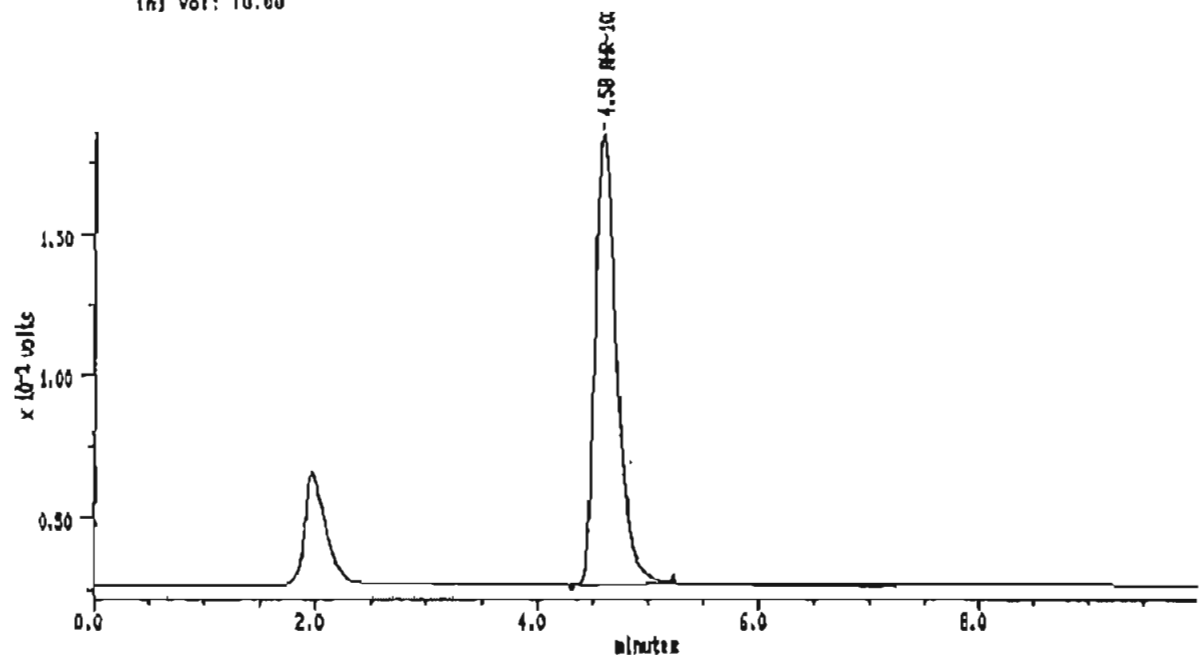
Amount: 0.000

DETECTOR1: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	BB	2243863	102380	100.00	AHR-10282B
TOTAL			2243863	102380		

32

Sample: A33 80°C-1W Channel: detector 1 Filename: INI-28 Chart Speed: Full Size
 Acquired: 20-FEB-2001 23:43 Method: B:VAHRVIXI3YIINITIAL Operator: S.S
 (n) Vol: 10.00



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MAXIMA 825 Custom Report

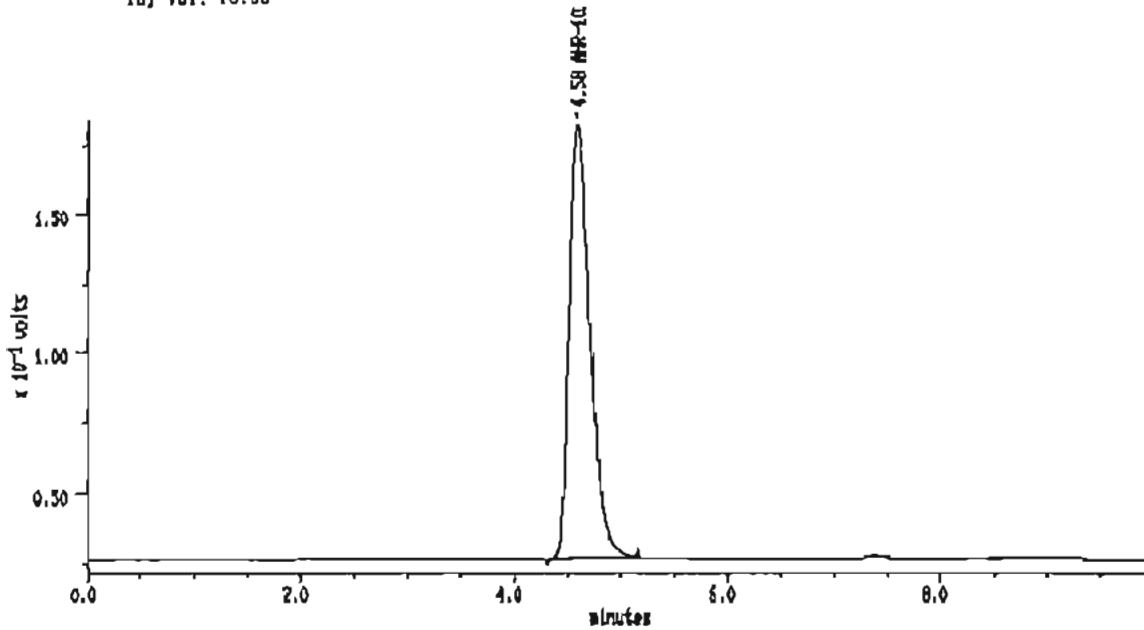
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SAMPLE: A33 80°C-1W Type: UNKN
 #32 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 20-FEB-2001 23:43 Filename: INI-28
 Rate: 2.0 points/sec Index: 28
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	83	2193124	187839	100.00	AHR-10282B
TOTAL			2193124	187839		

Sample: A34 80°C-1W Channel: detector 1 Filename: INI-30 Chart Speed: Full Size
 Acquired: 20-FEB-2001 23:54 Method: B:YAHYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 21-FEB-2001 0:05:02

SAMPLE: A34 80°C-1W

#22 In Method: AHR-10282B

Acquired: 20-FEB-2001 23:54

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNEX

Instrument: Instrument 1

Filename: INI-30

Index: 30

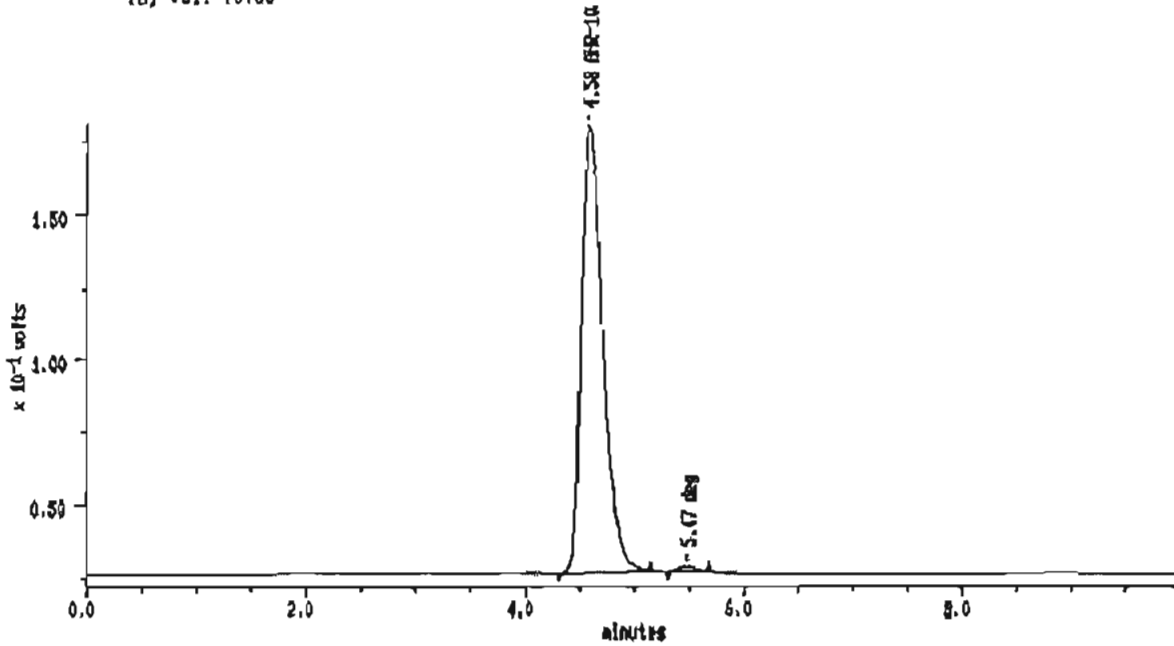
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.575	BB	2153242	158072	100.00	AHR-10282B
TOTAL			2153242	158072		

34

Sample: A36 80°C-1W Channel: detector 1 Filename: 1M1-31 Chart Speed: Full Size
 Acquired: 21-FEB-2001 0:08 Method: B:VAMRVIXI3V1INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 21-FEB-2001 0:18:28

SAMPLE: A36 80°C-1W

#34 In Method: AHR-10282B

Acquired: 21-FEB-2001 0:06

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: 1M1-31

Index: 31

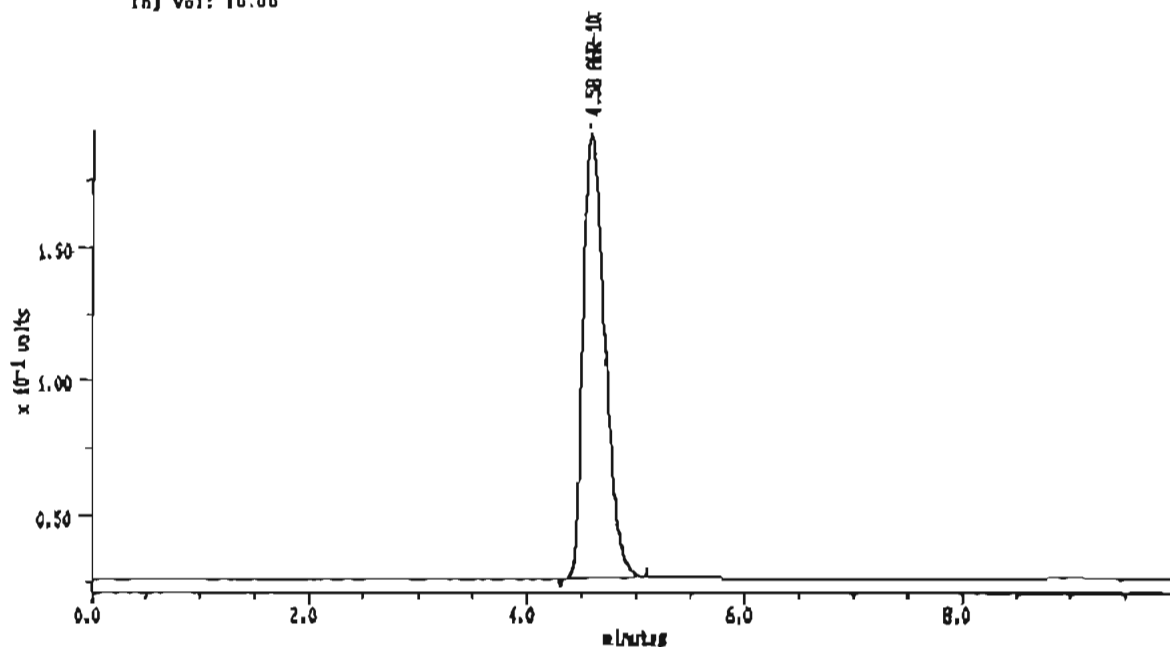
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.575	88	2118180	183489	88.98	AHR-10282B
2	6.487	88	21814	1937	1.02	deg
TOTAL			2139994	185426		

35

Sample: STD2 Channel: detector 1 Filename: INI-32 Chart Speed: Full Size
 Acquired: 21-FEB-2001 0:17 Method: B:VAIRYIKIQUINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 21-FEB-2001 0:27:58

SAMPLE: STD1

#35 In Method: AHR-10282B

Acquired: 21-FEB-2001 0:17

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-32

Index: 32

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.578	DB	2276153	185513	100.00	AHR-10282B
TOTAL			2276153	185513		

2/20 16:27 NO.21 PH 7.05 24.1°C	2/20 16:30 NO.31 PH 7.02 24.4°C	2/20 16:50 NO.41 PH 7.02 24.6°C
2/20 16:28 NO.22 PH 7.18 24.3°C	2/20 16:40 NO.32 PH 7.05 24.5°C	2/20 16:51 NO.42 PH 7.29 24.6°C
2/20 16:30 NO.23 PH 7.10 24.3°C	2/20 16:41 NO.33 PH 7.03 24.6°C	2/20 16:52 NO.43 PH 7.04 24.6°C
2/20 16:32 NO.24 PH 7.08 24.4°C	2/20 16:42 NO.34 PH 7.03 24.5°C	2/20 16:53 NO.44 PH 7.09 24.6°C
2/20 16:33 NO.25 PH 7.09 24.5°C	2/20 16:43 NO.35 PH 7.02 24.6°C	2/20 16:53 NO.45 PH 7.08 24.5°C
2/20 16:34 NO.26 PH 7.06 24.3°C	2/20 16:44 NO.36 PH 7.01 24.6°C	2/20 16:54 NO.46 PH 7.05 24.7°C
2/20 16:35 NO.27 PH 7.05 24.3°C	2/20 16:45 NO.37 PH 7.04 24.5°C	2/20 16:55 NO.47 PH 7.05 24.6°C
2/20 16:36 NO.28 PH 7.08 24.4°C	2/20 16:46 NO.38 PH 7.10 24.5°C	2/20 16:56 NO.48 PH 7.06 24.6°C
2/20 16:36 NO.29 PH 7.07 24.5°C	2/20 16:48 NO.39 PH 7.10 24.5°C	2/20 16:56 NO.49 PH 7.06 24.4°C
2/20 16:38 NO.30 PH 7.12 24.4°C	2/20 16:49 NO.40 PH 7.10 24.4°C	2/20 16:57 NO.50 PH 7.10 24.5°C

P2000B177 Lot No. 01K131 /Initial

This raw data is a duplicated copy as it could suffer the possibility of deterioration over time.

Therefore, there is no difference between this copy and the original copy.

2005.05.06 Shirou Sawa

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Collec(%)	Water	Initial	present
STD	1	S4-01	2303381						
STD	2	S4-18	2285689						
STD	mean		2284535	0.9980					
A-18	60°C-4W	S4-02	2379311	1.0394	101.39	94.51	6.79	8.5880	8.2530
A-19	80°C-4W	S4-03	2470948	1.0794	102.54	95.41	6.95	8.6173	8.2724
A-20	60°C-4W	S4-04	1329880	0.5809	55.01	51.27	6.79	8.6044	8.2682
A-21	80°C-4W	S4-05	1938745	0.8469	79.19	73.81	6.79	8.5498	8.2174
A-22	60°C-4W	S4-06	1594609	0.6968	67.87	63.19	6.89	8.5821	8.2425
A-23	60°C-4W	S4-07	2027430	0.8857	83.87	78.17	6.80	8.6440	8.3047
A-24	60°C-4W	S4-08	1833727	0.8011	77.88	72.44	6.74	8.6247	8.2897
A-25	60°C-4W	S4-09	1908933	0.8339	84.04	78.39	6.72	8.5719	8.2415
A-18	50°C-4W	S4-10	2277761	0.9950	97.05	94.13	3.01	8.6300	8.4800
A-19	50°C-4W	S4-11	2397283	1.0473	99.49	96.34	3.17	8.5270	8.3725
A-20	50°C-4W	S4-12	1527362	0.6872	63.18	61.18	3.16	8.4626	8.3103
A-21	50°C-4W	S4-13	2004949	0.8759	81.91	79.42	3.04	8.6138	8.4628
A-22	50°C-4W	S4-14	1725127	0.7536	73.43	71.03	3.27	8.5703	8.4093
A-23	50°C-4W	S4-15	2073253	0.9057	85.77	83.05	3.17	8.5336	8.3790
A-24	50°C-4W	S4-16	1887128	0.8244	79.94	77.40	3.18	8.4512	8.2986
A-25	60°C-4W	S4-17	1969902	0.8606	88.73	83.96	3.19	8.5877	8.4109

*Modified due to miscalculation.
 Re-edited in order to document the data necessary for calculation.
 Shirou Sawa, 6 May 2005*

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B		Test code	P2000B177		Test date	19 February 2001	
Test item							Tester	Shirou Sawa
STD AHR10282B 0.01996g/ 20mL x 2mL/20mLMP								
A18	60°C-4W	8.16	8.5880	8.2530	-	+	Yellow	
A19		8.17	8.6173	8.2724	-	-	Yellow	
A20		6.98	8.6044	8.2682	++	++	Orange yellow	
A21		6.89	8.5498	8.2174	++	++	Orange yellow	
A22		7.00	8.5821	8.2425	+	±	Yellow	
A23		6.97	8.6440	8.3047	+	+	Yellow	
A24		6.90	8.6247	8.2897	+	++	Orange yellow	
A25		6.95	8.5719	8.2415	+	+	Yellow	
A18	50°C-4W	8.19	8.6300	8.4800	-	-	Yellow	
A19		8.15	8.5270	8.39725	-	-	Yellow	
A20		7.00	8.4625	8.3103	-	+	Dark yellow	
A21		6.94	8.6136	8.4628	-	+	Dark yellow	
A22		6.99	8.5703	8.4093	+(r)	+	Yellow	
A23		6.95	8.5336	8.3790	-	+	Dark yellow	
A24		6.90	8.4512	8.2986	-	+	Dark yellow	
A25		7.00	8.5677	8.4109	-	+	Dark yellow	

2/19 16:11
NO. 8 P

2/19 16:12
NO. 9 P

2/19 16:13
NO. 1 P

2/19 16:14
NO. 2 P

2/19 16:15
NO. 3 P

2/19 16:16
NO. 4 P

2/19 16:17
NO. 5 P

2/19 16:17
NO. 5 P

2/19 16:18
NO. 7 P

2/19 16:19
NO. 8 P

2/19 16:19
NO. 9 P

2/19 16:20
NO. 10 P

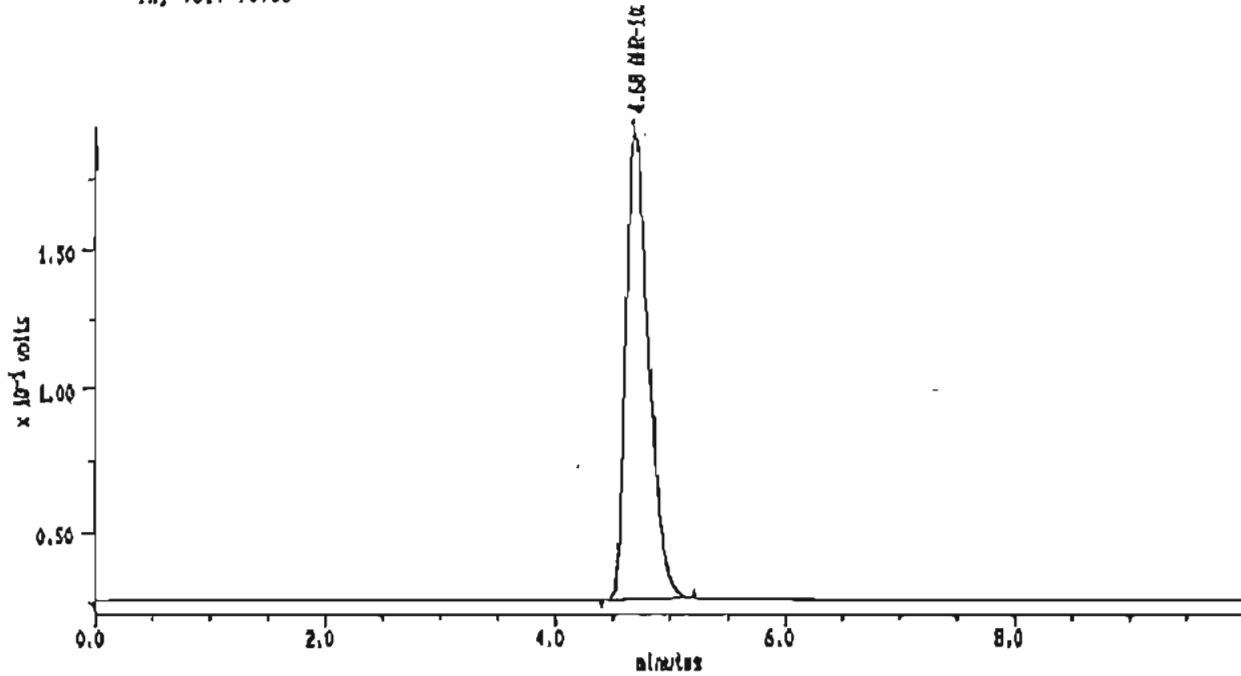
2/19 16:21
NO. 11 P

2/19 16:21
NO. 12 P

2/19 16:22
NO. 13 P

2/19 16:22
NO. 14 P

Sample: STD1 Channel: detector 1 Filename: S4-01 Chart Speed: Full Size
 Acquired: 19-FEB-101 21:02 Method: B:YAHRYIH18V80-4W Operator: S.S
 Inj Vol: 10.00



MAXIMA tel:1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 19-FEB-2001 21:13:03

SAMPLE: STD1

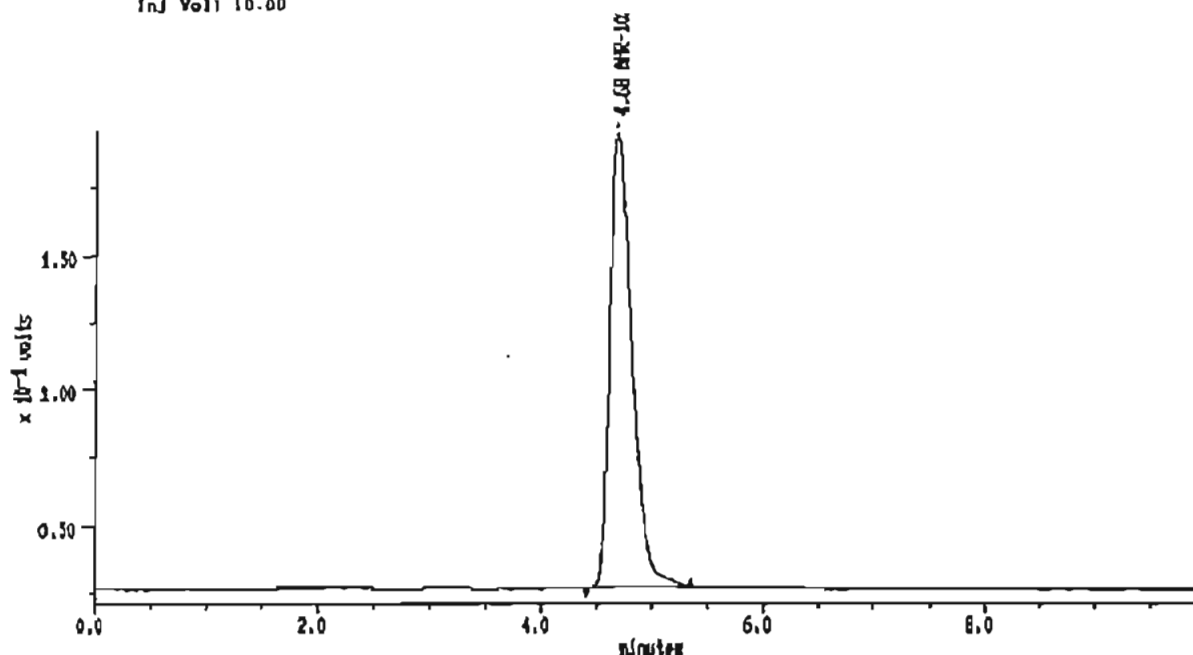
#4 In Method: AHR-10282B
 Acquired: 19-FEB-2001 21:02
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: S4-01
 Index: 18
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.675	BB	2303381	184826	100.00	AHR-10282B
TOTAL			2303381	184826		

Sample: A18 80°C-4W Channel: detector 1 Filename: S4-D2 Chart Speed: Full Size
 Acquired: 19-FEB-2001 21:13 Method: B:\ANALYSIS\H18Y80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 19-FEB-2001 21:24:27

SAMPLE: A18 80°C-4W

#5 in Method: AHR-10282B

Acquired: 19-FEB-2001 21:13

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: [instrument 1]

Filename: S4-D2

Index: 18

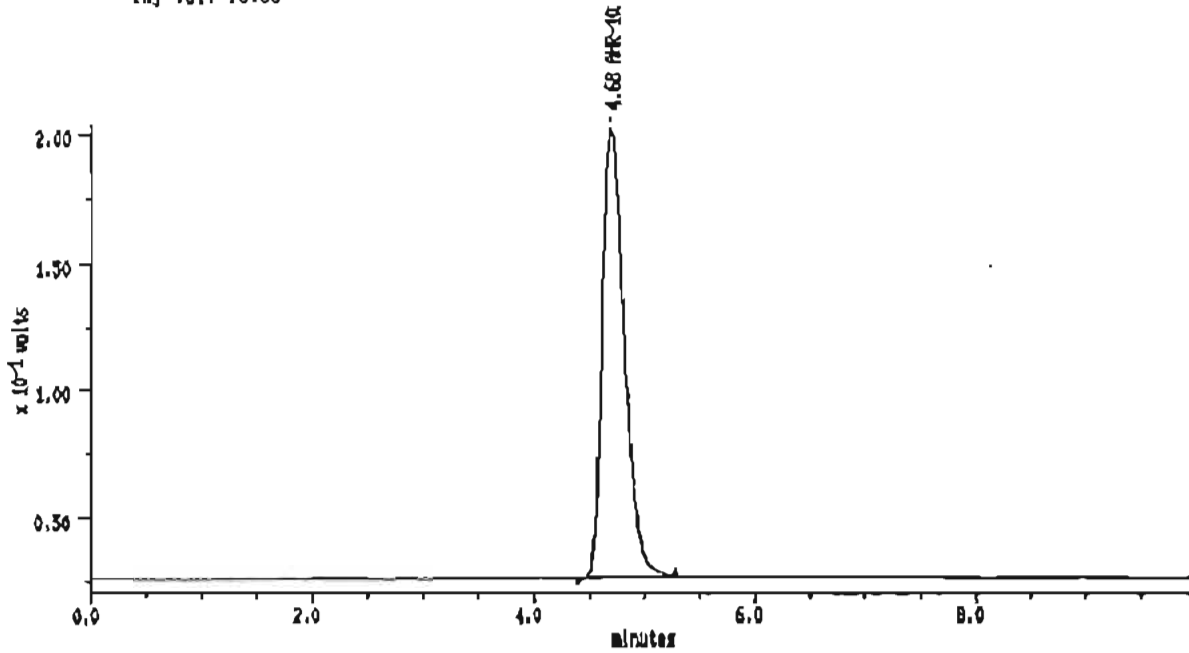
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.683	BB	2379311	187878	100.00	AHR-10282B
TOTAL			2379311	187878		

4

Sample: A19 80°C-4W Channel: detector 1 Filename: S4-03 Chart Speed: Full Size
 Acquired: 19-FEB-2001 21:25 Method: B:VAHRV1H16Y80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 19-FEB-2001 21:25:43

SAMPLE: A19 80°C-4W

#6 (n Method: AHR-10282B

Acquired: 19-FEB-2001 21:25

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: S4-03

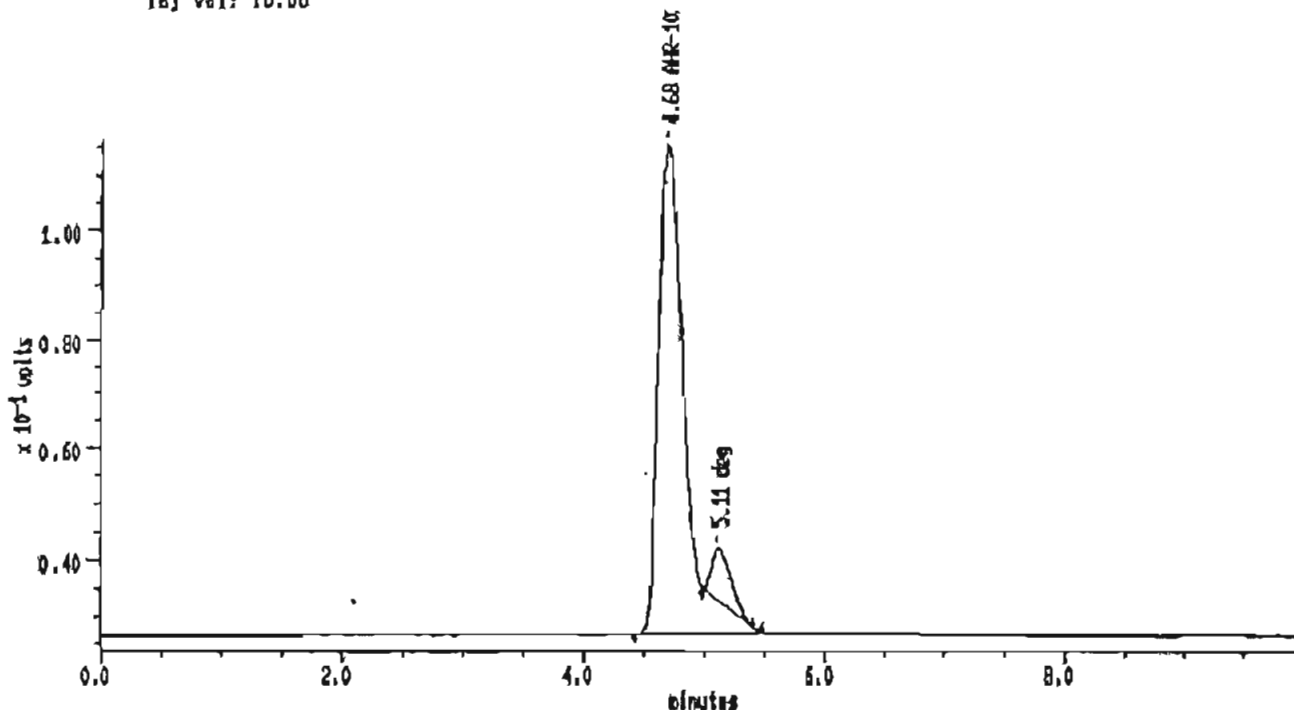
Index: 20

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.676	BB	2470948	176845	100.00	AHR-10282B
TOTAL			2470948	176845		

Sample: A20 80°C-4W Channel: detector 1 Filename: S4-04 Chart Speed: Full Size
 Acquired: 19-FEB-01 21:38 Method: B:VAHRV11116780-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 12:10:57

SAMPLE: A20 80°C-4W

#7 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-04

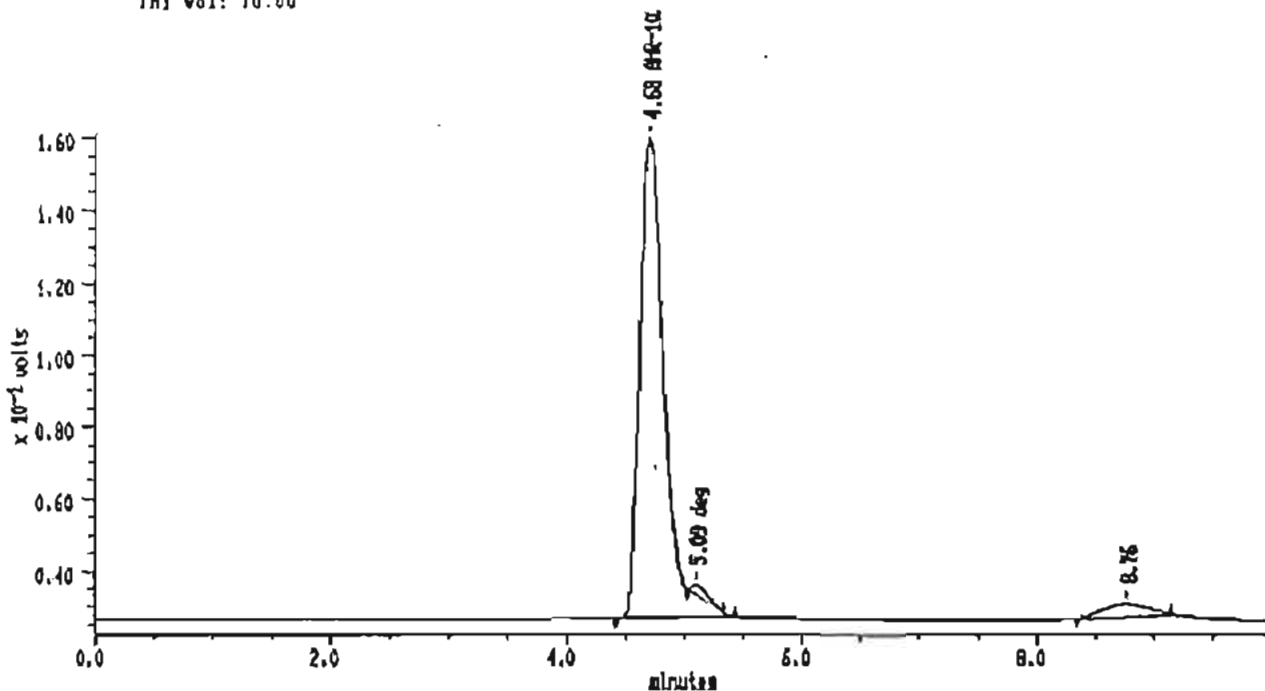
Index: 21

Injection Volume: 10.0

DETECTOR: detector 1

PE#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.683	OB	1329680	88819	92.74	AHR-10282B
2	5.108	SS	104020	9222	7.26	deg
TOTAL			1433700	97940		

Sample: A21 60°C-4W Channel: detector 1 Filename: S4-08 Chart Speed: Full Size
 Acquired: 19-FEB-10 21:47 Method: B:VAIRYIHTBY60-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 12:11:20

SAMPLE: A21 60°C-4W

#8 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:47

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URMN

Instrument: Instrument 1

Filename: S4-08

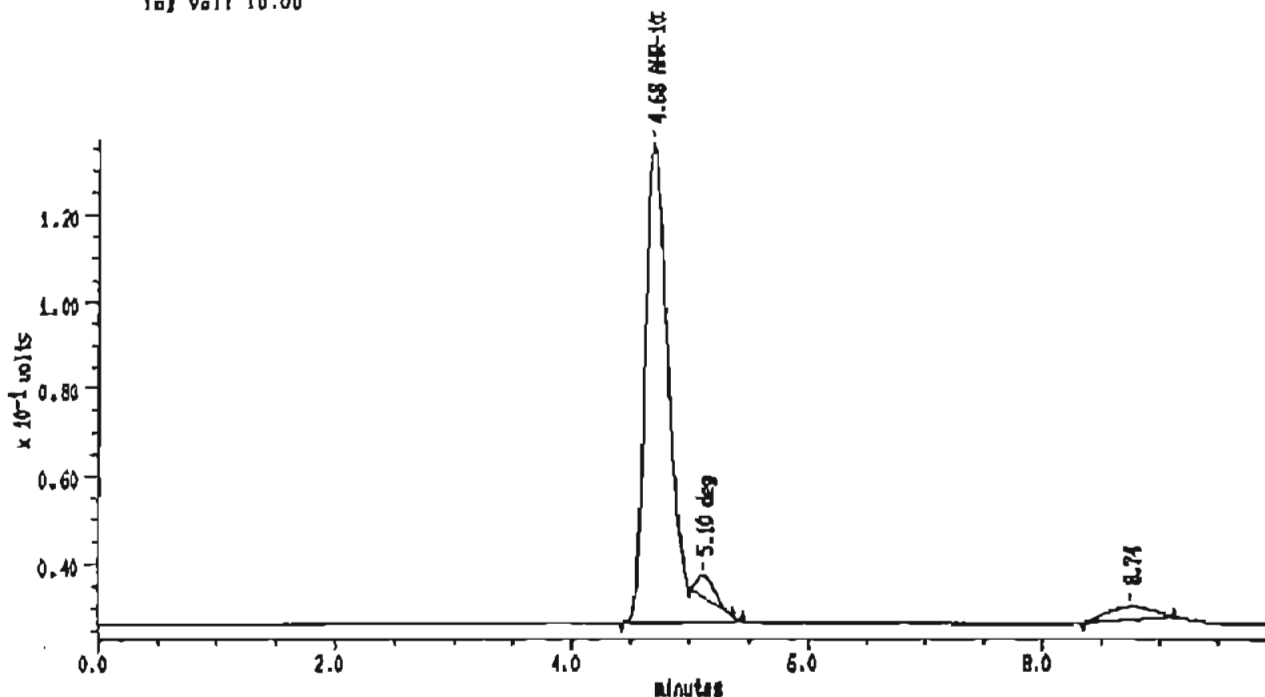
Index: 12

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.683	BB	1938745	133205	98.66	AHR-10282B
2	5.092	SS	26335	2598	1.34	deg
TOTAL			1965080	135792		

Sample: A22 80°C-4W Channel: detector 1 Filename: 54-08 Chart Speed: Full Size
 Acquired: 19-FEB-10 21:59 Method: B:VAHRV(1)16Y80-4W Operator: 9.9
 Inj Vol: 10.00



MAXIMA 101800 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 20-FEB-2001 12:11:58

SAMPLE: A22 80°C-4W

#9 In Method: AHR-10282B

Acquired: 10-FEB-2001 21:59

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: 9.9

Type: UNKN

Instrument: Instrument 1

Filename: 54-08

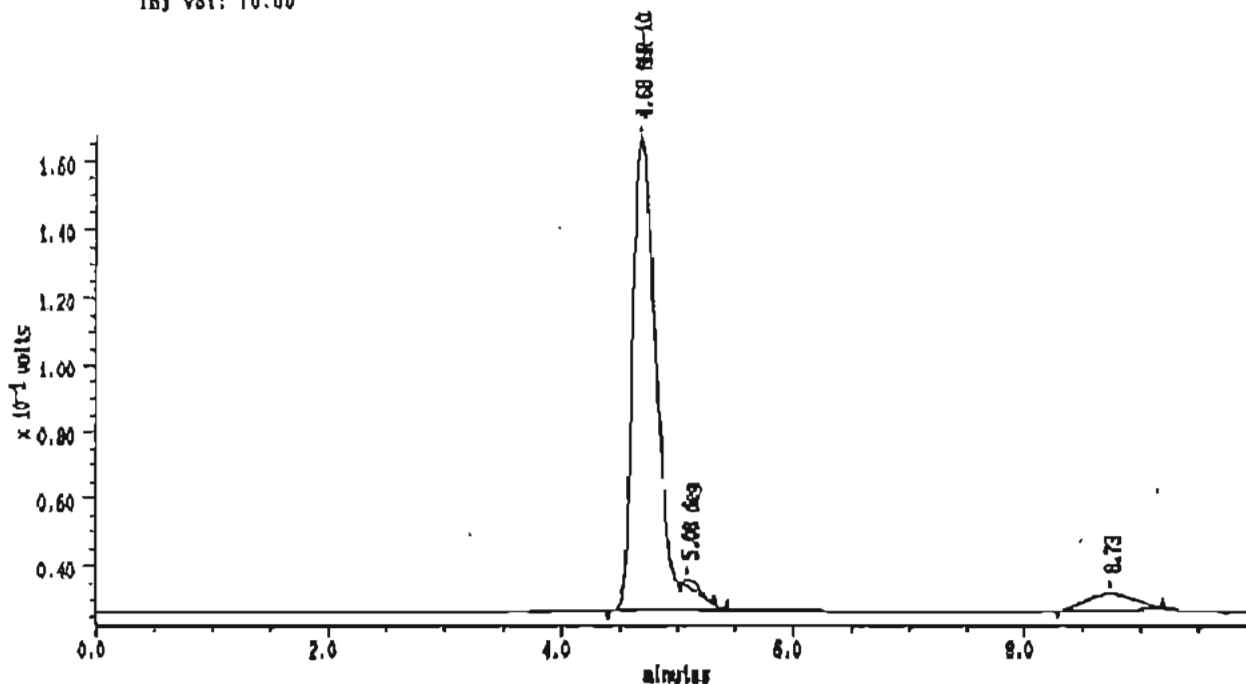
Index: 23

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.883	BB	1694609	108978	97.09	AHR-10282B
2	5.100	SS	47847	4542	2.91	deg
TOTAL			1642458	113519		

Sample: A23 00°C-4W Channel: detector 1 Filenamat: S4-07 Chart Speed: Full Size
 Acquired: 19-FEB-2001 22:10 Method: E:VAHRV1H15V60-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 12:12:24

SAMPLE: A23 80°C-4W

#10 In Method: **AHR-10282B**

Acquired: 19-FEB-2001 22:10

Rate: 2.0 points/sec

Duration: 10.090 minutes

Operator: S.S

Type: GNM

Instrument: Instrument 1

Filenam: S4-07

Index: 24

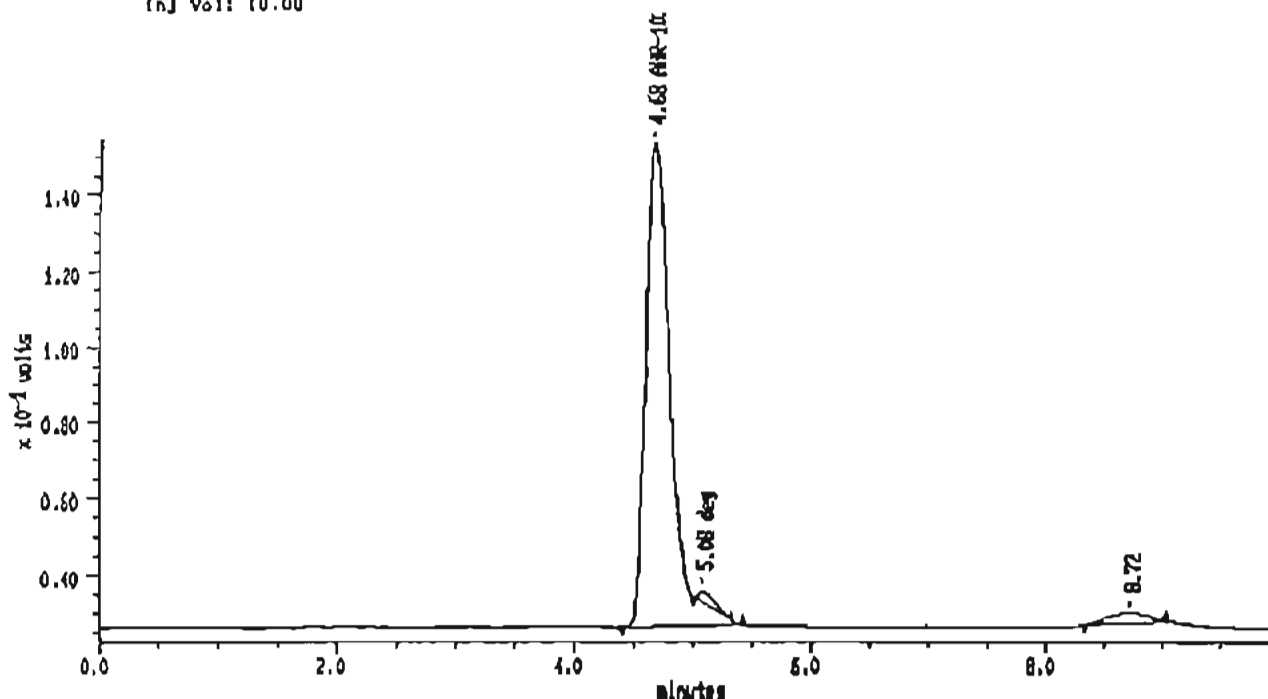
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.676	OB	2027430	140347	98.87	AHR-10282B
2	5.090	SS	23187	2268	1.13	dog
TOTAL			2060618	142615		

9

Sample: A24 80°C-4W Channel: detector 1 Filename: S4-08 Chart Speed: Full Size
 Acquired: 19-FEB-10 22:22 Method: B:VAHRY\H16V80-4W Operator: S.S
 Inj Vol: 10.00



MAXIMA Int 3998 Dynalco Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 10-FEB-2001 13:13:23

SAMPLE: A28 80°C-4W

012 in Method: AHR-10282B

Acquired: 10-FEB-2001 22:33

Rate: 2.0 psi/min/sec

Duration: 10.000 minutes

Operator: S.S

Type: UHPLC

Instrument: Instrument 1

Filename: S4-08

Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

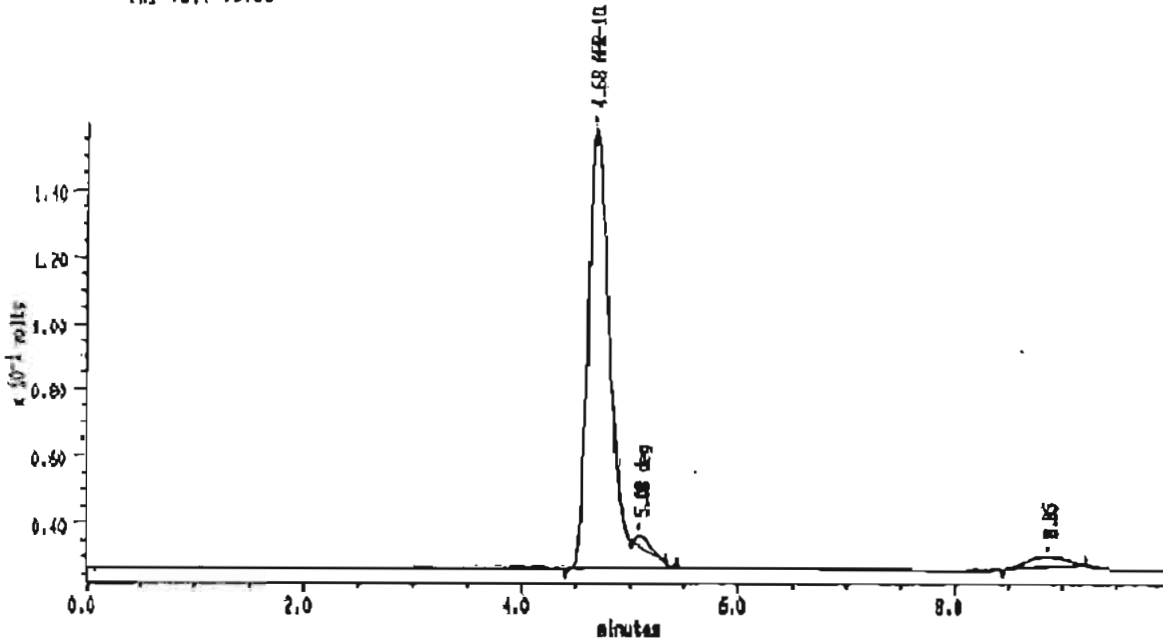
PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.676	DB	1908633	131639	98.44	AHR-10282B
2	5.083	DS	30288	2888	1.56	deg
TOTAL			1938921	134526		

10

Sample: A25 80°C-4W
 Acquired: 10-FEB-10 22:33
 (inj Vol: 10.00

Channel: detector 1
 Method: B:YAHRVINIGY80-GW

Filename: 54-00
 Chart Speed: Full Size
 Operator: S.S



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 12:10:22

SAMPLE: A25 80°C-4W

#12 In Method: AHR-10282B
 Acquired: 10-FEB-2001 22:33
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

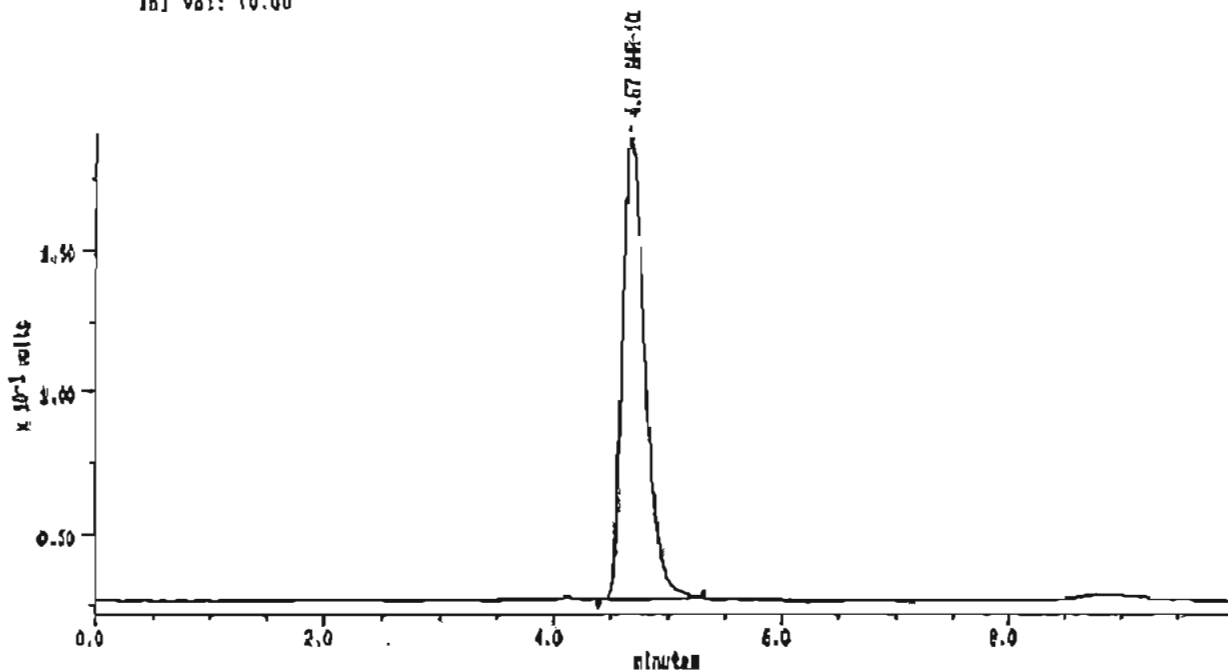
Type: UNKN
 Instrument: Instrument 1
 Filename: 54-00
 Index: 20
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.676	DB	1908930	131839	99.44	AHR-10282B
2	5.083	SS	30285	2888	1.56	dag
TOTAL			1939215	134727		

11

Sample: A10 50°C-4W Channel: detector 1 Filenamet: 54-10 Chart Speed: Full Size
 Acquired: 10-FEB-10 23:44 Method: B1YAHRYIHI8V00-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 10-FEB-2001 23:00:21

SAMPLE: A10 50°C-4W

#12 In Method: AHR-10282B

Acquired: 10-FEB-2001 23:44

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Inetrummit 1

Filenamet: 54-10

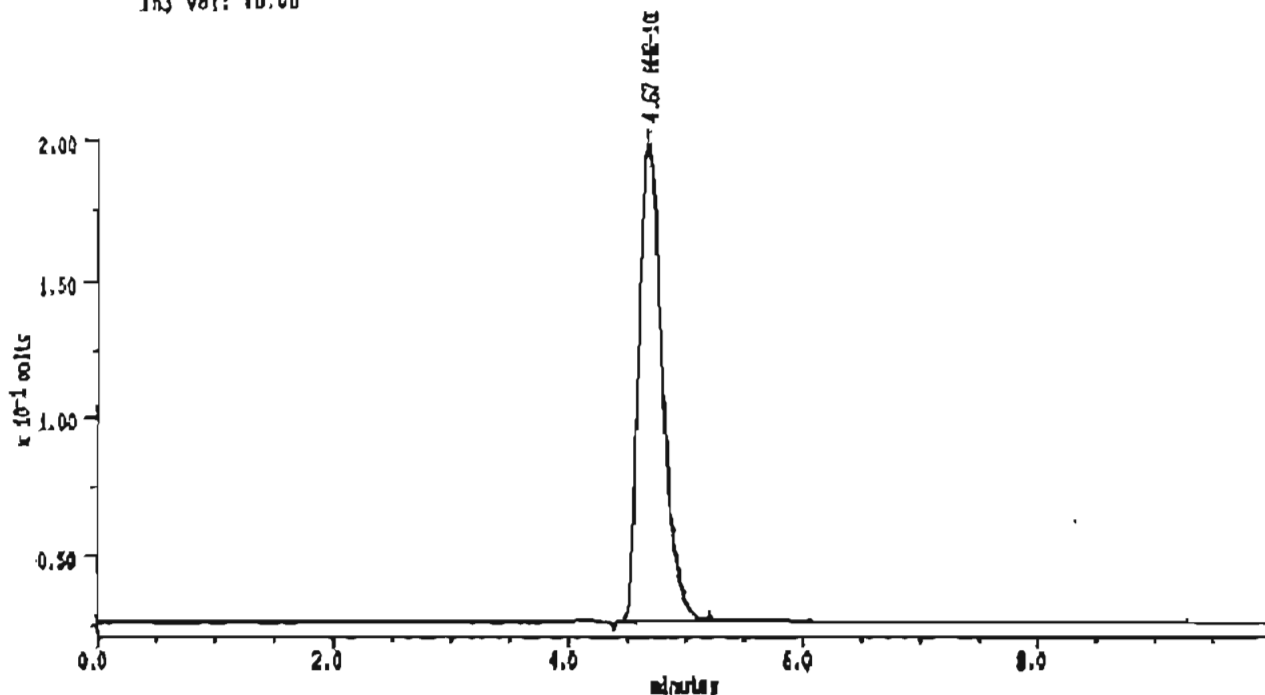
Index: 27

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.667	DB	2277701	102090	100.00	AHR-10282B
TOTAL			2277701	102090		

Sample: A10 80°C-4W Channel: detector 1 Filename: S4-11 Chart Speed: Full Size
 Acquired: 18-FEB-2001 22:58 Method: B:YAIRW:HSV00-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 19-FEB-2001 23:08:38

SAMPLE: A10 80°C-4W

014 In Method: AHR-10282B

Acquired: 18-FEB-2001 22:58

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: S4-11

Index: 28

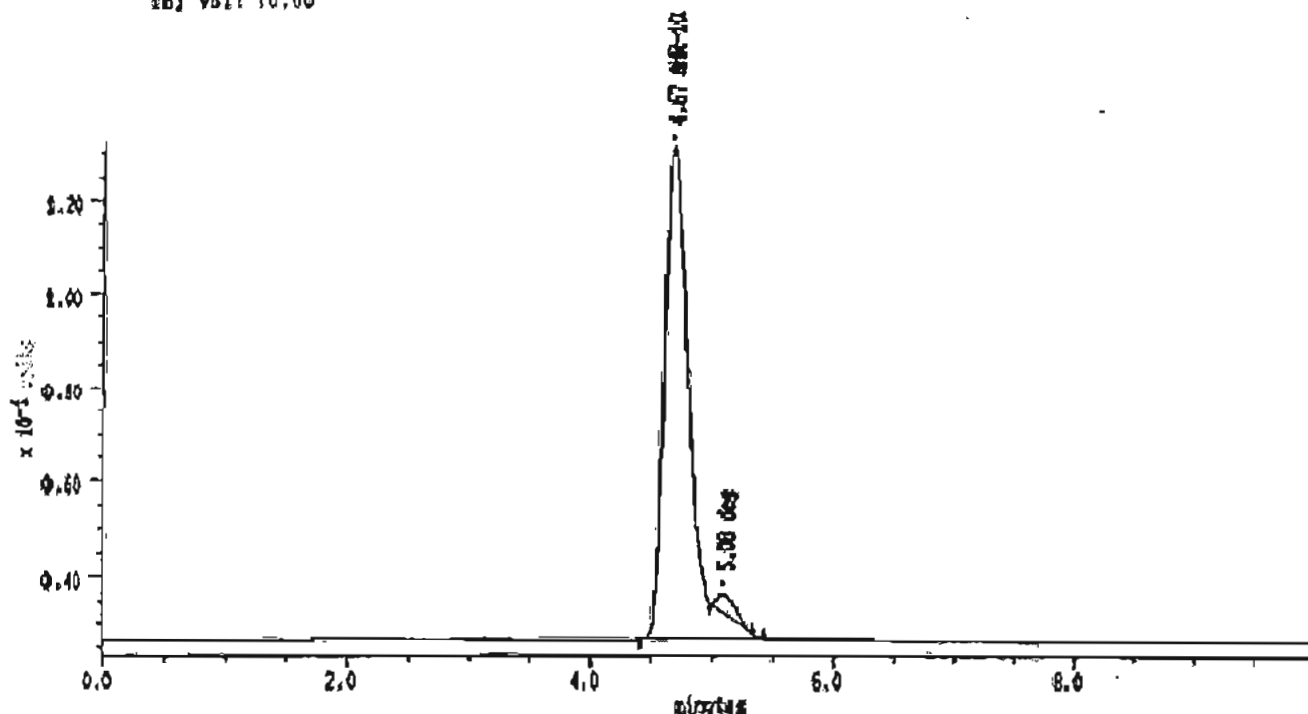
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.687	BB	2397283	172318	100.00	AHR-10282B
TOTAL			2397283	172318		

13

Sample: A20 60°C-4W Channel: detector 1 Filenames: S4-12 Chart Speed: Full Size
 Acquired: 18-FEB-01 23:07 Method: B:VARIYIM:6V80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 12:13:01

SAMPLE: A20 60°C-4W

#16 in Method: AHR-10282B

Acquired: 18-FEB-2001 23:07

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filenames: S4-12

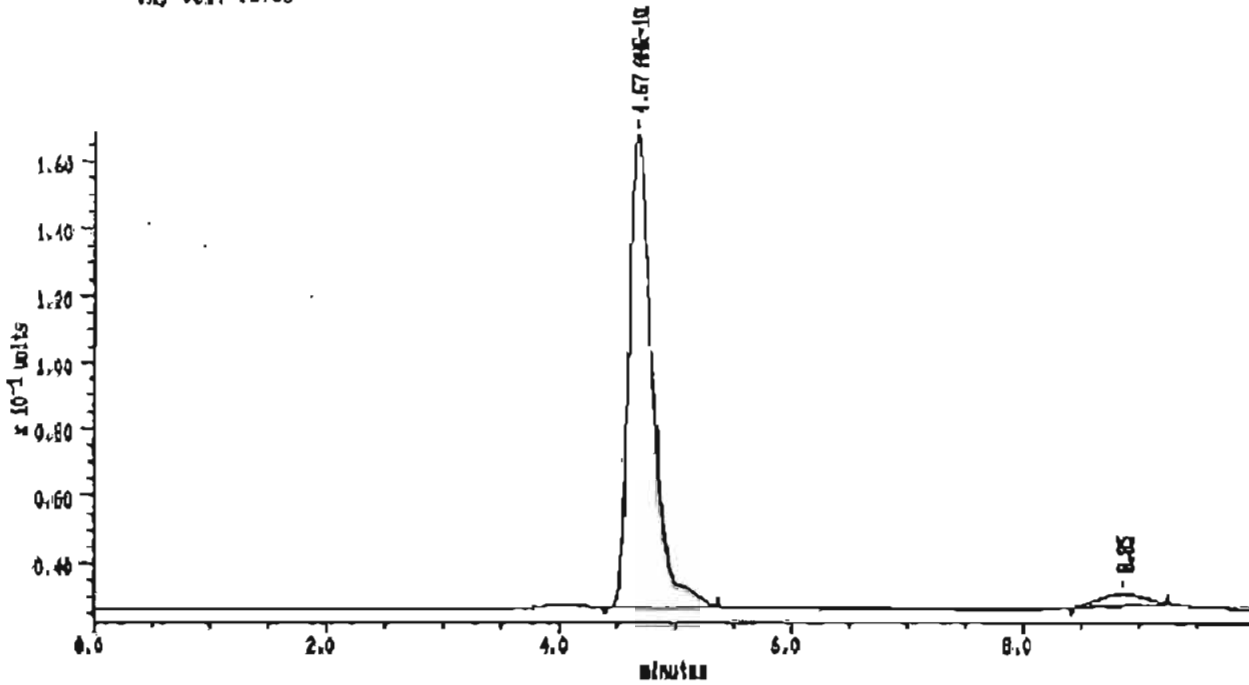
Index: 29

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.667	OB	1521363	104941	97.36	AHR-10282B
2	5.043	SB	41433	4052	2.64	ORG
TOTAL			1562796	108993		

Sample: A21 60°C-4W Channel: detector 1 Filename: S4-13 Chart Speed: Full Size
 Acquired: 19-FEB-2001 23:10 Method: D:\MIRV\8118V60-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 19-FEB-2001 23:29:20

SAMPLE: A21 60°C-4W

#18 in Method: AHR-10282B

Acquired: 19-FEB-2001 23:10

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-13

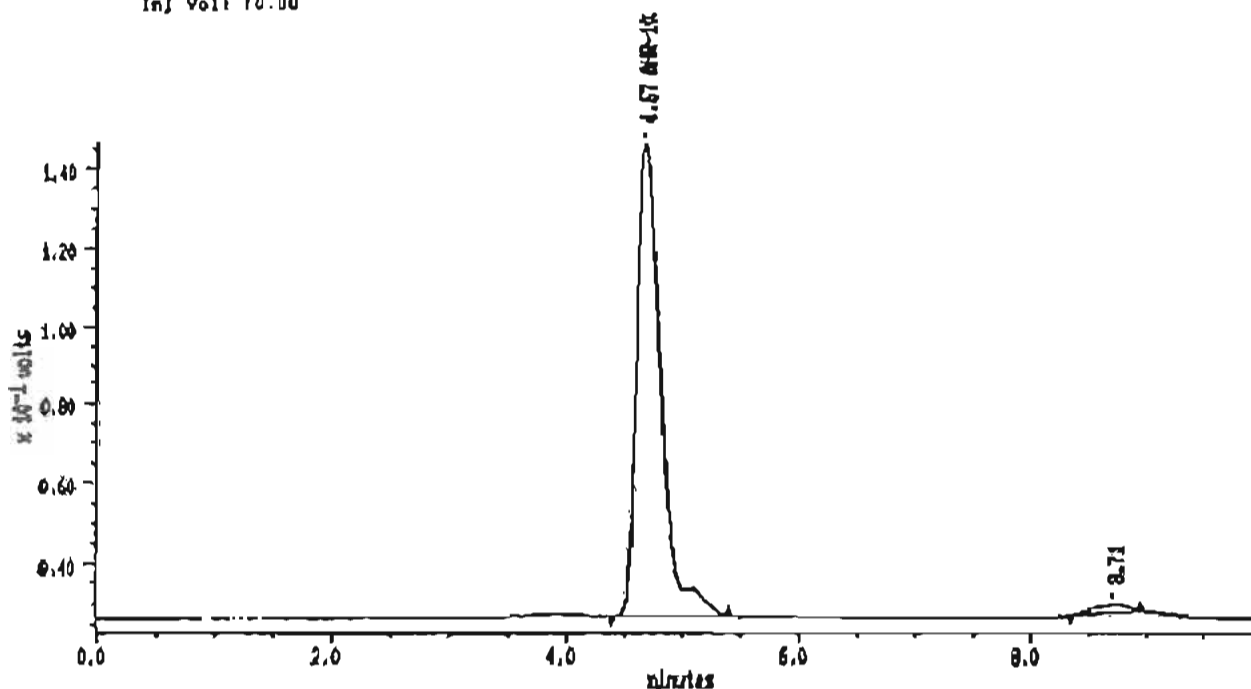
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.667	MD	2004948	149489	100.00	AHR-10282B
TOTAL			2004948	149489		

Sample: A22 60°C-4W Channel: detector 1 Filament: S4-14 Chart Speed: Full Size
 Acquired: 18-FEB-2001 23:30 Method: B:VAINRYLH:6V80-4W Operator: S.S
 Inj. Vol: 10.00



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MAXIMA 828 Custom Report

Printed: 18-FEB-2001 23:40:37

SAMPLE: A22 60°C-4W

#17 In Method: AHR-10282B

Acquired: 18-FEB-2001 23:30

Rate: 2.0 points/scan

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: S4-14

Index: 81

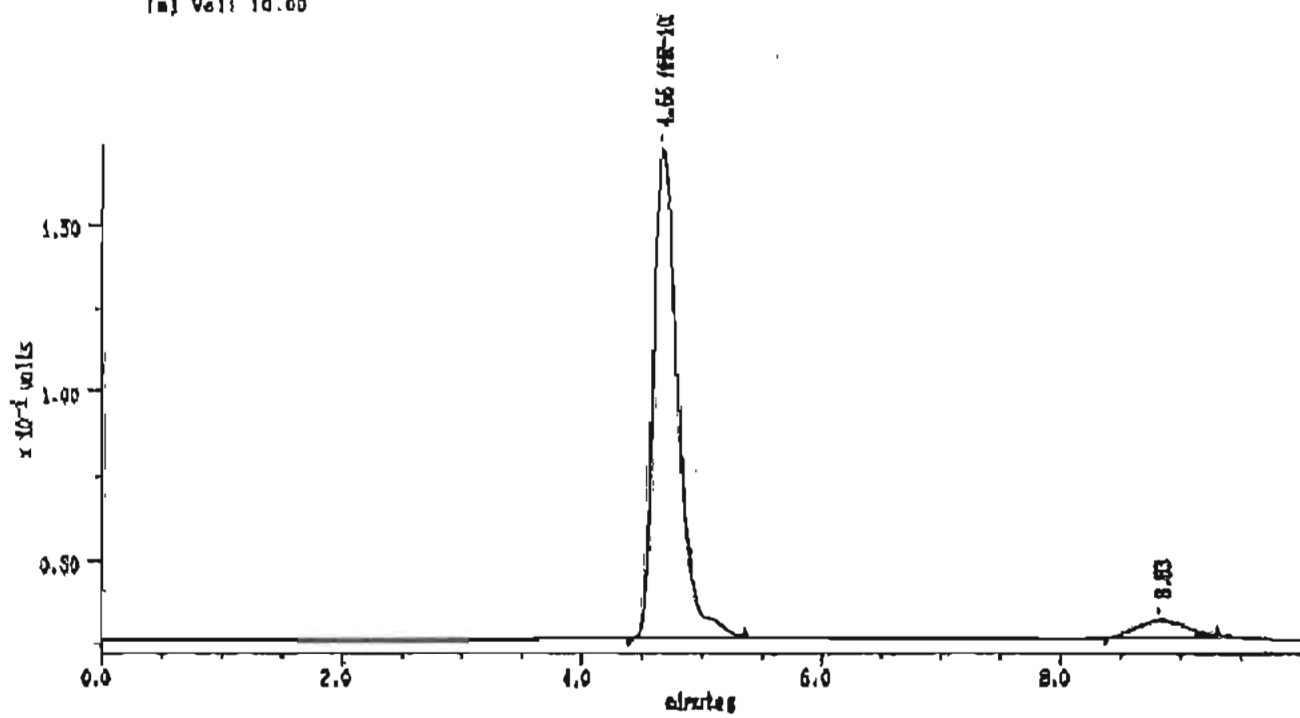
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.667	BB	1726127	119191	100.00	AHR-10282B
TOTAL			1726127	119191		

16

Sample: A23 60°C-4W Channel: detector 1 Filename: 64-16 Chart Speed: Full Size
 Acquired: 19-FEB-01 23:41 Method: D:VARRYDILEV80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 828 Custom Report

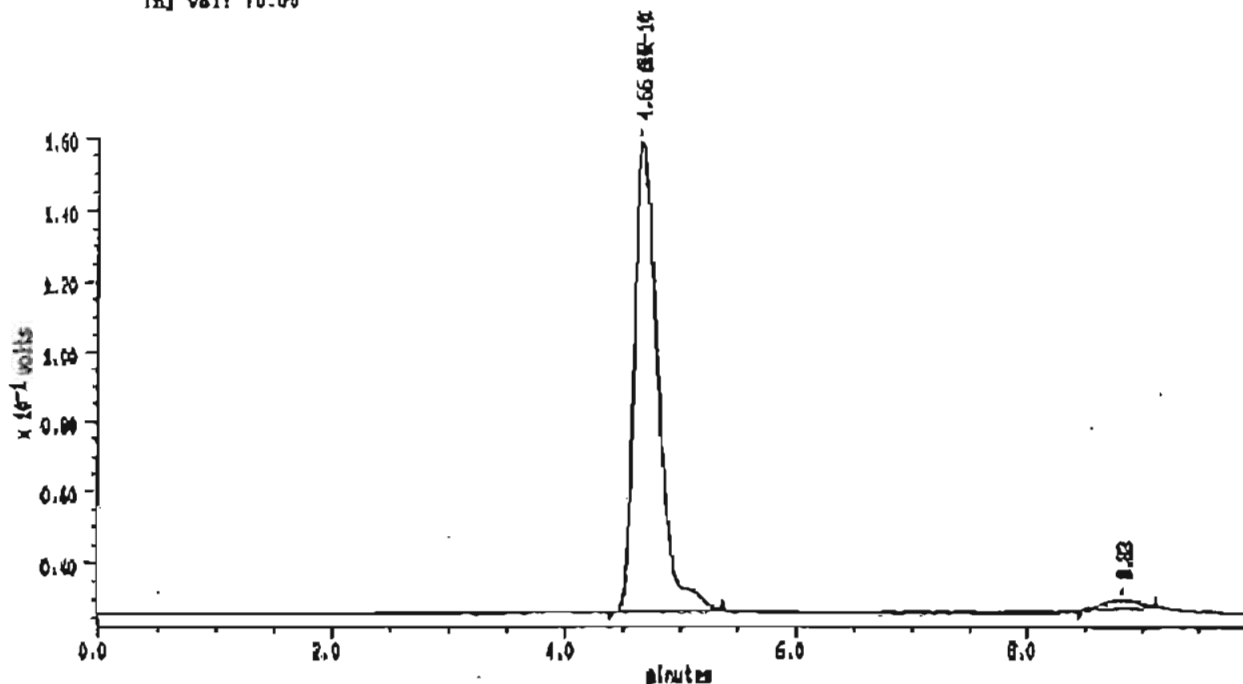
Printed: 19-FEB-2001 23:51:54

SAMPLE: A23 60°C-4W #10 In Method: AHR-10282B Acquired: 19-FEB-2001 23:41 Rate: 2.0 points/sec Duration: 10.000 minutes Operator: S.S	Type: UNKN Instrument: Instrument 1 Filename: 64-16 Index: 32 Injection Volume: 10.0
--	--

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.660	BB	2073253	145000	100.00	AHR-10282B
TOTAL			2073253	145000		

Sample: A24 80°C-4W Channel: detector 1 Filament: S4-18 Chart Speed: Full Size
 Acquired: 19-FEB-101 23:52 Method: B1YAHRY1H16V60-3W Operator: S.S
 Inj Vol: 10.00



MAXIMA 601888 Dynamic Solutions, Division of Hillborn

MAXIMA 825 Custom Report

Printed: 20-FEB-2001 0:02:11

SAMPLE: A24 80°C-4W

#19 in Method: AHR-10282B

Acquired: 19-FEB-2001 23:52

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: LHMW

Instrument: Instrument 1

Filament: S4-18

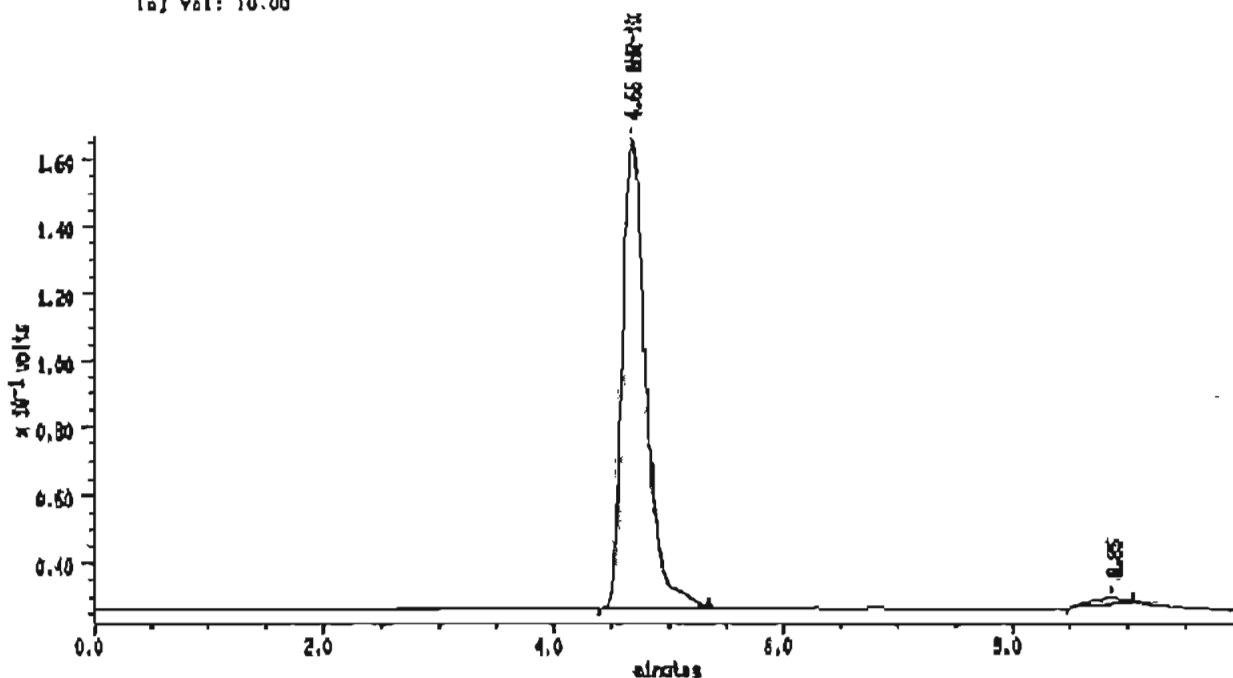
Index: 23

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.660	SB	1887128	131203	100.00	AHR-10282B
TOTAL			1887128	131203		

Sample: A26 60°C-4W Channel: detector 1 Filename: 54-17 Chart Speed: Full Size
 Acquired: 20-FEB-101 0:03 Method: D:\AIRV\1115V60-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 01:41:20

SAMPLE: A26 60°C-4W

#20 (in Method: AHR-10282B

Acquired: 20-FEB-2001 0:03

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: DRKM

Instrument: Instrument 1

Filename: 54-17

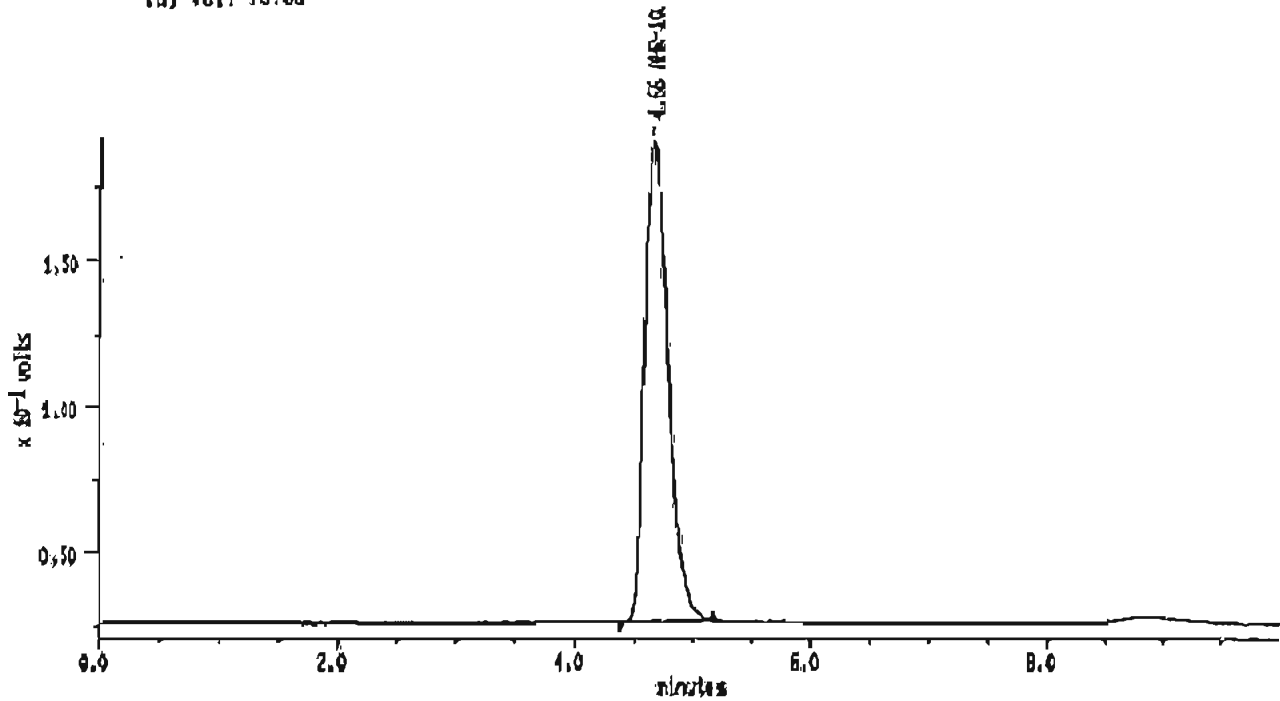
Injunct 34

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.668	DB	1888902	158783	100.00	AHR-10282B
TOTAL			1888902	158783		

Sample: STD Channel: detector 1 Filename: S4-18 Chart Speed: Full Size
 Acquired: 20-FEB-2001 0:18 Method: B:VAIRV/HLSY80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 20-FEB-2001 0:25:58

SAMPLE: STD

#21 In Method: AHR-10282B
 Acquired: 20-FEB-2001 0:18
 Rate: 7.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: URM
 Instrument: Instrument 1
 Filename: S4-18
 Index: 35
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.660	SS	2285889	18334	100.00	AHR-10282B
TOTAL			2285889	18334		

Test Record B (other) Form 7 (1 January 2000)

Test substance		AHR10282B	Test code		P2000B177		Test date		19 February 2001	
Test item						Tester		Shirou Sawa		
<i>STD AHR10282B 0.01996g/ 20mL x 2mL/20mLMP</i>										
A18	60°C-4W	8.16	8.5880	8.2530	-	+	Yellow			
A19		8.17	8.6173	8.2724	-	-	Yellow			
A20		6.98	8.6044	8.2682	++	++	Orange yellow			
A21		6.89	8.5498	8.2174	++	++	Orange yellow			
A22		7.00	8.5821	8.2425	+	±	Yellow			
A23		6.97	8.6440	8.3047	+	+	Yellow			
A24		6.90	8.6247	8.2897	+	++	Orange yellow			
A25		6.95	8.5719	8.2415	+	+	Yellow			
A18	50°C-4W	8.19	8.6300	8.4800	-	-	Yellow			
A19		8.15	8.5270	8.39725	-	-	Yellow			
A20		7.00	8.4625	8.3103	-	+	Dark yellow			
A21		6.94	8.6136	8.4628	-	+	Dark yellow			
A22		6.99	8.5703	8.4093	+(r)	+	Yellow			
A23		6.95	8.5336	8.3790	-	+	Yellow			
A24		6.90	8.4512	8.2986	-	+	Dark yellow			
A25		7.00	8.5677	8.4109	-	+	Dark yellow			
<p>P2000B177 Lot No. 01H151 60°C -4W This raw data is a duplicated copy as it could suffer the possibility of deterioration over time. Therefore, there is no difference between this copy and the original copy. 2005.05.06 Shirou Sawa</p>										

2/19 16:11
NO. 98 PH 2

2/19 16:12
NO. 99 PH 2

2/19 16:13
NO. 100 PH 2

2/19 16:14
NO. 2 PH 2

2/19 16:15
NO. 3 PH 2

2/19 16:16
NO. 4 PH 2

2/19 16:17
NO. 5 PH 2

2/19 16:17
NO. 6 PH 2

2/19 16:18
NO. 7 PH 2

2/19 16:19
NO. 8 PH 2

2/19 16:19
NO. 9 PH 2

2/19 16:20
NO. 10 PH 2

2/19 16:21
NO. 11 PH 2

2/19 16:21
NO. 12 PH 2

2/19 16:22
NO. 13 PH 2

2/19 16:22
NO. 14 PH 2

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Water Collect(%)	Initial	present
STD	1	V2-01	2310931					
STD	2	V2-26	2328564					
STD	mean		2319748	1.0095				
A-18	70°C-2W	V2-02	2423977	1.0549	102.90	95.01	7.67	8.8839 8.2978
A-19	70°C-2W	V2-03	2484866	1.0727	101.90	93.95	7.80	8.5682 8.1846
A-20	70°C-2W	V2-04	1889028	0.7350	69.60	64.57	7.22	8.6082 8.2487
A-21	70°C-2W	V2-05	2151512	0.8363	87.55	80.80	7.71	8.5344 8.1581
A-22	70°C-2W	V2-06	1898449	0.8253	80.42	74.55	7.30	8.6129 8.2507
A-23	70°C-2W	V2-07	2197150	0.9581	90.54	83.86	7.38	8.6428 8.2746
A-24	70°C-2W	V2-08	1992556	0.8671	84.08	77.55	7.77	8.4913 8.1152
A-25	70°C-2W	V2-09	2044175	0.8896	89.85	83.08	7.33	8.5906 8.2286
A-18	60°C-2W	V2-10	2301723	1.0017	97.71	94.66	3.12	8.6047 8.4501
A-19	60°C-2W	V2-11	2360197	1.0271	97.57	94.62	3.02	8.6026 8.4630
A-20	60°C-2W	V2-12	1751139	0.7621	72.17	70.12	2.84	8.6548 8.5126
A-21	60°C-2W	V2-13	2140159	0.9313	87.09	84.41	3.08	8.5464 8.3955
A-22	60°C-2W	V2-14	1951236	0.8491	82.73	80.05	3.24	8.5555 8.3965
A-23	60°C-2W	V2-15	2186038	0.9513	90.09	87.28	3.14	8.6875 8.5294
A-24	60°C-2W	V2-16	2007368	0.8736	84.71	82.08	3.10	8.6175 8.4634
A-25	60°C-2W	V2-17	2082273	0.9062	91.32	88.55	3.03	8.6289 8.4780
A-18	50°C-2W	V2-18	2237481	0.9737	94.88	93.44	1.62	8.5642 8.4844
A-19	50°C-2W	V2-19	2345444	1.0207	96.98	95.40	1.61	8.4536 8.3763
A-20	50°C-2W	V2-20	1839698	0.8008	75.81	74.65	1.53	8.5997 8.5240
A-21	50°C-2W	V2-21	2140699	0.9316	87.11	85.74	1.57	8.5511 8.4743
A-22	50°C-2W	V2-22	1963693	0.8546	83.27	81.99	1.54	8.6496 8.4742
A-23	50°C-2W	V2-23	2218206	0.9653	91.41	90.08	1.48	8.7794 8.7043
A-24	50°C-2W	V2-24	2081641	0.8972	87.00	85.73	1.46	8.5244 8.4531
A-25	50°C-2W	V2-25	2080594	0.9054	91.24	89.90	1.47	8.5409 8.4688

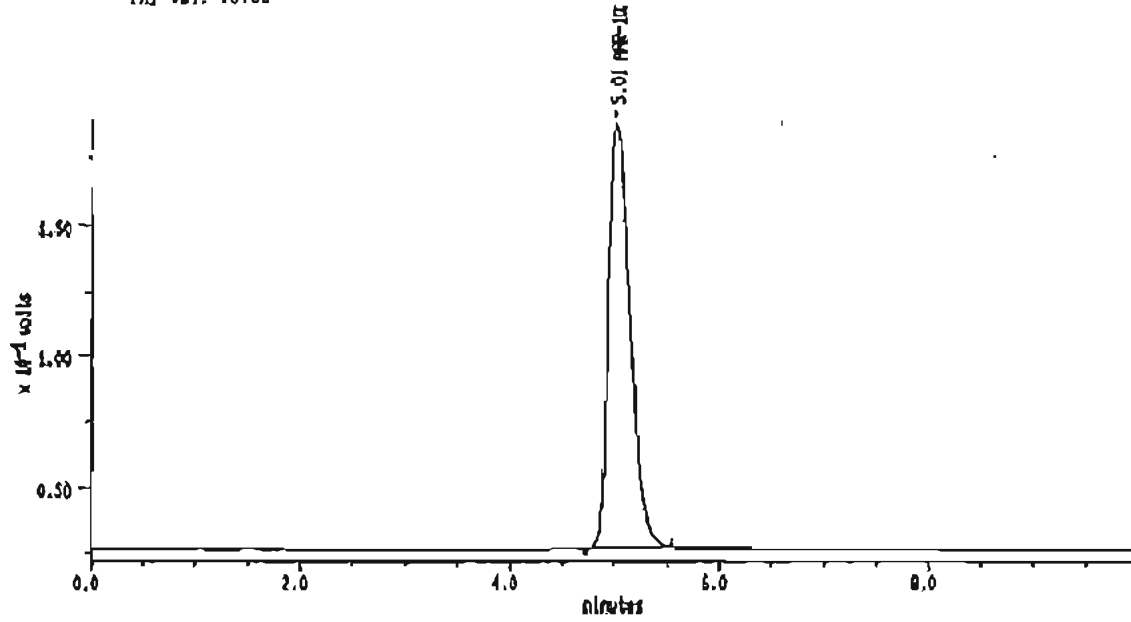
*Modified due to miscalculation.
 Re-edited in order to document the data necessary for calculation.
 Shirou Sawa, 6 May 2005*

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2000B177	Test date	05 February 2001		
Test item					Tester	Shirou Sawa	
0.02198/20mL							
0011 + 0.02019 g							
	Condition	PH	Weight change		Foreign matter	Turbidity	Color
A18	70°C-2W	8.13	8.6839	8.2978	-	+	Yellow
A19		8.15	8.5682	8.1845	-	-	Yellow
A20		6.95	8.6062	8.2487	++(r)	-	Dark yellow
A21		6.90	8.5344	8.2746	++(r)	-	Dark yellow
A22		6.96	8.26129	8.1581	++(r)	-	Yellow
A23		6.94	8.6428	8.2746	+(r)	-	Yellow
A24		6.87	8.4913	8.1152	++(r)	-	Dark yellow
A25		6.97	8.5906	8.2286	+(r)	-	Dark yellow
A18	60°C-2W	8.16	8.6074	8.4501	-	-	Yellow
A19		8.16	8.6026	8.4530	-	-	Yellow
A20		6.97	8.6548	8.5126	+(r)	-	Dark yellow
A21		6.94	8.5464	8.3955	+(r)	-	Dark yellow
A22		6.99	8.5555	8.3965	+(r)	+	Yellow
A23		6.97	8.6875	8.5294	+(r)	+	Yellow
A24		6.92	8.6175	8.4634	+(r)	++	Dark yellow
A25		7.00	8.6289	8.4780	+(r)	+	Yellow
A18	50°C-2W	8.15	8.5642	8.4844	-	-	Yellow
A19		8.17	8.4536	8.3763	-	-	Yellow
A20		6.99	8.5997	8.5240	±(r)	+	Dark yellow
A21		6.95	8.5511	8.4743	±	+	Yellow
A22		7.01	8.5496	8.4742	±	+	Yellow
A23		6.98	8.7794	8.7043	±	±	Yellow
A24		6.93	8.5224	8.4531	+	+	Yellow
A25		6.98	8.5409	8.54688	+	+	Yellow

2/05 18:35 NO. 90 PH 6.13 22.4°C	2/05 18:46 NO. 98 PH 6.16 22.6°C	2/05 18:52 NO. 96 PH 6.15 22.8°C
2/05 18:40 NO. 81 PH 6.15 22.4°C	2/05 18:47 NO. 99 PH 6.16 22.6°C	2/05 18:54 NO. 97 PH 6.17 22.5°C
2/05 18:40 NO. 82 PH 6.95 22.5°C	2/05 18:46 NO. 99 PH 6.97 22.5°C	2/05 18:56 NO. 99 PH 6.93 22.8°C
2/05 18:42 NO. 83 PH 6.93 22.6°C	2/05 18:49 NO. 91 PH 6.94 22.5°C	2/05 18:57 NO. 99 PH 6.95 22.6°C
2/05 18:42 NO. 84 PH 6.95 22.6°C	2/05 18:49 NO. 92 PH 6.99 22.5°C	2/05 18:58 NO. 1 PH 7.01 22.6°C
2/05 18:43 NO. 85 PH 6.94 22.6°C	2/05 18:50 NO. 93 PH 6.97 22.4°C	2/05 18:59 NO. 2 PH 6.95 22.6°C
2/05 18:44 NO. 85 PH 6.97 22.6°C	2/05 18:51 NO. 94 PH 6.92 22.4°C	2/05 19:00 NO. 3 PH 6.93 22.6°C
2/05 18:45 NO. 87 PH 6.97 22.6°C	2/05 18:52 NO. 95 PH 7.03 22.6°C	2/05 19:00 NO. 4 PH 6.93 22.4°C

Sample: STD1 Channel: detector 1 Filename: V2-01 Chart Speed: Full Size
 Acquired: 00-FEB-10 22:18 Method: B:YAH(Y)HIBY70-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (c)1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 6-FEB-2001 22:29:12

SAMPLE: STD1

#3 In Method: AHR-10282B

Acquired: 6-FEB-2001 22:18

Rate: 1.0 points/min

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-01

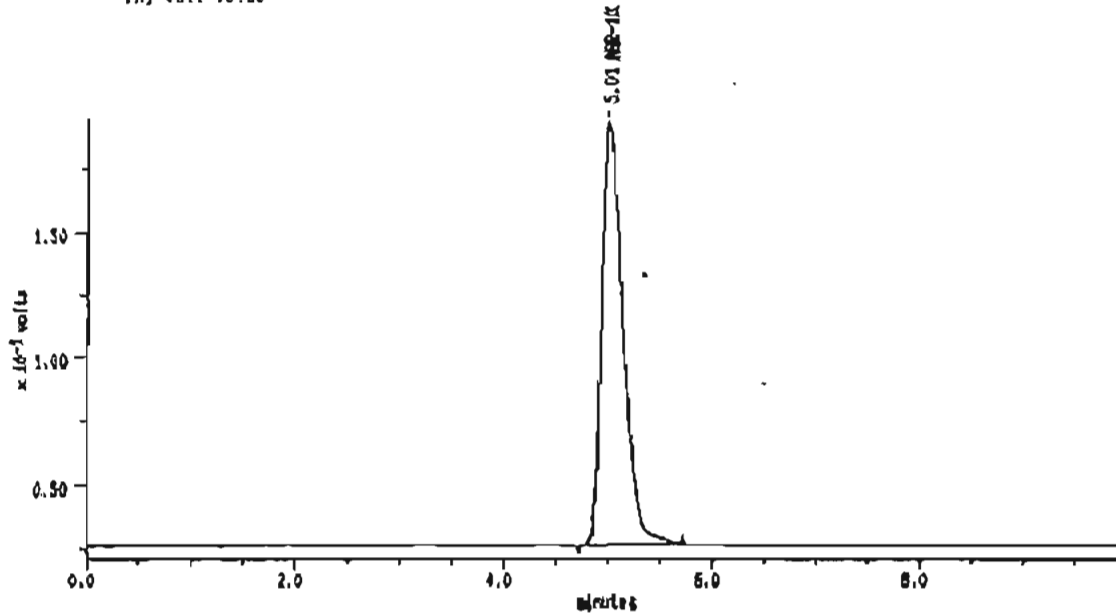
Index: 20

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	DB	2310931	161187	100.00	AHR-10282B
TOTAL			2310931	161187		

Sample: A18 70°C-2W Channel: detector 1 Filename: V2-02 Chart Speed: Full Size
 Acquired: 05-FEB-2001 22:30 Method: S:\ANALYSIS\16970-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1980 Dynafac Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 5-FEB-2001 22:46:38

SAMPLE: A18 70°C-2W

PK In Method: AHR-10282B

Acquired: 5-FEB-2001 22:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-02

Index: 27

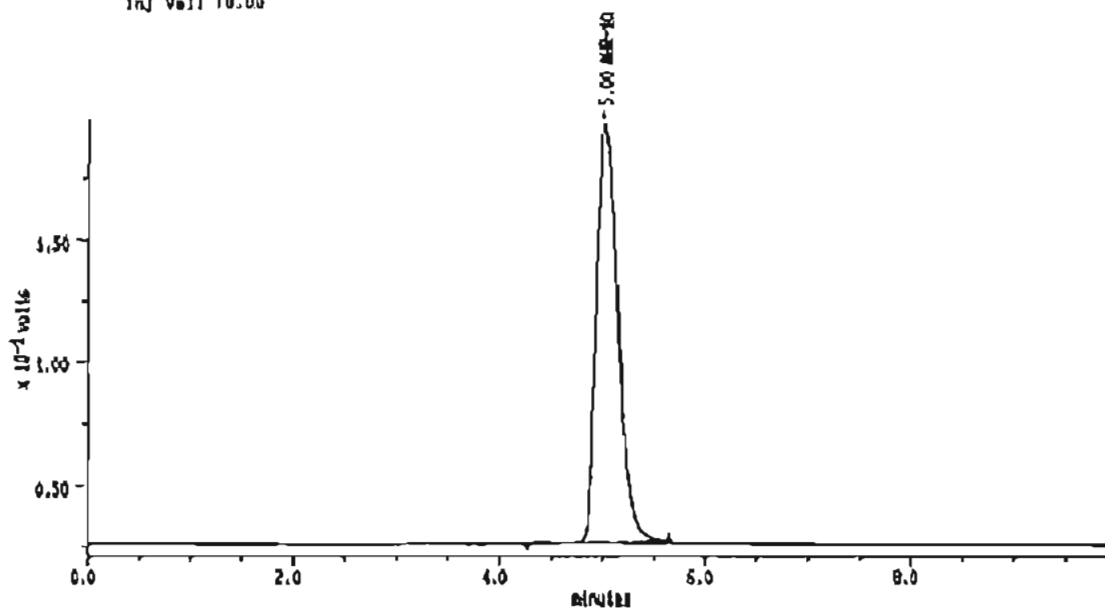
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	5.008	DS	2423877	108866	100.00	AHR-10282B
TOTAL			2423877	108866		

4

Sample: A19 70°C-28 Channel: detector 1 Filename: V2-03 Chart Speed: Full Size
 Acquired: 06-FEB-101 22:41 Method: B1(VA)IRV(H)KEY70-28 Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1990 Dynamic Solutions, Division of Millipore

MAXIMA 828 Custom Report

Printed: 0-FEB-1001 22:51:55

SAMPLE: A19 70°C-28

AS In Method: AHR-10282B

Acquired: 6-FEB-1001 22:41

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UHM

Instrument: Instrument 1

Filename: V2-03

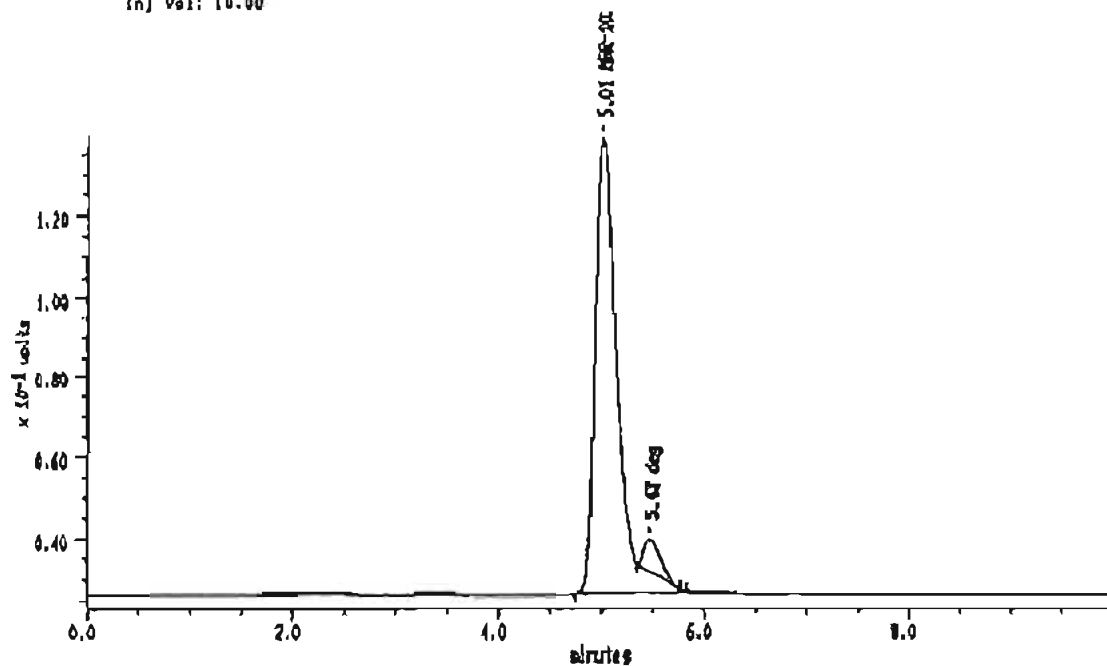
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	2486883	170447	100.00	AHR-10282B
TOTAL			2486883	170447		

Sample: A20 70°C-2W Channel: detector 1 Pileup: V2-04 Chart Speed: Full Size
 Acquired: 06-FEB-01 22:52 Method: A:VARRYHIBY70-2B Operator: S.S
 Inj Vol: 10.00



MAXIMA (a) 1990 Dynamic Solutions, Division of Millipore

MAXIMA 82B Custom Report

Printed: 6-FEB-2001 23:03:12

SAMPLE: A20 70°C-2W

as in Method: AHR-10282B

Acquired: 6-FEB-2001 22:52

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pileup: V2-04

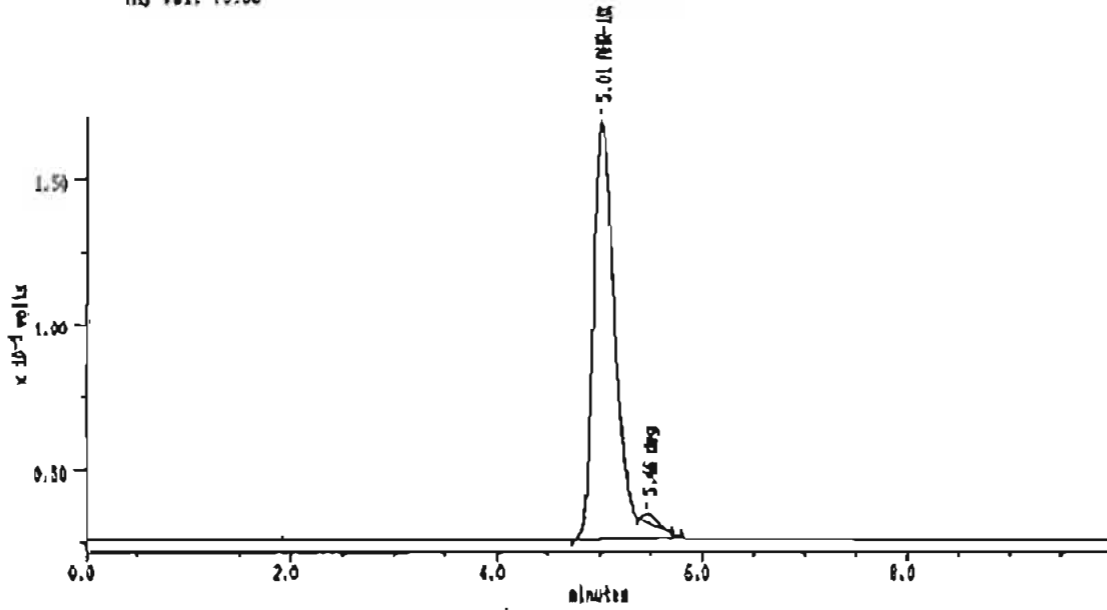
Index: 29

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	5.008	BB	1039028	112070	95.04	AHR-10282B
↑	5.487	ES	88144	7894	4.88	deg
TOTAL			1127172	118764		

Sample: ASI 70-C-20 Channel: detector 1 Pilenama: V2-06 Chart Speed: Full Size
 Acquired: 05-FEB-101 23:03 Method: B:YAHRYVHCY70-2# Operator: S.S
 Inj Vol: 10.00



MAXIMA 601890 Dynamic Software, Division of Millipore

MAXIMA 825 Custom Report

Printed: 5-FEB-2001 22:14:28

SAMPLE: ASI 70-C-20

#1 In Method: AHR-10282B

Acquired: 5-FEB-2001 23:03

Rate: 2.0 points/scan

Duration: 10.000 minutes

Operator: S.S

Type: LHM

Instrument: Instrument 1

Pilenama: V2-06

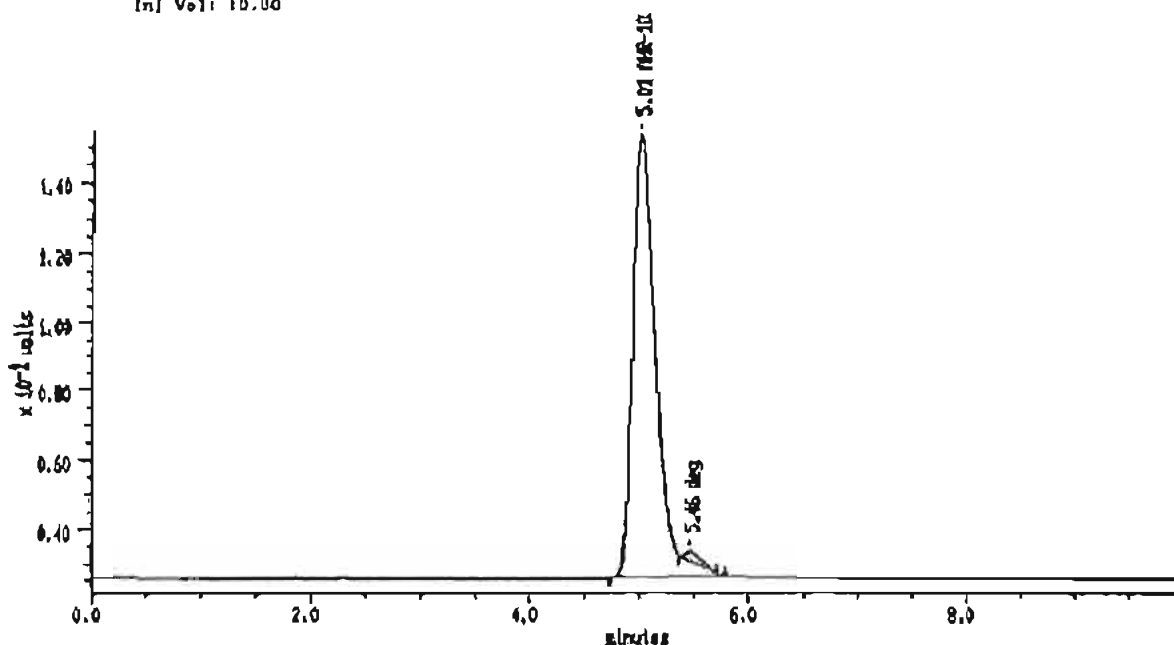
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	0.008	SB	2151512	144143	68.87	AHR-10282B
2	5.468	SS	26099	1828	1.33	deg
TOTAL			2180684	146269		

Sample: A22 70°C-2W Channel: detector 1 Pileup: V2-08 Chart Speed: Full Size
 Acquired: 08-PEB-101 23:18 Method: B:VAHRV1018710-2B Operator: S.S



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MAXIMA 825 Custom Report

Printed: 8-PEB-2001 23:26:48

SAMPLE: A22 70°C-2W

#0 In Method: AHR-10282B

Acquired: 8-PEB-2001 23:18

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pileup: V2-08

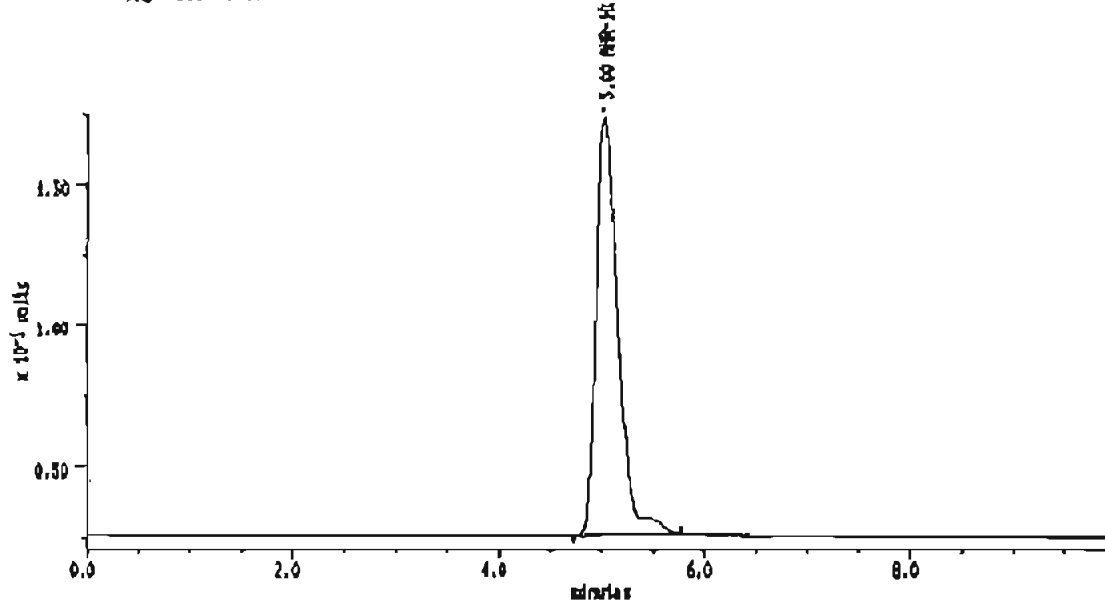
Index: 31

Injection Volume: 10.0

DETECTOR: Detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	7890449	127377	91.71	AHR-10282B
2	5.468	SS	24839	2426	1.29	deg
TOTAL			1921287	128602		

Sample: A23 10°C-2M Channel: detector 1 Filename: V2-07 Chart Speed: Full Size
 Acquired: 08-FEB-2001 23:28 Method: 0:YAHRY1118V30-2M Operator: S.5
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 8-FEB-2001 23:37:04

SAMPLE: A23 10°C-2M

#9 In Method: AHR-10282B

Acquired: 6-FEB-2001 23:28

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.5

Type: UNKN

Instrument: Instrument 1

Filename: V2-07

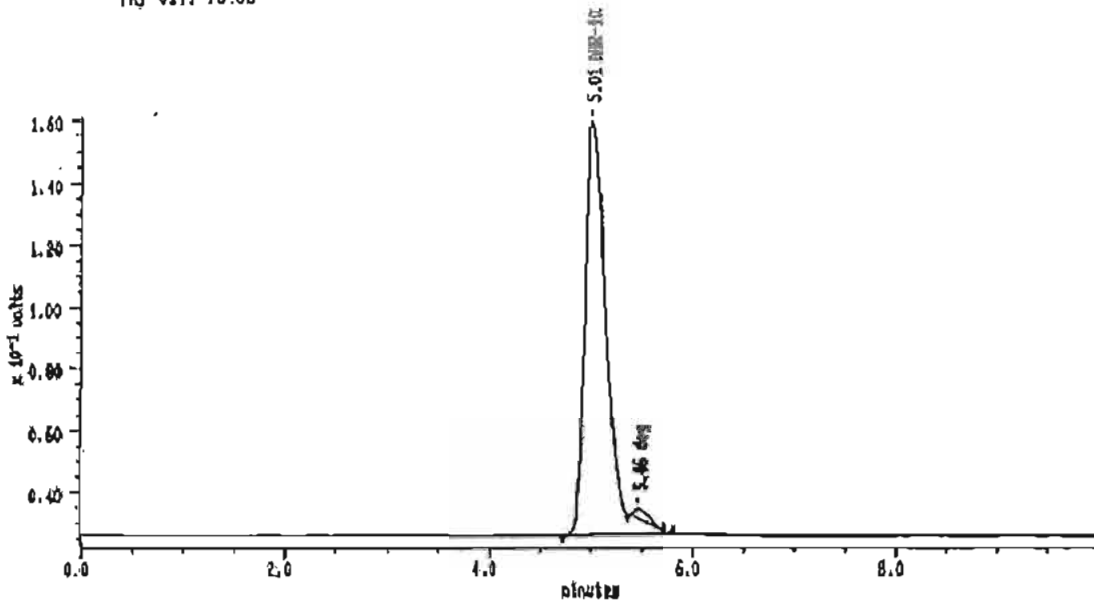
Index: 32

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	DB	2197150	147854	100.00	AHR-10282B
TOTAL			2197150	147854		

Sample: A24 70°C-2W Channel: detector 1 Filename: V2-08 Chart Speed: Full Size
 Acquired: 05-FEB-191 23:37 Method: B:VAHRV1H1EV70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 6-FEB-2001 23:49:28

SAMPLE: A24 70°C-2W

#10 In Method: AHR-10282B

Acquired: 6-FEB-2001 23:37

Rate: 2.0 points/min

Duration: 10,000 minutes

Operator: S.S

Type: DICH

Instrument: Instrument 1

Filename: V2-08

Index: 33

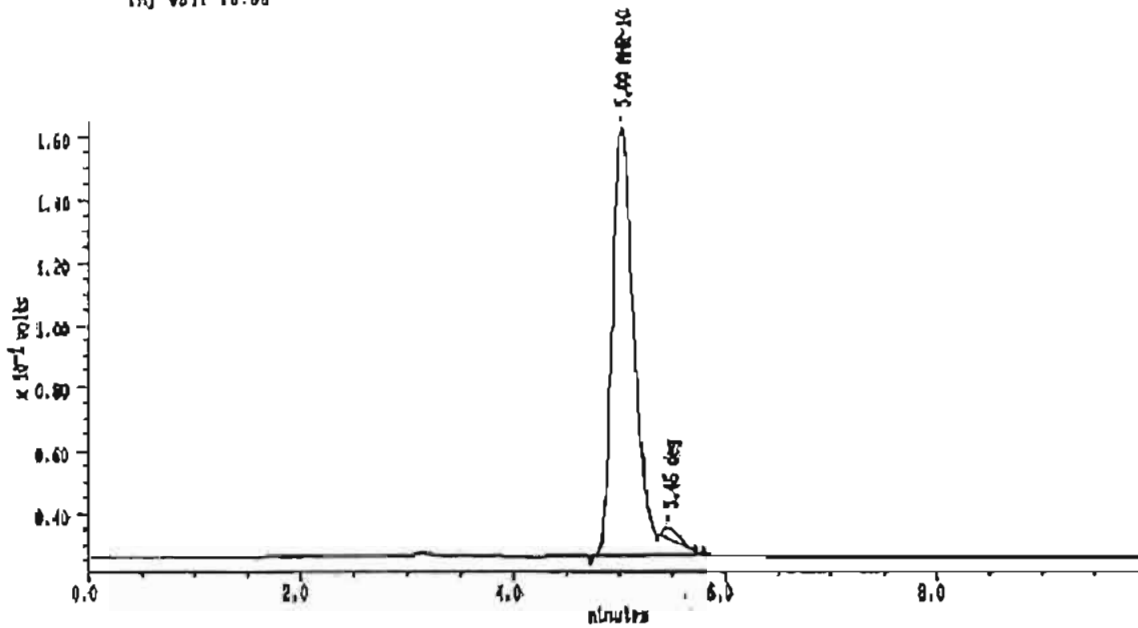
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	SB	1987388	102668	99.40	AHR-10282B
2	5.458	SS	32475	2674	1.60	dg
TOTAL			2020863	105342		

10

Sampler A25 70°C-2W Channel: detector 1 Pileup: V2-09 Chart Speed: Full Size
 Acquired: 05-FEB-2001 23:49 Method: D:\ANALYSIS\BY70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 6-FEB-2001 23:59:45

SAMPLE: A25 70°C-2W

File Method: AHR-10282B

Acquired: 6-FEB-2001 23:49

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNCH

Instrument: Instrument 1

Pileup: V2-09

Index: 34

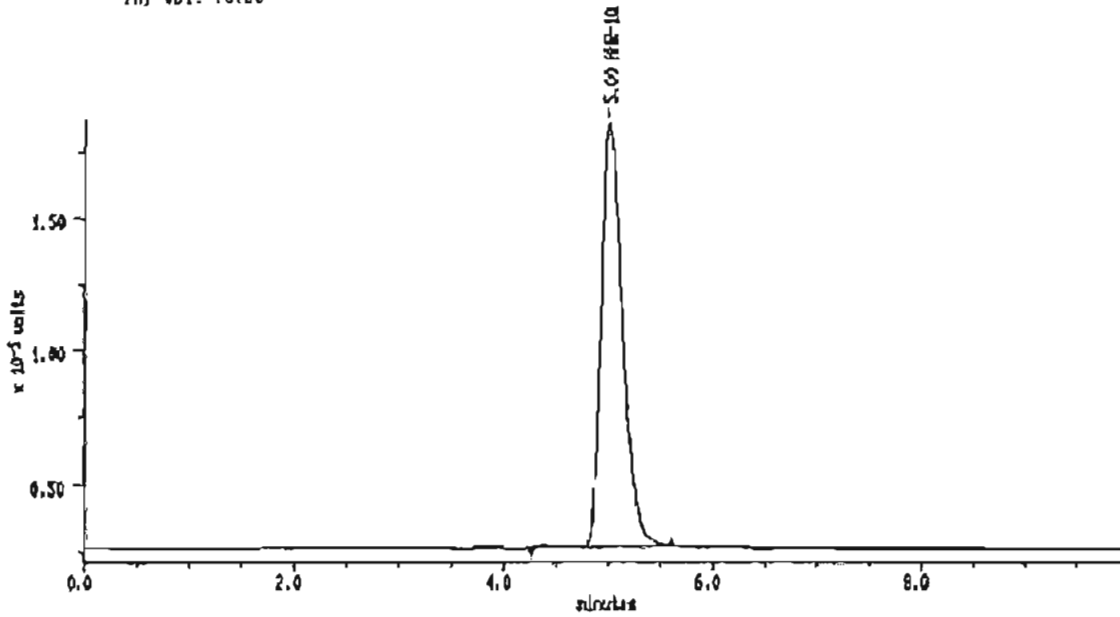
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	2044176	138477	88.18	AUH-10282B
2	5.458	SS	34138	2887	1.86	deg
TOTAL			2082808	140164		

11

Sample: A10 80°C-2W Channel: detector 1 Filament: V2-10 Chart Speed: Full Size
 Acquired: 08-FEB-2001 0:00 Method: B:TAHRYI16Y70-20 Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 8-FEB-2001 0:11:02

SAMPLES: A10 80°C-2W

#12 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:00

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filament: V2-10

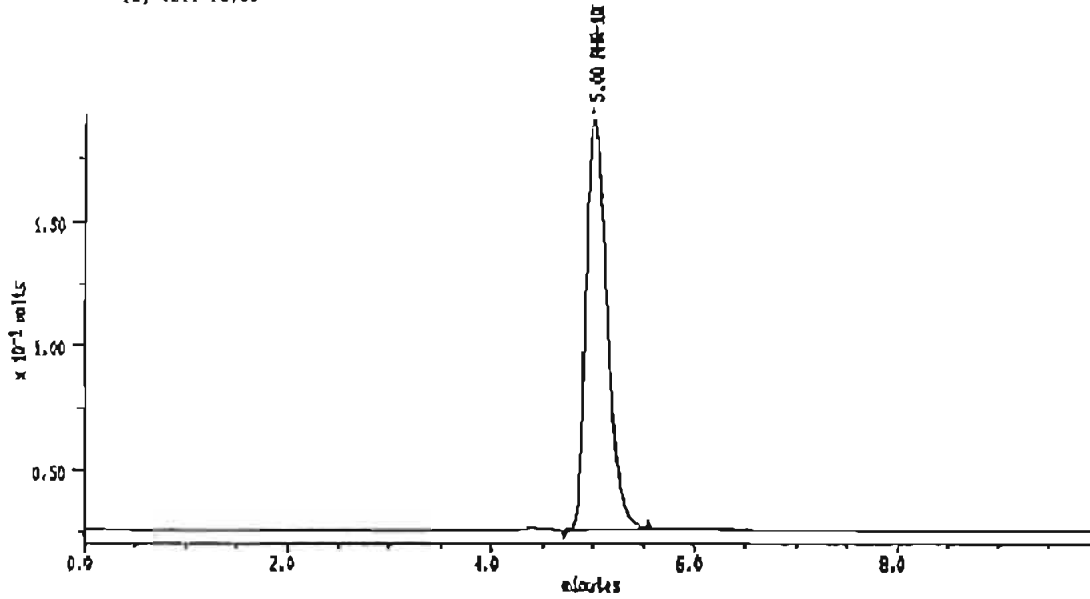
Index: 35

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	SS	2301723	150000	100.00	AHR-10282B
TOTAL			2301723	150000		

Sample: A19 60°C-2W Channel: detector 1 File Name: V2-11 Chart Speed: Full Scan
 Acquired: 06-FEB-2001 0:11 Method: D:\VALERY\HISTO-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1999 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 6-FEB-2001 0:22:13

SAMPLE: A19 60°C-2W

#10 In Method: AHR-10282B

Acquired: 6-FEB-2001 0:11

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

File Name: V2-11

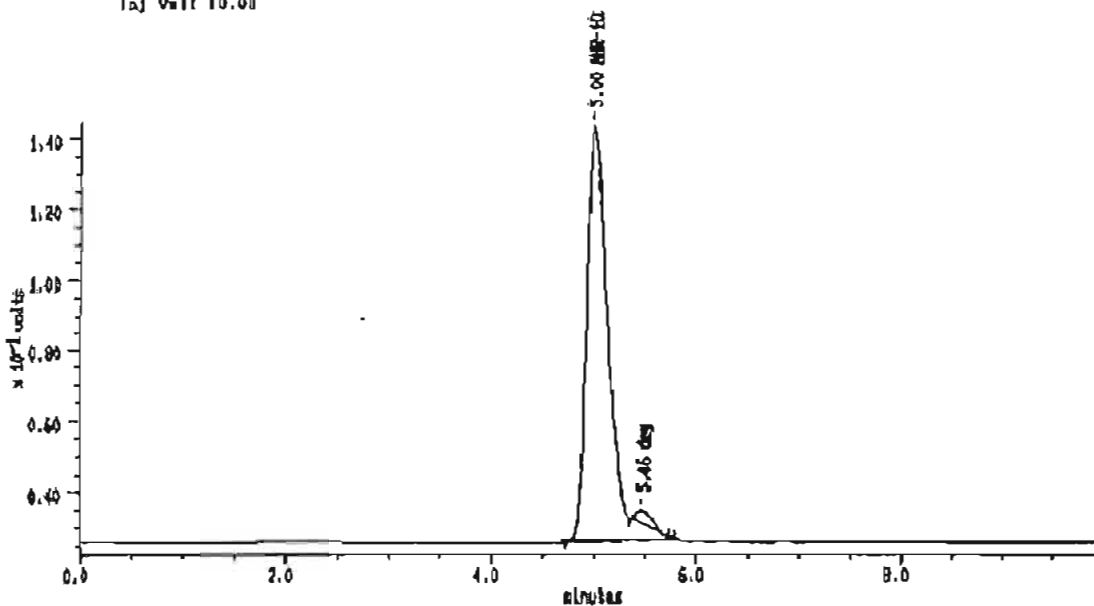
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	2360197	163976	100.00	AHR-10282B
TOTAL			2360197	163976		

Sample: A20 80°C-2W Channel: detector 1 Pileup: V2-12 Chart Speed: Full Size
 Acquired: 08-FEB-191 0:23 Method: B1YANRVIH75V70-2W Operator: S.S
 Inj. Vol: 10.00



MAXIMA 101880 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 8-FEB-2001 0:33:38

SAMPLE: A20 80°C-2W

#14 in Method: **AHR-10282B**

Acquired: 0-FEB-2001 0:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pileup: V2-12

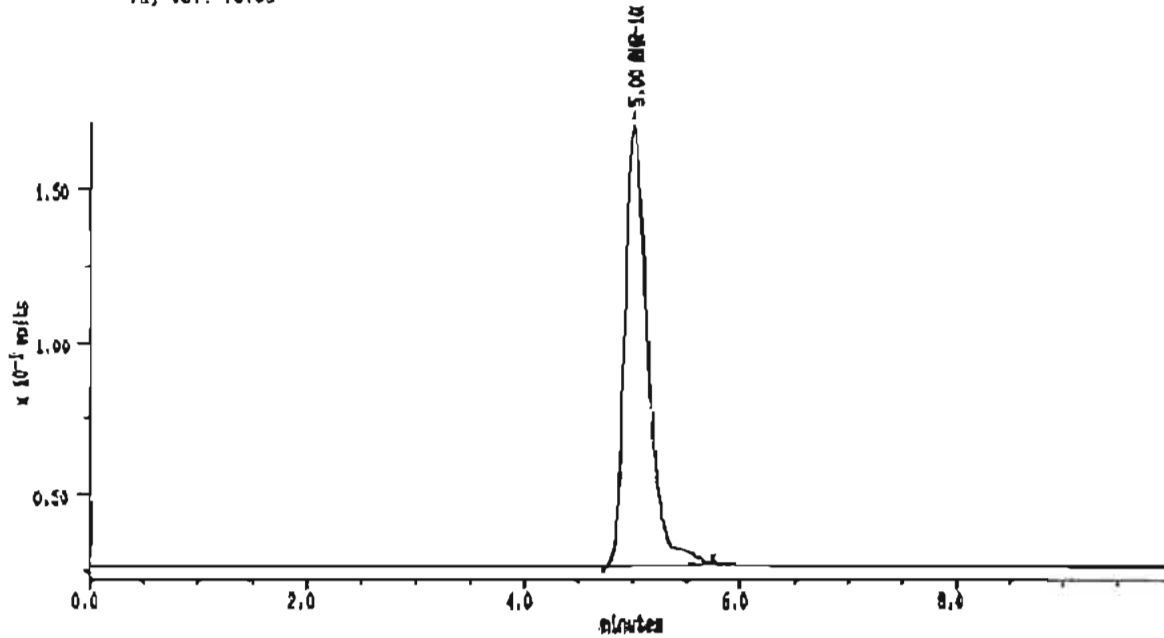
Index: 37

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	6.000	OB	1751139	117240	97.88	AHR-10282B
2	6.489	SE	41076	3832	2.54	det
TOTAL			1792215	121172		

Sample: A21 00°C-2W Channel: detector 1 Filenames: Y2-13 Chart Speed: Full Size
 Acquired: 08-FEB-10 0:34 Method: N1VAHRV1115W70-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1990 Dynamic Solutions, Division of Milligera

MAXIMA 828 Custom Report

Printed: 8-FEB-2001 0:48:01

SAMPLE: A21 00°C-2W

#16 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:34

Rate: 2.0 points/scan

Duration: 10.000 minutes

Operator: S.S

Type: UNKX

Instrument: Instrument 1

Filenames: Y2-13

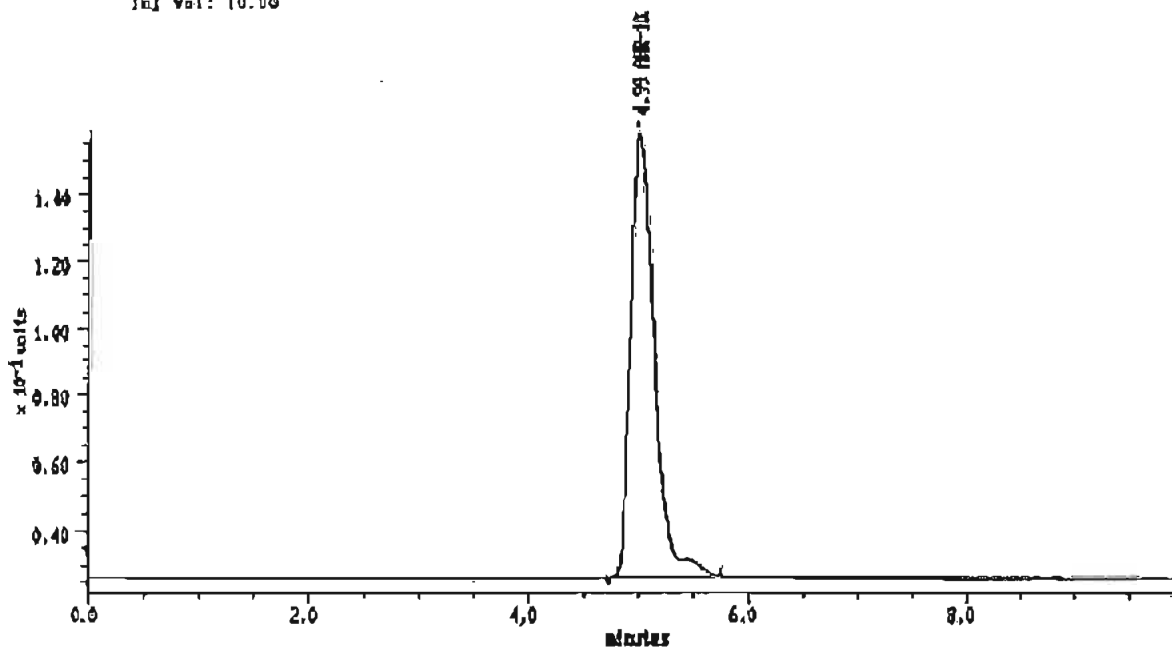
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BD	2140169	143778	100.00	AHR-10282B
TOTAL			2140169	143778		

Sample: A22 80°C-2W Channel: detector 1 Filename: V2-14 Chart Speed: Full Size
 Acquired: 08-FEB-10 0:45 Method: D:\ANALYSIS\70-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 6-FEB-2001 0:58:17

SAMPLE: A22 80°C-2W

MS In Method: AHR-10282B

Acquired: 6-FEB-2001 0:45

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-14

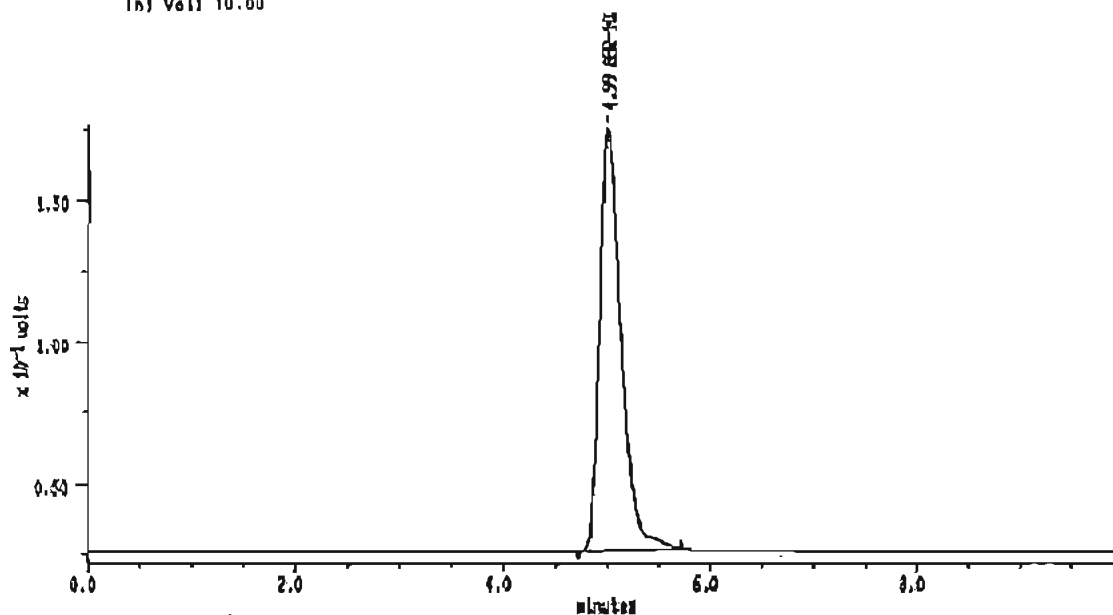
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	DB	1951230	131213	100.00	AHR-10282B
TOTAL			1951230	131213		

Sample: A23 60°C-2M Channel: detector 1 Pflmanar V2-16 Chart Speed: Full Size
 Acquired: 08-FEB-191 0:57 Method: BJTANRVIH18V70-2R Operator: S.S
 Inj Vol: 10.00



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MAXIMA 325 Custom Report

Printed: 8-FEB-2001 1:07:33

SAMPLE: A23 60°C-2M

File Method: AHR-10282B

Acquired: 8-FEB-2001 0:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Pflmanar: V2-16

Index: 40

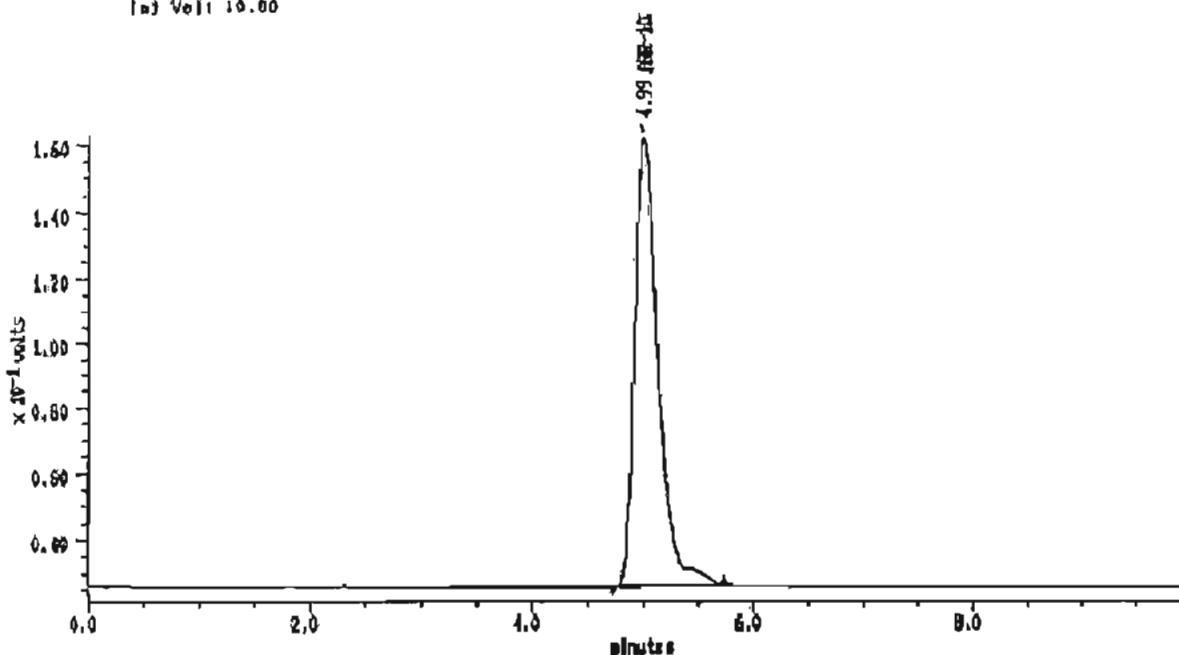
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.992	BB	210030	148146	100.00	AHR-10282B
TOTAL			210030	148146		

17

Sample: A24 10°C-2W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 08-FEB-10 1:08 Method: D:\AJORV\1\18\F10-2P Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 8-FEB-2001 11:18:50

SAMPLE: A24 10°C-2W

#10 in Method: AHR-10282B

Acquired: 8-FEB-2001 1:08

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-18

InjVol: 10

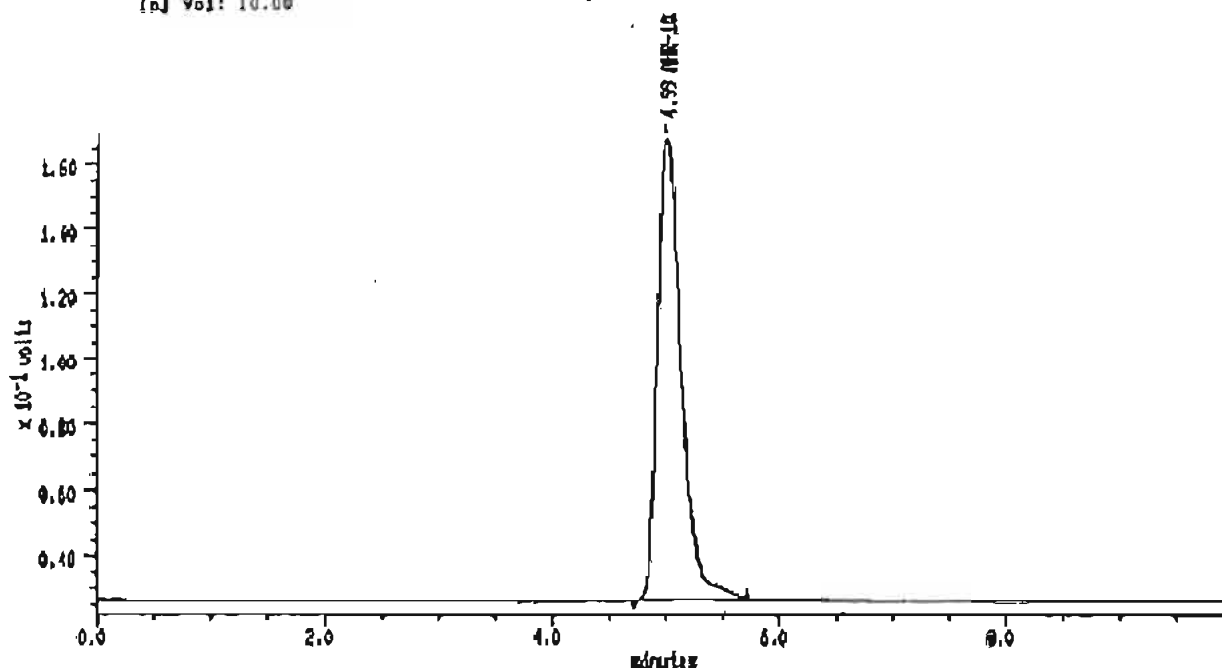
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.992	BB	2007388	135782	100.00	AHR-10282B
TOTAL			2007388	135782		

18

Sample: A25 80°C-2W Channel: detector 1 Filename: V2-17 Chart Speed: Full Size
 Acquired: 00-FEB-1991 1:18 Method: B:VAIRYIN16Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 6-FEB-2001 1:10:07

SAMPLE: A25 80°C-2W

File in Method: AHR-10282B

Acquired: 6-FEB-2001 1:19

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: GPC/M

Instrument: Instrument 1

Filename: V2-17

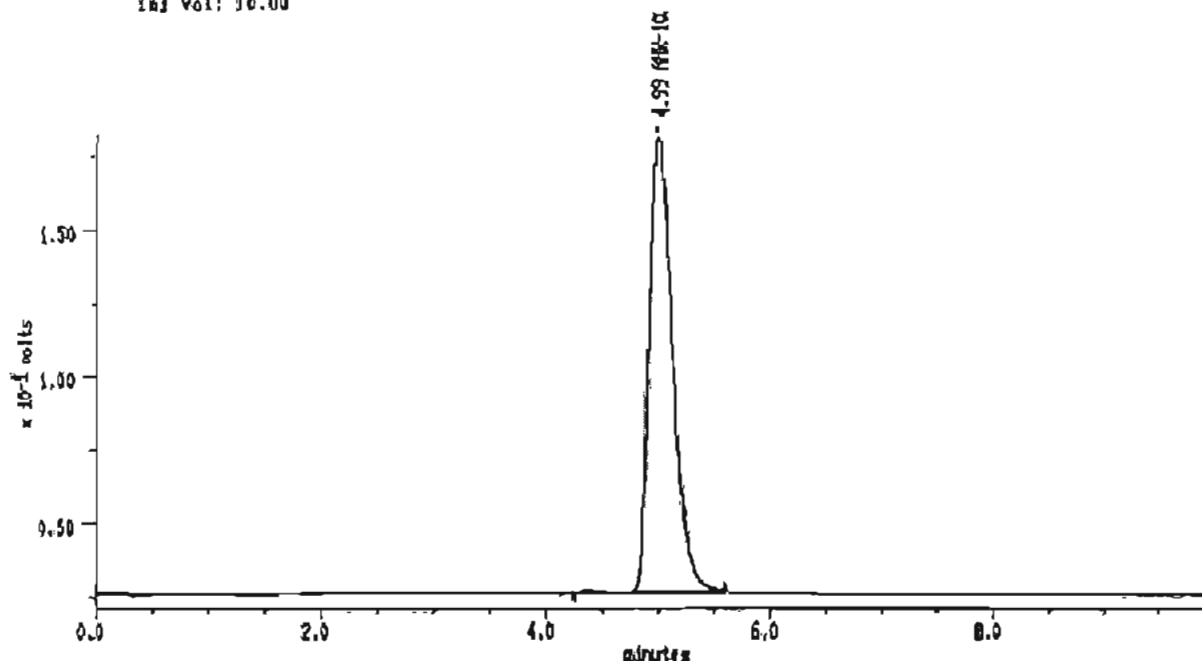
Index: 43

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.992	AD	2082273	141848	100.00	MIR-10292B
TOTAL			2082273	141848		

Sample: A18 50°C-2W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 08-FEB-01 1:30 Method: B:VAIRY\H16Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 8-FEB-2001 11:11:02

SAMPLE: A18 50°C-2W

#20 In Method: AHR-10282B

Acquired: 8-FEB-2001 1:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-18

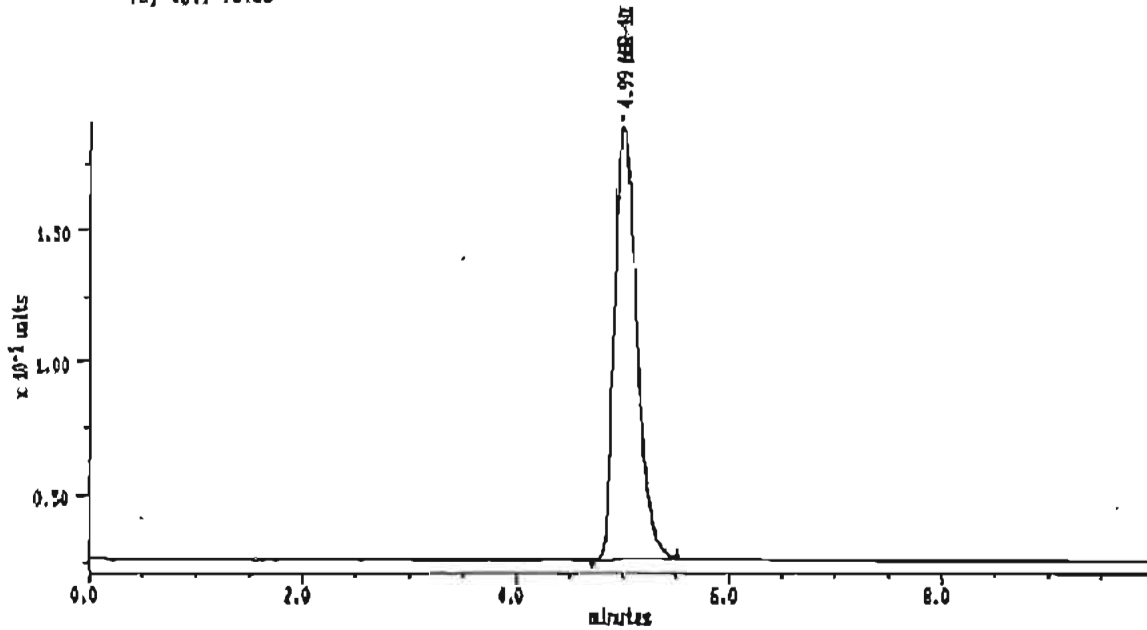
Index: 43

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	SB	2237401	154238	100.00	AHR-10282B
TOTAL			2237481	154238		

Sample: AIR 60°C-2W Channel: detector 1 Filename: V2-19 Chart Speed: Full Size
 Acquired: 08-FEB-2001 1:42 Method: D:\AJR\118\70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 8-FEB-2001 1:52:48

SAMPLE: AIR 60°C-2W

#21 in Method: AHR-10282B

Acquired: 8-FEB-2001 1:42

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: GUXN

Instrument: Instrument 1

Filename: V2-19

Index: 44

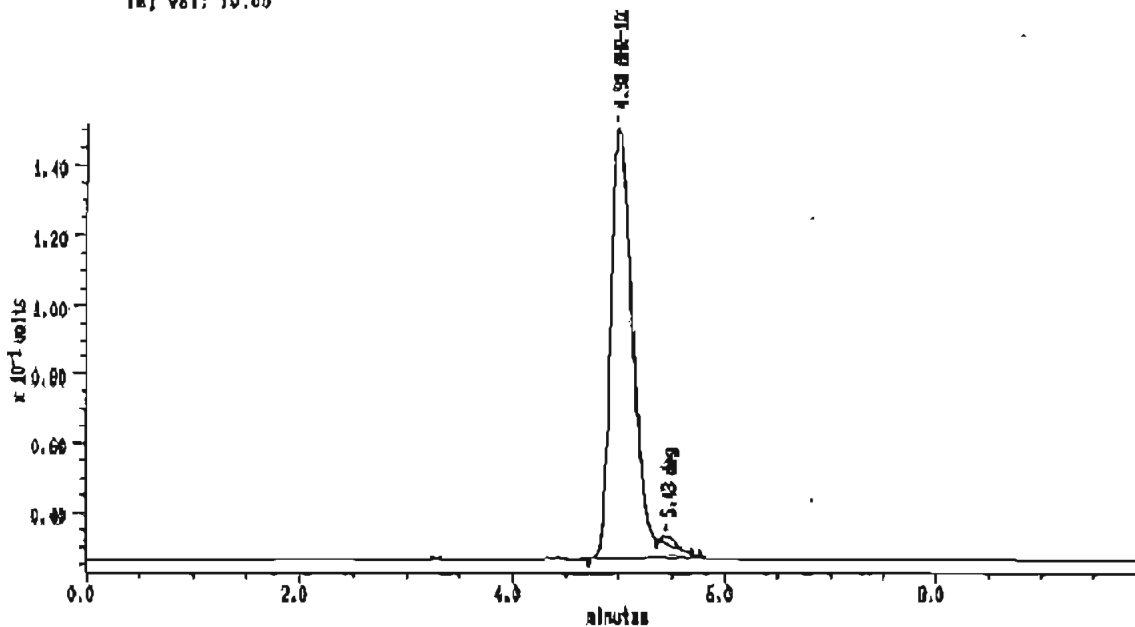
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	SB	2348444	183081	100.00	AIR-10282B
TOTAL			2348444	183081		

21

Sample: AZO 50°C-2W Channel: detector 1 Filename: V2-20 Chart Speed: Full Size
 Acquired: 06-FEB-10 1:53 Method: D:VAIRYI115W70-2W Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 6-FEB-2001 2:04:05

SAMPLES: AZO 50°C-2W

#22 in Method: AHR-10282B

Acquired: 6-FEB-2001 1:53

Rate: 5.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: V2-20

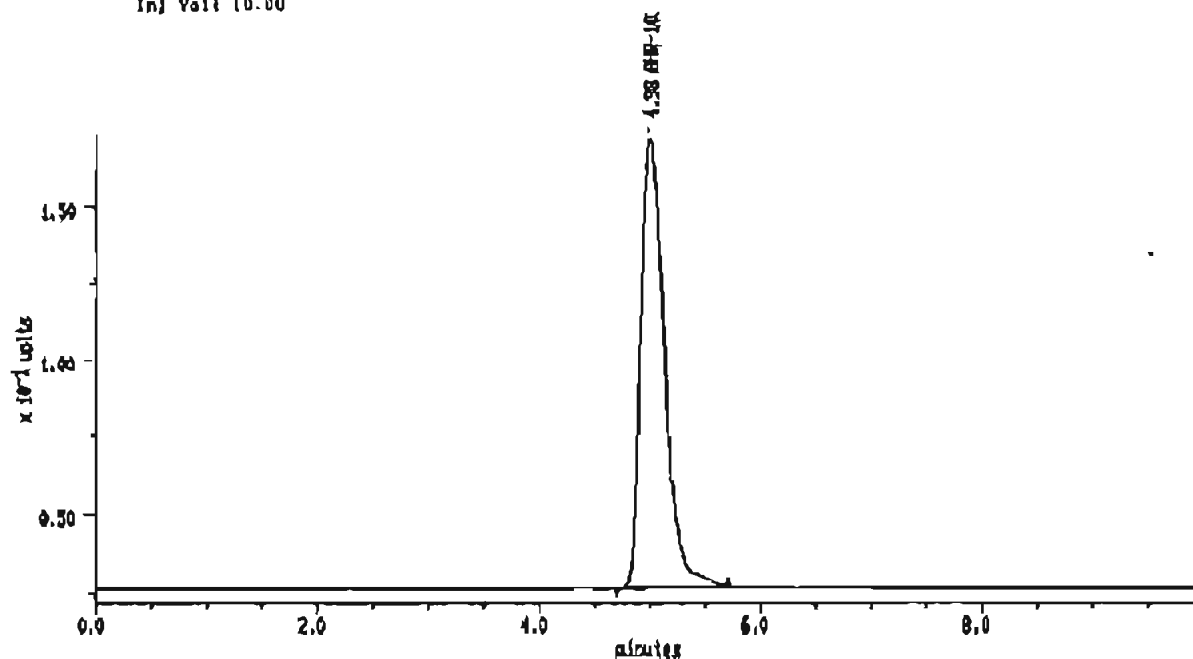
Index: 45

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	BB	1090004	124103	98.92	AHR-10282B
2	5.433	SS	21834	2107	1.10	deg
TOTAL			1091833	126210		

Sample: A21 60°C-2W Channel: detector 1 Filenames: V2-21 Chart Speed: Full Size
 Acquired: 08-FEB-10 2:04 Method: BIVAUHYIN10Y70-2W Operator: S.S
 Inj Volt: 10.00



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MAXIMA 825 Custom Report

Printed: 8-FEB-2001 2:15:23

SAMPLE: A21 60°C-2W

#23 In Method: AHR-10282B

Acquired: 8-FEB-2001 2:04

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UR2M

Instrument: Instrument 1

Filenames: V2-21

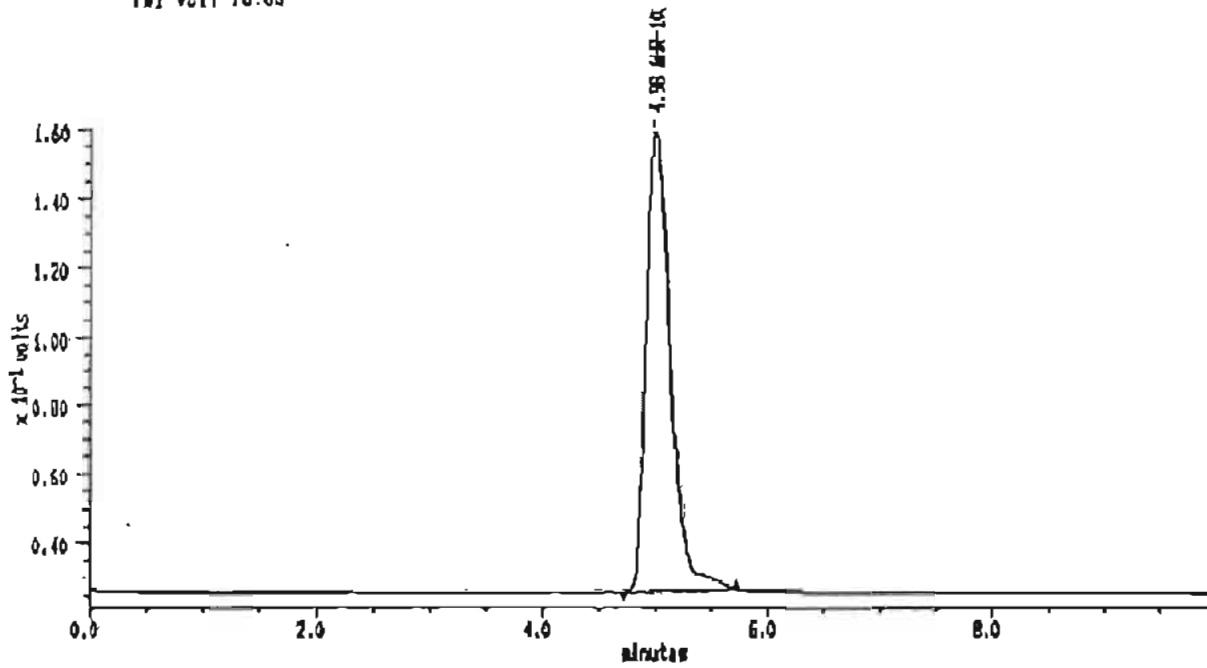
Index: 48

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	BB	2140898	146231	100.00	AHR-10282B
TOTAL			2140898	146231		

Sample: A22 80°C-2W Channel: detector 1 Filename: V3-22 Chart Speed: Full Size
 Acquired: 08-FEB-2001 2:18 Method: B:VAHRV1HLEBY70-2B Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 8-FEB-2001 2:28:08

SAMPLE: A22 80°C-2W

#24 In Method: AHR-10282B

Acquired: 8-FEB-2001 2:18

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V3-22

Inox: 47

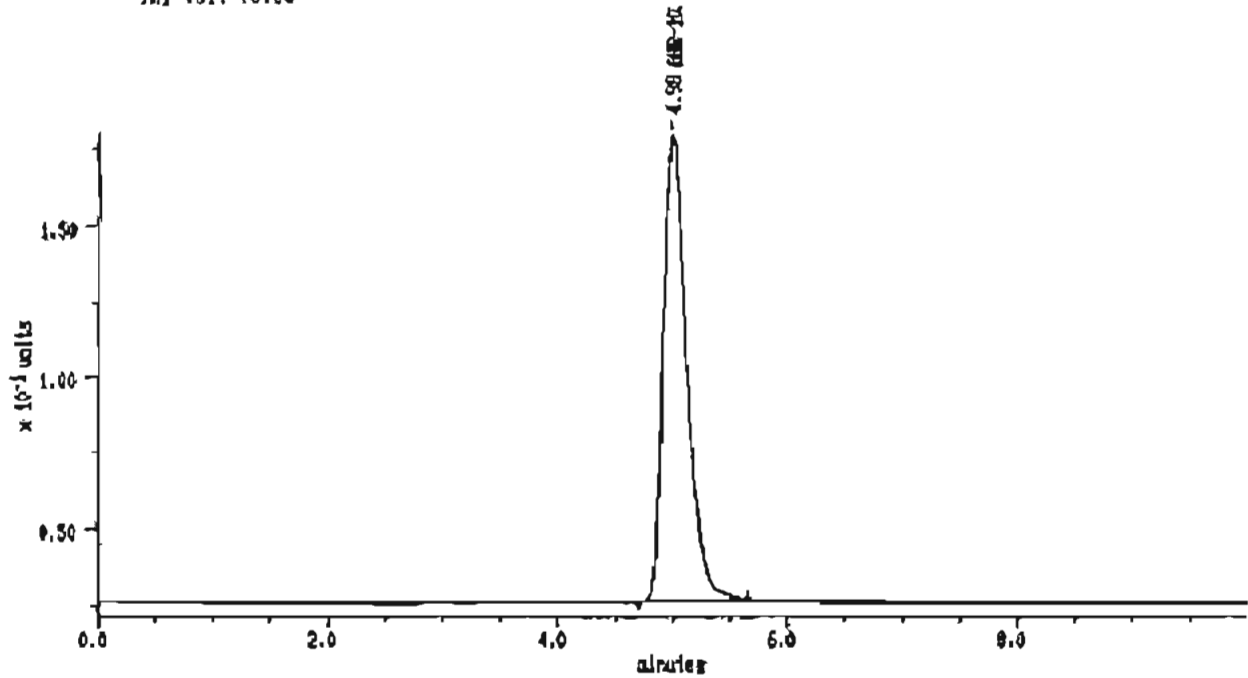
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	BB	1983893	133125	100.00	AHR-10282B
TOTAL			1983893	133125		

24

Sample: AZD 50°C-2W Channel: detector 1 Filename: V2-23 Chart Speed: Full Size
 Acquired: 00-FEB-01 2:37 Method: B:VAIUVI\H16V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 0-FEB-2001 2:58:13

SAMPLE: AZD 50°C-2W

#25 In Method: AHR-10282B

Acquired: 0-FEB-2001 2:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: QMKN

Instrument: Instrument 1

Filename: V2-23

Index: 48

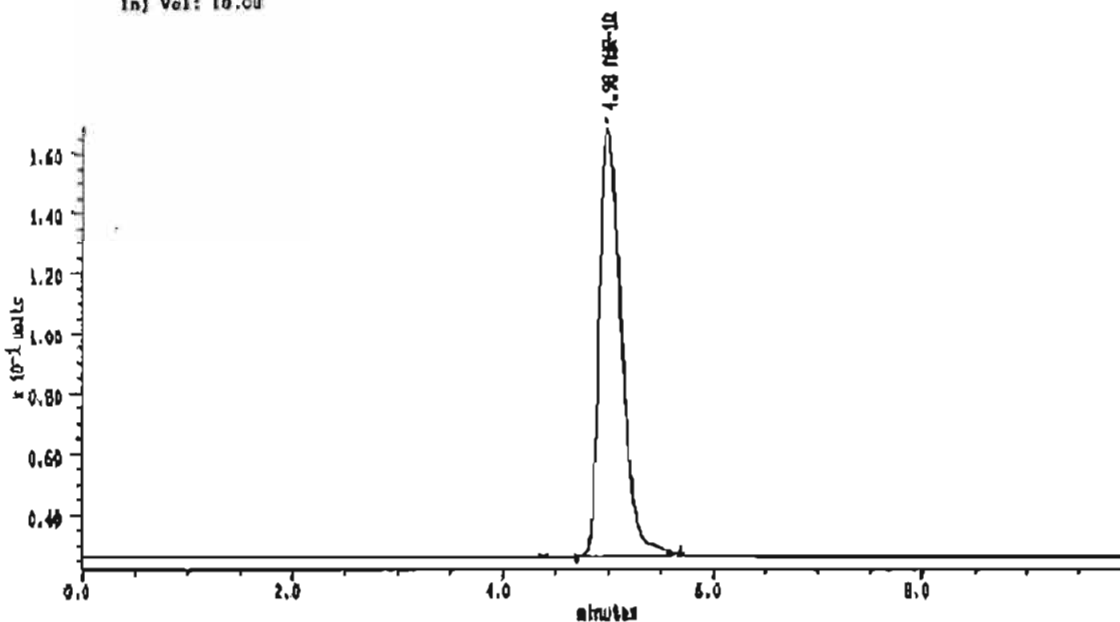
Injection Volume: 10.0

DETECTOR: (detector 1)

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.981	88	2218206	151731	100.00	AHR-10282B
TOTAL			2218206	151731		

25

Sample: A24 60°C-1W Channel: detector 1 Filename: V2-24 Chart Speed: Full Size
 Acquired: 08-FEB-10 1:39 Method: B:YARBYHIGYTO-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 8-FEB-2001 2:49:31

SAMPLE: A24 60°C-2W

#20 In Method: AHR-10282B

Acquired: 8-FEB-2001 2:39

Rate: 2.0 points/sec

Duration: 19.000 minutes

Operator: S.S

Type: WASH

Instrument: Instrument 1

Filename: V2-24

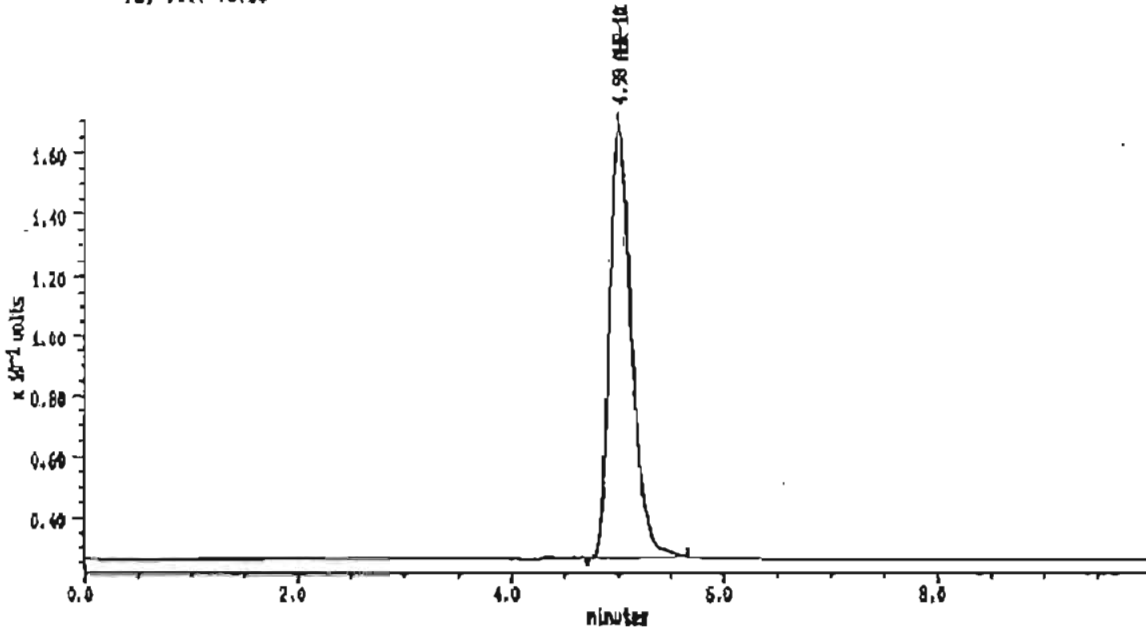
Index: 1

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.883	BB	2001841	141150	100.00	AHR-10282B
TOTAL			2001841	141150		

Sample: A25 50°C-2M Channel: detector 1 Filename: V1-25 Chart Speed: Full Size
 Acquired: 08-FEB-10 2:50 Method: D:\ANALYSIS\70-2M Operator: S.S
 Inj Vol: 10.00



MAXIMA (c)1980 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 8-FEB-2001 3:00:46

SAMPLE: A25 50°C-2M

#27 IA Method: AHR-10282B

Acquired: 8-FEB-2001 2:50

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: CHN

Instrument: Instrument 1

Filename: V1-25

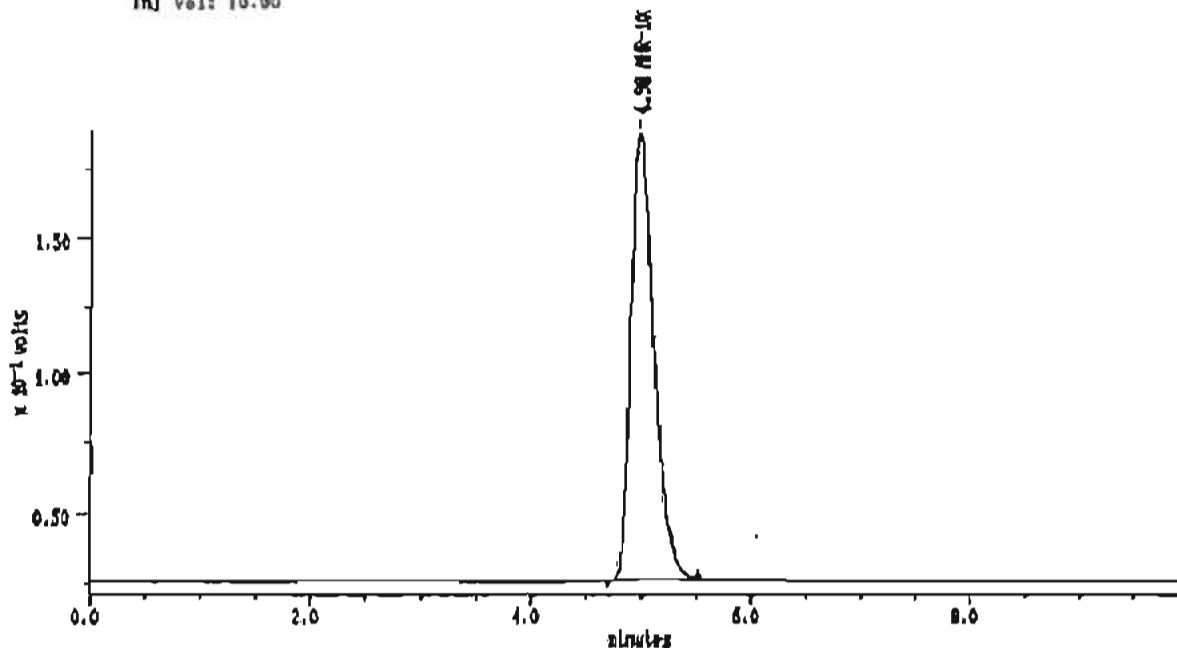
InjVol: 10.0

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.903	SD	2080804	142720	100.00	AHR-10282B
TOTAL			2080804	142720		

Sample: STD Channel: detector 1 Pileup: V2-28 Chart Speed: Full Size
 Acquired: 08-FEB-2001 3:01 Method: B1YAIRVIRI6Y70-2W Operator: S.S
 Inj Vol: 10.00



MAXIMA 101880 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 8-FEB-2001 3:12:18

SAMPLE: STD

Method: AHR-10282B

Acquired: 8-FEB-2001 3:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URXN

Instrument: Instrument 1

Pileup: V2-28

Index: 3

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.975	OB	2528584	181115	100.00	AHR-10282B
TOTAL			2528584	181115		

Bronuck Ophthalmic Solution Stability Test
 Lot No. 01H151

Test code: P2000B177
 Tester: Shirou Sawa
 Test date: 22 January 2001

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Collac(%)	Water	Initial present	
STD	1	IN-01	2217554						
STD	2	IN-26	2251148						
STD	mean		2234351	1.0005					
A-18	Initial	IN-02	2289485	1.0252	100				
A-19	Initial	IN-03	2350952	1.0527	100				
A-20	Initial	IN-04	2358326	1.0560	100				
A-21	Initial	IN-05	2388197	1.0694	100				
A-22	Initial	IN-06	2292061	1.0263	100				
A-23	Initial	IN-07	2358324	1.0560	100				
A-24	Initial	IN-08	2303165	1.0313	100				
A-25	Initial	IN-09	2216054	0.9923	100				
A-18	70°C-1W	IN-10	2242130	1.0040	97.93	94.32	3.69	8.6101	8.4269
A-19	70°C-1W	IN-11	2280528	1.0257	97.44	93.79	3.75	8.5729	8.3884
A-20	70°C-1W	IN-12	1951517	0.8739	82.76	79.67	3.86	8.5198	8.3317
A-21	70°C-1W	IN-13	2203526	0.9867	92.27	88.75	3.82	8.5739	8.3860
A-22	70°C-1W	IN-14	2082220	0.9324	90.85	87.33	3.87	8.5469	8.3574
A-23	70°C-1W	IN-15	2197753	0.9841	93.19	89.75	3.69	8.5791	8.3975
A-24	70°C-1W	IN-16	2095221	0.9382	90.97	87.58	3.73	8.5713	8.3879
A-25	70°C-1W	IN-17	2131377	0.9544	86.18	82.44	3.89	8.6647	8.3735
A-18	60°C-1W	IN-18	2234601	1.0006	97.60	95.88	1.76	8.5329	8.4468
A-19	60°C-1W	IN-19	2288796	1.0249	97.36	95.86	1.54	8.5779	8.5021
A-20	60°C-1W	IN-20	1948910	0.8727	82.64	81.28	1.65	8.5953	8.5137
A-21	60°C-1W	IN-21	2161205	0.9677	90.49	89.07	1.57	8.6548	8.4779
A-22	60°C-1W	IN-22	2091491	0.9365	91.25	89.73	1.67	8.5393	8.4579
A-23	60°C-1W	IN-23	2243646	1.0047	95.14	93.29	1.94	8.5813	8.4681
A-24	60°C-1W	IN-24	2079710	0.9313	90.30	88.84	1.62	8.5863	8.5064
A-25	60°C-1W	IN-25	2086854	0.9345	94.18	92.66	1.61	8.5624	8.4835

*Re-edited in order to document the data necessary for calculation.
 Shirou Sawa, 6 May 2005*

1/30

Preparation Record B (drug solution) Form 7 (15 November 2000)

Test substance	AHR10282B		Test code	P2000B177		Lot No.	0001H151		Preparation date	15 January 2001		Tester	Shirou Sawa		
Test item															
Amount manufactured	5mL colorless ampoules × 19			5mL colorless PP ×			×		×		×		×		
Formulation No.	A-18		A-19		A-20		A-21		A-22						
Ingredients and amounts	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Manufacturer Lot No.		
Ingredient	100 mL		100 mL		mL										
Bromfenac sodium	0.1	0.100	0.1	0.101	0.1	0.101	0.1	0.100	0.1	0.100	0.1	0.100			
Boric acid	1.1	1.099	1.1	1.102	1.5	1.496	1.5	1.502	1.5	1.497					
Borax	1.1	1.103	0.1	1.103	-		-		-		-				
Benzalkonium chloride	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	-				
Polysorbate 80	0.15	0.150	-		0.15	0.167	-		0.15	0.149	0.15	0.149			
Tyloxapol	-		0.15	0.14153	-		0.15	0.152	0.15	0.149	0.15	0.149			
Chlorhexidine gluconate	-		-		-		-		-		0.005	0.005			
pH	8.3	8.23	8.3	8.24	7	7.05	7	7.03	7	7.09					

1/15 15:48
NO.62 PH 8.23
22.6°C

1/15 15:52
NO.63 PH 8.24
22.5°C

1/15 15:55
NO.54 PH 7.05
22.5°C

1/15 16:02
NO.65 PH 7.93
22.5°C

1/15 16:08
NO.66 PH 7.89
22.9°C

1/15 16:12
NO.67 PH 7.88
22.8°C

1/15 16:14
NO.68 PH 6.99
22.8°C

1/15 16:25
NO.69 PH 7.88
22.8°C

Preparation Record B (drug solution) Form 7 (15 November 2000) Test substance		Test code		Lot No.		Preparation date		Tester	Shirou Sawa		
Test item											
Amount manufactured	5mL colorless ampoules ×		5mL colorless PP ×		5mL brown PP ×		×	×	×		
Formulation No.	A-23		A-24		A-25						
Ingredients and amounts	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Manufacturer Lot No.
	mL		mL		mL		mL				
<i>Bromfenac sodium</i>	0.1	0.101	0.1	0.100	0.1	0.101					
<i>Boric acid</i>	1.5	1.502	1.5	1.496	1.5	1.501					
<i>Borax</i>	-		-		-						
<i>Benzalkonium chloride</i>	-	-	0.005	0.005	0.005	0.005					
<i>Polysorbate 80</i>	-		-		-						
<i>Tylnxapol</i>	0.15	0.152	0.15	0.150	0.15	0.151					
<i>Chlorhexidine gluconate</i>	0.005	0.005	-		-						
<i>Methylglucamine</i>	-		0.3005	0.050	0.5	0.501					
<i>pH</i>	7	7.08		6.99		7.08					

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2000B177	Test date	22 January 2001		
Test item					Tester	Shirou Sawa	
STD solution 0.02001g/ 20mL x 2mL/20mLMP				22-Jan-2001	16:48:12		
				0021	+	0.02001 g	
		PH	Initial	Measurement	Turbidity	Foreign matter	Color
A18	Initial	8.23			-	-	Yellow
A19		8.22			-	-	↓
A20		8.7.02			-	-	↓
A21		6.98			-	-	↓
A22		7.05			-	-	↓
A23		6.99			-	-	↓
A24		6.93			-	-	↓
A25		7.00			-	-	↓
A18	70°C-1W	8.16	8.6101	8.4269	-	±	↓
A19		8.17	8.5729	8.3884	-	±	↓
A20		6.97	8.5196	8.3317	-	++(r)	Slight brown
A21		6.95	8.5739	8.3860	-	++(r)	Slight brown
A22		7.07	8.5469	8.3574	-	++(r)	Dark yellow
A23		7.05	8.5791	8.3975	+	+(r)	Yellow
A24		6.99	8.5713	8.3879	-	++(r)	Dark yellow
A25		7.07	8.5647	8.3735	+	+	Yellow
A18	60°C-1W	8.27	8.5329	8.4468	-	-	↓
A19		8.25	8.5779	8.5021	-	-	↓
A20		7.05	8.5953	8.5137	+	+(r)	Dark yellow
A21		7.03	8.5548	8.4779	+	±	↓
A22		7.08	8.5393	8.4579	+	+	Yellow
A23		7.07	8.5613	8.4661	+	±	↓
A24		6.96	8.5863	8.5064	+	±	Dark yellow
A25		7.08	8.5624	8.4835	+	±	Yellow

1/22 17:47
NO. 9 PH 6.23
24.3°C

1/22 17:47
NO. 10 PH 6.22
24.3°C

1/22 17:48
NO. 11 PH 7.02
24.3°C

1/22 17:49
NO. 12 PH 6.98
24.3°C

1/22 17:50
NO. 13 PH 7.05
24.3°C

1/22 17:50
NO. 14 PH 6.99
24.4°C

1/22 17:51
NO. 15 PH 6.95
24.4°C

1/22 17:52
NO. 16 PH 7.00
24.4°C

1/22 17:53
NO. 17 PH 6.16
24.2°C

1/22 17:54
NO. 18 PH 6.17
24.4°C

1/22 17:55
NO. 19 PH 6.97
24.5°C

1/22 17:55
NO. 20 PH 6.95
24.5°C

1/22 17:56
NO. 21 PH 7.07
24.4°C

1/22 18:00
NO. 22 PH 7.05
24.4°C

1/22 18:02
NO. 23 PH 6.99
24.4°C

1/22 18:05
NO. 24 PH 7.07
24.5°C

1/22 18:07
NO. 25 PH 6.27
24.4°C

1/22 18:09
NO. 26 PH 6.25
24.3°C

1/22 18:10
NO. 27 PH 7.05
24.3°C

1/22 18:12
NO. 28 PH 7.03
24.3°C

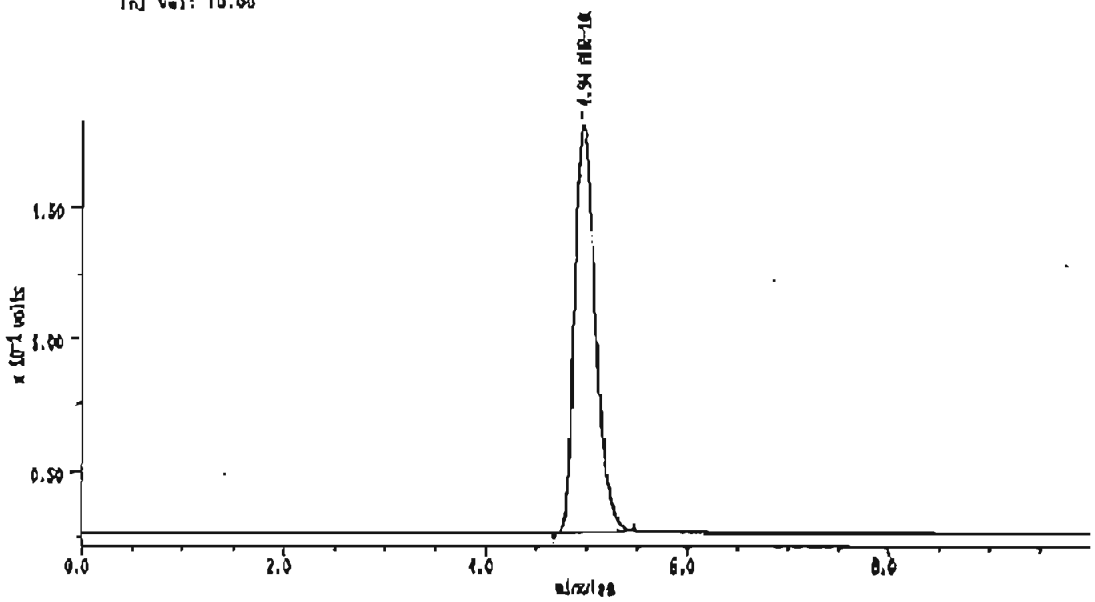
1/22 18:16
NO. 29 PH 7.23
24.3°C

1/22 18:18
NO. 30 PH 7.07
24.2°C

1/22 18:15
NO. 31 PH 6.95
24.2°C

1/22 18:21
NO. 32 PH 7.03
24.2°C

Sample: STD1 Channel: detector 1 Filename: 1N-01 Chart Speed: Full Size
 Acquired: 22-JAN-01 19:56 Method: D:\AHR\116\1\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 20:09:27

SAMPLE: 5761

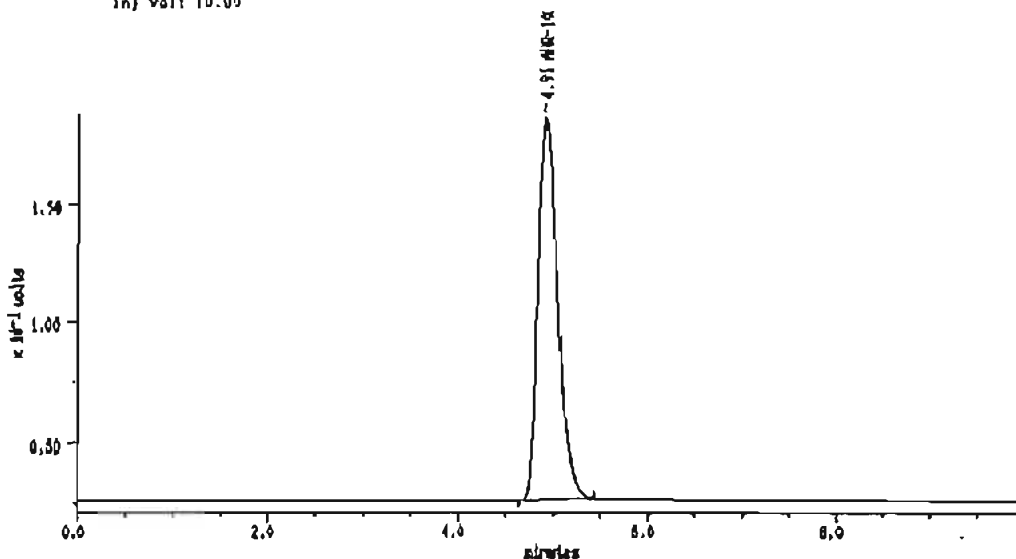
NO In Method: AHR-10282B
 Acquired: 22-JAN-2001 19:56
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: CHECK
 Instrument: Instrument 1
 Filename: 1N-01
 Index: 1
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.943	BB	2217554	154658	100.00	AHR-10282B
TOTAL			2217554	154658		

Sample: AIB INITIAL Channel: detector 1 Filenames: IX-02 Chart Speed: Full Size
 Acquired: 22-JAN-2001 20:12 Method: B:VALKYRIE\AIB\INITIAL Operator: S.S
 Inj. Volt: 10.00



MAXIMA Gel 1920 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 22-JAN-2001 20:22:53

SAMPLE: AIB INITIAL

Method: AHR-10282B

Acquired: 22-JAN-2001 20:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UHPLC

Instrument: Instrument 1

Filenames: IX-02

Index: 3

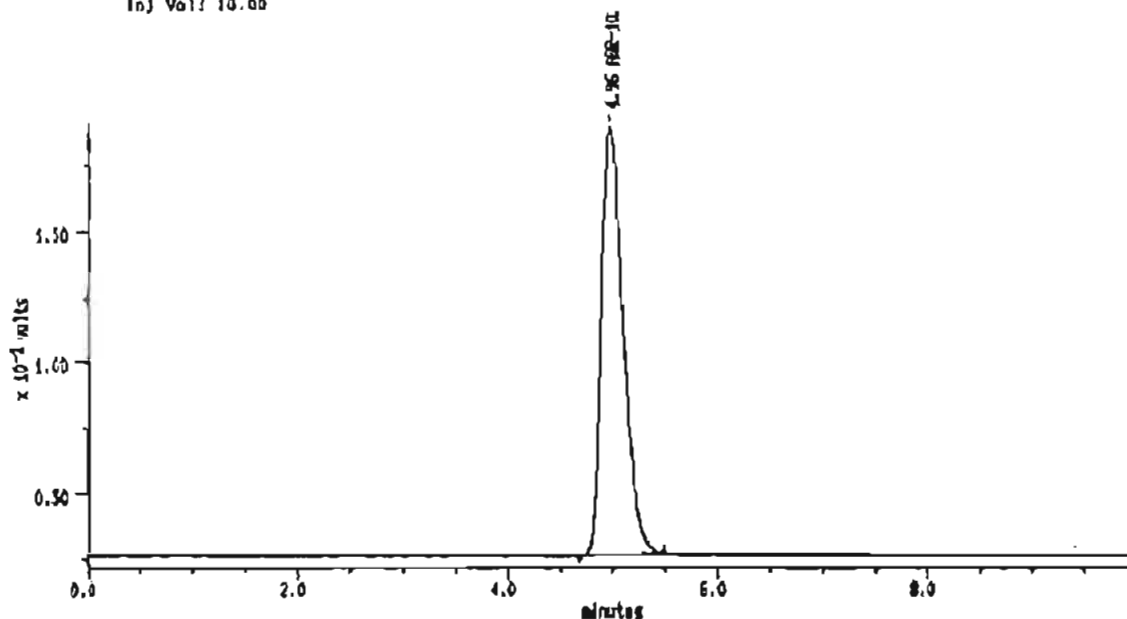
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.908	SB	2188485	159801	100.00	AIB-10282B
TOTAL			2289485	159801		

6

Sample: A39 INITIAL Channel: detector 1 Filenames: IN-03 Chart Speed: Full Size
 Acquired: 22-JAN-10 20:23 Method: D:\MSDCHEM\A39\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 20:34:10

SAMPLE: A39 INITIAL

MS (A Method: AHR-10282B

Acquired: 22-JAN-2001 20:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UHPLC

Instrument: Instrument 1

Filenames: IN-03

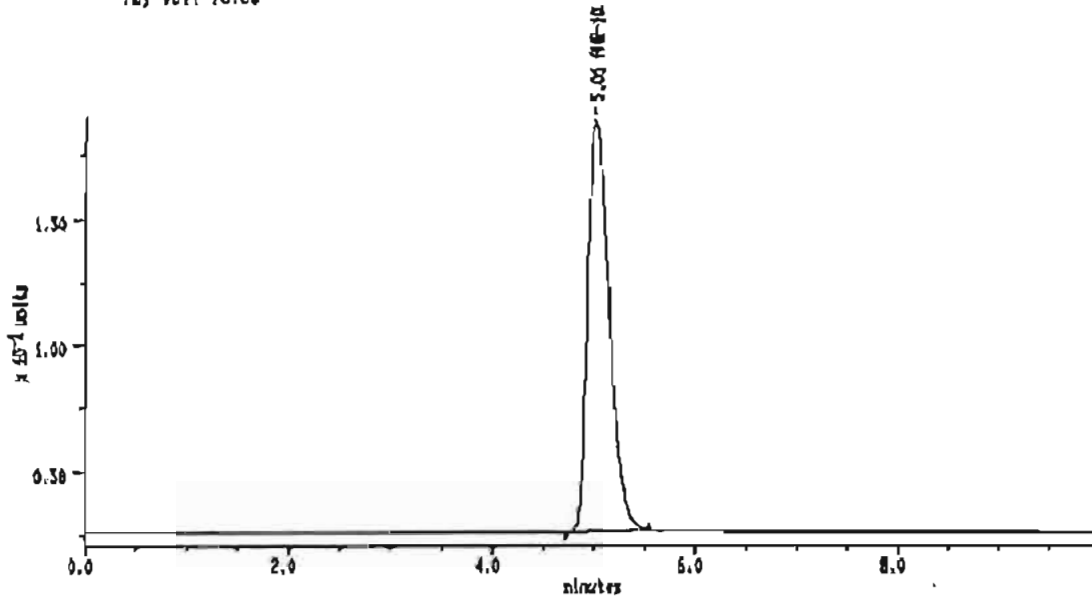
Index: 3

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.960	BB	2350052	102614	100.00	AHR-10282B
TOTAL			2350052	102614		

Sample: A20 INITIAL Channel: detector 1 File Name: 1N-04 Chart Speed: Full Size
 Acquired: 22-JAN-2001 20:34 Method: 8:VAJRWIMIS/INITIAL Operator: S.S
 Inj Volt: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 20:48:17

SAMPLE: A20 INITIAL

#8 in Method: AHR-10282B

Acquired: 22-JAN-2001 20:34

Rate: 1.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

File Name: 1N-04

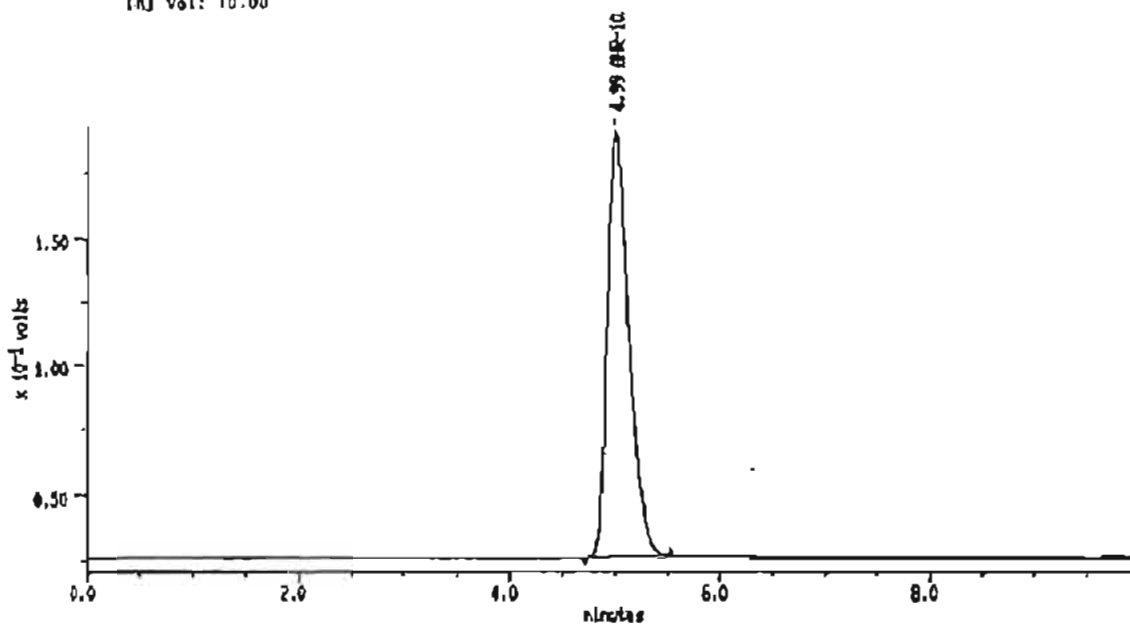
Inject: 1

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	2358328	181823	100.00	AHR-10282B
TOTAL			2358328	181823		

Sample: A21 INITIAL Channel: detector 1 Filename: 1X-06 Chart Speed: Full Size
 Acquired: 22-JAN-2001 20:46 Method: B:TAHRMINIBYINITIAL Operator: S.S
 (n) Vol: 10.00



MAXIMA (el1090 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 22-JAN-2001 20:56:42

SAMPLE: A21 INITIAL

#7 In Method: AHR-10282B

Acquired: 22-JAN-2001 20:46

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: 1X-06

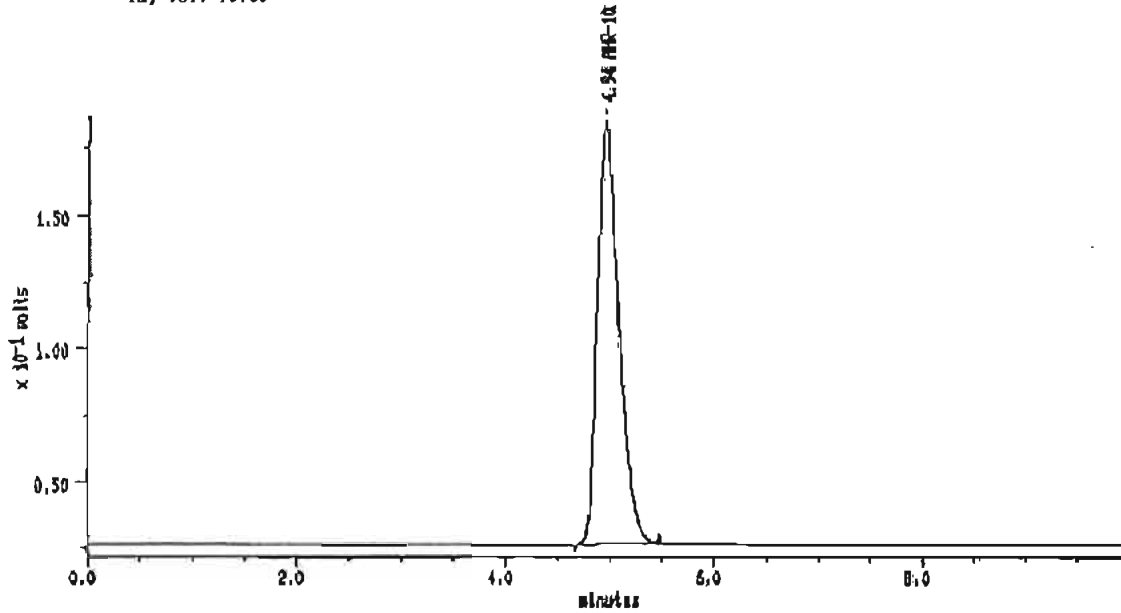
Index: 6

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.982	SD	2308187	106928	100.00	AHR-10282B
TOTAL			2308187	106928		

Sample: A22 INITIAL Channel: detector 1 Filenama: IX-06 Chart Speed: Full Size
 Acquired: 22-JAN-2001 20:57 Method: B:YAKRYIHININITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 : Custom Report

Printed: 22-JAN-2001 21:07:50

SAMPLE: A22 INITIAL

#8 In Method: AHR-10282B

Acquired: 22-JAN-2001 20:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URM

Instrument: Instrument 1

Filenama: IX-06

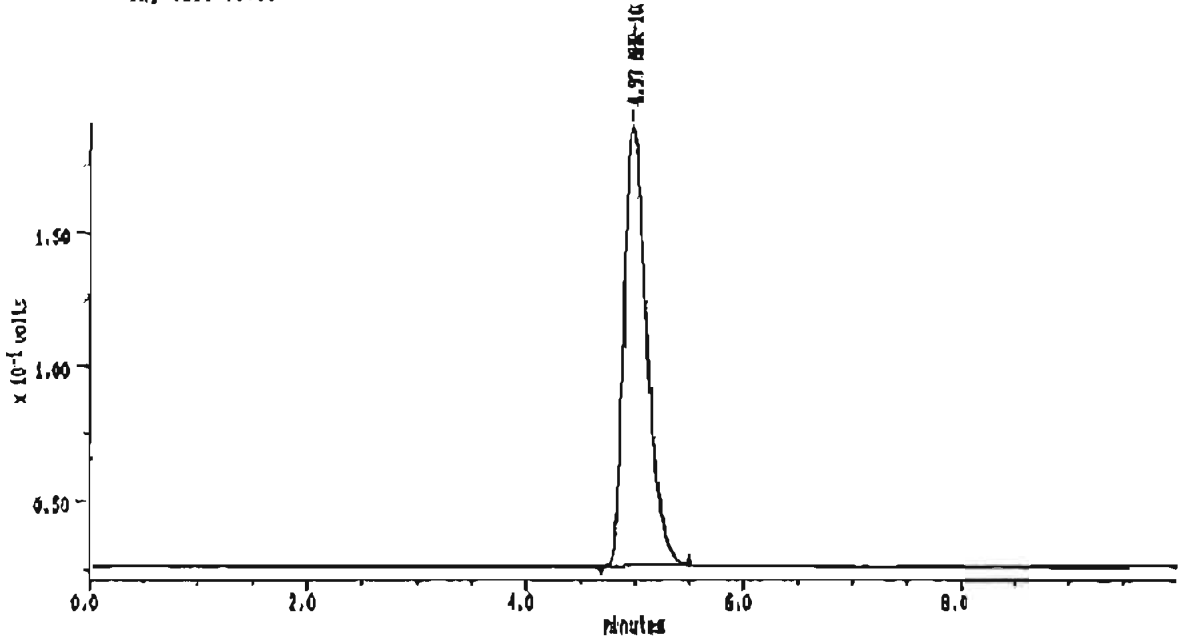
Index: 8

Injection Volume: 10.0

DETECTOR: detector 1

PX#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.942	88	2292001	158725	100.00	AHR-10282B
TOTAL			2292001	158725		

Sample: A23 INITIAL Channel: detector 1 Pileup: IN-07 Chart Speed: Full Size
 Acquired: 22-JAN-1991 21:08 Method: D:VARIYIHSYIINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 21:18:16

SAMPLE: A23 INITIAL

as is Method: AHR-10282B

Acquired: 22-JAN-2001 21:08

Rate: 2.0 ml/min

Duration: 10.000 minutes

Operator: S.S

Type: CONIC

Instrument: Instrument 1

Pileup: IN-07

Index: 7

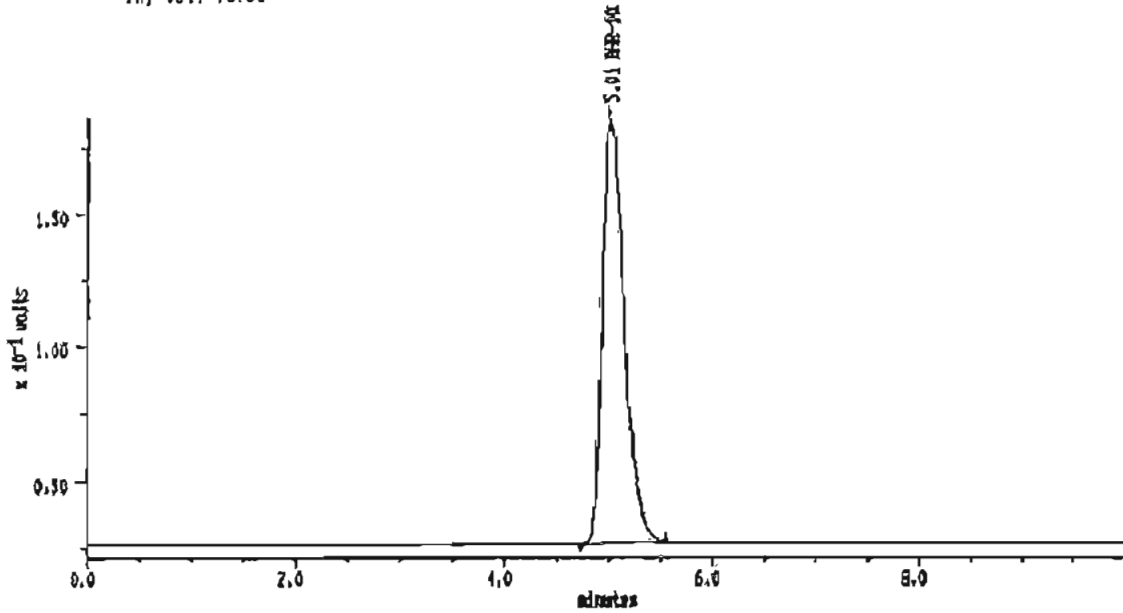
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.967	OB	2360324	102394	100.00	AHR-10282B
TOTAL			2360324	102394		

11

Sample: A24 INITIAL Channel: detector 1 Filename: IX-08 Chart Speed: Full Size
 Acquired: 22-JAN-01 21:20 Method: B:VALONTIRISVINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 21:30:32

SAMPLE: A24 INITIAL

#10 in Method: AHR-10282B

Acquired: 22-JAN-2001 21:20

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IX-08

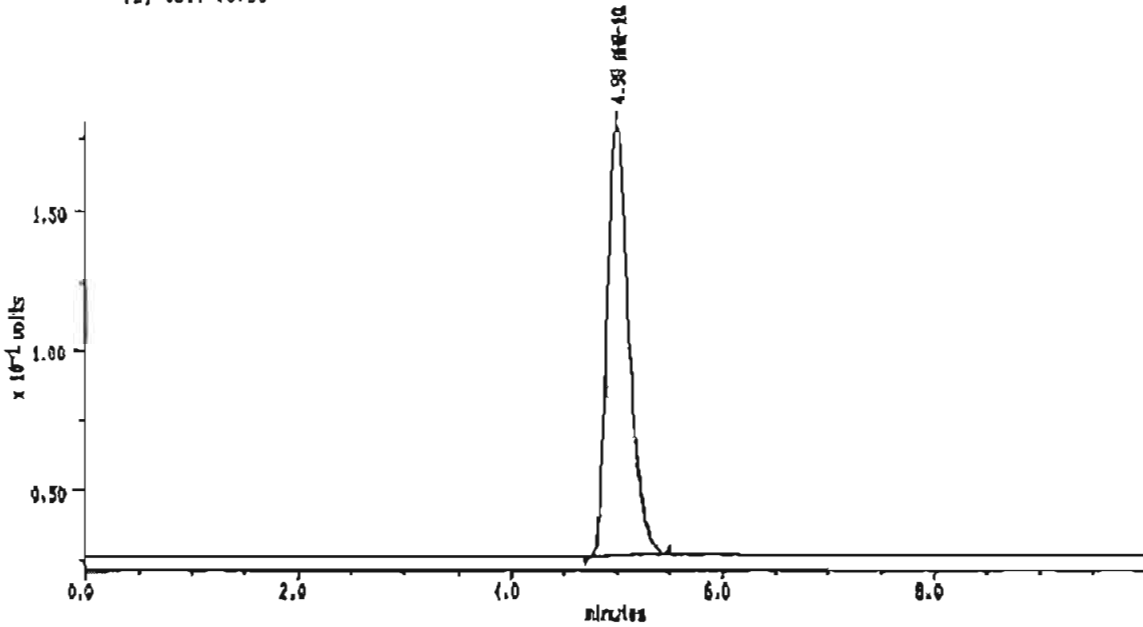
Index: 0

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time minutes	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	SS	2303165	150455	100.00	AHR-10282B
TOTAL			2303165	150455		

Sample: A26 INITIAL Channel: detector 1 Filament: IM-05 Chart Speed: Full Size
 Acquired: 22-JAN-2001 21:31 Method: G:VAURVIRI16VINITIAL Operator: S.S
 Inj Vol: 10.00



MAXIMA (c)1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 Custom Report

Printed: 22-JAN-2001 21:41:48

SAMPLE: A26 INITIAL

File Method: AHR-10282B

Acquired: 22-JAN-2001 21:31

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filament: IM-05

Inject: 9

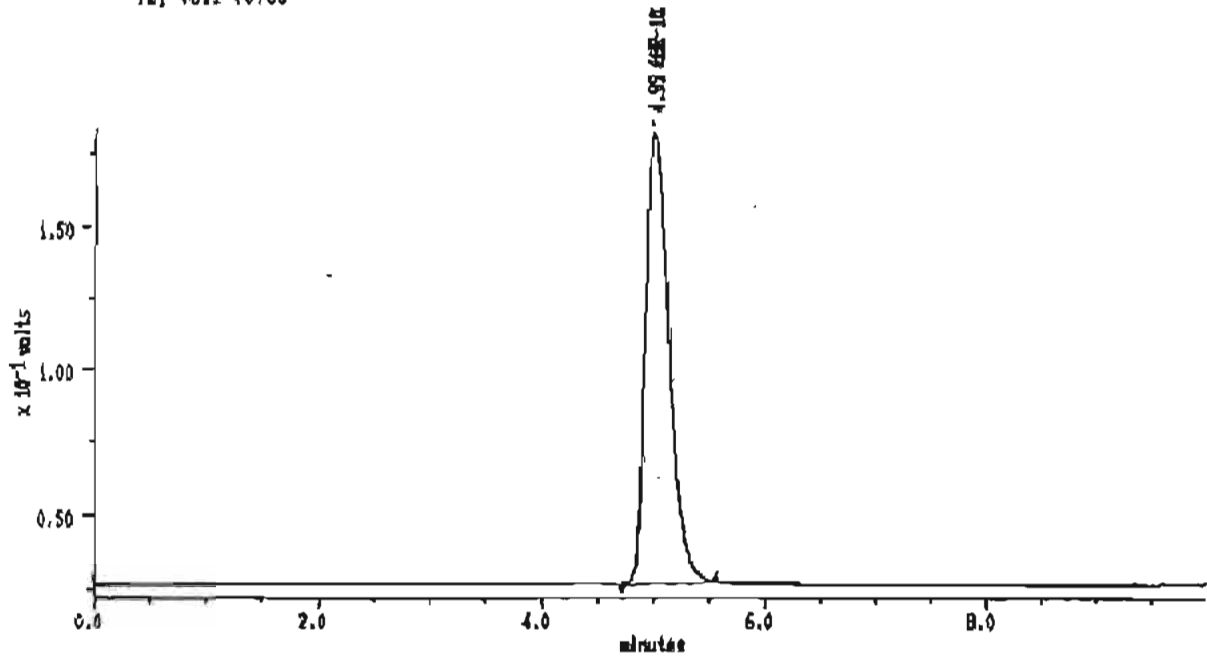
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.375	SM	2210364	103004	100.00	AHR-10282B
TOTAL			2210364	103004		

13

Sample: A18 70°C-1W Channel: detector 1 Filename: 18-10 Chart Speed: Full Size
 Acquired: 22-JAN-10 21:42 Method: D:\ANALYSIS\INITIAL Operator: S.S
 Inj Vol: 10.00



MAXIMA (c)1996 Dynamic Solutions, Division of Millipore

MAXIMA 825 ; Custom Report

Printed: 22-JAN-2001 21:53:04

SAMPLE: A18 70°C-1W

#12 in Method: AHR-10282B

Acquired: 22-JAN-2001 21:42

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNK

Instrument: Instrument 1

Filename: 18-10

Index: 10

Injection Volume: 10.0

DETECTOR: detector 1

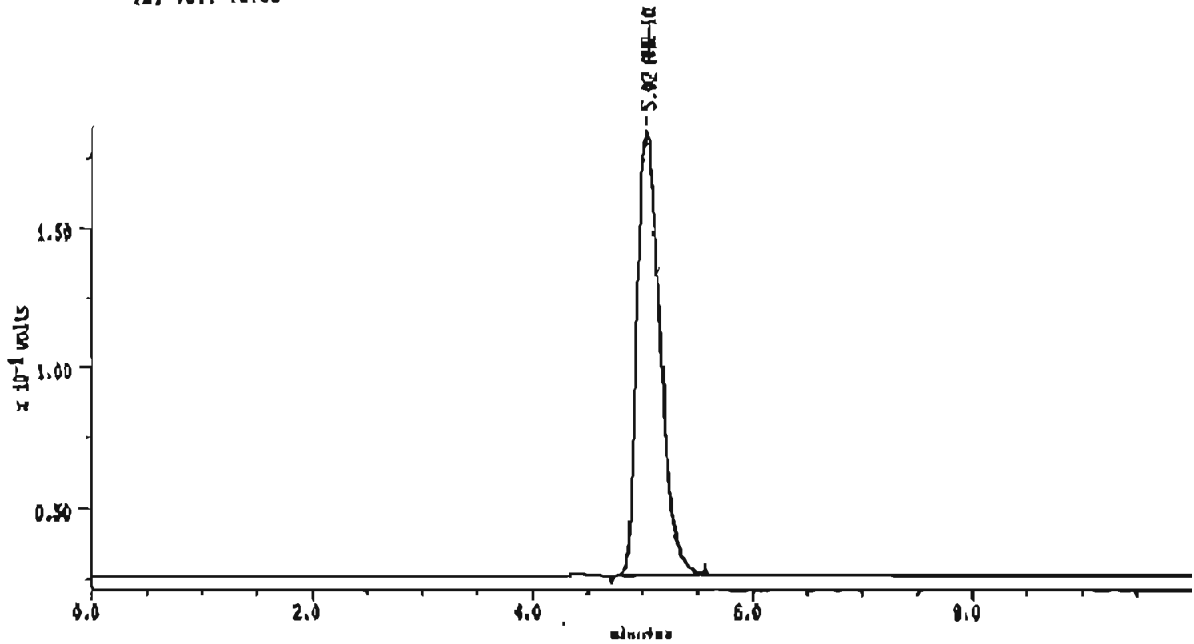
PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.957	AB	2242130	185010	100.00	AHR-10282B
TOTAL			2242130	185010		

14

Sample: A18 70°C-1W
 Acquired: 22-JAN-101 21:53
 Inj Volt: 10.00

Channel: detector 1
 Method: D:\AHR\YINH\8YINH\171AL

File Name: IN-11
 Chart Speed: Full Size
 Operator: S.S



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MAXIMA 825 Custom Report

Printed: 23-JAN-2001 13:00:58

SAMPLE: A18 70°C-1W

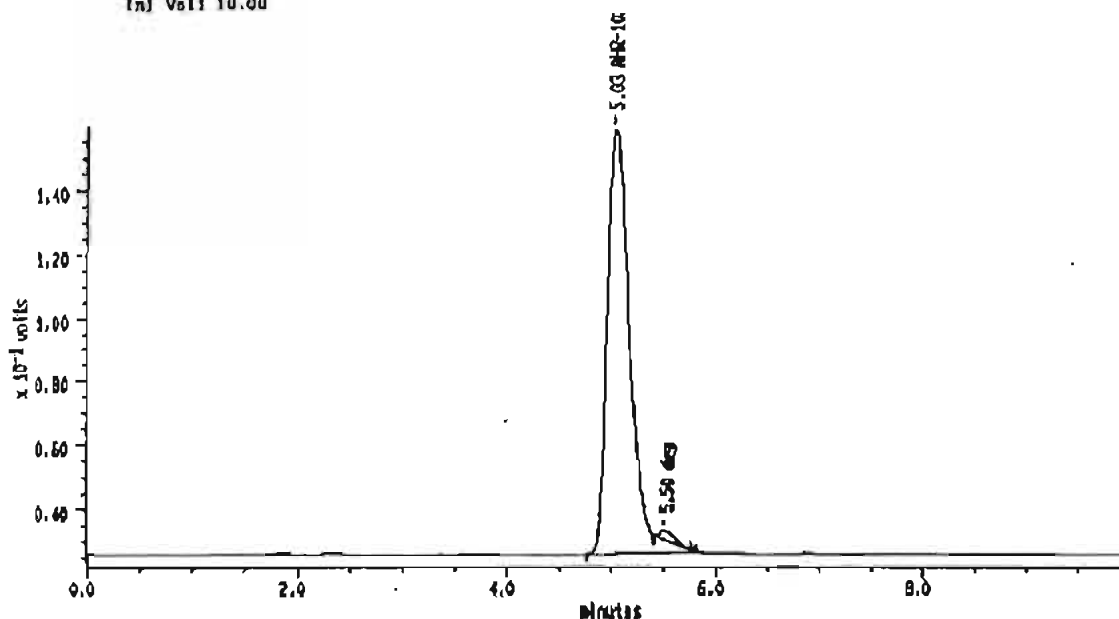
A18 in Method: AHR-10282B
 Acquired: 22-JAN-2001 21:53
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: QMCM
 Instrument: Instrument 1
 File Name: IN-11
 Index: 11
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	BB	2290528	166250	100.00	AHR-10282B
TOTAL			2290528	166250		

Sample: A20 70°C-1W Channel: detector 1 Filament: IX-12 Chart Speed: Full Size
 Acquired: 22-JAN-2001 22:05 Method: B:YAHRY1HISVIN1YIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 23:16:37

SAMPLE: A20 70°C-1W

#16 In Method: AHR-10282B

Acquired: 22-JAN-2001 22:05

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filament: IX-12

Index: 12

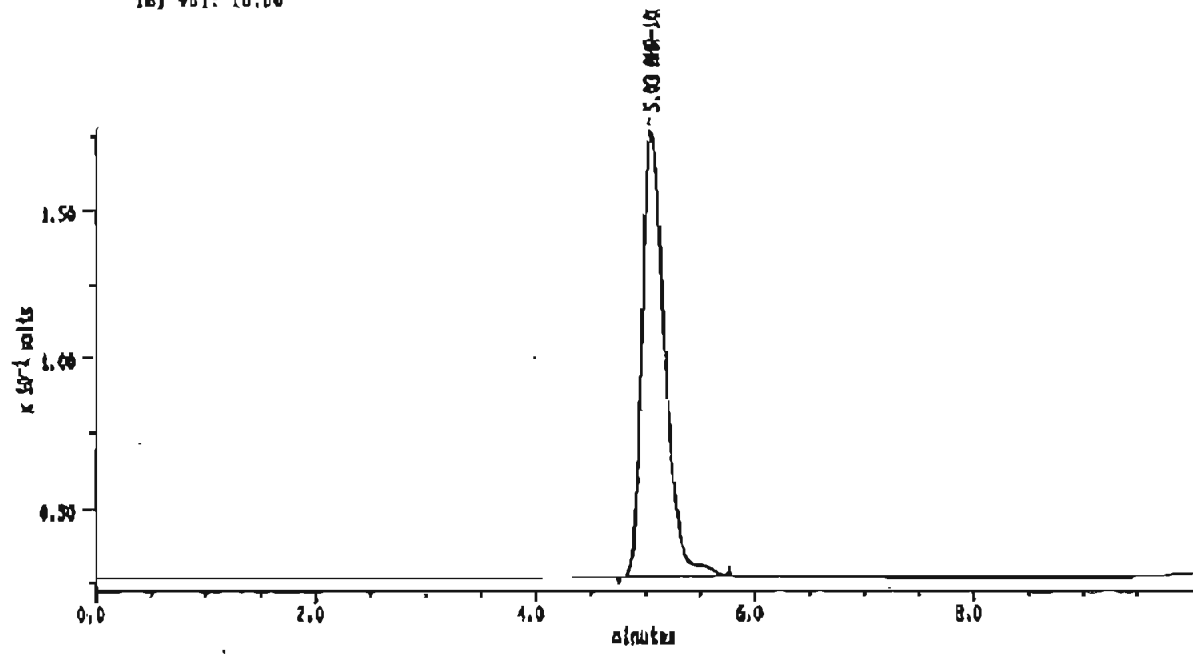
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.033	BB	1961817	102036	98.58	AHR-10282B
2	5.500	SS	28174	2000	1.42	dog
TOTAL			1990001	104124		

16

Sample: A31 70°C-1W Channel: detector 1 Filenama: IN-13 Chart Speed: Full Size
 Acquired: 22-JAN-2001 22:18 Method: BIVAPHYNINISYNTITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

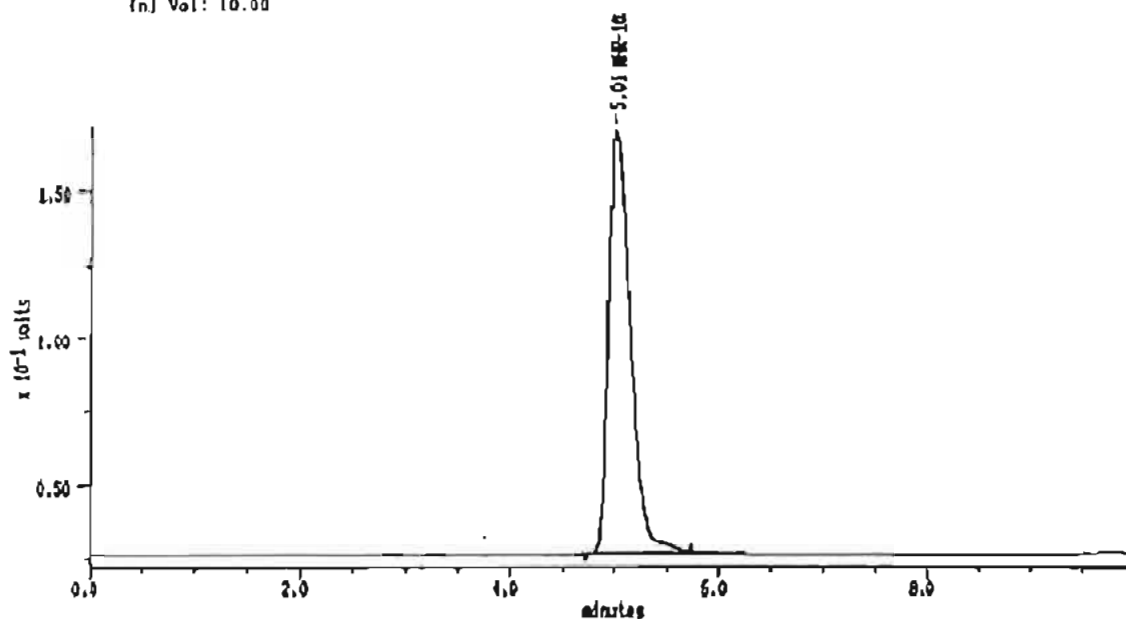
Printed: 22-JAN-2001 23:28:43

SAMPLE: A31 70°C-1W #15 In Method: AHR-10282B Acquired: 22-JAN-2001 22:18 Rate: 2.0 points/sec Duration: 10.000 minutes Operator: S.S	Type: UGXN Instrument: Instrument 1 Filenama: IN-13 Index: 13 Injection Volume: 10.0
--	--

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.003	BB	2203828	168123	100.00	AHR-10282B
TOTAL			2203828	168123		

Sample: A22 70°C-1W Channel: detector 1 File name: IN-14 Chart Speed: Full Size
 Acquired: 22-JAN-10 22:27 Method: B:VAHRVIMIGVINITIAL Operator: S.S
 (n) Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 22:38:08

SAMPLE: A22 70°C-1W

File In Method: AHR-10282B

Acquired: 22-JAN-2001 22:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

File name: IN-14

Index: 14

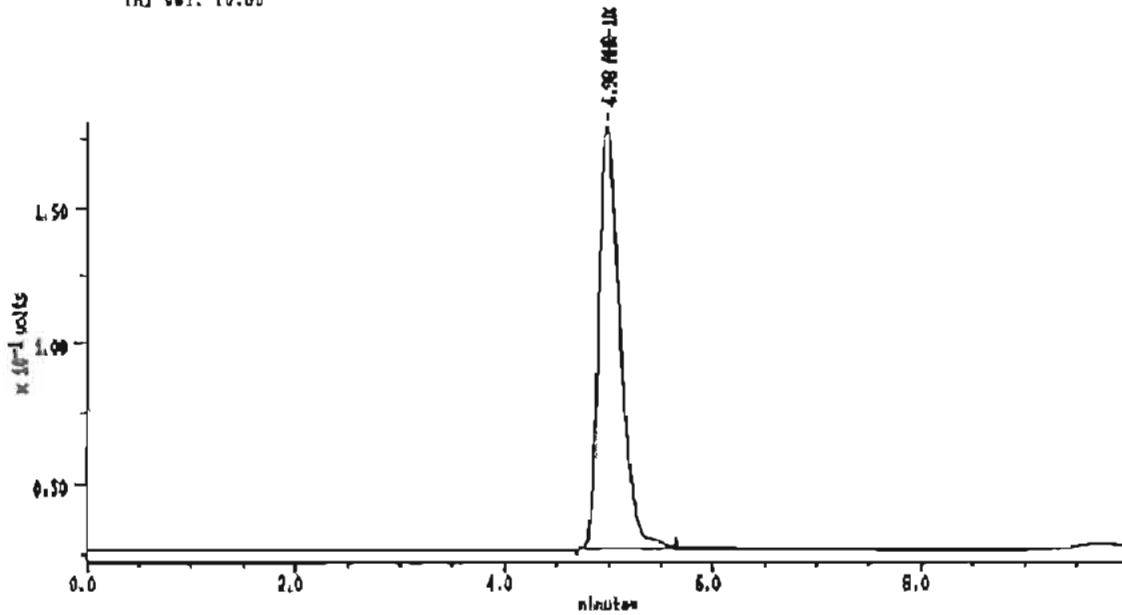
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	2082210	143738	100.00	AHR-10282B
TOTAL			2082210	143738		

18

Sample: A23 70°C-1W Channel: detector 1 Filename: 1W-15 Chart Speed: Full Size
 Acquired: 22-JAN-10 22:30 Method: B1YAHRYIHISVINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 23-JAN-2001 23:49:25

SAMPLE: A23 70°C-1W

Method: AHR-10282B

Acquired: 22-JAN-2001 22:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UMR

Instrument: Instrument 1

Filename: 1W-15

Index: 15

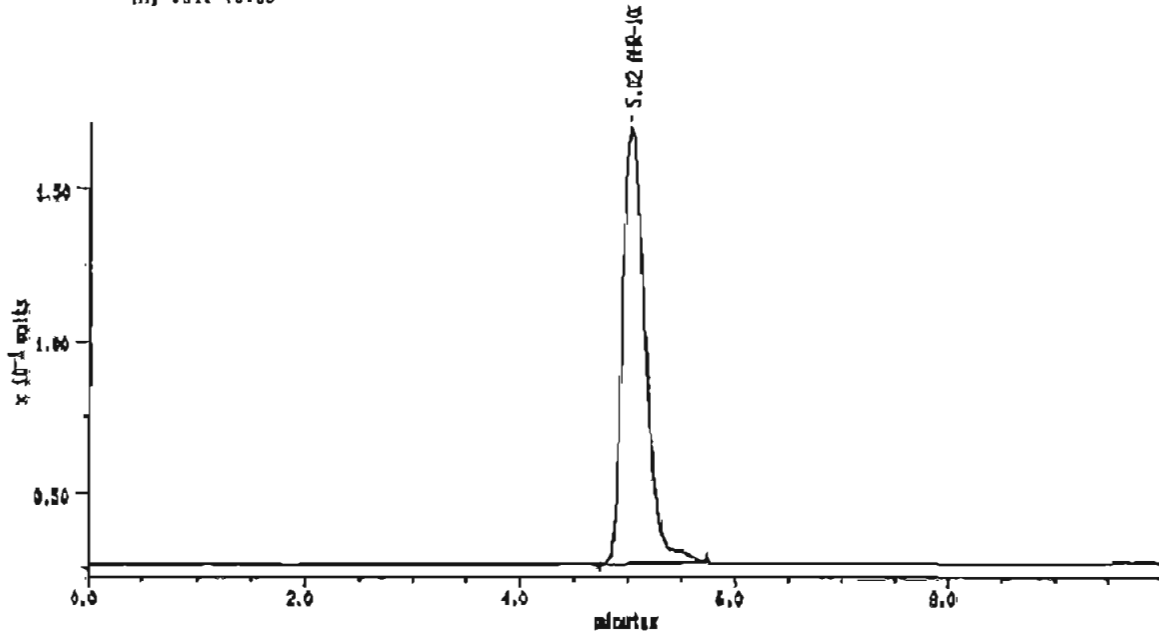
Injection Volume: 10.0

DETECTOR: detector 1

PEP	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.978	DB	2187783	152788	100.00	AHR-10282B
TOTAL			2187783	152788		

19

Sample: A24 70°C-1W Channel: detector 1 Pileman: IM-18 Chart Speed: Full Size
 Acquired: 22-JAN-2001 22:00 Method: B:YAHNYJHSYINVTIAL Operator: S.5
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 22:00:42

SAMPLE: A24 70°C-1W

File In Method: AHR-10282B

Acquired: 22-JAN-2001 22:00

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.5

Type: URRH

Instrument: Instrument 1

Pileman: IM-18

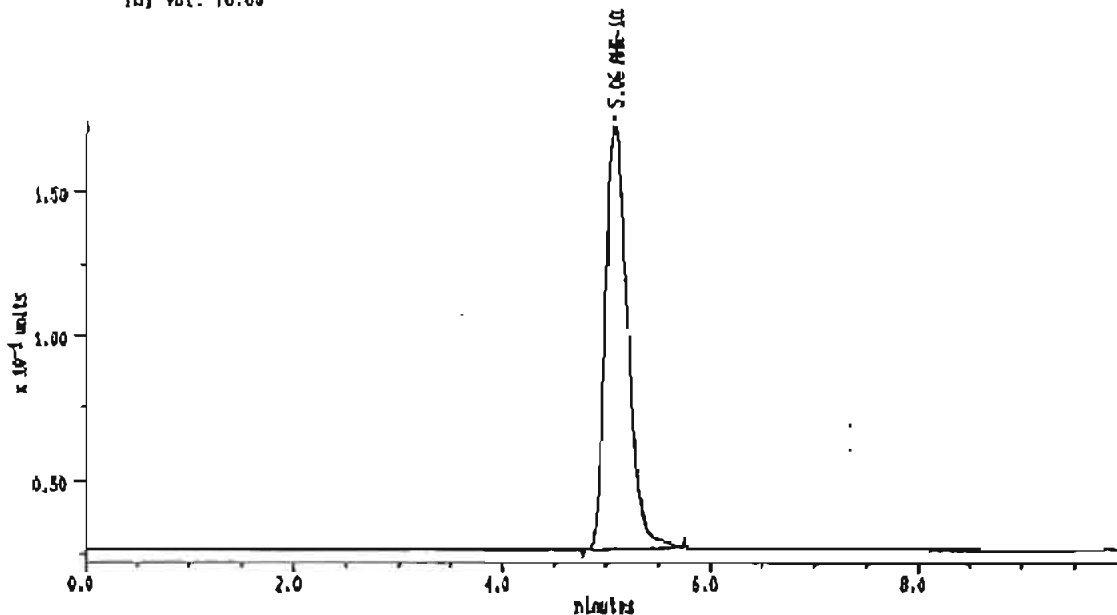
Index: 18

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	BB	7085221	143880	100.00	AHR-10282B
TOTAL			7085221	143880		

Sample: A25 70°C-1W Channel: detector 1 Pileonnet: IM-17 Chart Speed: Full Size
 Acquired: 22-JAN-2001 23:01 Method: GYANAVIHIBYINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 826 Custom Report

Printed: 22-JAN-2001 23:11:59

SAMPLE: A25 70°C-1W

#10 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URM

Instrument: Instrument 1

Pileonnet: IM-17

Index: 17

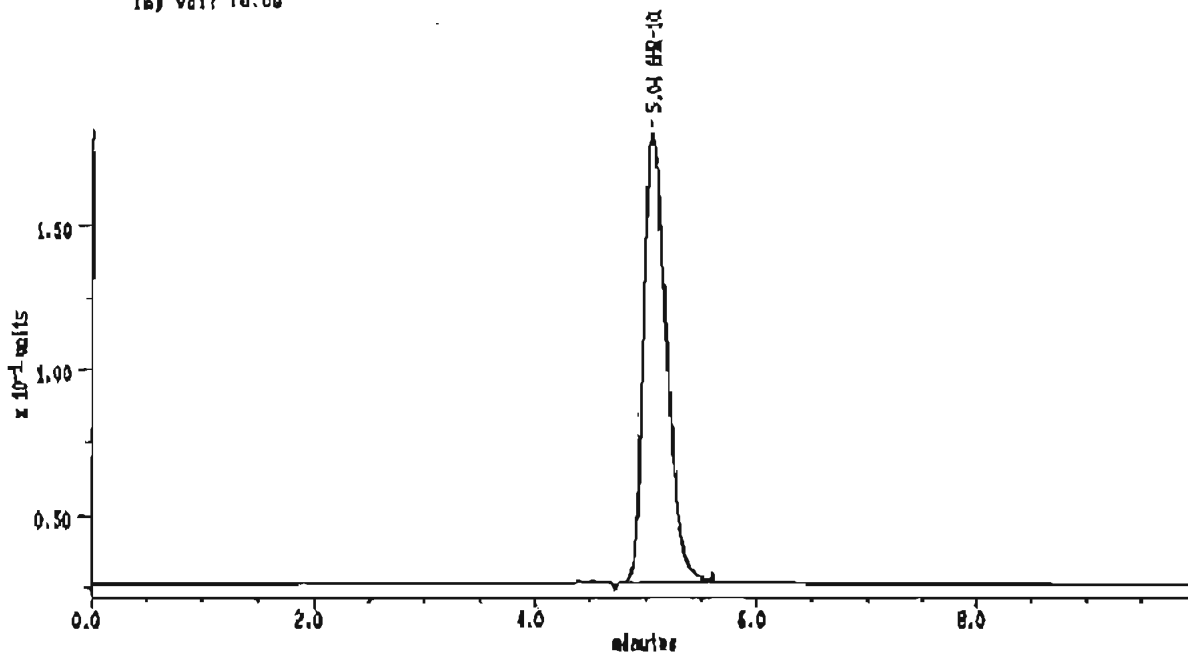
Injection Volume: 10.0

DETECTOR: Detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.060	BB	2131377	146034	100.00	AHR-10282B
TOTAL			2131377	146034		

21

Sample: A18 60°C-1W Channel: detector 1 Filename: IR-10 Chart Speed: Full Size
 Acquired: 22-JAN-01, 23:12 Method: 0:VAJRRYIHI6WIVIVIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 19-JAN-2001 12:09:18

SAMPLE: A18 60°C-1W

010 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:12

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IR-10

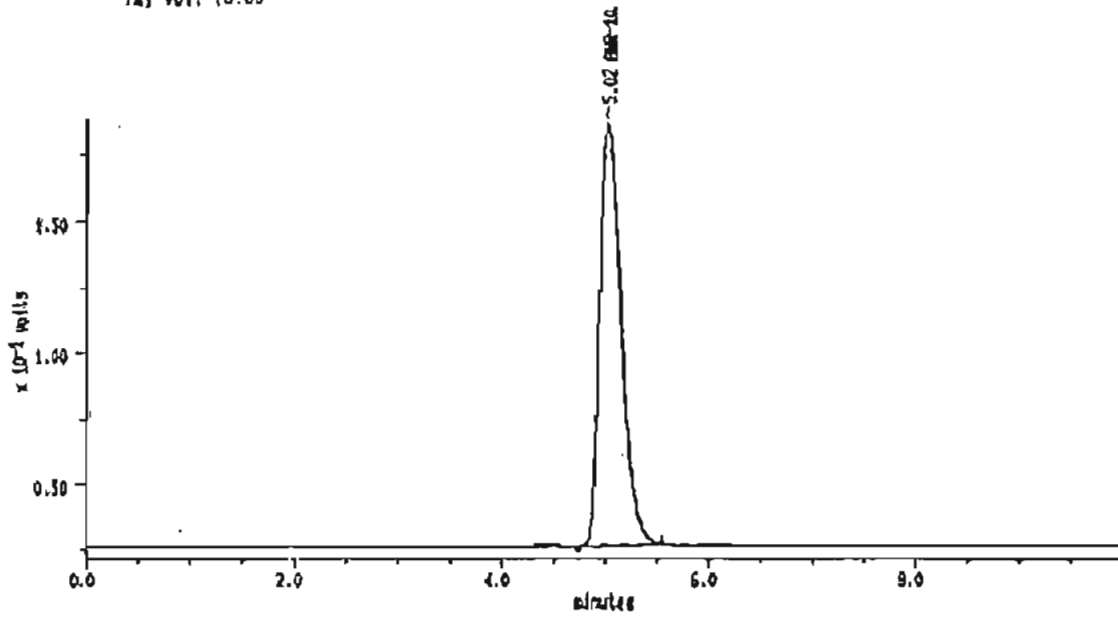
Index: 10

Injection Volume: 10.0

DETECTOR: detector 1

PLO	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.042	BB	2234801	165224	100.00	AHR-10282B
TOTAL			2234801	165224		

Sample: A19 80°C-19 Channel: detector 1 Filename: 1X-19 Chart Speed: Full Size
 Acquired: 22-JAN-2001 23:24 Method: D:VAIRAVI(P)S(V)MITYAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 23:34:33

SAMPLE: A19 80°C-19

#1 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:24

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: 1X-19

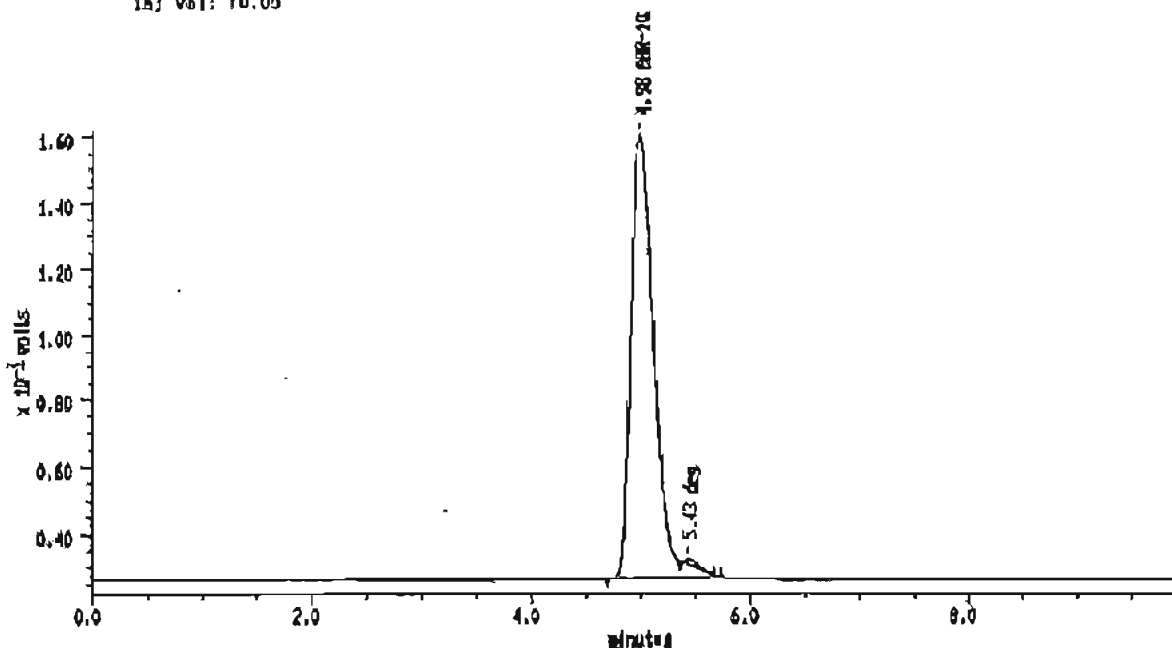
Inj Vol: 10

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	GS	2189798	180013	100.00	AHR-10282B
TOTAL			2189798	180013		

Sample: A20 80°C-1W Channel: detector 1 Pileup: IN-20 Chart Speed: Full Size
 Acquired: 22-JAN-2001 23:35 Method: B:VAMRV3H16VIRITIAL Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 22-JAN-2001 23:45:49

SAMPLE: A20 80°C-1W

433 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:35

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: CHEN

Instrument: Instrument 1

Pileup: IN-20

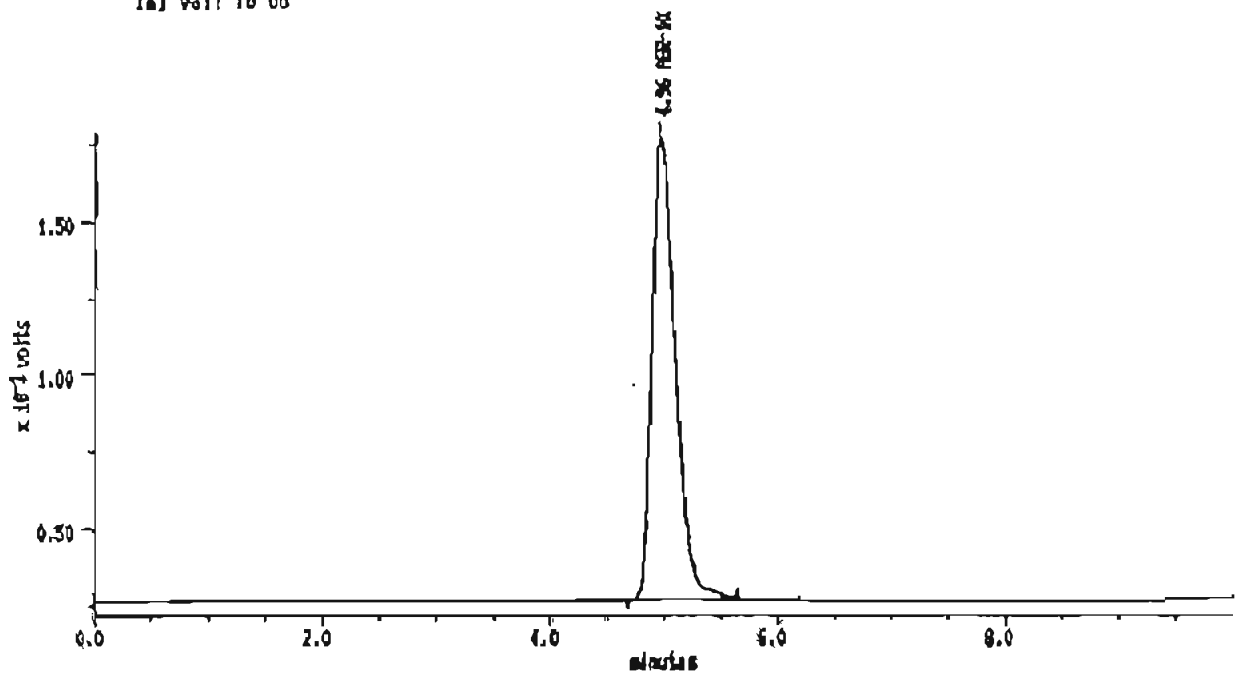
Index: 20

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.978	SB	1848910	130078	99.21	AHR-10282B
2	5.426	SI	15816	1583	0.79	deg
TOTAL			1864726	131661		

Sample: A21 80°C-1W Channel: detector 1 Filename: IN-21 Chart Speed: Full Size
 Acquired: 22-JAN-2001 23:48 Method: B:VAIRY\HIBV\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

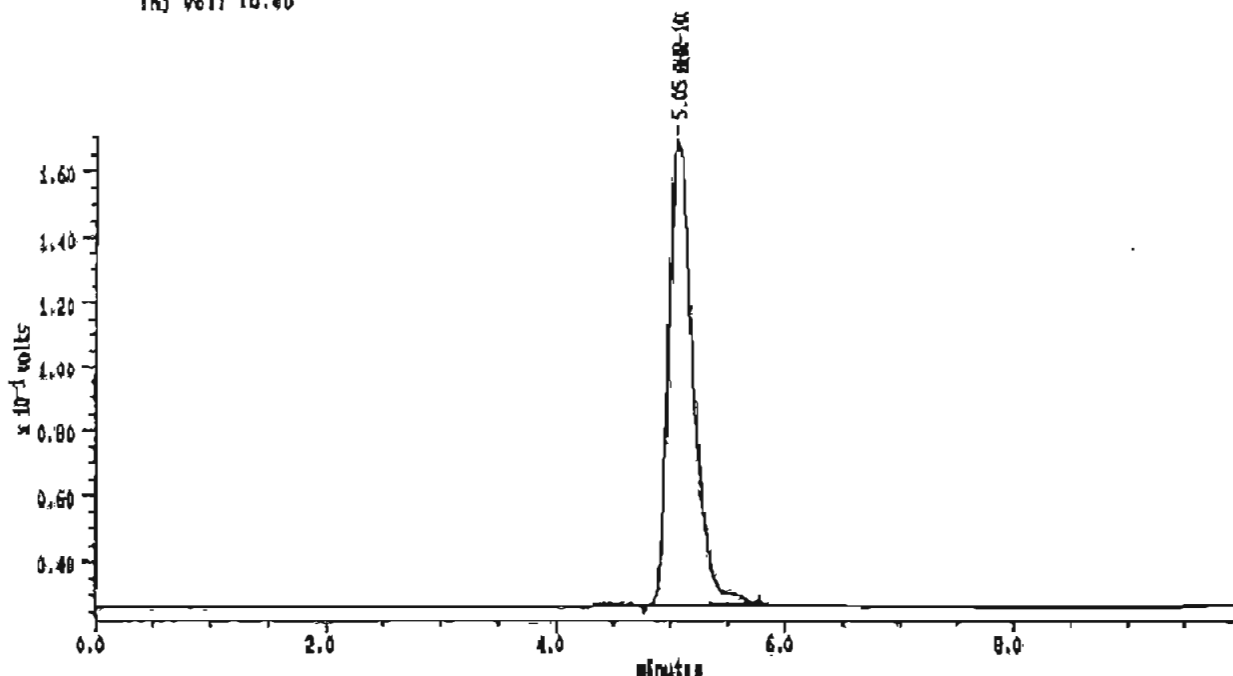
Printed: 22-JAN-2001 23:57:08

SAMPLE: A21 80°C-1W Type: USKN
 #23 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 22-JAN-2001 23:48 Filename: IN-21
 Rate: 2.0 points/sec Index: 21
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.968	DB	2181206	180005	100.00	AHR-10282B
TOTAL			2181206	180005		

Sample: A22 80°C-1X Channel: detector 1 Filename: IN-22 Chart Speed: Full Size
 Acquired: 22-JAN-10 23:57 Method: B:VARIATION:MINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 23-JAN-2001 0:08:22

SAMPLE: A22 80°C-1X

#24 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:57

Rate: 3.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URM

Instrument: Instrument 1

Filename: IN-22

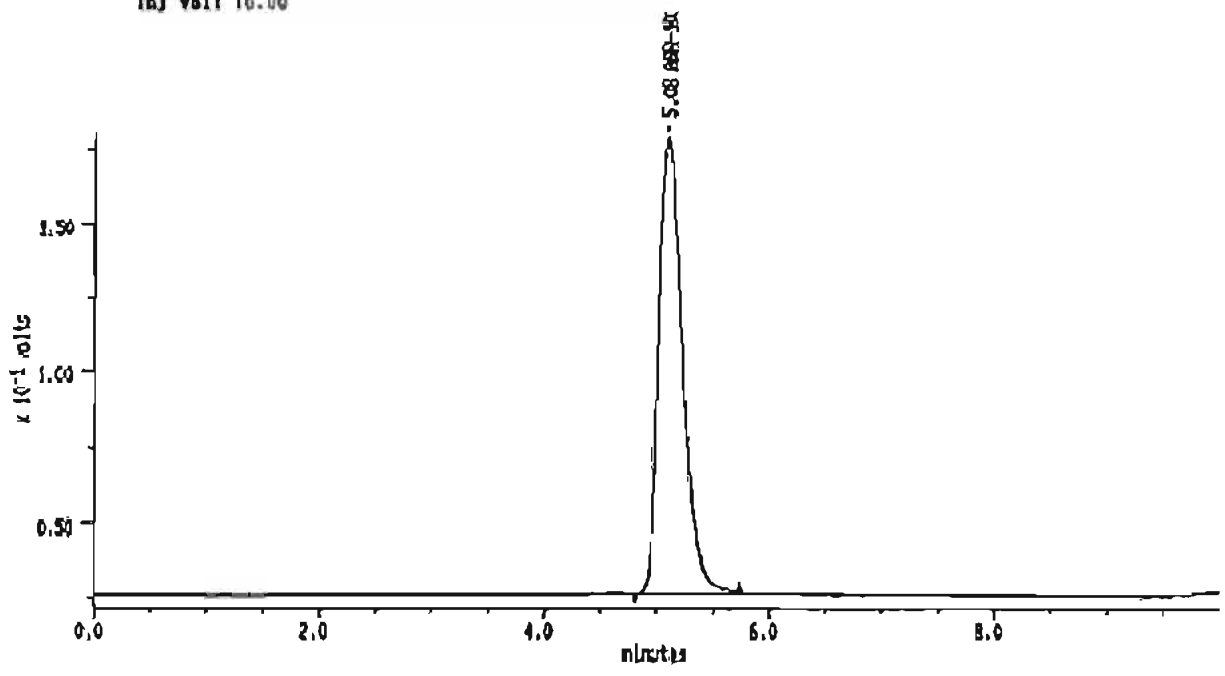
Index: 22

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.050	BD	2031481	142001	100.00	AHR-10282B
TOTAL			2031481	142001		

Sample: A23 80°C-1W Channel: detector 1 Filament: 1X-23 Chart Speed: Full Size
 Acquired: 23-JAN-2001 0:09 Method: B:VAMPHIHSY/INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

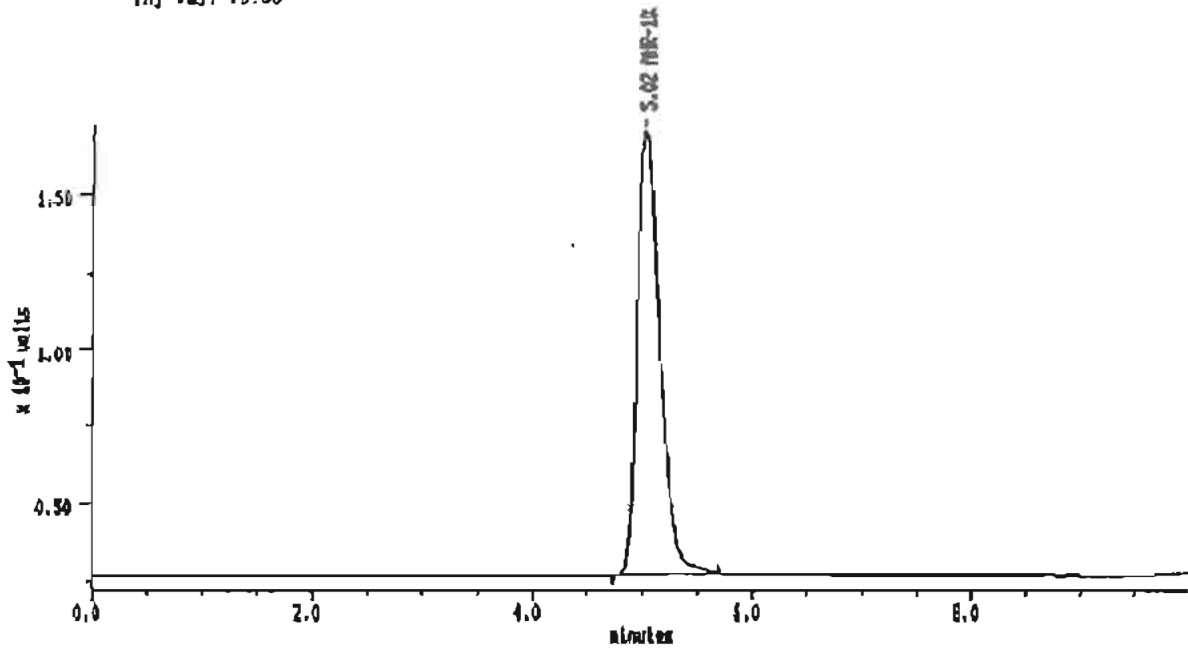
Printed: 23-JAN-2001 0:19:39

SAMPLE: A23 80°C-1W	Type: UNKN
278 (n Method: AHR-10282B	Instrument: Instrument 1
Acquired: 23-JAN-2001 0:09	Filament: 1X-23
Rate: 2.0 points/sec	Index: 23
Duration: 18.000 minutes	Injection Volume: 10.0
Operator: S.S	

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.083	88	2243048	162820	100.00	AHR-10282B
TOTAL			2243048	162820		

Sample: A24 80°C-1M Channel: detector 1 Filename: IN-24 Chart Speed: Full Size
 Acquired: 23-JAN-01 0:20 Method: B:YAHYVHISYINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

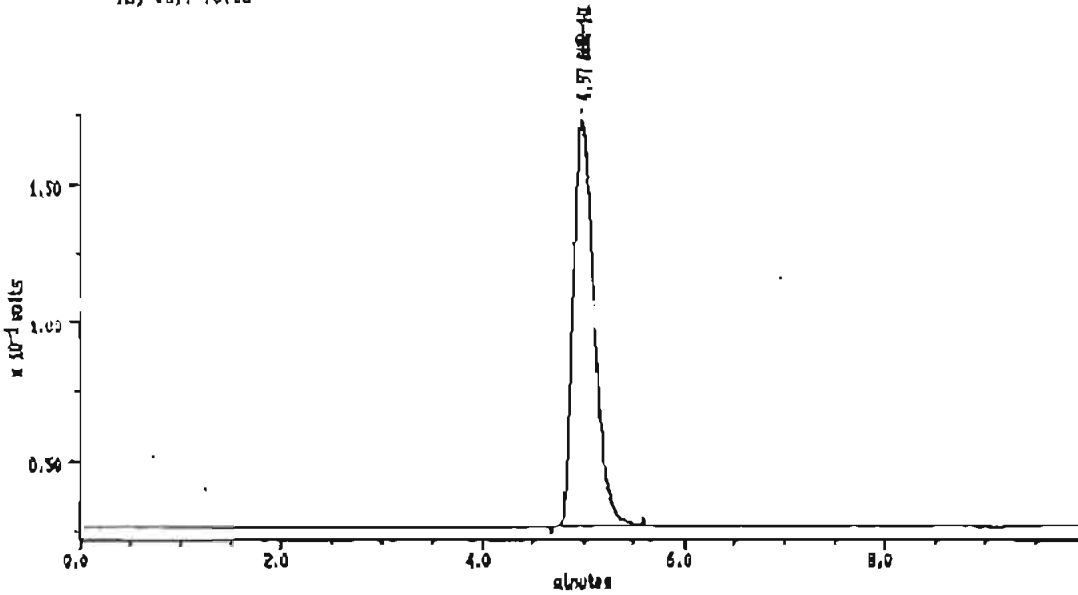
Printed: 23-JAN-2001 8:30:55

SAMPLE: A24 80°C-1M	Type: UNID
928 in Method: AHR - 10282B	Instrument: Instrument 1
Acquired: 23-JAN-2001 0:20	Filename: IN-24
Rate: 2.0 points/sec	Index: 28
Duration: 10.000 minutes	Injection Volume: 10.0
Operator: S.S	

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	SS	2076710	143989	100.00	AHR-10282B
TOTAL			2076710	143989		

Sample: A26 80°C-1V Channel: detector 1 Filtration: IM-26 Chart Speed: Full Size
 Acquired: 23-JAN-2001 0:31 Method: B:VAIRYACHHIVHIVYIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 23-JAN-2001 0:42:12

SAMPLE: A26 80°C-1V

x27 (a Method: AHR-10282B

Acquired: 23-JAN-2001 0:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URMK

Instrument: Instrument 1

Filtration: IM-26

Index: 25

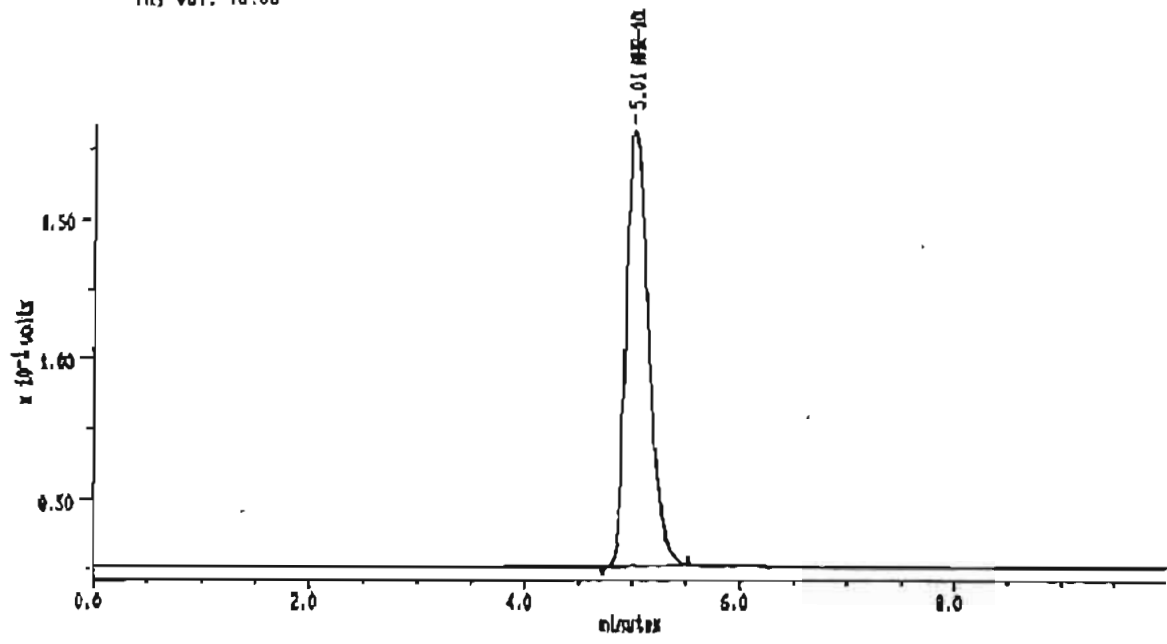
Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.987	BB	2000054	148042	100.00	AHR-10282B
TOTAL			2000054	148042		

29

Sampler: STD2 Channel: detector 1 Filename: IN-28 Chart Speed: Full Size
 Acquired: 23-JAN-101 0:47 Method: B:YAIRUBHISVINITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 Custom Report

Printed: 23-JAN-2001 0:53:38

SAMPLE: STD2

#28 In Method: AHR-10282B
 Acquired: 23-JAN-2001 0:42
 Run: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: QM4
 [Instrument: Instrument 1]
 Filename: IN-28
 Index: 28
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BD	236148	106690	100.00	AHR-10282B
TOTAL			236148	106690		

試験計画書

試験名：pH7におけるプロナック点眼液の製剤化検討

試験コード：P2000B177

試験系：なし

開発記号：AHR10282B

試験開始日：2000年12月07日

試験操作開始予定日：2000年12月07日

試験操作終了予定日：2000年03月15日

試験終了予定日：2001年03月30日

試験施設：千寿製薬株式会社 コーベクリエティブセンター
神戸市西区室谷一丁目5番4号

(業務分担)

試験責任者：澤 嗣郎

試験従事者：

試験物質：ブロムフェナクナトリウム

目的：ブロムフェナクナトリウムは低いpH領域では溶けにくく、不安定であるため、プロナック点眼液のpH(規格中心)は8.3に設定されている。涙液のpHは一般に7~7.4といわれており、プロナック点眼液のpHは点眼液で用いられる上限付近であると考えられるため、さらに低いpHでの処方化が望まれる。ブロムフェナクナトリウムは分子内に酢酸基をもつため、溶解度はpH6.5以上で増加する。また、プロナック点眼液の分解はほぼ酢酸基に関連して起こっているため、可溶化および安定化に対して酢酸基の制御が重要と考えられる。酢酸基の制御には対イオンの添加が考えられるが、ブロムフェナクナトリウムは第四級アンモニウム塩の添加により不溶性複合体を形成し白濁する。そこで、本実験では、複合体を形成しても水溶性を保つアミノ糖類を用いて、pH7におけるブロムフェナクナトリウムの可溶化および安定化を検討する。

試験方法：

1) 可溶化検討

酢酸(pH3~6)、リン酸(pH6~7)またはホウ酸(pH7~9)緩衝液にブロムフェナクナトリウムを過剰量添加し、N-メチルグルカミンまたは塩酸グルコサミンを0.1~1.0%となるように加え、塩酸でpHを調整する。この液をろ過し、ろ液中のブロムフェナクナトリウム濃度をHPLCで測定する。

2) 安定化検討

下記のブロムフェナクナトリウム点眼液に N-メチルグルカミンまたは塩酸グルコサミンを加え、pH7に調整する。N-メチルグルカミンまたは塩酸グルコサミンは、実験1)で pH6.5 で 0.1%ブロムフェナクナトリウムが溶解した量とする。この液を無色ポリプロピレン容器に充填し、70℃、60℃、40℃75%RH および 25℃60%RH に保存し、経時的にブロムフェナクナトリウム含量、pH、外観および不溶性異物について試験する。また、凍結融解を 10 回繰り返し、外観および不溶性異物を観察する。

	Rp-01	Rp-02	Rp-03	Rp-04
ブロムフェナクナトリウム	0.1 g	0.1 g	0.1 g	0.1 g
ホウ酸	1.1 g	-	1.1 g	-
ホウ砂	1.1 g	-	1.1 g	-
塩化ナトリウム	-	0.85g	-	0.85g
結晶リン酸二水素ナトリウム	-	0.1 g	-	0.1 g
ポリソルベート 80	0.15 g	0.15g	0.15g	0.15g
塩化ベンザルコニウム	0.005g	0.005g	0.005g	0.005g
ポリビニルピロリドン	-	-	2.0 g	2.0 g
亜硫酸水素ナトリウム	-	-	0.2g	0.2g
エデト酸ナトリウム	-	-	0.02g	0.02g
N-メチルグルカミン	q.s.	q.s.	q.s.	q.s.
塩酸グルコサミン	q.s.	q.s.	q.s.	q.s.
塩酸	q.s.	q.s.	q.s.	q.s.
精製水	q.s.	q.s.	q.s.	q.s.
全量	100mL	100mL	100mL	100mL
pH	7.0	7.0	7.0	7.0

3) HPLC 条件

検出器：紫外吸光光度計(測定波長：266nm)

カラム：Capcelpak AG-120

カラム温度：25℃付近の一定温度


移動相：リン酸二水素アンモニウム 1.98g を水 750mL に溶かし、リン酸を加えて pH7.3 に調整した後、アセトニトリル 250mL を混和する。

流速：ブロムフェナクナトリウムの溶出時間が約 18 分となるように調整する。

試料注入量：10μL


試験責任者の署名

2000年12月07日

澤 嗣郎 

応用開発 GM の署名

2000年12月08日

大馬 雅 

プロナック点眼液の安定性試験
Lot No.01K131

試験コード：P2000B177
試験実施者：澤 嗣郎
試験実施日：2001年04月02日

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Collec(%)	Water	Initial	present
STD	1	S2-01	2326333						
STD	2	S2-22	2321811						
STD	mean		2324072	1.0005					
A-26	60°C-2W	S2-02	2032330	0.8749	87.46	83.94	4.02	8.4304	8.2383
A-27	60°C-2W	S2-03	2214721	0.9534	96.62	93.03	3.72	8.4298	8.2521
A-28	60°C-2W	S2-04	2156136	0.9282	93.56	90.24	3.55	8.5538	8.3800
A-29	60°C-2W	S2-05	2217340	0.9546	92.39	89.22	3.43	8.5249	8.3580
A-30	60°C-2W	S2-06	2142178	0.9222	90.88	87.84	3.34	8.5115	8.3490
A-31	60°C-2W	S2-07	2041719	0.8789	86.55	83.53	3.49	8.5492	8.3785
A-32	80°C-2W	S2-08	1930369	0.8310	78.98	72.11	8.70	8.4060	8.2309
A-33	60°C-2W	S2-09	2070437	0.8913	88.93	85.67	3.67	8.4759	8.2989
A-34	60°C-2W	S2-10	2010017	0.8653	86.25	82.90	3.88	8.4139	8.2293
A-35	60°C-2W	S2-11	1990944	0.8571	84.79	81.68	3.67	8.5592	8.3792
A-26	50°C-4W	S2-12	1918766	0.8260	82.57	80.04	3.06	8.4391	8.2927
A-27	50°C-4W	S2-13	2176249	0.9369	94.94	91.62	3.50	8.4117	8.2453
A-28	50°C-4W	S2-14	2105338	0.9063	91.35	88.33	3.31	8.5416	8.3799
A-29	50°C-4W	S2-15	2137432	0.9202	89.06	86.05	3.38	8.4808	8.2981
A-30	50°C-4W	S2-16	2043998	0.8799	86.72	83.74	3.44	8.4297	8.2654
A-31	50°C-4W	S2-17	1933544	0.8324	81.97	79.44	3.09	8.5842	8.4126
A-32	50°C-4W	S2-18	1692208	0.7285	69.24	66.88	3.41	8.3947	8.2328
A-33	50°C-4W	S2-19	1842783	0.8364	83.45	80.85	3.12	8.4713	8.3210
A-34	50°C-4W	S2-20	1922369	0.8278	82.50	79.96	3.08	8.4168	8.2699
A-35	50°C-4W	S2-21	1927309	0.8297	82.08	79.45	3.20	8.5846	8.4266

計算に必要となるデータを記載した。
05.05.06 澤 嗣郎

試験物質名: AHR10282B 試験コード: P2000B177 試験年月日: 2001年04月02日

試験項目: 試験実施者: 澤 嗣郎

Lot No. 01K131

2-APR-2001 10:24:01

STD AHR10282B 0.02001g + MP → 20ml

001: 0.02001g

上試 2ml + MP → 20ml

濁り 不溶性物 色

A26	60C-2w	7.05	8.4304	8.2383	+	±	混濁
A27		7.10	8.4298	8.2521	±	+(n)	黄
A28		7.08	8.5538	8.3800	+	+(n)	
A29		7.06	8.5244	8.2580	+	+(n)	
A30		7.09	8.5115	8.3490	+	+(n)	
A31		7.07	8.5442	8.3785	+	+(n)	↓
A32		7.09	8.4060	8.2304	+	++(n)	混濁
A33		7.07	8.4759	8.2989	+	+(n)	
A34		7.11	8.4139	8.2293	+	+(n)	
A35		7.08	8.5592	8.3792	+	+(n)	↓
A26	50C-4w	7.06	8.4391	8.2927	+	±	↓
A27		7.11	8.4117	8.2453	+	++(n)	黄色
A28		7.10	8.5416	8.3799	+	+(n)	
A29		7.08	8.4606	8.2981	+	+(n)	
A30		7.09	8.4297	8.2654	+	+(n)	
A31		7.06	8.5642	8.4126	+	+(n)	↓
A32		7.10	8.3947	8.2328	++	++(n)	混濁
A33		7.12	8.4713	8.3210	+	+	黄
A34		7.11	8.4168	8.2699	+	++(n)	混濁
A35		7.14	8.5846	8.4266	+	+(n)	混濁

4/02 17:49
NO. 92 PH 7.05
24.1°C

4/02 17:51
NO. 93 PH 7.10
24.3°C

4/02 17:53
NO. 94 PH 7.02
24.3°C

4/02 17:55
NO. 95 PH 7.06
24.1°C

4/02 17:58
NO. 96 PH 7.03
24.1°C

4/02 17:59
NO. 97 PH 7.07
24.0°C

~~4/02 18:00~~
NO. 98 PH 7.07
23.9°C

4/02 18:01
NO. 99 PH 7.03
23.9°C

4/02 18:02
NO. 1 PH 7.07
23.9°C

4/02 18:04
NO. 2 PH 7.11
23.9°C

4/02 18:06
NO. 3 PH 7.03
23.6°C

4/02 18:07
NO. 4 PH 7.05
23.9°C

4/02 18:12
NO. 5 PH 7.11
24.2°C

4/02 18:14
NO. 6 PH 7.10
23.9°C

4/02 18:15
NO. 7 PH 7.08
23.9°C

4/02 18:16
NO. 8 PH 7.09
23.6°C

4/02 18:17
NO. 9 PH 7.03
23.6°C

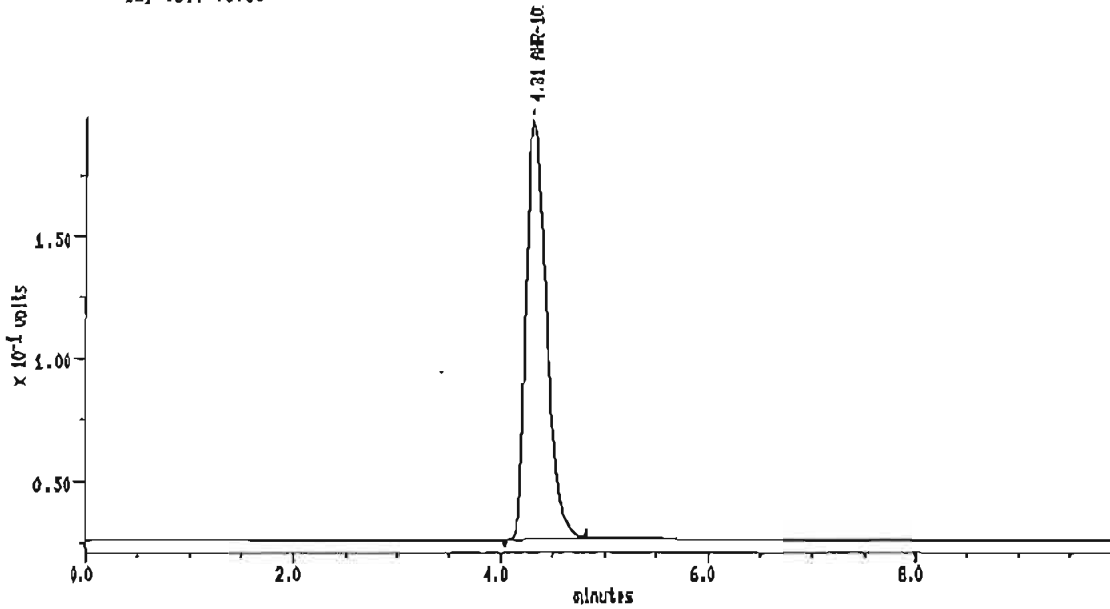
4/02 18:19
NO. 10 PH 7.10
23.7°C

4/02 18:21
NO. 11 PH 7.12
23.7°C

4/02 18:27
NO. 12 PH 7.11
23.7°C

4/02 18:26
NO. 13 PH 7.14
24.2°C

Sample: STD1 Channel: detector 1 Filename: S2-01 Chart Speed: Full Size
 Acquired: 02-APR-10 20:31 Method: B:YAJIRYIKI3Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 20:41:47

SAMPLE: STD1

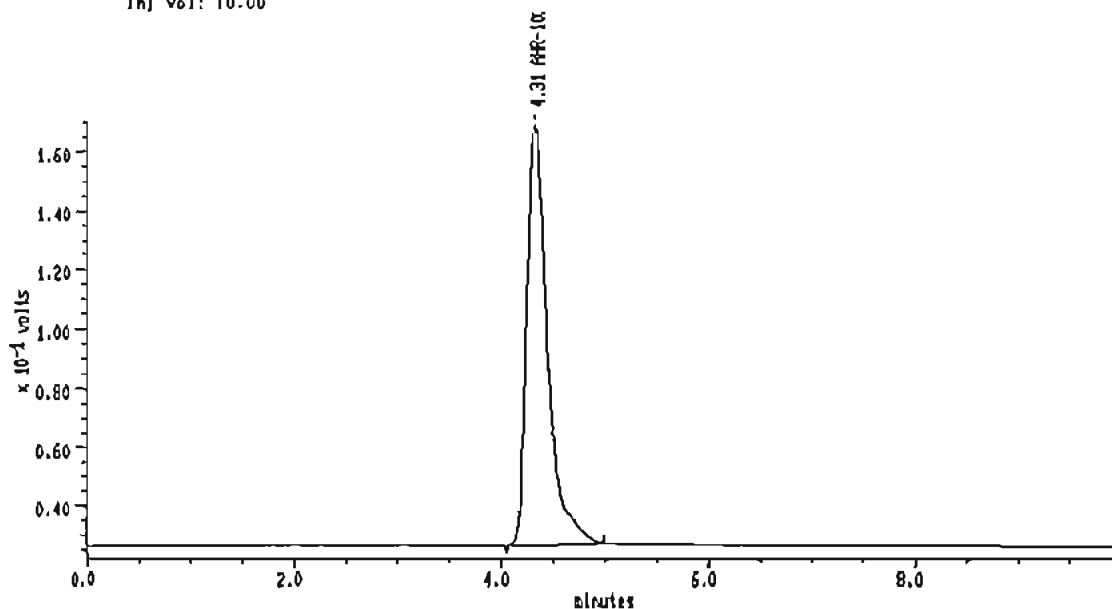
#1 (A Method: AHR-10282B
 Acquired: 2-APR-2001 20:31
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: S2-01
 Index: 28
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	BB	2328333	170545	100.00	AHR-10282B
TOTAL			2328333	170545		

Sample: A28 80°C-2W Channel: detector 1 Filename: S2-02 Chart Speed: Full Size
 Acquired: 02-APR-101 20:42 Method: B:YAHRYIK13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 20:53:05

SAMPLE: A28 80°C-2W

#2 In Method: AHR-10282B

Acquired: 2-APR-2001 20:42

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-02

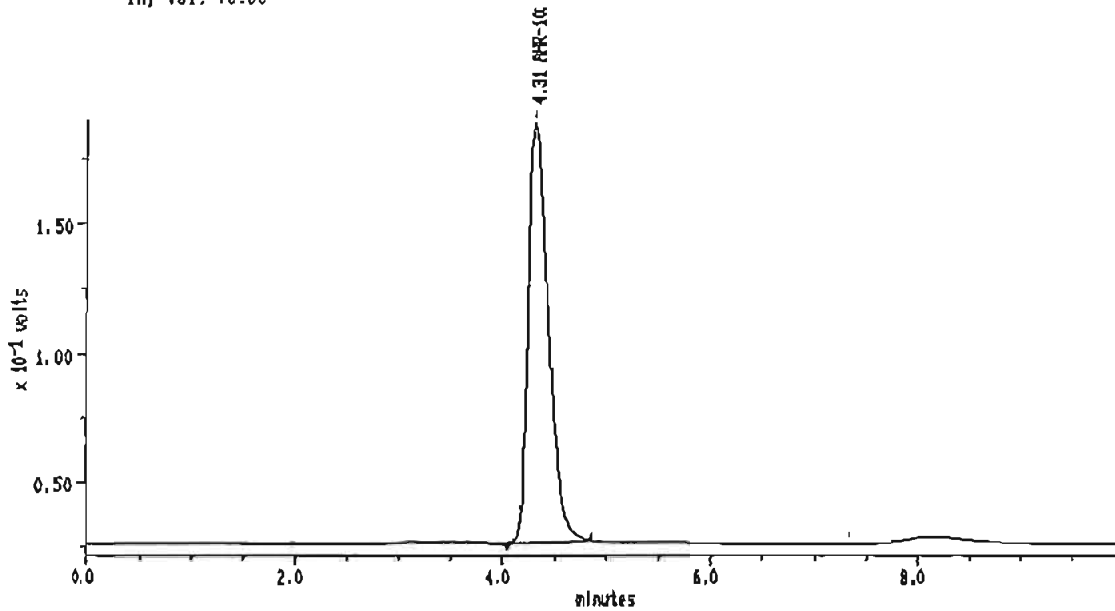
Index: 21

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	BB	2032330	142218	100.00	AHR-10282B
TOTAL			2032330	142218		

Sample: A27 80°C-2W Channel: detector 1 Filename: S2-03 Chart Speed: Full Size
 Acquired: 02-APR-10 20:53 Method: 8:VAHRYIKI3Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 21:04:18

SAMPLE: A27 80°C-2W

#3 In Method: AHR-10282B

Acquired: 2-APR-2001 20:53

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-03

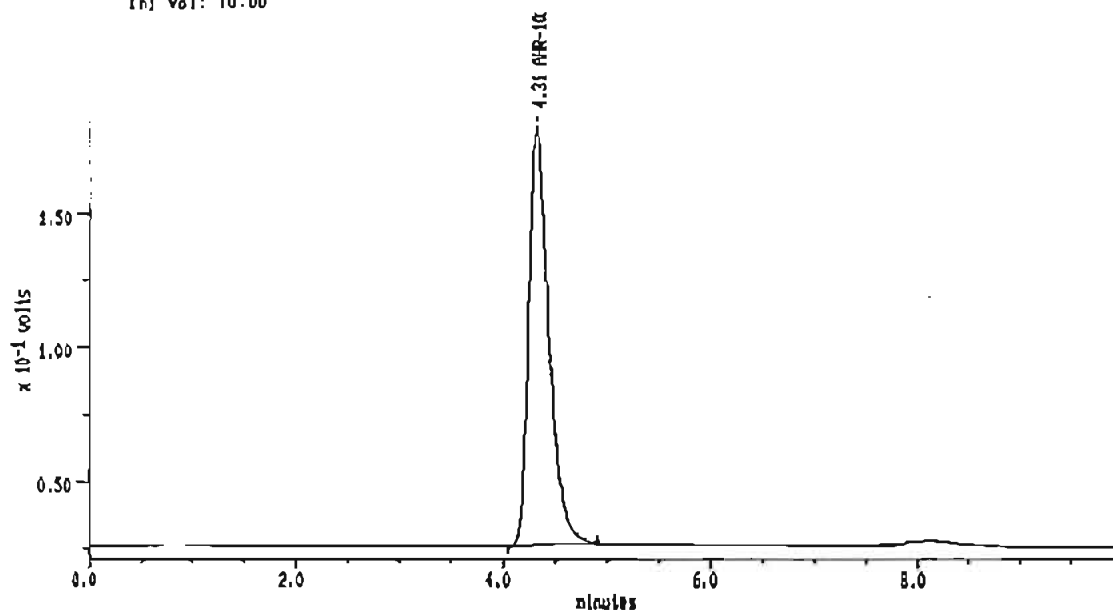
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	OB	2214721	181818	100.00	AHR-10282B
TOTAL			2214721	181818		

Sample: A28 60°C-2W Channel: detector 1 Filename: S2-04 Chart Speed: Full Size
 Acquired: 02-APR-10 21:05 Method: B:YAHRYIK13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 21:16:38

SAMPLE: A28 60°C-2W

#4 In Method: AHR-10282B

Acquired: 2-APR-2001 21:05

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-04

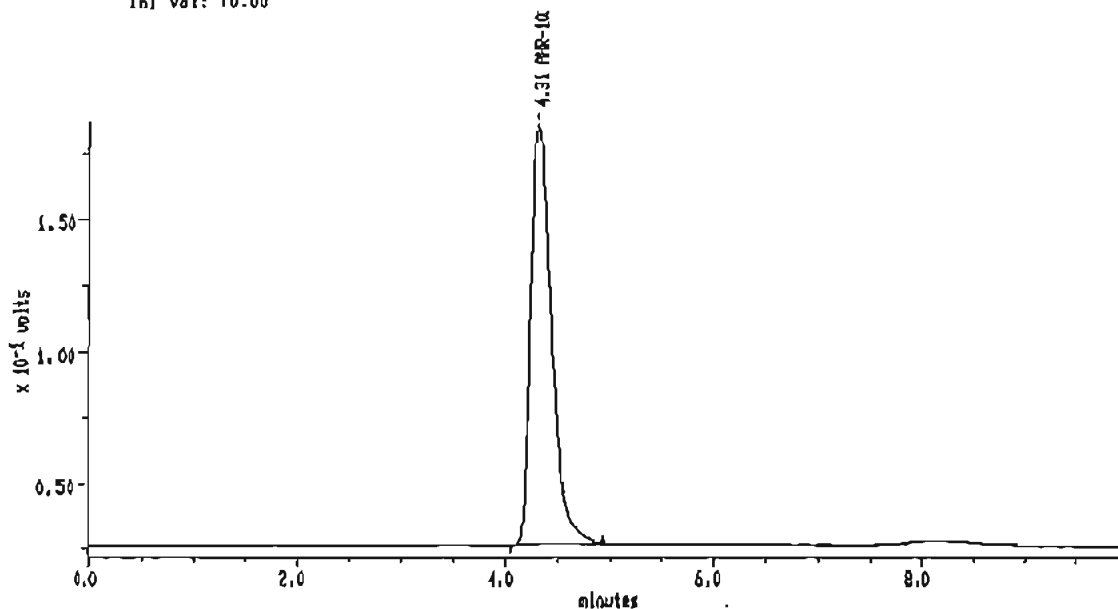
Index: 29

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	DB	2168138	158277	100.00	AHR-10282B
TOTAL			2168138	158277		

Sample: A29 60°C-2W Channel: detector 1 Filename: S2-05 Chart Speed: Full Size
 Acquired: 02-APR-10 21:10 Method: B:VAHRV1K13V60-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 21:28:52

SAMPLE: A29 60°C-2W

#5 In Method: AHR-10282B

Acquired: 2-APR-2001 21:10

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-05

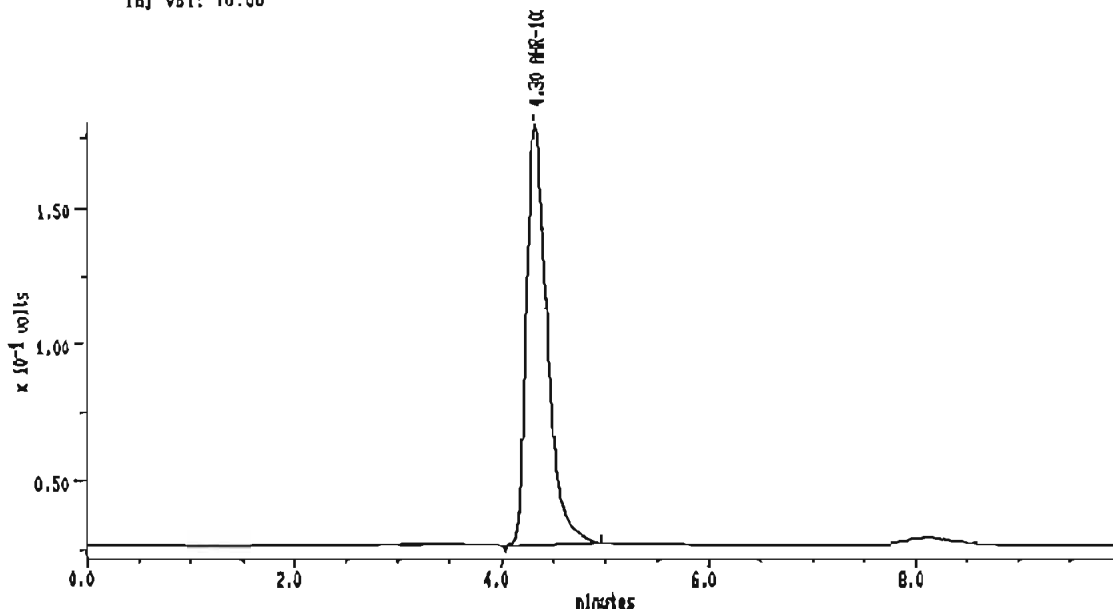
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	BB	2217340	159618	100.00	AHR-10282B
TOTAL			2217340	159618		

Sample: A30 80°C-2W Channel: detector 1 Filename: S2-08 Chart Speed: Full Size
 Acquired: 02-APR-10 21:27 Method: B:YAHRYIK13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 21:38:10

SAMPLE: A30 80°C-2W

#B In Method: AHR-10282B

Acquired: 2-APR-2001 21:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-08

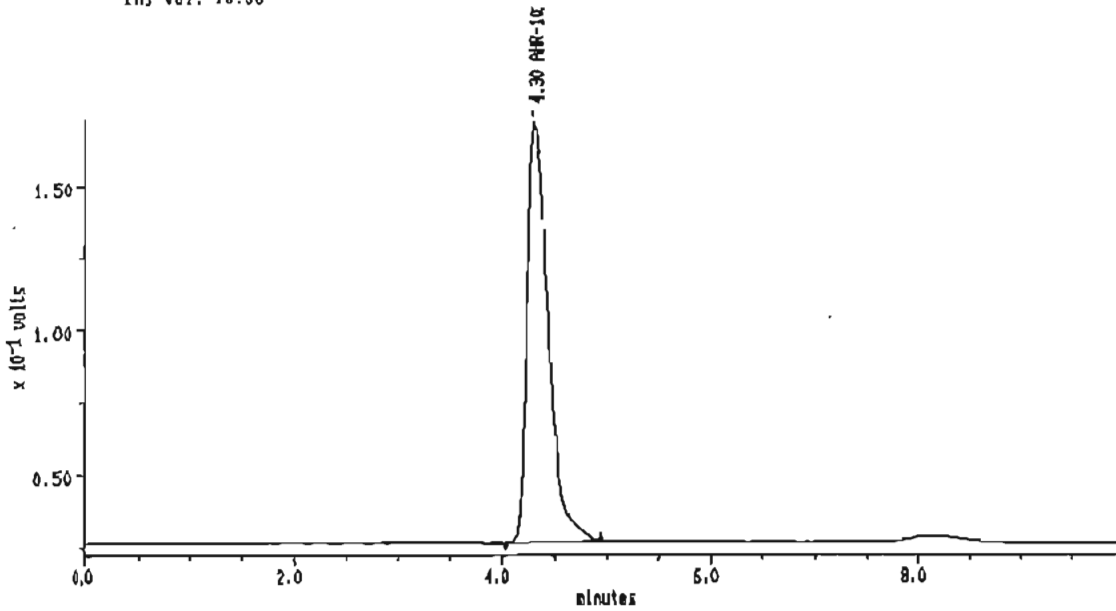
Index: 81

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	2142178	152831	100.00	AHR-10282B
TOTAL			2142178	152831		

Sample: A31 60°C-2W Channel: detector 1 Filename: S2-07 Chart Speed: Full Size
 Acquired: 02-APR-2001 21:38 Method: B:YAJRWIK13Y00-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 21:49:28

SAMPLE: A31 60°C-2W

#7 In Method: AHR-10282B

Acquired: 2-APR-2001 21:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-07

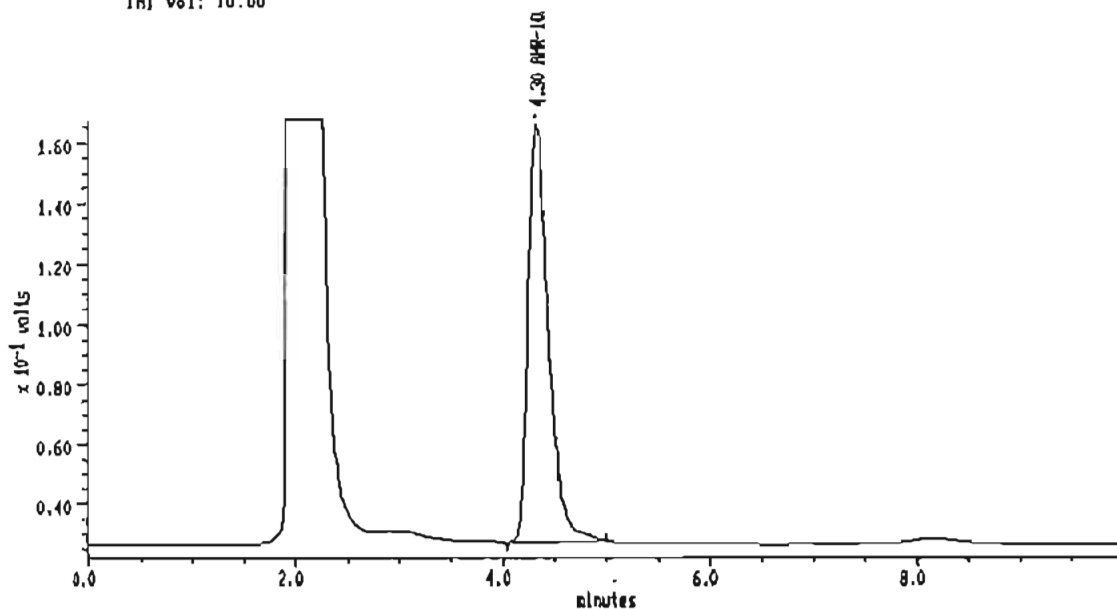
Index: 32

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	2041719	145631	100.00	AHR-10282B
TOTAL			2041719	145631		

Sample: A32 60°C-2W Channel: detector 1 Filename: S2-08 Chart Speed: Full Size
 Acquired: 02-APR-101 21:50 Method: B:YABRYIK13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 22:00:43

SAMPLE: A32 00°C-2W

#8 In Method: AHR-10282B

Acquired: 2-APR-2001 21:50

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-08

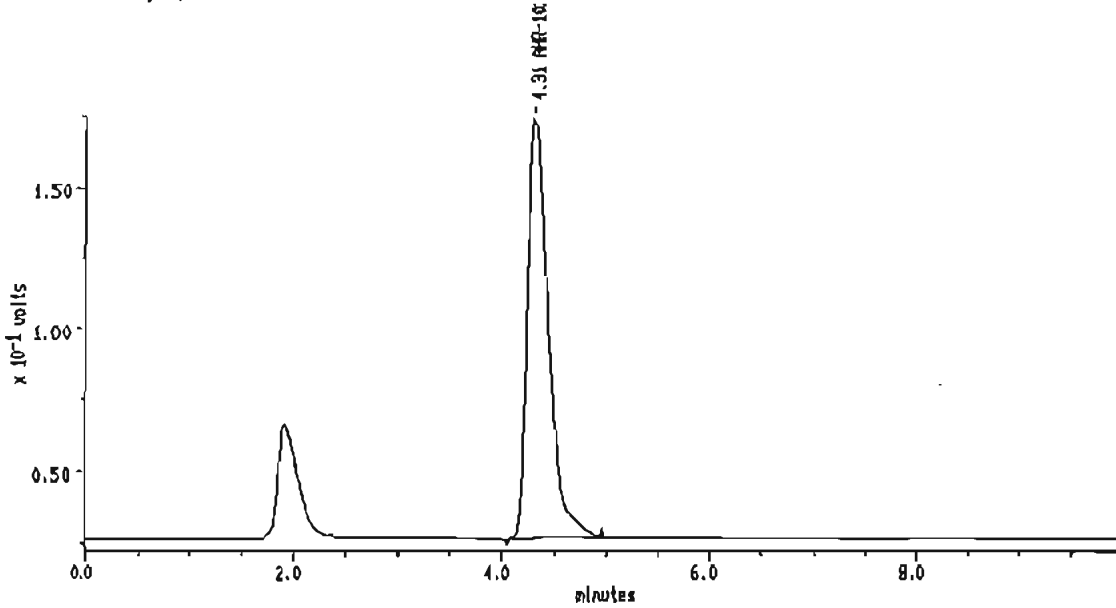
Index: 33

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	1930389	138435	100.00	AHR-10282B
TOTAL			1930389	138435		

Sample: A33 60°C-2W Channel: detector 1 Filename: S2-09 Chart Speed: Full Size
 Acquired: 02-APR-2001 22:01 Method: B:VAUGRYIK13V80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 22:11:59

SAMPLE: A33 60°C-2W

#9 In Method: AHR-10282B

Acquired: 2-APR-2001 22:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-09

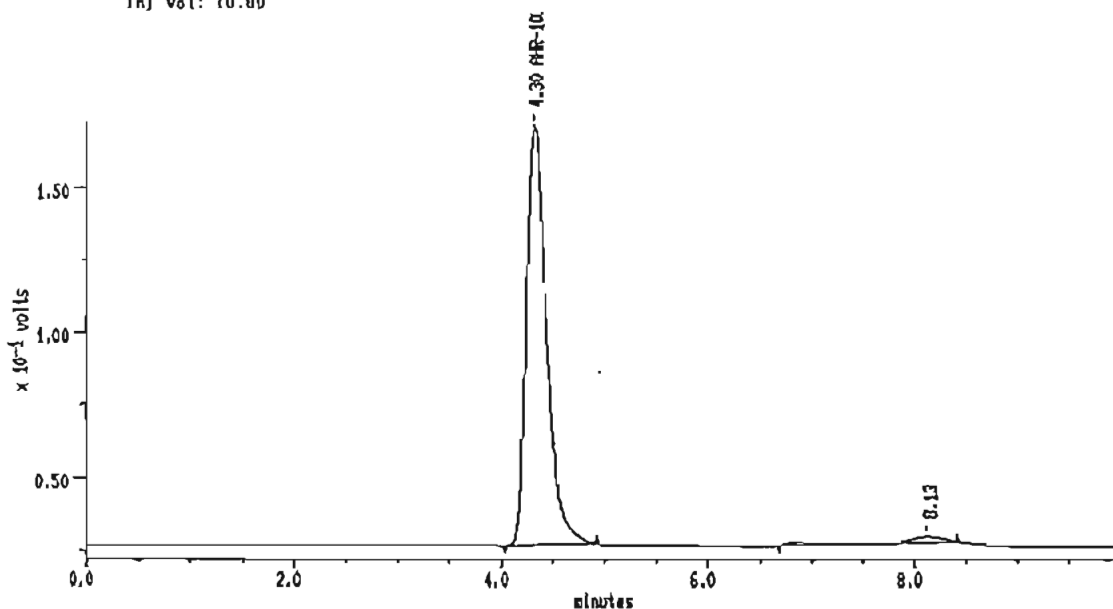
Index: 34

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.308	98	2070437	147508	100.00	AHR-10282B
TOTAL			2070437	147508		

Sample: A34 80°C-2W Channel: detector 1 Filename: S2-10 Chart Speed: Full Size
 Acquired: 02-APR-101 22:12 Method: B:YAHRVIX13Y86-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 22:23:18

SAMPLE: A34 80°C-2W

10 In Method: AHR-10282B

Acquired: 2-APR-2001 22:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-10

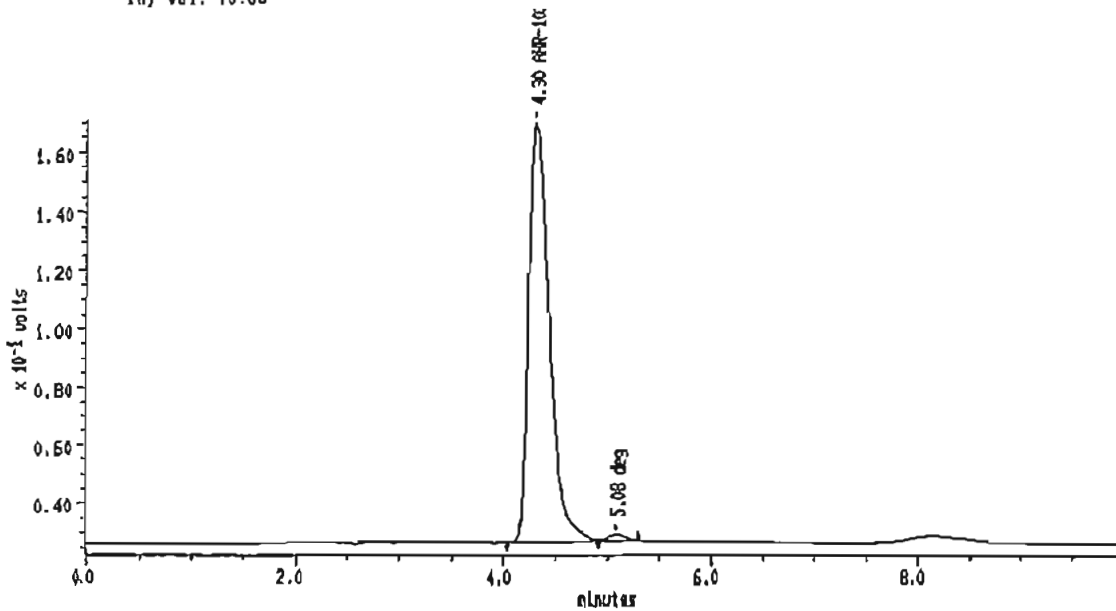
Index: 36

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	4.300	B0	2010017	145053	100.00	AHR-10282B
TOTAL			2010017	145053		

Sample: A35 60°C-2W Channel: detector 1 Filename: S2-11 Chart Speed: Full Size
 Acquired: 02-APR-2001 22:24 Method: B:VAHRV1K13Y60-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 22:34:32

SAMPLE: A35 60°C-2W

#11 in Method: AHR-10282B

Acquired: 2-APR-2001 22:24

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-11

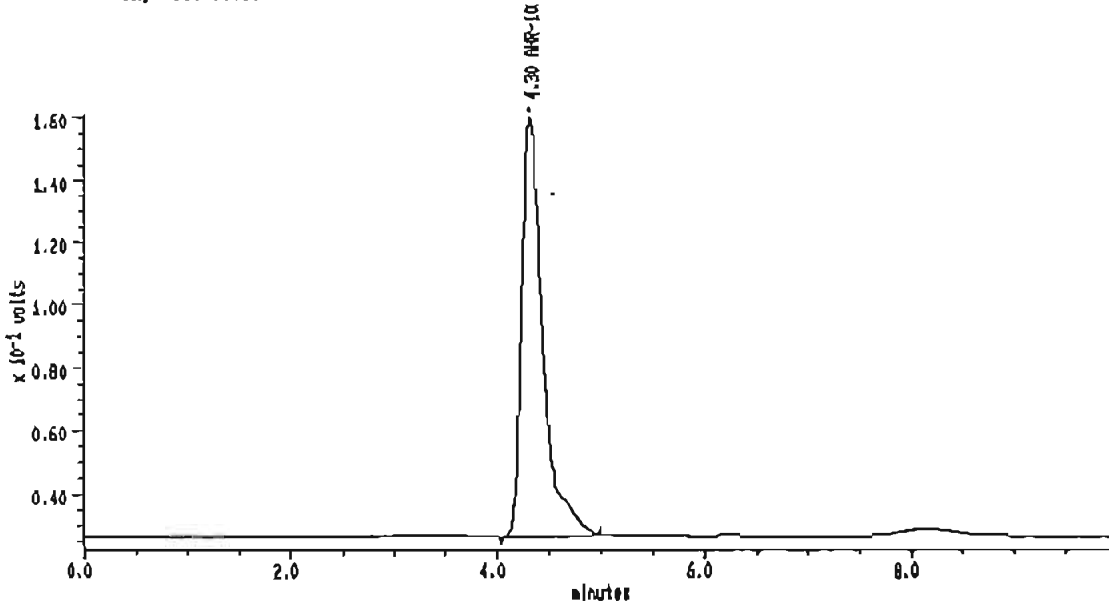
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	1890944	143218	98.91	AHR-10282B
2	5.076	SS	21895	1873	1.09	deg
TOTAL			2012839	145090		

Sample: A28 50°C-4W Channel: detector 1 Filename: S2-12 Chart Speed: Full Size
 Acquired: 02-APR-01 22:36 Method: B:YAHRYIK13V60-2W Operator: S.S
 Inj Volt: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 22:46:49

SAMPLE: A28 50°C-4W

#12 In Method: AHR-10282B

Acquired: 2-APR-2001 22:36

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-12

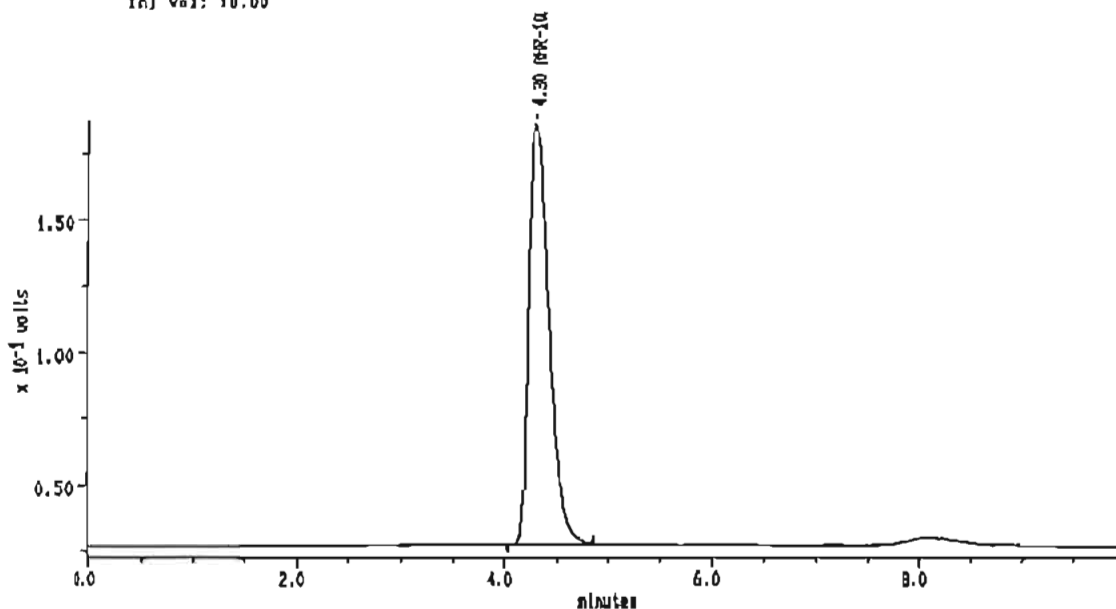
Index: 37

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	BB	1918788	133305	100.00	AHR-10282B
TOTAL			1918788	133305		

Sample: A27 50°C-4W Channel: detector 1 Filename: S2-13 Chart Speed: Full Size
 Acquired: 02-APR-101 22:46 Method: B:YAHRYJKI3V80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 22:57:05

SAMPLE: A27 50°C-4W

#13 In Method: AHR-10282B

Acquired: 2-APR-2001 22:40

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-13

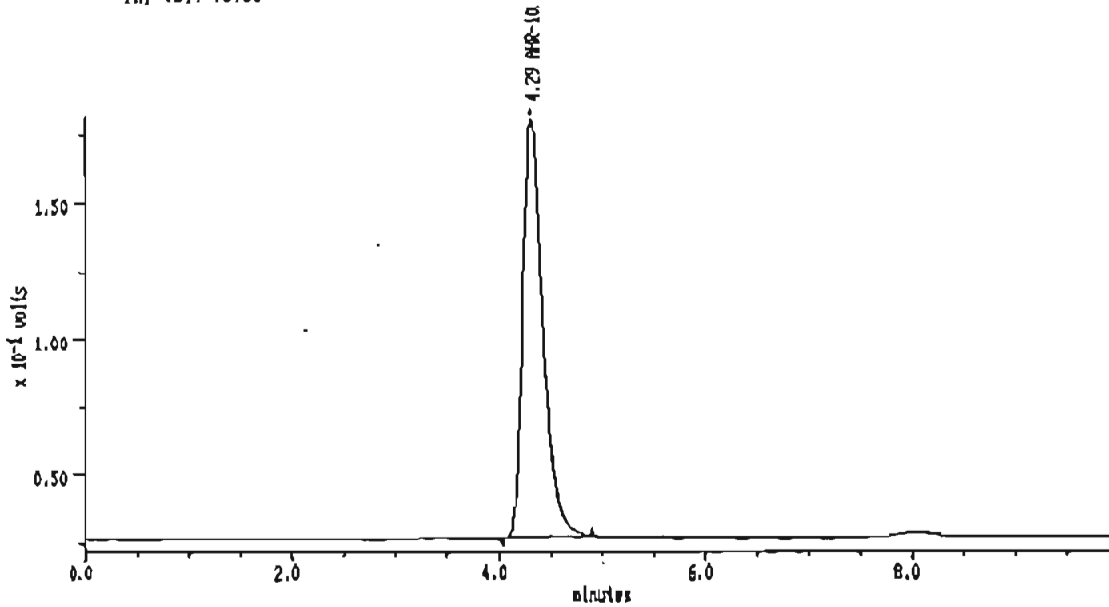
Index: 08

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.300	SB	2176249	159480	100.00	AHR-10282B
TOTAL			2176249	159480		

Sample: A28 50°C-4# Channel: detector 1 Filename: S2-14 Chart Speed: Full Size
 Acquired: 02-APR-01 22:57 Method: 8:YAHRYIKI3V80-29 Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

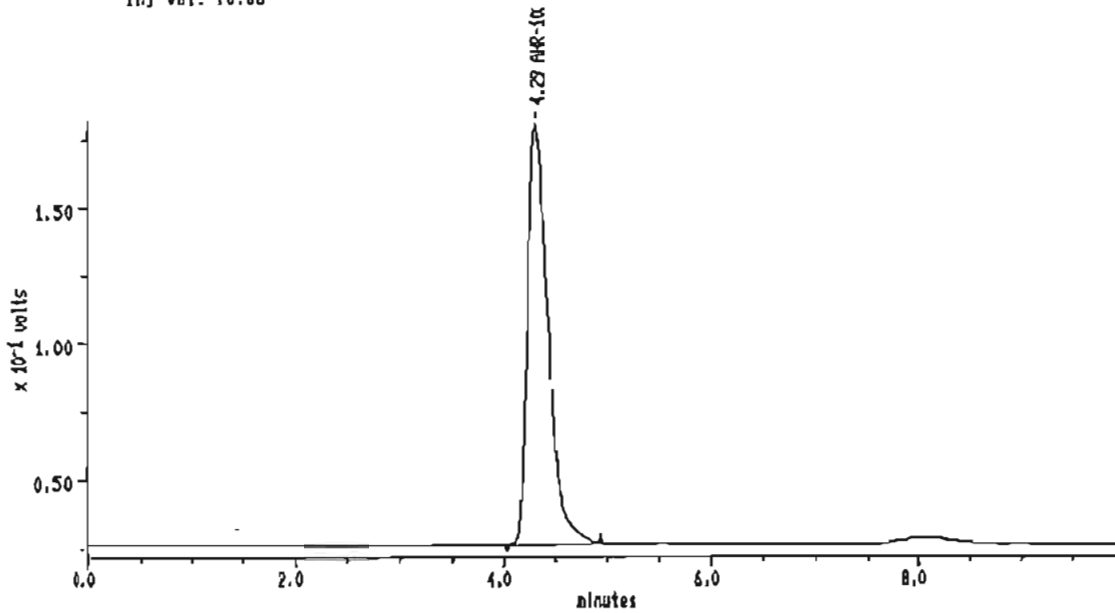
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SAMPLE: A28 50°C-4# Type: UNKNOWN
 414 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 2-APR-2001 22:57 Filename: S2-14
 Rate: 2.0 points/sec Index: 38
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	88	2105338	153388	100.00	AHR-10282B
TOTAL			2105338	153388		

Sample: A29 50°C-4W Channel: detector 1 Filename: S2-15 Chart Speed: Full Size
 Acquired: 02-APR-10 23:09 Method: B:YAIIRYIKI3V80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 23:19:38

SAMPLE: A29 50°C-4W

#15 In Method: AHR-10282B

Acquired: 2-APR-2001 23:09

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-15

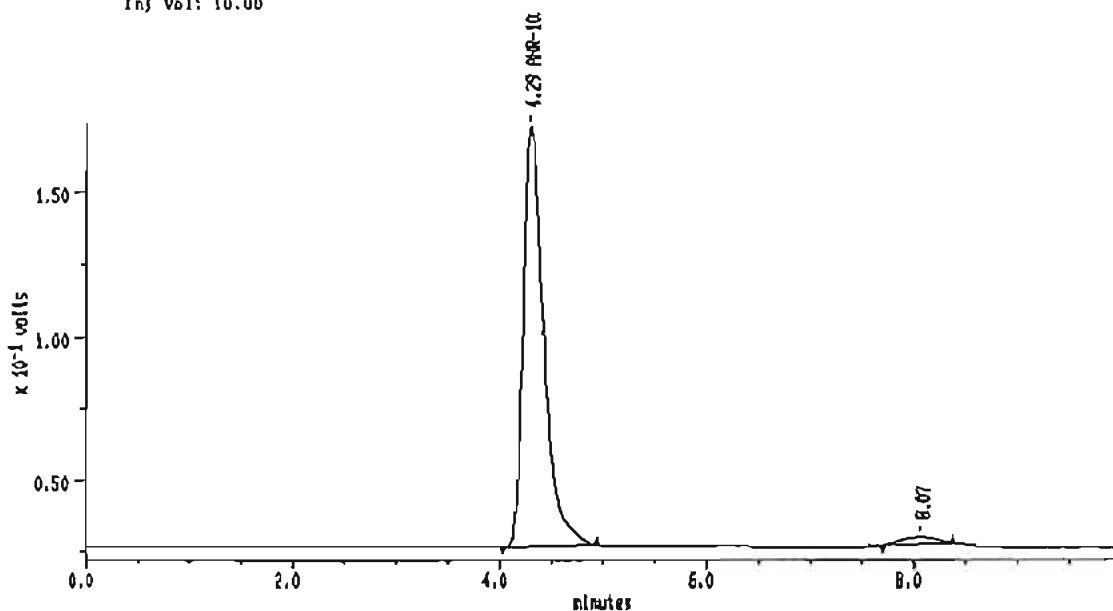
Index: 40

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BD	2137432	154351	100.00	AHR-10282B
TOTAL			2137432	154351		

Sample: A30 E0°C-4W Channel: detector 1 Filename: S2-18 Chart Speed: Full Size
 Acquired: 02-APR-101 23:20 Method: B:YAHRYIK13Y80-2W Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 2-APR-2001 23:30:58

SAMPLE: A30 E0°C-4W

#18 In Method: AHR-10282B

Acquired: 2-APR-2001 23:20

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-18

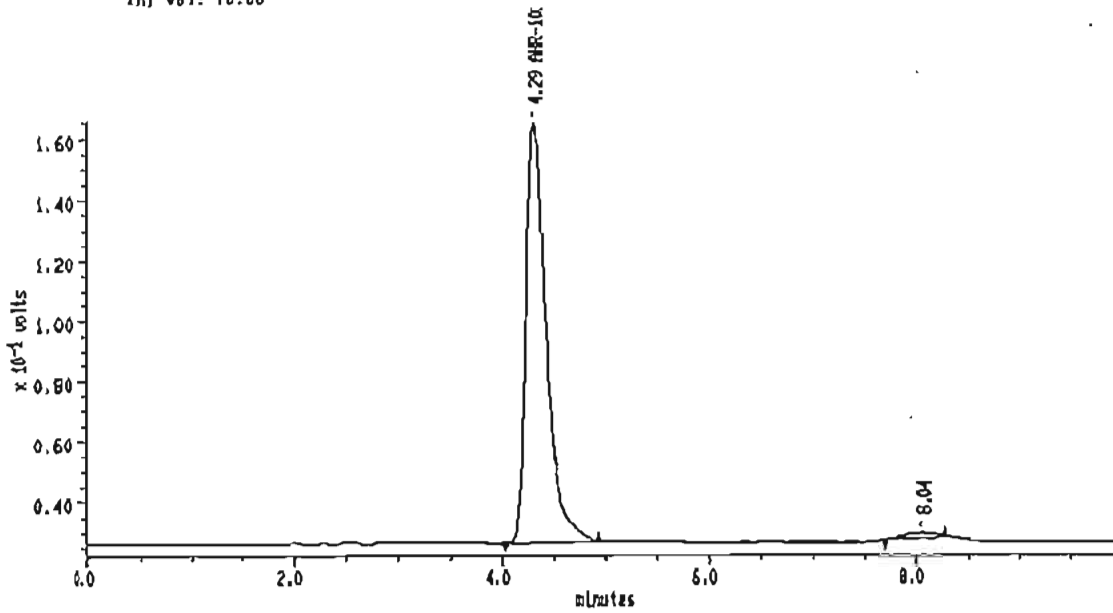
Index: 41

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.282	BB	2043998	146010	100.00	AHR-10282B
TOTAL			2043998	146010		

Sample: A31 50°C-4W Channel: detector 1 Filename: S2-17 Chart Speed: Full Size
 Acquired: 02-APR-101 23:31 Method: D:VAHRV1K13V80-2W Operator: S.S
 Inj Vol: 10.00



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Printed: 2-APR-2001 23:42:13

SAMPLE: A31 50°C-4W

#17 In Method: AHR-10282B

Acquired: 2-APR-2001 23:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-17

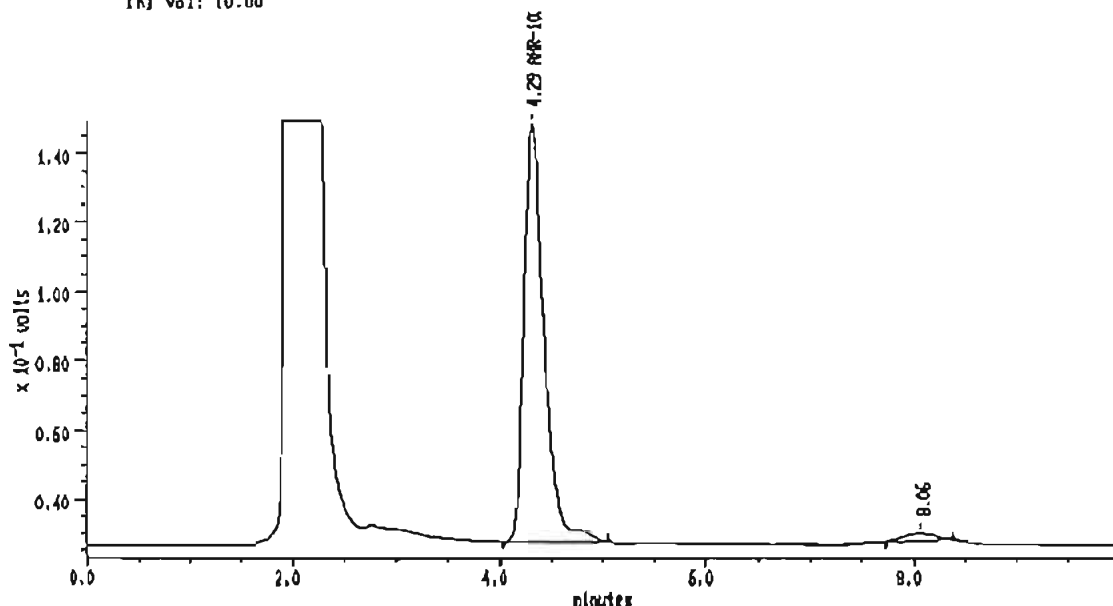
Index: 42

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BB	1933544	138348	100.00	AHR-10282B
TOTAL			1933544	138348		

Sample: A32 50°C-4W Channel: detector 1 Filename: S2-18 Chart Speed: Full Size
 Acquired: 02-APR-101 23:43 Method: B:YAHVVIX13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

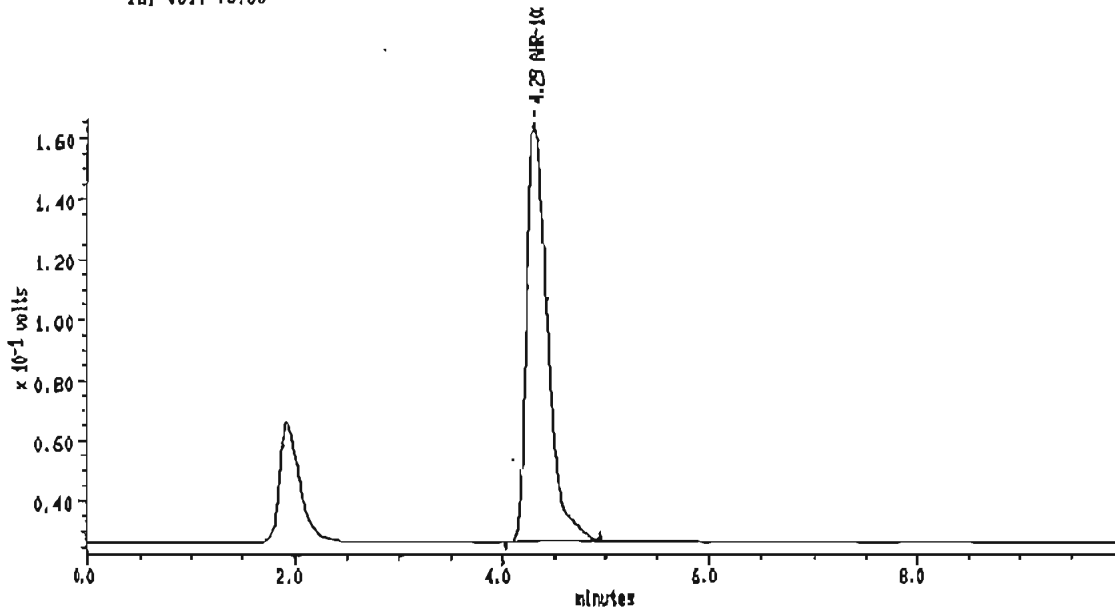
Printed: 2-APR-2001 23:53:30

SAMPLE: A32 50°C-4W Type: UNKN
 #18 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 2-APR-2001 23:43 Filename: S2-18
 Rate: 2.0 points/sec Index: 43
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S Amount: 0.000

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	UB	1892208	120875	100.00	AHR-10282B
TOTAL			1892208	120875		

Sample: A33 50°C-4W Channel: detector 1 Filename: S2-19 Chart Speed: Full Size
 Acquired: 02-APR-10 23:54 Method: B:YAHRYIK13Y80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 3-APR-2001 0:04:47

SAMPLE: A33 50°C-4W

#19 in Method: AHR-10282B

Acquired: 2-APR-2001 23:54

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-19

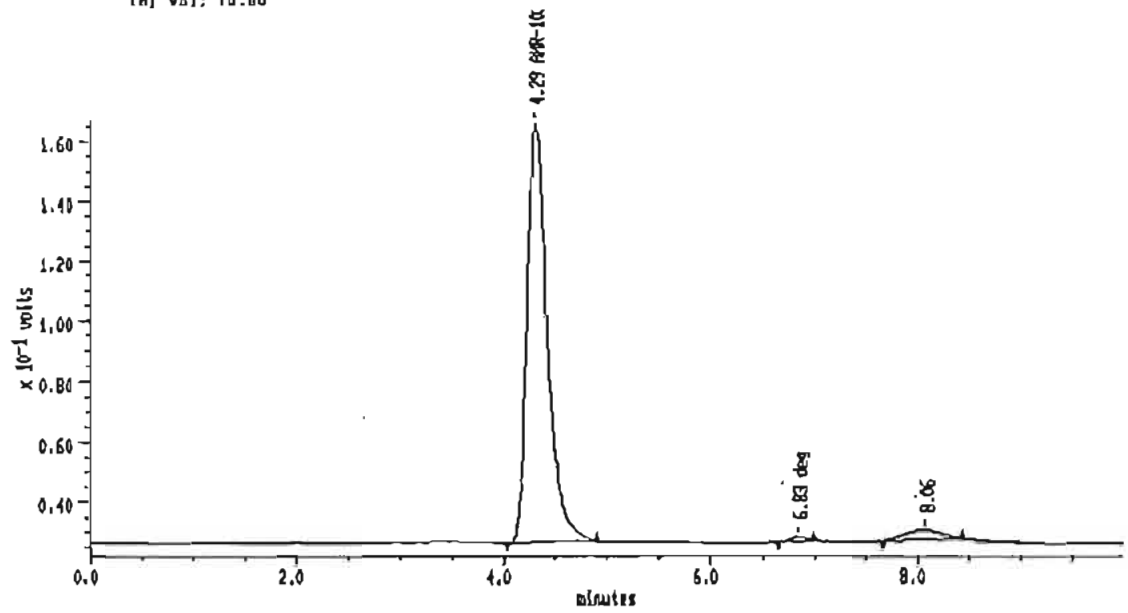
Index: 44

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BB	1942783	138393	100.00	AHR-10282B
TOTAL			1942783	138393		

Sample: A34 60°C-4W Channel: detector 1 Filename: S2-20 Chart Speed: Full Size
 Acquired: 03-APR-2001 0:05 Method: B:YAHRYIK13Y80-2W Operator: S.S
 [n] Vol: 10.00



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MAXIMA 825 カスタムレポート

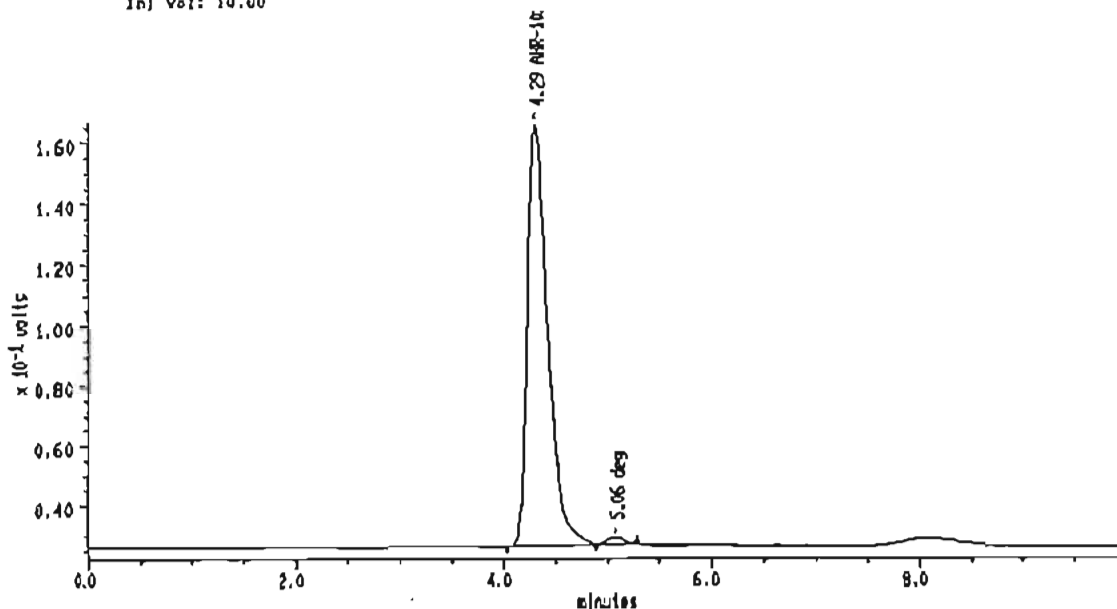
Printed: 3-APR-2001 0:10:04

SAMPLE: A34 60°C-4W Type: UNKN
 #20 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 3-APR-2001 0:05 Filename: S2-20
 Rate: 2.0 points/sec Index: 45
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BB	1922389	139185	99.18	AHR-10282B
2	8.033	BB	15859	1272	0.82	deg
TOTAL			1938228	140457		

Sample: A35 50°C-4W Channel: detector 1 Filename: S2-21 Chart Speed: Full Size
 Acquired: 03-APR-2001 0:16 Method: B:YAHRYIK13V80-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 3-APR-2001 0:27:21

SAMPLE: A35 50°C-4W

#21 ID Method: AHR-10282B

Acquired: 3-APR-2001 0:16

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-21

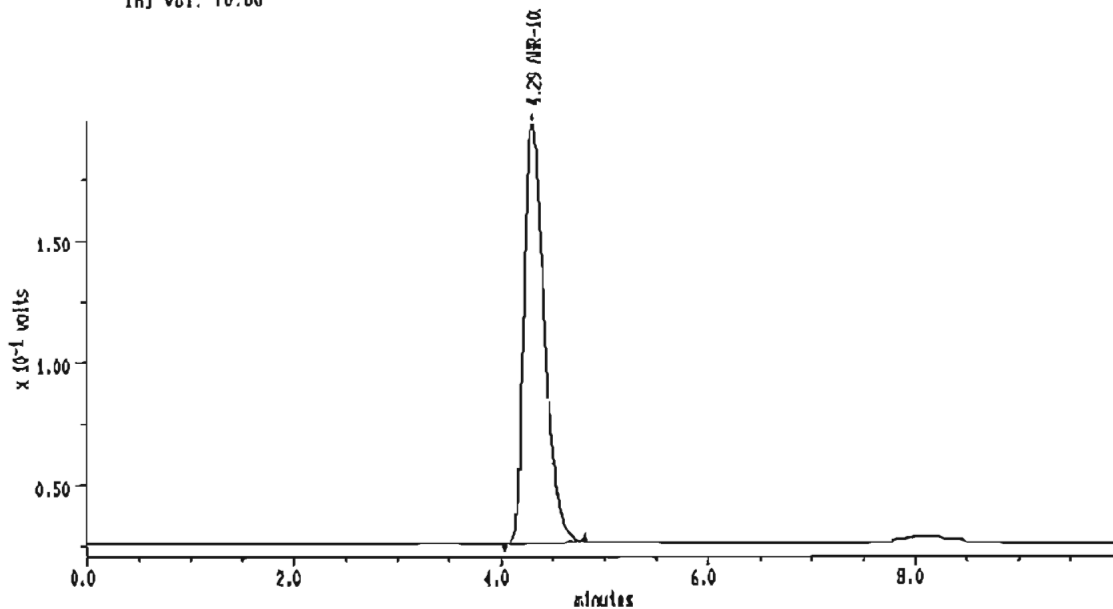
Index: 48

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.292	BD	1927309	138800	90.83	AHR-10282B
2	5.058	SS	22848	1049	1.17	deg
TOTAL			1950167	140835		

Sample: ST02 Channel: detector 1 Filename: S2-22 Chart Speed: Full Size
 Acquired: 03-APR-101 0:28 Method: B:YAIRVIRIKI3Y60-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 3-APR-2001 0:38:50

SAMPLE: ST02

#22 In Method: AHR-10282B

Acquired: 3-APR-2001 0:28

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S2-22

Index: 47

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.282	BB	2321811	170812	100.00	AIR-10282B
TOTAL			2321811	170812		

4/02 17:49
NO.92 PH 7.05
24.1°C

4/02 18:07
NO. 4 PH 7.06
23.9°C

4/02 17:51
NO.93 PH 7.10
24.3°C

4/02 18:12
NO. 5 PH 7.11
24.2°C

4/02 17:53
NO.94 PH 7.08
24.3°C

4/02 18:14
NO. 6 PH 7.10
23.9°C

4/02 17:55
NO.95 PH 7.05
24.1°C

4/02 18:15
NO. 7 PH 7.08
23.8°C

4/02 17:56
NO.96 PH 7.09
24.1°C

4/02 18:16
NO. 8 PH 7.09
23.6°C

4/02 17:59
NO.97 PH 7.07
24.0°C

4/02 18:17
NO. 9 PH 7.05
23.6°C

4/02 18:00
NO.98 PH 7.07
23.9°C

4/02 18:19
NO.10 PH 7.10
23.7°C

4/02 18:01
NO.99 PH 7.09
23.9°C

4/02 18:21
NO.11 PH 7.12
23.7°C

4/02 18:02
NO. 1 PH 7.07
23.9°C

4/02 18:23
NO.12 PH 7.11
23.7°C

4/02 18:04
NO. 2 PH 7.11
23.9°C

4/02 18:26
NO.13 PH 7.14
24.2°C

4/02 18:06
NO. 3 PH 7.09
23.6°C

P2000B177 Lot No. 01K131 66C-2W

本生データは経時的な劣化のおそれがあるため、複写しました。
従って、原本と相違ありません。

'05.05.06 澤 嗣郎

プロナック点眼液の安定性試験
Lot No.01K131

試験コード：P2000B177
試験実施者：澤 嗣郎
試験実施日：2001年03月23日

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Water Collec(%)	Initial	present
STD	1	V2-01	2290068					
STD	2	V2-22	2319969					
STD	mean		2305019	0.9975				
A-26	70°C-2W	V2-02	2063024	0.8928	89.24	82.40	7.67	8.4915 8.1202
A-27	70°C-2W	V2-03	2247015	0.9724	98.54	90.69	7.97	8.3951 8.0171
A-28	70°C-2W	V2-04	2213332	0.9578	96.54	89.01	7.80	8.5470 8.1654
A-29	70°C-2W	V2-05	2240698	0.9697	93.85	84.93	9.50	8.4962 8.0358
A-30	70°C-2W	V2-06	2175407	0.9414	92.78	83.08	10.46	8.4388 7.9380
A-31	70°C-2W	V2-07	2034107	0.8803	86.69	79.82	7.92	8.4972 8.1133
A-32	70°C-2W	V2-08	1628485	0.7039	66.90	61.18	8.55	8.4194 8.0117
A-33	70°C-2W	V2-09	2080969	0.9005	89.84	82.51	8.16	8.4398 8.0487
A-34	70°C-2W	V2-10	2003119	0.8669	86.41	79.60	7.88	8.4808 8.1003
A-35	70°C-2W	V2-11	1915502	0.8289	82.00	75.89	7.45	8.5033 8.1417
A-26	80°C-4W	V2-12	1854161	0.8024	80.21	74.86	6.87	8.4743 8.1527
A-27	80°C-4W	V2-13	2194535	0.9497	96.24	89.64	6.86	8.4358 8.1075
A-28	60°C-4W	V2-14	2111436	0.9137	92.10	85.98	6.67	8.5133 8.1889
A-29	60°C-4W	V2-15	2112402	0.9141	88.47	82.01	7.30	8.4560 8.1051
A-30	60°C-4W	V2-16	2040435	0.8830	87.02	80.71	7.25	8.4431 8.0958
A-31	60°C-4W	V2-17	1868891	0.8092	79.68	74.12	6.98	8.6017 8.2564
A-32	60°C-4W	V2-18	1243872	0.5383	51.16	47.37	7.41	8.4342 8.0797
A-33	60°C-4W	V2-19	1905780	0.8247	82.28	78.82	6.63	8.4998 8.1784
A-34	60°C-4W	V2-20	1850867	0.8010	79.84	74.04	7.26	8.4355 8.0883
A-35	60°C-4W	V2-21	1813088	0.7846	77.62	72.33	6.81	8.6422 8.3022

計算に必要となる値を記載するため、再計算した。
105.05.06 澤 嗣郎

試験物質名: AHK10282B		試験コード: P2000B/77		試験年月日: 200 / 年 03 月 23 日		
試験項目:				試験実施者: 澤 嗣郎		
Lot No. 01K131						
STD 0.01995g AHK10282B + MP → 20ml						
上記 2ml + MP → 20ml						
		pH		濁り	異物 色	
A-26	70C-2W	6.89	8.4915 8.1202	+	+	淡黄色
A-27		7.00	8.3951 8.0171	+	+	黄色
A-28		7.00	8.5470 8.1654	+	+	黄色
A-29		6.96	8.4962 8.0358	+	+	濃黄色
A-30		7.00	8.4388 7.9380	+	+	(r)
A-31		6.93	8.4972 8.1133	+	+	(r)
A-32		7.00	8.4194 8.0117	+	+	
A-33		6.99	8.4396 8.0487	+	+	(r) ↓
A-34		6.95	8.4808 8.1003	+	+	(r) 黄色
A-35		7.03	8.5033 8.1417	+	+	(r) ↓
A-26	68C-4W	7.00	8.4743 8.1527	+	+	淡黄色
A-27		6.99	8.4358 8.1075	+	+	(r) 黄色
A-28		6.96	8.5133 8.1889	+	+	(r) 黄色
A-29		6.94	8.4560 8.1051	+	+	(r) 淡黄色
A-30		6.96	8.4431 8.0958	+	+	(r) ↓
A-31		6.93	8.6017 8.2564	+	+	(r) 黄色
A-32		7.01	8.4342 8.0797	++	+	(r) 淡黄色
A-33		7.00	8.4998 8.1784	++	+	(r)
A-34		7.02	8.4355 8.0883	+	+	(r) ↓
A-35		7.04	8.6422 8.3022	+	+	(r) ↓

3/23 17:07
NO.65 PH 6.89
25.2°C

3/23 17:08
NO.66 PH 7.00
25.6°C

3/23 17:09
NO.67 PH 7.00
25.7°C

3/23 17:10
NO.68 PH 6.95
25.7°C

3/23 17:11
NO.69 PH 7.00
25.7°C

3/23 17:12
NO.70 PH 6.93
25.7°C

3/23 17:14
NO.71 PH 7.00
25.6°C

3/23 17:16
NO.72 PH 6.99
25.6°C

3/23 17:17
NO.73 PH 6.95
25.6°C

3/23 17:18
NO.74 PH 7.03
25.7°C

3/23 17:20
NO.75 PH 7.00
25.8°C

3/23 17:22
NO.76 PH 6.99
25.9°C

3/23 17:23
NO.77 PH 6.95
26.0°C

3/23 17:24
NO.78 PH 6.94
25.8°C

3/23 17:24
NO.79 PH 6.96
25.8°C

3/23 17:25
NO.80 PH 6.95
25.8°C

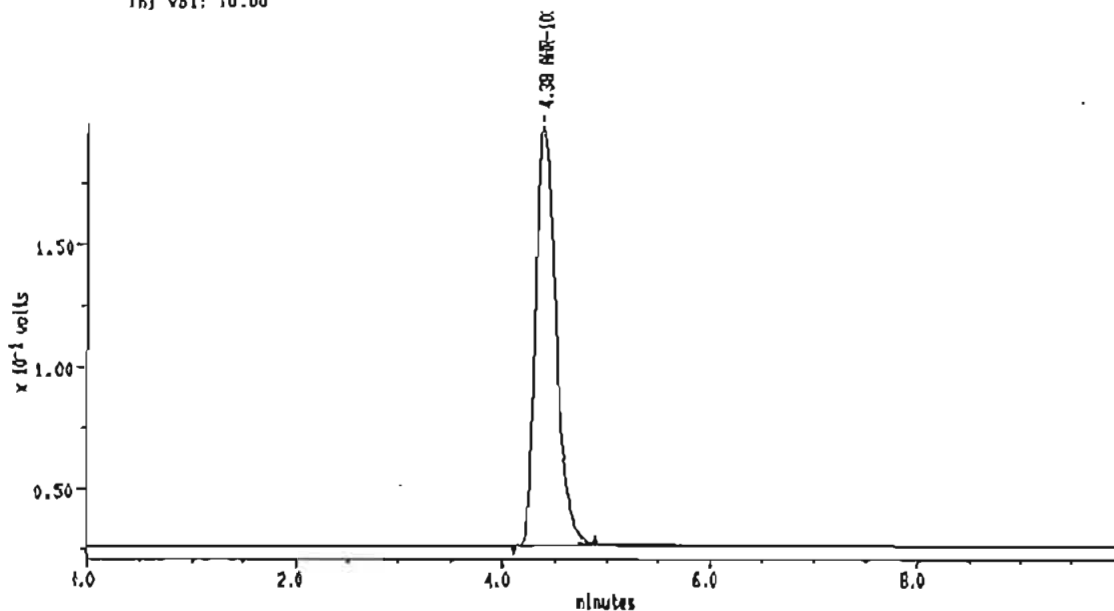
3/23 17:28
NO.81 PH 7.01
25.7°C

3/23 17:29
NO.82 PH 7.00
25.9°C

3/23 17:31
NO.83 PH 7.02
25.8°C

3/23 17:31
NO.84 PH 7.04
25.8°C

Sample: STD1 Channel: detector 1 Filename: V2-01 Chart Speed: Full Size
 Acquired: 23-MAR-2001 21:48 Method: B:YAHRY1K13Y70-2 Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 17:08:21

SAMPLE: STD1

#1 In Method: AHR-10282B

Acquired: 23-MAR-2001 21:48

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-01

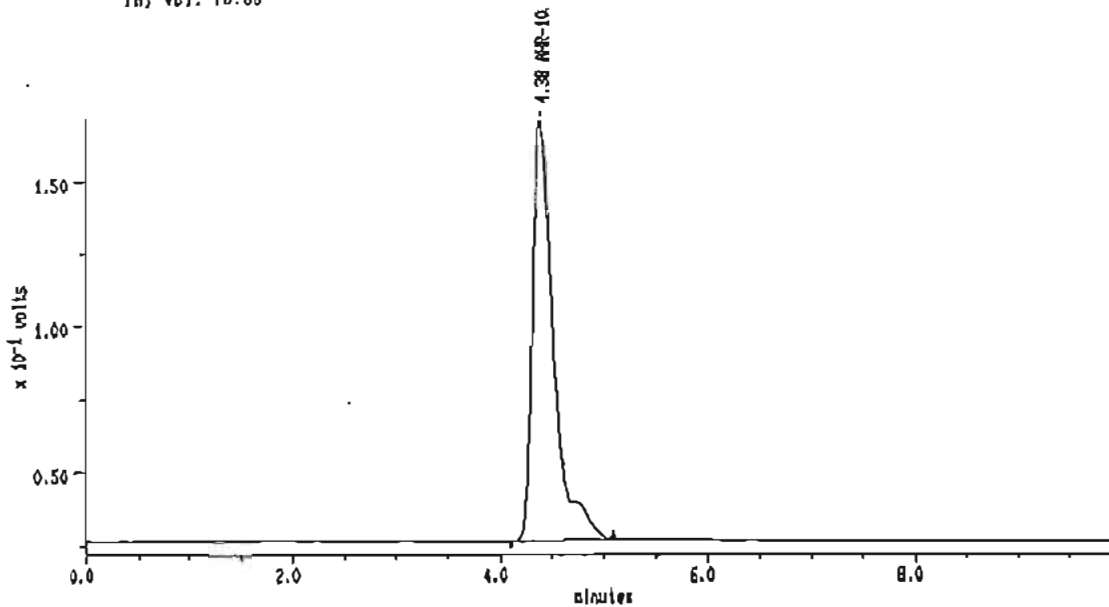
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.383	BB	2290088	171093	100.00	AHR-10282B
TOTAL			2290088	171093		

Sample: A26 70°C-2W Channel: detector 1 Filename: V2-02 Chart Speed: Full Size
 Acquired: 23-MAR-01 21:59 Method: B:YAHRYIK13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

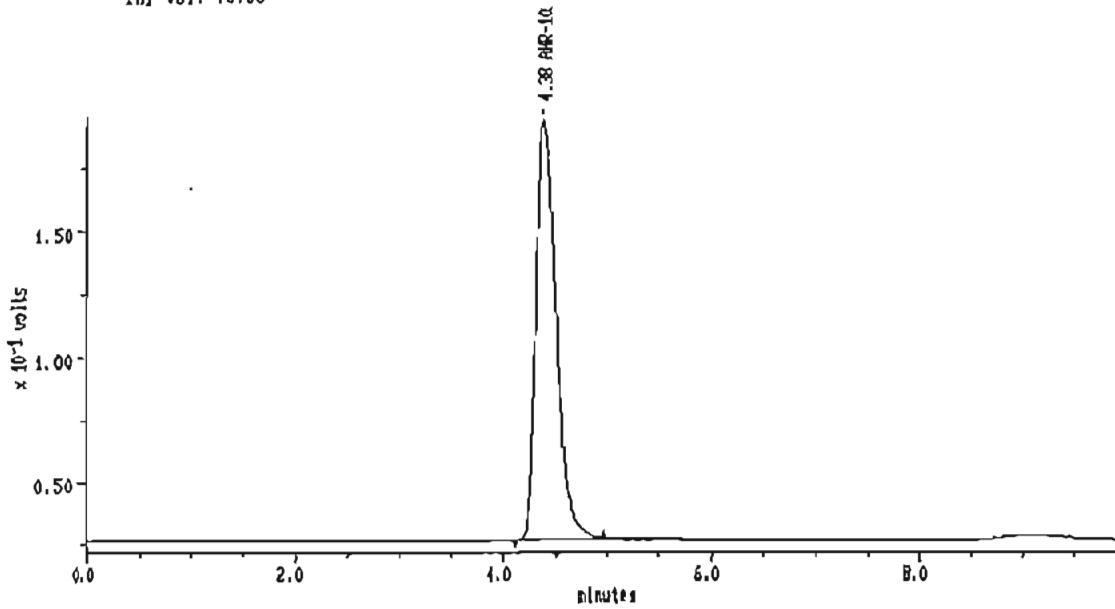
Printed: 28-MAR-2001 12:08:51

SAMPLE: A26 70°C-2W Type: CNKN
 #2 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 23-MAR-2001 21:59 Filename: V2-02
 Rate: 2.0 points/sec Index: 27
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.383	BB	2083024	143788	100.00	AHR-10282B
TOTAL			2083024	143788		

Sample: A27 70°C-2W Channel: detector 1 Filename: V2-03 Chart Speed: Full Size
 Acquired: 23-MAR-2001 22:11 Method: 0:VAJRY1K13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:08:21

SAMPLE: A27 70°C-2W

#3 In Method: AHR-10282B

Acquired: 23-MAR-2001 22:11

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-03

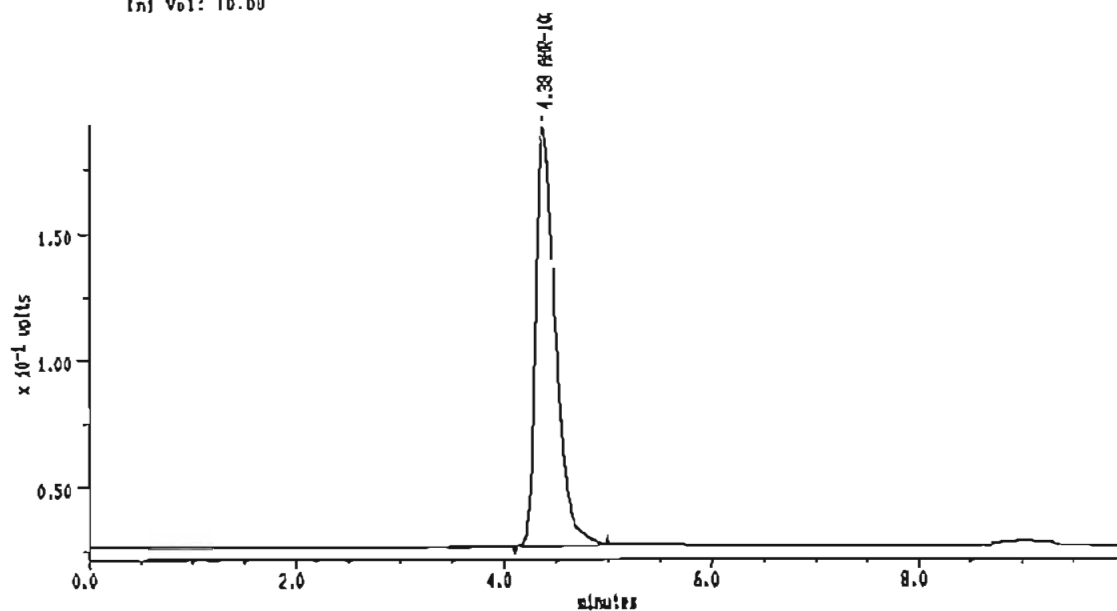
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.375	DB	2247015	187844	100.00	AHR-10282B
TOTAL			2247015	187844		

Sample: A20 70°C-2W Channel: detector 1 Filename: V2-04 Chart Speed: Full Size
 Acquired: 23-MAR-2001 22:22 Method: 8:VAIRYIKI3V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:09:52

SAMPLE: A20 70°C-2W

Method: AHR-10282B

Acquired: 23-MAR-2001 22:22

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-04

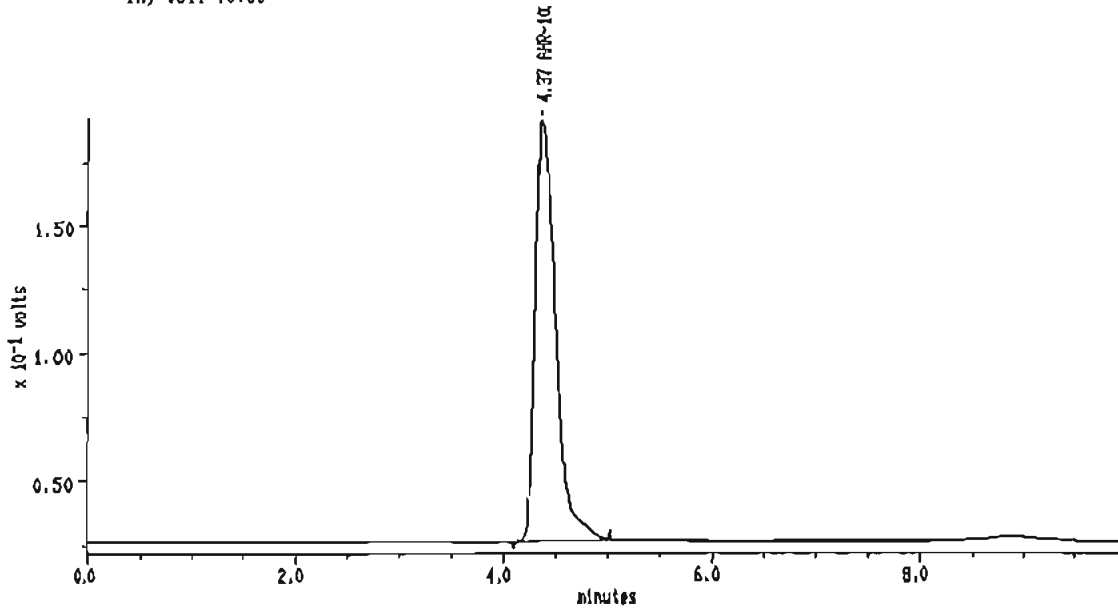
Index: 29

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.375	BB	2213332	183942	100.00	AHR-10282B
TOTAL			2213332	183942		

Sample: A29 70°C-2W Channel: detector 1 Filename: V2-06 Chart Speed: Full Size
 Acquired: 23-MAR-01 22:33 Method: B:YAHIRYIK13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-MAR-2001 12:10:23

SAMPLE: A29 70°C-2W

#5 In Method: AHR-10282B

Acquired: 23-MAR-2001 22:33

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-06

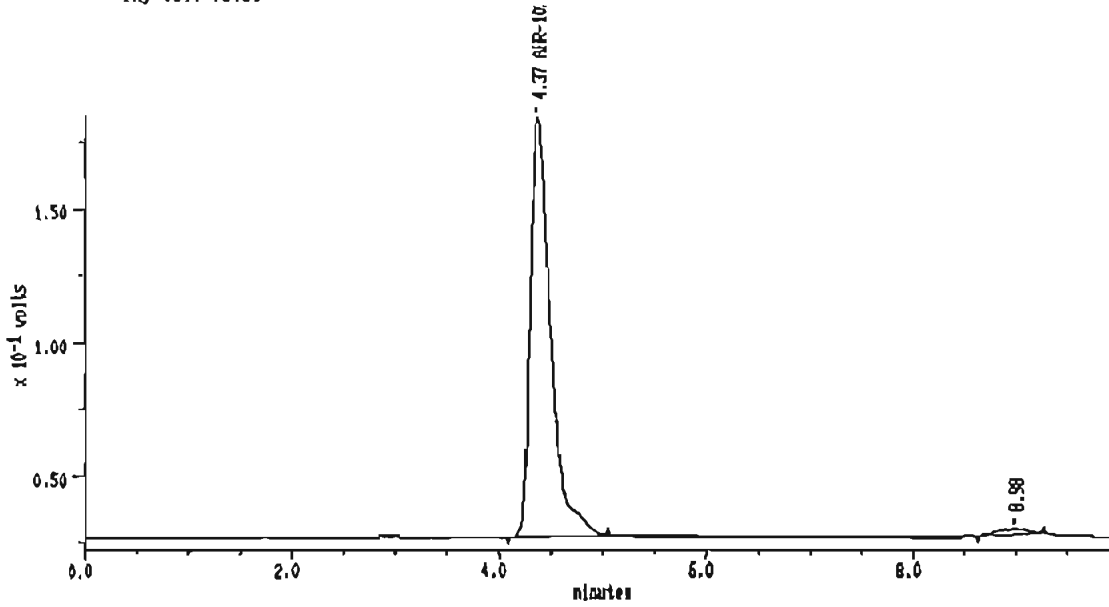
Index: 90

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.367	BB	2240898	184307	100.00	AHR-10282B
TOTAL			2240898	184307		

Sample: A30 70°C-2W Channel: detector 1 File name: V2-08 Chart Speed: Full Size
 Acquired: 23-MAR-2001 22:45 Method: 8:VAHRV1K13Y70-2 Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:10:64

SAMPLE: A30 70°C-2W

#6 In Method: AHR-10282B

Acquired: 23-MAR-2001 22:45

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

File name: V2-08

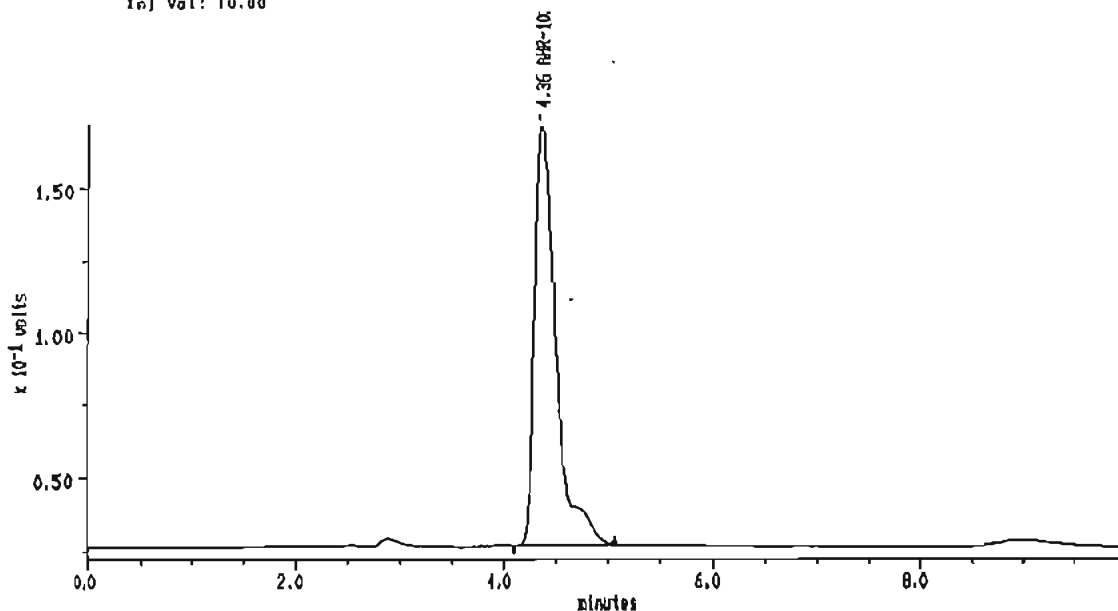
Index: 31

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.367	DB	2176407	158906	100.00	AHR-10282B
TOTAL			2176407	158906		

Sample: A31 70°C-2W Channel: detector 1 Filename: V2-07 Chart Speed: Full Size
 Acquired: 23-MAR-2001 22:58 Method: B:VAHRYIK13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:11:24

SAMPLE: A31 70°C-2W

#7 In Method: AHR-10282B

Acquired: 23-MAR-2001 22:58

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-07

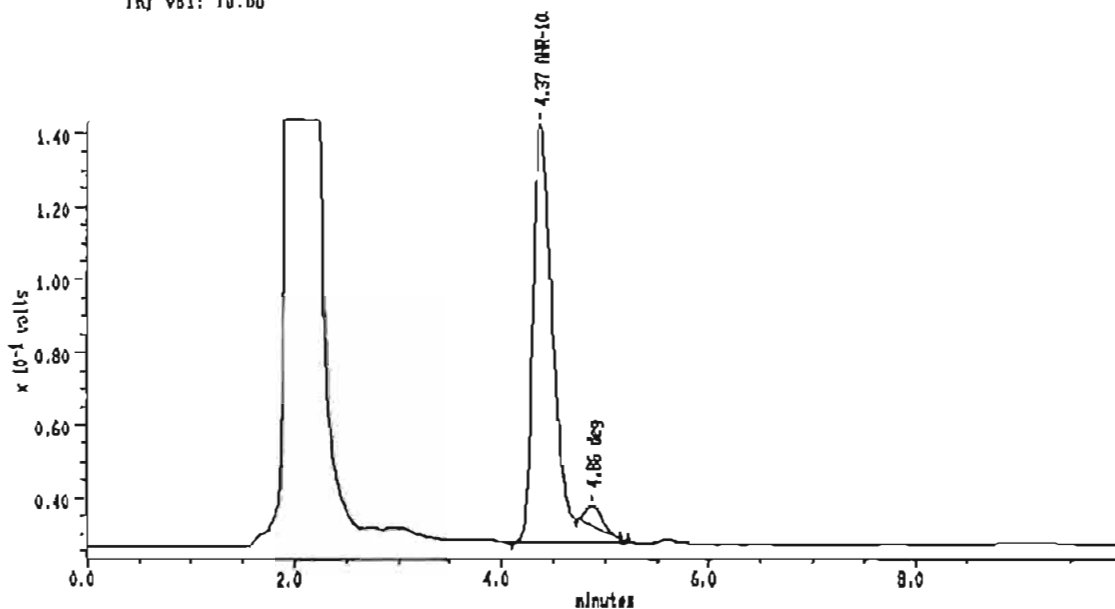
Index: 32

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.358	DB	2034107	143809	100.00	AHR-10282B
TOTAL			2034107	143809		

Sample: A32 70°C-2W Channel: detector 1 Filename: V2-08 Chart Speed: Full Size
 Acquired: 23-MAR-101 23:07 Method: D:YAHRYIK13V70-2W Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:11:66

SAMPLE: A32 70°C-2W

#8 In Method: AHR-10282B

Acquired: 23-MAR-2001 23:07

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-08

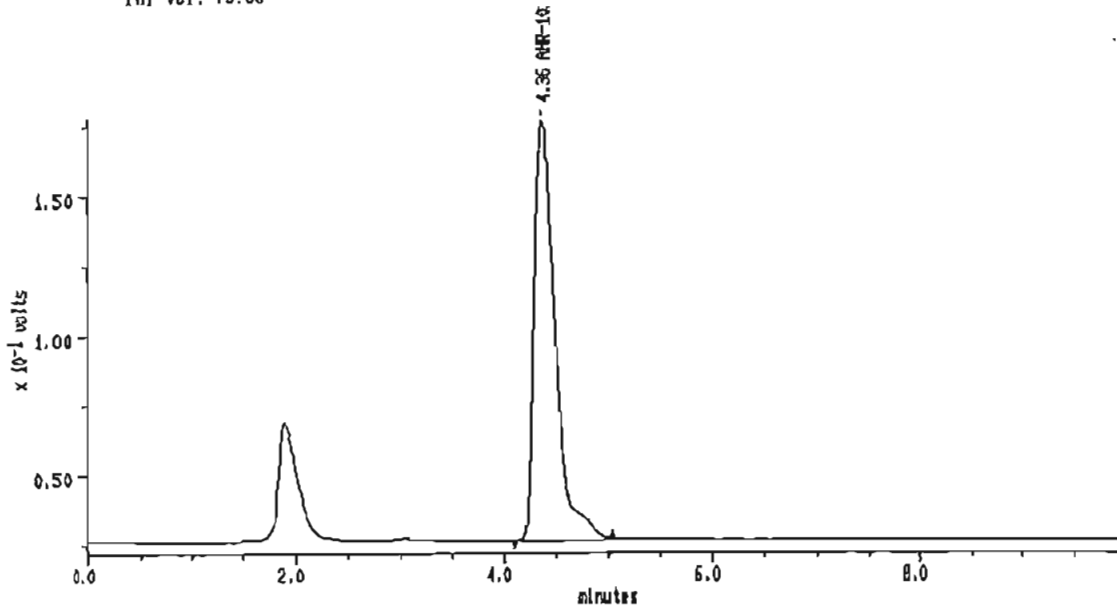
Index: 33

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.387	BB	1828486	114940	88.28	AHR-10282B
2	4.858	SS	83129	6450	3.74	deg
TOTAL			1889594	120390		

Sample: A33 70°C-2W Channel: detector 1 Filename: V2-09 Chart Speed: Full Size
 Acquired: 23-MAR-01 23:19 Method: B:VAJRYIKI3Y7D-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:12:28

SAMPLE: A33 70°C-2W

#9 in Method: AHR-10282B

Acquired: 23-MAR-2001 23:19

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-09

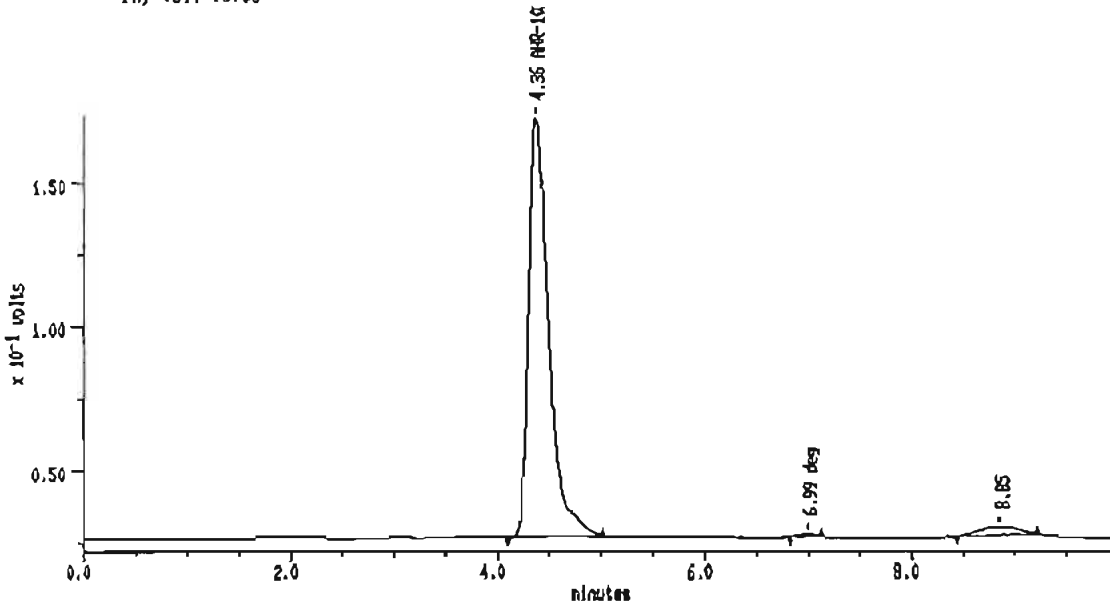
Index: 34

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.358	BB	2080989	149986	100.00	AHR-10282B
TOTAL			2080989	149986		

Sample: A34 70°C-2W Channel: detector 1 Filenames: V2-10 Chart Speed: Full Size
 Acquired: 23-MAR-10 23:30 Method: B:YAHRYIX13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:12:58

SAMPLE: A34 70°C-2W

=10 In Method: AHR-10282B

Acquired: 23-MAR-2001 23:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filenames: V2-10

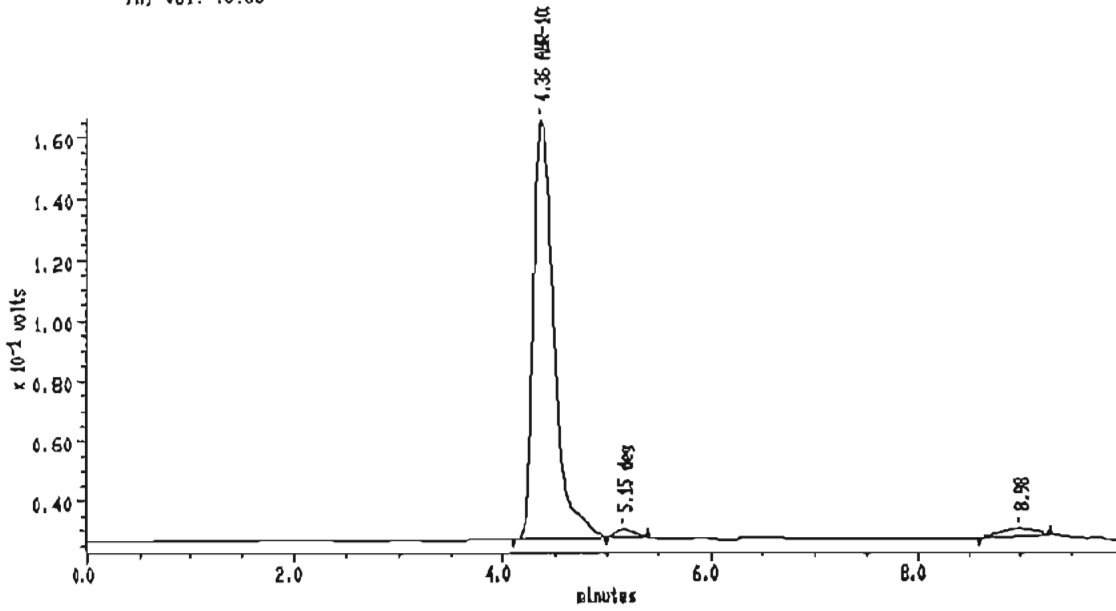
Index: 35

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.358	BB	2003119	145778	99.40	AHR-10282B
2	6.992	BB	12177	1039	0.60	deg
TOTAL			2015296	146815		

Sample: A36 70°C-2W Channel: detector 1 Filename: V2-11 Chart Speed: Full Size
 Acquired: 23-MAR-2001 23:41 Method: B:YAJIRYIK13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:13:30

SAMPLE: A36 70°C-2W

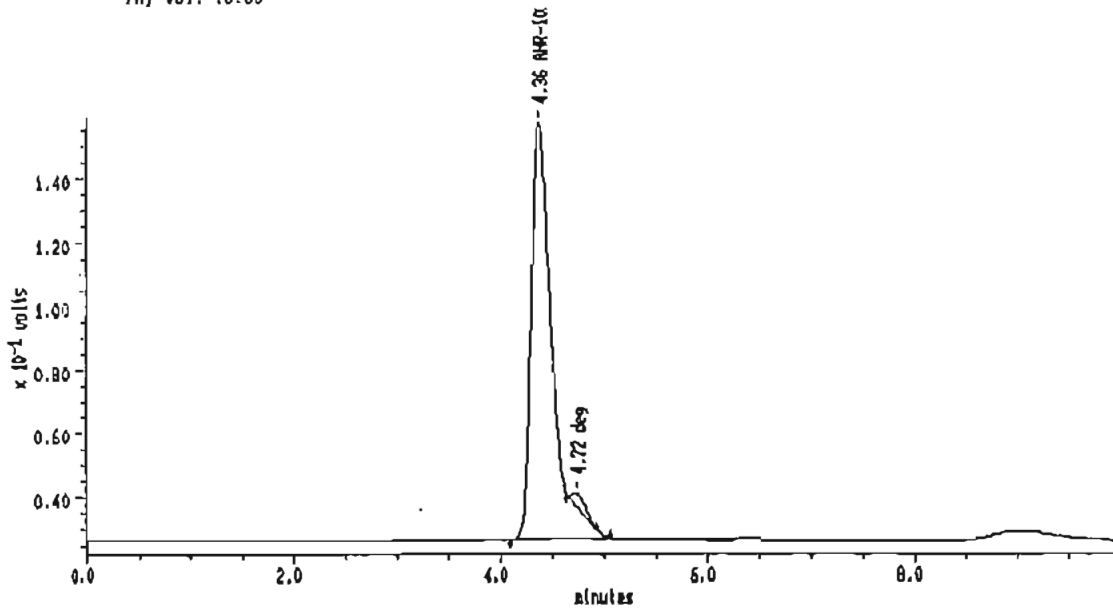
#11 (n Method: AHR-10282B
 Acquired: 23-MAR-2001 23:41
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument)
 Filename: V2-11
 Index: 38
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.368	BD	1916602	138408	98.84	AHR-10282B
2	5.150	SS	26468	2224	1.38	deg
TOTAL			1941967	140832		

Sample: A28 60°C-4W Channel: detector 1 Filename: V2-12 Chart Speed: Full Size
 Acquired: 23-MAR-10 23:52 Method: B:YAMUYIK13V70-2W Operator: S.S
 Inf Vol: 10.00



MAXIMA 101890 Dynamic Solutions, Division of Millipore

MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:30:07

SAMPLE: A28 60°C-4W

#12 in Method: AHR-10282B

Acquired: 23-MAR-2001 23:52

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-12

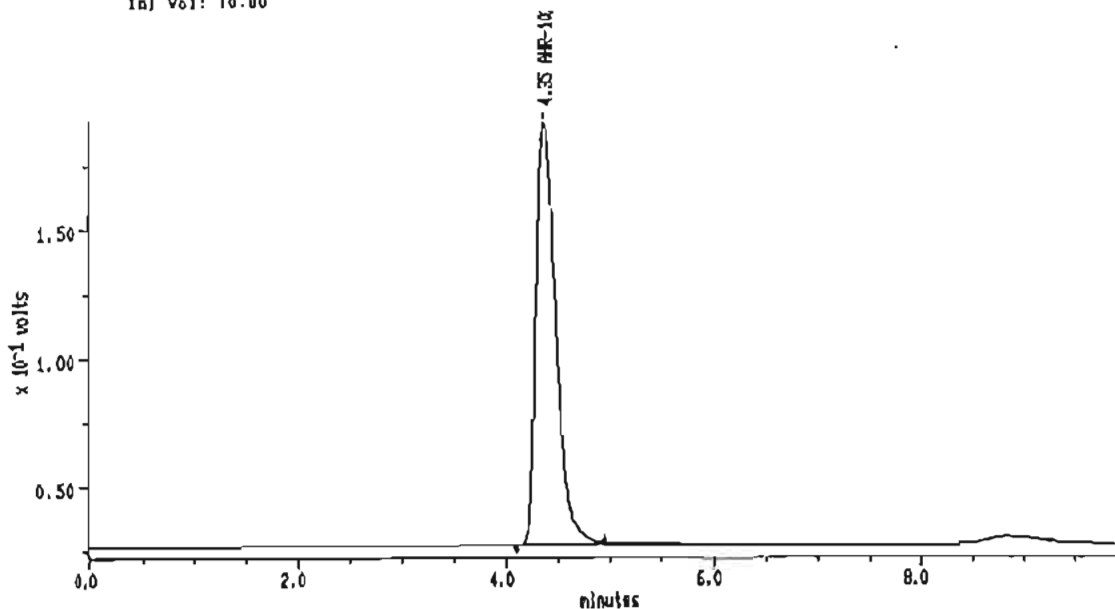
Index: 37

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.360	BB	1854101	130520	98.02	AHR-102820
2	4.717	SS	37403	4260	1.98	deg
TOTAL			1891644	134778		

Sample: A27 80°C-4W Channel: detector 1 Filename: V2-13 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:04 Method: 8:VAHRVIK13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:14:33

SAMPLE: A27 80°C-4W

#13 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:04

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-13

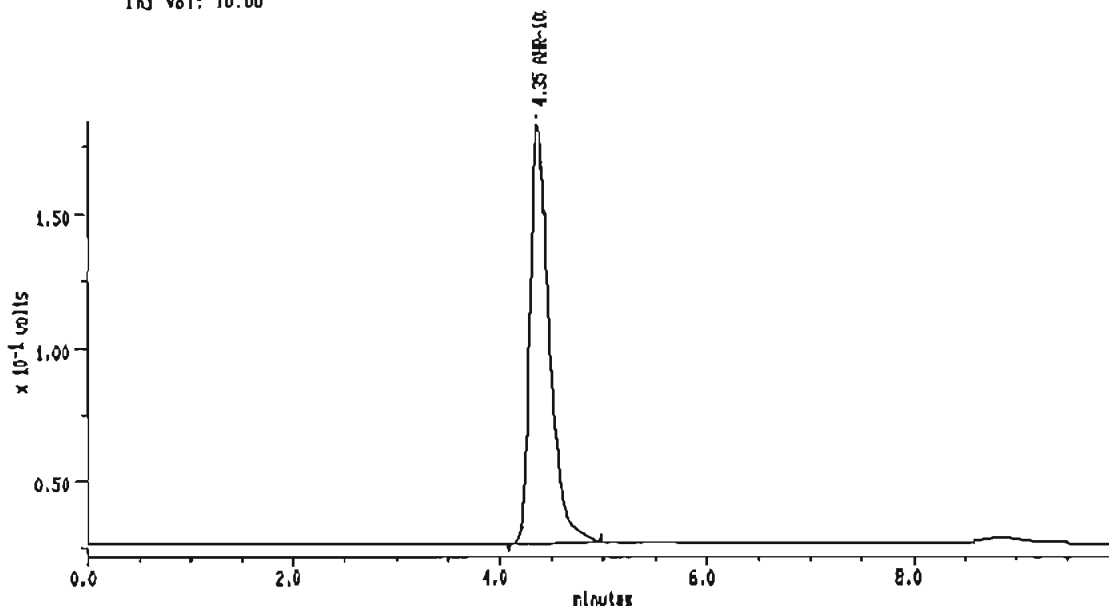
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	BB	2194535	184133	100.00	AHR-102828
TOTAL			2194535	184133		

Sample: A28 60°C-4# Channel: detector 1 Filename: V2-14 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:16 Method: 8:VAHRV1K13Y70-2# Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:16:03

SAMPLE: A28 60°C-4#

#14 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:16

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-14

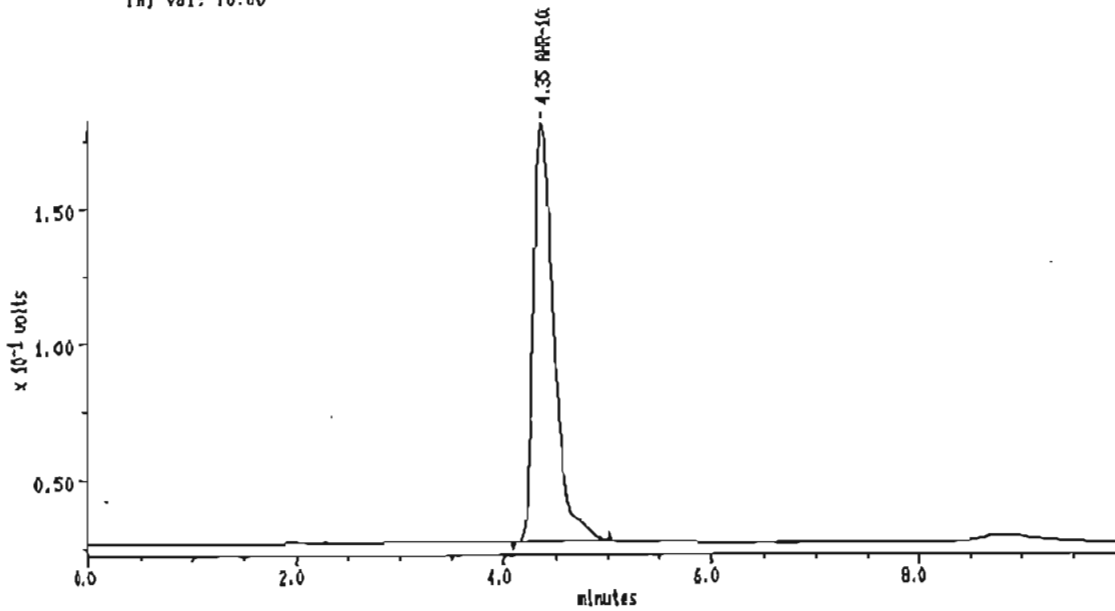
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.360	68	2111438	158232	100.00	AHR-10282B
TOTAL			2111438	158232		

Sample: A29 60°C-4W Channel: detector 1 Filename: V2-16 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:27 Method: B:VAHRV1K13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 826 カスタムレポート

Printed: 28-MAR-2001 12:15:34

SAMPLE: A29 60°C-4W

#15 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-16

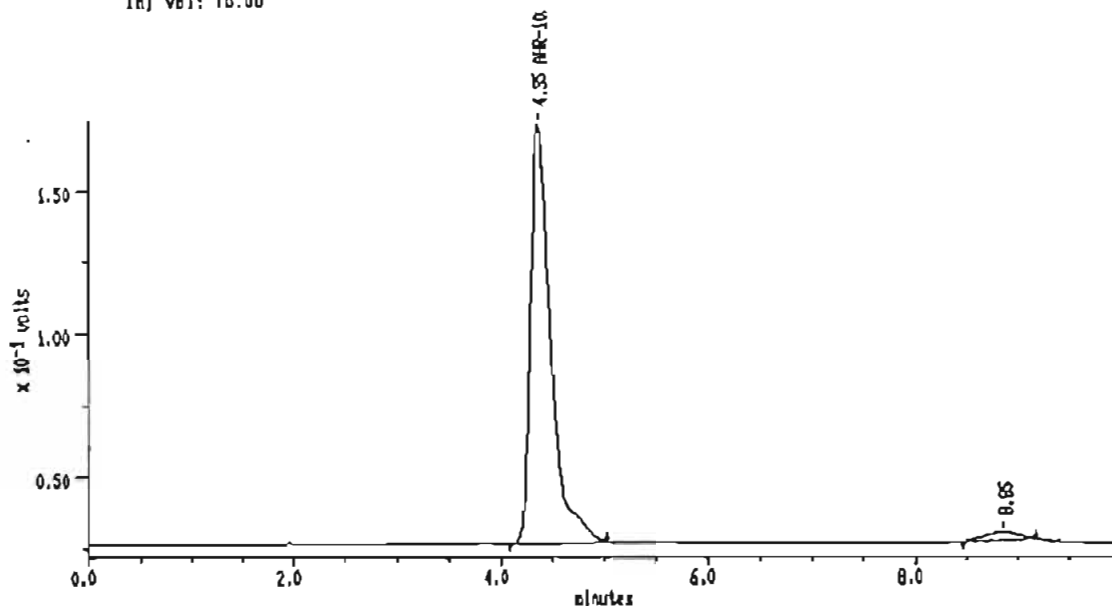
Index: 40

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	BB	2112402	164392	100.00	AHR-10282B
TOTAL			2112402	164392		

Sample: A20 60°C-4W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:38 Method: B:VAHRYIK13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

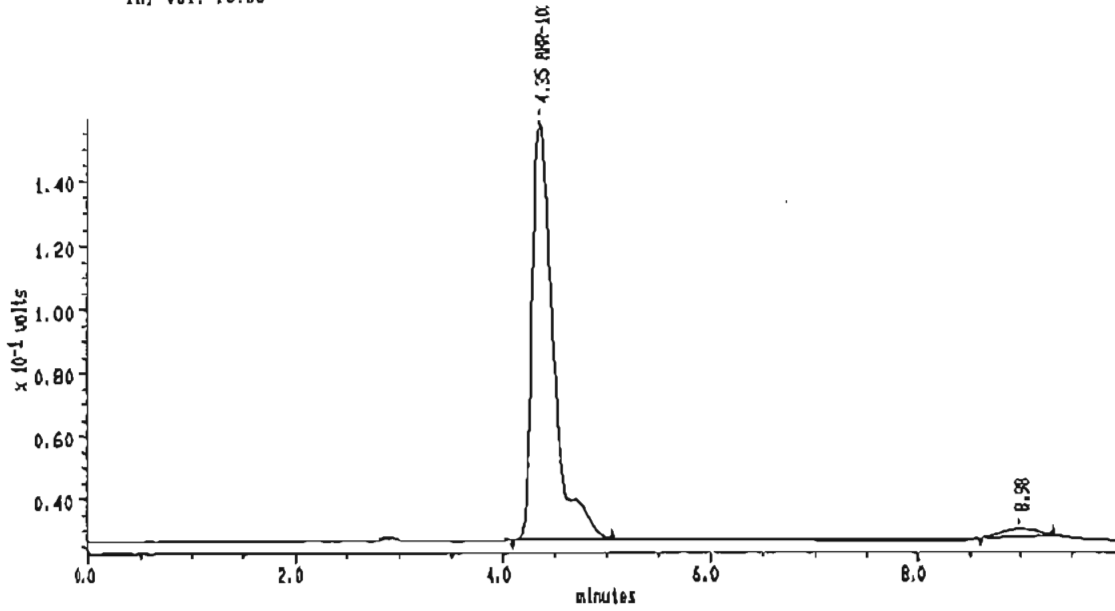
Printed: 28-MAR-2001 12:16:05

SAMPLE: A20 60°C-4W Type: UNKN
 F18 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 24-MAR-2001 0:38 Filename: V2-18
 Rate: 2.0 points/sec Index: 41
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.360	BB	2040435	148305	100.00	AHR-10282B
TOTAL			2040435	148305		

Sample: A31 80°C-4W Channel: detector 1 Filename: V2-17 Chart Speed: Full Size
 Acquired: 24-MAR-2001 0:49 Method: B:VAHRV\K13V7D-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:18:37

SAMPLE: A31 80°C-4W

#17 In Method: AHR-10282B

Acquired: 24-MAR-2001 0:49

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-17

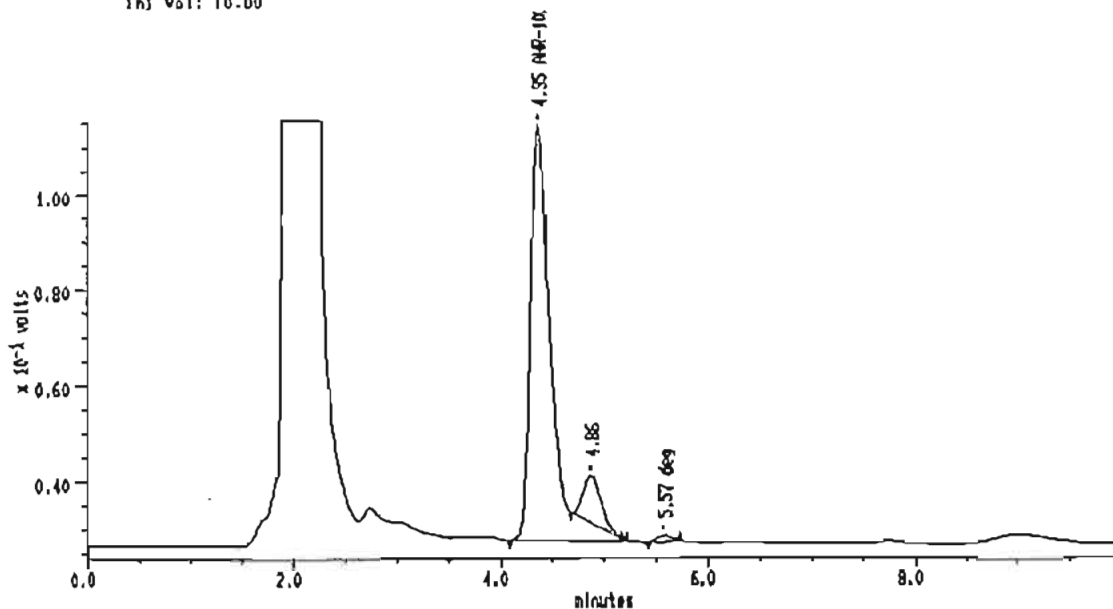
Index: 42

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	OB	1869891	131403	100.00	AHR-10282B
TOTAL			1869891	131403		

Sample: A32 60°C-4W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 24-MAR-10 1:01 Method: B:YAHRYIK13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:17:00

SAMPLE: A32 60°C-4W

#18 In Method: AHR-10282B

Acquired: 24-MAR-2001 1:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-18

Index: 43

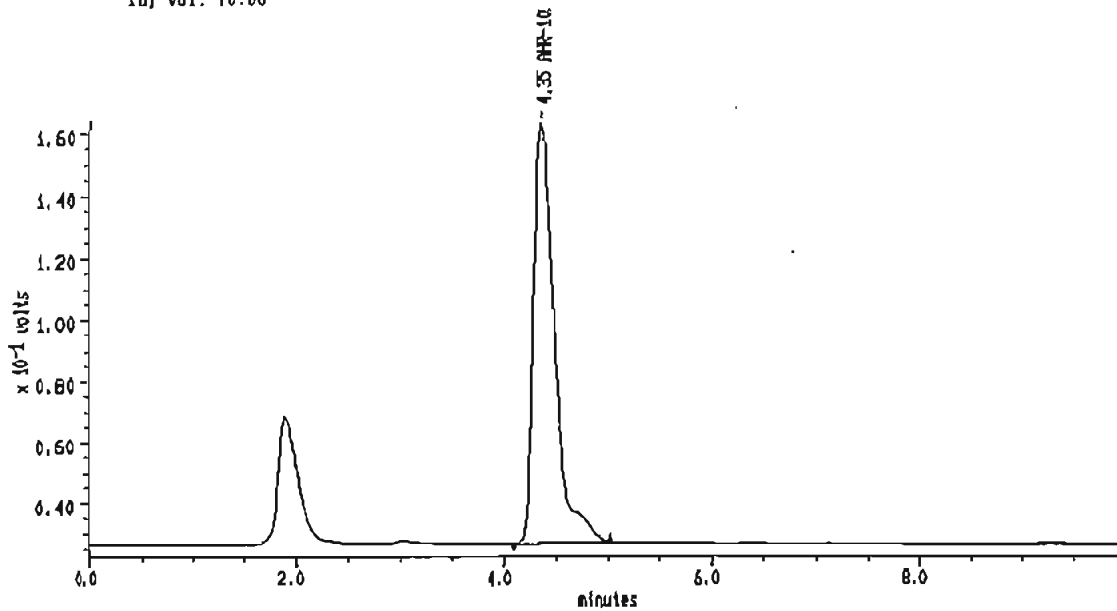
Injection Volume: 10.0

Amount: 0.000

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	BB	1243872	86988	88.98	AHR-10282B
3	5.587	BD	13118	1188	1.04	deg
TOTAL			1266990	88172		

Sample: A33 80°C-4W Channel: detector 1 Filenames: V2-19 Chart Speed: Full Size
 Acquired: 24-MAR-10 1:12 Method: B:YAHRYIKI3Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

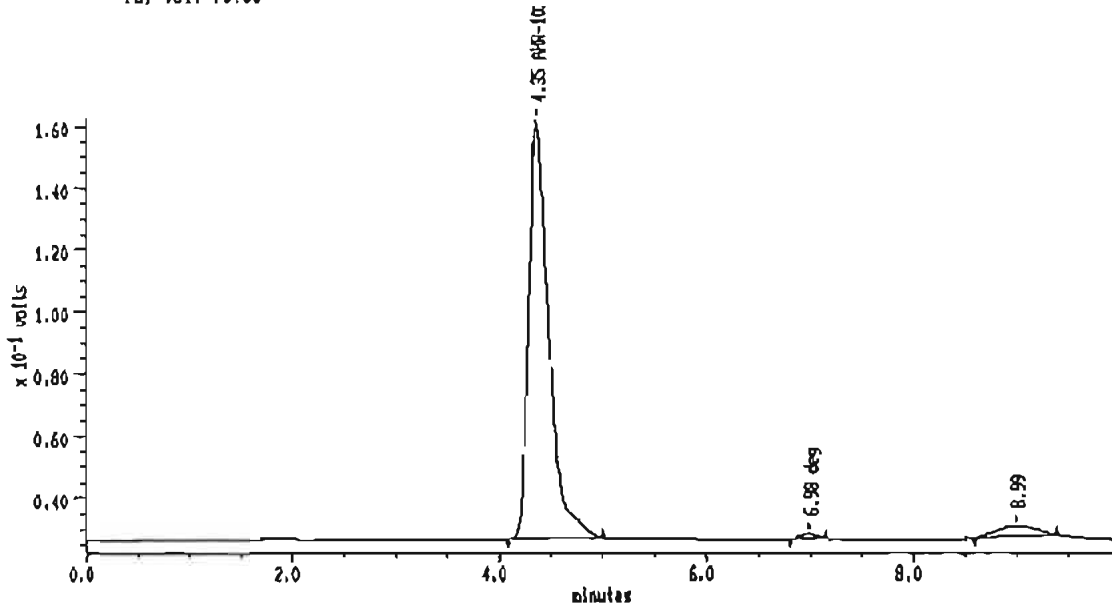
Printed: 28-MAR-2001 12:17:39

SAMPLE: A33 80°C-4W Type: UNKN
 =19 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 24-MAR-2001 1:12 Filenames: V2-19
 Rate: 2.0 points/sec Index: 44
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	SD	1805780	138520	100.00	AHR-10282B
TOTAL			1805780	138520		

Sample: A34 80°C-4W Channel: detector 1 Filename: V2-20 Chart Speed: Full Size
 Acquired: 24-MAR-101 1:23 Method: 8:VAIRV1K13V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

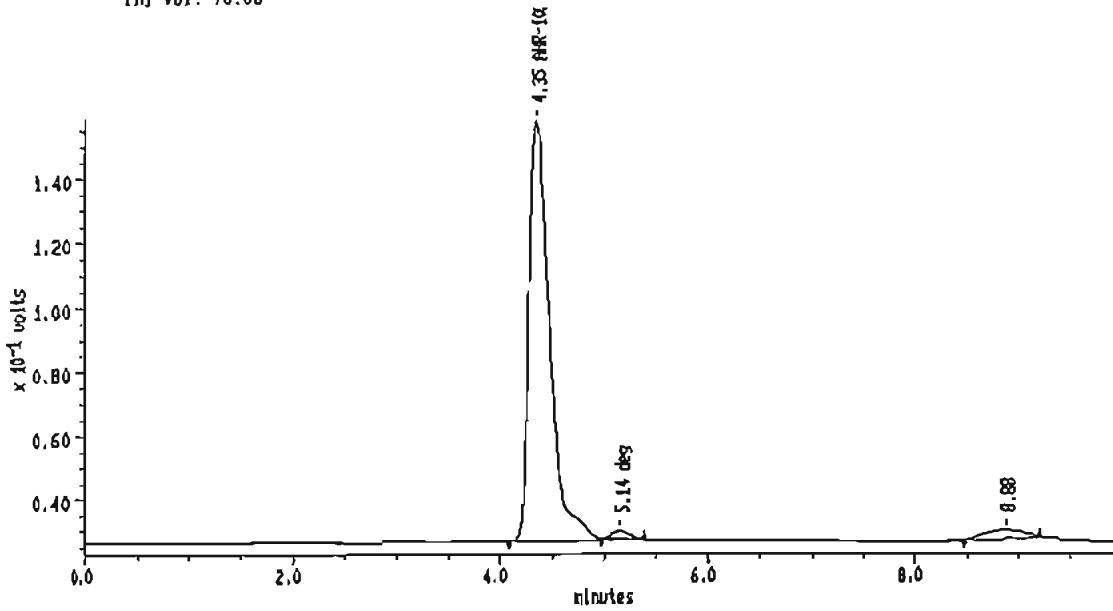
Printed: 28-MAR-2001 12:18:11

SAMPLE: A34 80°C-4W Type: LNKV
 #20 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 24-MAR-2001 1:23 Filename: V2-20
 Rate: 2.0 points/sec Index: 45
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.350	OB	1850867	134741	99.01	AHR-10282B
2	8.976	OB	18307	1386	0.99	deg
TOTAL			1869174	136136		

Sample: A35 60°C-4W Channel: detector 1 Filename: V2-21 Chart Speed: Full Size
 Acquired: 24-MAR-2001 1:35 Method: B:VAHRY1X13Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:18:43

SAMPLE: A35 60°C-4W

#21 In Method: AHR-10282B

Acquired: 24-MAR-2001 1:35

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-21

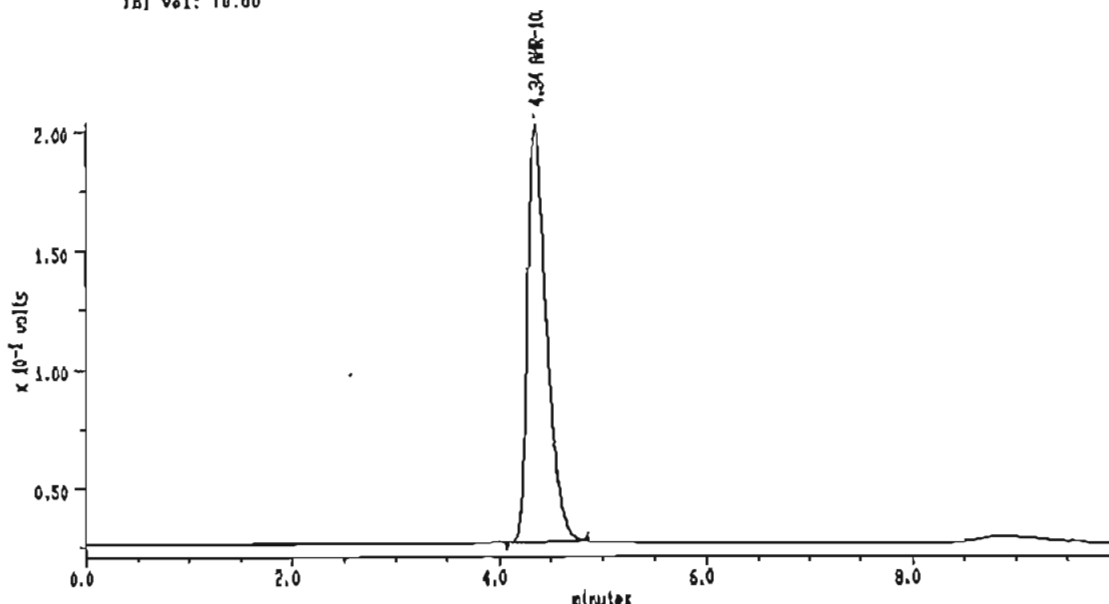
Inbox: 48

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.360	BB	1813088	130724	98.53	AHR-10282B
2	6.142	SS	27101	2288	1.47	deg
TOTAL			1840189	133010		

Sample: STD2 Channel: detector 1 Filename: V2-22 Chart Speed: Full Size
 Acquired: 24-MAR-2001 1:40 Method: B:YAJIRYIK13V70-2# Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 28-MAR-2001 12:19:14

SAMPLE: STD2

Method: AHR-10282B

Acquired: 24-MAR-2001 1:40

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-22

Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.342	BB	2319889	176137	100.00	AHR-10282B
TOTAL			2319889	176137		

プロナック点眼液の安定性試験
Lot No.01K131

試験コード：P2000B177
試験実施者：澤 嗣郎
試験実施日：2001年02月20日

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Water Collec(%)	Initial	present	
STD	1	INI-01	2286918						
STD	2	INI-32	2276153						
STD	mean		2281536	0.8950					
A-26	Initial	INI-02	2293965	1.0004	100				
A-27	Initial	INI-03	2262847	0.9868	100				
A-28	Initial	INI-04	2274923	0.9921	100				
A-29	Initial	INI-05	2369117	1.0332	100				
A-30	Initial	INI-06	2326638	1.0147	100				
A-31	Initial	INI-07	2328545	1.0155	100				
A-32	Initial	INI-08	2412438	1.0521	100				
A-33	Initial	INI-09	2298387	1.0023	100				
A-34	Initial	INI-10	2300355	1.0032	100				
A-35	Initial	INI-11	2317836	1.0108	100				
A-26	70°C-1W	INI-12	2132144	0.9298	92.94	89.49	3.71	8.3664	8.1916
A-27	70°C-1W	INI-13	2253964	0.9830	89.61	95.88	3.74	8.3824	8.2055
A-28	70°C-1W	INI-14	2243961	0.9786	98.64	95.01	3.68	8.4773	8.2997
A-29	70°C-1W	INI-15	2312544	1.0085	97.61	94.30	3.39	8.4742	8.3106
A-30	70°C-1W	INI-16	2258611	0.9850	97.07	93.45	3.73	8.5162	8.3347
A-31	70°C-1W	INI-17	2195956	0.9577	94.31	91.14	3.38	8.5820	8.4165
A-32	70°C-1W	INI-18	2197422	0.9583	91.08	87.70	3.71	8.4924	8.3126
A-33	70°C-1W	INI-19	2186825	0.9537	95.15	91.55	3.78	8.4307	8.2501
A-34	70°C-1W	INI-20	2165761	0.9445	94.15	90.90	3.45	8.4785	8.3119
A-35	70°C-1W	INI-21	2115392	0.9225	91.26	87.80	3.79	8.5490	8.3635
A-26	60°C-1W	INI-22	2117458	0.9234	92.30	90.67	1.77	8.3539	8.2705
A-27	60°C-1W	INI-23	2250178	0.9813	99.44	97.70	1.75	8.4678	8.3837
A-28	60°C-1W	INI-24	2211902	0.9646	97.23	95.47	1.81	8.4976	8.4099
A-29	60°C-1W	INI-25	2290389	0.9989	96.68	95.15	1.58	8.5264	8.4495
A-30	60°C-1W	INI-26	2227585	0.9715	95.74	94.19	1.62	8.4971	8.4186
A-31	60°C-1W	INI-27	2170690	0.9467	93.23	91.58	1.77	8.5363	8.4500
A-32	60°C-1W	INI-28	2243853	0.9786	93.01	91.35	1.79	8.4164	8.3310
A-33	60°C-1W	INI-29	2193124	0.9564	95.42	93.75	1.75	8.4560	8.3718
A-34	60°C-1W	INI-30	2153242	0.9390	93.60	92.08	1.62	8.4995	8.4210
A-35	60°C-1W	INI-31	2116180	0.9229	91.30	89.65	1.81	8.6061	8.5163

計算に必要なデータは記載済み再編集して。
2005.06 澤 嗣郎

試験物質名: AHR10282B	試験コード: P2000B177	試験年月日: 2000年02月20日
試験項目:		試験実施者: 深 岡郎

STD 0.01990g AHR10282B 20ml x 2 = 40ml MP . 20-Feb-2001 . 14:30:25

001: + 0.01990g

異物(例)

A24 initial	7.05			±	-	深黄
A27	7.10			-	-	
A28	7.10			-	-	
A29	7.08			-	-	
A30	7.09			-	-	
A31	7.06			-	-	
A32	7.05			-	-	
A33	7.08			-	-	
A34	7.07			-	-	
A35	7.12			-	-	↓
A26 70C- 27 ^{1W}	7.02	8.3664	8.1916	±	+	深黄
A27	7.05	8.3824	8.2055	+G)	-	黄
A28	7.03	8.4773	8.2997	+	+	黄
A29	7.03	8.4742	8.3106	±	+	黄
A30	7.02	8.5162	8.3347	±	+	黄
A31	7.01	8.5820	8.4165	±	+	黄
A32	7.04	8.4924	8.3126	+G)	±	深黄
A33	7.10	8.4307	8.2501	±	+	黄
A34	7.10	8.4785	8.3119	±	+	黄
A35	7.10	8.5490	8.3625	±	+	黄
A26 60C- 27 ^{1W}	7.02	8.3539	8.2705	±	+	深黄
A27	7.08	8.4678	8.3837	+	±	黄
A28	7.04	8.4976	8.4099	±	±	黄
A29	7.09	8.5264	8.4495	-	±	黄
A30	7.08	8.4971	8.4186	-	+	黄
A31	7.05	8.5363	8.4500	±	+	黄
A32	7.05	8.4164	8.3310	+G)	±	深黄
A33	7.06	8.4560	8.3718	±	+	黄
A34	7.06	8.4995	8.4210	±	+	黄
A35	7.10	8.6061	8.5163	±	+	黄

1/10/02
2002/12

2/20 16:27
NO.21 PH 7.05
24.1°C

2/20 16:28
NO.22 PH 7.10
24.3°C

2/20 16:30
NO.23 PH 7.10
24.3°C

2/20 16:32
NO.24 PH 7.08
24.4°C

2/20 16:33
NO.25 PH 7.09
24.5°C

2/20 16:34
NO.26 PH 7.06
24.3°C

2/20 16:35
NO.27 PH 7.05
24.3°C

2/20 16:36
NO.28 PH 7.06
24.4°C

2/20 16:36
NO.29 PH 7.07
24.5°C

2/20 16:38
NO.30 PH 7.12
24.4°C

2/20 16:39
NO.31 PH 7.02
24.5°C

2/20 16:40
NO.32 PH 7.05
24.5°C

2/20 16:41
NO.33 PH 7.03
24.6°C

2/20 16:42
NO.34 PH 7.03
24.5°C

2/20 16:43
NO.35 PH 7.02
24.6°C

2/20 16:44
NO.36 PH 7.01
24.6°C

2/20 16:45
NO.37 PH 7.04
24.5°C

2/20 16:46
NO.38 PH 7.10
24.6°C

2/20 16:48
NO.39 PH 7.10
24.5°C

2/20 16:49
NO.40 PH 7.10
24.4°C

2/20 16:50
NO.41 PH 7.02
24.6°C

2/20 16:51
NO.42 PH 7.03
24.6°C

2/20 16:52
NO.43 PH 7.04
24.6°C

2/20 16:53
NO.44 PH 7.09
24.6°C

2/20 16:53
NO.45 PH 7.08
24.5°C

2/20 16:54
NO.46 PH 7.05
24.7°C

2/20 16:55
NO.47 PH 7.05
24.6°C

2/20 16:56
NO.48 PH 7.06
24.6°C

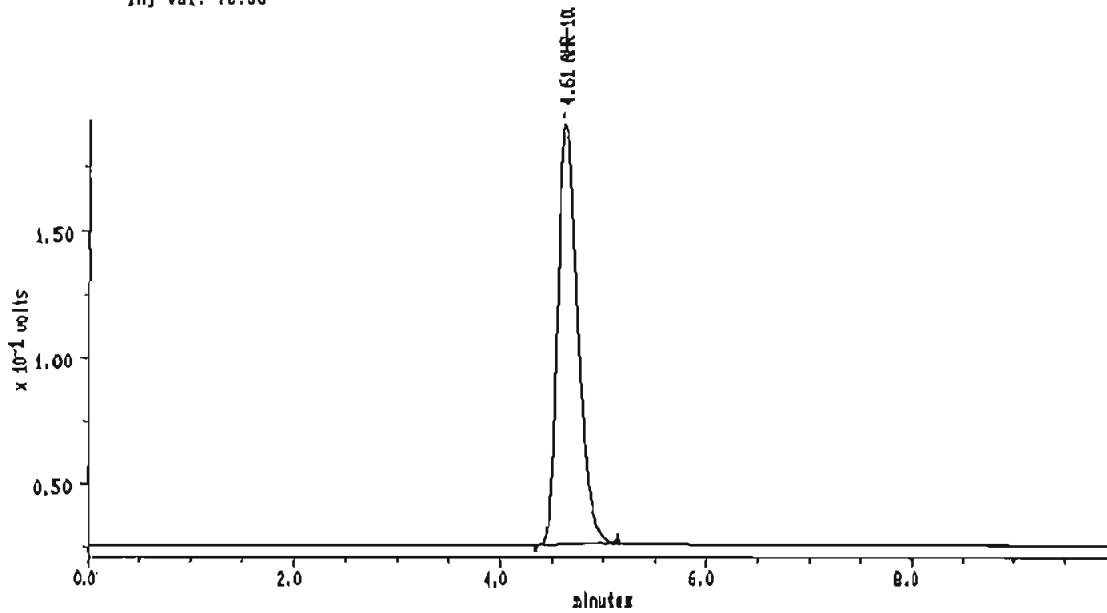
2/20 16:56
NO.49 PH 7.06
24.4°C

2/20 16:57
NO.50 PH 7.10
24.5°C

NO. 5
S 231
NO. 43
13 1:27
NO. 14 71
13 1:30
NO. 5 25
13 1:31
NO. 11 22.7
13 1:32
NO. 11 21
13 1:33
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13 1:58
NO. 11 21
13 1:59
NO. 11 21
13 2:00
NO. 11 21

試験物質名					試験コード					ロット番号					調製年月日	20	年	月	日	試験実施者	澤 嗣郎	
試験題目																						
製造量	無色77 [※] 15mL×	本	無色PP5mL×	本	褐色PP5mL×	本	×	本	×	本	×	本	×	本								
処方番号	31				32				33				34				35					
成分及び分量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	製造メーカー					
成分名	mL	(g)	mL	(g)	mL	(g)	mL	(g)	mL	(g)	mL	(g)	mL	(g)	mL	(g)	ロット番号					
ブドウ糖	0.1	0.102	0.1	0.102	0.1	0.100	0.1	0.100	0.1	0.101												
ホウ酸	1.6	1.596	1.6	1.610	1.6	1.606	1.6	1.596	1.6	1.598												
塩化ベンザルコニウム	0.005		0.005		0.005		0.005		0.005													
4-ヒドロキシベンゾ酸	0.15	0.14984	0.15	0.14980	0.15	0.15152	0.15	0.1498	0.15	0.14961												
α-ヒドロキシカルボン酸	1.0	0.999																				
ソルビトール			0.3	0.302																		
グリセリン					0.1	0.100																
イソプロピルアルコール							0.5	0.499														
アセチルサリチル酸									0.5	0.502												

Sample: STD1 Channel: detector 1 Filename: IN1-01 Chart Speed: Full Size
 Acquired: 20-FEB-2001 18:27 Method: B:VAHRV1K13V1R1TIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 18:37:32

SAMPLE: STD1

#4 in Method: AHR-10282B

Acquired: 20-FEB-2001 18:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-01

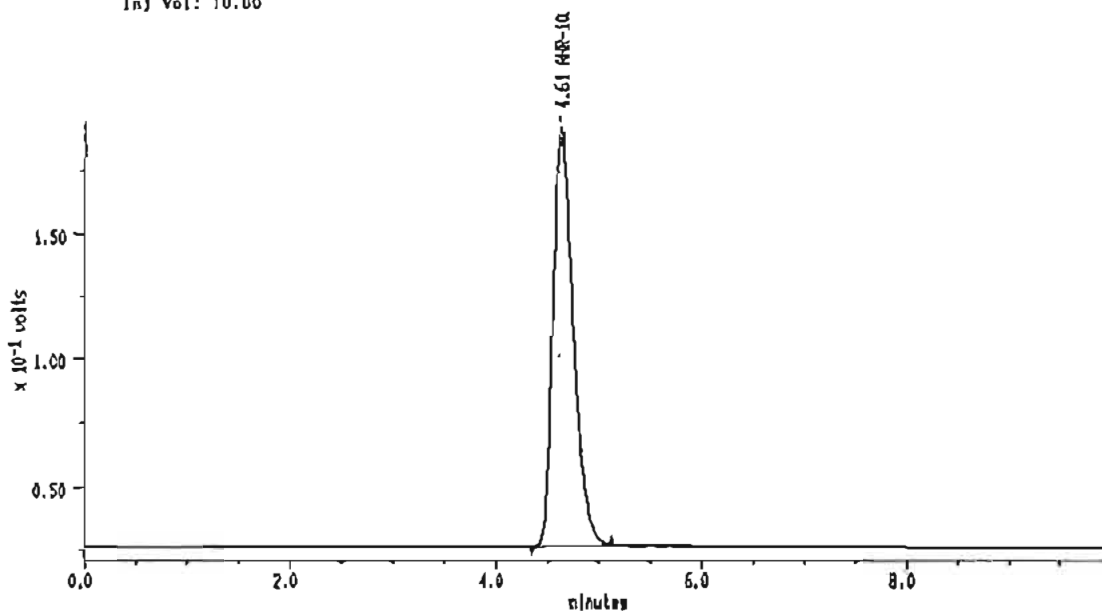
Index: 1

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.608	88	2288918	166263	100.00	AHR-10282B
TOTAL			2288918	166263		

Sample: A28 INITIAL Channel: detector 1 Filename: INI-02 Chart Speed: Full Size
 Acquired: 20-FEB-2001 18:38 Method: S-YAHRV1K13VINI1IAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 18:48:48

SAMPLE: A28 INITIAL

#5 In Method: AHR-10282B

Acquired: 20-FEB-2001 18:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-02

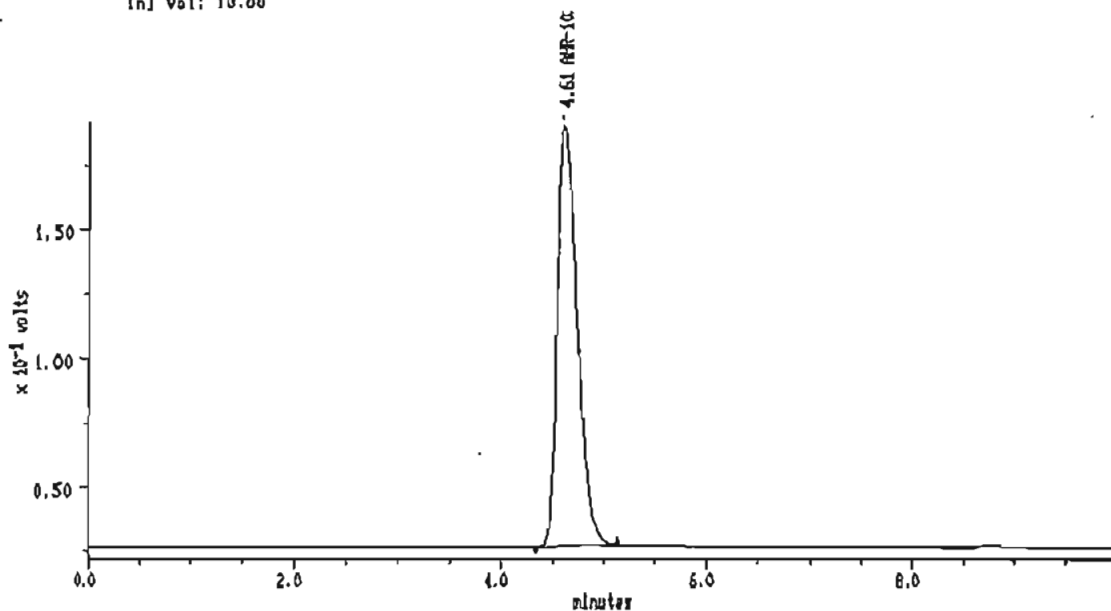
Index: 2

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.809	BB	2293985	185920	100.00	AHR-10282B
TOTAL			2293985	185920		

Sample: A27 INITIAL Channel: detector 1 Filename: INI-03 Chart Speed: Full Size
 Acquired: 20-FEB-2001 18:49 Method: B:YAHRYIK13V\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 19:00:03

SAMPLE: A27 INITIAL

#8 In Method: AHR-10282B

Acquired: 20-FEB-2001 18:49

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-03

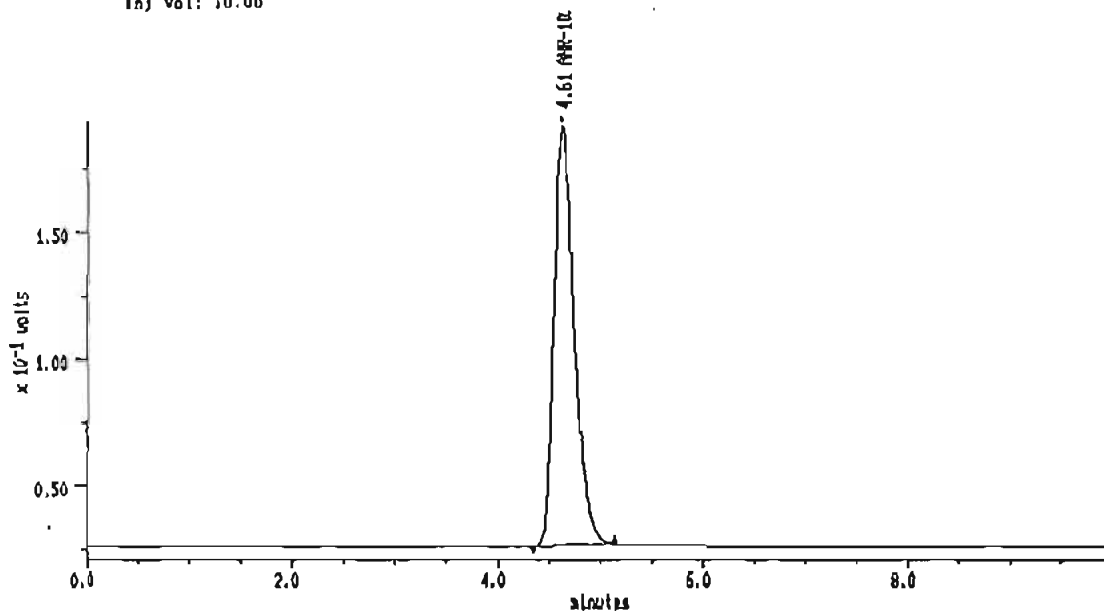
Index: 3

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.808	DB	2282847	183883	100.00	AHR-10282B
TOTAL			2282847	183883		

Sample: A28 INITIAL Channel: detector 1 Filename: INI-04 Chart Speed: Full Size
 Acquired: 20-FEB-2001 19:00 Method: B:VAHRVIR)3V(INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 19:11:20

SAMPLE: A28 INITIAL

#7 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:00

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-04

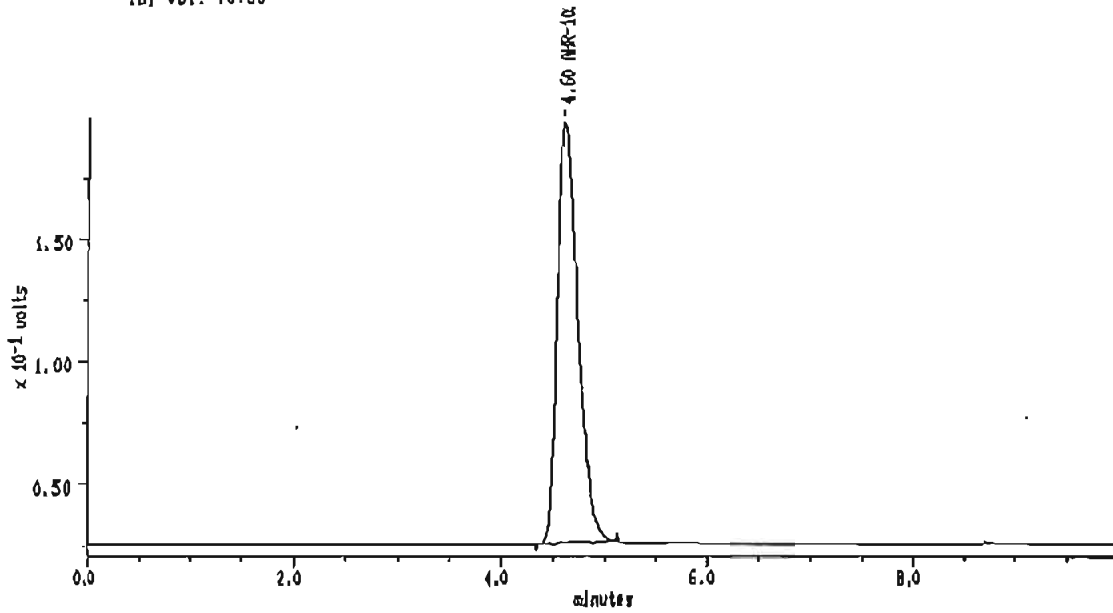
Index: 4

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.608	OB	2274923	164712	100.00	AHR-10282B
TOTAL			2274923	164712		

Sample: A29 INITIAL Channel: detector 1 Filename: INI-06 Chart Speed: Full Size
 Acquired: 20-FEB-2001 19:12 Method: B:YAHRYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 19:22:37

SAMPLE: A29 INITIAL

#8 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-06

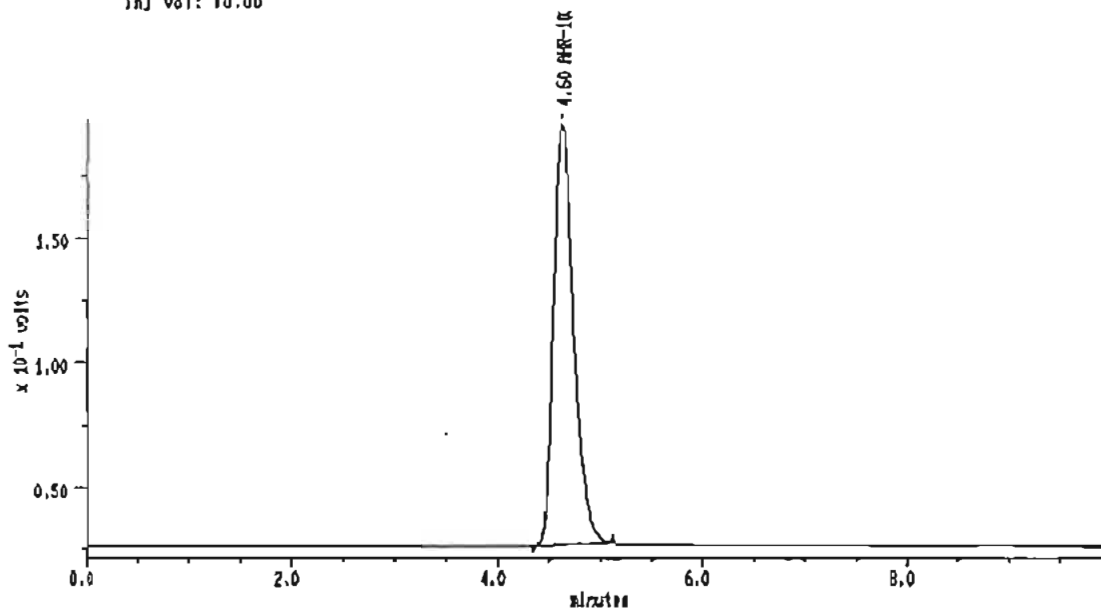
Index: 6

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	DB	2309117	171584	100.00	AHR-10282B
TOTAL			2309117	171584		

Sample: A30 INITIAL Channel: detector 1 Filename: IN1-08 Chart Speed: Full Size
 Acquired: 20-FEB-101 19:23 Method: 0:VAIRYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 19:33:54

SAMPLE: A30 INITIAL

#9 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-08

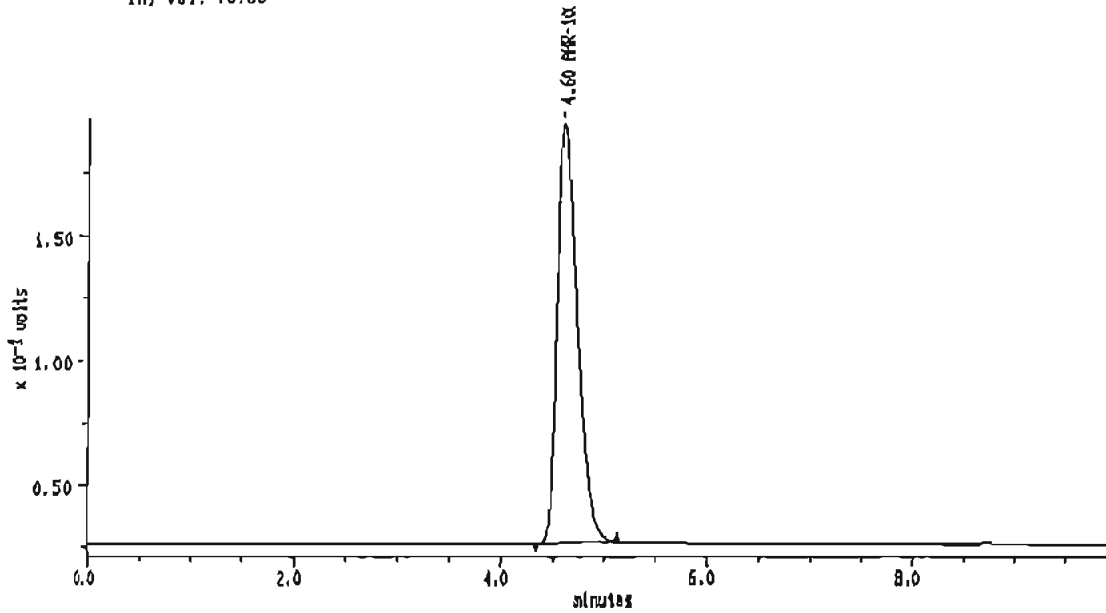
Index: 0

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2326838	168594	100.00	AHR-10282B
TOTAL			2326838	168594		

Sample: A31 INITIAL Channel: detector 1 Filename: INI-07 Chart Speed: Full Size
 Acquired: 20-FEB-2001 19:34 Method: B:VAHRVIKICV\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 18:45:11

SAMPLE: A31 INITIAL

#10 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:34

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-07

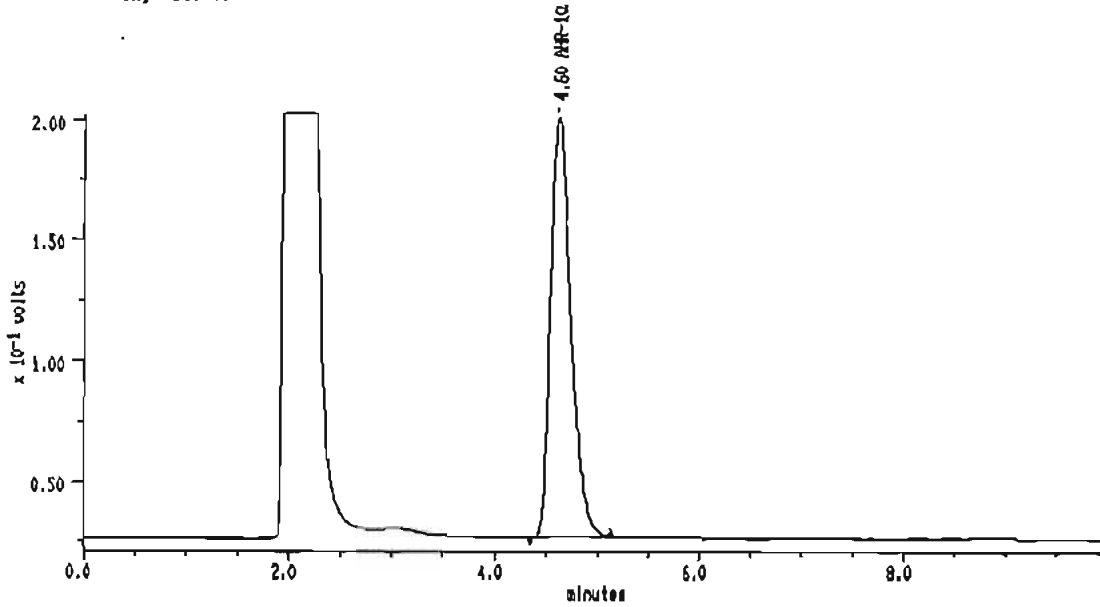
Index: 7

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	80	2328545	180265	100.00	AHR-10282B
TOTAL			2328545	180265		

Sample: A32 INITIAL Channel: detector 1 Filename: IMI-08 Chart Speed: Full Size
 Acquired: 20-FEB-10 19:45 Method: 0:YAHRYIK13VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 18:58:28

SAMPLE: A32 INITIAL

#11 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:45

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IMI-08

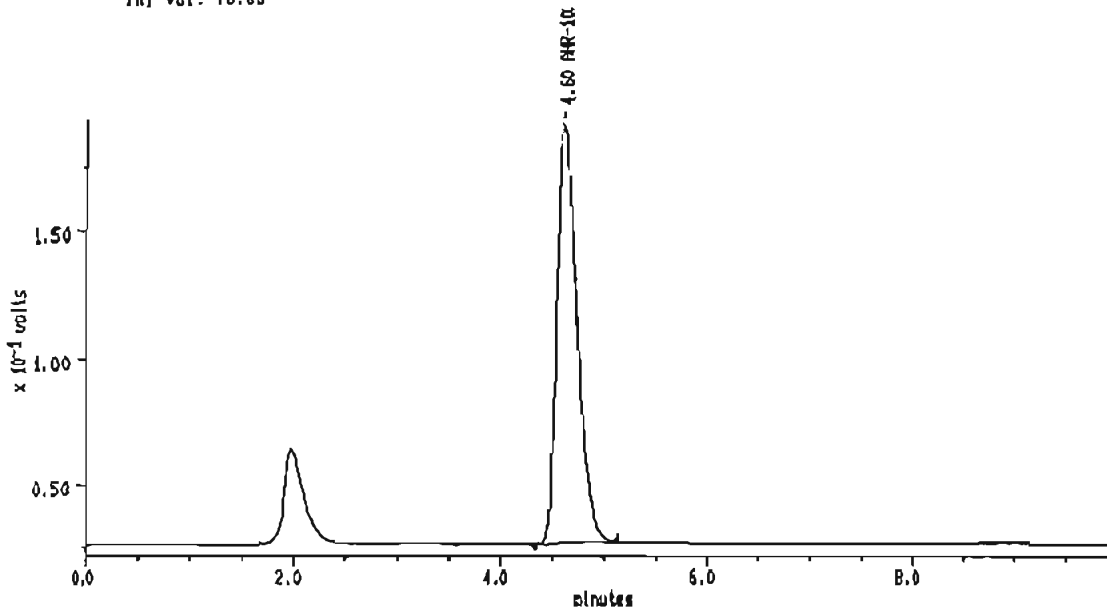
Index: 8

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	SD	2412438	174182	100.00	AHR-10282B
TOTAL			2412438	174182		

Sample: A33 INITIAL Channel: detector 1 Filename: IN1-09 Chart Speed: Full Size
 Acquired: 20-FEB-10 19:57 Method: 8:VAHRYIKI3YINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 20:07:45

SAMPLE: A33 INITIAL

012 In Method: AHR-10282B

Acquired: 20-FEB-2001 19:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-09

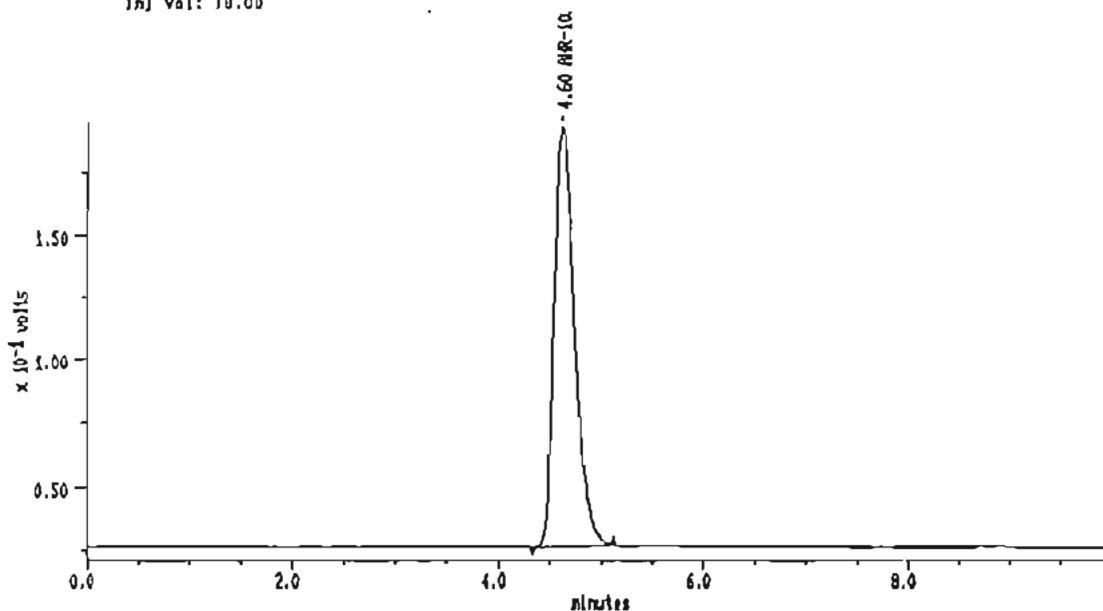
Index: 9

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	SD	2298387	168127	100.00	AHR-10282B
TOTAL			2298387	168127		

Sample: A34 INITIAL Channel: detector 1 Filename: IN1-10 Chart Speed: Full Size
 Acquired: 20-FEB-101 20:08 Method: B:YAHRYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 20:19:02

SAMPLE: A34 INITIAL

#13 In Method: AHR-10282B

Acquired: 20-FEB-2001 20:08

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-10

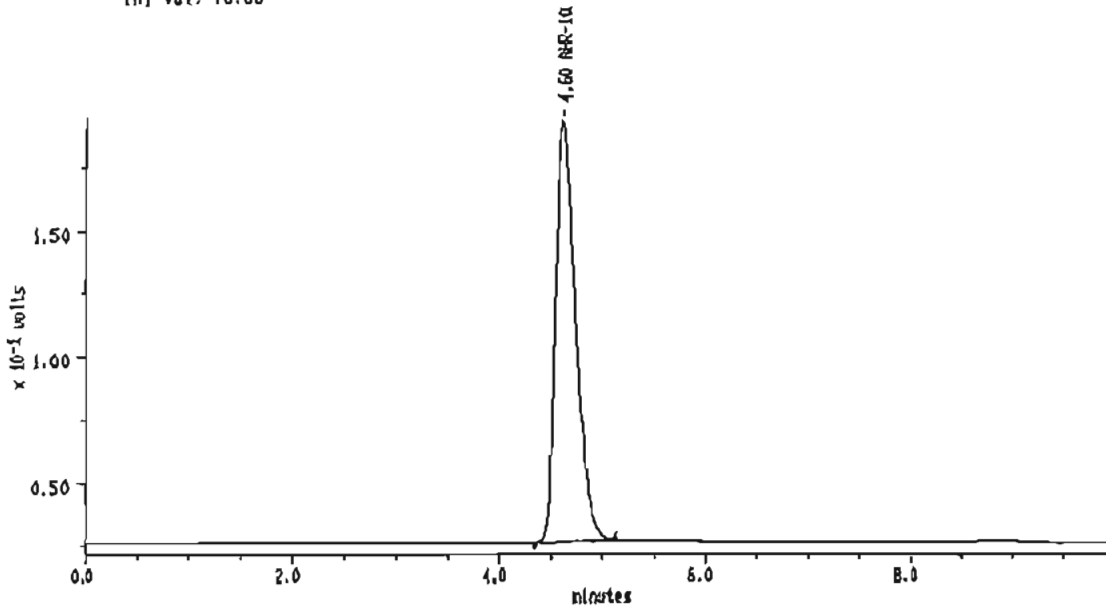
Index: 10

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2300365	180136	100.00	AHR-10282B
TOTAL			2300365	180136		

Sample: A35 INITIAL Channel: detector 1 Filename: INI-11 Chart Speed: Full Size
 Acquired: 20-FEB-2001 20:19 Method: B:VAHRVIXI13YIINITIAL Operator: S.S
 [n] Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 20:30:19

SAMPLE: A35 INITIAL

#14 in Method: AHR-10282B

Acquired: 20-FEB-2001 20:19

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-11

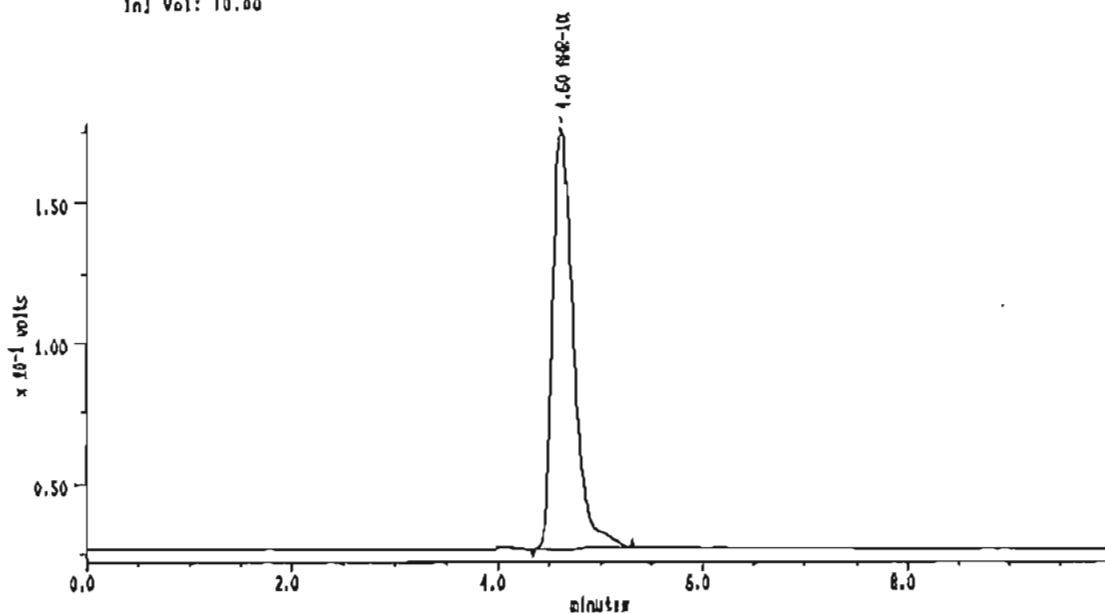
Index: 11

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2317838	187378	100.00	AHR-10282B
TOTAL			2317838	187378		

Sample: A28 70°C-1W Channel: detector 1 Filename: INI-12 Chart Speed: Full Size
 Acquired: 20-FEB-2001 20:31 Method: G:VAHRVXK13VINI1AL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 20:41:38

SAMPLE: A28 70°C-1W

#15 In Method: AHR-10282B

Acquired: 20-FEB-2001 20:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-12

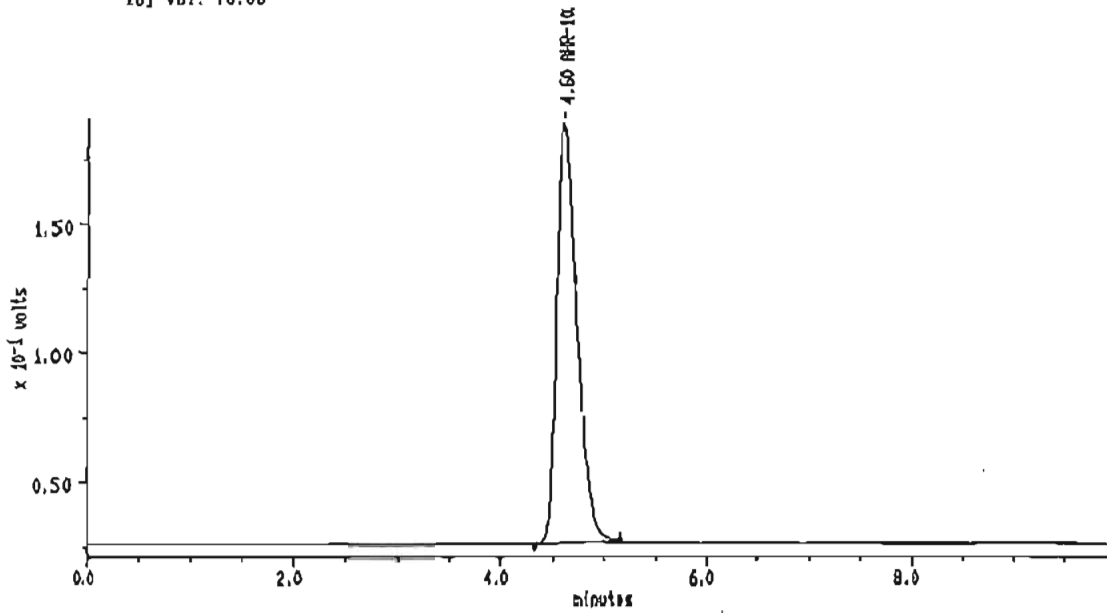
Index: 12

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2132144	149609	100.00	AHR-102828
TOTAL			2132144	149609		

Sample: A27 70°C-1W Channel: detector 1 Filename: IN1-13 Chart Speed: Full Size
 Acquired: 20-FEB-2001 20:42 Method: 8:YAMRVIKIJYINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 20:52:53

SAMPLE: A27 70°C-1W

#18 in Method: AHR-10282B

Acquired: 20-FEB-2001 20:42

Rate: 2.0 points/sec

Duration: 30.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-13

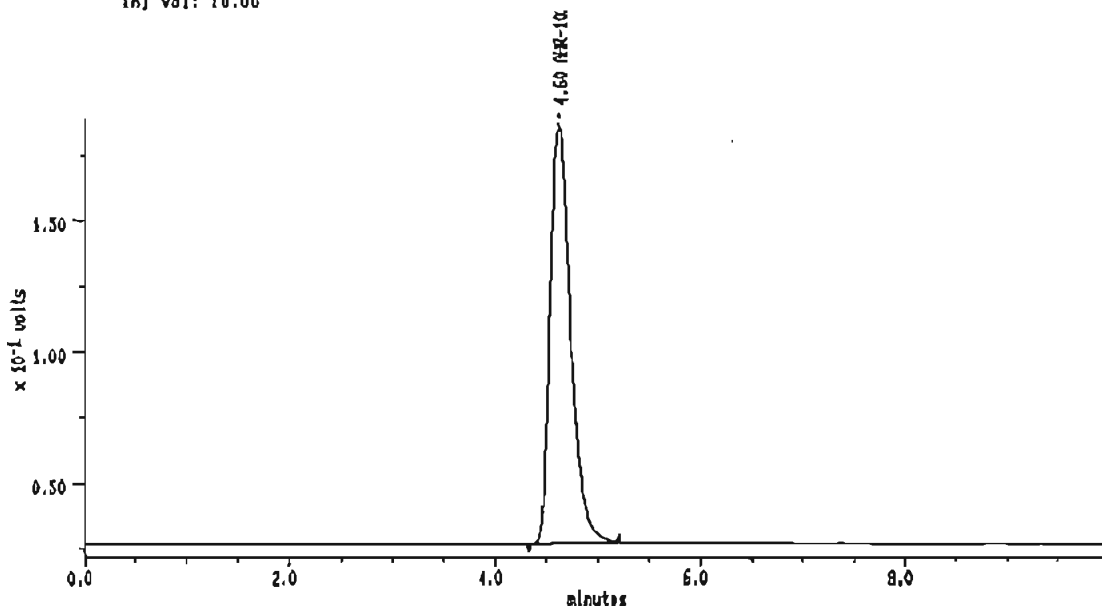
Index: 13

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2263984	182371	100.00	AHR-10282B
TOTAL			2263984	182371		

Sample: A28 70°C-1W Channel: detector 1 Pilename: INI-14 Chart Speed: Full Size
 Acquired: 20-FEB-01 20:53 Method: B:YAHRYIKIJOYINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 21:04:11

SAMPLE: A28 70°C-1W

Method: AHR-10282B

Acquired: 20-FEB-2001 20:53

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: DNKN

Instrument: Instrument 1

Pilename: INI-14

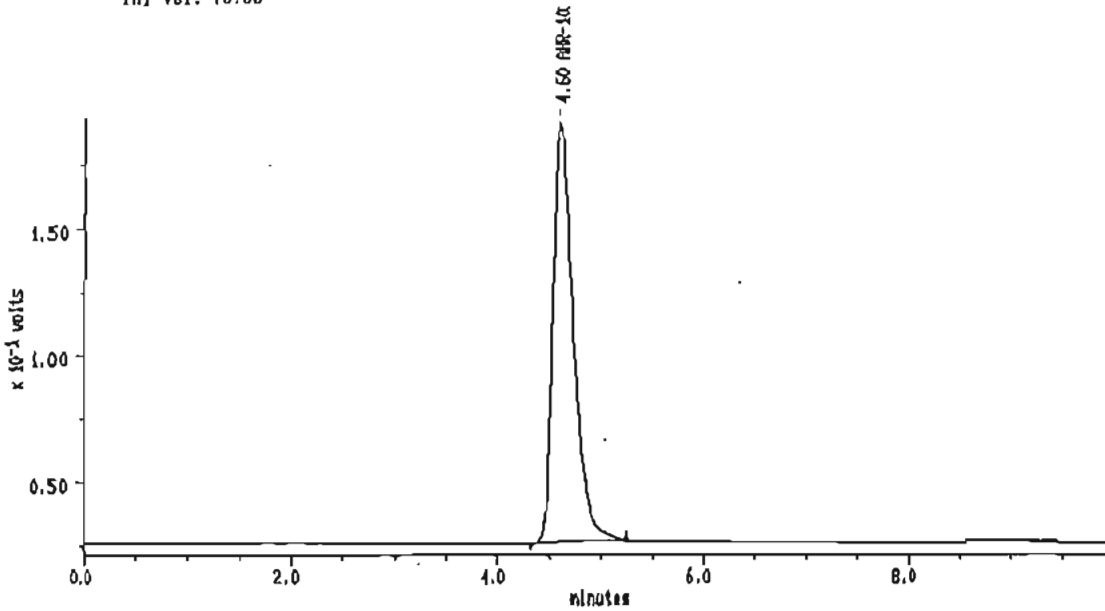
Index: 14

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BD	2243981	180857	100.00	AHR-10282B
TOTAL			2243981	180857		

Sampler: A29 70°C-1W Channel: detector 1 Pilenname: INI-16 Chart Speed: Full Size
 Acquired: 20-FEB-01 21:04 Method: 0:YAJIRYIKISYINIJIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 21:16:28

SAMPLE: A29 70°C-1W

#18 In Method: AHR-10282B

Acquired: 20-FEB-2001 21:04

Rate: 2.0 points/sec

Duration: 10.008 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pilenname: INI-16

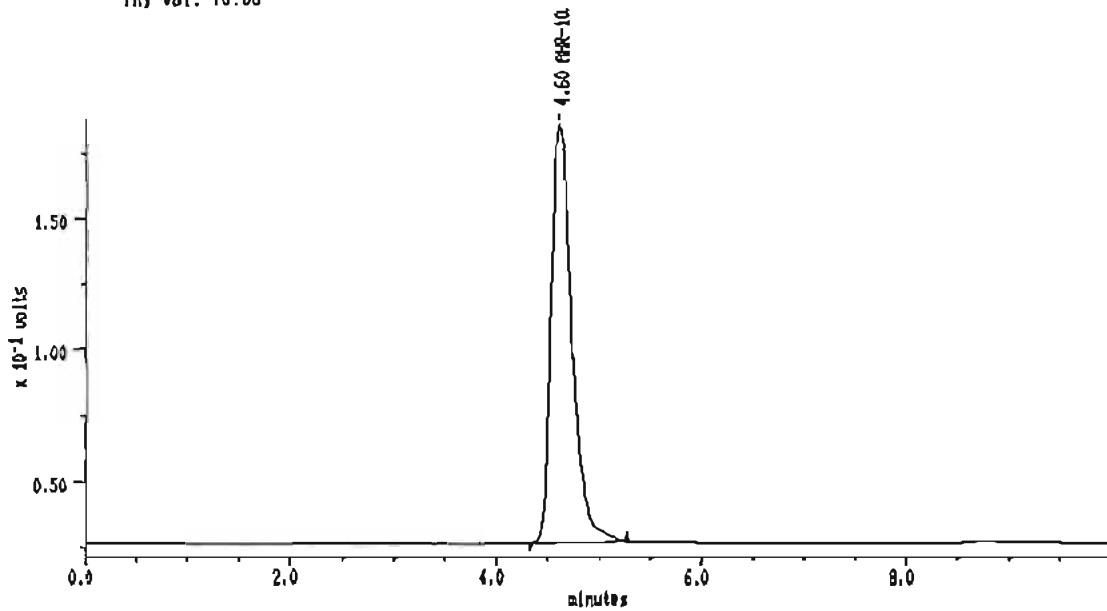
Index: 15

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	88	2312644	164798	100.00	AHR-10282B
TOTAL			2312644	164798		

Sample: A30 70°C-1W Channel: detector 1 Pileonno: INI-18 Chart Speed: Full Size
 Acquired: 20-FEB-01 21:18 Method: B:YAJIRVIX13VINIHAL Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 21:28:42

SAMPLE: A30 70°C-1W

#19 in Method: AHR-10282B

Acquired: 20-FEB-2001 21:18

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pileonno: INI-18

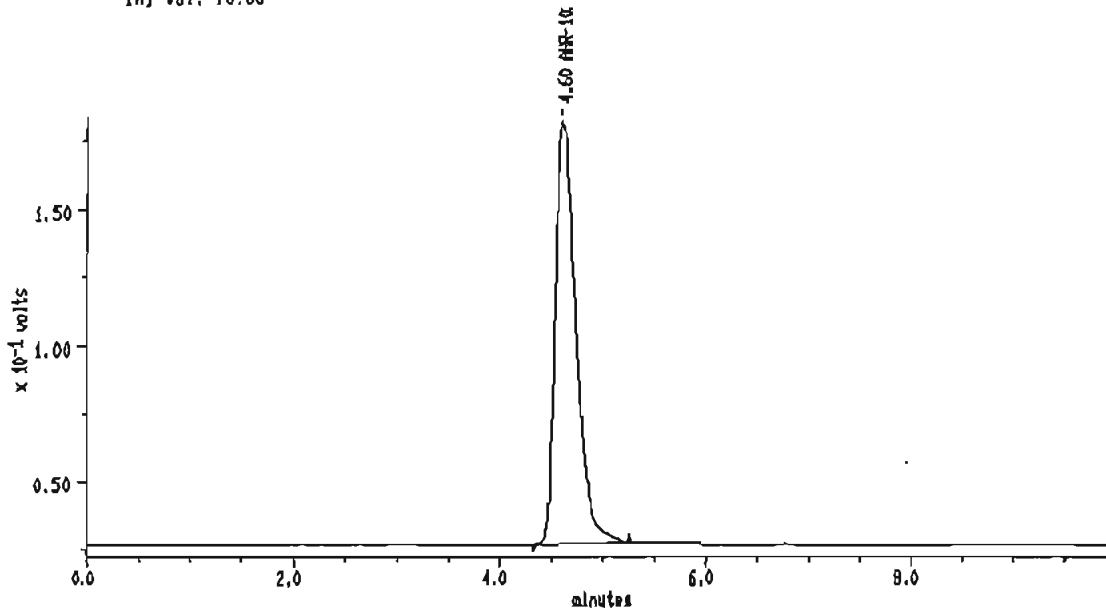
Index: 18

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	00	2268811	180085	100.00	AHR-10282B
TOTAL			2268811	180085		

Sample: A31 70°C-1W Channel: detector 1 Filename: INI-17 Chart Speed: Full Size
 Acquired: 20-FEB-2001 21:27 Method: B:YAHRYIKI3VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 21:38:00

SAMPLE: A31 70°C-1W

#20 In Method: AHR-10282B

Acquired: 20-FEB-2001 21:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-17

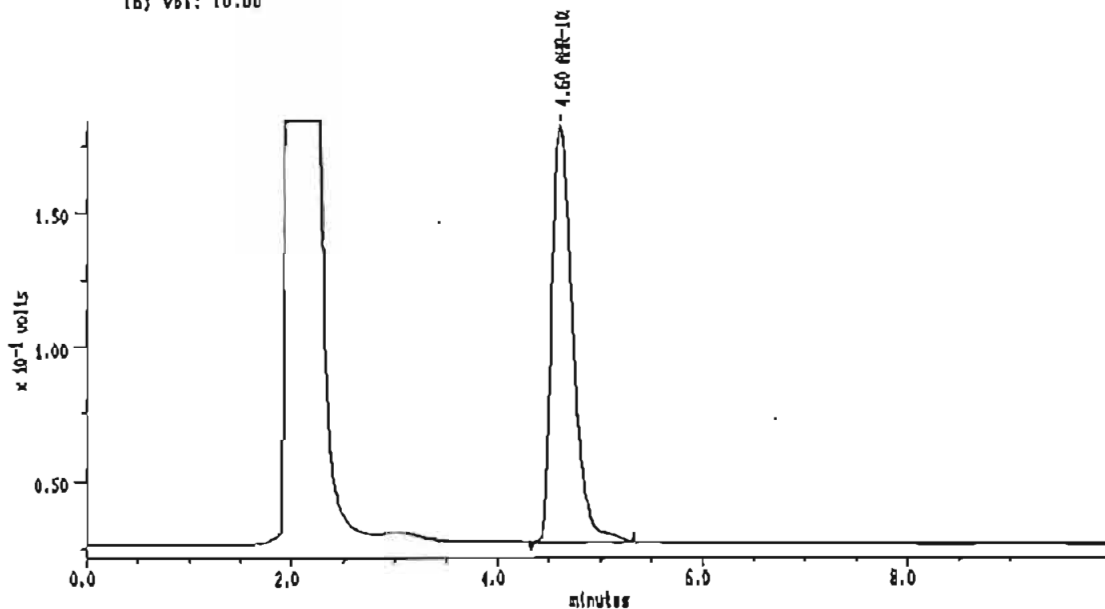
Index: 17

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2195958	166705	100.00	AHR-10282B
TOTAL			2195958	166705		

Sample: A32 70°C-1W Channel: detector 1 Filename: INI-18 Chart Speed: Full Size
 Acquired: 20-FEB-10 21:38 Method: B:VAHRVIXI3VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 21:49:17

SAMPLE: A32 70°C-1W

#21 In Method: AHR-10282B

Acquired: 20-FEB-2001 21:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-18

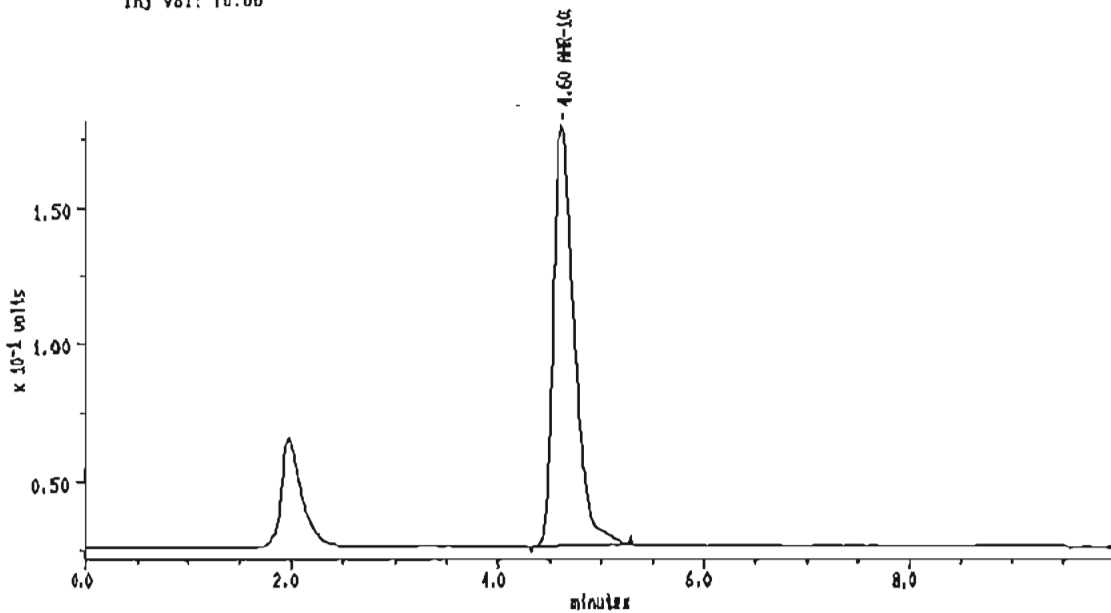
Index: 18

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.800	BB	2197422	156218	100.00	AHR-10282B
TOTAL			2197422	156218		

Sample: A33 70°C-1W Channel: detector 1 Filename: INI-19 Chart Speed: Full Size
 Acquired: 20-FEB-2001 21:50 Method: B:VAURVIXI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 22:00:33

SAMPLE: A33 70°C-1W

#22 In Method: AHR-10282B

Acquired: 20-FEB-2001 21:50

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-19

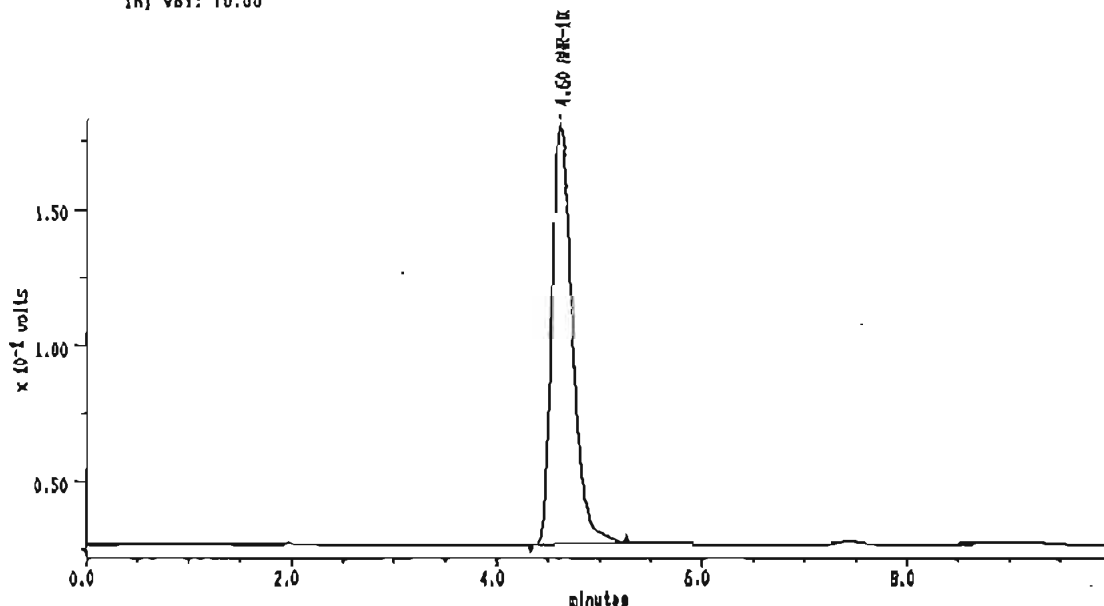
Index: 19

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.800	80	2188925	153754	100.00	AHR-10282B
TOTAL			2188925	153754		

Sample: A34 10°C-1W Channel: detector 1 Filename: INI-20 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:01 Method: B:YAHHRVIX13YINIZIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 22:11:51

SAMPLE: A34 10°C-1W

#20 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-20

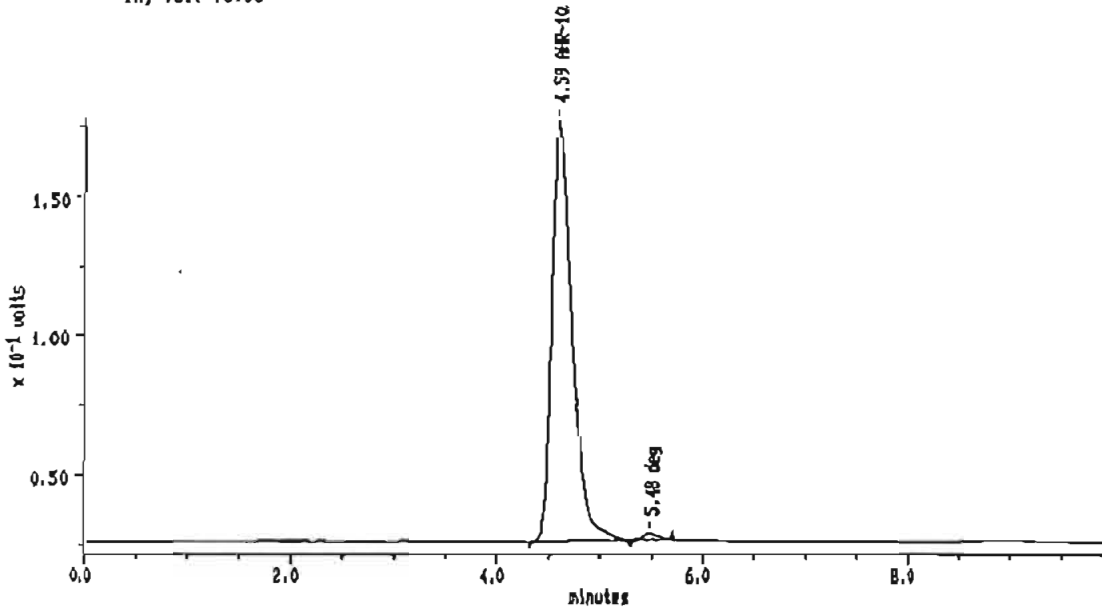
Index: 20

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.600	BB	2185781	154873	100.00	AHR-10282B
TOTAL			2185781	154873		

Sample: A35 70°C-1W Channel: detector 1 Filename: INI-21 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:12 Method: B:WAHRYIKI3VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 22:23:07

SAMPLE: A35 70°C-1W

#24 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-21

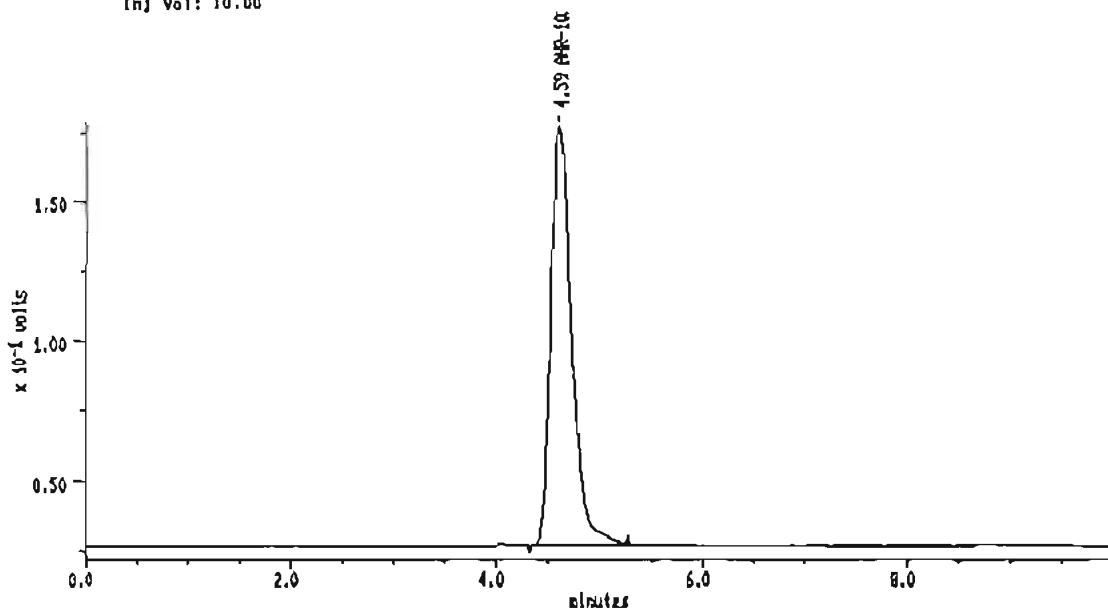
Index: 21

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.692	DB	2116392	160851	98.88	AHR-10202B
2	6.483	SS	23941	1953	1.12	deg
TOTAL			2139332	162804		

Sample: A28 80°C-1W Channel: detector 1 Filename: INI-22 Chart Speed: Full Size
 Acquired: 20-FEB-101 22:23 Method: B:YAHRYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 22:34:25

SAMPLE: A28 80°C-1W

#25 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-22

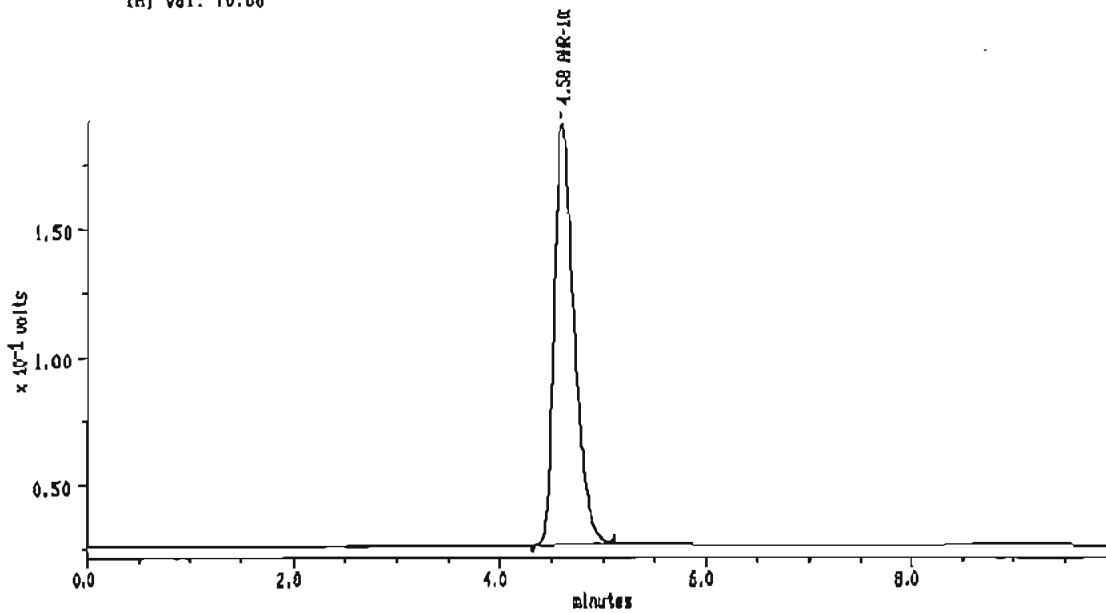
Index: 22

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.632	BD	2117458	150742	100.00	AHR-102828
TOTAL			2117458	150742		

Sample: A27 80°C-1W Channel: detector 1 Filename: INI-23 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:36 Method: B:VAJIRVIK13VJNITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 22:46:41

SAMPLE: A27 80°C-1W

#28 in Method: AHR-10282B

Acquired: 20-FEB-2001 22:35

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-23

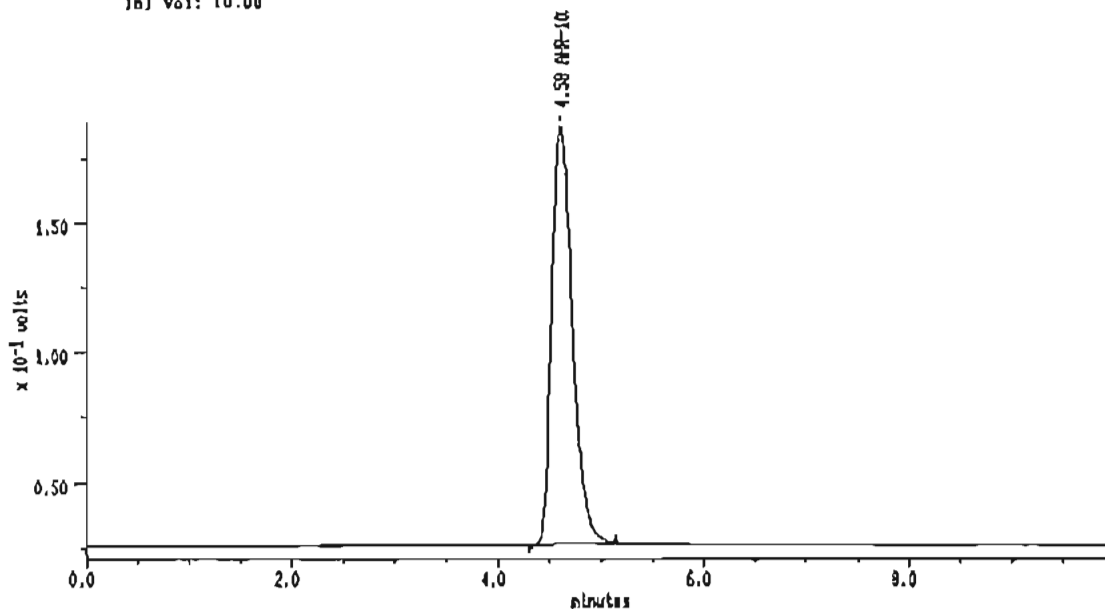
Index: 23

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	BB	2250178	183888	100.00	AHR-10282B
TOTAL			2250178	183888		

Sample: A28 80°C-1W Channel: detector 1 Filename: IN1-24 Chart Speed: Full Size
 Acquired: 20-FEB-01 22:48 Method: 8:VAIR(V)KISVINIJJAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 22:68:68

SAMPLE: A28 80°C-1W

#27 In Method: AHR-10282B

Acquired: 20-FEB-2001 22:48

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-24

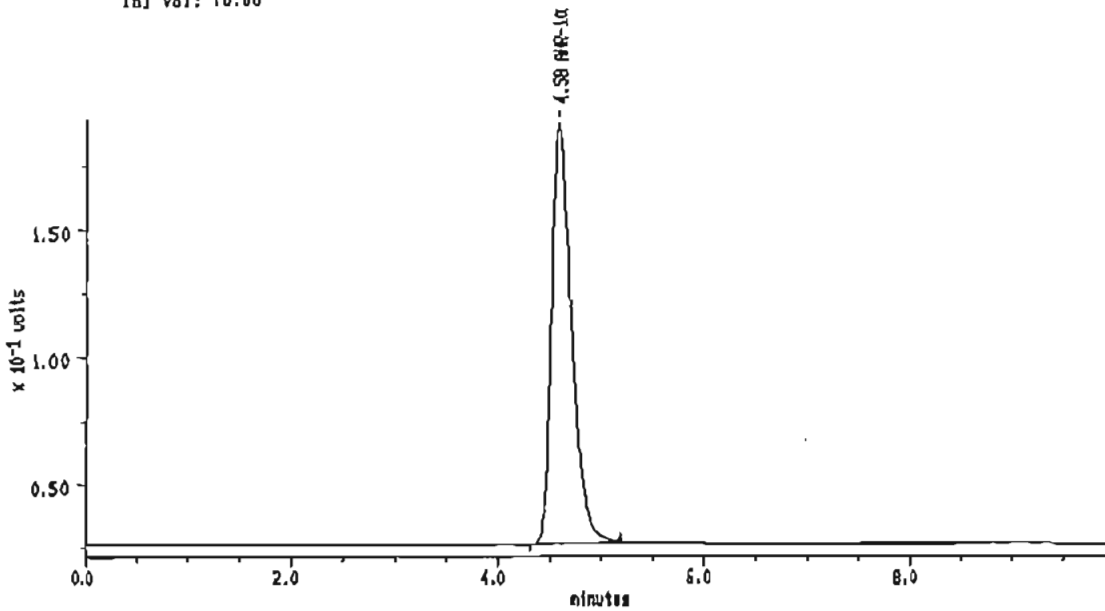
Index: 24

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.683	DB	2211902	180832	100.00	AHR-10282B
TOTAL			2211902	180832		

Sample: A29 80°C-1W Channel: detector 1 Filename: INT-25 Chart Speed: Full Size
 Acquired: 20-FEB-2001 22:57 Method: B:VAHRV(X)3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 23:08:15

SAMPLE: A29 80°C-1W

#28 in Method: AHR-10282B

Acquired: 20-FEB-2001 22:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INT-25

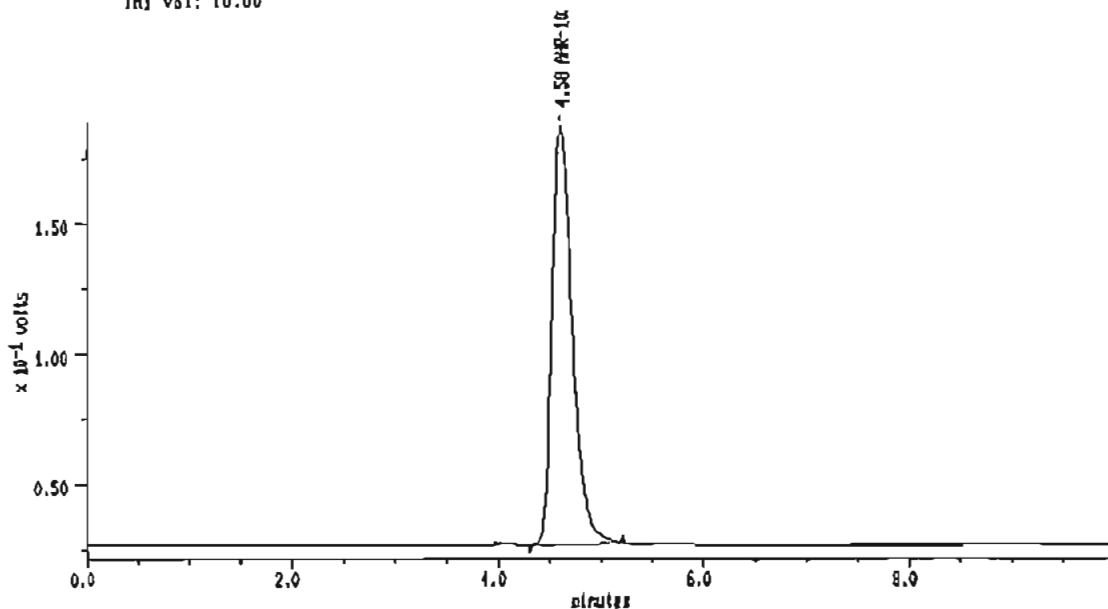
Index: 26

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	BB	2290389	185439	100.00	AHR-10282B
TOTAL			2290389	185439		

Sample: A30 80°C-1W Channel: detector 1 Filename: INI-28 Chart Speed: Full Size
 Acquired: 20-FEB-01 23:09 Method: B:YAHYIKI0VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 23:19:32

SAMPLE: A30 80°C-1W

#29 (n Method: AHR-10282B

Acquired: 20-FEB-2001 23:09

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-28

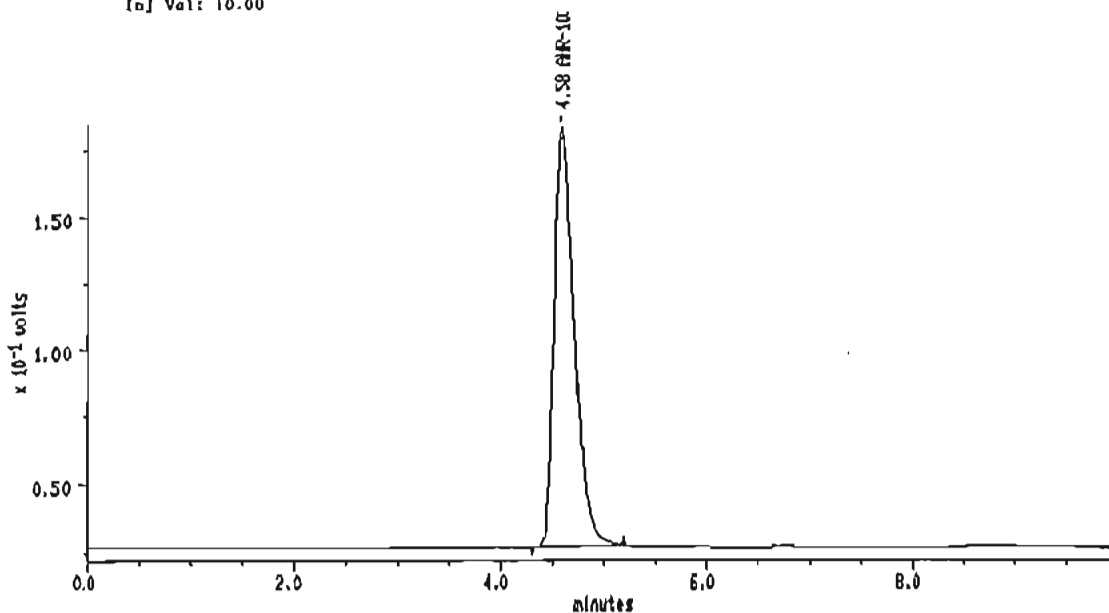
Inj Vol: 10.0

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	BB	2227606	180837	100.00	AHR-10282B
TOTAL			2227606	180837		

Sample: A3) 80°C-1W Channel: Detector 1 Filename: IN1-27 Chart Speed: Full Size
 Acquired: 20-FEB-2001 23:20 Method: D:VAHRY\K13V\INITIAL Operator: S.S
 Inj Val: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 23:30:48

SAMPLE: A3) 80°C-1W

#30 In Method: AHR-10282B

Acquired: 20-FEB-2001 23:20

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN1-27

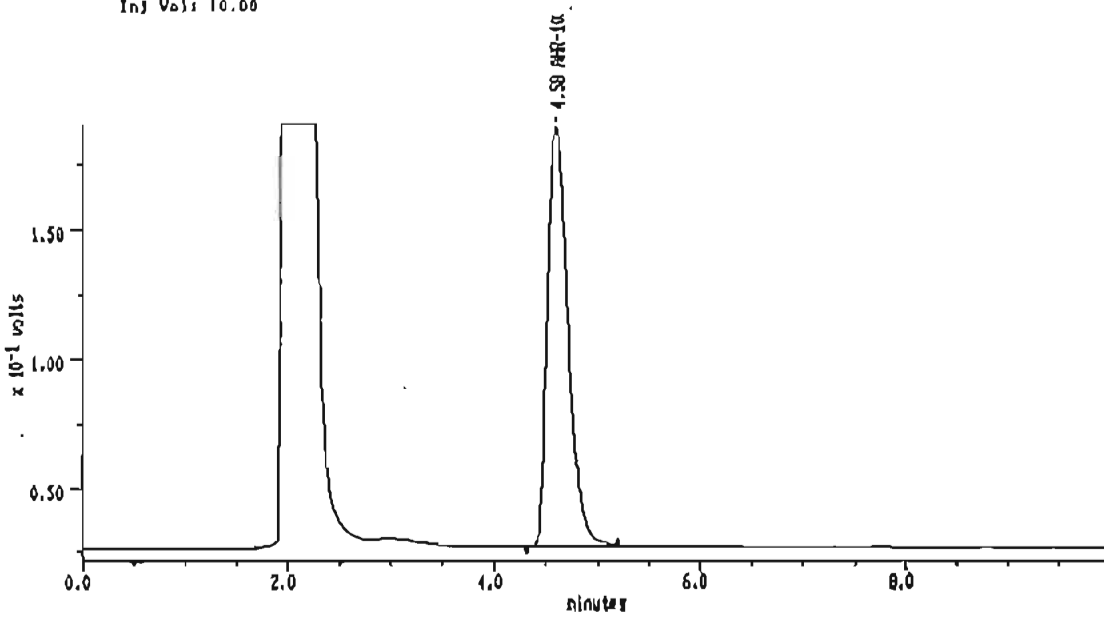
Index: 27

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.576	DB	2170890	168770	100.00	AHR-10282B
TOTAL			2170890	168770		

Sample: A32 60°C-1W Channel: detector 1 Filename: INI-28 Chart Speed: Full Size
 Acquired: 20-FEB-2001 23:31 Method: B:VAHRVIXI3YINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 23:42:13

SAMPLE: A32 60°C-1W

#31 (n Method: AHR-10282B

Acquired: 20-FEB-2001 23:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-28

Index: 28

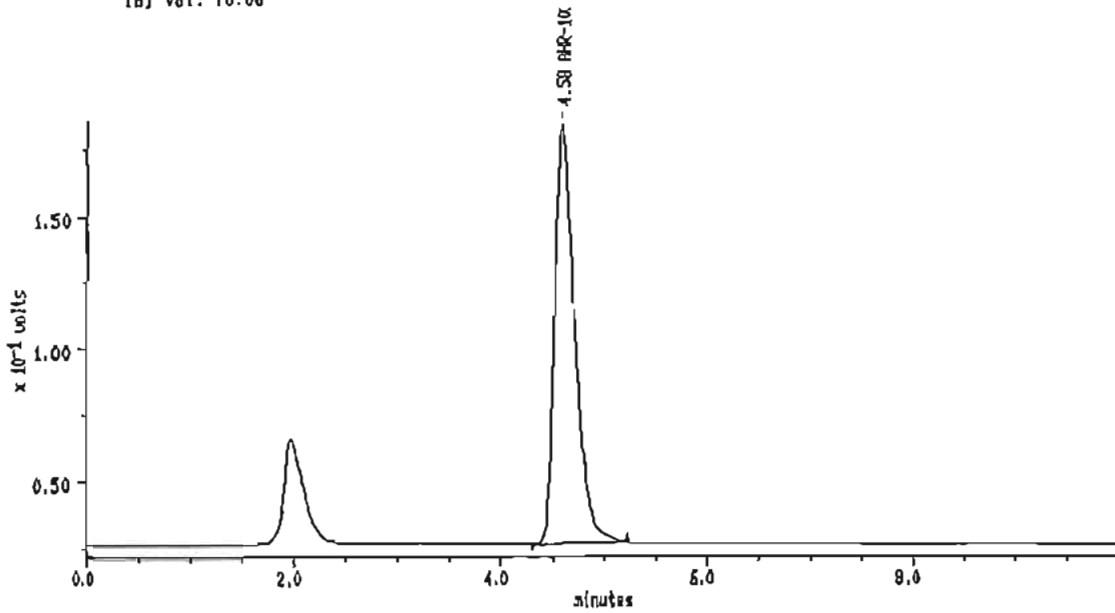
Injection Volume: 10.0

Amount: 0.000

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.583	BB	2243853	182390	100.00	AJR-10282B
TOTAL			2243853	182390		

Sample: A33 80°C-1W Channel: detector 1 Filename: INI-29 Chart Speed: Full Size
 Acquired: 20-FEB-2001 23:43 Method: 8:YAKRYIKI3VINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 23:63:38

SAMPLE: A33 80°C-1W

#32 In Method: AHR-10282B

Acquired: 20-FEB-2001 23:43

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-29

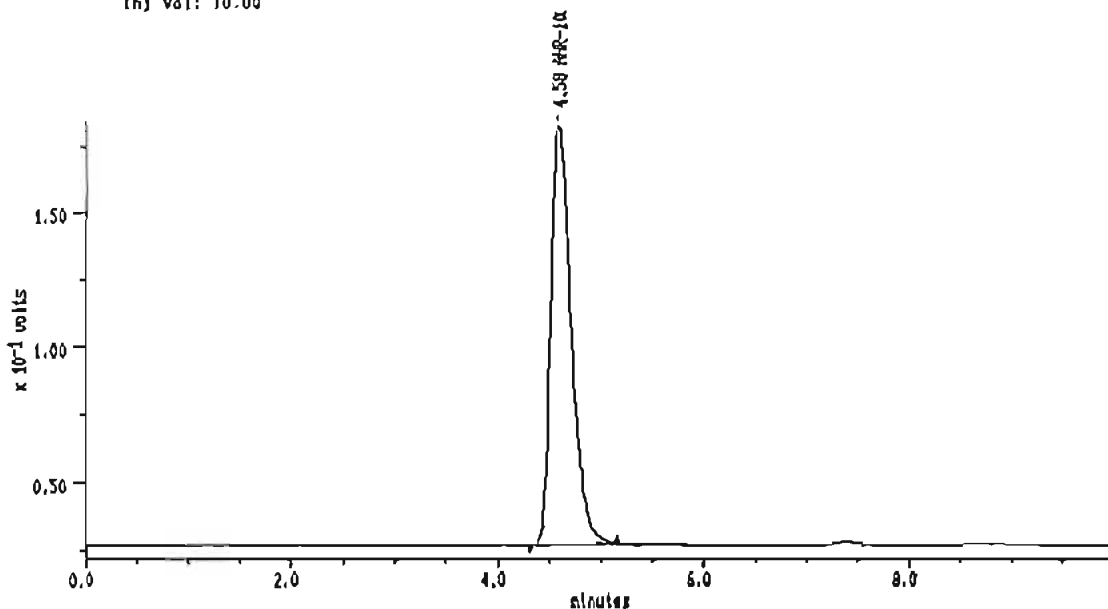
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.693	88	2193124	167839	100.00	AHR-10282B
TOTAL			2193124	167839		

Sample: A34 80°C-1W Channel: detector 1 Filename: INI-30 Chart Speed: Full Size
 Acquired: 20-FEB-01 23:54 Method: B:YAHRVIKI3VINJ1AL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 21-FEB-2001 0:06:02

SAMPLE: A34 80°C-1W

#33 in Method: AHR-10282B

Acquired: 20-FEB-2001 23:54

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: INI-30

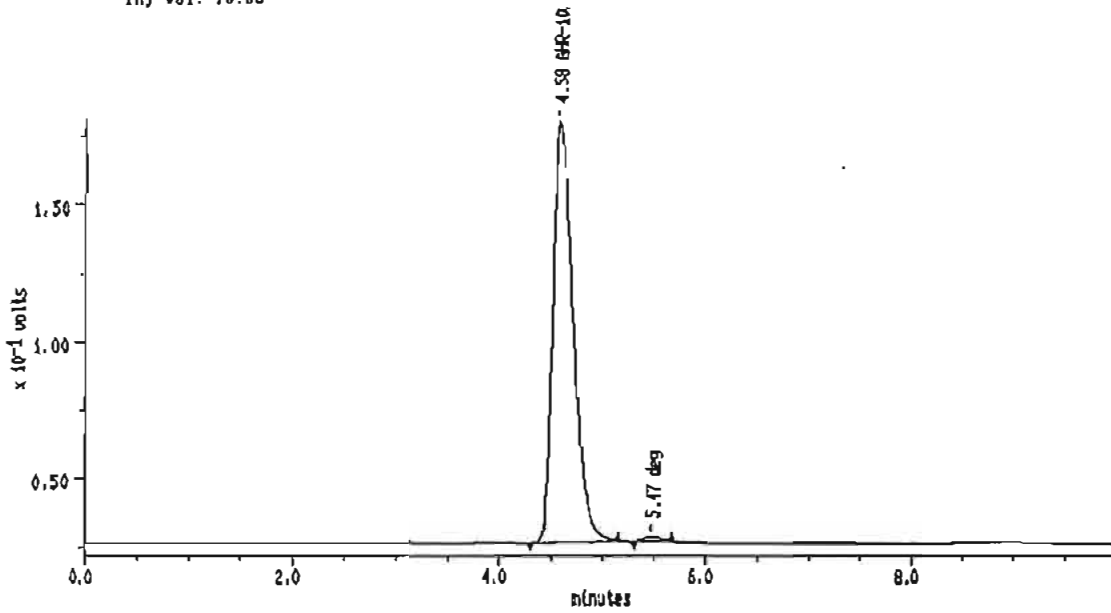
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.575	BB	2153242	168022	100.00	AHR-10282B
TOTAL			2153242	168022		

Sample: A35 80°C-1W Channel: detector 1 File Name: INI-31 Chart Speed: Full Size
 Acquired: 21-FEB-2001 0:06 Method: B:VAURVIX13VINITTAL Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 21-FEB-2001 0:18:28

SAMPLE: A35 80°C-1W

#34 In Method: AHR-10282B

Acquired: 21-FEB-2001 0:06

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

File Name: INI-31

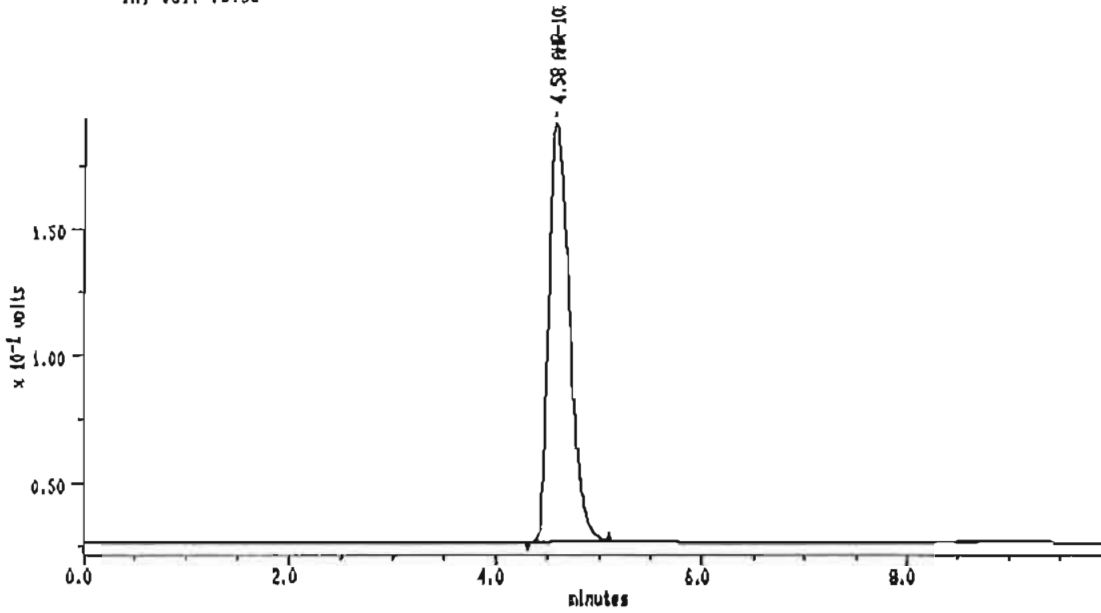
Index: 31

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.575	BB	2118180	163469	98.98	AHR-10282B
2	5.487	BB	21814	1737	1.02	deg
TOTAL			2137994	165197		

Sample: STD2 Channel: detector 1 Filename: INI-32 Chart Speed: Full Size
 Acquired: 21-FEB-2001 0:17 Method: B:VAHRV\K13\INITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 21-FEB-2001 0:27:58

SAMPLE: STD2

#35 In Method: AHR-10282B
 Acquired: 21-FEB-2001 0:17
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: INI-32
 Index: 32
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.575	00	2278153	186513	100.00	AHR-102828
TOTAL			2278153	186513		

2/20 16:27
NO.21 PH 7.05
24.1°C

2/20 16:28
NO.22 PH 7.10
24.3°C

2/20 16:30
NO.23 PH 7.10
24.3°C

2/20 16:32
NO.24 PH 7.00
24.4°C

2/20 16:33
NO.25 PH 7.09
24.5°C

2/20 16:34
NO.26 PH 7.06
24.3°C

2/20 16:35
NO.27 PH 7.05
24.3°C

2/20 16:36
NO.28 PH 7.08
24.4°C

2/20 16:36
NO.29 PH 7.07
24.5°C

2/20 16:38
NO.30 PH 7.12
24.4°C

2/20 16:39
NO.31 PH 7.02
24.4°C

2/20 16:40
NO.32 PH 7.05
24.5°C

2/20 16:41
NO.33 PH 7.03
24.6°C

2/20 16:42
NO.34 PH 7.03
24.5°C

2/20 16:43
NO.35 PH 7.02
24.6°C

2/20 16:44
NO.36 PH 7.01
24.6°C

2/20 16:45
NO.37 PH 7.04
24.5°C

2/20 16:46
NO.38 PH 7.10
24.6°C

2/20 16:48
NO.39 PH 7.10
24.5°C

2/20 16:49
NO.40 PH 7.10
24.4°C

2/20 16:50
NO.41 PH 7.02
24.6°C

2/20 16:51
NO.42 PH 7.02
24.6°C

2/20 16:52
NO.43 PH 7.04
24.6°C

2/20 16:53
NO.44 PH 7.09
24.6°C

2/20 16:53
NO.45 PH 7.08
24.5°C

2/20 16:54
NO.46 PH 7.05
24.7°C

2/20 16:55
NO.47 PH 7.05
24.6°C

2/20 16:56
NO.48 PH 7.06
24.6°C

2/20 16:56
NO.49 PH 7.06
24.4°C

2/20 16:57
NO.50 PH 7.10
24.5°C

P2000B177 Lot No. 01K13/ Unit 1/

本生データは経時的な劣化のおそれがあるため、複写しました。
従って、原本と相違ありません。

05.05.06 澤 嗣郎

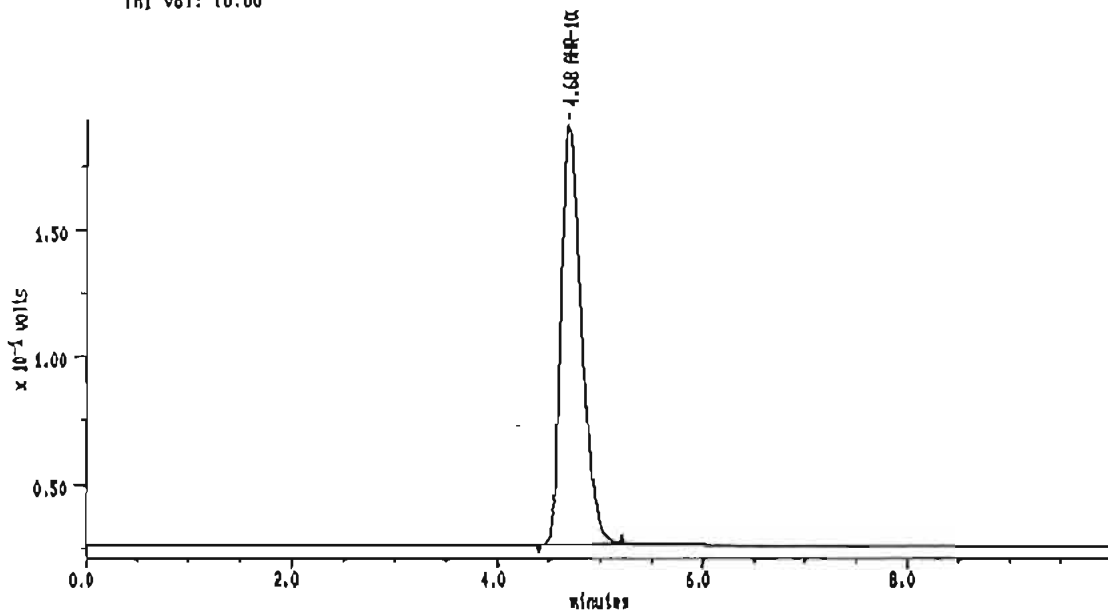
プロナック点眼液の安定性試験
Lot No.01H151

試験コード：P2000B177
試験実施者：澤 嗣郎
試験実施日：2001年02月19日

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Colloc(%)	Water	Initial	present
STD	1	S4-01	2303381						
STD	2	S4-18	2266689						
STD	mean		2284535	0.9980					
A-18	60°C-4W	S4-02	2379311	1.0394	101.39	94.51	6.79	8.5880	8.2530
A-19	60°C-4W	S4-03	2470948	1.0784	102.54	95.41	6.95	8.6173	8.2724
A-20	60°C-4W	S4-04	1329880	0.5809	55.01	51.27	6.79	8.6044	8.2882
A-21	60°C-4W	S4-05	1938745	0.8469	79.19	73.81	6.79	8.5498	8.2174
A-22	60°C-4W	S4-06	1594609	0.6966	67.87	63.19	6.89	8.5821	8.2425
A-23	60°C-4W	S4-07	2027430	0.8857	83.87	78.17	6.80	8.6440	8.3047
A-24	60°C-4W	S4-08	1833727	0.8011	77.68	72.44	6.74	8.6247	8.2897
A-25	60°C-4W	S4-09	1908933	0.8339	84.04	78.39	6.72	8.5719	8.2415
A-18	50°C-4W	S4-10	2277761	0.9950	97.05	94.13	3.01	8.6300	8.4800
A-19	50°C-4W	S4-11	2397283	1.0473	99.49	96.34	3.17	8.5270	8.3725
A-20	50°C-4W	S4-12	1527362	0.6672	63.18	61.18	3.16	8.4625	8.3103
A-21	50°C-4W	S4-13	2004949	0.8759	81.91	79.42	3.04	8.6136	8.4628
A-22	50°C-4W	S4-14	1725127	0.7536	73.43	71.03	3.27	8.5703	8.4093
A-23	50°C-4W	S4-15	2073253	0.9057	86.77	83.05	3.17	8.5336	8.3790
A-24	50°C-4W	S4-16	1887128	0.8244	79.94	77.40	3.18	8.4512	8.2986
A-25	50°C-4W	S4-17	1969902	0.8606	86.73	83.96	3.19	8.5677	8.4109

計算ミスがあったため訂正した。
計算に使用するデータを記載するに再編集した。
05.05.06 澤 嗣郎

Sample: STD1 Channel: detector 1 Filename: S4-01 Chart Speed: Full Size
 Acquired: 19-FEB-101 21:02 Method: B:YHRV11115V80-49 Operator: S.S
 [inj Vol]: 10.00



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MAXIMA 825 カスタムレポート

Printed: 19-FEB-2001 21:13:03

SAMPLE: STD1

#4 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:02

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: DNKN

Instrument: Instrument 1

Filename: S4-01

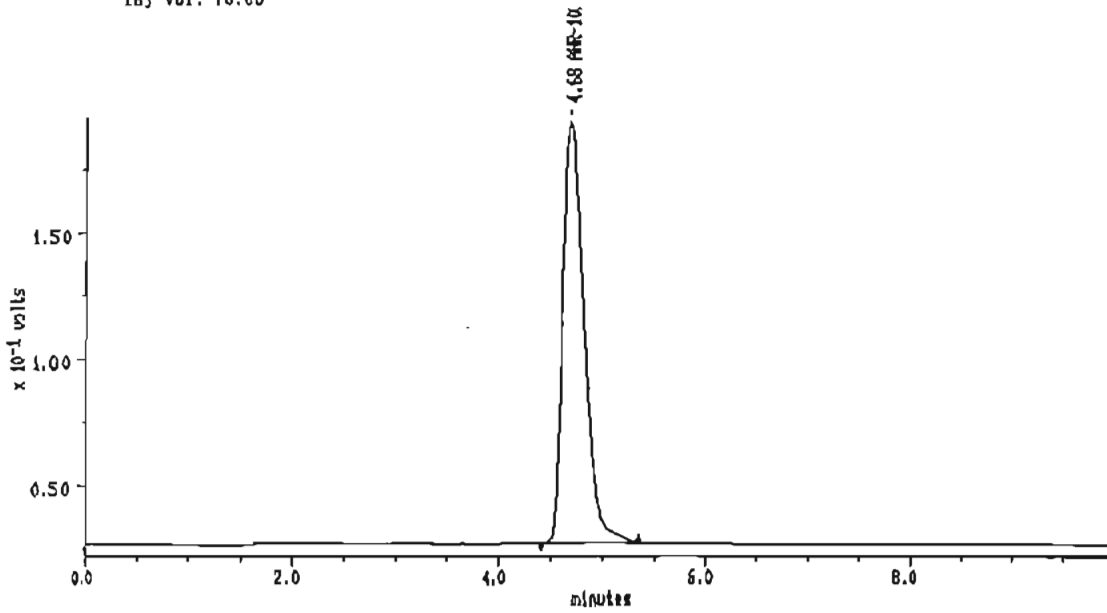
Index: 18

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.676	BB	2303381	164825	100.00	AHR-10282B
TOTAL			2303381	164825		

Sample: A10 60°C-4# Channel: detector 1 Filenane: S4-02 Chart Speed: Full Size
 Acquired: 19-FEB-101 21:13 Method: B:YAJRVINIGY80-4# Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 19-FEB-2001 21:24:27

SAMPLE: A10 60°C-4#

#5 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:13

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filenane: S4-02

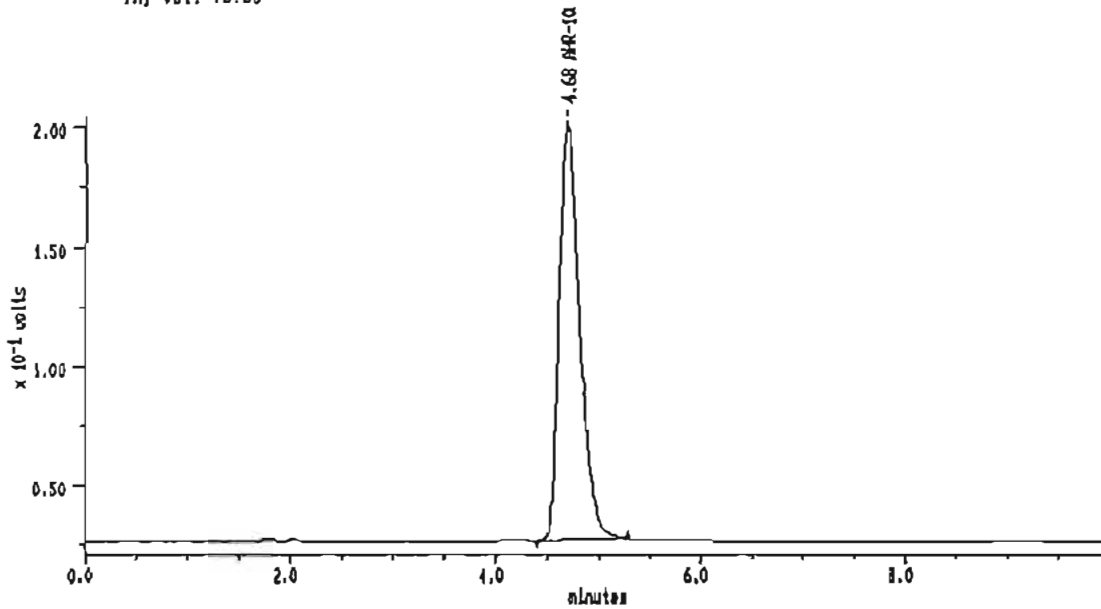
Index: 10

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.603	BB	2379311	107070	100.00	AHR-10282B
TOTAL			2379311	107070		

Sample: A19 00°C-4W Channel: detector 1 Filename: S4-03 Chart Speed: Full Size
 Acquired: 19-FEB-2001 21:26 Method: 8:YHRVH15Y00-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 19-FEB-2001 21:35:43

SAMPLE: A19 00°C-4W

#8 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:26

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-03

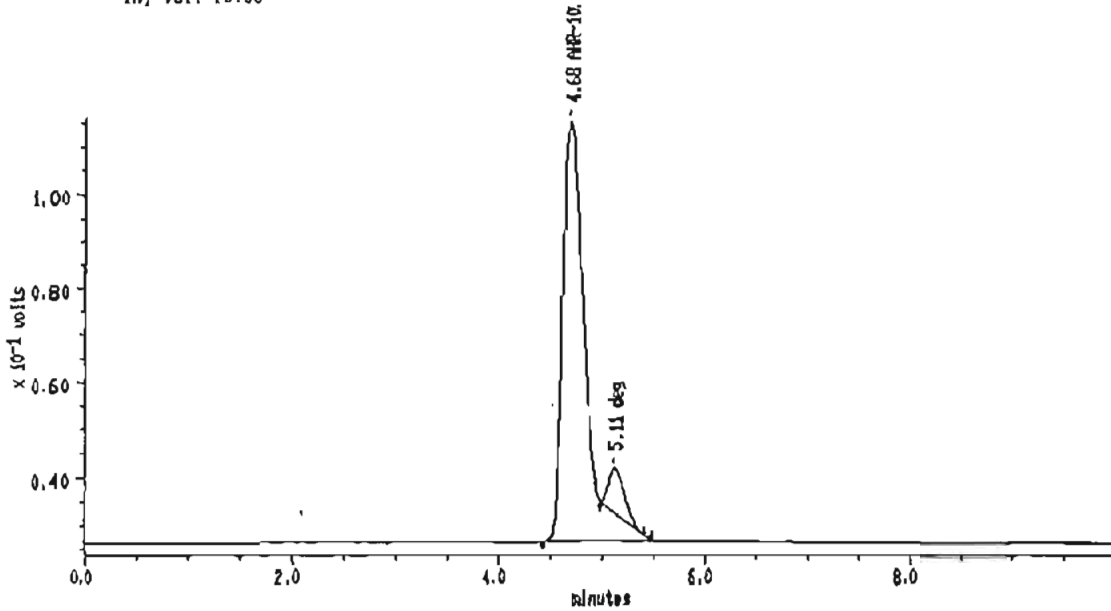
Index: 20

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.675	BB	2470948	176845	100.00	AHR-10282B
TOTAL			2470948	176845		

Sample: A20 80°C-4W Channel: detector 1 Filename: S4-04 Chart Speed: Full Size
 Acquired: 19-FEB-2001 21:38 Method: 8:VAHRV11116V80-4W Operator: S.S
 Inj Volt: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 12:10:57

SAMPLE: A20 80°C-4W

#7 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-04

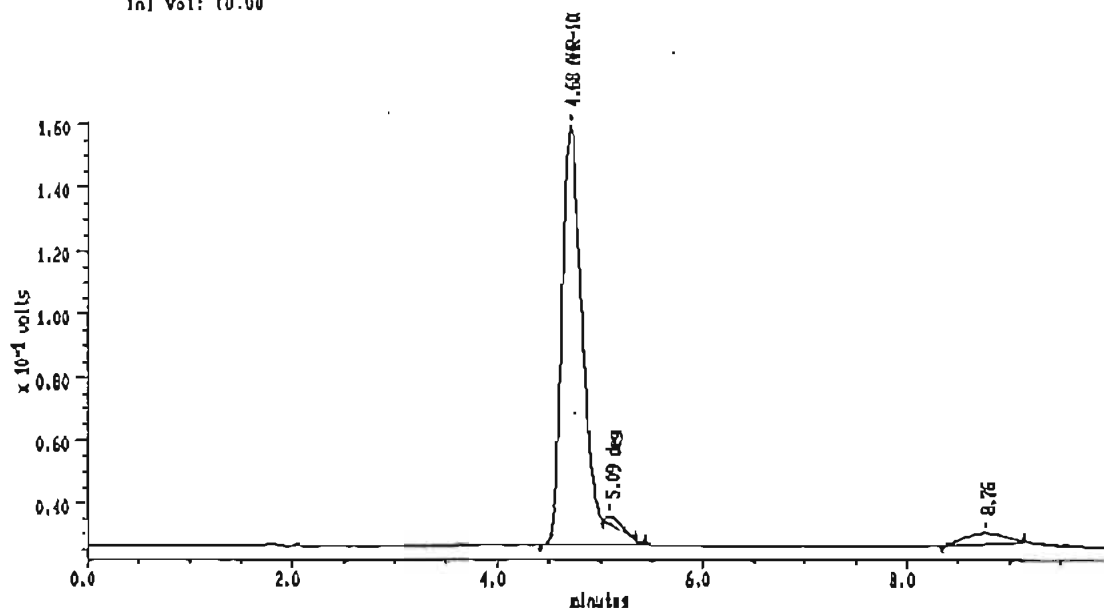
Index: 21

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.683	BB	1329880	88819	92.74	AHR-10282B
2	5.108	SS	104020	9222	7.28	deg
TOTAL			1433700	97840		

Sample: A21 80°C-4W Channel: detector 1 Filename: S4-05 Chart Speed: Full Size
 Acquired: 19-FEB-2001 21:47 Method: B:VAHRV)HISV80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 12:11:20

SAMPLE: A21 80°C-4W

#0 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:47

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-05

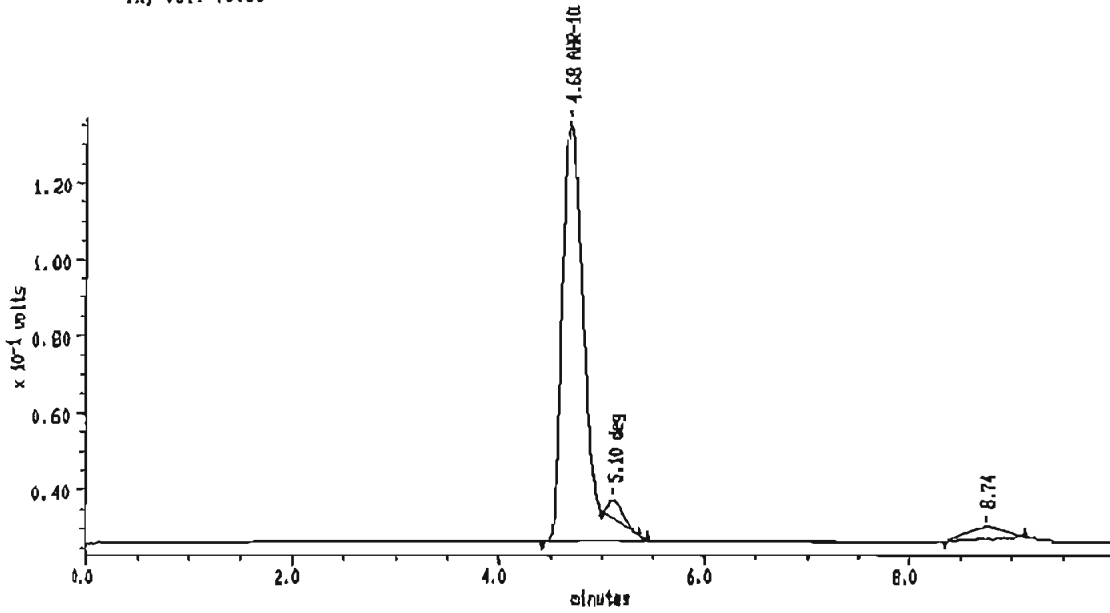
Index: 22

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.683	BB	1338745	133208	98.68	AHR-10282B
2	5.092	SS	28335	2588	1.34	deg
TOTAL			1367080	136792		

Sample: A22 80°C-4W Channel: detector 1 Filename: S4-08 Chart Speed: Full Size
 Acquired: 19-FEB-10 21:59 Method: B:YAHRYI1116V80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 12:11:55

SAMPLE: A22 80°C-4W

#9 In Method: AHR-10282B

Acquired: 19-FEB-2001 21:59

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-08

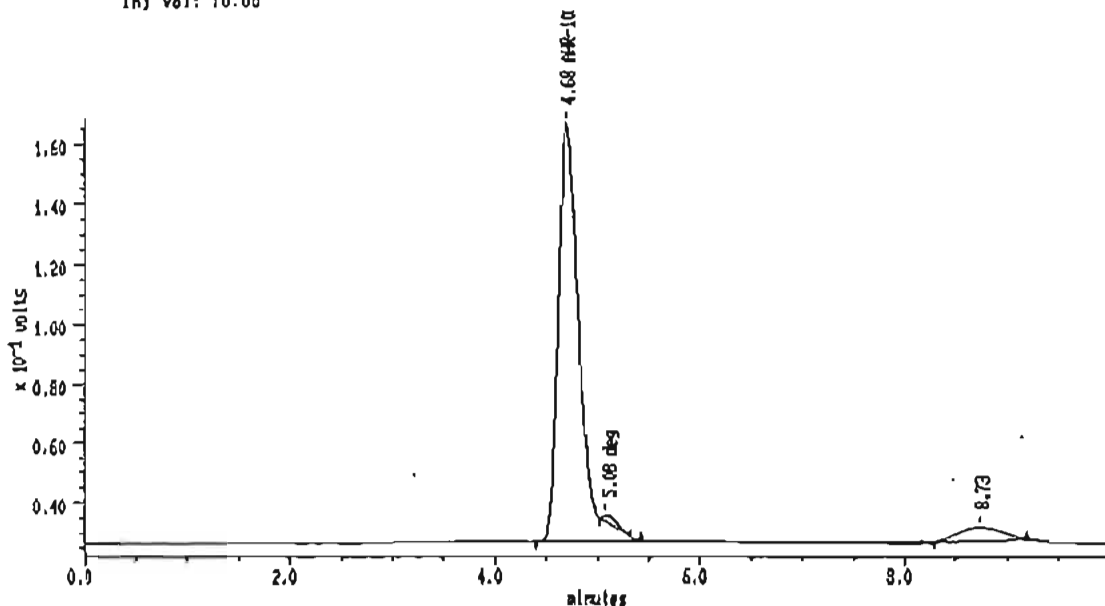
Index: 23

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.883	BB	1694809	109978	97.09	AHR-10282B
2	5.100	SS	47847	4542	2.91	deg
TOTAL			1842456	113619		

Sample: A23 80°C-4W Channel: detector 1 Filename: S4-07 Chart Speed: Full Size
 Acquired: 19-FEB-2001 22:10 Method: 0:YAHRVJH)EV80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 12:12:24

SAMPLE: A23 80°C-4W

#10 In Method: AHR-10282B

Acquired: 19-FEB-2001 22:10

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-07

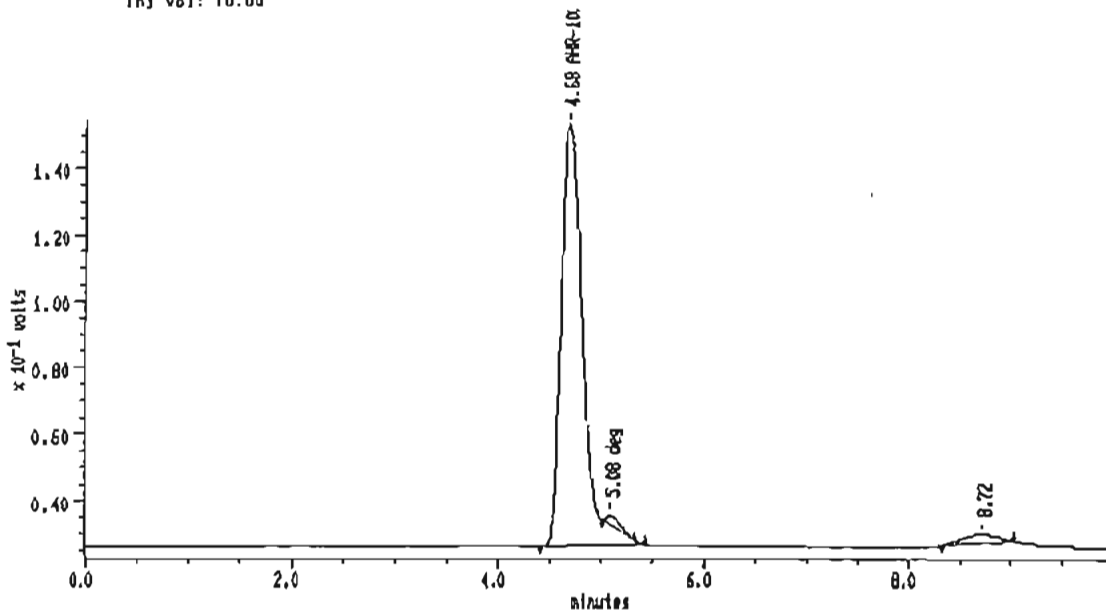
Index: 24

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.076	BB	2027430	140347	88.87	AHR-10282B
2	6.083	SS	23187	2268	1.13	deg
TOTAL			2050618	142615		

Sample: A24 80°C-4W Channel: detector 1 Filename: S4-08 Chart Speed: Full Size
 Acquired: 19-FEB-10J 22:22 Method: 8:YAHRYIH16V80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 12:12:53

SAMPLE: A24 80°C-4W

#11 In Method: AHR-10282B

Acquired: 19-FEB-2001 22:22

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-08

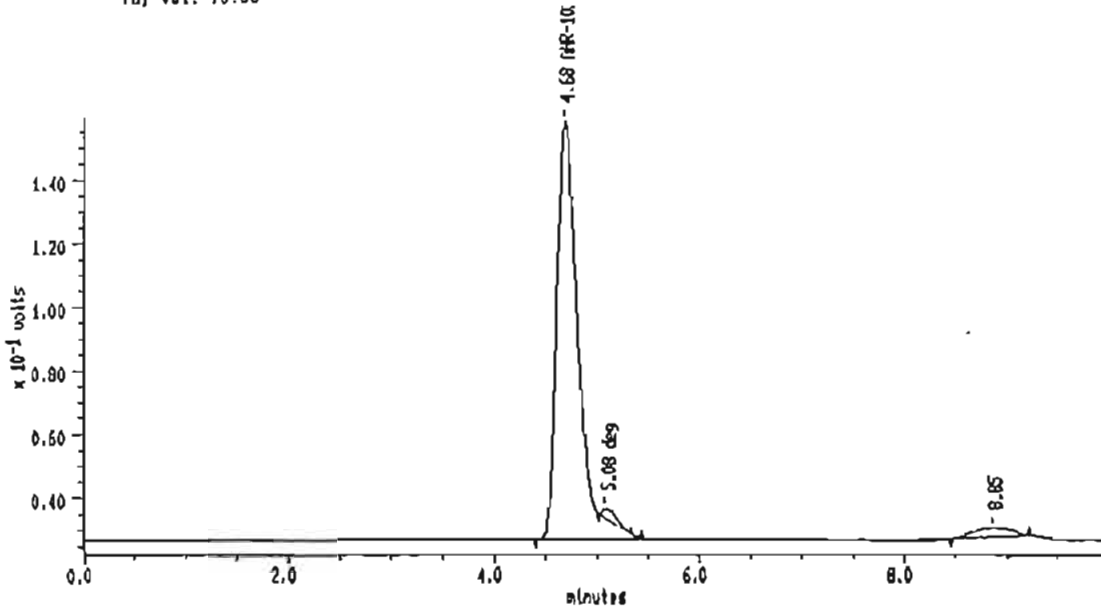
Index: 25

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.676	BB	1833727	128887	98.57	AHR-10282B
2	6.003	SS	28810	2887	1.43	deg
TOTAL			1880337	129534		

Sample: A25 80°C-4W Channel: detector 1 Filename: S4-09 Chart Speed: Full Size
 Acquired: 19-FEB-10 22:33 Method: B:YAHRYIHI15V60-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 12:13:22

SAMPLE: A25 80°C-4W

#12 In Method: AHR-10282B

Acquired: 19-FEB-2001 22:33

Rate: 2.0 points/deg

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-08

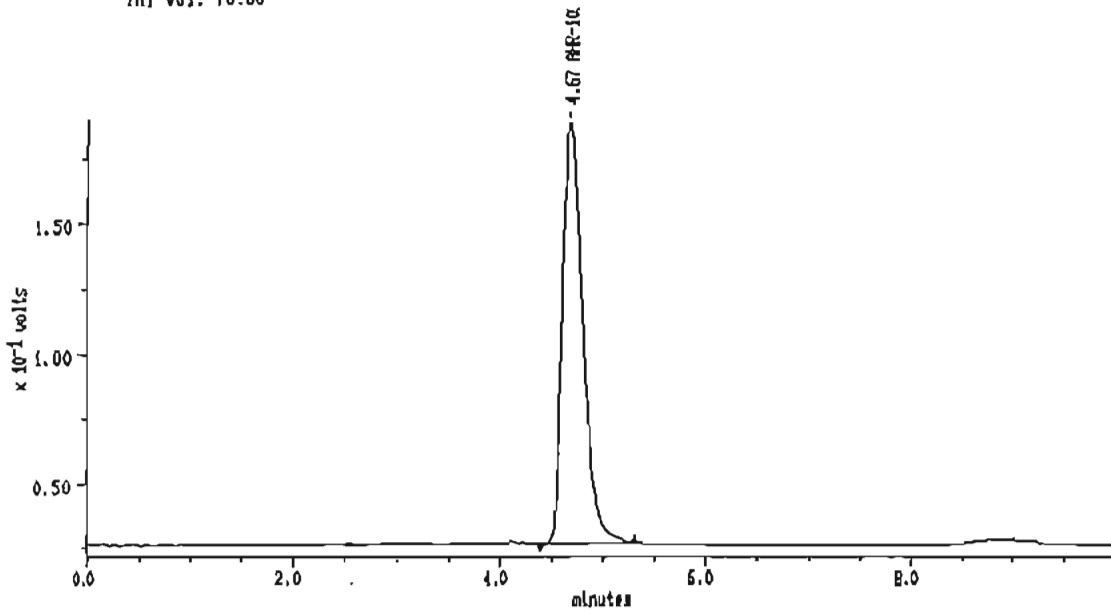
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.676	BD	1908933	131639	98.44	AHR-10282B
2	5.083	SS	30286	2986	1.56	deg
TOTAL			1939198	134505		

Sample: A18 50°C-4W Channel: detector 1 Filename: S4-10 Chart Speed: Full Size
 Acquired: 19-FEB-2001 22:44 Method: 0:YANRVIH16V80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

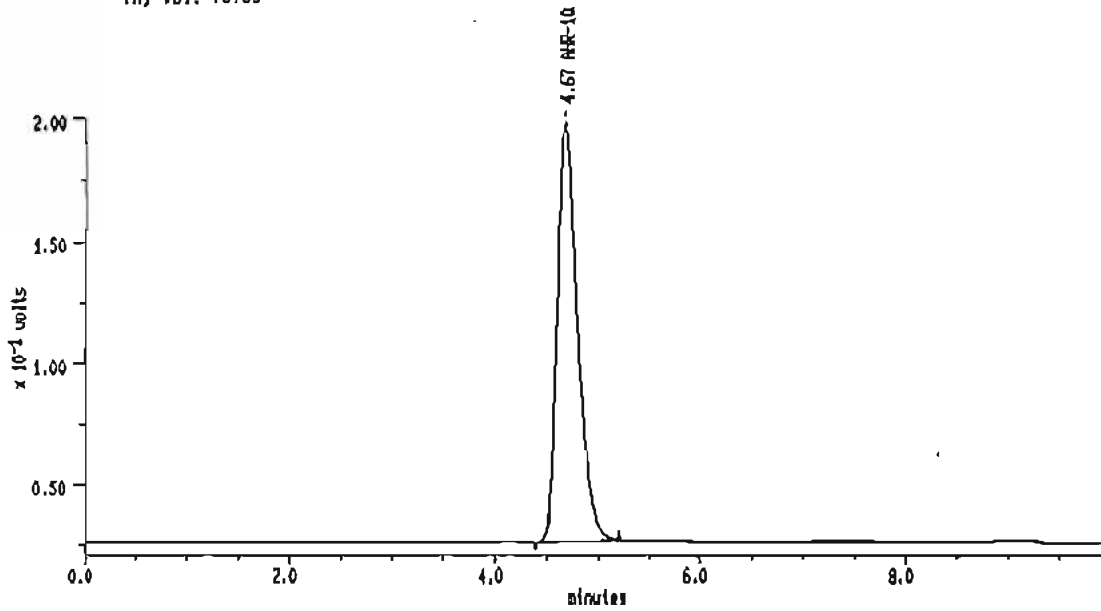
Printed: 19-FEB-2001 22:56:21

SAMPLE: A18 50°C-4W Type: UNKN
 #13 in Method: AHR-10282B Instrument: Instrument 1
 Acquired: 19-FEB-2001 22:44 Filename: S4-10
 Rate: 2.0 points/sec Index: 27
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.807	BB	2277701	182898	100.00	AHR-10282B
TOTAL			2277701	182898		

Sample: A19 50°C-4W Channel: detector 1 Filename: S4-11 Chart Speed: Full Size
 Acquired: 19-FEB-10 22:58 Method: B:VAHRY1H15Y86-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 19-FEB-2001 23:08:38

SAMPLE: A19 50°C-4W

#14 In Method: AHR-10282B

Acquired: 19-FEB-2001 22:58

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-11

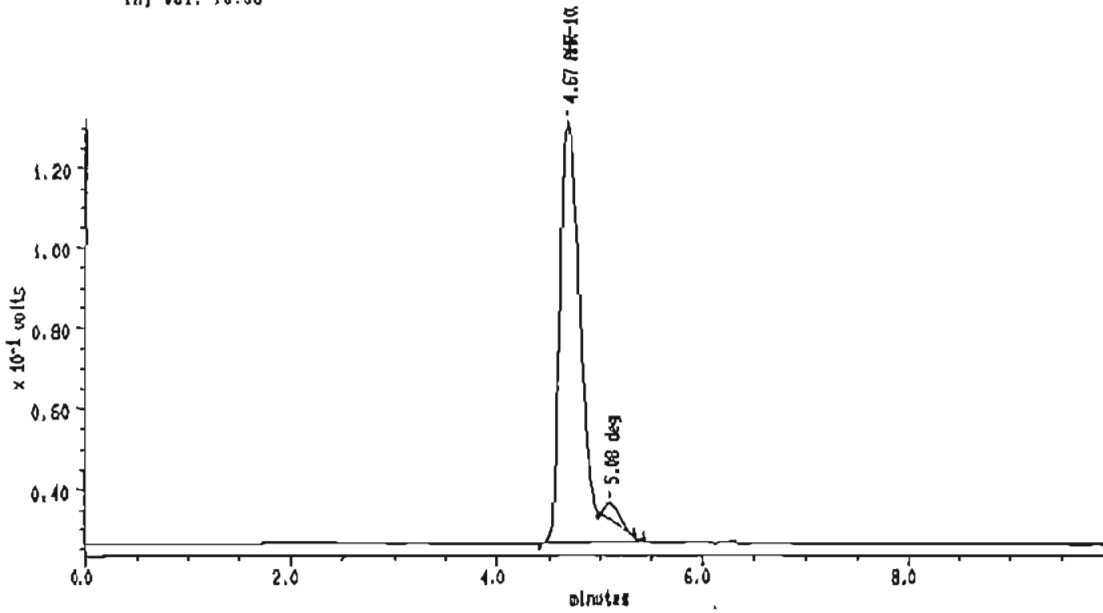
Index: 20

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.687	DD	2397283	172318	100.00	AHR-10282B
TOTAL			2397283	172318		

Sample: A20 50°C-4W Channel: detector 1 Filename: S4-12 Chart Speed: Full Size
 Acquired: 19-FEB-10 23:07 Method: B:VAHRV1H15Y80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 12:13:61

SAMPLE: A20 50°C-4W

#15 In Method: AHR-10282B

Acquired: 19-FEB-2001 23:07

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-12

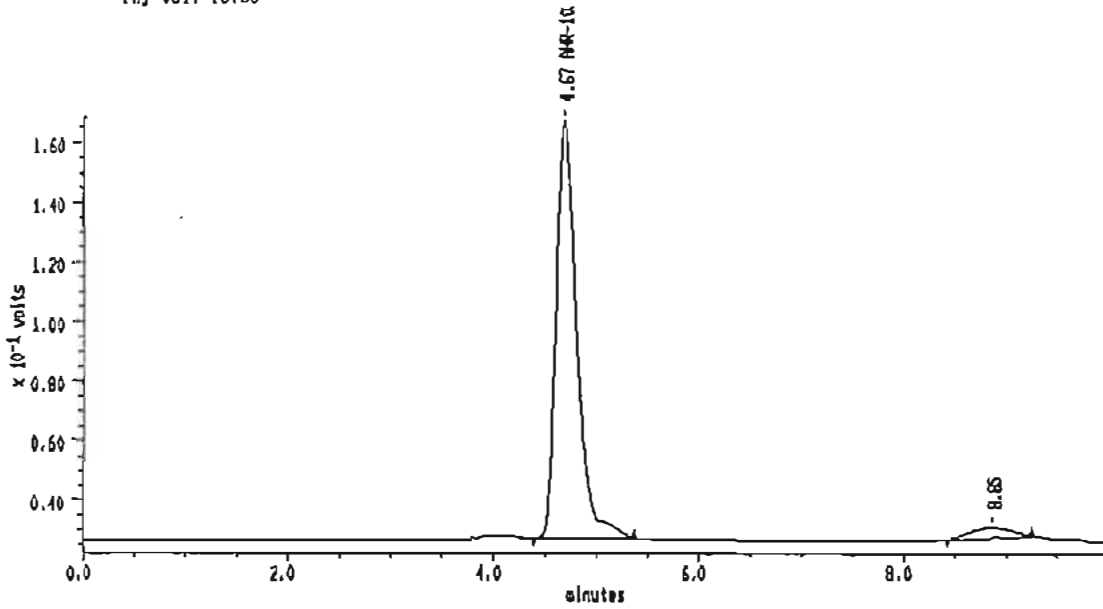
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.687	86	1527382	104941	97.38	AHR-10282B
2	5.083	SS	41432	4052	2.84	deg
TOTAL			1568794	108992		

Sample: A21 50°C-4W Channel: detector 1 Pilenax: S4-13 Chart Speed: Full Size
 Acquired: 19-FEB-101 23:18 Method: B:YAHRYIH16Y60-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 19-FEB-2001 23:29:20

SAMPLE: A21 50°C-4W

#16 in Method: AHR-10282B

Acquired: 19-FEB-2001 23:18

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instruamt: Instrument 1

Pilenax: S4-13

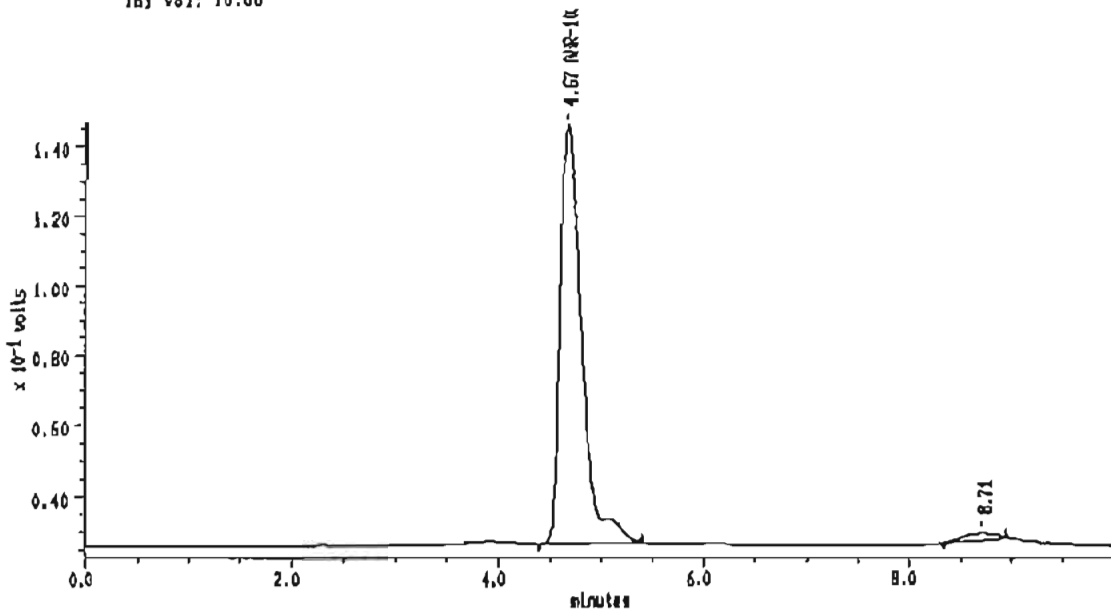
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.667	DB	2004949	140489	100.00	AHR-10282B
TOTAL			2004949	140489		

Sample: A22 60°C-4W Channel: detector 1 Filename: S4-14 Chart Speed: Full Size
 Acquired: 19-FEB-01 23:30 Method: 8:YAIRYJH15780-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 19-FEB-2001 23:40:37

SAMPLE: A22 60°C-4W

#17 In Method: AHR-10282B

Acquired: 19-FEB-2001 23:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-14

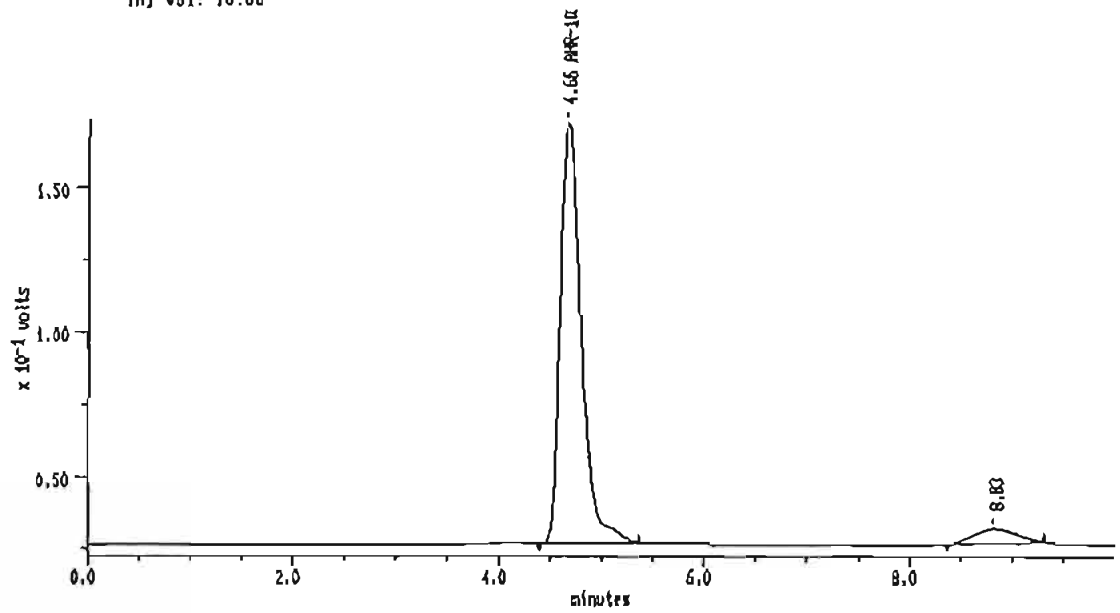
Index: 31

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.887	BB	1726127	119191	100.00	AHR-10282B
TOTAL			1726127	119191		

Sample: A23 50°C-4W Channel: detector 1 Filename: S4-16 Chart Speed: Full Size
 Acquired: 19-FEB-10 23:41 Method: B:VAHRY11115V80-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 19-FEB-2001 23:51:54

SAMPLE: A23 50°C-4W

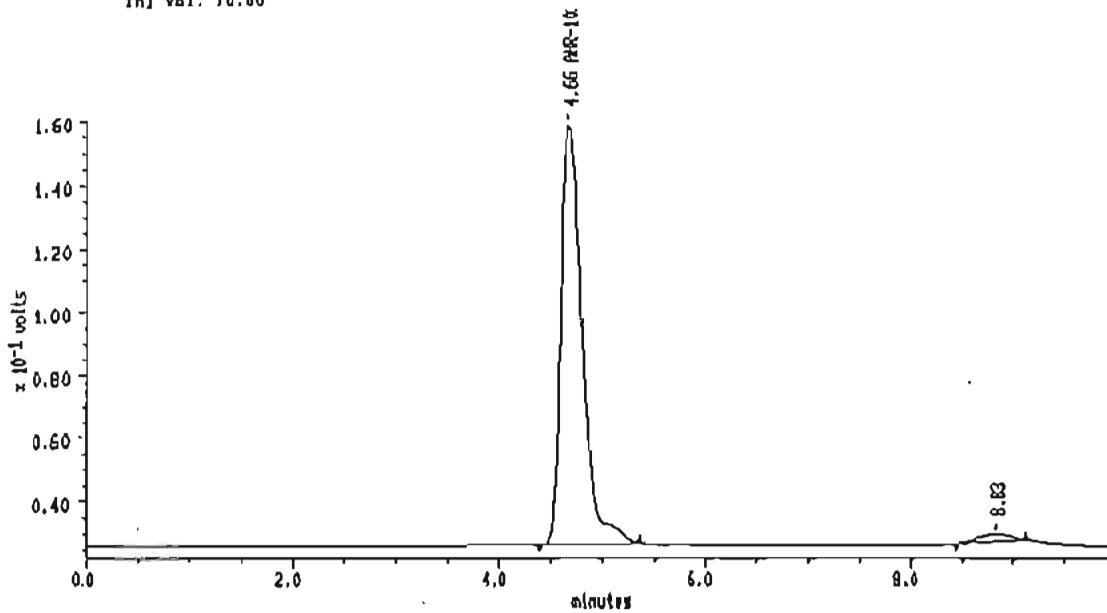
#18 In Method: **AHR-10282B**
 Acquired: 19-FEB-2001 23:41
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: S4-16
 Index: 32
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.658	BB	2073253	146808	100.00	AHR-10282B
TOTAL			2073253	146808		

Sample: A24 50°C-4W Channel: detector 1 Filename: S4-18 Chart Speed: Full Size
 Acquired: 19-FEB-2001 23:52 Method: B:VAHRV(H)5Y60-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 0:03:11

SAMPLE: A24 50°C-4W

#19 in Method: AHR-10282B

Acquired: 19-FEB-2001 23:52

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-18

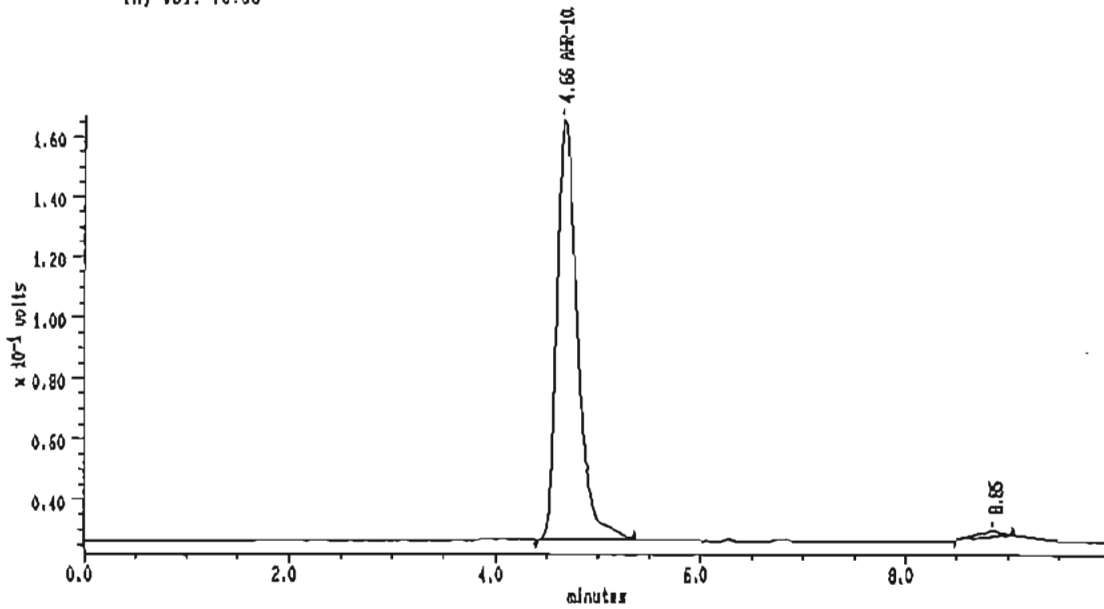
Index: 33

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.668	DB	1887128	132203	100.00	AHR-10282B
TOTAL			1887128	132203		

Sample: A25 50°C-4W Channel: detector 1 Filename: S4-17 Chart Speed: Full Size
 Acquired: 20-FEB-2001 0:03 Method: B:YAMRYIH15V60-4W Operator: S.S
 (n) Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 0:14:28

SAMPLE: A25 50°C-4W

#20 In Method: AHR-10282B

Acquired: 20-FEB-2001 0:03

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: S4-17

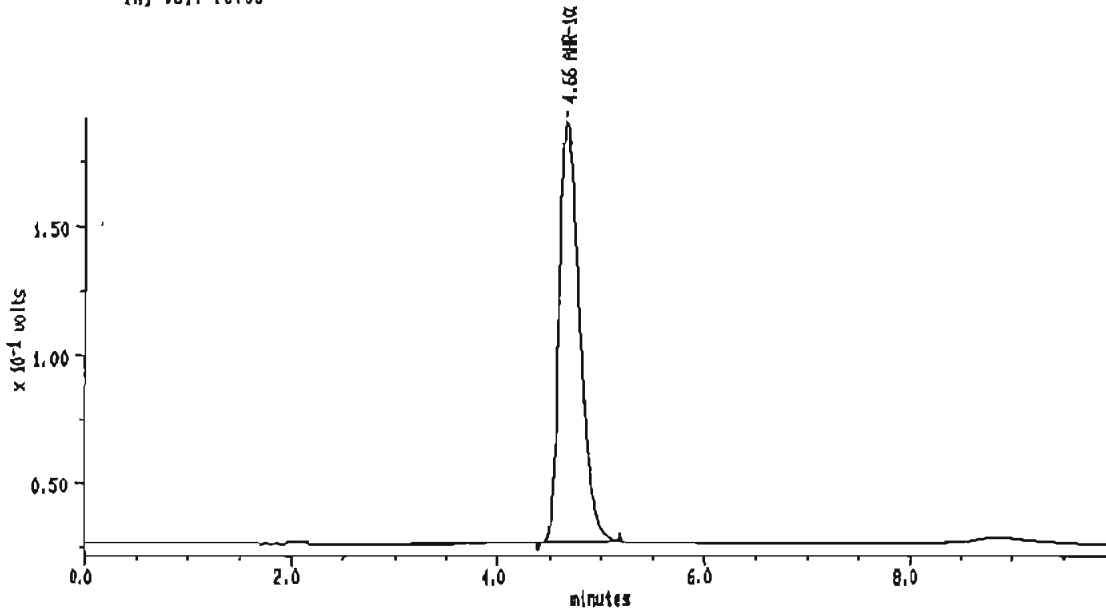
Index: 34

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.658	BB	1989902	138783	100.00	AHR-10282B
TOTAL			1989902	138783		

Sample: STD Channel: detector 1 Filename: 54-18 Chart Speed: Full Size
 Acquired: 20-FEB-2001 0:15 Method: D:\AJR\VINI16Y00-4W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 20-FEB-2001 0:25:00

SAMPLE: STD

#21 In Method: AHR-10282B

Acquired: 20-FEB-2001 0:15

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: 54-18

Index: 35

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.668	BB	2286889	183334	100.00	AHR-10282B
TOTAL			2286889	183334		

試験物質名: AHR10282B	試験コード: P2000B177	試験年月日: 2001年02月19日
試験項目:		試験実施者: 澤 嗣郎
ST10 AHR10282B 0.01995g/20ml X2-2/20ml MP		

								2/19 16:11 NO.93 PH 8.15 24.3°C
A18	60°C-KW	8.16	8.5880	8.2530	-	+	黄	
A19		8.17	8.6173	8.2724	-	-	黄	2/19 16:12 NO.99 PH 8.17 24.4°C
A20		6.98	8.6044	8.2682	++	++	橙黄	
A21		6.89	8.5498	8.2174	++	++	橙黄	2/19 16:13 NO. 1 PH 6.98 24.4°C
A22		7.00	8.5821	8.2425	+	±	黄	
A23		6.97	8.6440	8.3047	+	+	黄	2/19 16:14 NO. 2 PH 6.89 24.6°C
A24		6.90	8.6247	8.2897	+	++	橙黄	
A25		6.95	8.5719	8.2415	+	+	黄	2/19 16:15 NO. 3 PH 7.00 24.6°C
A18	50°C-KW	8.19	8.6300	8.4800	-	-	黄	
A19		8.15	8.5270	8.32725	-	-	黄	2/19 16:16 NO. 4 PH 6.97 24.6°C
A20		7.00	8.4625	8.3103	-	+	橙黄	
A21		6.94	8.6136	8.4628	-	+	橙黄	2/19 16:17 NO. 5 PH 6.90 24.5°C
A22		6.99	8.5703	8.4093	(+) +		黄	
A23		6.95	8.5336	8.3790	-	+	黄	2/19 16:17 NO. 6 PH 6.95 24.6°C
A24		6.90	8.4512	8.2986	-	+	橙黄	
A25		7.00	8.5677	8.4109	-	+	橙黄	2/19 16:18 NO. 7 PH 8.19 24.3°C
								2/19 16:19 NO. 8 PH 8.15 24.3°C
								2/19 16:19 NO. 9 PH 7.00 24.3°C
P2000B177 Lot No. 01H151 60°C-KW								2/19 16:20 NO. 10 PH 6.94 24.3°C
本生データは経時的な劣化のおそれがあるため、複写しました。 従って、原本と相違ありません。								2/19 16:21 NO. 11 PH 6.99 24.4°C
'05.05.06 澤 嗣郎								2/19 16:21 NO. 12 PH 6.95 24.4°C
								2/19 16:22 NO. 13 PH 6.90 24.3°C
								2/19 16:22 NO. 14 PH 7.00 24.3°C

プロナック点眼液の安定性試験
Lot No.01H151

試験コード：P2000B177
試験実施者：澤 嗣郎
試験実施日：2001年02月05日

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Water Collec(%)	Initial	present
STD	1	V2-01	2310931					
STD	2	V2-26	2328564					
STD	mean		2319749	1.0095				
A-18	70°C-2W	V2-02	2423977	1.0549	102.90	95.01	7.67	8.6839 8.2978
A-19	70°C-2W	V2-03	2484866	1.0727	101.90	93.95	7.80	8.5682 8.1845
A-20	70°C-2W	V2-04	1889028	0.7350	69.60	64.57	7.22	8.6082 8.2487
A-21	70°C-2W	V2-05	2151512	0.9363	87.55	80.80	7.71	8.5344 8.1581
A-22	70°C-2W	V2-06	1896449	0.8253	80.42	74.55	7.30	8.6129 8.2507
A-23	70°C-2W	V2-07	2197150	0.9581	90.54	83.86	7.38	8.6428 8.2746
A-24	70°C-2W	V2-08	1992556	0.8671	84.08	77.55	7.77	8.4913 8.1152
A-25	70°C-2W	V2-09	2044175	0.8896	89.65	83.08	7.33	8.5906 8.2286
A-18	60°C-2W	V2-10	2301723	1.0017	97.71	94.66	3.12	8.6047 8.4501
A-19	60°C-2W	V2-11	2360197	1.0271	97.57	94.62	3.02	8.6026 8.4530
A-20	60°C-2W	V2-12	1751139	0.7621	72.17	70.12	2.84	8.6548 8.5126
A-21	60°C-2W	V2-13	2140159	0.9313	87.09	84.41	3.08	8.5464 8.3955
A-22	60°C-2W	V2-14	1951236	0.8491	82.73	80.05	3.24	8.5555 8.3965
A-23	60°C-2W	V2-15	2186038	0.9513	90.09	87.26	3.14	8.6875 8.5294
A-24	60°C-2W	V2-16	2007368	0.8736	84.71	82.08	3.10	8.6175 8.4634
A-25	60°C-2W	V2-17	2082273	0.9062	91.32	88.55	3.03	8.6289 8.4780
A-18	50°C-2W	V2-18	2237481	0.9737	94.98	93.44	1.82	8.5642 8.4844
A-19	50°C-2W	V2-19	2345444	1.0207	96.98	95.40	1.61	8.4536 8.3763
A-20	50°C-2W	V2-20	1839698	0.8006	75.81	74.65	1.53	8.5997 8.5240
A-21	50°C-2W	V2-21	2140698	0.9316	87.11	85.74	1.57	8.5511 8.4743
A-22	50°C-2W	V2-22	1963693	0.8546	83.27	81.99	1.54	8.5496 8.4742
A-23	50°C-2W	V2-23	2218206	0.9653	91.41	90.08	1.48	8.7794 8.7043
A-24	50°C-2W	V2-24	2061641	0.8972	87.00	85.73	1.46	8.5244 8.4531
A-25	50°C-2W	V2-25	2080594	0.9054	91.24	89.90	1.47	8.5409 8.4688

計算ミスが原因と認められ訂正した。
計算に必要なデータを記載するため、再編集した。
05.05.06 澤 嗣郎

試験物質名: AHK0282B	試験コード: P2000 B177	試験年月日: 2001年02月05日
試験項目:		試験実施者: 澤 副郎

0.02019 8/27

DOI: + 0.02019 9

条件	PH	重量変化	異物	色
A18 70C-2W	8.13	8.6839 8.2978	- +	黄色
A19	8.15	8.5682 8.1845	- -	黄色
A20	6.95	8.6062 8.2487	+m -	浓黄色
A21	6.90	8.5344 8.1581	+t -	浓黄色
A22	6.96	8.26129 8.2507	+t -	黄色
A23	6.94	8.6428 8.2746	+t -	黄色
A24	6.87	8.4913 8.1152	+t -	浓黄色
A25	6.97	8.5906 8.2286	+t -	浓黄色
A18 60C-2W	8.16	8.6074 8.4501	- -	黄色
A19	8.16	8.6026 8.4530	- -	黄色
A20	6.97	8.6548 8.5126	+t -	浓黄色
A21	6.94	8.5464 8.3955	+t -	浓黄色
A22	6.99	8.5555 8.3965	+t +	黄色
A23	6.97	8.6875 8.5294	+t +	黄色
A24	6.92	8.6175 8.4634	+t +	浓黄色
A25	7.00	8.6289 8.4780	+t +	黄色
A18 50C-2W	8.15	8.5642 8.4844	- -	黄色
A19	8.17	8.4536 8.3763	- -	黄色
A20	6.99	8.5997 8.5240	+t +	浓黄色
A21	6.95	8.5511 8.4743	t +	黄色
A22	7.01	8.5496 8.4742	t +	黄色
A23	6.98	8.7794 8.7043	t t	黄色
A24	6.93	8.5244 8.4531	t +	黄色
A25	6.98	8.5409 8.5468	t +	黄色

2/05 18:39
NO. 90 PH 8.13
22.4°C

2/05 18:40
NO. 91 PH 8.15
22.4°C

2/05 18:40
NO. 92 PH 6.93
22.5°C

2/05 18:42
NO. 93 PH 6.90
22.6°C

2/05 18:42
NO. 94 PH 6.95
22.6°C

2/05 18:43
NO. 95 PH 6.94
22.6°C

2/05 18:44
NO. 96 PH 5.97
22.6°C

2/05 18:45
NO. 97 PH 6.97
22.6°C

2/05 18:46
NO. 98 PH 6.16
22.6°C

2/05 18:47
NO. 99 PH 8.16
22.6°C

2/05 18:48
NO. 90 PH 5.97
22.5°C

2/05 18:49
NO. 91 PH 6.94
22.5°C

2/05 18:49
NO. 92 PH 6.99
22.5°C

2/05 18:50
NO. 93 PH 6.97
22.4°C

2/05 18:51
NO. 94 PH 6.92
22.4°C

2/05 18:52
NO. 95 PH 7.93
22.6°C

2/05 18:52
NO. 96 PH 8.15
22.8°C

2/05 18:54
NO. 97 PH 8.17
22.6°C

2/05 18:56
NO. 98 PH 6.93
22.8°C

2/05 18:57
NO. 99 PH 6.93
22.6°C

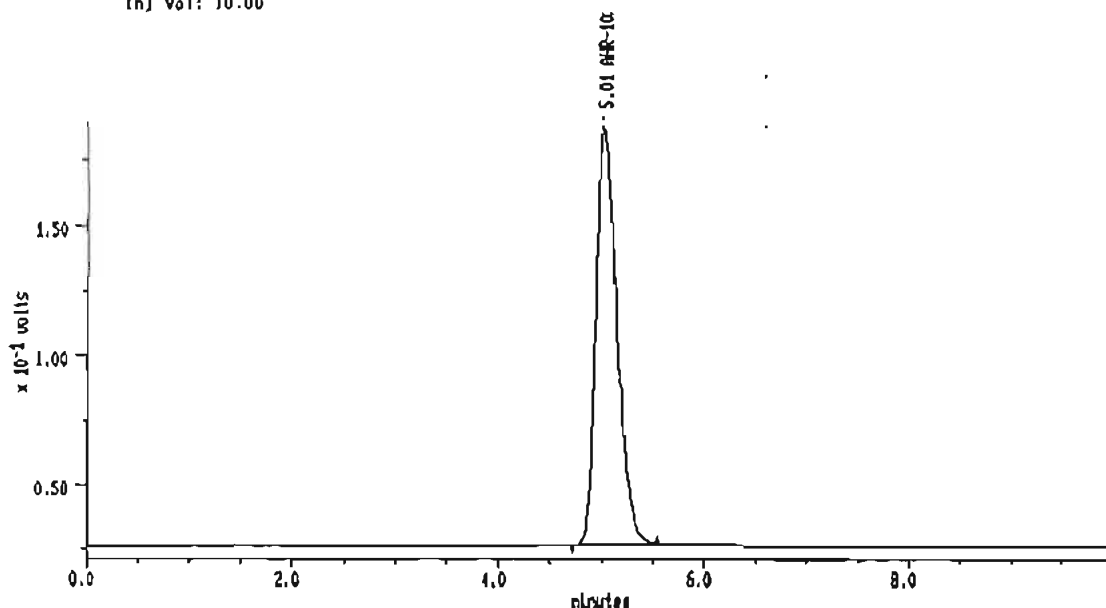
2/05 18:58
NO. 1 PH 7.91
22.6°C

2/05 18:59
NO. 2 PH 6.95
22.6°C

2/05 19:00
NO. 3 PH 5.93
22.5°C

2/05 19:00
NO. 4 PH 6.93
22.4°C

Sample: STD1 Channel: detector 1 Pilenama: V2-01 Chart Speed: Full Size
 Acquired: 05-FEB-101 22:18 Method: B:VAHRY)H15V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 5-FEB-2001 22:29:22

SAMPLE: STD1

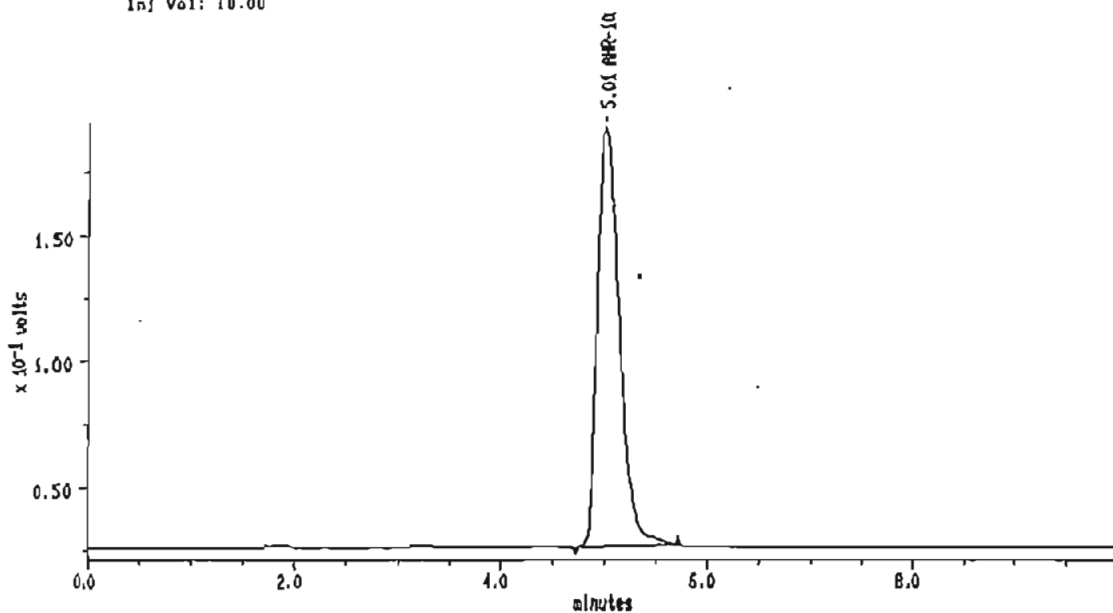
#3 in Method: AHR-10282B
 Acquired: 5-FEB-2001 22:18
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Pilenama: V2-01
 Index: 28
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	DD	2310931	181197	100.00	AHR-10282B
TOTAL			2310931	181197		

Sample: A18 70°C-2W Channel: detector 1 Filename: V2-02 Chart Speed: Full Size
 Acquired: 05-FEB-10 22:30 Method: B:YAHRYIII15V70-2W Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 5-FEB-2001 22:40:38

SAMPLE: A18 70°C-2W

#4 In Method: AHR-10282B

Acquired: 5-FEB-2001 22:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-02

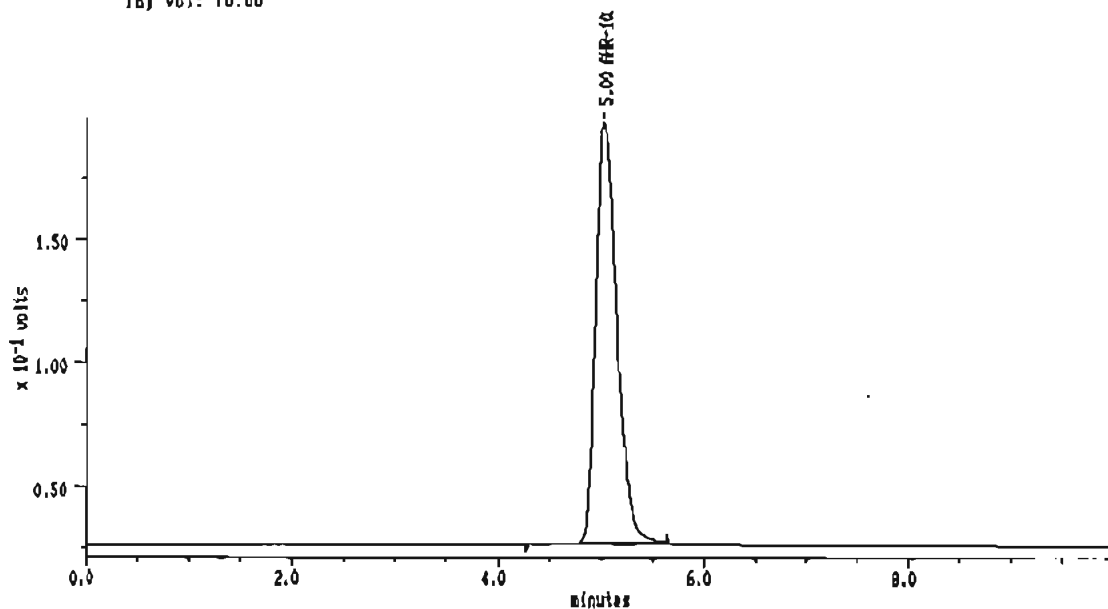
Index: 27

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	2423977	188555	100.00	AHR-10282B
TOTAL			2423977	188566		

Sample: A19 70°C-2W Channel: detector 1 Filename: V2-03 Chart Speed: Full Size
 Acquired: 06-FEB-10 22:41 Method: B:VAHRV1H16V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 6-FEB-2001 22:41:56

SAMPLE: A19 70°C-2W

#5 In Method: AHR-10282B

Acquired: 6-FEB-2001 22:41

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-03

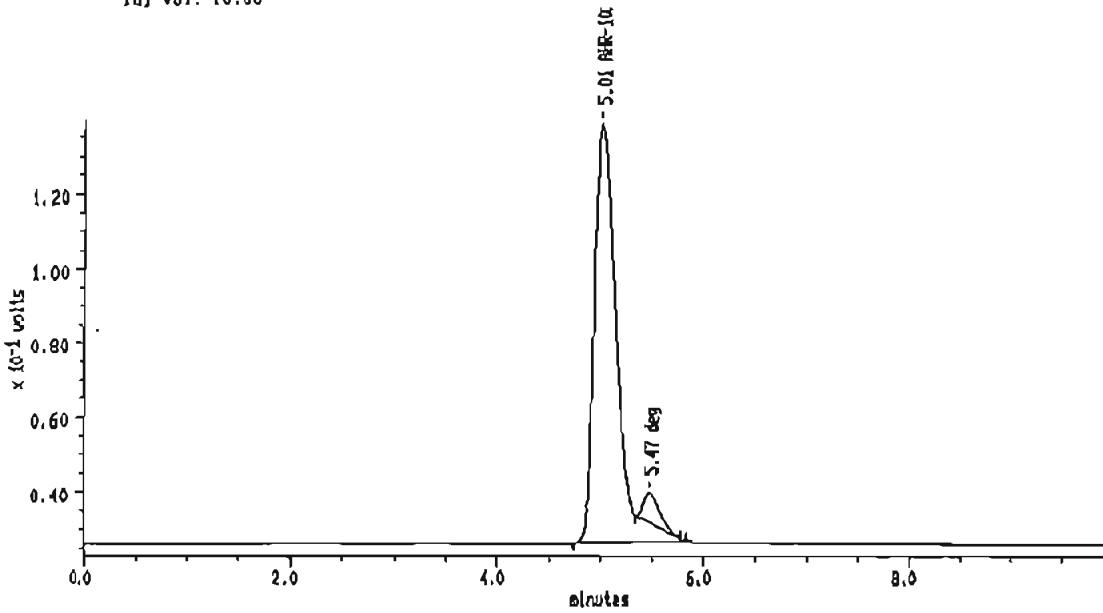
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	2484888	170447	100.00	AHR-10282B
TOTAL			2484888	170447		

Sample: A20 70°C-2W Channel: detector 1 Filename: V2-04 Chart Speed: Full Size
 Acquired: 05-FEB-10 22:52 Method: 0:VAIRY\1116Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 6-FEB-2001 23:03:13

SAMPLE: A20 70°C-2W

#8 In Method: AHR-10282B

Acquired: 6-FEB-2001 22:52

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-04

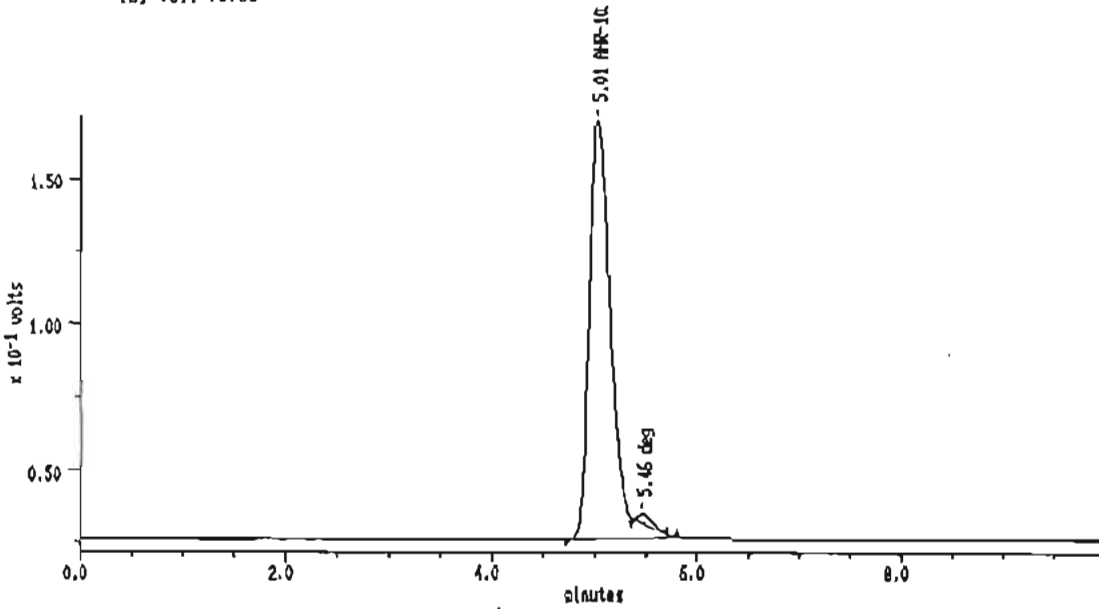
Index: 28

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	SS	1689028	112070	95.04	AHR-10282B
2	5.487	SS	88144	7884	4.98	deg
TOTAL			1777172	119954		

Sample: A21 70°C-2W Channel: detector 1 Filename: V2-05 Chart Speed: Full Size
 Acquired: 05-FEB-101 23:03 Method: B:YAHRYIHI15Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 5-FEB-2001 23:14:29

SAMPLE: A21 70°C-2W

#7 In Method: AHR-10282B

Acquired: 5-FEB-2001 23:03

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-05

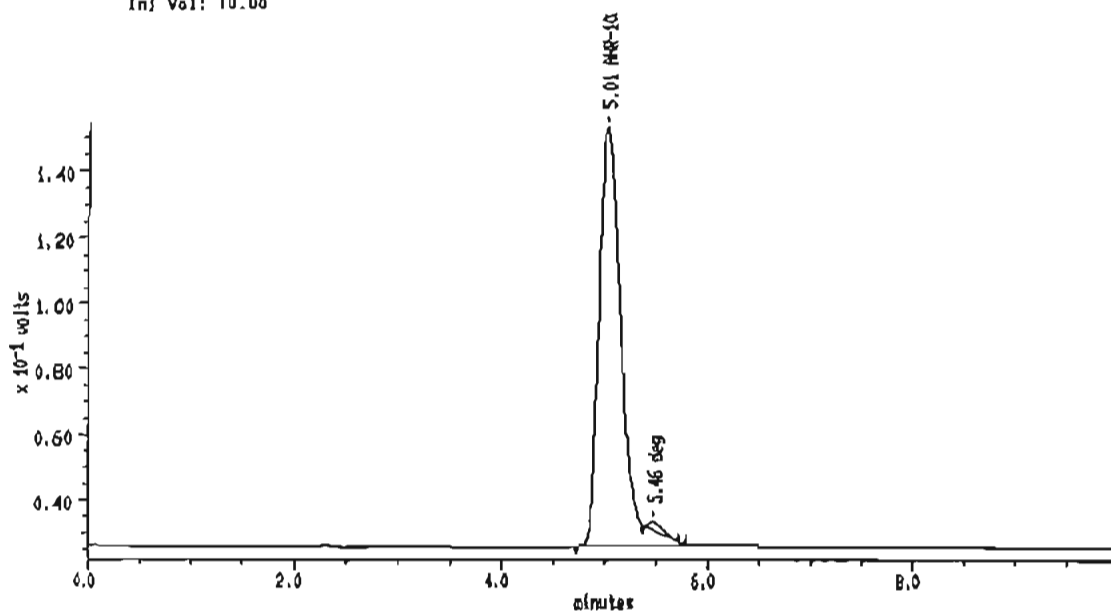
Index: 30

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	6.008	OB	2151612	144143	98.87	AHR-10282B
2	6.450	SS	29083	2828	1.33	deg
TOTAL			2180594	148989		

Sample: A22 70°C-2W Channel: detector 1 Filename: V2-08 Chart Speed: Full Size
 Acquired: 05-FEB-01 23:15 Method: B:YAHKRV1H15V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 6-FEB-2001 23:26:48

SAMPLE: A22 70°C-2W

#8 In Method: AHR-10282B

Acquired: 6-FEB-2001 23:15

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-08

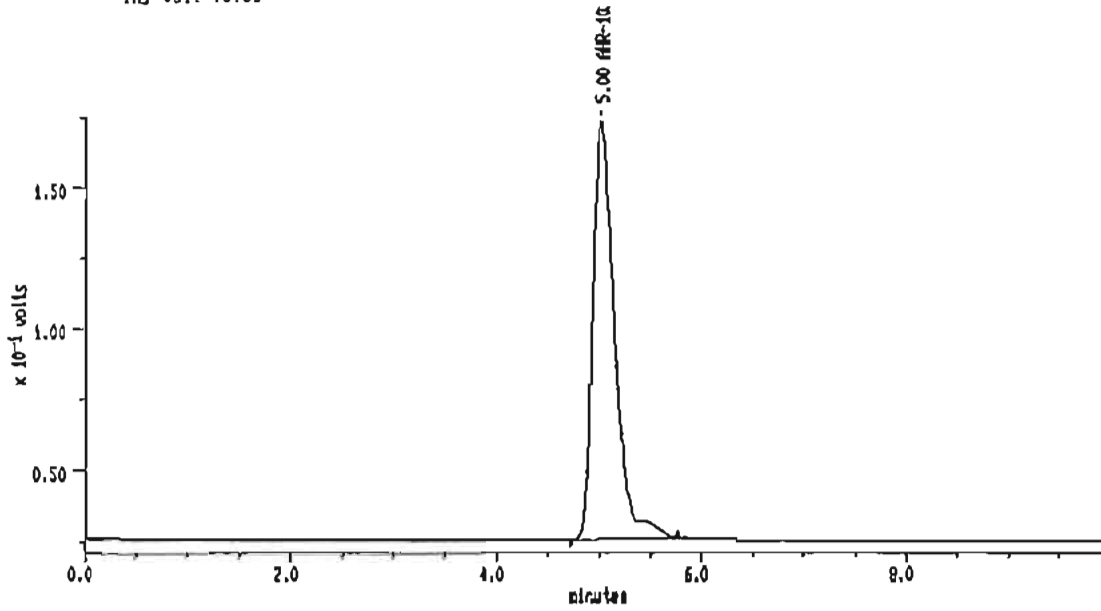
Index: 31

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	1008449	127377	98.71	AHR-102828
2	5.468	SS	24038	2426	1.29	deg
TOTAL			1021287	129802		

Sample: A23 70°C-2W Channel: detector 1 Filename: V2-07 Chart Speed: Full Size
 Acquired: 05-FEB-2001 23:28 Method: B:YHRV1H15Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 6-FEB-2001 23:37:04

SAMPLE: A23 70°C-2W

#9 In Method: AHR-10282B

Acquired: 6-FEB-2001 23:28

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-07

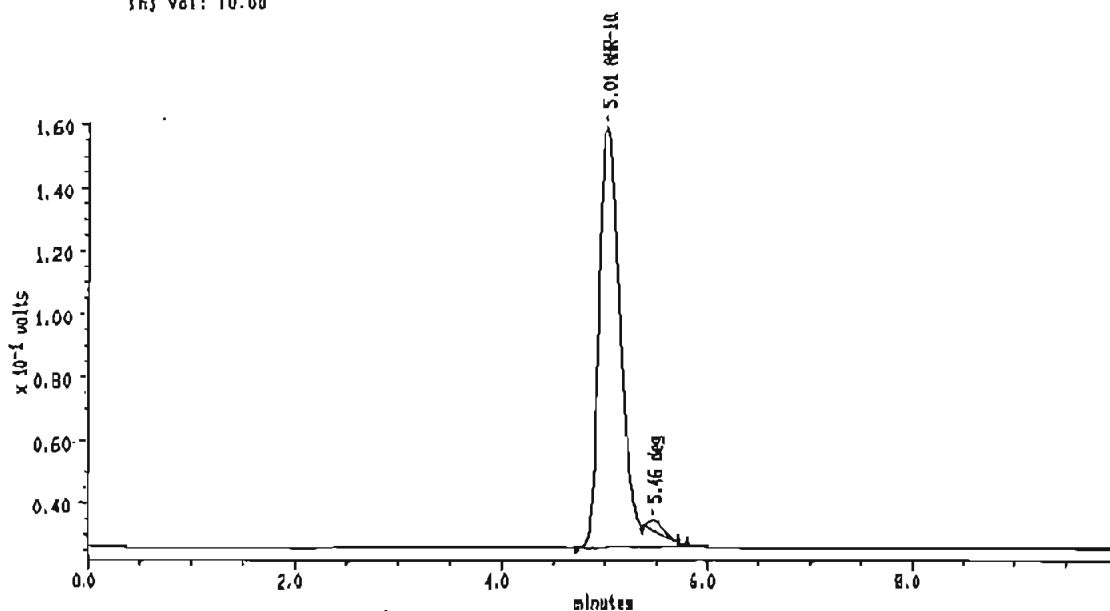
Index: 32

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	2197160	147854	100.00	AHR-10282B
TOTAL			2197160	147854		

Sample: A24 70°C-2W Channel: detector 1 Filename: V2-08 Chart Speed: Full Size
 Acquired: 05-FEB-2001 23:37 Method: B:VAHRYIHI6V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 5-FEB-2001 23:48:28

SAMPLE: A24 70°C-2W

#10 In Method: AHR-10282B

Acquired: 5-FEB-2001 23:37

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-08

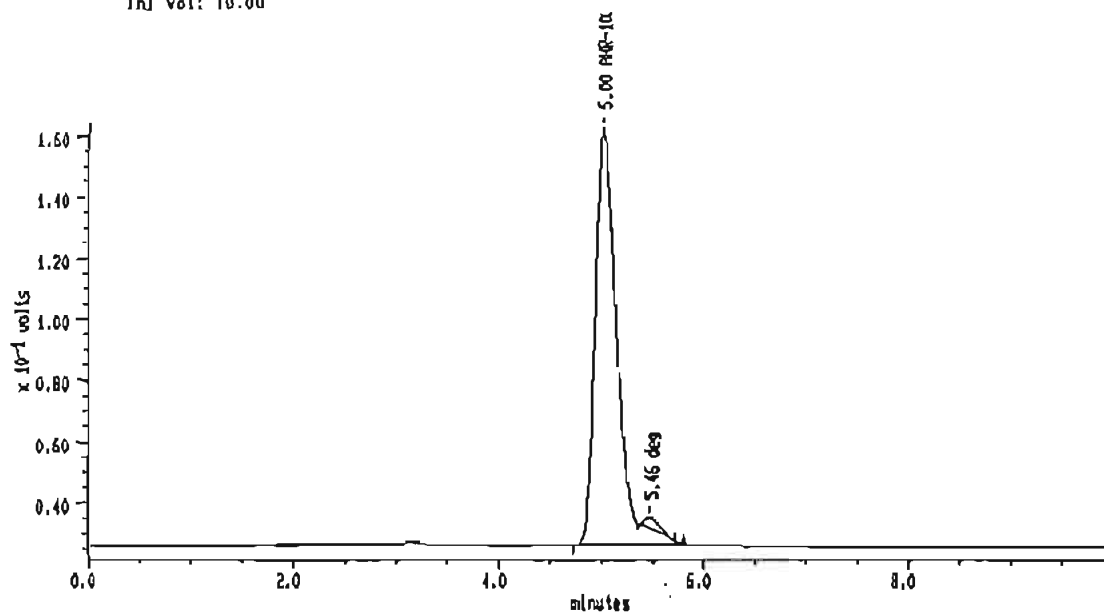
Index: 33

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	1992658	133588	98.40	AHR-10282B
2	5.458	SS	32475	3074	1.60	deg
TOTAL			2025031	136660		

Sample: A26 70°C-2W Channel: detector 1 Filename: V2-09 Chart Speed: Full Size
 Acquired: 05-FEB-101 23:49 Method: B:YAHRY\HLSY70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 5-FEB-2001 23:58:45

SAMPLE1: A26 70°C-2W

#11 in Method: AHR-10282B

Acquired: 5-FEB-2001 23:49

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-09

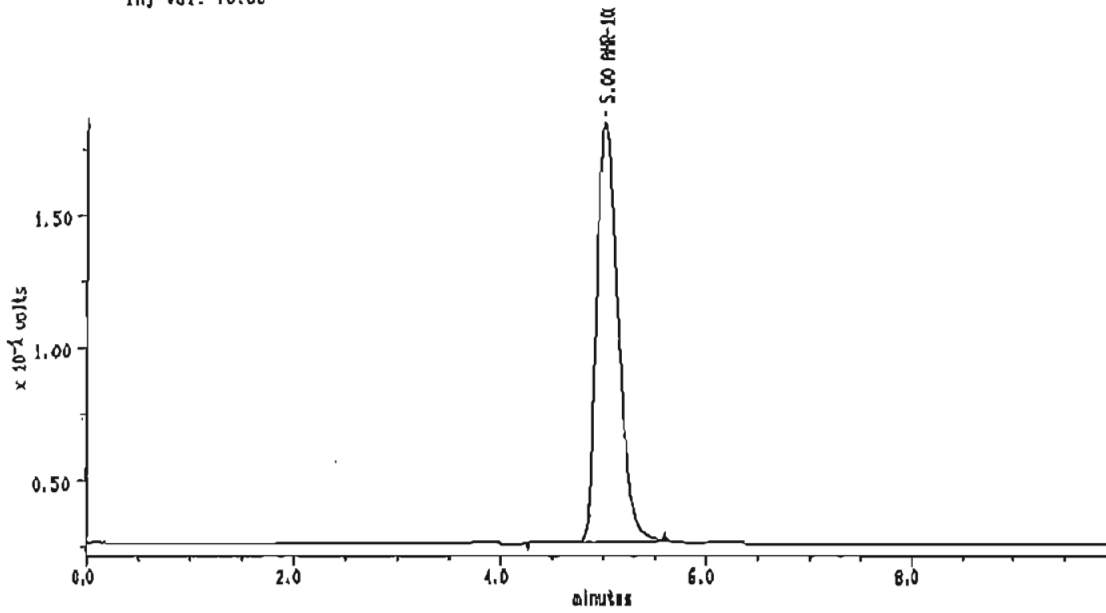
Index: 34

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	DB	2044176	138437	98.15	AHR-10282B
2	5.466	SS	38435	3887	1.85	deg
TOTAL			2082609	140104		

Sample: A18 80°C-2W Channel: detector 1 Filename: V2-10 Chart Speed: Full Size
 Acquired: 08-FEB-2001 0:00 Method: B:YAHRYIII15Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 0:11:02

SAMPLE: A18 80°C-2W

#12 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:00

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-10

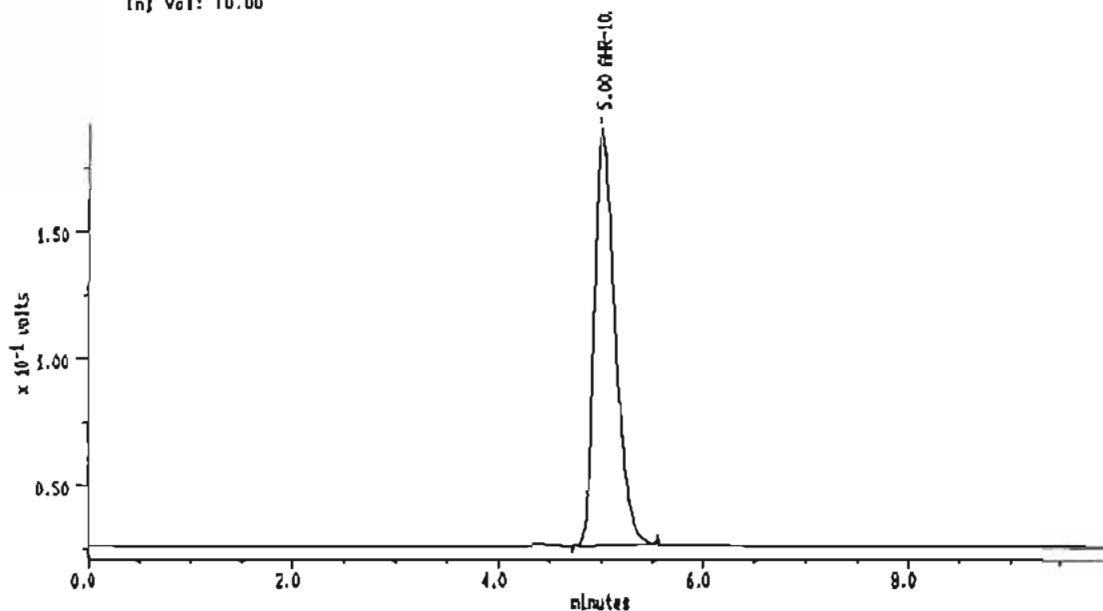
Index: 35

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	2301723	158880	100.00	AHR-10282B
TOTAL			2301723	158880		

Sample: A19 00°C-2W Channel: detector 1 Filename: V2-11 Chart Speed: Full Size
 Acquired: 08-FEB-2001 0:11 Method: B:VAIRY1H15V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 0:22:19

SAMPLE: A19 00°C-2W

#13 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:11

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-11

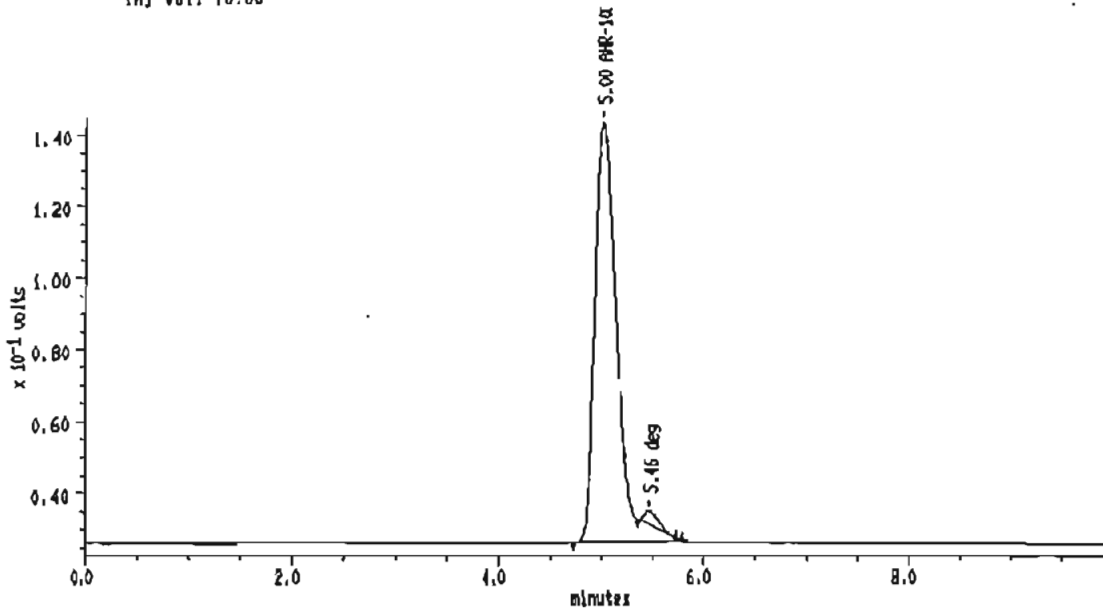
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Compound Name
1	5.000	BB	2380197	183978	100.00	AHR-10282B
TOTAL			2380197	183978		

Sample: A20 80°C-2W Channel: detector 1 Filename: V2-12 Chart Speed: Full Size
 Acquired: 08-FEB-2001 0:23 Method: B:YARRV\HIEV70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 0:33:38

SAMPLE: A20 80°C-2W

#14 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-12

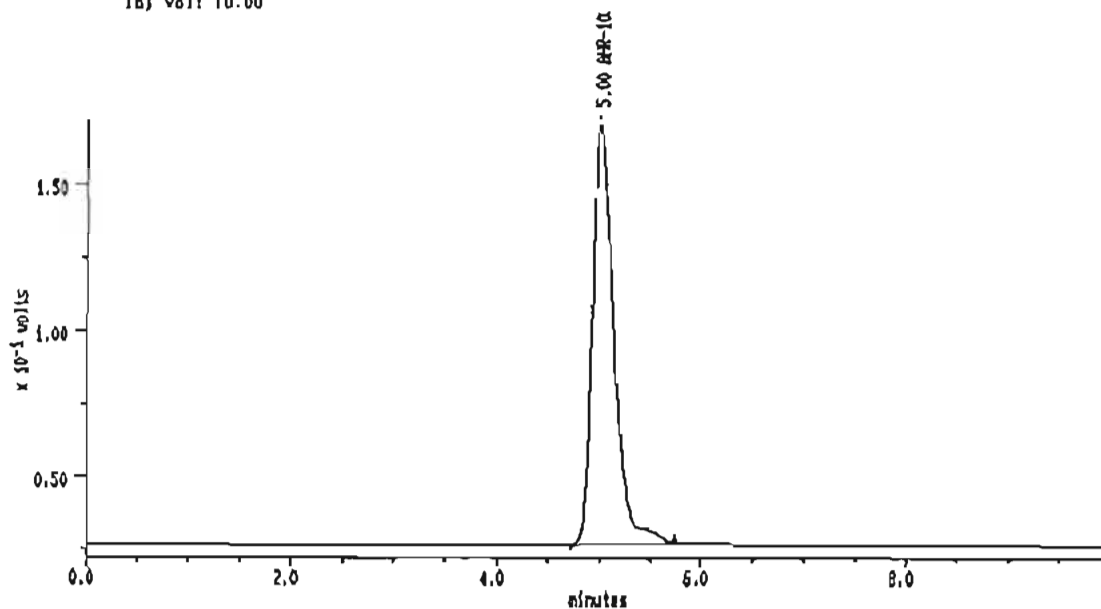
Index: 37

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	DB	1761139	117240	97.88	AHR-10282B
2	5.488	SS	41976	3932	2.34	deg
TOTAL			1793115	121172		

Sample: A21 80°C-2W Channel: detector 1 Filename: Y2-13 Chart Speed: Full Size
 Acquired: 08-FEB-101 0:34 Method: 8:YAHRVJH15V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 0:45:01

SAMPLE: A21 80°C-2W

416 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:34

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: Y2-13

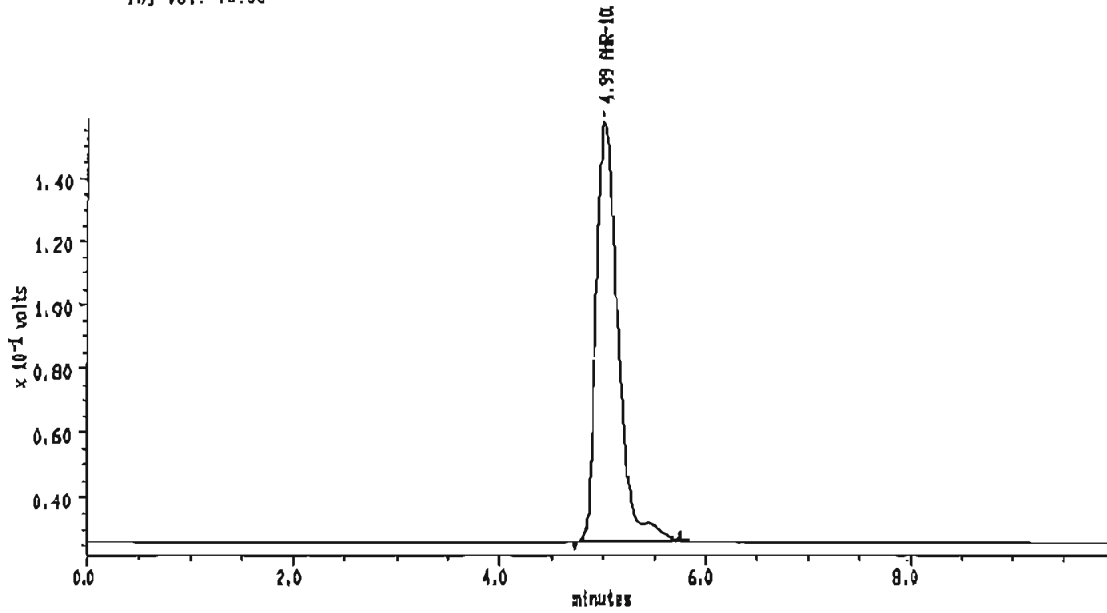
Index: 38

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.000	BB	2140159	143779	100.00	AHR-10282B
TOTAL			2140159	143779		

Sample: A22 60°C-2W Channel: detector 1 Filename: V2-14 Chart Speed: Full Size
 Acquired: 08-FEB-2001 0:45 Method: 8:VAHRV1H16V70-2W Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 0:58:17

SAMPLE: A22 60°C-2W

#18 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:45

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-14

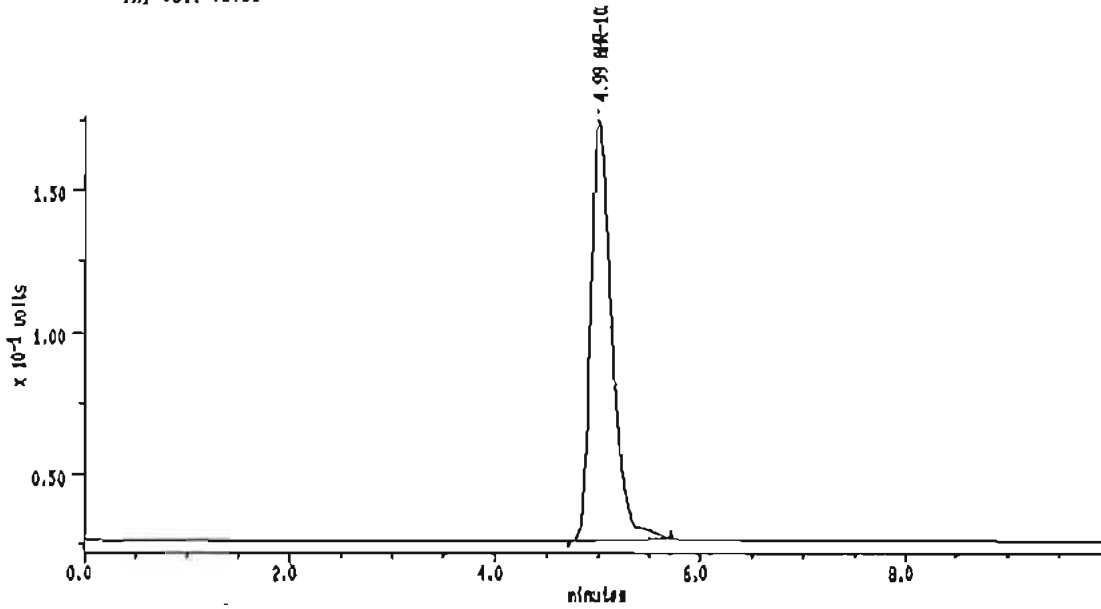
Index: 39

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time [minutes]	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	DB	1951238	131213	100.00	AHR-10282B
TOTAL			1951238	131213		

Sample: A23 80°C-2W Channel: detector 1 Filename: V2-15 Chart Speed: Full Size
 Acquired: 00-FEB-10 0:57 Method: B:YHRV1H15Y70-2R Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 1:07:33

SAMPLE: A23 80°C-2W

#17 In Method: AHR-10282B

Acquired: 8-FEB-2001 0:57

Rate: 2.0 points/min

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-15

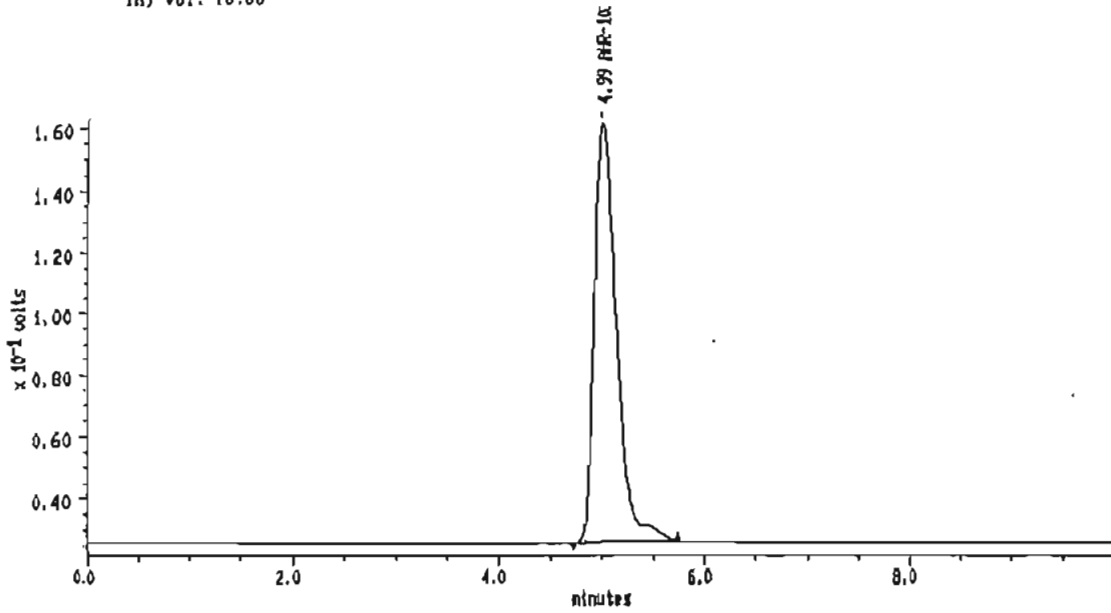
Index: 40

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	BB	2188038	149145	100.00	AHR-10282B
TOTAL			2188038	149145		

Sample: A24 80°C-2W Channel: detector 1 Filename: V2-18 Chart Speed: Full Size
 Acquired: 06-FEB-18 1:08 Method: D:YAHRYIH16V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 1:18:50

SAMPLE: A24 80°C-2W

#18 In Method: AHR-10282B

Acquired: 8-FEB-2001 1:08

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-18

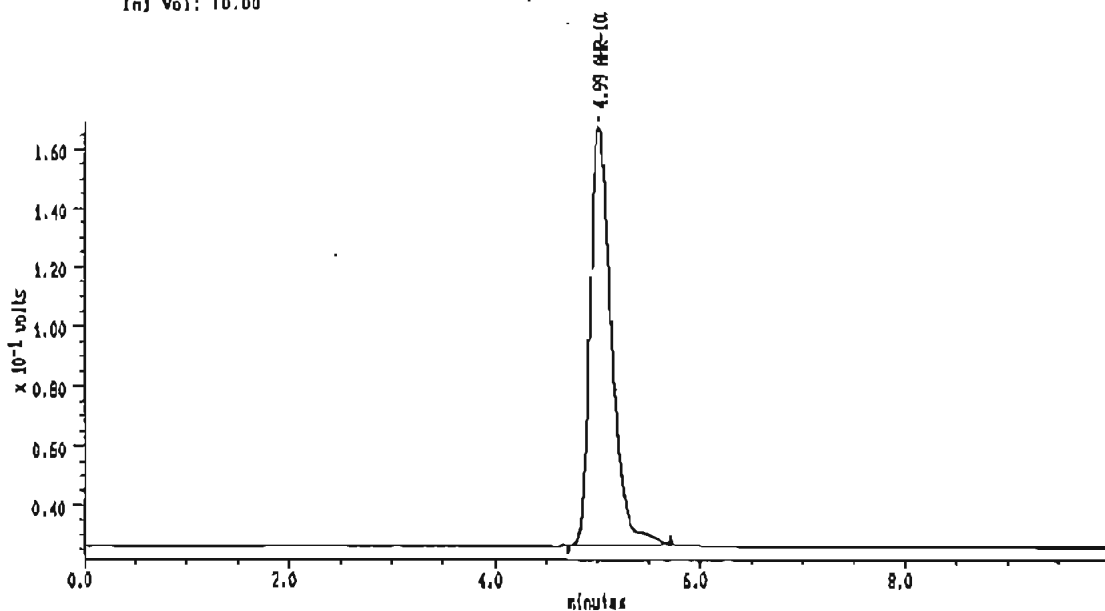
Index: 41

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	BB	2007388	135782	100.00	AHR-10282B
TOTAL			2007388	135782		

Sample: A25 80°C-2W Channel: detector 1 Pileoname: V2-17 Chart Speed: Full Size
 Acquired: 08-FEB-2001 1:19 Method: B:VAHRY11115V70-2W Operator: S.S
 [n] Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 1:30:07

SAMPLE: A25 80°C-2W

#19 In Method: AHR-10282B

Acquired: 8-FEB-2001 1:19

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pileoname: V2-17

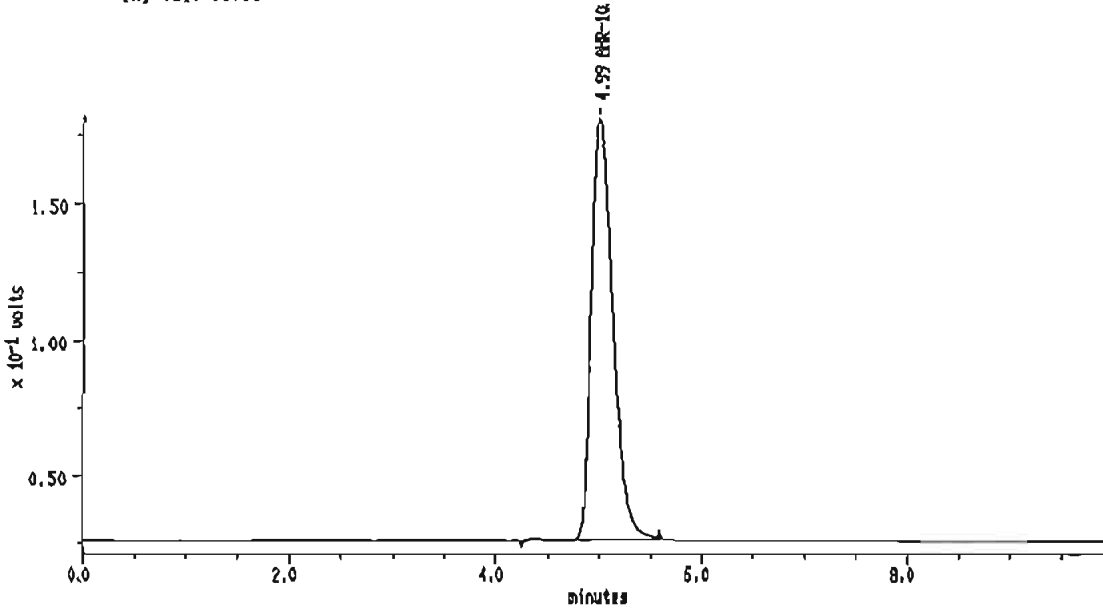
Index: 42

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	BB	2082273	141648	100.00	AHR-10282B
TOTAL			2082273	141648		

Sample: A18 60°C-2W Channel: detector 1 Filament: V2-18 Chart Speed: Full Size
 Acquired: 08-FEB-10 1:30 Method: D:VAIRV1H16Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 1:41:32

SAMPLE: A18 60°C-2W

#20 In Method: AHR-10282B

Acquired: 8-FEB-2001 1:30

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filament: V2-18

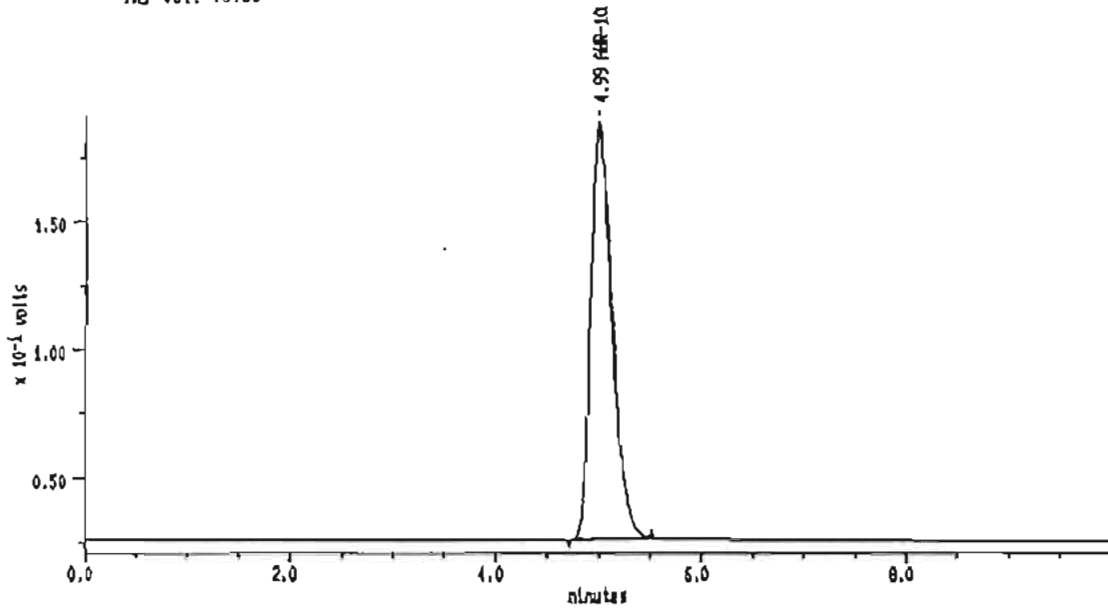
Index: 43

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	BB	2237481	154238	100.00	AHR-10282B
TOTAL			2237481	154238		

Sample: A19 50°C-2W Channel: detector 1 Filename: V2-19 Chart Speed: Full Size
 Acquired: 08-FEB-101 1:42 Method: B:YAHRY11115V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 1:52:49

SAMPLE: A19 50°C-2W

#21 In Method: AHR-10282B

Acquired: 8-FEB-2001 1:42

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-19

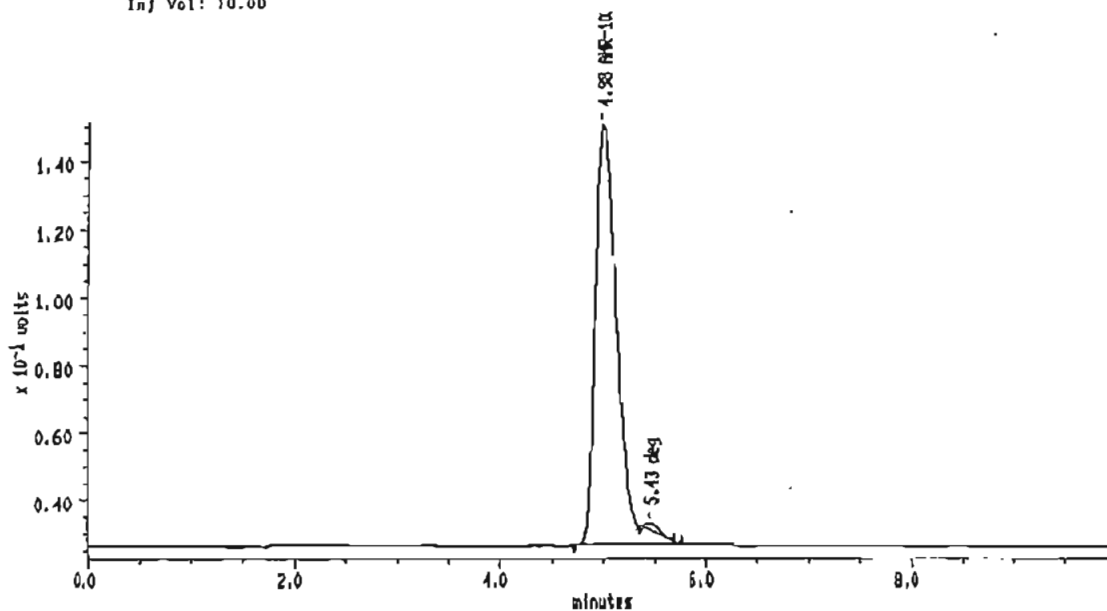
Index: 44

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	DB	2345444	183082	100.00	AHR-10282B
TOTAL			2345444	183082		

Sample: A20 50°C-2W Channel: detector 1 Filename: V2-20 Chart Speed: Full Size
 Acquired: 08-FEB-2001 1:53 Method: D:\YANRVJ\116V70-2R Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 2:04:08

SAMPLE: A20 50°C-2W

#22 In Method: AHR-10282B

Acquired: 8-FEB-2001 1:53

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-20

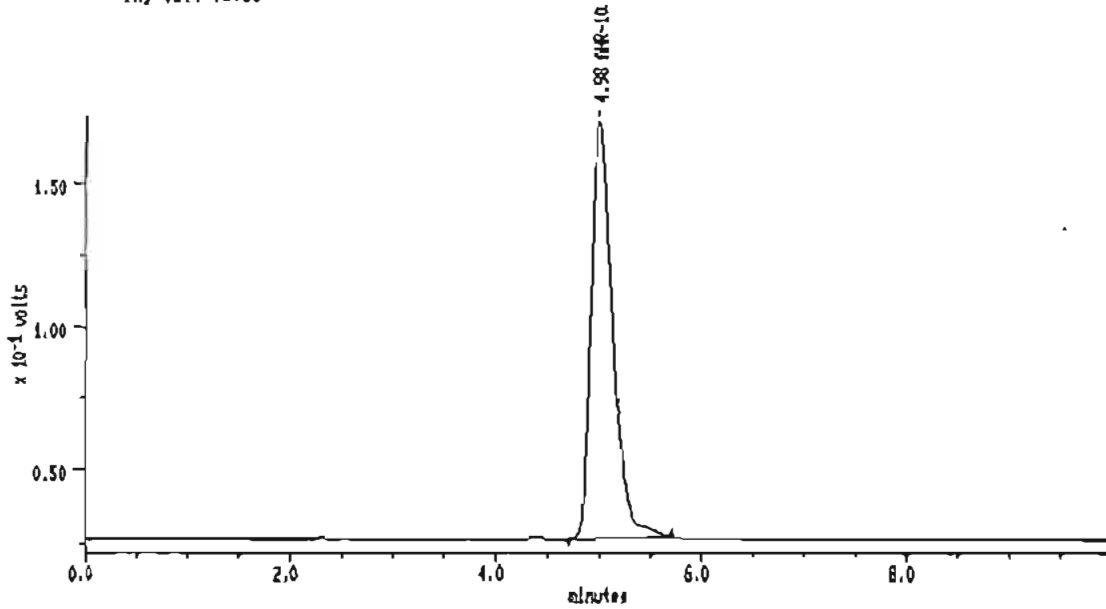
Index: 46

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	BB	1839898	124103	98.82	AHR-10282B
2	5.433	SS	21934	2107	1.18	dog
TOTAL			1861833	126210		

Sample: A21 60°C-2W Channel: detector 1 Filename: V2-21 Chart Speed: Full Size
 Acquired: 08-FEB-101 2:04 Method: B:VAHRV1H1EV70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 2:15:23

SAMPLE: A21 60°C-2W

423 In Method: AHR-10282B

Acquired: 8-FEB-2001 2:04

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-21

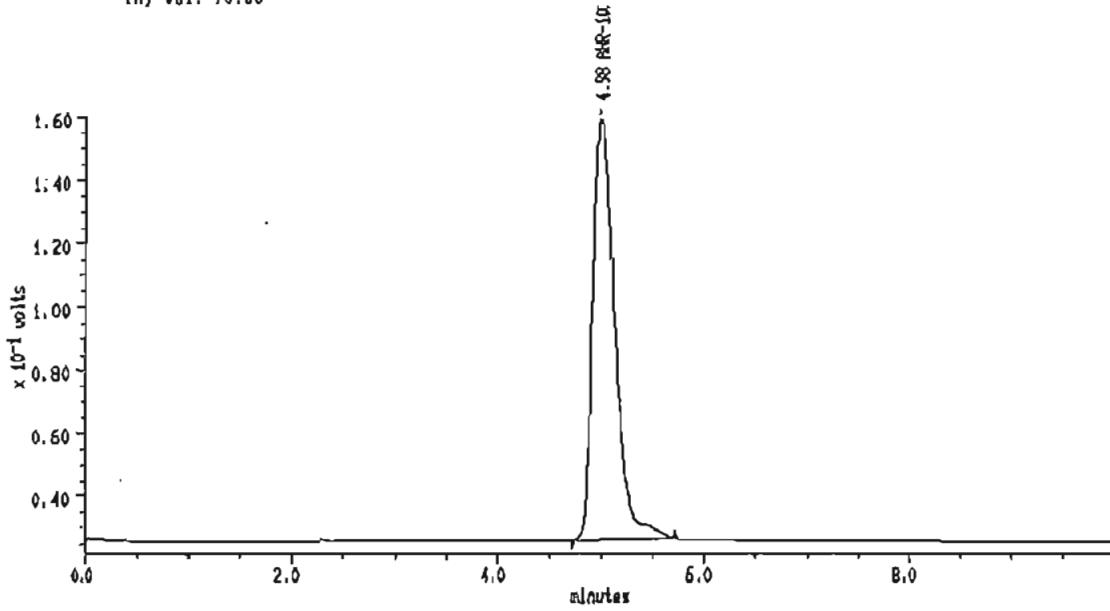
Index: 48

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	BB	2140898	146231	100.00	AHR-10182B
TOTAL			2140898	146231		

Sample: A22 50°C-2W Channel: detector 1 Filename: V2-22 Chart Speed: Full Size
 Acquired: 08-FEB-101 2:18 Method: 8:VAHRV\H16V70-2W Operator: S.S
 [n] Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 2:26:48

SAMPLE: A22 50°C-2W

#24 In Method: AHR-10282B

Acquired: 8-FEB-2001 2:18

Rate: 2.0 points/min

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-22

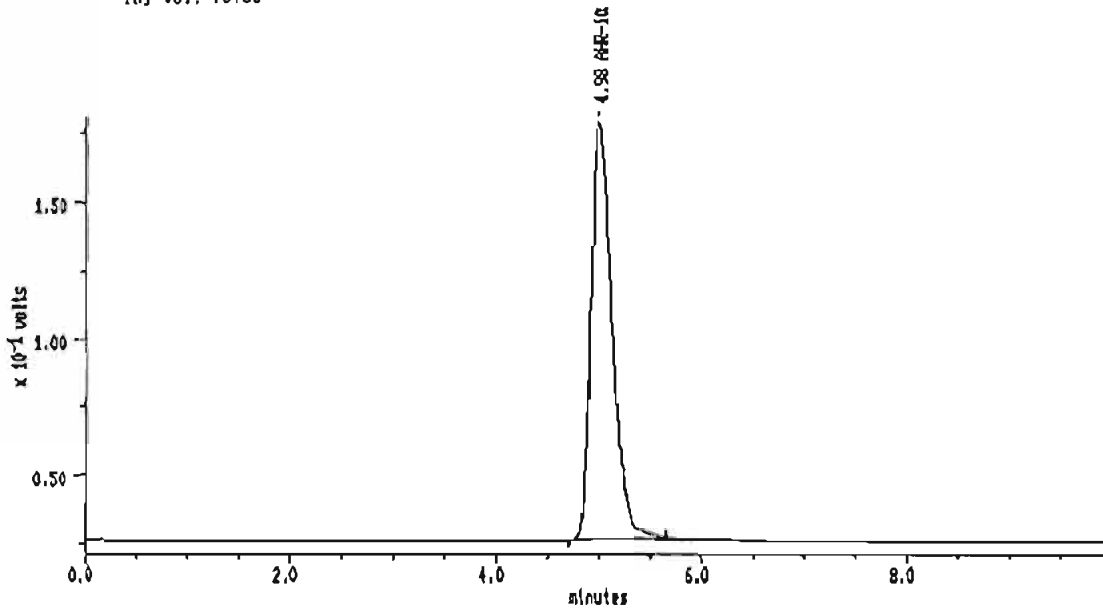
Index: 47

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	DB	1983893	133126	100.00	AHR-10282B
TOTAL			1983893	133126		

Sample: A23 60°C-2W Channel: detector 1 Filename: V2-23 Chart Speed: Full Size
 Acquired: 08-FEB-2001 2:27 Method: B:YAHRY1H16V70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 0-FEB-2001 2:38:13

SAMPLE: A23 60°C-2W

#25 In Method: AHR-10282B

Acquired: 0-FEB-2001 2:27

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-23

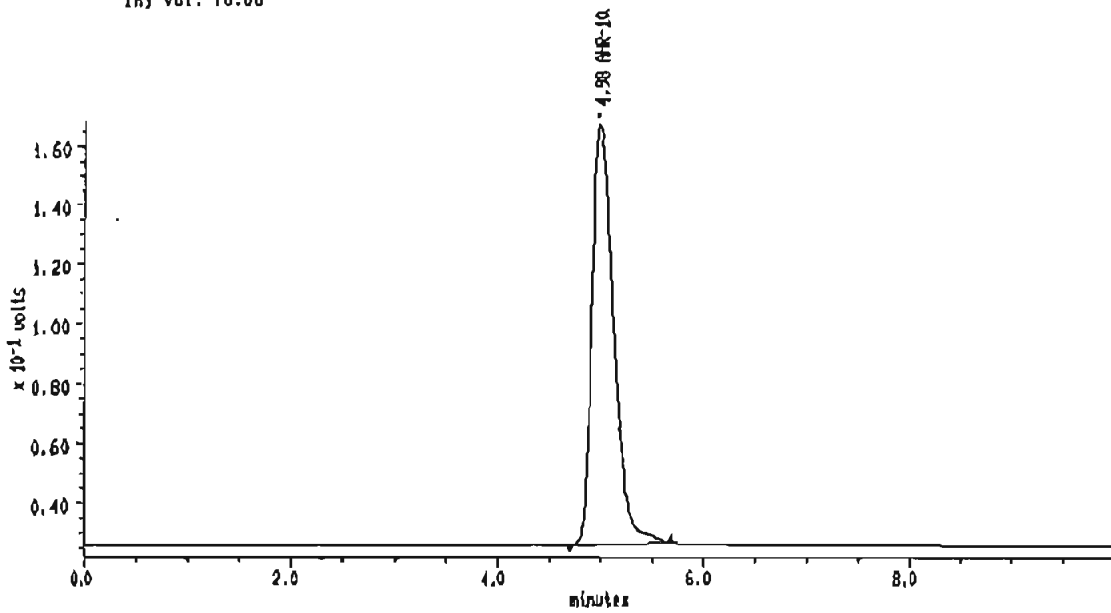
Index: 49

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	00	2218208	152731	100.00	AHR-10282B
TOTAL			2218208	152731		

Sample: A24 50°C-2W Channel: detector 1 Filename: V2-24 Chart Speed: Full Size
 Acquired: 06-FEB-2001 2:39 Method: B:WAHRYIHI15Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 0-FEB-2001 2:49:31

SAMPLE: A24 50°C-2W

#28 in Method: AHR-10282B

Acquired: 0-FEB-2001 2:39

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-24

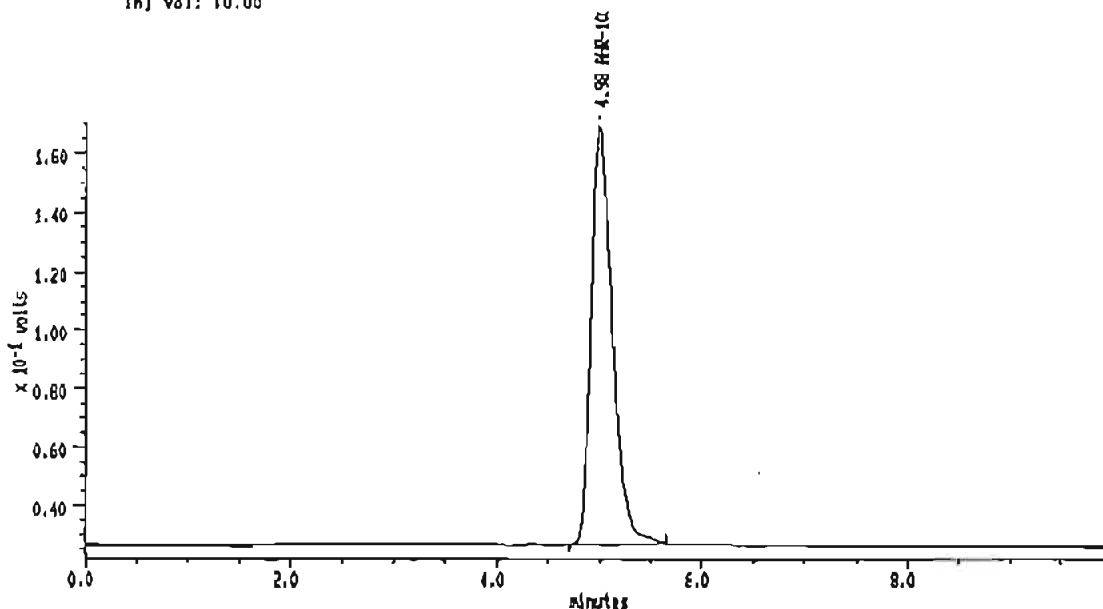
Index: 1

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	DB	2081841	141150	100.00	AHR-10282B
TOTAL			2081841	141150		

Sample: A25 50°C-2# Channel: detector 1 Filename: V2-25 Chart Speed: Full Size
 Acquired: 00-FEB-10 2:50 Method: 8-YAHRV1H15V70-2# Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 3:00:48

SAMPLE: A25 50°C-2#

#27 In Method: AHR-10282B

Acquired: 8-FEB-2001 2:50

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: V2-26

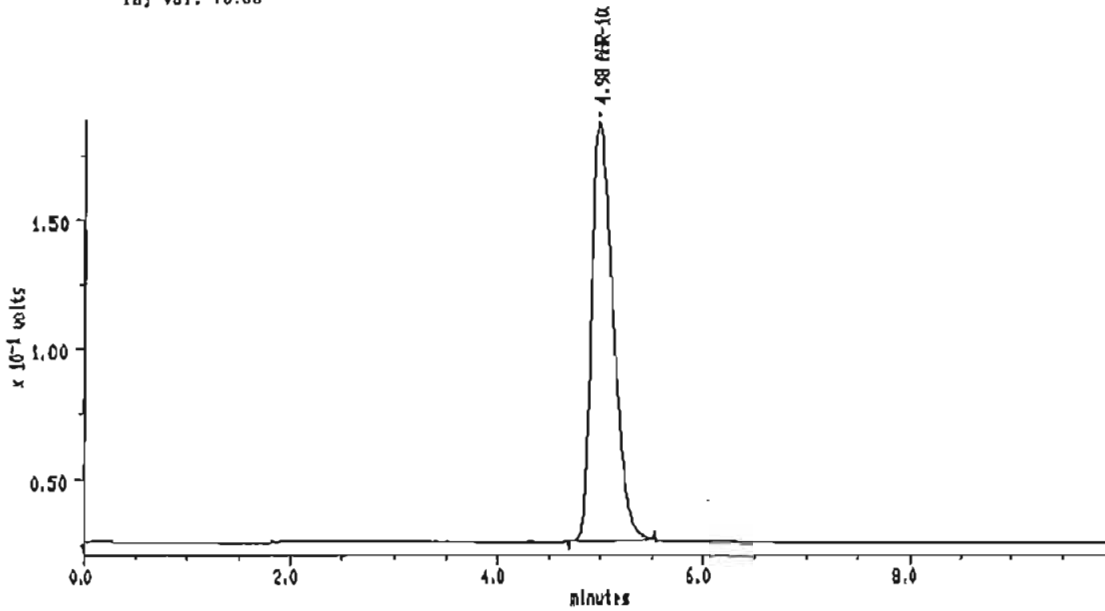
Index: 2

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.983	BB	2080594	142728	100.00	AHR-10282B
TOTAL			2080594	142728		

Sample: STD Channel: detector 1 Filename: V2-28 Chart Speed: Full Size
 Acquired: 08-FEB-101 3:01 Method: B:VAHRYI116Y70-2W Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 8-FEB-2001 3:12:16

SAMPLE: STD

#28 In Method: AHR-10282B
 Acquired: 8-FEB-2001 3:01
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: V2-28
 Index: 3
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.976	BB	2328584	181116	100.00	AHR-10282B
TOTAL			2328584	181116		

プロナック点眼液の安定性試験
Lot No.01H151

試験コード：P2000B177
試験実施者：澤 嗣郎
試験実施日：2001年01月22日

	ID	Chromato No	Peak Area	Conc. (mg/mL)	Remaining (%)	Water Collec(%)	Initial	present
STD	1	IN-01	2217554					
STD	2	IN-26	2251148					
STD	mean		2234351	1.0005				
A-18	Initial	IN-02	2289485	1.0252	100			
A-19	Initial	IN-03	2350952	1.0527	100			
A-20	Initial	IN-04	2358328	1.0560	100			
A-21	Initial	IN-05	2388197	1.0694	100			
A-22	Initial	IN-06	2292061	1.0263	100			
A-23	Initial	IN-07	2358324	1.0560	100			
A-24	Initial	IN-08	2303165	1.0313	100			
A-25	Initial	IN-09	2216054	0.9923	100			
A-18	70°C-1W	IN-10	2242130	1.0040	97.93	94.32	3.69	8.6101 8.4269
A-19	70°C-1W	IN-11	2290529	1.0257	97.44	93.79	3.75	8.5729 8.3884
A-20	70°C-1W	IN-12	1951517	0.8739	82.76	79.57	3.86	8.5196 8.3317
A-21	70°C-1W	IN-13	2203525	0.9867	92.27	88.75	3.82	8.5739 8.3860
A-22	70°C-1W	IN-14	2082220	0.9324	90.85	87.33	3.87	8.5469 8.3574
A-23	70°C-1W	IN-15	2197753	0.9841	93.19	89.75	3.69	8.5791 8.3975
A-24	70°C-1W	IN-16	2095221	0.9382	90.97	87.58	3.73	8.5713 8.3879
A-25	70°C-1W	IN-17	2131377	0.9544	96.18	92.44	3.89	8.5847 8.3735
A-18	60°C-1W	IN-18	2234801	1.0006	97.80	95.88	1.76	8.5329 8.4468
A-19	60°C-1W	IN-19	2288796	1.0249	97.36	95.86	1.54	8.5779 8.5021
A-20	60°C-1W	IN-20	1948910	0.8727	82.64	81.28	1.65	8.5953 8.5137
A-21	60°C-1W	IN-21	2161205	0.9677	90.49	89.07	1.57	8.5548 8.4779
A-22	60°C-1W	IN-22	2091491	0.9365	91.25	89.73	1.67	8.5393 8.4579
A-23	60°C-1W	IN-23	2243646	1.0047	95.14	93.29	1.94	8.5613 8.4661
A-24	60°C-1W	IN-24	2079710	0.9313	90.30	88.84	1.62	8.5863 8.5064
A-25	60°C-1W	IN-25	2086854	0.9345	94.18	92.66	1.61	8.5824 8.4835

計算に必要データは記載済み、再編集して
050506 澤 嗣郎

試験物質名	AHR10282B	試験コード	P2000B/77	ロット番号	01H151	調製年月日	2007年01月15日	試験実施者	澤 嗣郎
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試験題目

製造量	無色77mL×	1本	無色PP5mL×	19本	褐色PP5mL×	本	×	本	×	本	×	本
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処方番号	A-18		A-19		A-20		A-21		A-22		製造メーカー ロット番号
成分及び分量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	
成分名	100 mL	(g)	100 mL	(g)	mL	(g)	mL	(g)	mL	(g)	
77mLエナリウム	0.1	0.100	0.1	0.101	0.1	0.101	0.1	0.100	0.1	0.100	
ホウ酸	1.1	1.099	1.1	1.102	1.5	1.496	1.5	1.502	1.5	1.497	
ホウ砂	1.1	1.103	0.1	1.103	—	—	—	—	—	—	
塩化ベンザルコウム	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	—	—	
ホリソルベ-180	0.15	0.150	—	—	0.15	0.167	—	—	0.15	0.149	
チロキサポール	—	—	0.15	0.153	—	—	0.15	0.152	0.15	0.152	
77mLエナリウム	—	—	—	—	—	—	—	—	0.005	0.005	
pH	8.3	8.23	8.3	8.24	7	7.05	7	7.03	7	7.09	

1/15 15:48
NO.62 PH 8.23
22.5°C

1/15 15:52
NO.63 PH 8.24
22.5°C

1/15 15:55
NO.64 PH 7.05
22.5°C

1/15 16:02
NO.65 PH 7.93
22.5°C

1/15 16:08
NO.66 PH 7.09
22.8°C

1/15 16:12
NO.67 PH 7.08
22.8°C

1/15 16:14
NO.68 PH 6.99
22.8°C

1/15 16:25
NO.69 PH 7.08
23.0°C

試験物質名		試験コード		ロット番号		調製年月日		年		月		日		試験実施者		澤 嗣郎	
試験題目		無色PP5mL×		本褐色PP5mL×		×		×		×		×		×		×	
製造番号		無色PP5mL×		本褐色PP5mL×		×		×		×		×		×		×	
処方番号		A-23		A-24		A-25											
成分及び分量		仕込量 g		仕込量 g		仕込量 g		仕込量 g		仕込量 g		仕込量 g		秤取量 (g)		秤取量 (g)	
成分名		mL		mL		mL		mL		mL		mL		mL		mL	
7-α-エチルチオフラム		0.1	0.101	0.1	0.100	0.1	0.101	0.1	0.100	0.1	0.101	0.1	0.100	0.1	0.101	0.1	0.100
ホウ酸		1.5	1.502	1.5	1.496	1.5	1.501	1.5	1.496	1.5	1.501	1.5	1.496	1.5	1.501	1.5	1.496
ホウ砂		—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
塩化ベンザルコニウム		—	—	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005
ホリソルベート80		—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
チロシオール		0.15	0.152	0.15	0.150	0.15	0.151	0.15	0.150	0.15	0.151	0.15	0.150	0.15	0.151	0.15	0.150
カルコニウムチロシン		0.005	0.005	—	—	—	—	—	—	—	—	—	—	—	—	—	—
Xチルカルカミン		—	—	0.005	0.005	0.5	0.501	0.5	0.501	0.5	0.501	0.5	0.501	0.5	0.501	0.5	0.501
PHI		7	7.08		6.99		7.08		6.99		7.08		6.99		7.08		6.99

試験物質名: AHR10282B	試験コード: P2000B177	試験年月日: 2007年01月22日
試験項目:		試験実施者: 深 桐郎
標準濃度 0.02001g/50ml x 2ml/20ml H1		
PH initial 測定時 濁り 異物 色		
A18 initial 8.23	- - 藍色	22-Jan-2001 16:48:12
A19 8.22	- -	002: + 0.02001 g
A20 87.02	- -	
A21 6.98	- -	
A22 7.05	- -	
A23 6.99	- -	
A24 6.93	- -	
A25 7.00	- -	
A18 70c/w 8.16 8.6101 8.4269	- ±	
A19 8.17 8.5729 8.3884	- ± ↓	
A20 6.97 8.5196 8.3317	- ++(+) 深藍色	
A21 6.95 8.5739 8.3860	- ++(+) 深藍色	
A22 7.07 8.5469 8.3574	- ++(+) 深藍色	
A23 7.05 8.5791 8.3975	+ +(+) 黄色	
A34 6.99 8.5713 8.3879	- ++(+) 深黄色	
A25 7.07 8.5647 8.3735	+ + 黄色	
A18 60c/w 8.27 8.5329 8.4468	- -	
A19 8.25 8.5779 8.5021	- - ↓	
A20 7.05 8.5453 8.5137	+ +(+) 深黄色	
A21 7.03 8.5548 8.4719	+ ± ↓	
A22 7.08 8.5393 8.4579	+ + 黄色	
A23 7.07 8.5613 8.4661	+ ± ↓	
A24 6.96 8.5863 8.5064	+ ± 深黄色	
A25 7.08 8.5624 8.4835	+ ± 黄色	

1/22 17:47
NO. 9 PH 8.25
24.3°C

1/22 17:47
NO. 10 PH 8.22
24.3°C

1/22 17:48
NO. 11 PH 7.92
24.2°C

1/22 17:49
NO. 12 PH 8.98
24.3°C

1/22 17:50
NO. 13 PH 7.95
24.3°C

1/22 17:50
NO. 14 PH 6.99
24.4°C

1/22 17:51
NO. 15 PH 6.97
24.4°C

1/22 17:52
NO. 16 PH 7.99
24.4°C

1/22 17:53
NO. 17 PH 9.16
24.4°C

1/22 17:54
NO. 18 PH 8.17
24.4°C

1/22 17:55
NO. 19 PH 8.97
24.4°C

1/22 17:55
NO. 20 PH 6.95
24.5°C

1/22 17:58
NO. 21 PH 7.97
24.4°C

1/22 18:00
NO. 22 PH 7.95
24.4°C

1/22 18:02
NO. 23 PH 6.99
24.4°C

1/22 18:05
NO. 24 PH 7.97
24.5°C

1/22 18:07
NO. 25 PH 8.27
24.4°C

1/22 18:09
NO. 26 PH 8.25
24.3°C

1/22 18:10
NO. 27 PH 7.95
24.3°C

1/22 18:12
NO. 28 PH 7.93
24.3°C

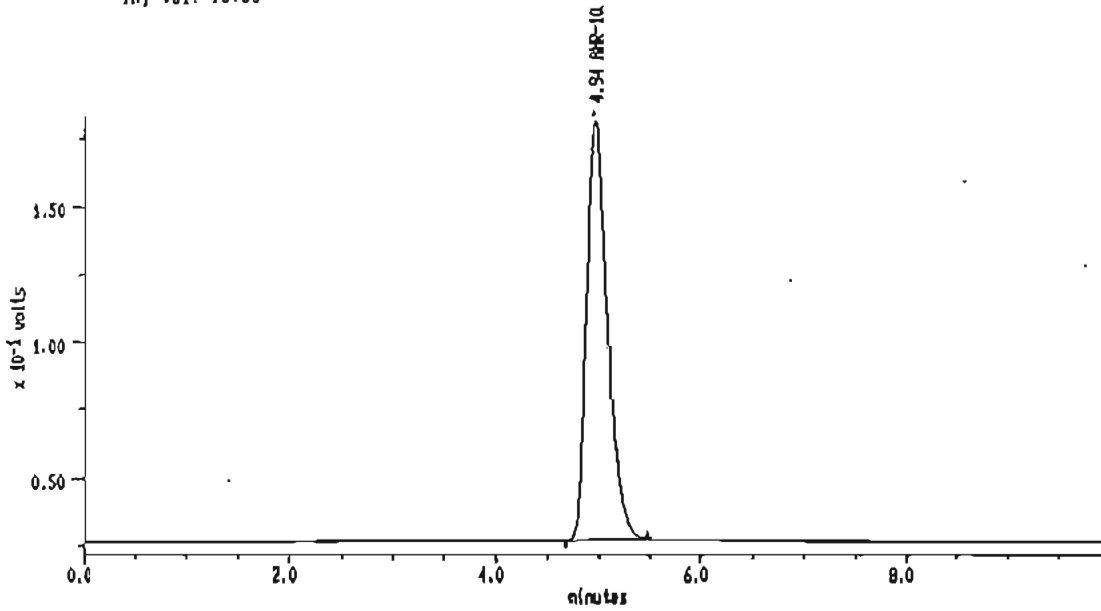
1/22 18:16
NO. 29 PH 7.93
24.3°C

1/22 18:19
NO. 30 PH 7.97
24.2°C

1/22 18:19
NO. 31 PH 6.96
24.2°C

1/22 18:21
NO. 32 PH 7.98
24.2°C

Sample: STD1 Channel: detector 1 Filename: 1R-01 Chart Speed: Full Size
 Acquired: 22-JAN-2001 19:58 Method: B:YAHRYIIIISYIKITJAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 20:08:27

SAMPLE: STD1

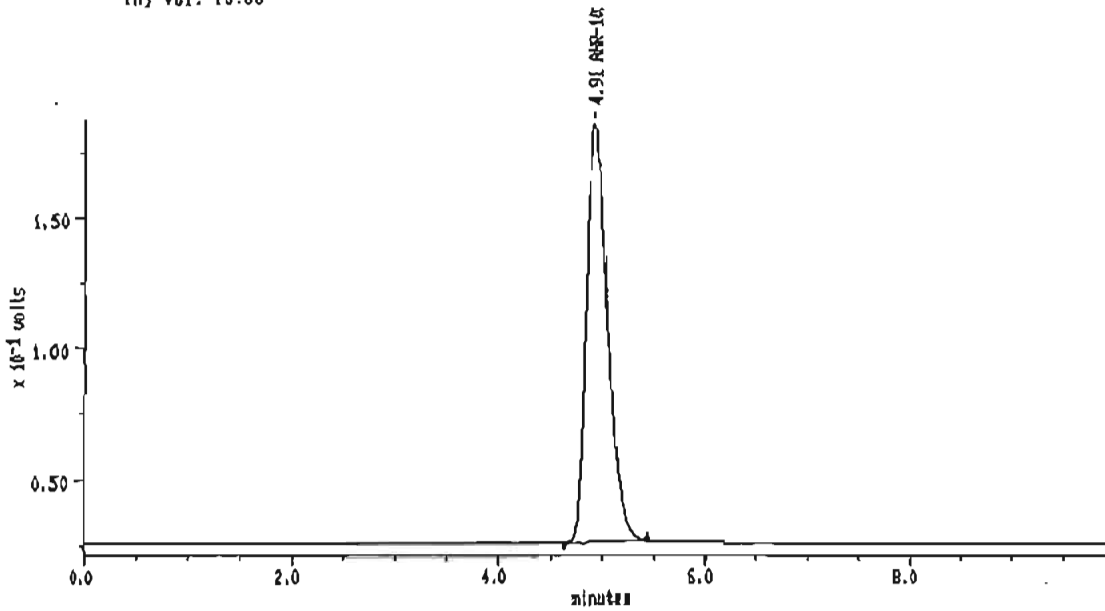
#3 In Method: AHR-10282B
 Acquired: 22-JAN-2001 19:58
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filename: 1R-01
 Index: 1
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.942	BS	2217554	164568	100.00	AHR-10282B
TOTAL			2217554	164568		

Sample: A10 INITIAL Channel: detector 1 Filename: IN-02 Chart Speed: Full Size
 Acquired: 22-JAN-10 20:12 Method: D:YAHRY\H15V\INITIAL Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1990 Dynalco Solutions, Division of Millipore

MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 20:22:53

SAMPLE# A10 INITIAL

#4 In Method: AHR-10282B

Acquired: 22-JAN-2001 20:12

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-02

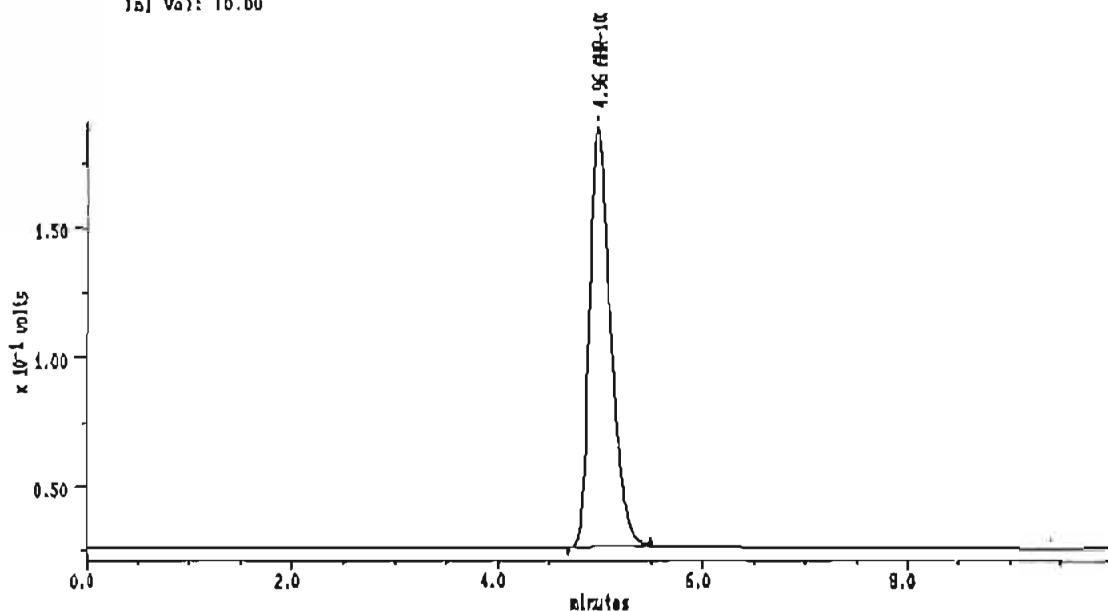
Index: 2

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.908	BB	2289486	169801	100.00	AHR-10282B
TOTAL			2289486	169801		

Sample: A19 INITIAL Channel: detector 1 Filename: IN-03 Chart Speed: Full Size
 Acquired: 22-JAN-2001 20:23 Method: B:VAHRYIHHI6VINI1IAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 20:34:10

SAMPLE: A19 INITIAL

#5 In Method: AHR-10282B

Acquired: 22-JAN-2001 20:23

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-03

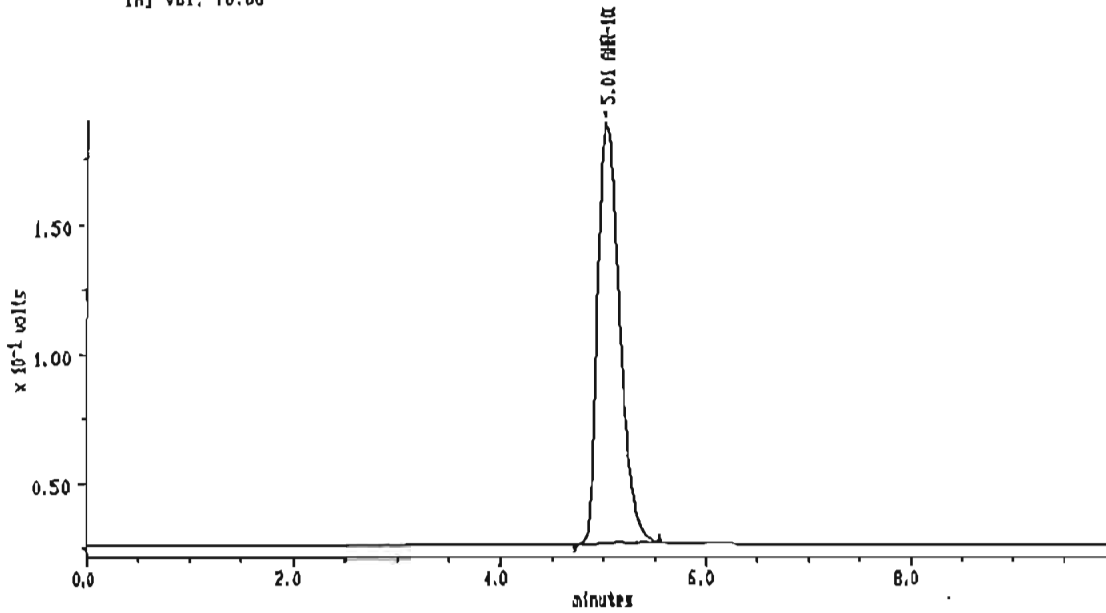
Index: 3

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.968	DB	2360952	182814	100.00	AHR-10282B
TOTAL			2360952	182814		

Sample: A20 INITIAL Channel: detector 1 Filename: IN-04 Chart Speed: Full Size
 Acquired: 22-JAN-2001 20:34 Method: B:VAHRV(H)IEV(INITIAL) Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 20:46:27

SAMPLE: A20 INITIAL

#8 In Method: AHR-10282B

Acquired: 22-JAN-2001 20:34

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-04

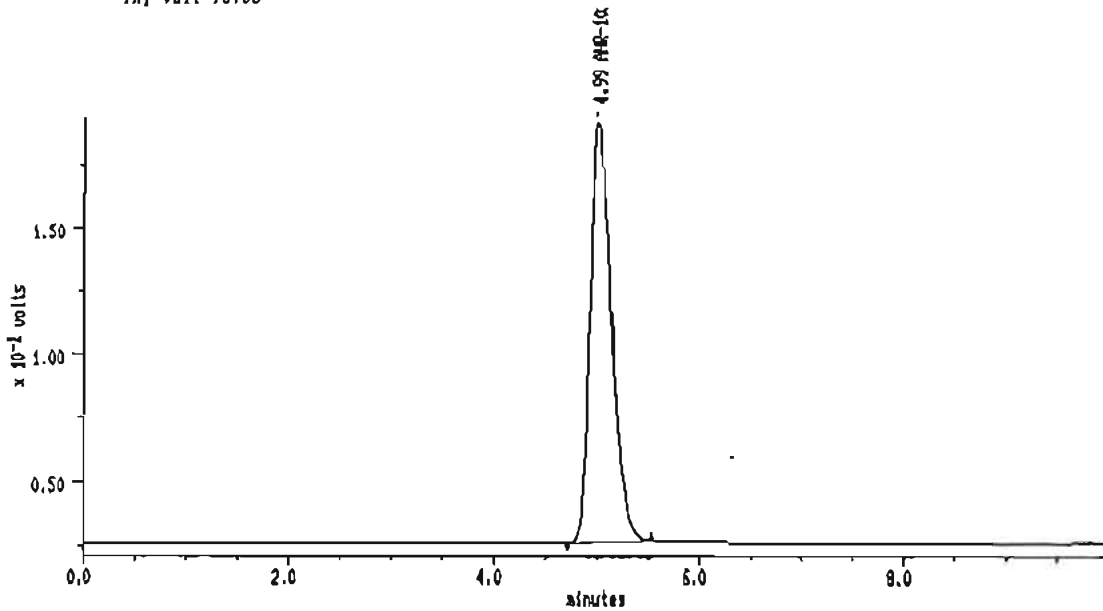
Index: 4

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BD	2368328	181923	100.00	AHR-10282B
TOTAL			2368328	181923		

Sample: A21 INITIAL Channel: detector 1 Filename: IN-05 Chart Speed: Full Size
 Acquired: 22-JAN-101 20:48 Method: B:YASRV\HIBYINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 20:58:42

SAMPLE: A21 INITIAL

#7 In Method: AHR-10282B

Acquired: 22-JAN-2001 20:48

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-05

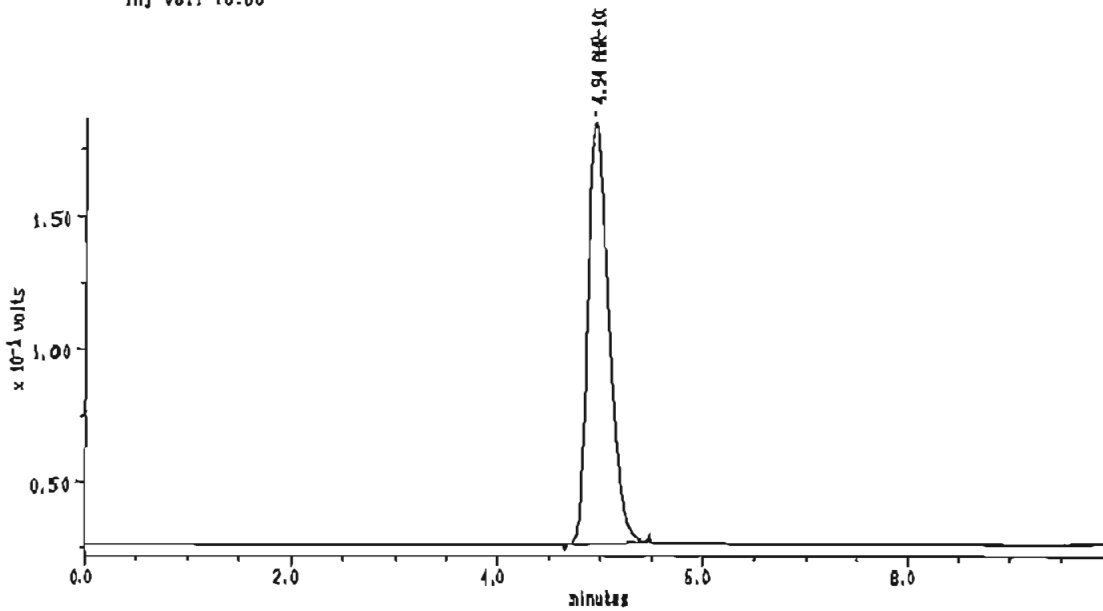
Index: 5

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	B0	2388197	185329	100.00	AHR-10282B
TOTAL			2388197	185329		

Sample: A22 (N)TJAL Channel: detector 1 Filename: IN-08 Chart Speed: Full Size
 Acquired: 22-JAN-101 20:57 Method: B:VAHRVJIII6YINITJAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 21:07:58

SAMPLE: A22 INITIAL

#0 in Method: AHR-10282B

Acquired: 22-JAN-2001 20:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-08

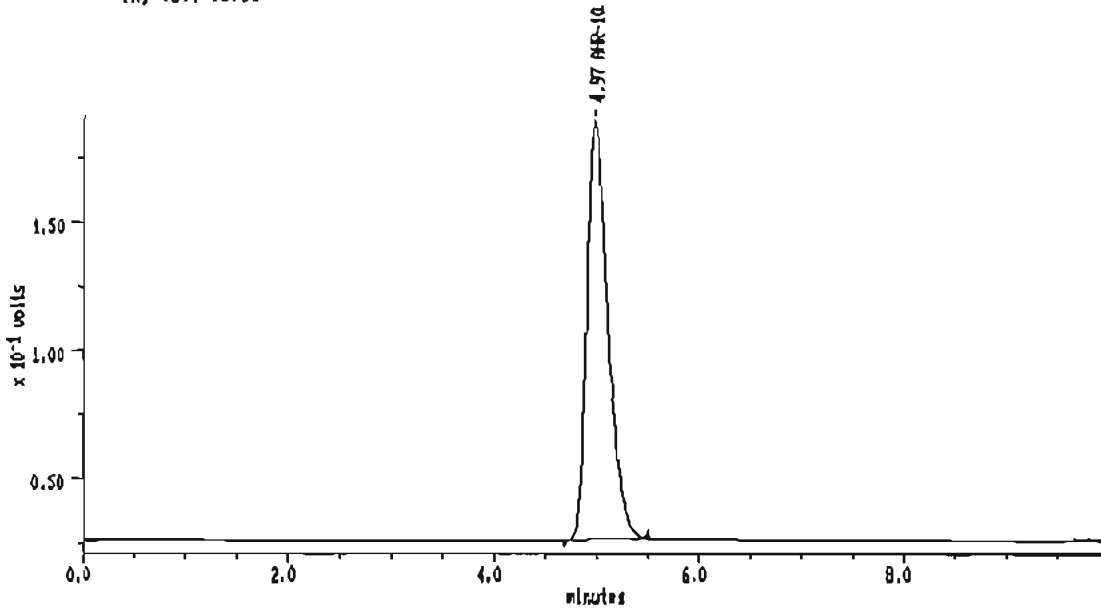
Index: 8

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.942	BB	2292081	168726	100.00	AHR-10282B
TOTAL			2292081	168726		

Sample: A23 INITIAL Channel: detector 1 Filename: IN-07 Chart Speed: Full Size
 Acquired: 22-JAN-01 21:08 Method: 8:YAHBYIH16VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 21:18:16

SAMPLE: A23 INITIAL

#9 In Method: AHR-10282B

Acquired: 22-JAN-2001 21:08

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-07

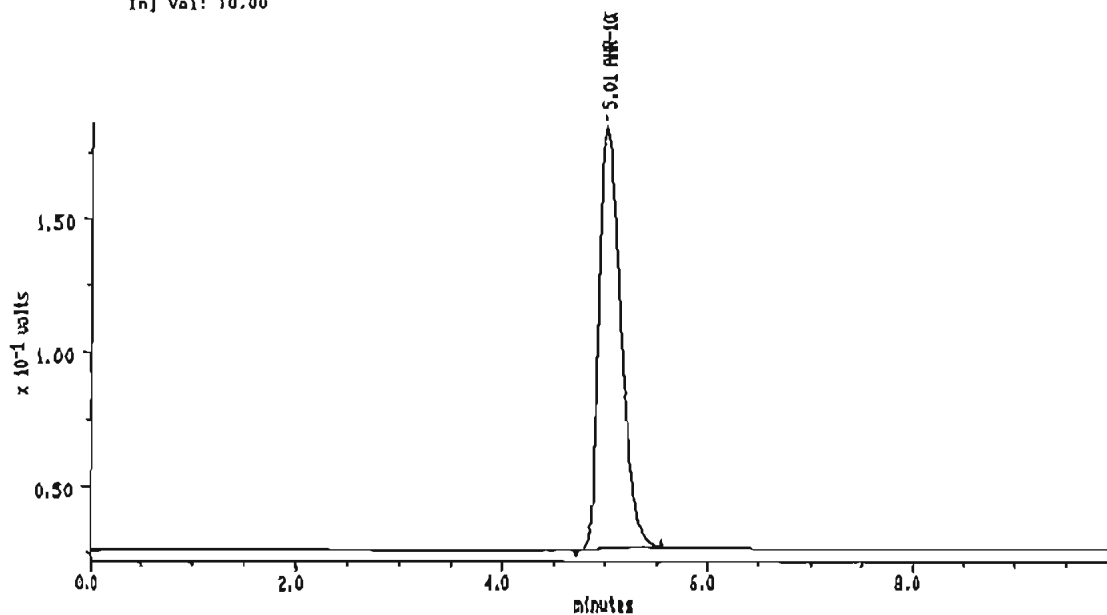
Index: 7

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.967	BB	2358324	182394	100.00	AHR-10282B
TOTAL			2358324	182394		

Sample: A24 INITIAL Channel: detector 1 Filename: IN-08 Chart Speed: Full Size
 Acquired: 22-JAN-101 21:20 Method: D:YAHRYIH18YINITIAL Operator: S.S
 Inj Val: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 21:30:02

SAMPLE: A24 INITIAL

#10 In Method: AHR-10282B

Acquired: 22-JAN-2001 21:20

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-08

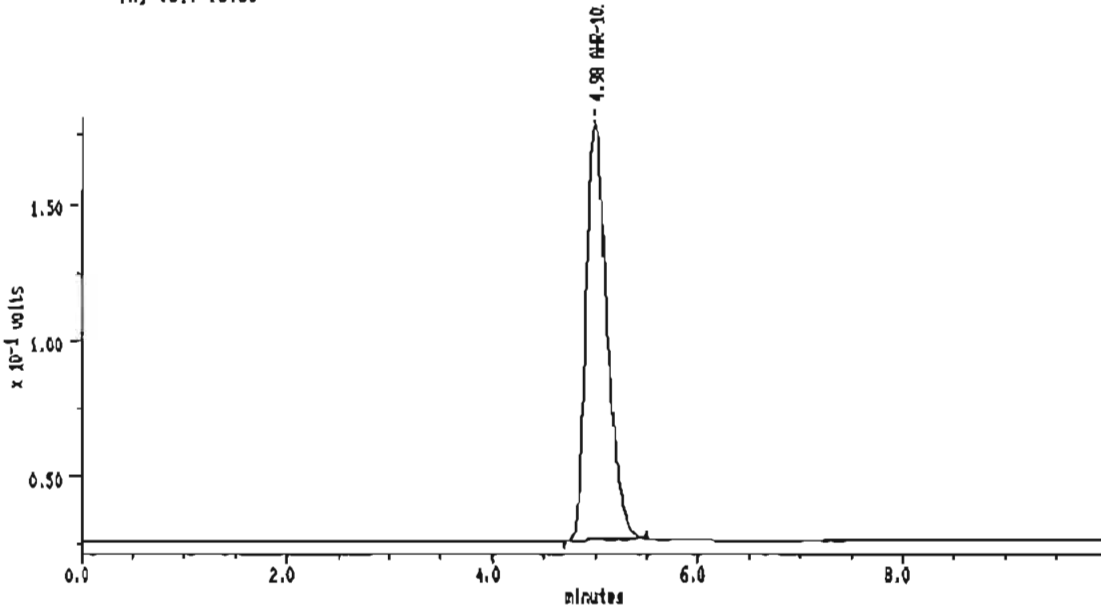
Index: 8

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	2303185	158466	100.00	AHR-10282B
TOTAL			2303185	158466		

Sample: A26 INITIAL Channel: detector 1 Filename: IN-09 Chart Speed: Full Size
 Acquired: 22-JAN-01 21:31 Method: B:YHRVIBISVINIJIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 21:41:49

SAMPLE: A26 INITIAL

File In Method: AHR-10282B

Acquired: 22-JAN-2001 21:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-09

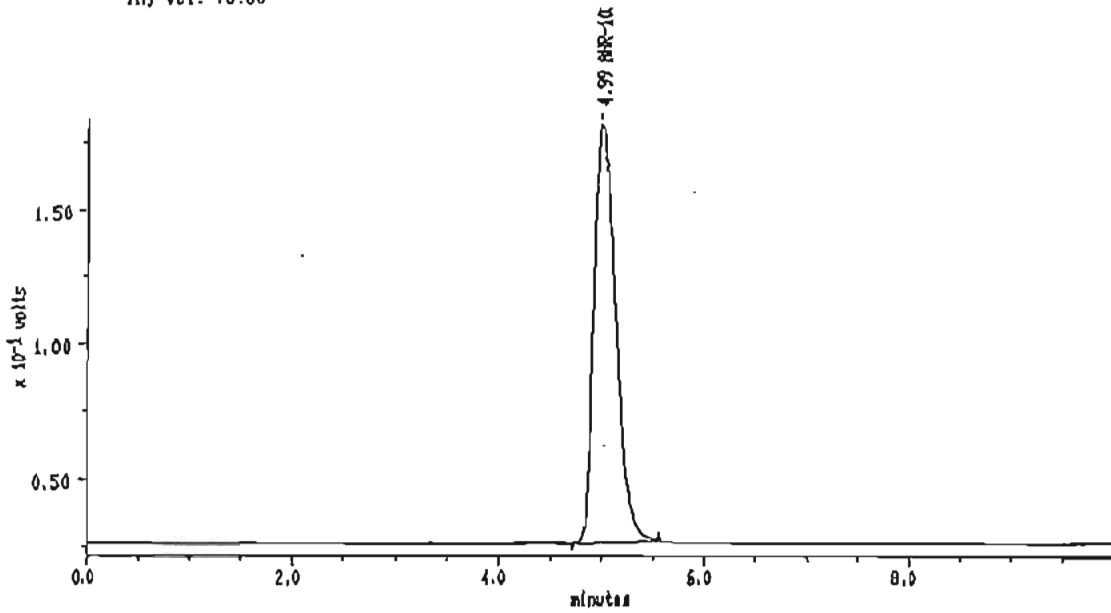
Index: 9

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.976	BB	2218054	163884	100.00	AHR-10282B
TOTAL			2218054	163884		

Sample: A18 70°C-1W Channel: detector 1 Filename: IN-10 Chart Speed: Full Size
 Acquired: 22-JAN-2001 21:42 Method: B:VAHRVHIEVINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 21:63:04

SAMPLE: A18 70°C-1W

#12 In Method: AHR-10282B

Acquired: 22-JAN-2001 21:42

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-10

Index: 10

Injection Volume: 10.0

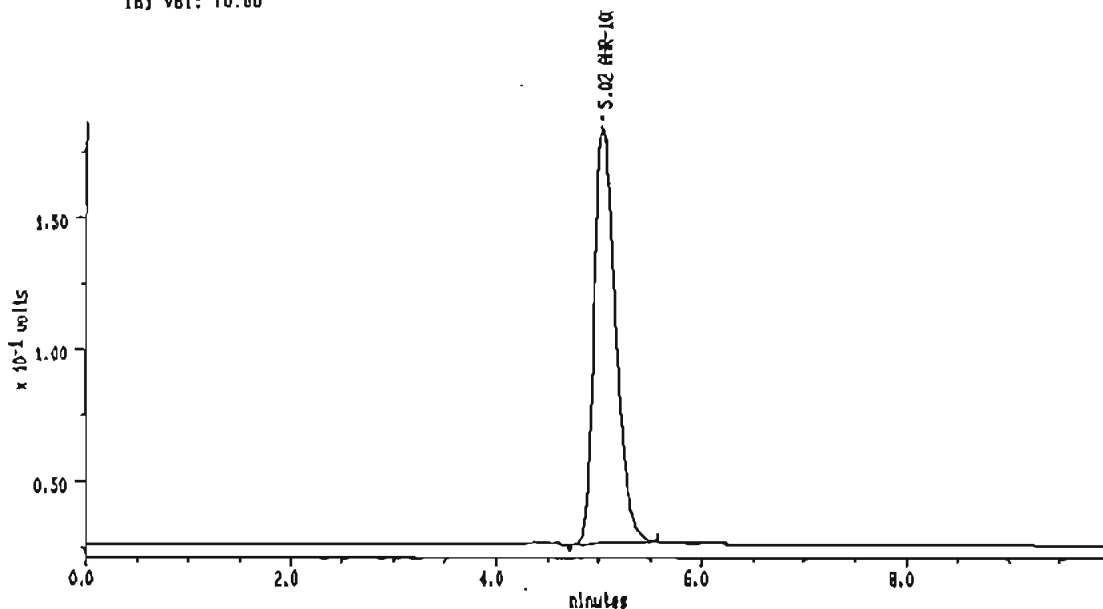
DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.992	BB	2242130	166818	100.00	AHR-10282B
TOTAL			2242130	166818		

Sample: A19 70°C-1W
Acquired: 22-JAN-01 21:53
Inj Vol: 10.00

Channel: detector 1
Method: D:YAHRYIHHBYJNIT/AL

Filename: IN-11
Chart Speed: Full Size
Operator: S.S



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MAXIMA 825 カスタムレポート

Printed: 23-JAN-2001 12:08:58

SAMPLE: A19 70°C-1W

#13 In Method: AHR-10282B

Acquired: 22-JAN-2001 21:53

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKNOWN

Instrument: Instrument 1

Filename: IN-11

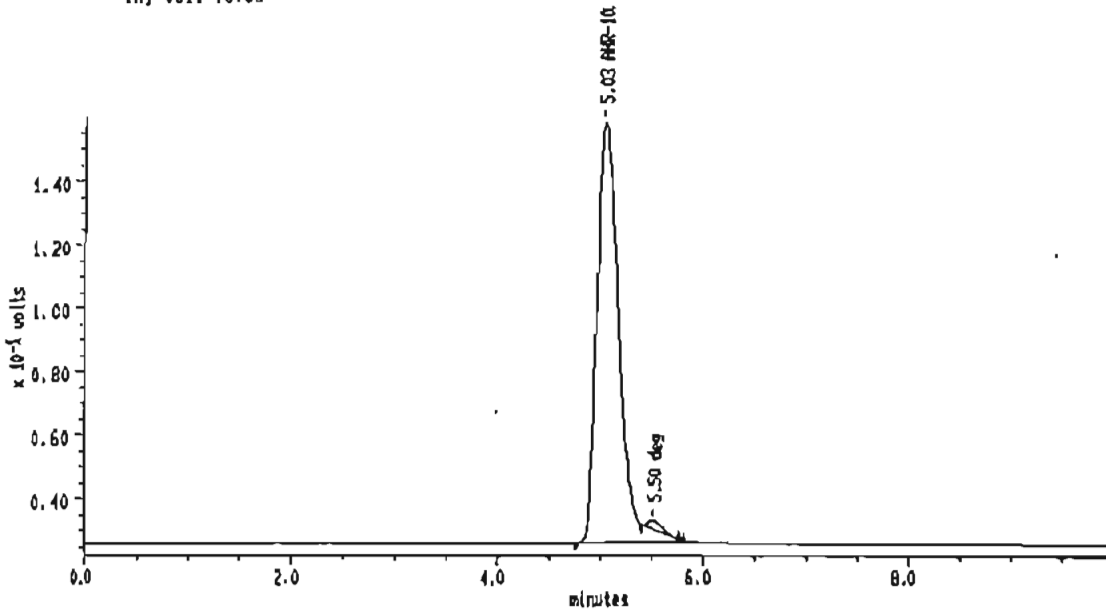
Index: 11

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	BB	2290529	150280	100.00	AIR-10282B
TOTAL			2290628	150280		

Sample: A20 70°C-1W Channel: detector 1 Filename: 1N-12 Chart Speed: Full Size
 Acquired: 22-JAN-2001 22:05 Method: D:\AHRV\H15\N171AL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 22:18:37

SAMPLE: A20 70°C-1W

#14 In Method: AHR-10282B

Acquired: 22-JAN-2001 22:05

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: URM

Instrument: Instrument 1

Filename: 1N-12

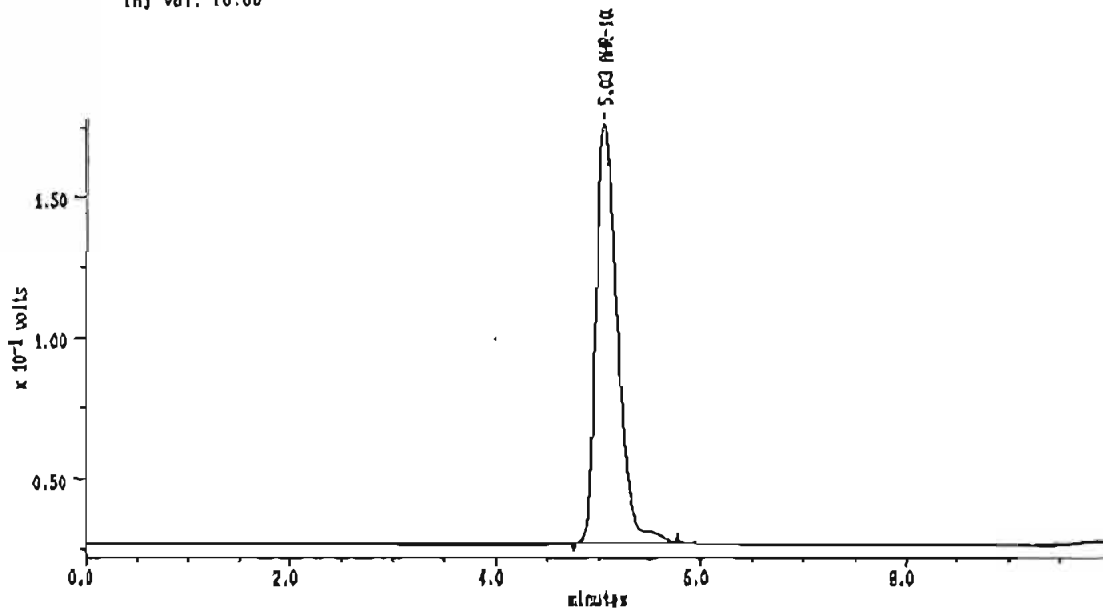
Index: 12

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.031	RD	1951517	132030	98.58	AHR-10282B
2	6.500	SS	28174	2690	1.42	deg
TOTAL			1979691	134726		

Sample: A21 70°C-1W Channel: detector 1 Filename: IN-13 Chart Speed: Full Size
 Acquired: 22-JAN-01 22:18 Method: B:YAHRYIHISVIN7IAL Operator: S.S
 [inj Vol]: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 22:28:53

SAMPLE: A21 70°C-1W

#16 (n Method: AHR-10282B

Acquired: 22-JAN-2001 22:18

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-13

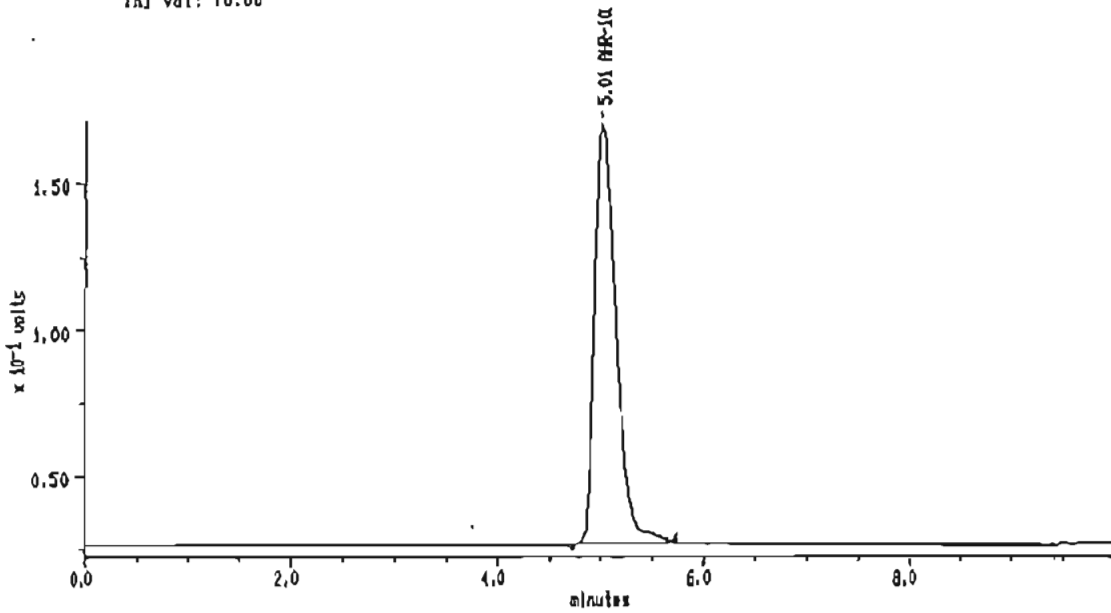
Index: 13

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.033	DB	2203526	150129	100.00	AHR-10282B
TOTAL			2203526	150129		

Sample: A22 70°C-1W Channel: detector 1 Filename: 1N-14 Chart Speed: Full Size
 Acquired: 22-JAN-10 22:27 Method: G:YAHRYIHIEVINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

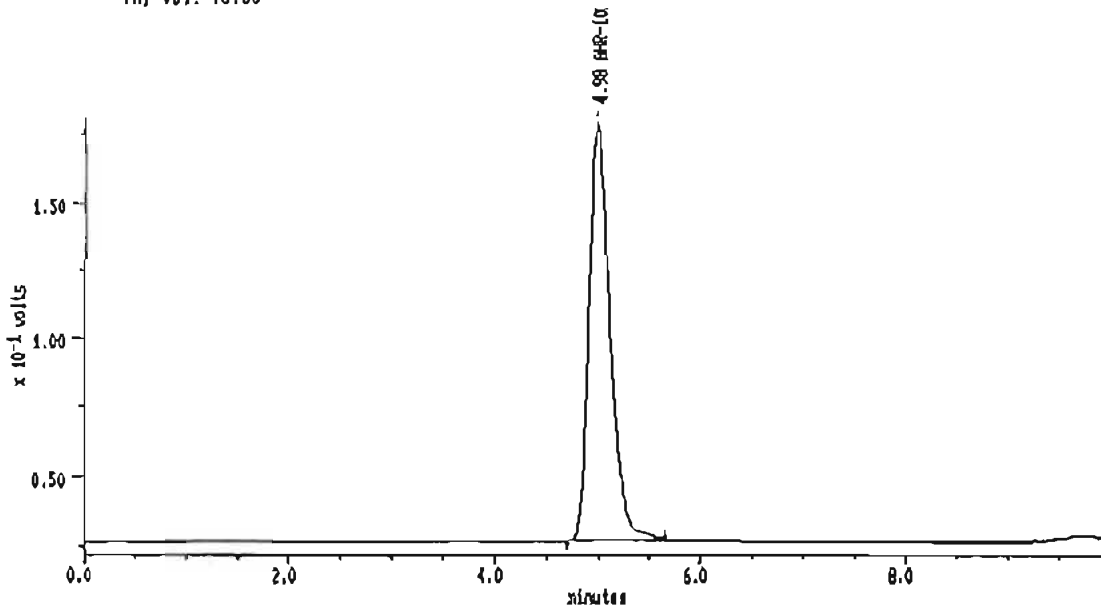
Printed: 22-JAN-2001 22:38:09

SAMPLE: A22 70°C-1W Type: UNKN
 #18 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 22-JAN-2001 22:27 Filename: 1N-14
 Rate: 2.0 points/sec Index: 14
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	2082220	143738	100.00	AHR-10282B
TOTAL			2082220	143738		

Sample: A23 70°C-1W Channel: detector 1 Filename: IN-16 Chart Speed: Full Size
 Acquired: 22-JAN-101 22:38 Method: D:YAKRVIH16VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 22:49:25

SAMPLE: A23 70°C-1W

#17 In Method: AHR-10282B

Acquired: 22-JAN-2001 22:38

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-16

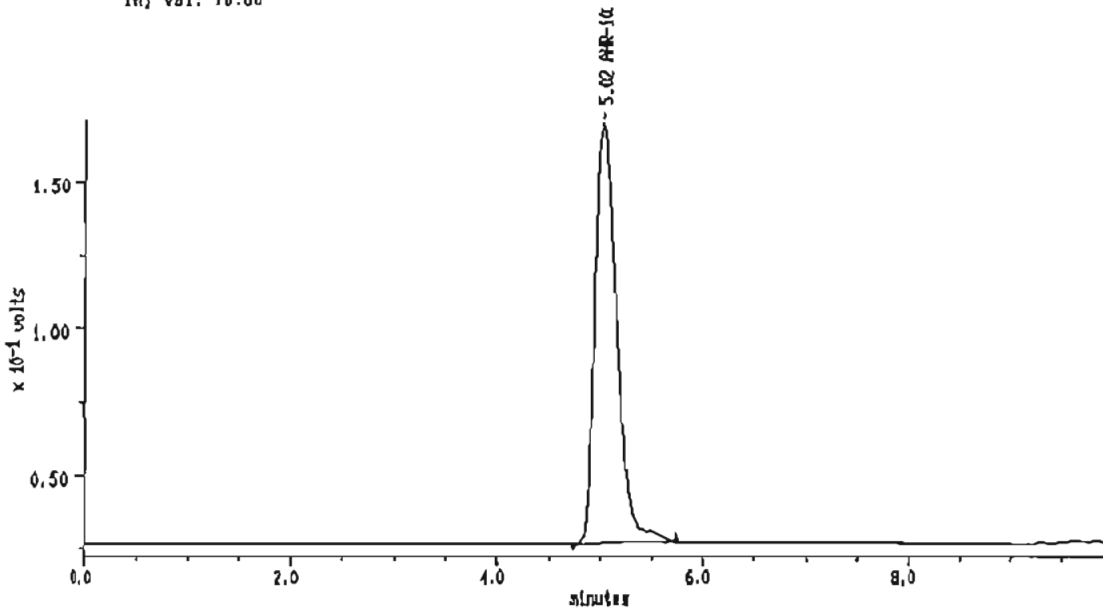
Index: 15

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.976	BB	2197763	162788	100.00	AHR-10282B
TOTAL			2197763	162788		

Sample: A24 70°C-1W Channel: detector 1 Filename: IN-18 Chart Speed: Full Size
 Acquired: 22-JAN-2001 22:58 Method: 8:VAHRVIII16YINIJAL Operator: S.S
 Inf Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 23:00:42

SAMPLE: A24 70°C-1W

#18 In Method: AHR-10282B

Acquired: 22-JAN-2001 22:58

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-18

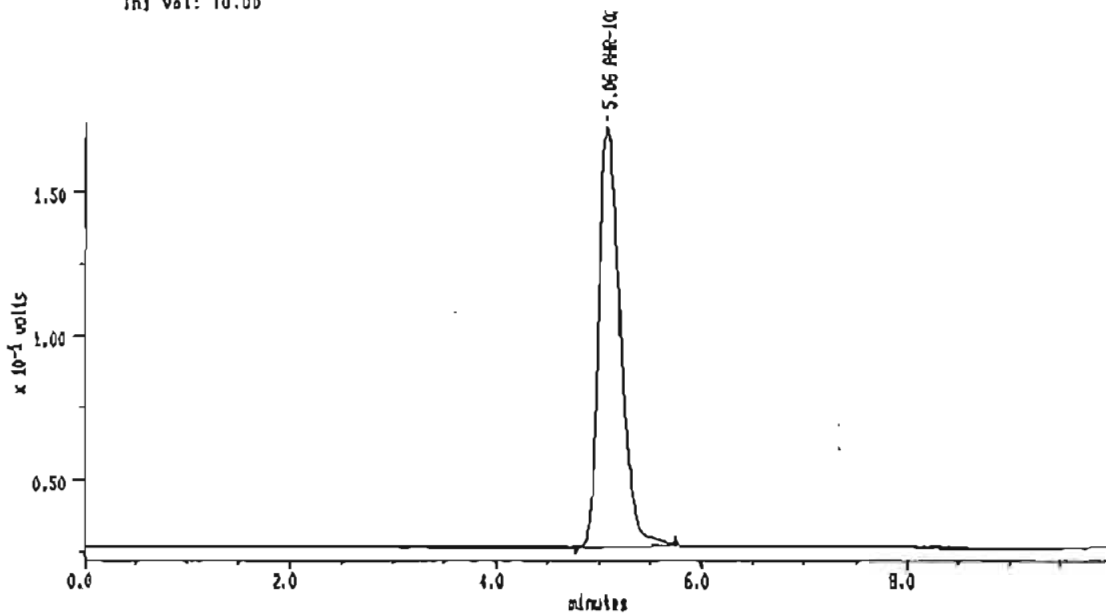
Index: 18

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	BD	2095221	143690	100.00	AHR-10282B
TOTAL			2095221	143690		

Sample: A26 10° C-1W Channel: detector 1 Pilename: IN-17 Chart Speed: Full Size
 Acquired: 22-JAN-01 23:01 Method: B:YAJIRVIRHISVINJIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 23:11:59

SAMPLE: A25 10° C-1W

#19 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:01

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Pilename: IN-17

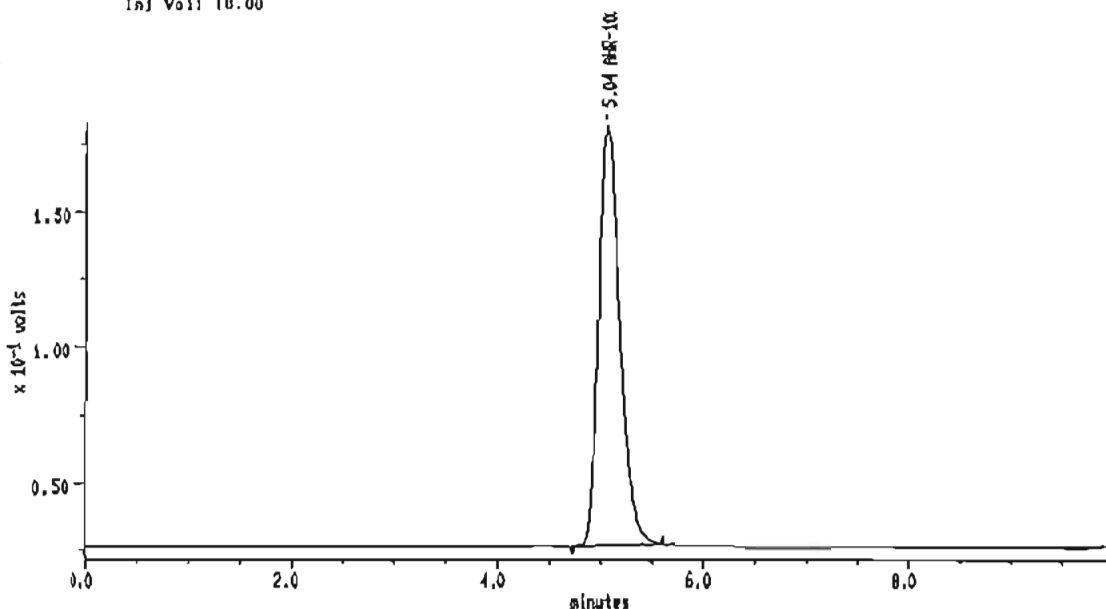
Index: 17

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	6.068	BB	2131377	145834	100.00	AHR-10282B
TOTAL			2131377	145834		

Sample: A18 80°C-1W Channel: detector 1 Filename: 1N-18 Chart Speed: Full Size
 Acquired: 22-JAN-2001 23:12 Method: B:VAHRYIHI16VINIITJIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

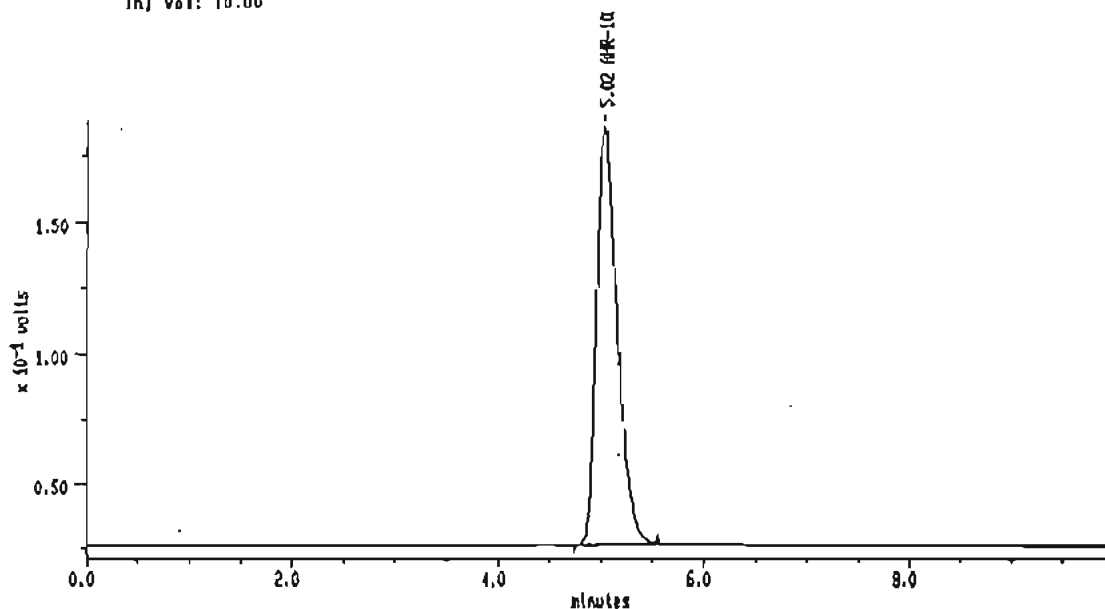
Printed: 23-JAN-2001 12:09:18

SAMPLE: A18 80°C-1W Type: UNKN
 #20 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 22-JAN-2001 23:12 Filename: 1N-18
 Rate: 2.0 points/sec Index: 18
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.042	DD	2234801	156224	100.00	AHR-10282B
TOTAL			2234801	156224		

Sample: A19 80°C-1W Channel: detector 1 Filename: 1N-19 Chart Speed: Full Size
 Acquired: 22-JAN-01 23:24 Method: B:YAHRYIH16VINIJIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 23:34:33

SAMPLE: A19 80°C-1W

#21 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:24

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: 1N-19

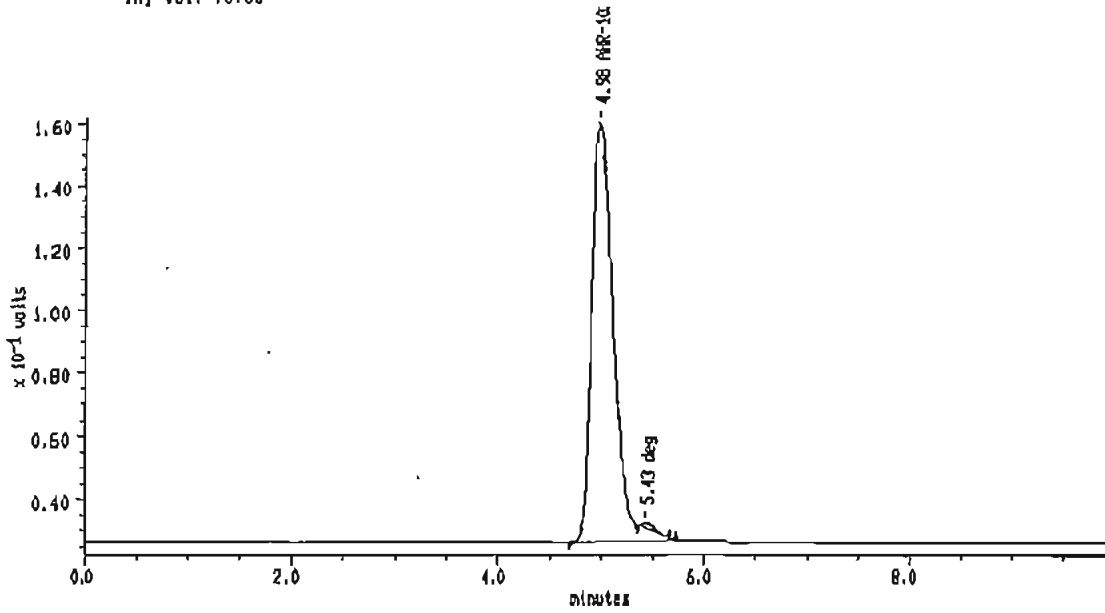
Index: 19

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	BB	2288798	180013	100.00	AHR-10282B
TOTAL			2288798	180013		

Sample: A20 80°C-1W Channel: detector 1 Filename: IN-20 Chart Speed: Full Size
 Acquired: 22-JAN-101 23:35 Method: B:VAHRVHIIH6YINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

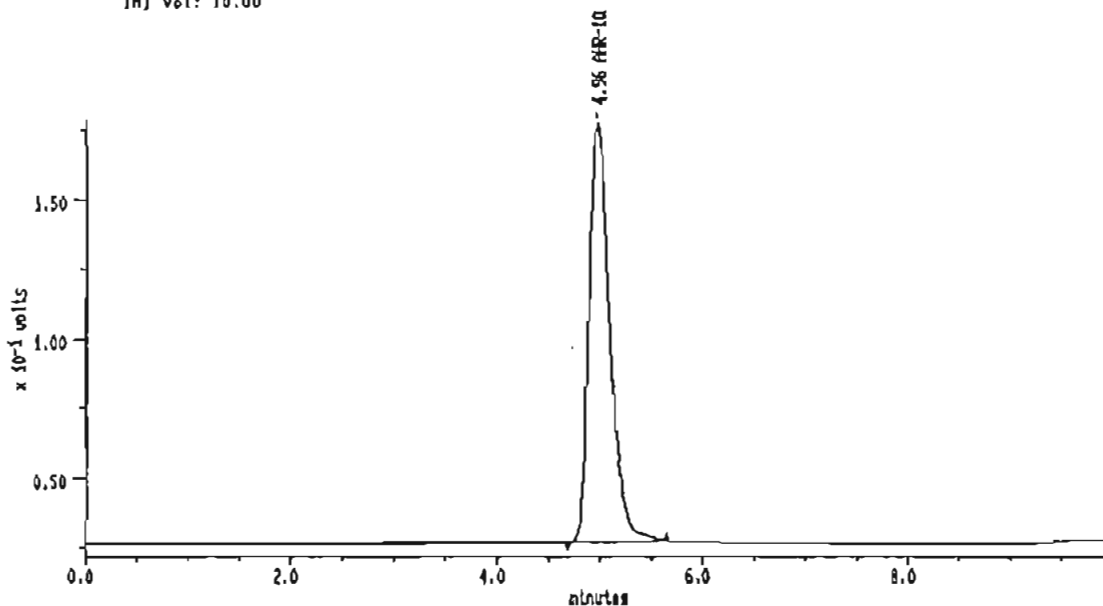
Printed: 22-JAN-2001 23:45:48

SAMPLE: A20 80°C-1W Type: UNKN
 #22 In Method: AHR-10282B Instrument: Instrument 1
 Acquired: 22-JAN-2001 23:35 Filename: IN-20
 Rate: 2.0 points/sec Index: 20
 Duration: 10.000 minutes Injection Volume: 10.0
 Operator: S.S

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.976	BB	1948910	133878	99.21	AHR-10282B
2	6.426	SS	16018	1583	0.79	deg
TOTAL			1984625	135441		

Sample: A21 80°C-1W Channel: detector 1 Filename: IX-21 Chart Speed: Full Size
 Acquired: 22-JAN-101 23:48 Method: B:YAHRYIH16VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 22-JAN-2001 23:67:06

SAMPLE: A21 80°C-1W

#23 in Method: AHR-10282B

Acquired: 22-JAN-2001 23:48

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IX-21

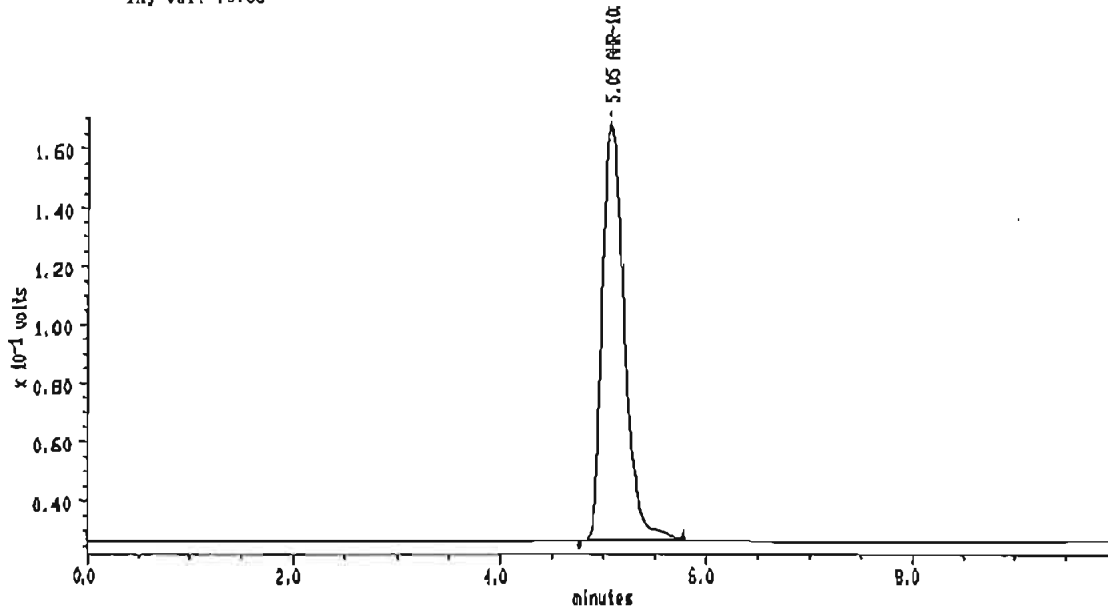
Index: 21

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.968	BB	2181206	160885	100.00	AHR-10282B
TOTAL			2181206	160886		

Sample: A22 80°C-1W Channel: detector 1 Filename: IN-22 Chart Speed: Full Size
 Acquired: 22-JAN-2001 23:57 Method: BrVAHRV1H16VINIITAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 23-JAN-2001 0:08:22

SAMPLE: A22 80°C-1W

#24 In Method: AHR-10282B

Acquired: 22-JAN-2001 23:57

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-22

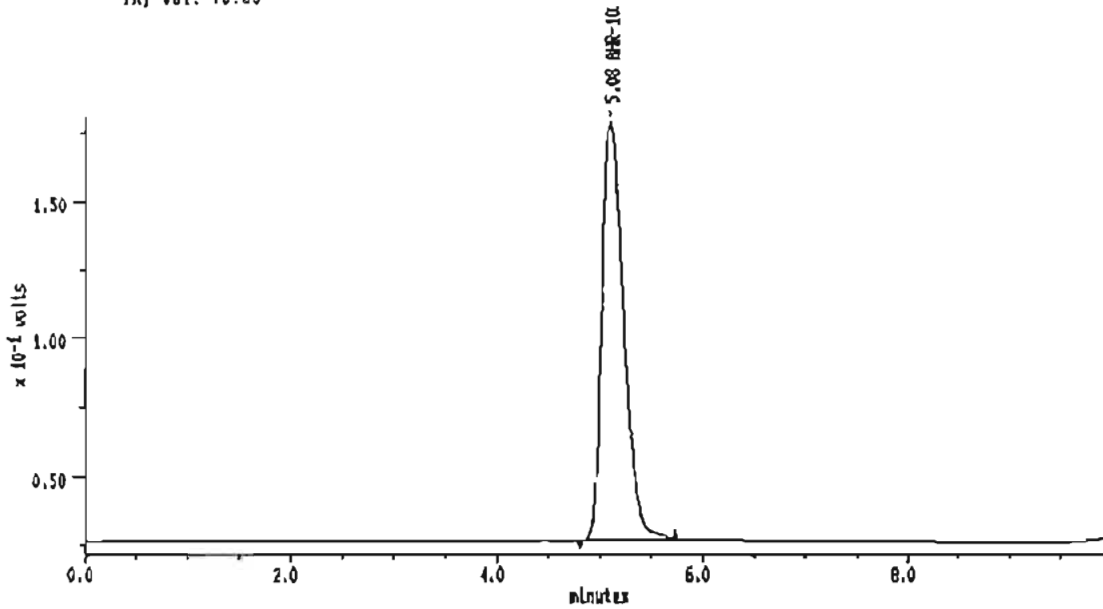
Index: 22

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.050	BD	2091491	142081	100.00	AHR-10282B
TOTAL			2091491	142081		

Sample: A23 80°C-1W Channel: detector 1 Filename: IN-23 Chart Speed: Full Size
 Acquired: 23-JAN-2001 0:09 Method: B:YANRV1H15XINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 23-JAN-2001 0:19:39

SAMPLE: A23 80°C-1W

#25 In Method: AHR-10282B

Acquired: 23-JAN-2001 0:09

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-23

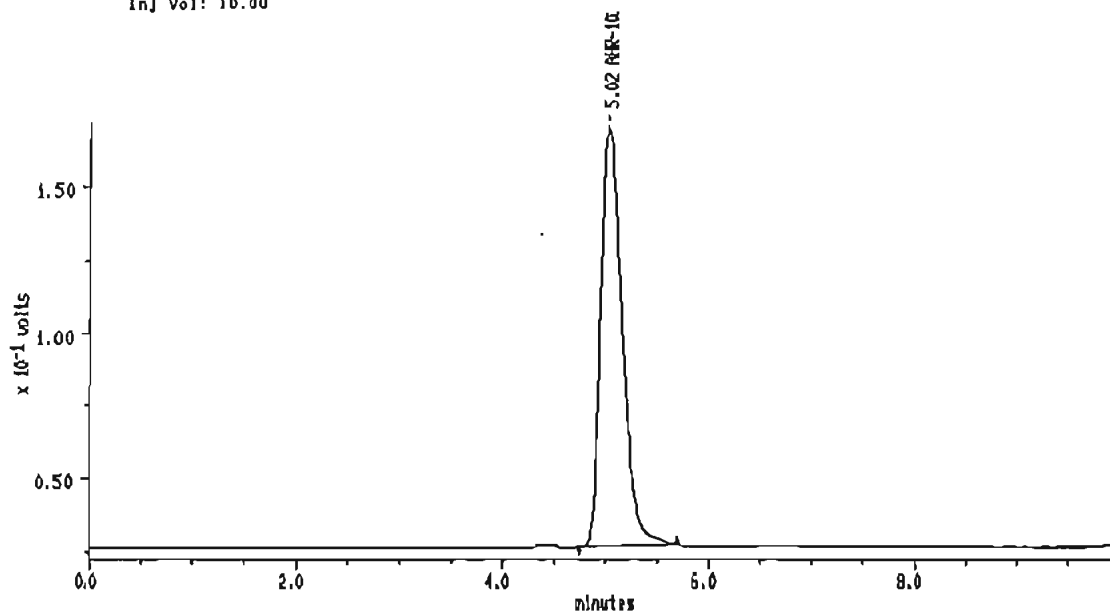
Index: 23

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.083	DD	2243848	162820	100.00	AHR-101820
TOTAL			2243848	162820		

Sample: A24 60°C-1W Channel: detector 1 Filename: IN-24 Chart Speed: Full Size
 Acquired: 23-JAN-2001 0:20 Method: B:YAHRYVHHSVINITIAL Operator: S.S
 Inj Vol: 10.00



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MAXIMA 825 カスタムレポート

Printed: 23-JAN-2001 0:30:55

SAMPLE: A24 60°C-1W

#28 In Method: AHR-10282B

Acquired: 23-JAN-2001 0:20

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-24

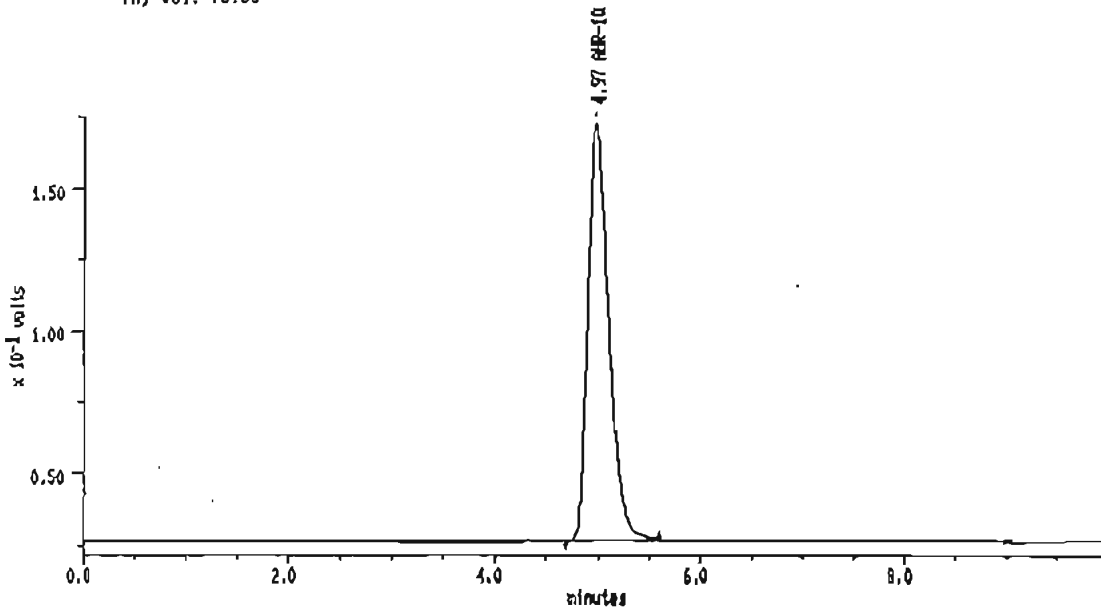
Index: 24

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.017	BB	2079710	143989	100.00	AHR-10282B
TOTAL			2079710	143989		

Sample: A26 80°C-1W Channel: detector 1 Filename: IN-25 Chart Speed: Full Size
 Acquired: 23-JAN-2001 0:31 Method: B:YAHRYIH)5VINITIAL Operator: S.S
 (n) Vol: 10.00



MAXIMA (c)1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 カスタムレポート

Printed: 23-JAN-2001 0:42:12

SAMPLE: A26 80°C-1W

#27 In Method: AHR-10282B

Acquired: 23-JAN-2001 0:31

Rate: 2.0 points/sec

Duration: 10.000 minutes

Operator: S.S

Type: UNKN

Instrument: Instrument 1

Filename: IN-25

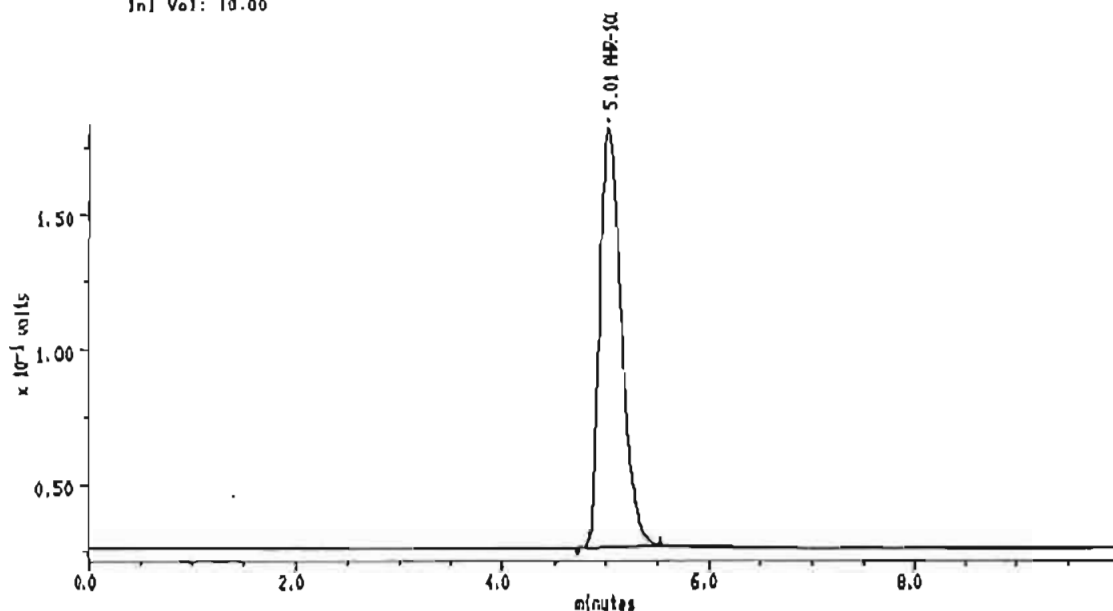
Index: 26

Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	4.987	BB	2088864	148842	100.00	AHR-10282B
TOTAL			2088864	148842		

Sample: STD2 Channel: detector 1 Filanase: IN-28 Chart Speed: Full Size
 Acquired: 23-JAN-101 0:42 Method: B:VAHRVHISVINIITAL Operator: S.S
 Inj Vol: 10.00



MAXIMA (c) 1990 Dynamic Solutions, Division of Millipore

MAXIMA 825 カスタムレポート

Printed: 23-JAN-2001 0:53:38

SAMPLE: STD2

#28 In Method: AHR-10282B
 Acquired: 23-JAN-2001 0:42
 Rate: 2.0 points/sec
 Duration: 10.000 minutes
 Operator: S.S

Type: UNKN
 Instrument: Instrument 1
 Filanase: IN-28
 Index: 28
 Injection Volume: 10.0

DETECTOR: detector 1

PK#	Retention Time (minutes)	Type	Peak Area	Peak Height	Area Percent	Component Name
1	5.008	BB	2251148	155890	100.00	AHR-10282B
TOTAL			2251148	155890		

APPENDIX B

CERTIFICATION OF TRANSLATION

The undersigned, Ryan Malcho, whose address is 2192 Queen St. E., No. 64,
Toronto, Canada declares and states as follows:

I am well acquainted with the English and Japanese languages; I have in the past translated numerous Japanese documents of legal and/or technical content into English,

I have been requested to translate into English the attached Japanese documents titled:

- Exhibit A_P2000B177 data relied on.pdf
- Exhibit B_P2002B116 data relied on.pdf
- Exhibit C_P2002B131 data relied on.pdf

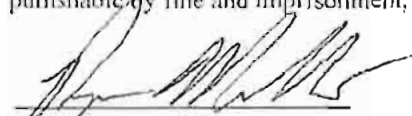
To copies of these Japanese documents I therefore attach the English translations and my Certification of Translation.

I hereby certify that the English translations of the attached documents titled

- Exhibit A_P2000B177 data relied on.pdf
- Exhibit B_P2002B116 data relied on.pdf
- Exhibit C_P2002B131 data relied on.pdf

are, to the best of my knowledge and ability, accurate translations.

And I declare further that all statements made herein of my own knowledge are true, that all statements made on information and belief are believed to be true, and that false statements and the like are punishable by fine and imprisonment, or both, under Section 1001 of Title 18 of the United States Code.



Ryan Malcho

Nov 16, 2015

Date

Test Protocol

Name of test: Study of the Preservatives-Effectiveness of Bronuck Ophthalmic Solution

Test code: P2002B116

Test system: None

Development code: AHR10282B

Test start date: 29 May 2002

Scheduled start date of test operations: 5 June 2002

Scheduled end date of test operations: 17 July 2002

Scheduled test end date: 5 December 2002

Test facility: Kobe Creative Center, Senju Pharmaceutical Co., Ltd.

1-5-4 Murotani, Nishi-ku, Kobe-shi

(Distribution of work duties)

Study director: Shuhei Fujita (creation of test protocol, creation of test operations and final report)

Test substance: Bronuck Ophthalmic Solution, Lot No. A007

Formulation (in 1mL)

Bromfenac sodium hydrate	1mg
Boric acid	11mg
Borax	11mg
Dried sodium sulfite	2mg
Sodium edetate	0.2mg
Povidone (polyvinylpyrrolidone K30)	20mg
Polysorbate 80	1.5mg
Benzalkonium chloride (10 W/V%) (0.05mg as benzalkonium chloride)	0.5µL
Sodium hydroxide	q.s.
Purified water	q.s.

Purpose: A preservatives-effectiveness study has already been performed on Bronuck Ophthalmic Solution in accordance with the Japanese Pharmacopocia, and it was confirmed that preservatives-effectiveness met the standards thereof. This time, a preservatives-effectiveness study is being performed in accordance with the European Pharmacopoeia in view of the plan to license this formulation out to Europe, in order to confirm whether or not preservatives-effectiveness meets the standards thereof.

Test method: Perform according to the preservatives-effectiveness test method in the European Pharmacopoeia. That is, dispense 10mL of the test sample into each of five sterilized, stoppered test tubes, and inoculate each one of the following test bacteria or fungi into each test tube containing the samples such that the quantity of bacteria is 10^6 CFU/mL and the quantity of fungi is 10^5 CFU/mL. Store the inoculated test tubes containing the samples at 20 to 25°C, and sample them after 6 hours, 24 hours, 7 days, 14 days, 21 days, and 28 days. Collect 0.5mL of the samples from each test tube, dilute 10×, 100×, and 1000× with 4.5mL of sterile physiological saline solution, and inoculate 1mL into petri dishes. Dispense 15 to 20mL of soybean-casein digest agar media (SCD agar media) containing an inactivating agent (0.1% lecithin, 0.7% polysorbate 80) into the bacteria, and dispense 15 to 20mL of Sabouraud glucose agar media containing an inactivating agent (0.1% lecithin, 0.7% polysorbate 80) into the fungi, culture under the following conditions, and confirm the viable bacteria counts.

	Test strains	Culturing conditions
Bacteria	<i>Staphylococcus aureus</i> ATCC 6538	30~35°C
	<i>Escherichia coli</i> ATCC 8739	
	<i>Pseudomonas aeruginosa</i> ATCC 9027	
Fungi	<i>Candida albicans</i> ATCC 10231	20~25°C
	<i>Aspergillus niger</i> ATCC 16404	

Equipment: SANYO incubator MIR-251
 SANYO incubator MIR-551
 Spectrophotometer MILTONROY COMPANY SPECTRONIC 601
 Balance Sartorius LC620S
 TOMY HIGH-PRESSURE STEAM STERILIZER BS-325

Criteria: According to the European Pharmacopoeia. That is, Criteria A of the European Pharmacopoeia are met if the viable bacteria counts are 1/100 of the inoculated bacteria or lower after 6 hours, 1/1000 of the inoculated bacteria or lower after 24 hours, and no viable bacteria detected after 28 days, and if the viable fungi counts are 1/100 of the inoculated fungi or lower after 7 days, and the same level or lower thereafter. Criteria B of the European Pharmacopoeia are met if the viable bacteria counts are 1/10 of the inoculated bacteria or lower after 24 hours, 1/1000 of the inoculated bacteria or lower after 7 days, and the same level or lower thereafter, and if the viable fungi counts are 1/10 of the inoculated fungi or lower after 14 days, and the same level or lower thereafter.

Signature of study director	<u>29 May 2002</u>	<u>Shuhei Fujita [seal]</u>
Signature of formulation & analysis GM	<u>29 May 2002</u>	<u>Katsuhiko Inada [seal]</u>
Approval of laboratory director	<u>30 May 2002</u>	<u>Akira Ohtori [seal]</u>

Preservatives-Effectiveness Test Record

Test period: 5 June 2002 ~ 10 July 2002
 Test code: P2002B116
 Tester: Shuhei Fujita [seal]
 Study director: Shuhei Fujita [seal]
 Test substance: Bronuck Ophthalmic Solution, Lot No. A007

Formulation

(in 1mL)
 Bromfenac sodium hydrate 1mg
 Boric acid 11mg
 Borax 11mg
 Dried sodium sulfite 2mg
 Sodium edetate 0.2mg
 Povidone (polyvinylpyrrolidone K30) 20mg
 Polysorbate 80 1.5mg
 Benzalkonium chloride (10 W/V%)
 (0.05mg as benzalkonium chloride) 0.5μL
 Sodium hydroxide q.s.
 Purified water q.s.

Results

Test bacteria/fungi inoculation date: 5 June 2002

	No. inoculated	Test date					
		06/05/02	06/06/02	06/12	06/19	06/26	07/03
Count date	06/07	06/07	06/07	06/17	06/24	06/28	07/05
<i>S.aureus</i> *1	4.3 × 10 ⁶	4.0 × 10 ⁶	3.1 × 10 ⁶	7.0 × 10 ⁵	0	0	0
Count date	06/07	06/07	06/07	06/17	06/24	06/28	07/05
<i>E.coli</i> *1	6.9 × 10 ⁶	2.5 × 10 ³	0	0	0	0	0
Count date	06/07	06/07	06/07	06/17	06/24	06/28	07/05
<i>P.aeruginosa</i> *1	1.1 × 10 ⁷	0	0	0	0	0	0
Count date	06/07	06/07	06/07	06/17	06/24	06/28	07/05
<i>C.albicans</i> *2	3.2 × 10 ⁵	1	1	4.7 × 10 ³	0	0	0
Count date	06/12	06/12	06/12	06/19	06/26	07/03	07/10
<i>A.niger</i> *2	1.1 × 10 ⁵	1	1	6.0 × 10 ³	3.0 × 10 ³	0	0

unit : CFU/mL

Equipment: SANYO incubator MIR-551, equipment number 400061, temperature setting 30~35°C *1 Test bacteria/fungi culturing equipment
 SANYO incubator MIR-251, equipment number 300257, temperature setting 20~25°C *2 Test bacteria/fungi culturing equipment

試験計画書

試験名： プロナック点眼液の保存効力試験

試験コード： P2002B116

試験系： なし

開発記号： AHR10282B

試験開始日 : 2002 年 05 月 29 日

試験操作開始予定日 : 2002 年 06 月 05 日

試験操作終了予定日 : 2002 年 07 月 17 日

試験終了予定日 : 2002 年 12 月 05 日

試験施設 : 千寿製薬株式会社 コーベクリエティブセンター
神戸市西区室谷一丁目 5 番 4 号

(業務分担)

試験責任者： 藤田 修平 試験計画書作成、試験操作及び最終報告書作成

試験物質 : プロナック点眼液 Lot No.A007

処方 (1mL 中)

ブロムフェナクナトリウム水和物	1mg
ホウ酸	11mg
ホウ砂	11mg
乾燥亜硫酸ナトリウム	2mg
エデト酸ナトリウム	0.2mg
ポビドン (ポリアクリル酸ナトリウム K30)	20mg
ポリソルベート 80	1.5mg
塩化ベンザルコニウム液 (10W/V%) (塩化ベンザルコニウムとして 0.05mg)	0.5 μ L
水酸化ナトリウム	適量
精製水	適量

目的：プロナック点眼液は以前、日本薬局方に準じて保存効力試験を行い保存効力がその基準に適合であったことを確認している。今回、本処方の海外（ヨーロッパ）への導出を考えており、ヨーロッパ薬局方に準じて試験を行い、保存効力がその基準に適合するかを確認する。

試験方法：ヨーロッパ薬局方の保存効力試験法に準じる。即ち、5本の滅菌された共栓付き試験管に本試験のサンプルを10mLずつ分注し、下記の試験菌を細菌は 10^6 CFU/mL、真菌は 10^6 CFU/mLになるよう、サンプル入り試験管1本につき1菌種を接種する。菌を接種したサンプル入り試験管を20~25°Cで保管し、6時間後、24時間後、7日後、14日後、21日後及び28日後にサンプリングを行う。各試験管からサンプルを0.5mL分取し、4.5mLの滅菌生理食塩液で10倍、100倍及び1000倍希釈を行いシャーレに1mL接種し、細菌には不活化剤（レシチン0.1% ポリソルベート80 0.7%）入りソイビーン・カゼイン・ダイジェスト寒天培地（SCD寒天培地）を15~20mL分注し、真菌には不活化剤（レシチン0.1% ポリソルベート80 0.7%）入りサブロー・ブドウ糖寒天培地を15~20mL分注し、下記の条件下で培養を行い生菌数を確認する。

	被検菌株	培養条件
細菌	<i>Staphylococcus aureus</i> ATCC 6538	30~35°C
	<i>Escherichia coli</i> ATCC 8739	
	<i>Pseudomonas aeruginosa</i> ATCC 9027	
真菌	<i>Candida albicans</i> ATCC 10231	20~25°C
	<i>Aspergillus niger</i> ATCC 16404	

使用機器：SANYO インキュベータ MIR-251
 SANYO インキュベータ MIR-551
 分光光度計 MILTONROY COMPANY SPECTRONIC 601
 天秤ザルトリウス LC620S
 TOMY HIGH-PRESSURE STEAM STERILIZER BS-325

判定基準：ヨーロッパ薬局方に準ずる。即ち、A基準では細菌は6時間後に生菌数が接種菌数の1/100以下、24時間後に接種菌数の1/1000以下、28日後に生菌が検出されないこと、また、真菌は7日後に接種菌数の1/100以下、以降は7日後と同レベルかそれ以下ならば、ヨーロッパ薬局方A基準に適合とする。B基準では細菌は24時間後に生菌数が接種菌数の1/10以下、7日後に接種菌数の1/1000以下、以降は同レベルかそれ以下であること、また、真菌は14日後に生菌数が接種菌数の1/10以下、以降は同レベルかそれ以下ならば、ヨーロッパ薬局方B基準に適合とする。

試験責任者の署名 2002年05月29日 藤田 修平 (藤田)

製剤・分析GMの署名 2002年05月29日 稲田 勝弘 (稲田)

開発研究所長の承認 2002年05月30日 大橋 聡 (大橋)

保存効力試験記録書

試験期間: '02.06.05~'02.07.10

試験コード: P2002B116

試験実施者: 藤田 修平 (印)

試験責任者: 藤田 修平 (印)

試験物質: プロナック点眼液 Lot No. A007

処方

(1 mL 中)

7-DM7エチナトリウム水和物	1mg
芍薬	11mg
芍砂	11mg
乾燥亜硫酸ナトリウム	2mg
アスコルビン酸ナトリウム	0.2mg
ホピトロン (ホピトロン K30)	20mg
ポリリハート 80	1.5mg
塩化ベンザルコニウム液 (10w/v%)	0.5μL
(塩化ベンザルコニウムとして 0.05mg)	
水酸化ナトリウム	適量
精製水	適量

結果

試験菌接種日 '02年 06月 05日

	接種菌数	試験実施日					
		06/05 ^(6h)	06/06 ^(24h)	06/12	06/19	06/26	07/03
菌数確認日	06/07	06/07	06/07	06/17	06/24	06/28	07/05
<i>S.aureus</i> *1	4.3×10 ⁶	4.0×10 ⁶	3.1×10 ⁶	7.0×10 ⁵	0	0	0
菌数確認日	06/07	06/07	06/07	06/17	06/24	06/28	07/05
<i>E.coli</i> *1	6.9×10 ⁶	2.5×10 ³	0	0	0	0	0
菌数確認日	06/07	06/07	06/07	06/17	06/24	06/28	07/05
<i>P.aeruginosa</i> *1	1.1×10 ⁷	0	0	0	0	0	0
菌数確認日	06/07	1	1	06/17	06/24	06/28	07/05
<i>C.albicans</i> *2	3.2×10 ⁵			4.7×10 ³	0	0	0
菌数確認日	06/12	1	1	06/19	06/26	07/03	07/10
<i>A.niger</i> *2	1.1×10 ⁵			6.0×10 ³	3.0×10 ³	0	0

unit: CFU/mL

使用機器: SANYO イキハータ MIR-551 機器番号 400061 温度設定 30~35°C *1 試験菌培養機器

SANYO イキハータ MIR-251 機器番号 300257 温度設定 20~25°C *2 試験菌培養機器

APPENDIX C

CERTIFICATION OF TRANSLATION

The undersigned, Ryan Malcho, whose address is 2192 Queen St. E., No. 64, declares and states as follows:
Toronto, Canada

I am well acquainted with the English and Japanese languages; I have in the past translated numerous Japanese documents of legal and/or technical content into English.

I have been requested to translate into English the attached Japanese documents titled:

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- Exhibit B_P2002B116 data relied on.pdf
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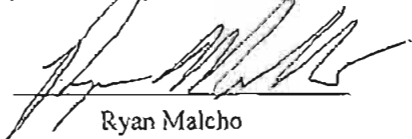
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are, to the best of my knowledge and ability, accurate translations.

And I declare further that all statements made herein of my own knowledge are true, that all statements made on information and belief are believed to be true, and that false statements and the like are punishable by fine and imprisonment, or both, under Section 1001 of Title 18 of the United States Code.


Ryan Malcho

Nov 16, 2015
Date

Test Protocol

Name of test: Formulation Design for Bronuck Ophthalmic Solution in Compliance with EP

Test code: P2002B131

Test system: None

Development code: AHR10282B

Test start date: 26 June 2002

Scheduled start date of test operations: 26 June 2002

Scheduled end date of test operations: 28 December 2002

Scheduled test end date: 31 January 2003

Test facility: Kobe Creative Center, Senju Pharmaceutical Co., Ltd.

1-5-4 Murotani, Nishi-ku, Kobe-shi

(Split of work duties)

Study director: Shirou Sawa (creation of test protocol, test operations and creation of final report)

Study personnel: Shuhei Fujita (test operations)

Test substance: Bromfenac sodium

Purpose: Bronuck Ophthalmic Solution is an anti-inflammatory developed by our company, and is under review for exportation to Europe. However, the preservatives-effectiveness of the current formulations does not comply with the EP, so its preservatives, additives, and pH are being optimized through this research in order to enhance its preservatives-effectiveness.

Test method:

1) Stability study

Vary the amount of the tyloxapol, the amount of the benzalkonium chloride, and the pH of Bronuck Ophthalmic Solution and observe its external appearance. Fill the formulations that did not become cloudy into colorless polypropylene containers, and stored at 70°C, 60°C, and 40°C at 75% RH. Perform tests on the bromfenac sodium content, pH, external appearance, and foreign insoluble matter of the solution over time. Also, freeze and thaw the solution 10 times, and observe its external appearance and foreign insoluble matter.

<HPLC conditions>

Detector: Ultraviolet absorption photometer (wavelength: 266nm)

Column: Capcelpak AG-120

Column temperature: A constant temperature around 25°C

Mobile phase: Dissolve 1.98g of ammonium dihydrogen phosphate into 750mL of water, add phosphoric acid to adjust the pH to 7.3, and then mix in 250mL of acetonitrile.

Flow rate: Adjust so that the elution time of bromfenac sodium is about 18 minutes.

Sample injection amount: 10µL

Formulation	
Bromfenac sodium	0.1g
Benzalkonium chloride	0.005g~0.01g
Tyloxapol	0.02~0.15g
Povidone (K-30)	2.0g
Sodium edetate	0.02g
Boric acid	q.s.
Borax	q.s.
Purified water	q.s.
pH	7.0~8.3
Total amount	100mL

Adjust the amount of boric acid and borax to suit the pH and osmotic pressure.

2) Preservatives-effectiveness study

Use the formulations whose stability was confirmed in Experiment 1) as the test sample. Perform the test according to the EP. That is, dispense the test sample into five sterilized, stoppered test tubes. Then, prepare the following test bacteria/fungi such that the quantity of bacteria is 10^8 CFU/mL and the quantity of fungi is 10^7 CFU/mL, and inoculate one species into each test tube containing the sample such that the quantity of bacteria is 10^6 CFU/mL and the quantity of fungi is 10^5 CFU/mL after inoculation. Store the inoculated test tubes containing the samples at 20 to 25°C, and sample them after 6 hours, 24 hours, 1 day, 2 days, 3 days, and 4 days. Collect 0.5mL of the samples from each test tube, dilute 10× one to three times with 4.5mL of sterile physiological saline solution, and inoculate 1mL into petri dishes. Dispense 15 to 20mL of soybean-casein digest agar media (SCD agar media) containing an inactivating agent (0.1% lecithin, 0.7% polysorbate 80) into the bacteria, and dispense 15 to 20mL of Sabouraud glucose agar media containing an inactivating agent (0.1% lecithin, 0.7% polysorbate 80) into the fungi, culture under the following conditions, and confirm the viable bacteria counts.

	Test strains	Culturing conditions
Bacteria	<i>Staphylococcus aureus</i> ATCC 6538	30~35°C
	<i>Escherichia coli</i> ATCC 8739	
	<i>Pseudomonas aeruginosa</i> ATCC 9027	
Fungi	<i>Candida albicans</i> ATCC 10231	20~25°C
	<i>Aspergillus niger</i> ATCC 16404	

Signature of study director

26 June 2002

Shirou Sawa [sea]

Signature of application development GM

26 June 2002

Akira Ohtori [sea]

FORM Protocol-5 (Version 5, 1 February 2000)

Notification of Change to Test Protocol No. 1

Name of test: Formulation Design for Bronuck Ophthalmic Solution in Compliance with EP

Test code: P2002B131

Date of change: 28 February 2003

Before change:

Scheduled end date of test operations: 28 December 2002

Scheduled test end date: 31 January 2003

After change:

Scheduled end date of test operations: 25 April 2003

Scheduled test end date: 30 May 2003

Reason for change:

The results of the formulation study revealed that preservatives-effectiveness met Criteria A of the European Pharmacopoeia, and a stable formulation could be established in polypropylene containers. The test period is being prolonged in order to confirm stability in polypropylene containers, which are often used overseas.

Signature of study director

28 February 2003

Shirou Sawa [seal]

Signature of application development GM

28 February 2003

Akira Ohtori [seal]

Preservatives-Effectiveness Test Record

Test period: 3 July 2002 ~ 7 August 2002
 Test code: P2002B131
 Tester: Shuhei Fujita [seal]
 Study director: Shirou Sawa [seal]
 Test substance: Bronuck Ophthalmic Solution, Lot No. 02S021

Formulation

(in 100mL) *A-01*
 Bromfenac sodium 0.1g
 Boric acid 1.1g
 Borax 1.1g
 Benzalkonium chloride 0.005g
 Tyloxapol 0.02g
 Povidone (polyvinylpyrrolidone K30) 2g
 Sodium edetate 0.02g
 Sodium hydroxide q.s.
 Purified water q.s.

Results

Test bacteria/fungi inoculation date: 3 July 2002

	No. inoculated	Test date					
		07/03/02	07/04/02	07/10	07/17	07/24	07/31
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>S.aureus</i> *1	2.1×10^6	3.0×10^1	0	0	0	0	0
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>E.coli</i> *1	1.5×10^6	0	0	0	0	0	0
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>P.aeruginosa</i> *1	5.8×10^6	0	0	0	0	0	0
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>C.albicans</i> *2	3.2×10^5	1	1	0	0	0	0
Count date	07/10	07/10	07/10	07/17	07/24	07/31	08/07
<i>A.niger</i> *2	1.8×10^5	1	1	0	0	0	0

unit : CFU/mL

Equipment: SANYO incubator MIR-551, equipment number 400061, temperature setting 30~35°C *1 Test bacteria/fungi culturing equipment
 SANYO incubator MIR-251, equipment number 300257, temperature setting 20~25°C *2 Test bacteria/fungi culturing equipment

Preservatives-Effectiveness Test Record

Test period: 3 July 2002 ~ 7 August 2002
 Test code: P2002B131
 Tester: Shuhei Fujita [seal]
 Study director: Shirou Sawa [seal]
 Test substance: Bronuck Ophthalmic Solution, Lot No. 02S021

Formulation

(in 100mL) A-02
 Bromfenac sodium 0.1g
 Boric acid 1.1g
 Borax 1.1g
 Benzalkonium chloride 0.005g
 Tyloxapol 0.05g
 Povidone (polyvinylpyrrolidone K30) 2g
 Sodium edetate 0.02g
 Sodium hydroxide q.s.
 Purified water q.s.

Results

Test bacteria/fungi inoculation date: 3 July 2002

	No. inoculated	Test date					
		07/07/02	07/07/02	07/10	07/17	07/24	07/31
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>S.aureus</i> *1	2.1×10^6	1.7×10^5	2.0×10^1	0	0	0	0
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>E.coli</i> *1	6.5×10^6	0	0	0	0	0	0
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>P.aeruginosa</i> *1	5.8×10^6	0	0	0	0	0	0
Count date	07/08	07/08	07/08	07/12	07/22	07/26	08/02
<i>C.albicans</i> *2	3.2×10^5	1	1	0	0	0	0
Count date	07/10	07/10	07/10	07/17	07/24	07/31	08/07
<i>A.niger</i> *2	1.8×10^5	1	1	0	0	0	0

unit : CFU/mL

Equipment: SANYO incubator MIR-551, equipment number 400061, temperature setting 30~35°C *1 Test bacteria/fungi culturing equipment
 SANYO incubator MIR-251, equipment number 300257, temperature setting 20~25°C *2 Test bacteria/fungi culturing equipment

Preparation Record B (medicinal solution) Form 7 (15 November 2000)

Test substance	AHR10282B	Test code	P2002B131	Lot No.	025021	Preparation date	2 July 2002	Tester	Shirou Sawa	
Test item	Samples for preservatives-effectiveness study									
Amount manufactured	5mL colorless ampoules x	5mL colorless PP x	5mL brown PP x	100mL glass bottles x		x		x		
Formulation No.	A-01		A-02							
Ingredients and amounts	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)			Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Manufacturer Lot No.
Ingredient	100mL		100mL							
Bromfenac sodium	0.1	0.10005	0.1	0.10001	2-Jul-2002 18:36:45					
Boric acid	1.1	1.099	1.1	1.101	001: + 0.02007 g					
Borax	1.1	1.100	1.1	1.100	002: + 0.02010 g					0C2617
Benzalkonium chloride	0.005	0.005	0.005	0.005	003: - 0.02020 g					01E008
Tyloxapol	0.02	0.02020	0.05	0.05019	004: - 0.05019 g					00P027
Povidone (K-30)	2.0	1.998	2.0	1.999	005: + 0.10005 g					02B022
Sodium edetate	0.02	0.02007	0.02	0.02010	006: + 0.10001 g					E65703A07
Purified water	q.s.	q.s.	q.s.	q.s.	2-Jul-2002 18:49:32					01R012
					001: + 1.100 g					98N012A
					002: + 1.100 g					
					003: + 1.998 g					
					004: + 1.999 g					
					005: + 1.099 g					
					006: + 1.101 g					
					02/ 7/02 18:58					
					pH 8.19					
					ATC 26.2°C					
					02/ 7/02 19:52					
					pH 8.28					
					ATC 26.2°C					
pH	8.2	8.19	8.2	8.20						

Interaction of gatifloxacin and bromfenac sodium
 Stability of bromfenac sodium

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 5 July 2002

ID	Chromato No.	AM	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.L.M.	Turbid	Permeation (%)
STD	AH2S051.C01	2295054	1378449	1.6650	0.10008						
A-01	Initial	AH2S051.C02	2274085	1375224	1.6538	0.09939	100.00	8.17	—	—	
A-02	Initial	AH2S051.C03	2269283	1358788	1.6701	0.10039	100.00	8.18	—	—	
A-03	Initial	AH2S051.C04	2269941	1362568	1.6823	0.09992	100.00	8.16	—	—	
A-04	Initial	AH2S051.C05	2263909	1357737	1.6674	0.10022	100.00	8.15	—	—	

Preparation Record B (medicinal solution) Form 7 (15 November 2000)

Test substance	AHR10282B	Test code	P2002B131	Lot No.	025041	Preparation date	4 July 2002	Tester	Shiroo Sawa		
Test item											
Amount manufactured	5mL colorless ampoules ×	5mL colorless PP ×	5mL brown PP ×	x	x	x					
Formulation No.	A-01		A-02		A-03		A-04				
Ingredients and amounts	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Amount of starting material (g)	Weighed amount (g)	Manufacturer Lot No.
Ingredient	100mL		100mL		100mL		100mL				
Bromfenac sodium	0.1	0.10000	0.1	0.10000	0.1	0.10002	0.1	0.10000			0C2647
Boric acid	1.1	1.100	1.1	1.102	1.1	1.100	1.1	1.100			01E028
Borax	1.1	1.100	1.1	1.099	1.1	1.102	1.1	1.100			00P027
Benzalkonium chloride	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005			03B022
Tyloxapol	0.02	0.02013	0.05	0.05019	0.03	0.03013	0.02	0.02008			E65703A07
Povidone (K-30)	2.0	2.003	2.0	2.002	2.0	2.000	1.0	1.004			01R012
Sodium edetate	0.02	0.02003	0.02	0.02004	0.02	0.02003	0.02	0.02001			98N012A
Purified water											
pH	8.2	8.15	8.2	8.15	8.2	8.14	8.2	8.11			

4-Jul-2002 17:37:41		4-Jul-2002 17:35:18			
001:	- 0.02013 g	001:	+ 1.100 g	102/ 7/04 19:17	
002:	- 0.05019 g	002:	+ 1.102 g	PH 8.15	
003:	- 0.03013 g	003:	+ 1.100 g	ATC 26.9°C	
004:	- 0.02008 g	004:	+ 1.100 g	102/ 7/04 19:19	
005:	+ 0.02003 g	005:	+ 2.003 g	PH 8.15	
006:	+ 0.02004 g	006:	+ 2.002 g	ATC 26.2°C	
007:	+ 0.02003 g	007:	+ 2.000 g	102/ 7/04 19:22	
008:	+ 0.02001 g	008:	+ 1.004 g	PH 8.14	
009:	+ 0.10000 g	009:	+ 1.100 g	ATC 26.9°C	
010:	+ 0.10000 g	010:	+ 1.099 g		
011:	+ 0.10002 g	011:	+ 1.102 g		
012:	+ 0.10000 g	012:	+ 1.100 g		

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2002B131	Test date	5 July 2002
Test item				Tester	Shirou Sawa
<i>STD1, IS: 4 July 2002 Preparation used.</i>					
<i>(AHR10282B 0.02502g + mobile phase → 25mL)</i>					
<i>STD2: 2mL of above solution + 2mL of IS + mobile phase → 20mL)</i>					
<i>IS: (0.03320g of methyl p-hydroxybenzoate + mobile phase → 100mL)</i>					
		<i>pH</i>	<i>External appearance</i>		
A-01	<i>Initial</i>	8.17	—		
A-02	<i>Initial</i>	8.16	—		
A-03	<i>Initial</i>	8.15	—		
A-04	<i>Initial</i>	8.15	—		

'02/ 7/05 17:38	
PH 8.17	
ATC 25.5°C	
'02/ 7/05 17:40	
PH 8.16	
ATC 26.1°C	
'02/ 7/05 17:41	
PH 8.15	
ATC 26.2°C	
'02/ 7/05 17:42	
PH 8.15	
ATC 26.4°C	

17:26:18

Sample: STD

ID:

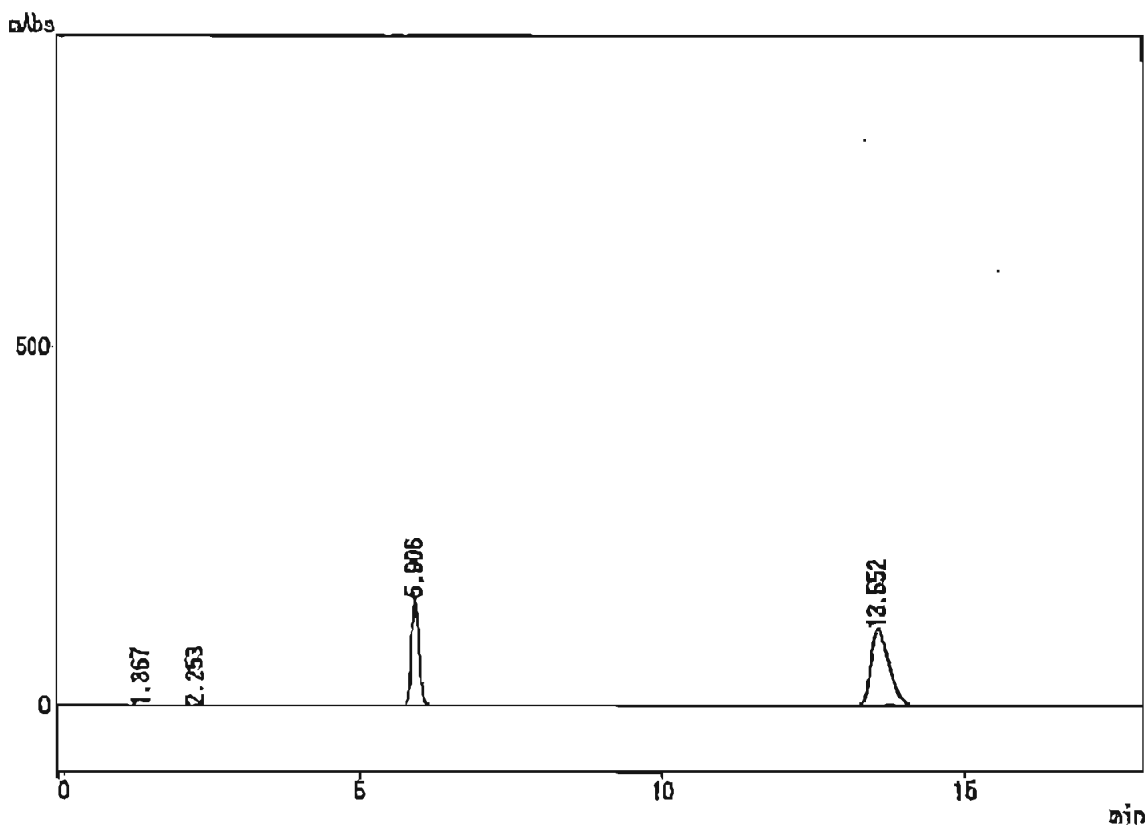
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S051.C01



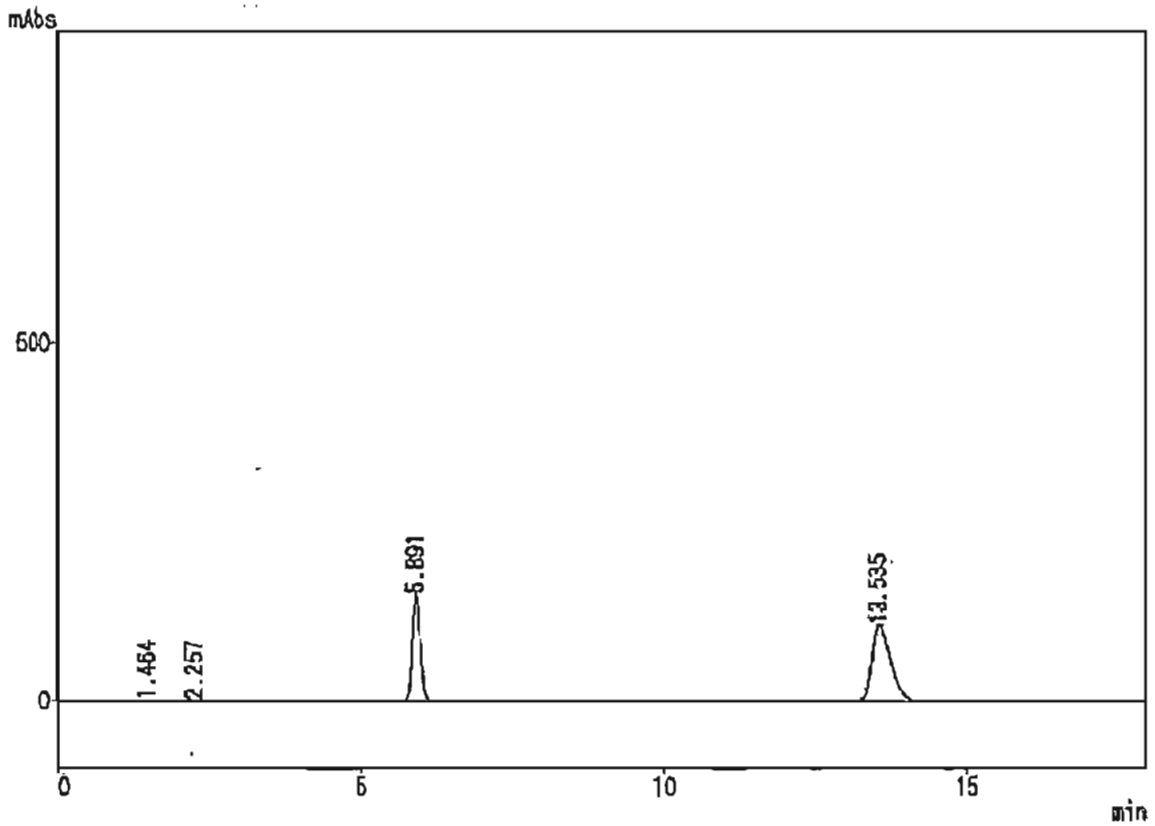
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.367	1222	104	V		0.0332	
2	2.253	1810	251			0.0492	
3	5.806	1378449	164339			37.4831	
4	13.552	2295054	108334	S		62.4244	
		3676538	263028			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=81 Data=AH2S051.D02 02/07/05 17:45:40

Sample: A-01
ID: Initial
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S051.C02



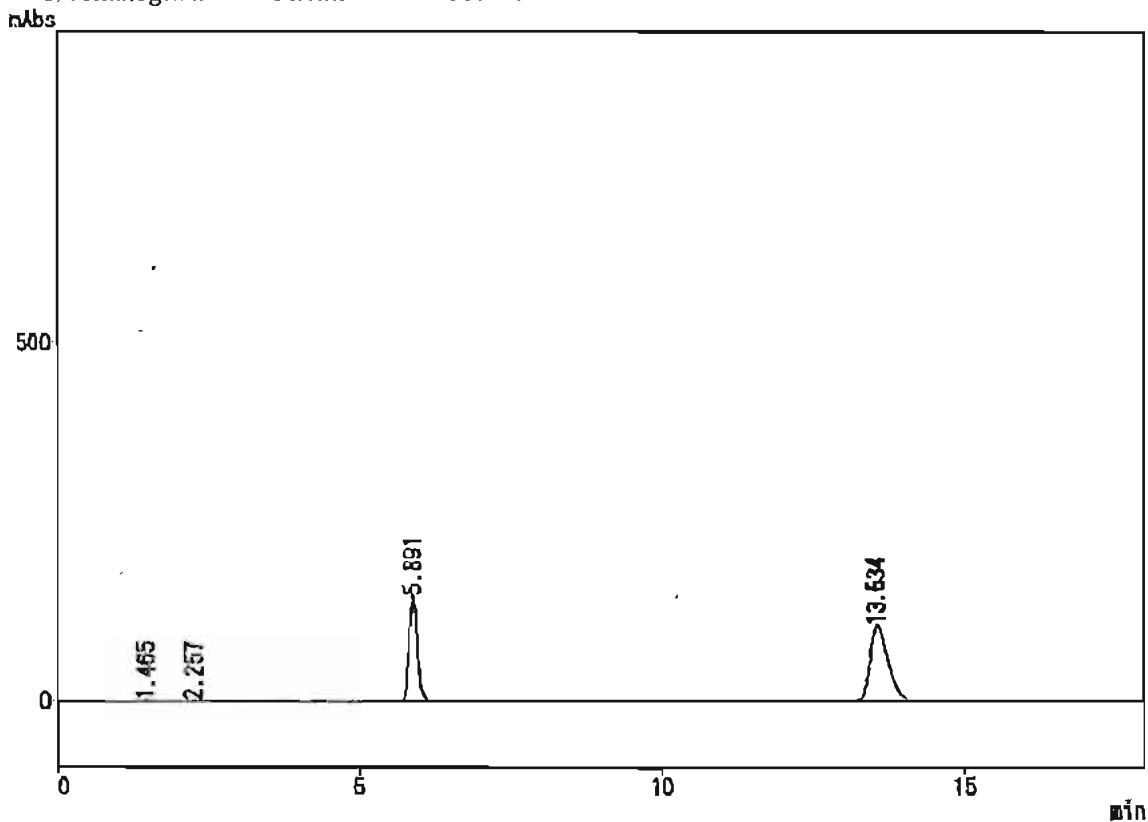
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.464	6178	684	V		0.1688	
2	2.257	3091	383	V		0.0845	
3	5.891	1375224	151679			37.5890	
4	13.535	2274086	108746			62.1576	
		3658577	259471			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=82 Data=AH2S051.D03 02/07/05
18:05:04

Sample: A-02
ID: Initial
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: !AHR!028.MET

*** Chromatogram *** Filename: AH2S051.C03

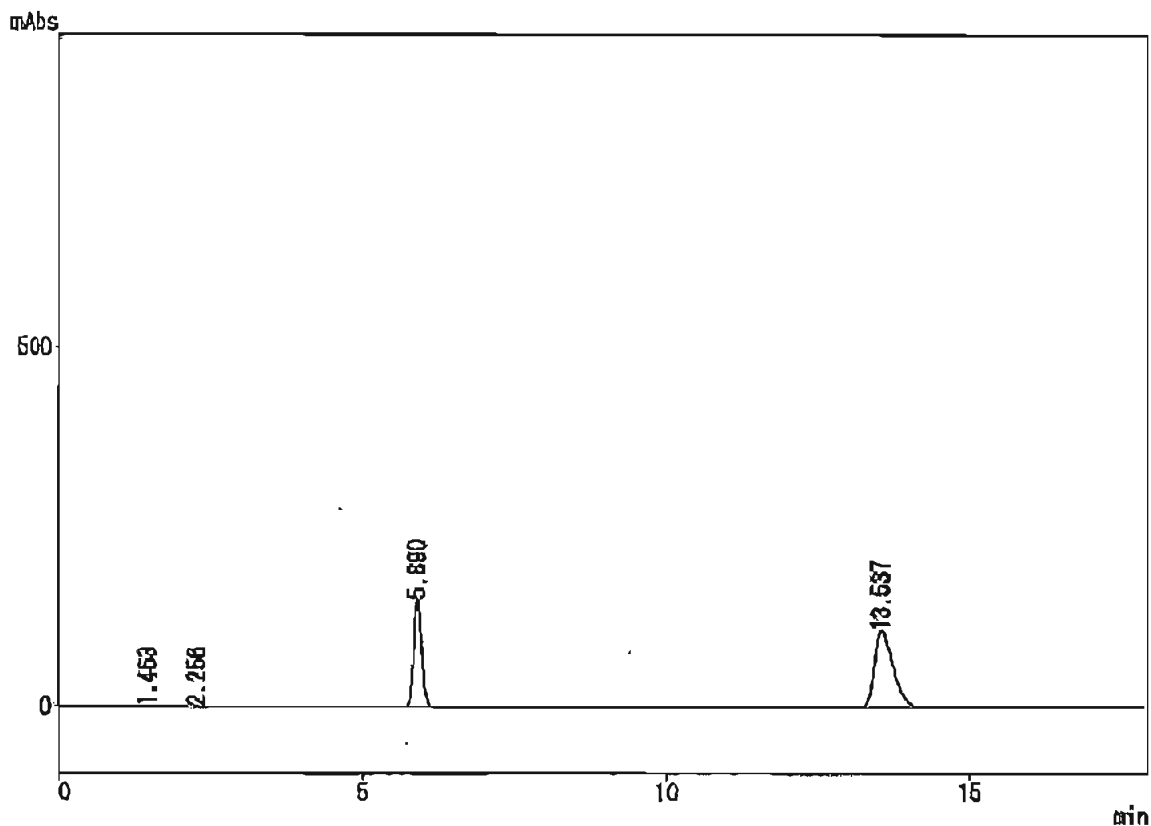


*** Peak Report ***

PKID	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.465	5968	668	V		0.1814	
2	2.257	2050	283	V		0.0564	
3	5.891	1358788	160061			37.3702	
4	13.534	2269283	106534			62.4121	
		3635969	267545			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=83 Data=AH2S051.D04 02/07/05 18:24:24
 Sample: A-03
 ID: Initial
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S051.C04

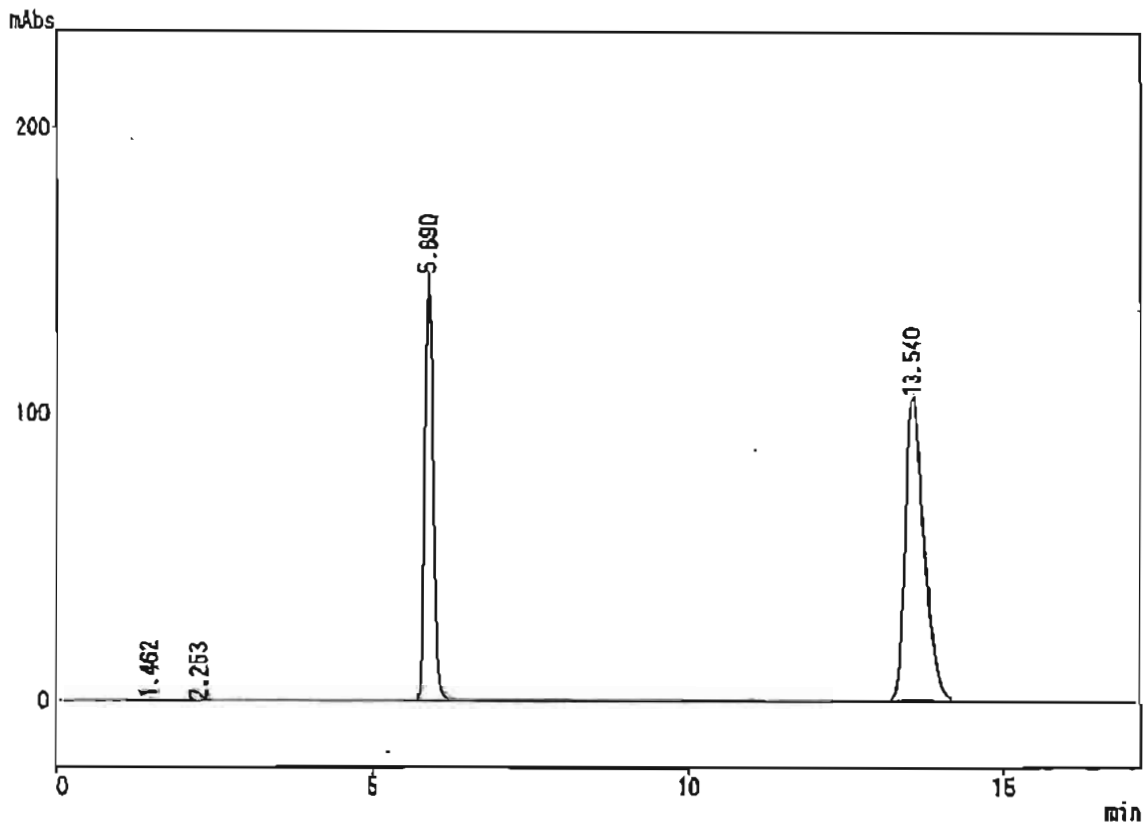


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.483	8481	707	V		0.1773	
2	2.256	1704	223	V		0.0468	
3	5.890	1365588	150905			37.4778	
4	13.537	2269841	108639			62.2982	
		3643673	258474			100.0000	

Sample: A-04
 ID: Initial
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S051.C05



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.462	7533	787	V		0.2075	
2	2.253	1708	194	V		0.0470	
3	5.890	1357737	150024	S		37.3941	
4	13.540	2263909	106350			62.3514	
		3630886	257338			100.0000	

Stability of bromfenac sodium
 Lot No. 02S051

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 12 July 2002

ID	Chromato No.	AM	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Corrosion (%)	pH	FLM	Turbid
STD	AH2S12.C01	4618881	2811272	1.6430						
STD	AH2S12.C10	4540394	2758913	1.6457						
STD	Mean			1.6444	0.10008					
A-01	70°C-1W	AH2S12.C02	4593092	2811409	1.6337	0.09943	100.04	98.32	8.15	--
A-02	70°C-1W	AH2S12.C03	4535814	2784898	1.6288	0.09913	99.74	95.20	8.18	--
A-03	70°C-1W	AH2S12.C04	4555172	2797288	1.6284	0.09911	99.19	95.64	8.16	--
A-04	70°C-1W	AH2S12.C05	4621623	2784880	1.6596	0.10100	100.78	97.27	8.16	--
A-01	60°C-1W	AH2S12.C06	4492662	2783499	1.6257	0.09994	99.55	98.10	8.16	--
A-02	60°C-1W	AH2S12.C07	4476678	2762011	1.6204	0.09862	99.24	96.86	8.17	--
A-03	60°C-1W	AH2S12.C08	4480284	2765521	1.6201	0.09860	99.68	97.27	8.17	--
A-04	60°C-1W	AH2S12.C09	4540394	2758913	1.6457	0.10016	99.94	98.48	8.17	--

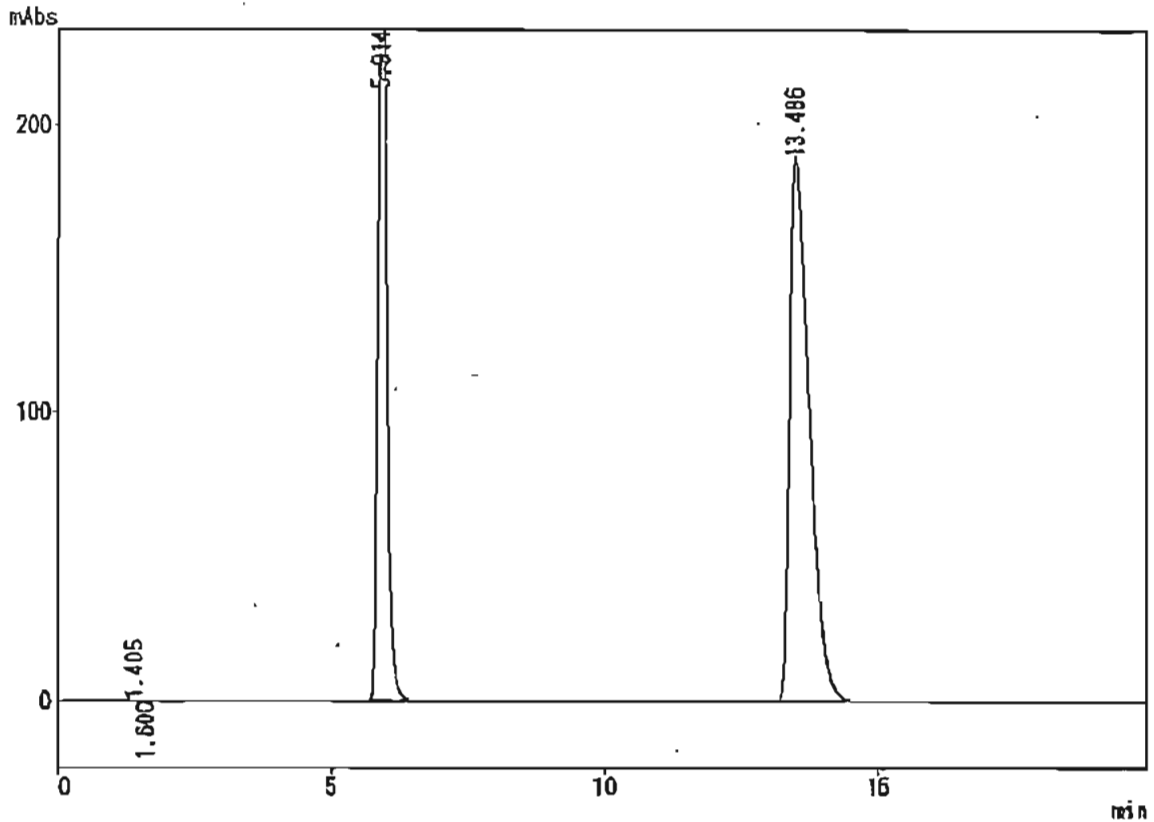
Error corrected 11 July 2002, Sawa

Test Record B (other) Form 7 (1 January 2000)

Test substance	P200 AHR10282B	Test code	P2002B131	Test date	12 July 2002																																																						
Test item				Tester	Shirou Sawa																																																						
IS: 4 July 2002 Preparation used																																																											
STD1: 4 July 2002 Preparation used																																																											
STD2: 2mL of STD1 + 2mL of IS + mobile phase → 20mL																																																											
Sample: 2mL of this product + 2mL of IS + mobile phase → 20mL																																																											
<table border="1"> <thead> <tr> <th colspan="2"></th> <th colspan="4">pH</th> </tr> </thead> <tbody> <tr> <td>A-01</td> <td>70°C-1W</td> <td>8.15</td> <td>—</td> <td>8.7775</td> <td>8.5867</td> </tr> <tr> <td>A-02</td> <td>↓</td> <td>8.16</td> <td>—</td> <td>9.2164</td> <td>9.0164</td> </tr> <tr> <td>A-03</td> <td>↓</td> <td>8.16</td> <td>—</td> <td>8.9999</td> <td>8.8082</td> </tr> <tr> <td>A-04</td> <td>↓</td> <td>8.16</td> <td>—</td> <td>9.0014</td> <td>8.8152</td> </tr> <tr> <td>A-01</td> <td>60°C-1W</td> <td>8.16</td> <td>—</td> <td>9.0410</td> <td>8.9622</td> </tr> <tr> <td>A-02</td> <td>↓</td> <td>8.17</td> <td>—</td> <td>9.1819</td> <td>9.0927</td> </tr> <tr> <td>A-03</td> <td>↓</td> <td>8.17</td> <td>—</td> <td>8.9124</td> <td>8.8374</td> </tr> <tr> <td>A-04</td> <td>↓</td> <td>8.17</td> <td>—</td> <td>8.9412</td> <td>8.8640</td> </tr> </tbody> </table>								pH				A-01	70°C-1W	8.15	—	8.7775	8.5867	A-02	↓	8.16	—	9.2164	9.0164	A-03	↓	8.16	—	8.9999	8.8082	A-04	↓	8.16	—	9.0014	8.8152	A-01	60°C-1W	8.16	—	9.0410	8.9622	A-02	↓	8.17	—	9.1819	9.0927	A-03	↓	8.17	—	8.9124	8.8374	A-04	↓	8.17	—	8.9412	8.8640
		pH																																																									
A-01	70°C-1W	8.15	—	8.7775	8.5867																																																						
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A-03	↓	8.17	—	8.9124	8.8374																																																						
A-04	↓	8.17	—	8.9412	8.8640																																																						
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7/12 13:39	NO.39	pH	8.15	25.9°C																																																							
7/12 13:40	NO.40	pH	8.16	25.9°C																																																							
7/12 13:41	NO.41	pH	8.16	25.9°C																																																							
7/12 13:42	NO.42	pH	8.16	25.9°C																																																							
7/12 13:42	NO.43	pH	8.16	26.1°C																																																							
7/12 13:43	NO.44	pH	8.17	26.4°C																																																							
7/12 13:44	NO.45	pH	8.17	26.1°C																																																							
7/12 13:44	NO.46	pH	8.17	25.8°C																																																							

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=2 Data=AH2S12.D01 02/07/12 12:33:20
 Sample: STD
 ID:
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C01

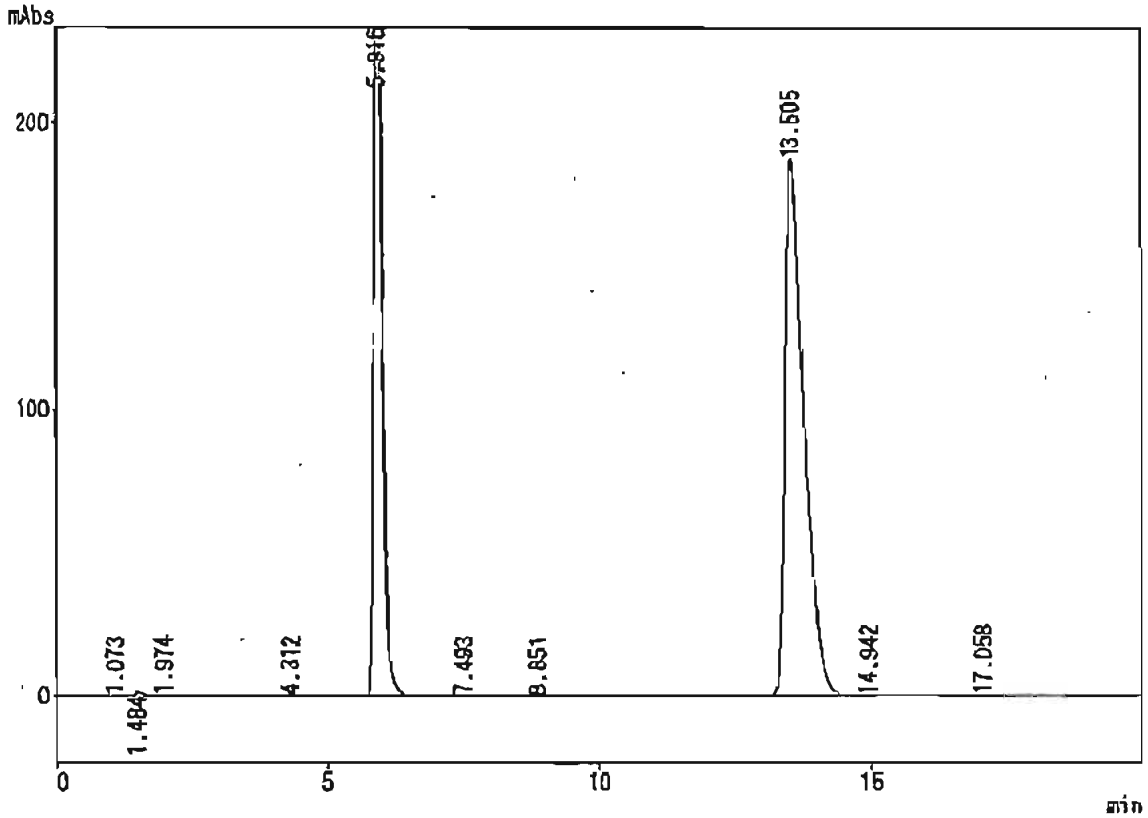


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IONO	CONC	NAME
1	1.405	1859	149	V		0.0250	
2	1.600	2281	229	V		0.0304	
3	5.914	2811272	277954			37.8150	
4	13.486	4618881	188637			62.1298	
		7434273	468989			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=3 Data=AH2S12.D02 02/07/12 12:53:26
 Sample: A-01
 ID: 70°C-1W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C02



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.073	1468	177			0.0197	
2	1.484	18768	1481	V		0.2251	
3	1.974	4428	218	V		0.0594	
4	4.312	2083	242			0.0277	
5	5.916	2811409	278271	S		37.7584	
6	7.493	4002	325	T		0.0537	
7	8.851	1084	78			0.0146	
8	13.505	4593092	187181	S		61.6672	
9	14.942	2539	146	T		0.0341	
10	17.058	8945	355			0.1201	
		7445783	468452			100.0000	

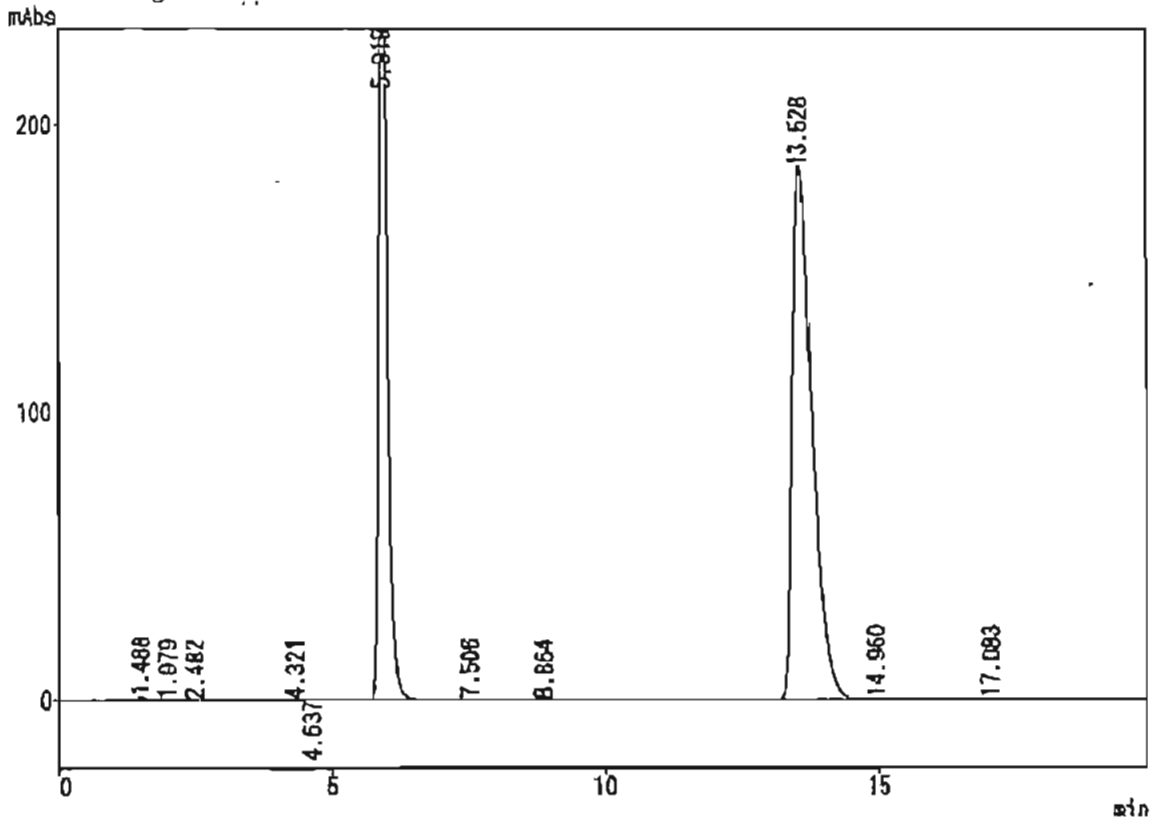
3 - 1/1

02/07/12 13:13:38

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=4 Data=AH2S12.D03 02/07/12
 13:13:32

Sample: A-02
 ID: 70°C-1W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C03

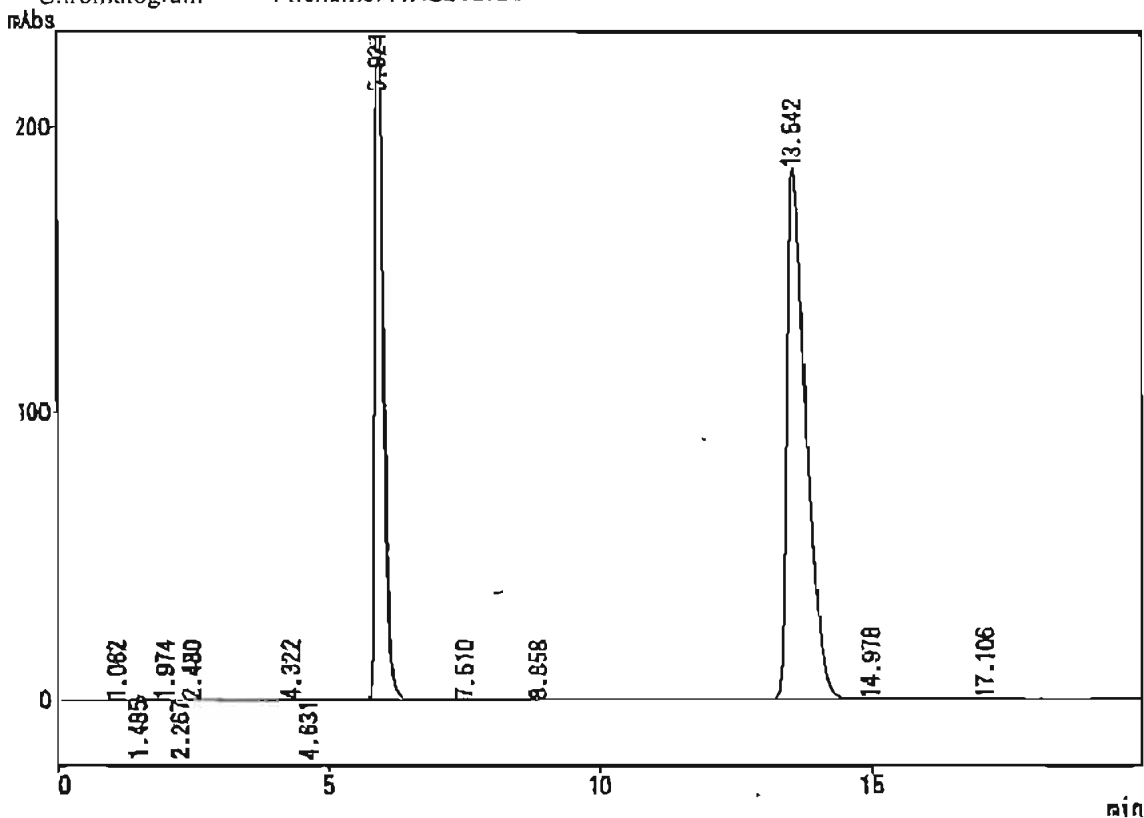


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.488	14720	1319	V		0.2000	
2	1.979	3811	212	V		0.0491	
3	2.482	1023	92	V		0.0139	
4	4.321	2268	238			0.0308	
5	4.637	1092	84	V		0.0148	
6	5.919	2784698	276050	S		37.8364	
7	7.506	4073	333	T		0.0553	
8	8.864	1095	75			0.0149	
9	13.528	4535814	185022	S		81.6292	
10	14.960	2391	141	T		0.0326	
11	17.083	9361	368			0.1231	
		7359843	463933			100.0000	

Sample: A-03
 ID: 70°C-1W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C04

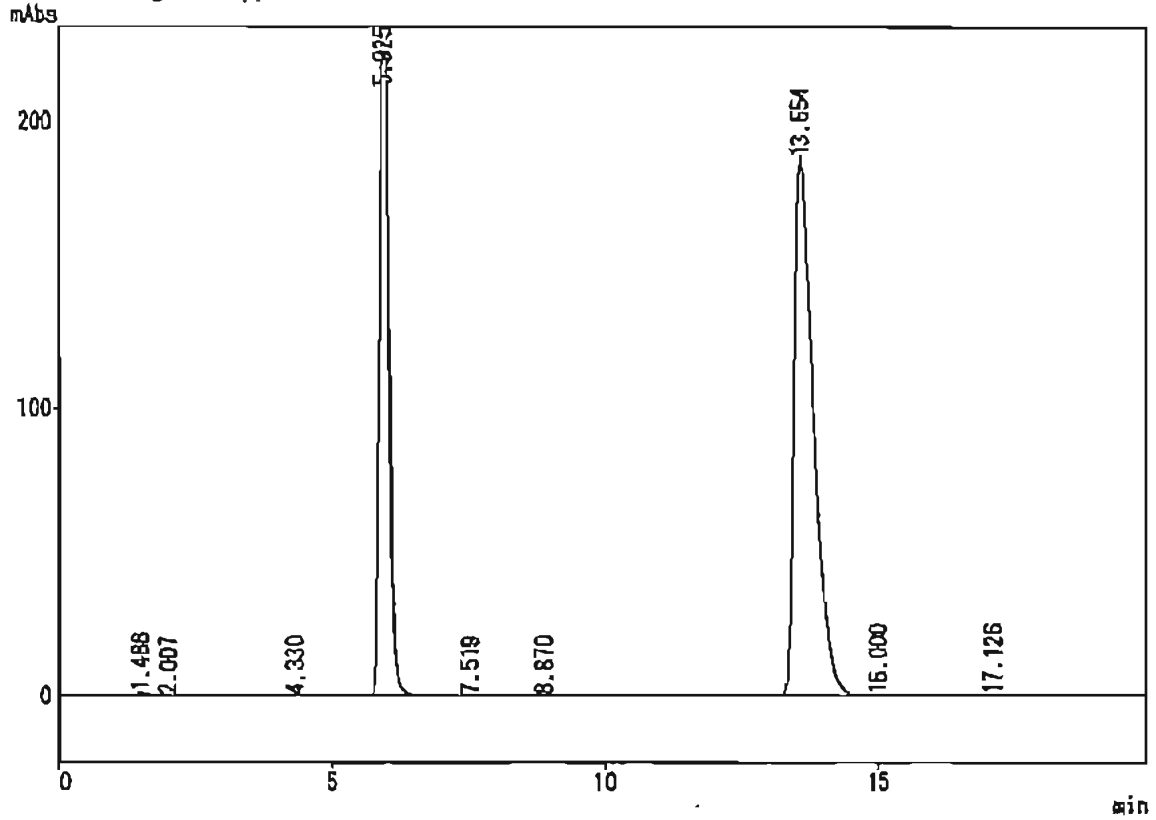


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.082	1198	138	V		0.0182	
2	1.485	16951	1483	V		0.2281	
3	1.974	3712	222	V		0.0502	
4	2.287	1155	130	V		0.0158	
5	2.480	1704	117	V		0.0230	
8	4.322	2334	264	V		0.0318	
7	4.831	1214	85	V		0.0184	
8	5.921	2797288	277876	SV		37.8148	
9	7.510	3857	317	T		0.0521	
10	8.858	1245	81	V		0.0168	
11	13.642	4555172	185511	S		81.6783	
12	14.978	2782	160	T		0.0378	
13	17.106	8780	358	V		0.1184	
		7397371	468737			100.0000	

Sample: A-04
 ID: 70°C-1W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C05

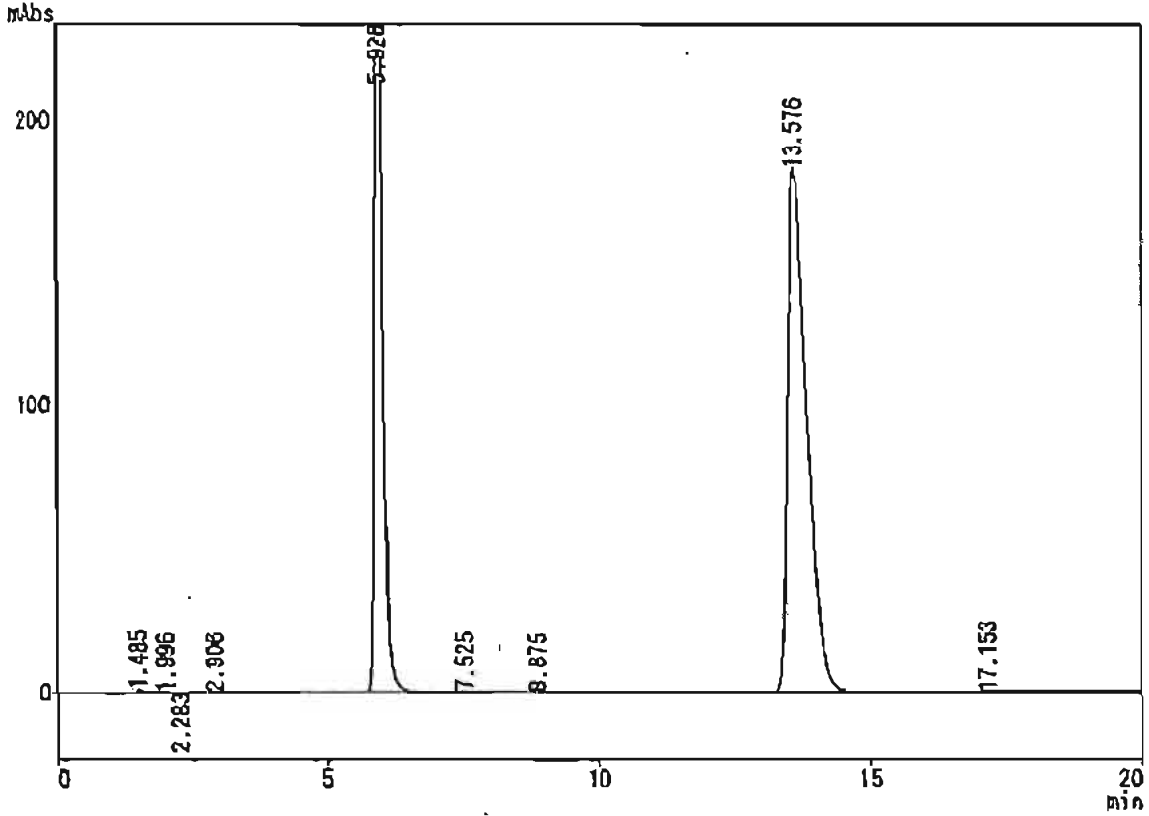


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.488	13817	1274	V		0.1857	
2	2.007	1828	103	V		0.0246	
3	4.330	1887	239			0.0264	
4	5.925	2784880	276061	S		37.4330	
5	7.519	1583	128	T		0.0213	
6	8.870	1531	105			0.0208	
7	13.554	4821523	187705	S		62.1202	
8	15.000	4984	261	T		0.0887	
9	17.126	7833	314			0.1026	
		7439645	468191			100.0000	

Sample: A-01
 ID: 60°C-1W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C06

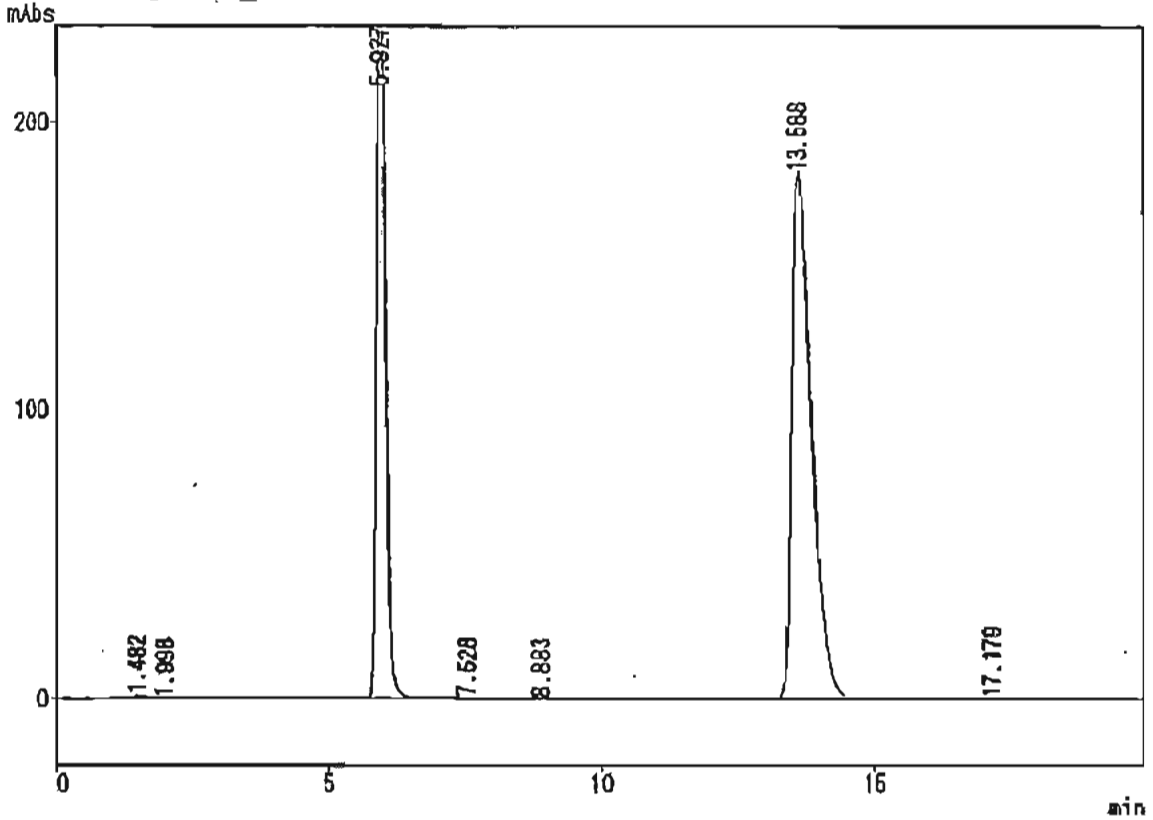


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.485	13490	1165	V		0.1851	
2	1.896	2340	183	V		0.0321	
3	2.283	1708	111	V		0.0234	
4	2.906	1318	117	V		0.0181	
5	5.928	2763499	274808			37.9275	
6	7.525	4030	317			0.0553	
7	8.875	1984	139			0.0272	
8	13.576	4492682	183211			61.8596	
9	17.163	6218	214			0.0718	
		7288287	460245			100.0000	

Sample: A-02
ID: 60°C-1W
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: !AHRJ028.MET

*** Chromatogram *** Filename: AH2S12.C07

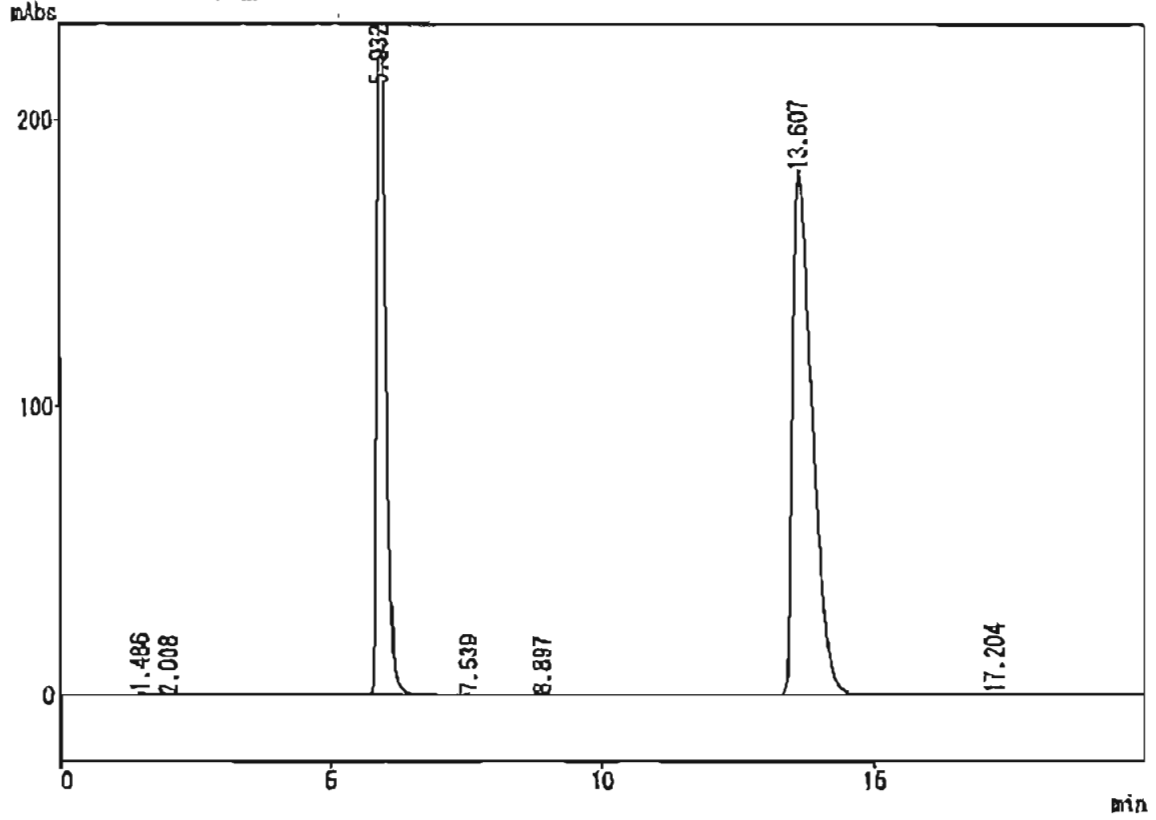


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MX	IDNO	CONC	NAME
1	1.482	13709	1269	V		0.1887	
2	1.998	2000	124	V		0.0275	
3	5.927	2762011	274729			38.0170	
4	7.528	4308	339			0.0693	
5	8.883	2130	148			0.0293	
6	13.588	4475578	182534			61.6030	
7	17.179	5462	221			0.0752	
		7265198	459354			100.0000	

Sample: A-03
ID: 60°C-1W
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: LAHR1028.MET

*** Chromatogram ***_ Filename: AH2S12.C08



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.486	11360	973	V		0.1563	
2	2.008	2312	130	V		0.0318	
3	6.932	2765521	274583			38.0385	
4	7.539	3910	309			0.0538	
5	8.897	2214	156			0.0304	
6	13.607	4480284	182403			61.6244	
7	17.204	4710	197			0.0648	
		7270311	458751			100.0000	

Sample: STD

ID:

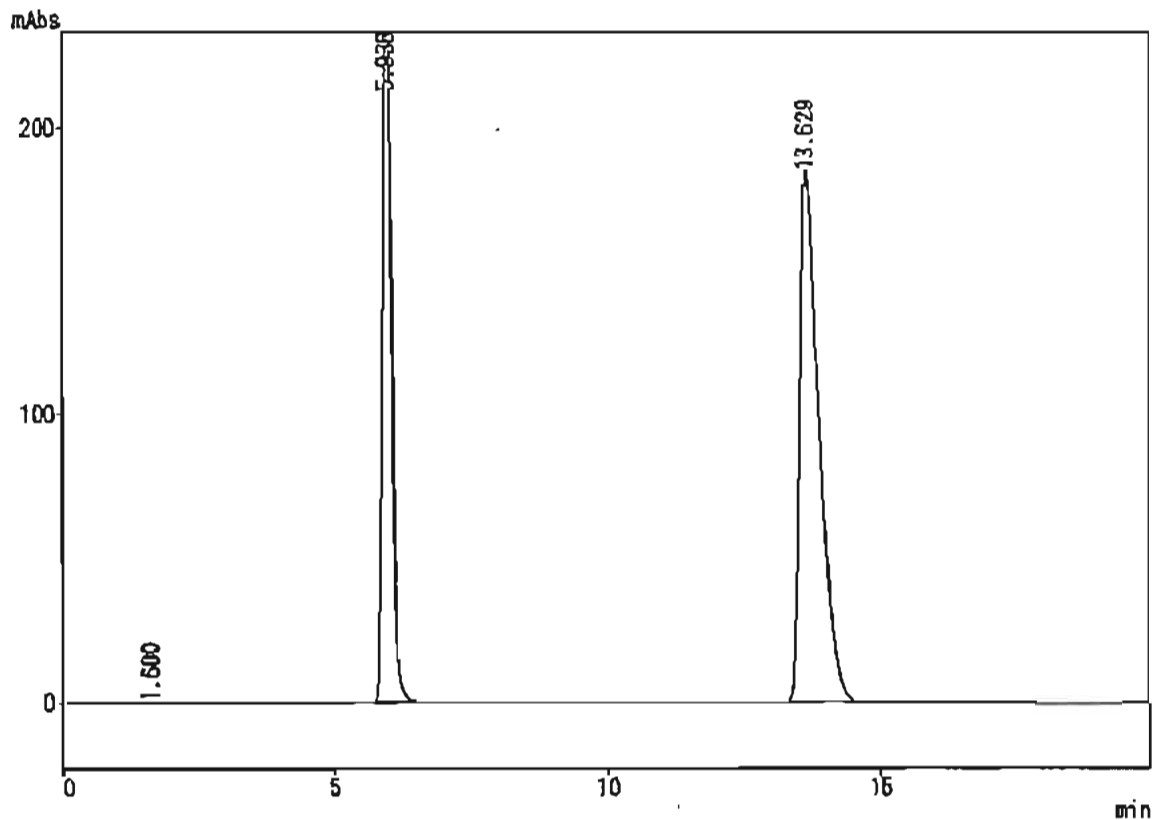
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C10



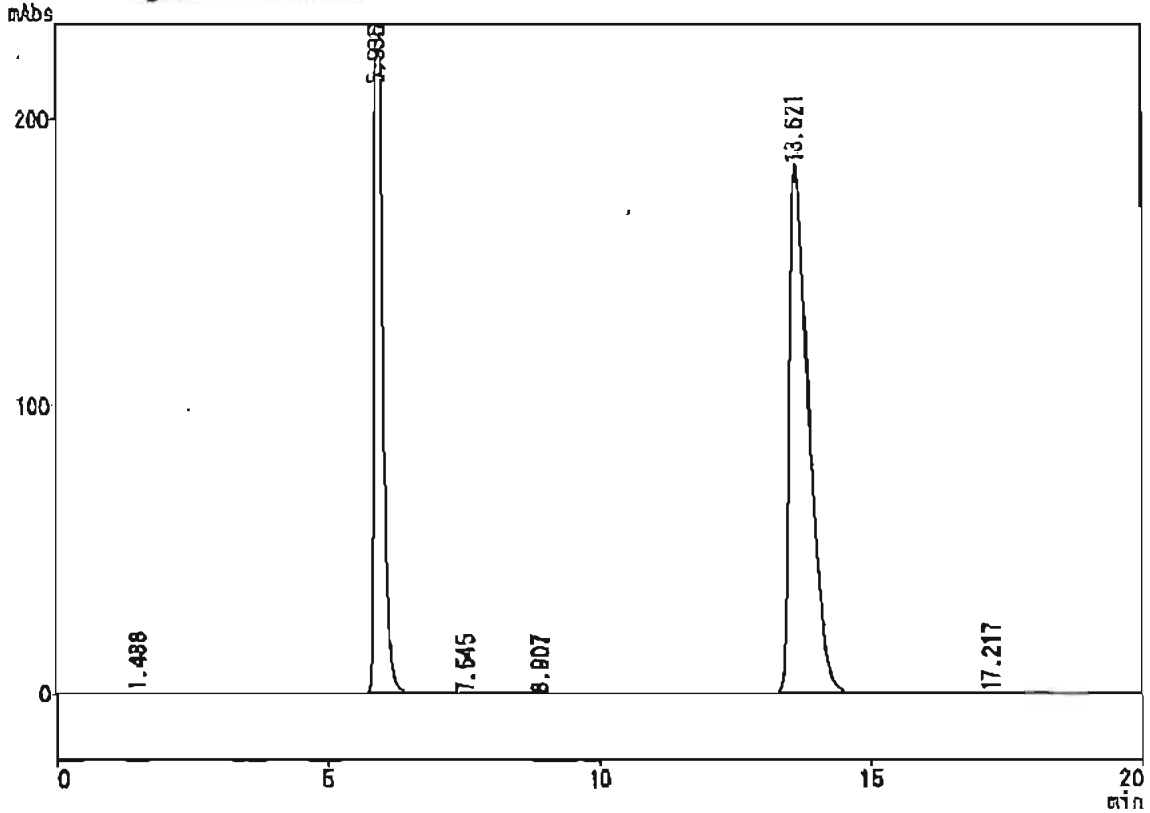
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.600	5243	312	V		0.0716	
2	5.938	2782338	273550			37.7314	
3	13.629	4553477	184898			62.1870	
		7321058	458768			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=10 Data=AH2S12.D09 02/07/12 15:14:08

Sample: A-04
ID: 60°C-1W
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S12.C09



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.488	12318	1081	SV		0.1683	
2	5.936	2768913	274327			37.6900	
3	7.545	1628	120			0.0209	
4	8.907	2378	183			0.0326	
5	13.621	4540384	184383			62.0270	
6	17.217	4483	187			0.0614	
		7320023	460280			100.0000	

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02/07/12 15:34:20

12

Stability of bromfenac sodium
Lot No. 02S051

Test code: P2002B131
Tester: Shirou Sawa
Test date: 19 July 2002

ID	Chromato No.	AM	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.L.M.	Turbid	Permeation (%)
STD	AH2S19.C01	4590898	2771294	1.8688							
STD	AH2S19.C10	4541555	2747359	1.8531							
STD	Mean			1.8549	0.10010						
A-01	70°C-2W	AH2S19.C02	4601736	2766631	1.8693	0.10097	101.59	94.31	8.17	—	7.17
A-02	70°C-2W	AH2S19.C03	4586591	2750687	1.8565	0.10020	99.81	93.00	8.18	—	6.82
A-03	70°C-2W	AH2S19.C04	4588564	2761893	1.8616	0.10051	100.69	93.29	8.15	—	7.26
A-04	70°C-2W	AH2S19.C05	4677878	2747028	1.7029	0.10300	102.77	95.22	8.15	—	7.35
A-01	60°C-2W	AH2S19.C06	4497171	2741726	1.6366	0.09899	98.60	98.47	8.17	—	3.14
A-02	60°C-2W	AH2S19.C07	4479387	2744786	1.8320	0.09871	98.33	95.25	8.16	—	3.13
A-03	60°C-2W	AH2S19.C08	4477249	2746480	1.6302	0.09861	98.69	95.79	8.16	—	2.94
A-04	60°C-2W	AH2S19.C09	4565943	2732765	1.6712	0.10109	100.87	97.66	8.16	—	3.18

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2002B131	Test date	19 July 2002
Test item				Tester	Shirou Sawa
IS: 15 July 2002 Preparation used. 0.03314g of methyl p-hydroxybenzoate + mobile phase → 100mL					
STD1: 15 July 2002 Preparation used. 0.02002g of AHR10282B + mobile phase → 20mL					
STD1: 4 July 2002 Preparation used					
STD2: 2mL of STD1 + 2mL of IS → 20mL → 20mL inject					
		pH	Ext. app.	Weight change	
A-01	70°C-2W	8.17	—	8.9141	8.5367
A-02	↓	8.16	—	9.1572	8.7816
A-03	↓	8.15	—	8.9010	8.5199
A-04	↓	8.15	—	8.8113	8.4323
A-01	60°C-2W	8.17	—	9.0512	8.8819
A-052*1	↓	8.16	—	9.1321	8.9608
A-063*1	↓	8.16	—	8.8133	8.6616
A-074*1	↓	8.16	—	8.8588	8.6930
*1 errors corrected					
'02.07.19 Sawa					

7/19 17:26 pH 8.17
NO.45 25.1°C

7/19 17:27
NO.46 pH 8.15
25.4°C

7/19 17:29
NO.47 pH 8.15
25.5°C

7/19 17:29
NO.48 pH 8.15
25.6°C

7/19 17:30
NO.49 pH 8.17
25.9°C

7/19 17:30
NO.50 pH 8.15
26.0°C

7/19 17:31
NO.51 pH 8.15
25.8°C

7/19 17:32
NO.52 pH 8.16
25.8°C

Sample: STD

ID:

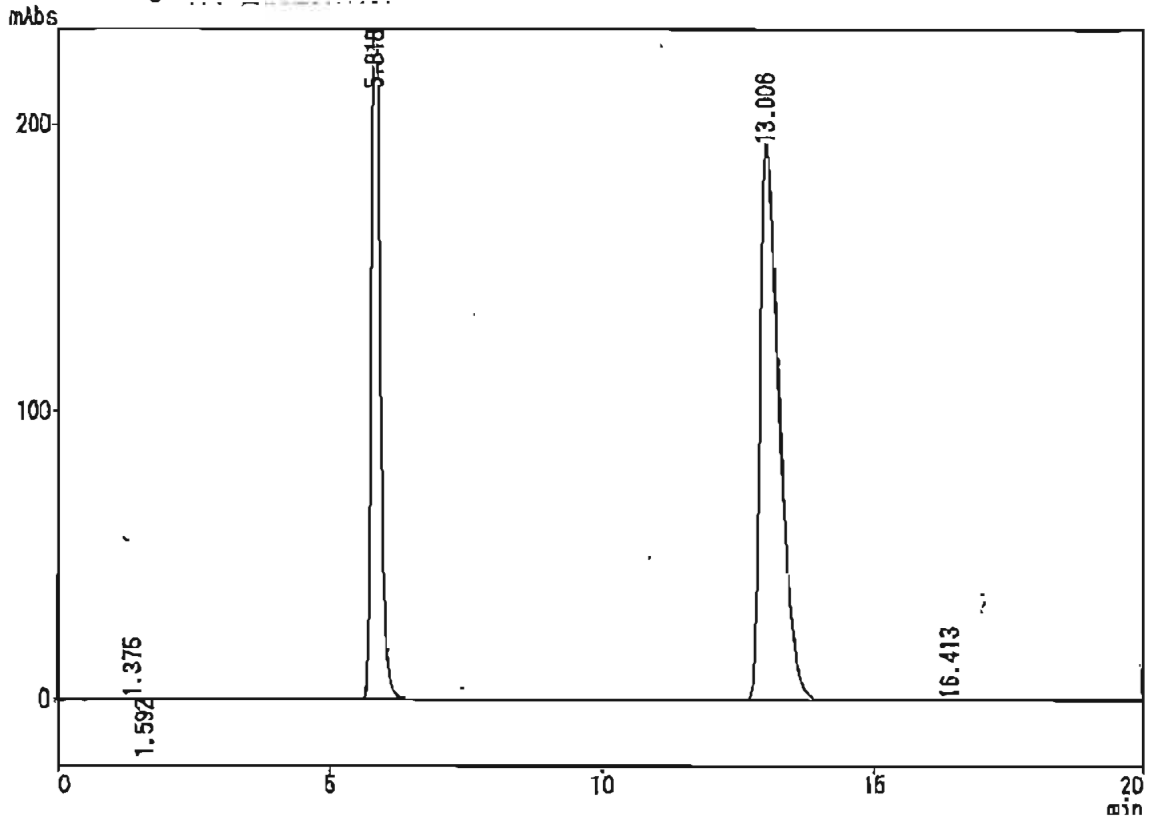
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: IAHRI028.MET

*** Chromatogram *** Filename: AH2S19.C01



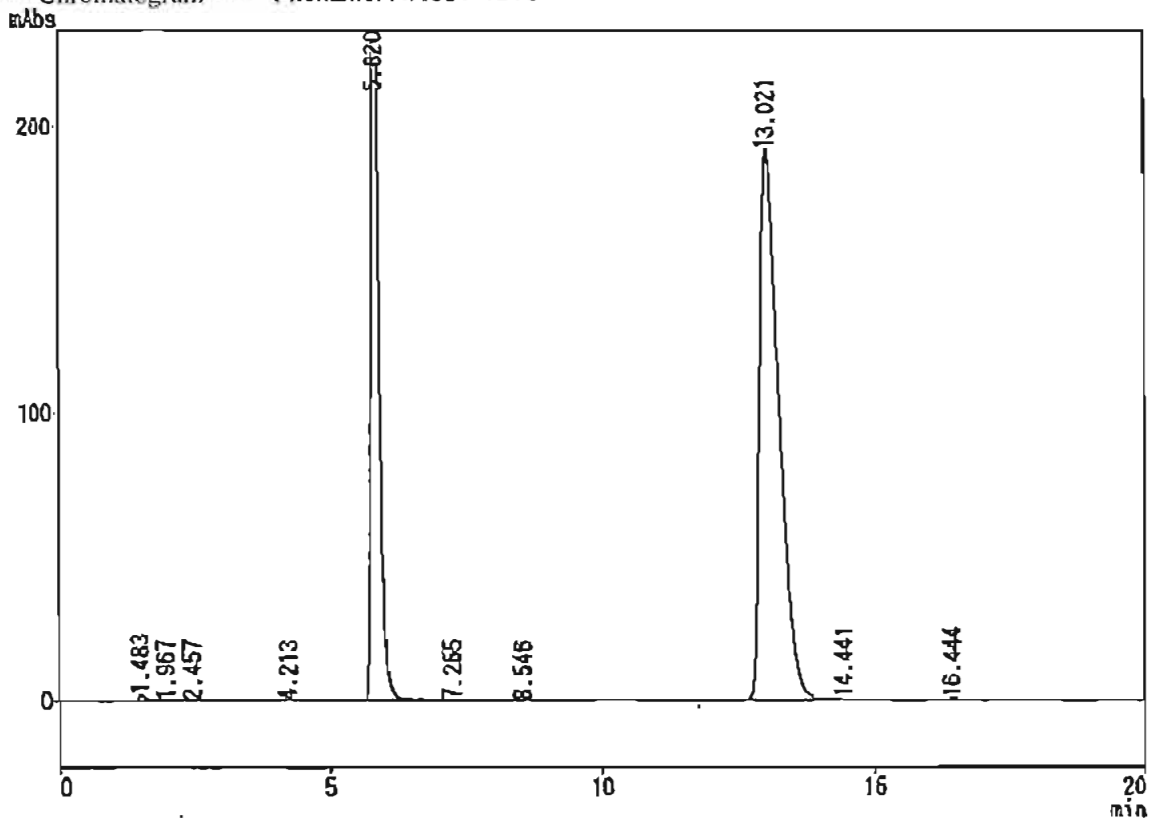
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.378	4314	398	V		0.0585	
2	1.592	2740	230	V		0.0372	
3	5.818	2771294	278151			37.6007	
4	13.006	4590898	193320			62.2890	
6	16.413	1077	65			0.0148	

7970323 472183 100.0000

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=3 Data=AH2S19.D02 02/07/19 15:17:00
 Sample: A-01
 ID: 70°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C02



*** Peak Report ***

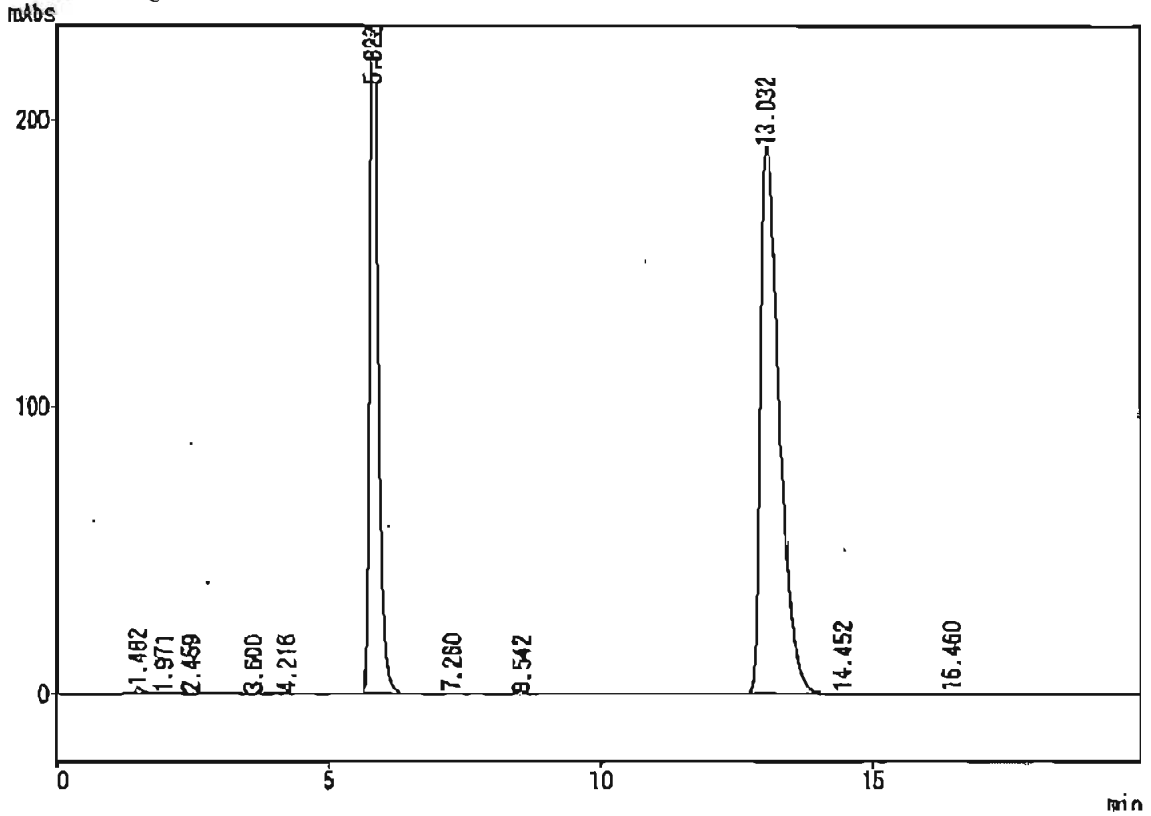
PKID	TIME	AREA	HEIGHT	PK	IDRO	CONC	NAME
1	1.483	25298	2357	V		0.3408	
2	1.987	6279	324	V		0.0845	
3	2.457	1722	156	V		0.0232	
4	4.213	3119	372			0.0420	
5	5.820	2758631	276295	S		37.1160	
6	7.255	5747	472	T		0.0774	
7	8.546	1140	75			0.0163	
8	13.021	4601736	192331	S		61.9590	
9	14.441	8282	454	T		0.1116	
10	16.444	17113	712			0.2304	
		7427065	473548			100.0000	

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02/07/19 15:37:10

Sample: A-02
 ID: 70°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C03

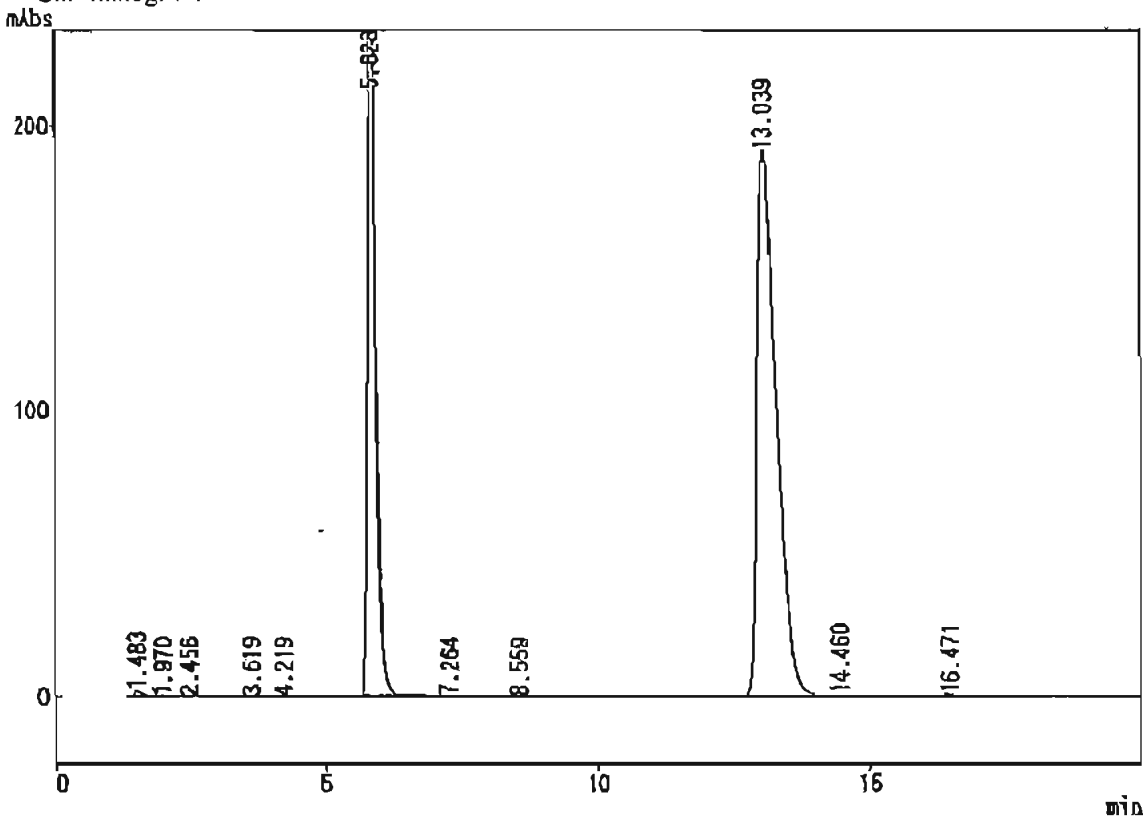


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.482	26318	2458	V		0.3567	
2	1.971	6439	335	V		0.0873	
3	2.459	1857	155	V		0.0225	
4	3.800	1804	70			0.0245	
5	4.216	2583	333			0.0350	
6	5.822	2750687	275772	S		37.2818	
7	7.200	6074	504	T		0.0823	
8	8.542	1264	88			0.0171	
9	13.032	4556591	190788	S		81.7580	
10	14.452	8073	439	T		0.1094	
11	16.460	16647	709			0.2258	
		7378138	471629			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=5 Data=AH2S19.D04 02/07/19 15:57:12
 Sample: A-03
 ID: 70°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C04



*** Peak Report ***

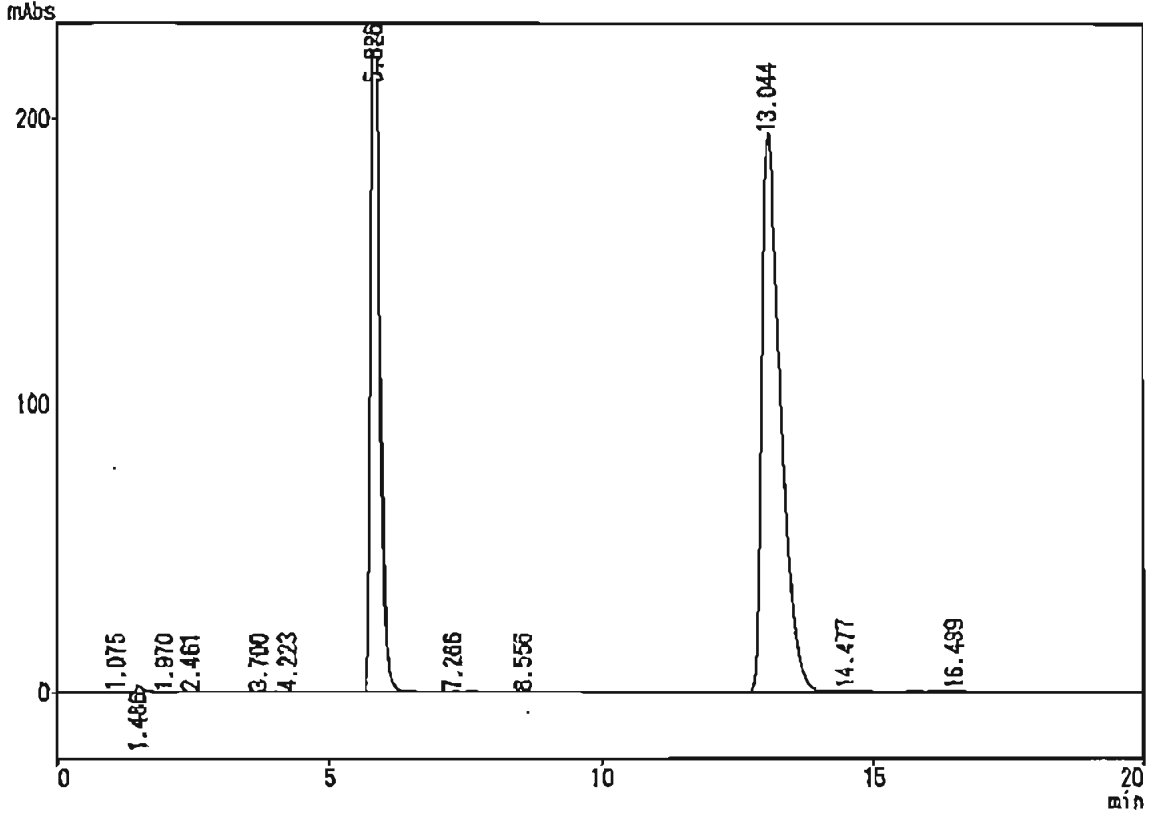
PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.483	24748	2279	V		0.3336	
2	1.970	6381	328	V		0.0857	
3	2.456	1616	155	V		0.0218	
4	3.619	1453	70	V		0.0196	
5	4.219	2654	344			0.0358	
6	5.823	2761593	276799	S		37.2209	
7	7.264	5734	471	T		0.0779	
8	8.558	1420	99			0.0191	
9	13.039	4588534	191739	S		61.8452	
10	14.460	8359	459	T		0.1127	
11	16.471	16841	710			0.2283	
		7419464	473452			100.0000	

S - 1/1

02/07/19 16:17:27

Sample: A-04
 ID: 70°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C05

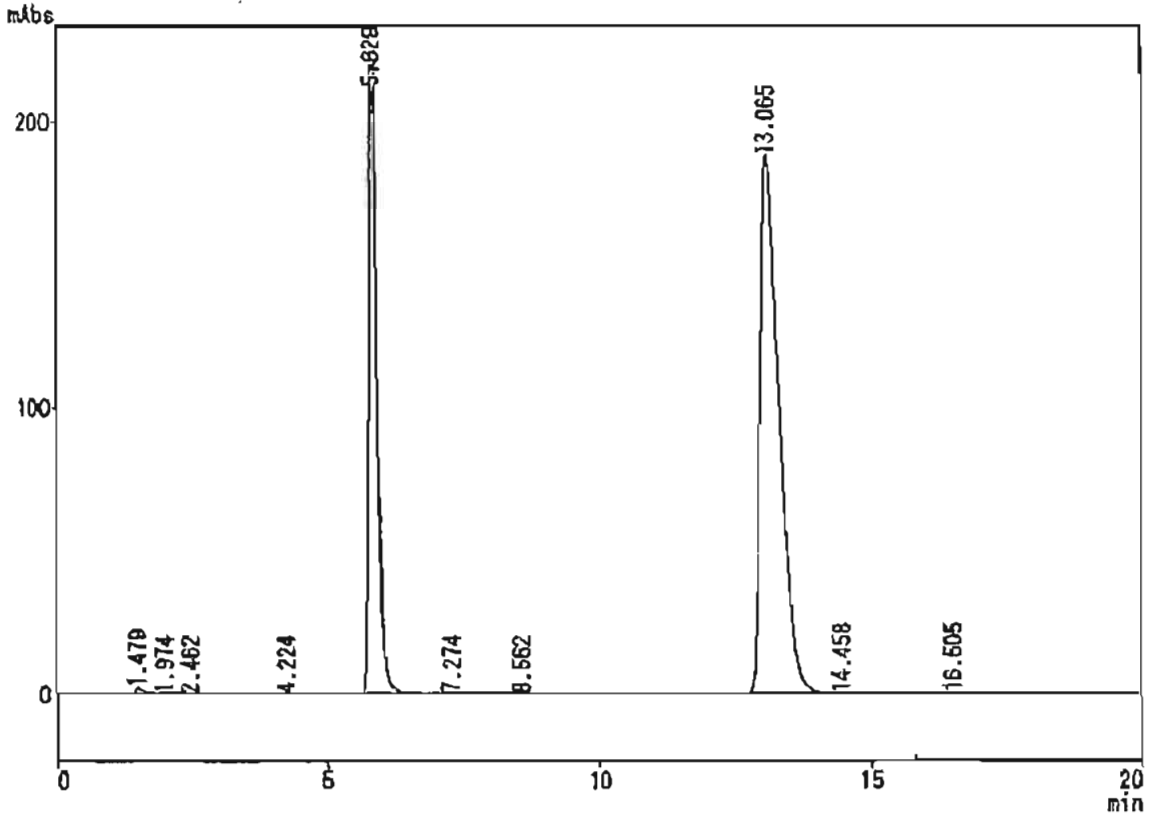


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.075	1514	184			0.0202	
2	1.488	22820	2141	V		0.3045	
3	1.870	4598	211	V		0.0614	
4	2.461	1823	150	V		0.0243	
5	3.700	1404	67	V		0.0187	
6	4.223	2997	378			0.0400	
7	6.828	2747028	275579	S		36.6538	
8	7.286	2468	205	T		0.0329	
9	8.556	1275	101			0.0170	
10	13.044	4877878	194977	S		62.4182	
11	14.477	13740	703	T		0.1833	
12	16.489	16930	688			0.2259	
		7494576	475395			100.0000	

Sample: A-01
 ID: 60°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: JAHRI028.MET

*** Chromatogram *** Filename: AH2S19.C06

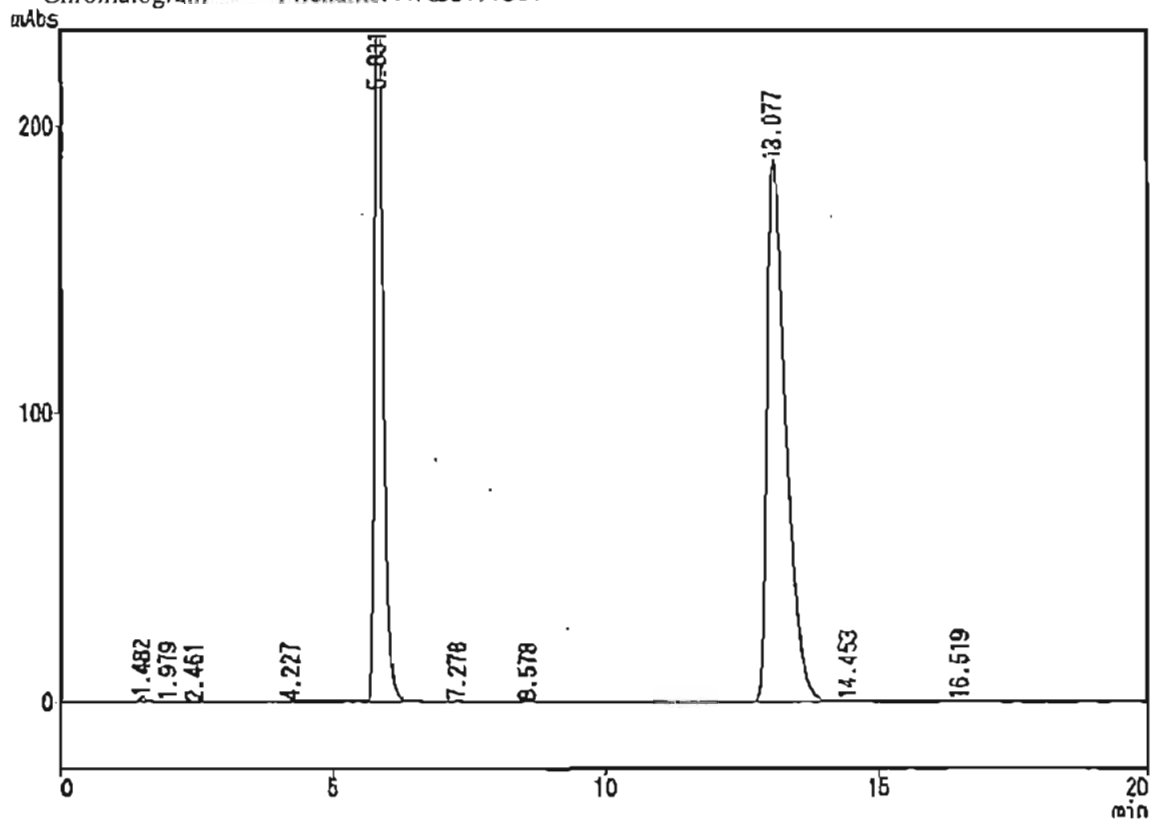


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.479	27607	2569	V		0.3792	
2	1.974	5439	272	V		0.0747	
3	2.462	1497	124	V		0.0206	
4	4.224	1013	132			0.0139	
5	6.829	2741725	275249	S		37.6598	
6	7.274	5228	428	T		0.0718	
7	8.562	1720	126			0.0237	
8	13.065	4487171	188231	S		61.6349	
9	14.458	1675	98	T		0.0230	
10	16.505	7163	301			0.0984	
		7280245	467529			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=8 Data=AH2S19.D07 02/07/19 16:57:30
 Sample: A-02
 ID: 60°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C07



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.482	18274	1787	V		0.2515	
2	1.979	4011	204	V		0.0552	
3	2.461	1057	98	V		0.0146	
4	4.227	1108	138			0.0152	
5	5.831	2744988	275425	S		37.7808	
6	7.278	5494	451	T		0.0758	
7	8.578	1784	130			0.0245	
8	13.077	4479387	187789	S		81.8521	
9	14.453	1451	74	T		0.0200	
10	16.519	8036	334			0.1106	
		7265588	466427			100.0000	

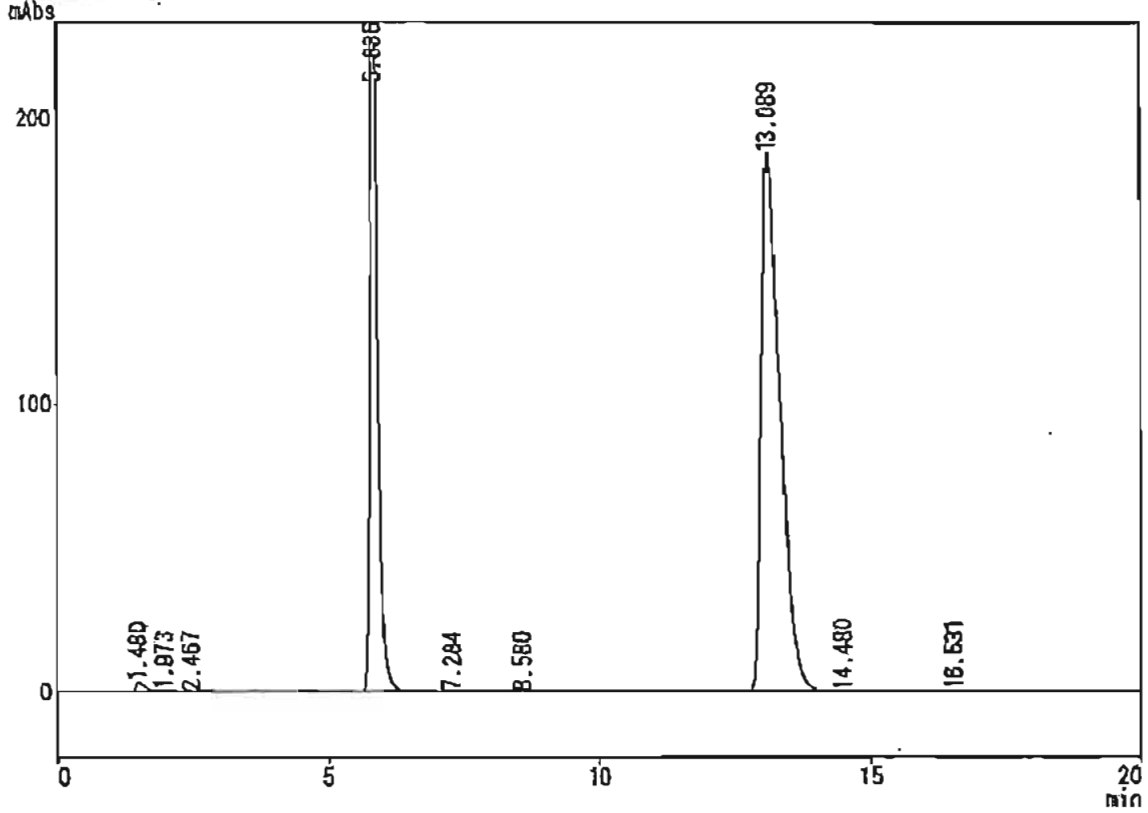
8 - 1/1

02/07/19 17:17:37

9

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=9 Data=AH2S19.D08 02/07/19 17:17:36
 Sample: A-03
 ID: 60°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C08



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.480	42471	3394	V		0.5824	
2	1.973	8211	412	V		0.1126	
3	2.467	2729	185	V		0.0374	
4	5.835	2748480	276743	S		37.8598	
5	7.284	5180	422	T		0.0710	
6	8.580	2189	154			0.0300	
7	13.089	4477249	187667	S		61.3917	
8	14.480	1446	83	T		0.0198	
9	16.531	6962	294			0.0856	
		7292918	468353			100.0000	

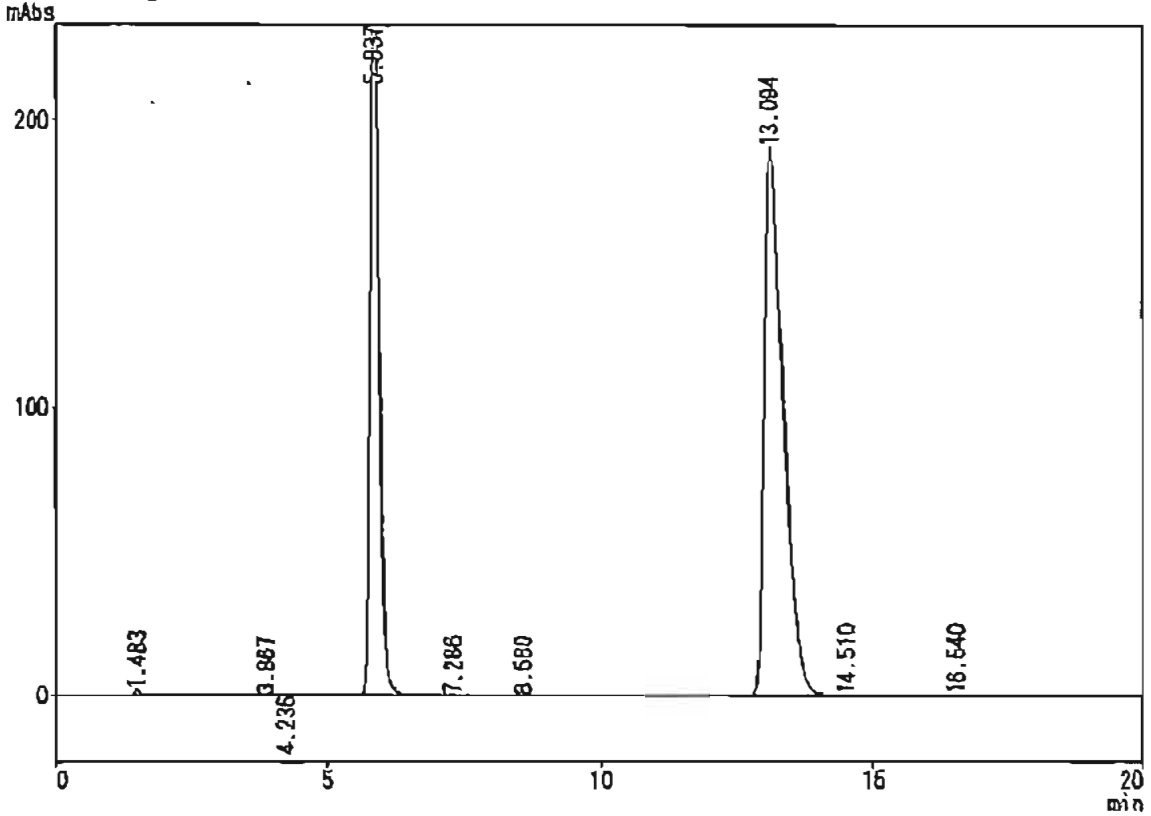
9 - 1/1

02/07/19 17:37:50

10

Sample: A-04
 ID: 60°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C09

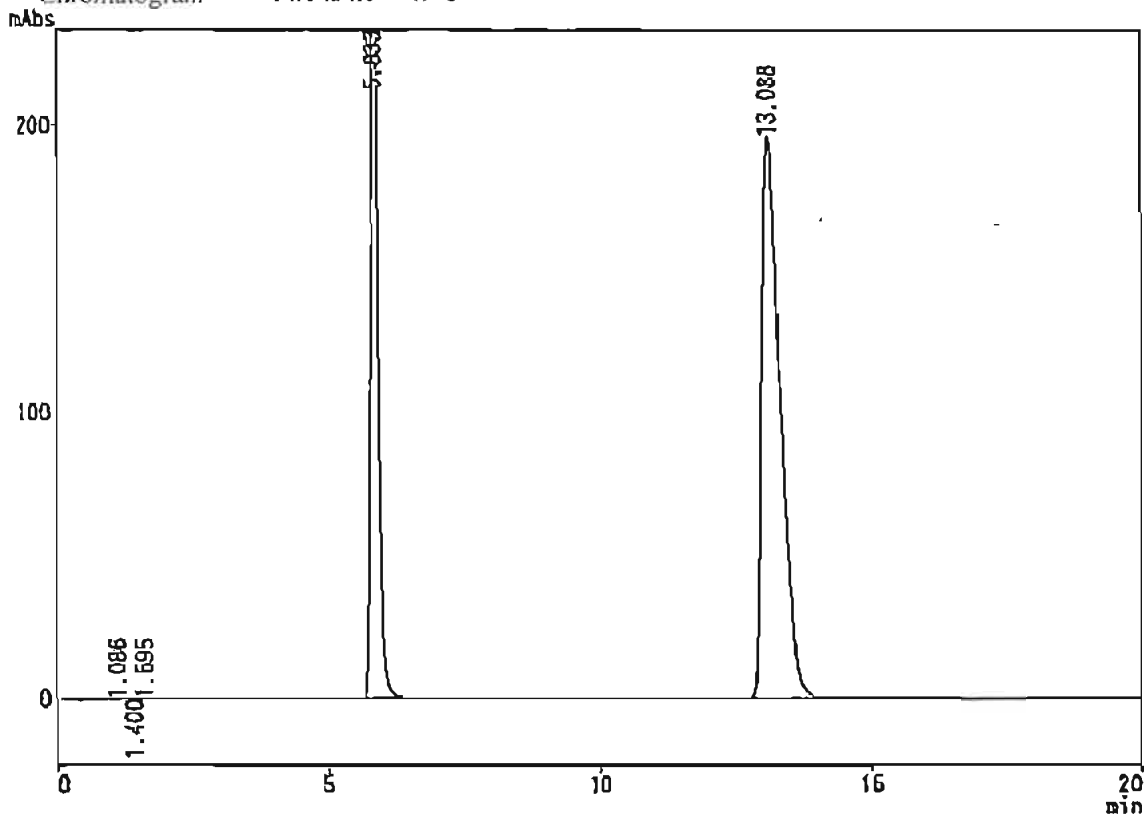


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.483	18570	2003	V		0.2531	
2	3.887	1451	64	V		0.0198	
3	4.236	1958	182	V		0.0267	
4	5.837	2732385	274668	S		37.2440	
6	7.286	2101	177	T		0.0286	
6	8.580	2217	158	T		0.0302	
7	13.094	4585943	190680	S		62.2508	
8	14.510	3845	210	T		0.0524	
9	16.540	8940	293	T		0.0848	
		7338388	488445			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=12 Data=AH2S19.D10 02/07/19 18:29:46
 Sample: STD
 ID:
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2S19.C10



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.088	1128	132			0.0155	
2	1.400	1487	143	V		0.0201	
3	1.595	2467	217	V		0.0338	
4	6.833	2747359	294572			37.6881	
5	13.088	4541555	198213			52.2645	
		7293878	491277			100.0000	

12 - 1/1

02/07/19 18:49:51

12

Stability of bromfenac sodium
 Lot No. 02S051

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 5 August 2002

ID	Chromato No.	AHP	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Corrosion (%)	pH	F.I.M.	Turbid Formation (%)	
STD	AH2T05.C01	2285663	1361530	1.6787							
STD	AH2T05.C06	2275845	1353252	1.6816							
STD	Mean			1.6802	0.10030						
A-01	60°C-4W	AH2T05.C02	2238745	1353782	1.6522	0.09863	99.24	82.67	8.16	--	6.72
A-02	60°C-4W	AH2T05.C03	2207348	1353829	1.6304	0.09733	98.95	90.93	8.18	--	6.21
A-03	80°C-4W	AH2T05.C04	2232104	1354214	1.6483	0.09840	98.48	91.97	8.15	--	6.61
A-04	80°C-4W	AH2T05.C05	2298583	1353639	1.6974	0.10133	101.11	94.28	8.16	--	6.78

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2002B131	Test date	5 August 2002
Test item				Tester	Shirou Sawa
STD: 0.02006g of AHR10282B + mobile phase → 20mL					
2mL of above solution + 2mL of IS + mobile phase → 20mL					
Sample: 2mL of this product + 2mL of IS + mobile phase → 20mL					
Errors c					
	pH	Ext. app.	Weight change	'02.08.0	
A-05	Initial	8.19	---	8.5568	8.56
A-06	Initial	8.19	---	8.4745	
A-05	70°C-2W	8.14	- +	8.4877	8.09249
A-06	70°C-2W	8.15	---	8.4894	8.09057
A-05	60°C-2W	8.17	---	8.4986	8.31388
A-06	60°C-2W	8.17	---	8.4796	8.29993
A-01	60°C-4W	8.15	---	8.9191	8.5653
A-02	60°C-4W	8.16	---	9.0937	8.7557
A-03	60°C-4W	8.15	---	8.8635	8.5192
A-04	60°C-4W	8.16	---	8.8862	8.5331
<p>8/05 15:32 NO.18 PH 8.15 27.5°C</p> <p>8/05 15:34 NO.19 PH 8.16 27.7°C</p> <p>8/05 15:34 NO.20 PH 8.15 27.5°C</p> <p>8/05 15:35 NO.21 PH 8.15 27.3°C</p> <p>8/05 15:36 NO.22 PH 8.15 27.6°C</p> <p>8/05 15:37 NO.23 PH 8.15 27.6°C</p> <p>8/05 15:38 NO.24 PH 8.15 27.2°C</p> <p>8/05 15:39 NO.25 PH 8.15 27.9°C</p> <p>8/05 15:40 NO.26 PH 8.17 27.7°C</p> <p>8/05 15:42 NO.27 PH 8.17 27.8°C</p>					

Sample: STD

ID:

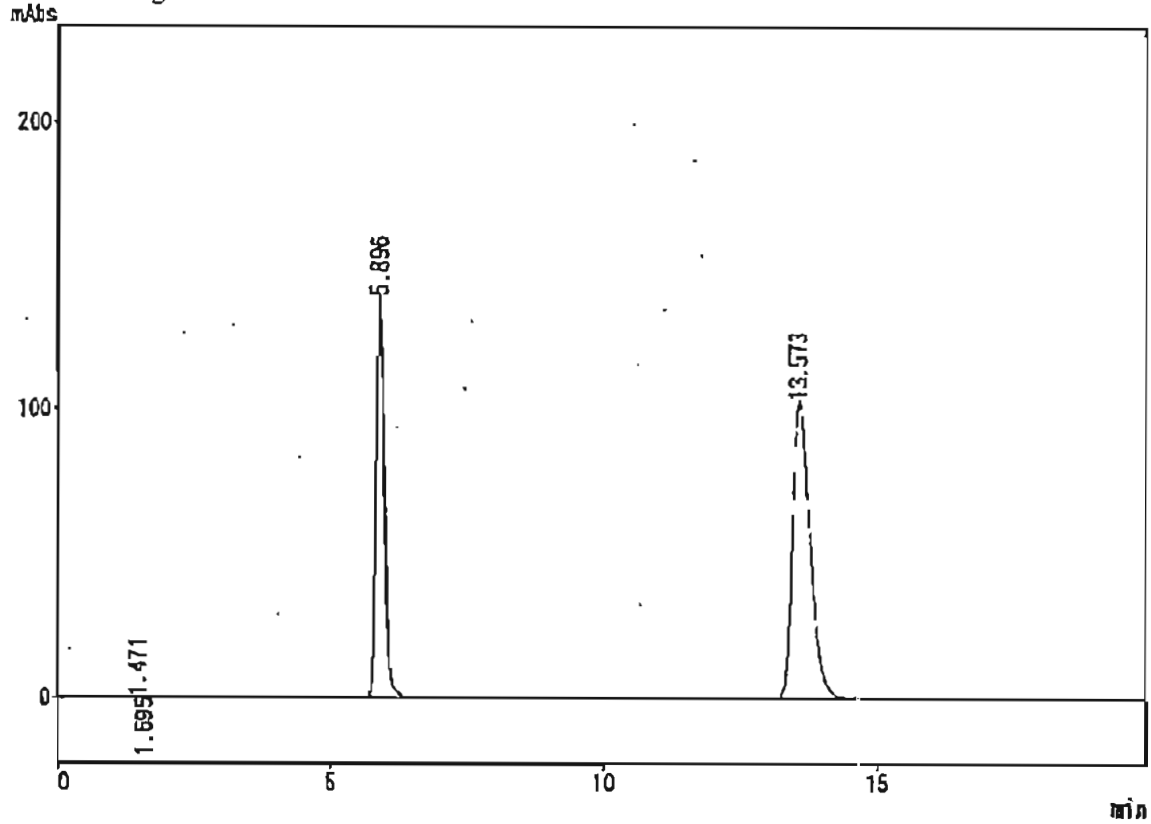
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: IAHRI028.MET

*** Chromatogram *** Filename: AH2T05.C01



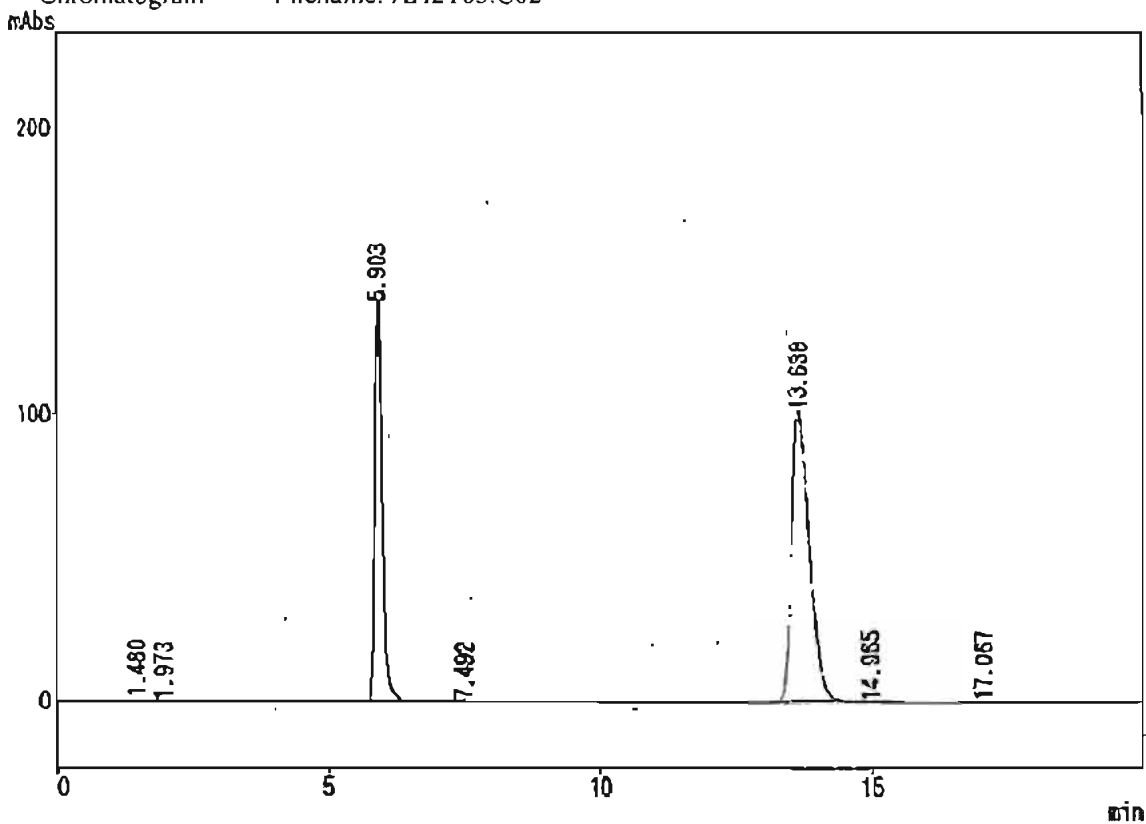
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.471	2112	169	V		0.0579	
2	1.595	2024	197	V		0.0554	
3	5.896	1361530	140426			37.2886	
4	13.573	2285863	103113			82.5981	
		3651329	243904			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=3 Data=AH2T05.D02 02/08/05 16:13:42

Sample: A-01
ID: 60°C-4W
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2T05.C02



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	NK	IDNO	COND	NAME
1	1.480	8402	878	V		0.2324	
2	1.973	2141	108	V		0.0592	
3	5.903	1353782	139760			37.4482	
4	7.492	3716	301			0.1028	
5	13.638	2236746	101102			61.8694	
6	14.965	3114	174			0.0881	
7	17.067	7372	288			0.2039	

3616271 242621 100.0000

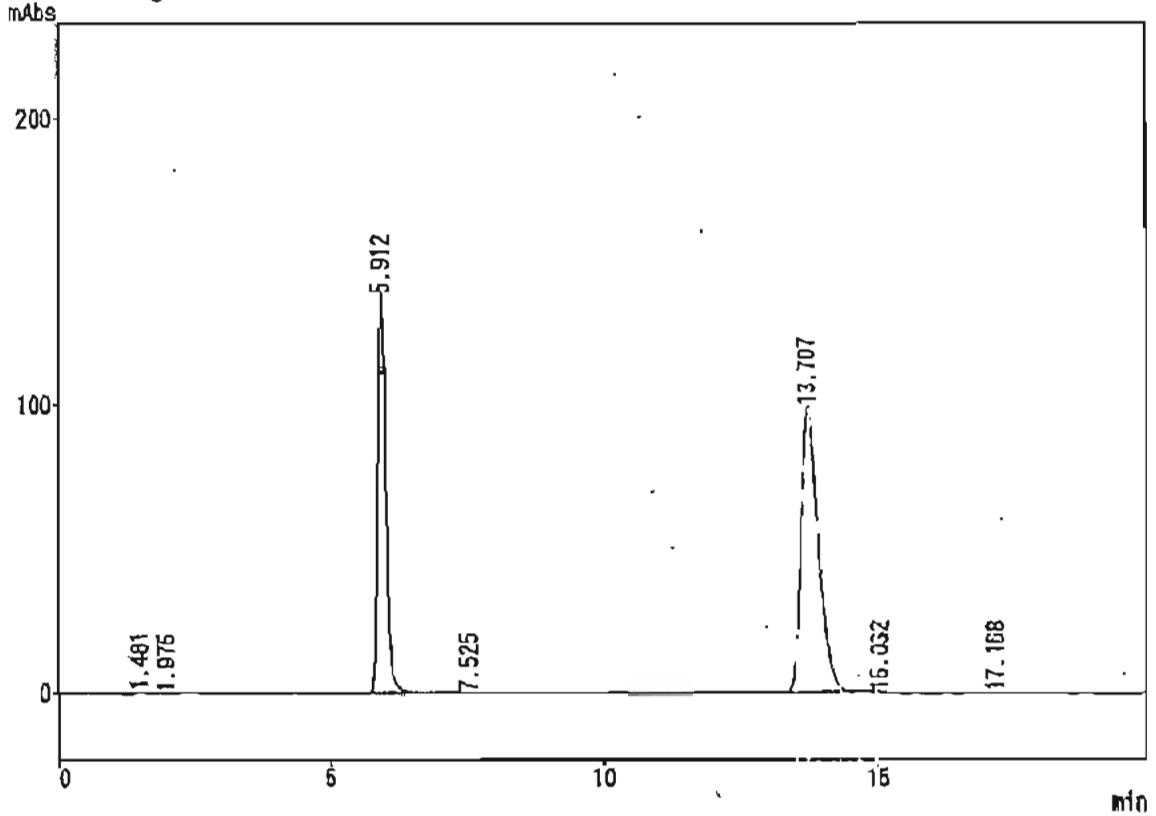
3 - 1/1

02/08/05 16:33:57

4

Sample: A-02
ID: 60°C-4W
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2T05.C03

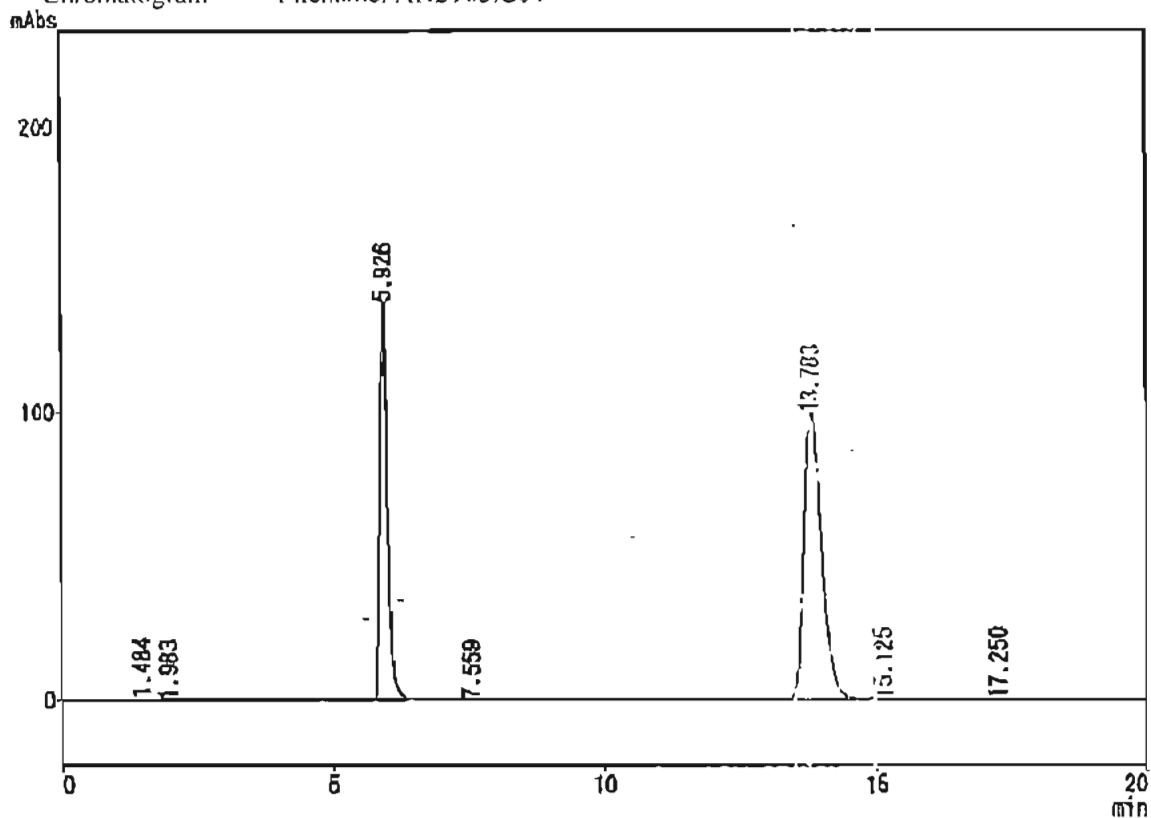


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.481	8013	858	V		0.2234	
2	1.975	2081	118	V		0.0580	
3	5.912	1353829	139609			37.7542	
4	7.525	3736	310			0.1042	
5	13.707	2207348	99386			61.6582	
6	15.032	3013	165			0.0840	
7	17.168	7885	316			0.2100	
		3585905	240768			100.0000	

Sample: A-03
 ID: 60°C-4W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2T05.C04



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.484	8194	835	V		0.2285	
2	1.983	2091	101	V		0.0578	
3	5.926	1354214	138961			37.4328	
4	7.569	3522	293			0.0974	
5	13.783	2232104	99359			61.6991	
6	15.125	10454	363	V		0.2890	
7	17.250	7148	291			0.1976	
		3617726	240204			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=6 Data=AH2T05.D05 02/08/05 17:14:00

Sample: A-04

ID: 60°C-4W

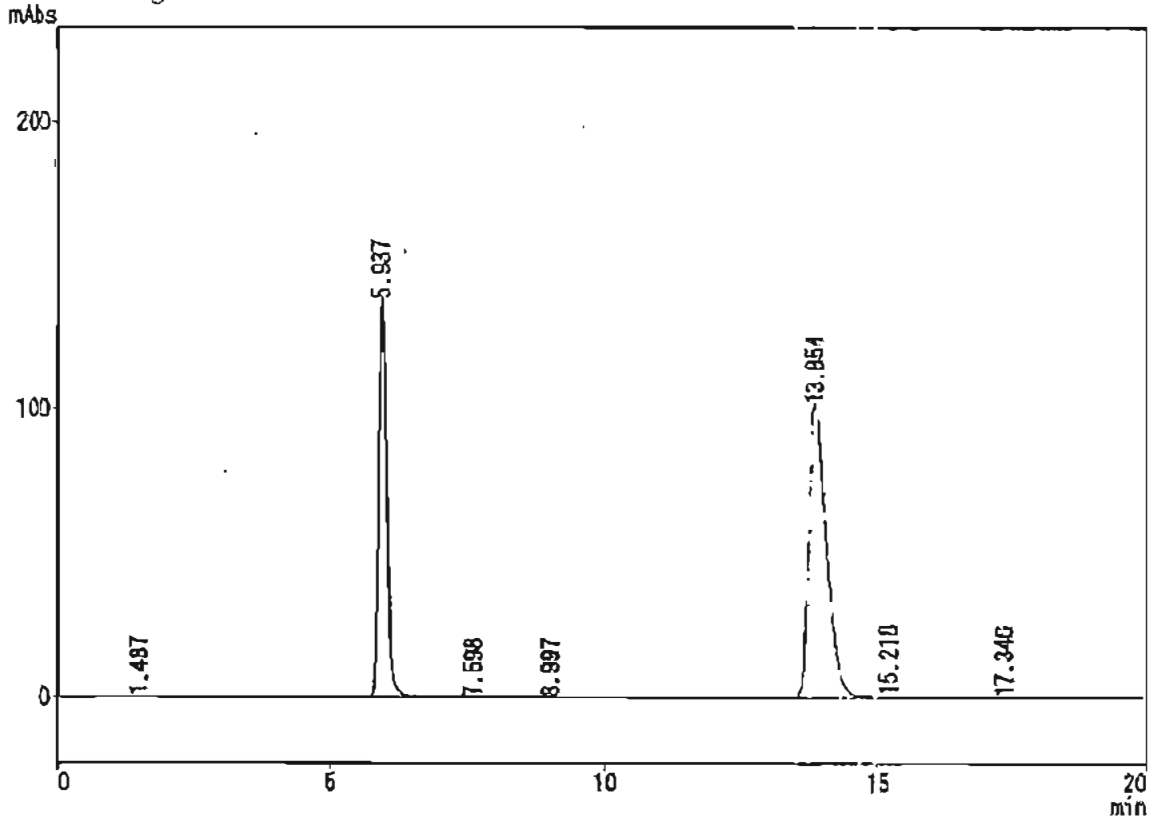
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2T05.C05



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.487	6295	762			0.1711	
2	5.937	1353039	139054			38.7798	
3	7.598	1763	143			0.0477	
4	8.997	1041	78			0.0283	
5	13.854	2296583	101688			62.4279	
6	15.218	12282	462	V		0.3339	
7	17.348	7785	308			0.2118	
		3678778	242479			100.0000	

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02/08/05 17:34:15

7

17:34:08

Sample: STD

ID:

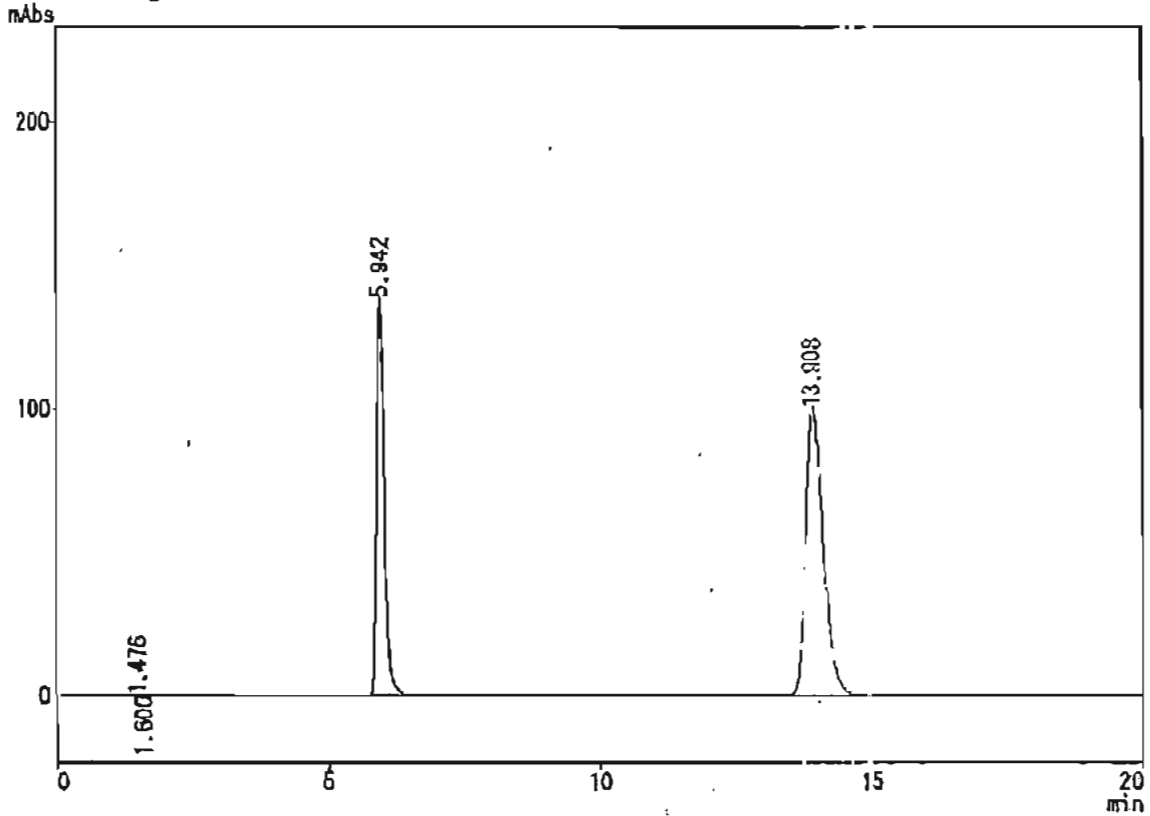
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2T05.C06



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.476	2198	184	V		0.0804	
2	1.600	1908	119	V		0.0360	
3	5.942	1353252	138822			37.2551	
4	13.908	2275645	100380			62.6485	
		3632399	239505			100.0000	

Stability of bromfenac sodium
 Lot No. 02S051

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 10 September 2002

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	FIM	Turbid	Permeation (%)
STD	AH2W10.C01	4619110	2799031	1.6503							
STD	AH2W10.C10	4680170	2784914	1.6479							
STD	Mean			1.6491	0.10060						
A-01	40°C-2M AH2W10.C02	4400120	2802884	1.5699	0.09577	96.36	95.89	8.18	—	—	0.49
A-02	40°C-2M AH2W10.C03	4371874	2780031	1.5569	0.09539	95.22	94.75	8.21	—	—	0.49
A-03	40°C-2M AH2W10.C04	4370724	2772844	1.5783	0.09616	96.24	95.69	8.18	—	—	0.57
A-04	40°C-2M AH2W10.C05	4438004	2775214	1.5992	0.09759	97.35	96.83	8.17	—	—	0.53

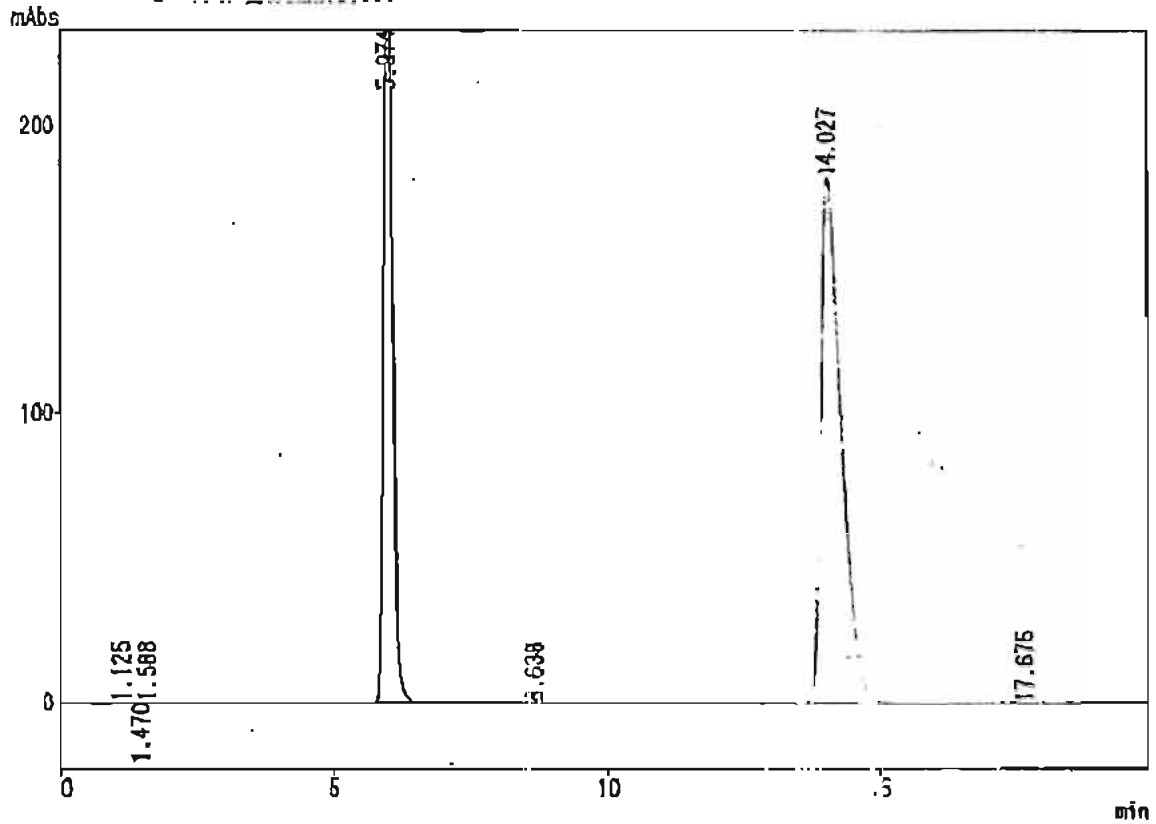
Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2002B131	Test date	10 September 2002
Test item				Tester	Shirou Sawa
IS: 0.020123333g of methyl p-hydroxybenzoate + diluted mobile phase → 100mL					
STD: 0.02012g of AHR10282B + mobile phase → 20mL					
10-Sep-2002 11:49:09					
001:R + 0.02012 g					
		pH	Ext. app.	Weight change	
A-01	40°C-2M	8.18	—	9.1335	9.1066
A-02	↓	8.21	—	9.1203	9.0934
A-03	↓	8.18	—	8.7819	8.7526
A-04	↓	8.17	—	8.9829	8.9547
A-05	60°C-1W	8.16	—	8.5396	8.4403
A-06	60°C-1W	8.17	—	8.4586	8.3582
A-05	60°C-4W	8.13	—	8.4889	8.1719
A-06	60°C-4W	8.14	—	8.4207	8.0996
9/10 16:59 NO.25 PH 8.18 26.4°C					
9/10 17:00 NO.26 PH 8.21 26.7°C					
9/10 17:00 NO.27 PH 8.18 25.8°C					
9/10 17:01 NO.28 PH 8.17 26.9°C					
9/10 17:03 NO.29 PH 8.16 26.9°C					
9/10 17:04 NO.30 PH 8.17 26.8°C					
9/10 17:05 NO.31 PH 8.13 25.9°C					
9/10 17:06 NO.32 PH 8.14 27.0°C					

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=2 Data=AH2W10.D01 02/09/10
 15:44:50

Sample: STD
 ID:
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2W10.C01



*** Peak Report ***

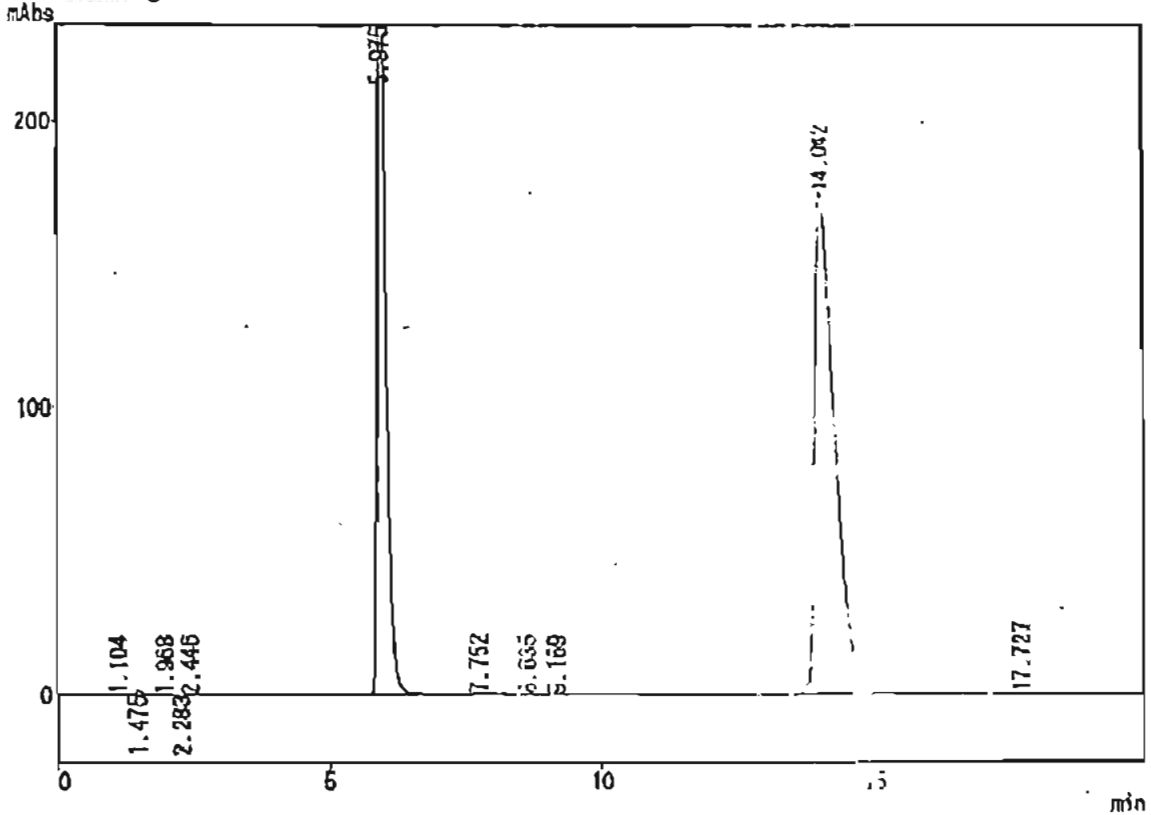
PKNO	TIME	AREA	HEIGHT	MK	ID/C	CONC	NAME
1	1.125	1083	78			0.0146	
2	1.470	2163	204	V		0.0291	
3	1.588	1489	159	V		0.0200	
4	5.974	2799031	280500	S		37.6629	
5	8.838	9408	383			0.1266	
6	14.027	4618110	181975			62.1368	
7	17.875	1485	81			0.0200	
		7433771	483380			100.0000	

2-1/1

02/09/10 16:05:06

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=3 Data=AH2W10.D02 02/09/10 16:04:56
 Sample: A-01
 ID: 40°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2W10.C02

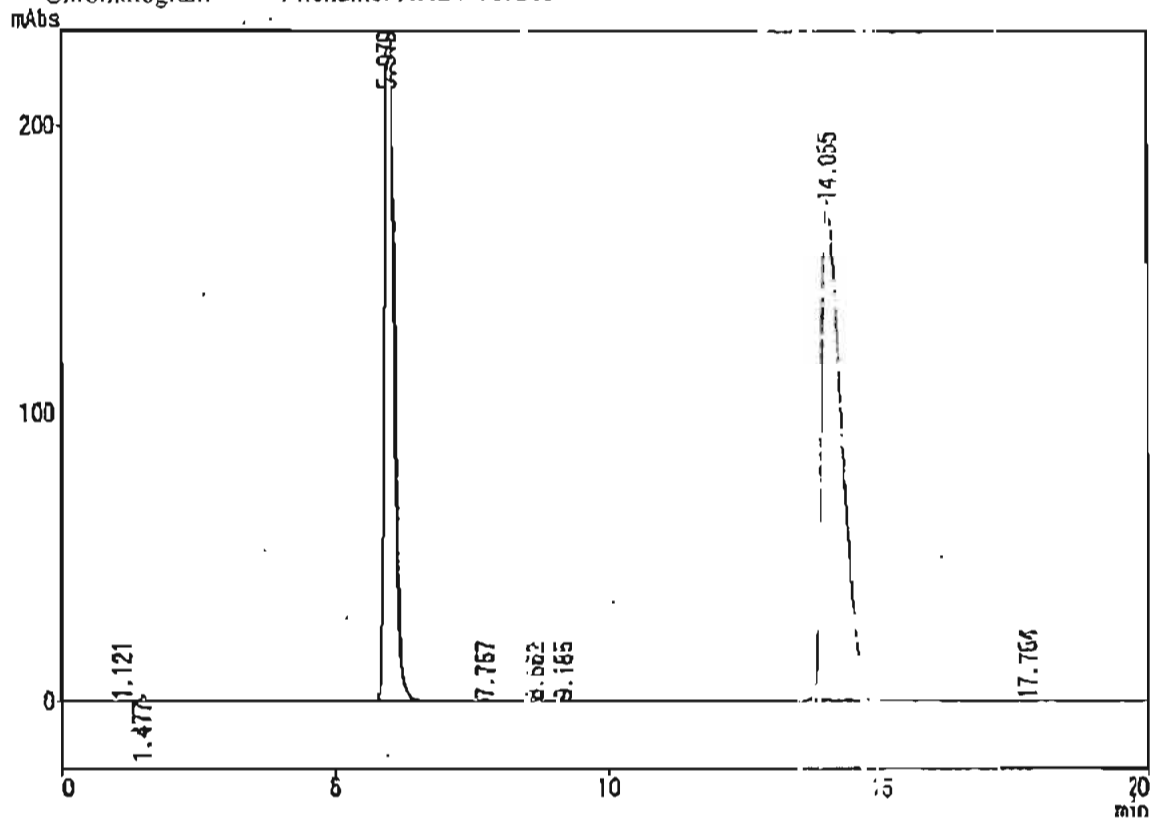


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	ID#	CONC	NAME
1	1.104	1871	129			0.0231	
2	1.476	13160	1508	V		0.1917	
3	1.988	1674	151	V		0.0231	
4	2.283	1000	84	V		0.0138	
5	2.446	1044	104	V		0.0144	
6	5.975	2802864	281181			38.7058	
7	7.752	6079	474			0.0839	
8	8.635	5958	282			0.0823	
9	9.169	2575	149	V		0.0356	
10	14.042	4400120	174275	S		60.7628	
11	17.727	5319	209			0.0734	
		7241482	458524			100.0000	

Sample: A-02
 ID: 40°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2W10.C03



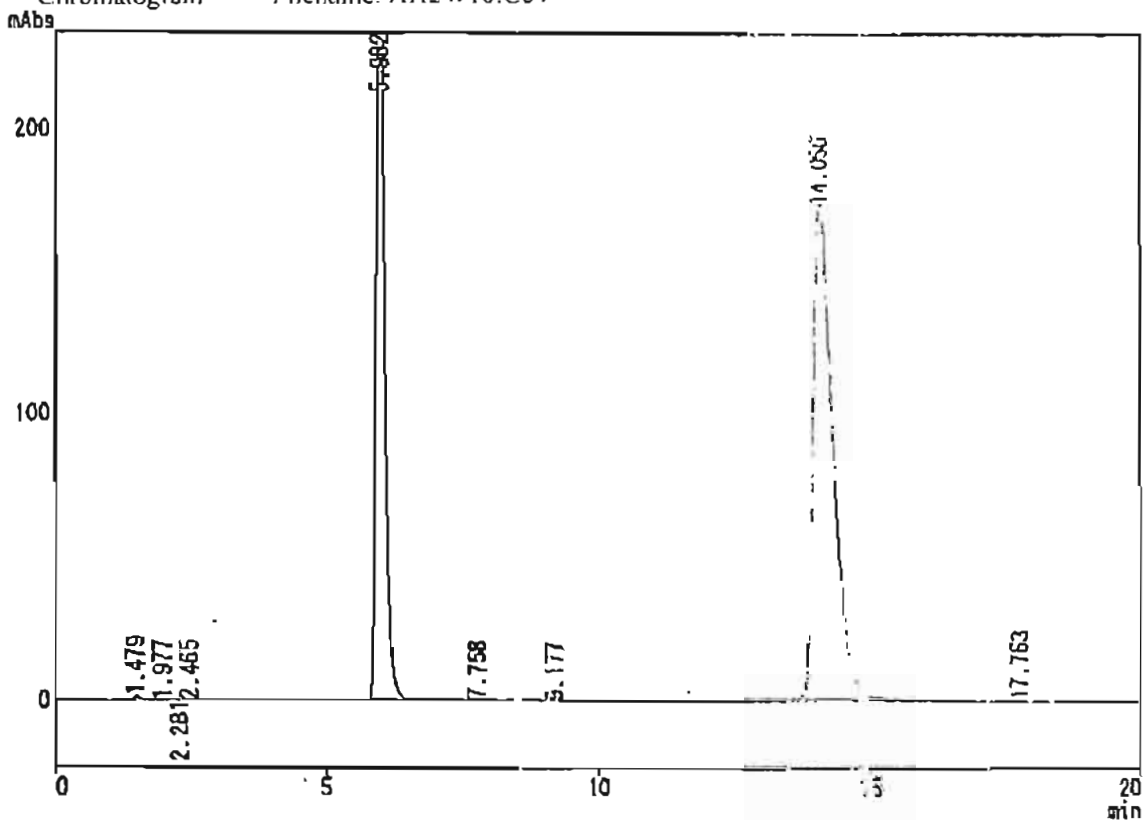
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	ID:G	CONC	SAME
1	1.121	1630	119			0.0213	
2	1.477	13680	1897	V		0.1802	
3	5.979	2790031	280527	V		38.7821	
4	7.757	6303	503			0.0876	
5	8.862	2925	135			0.0407	
6	9.185	2468	128	V		0.0343	
7	14.055	4371874	173463			60.7673	
8	17.764	6513	214			0.0766	

7194122 458783 100.0000

Sample: A-03
 ID: 40°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2W10.C04

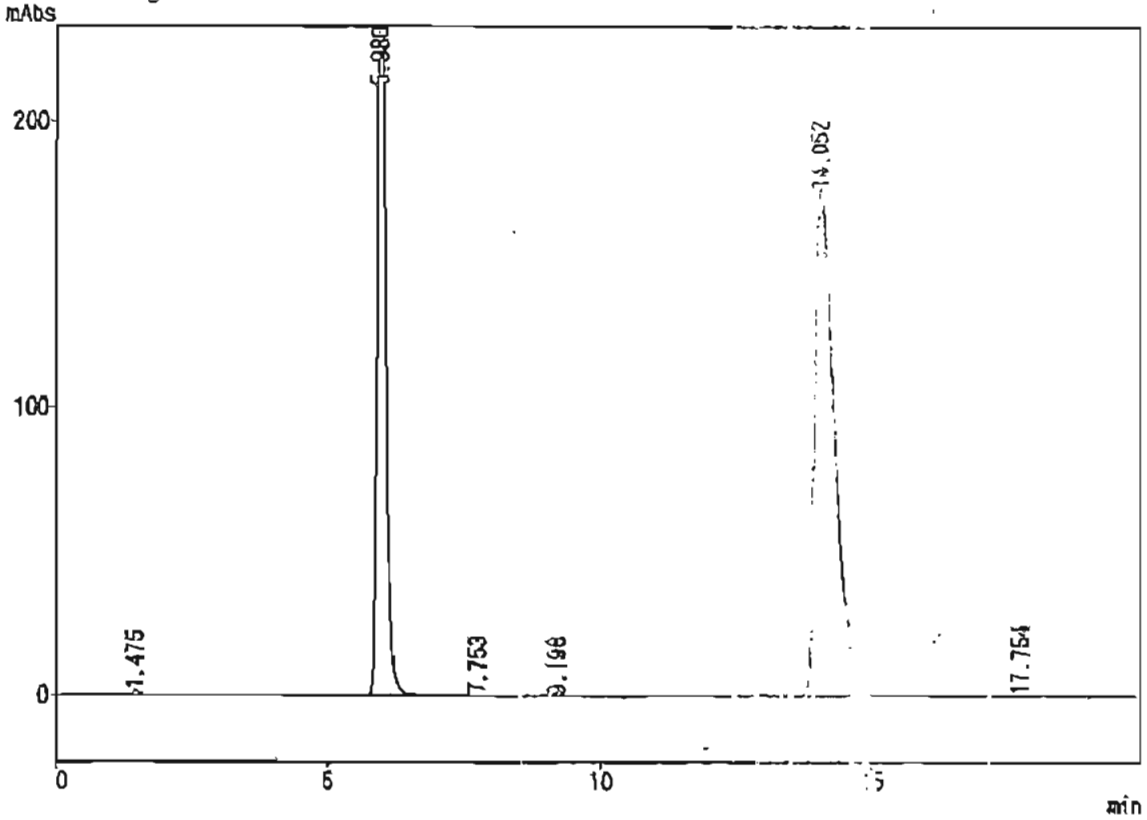


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	DIAG	CONC	NAME
1	1.479	13582	1590	V		0.1893	
2	1.977	1971	157	V		0.0276	
3	2.281	1187	103	V		0.0165	
4	2.465	1225	98	V		0.0171	
5	5.982	2772844	278821	V		38.6498	
6	7.758	8321	486			0.0881	
7	8.177	1370	102			0.0191	
8	14.058	4370724	173373			60.9221	
9	17.763	5063	199			0.0706	
		7174285	454939			100.0000	

Sample: A-04
 ID: 40°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2W10.C05



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MX	IOI-C	CONC	NAME
1	1.475	16535	1704	SV		0.2284	
2	5.980	2775214	280141	V		38.3349	
3	7.753	2827	230			0.0391	
4	9.198	1552	111			0.0214	
5	14.052	4438004	176335			61.3035	
6	17.754	5262	210			0.0727	
		7239394	458731			100.0000	

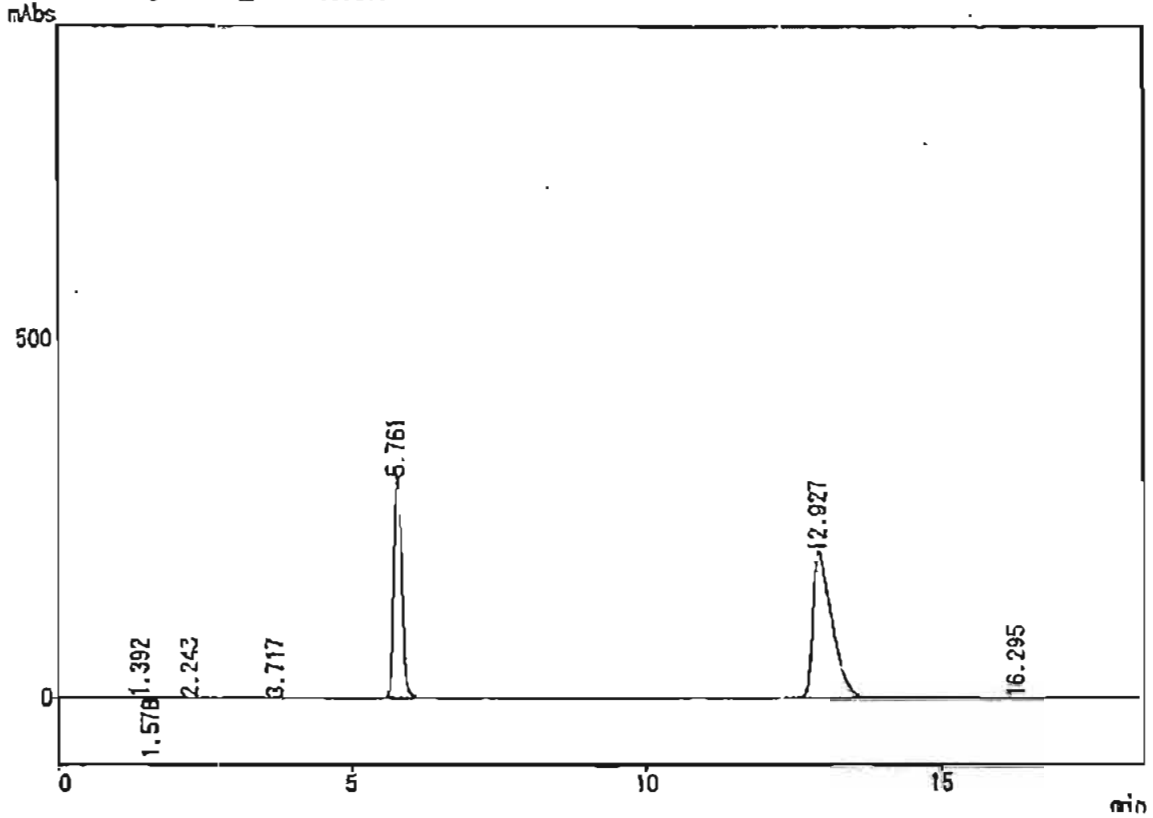
Stability of bromfenac sodium
Lot No. 02S051

Test code: P2002B131
Tester: Shirou Sawa
Test date: 13 November 2002

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	AH2Y13.C17	4588030	2793072	1.6355							
STD	AH2Y13.C22	4588150	2785108	1.6402							
STD	Mean			1.6379	0.09980						
A-01	40°C-4M AH2Y13.C18	4306875	2788894	1.5443	0.09410	94.68	93.59	8.19	—	—	1.15
A-02	40°C-4M AH2Y13.C19	4283038	2781720	1.5397	0.09382	93.48	92.50	8.19	—	—	1.03
A-03	40°C-4M AH2Y13.C20	4281429	2779313	1.5405	0.09387	93.95	92.95	8.19	—	—	1.08
A-04	40°C-4M AH2Y13.C21	4407948	2776019	1.5879	0.09875	98.54	95.50	8.20	—	—	1.08

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=92 Data=AH2Y13.D17 02/11/13 19:53:42
 Sample: STD
 ID:
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C17



*** Peak Report ***

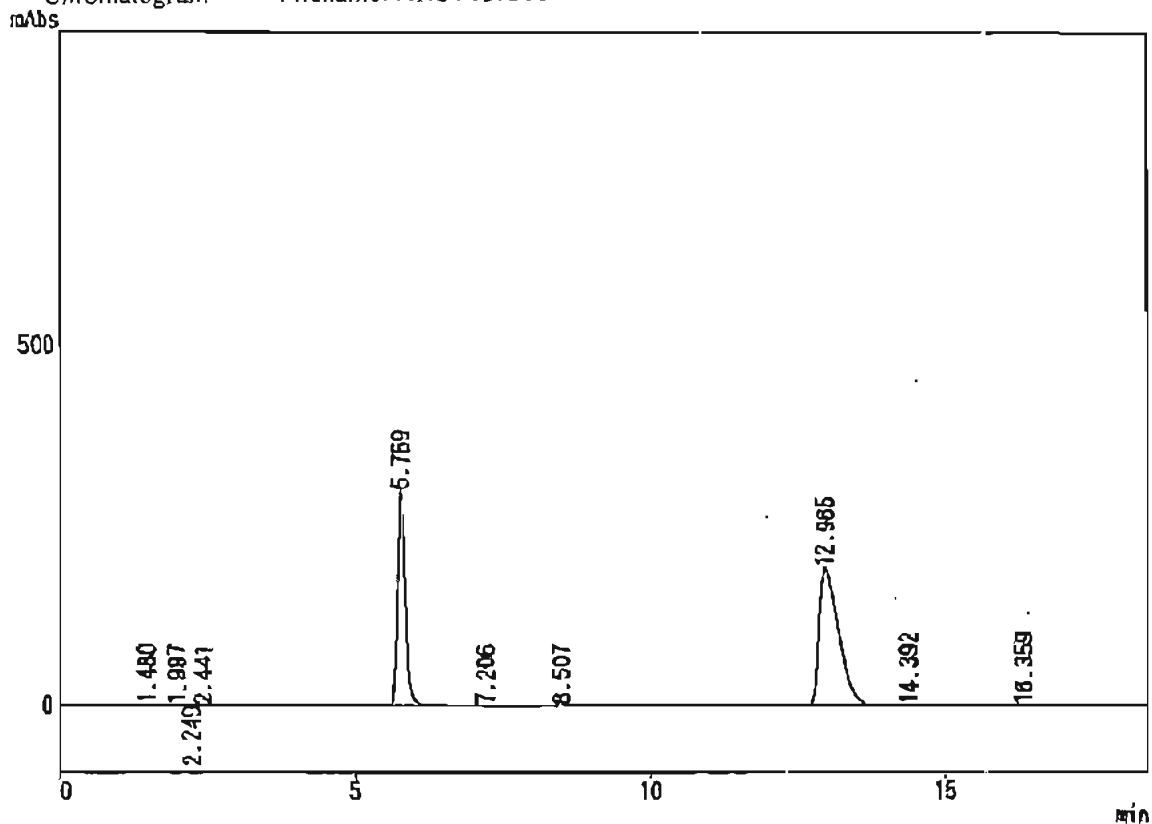
PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.392	1169	135	V		0.0159	
2	1.578	1403	160	V		0.0190	
3	2.243	2485	346			0.0337	
4	3.717	5849	189			0.0793	
5	5.761	2793072	308801			37.8821	
6	12.927	4568030	202625	S		61.9558	
7	16.295	1065	58			0.0144	
		7373072	510315			100.0000	

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02/11/13 20:12:23

Sample: A-01
 ID: 40°C-4M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C18



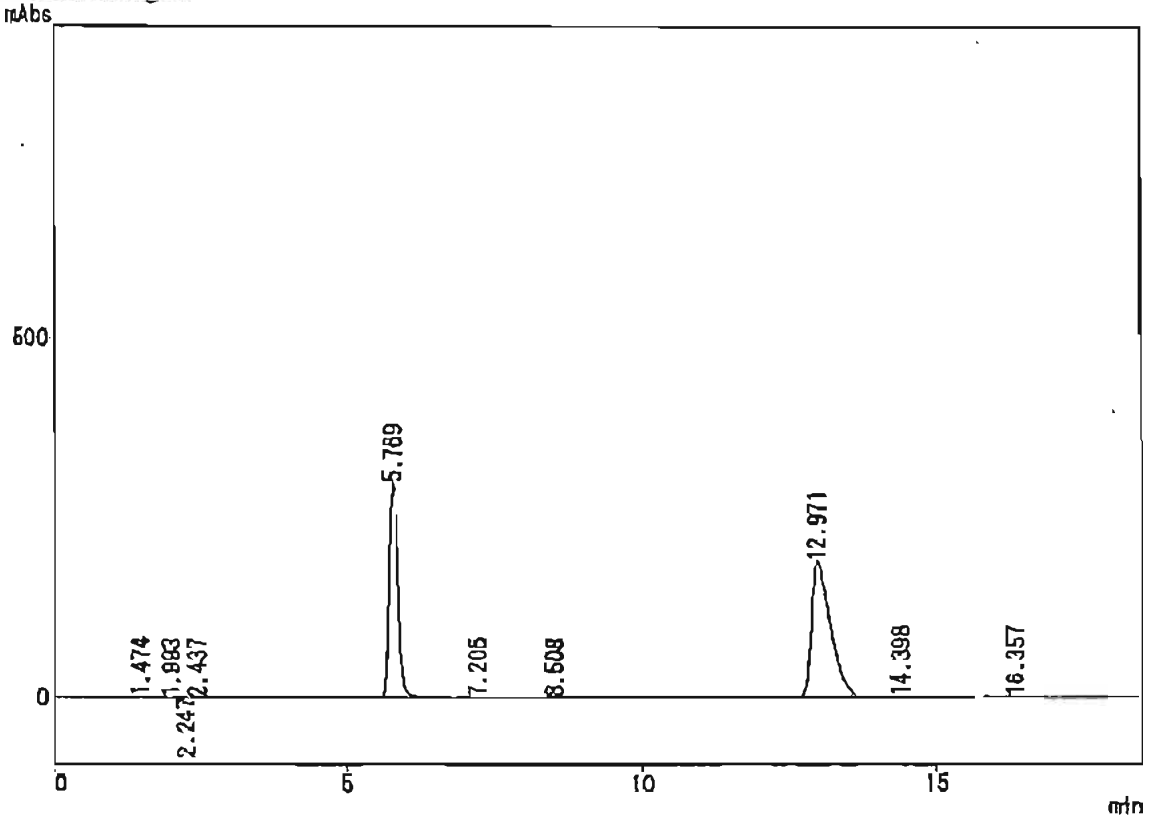
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MX	IDNO	CONC	NAME
1	1.480	12300	1194	V		0.1723	
2	1.997	2187	205	V		0.0308	
3	2.249	4370	611	V		0.0812	
4	2.441	1458	149	V		0.0204	
5	5.769	2788894	289943	S		39.0672	
6	7.206	8288	708	T		0.1158	
7	8.507	2288	162			0.0320	
8	12.965	4308875	190172	S		60.3313	
9	14.392	3480	209	T		0.0487	
10	18.359	8592	367			0.1204	

7:38710 493622 100.0000

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=94 Data=AH2Y13.D19 02/11/13 20:33:40
 Sample: A-02
 ID: 40°C-4M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C19



*** Peak Report ***

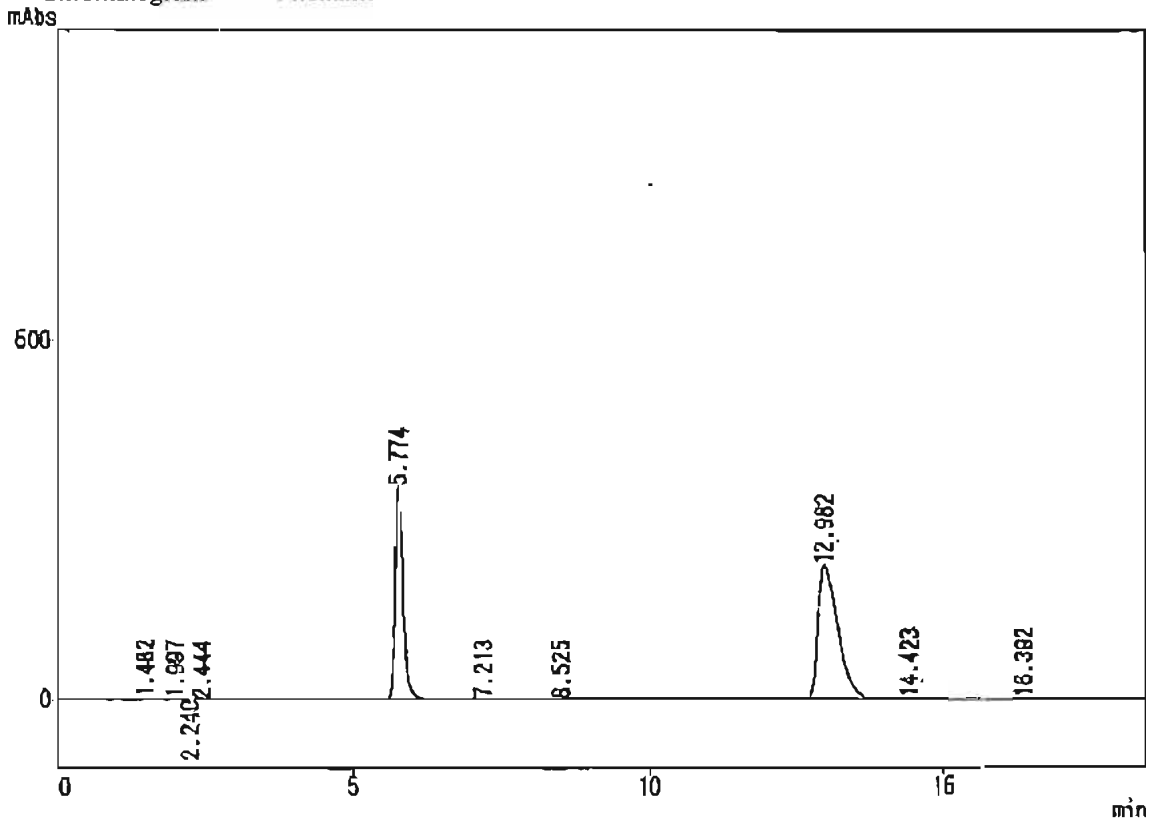
PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.474	16063	1662	V		0.2259	
2	1.893	2329	217	V		0.0327	
3	2.247	4034	463	V		0.0587	
4	2.437	1444	145	V		0.0203	
5	5.769	2781720	299235	S		39.1128	
6	7.206	8885	781	T		0.1249	
7	8.508	2448	172			0.0344	
8	12.971	4283036	189293	S		60.2223	
9	14.398	3050	189	T		0.0429	
10	16.357	9032	368			0.1270	
		7.12042	492524			100.0000	

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02/11/13 20:52:18

Sample: A-03
 ID: 40°C-4M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C20



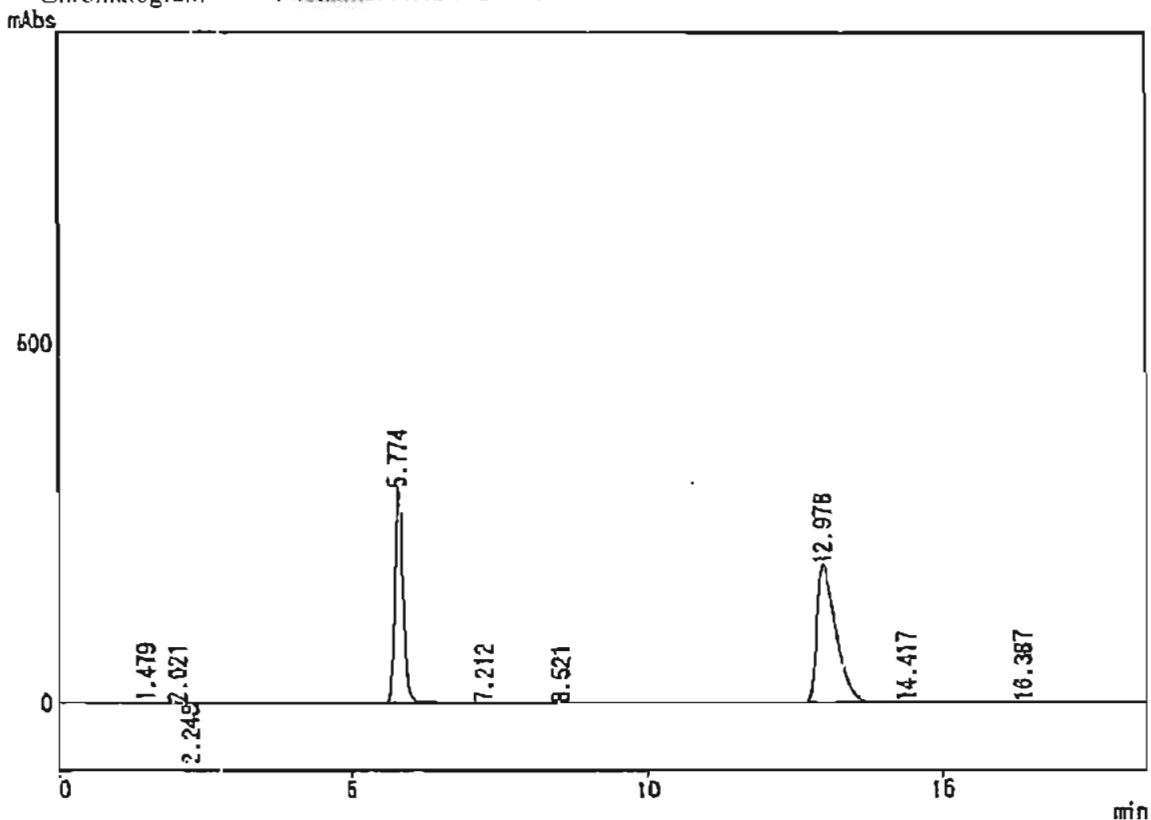
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.482	10532	1104	V		0.1483	
2	1.997	2091	199	V		0.0294	
3	2.249	3685	418	V		0.0519	
4	2.444	1451	139	V		0.0204	
5	5.774	2779313	298508	S		39.1381	
6	7.213	8413	720	T		0.1185	
7	8.525	2397	170			0.0338	
8	12.982	4281426	189051	S		60.2908	
9	14.423	3504	213	T		0.0493	
10	18.392	8483	366			0.1195	

7.01294 490885 100.0000

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=96 Data=AH2Y13.D21 02/11/13 21:13:34
 Sample: A-04
 ID: 40°C-4M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C21



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.479	12894	1368	V		0.1785	
2	2.021	1395	135			0.0193	
3	2.248	2841	338	V		0.0393	
4	5.774	2778019	298879	V		38.4233	
5	7.212	4536	394			0.0528	
6	8.521	2141	157			0.0298	
7	12.978	4407946	103885	S		61.0110	
8	14.417	7349	403	T		0.1017	
9	16.387	9719	419			0.1345	
		7224840	495974			100.0000	

96 - 1/1

02/11/13 21:32:14

Sample: STD

ID:

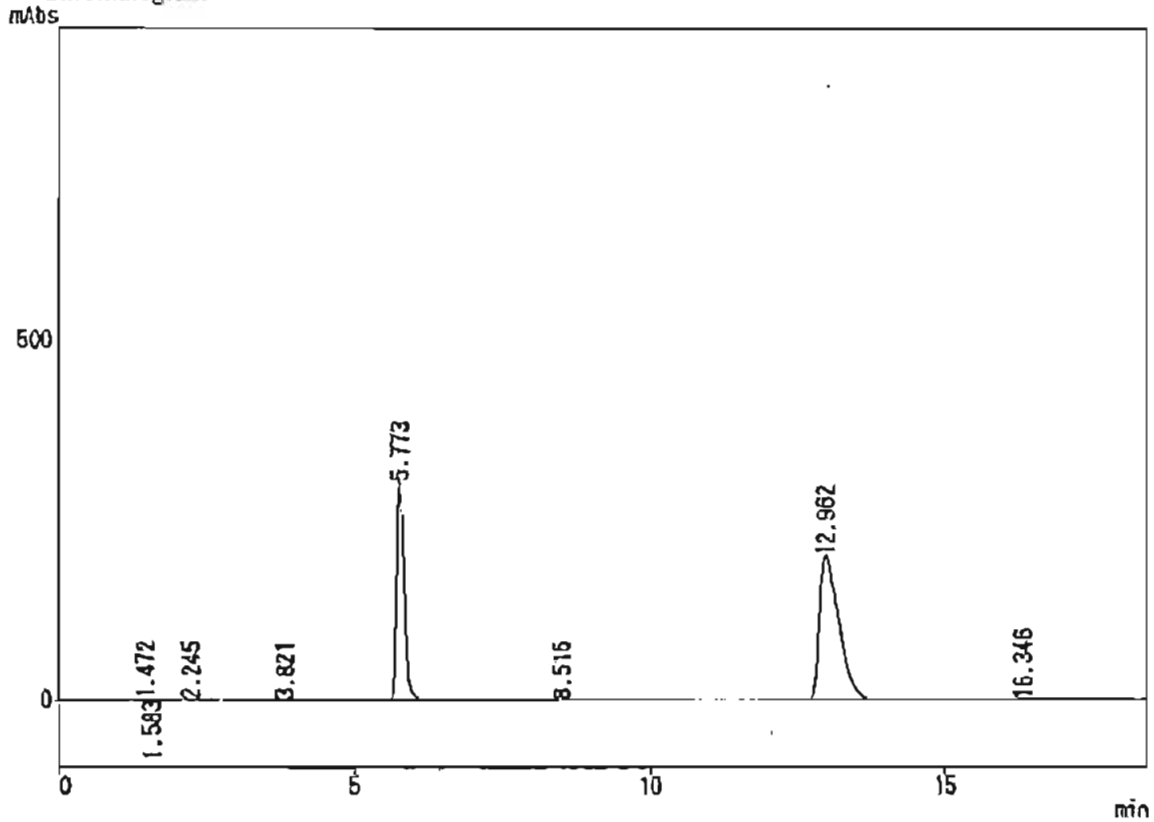
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C22



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.472	3388	280	V		0.0460	
2	1.583	1918	247	V		0.0281	
3	2.245	1315	198			0.0179	
4	3.821	1523	78			0.0207	
6	5.773	2785109	305132			37.8223	
6	8.515	1094	83			0.0148	
7	12.962	4568160	202151	S		62.0363	
8	16.346	1173	61			0.0159	
		7383670	508228			100.0000	

Stability of bromfenac sodium
Lot No. 02S051

Test code: P2002B131
Tester: Shirou Sawa
Test date: 17 January 2003

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	FLM	Turbid
STD	AH3H17.C01	4548885	2846553	1.5984						
STD	AH3H17.C08	4585385	2858747	1.5970						
STD	Mean			1.5977	0.10085					
A-01	40°C-6M	AH3H17.C02	4276662	2872101	1.4086	0.09405	94.64	92.90	8.17	-
A-02	40°C-6M	AH3H17.C03	4243563	2866430	1.4804	0.09354	93.18	91.53	8.18	-
A-03	40°C-6M	AH3H17.C04	4244241	2862118	1.4828	0.09370	93.78	92.11	8.17	-
A-04	40°C-6M	AH3H17.C05	4386572	2871808	1.5275	0.09651	98.30	94.58	8.17	+

Stability of bromfenac sodium
 Lot No. 02X221

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 17 January 2003

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid
STD	AH3H17.C08	4568385	2868747	1.5970						
STD	AH3H17.C15	4558189	2852477	1.5973						
STD	Mean			1.5972	0.10095					
A-01	40°C-2M AH3H17.C09	4301017	2860103	1.5318	0.09602	95.45	84.01	8.17	—	—
A-03	40°C-2M AH3H17.C10	4407379	2857141	1.5426	0.09750	97.40	95.84	8.16	—	—
BF	40°C-2M AH3H17.C11	4447937	2867651	1.5511	0.09804	96.07	95.53	8.23	—	—
A-01	25°C-2M AH3H17.C12	4455271	2874184	1.5501	0.09797	98.59	86.20	8.17	—	—
A-03	25°C-2M AH3H17.C13	4468866	2860635	1.5615	0.09869	98.59	88.25	8.16	—	—
BF	25°C-2M AH3H17.C14	4639904	2862632	1.5859	0.10024	98.23	87.93	8.23	—	—

Sample: STD

ID:

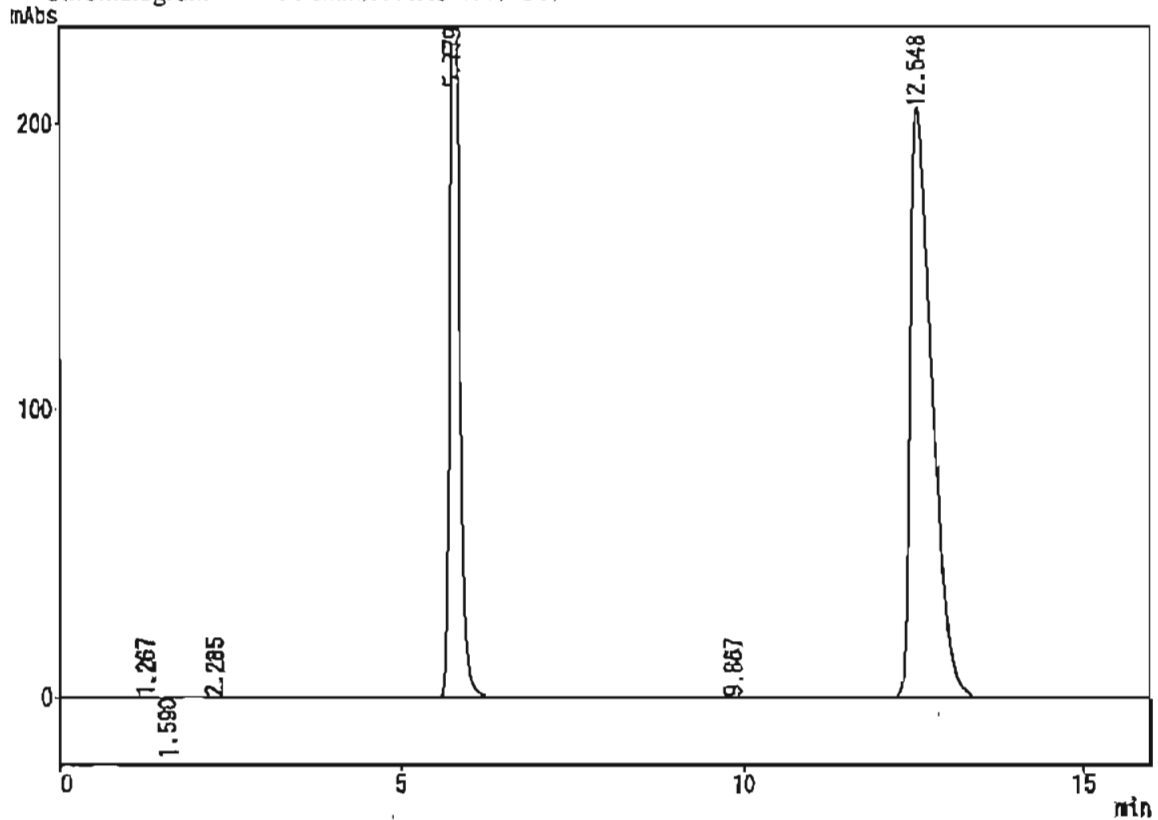
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C01

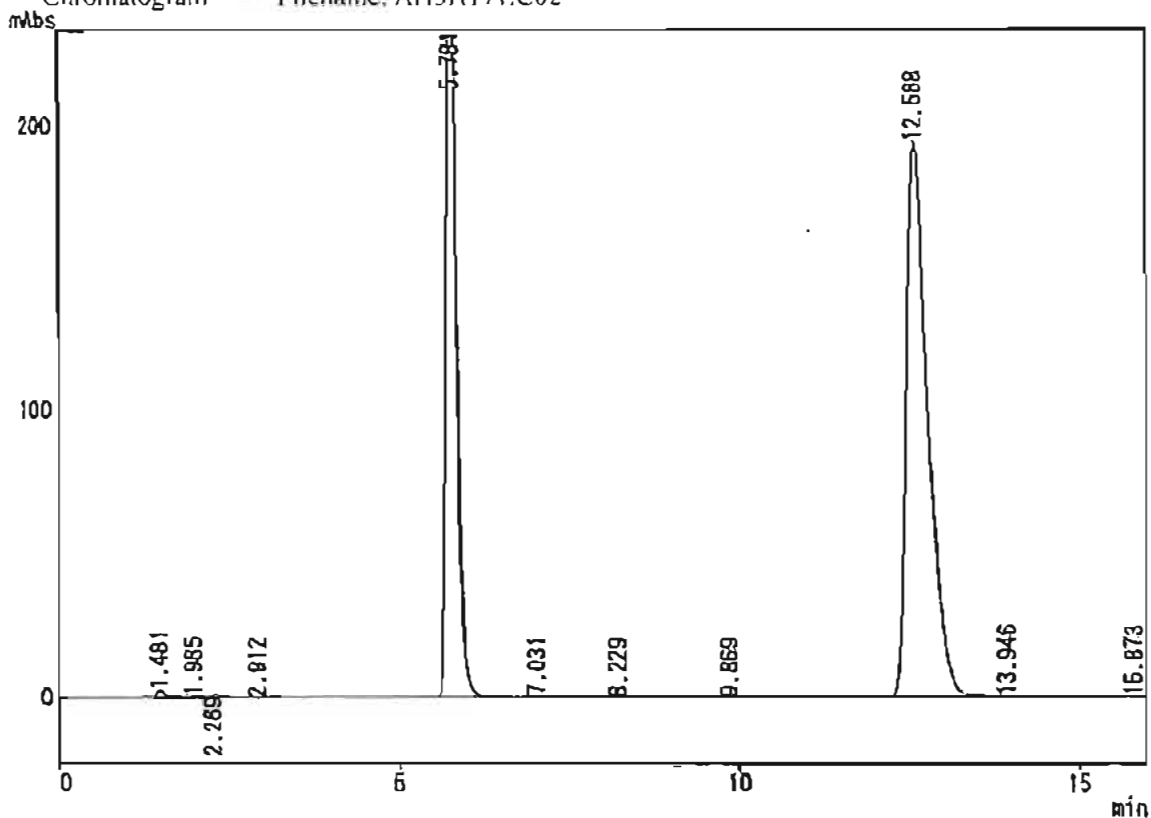


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.267	2015	103			0.0272	
2	1.590	1854	138	V		0.0250	
3	2.285	1836	245	V		0.0248	
4	5.779	2846553	306129			38.4442	
5	9.867	2251	146			0.0304	
6	12.548	4549865	205221			61.4483	
		7404374	511984			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=35 Data=AH3H171.D02 03/01/17 18:01:18
 Sample: A-01
 ID: 40°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C02

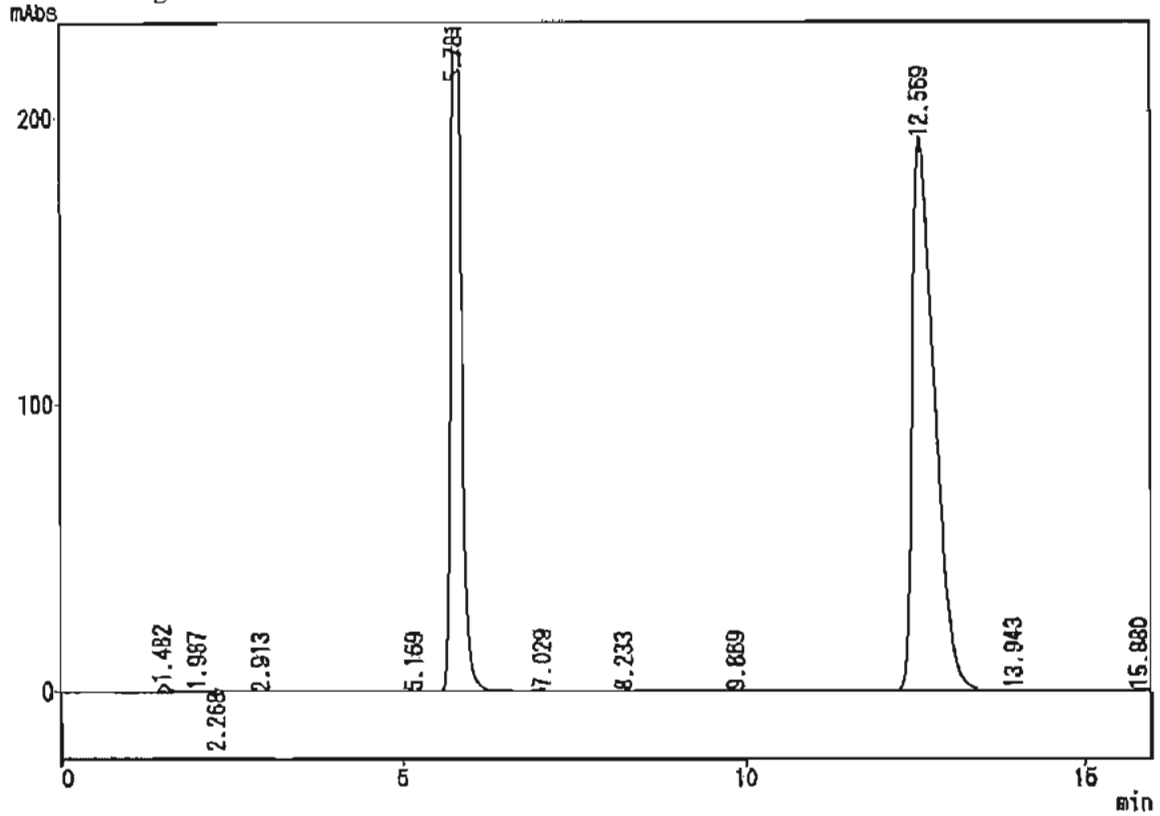


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	24769	2781	V		0.3433	
2	1.985	3280	295	V		0.0452	
3	2.269	12251	1138	V		0.1698	
4	2.812	2039	158	V		0.0283	
6	5.781	2872191	305372	S		39.8090	
6	7.031	9557	843	T		0.1325	
7	8.229	2437	175			0.0338	
8	9.869	3643	235			0.0481	
9	12.588	4275582	194154	S		59.2802	
10	13.946	6805	410	T		0.0957	
11	16.873	2399	175			0.0332	
		7214933	505718			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=36 Data=AH3H171.D03 03/01/17 18:18:46
 Sample: A-02
 ID: 40°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C03

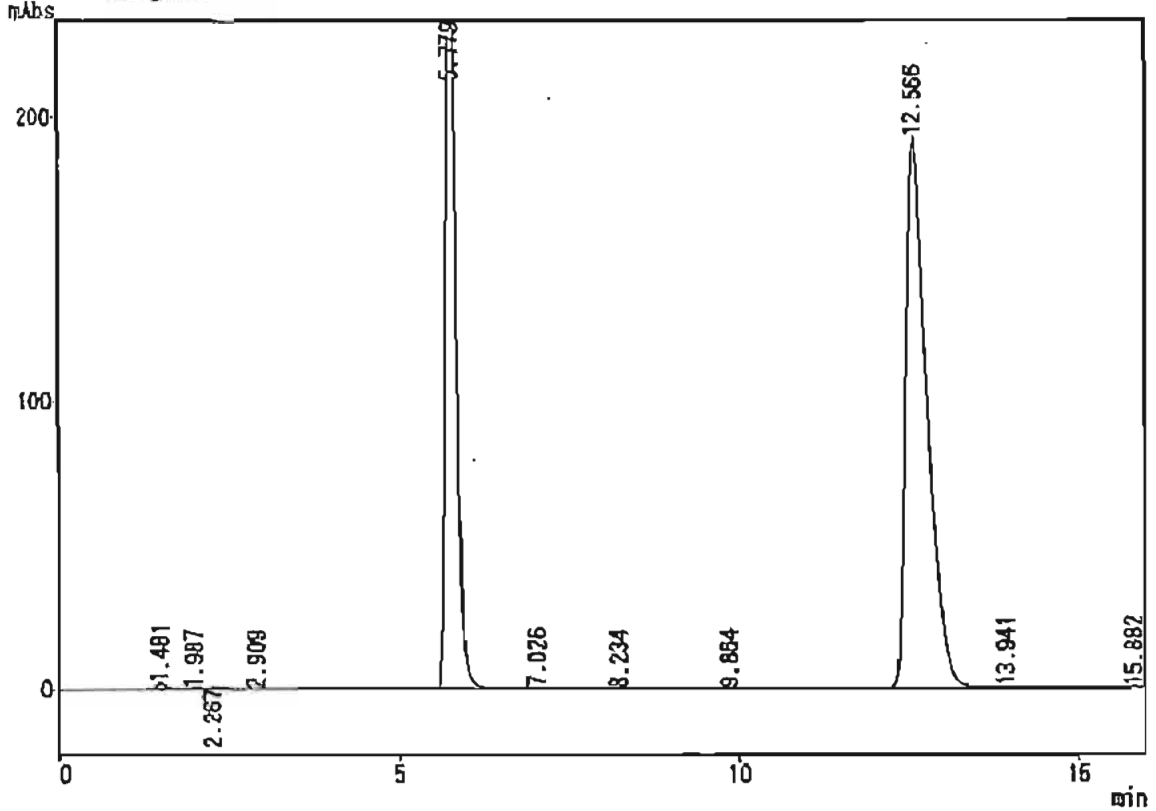


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.482	23146	2451	V		0.3225	
2	1.887	3457	291	V		0.0482	
3	2.268	10912	964	V		0.1521	
4	2.913	1941	150	V		0.0270	
5	5.169	2339	112			0.0328	
6	5.781	2866430	305065	SV		89.8444	
7	7.029	9798	870	T		0.1365	
8	8.233	2254	169			0.0314	
9	9.869	3856	240			0.0510	
10	12.669	4243563	193000	S		59.1351	
11	13.943	6426	381	T		0.0895	
12	15.880	2135	158			0.0298	
		7176053	503849			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=37 Data=AH3H171.D04 03/01/17 18:36:14
 Sample: A-03
 ID: 40°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C04



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.481	22004	2406	V		0.3070	
2	1.987	3113	277	V		0.0434	
3	2.267	9838	812	V		0.1372	
4	2.909	2135	154	V		0.0299	
5	6.779	2882118	305025	S		39.9298	
6	7.026	9443	837	T		0.1317	
7	8.234	2230	168			0.0311	
8	8.864	3765	242			0.0524	
9	12.566	4244241	192968	S		59.2120	
10	13.941	6633	393	T		0.0925	
11	15.882	2370	162			0.0331	

7187879

503443

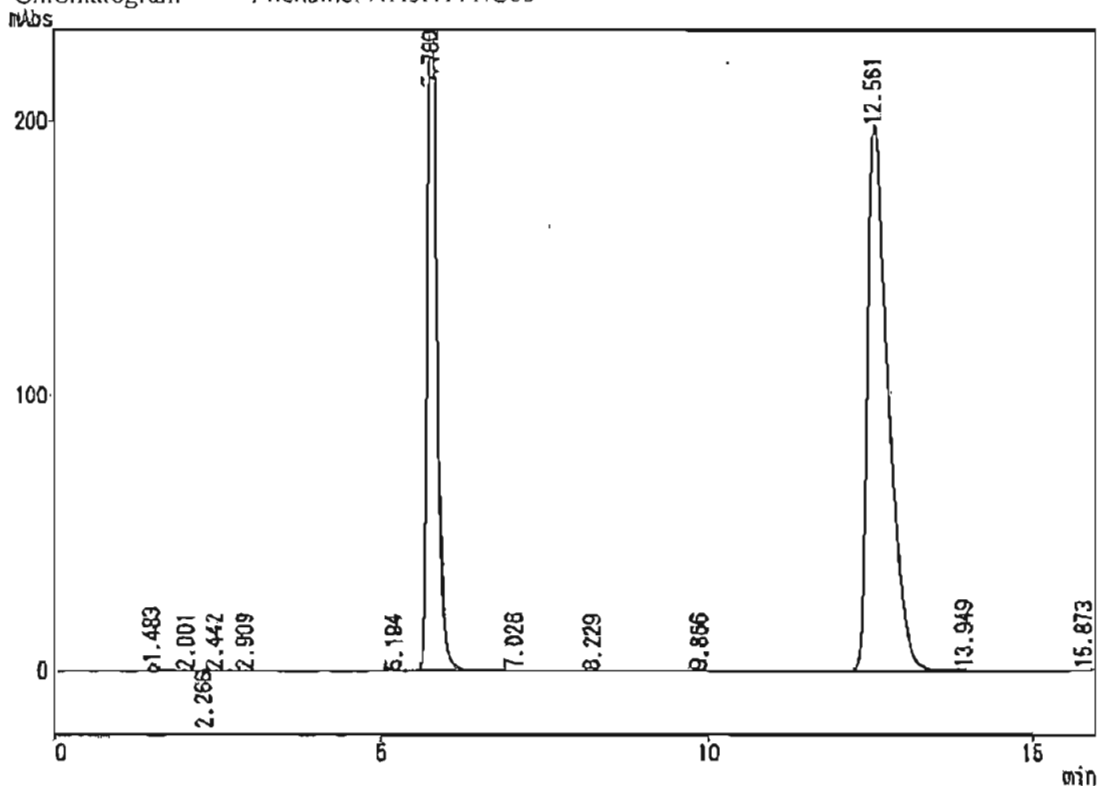
100.0000

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03/01/17 18:52:26

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=38 Data=AH3H171.D05 03/01/17 18:53:40
 Sample: A-04
 ID: 40°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C05



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.483	22252	2489	V		0.3039	
2	2.001	2292	185	V		0.0313	
3	2.266	5344	681	V		0.0730	
4	2.442	1877	201	V		0.0256	
5	2.909	1562	132	V		0.0213	
6	5.194	2724	128			0.0372	
7	5.789	2071008	305042	SV		99.2232	
8	7.026	5364	475	T		0.0733	
9	8.229	2204	164			0.0301	
10	9.866	3854	249			0.0628	
11	12.561	4386572	198560	S		59.9119	
12	13.949	13021	709	T		0.1778	
13	15.873	2832	214			0.0387	

7321705

510007

100.0000

38 - 1/1

03/01/17 19:09:48

Sample: STD

ID:

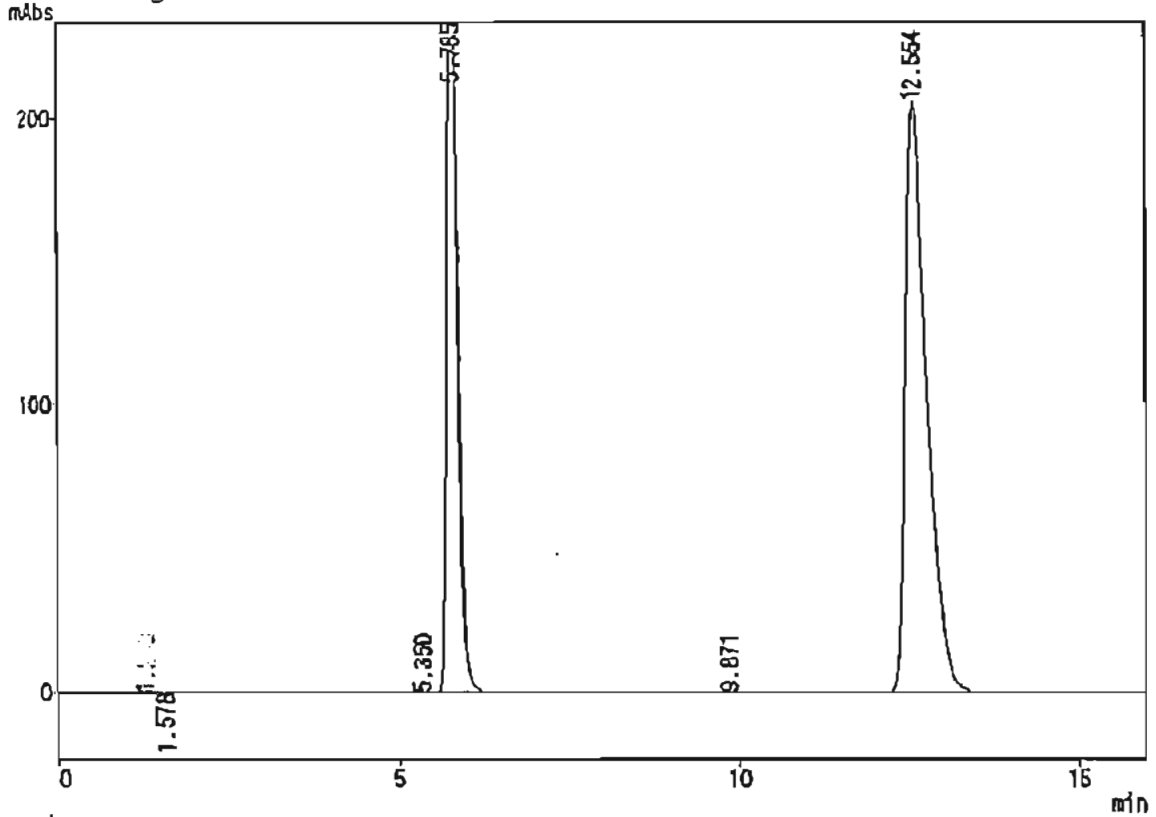
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C08

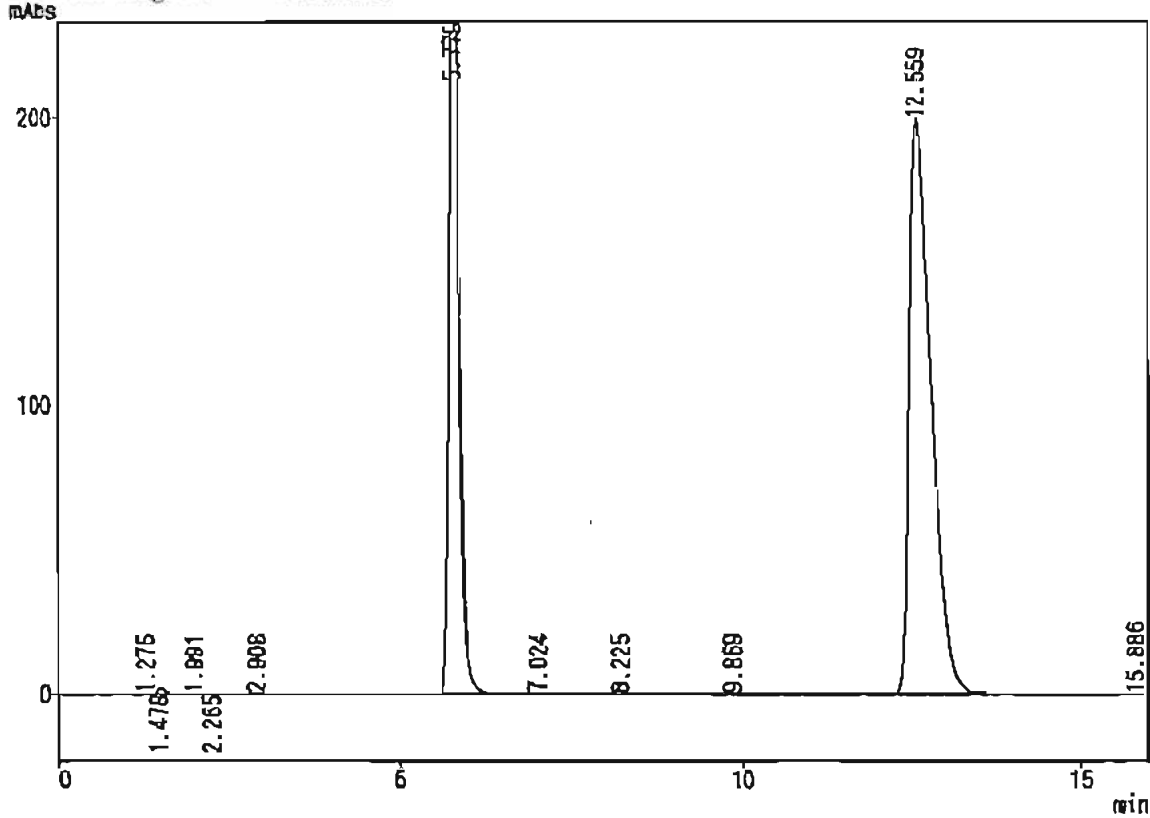


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	WK	IDNO	CONC	NAME
1	1.280	2421	137			0.0328	
2	1.578	2298	177	V		0.0309	
3	5.350	4420	178			0.0594	
4	5.785	2858747	308832	SV		38.4480	
6	9.871	2080	143			0.0280	
8	12.554	4586386	205732			61.4011	
		7435350	513197			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=42 Data=AH3H171.D09 03/01/17 20:03:30
 Sample: A-01
 ID: 40°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C09

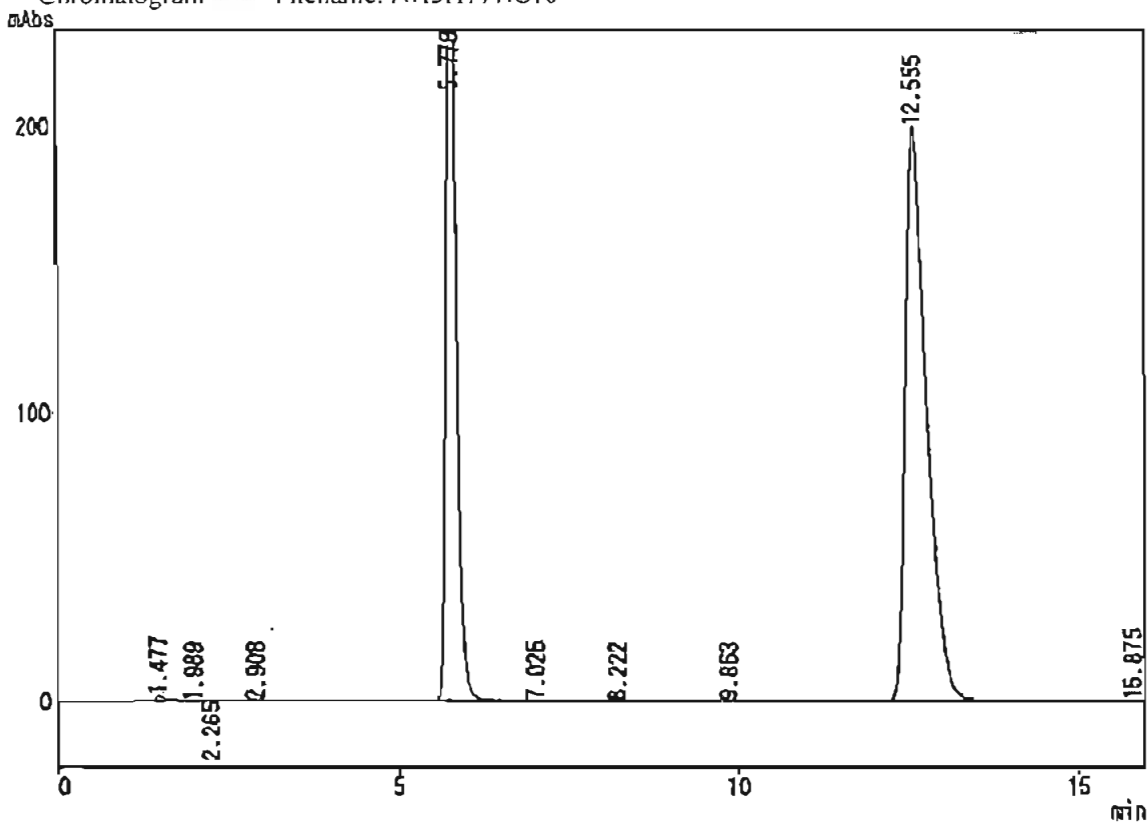


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.278	1781	120			0.0242	
2	1.478	20171	2257	V		0.2788	
3	1.991	2144	209	V		0.0294	
4	2.265	6473	477	V		0.0888	
5	2.808	2653	289	V		0.0364	
6	5.779	2860103	304472	S		39.2444	
7	7.024	5170	487	T		0.0761	
8	8.225	2784	205			0.0382	
9	9.869	4035	268			0.0554	
10	12.559	4381017	199015			60.1134	
11	15.886	1310	90			0.0180	
		7287920	507899			100.0000	

CLASS-I.C10 Ver=1.62 System No=1 Ch=1 Report No=43 Data=AH3H171.D10 03/01/17 20:21:02
 Sample: A-03
 ID: 40°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C10



*** Peak Report ***

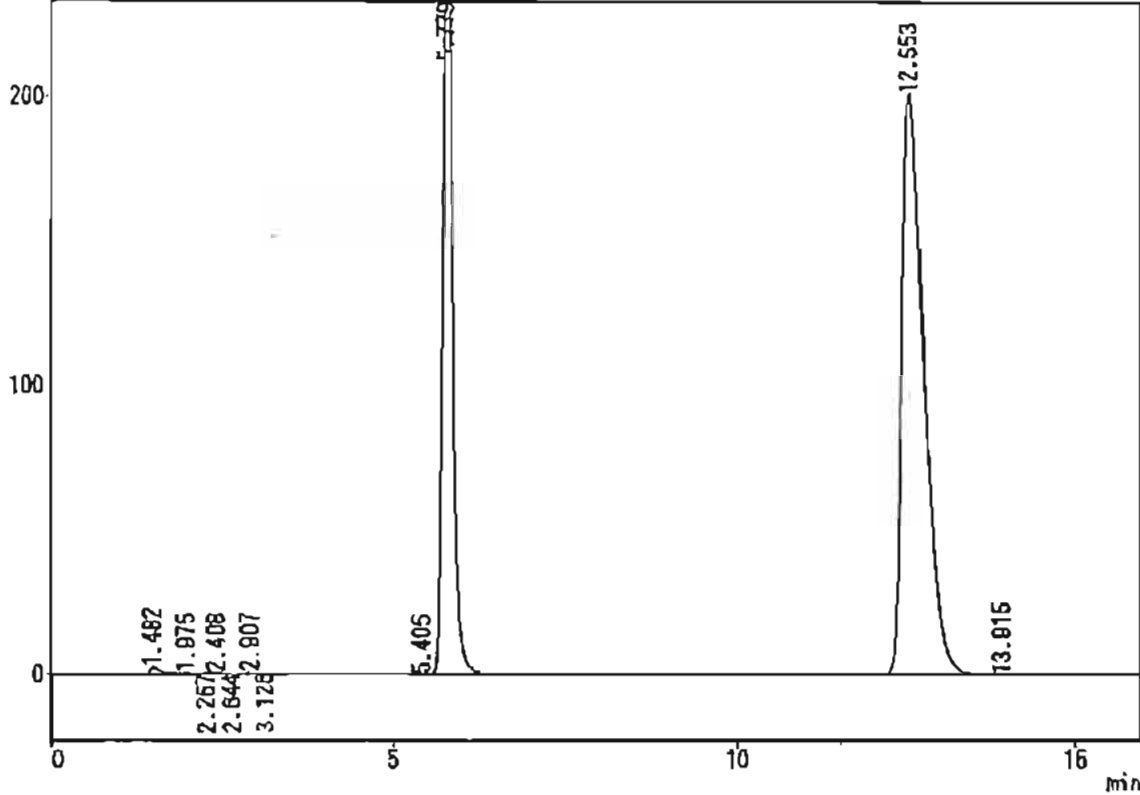
PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.477	19859	2218	V		0.2717	
2	1.989	2205	226	V		0.0302	
3	2.265	6553	449	V		0.0898	
4	2.908	3188	322	V		0.0436	
5	5.778	2867141	304490	S		39.0859	
6	7.025	5594	498	T		0.0765	
7	8.222	2990	226			0.0410	
8	9.863	3790	249			0.0518	
9	12.555	4407379	200024			60.2933	
10	15.876	1193	90			0.0163	
		7309899	608789			100.0000	

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03/01/17 20:37:10

Sample: BF
 ID: 40°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C11
 mAbs

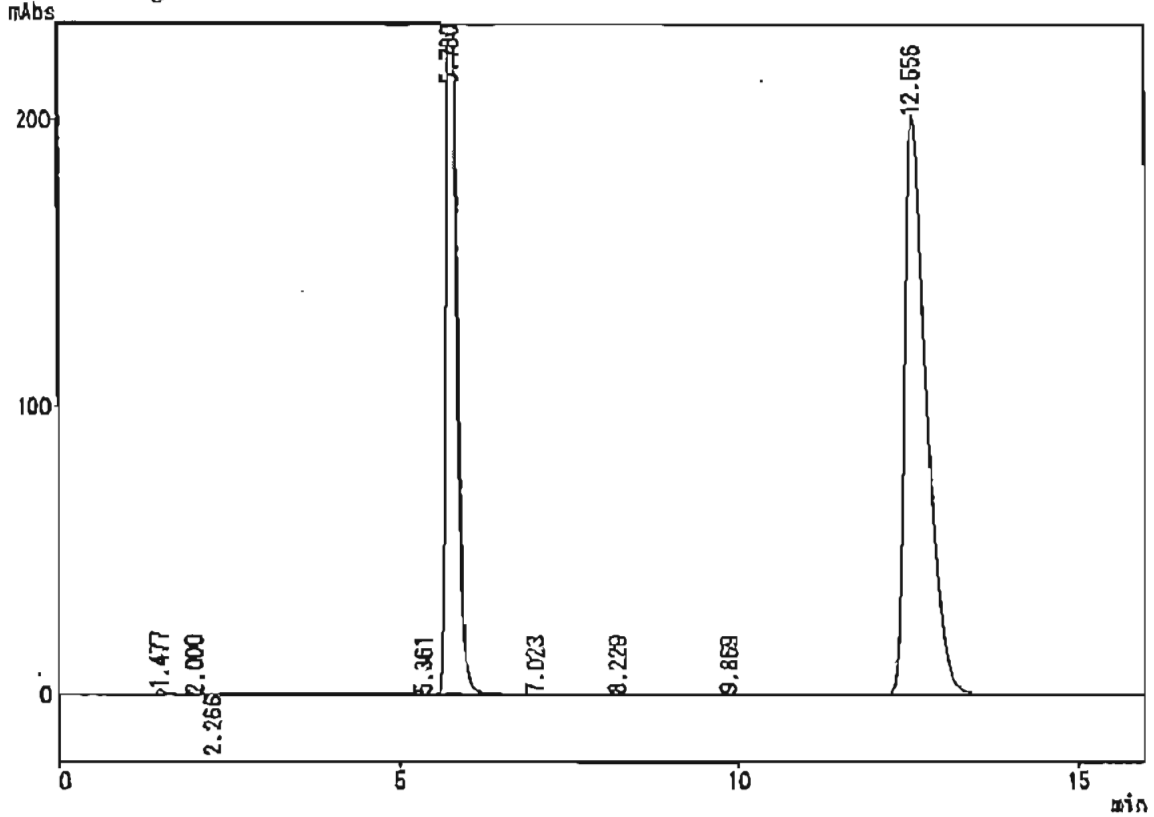


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	26094	2268	V		0.3544	
2	1.975	2029	250	V		0.0278	
3	2.267	3747	423	V		0.0509	
4	2.408	4698	560	V		0.0638	
5	2.644	1004	125	V		0.0136	
6	2.907	5257	673	V		0.0714	
7	3.126	1155	118	V		0.0157	
8	5.405	1033	78	V		0.0140	
9	5.779	2887851	305264	SV		38.9528	
10	12.553	4447937	201265	S		60.4188	
11	13.915	1281	77	T		0.0171	
		7361865	511100			100.0000	

Sample: A-01
 ID: 25°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHRT028.MET

*** Chromatogram *** Filename: AH3H171.C12

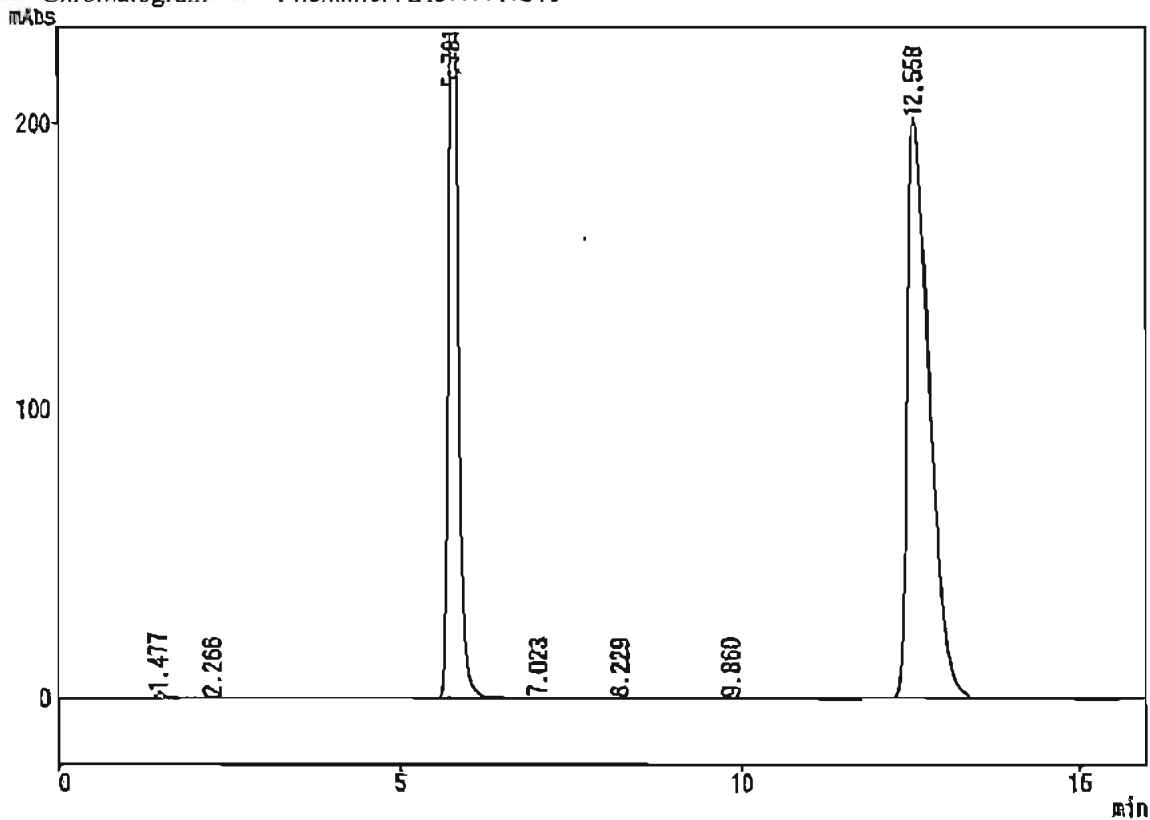


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.477	18139	1913	V		0.2462	
2	2.000	1561	139			0.0212	
3	2.266	3572	325	V		0.0485	
4	5.361	1580	74			0.0215	
5	5.780	2874184	306122	SV		39.0170	
8	7.023	3683	322	T		0.0500	
7	8.229	3994	295			0.0542	
8	9.869	4506	294	V		0.0812	
8	12.556	4455271	201390			60.4802	
		7366490	510873			100.0000	

Sample: A-03
 ID: 25°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C13

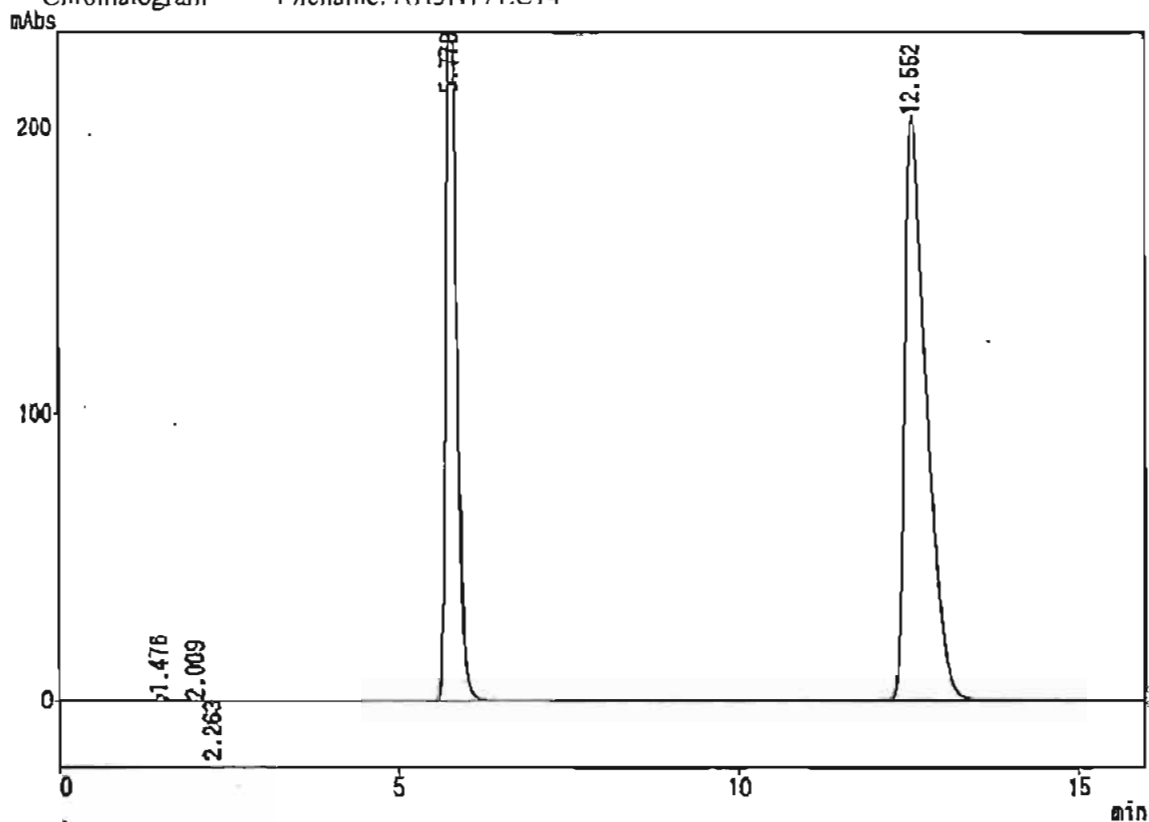


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.477	17873	1959	SV		0.2428	
2	2.266	2882	287	V		0.0364	
3	5.781	2860635	304234	S		38.8652	
4	7.023	3983	348	T		0.0541	
5	8.229	3943	298			0.0536	
6	9.860	4420	292			0.0601	
7	12.558	4166868	201701			60.6878	
		7380402	509116			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=47 Data=AH3H171.D14 03/01/17 21:30:56
 Sample: BF
 ID: 25°C-2M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C14



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.476	22168	2087	V		0.2982	
2	2.009	1033	130			0.0139	
3	2.263	4259	322	V		0.0573	
4	5.778	2862632	304977	V		38.5281	
5	12.562	4639904	204556			61.1025	
		7429908	512071			100.0000	

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03/01/17 21:47:09

18

Sample: STD

ID:

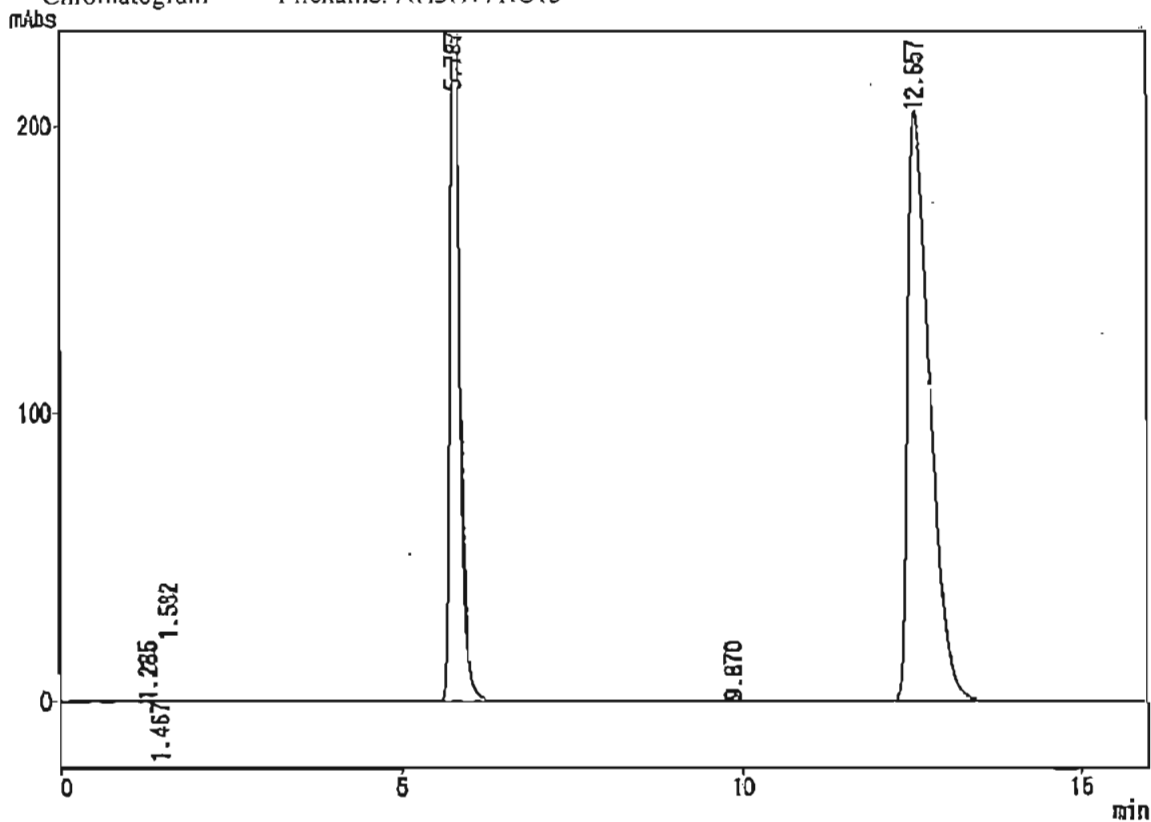
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3H171.C15



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.285	2140	133			0.0289	
2	1.467	1141	140	V		0.0154	
3	1.592	2551	168	V		0.0344	
4	6.787	2852477	306429	S		38.4601	
5	9.870	2239	152			0.0302	
6	12.657	4556169	206242			61.4311	
		7416717	512264			100.0000	

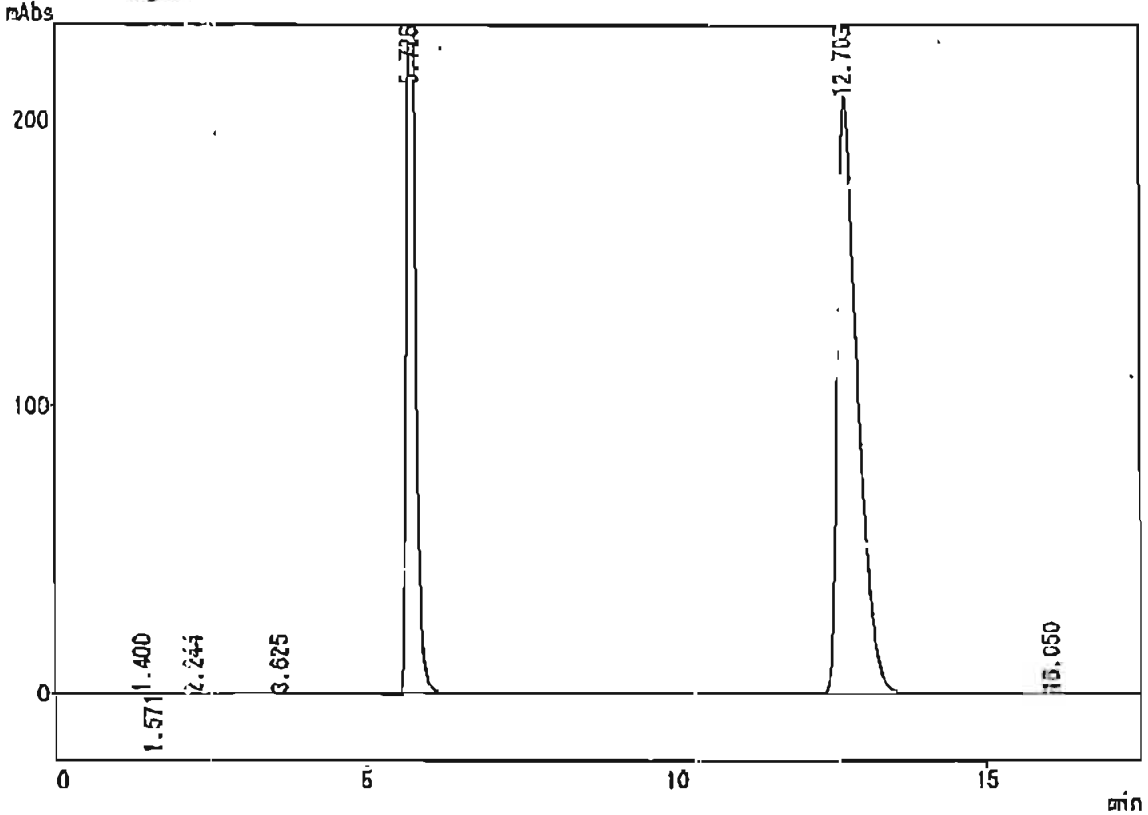
Stability of bromfenac sodium
 Lot No. 02X221

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 20 November 2002

ID	Chromata No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.M.	Turbid	Permeation (%)
STD	AH2Y20.004	4877892	2804301	1.6689							
STD	AH2Y20.008	4593438	2761858	1.6633							
STD	Mean			1.6651	0.10115						
A-01	60°C-4W	AH2Y20.C05	4525804	2774228	1.6314	0.09910	97.70	93.81	8.23	—	4.19
A-03	60°C-4W	AH2Y20.C06	4629548	2821284	1.6409	0.09968	98.58	95.07	8.21	—	4.53
BF	50°C-4W	AH2Y20.C07	4474110	2789530	1.6038	0.09743	95.47	91.45	8.31	—	4.21

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=14 Data=AH2Y20.D04 02/11/20 21:21:56
 Sample: STD
 JD:
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: \AHR\028.MET

*** Chromatogram *** Filename: AH2Y20.C04



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IOBO	CONC	NAME
1	1.403	2147	169	V		0.0206	
2	1.571	2106	207	V		0.0211	
3	2.244	1267	179	V		0.0109	
4	3.625	3761	124	V		0.0502	
5	5.726	2898301	306651			37.4440	
6	12.703	4577892	208651			62.4163	
7	16.050	1190	63			0.0169	
		Tot:4663	518045			100.0000	

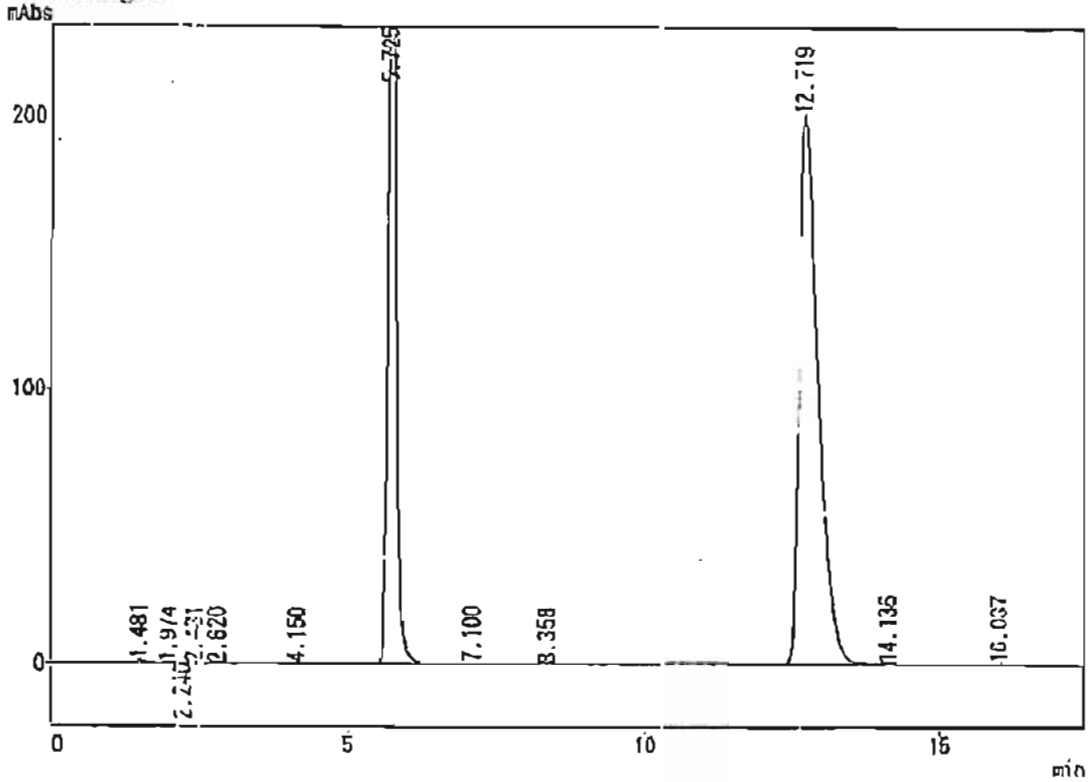
14 - 1/1

02/11/20 21:39:41

7

Sample: A-01
 ID: 60°C-4W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y20.C05

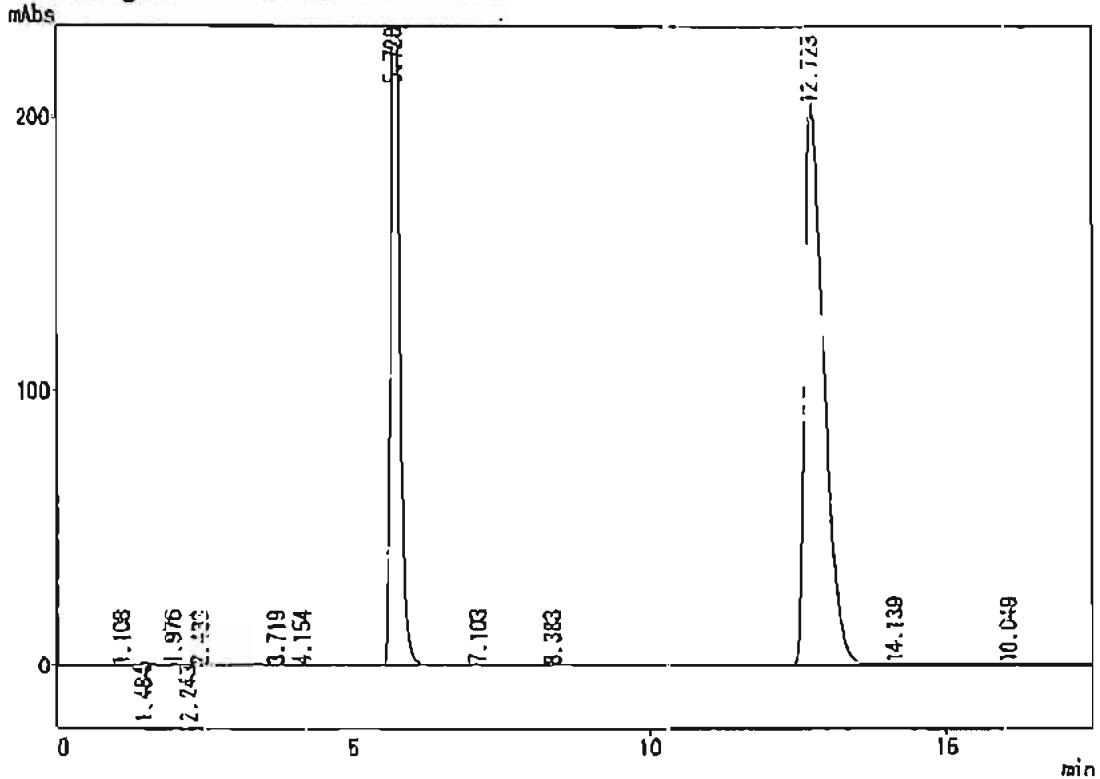


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	15097	1408	V		0.2062	
2	1.974	3630	324	V		0.0462	
3	2.240	3745	327	V		0.0519	
4	2.431	2645	208	V		0.0379	
5	2.820	2457	138	V		0.0334	
6	4.150	1780	229			0.0239	
7	5.725	2774228	299533	SV		37.8915	
8	7.100	6593	578	T		0.0896	
9	8.358	1237	93			0.0168	
10	12.719	4525804	201610	S		51.5009	
11	14.135	6843	348	T		0.0794	
12	16.037	15761	881			0.2142	
		7258801	505477			100.0010	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=16 Data=AH2Y20.D06 02/11/20 22:00:54
 Sample: A-03
 ID: 60°C-4W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y20.C06



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MR	IDNO	CONC	NAME
1	1.108	1020	107			0.0136	
2	1.484	14102	1313	V		0.1876	
3	1.976	3232	300	V		0.0431	
4	2.243	3048	274	V		0.0406	
5	2.433	1022	159	V		0.0216	
6	3.719	1247	57	V		0.0166	
7	4.154	1524	215			0.0203	
8	5.728	3921284	304786	SV		37.5708	
9	7.103	6684	588	T		0.0866	
10	8.383	1278	97			0.0170	
11	12.723	4529549	205249	S		61.6607	
12	14.139	5246	314	T		0.0899	
13	18.049	17628	748			0.2348	
		7507440	514204			100.0010	

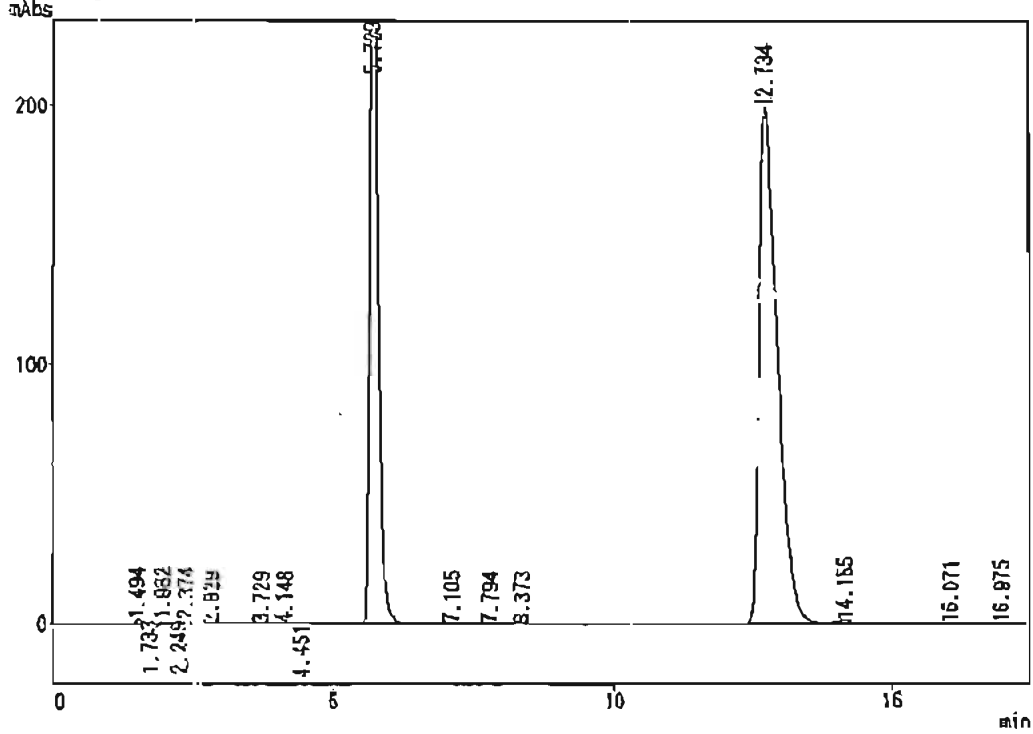
16 - 1/1

02/11/20 22:18:37

9

Sample: BF
 ID: 60°C-4W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y20.C07



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.494	17779	1828	V		0.2423	
2	1.733	4220	518	V		0.0575	
3	1.862	5283	536	V		0.0720	
4	2.249	2021	280	V		0.0275	
5	2.374	3938	349	V		0.0537	
6	2.829	1670	125	V		0.0218	
7	3.729	1250	58			0.0150	
8	4.148	2329	322			0.0317	
9	4.451	1789	236			0.0244	
10	5.729	239630	301768	S		39.0146	
11	7.105	3387	305	T		0.0462	
12	7.794	1309	120			0.0178	
13	8.373	1585	120			0.0218	
14	12.734	4474110	199580	S		60.9682	
15	14.155	23417	1200	T		0.3191	
16	15.071	2688	131			0.0368	
17	15.875	1905	86			0.0260	
		7338311	507357			100.0000	

Sample: STD

ID:

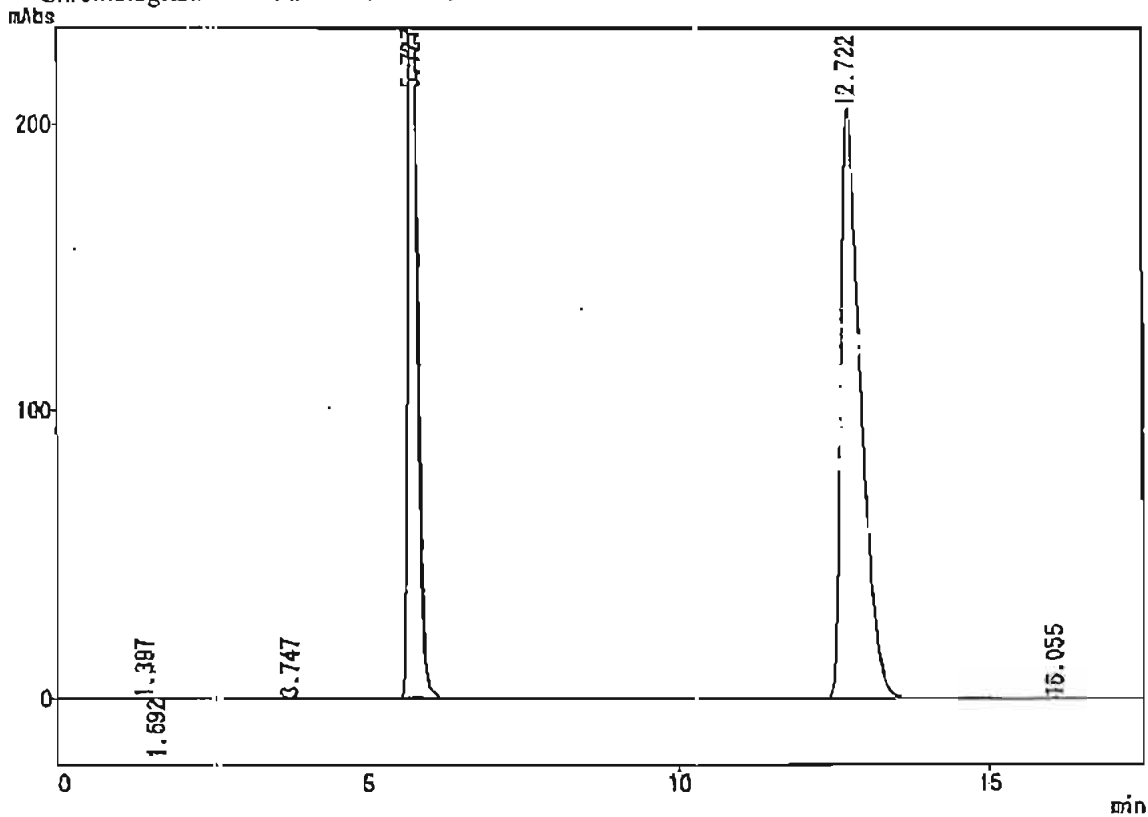
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y20.C08



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.397	1077	142	V		0.0146	
2	1.592	1020	123	V		0.0138	
3	3.747	8288	284			0.1115	
4	5.727	2781658	303946	S		37.4870	
5	12.722	4593438	206133	S		62.3517	
6	18.055	1503	72			0.0214	

7106983 510680 100.0010

Stability of bromfenac sodium
 Lot No. 02X221

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 22 October 2002

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.M.	Turbid	Permeation (%)
STD	AH2X22.C01	4613938	3002770	1.4987							
STD	AH2X22.C05	4603913	3072540	1.4984							
STD	Mean			1.4978	0.10015						
A-01	Initial	AH2X22.C02	4651972	3067148	1.5167	0.10143	100.00	8.20	—	—	
A-03	Initial	AH2X22.C03	4625716	3090209	1.4969	0.10010	100.00	8.27	—	—	
BF	Initial	AH2X22.C04	4665202	3067080	1.5280	0.10205	100.00	8.20	—	—	

Test substance	AHR10282B	Test code	P2002B131	Lot No.	02X221	Preparation date	22 October 2002	Tester	Shiro Sawa
Test item									
Amount manufactured	5mL ampoules ×	colorless	5mL colorless PP ×	5mL brown PP ×		x	x	x	
Formulation No.	A-01		A-03		Bronuck				
Ingredients and amounts	Amount of starting material (g)	Weighted amount (g)	Amount of starting material (g)	Weighted amount (g)	Amount of starting material (g)	Weighted amount (g)	22-Oct-2002 11:13:12	Weighted amount (g)	Manufacturer Lot No.
Ingredient	100mL		100mL		100mL				
Bromfenac sodium	0.1	0.10013	0.1	0.10007	0.1	0.10000	001:H + 0.10013 g		0C2647
Boric acid	1.1	1.103	1.1	1.100	1.1	1.103	002:H + 0.10007 g		01E008
Borax	1.1	1.100	1.1	1.100	1.1	1.099	003:H + 0.10000 g		00N022
Benzalkonium chloride	0.005	0.005	0.005	0.005	0.005	0.005			02B022
Tylosapol	0.02	0.02015	0.03	0.03007	—	—			E63703A07
Povidone (K-30)	2.0	2.003	2.0	2.004	2.0	2.003			01R012
Sodium edetate	0.02	0.02000	0.02	0.01997	0.02	0.02005			02A007
Polysorbate 80					0.15	0.15234			997023
Sodium sulfite					0.2	0.19993			
							22-Oct-2002 10:49:27		
							002: + 2.003 g		
							003: + 2.004 g		
							004: + 2.008 g		
							005: + 1.100 g		
							006: + 1.100 g		
							007: + 1.099 g		
							008: + 1.103 g		
							009: + 1.100 g		
							010: + 1.103 g		
							22-Oct-2002 10:19:34		
							003:H - 0.02015 g		
							004:H - 0.03007 g		
							005:H - 0.15234 g		
							006:H + 0.02000 g		
							007:H + 0.01997 g		
							008:H + 0.02005 g		
							009:H + 0.19993 g		
pH	8.3	8.17	8.3	8.21	8.3	8.28			

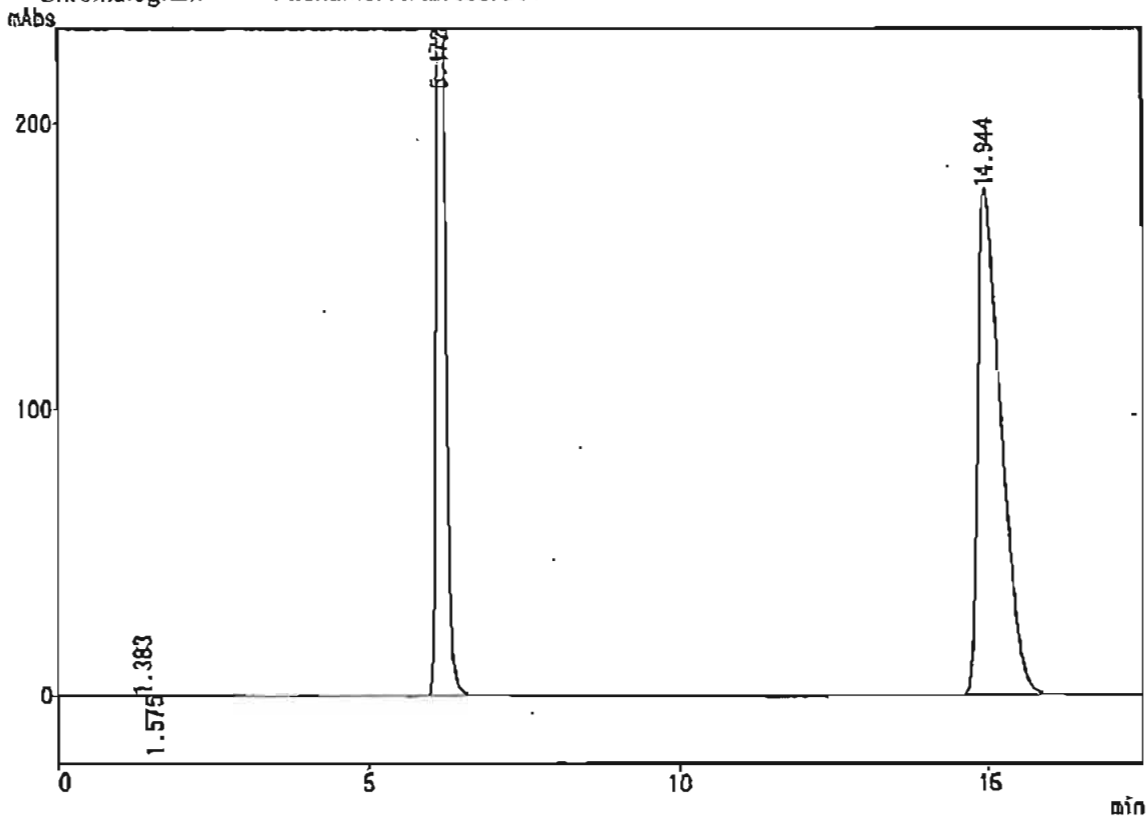
10/22 11:31
 NO.29 pH 8.17
 25.6°C

10/22 11:34
 NO.30 pH 8.21
 25.5°C

10/22 11:35
 NO.31 pH 8.23
 26.0°C

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=2 Data=AH2X22.D01 02/10/22 17:46:14
 Sample: STD
 ID:
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2X22.C01



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.383	2041	135			0.0285	
2	1.575	3334	216	V		0.0433	
3	6.172	3082770	321671			40.0251	
4	14.944	4613938	176966			59.9051	
		7702083	498989			100.0000	

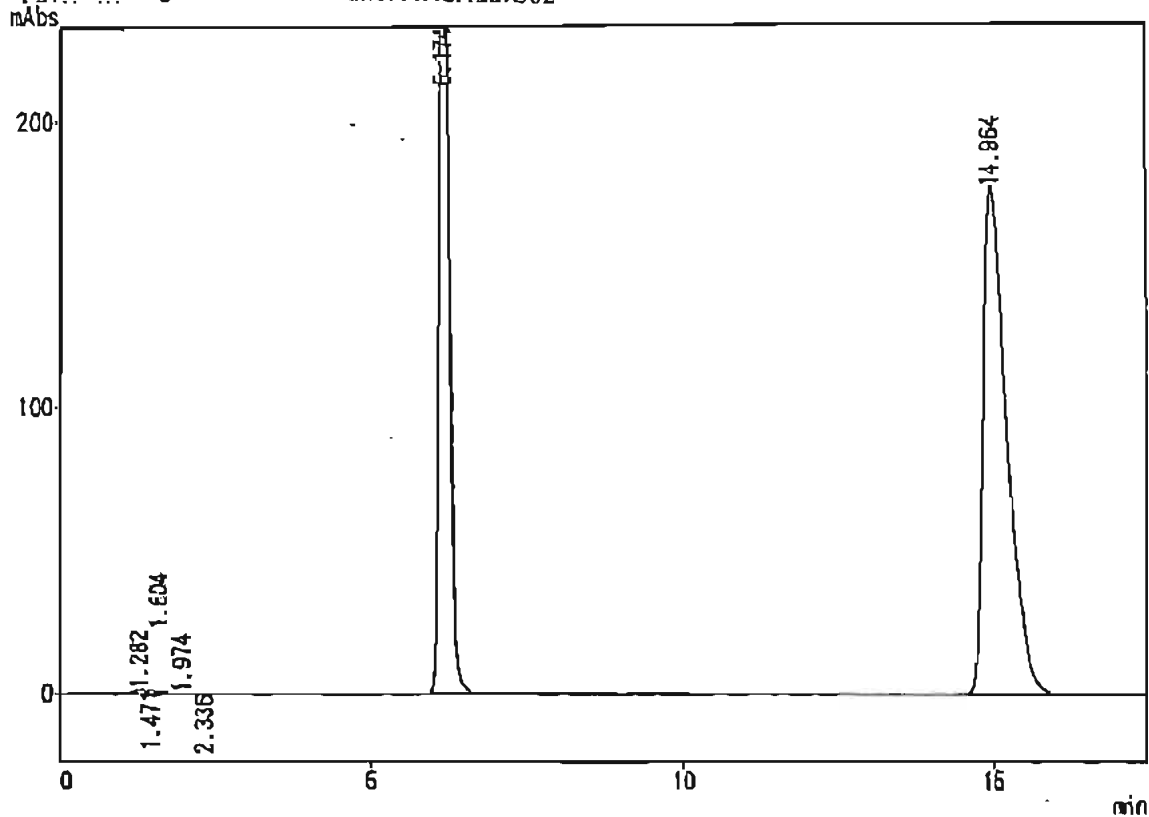
2 - 1/1

02/10/22 18:03:53

4

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=3 Data=AH2X22.D02 02/10/22 18:05:44
 Sample: A-01
 ID: Initial
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2X22.C02



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.282	18032	1319			0.2325	
2	1.471	7987	1140	V		0.1030	
3	1.604	8077	815	V		0.1041	
4	1.974	1025	104	V		0.0132	
5	2.336	1824	177	V		0.0235	
6	6.174	3067148	314342	S		39.5452	
7	14.964	4651972	178635			59.9785	
		7756064	494533			100.0000	

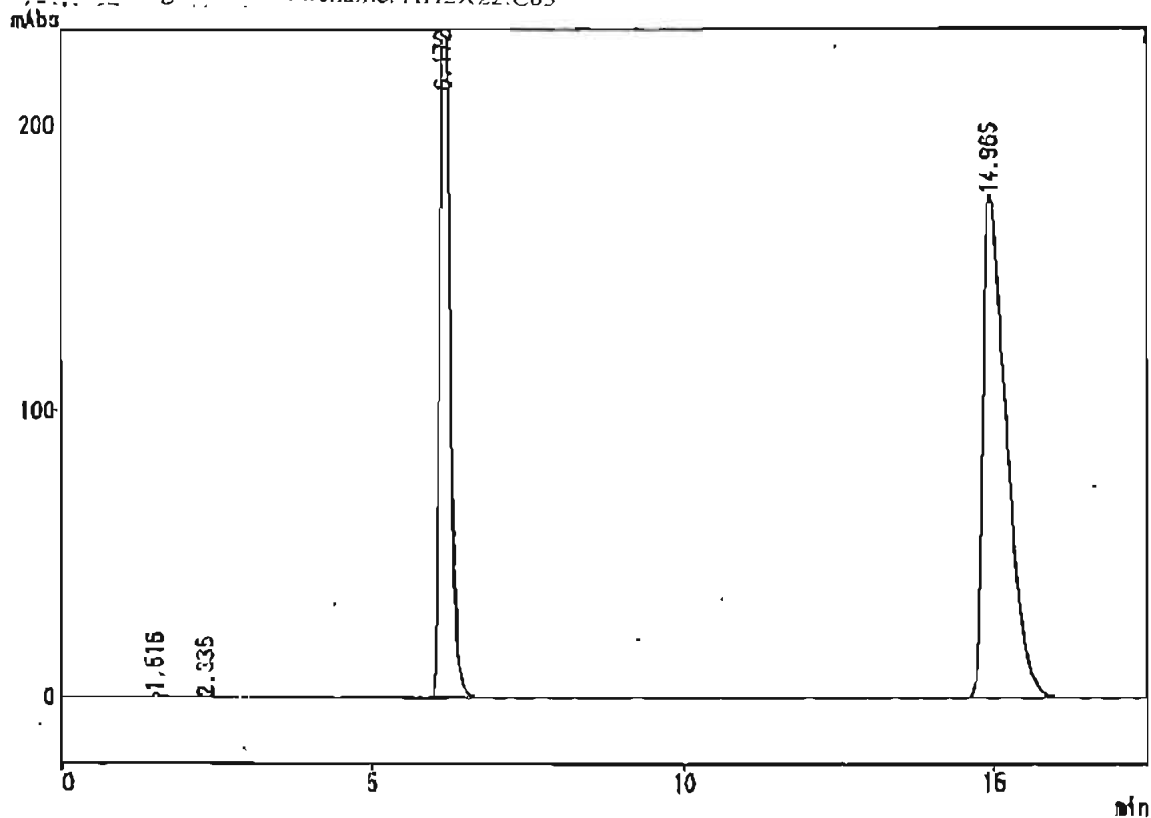
3 - 1/1

02/10/22 18:23:25

5

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=4 Data=AH2X22.D03 02/10/22 18:25:12
 Sample: A-03
 ID: Initial
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2X22.C03



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.516	15418	1678	SV		0.1994	
2	2.335	1650	169	V		0.0200	
3	6.172	3090209	317009			39.9619	
4	14.965	4825716	175708			59.8187	
		7732891	494563			100.0000	

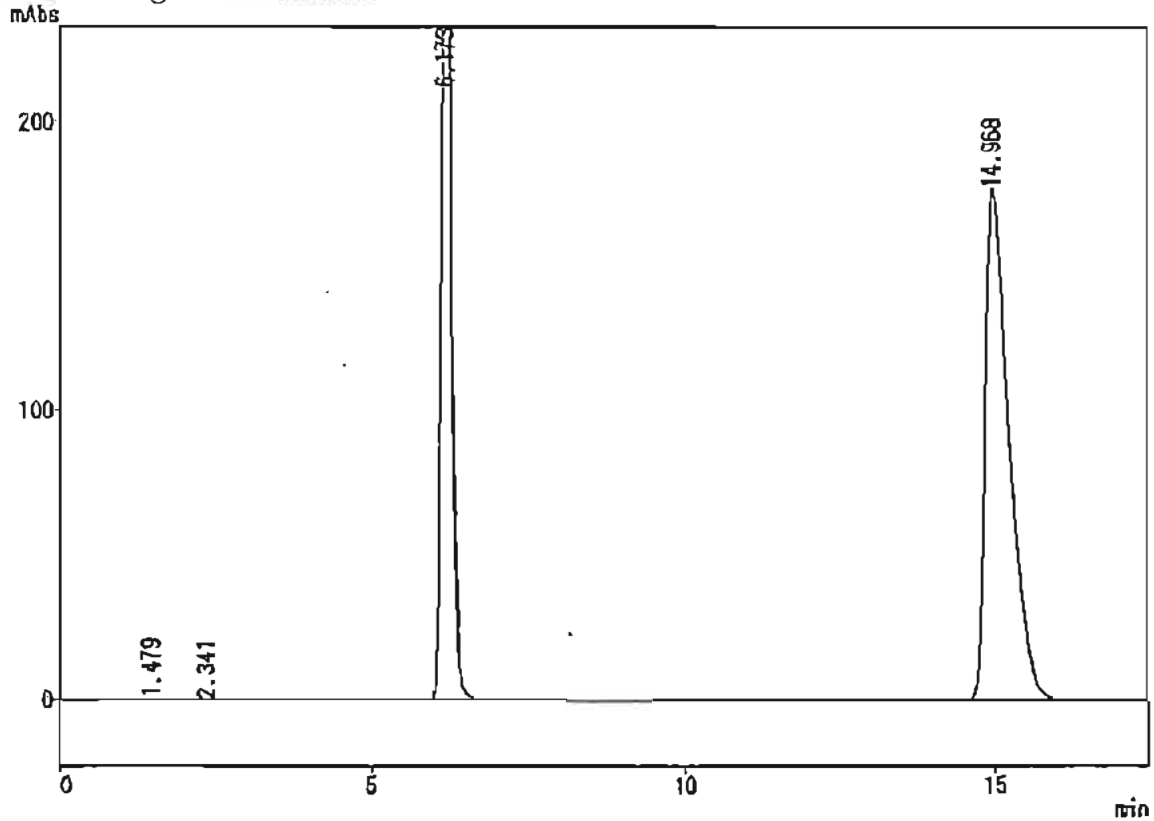
4 - 1/1

02/10/22 18:42:58

6

Sample: BF
 ID: Initial
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2X22.C04

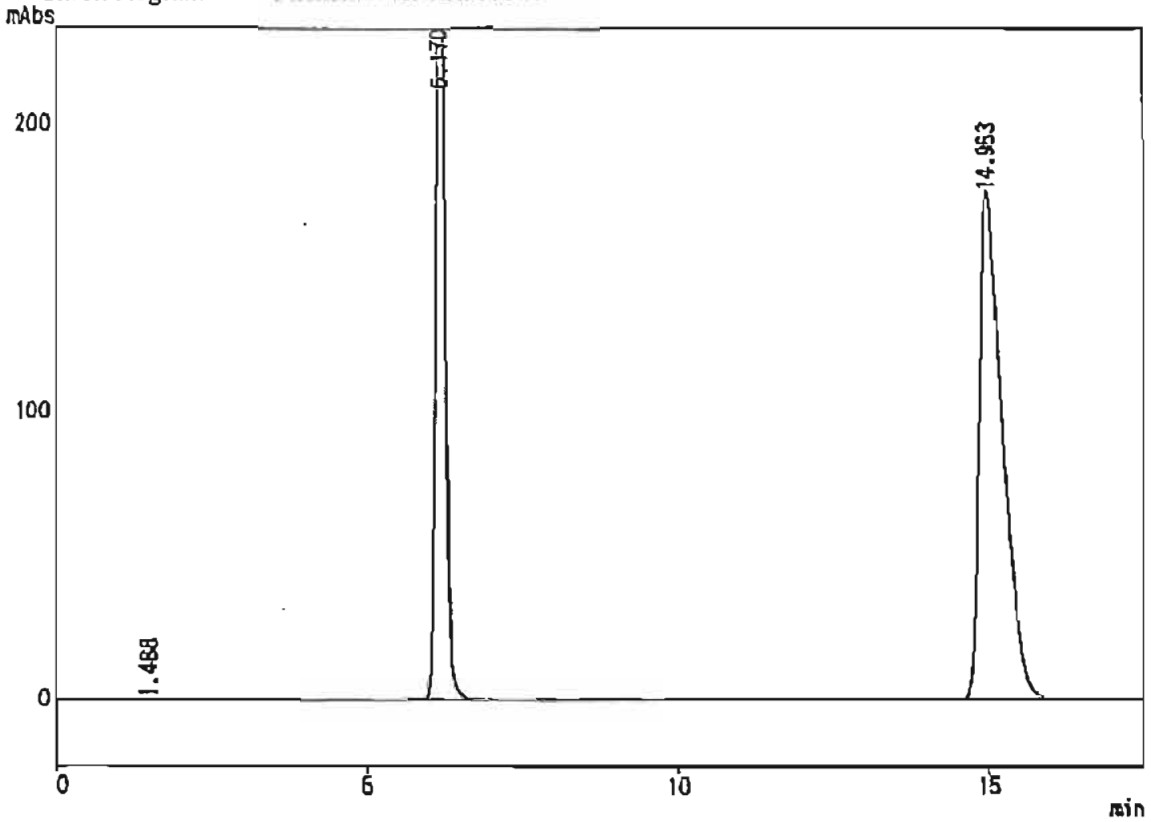


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.479	8870	711	V		0.0889	
2	2.341	1764	147	V		0.0228	
3	6.173	3057080	313853			39.5438	
4	14.968	4865202	176971			60.3448	
		7730916	491482			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=6 Data=AH2X22.D05 02/10/22 19:04:12
 Sample: STD
 ID:
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: IAHRI028.MET

*** Chromatogram *** Filename: AH2X22.C05



*** Peak Report ***

PK#	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.488	4818	233	V		0.0627	
2	8.170	3072549	320993			40.0005	
3	14.953	4603915	178570			58.9388	
		7681282	497796			100.0000	

6 - 1/1

02/10/22 19:21:53

8

Stability of bromfenac sodium
 Lot No. 02X221

Test code: P2002B131
 Tester: Shirou Sawa
 Test date: 13 November 2002

STD	Mean	Chromato No.	AHR	Peak Area		Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
				IS	Ratio							
		AH2Y13.013	4579483	2805042	1.6320	0.10015						
A-01	60°C-2W	AH2Y13.014	4525801	2800112	1.6163	0.09919	87.78	94.33	8.23	—	—	3.54
A-03	60°C-2W	AH2Y13.015	4518470	2783708	1.6232	0.09961	89.51	95.88	8.21	—	—	3.55
BF	60°C-2W	AH2Y13.016	4452342	2778799	1.6023	0.09823	88.35	92.97	8.31	—	—	3.51

Sample: STD

ID:

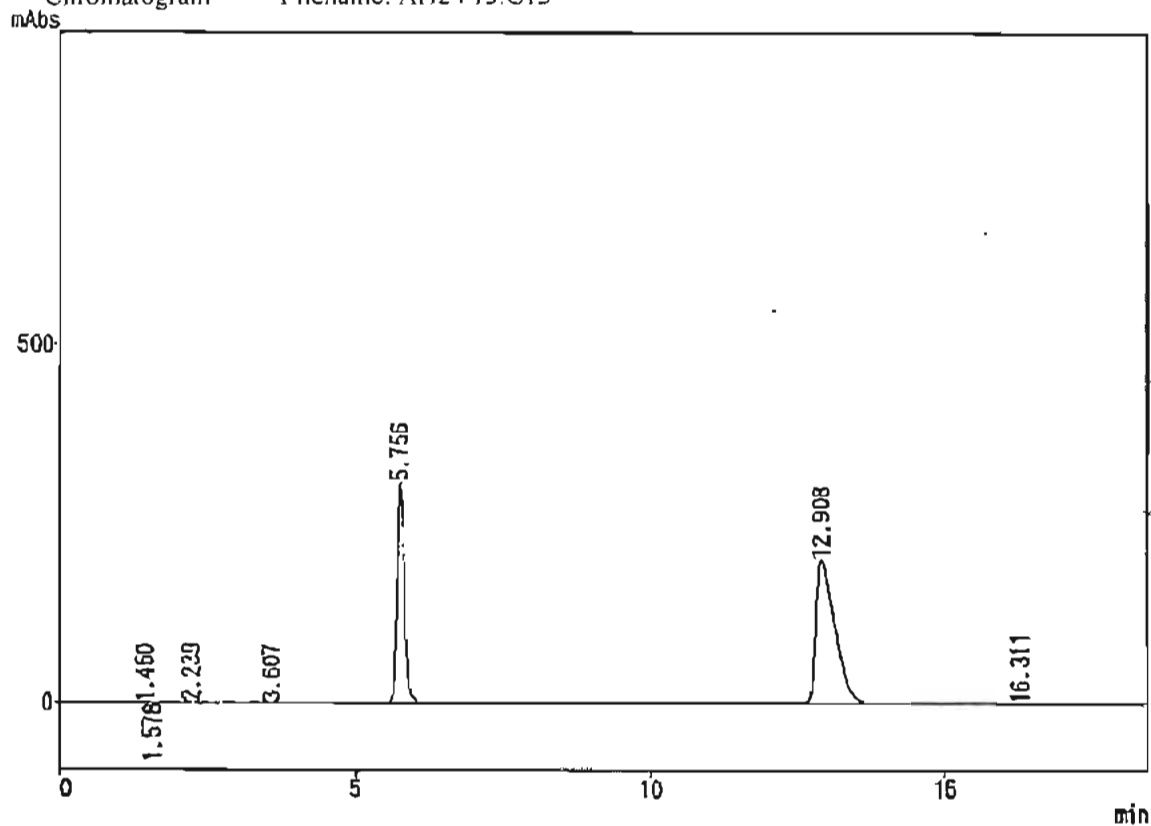
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C13



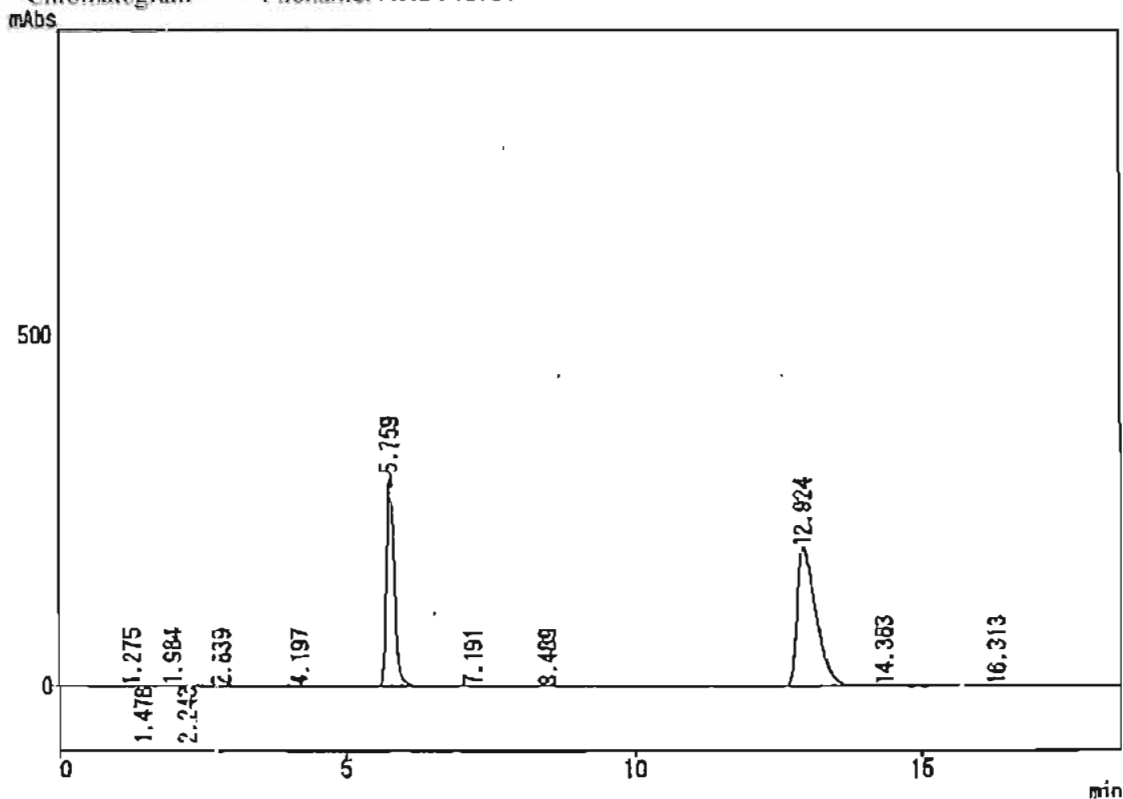
*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.450	2039	152	V		0.0275	
2	1.678	1432	174	V		0.0193	
3	2.239	8772	1145	SV		0.1185	
4	3.607	4543	152			0.0814	
5	5.756	2006042	308208			37.9009	
6	12.908	4579463	203270	S		61.8543	
7	16.311	1338	67			0.0181	

7403829 513165 100.0000

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No. 89 Data=AH2Y13.D14 02/11/13 18:53:46
 Sample: A-01
 ID: 60°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C14



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.275	1843	145			0.0249	
2	1.478	18100	1534	V		0.2180	
3	1.984	3388	288	V		0.0459	
4	2.243	11878	1147	V		0.1800	
5	2.839	1298	96	V		0.0178	
6	4.197	1489	191			0.0202	
7	5.759	2100112	300917	S		37.9081	
8	7.191	5862	502	T		0.0792	
9	8.489	2302	105			0.0312	
10	12.924	4525901	198764	S		81.2887	
11	14.363	3920	237	T		0.0531	
12	16.313	12883	544			0.1744	

7386867

504530

100.0000

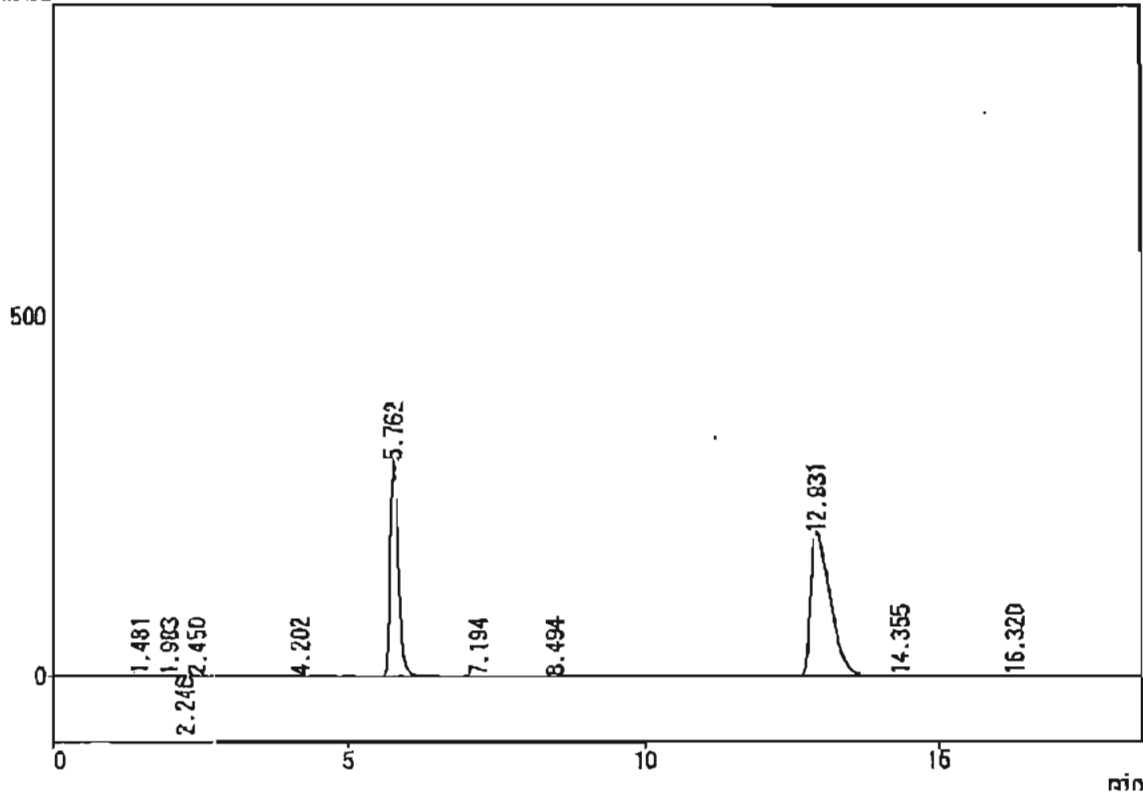
89 - 1/1

02/11/13 19:12:24

4

Sample: A-03
 ID: 60°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C15
 mAbs

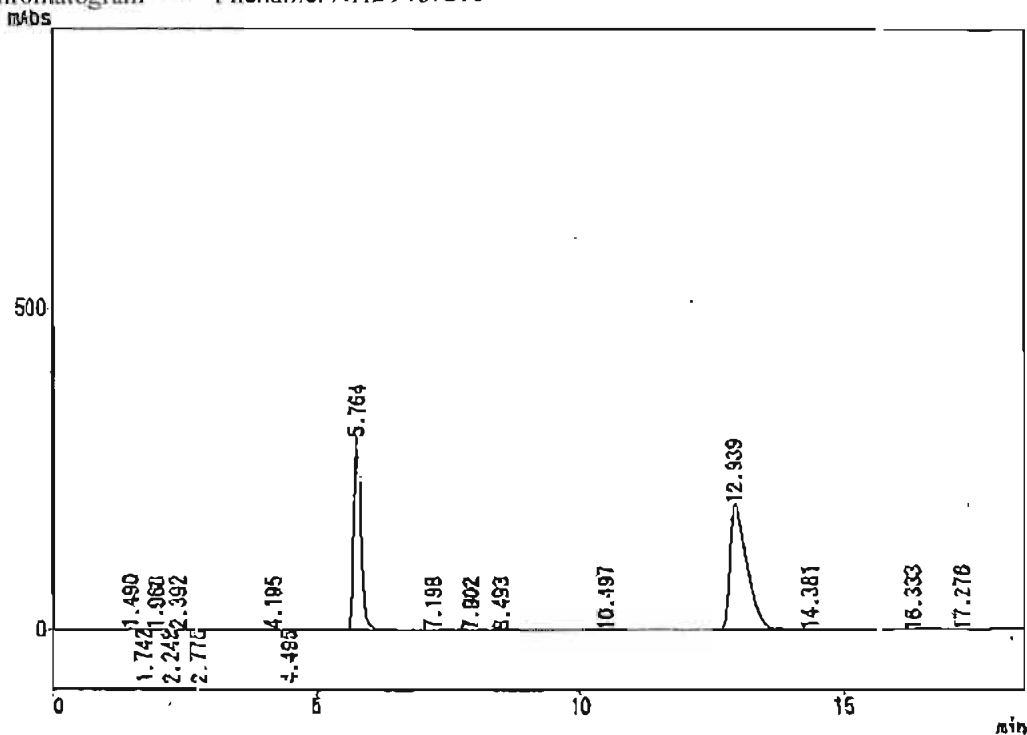


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MX	IDNO	CONC	NAME
1	1.481	14369	1399	V		0.1954	
2	1.983	3016	267	V		0.0410	
3	2.246	6609	831	V		0.0907	
4	2.450	1815	179	V		0.0247	
5	4.202	1308	176			0.0178	
6	5.762	2783706	299623	S		37.8508	
7	7.194	5833	506	T		0.0793	
8	8.494	2510	178			0.0342	
9	12.931	4518470	198351	S		61.4385	
10	14.355	3862	216	T		0.0498	
11	16.320	13102	560			0.1781	
		7354464	502288			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=91 Data=AH2Y13.D16 02/11/13 19:33:44
 Sample: BF
 ID: 60°C-2W
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH2Y13.C16



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.490	17354	1547	V		0.2379	
2	1.742	3587	468	V		0.0492	
3	1.868	4314	472	V		0.0591	
4	2.248	5502	744	V		0.0754	
5	2.392	4180	392	V		0.0570	
8	2.775	1153	95	V		0.0158	
7	4.195	2027	276			0.0278	
8	4.495	1818	244			0.0283	
9	5.764	2778799	289685			38.0918	
10	7.198	2384	209			0.0324	
11	7.802	1173	107			0.0161	
12	8.493	1552	114			0.0213	
13	10.497	1005	79			0.0138	
14	12.939	4452342	198001	S		61.0327	
15	14.381	14233	730	T		0.1951	
16	16.333	1856	80			0.0254	
17	17.278	1674	77			0.0229	
		7195013	501328			100.0000	

Stability of bromfenac sodium
Lot No. 02X221

Test code: P2002B131
Tester: Shirou Sawa
Test date: 22 April 2003

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid
STD	AH3N22.C01	4553327	2819852	1.6147						
STD	AH3N22.C11	4557788	2834325	1.6081						
STD	Mean			1.6114	0.10100					
A-01	40°C-6M	AH3N22.C02	4285770	2845412	1.5002	0.09441	93.08	91.85	8.29	--
A-03	40°C-8M	AH3N22.C03	4224937	2814027	1.5014	0.09411	94.02	92.88	8.30	--
BF	40°C-6M	AH3N22.C04	4222368	2838573	1.4875	0.09321	91.36	90.02	8.33	--
A-01	25°C-4M	AH3N22.C05	4427810	2839972	1.6591	0.09772	96.34	95.97	8.30	--
A-03	25°C-4M	AH3N22.C06	4409879	2840521	1.5528	0.09731	97.21	96.84	8.31	--
BF	25°C-4M	AH3N22.C07	4470457	2832712	1.5782	0.09862	96.93	96.58	8.37	--
A-01	25°C-6M	AH3N22.C08	4388198	2823021	1.5473	0.09898	95.81	94.95	8.30	--
A-03	25°C-6M	AH3N22.C09	4404588	2826921	1.5581	0.09768	97.56	98.95	8.30	--
BF	25°C-6M	AH3N22.C10	4486855	2852405	1.5730	0.09859	96.81	95.99	8.35	--

Test Record B (other) Form 7 (1 January 2000)

Test substance	AHR10282B	Test code	P2002B131	Test date	22 April 2003
Test item				Tester	Shirou Sawa

IS: 0.01676g of methyl p-hydroxybenzoate + mobile phase → 50mL

STD1: 0.02020g of AHR10282B + mobile phase → 20mL

STD2: 2mL of STD1 (AHR) + 2mL of IS (AHR) + mobile phase → 20mL

Sample: 2mL of this product + 2mL of IS (AHR) + mobile phase → 20mL

22-Apr-2003	10:11:24
004:N	+ 0.02020_9
005:N	+ 0.01676_9

		pH	Ext. app.	Weight change		
A-01	40°C-6M	8.29	—	9.3565	9.2686	
A-03	↓	8.30	—	9.4002	9.3186	
BF	↓	8.33	—	9.3570	9.2730	
A-01	25°C-4M	8.30	—	9.4249	9.4032	4/22 13:48
A-03	↓	8.31	—	9.3834	9.3619	NO.62 PH 8.29
BF	↓	8.37	—	9.2564	9.2364	26.2°C
A-01	25°C-6M	8.30	—	9.3086	9.2698	4/22 13:49
A-03	↓	8.30	—	9.3720	9.3360	NO.63 PH 8.33
BF	↓	8.35	—	9.3372	9.3006	26.7°C
						4/22 13:50
						NO.64 PH 8.35
						26.7°C
						4/22 13:51
						NO.65 PH 8.33
						26.7°C
						4/22 13:53
						NO.66 PH 8.31
						26.8°C
						4/22 13:54
						NO.67 PH 8.37
						26.7°C
						4/22 13:55
						NO.68 PH 8.30
						26.8°C
						4/22 13:56
						NO.69 PH 8.33
						26.8°C
						4/22 13:56
						NO.70 PH 8.35
						26.6°C

Sample: TEST

ID:

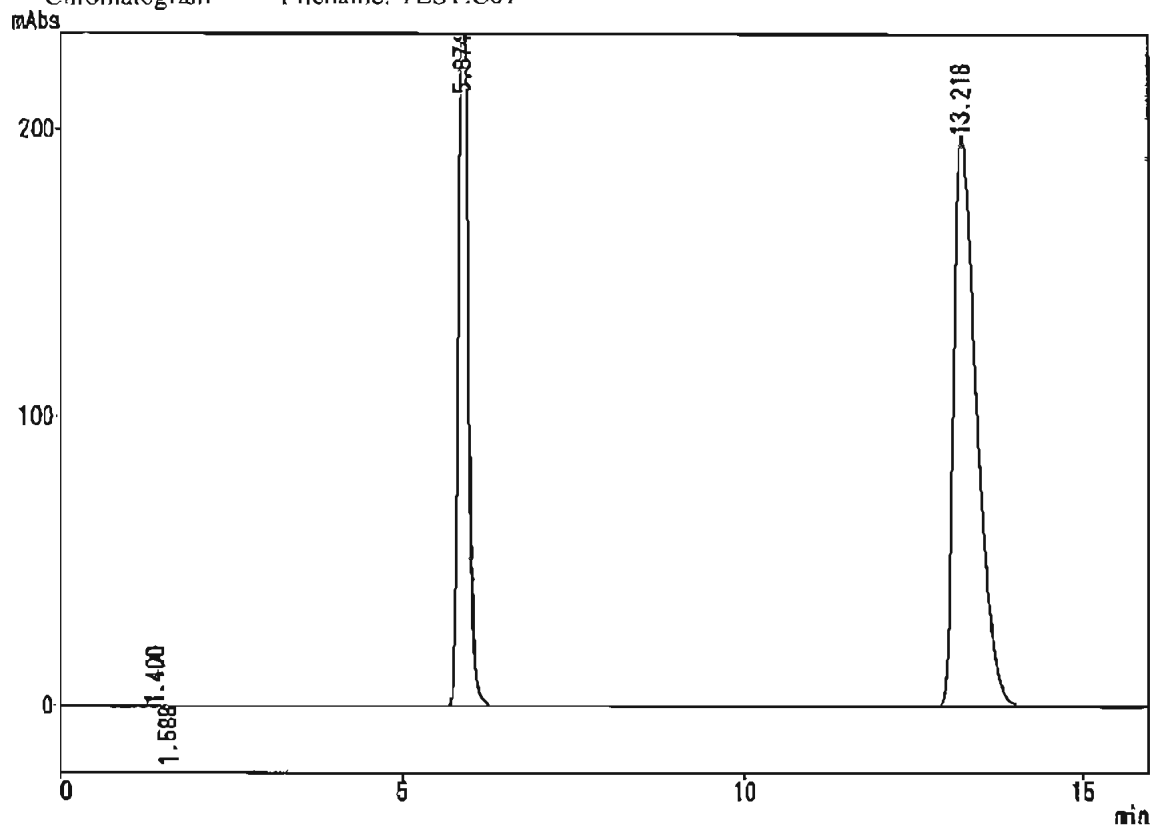
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: TEST.C01



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.400	1828	124	V		0.0248	
2	1.588	1108	122	V		0.0150	
3	5.874	2820808	304780			38.2274	
4	13.218	4556271	198298			81.7328	
		7379013	503324			100.0000	

Sample: STD

ID:

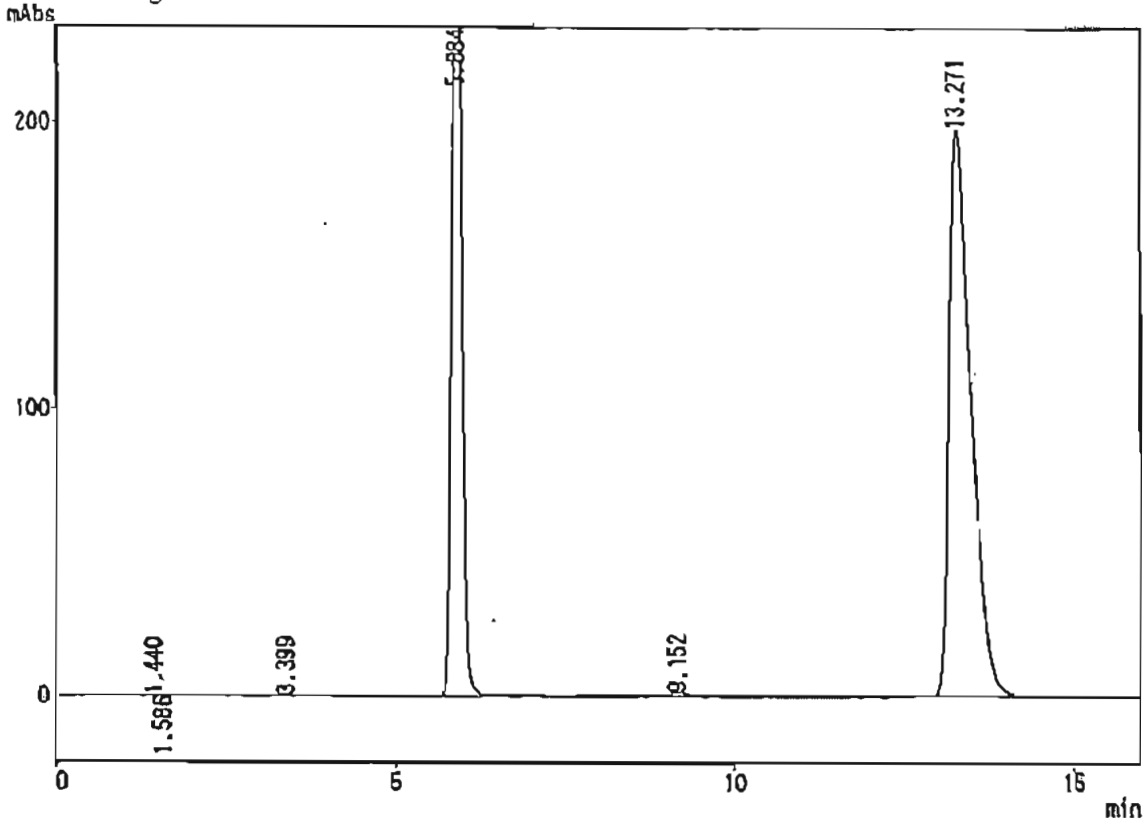
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHR1028.MET

*** Chromatogram *** Filename: A113N22.C01

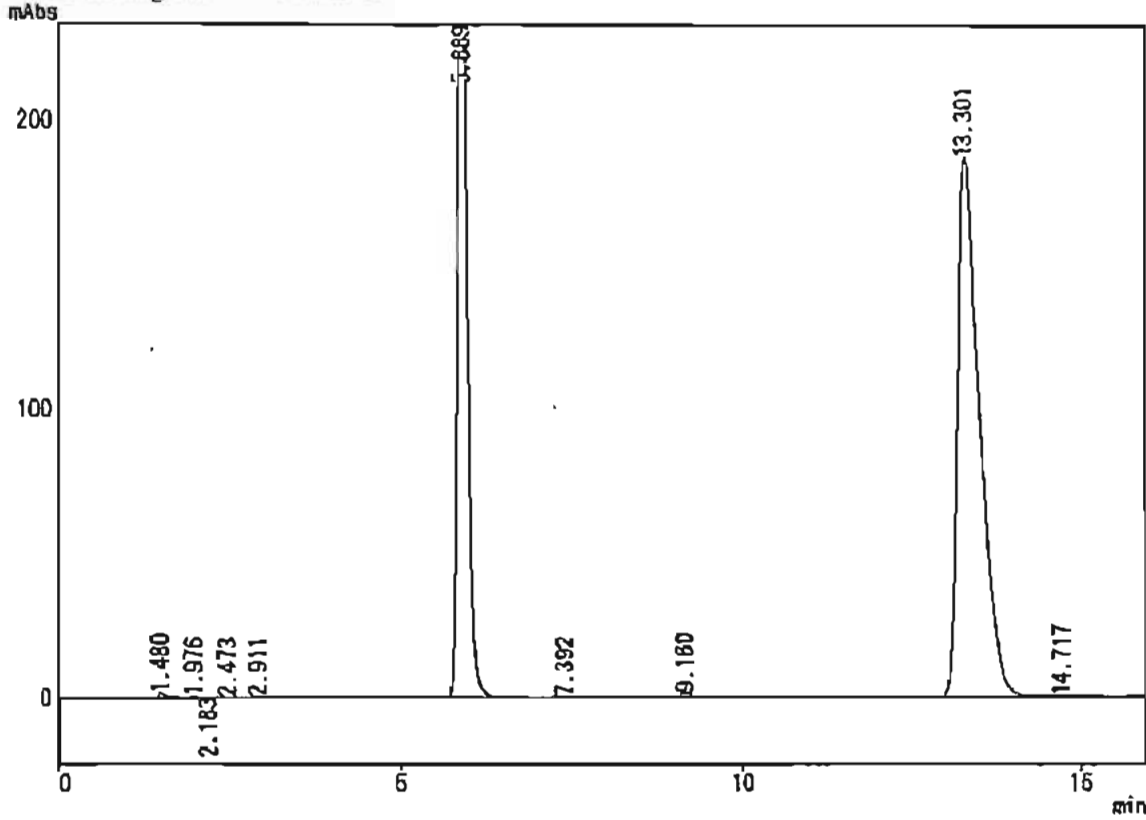


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.440	1443	128	V		0.0195	
2	1.586	1715	130	V		0.0232	
3	3.399	1194	184			0.0181	
4	5.884	2819952	304679			38.1138	
5	9.152	21145	1693			0.2858	
6	13.271	4553327	188023			61.5418	
		7398775	504715			100.0000	

Sample: A01
 ID: 40°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C02

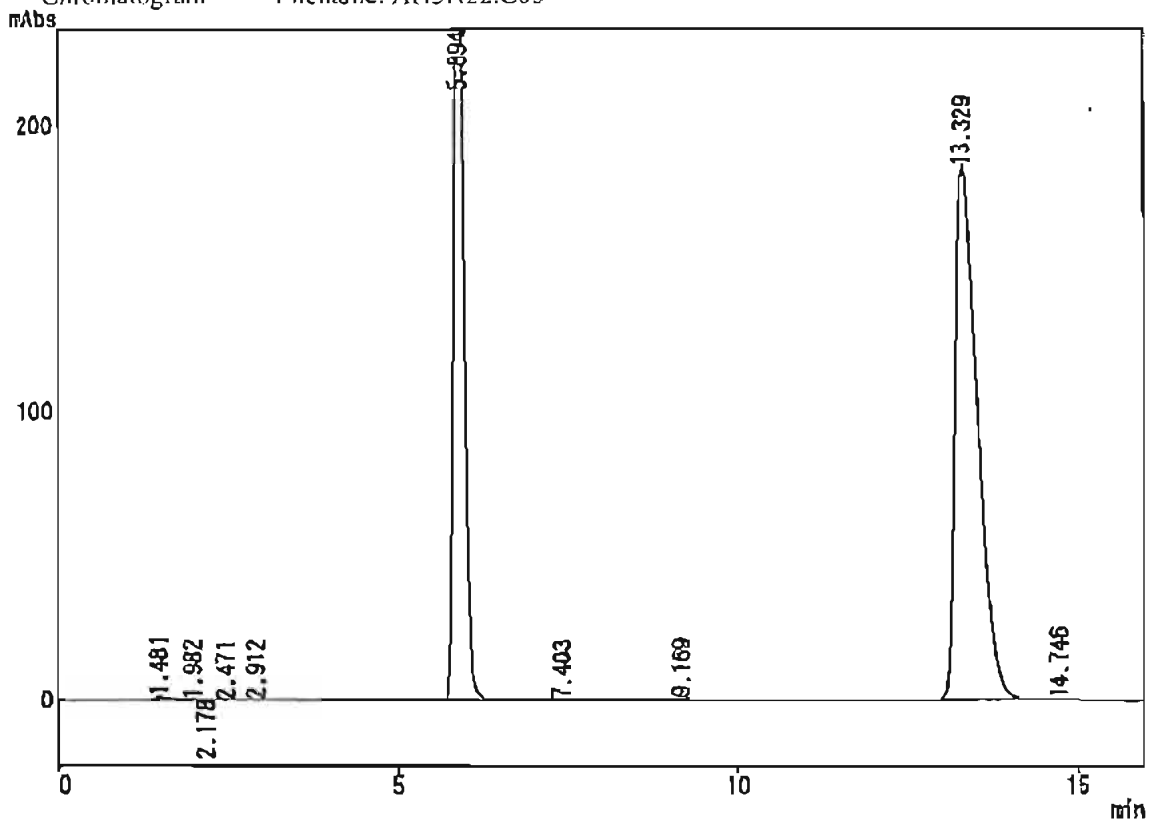


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.480	16435	1525	V		0.2145	
2	1.976	3117	265	V		0.0433	
3	2.183	1677	207	V		0.0219	
4	2.473	2311	172	V		0.0321	
5	2.911	3517	381	V		0.0489	
6	5.889	2845412	308190			39.5437	
7	7.392	7708	652			0.1071	
8	9.160	24302	1787	V		0.3377	
9	13.301	4285770	186873	S		59.5609	
10	14.717	6485	343	TV		0.0898	
		7195813	498398			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=32 Data=AH3N22.D03 03/04/22 13:08:18
 Sample: A03
 ID: 40°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C03



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	15393	1648	V		0.2170	
2	1.982	2418	226	V		0.0341	
3	2.178	1298	176	V		0.0183	
4	2.471	1399	134	V		0.0197	
5	2.912	2357	341	V		0.0332	
6	5.894	2814027	302517			39.6685	
7	7.403	7828	678			0.1104	
8	9.169	18125	1348	V		0.2556	
9	13.329	4224937	185839			69.6577	
10	14.746	8080	339			0.0857	

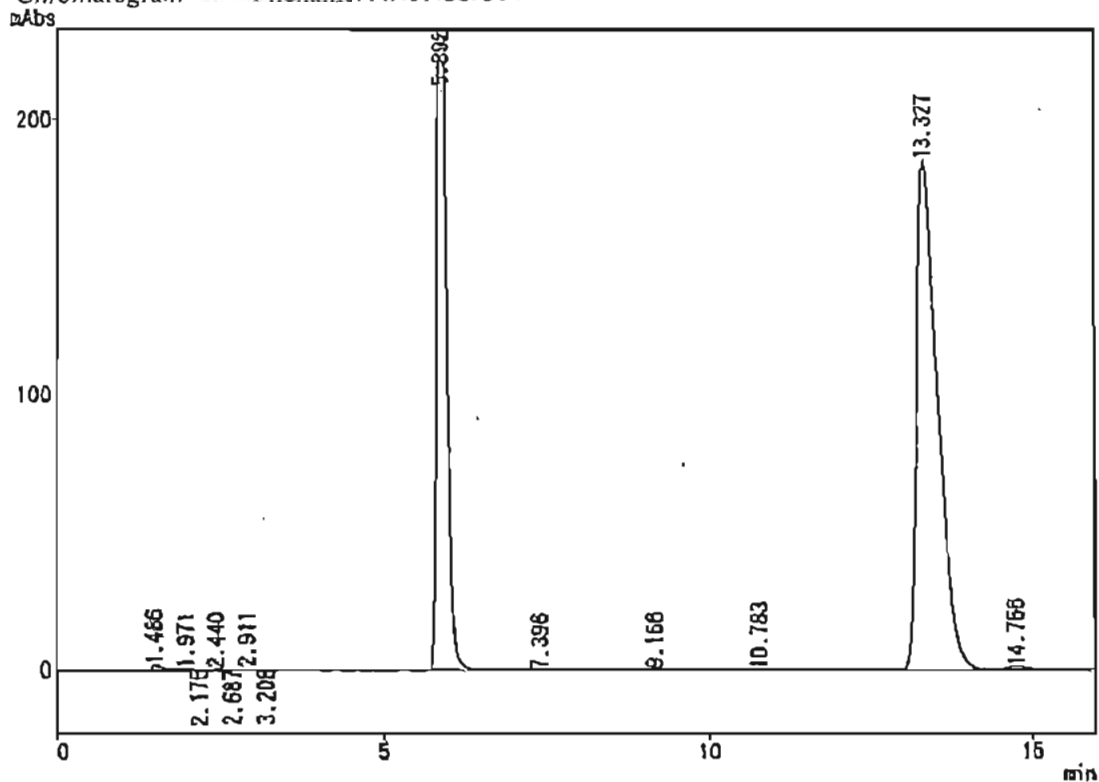
7093860 492945 100.0000

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03/04/22 13:24:30

Sample: BF
 ID: 40°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: \AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C04

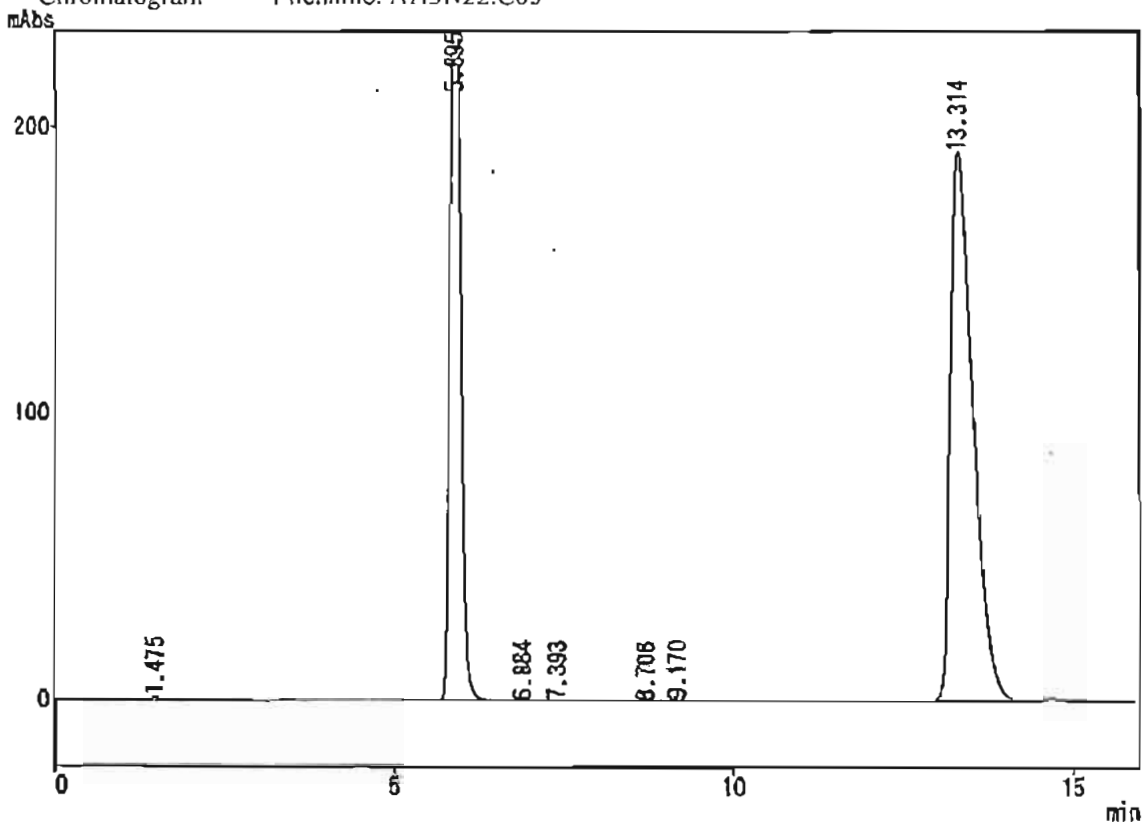


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.488	23939	1819	V		0.3343	
2	1.971	4231	505	V		0.0591	
3	2.175	1414	211	V		0.0197	
4	2.440	4989	444	V		0.0694	
5	2.687	1271	169	V		0.0177	
6	2.911	7124	917	V		0.0995	
7	3.208	1311	127	V		0.0183	
8	5.892	2848573	304476	S		39.7811	
9	7.396	2201	197	T		0.0307	
10	9.166	9418	711	V		0.1315	
11	10.783	2921	191	V		0.0408	
12	13.327	4222386	184975			58.9668	
13	14.758	30858	1228	V		0.4309	
		7180615	495969			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=34 Data=AH3N22.D05 03/04/22 13:43:14
 Sample: A01
 ID: 25°C-4M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C05

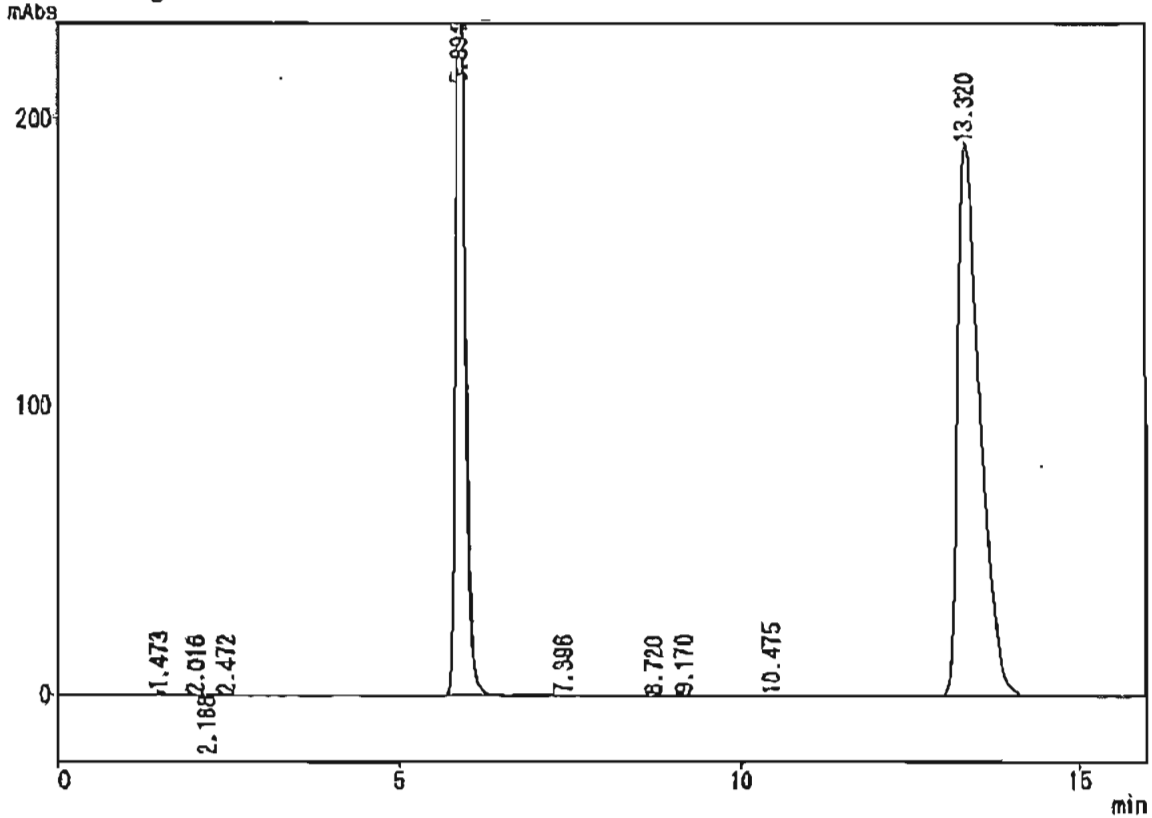


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.475	11771	1337	SV		0.1810	
2	5.895	2839972	305128			38.8489	
3	6.884	12107	373	V		0.1658	
4	7.393	6430	471	V		0.0880	
5	8.708	1873	128			0.0229	
6	9.170	10537	794	V		0.1441	
7	13.314	4427810	192161			60.5695	
		7310300	500380			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=35 Data=AH3N22.D06 03/04/22 14:00:42
 Sample: A03
 ID: 25°C-4M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C06



*** Peak Report ***

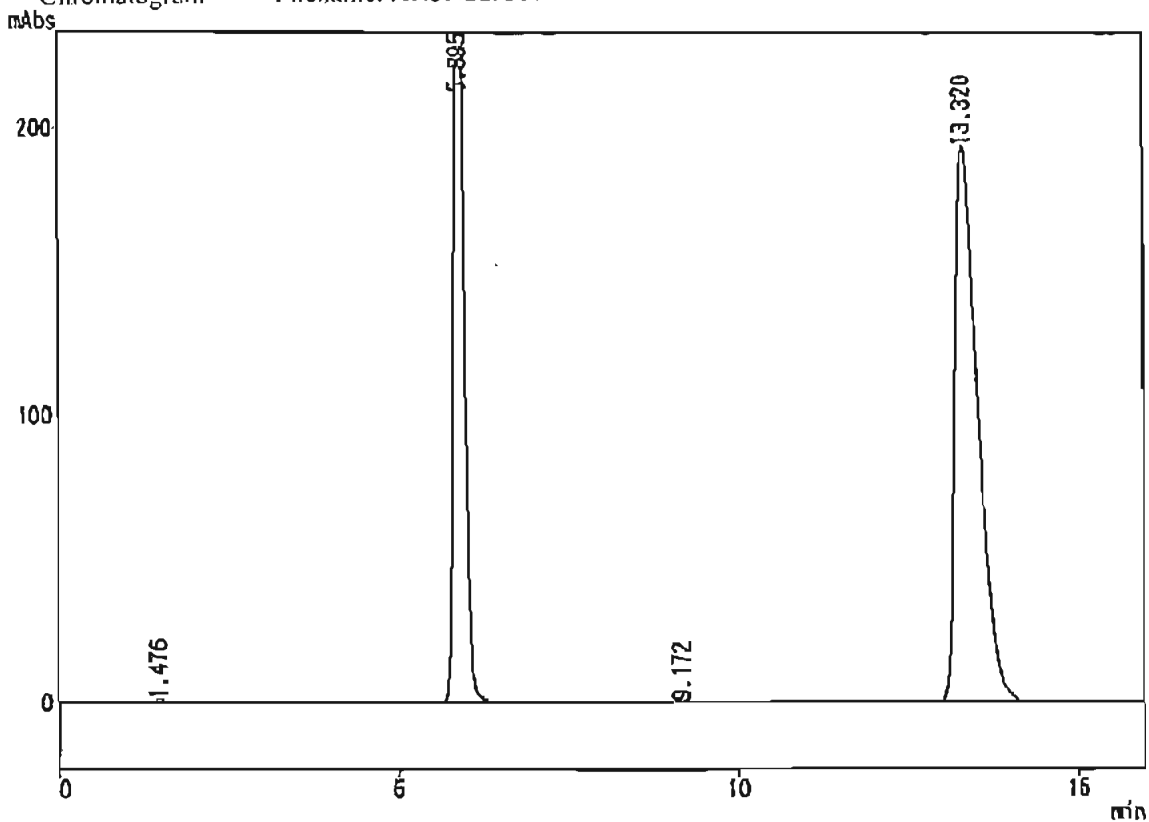
PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.473	11713	1383	SV		0.1609	
2	2.016	1258	117	T		0.0173	
3	2.188	1064	103	TV		0.0148	
4	2.472	1051	82	TV		0.0144	
5	5.894	2840521	305524			39.0137	
6	7.396	4561	380			0.0628	
7	8.720	1872	144			0.0271	
8	9.170	7400	553	V		0.1018	
9	10.475	1409	84			0.0193	
10	13.320	4409879	191505			60.5684	
		7280826	499875			100.0000	

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03/04/22 14:16:58

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=36 Data=AH3N22.D07 03/04/22 14:18:12
 Sample: BF
 ID: 25°C-4M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C07



*** Peak Report ***

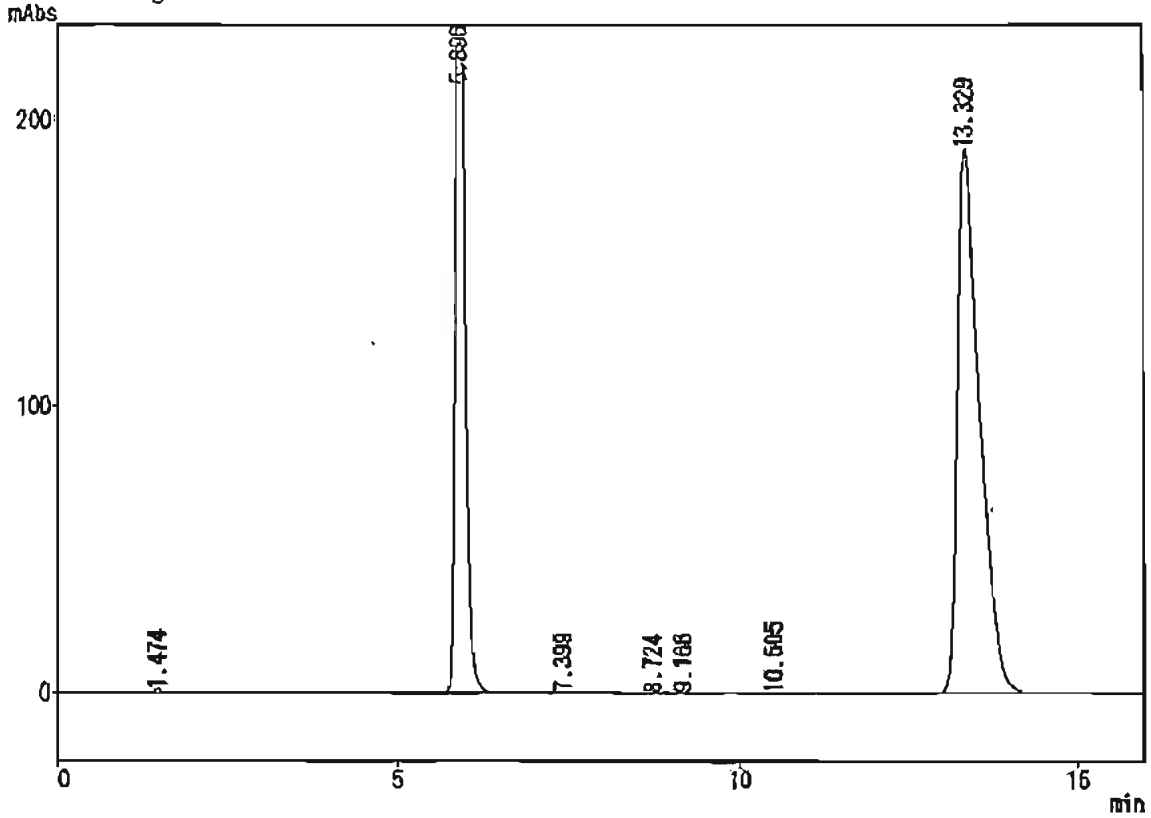
PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.476	14738	1308	V		0.2013	
2	5.895	2832712	304412			38.6819	
3	9.172	5195	398			0.0709	
4	13.320	4470457	183753	S		81.0459	
		7323102	499870			100.0000	

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03/04/22 14:34:21

Sample: A01
 ID: 25°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C08

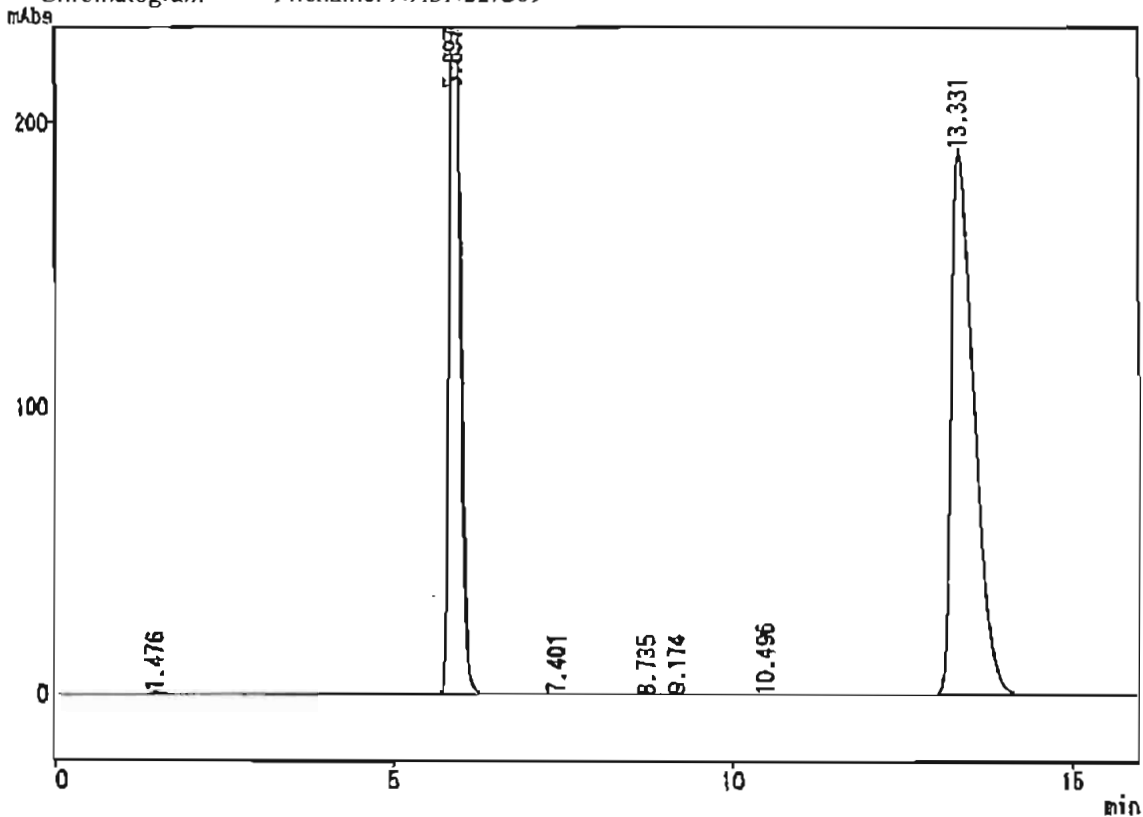


*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.474	10357	1271	V		0.1438	
2	5.898	2823021	303876			39.1311	
3	7.398	5142	430			0.0713	
4	8.724	1643	122			0.0228	
5	9.168	4570	348	V		0.0833	
8	10.505	1338	84			0.0185	
7	13.329	4388198	189924			60.5494	
		7214289	498062			100.0000	

CLASS-LC10 Ver=1.62 System No=1 Ch=1 Report No=38 Data=AH3N22.D09 03/04/22 14:53:08
 Sample: A03
 ID: 25°C-6M
 Type: Unknown sample
 Detector: SPD-10A single
 Operator: Shirou Sawa
 Method: IAH1028.MET

*** Chromatogram *** Filename: AH3N22.C09



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.476	9893	1142	V		0.1363	
2	5.897	2826921	304114			38.9537	
3	7.401	5145	438			0.0709	
4	8.735	1792	134			0.0247	
5	9.174	7142	534	V		0.0984	
8	10.496	1658	109			0.0230	
7	13.331	4404577	191088			60.6930	
		7257137	497658			100.0000	

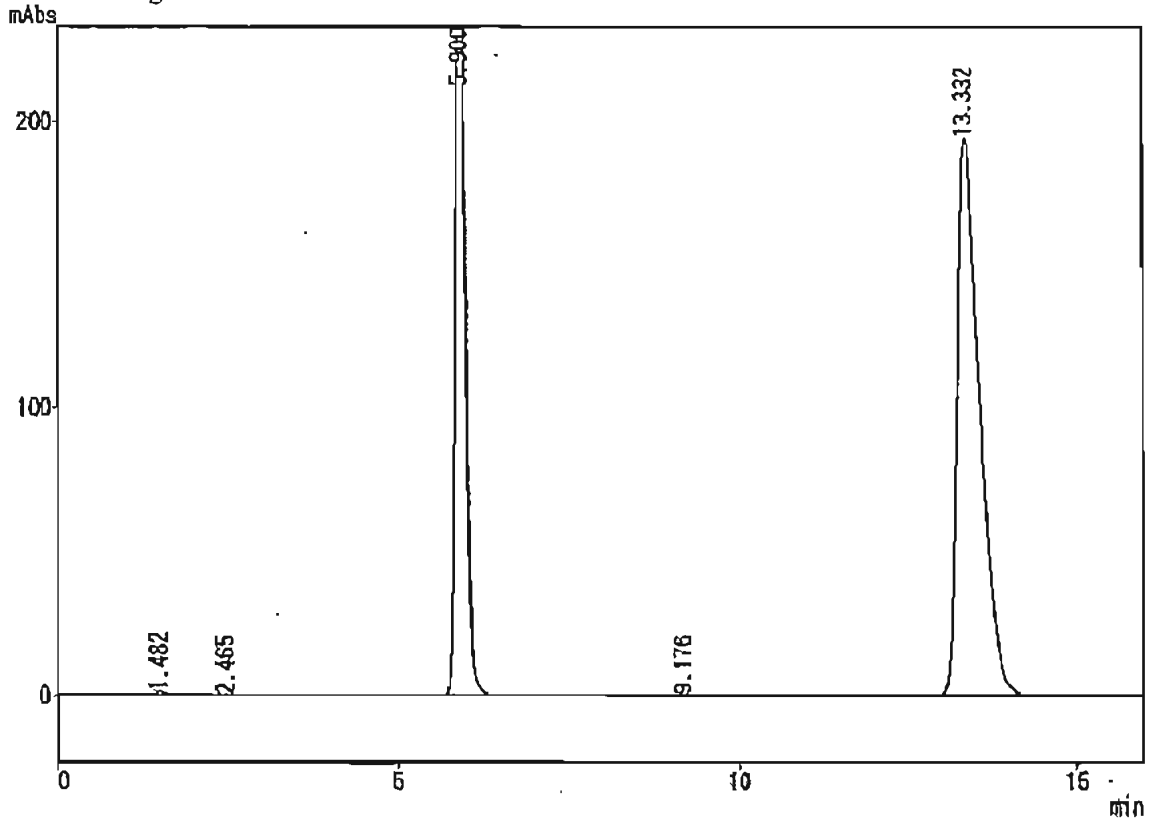
38 - 1/1

03/04/22 15:09:16

12

Sample: BF
ID: 25°C-6M
Type: Unknown sample
Detector: SPD-10A single
Operator: Shirou Sawa
Method: !AHR1028.MET

*** Chromatogram *** Filename: AH3N22.C10



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	15801	1277	SV		0.2119	
2	2.465	1935	183	V		0.0283	
3	5.900	2862405	305281			38.7608	
4	9.176	4107	311			0.0558	
5	13.332	4486855	194369			60.9552	
		7380903	601420			100.0000	

Sample: STD

ID:

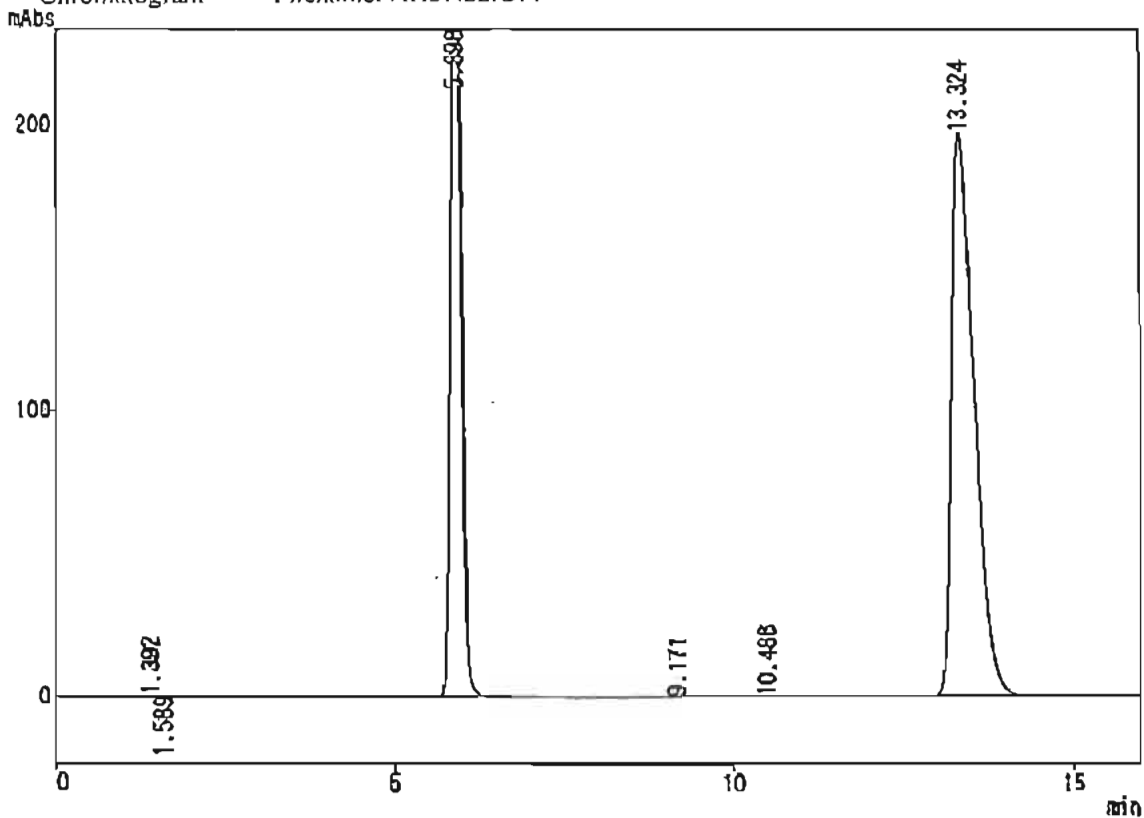
Type: Unknown sample

Detector: SPD-10A single

Operator: Shirou Sawa

Method: !AHRI028.MET

*** Chromatogram *** Filename: AH3N22.C11



*** Peak Report ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.392	1803	131	V		0.0244	
2	1.589	1957	149	V		0.0266	
3	5.898	2034325	305722			38.2981	
4	9.171	2688	202	V		0.0380	
6	10.488	2142	135			0.0289	
8	13.324	4557785	197383			81.5860	

7400689 503701 100.0000

試験計画書

試験名：EP に適合するプロナック点眼液の処方設計

試験コード：P2002B131

試験系：なし

開発記号：AHR10282B

試験開始日：2002年06月26日

試験操作開始予定日：2002年06月26日

試験操作終了予定日：2002年12月28日

試験終了予定日：2003年01月31日

試験施設：千寿製薬株式会社 コーベクリエイティブセンター
神戸市西区室谷一丁目5番4号

(業務分担)

試験責任者：澤 嗣郎 試験計画書作成、試験操作および報告書作成

試験従事者：藤田 修平 試験操作

試験物質：ブロムフェナクナトリウム

目的：プロナック点眼液は自社で開発した抗炎症剤であり、ヨーロッパへの導出を検討中である。しかし、現処方では保存効力が EP の基準に適合していないため、本研究では保存効力を強化するよう、保存剤、添加剤および pH の最適化を行う。

試験方法：

1) 安定性試験

下記のブロムフェナクナトリウム点眼液のチロキサポール配合量、塩化ベンザルコニウム配合量および pH を種々変えて、外観を観察する。濁らなかつた処方を無色ポリプロピレン容器に充填し、70℃、60℃、40℃75%RH に保存後、経時的にブロムフェナクナトリウム含量、pH、外観および不溶性異物について試験する。また、凍結融解を10回繰り返し、外観および不溶性異物を観察する。

<HPLC 条件>

検出器：紫外吸光光度計(測定波長：266nm)

カラム：Capcelpak AG-120

カラム温度：25℃付近の一定温度

移動相：リン酸二水素アンモニウム 1.98g を水 750mL に溶かし、リン酸を加えて pH7.3 に調整した後、アセトニトリル 250mL を混和する。

流速：ブロムフェナクナトリウムの溶出時間が約 18 分となるように調整する。

試料注入量：10μL

試験計画書の変更書 No.1

試験名: EP に適合するプロナック点眼液の処方設計

試験コード: P2002B131

変更日: 2003年02月28日

変更前:

試験操作終了予定日 : 2002年12月28日

試験終了予定日 : 2003年01月31日

変更後:


試験操作終了予定日 : 2003年04月25日

試験終了予定日 : 2003年05月30日

変更理由:

製剤化検討の結果、保存効力がヨーロッパ薬局方の A 基準に適合し、ポリプロピレン容器では安定な処方が設定できた。さらに、海外でよく用いられるポリエチレン容器での安定性を確認するため、試験期間の延長を行う。

試験責任者の署名 2003年02月28日 澤 嗣郎 

応用開発 GM の承認 2003年02月28日 大崎 聡 

保存効力試験記録書

試験期間： 02.07.03 ~ 02.08.07

試験コード： P2002B131

試験実施者： 藤田 修平 (印)

試験責任者： 澤 嗣司 (印)

試験物質： 7-β-D-エタナトリアム点眼液 Lot No. 02502-1

処方

- (100 mL 中) A-01
- 7-β-D-エタナトリアム 0.1g
- 砂糖 1.1g
- 砂糖 1.1g
- 塩化ベンゾルニウム 0.005g
- キシトール 0.02g
- ポピドン (ホリビニール) K30 2g
- イソトシナトリアム 0.02g
- 水酸化ナトリウム 適量
- 精製水 適量

結果

試験菌接種日、02年07月03日

菌数確認日	接種菌数	試験実施日												
		07103 (株)	07104 (株)	07108	07110	07112	07117	07124	07131	07108	07112			
S.aureus*1	2.1x10 ⁶	0	0	0	0	0	0	0	0	0	0	0	0	0
菌数確認日	07108	07108	07108	07112	07112	07122	07126	08102	07108	07112				
E.coll*1	6.5x10 ⁶	0	0	0	0	0	0	0	0	0	0	0	0	0
菌数確認日	07108	07108	07108	07112	07112	07122	07126	08102	07108	07112				
P.aeruginosa*1	5.8x10 ⁶	0	0	0	0	0	0	0	0	0	0	0	0	0
菌数確認日	07108	07108	07108	07112	07112	07122	07126	08102	07108	07112				
C.albicans*2	3.2x10 ⁵	0	0	0	0	0	0	0	0	0	0	0	0	0
菌数確認日	07110	07110	07110	07117	07117	07124	07131	08107	07110	07117				
A.niger*2	1.8x10 ⁵	0	0	0	0	0	0	0	0	0	0	0	0	0

unit : CFU/mL

使用機器： SANYO イキュバータ MIR-551 機器番号 400061 温度設定 30~35°C *1 試験菌培養機器

SANYO イキュバータ MIR-251 機器番号 300257 温度設定 20~25°C *2 試験菌培養機器

保存効力試験記録書

試験期間: 02.07.03 ~ 02.08.07

試験コード: P2002B131

試験実施者: 藤田 悠平

試験責任者: 澤 尚徳

試験物質: 7047エタクトリウム点眼液 Lot No. 02S021

処方

- (100 mL 中) A-02
- 7047エタクトリウム 0.1g
- 砂酸 1.1g
- 砂砂 1.1g
- 塩化ベンゾルニウム 0.005g
- 和村ポール 0.05g
- ポピドン (ポリビニルピリドン K30) 2g
- エト酸トリウム 0.02g
- 水酸化ナトリウム 適量
- 精製水 適量

結果

試験菌接種日 02年07月03日

菌数確認日	接種菌数	試験実施日					
		07/03/04	07/04/04	07/10	07/17	07/24	07/31
菌数確認日	07/08	07/08	07/12	07/12	07/12	07/12	07/12
<i>S.aureus</i> *1	2.1 × 10 ⁶	1.7 × 10 ⁵	2.0 × 10 ¹	0	0	0	0
菌数確認日	07/08	07/08	07/12	07/12	07/12	07/12	07/12
<i>E.coli</i> *1	6.5 × 10 ⁶	0	0	0	0	0	0
菌数確認日	07/08	07/08	07/12	07/12	07/12	07/12	07/12
<i>P.aeruginosa</i> *1	5.8 × 10 ⁶	0	0	0	0	0	0
菌数確認日	07/08	07/08	07/12	07/12	07/12	07/12	07/12
<i>C.albicans</i> *2	3.2 × 10 ⁵	1	1	0	0	0	0
菌数確認日	07/10	07/10	07/17	07/17	07/17	07/17	07/17
<i>A.niger</i> *2	1.8 × 10 ⁵	1	1	0	0	0	0

unit: CFU/mL

使用機器: SANYO インキュベータ MIR-551 機器番号 400061

温度設定 30~35°C *1 試験菌培養機器

SANYO インキュベータ MIR-251 機器番号 300257

温度設定 20~25°C *2 試験菌培養機器

試験物質名	AHR10282B	試験コード	P2002B/31	ロット番号	025021	調製年月日	2002年07月02日	試験実施者	澤 前郎
試験題目	保存効力試験用サンプリ								
製造量	無色77.15mL X	—	本	無色PP5mL X	—	本	025021	×	本
処方番号	A-01	A-02		A-03		A-04		A-05	
成分及び分量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g
成分名	100 mL	(g)	100 mL	(g)	100 mL	(g)	100 mL	(g)	100 mL
7-β-D-エプタトリン	0.1	0.10005	0.1	0.10001	0.1	0.10001	0.1	0.10001	0.1
ホウ酸	1.1	1.099	1.1	1.101	1.1	1.101	1.1	1.101	1.1
ホウ砂	1.1	1.100	1.1	1.100	1.1	1.100	1.1	1.100	1.1
塩化ベンザルコニウム	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005
4-12チサホル	0.02	0.02020	0.05	0.05019	0.05	0.05019	0.05	0.05019	0.05
ホモトロン (K-30)	2.0	2.1998	2.0	1.999	2.0	1.999	2.0	1.999	2.0
エテトリン	0.02	0.02007	0.02	0.02010	0.02	0.02010	0.02	0.02010	0.02
精製水	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5	8.5
PH	8.2	8.19	8.2	8.20	8.2	8.20	8.2	8.20	8.2

ガテフロキサシンとブロムフェナクナトリウムの相互作用
ブロムフェナクナトリウムの安定性

試験コード: P2002B131
試験実施者: 塚 嗣郎
試験実施日: 2002年07月05日

ID	Chromato No.	AM	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	AH2S051.C01	2295054	1378449	1.6550	0.10008						
A-01	Initial	AH2S051.C02	2274085	1375224	1.6536	0.09939	100.00	8.17	-	-	
A-02	Initial	AH2S051.C03	2269283	1358758	1.6701	0.10039	100.00	8.16	-	-	
A-03	Initial	AH2S051.C04	2269941	1365558	1.6623	0.09992	100.00	8.15	-	-	
A-04	Initial	AH2S051.C05	2263909	1357737	1.6674	0.10022	100.00	8.15	-	-	

試験物質名	AHR10282B	試験コード	P2002B131	ロット番号	025041	調製年月日	2002年07月04日	試験実施者	澤 嗣郎
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試験題目

製造量	無色77' 15mL×	本	無色PP5mL×	本	褐色PP5mL×	本	×	本	×	本	×	本
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処方番号	A-01		A-02		A-03		A-04				
成分及び分量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	仕込量 g	秤取量	製造メーカー
成分名	100 mL	(g)	100 mL	(g)	100 mL	(g)	100 mL	(g)	mL	(g)	ロット番号
7-β-ヒドロキシステロイド	0.1	0.10000	0.1	0.10000	0.1	0.10002	0.1	0.10000			OC 2647
ホウ酸	1.1	1.100	1.1	1.102	1.1	1.100	1.1	1.100			01E028
ホウ砂	1.1	1.100	1.1	1.099	1.1	1.102	1.1	1.100			00P027
塩化ベンザルコニウム	0.005	0.005	0.005	0.005	0.005	0.005	0.005	0.005			02B022
4-オキシサポール	0.02	0.02013	0.05	0.05019	0.03	0.03013	0.02	0.02008			E65703A07
ホセチン(K-30)	2.0	2.003	2.0	2.002	2.0	2.000	1.0	1.004			01R012
イソトロンナリウム	0.02	0.02003	0.02	0.02004	0.02	0.02003	0.02	0.02001			98N012A
精製水											
pH	8.2	8.15	8.2	8.15	8.2	8.14	8.2	8.14			

4-Jul-2002 17:37:41

001:	-	0.02013	g
002:	-	0.05019	g
003:	-	0.03013	g
004:	-	0.02008	g
005:	+	0.02003	g
006:	+	0.02004	g
007:	+	0.02003	g
008:	+	0.02001	g
009:	+	0.10000	g
010:	+	0.10000	g
011:	+	0.10002	g
012:	+	0.10000	g

4-Jul-2002 17:35:18

001:	+	1.100	g
002:	+	1.102	g
003:	+	1.100	g
004:	+	1.100	g
005:	+	2.003	g
006:	+	2.002	g
007:	+	2.000	g
008:	+	1.004	g
009:	+	1.100	g
010:	+	1.099	g
011:	+	1.102	g
012:	+	1.100	g

001/ 7/04 19:17
PH 8.15
ATC 26.5°C

001/ 7/04 19:19
PH 8.15
ATC 26.4°C

001/ 7/04 19:22
PH 8.14
ATC 26.2°C

002/ 7/04 19:24
PH 8.14
ATC 26.0°C

試験物質名: AHR10282B 試験コード: P7002B131 試験年月日: 2002年07月05日

試験項目: 試験実施者: 澤 嗣郎

STD1, IS: 02.07.04 調製品を用いた.

(AHR10282B 0.02502g + 移動相 → 25ml)

STD2 上液 2ml + IS 2ml + 移動相 → 20ml

IS (P-ヒト肝臓抽出液 X ALU 0.03320g + 移動相 → 100ml)

	pH	外觀
A-01 initial	8.17	—
A-02	8.16	—
A-03	8.15	—
A-04	8.15	—

'02/ 7/05 17:38
pH 8.17
ATC 25.5°C

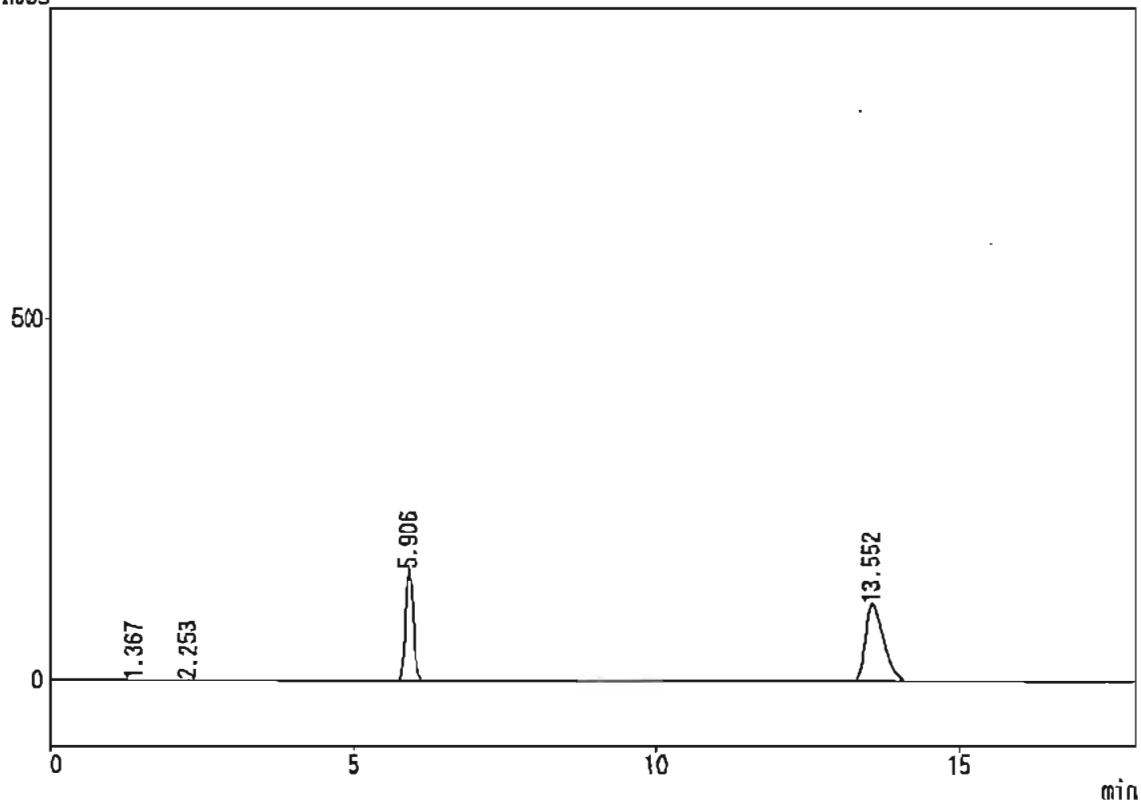
'02/ 7/05 17:40
pH 8.16
ATC 26.1°C

'02/ 7/05 17:41
pH 8.15
ATC 26.2°C

'02/ 7/05 17:42
pH 8.15
ATC 26.4°C

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレータ名 : 浅 嗣郎
 メソッド名 : !AHR1028.MET

*** カマクログ *** ファイル名:AH2S051.C01
 mAbs

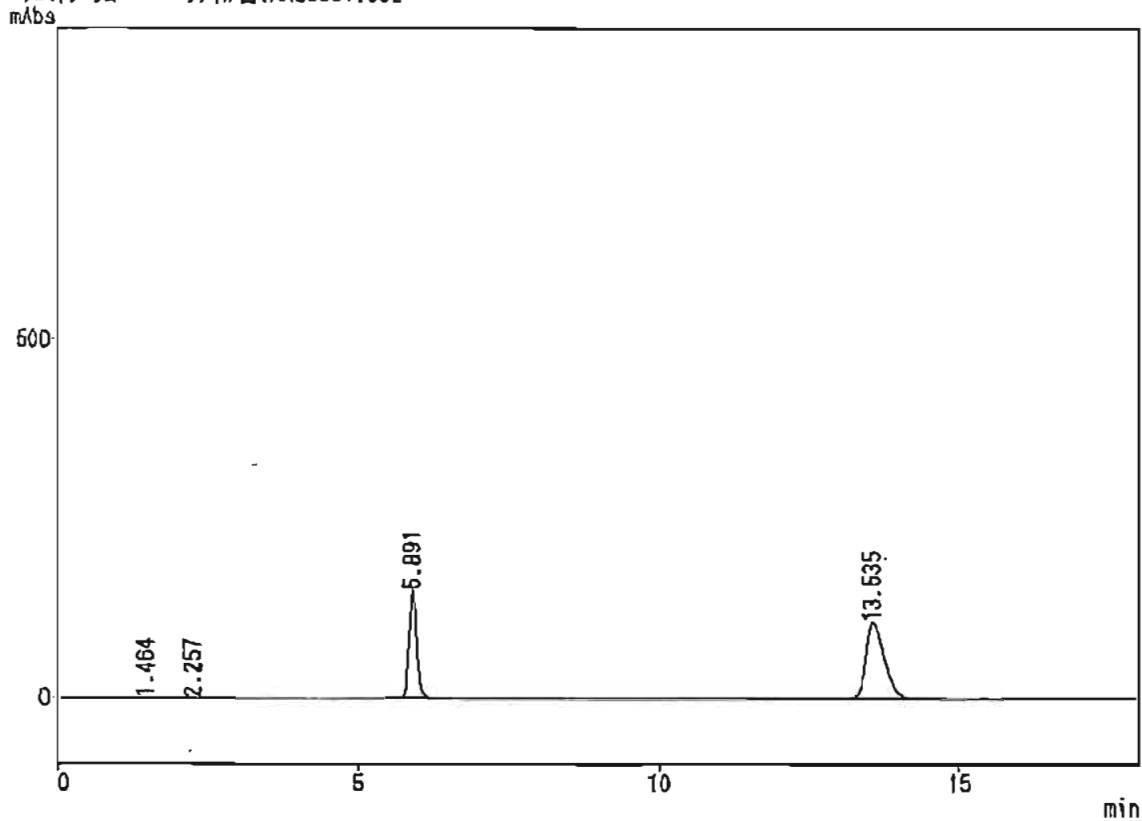


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.367	1222	104	V		0.0332	
2	2.253	1810	251			0.0492	
3	5.906	1378449	154339			37.4831	
4	13.552	2295054	108334	S		62.4244	
		3676536	263028			100.0000	

サンプル : A-01
 ID : Initial
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレータ名 : 滝 嗣郎
 メソッド名 : !AHR1028.MET

*** カロマトグラム *** ファイル名:AH2S051.002

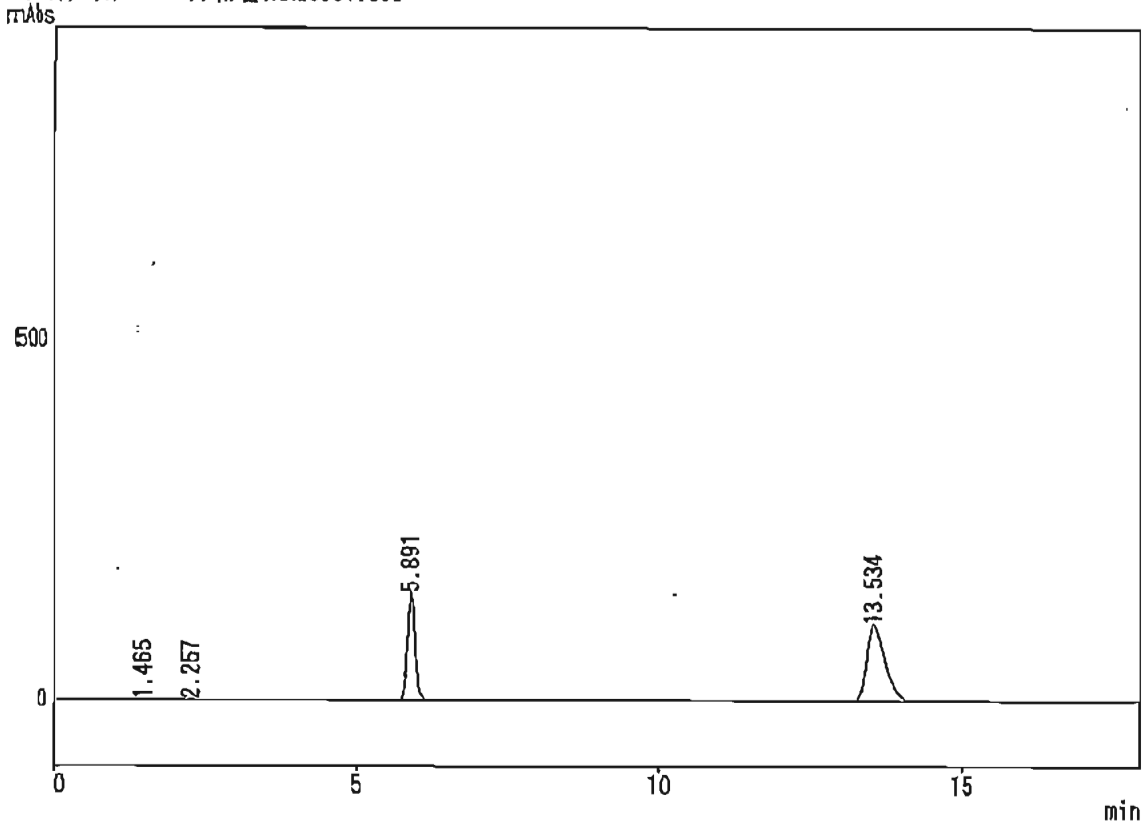


*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	MK	IONO	CONC	NAME
1	1.464	6178	664	V		0.1888	
2	2.257	3091	383	V		0.0845	
3	5.891	1375224	151679			37.6890	
4	13.535	2274085	106745			82.1578	
		3858577	269471			100.0000	

サンプル : A-02
 ID : Initial
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロリグラム *** ファイル名:AH2S051.C03

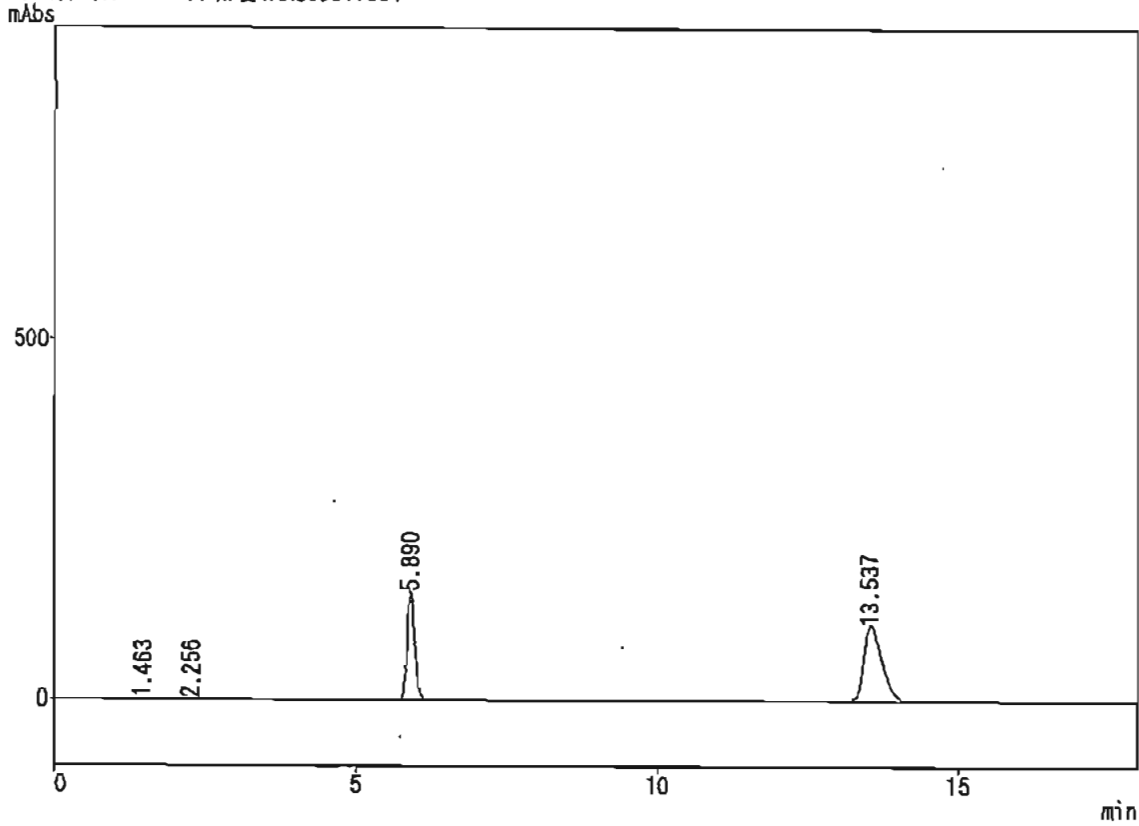


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.465	5868	668	V		0.1614	
2	2.257	2050	283	V		0.0564	
3	5.891	1358768	150061			37.3702	
4	13.534	2269283	106534			62.4121	
		3635969	257545			100.0000	

サンプル : A-03
 ID : Initial
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : IAHRT028.MET

*** カロマトグラム *** ファイル名:AH2S051.C04

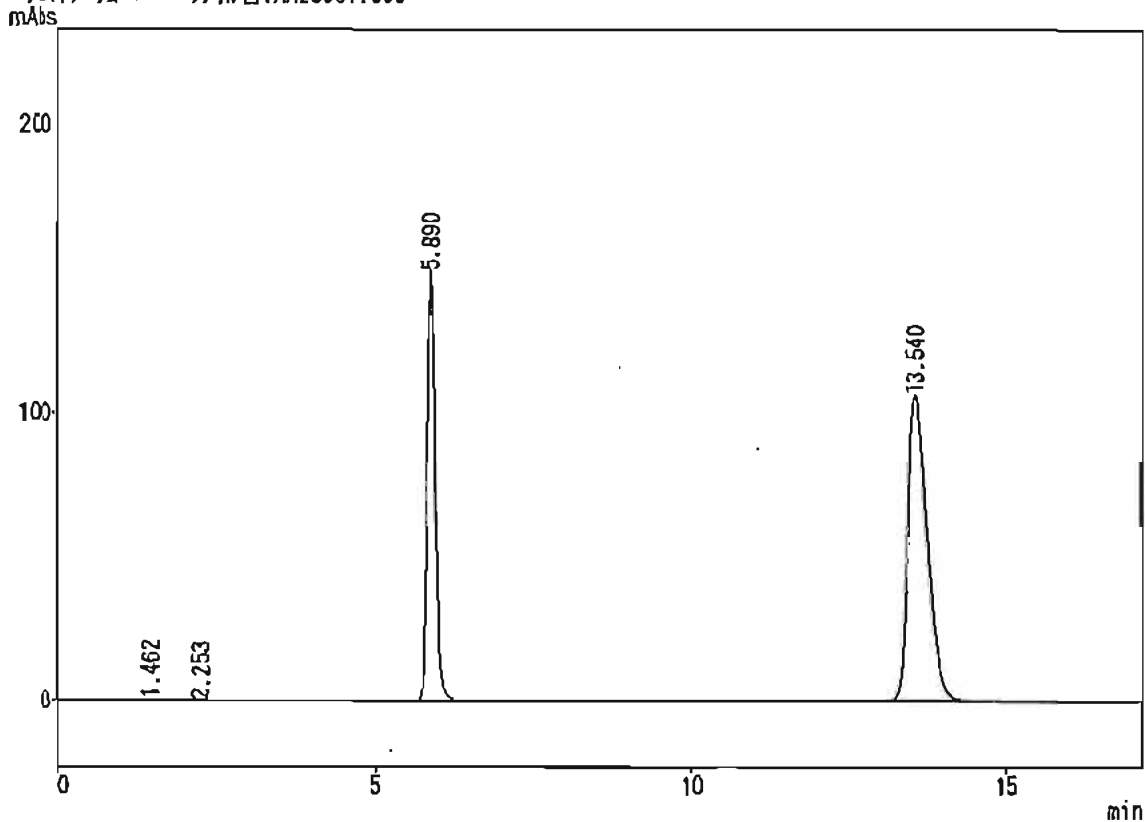


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.463	8461	707	V		0.1773	
2	2.256	1704	223	V		0.0468	
3	5.890	1365568	150905			37.4778	
4	13.537	2269941	106639			62.2982	
		3643673	258474			100.0000	

サンプル : A-04
 ID : Initial
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレーター名 : 澤 嗣郎
 プリント名 : IHR1028.MET

*** カマトグラム *** ファイル名:AH2S051.C05



*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.462	7533	767	V		0.2076	
2	2.253	1708	194	V		0.0470	
3	5.890	1357737	160024	S		37.3941	
4	13.540	2283909	106350			62.3514	
		3630886	257336			100.0000	

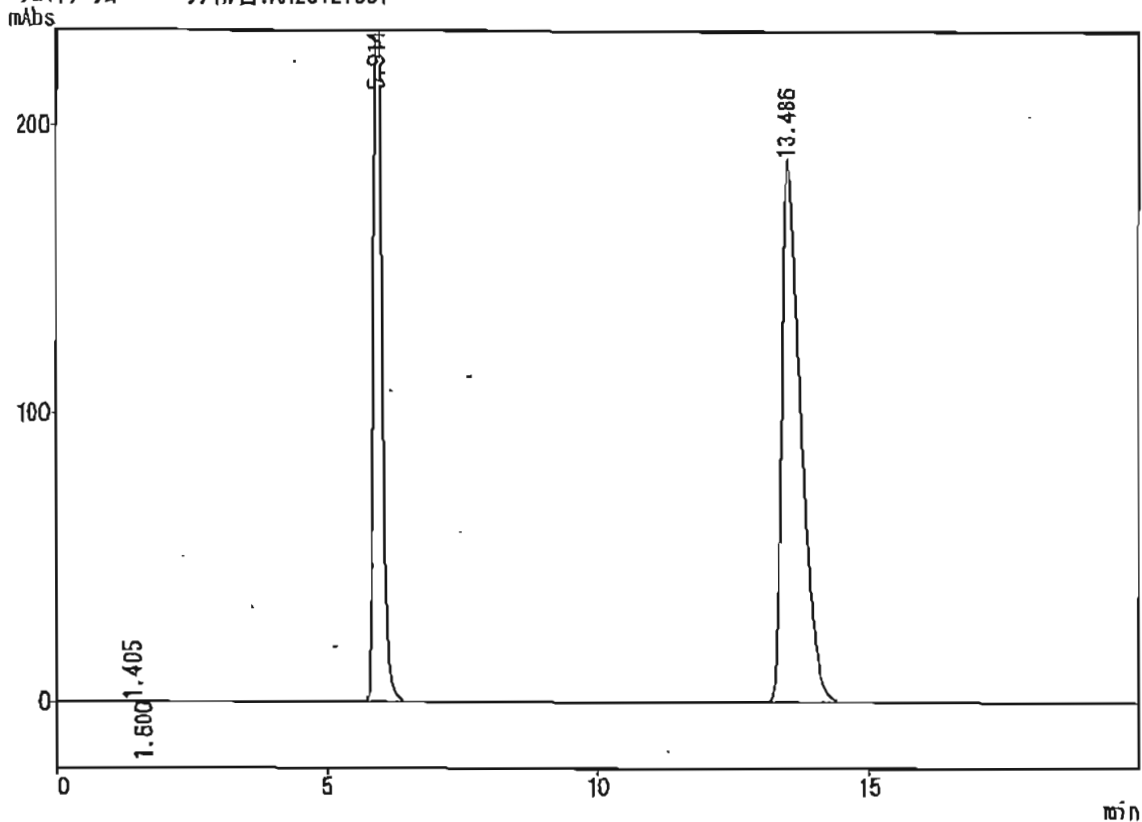
プロムフェナクナトリウムの安定性
Lot No.02SD51

試験コード:P2002B131
試験実施者: 澤 嗣郎
試験実施日:2002年07月12日

ID	Chromato No.	AM	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.L.M.	Turbid
STD	AH2S12.C01	4618881	2811272	1.6430						
STD	AH2S12.C10	4540394	2758913	1.6457						
STD	Mean			1.6444	0.10008					
A-01	70°C-1W AH2S12.C02	4593082	2811409	1.6337	0.09943	100.04	96.32	8.15	-	-
A-02	70°C-1W AH2S12.C03	4535814	2784696	1.6288	0.09913	98.74	95.20	8.16	-	-
A-03	70°C-1W AH2S12.C04	4555172	2797288	1.6284	0.09911	99.19	95.64	8.16	-	-
A-04	70°C-1W AH2S12.C05	4621523	2784880	1.6595	0.10100	100.78	97.27	8.16	-	-
A-01	60°C-1W AH2S12.C06	4492682	2763499	1.6257	0.09894	99.55	98.10	8.16	-	-
A-02	60°C-1W AH2S12.C07	4475578	2762011	1.6204	0.09862	98.24	96.66	8.17	-	-
A-03	60°C-1W AH2S12.C08	4480284	2765521	1.6201	0.09860	98.68	97.27	8.17	-	-
A-04	60°C-1W AH2S12.C09	4540394	2758913	1.6457	0.10016	99.94	98.48	8.17	-	-

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 サンプル名 : IAHRI02B.MET

*** 検出グラフ *** ファイル名:AH2S12.C01

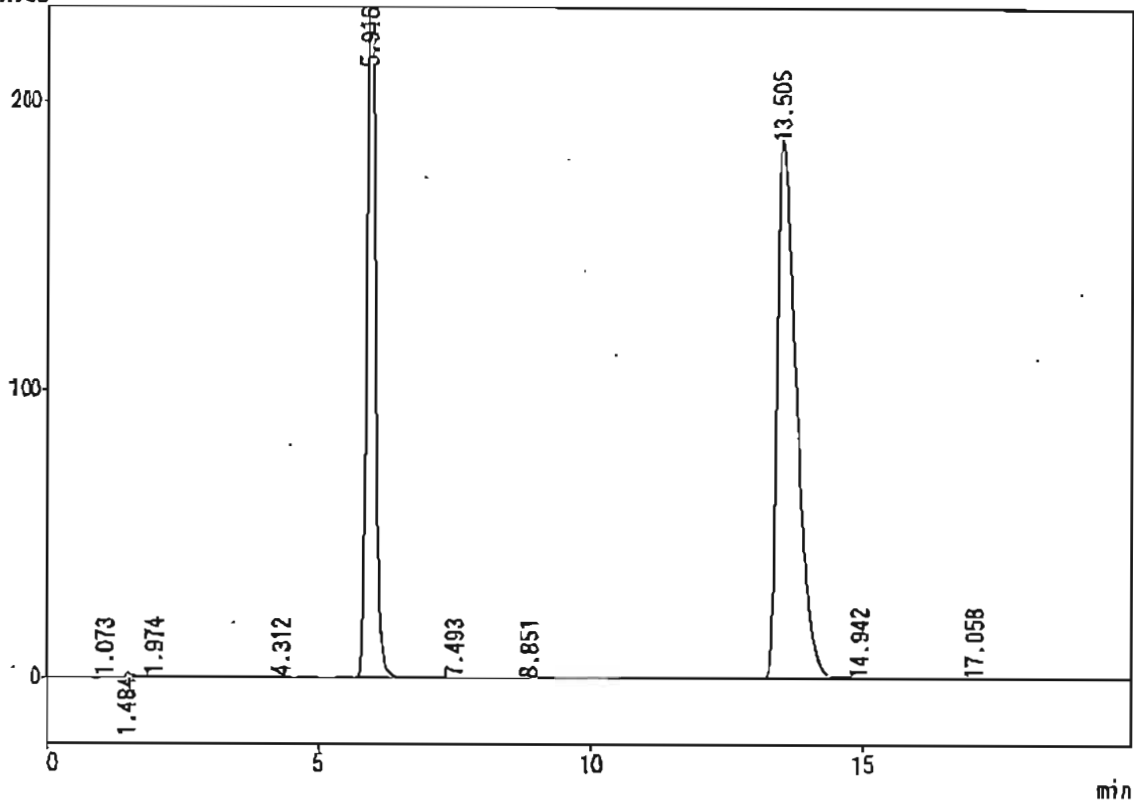


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.405	1859	149	V		0.0250	
2	1.600	2261	229	V		0.0304	
3	5.914	2811272	277954			37.8150	
4	13.486	4518881	188637			62.1296	
		7434273	466969			100.0000	

サンプル : A-01
 ID : 70°C-1W
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 サンプル名 : 漆 副郎
 サンプル名 : !AHR1028.MET

*** カマトラム *** ファイル名:AH2S12.C02
 mAbs

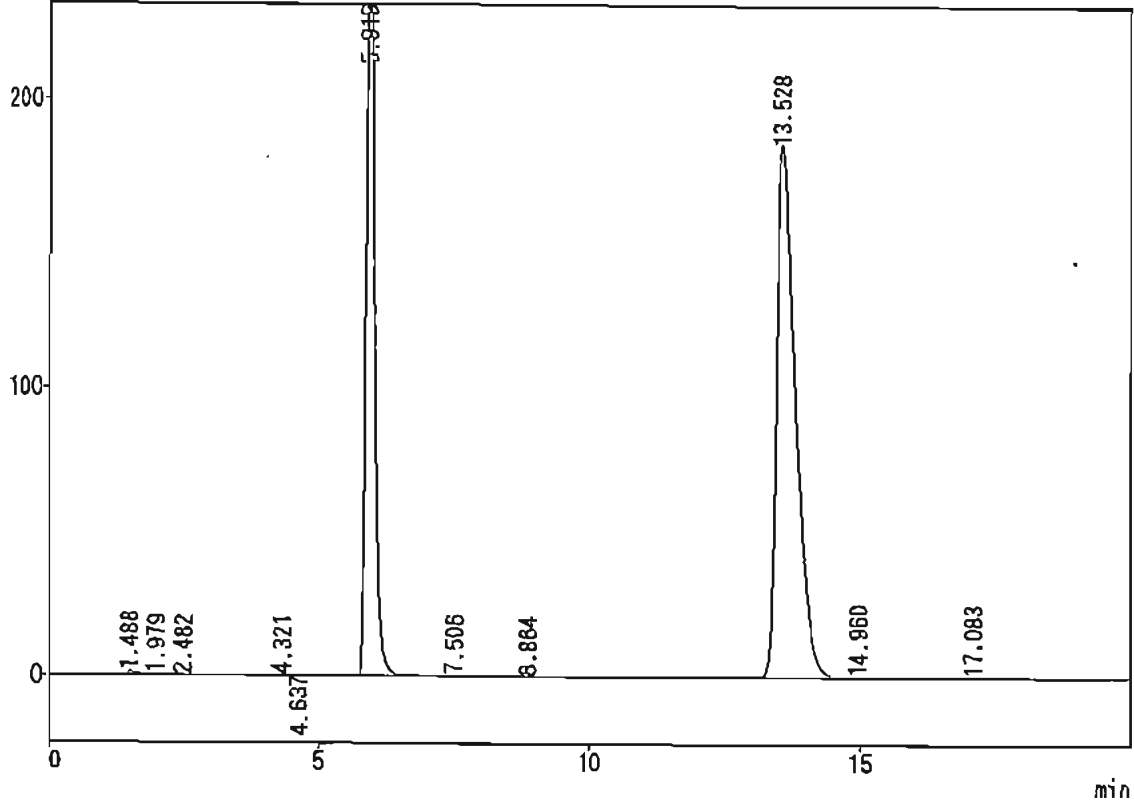


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.073	1466	177			0.0197	
2	1.484	16768	1481	V		0.2251	
3	1.974	4426	216	V		0.0594	
4	4.312	2063	242			0.0277	
5	5.916	2811409	278271	S		37.7584	
6	7.493	4002	325	T		0.0537	
7	8.851	1084	78			0.0146	
8	13.505	4593092	187161	S		61.6872	
9	14.942	2539	146	T		0.0341	
10	17.058	8945	355			0.1201	
		7445783	468452			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=4 テータ=AH2S12.003 02/07/12 13:13:32
 サンプル : A-02
 ID : 70°C-1W
 タイプ : 未知試料
 検出器 : SPD-10A シグナル
 オペレータ名 : 澤 嗣郎
 サンプル名 : IAHRT028.MET

*** カマトグラム *** ファイル名:AH2S12.C03
 mAbs



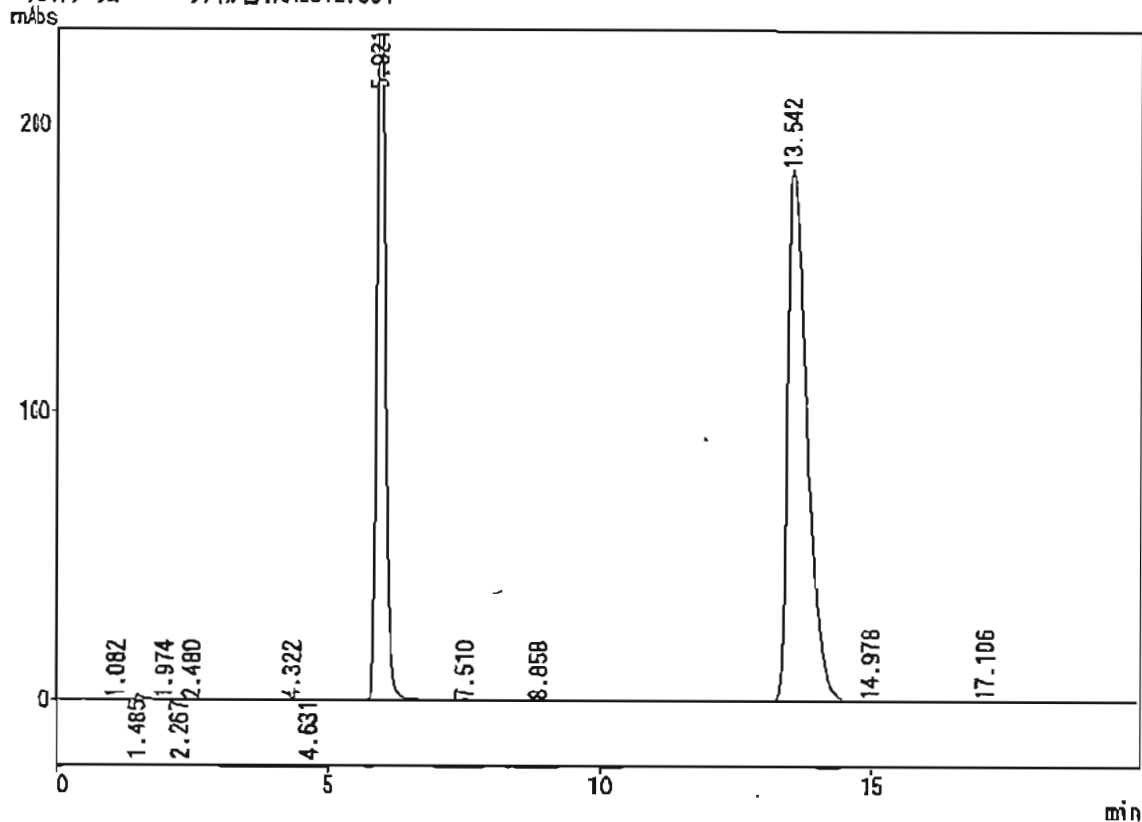
*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.488	14720	1319	V		0.2000	
2	1.979	3811	212	V		0.0491	
3	2.482	1023	92	V		0.0139	
4	4.321	2268	238			0.0308	
5	4.637	1092	84	V		0.0148	
6	5.919	2784696	276050	S		37.8364	
7	7.506	4073	333	T		0.0553	
8	8.864	1095	75			0.0149	
9	13.528	4535814	185022	S		61.6292	
10	14.960	2391	141	T		0.0325	
11	17.083	9081	368			0.1231	
		7359843	463933			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=5 テーマ=AH2S12.D04 02/07/12 13:33:38

サンプル : A-03
 ID : 70°C-1W
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カリブレーション *** ファイル名:AH2S12.C04



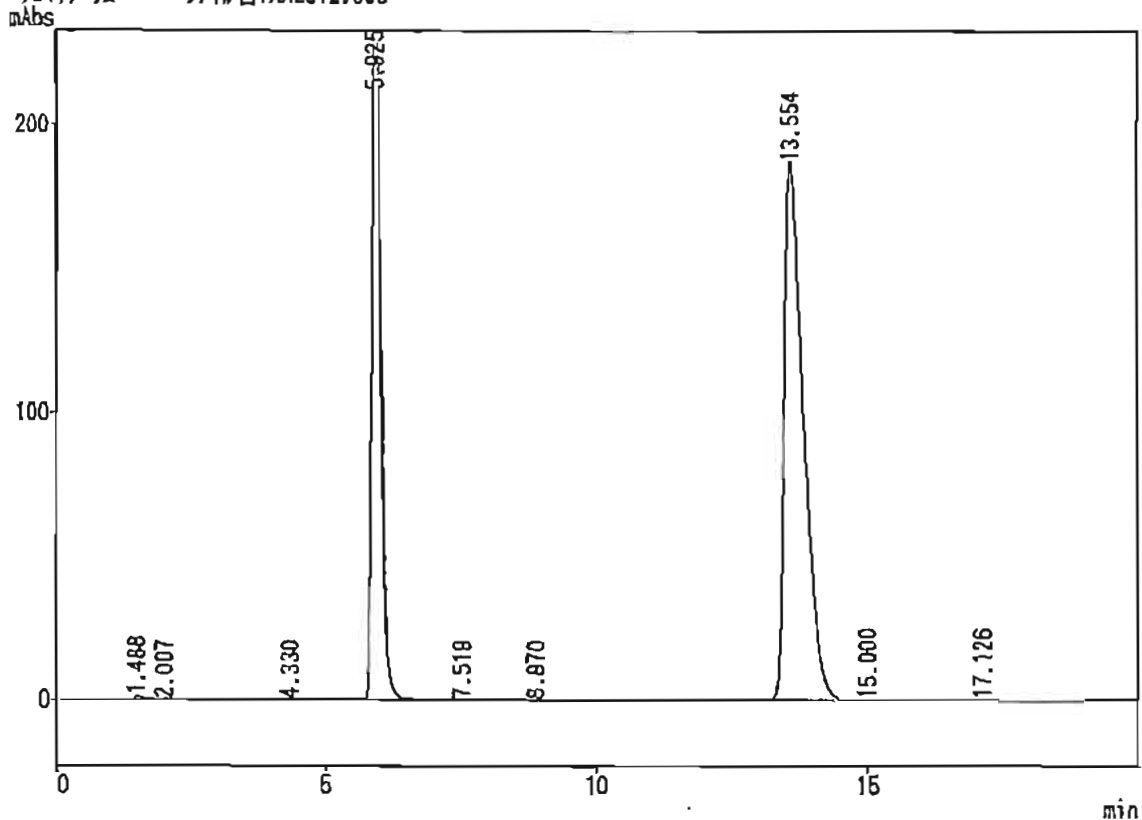
*** ヒートレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.082	1198	138	V		0.0182	
2	1.485	16951	1483	V		0.2291	
3	1.974	3712	222	V		0.0502	
4	2.267	1155	130	V		0.0156	
5	2.480	1704	117	V		0.0230	
6	4.322	2334	254			0.0316	
7	4.631	1214	85	V		0.0164	
8	5.921	2797288	277875	SV		37.8146	
9	7.510	3857	317	T		0.0521	
10	8.858	1245	91			0.0168	
11	13.542	4555172	185511	S		61.5783	
12	14.978	2782	160	T		0.0376	
13	17.108	8760	356			0.1184	

7397371 466737 100.0000

サンプル : A-04
 ID : 70°C-1W
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カリブレーション *** ファイル名:AH2S12.C05



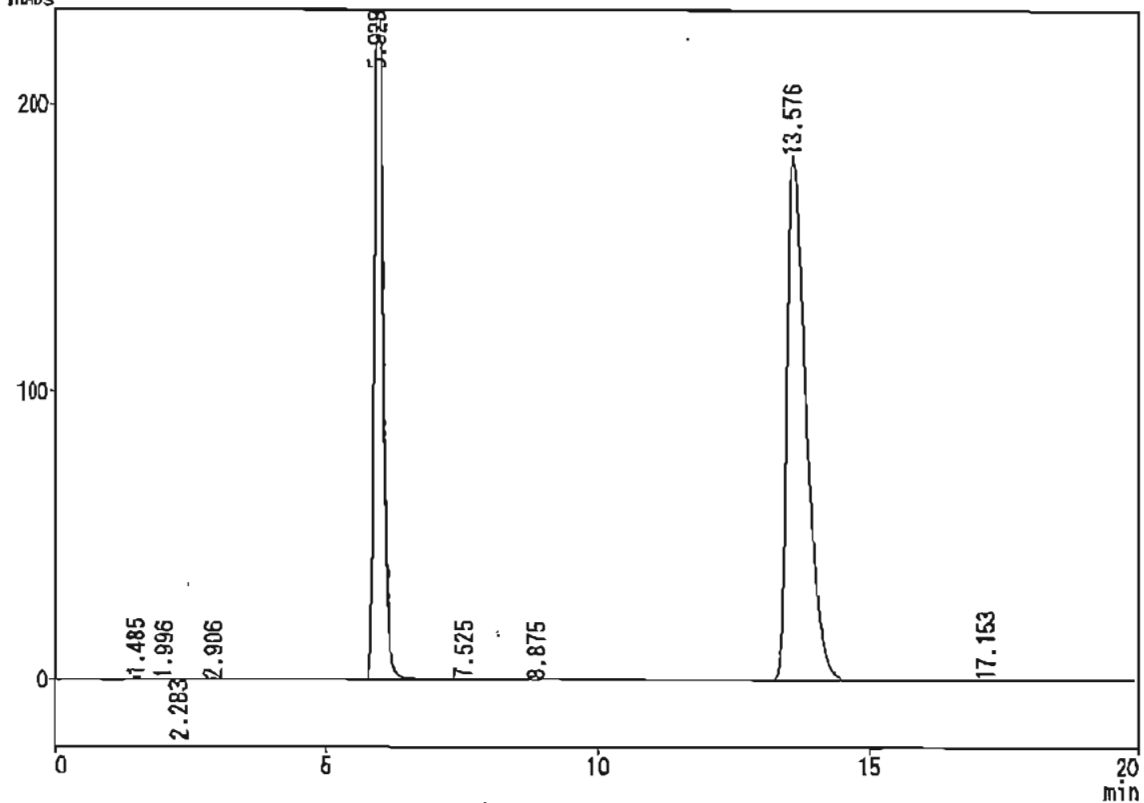
*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.488	13817	1274	V		0.1857	
2	2.007	1828	103	V		0.0246	
3	4.330	1887	239			0.0254	
4	5.925	2784880	276061	S		37.4330	
5	7.518	1583	128	T		0.0213	
6	8.870	1631	105			0.0208	
7	13.554	4621523	187705	S		62.1202	
8	15.000	4964	281	T		0.0667	
9	17.126	7633	314			0.1026	
		7439645	468191			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 検体番号=7 テキスト=AH2S12.D06 02/07/12 14:13:50

サンプル名 : A-01
 ID : 60°C-1W
 タイプ : 未知試料
 検出器 : SPD-10A シンチレーション
 オペレーター名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カマトグラム *** ファイル名:AH2S12.C06
 mAbs

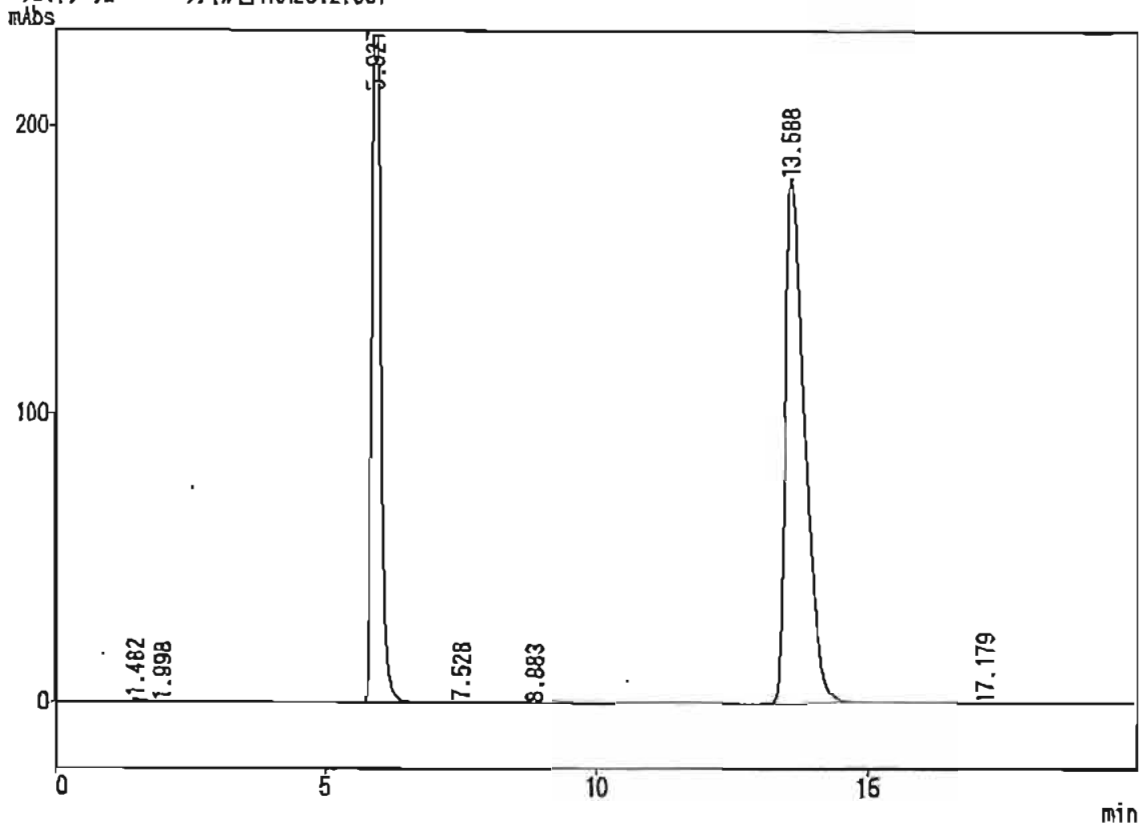


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.485	13490	1165	V		0.1851	
2	1.996	2340	183	V		0.0321	
3	2.283	1708	111	V		0.0234	
4	2.906	1316	117	V		0.0181	
5	5.928	2763499	274808			37.9275	
6	7.525	4030	317			0.0553	
7	8.875	1984	139			0.0272	
8	13.576	4492682	183211			61.6596	
9	17.153	5218	214			0.0716	
		7288267	460245			100.0000	

シフト名 : A-02
 ID : 60°C-1W
 タイプ : 未知試料
 検出器 : SPD-10A シフト名
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カマトグラム *** ファイル名:AH2S12.C07



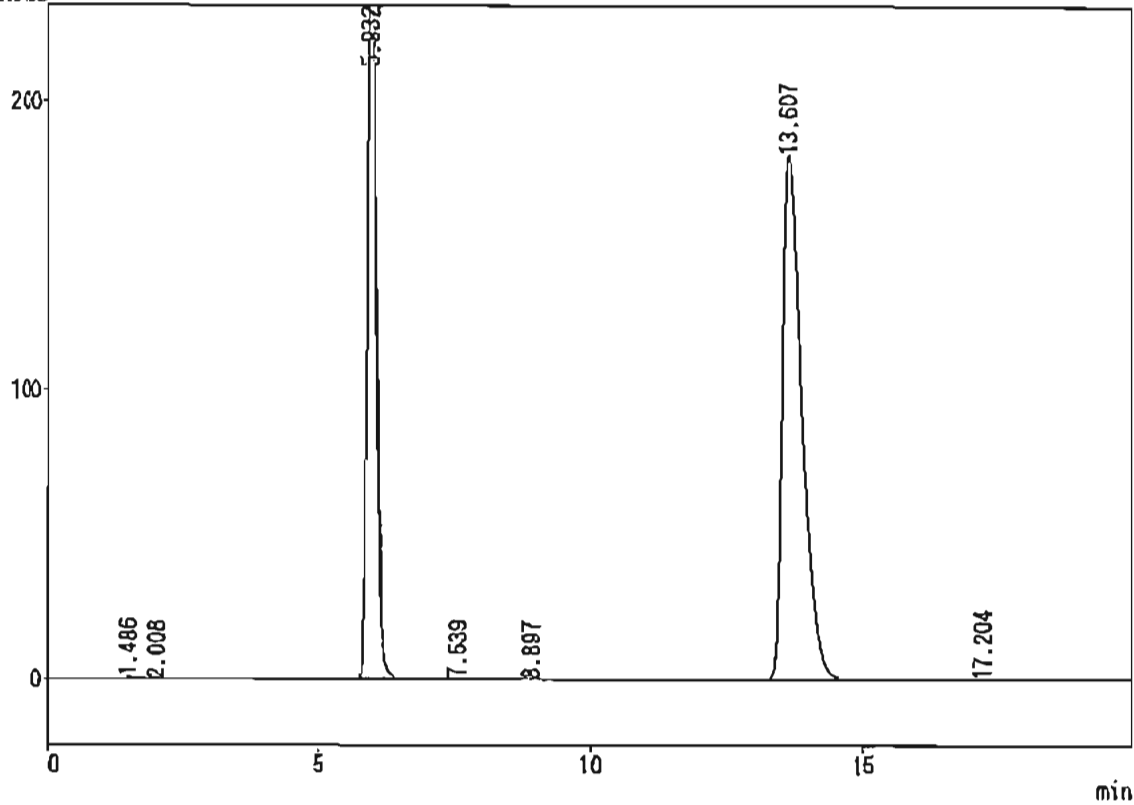
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	13709	1259	V		0.1887	
2	1.998	2000	124	V		0.0275	
3	5.927	2762011	274729			38.0170	
4	7.528	4308	339			0.0593	
6	8.883	2130	148			0.0293	
6	13.588	4475578	182534			61.6030	
7	17.179	5462	221			0.0752	
		7265198	459354			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=9 データ=AH2S12.D08 02/07/12 14:54:02

サンプル : A-03
ID : 50°C-1W
タイプ : 未知試料
検出器 : SPD-10A シンクル
オペレーター名 : 澤 嗣郎
メソッド名 : !AHR1028.MET

*** カマトグラム *** ファイル名:AH2S12.C08
mAbs



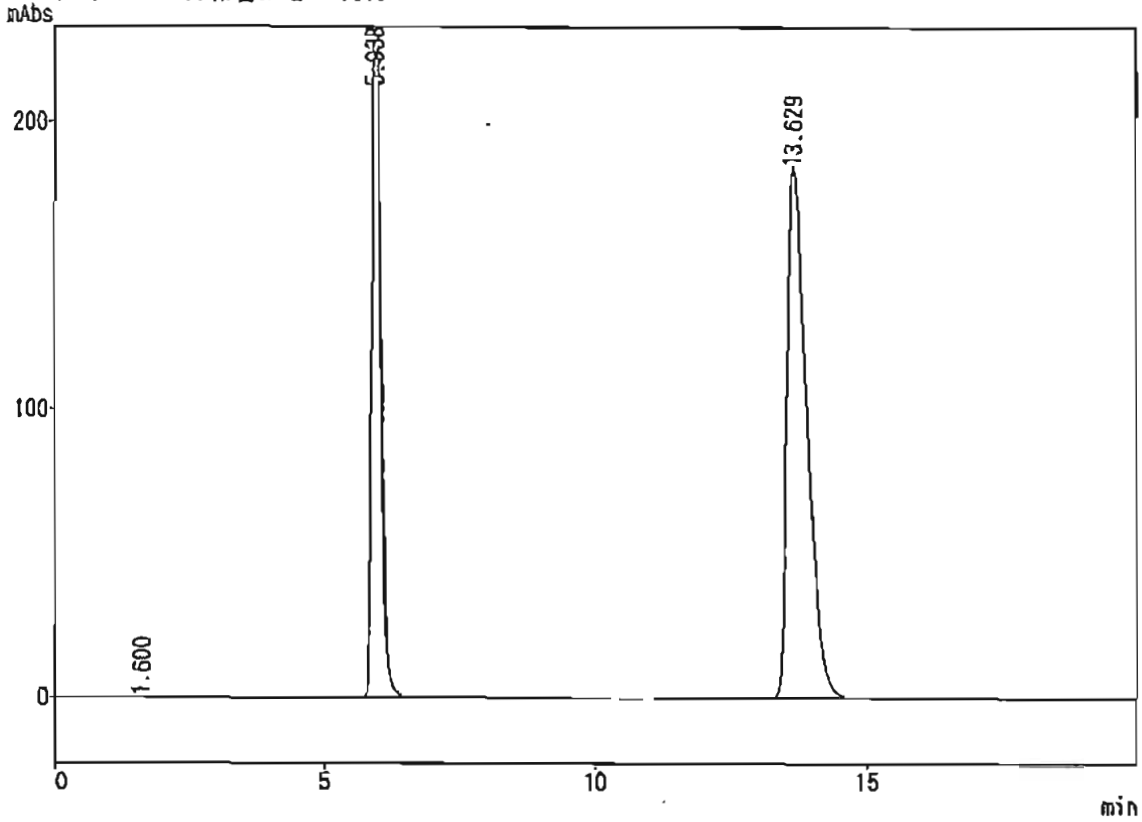
*** レポート ***

PKNO	TIME	AREA	HEIGHT	MX	IDNO	CONC	NAME
1	1.486	11360	973	V		0.1563	
2	2.008	2312	130	V		0.0318	
3	5.932	2765521	274583			38.0385	
4	7.539	3910	309			0.0538	
5	8.897	2214	156			0.0304	
6	13.607	4480284	182403			61.6244	
7	17.204	4710	197			0.0648	

7270311 458751 100.0000

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 検体番号=11 データ=AH2S12.D10 02/07/12 15:34:14
 サンプル名 : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロマトグラム *** ファイル名:AH2S12.C10



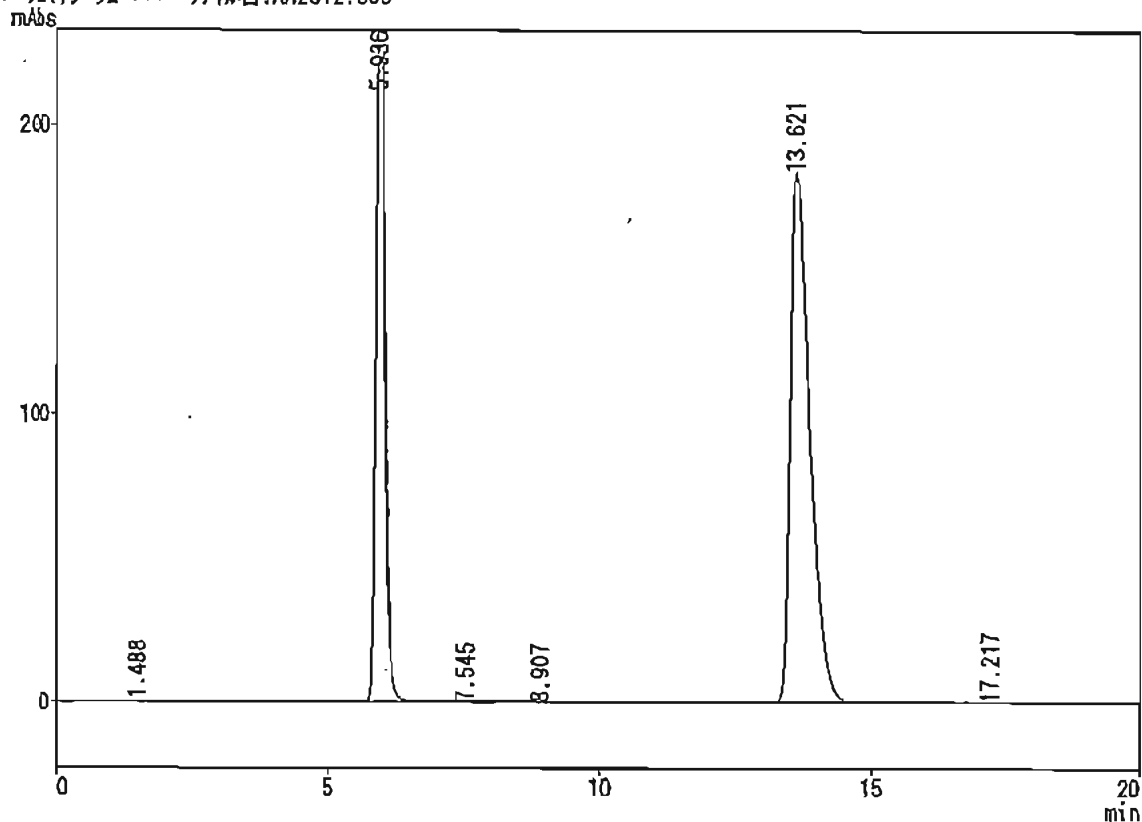
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.600	6243	312	V		0.0716	
2	5.938	2782336	273550			37.7314	
3	13.629	4553477	184896			62.1970	
		7321056	458768			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=10 データ=AH2S12.D09 02/07/12 15:14:08

サンプル : A-04
ID : 60°C-1W
タイプ : 未知試料
検出器 : SPD-10A シンクル
オペレーター名 : 澤 嗣郎
メソッド名 : !AHR1028.MET

*** 加算クロム *** ファイル名:AH2S12.C09



*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.488	12318	1081	SV		0.1683	
2	5.936	2758913	274327			37.6900	
3	7.545	1528	120			0.0209	
4	8.907	2378	163			0.0325	
5	13.621	4540394	184383			62.0270	
6	17.217	4493	187			0.0614	

7320023 460260 100.0000

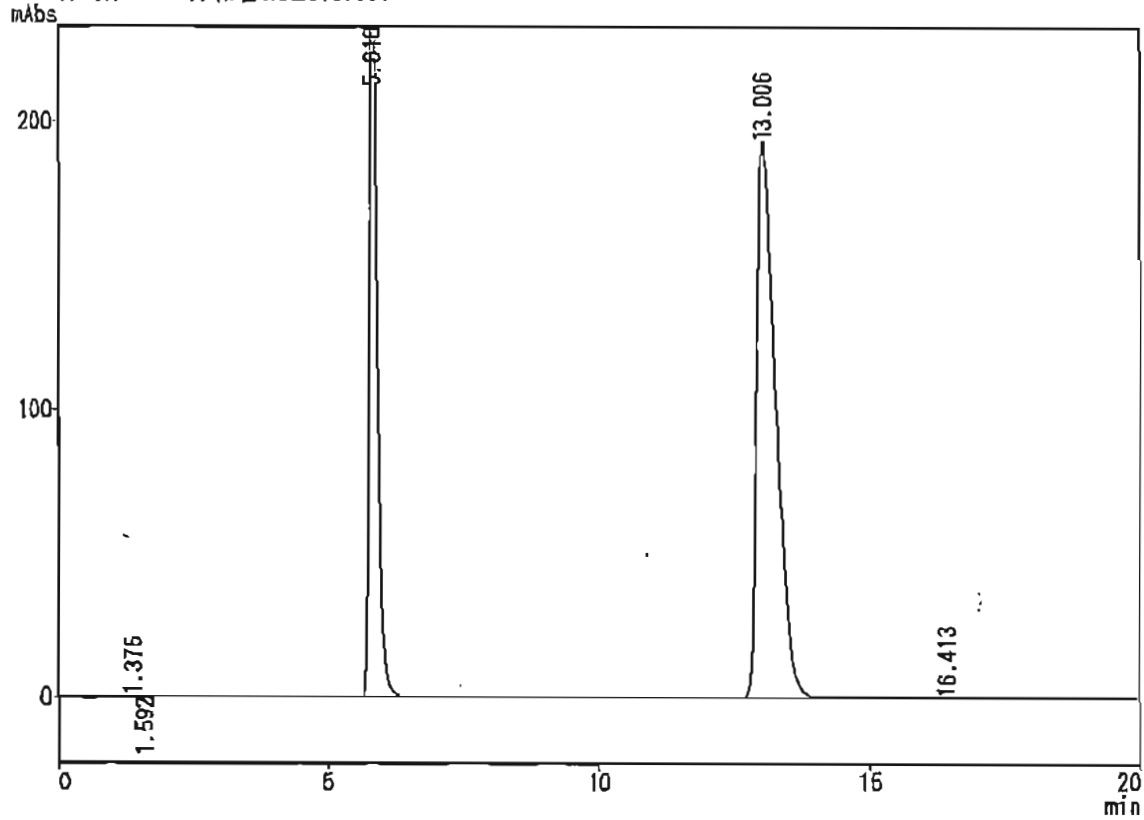
プロムフェナクナトリウムの安定性
Lot No:02S051

試験コード:P2002B131
試験実施者: 湯 原 郎
試験実施日:2002年07月19日

ID	Chromato No.	AM	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.L.M.	Turbid	Permeation (%)
STD	AH2S19.C01	4590898	2771294	1.6586							
STD	AH2S19.C10	4541555	2747359	1.6531							
STD	Mean			1.6549	0.10010						
A-01	70°C-2W AH2S19.C02	4601736	2756631	1.6693	0.10097	101.59	94.31	8.17	-	-	7.17
A-02	70°C-2W AH2S19.C03	4556591	2750687	1.6565	0.10020	99.81	93.00	8.18	-	-	6.82
A-03	70°C-2W AH2S19.C04	4588584	2761593	1.6616	0.10051	100.59	93.29	8.16	-	-	7.26
A-04	70°C-2W AH2S19.C05	4677976	2747028	1.7029	0.10300	102.77	95.22	8.15	-	-	7.35
A-01	60°C-2W AH2S19.C06	4487171	2741725	1.6366	0.09899	99.60	96.47	8.17	-	-	3.14
A-02	60°C-2W AH2S19.C07	4479387	2744786	1.6320	0.09871	98.33	95.25	8.16	-	-	3.13
A-03	60°C-2W AH2S19.C08	4477249	2746480	1.6302	0.09861	98.69	95.79	8.16	-	-	2.94
A-04	60°C-2W AH2S19.C09	4566943	2732765	1.6712	0.10109	100.87	97.66	8.16	-	-	3.18

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : IASHR102B.MET

*** カログララム *** ファイル名:AH2S19.C01



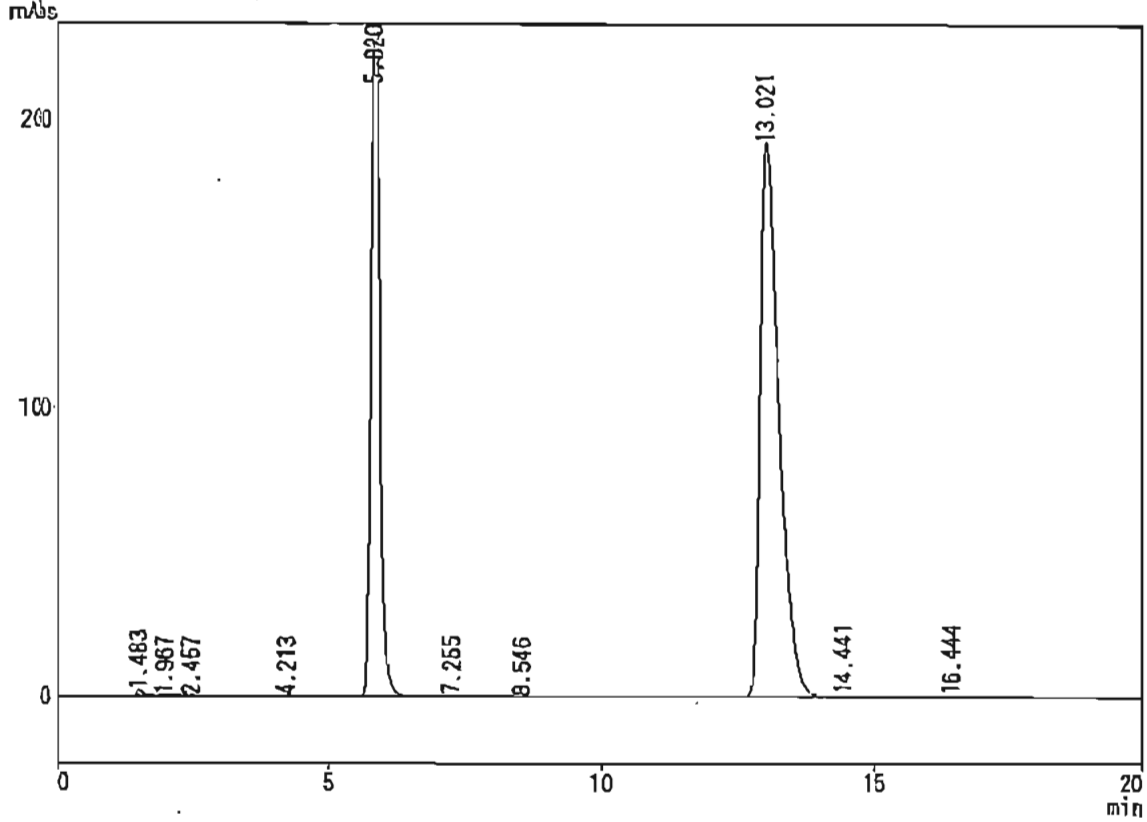
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.375	4314	398	V		0.0585	
2	1.592	2740	230	V		0.0372	
3	5.818	2771294	278151			37.6007	
4	13.006	4590898	193329			62.2890	
5	16.413	1077	55			0.0146	
		7370323	472163			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 ピーク番号=3 データ=AH2S19.D02 02/07/19 15:17:00

サンプル : A-01
ID : 70°C-2W
タイプ : 未知試料
検出器 : SPD-10A シンクル
オペレータ名 : 滝 嗣郎
メソッド名 : !AHR1028.MET

*** カマフラム *** ファイル名:AH2S19.C02



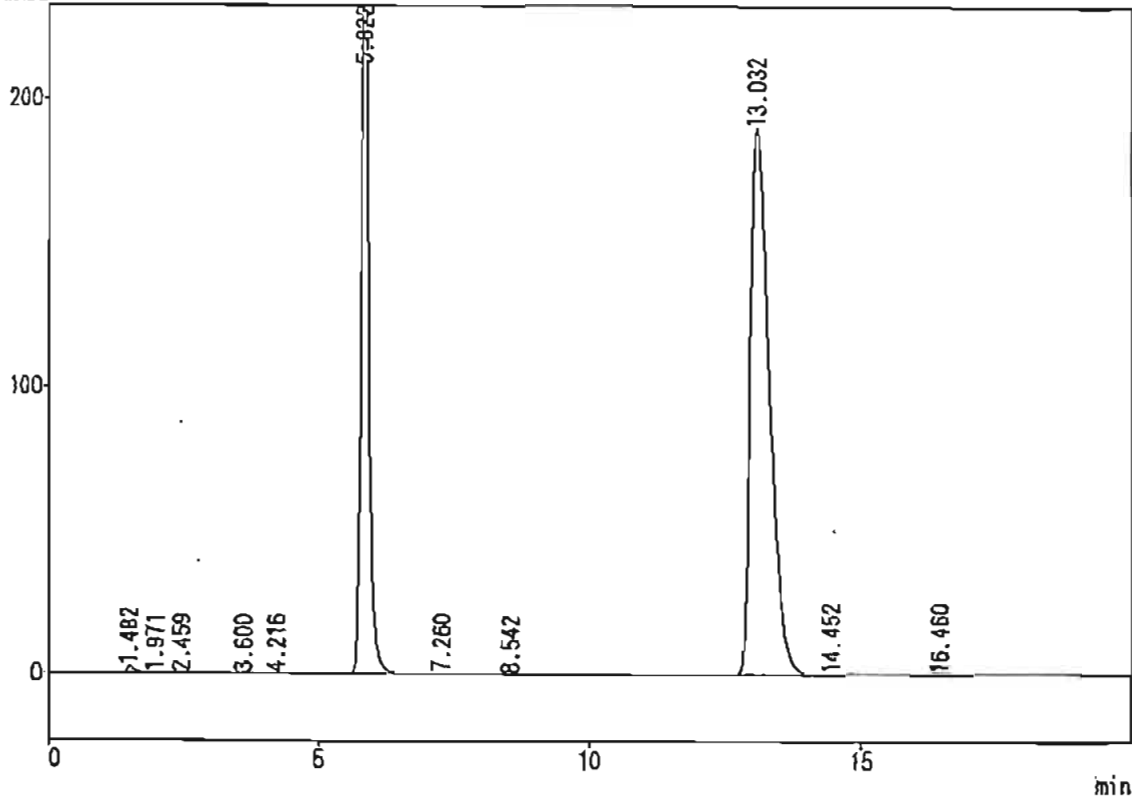
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MX	IDNO	CONC	NAME
1	1.483	26298	2357	V		0.3406	
2	1.967	6279	324	V		0.0845	
3	2.457	1722	158	V		0.0232	
4	4.213	3119	372			0.0420	
5	6.820	2756831	278295	S		37.1160	
6	7.255	5747	472	T		0.0774	
7	8.546	1140	75			0.0153	
8	13.021	4601736	192331	S		61.9590	
9	14.441	8282	454	T		0.1115	
10	16.444	17113	712			0.2304	

7427065 473548 100.0000

サンプル : A-02
 ID : 70°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレーター名 : 澤 嗣郎
 メソッド名 : IADR1028.MET

*** カリブレーション *** ファイル名:AH2S19.C03
 mAbs



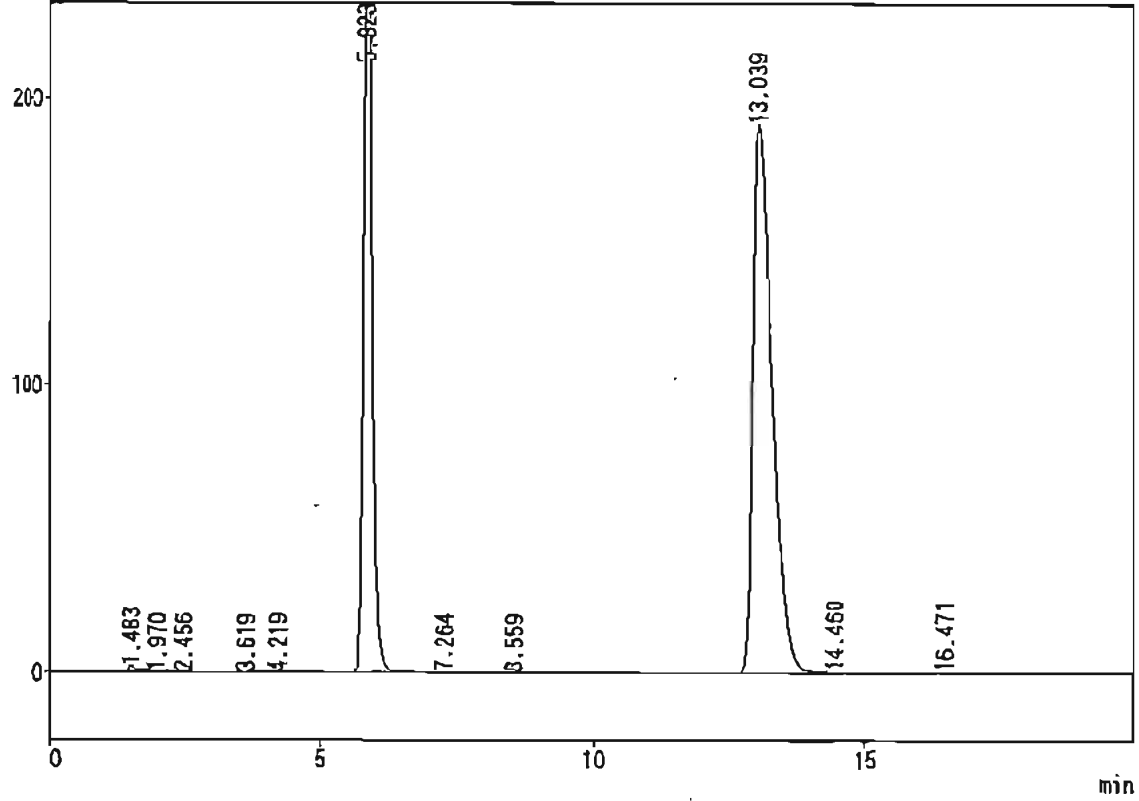
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	26318	2456	V		0.3567	
2	1.971	6439	335	V		0.0873	
3	2.459	1657	155	V		0.0225	
4	3.600	1804	70			0.0245	
5	4.216	2583	333			0.0350	
6	5.822	2750687	275772	S		37.2816	
7	7.260	6074	504	T		0.0823	
8	8.542	1264	88			0.0171	
9	13.032	4556591	190768	S		61.7580	
10	14.452	8073	439	T		0.1094	
11	16.460	16647	709			0.2256	
		7378138	471629			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 ポート番号=5 データ=AH2S19.D04 02/07/19 15:57:12

サンプル : A-03
 ID : 70°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : ICHR102B.MET

*** カリブレーション *** ファイル名:AH2S19.C04
 mAbs



*** ピークレポート ***

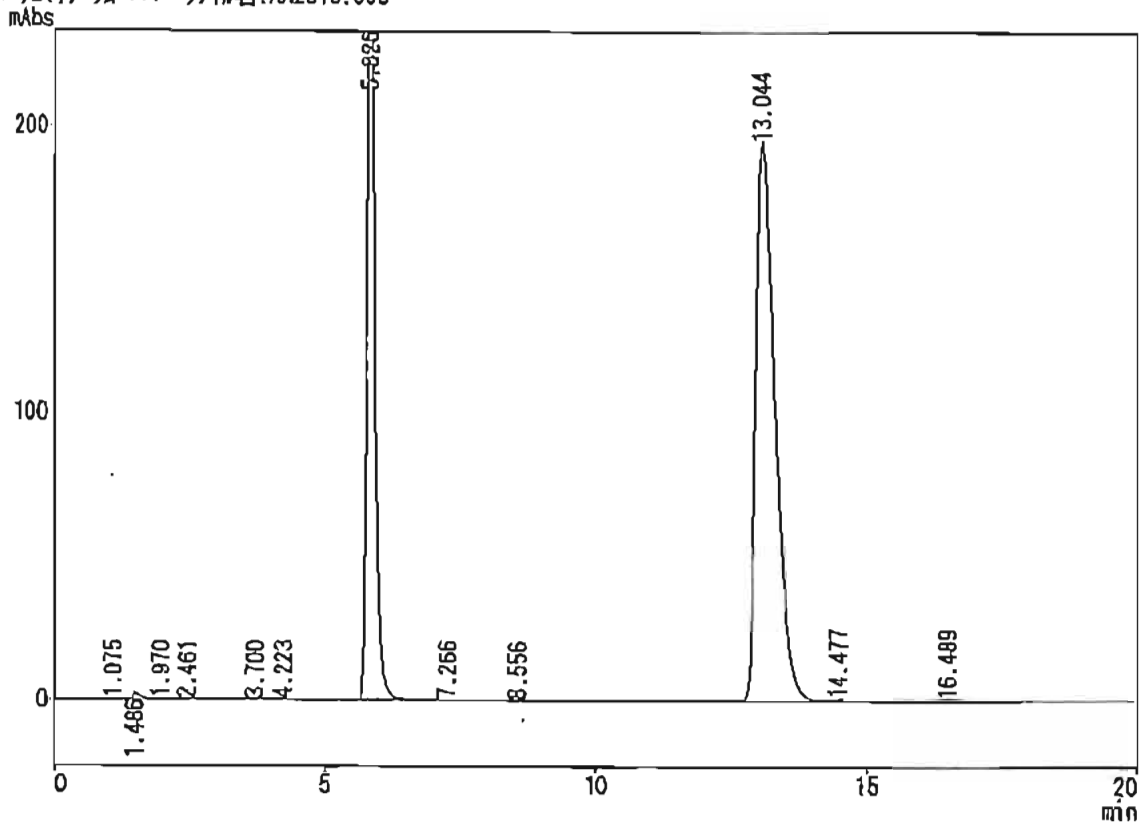
PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.483	24748	2279	V		0.3336	
2	1.970	6361	328	V		0.0857	
3	2.456	1816	155	V		0.0218	
4	3.619	1453	70	V		0.0196	
5	4.219	2854	344			0.0358	
6	5.823	2761593	276799	S		37.2209	
7	7.264	5734	471	T		0.0773	
8	8.559	1420	99			0.0191	
9	13.039	4588584	191739	S		61.8452	
10	14.460	8359	459	T		0.1127	
11	16.471	16941	710			0.2283	

7419464 473452 100.0000

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=6 データ=AH2S19.D05 02/07/19 16:17:20

サンプル : A-04
 ID : 70°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロマトグラム *** ファイル名:AH2S19.C05

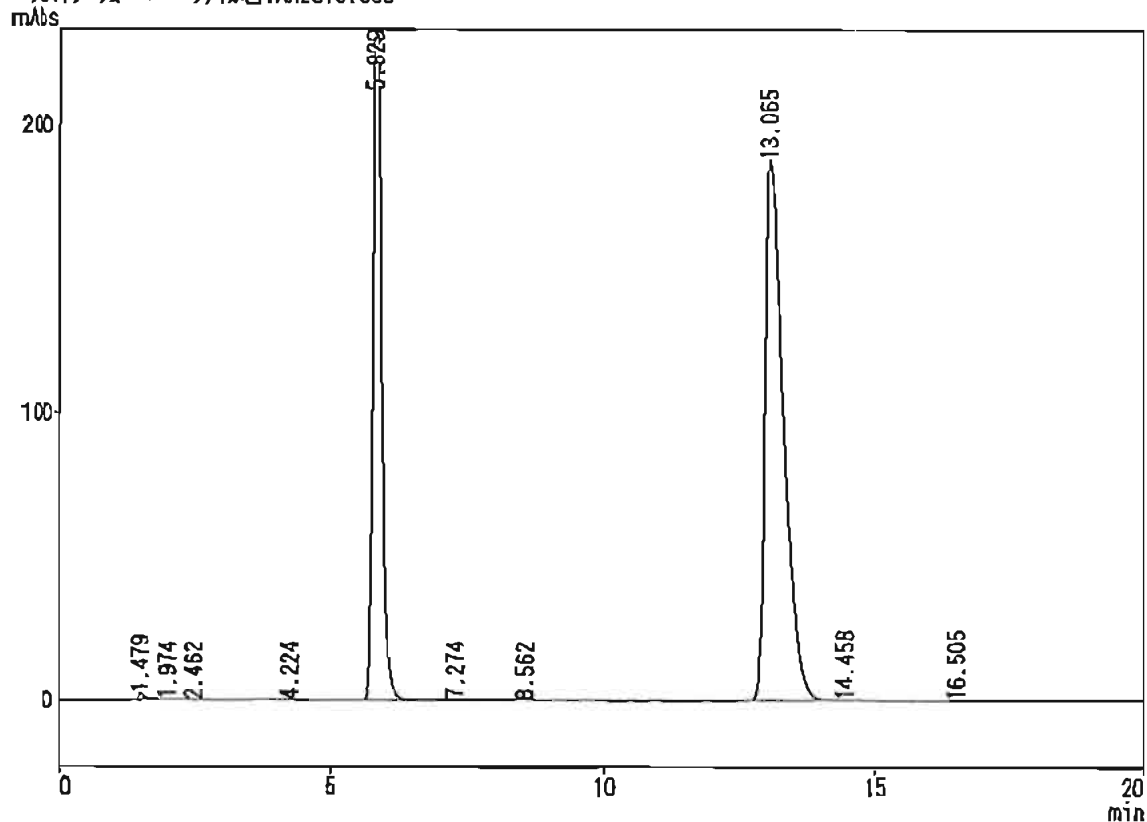


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.075	1514	184			0.0202	
2	1.486	22820	2141	V		0.3045	
3	1.970	4598	211	V		0.0614	
4	2.461	1823	150	V		0.0243	
5	3.700	1404	67	V		0.0187	
6	4.223	2997	379			0.0400	
7	5.826	2747028	275579	S		36.6536	
8	7.266	2468	205	T		0.0329	
9	8.556	1275	101			0.0170	
10	13.044	4677978	194977	S		62.4182	
11	14.477	13740	703	T		0.1833	
12	16.489	16930	698			0.2259	
		7494575	475395			100.0000	

サンプル : A-01
 ID : 60°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** 加算プログラム *** ファイル名:AH2S19.C06



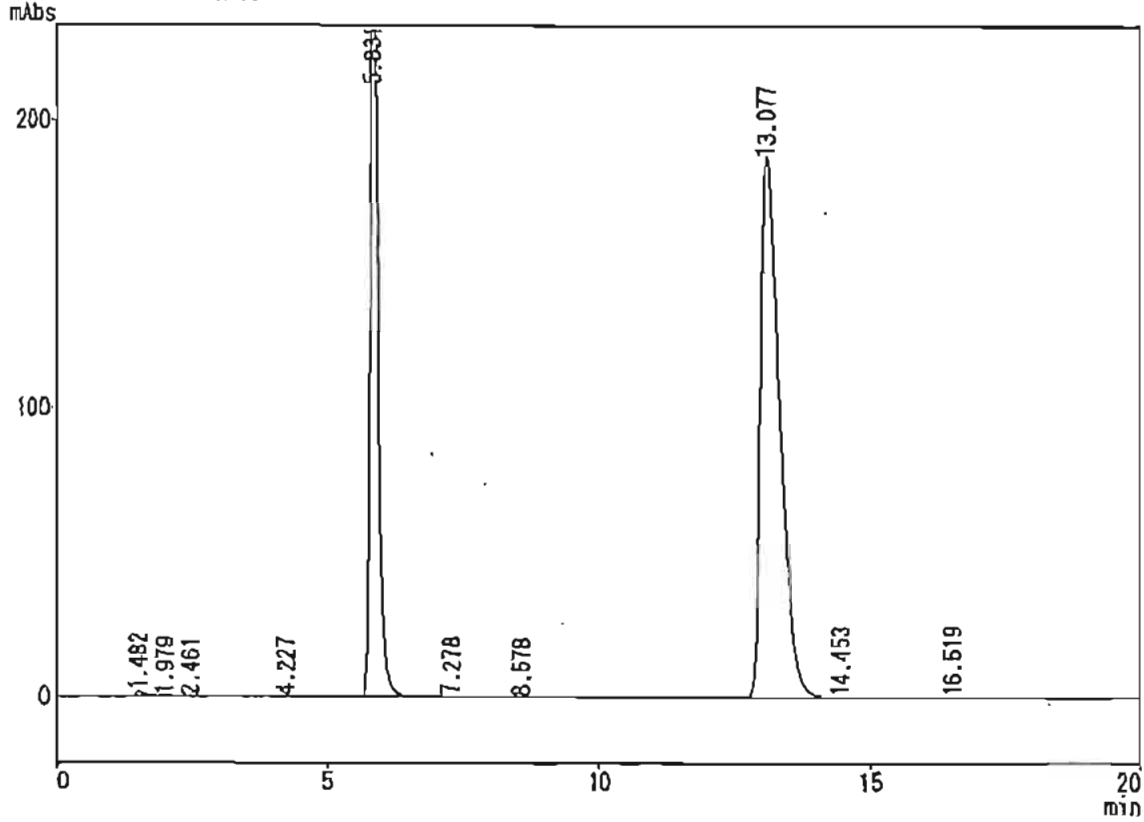
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IONO	CONC	NAME
1	1.479	27607	2569	V		0.3792	
2	1.974	6439	272	V		0.0747	
3	2.462	1497	124	V		0.0206	
4	4.224	1013	132			0.0139	
5	5.829	2741725	275249	S		37.6598	
6	7.274	5228	428	T		0.0718	
7	8.562	1728	126			0.0237	
8	13.065	4487171	188231	S		61.6349	
9	14.468	1675	98	T		0.0230	
10	16.505	7163	301			0.0984	

7280245 467529 100.0000

サンプル : A-02
 ID : 60°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : IHR1028.MET

*** カロマトグラム *** ファイル名:AH2S19.C07



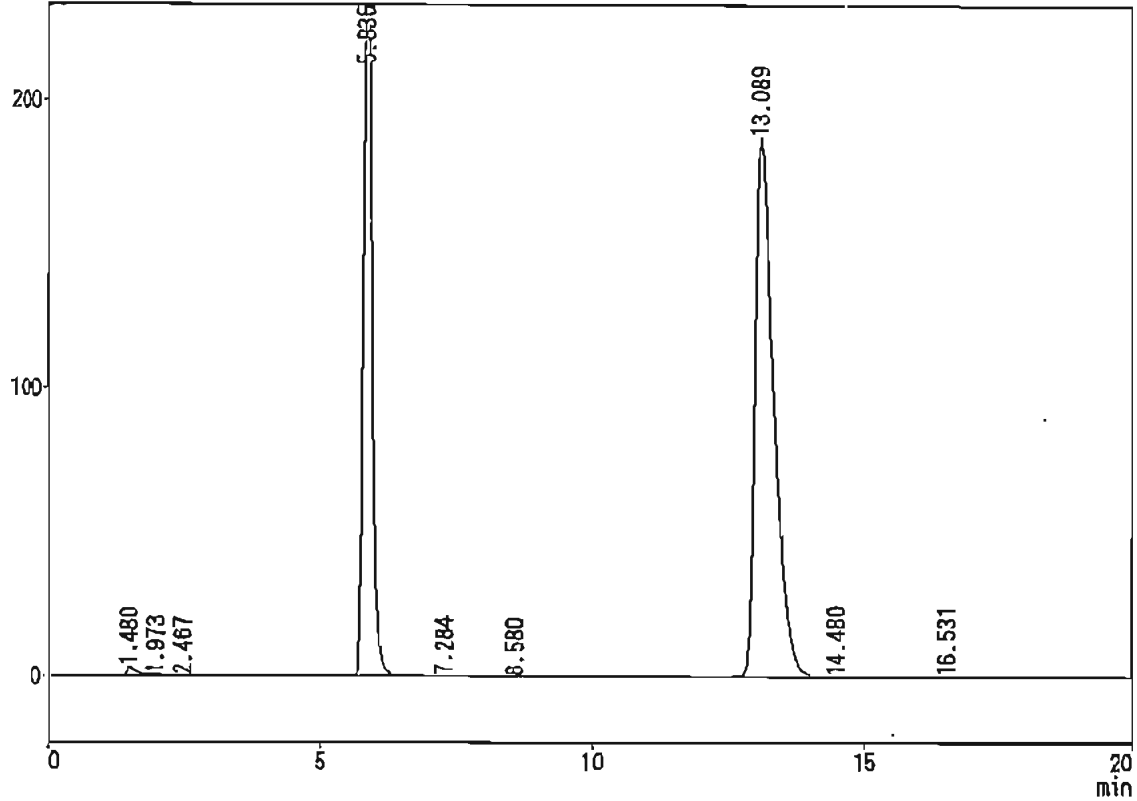
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	18274	1787	V		0.2515	
2	1.979	4011	204	V		0.0552	
3	2.461	1057	96	V		0.0146	
4	4.227	1108	138			0.0152	
5	5.831	2744986	275425	S		37.7808	
6	7.278	5494	451	T		0.0756	
7	8.578	1784	130			0.0245	
8	13.077	4479387	187789	S		61.6521	
9	14.453	1451	74	T		0.0200	
10	16.519	8035	334			0.1106	
		7265588	465427			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=9 データ=AH2S19.D08 02/07/19 17:17:36

サンプル : A-03
ID : 60°C-2W
タイプ : 未知試料
検出器 : SPD-10A シンク
オペレーター名 : 澤 嗣郎
メソッド名 : ICHR1028.MET

*** カラム *** ファイル名:AH2S19.C08
mAbs



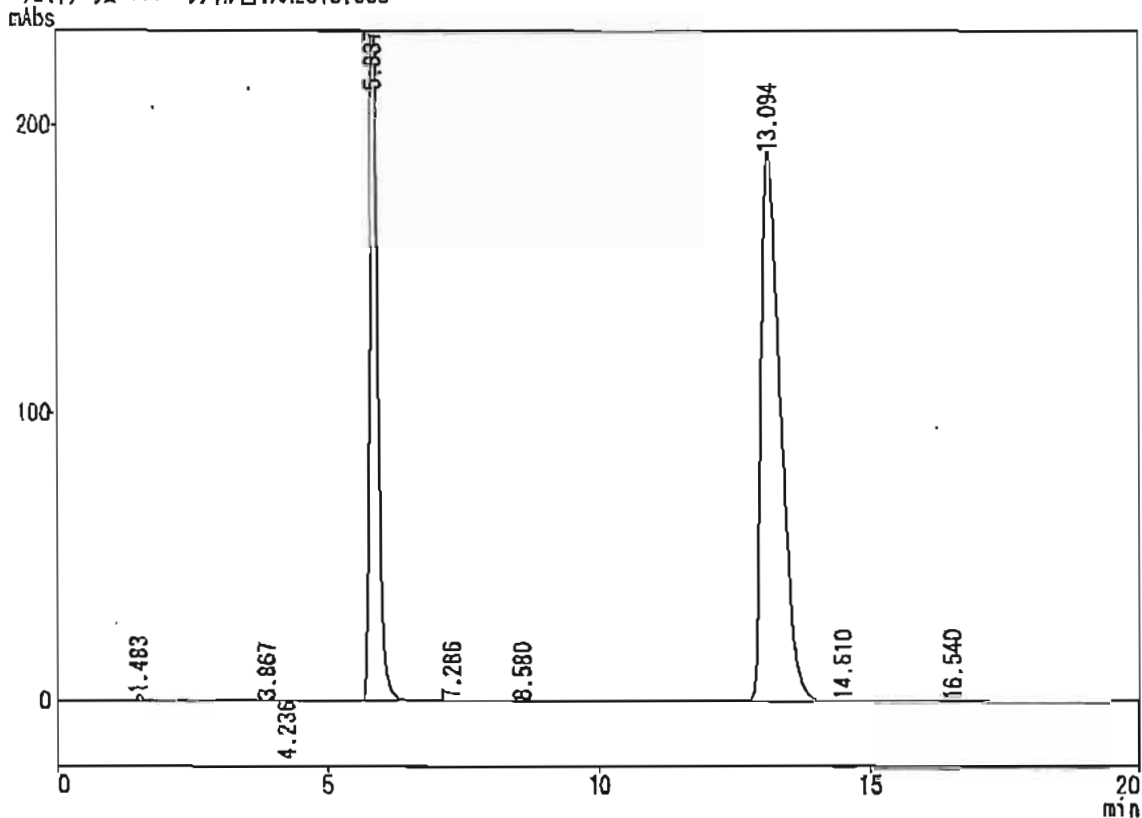
*** レポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.480	42471	3394	V		0.5824	
2	1.973	8211	412	V		0.1128	
3	2.467	2729	185	V		0.0374	
4	5.835	2746480	275743	S		37.6596	
5	7.284	5180	422	T		0.0710	
6	8.580	2189	154			0.0300	
7	13.089	4477249	187667	S		61.3917	
8	14.480	1446	83	T		0.0198	
9	16.531	6962	294			0.0955	

7292918 468353 100.0000

サンプル : A-04
 ID : 60°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カリブレーション *** ファイル名:AH2S19.C09

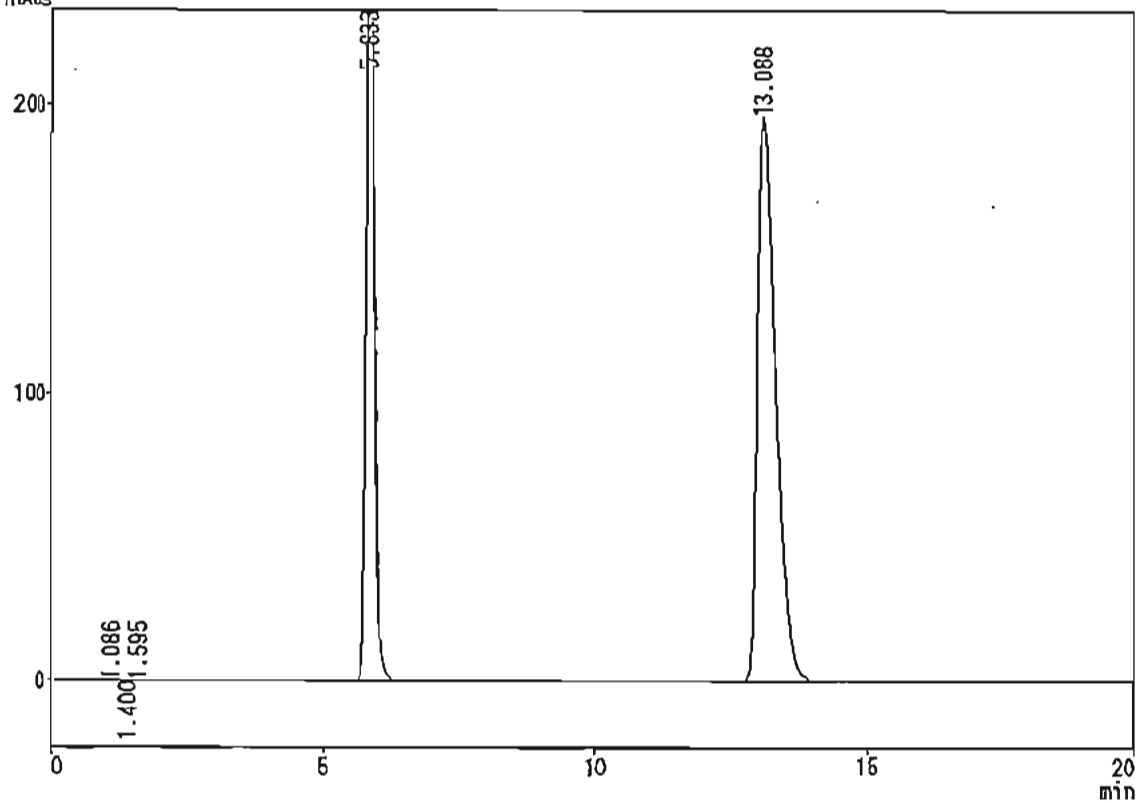


*** レポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.483	18570	2003	V		0.2531	
2	3.867	1451	64	V		0.0198	
3	4.236	1956	192	V		0.0267	
4	5.837	2732365	274669	S		37.2440	
5	7.286	2101	177	T		0.0288	
6	8.580	2217	168			0.0302	
7	13.094	4568943	190680	S		62.2506	
8	14.510	3845	210	T		0.0524	
9	16.540	6940	293			0.0946	
		7338386	468445				
				100.0000			

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレーター名 : 澤 嗣郎
 ネット名 : !AHR1028.MET

*** カラム *** ファイル名:AH2S19.C10
 mAbs



*** ピークリスト ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.086	1129	132			0.0155	
2	1.400	1467	143	V		0.0201	
3	1.595	2467	217	V		0.0338	
4	5.833	2747359	294572			37.8661	
5	13.088	4541555	196213			62.2645	

7293976 491277 100.0000

ブロムフェナクナトリウムの安定性
Lot No.02S051

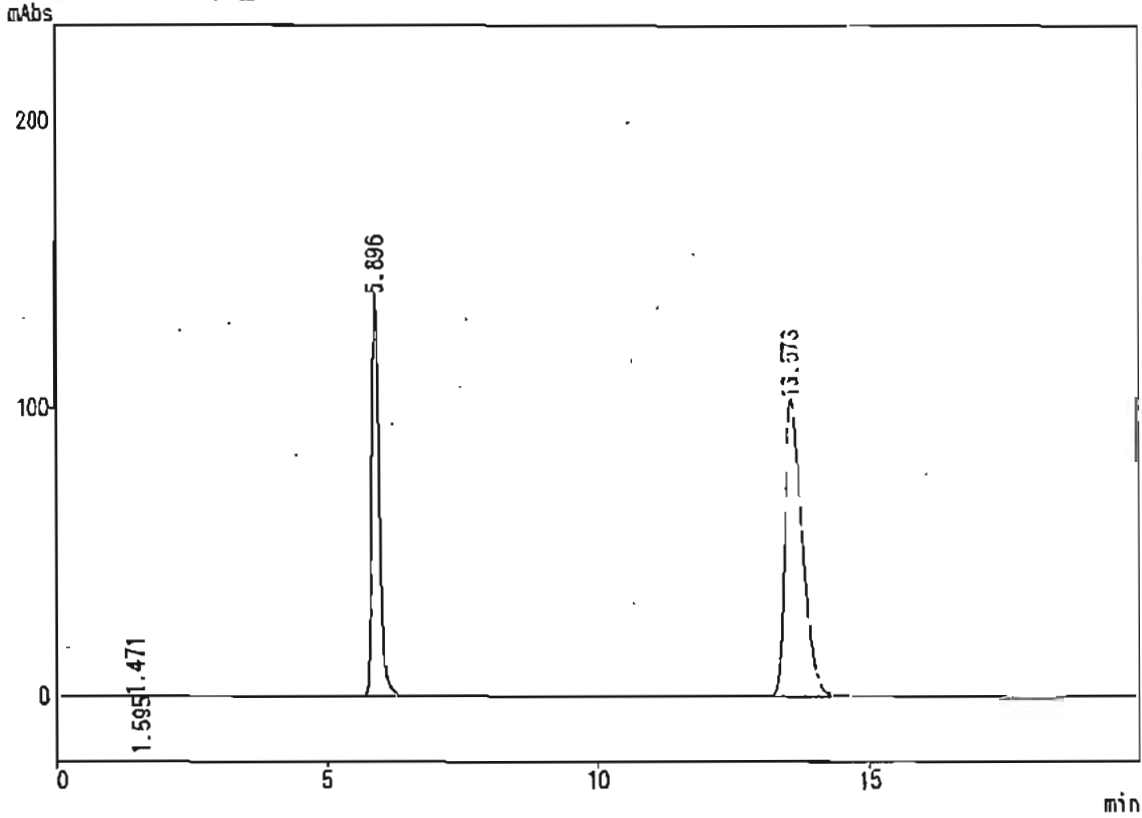
試験コード: P2002B131
試験実施者: 澤 嗣郎
試験実施日: 2002年08月05日

ID	Chromato No	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	AH2T05.C01	2285663	1361530	1.6787							
STD	AH2T05.C06	2275845	1353252	1.6816							
STD	Mean			1.6802	0.10030						
A-01	00°C-4W AH2T05.C02	2296745	1353782	1.6622	0.09863	99.24	02.67	8.15	-	-	6.72
A-02	60°C-4W AH2T05.C03	2207348	1353829	1.6304	0.09733	96.95	80.93	8.18	-	-	6.21
A-03	60°C-4W AH2T05.C04	2232104	1354214	1.6483	0.09840	98.48	91.97	8.15	-	-	6.61
A-04	60°C-4W AH2T05.C05	2296583	1353039	1.6974	0.10133	101.11	94.29	8.16	-	-	6.75

試験物質名: AHR10282B	試験コード: P2002B13/	試験年月日: 2002年08月05日		
試験項目:	試験実施者: 沼 銅郎			
STD AHR10282B 002006g + 移動相 → 20ml	...5-Aug-2002... 11:05:12			
上液 2ml + IS 2ml + 移動相 → 20ml	006: 0.02006 g			
Sample 本: 2ml + IS 2ml + 移動相 → 20ml				
	pH	外觀	重量変化	
A-05 Initial	8.19	--	8.5568 8.5568	8/05 15:32 PH 8.15 NO.18 27.6°C
A-06 Initial	8.19	--	8.4745	
A-05 70°C-2w	8.14	-+	8.4877 8.09249	8/05 15:34 PH 8.16 NO.19 27.7°C
A-06 70°C-2w	8.15	--	8.4894 8.09057	
A-05 60°C-2w	8.17	--	8.4986 8.31388	8/05 15:34 PH 8.15 NO.20 27.5°C
A-06 60°C-2w	8.17	--	8.4796 8.29993	
A-01 60°C-4w	8.15	--	8.9191 8.5653	8/05 15:35 PH 8.16 NO.21 27.3°C
A-02 60°C-4w	8.16	--	9.0937 8.7557	
A-03 60°C-4w	8.15	--	8.8635 8.5192	8/05 15:36 PH 8.19 NO.22 27.6°C
A-04 60°C-4w	8.16	--	8.8862 8.5331	8/05 15:37 PH 8.19 NO.23 27.6°C
				8/05 15:38 PH 8.14 NO.24 27.2°C
				8/05 15:39 PH 8.15 NO.25 27.9°C
				8/05 15:40 PH 8.17 NO.26 27.7°C
				8/05 15:42 PH 8.17 NO.27 27.8°C

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 サンプル名 : 潘 嗣郎
 メソッド名 : !AHR1028.MET

*** カロリグラム *** ファイル名:AH2T05.C01

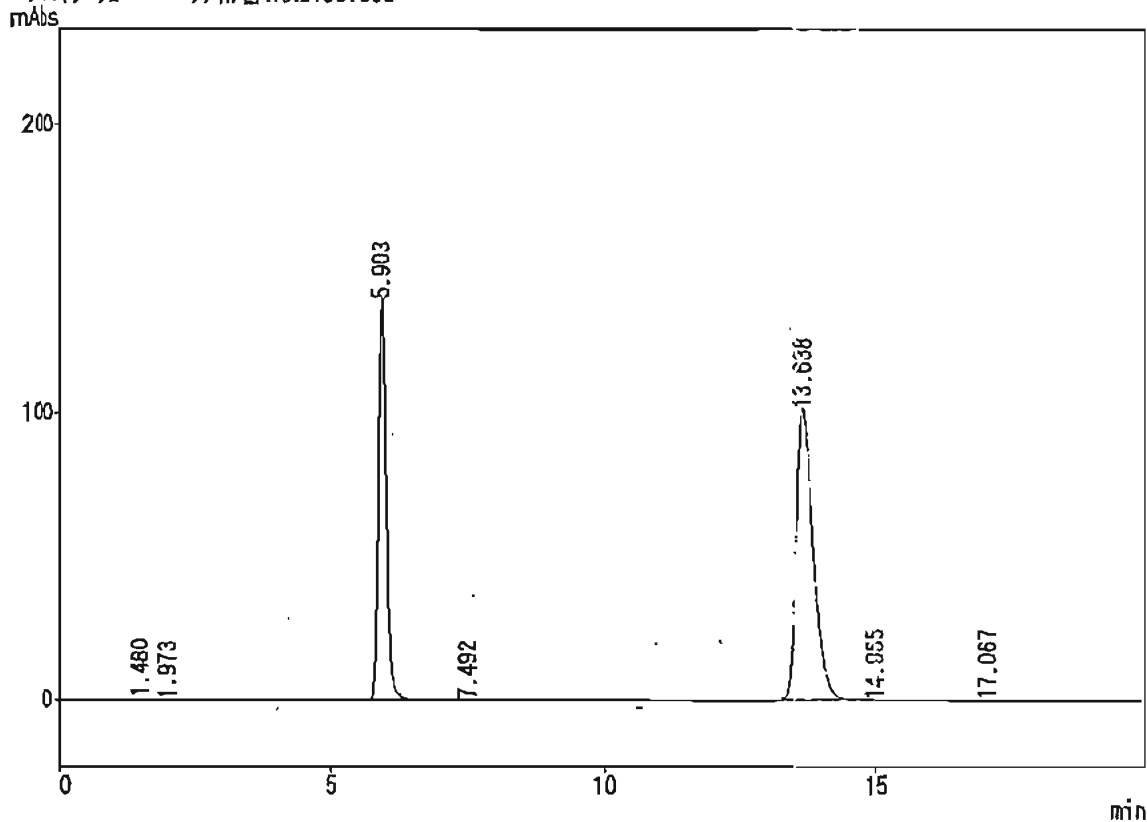


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.471	2112	189	V		0.0579	
2	1.595	2024	197	V		0.0554	
3	5.896	1361530	140425			37.2886	
4	13.573	2285663	103113			62.5981	
		3651329	243904			100.0000	

サンプル : A-01
 ID : 60°C-4W
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カマトラム *** ファイル名:AH2T05.C02

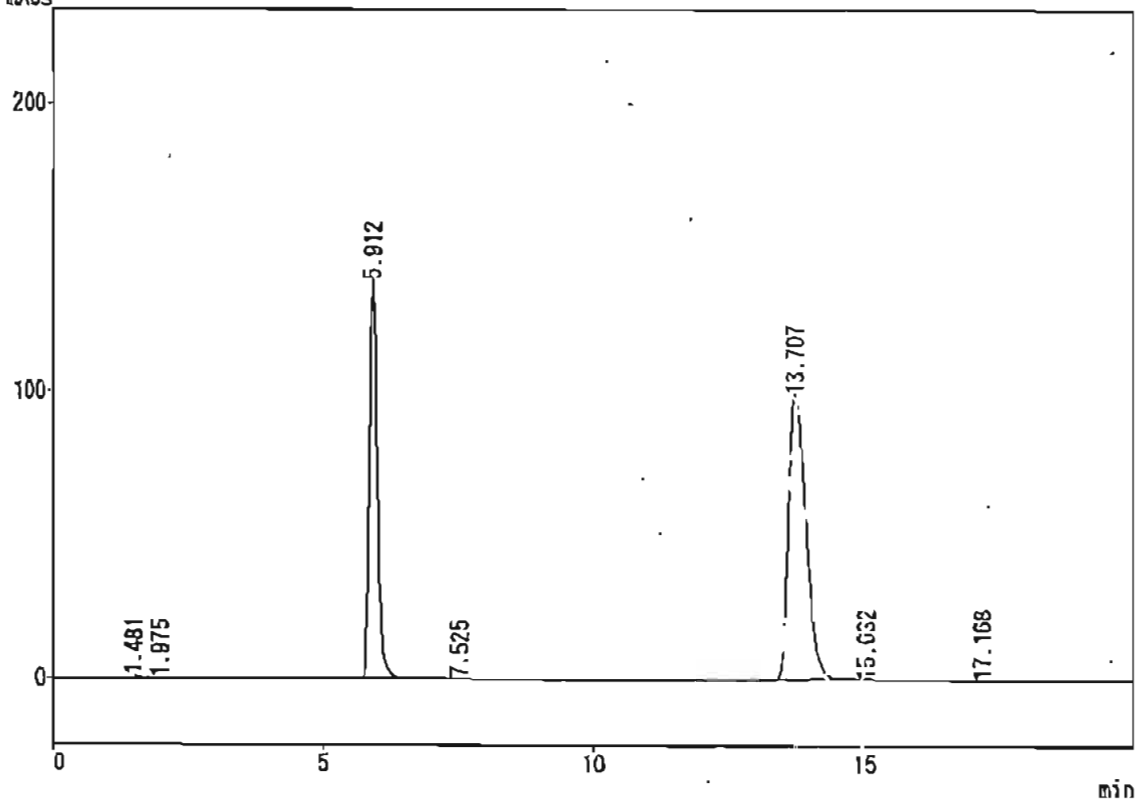


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.480	8402	878	V		0.2324	
2	1.973	2141	108	V		0.0592	
3	5.903	1353782	139760			37.4462	
4	7.492	3715	301			0.1028	
5	13.638	2236745	101102			61.8694	
6	14.965	3114	174			0.0861	
7	17.067	7372	298			0.2039	
		3616271	242621			100.0000	

サンプル : A-02
 ID : 60°C-4W
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 藤 嗣郎
 サンプル名 : !AHR1028.MET

*** カリブレーション *** ファイル名:AH2T05.C03
 mAbs

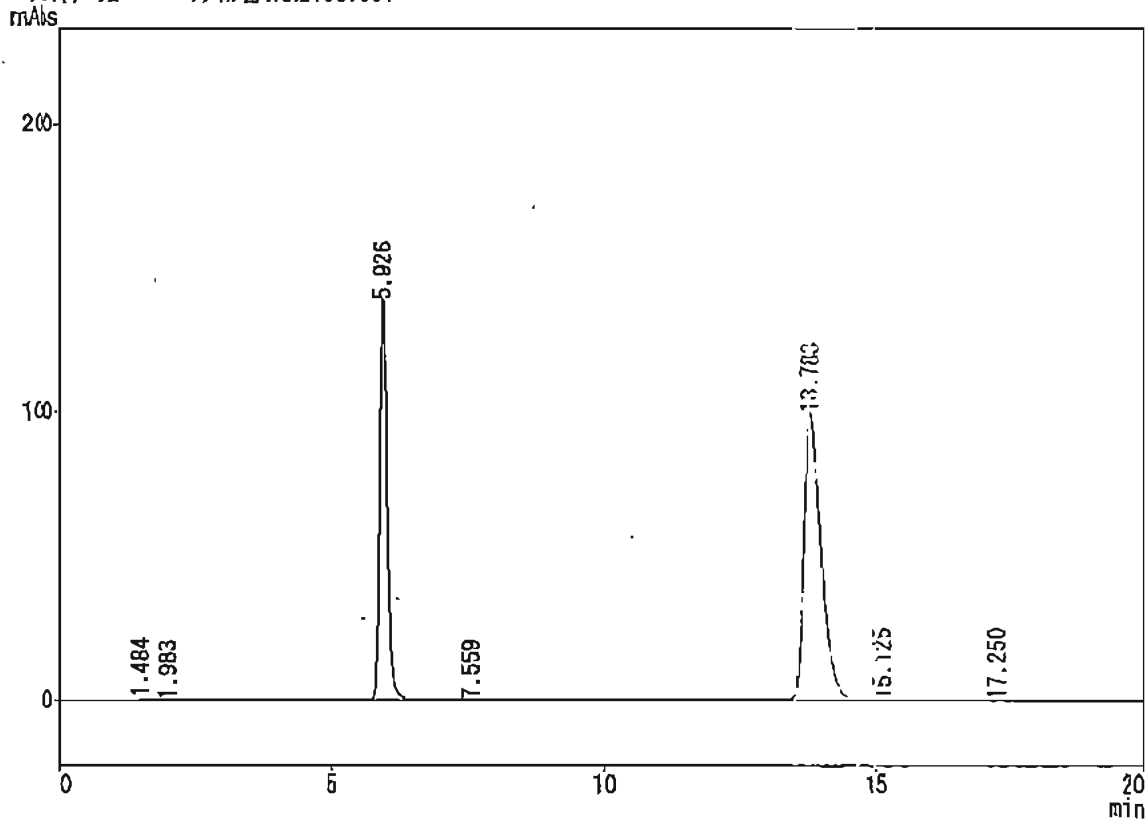


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	8013	856	V		0.2234	
2	1.975	2081	116	V		0.0580	
3	5.912	1353829	139609			37.7542	
4	7.525	3736	310			0.1042	
5	13.707	2207348	99386			61.5562	
6	15.032	3013	165			0.0840	
7	17.188	7886	318			0.2199	
		3585906	240758			100.0000	

サンプル : A-03
 ID : 60°C-4W
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロトグラム *** ファイル名:AH2T05.C04

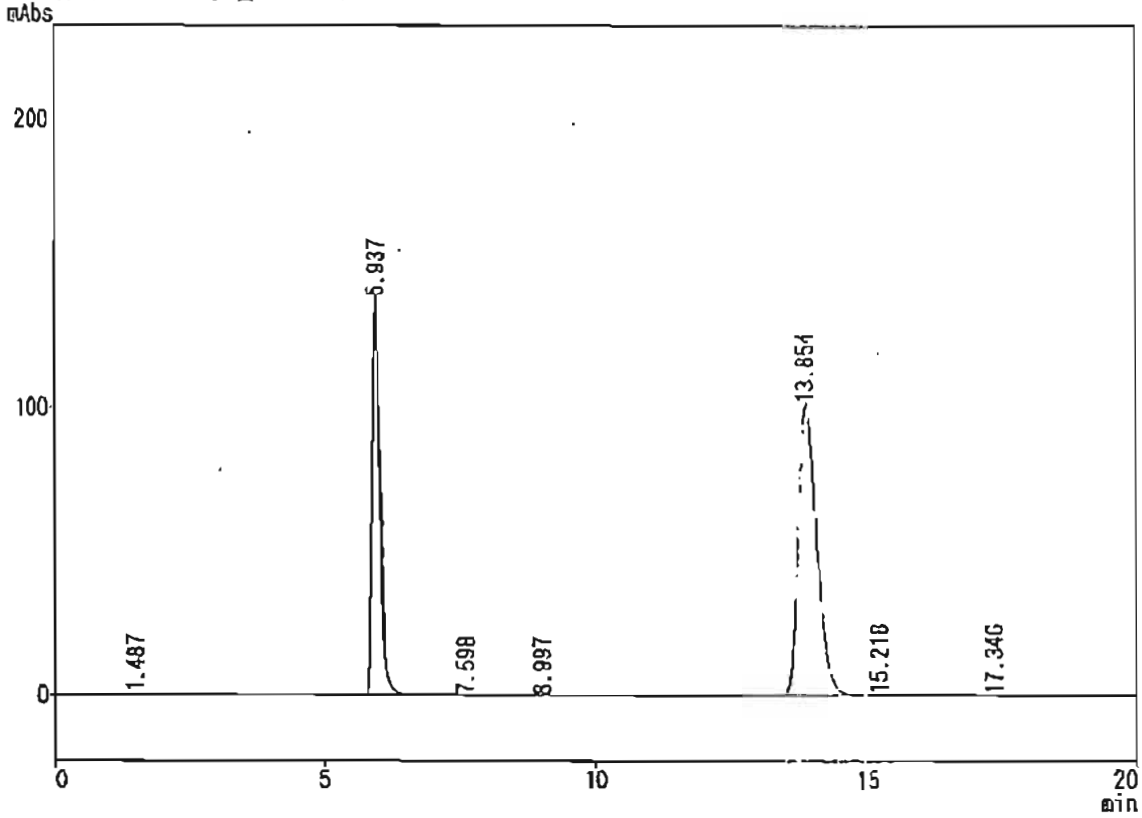


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.484	8194	835	V		0.2265	
2	1.983	2091	101	V		0.0578	
3	5.926	1354214	138981			37.4328	
4	7.559	3522	293			0.0974	
5	13.783	2232104	99359			61.6991	
6	15.125	10454	363	V		0.2890	
7	17.250	7146	291			0.1975	
		3617726	240204			100.0000	

サンプル : A-04
 ID : 60°C-4W
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : IHR1028.MET

*** クロマトグラム *** ファイル名: AH2T05.C05



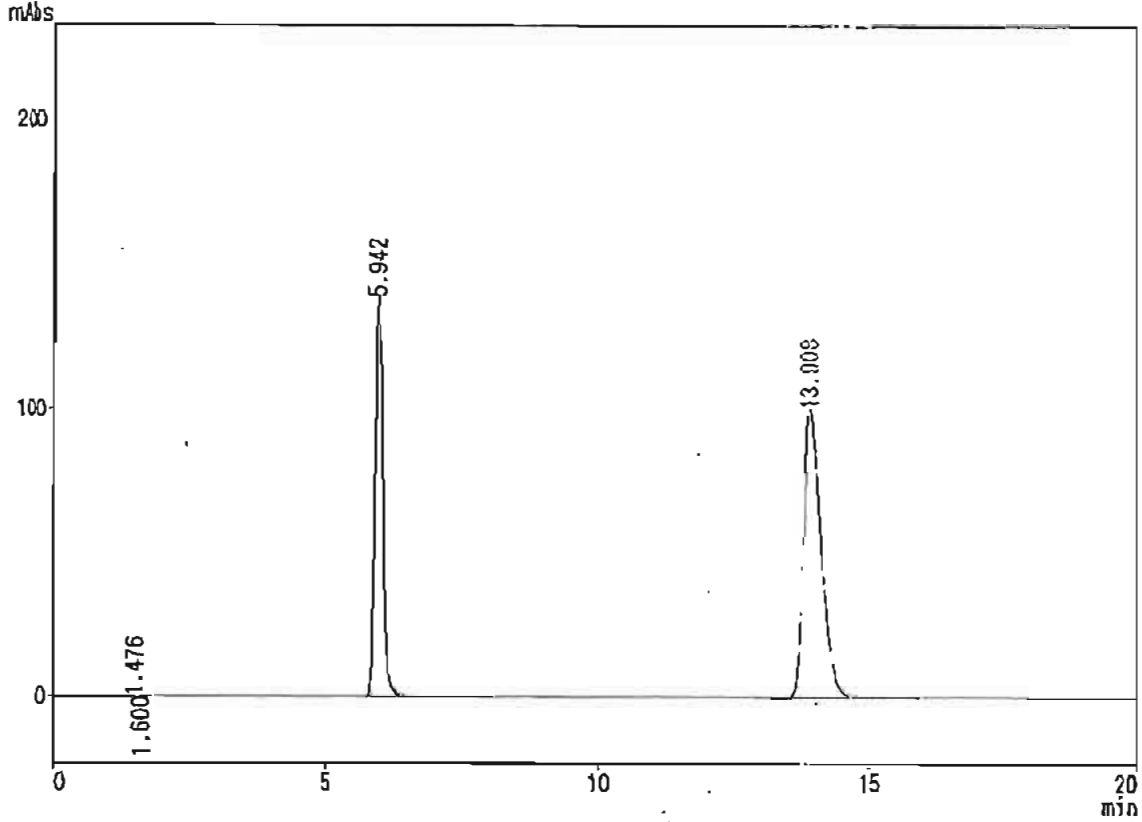
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.487	6295	752			0.1711	
2	5.937	1353039	139054			38.7796	
3	7.598	1753	143			0.0477	
4	8.997	1041	76			0.0283	
5	13.854	2296583	101886			62.4279	
6	15.218	12282	462	V		0.3339	
7	17.346	7785	306			0.2116	

3878778 242479 100.0000

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレーター名 : 澤 嗣郎
 メソッド名 : IAHRT028.MET

*** 加算グラム *** ファイル名:AH2T05.C06



*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.476	2196	184	V		0.0604	
2	1.600	1306	119	V		0.0360	
3	5.942	1353252	138822			37.2551	
4	13.908	2275645	100380			62.6485	
		3632399	239505			100.0000	

プロムフェナクナトリウムの安定性
Lot No.:02S051

試験コード:P2002B131
試験実施者: 澤 嗣郎
試験実施日:2002年09月10日

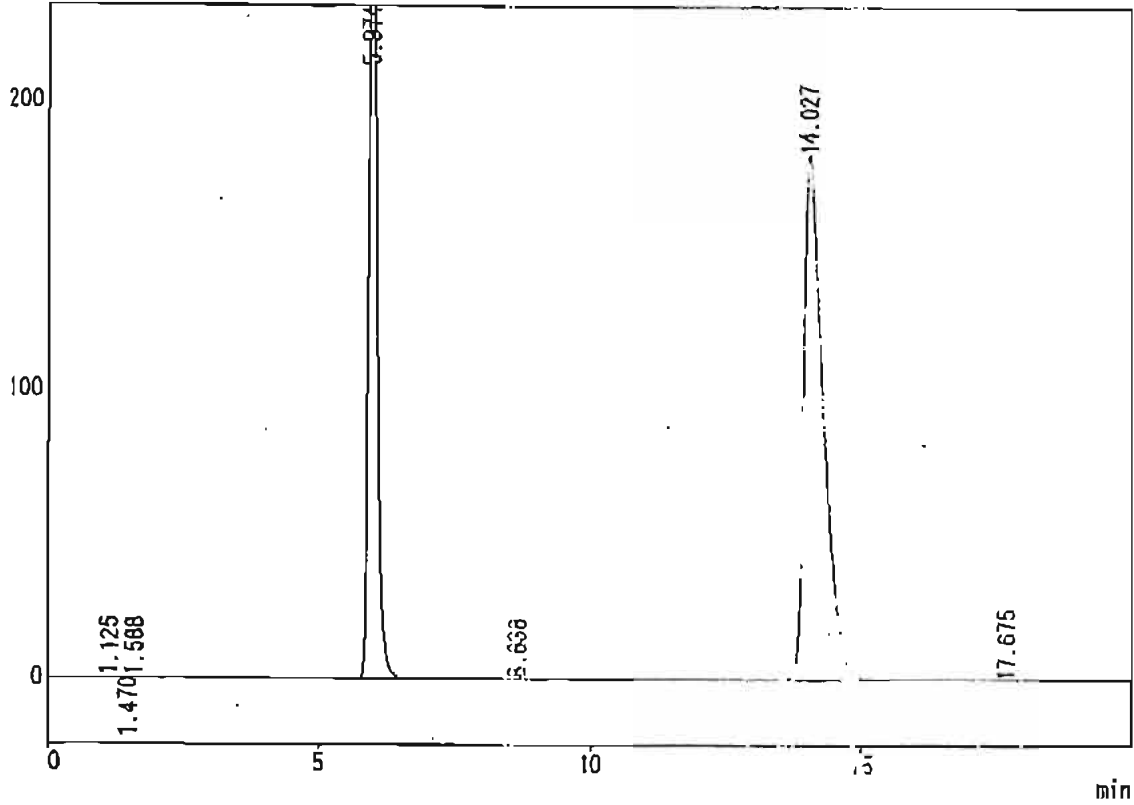
ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	AH2W10.C01	4619110	2799031	1.6503							
STD	AH2W10.C10	4589170	2784914	1.6479							
STD	Mean			1.6491	0.10060						
A-01	40°C-2M AH2W10.C02	4400120	2802864	1.5699	0.09577	96.38	95.89	8.18	-	-	0.49
A-02	40°C-2M AH2W10.C03	4371674	2790031	1.5668	0.09559	95.22	94.75	8.21	-	-	0.49
A-03	40°C-2M AH2W10.C04	4370724	2772844	1.5763	0.09616	96.24	95.69	8.18	-	-	0.67
A-04	40°C-2M AH2W10.C05	4438004	2775214	1.5992	0.09756	97.35	96.83	8.17	-	-	0.53

試験物質名: AHR10282B	試験コード: P2002B13/	試験年月日: 2002年09月10日
試験項目:		試験実施者: 澤 嗣郎
IS P- ϵ -12- β -D-グルコサール 0.02012g + 希釈液相 \rightarrow 10ml		
STD AHR10282B 0.02012g + 希釈液相 \rightarrow 20ml		
		10-Sep-2002 11:49:07
		001:N + 0.02012g

		PH	外観	重量	重量	
A-01	40°C-2M	8.18	-	9.1335	9.1066	9/10 16:59 NO.25 PH 8.18 26.4°C
A-02	↓	8.21	-	9.1203	9.0934	
A-03	↓	8.18	-	8.7819	8.7526	9/10 17:00 NO.26 PH 8.21 25.7°C
A-04	↓	8.17	-	8.9829	8.9547	
						9/10 17:00 NO.27 PH 8.18 26.8°C
A-05	60°C-1W	8.16	-	8.5396	8.4403	9/10 17:01 NO.28 PH 8.17 26.9°C
A-06	60°C-1W	8.17	-	8.4586	8.3582	
A-05	60°C-4W	8.13	-	8.4889	8.1719	9/10 17:03 NO.29 PH 8.16 25.8°C
A-06	60°C-4W	8.14	-	8.4207	8.0996	
						9/10 17:04 NO.30 PH 8.17 26.8°C
						9/10 17:05 NO.31 PH 8.13 26.9°C
						9/10 17:06 NO.32 PH 8.14 27.0°C

CLASS-LC10 Ver.=1.82 システム番号=1 Ch=1 レポート番号=2 データ=AH2W10.D0: 02/09/10 15:44:50
 サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレーター名 : 澤 嗣郎
 メソッド名 : ICHR1028.MET

*** クロマトグラム *** ファイル名:AH2W10.C01
 mAbs

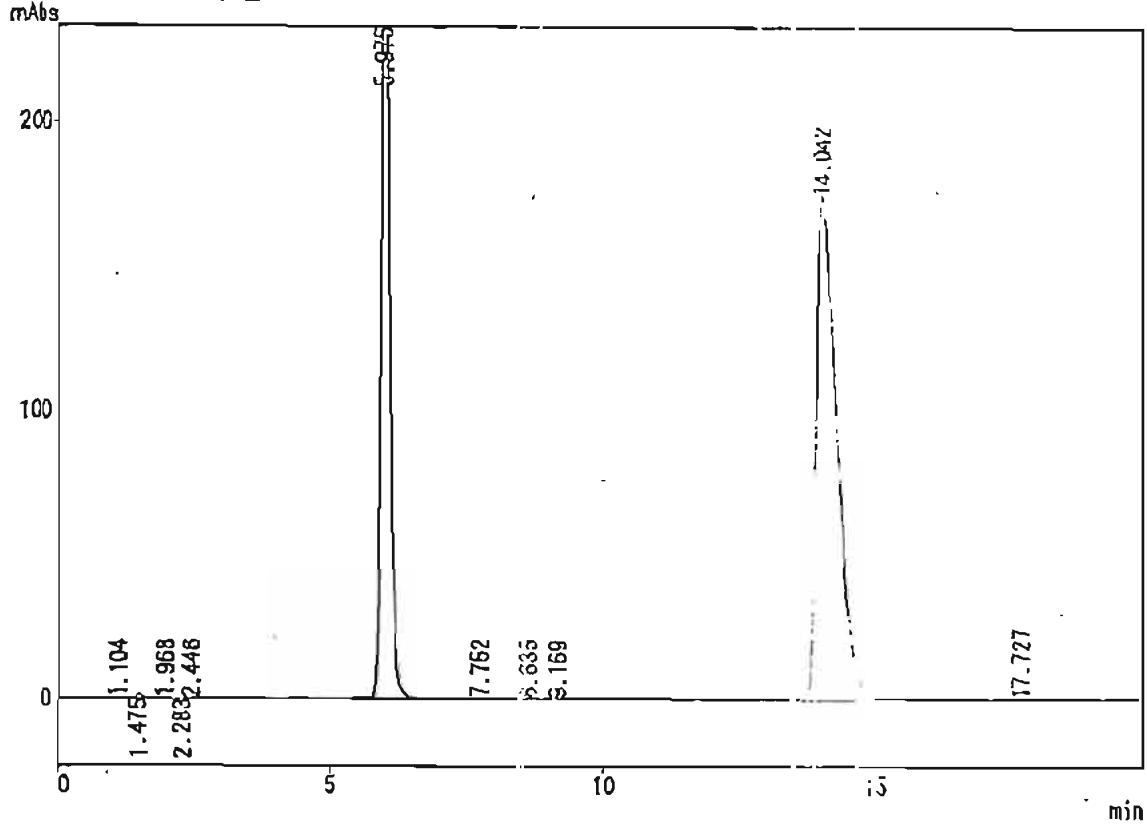


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDEG	CONC	NAME
1	1.125	1083	78			0.0146	
2	1.470	2183	204	V		0.0291	
3	1.588	1489	159	V		0.0200	
4	5.974	2799031	280500	S		37.6529	
5	8.638	9408	383			0.1266	
6	14.027	4619110	181975			62.1368	
7	17.675	1486	61			0.0200	
		7433771	463360			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 検体番号=3 データ=AH2W10.D02 02/09/10 16:04:56
 サンプル : A-01
 ID : 40°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 添 嗣郎
 メソッド名 : !AHR102B.MET

*** カロトグラム *** ファイル名:AH2W10.C02

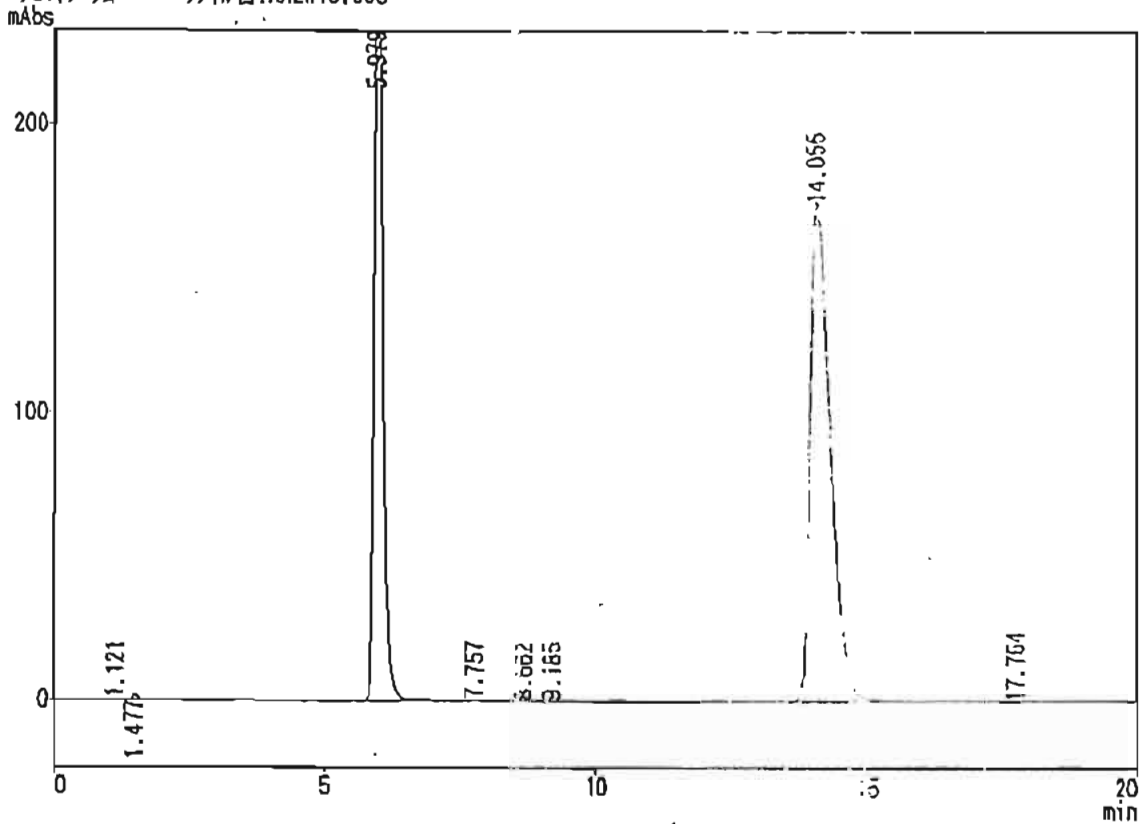


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.104	1671	129			0.0231	
2	1.475	13160	1508	V		0.1817	
3	1.968	1674	151	V		0.0231	
4	2.283	1000	84	V		0.0138	
5	2.448	1044	104	V		0.0144	
6	6.975	2802884	281181			38.7058	
7	7.752	6079	474			0.0839	
8	8.835	5958	282			0.0823	
9	9.169	2575	149	V		0.0356	
10	14.042	4400120	174275	S		60.7829	
11	17.727	5319	209			0.0734	
		7241462	458524			100.0000	

サンプル : A-02
 ID : 40°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 プット名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH2W10.C03



*** ヒートマップ ***

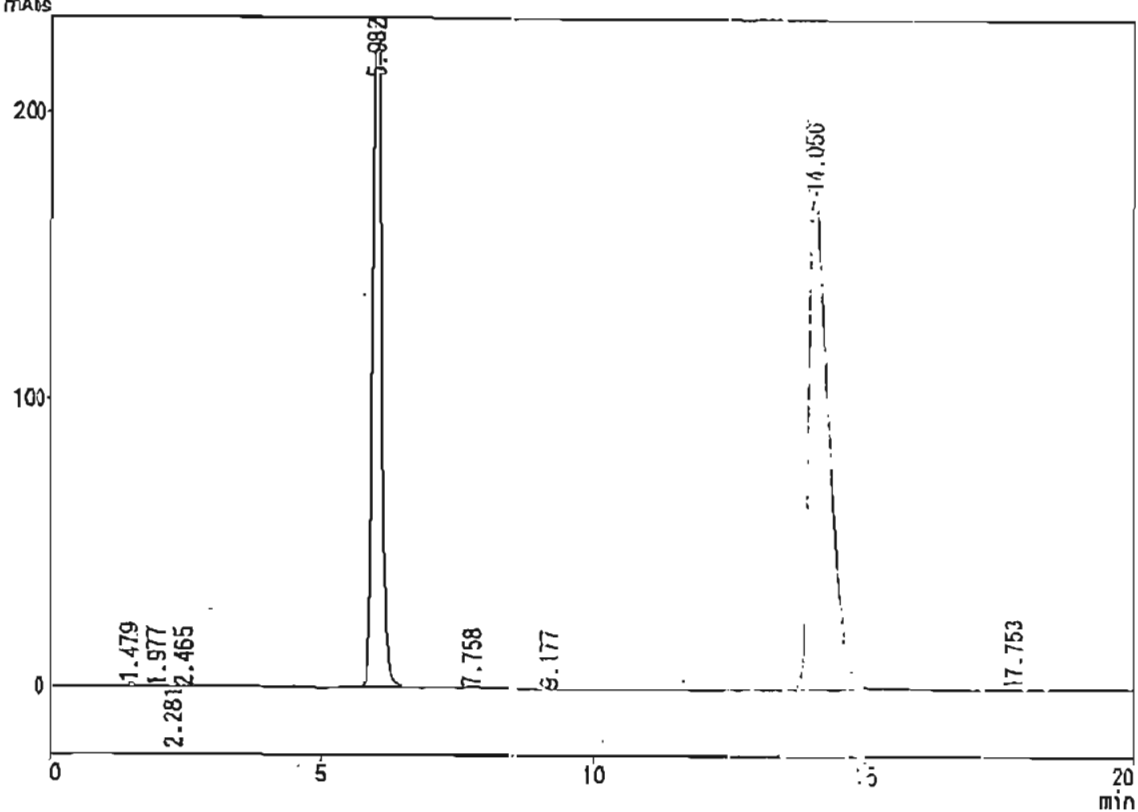
PKNO	TIME	AREA	HEIGHT	MK	IDL.G	CONC	NAME
1	1.121	1530	119			0.0213	
2	1.477	13880	1697	V		0.1902	
3	5.979	2790031	280527	V		38.7821	
4	7.757	6303	503			0.0876	
5	8.662	2925	135			0.0407	
6	9.185	2468	128	V		0.0343	
7	14.055	4371674	173463			60.7673	
8	17.754	5513	214			0.0766	

7194122 456783 100.0000

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 検出器=SPD-10A データ=AH2W10.L04 02/09/10 16:45:08

サンプル : A-03
 ID : 40°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 ハンドラー名 : 澤 嗣郎
 サンプル名 : !AHR1028.MET

*** カリブレーション *** ファイル名:AH2W10.C04
 mAbs



*** ピークレポート ***

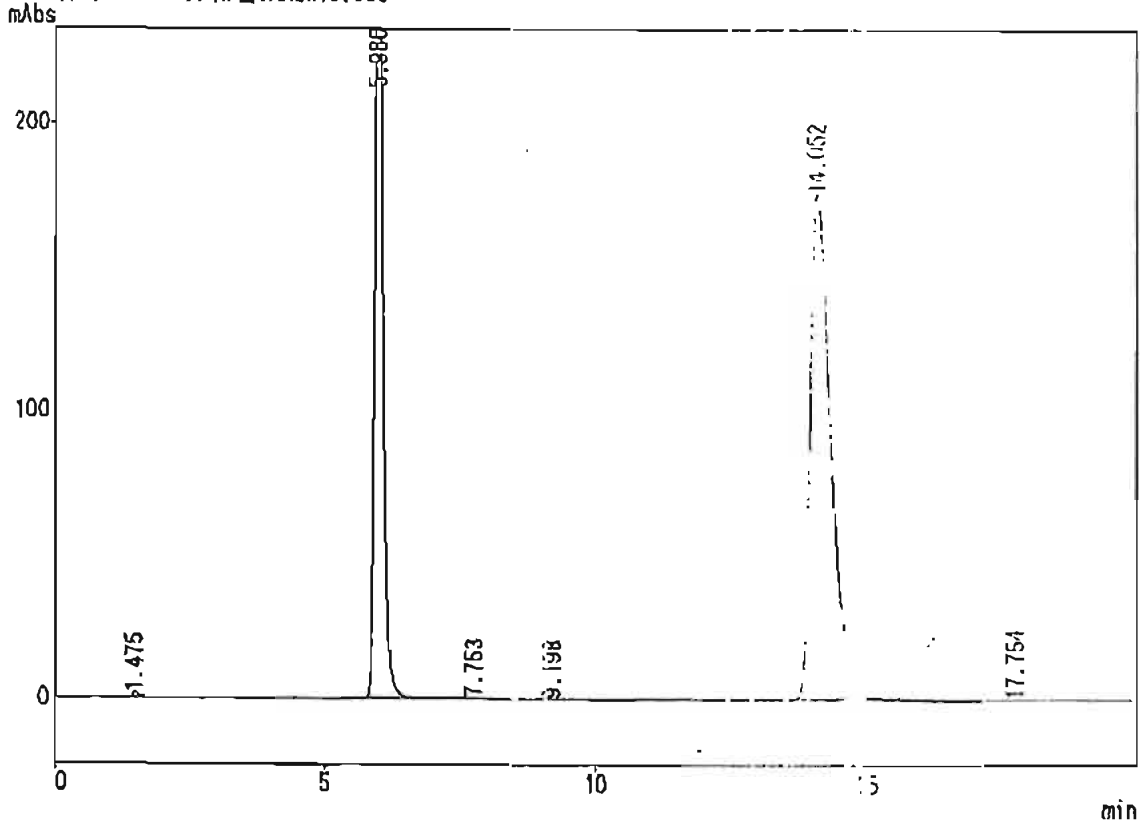
PKNO	TIME	AREA	HEIGHT	MK	IDIG	CONC	NAME
1	1.479	13582	1590	V		0.1893	
2	1.977	1971	157	V		0.0275	
3	2.281	1187	103	V		0.0165	
4	2.465	1225	98	V		0.0171	
5	5.982	2772844	278821	V		38.6498	
6	7.758	6321	496			0.0881	
7	9.177	1370	102			0.0191	
8	14.056	4370724	173373			60.9221	
9	17.753	5083	199			0.0706	

7174285 454939 100.0000

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 検出器番号=0 データ=AH2W10.C05 02/09/10 17:05:14

サンプル : A-04
ID : 40°C-2M
タイプ : 未知試料
検出器 : SPD-10A シンクル
オペレーター名 : 澤 嗣郎
メソッド名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH2W10.C05



*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDI-G	CONC	LABE
1	1.475	16535	1704	SV		0.2284	
2	5.980	2775214	280141	V		38.3349	
3	7.753	2827	230			0.0391	
4	9.198	1552	111			0.0214	
5	14.052	4438004	176335			61.3035	
6	17.754	5262	210			0.0727	
		7239394	458731			100.0000	

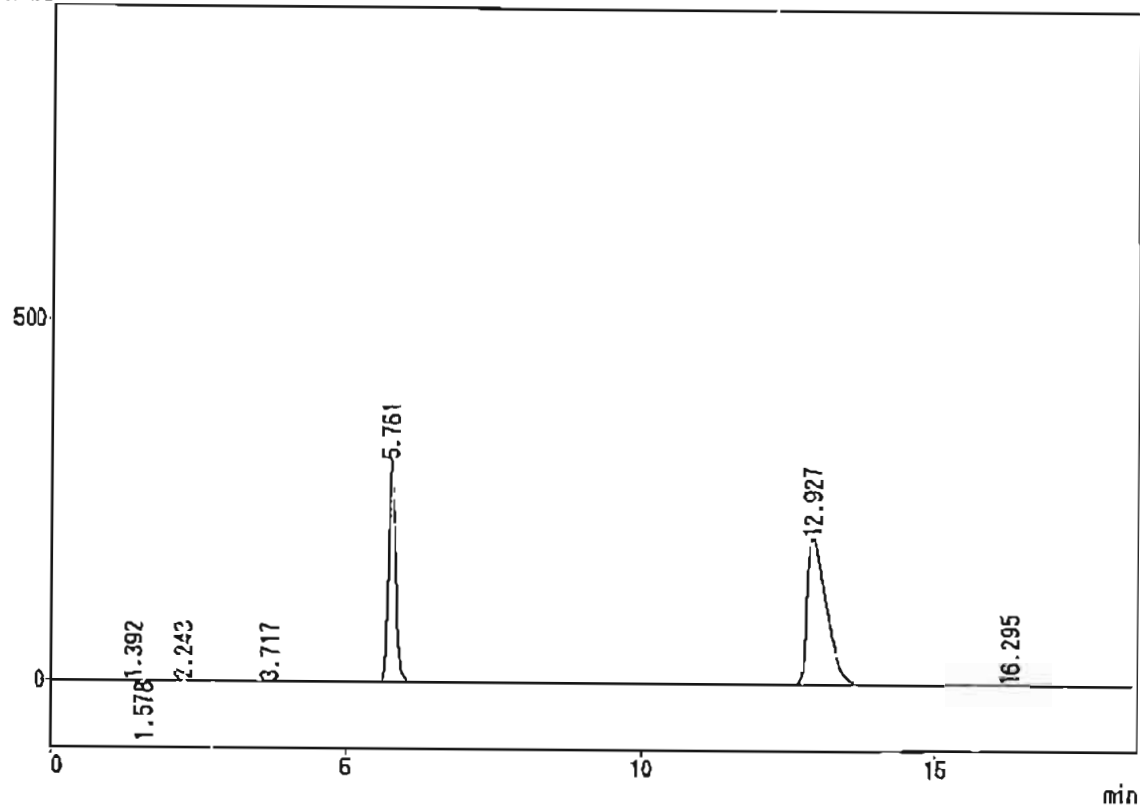
ブロムフェナクナトリウムの安定性
Lot No.02S051

試験コード:P2002B131
試験実施者: 澤 嗣郎
試験実施日:2002年11月13日

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	AH2Y13.C17	4568030	2793072	1.6355							
STD	AH2Y13.C22	4568150	2785109	1.6402							
STD	Mean			1.6379	0.09980						
A-01	40°C-4M AH2Y13.C18	4306875	2788894	1.5443	0.09410	94.68	93.59	8.19	—	—	1.15
A-02	40°C-4M AH2Y13.C19	4283036	2781720	1.5397	0.09382	93.46	92.50	8.19	—	—	1.03
A-03	40°C-4M AH2Y13.C20	4281426	2779313	1.5405	0.09387	93.95	92.95	8.19	—	—	1.06
A-04	40°C-4M AH2Y13.C21	4407948	2776019	1.5879	0.09675	96.54	95.50	8.20	—	—	1.08

CLASS-LC10 Ver.=1.02 システム番号=1 Ch=1 検体番号=92 データ=AH2Y13.D17 02/11/13 19:53:42
 サンプル : STD
 IO :
 タイプ : 未知試料
 検出器 : SPD-10A シンガム
 分析機名 : 澤 嗣郎
 サンプル名 : 'HR1028.MET

*** カロリグラム *** ファイル名:AH2Y13.C17
 mAbs

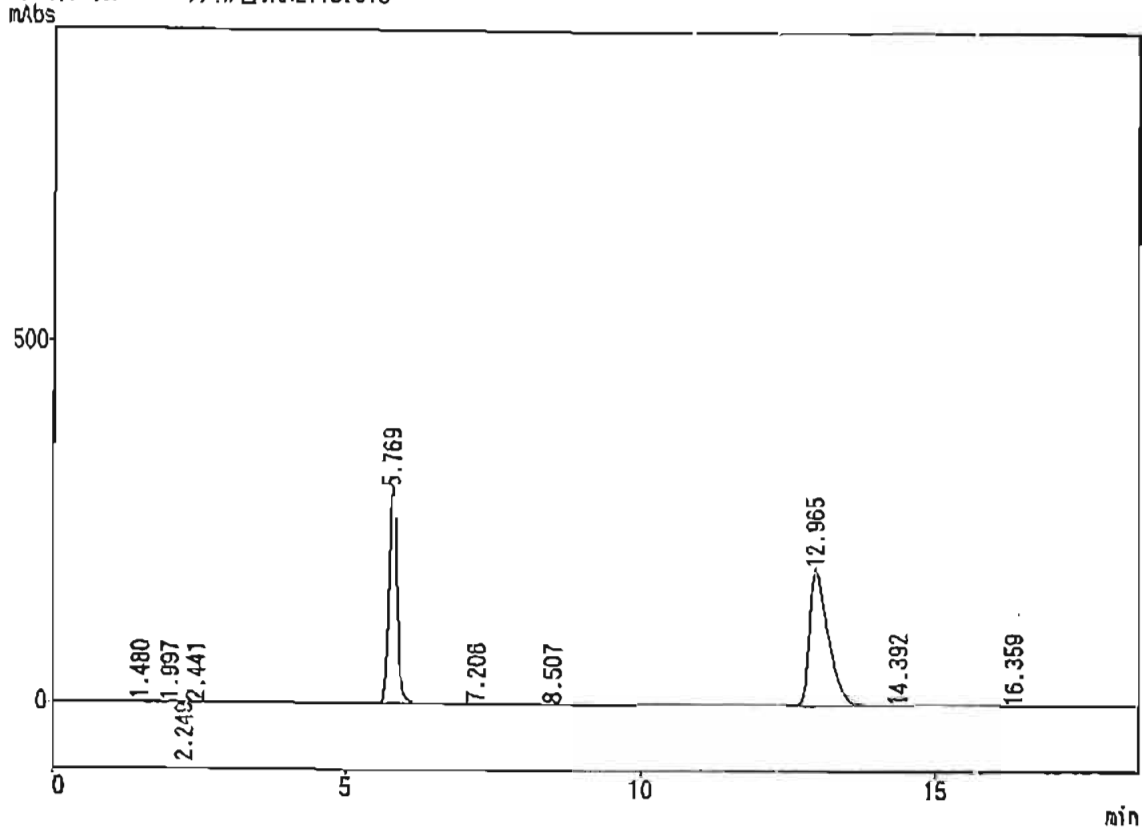


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.392	1169	135	V		0.0159	
2	1.578	1403	160	V		0.0190	
3	2.243	2485	346			0.0337	
4	3.717	5849	189			0.0793	
5	5.761	2793072	306801			37.8821	
6	12.927	4568030	202625	S		61.9556	
7	16.295	1065	58			0.0144	
		7373072	510316			100.0000	

サンプル : A-01
 ID : 40°C-4H1
 タイプ : 未知試料
 検出器 : SPD-10A シンガム
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロマトグラム *** ファイル名:AH2Y13.C18



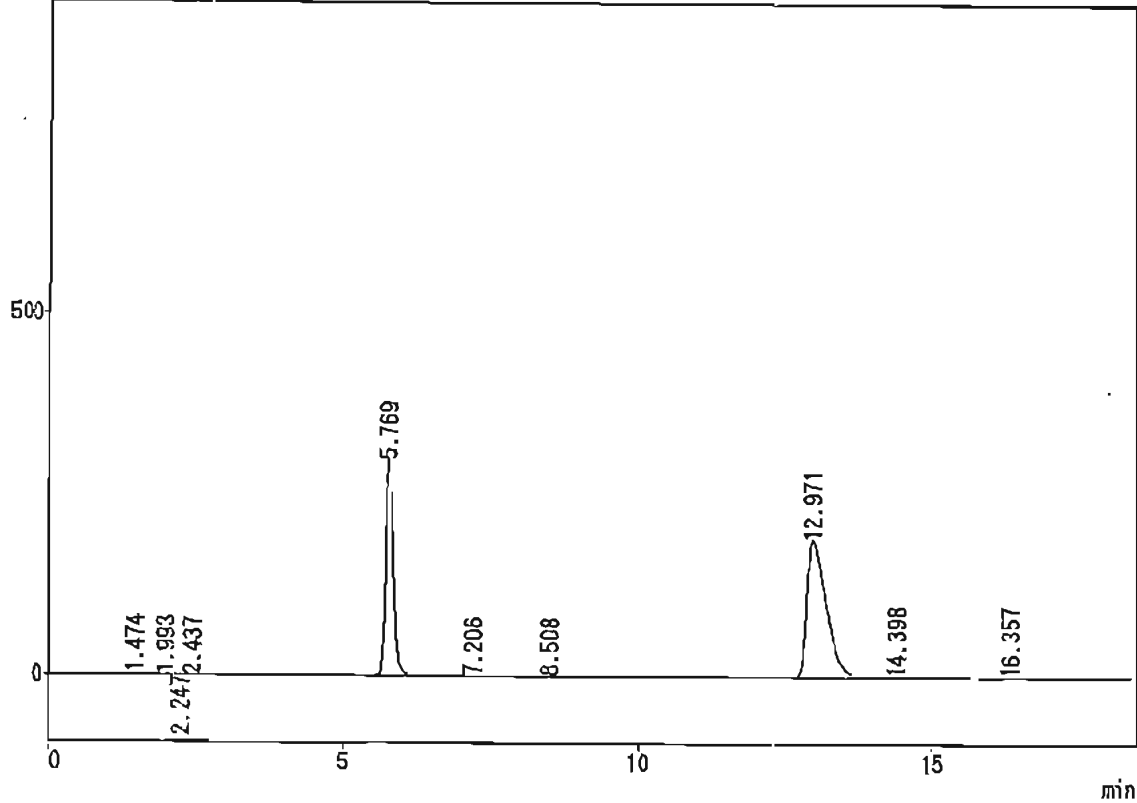
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.480	12300	1194	V		0.1723	
2	1.997	2187	205	V		0.0308	
3	2.249	4370	511	V		0.0612	
4	2.441	1458	149	V		0.0204	
5	5.769	2788894	299943	S		39.0672	
6	7.206	8268	708	T		0.1158	
7	8.507	2288	162			0.0320	
8	12.965	4308875	190172	S		60.3313	
9	14.392	3480	209	T		0.0487	
10	16.359	8592	367			0.1204	
		7.38710	493622			100.0000	

CLASS-LC10 Ver.=1.52 システム番号=1 Ch=1 レポート番号=94 データ=AH2Y13.019 02/11/13 20:33:40

サンプル : A-02
 ID : 40°C-4M
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレータ名 : 澤 詞郎
 メソッド名 : !AHR1028.MET

*** カロリグラム *** ファイル名:AH2Y13.C19
 mAbs

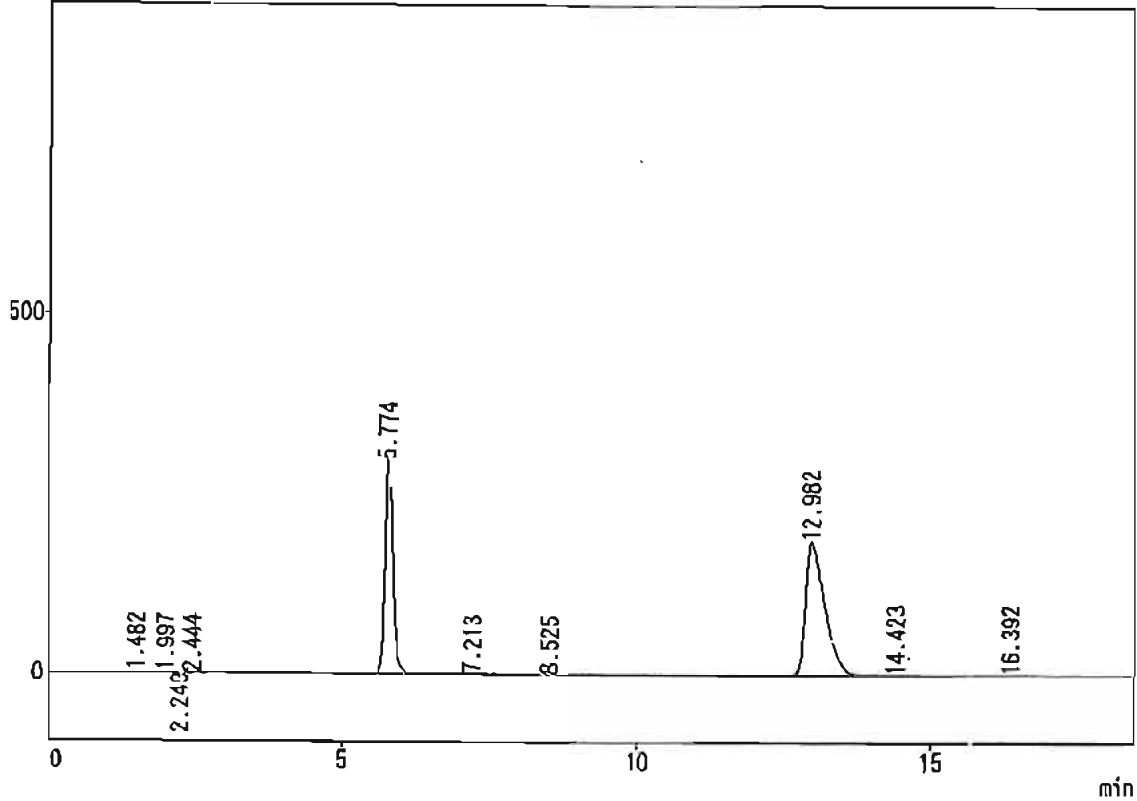


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.474	16063	1662	V		0.2259	
2	1.993	2329	217	V		0.0327	
3	2.247	4034	463	V		0.0567	
4	2.437	1444	145	V		0.0203	
5	5.769	2781720	299235	S		39.1128	
6	7.206	8886	761	T		0.1249	
7	8.508	2448	172			0.0344	
8	12.971	4283036	189293	S		60.2223	
9	14.398	3050	189	T		0.0429	
10	16.357	9032	386			0.1270	
		7.12042	492524			100.0000	

サンプル : A-03
 ID : 40°C-4H
 タイプ : 未知試料
 検出器 : SPD-10A シンガム
 オペレーター名 : 藤 嗣郎
 メソッド名 : I_AHR102B.MET

*** クロマトグラム *** ファイル名:AH2Y13.C20
 mAbs



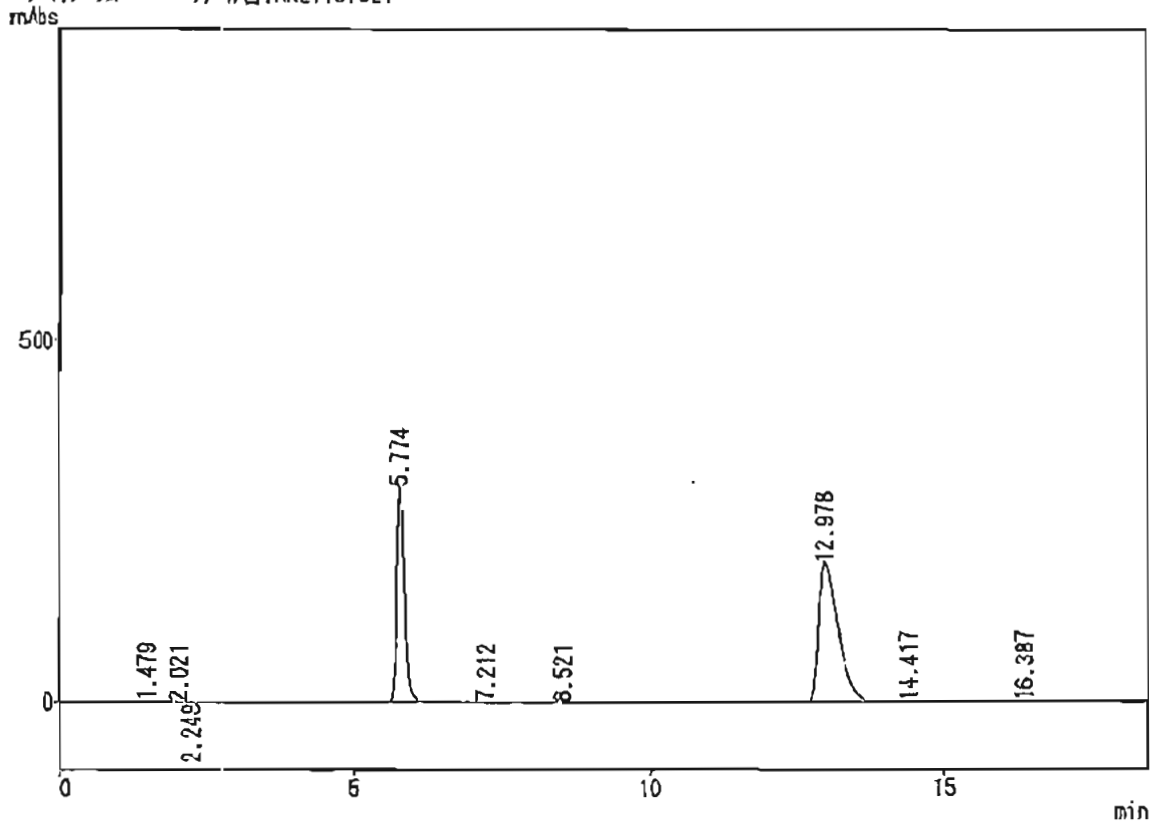
*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	10532	1104	V		0.1483	
2	1.997	2091	199			0.0294	
3	2.249	3685	416	V		0.0519	
4	2.444	1451	139	V		0.0204	
5	5.774	2779313	298508	S		39.1381	
6	7.213	8413	720	T		0.1185	
7	8.525	2397	170			0.0338	
8	12.982	4281426	189051	S		60.2908	
9	14.423	3504	213	T		0.0493	
10	16.392	8483	366			0.1195	
		7101294	490885			100.0000	

CLASS-LC10 Ver.=1.02 システム番号=1 Ch=1 レポート番号=96 データ=AH2Y13.D21 02/11/13 21:13:34

サンプル : A-04
 ID : 40°C-4M
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレーター名 : 澤 嗣郎
 サンプル名 : !AHR1028.MET

*** カリブレーション *** ファイル名:AH2Y13.C21

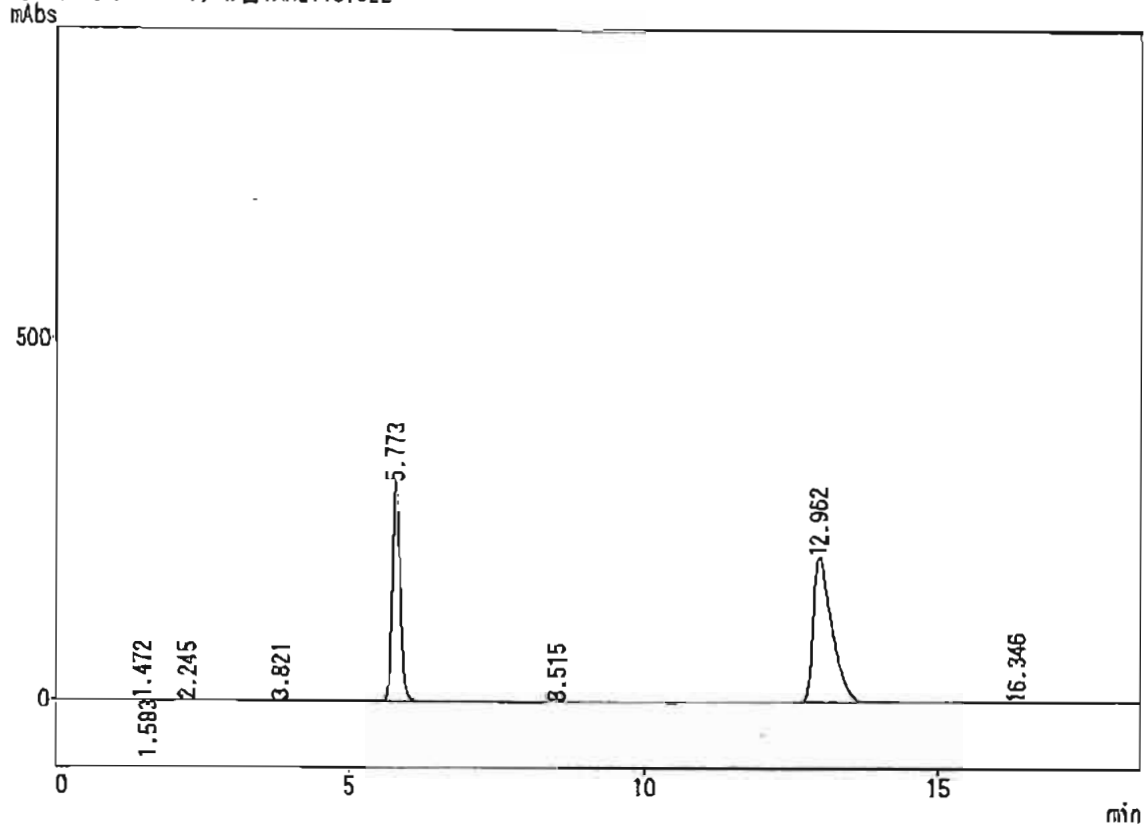


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.479	12894	1366	V		0.1785	
2	2.021	1395	135			0.0193	
3	2.248	2841	338	V		0.0393	
4	5.774	2778019	298879	V		38.4233	
5	7.212	4536	394			0.0628	
6	8.521	2141	157			0.0295	
7	12.978	4407946	193885	S		61.0110	
8	14.417	7349	403	T		0.1017	
9	16.387	9719	419			0.1345	
		7224840	495974			100.0000	

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 深 嗣郎
 ネット名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH2Y13.C22



*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.472	3388	280	V		0.0460	
2	1.583	1918	247	V		0.0261	
3	2.245	1315	196			0.0179	
4	3.821	1523	78			0.0207	
5	5.773	2785109	305132			37.8223	
6	8.515	1094	83			0.0148	
7	12.962	4588150	202151	S		62.0363	
8	18.346	1173	61			0.0159	
		7363870	508228			100.0000	

プロムフェナクナトリウムの安定性
 Lot No.:02S051

試験コード: P2002B131
 試験実施者: 澤 嗣郎
 試験実施日: 2003年01月17日

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid
STD	AH3H17.C01	4549865	2846553	1.5984						
STD	AH3H17.C08	4565385	2858747	1.5970						
STD	Mean			1.5977	0.10095					
A-01	40°C-6M AH3H17.C02	4275582	2872191	1.4886	0.09408	94.64	92.90	8.17	-	-
A-02	40°C-6M AH3H17.C03	4243563	2866430	1.4804	0.09354	93.18	91.53	8.18	-	-
A-03	40°C-6M AH3H17.C04	4244241	2862118	1.4829	0.09370	93.78	92.11	8.17	-	-
A-04	40°C-6M AH3H17.C05	4386572	2871808	1.5275	0.09651	96.30	94.66	8.17	+	-

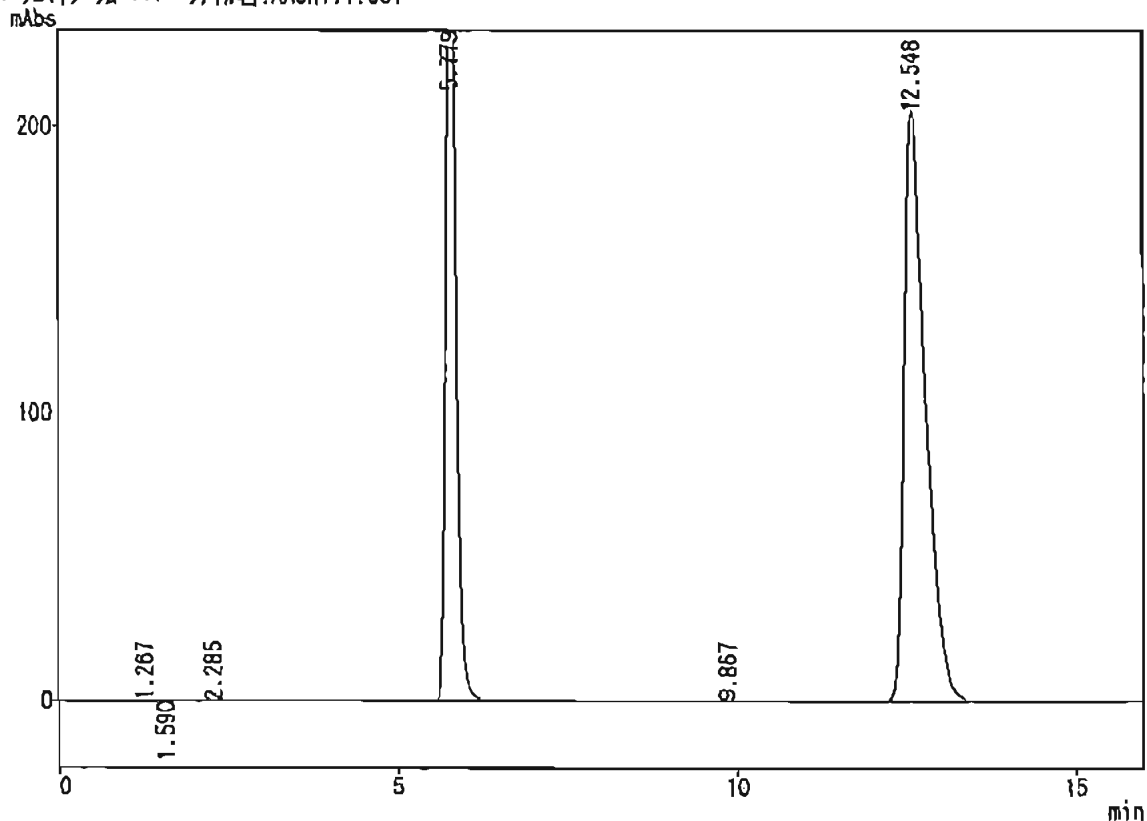
プロムフェナクナトリウムの安定性
Lot No.:02X221

試験コード: P2002B131
試験実施者: 澤 嗣郎
試験実施日: 2003年01月17日

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid
STD	AH3H17.C08	4565385	2858747	1.5970						
STD	AH3H17.C15	4556169	2852477	1.5973						
STD	Mean			1.5972	0.10095					
A-01	40°C-2M AH3H17.C09	4381017	2860103	1.5318	0.09682	95.45	94.91	8.17	—	—
A-03	40°C-2M AH3H17.C10	4407378	2857141	1.5428	0.09750	97.40	96.84	8.16	—	—
BF	40°C-2M AH3H17.C11	4447937	2867651	1.5511	0.09804	96.07	95.53	8.23	—	—
A-01	25°C-2M AH3H17.C12	4465271	2874184	1.5501	0.09797	96.59	96.26	8.17	—	—
A-03	25°C-2M AH3H17.C13	4468866	2860635	1.5815	0.09869	98.59	98.25	8.15	—	—
BF	25°C-2M AH3H17.O14	4539804	2862632	1.5859	0.10024	98.23	97.93	8.23	—	—

サンプル名 : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シックル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR102B.MET

*** クロマトグラム *** ファイル名:AH3H171.C01

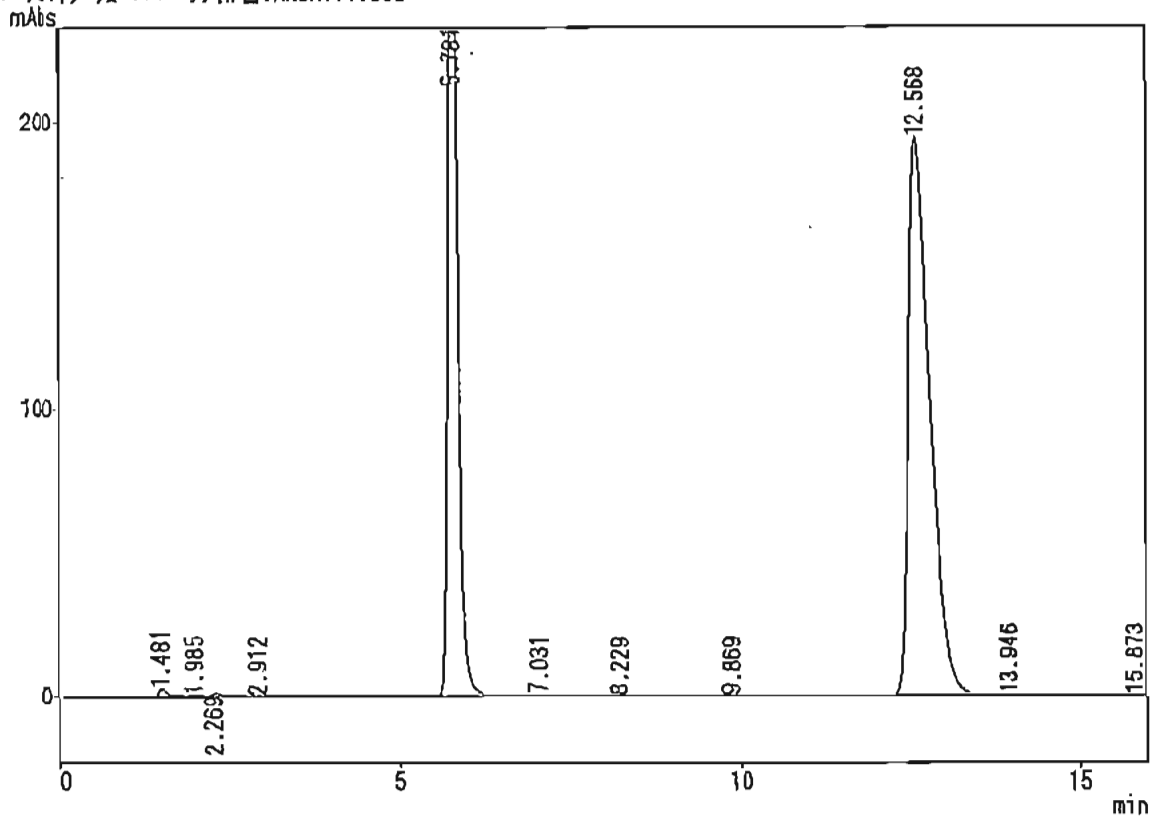


*** レポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.267	2015	103			0.0272	
2	1.590	1854	138	V		0.0250	
3	2.285	1836	245	V		0.0248	
4	5.779	2846553	308129			38.4442	
6	9.867	2251	146			0.0304	
6	12.548	4549865	205221			61.4483	
		7404374	511984			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 検出器番号=35 データ=AH3H171.D02 03/01/17 18:01:18
 サンプル名 : A-01
 ID : 40°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロマトグラム *** ファイル名:AH3H171.C02



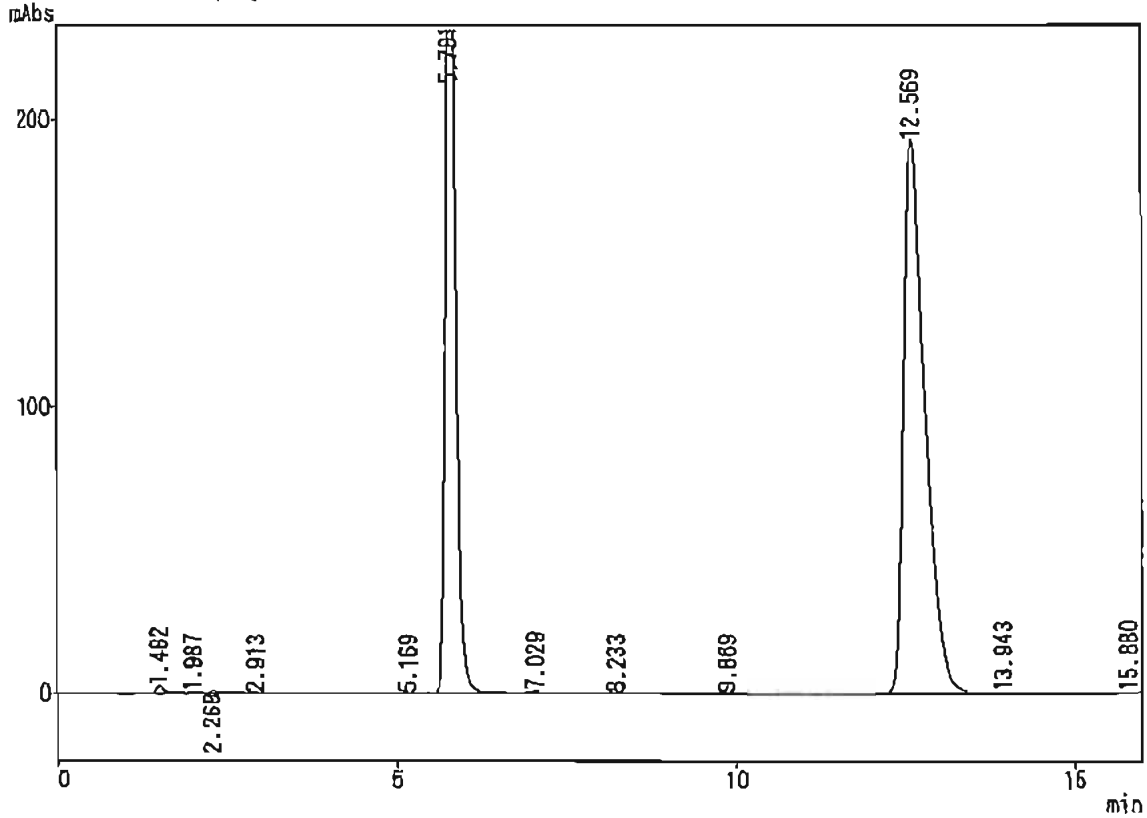
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	24769	2761	V		0.3433	
2	1.985	3260	295	V		0.0452	
3	2.269	12251	1138	V		0.1698	
4	2.912	2039	158	V		0.0283	
5	5.781	2872191	305372	S		39.8090	
6	7.031	9557	843	T		0.1325	
7	8.229	2437	175			0.0338	
8	9.869	3543	235			0.0491	
9	12.568	4275582	194154	S		59.2602	
10	13.946	6905	410	T		0.0957	
11	15.873	2399	175			0.0332	

7214933 505716 100.0000

サンプル : A-02
 ID : 40°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロマトグラム *** ファイル名:AH3H171.C03

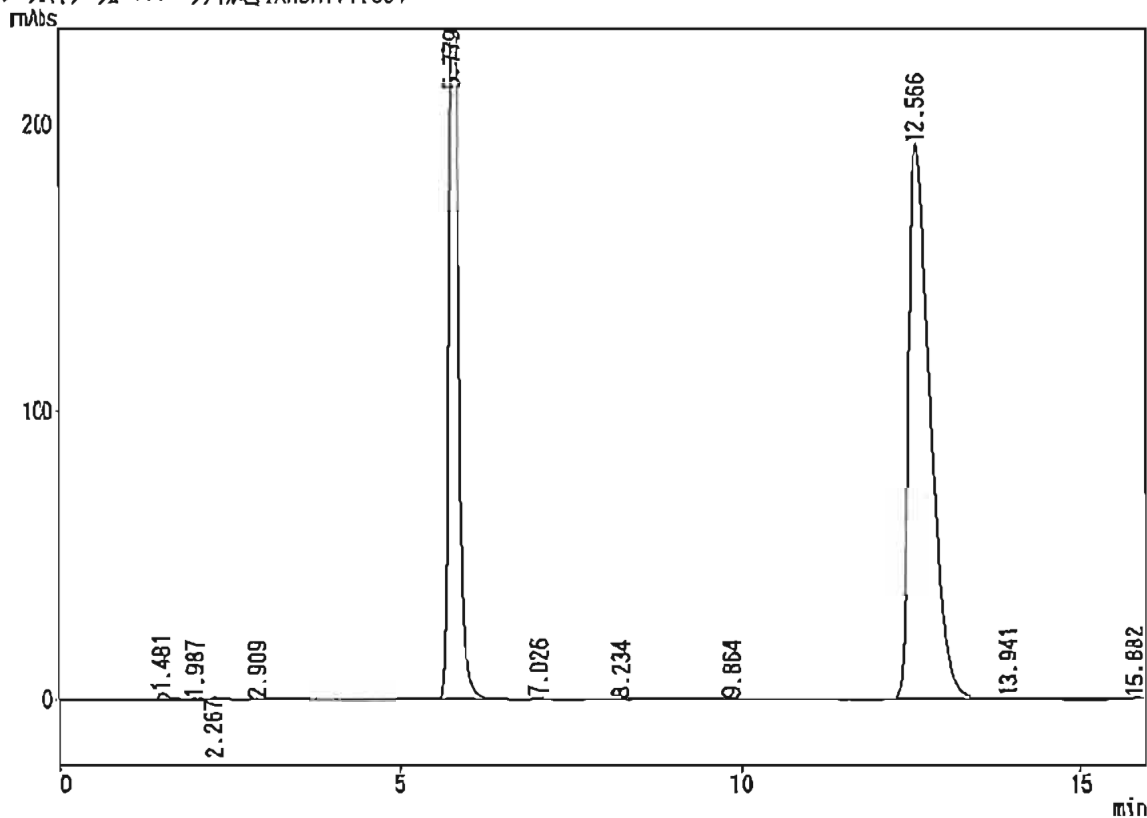


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	23146	2451	V		0.3225	
2	1.987	3457	291	V		0.0482	
3	2.268	10912	984	V		0.1521	
4	2.913	1941	150	V		0.0270	
5	5.169	2339	112			0.0328	
6	5.781	2866430	305085	SV		39.9444	
7	7.029	9798	870	T		0.1365	
8	8.233	2254	189			0.0314	
9	9.869	3656	240			0.0510	
10	12.569	4243563	193000	S		59.1351	
11	13.943	6425	381	T		0.0895	
12	15.880	2135	158			0.0298	
		7176053	503849			100.0000	

サンプル : A-03
 ID : 40°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カマトラム *** ファイル名:AH3H171.C04



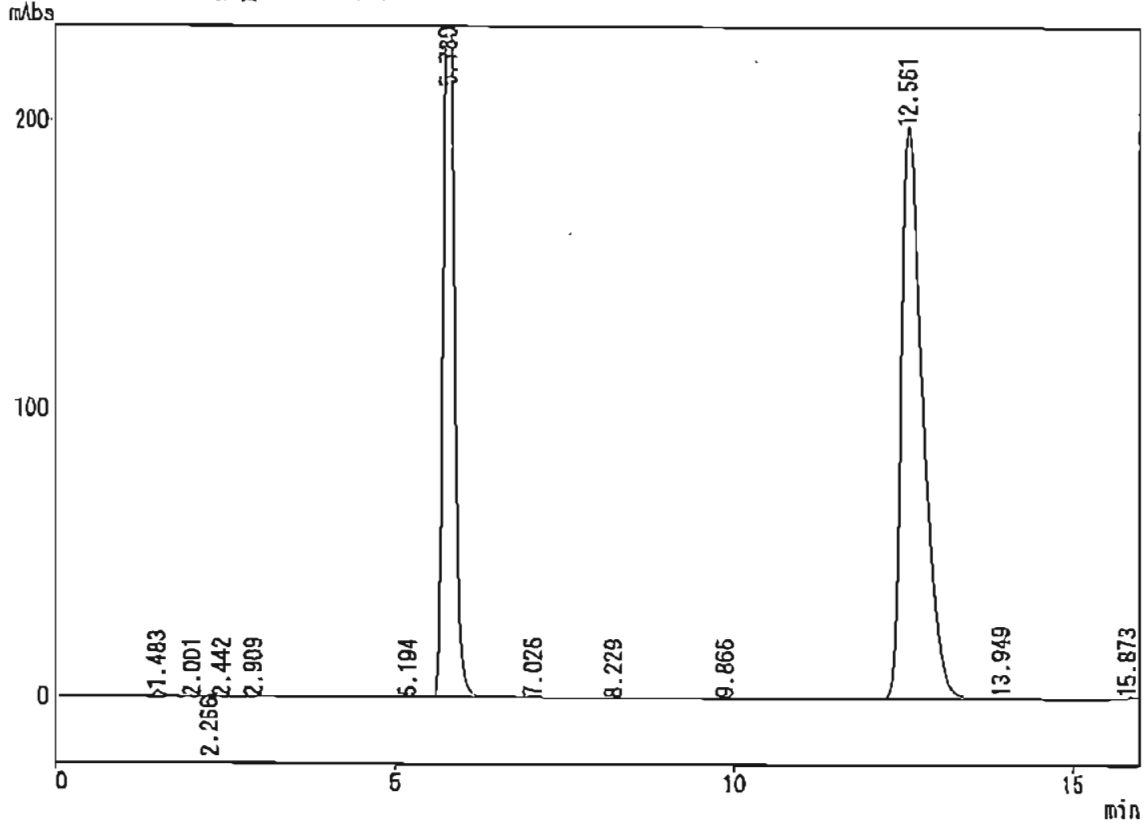
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	22004	2405	V		0.3070	
2	1.987	3113	277	V		0.0434	
3	2.267	9835	812	V		0.1372	
4	2.909	2135	154	V		0.0298	
5	5.779	2862118	305025	S		39.9298	
6	7.026	9443	837	T		0.1317	
7	8.234	2230	168			0.0311	
8	9.864	3755	242			0.0524	
9	12.566	4244241	192868	S		59.2120	
10	13.941	6633	393	T		0.0925	
11	15.882	2370	162			0.0331	

7167879 503443 100.0000

サンプル : A-04
 ID : 40°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 プリント名 : !AHR1028.MET

*** カロリグラム *** ファイル名:AH3H171.005

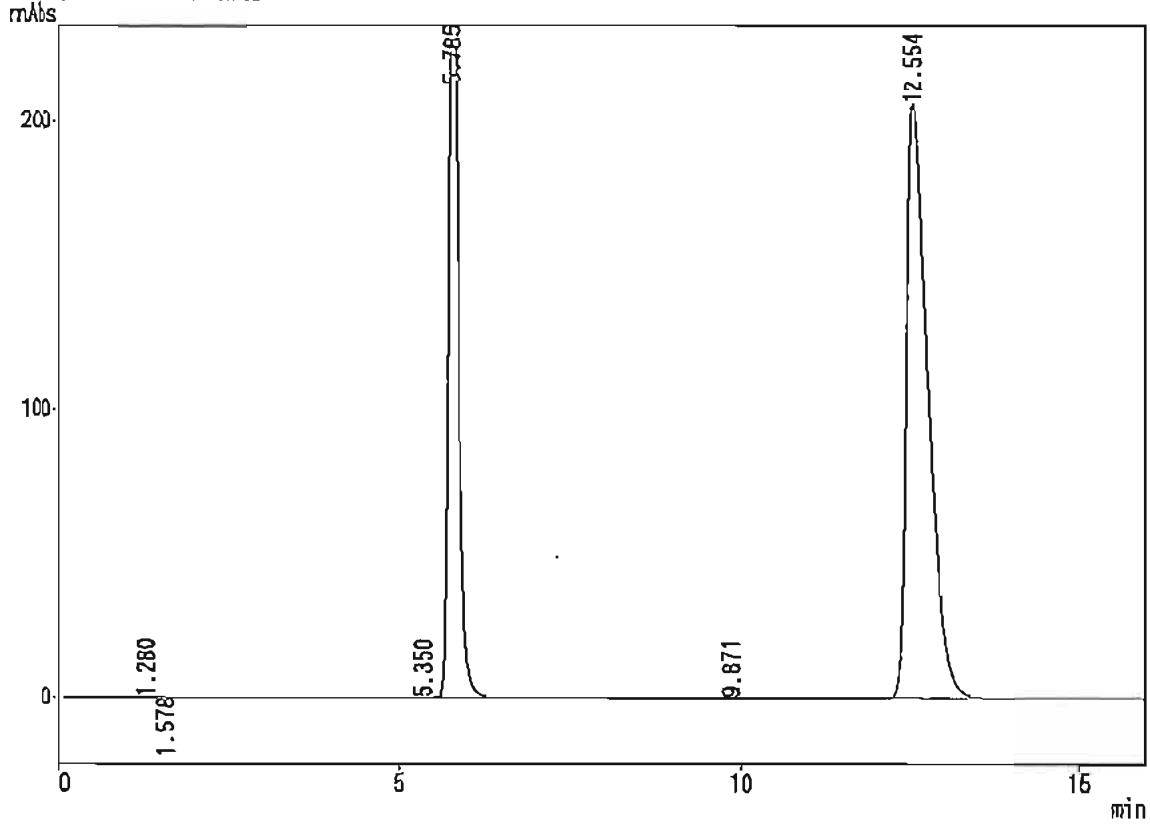


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.483	22252	2489	V		0.3039	
2	2.001	2292	185	V		0.0313	
3	2.266	5344	661	V		0.0730	
4	2.442	1877	201	V		0.0256	
5	2.909	1562	132	V		0.0213	
6	5.194	2724	128			0.0372	
7	5.780	2871808	305842	SV		39.2232	
8	7.026	5384	475	T		0.0733	
9	8.229	2204	164			0.0301	
10	9.866	3854	249			0.0526	
11	12.561	4386572	198560	S		59.9119	
12	13.949	13021	709	T		0.1778	
13	15.873	2832	214			0.0387	
		7321705	510007			100.0000	

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 漆 嗣郎
 メソッド名 : !AHR1028.MET

*** カマトグラム *** ファイル名:AH3H171.C08

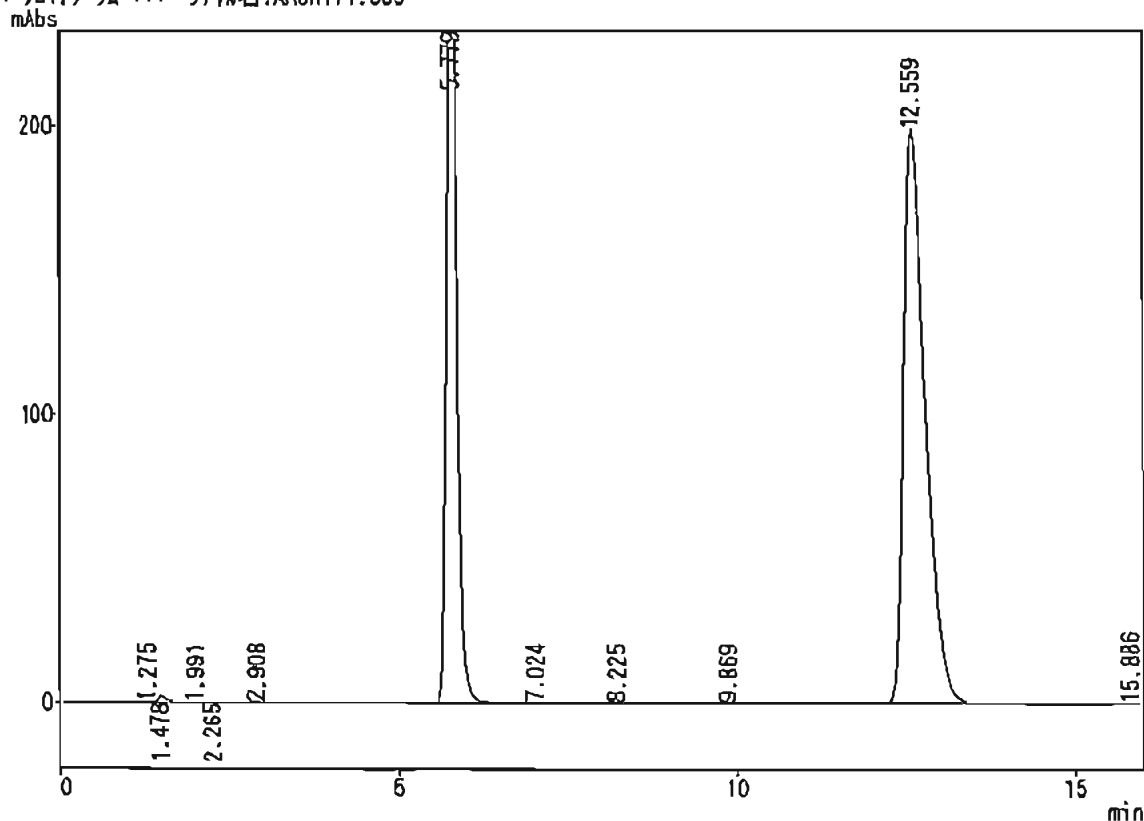


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.280	2421	137			0.0326	
2	1.578	2298	177	V		0.0309	
3	5.350	4420	176			0.0594	
4	5.785	2858747	306832	SV		38.4480	
5	9.871	2080	143			0.0280	
6	12.554	4565385	205732			61.4011	
		7435350	513197			100.0000	

サンプル : A-01
 ID : 40°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シックル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR102B.MET

*** カロマトグラム *** ファイル名:AH3H171.C09

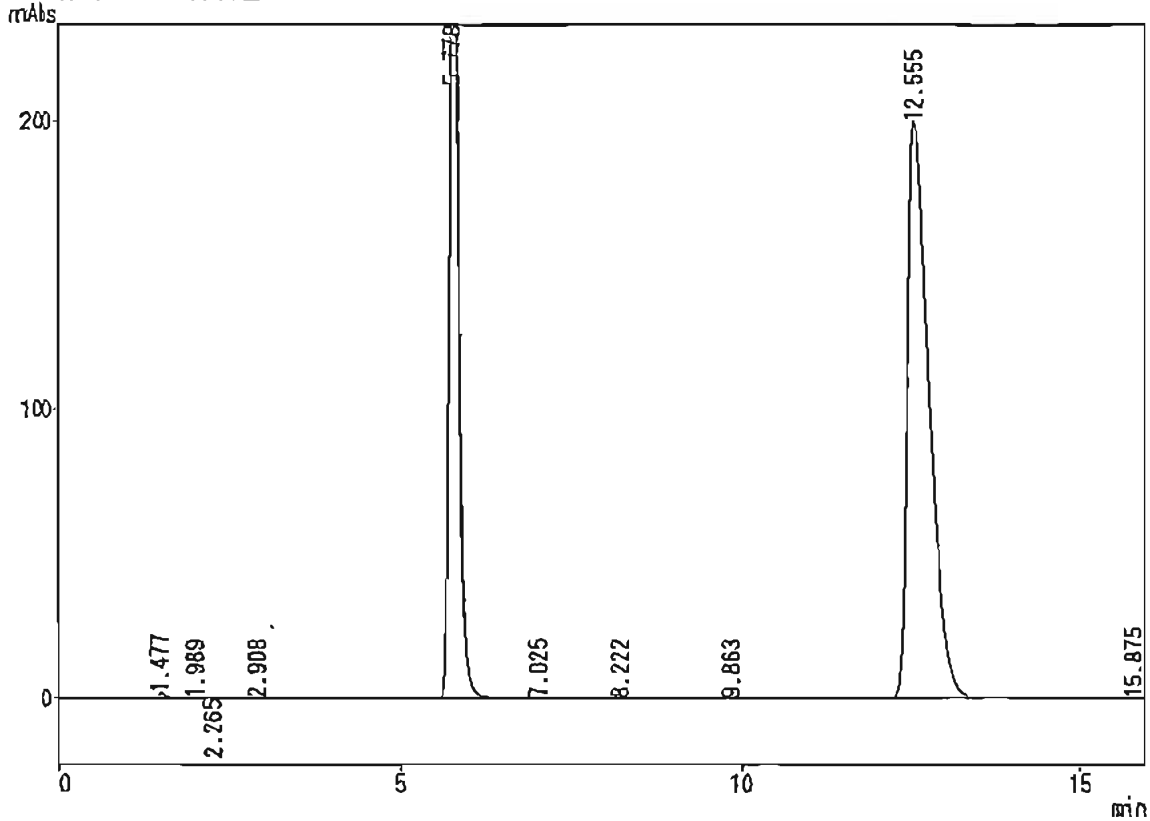


*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.275	1761	120			0.0242	
2	1.478	20171	2257	V		0.2768	
3	1.991	2144	209	V		0.0294	
4	2.265	6473	477	V		0.0888	
5	2.908	2853	299	V		0.0364	
6	5.779	2860103	304472	S		39.2444	
7	7.024	5470	487	T		0.0751	
8	8.225	2784	206			0.0382	
9	9.869	4035	268			0.0554	
10	12.559	4381017	199015			60.1134	
11	15.886	1310	90			0.0180	
		7287920	507899			100.0000	

サンプル : A-03
 ID : 40°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロリグラム *** ファイル名:AH3H171.C10

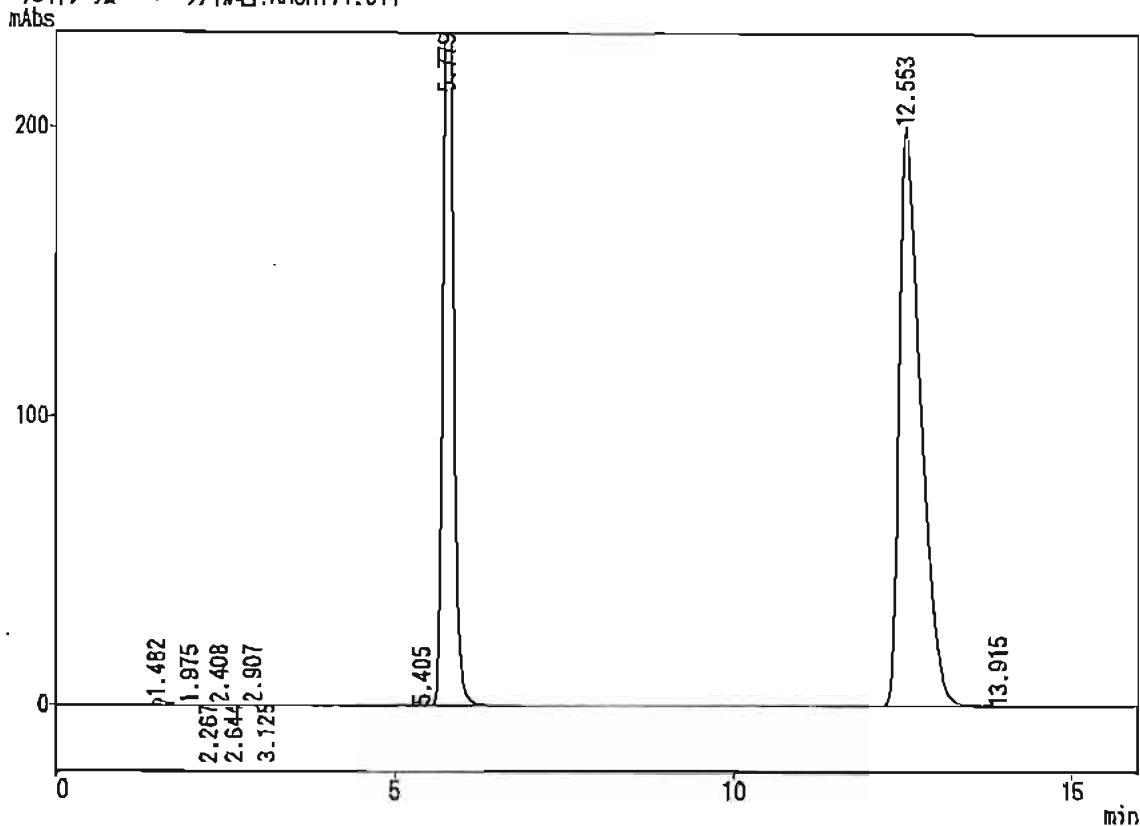


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.477	19859	2218	V		0.2717	
2	1.989	2205	226	V		0.0302	
3	2.265	6553	449	V		0.0896	
4	2.908	3188	322	V		0.0436	
5	5.778	2857141	304490	S		39.0859	
6	7.025	5594	496	T		0.0765	
7	8.222	2988	225			0.0410	
8	9.863	3790	249			0.0518	
9	12.555	4407379	200024			60.2933	
10	15.875	1193	90			0.0163	
		7309899	508789			100.0000	

サンプリング : 8F
 ID : 40°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 ソフト名 : !AHR1028.MET

*** カラム情報 *** ファイル名:AH3H171.C11



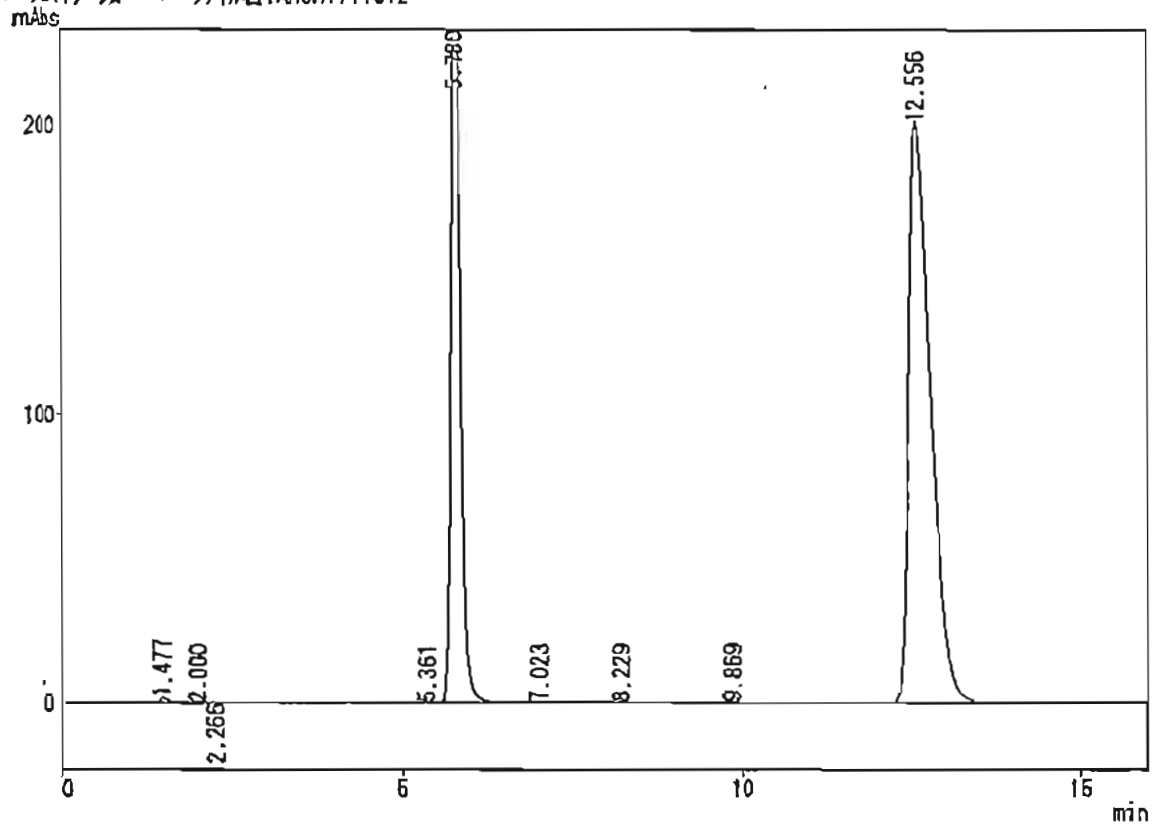
*** レポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	26094	2268	V		0.3544	
2	1.975	2029	250	V		0.0276	
3	2.267	3747	423	V		0.0509	
4	2.408	4698	560	V		0.0638	
5	2.644	1004	125	V		0.0136	
6	2.907	5257	673	V		0.0714	
7	3.125	1155	118	V		0.0157	
8	5.405	1033	78	V		0.0140	
9	5.779	2867651	305264	SV		38.9528	
10	12.553	4447937	201266	S		60.4186	
11	13.915	1261	77	T		0.0171	

7361865 511100 100.0000

サンプル : A-01
 ID : 25°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレーター名 : 澤 嗣郎
 メソッド名 : LAHR1028.MET

*** カロマトグラム *** ファイル名:AH3H171.C12

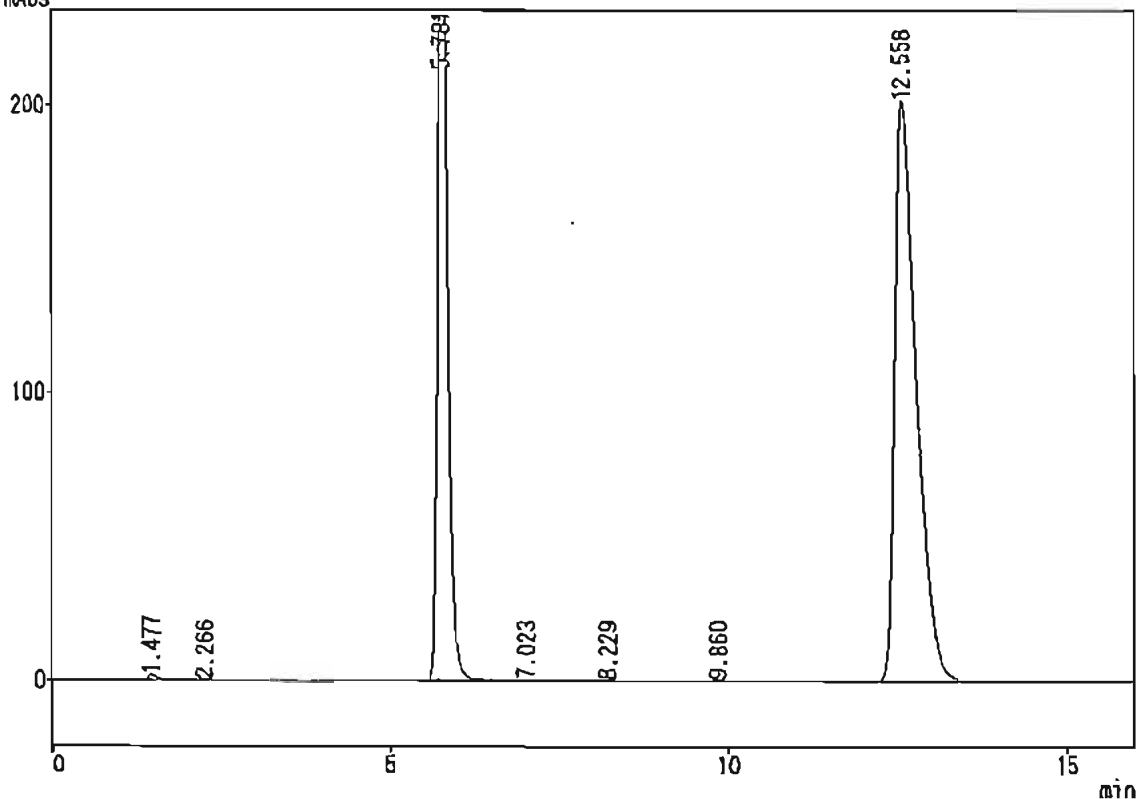


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.477	18139	1913	V		0.2462	
2	2.000	1561	139			0.0212	
3	2.266	3572	325	V		0.0485	
4	5.361	1580	74			0.0215	
6	5.780	2874184	306122	SV		39.0170	
6	7.023	3883	322	T		0.0500	
7	8.229	3994	295			0.0542	
8	9.869	4506	294	V		0.0612	
9	12.556	4455271	201390			60.4802	
		7366490	510873			100.0000	

サンプル : A-03
 ID : 25°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレーター名: 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロマトグラム *** ファイル名:AH3H171.C13
 mAbs

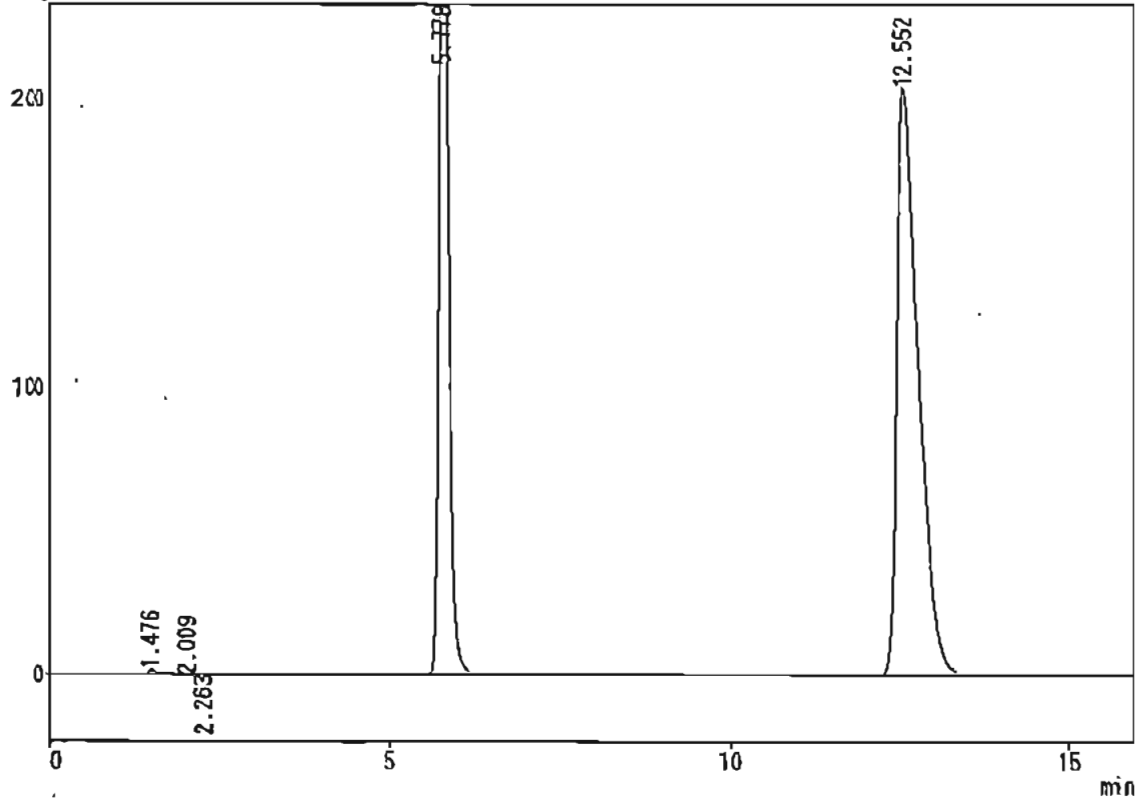


*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.477	17873	1959	SV		0.2428	
2	2.266	2682	287	V		0.0364	
3	5.781	2860635	304234	S		38.8852	
4	7.023	3983	348	T		0.0541	
5	8.229	3943	296			0.0536	
6	9.860	4420	292			0.0601	
7	12.558	4465866	201701			60.6878	
		7360402	609116			100.0000	

サンプル : BF
 ID : 25°C-2M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレーター名 : 澤 龍郎
 メソッド名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH3H171.C14
 mAbs

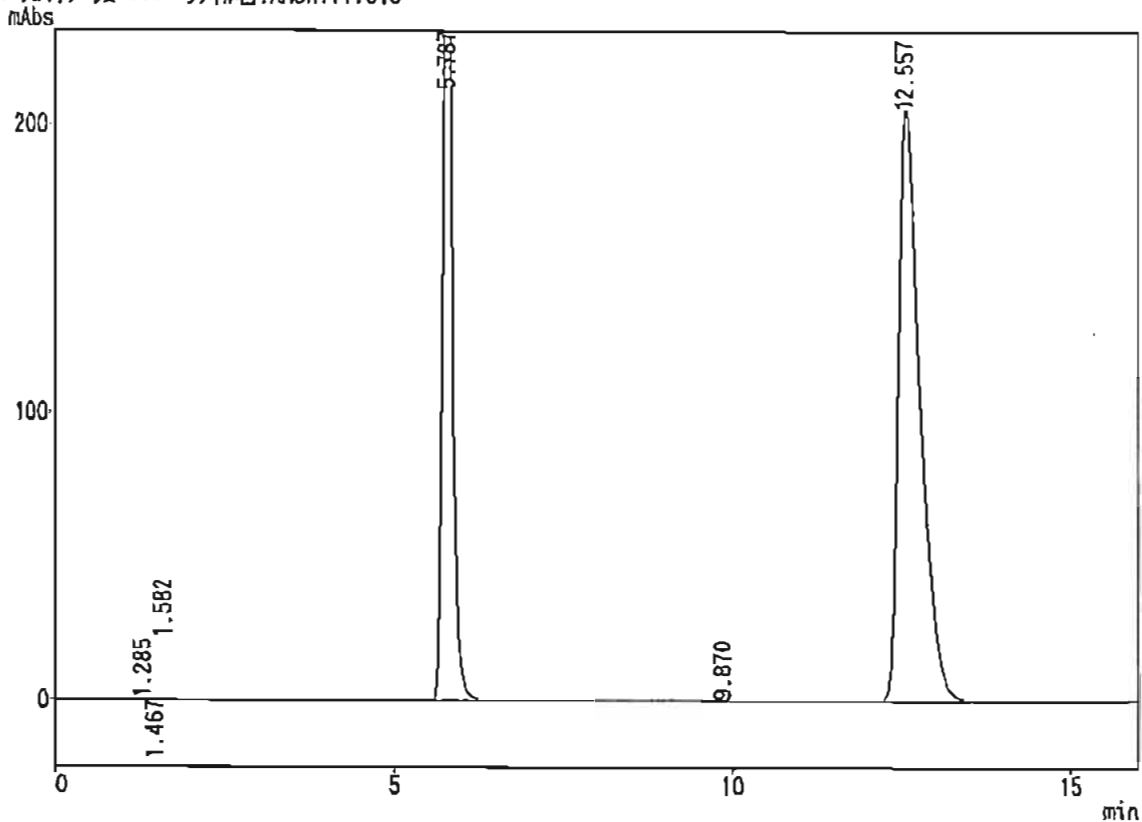


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.476	22158	2087	V		0.2982	
2	2.009	1033	130			0.0139	
3	2.263	4259	322	V		0.0573	
4	6.778	2882832	304977	V		38.5281	
5	12.552	4539904	204556			61.1025	
		7429886	512071			100.0000	

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレータ名 : 澤 嗣郎
 メソッド名 : IAHRT028.MET

*** カリブレーション *** ファイル名:AH3H171.C15



*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.285	2140	133			0.0289	
2	1.467	1141	140	V		0.0164	
3	1.582	2551	168	V		0.0344	
4	5.787	2852477	306429	S		38.4601	
5	9.870	2239	152			0.0302	
6	12.557	4556169	205242			61.4311	
		7416717	512264			100.0000	

プロムフェナクナトリウムの安定性
 Lot No.02X221

試験コード:P2002B131
 試験実施者: 澤 嗣郎
 試験実施日:2002年11月20日

ID	Chromato No.	AHR	Peak Area IS	Ratio	Cono. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	AH2Y20.C04	4677892	2806301	1.6869							
STD	AH2Y20.C08	4593438	2761658	1.6633							
STD	Mean			1.6851	0.10115						
A-01	60°C-4W AH2Y20.C05	4525804	2774228	1.6314	0.09910	97.70	83.61	8.23	—	—	4.19
A-03	60°C-4W AH2Y20.C06	4629549	2821284	1.6409	0.09968	99.58	95.07	8.21	—	—	4.53
BF	60°C-4W AH2Y20.C07	4474110	2789630	1.6038	0.09743	96.47	91.45	8.31	—	—	4.21

試験物質名	AHR10282B	試験コード	P2002B131	試験年月日	2002年11月20日	
試験項目					試験実施者	澤 嗣郎
IS P-ヒドロキシ安息香酸X凡 0.03354g		+移動相	→	100ml		
STD1	AHR10282B 0.02023g	+移動相	→	20ml		
STD2	STD1 2ml + IS 2ml	+移動相	→	20ml		
Sample	本品 2ml + IS 2ml	+移動相	→	20ml		

---20-Nov-2002--- 18:19:23
 001:N + 0.02023g
 002:N + 0.03354g

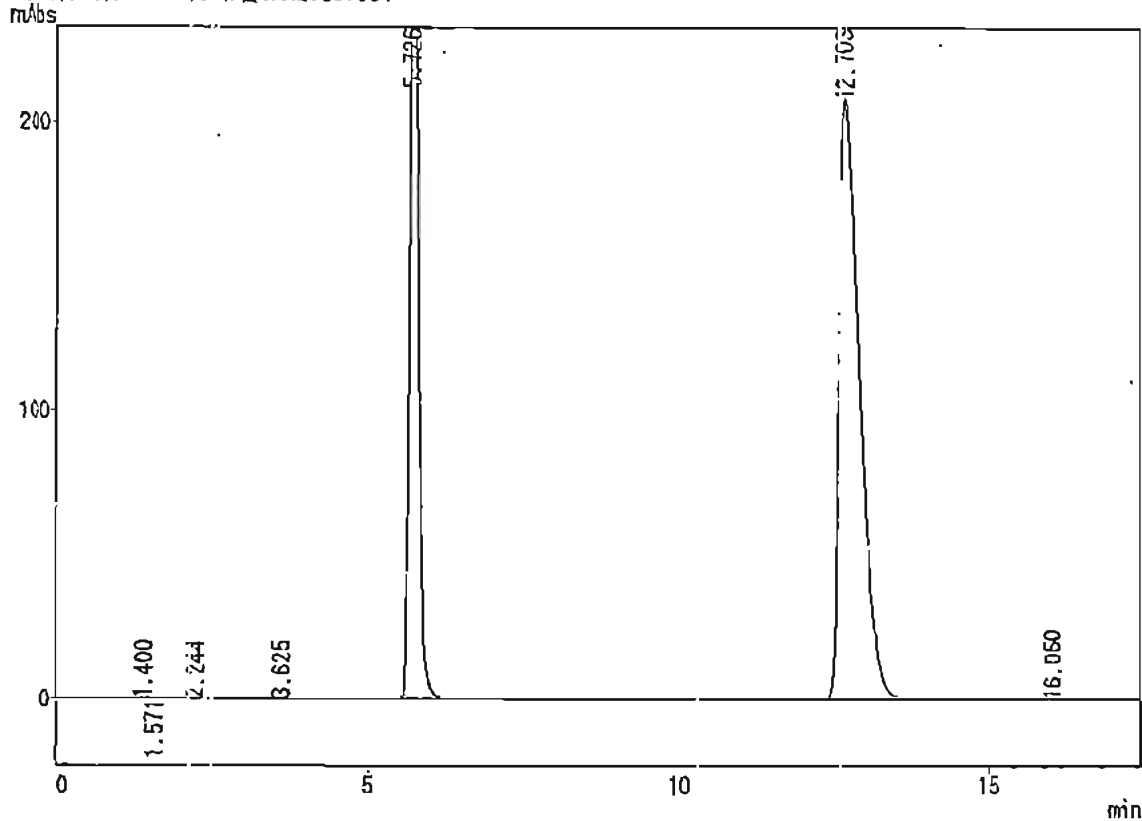
PH

A-05	40°C-4M	8.22	+	8.5299	8.4660
A-06	40°C-4M	8.19	+	8.5405	8.4808
A-01	60°C-4w	8.16	-	9.2414	9.0071
A-03	60°C-4w	8.16	-	9.2667	9.0125
BF	60°C-4w	8.21	-	9.3069	9.0687

11/20 20:02
 11/20 20:02
 11/20 20:07
 11/20 20:07
 11/20 20:07
 11/20 20:06

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレーター名 : 藤 嗣郎
 メソッド名 : IAHRT028.MET

*** カロリグラム *** ファイル名:AH2Y20.C04



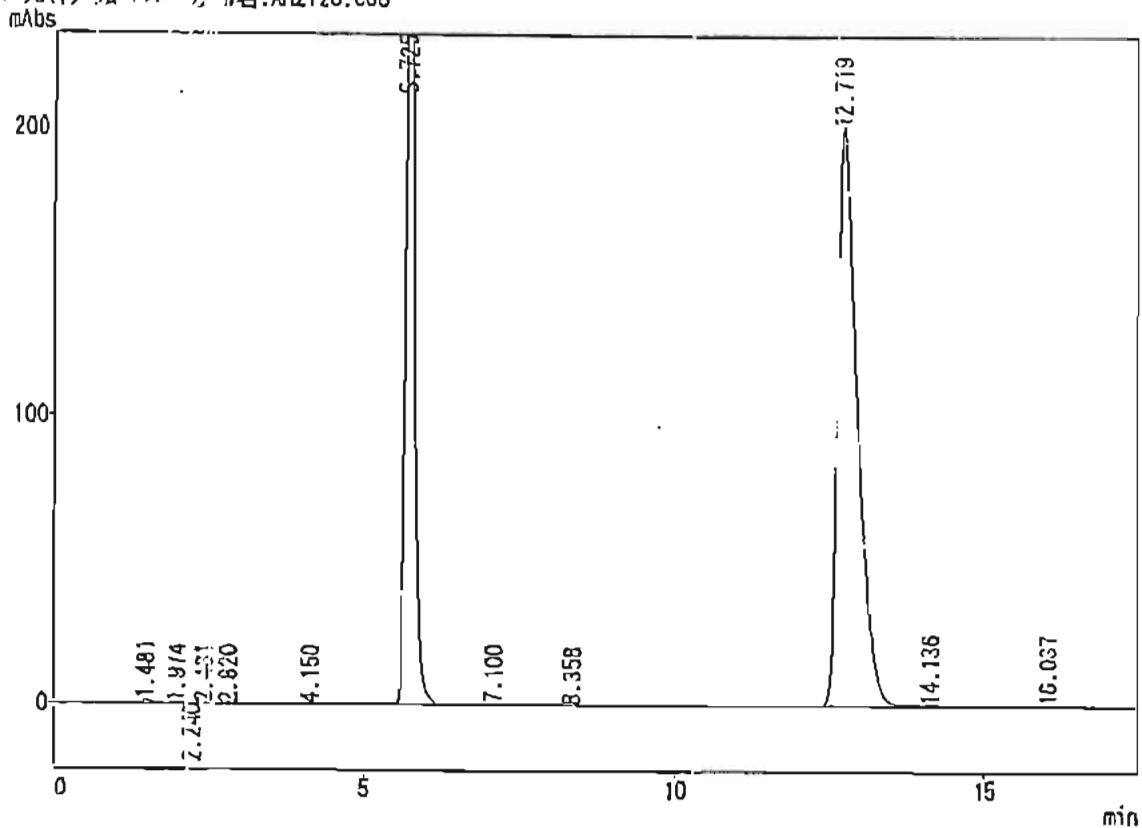
*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.400	2147	189	V		0.0216	
2	1.571	2105	207	V		0.0211	
3	2.244	1267	179	V		0.0109	
4	3.625	3761	124	V		0.0502	
5	5.726	2806301	306851			37.4440	
6	12.703	4577892	208851			62.4103	
7	16.050	1190	63			0.0119	

7494663 516045 100.0000

サンプル : A-01
 ID : 60°C-41j
 タイプ : 未知試料
 検出器 : SPD-10A 検出器
 ホール名 : 海 嗣郎
 サンプル名 : !AHR1028.MET

*** カラクリ *** データ名:AH2Y20.C06

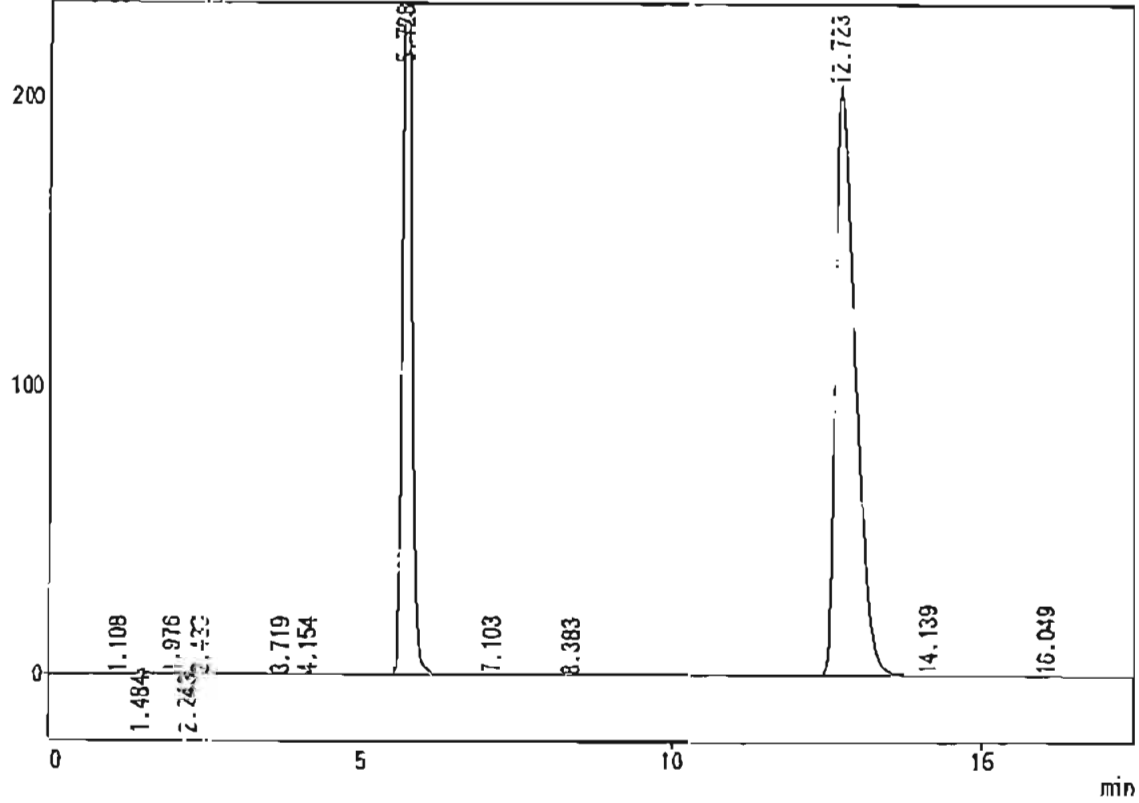


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	15097	1408	V		0.2062	
2	1.974	3630	324	V		0.0492	
3	2.240	3745	327	V		0.0509	
4	2.431	2645	208	V		0.0356	
5	2.820	2457	138	V		0.0334	
6	4.150	1760	229			0.0238	
7	5.725	2774228	299533	SV		37.6915	
8	7.100	6593	578	T		0.0816	
9	8.358	1237	93			0.0168	
10	12.719	4525804	201610	S		61.5079	
11	14.136	5843	348	T		0.0714	
12	16.037	15761	681			0.2142	
		7258801	505477			100.0000	

CLASS-LC10 Ver.=1.52 システム番号=1 Ch=1 ピーク番号=16 データ名=AH2Y20.C06 02/11/20 22:00:54
 サンプル : A-03
 ID : 60°C-4V
 タイプ : 未知試料
 検出器 : SPD-10A UV-210
 分析機名 : 藤 嗣郎
 ネット名 : LAHR1028.NET

*** カロリグラム *** ファイル名:AH2Y20.C06
 mAbs



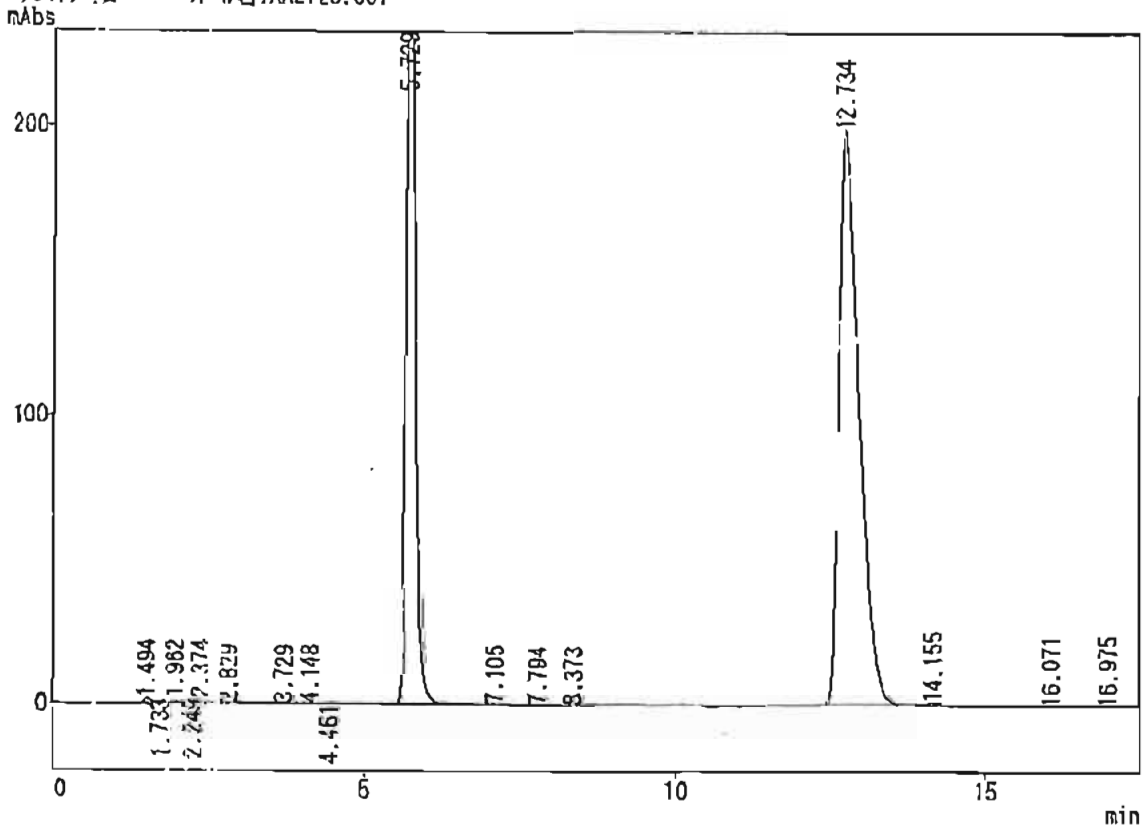
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.108	1020	107			0.0136	
2	1.484	14102	1313	V		0.1878	
3	1.976	3232	300	V		0.0431	
4	2.243	3048	274	V		0.0406	
5	2.433	1822	159	V		0.0216	
6	3.719	1247	57	V		0.0166	
7	4.154	1524	215			0.0203	
8	5.728	2021284	304786	SV		37.5788	
9	7.103	6664	588	T		0.0868	
10	8.383	1278	97			0.0170	
11	12.723	1929549	205249	S		61.6662	
12	14.139	5246	314	T		0.0868	
13	16.049	17628	748			0.2378	

7507440 514204 100.0000

サンプル : BF
 ID : 60°C-4W
 タイプ : 未知試料
 検出器 : SPD-10A 蛍光
 オペレーター名 : 澄 調郎
 ソフト名 : LAHR102B.MET

*** カマトク法 *** ファイル名:AH2Y20.C07



*** ヒートマップ ***

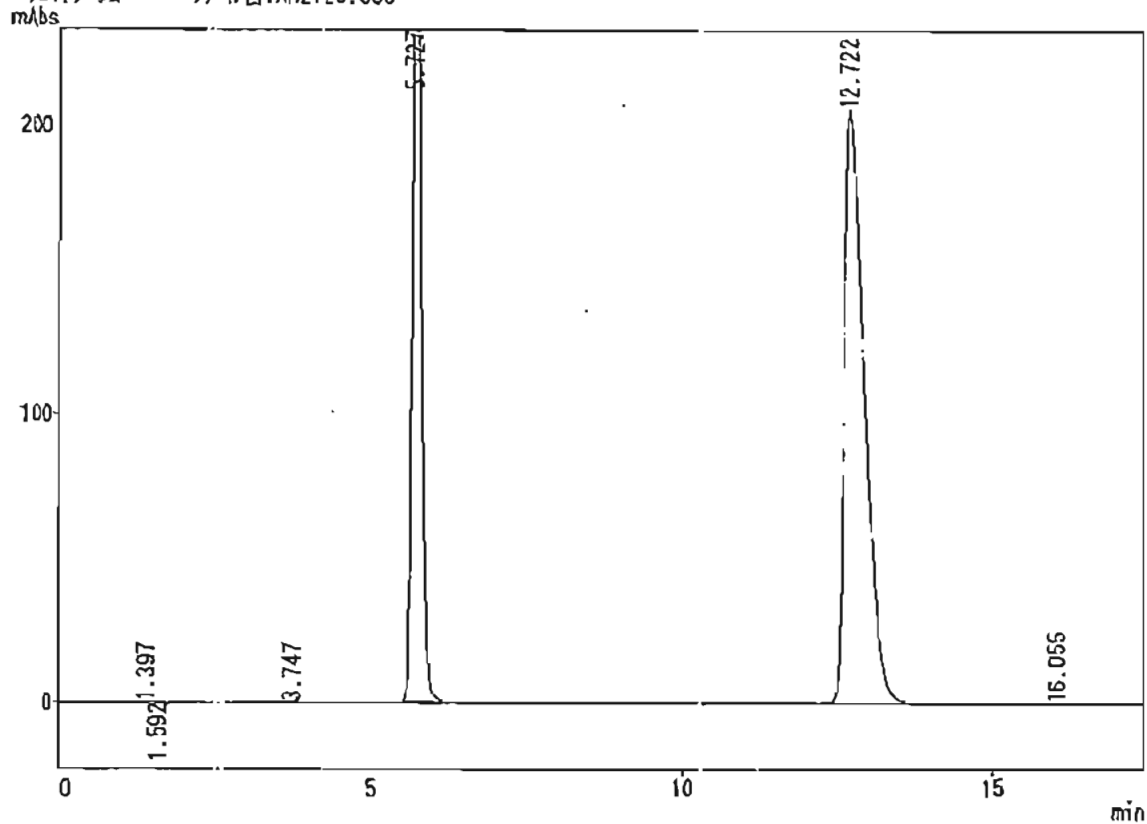
PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.494	17779	1628	V		0.2423	
2	1.733	4220	518	V		0.0575	
3	1.962	5283	536	V		0.0720	
4	2.219	2021	280	V		0.0275	
5	2.374	3938	349	V		0.0537	
6	2.829	1670	125	V		0.0228	
7	3.729	1250	58			0.0170	
8	4.148	2329	322			0.0317	
9	4.451	1789	236			0.0214	
10	5.729	2739630	301768	S		38.0146	
11	7.105	3387	305	T		0.0462	
12	7.704	1309	120			0.0178	
13	8.373	1585	120			0.0216	
14	12.734	4474110	199580	S		60.9692	
15	14.155	23417	1200	T		0.3191	
16	16.071	2688	131			0.0366	
17	16.975	1905	88			0.0260	

7238311 507357 100.0000

CLASS-LC10 Ver.=1.52 システム番号=1 Ch=1 レポート番号=18 テキスト=AH2Y;0.008 02/11/20 22:39:48

サンプル名 : STD
ID :
タイプ : 未知試料
検出器 : SPD-10A システム
オペレーター名 : 渡 嗣郎
プリント名 : LAHR1028.MET

*** カリブレーション *** ファイル名:AH2Y20.C08



*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.397	1077	142	V		0.0146	
2	1.592	1020	123	V		0.0138	
3	3.747	8288	264			0.1125	
4	5.727	2761658	303946	S		37.4870	
5	12.722	4593438	206133	S		62.3577	
6	18.055	1503	72			0.0204	

T:66983 510680 100.0010

プロムフェナクナトリウムの安定性
Lot No.02X221

試験コード:P2002B131
試験実施者: 澤 野郎
試験実施日:2002年10月22日

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	AH2X22.C01	4613938	3082770	1.4967							
STD	AH2X22.C05	4603915	3072549	1.4984							
STD	Mean			1.4976	0.10015						
A-01	Initial	AH2X22.C02	4631972	3067148	1.5167	0.10143	100.00	100.00	8.20	—	—
A-03	Initial	AH2X22.C03	4625716	3090209	1.4969	0.10010	100.00	100.00	8.27	—	—
BF	Initial	AH2X22.C04	4685202	3057080	1.5260	0.10205	100.00	100.00	8.20	—	—

試験物質名	AHR10282B	試験コード	P2002B131	試験年月日	2002年10月22日
試験項目				試験実施者	澤 嗣郎
IS:	p-トウモロコシ酸x11L	0.01799g + 移動相	→	50ml	22-Oct-2002 - 16:12:55
STD1:	AHR10282B	0.02003g + 移動相	→	20ml	003: N + 0.01799g
STD2:	STD1 2ml + IS 2ml	+ 移動相	→	20ml	
Sample	本品 2ml + IS 2ml	+ 移動相	→	20ml	22-Oct-2002 16:04:42

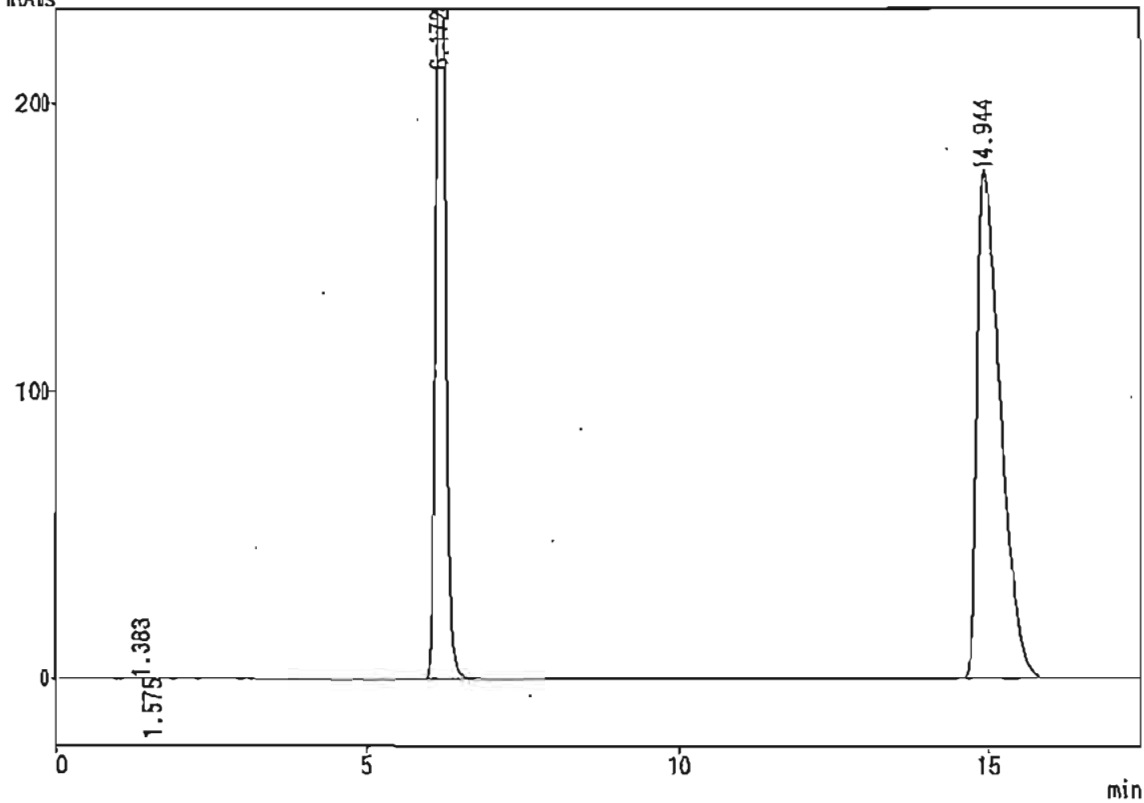
DO1:	H	+ 0.02003g	
------	---	------------	--

	pH	外觀
A-01 initial	8.20	—
A-03 ↓	8.27	—
BF	8.20	—

10/22 17:47	NO.39	pH 8.22	24.5°C
10/22 17:48	NO.40	pH 8.27	24.7°C
10/22 17:49	NO.41	pH 8.20	24.9°C

サンプル名 : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレーター名 : 藤 嗣郎
 サンプル名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH2X22.C01
 mAbs

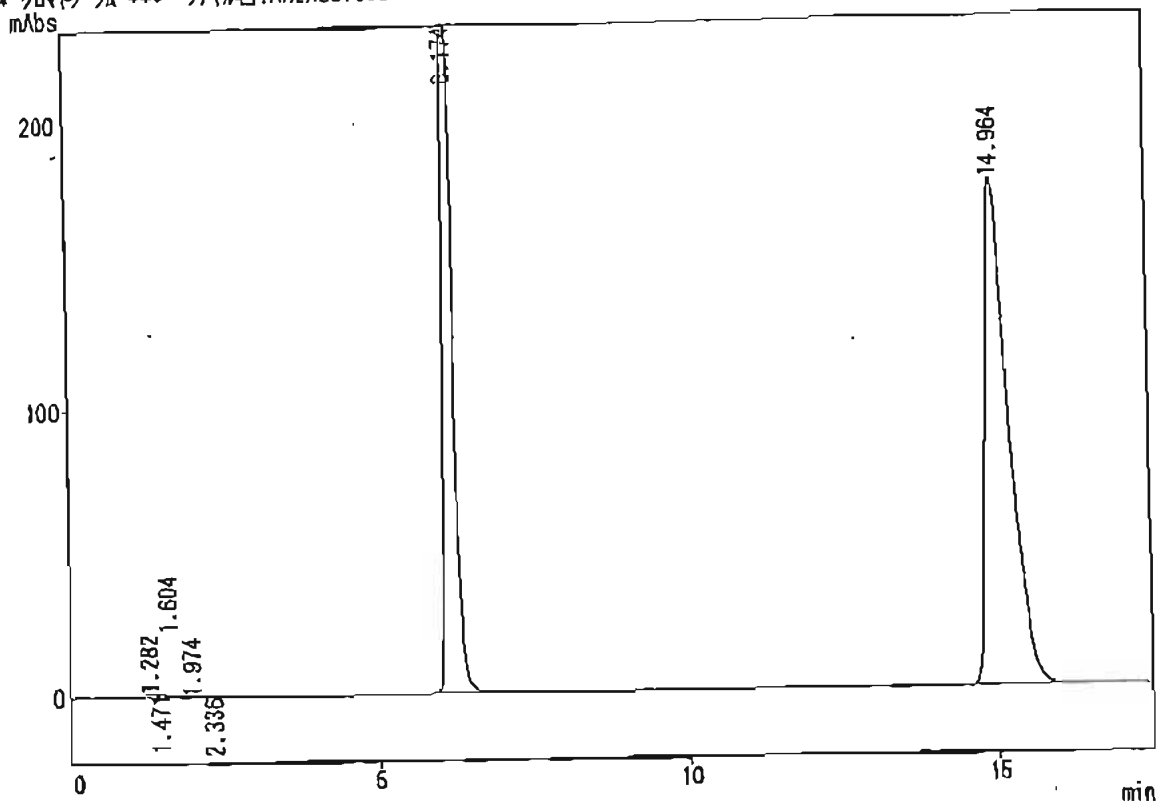


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.383	2041	135			0.0265	
2	1.575	3334	216	V		0.0433	
3	6.172	3082770	321671			40.0251	
4	14.944	4613938	176966			59.9051	
		7702083	498989			100.0000	

サンプル : A-01
 ID : Initial
 タイプ : 未知試料
 検出器 : SPD-10A シカール
 オペレーター名 : 漆 嗣郎
 サンプル名 : IAHRT02B.MET

*** クロマトグラム *** ファイル名:AH2X22.C02

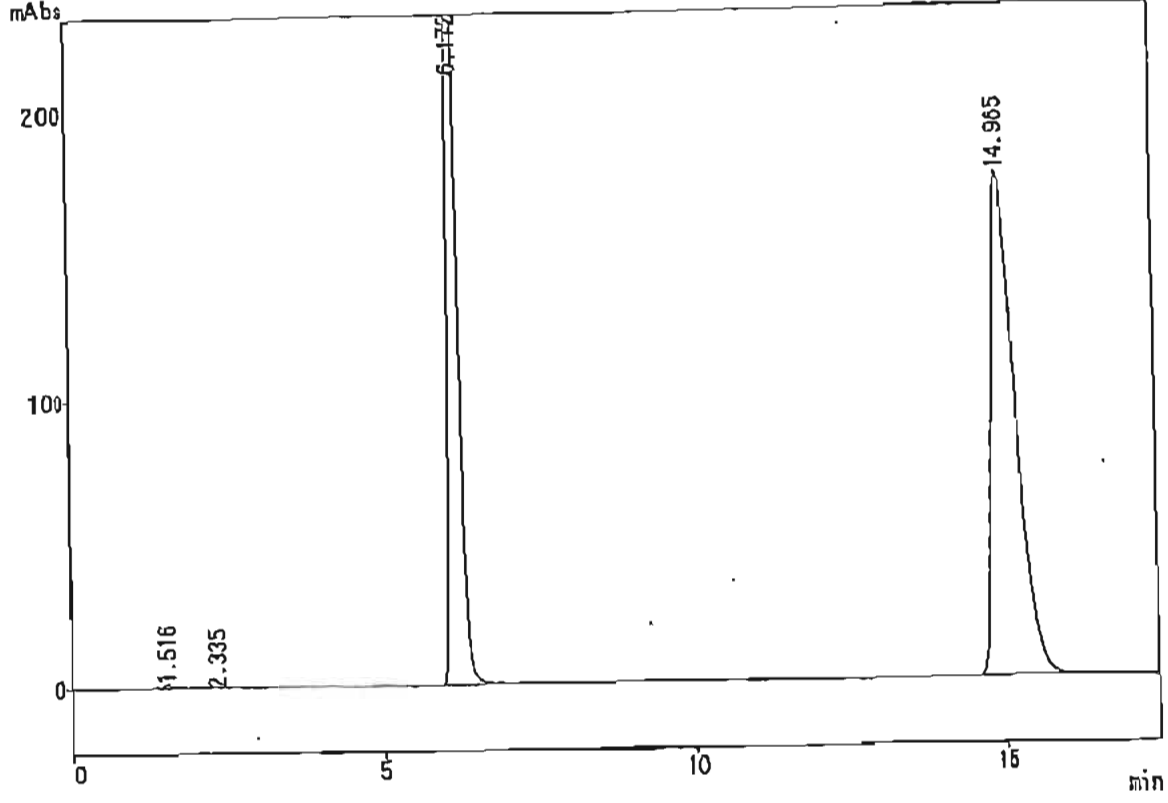


*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.282	18032	1319			0.2325	
2	1.471	7987	1140	V		0.1030	
3	1.604	8077	815	V		0.1041	
4	1.974	1025	104	V		0.0132	
5	2.338	1824	177	V		0.0235	
6	6.174	3087148	314342	S		39.5452	
7	14.964	4651972	176635			59.9785	
		7756064	494533			100.0000	

サンプル : A-03
 ID : Initial
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレータ名 : 澤 嗣郎
 メソッド名 : IHR1028.MET

*** クロマトグラム *** ファイル名:AH2X22.C03

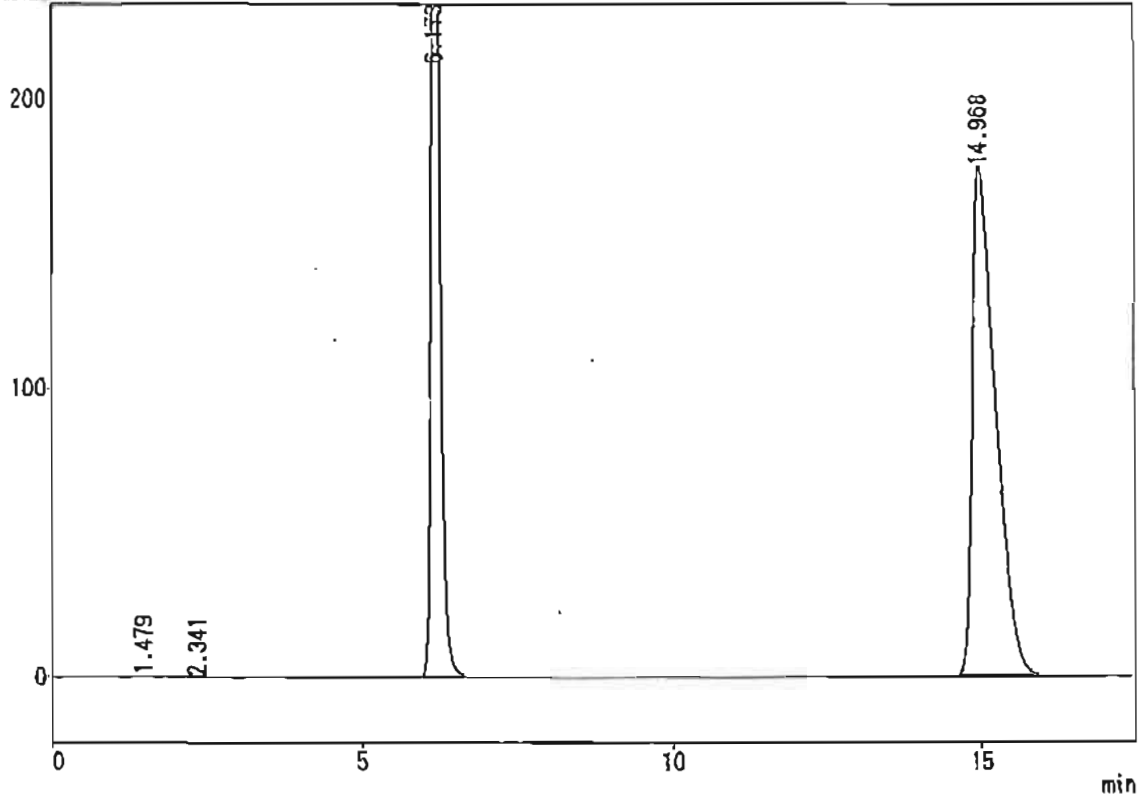


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.516	15418	1678	SV		0.1994	
2	2.335	1550	169	V		0.0200	
3	6.172	3090209	317009			39.9819	
4	14.965	4826716	175708			59.8187	
		7732891	494563			100.0000	

サンプル : BF
 ID : Initial
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレータ名 : 澤 嗣郎
 メソッド名 : LAHR1028.MET

*** クロマトグラム *** ファイル名: AH2X22.C04
 mAbs

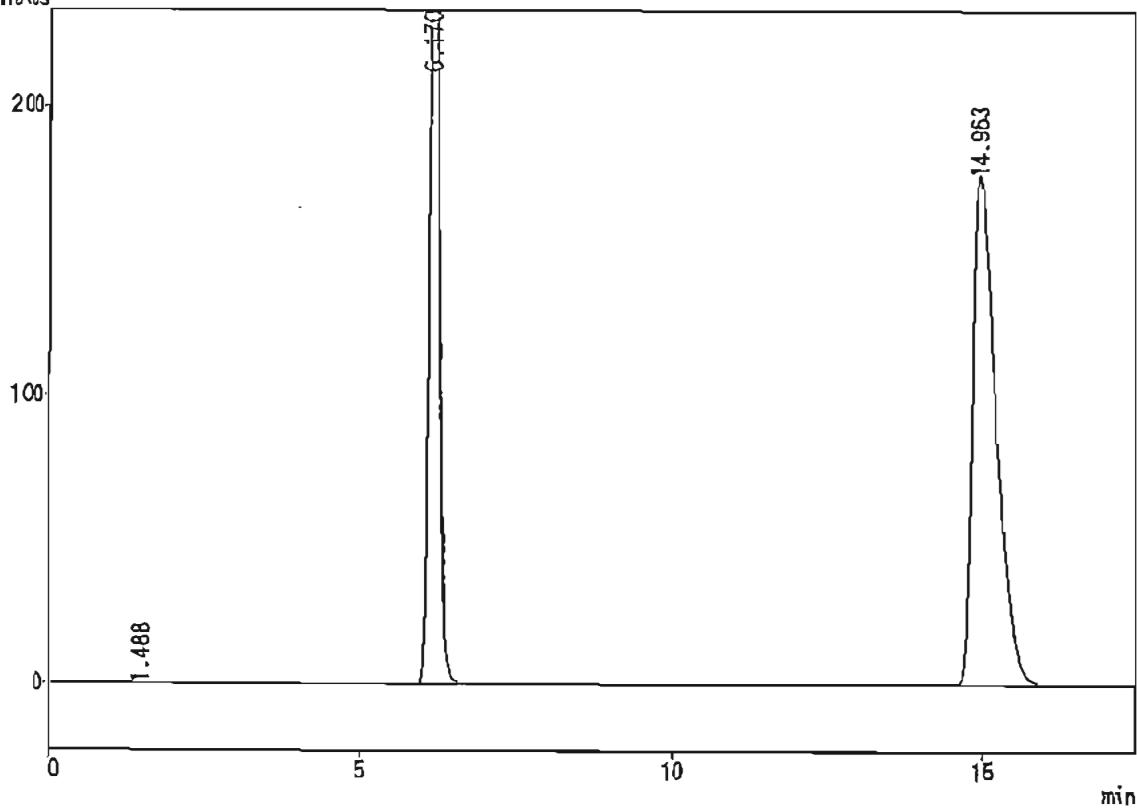


*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.479	6870	711	V		0.0889	
2	2.341	1764	147	V		0.0228	
3	6.173	3057080	313853			39.5438	
4	14.968	4665202	176971			60.3448	
		7730915	491482			100.0000	

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレーター名 : 澤 嗣郎
 メソッド名 : !AHR102B.MET

*** カリブレーション *** ファイル名:AH2X22.C05
 mAbs



*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.488	4818	233	V		0.0627	
2	6.170	3072549	320993			40.0005	
3	14.963	4603915	176570			59.9368	
		7681282	497796			100.0000	

2

プロムフェナクナトリウムの安定性
Lot No.02X221

試験コード: P2002B131
試験実施者: 澤 嗣郎
試験実施日: 2002年11月13日

ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid	Permeation (%)
STD	Mean	AH2Y13.C13	4579463	2806042	1.6320	0.10015					
A-01	60°C-2W	AH2Y13.C14	4525901	2800112	1.6163	0.09919	97.79	94.33	8.23	—	3.54
A-03	60°C-2W	AH2Y13.C15	4518470	2783708	1.6232	0.09981	99.51	95.98	8.21	—	3.55
BF	60°C-2W	AH2Y13.C16	4452342	2778799	1.6023	0.09833	96.35	92.97	8.31	—	3.61

試験物質名: AHR10282B 試験コード: P2002B131 試験年月日: 2002年11月12日

試験項目: 試験実施者: 澤 嗣郎

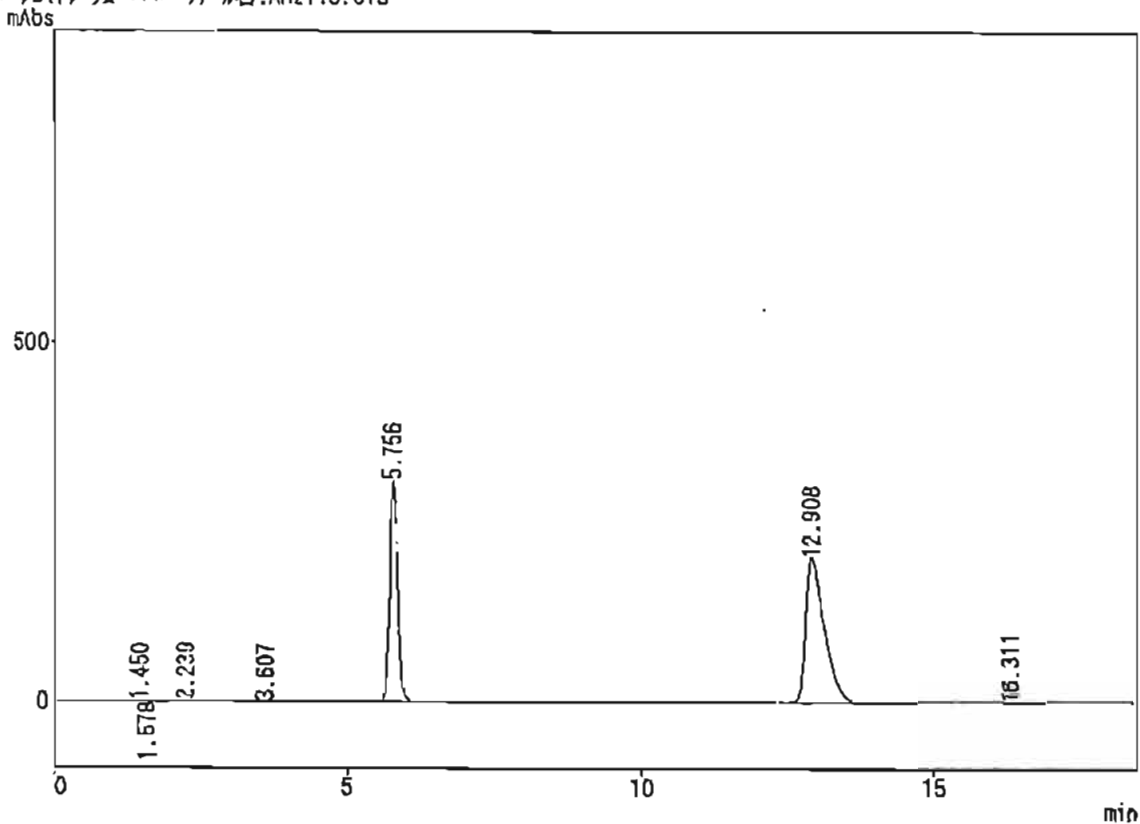
IS p-ヒドロキシ安息香酸メチル 0.03354g + 移動相 → 100ml
 STD1 7-ロウレンチウム 0.01996g + 移動相 → 20ml
 STD2 STD1 2ml + IS 2ml + 移動相 → 20ml
 Sample 本品 2ml + IS 2ml + 移動相 → 20ml

		pH	外観	重量変化	
A-01	60°C-2W	8.23	--	9.3101	9.1097
A-03	60°C-2W	8.21	--	9.2939	9.0934
BL	60°C-2W	8.31	--	9.3582	9.1580

11/12 15:04
 NO.47 8.27
 20.00
 11/12 15:35
 NO.48 8.21
 20.00
 11/12 15:04
 NO.45 8.31
 20.00

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 ホスト名 : 濠 嗣郎
 メソッド名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH2Y13.C13



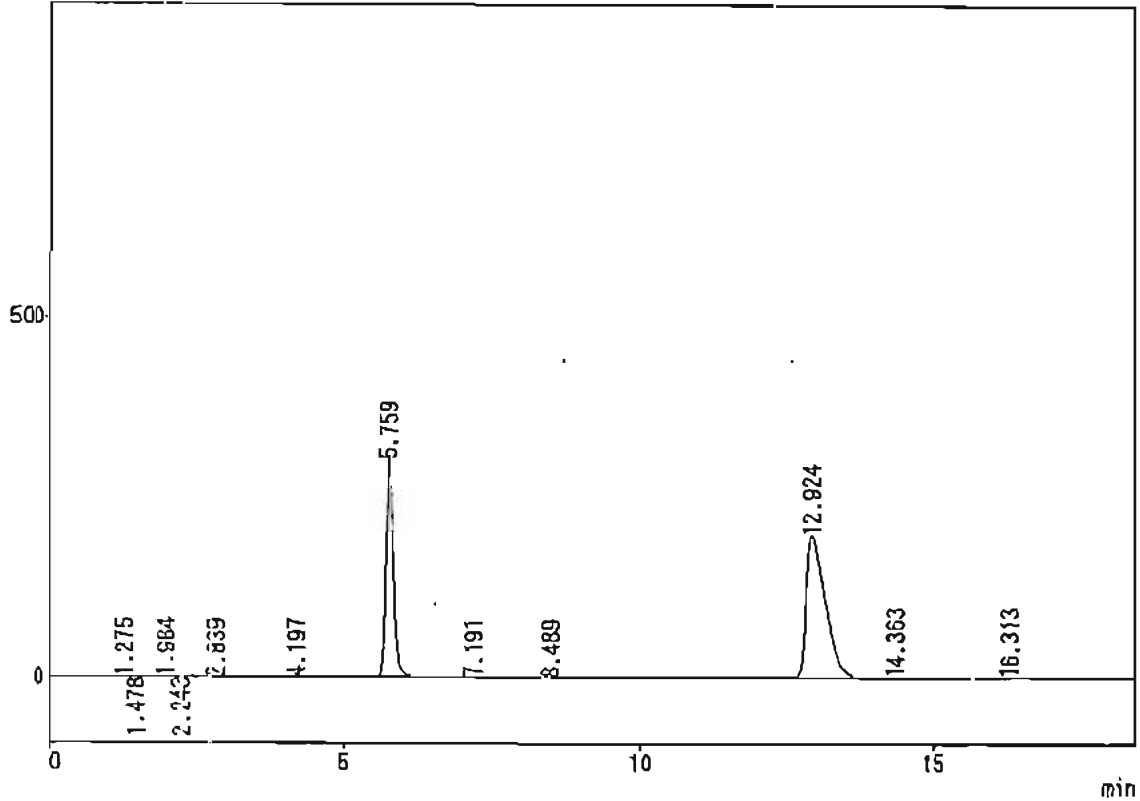
*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.450	2039	152	V		0.0275	
2	1.578	1432	174	V		0.0193	
3	2.239	8772	1145	SV		0.1185	
4	3.607	4643	152			0.0814	
5	5.756	2806042	308206			37.9009	
6	12.908	4579483	203270	S		61.8543	
7	16.311	1338	67			0.0181	
		7403629	513165			100.0000	

CLASS-LC10 Ver.=1.32 システム番号=1 Ch=1 レポート番号=89 テータ=AH2Y13.D14 (02/11/13 18:53:46)

サンプル : A-01
 ID : 60°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレータ名 : 深 崎郎
 プリント名 : LAHR1028.MET

*** カマトラム *** ファイル名:AH2Y13.C14
 mAbs

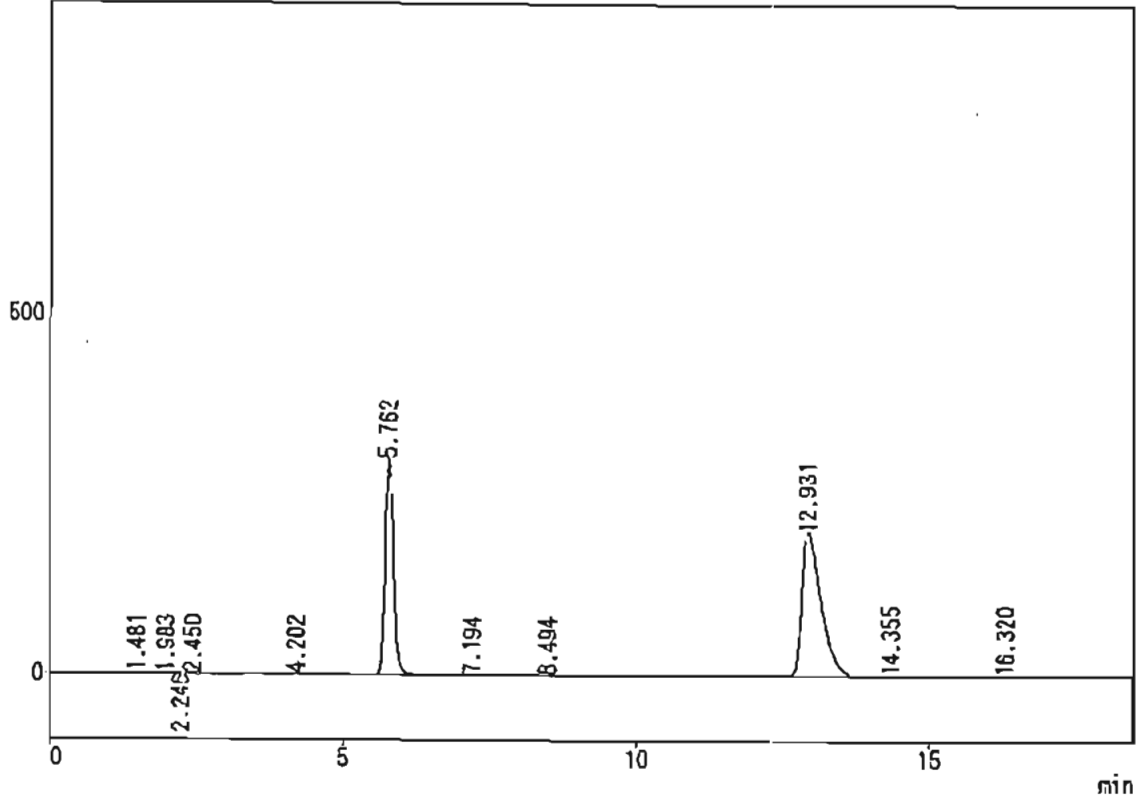


*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	PK	IDNO	CONC	NAME
1	1.275	1843	145			0.0249	
2	1.478	16100	1534	V		0.2180	
3	1.984	3388	298	V		0.0459	
4	2.243	11878	1147	V		0.1608	
5	2.839	1298	96	V		0.0176	
6	4.197	1489	191			0.0202	
7	5.759	2800112	300917	S		37.9061	
8	7.191	5852	502	T		0.0792	
9	8.489	2302	165			0.0312	
10	12.924	4525901	198754	S		61.2687	
11	14.363	3920	237	T		0.0531	
12	16.313	12883	544			0.1744	
		7386987	504530			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=90 テーマ=AH2Y13.D15 02/11/13 19:13:46
 サンプル : A-03
 ID : 60°C-2W
 タイプ : 未知試料
 検出器 : SPD-10A シンチレーション
 オペレータ名: 澤 嗣郎
 サンプル名: !AHR1028.MET

*** カロリグラム *** ファイル名:AH2Y13.C15
 mAbs



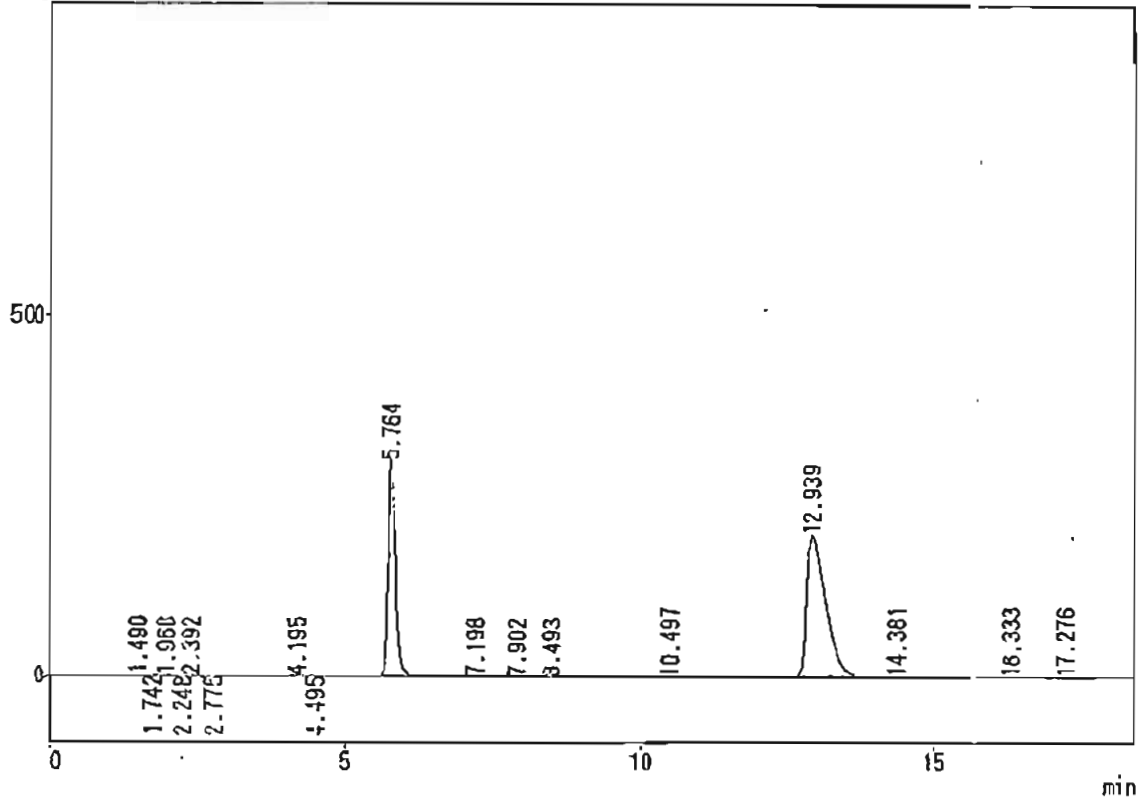
*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	14369	1389	V		0.1954	
2	1.983	3016	287	V		0.0410	
3	2.248	8669	831	V		0.0907	
4	2.450	1815	179	V		0.0247	
5	4.202	1308	176			0.0178	
6	5.762	2783708	289623	S		37.8506	
7	7.194	5833	506	T		0.0793	
8	8.494	2516	178			0.0342	
9	12.931	4518470	198351	S		61.4385	
10	14.355	3682	216	T		0.0498	
11	16.320	13102	560			0.1781	
		7354464	502286			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=91 レポート名=AH2Y13.D16 02/11/13 19:33:44

サンプル名 : BF
 ID : 60°C-2H
 タイプ : 未知試料
 検出器 : SPD-10A システム名
 サンプル名 : 澤 嗣郎
 サンプル名 : !AHR1028.MET

*** カラム *** カラム名:AH2Y13.C16
 mAbs



*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.490	17354	1547	V		0.2379	
2	1.742	3587	466	V		0.0492	
3	1.968	4314	472	V		0.0591	
4	2.248	5502	744	V		0.0754	
5	2.392	4160	392	V		0.0570	
6	2.775	1153	95	V		0.0158	
7	4.195	2027	278			0.0278	
8	4.495	1919	244			0.0263	
9	5.764	2778799	299605			38.0910	
10	7.198	2364	209			0.0324	
11	7.902	1173	107			0.0161	
12	8.493	1552	114			0.0213	
13	10.497	1005	79			0.0138	
14	12.939	4452342	196001	S		61.0327	
15	14.381	14233	730	T		0.1951	
16	16.333	1856	88			0.0254	
17	17.276	1674	77			0.0229	

7295013 501326 100.0000

プロムフェナクナトリウムの安定性
Lot No.:02X221

試験コード:P2002B131
試験実施者: 澤 嗣郎
試験実施日:2003年04月22日

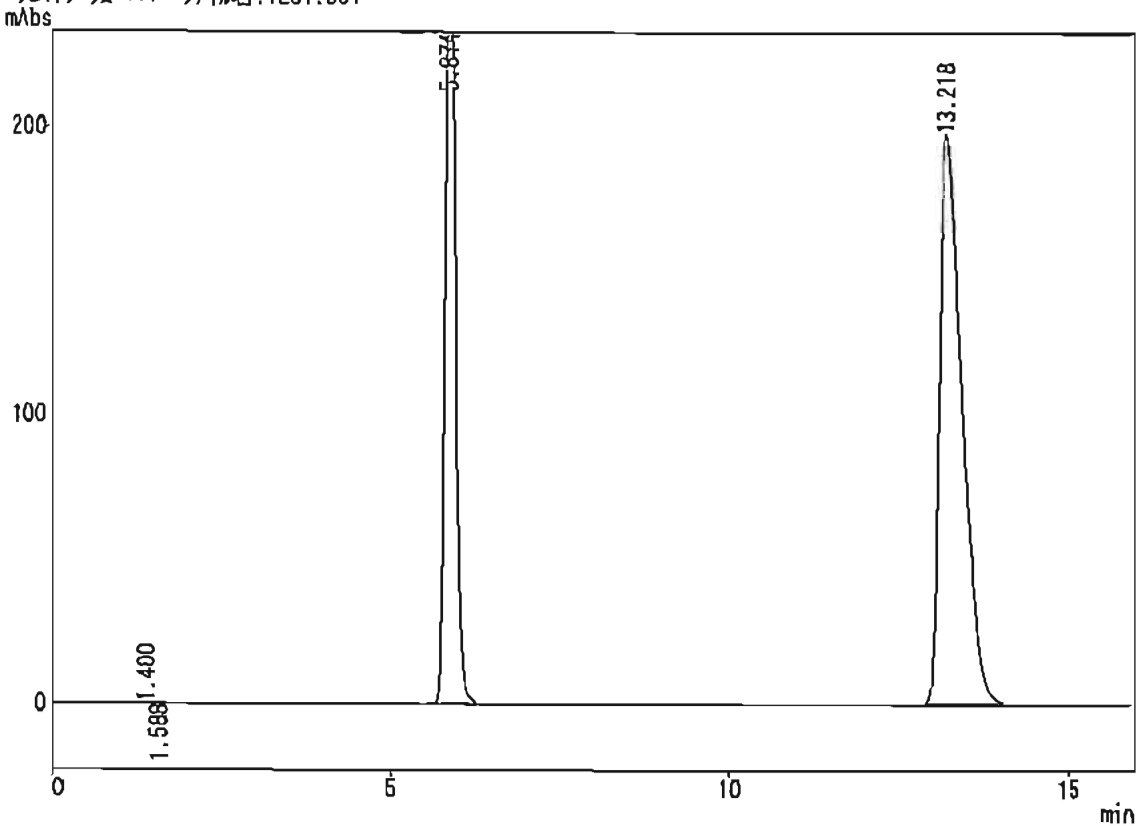
ID	Chromato No.	AHR	Peak Area IS	Ratio	Conc. (mg/mL)	Remaining (%)	Correction (%)	pH	F.I.M.	Turbid
STD	AH3N22.C01	4553327	2819952	1.6147						
STD	AH3N22.C11	4557786	2834325	1.6081						
STD	Mean			1.6114	0.10100					
A-01	40°C-6M AH3N22.C02	4285770	2845412	1.5062	0.09441	93.08	91.65	8.29	—	—
A-03	40°C-6M AH3N22.C03	4224937	2814027	1.6014	0.09411	94.02	92.88	8.30	—	—
BF	40°C-6M AH3N22.C04	4222386	2838573	1.4875	0.09323	91.36	90.02	8.33	—	—
A-01	25°C-4M AH3N22.C05	4427810	2839972	1.5591	0.09772	96.34	95.97	8.30	—	—
A-03	25°C-4M AH3N22.C06	4409879	2840521	1.5525	0.09731	97.21	96.84	8.31	—	—
BF	25°C-4M AH3N22.C07	4470457	2832712	1.5782	0.09892	96.93	96.58	8.37	—	—
A-01	25°C-6M AH3N22.C08	4368198	2823021	1.5473	0.09698	95.61	94.95	8.30	—	—
A-03	25°C-6M AH3N22.C09	4404588	2826921	1.5581	0.09768	97.58	96.95	8.30	—	—
BF	25°C-6M AH3N22.C10	4486855	2852405	1.5730	0.09859	96.81	95.99	8.35	—	—

試験物質名: AHR10282B	試験コード: P2002B131	試験年月日: 2003年04月22日
試験項目:		試験実施者: 澤 嗣郎
IS p-ヒドロキシ安息香酸X4H 0.01676g + 移動相 → 50ml		
STD1 AHR10282B 0.02020g + 移動相 → 20ml	22-APR-2003 10:11:24	
STD2 STD1 2ml + IS 2ml + 移動相 → 20ml	004:N + 0.02020g	
Sample 本品 2ml + IS 2ml + 移動相 → 20ml	005:N + 0.01676g	

		PH	外観	重量変化	
A-01	40°C6M	8.29	-	9.3565 9.2686	4/22 13:48
A-03	↓	8.30	-	9.4002 9.3186	NO.62 PH 8.29 26.2°C
BF	↓	8.33	-	9.3570 9.2730	
A-01	25°C4M	8.30	-	9.4249 9.4032	4/22 13:49
A-03	↓	8.31	-	9.3834 9.3619	NO.63 PH 8.33 26.7°C
BF	↓	8.37	-	9.2564 9.2364	4/22 13:50
A-01	25°C6M	8.30	-	9.3086 9.2698	NO.64 PH 8.33 26.7°C
A-03	↓	8.30	-	9.3720 9.3360	4/22 13:51
BF	↓	8.35	-	9.3372 9.3006	NO.65 PH 8.33 26.7°C
					4/22 13:52
					NO.66 PH 8.33 26.8°C
					4/22 13:54
					NO.67 PH 8.37 26.7°C
					4/22 13:55
					NO.68 PH 8.33 26.8°C
					4/22 13:56
					NO.69 PH 8.33 26.5°C
					4/22 13:56
					NO.70 PH 8.35 26.8°C

サンプル : TEST
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレーター名 : 澤 嗣郎
 サンプル名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:TEST.C01

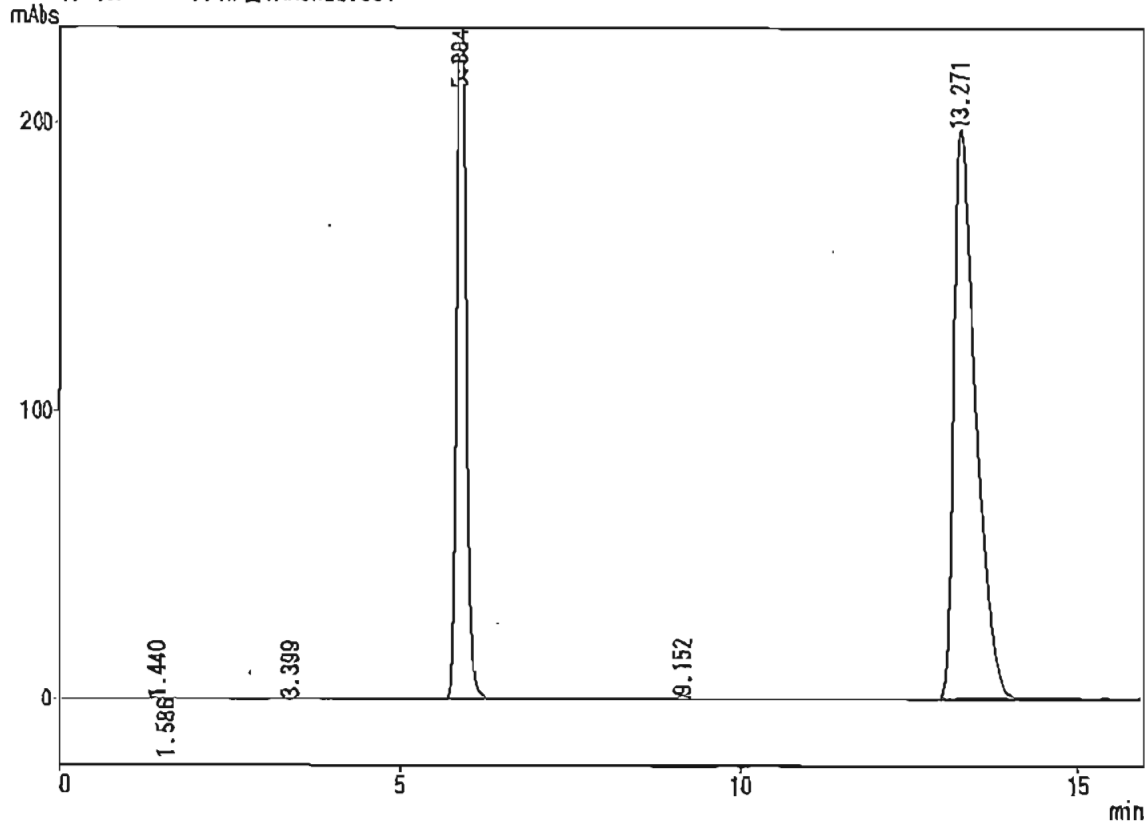


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.400	1828	124	V		0.0248	
2	1.588	1108	122	V		0.0150	
3	5.874	2820808	304780			38.2274	
4	13.218	4555271	198298			61.7328	
		7379013	503324			100.0000	

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレータ名 : 澤 嗣郎
 ソフト名 : !AHR1028.MET

*** カマトグラム *** ファイル名:AH3N22.C01

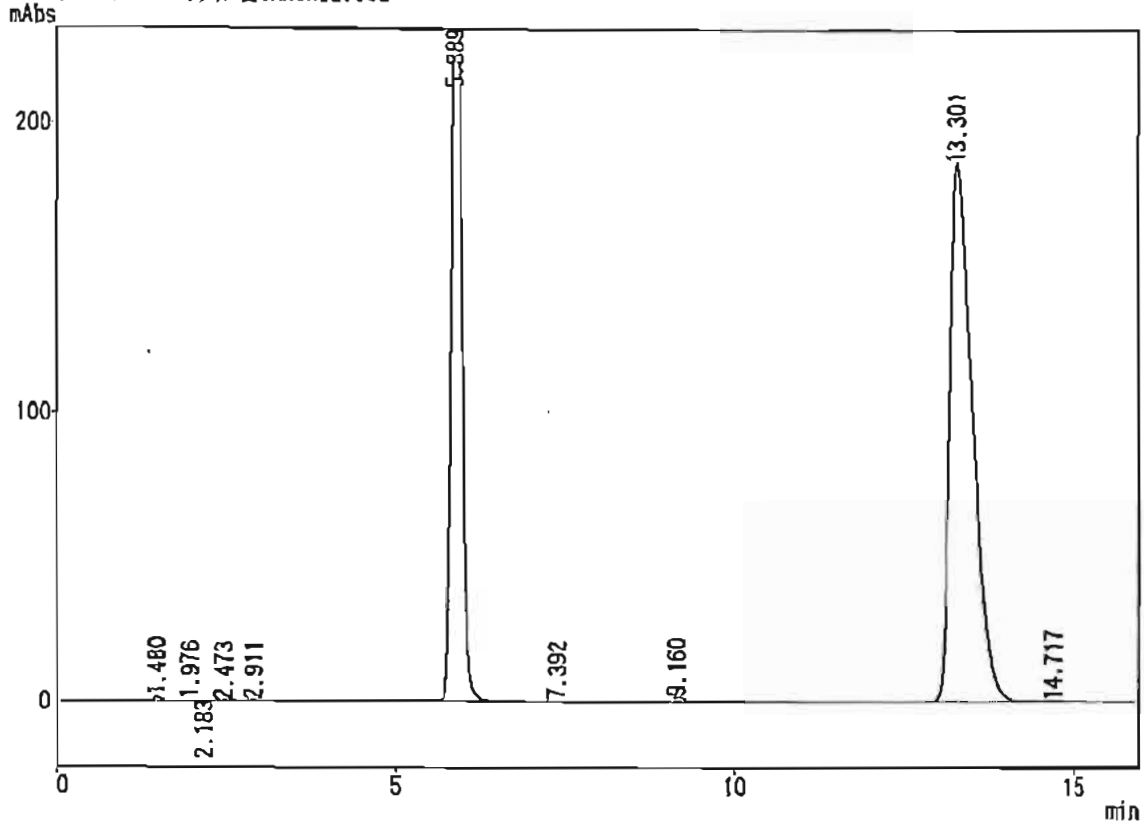


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.440	1443	128	V		0.0195	
2	1.586	1715	130	V		0.0232	
3	3.399	1194	184			0.0161	
4	5.884	2819952	304679			38.1138	
5	9.152	21145	1593			0.2858	
6	13.271	4553327	198023			61.5416	
		7398775	504715			100.0000	

サンプル : A01
 ID : 40°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シンク
 オペレーター名 : 澤 嗣郎
 メソッド名 : ICHR1028.MET

*** クロマトグラム *** ファイル名:AH3N22.C02

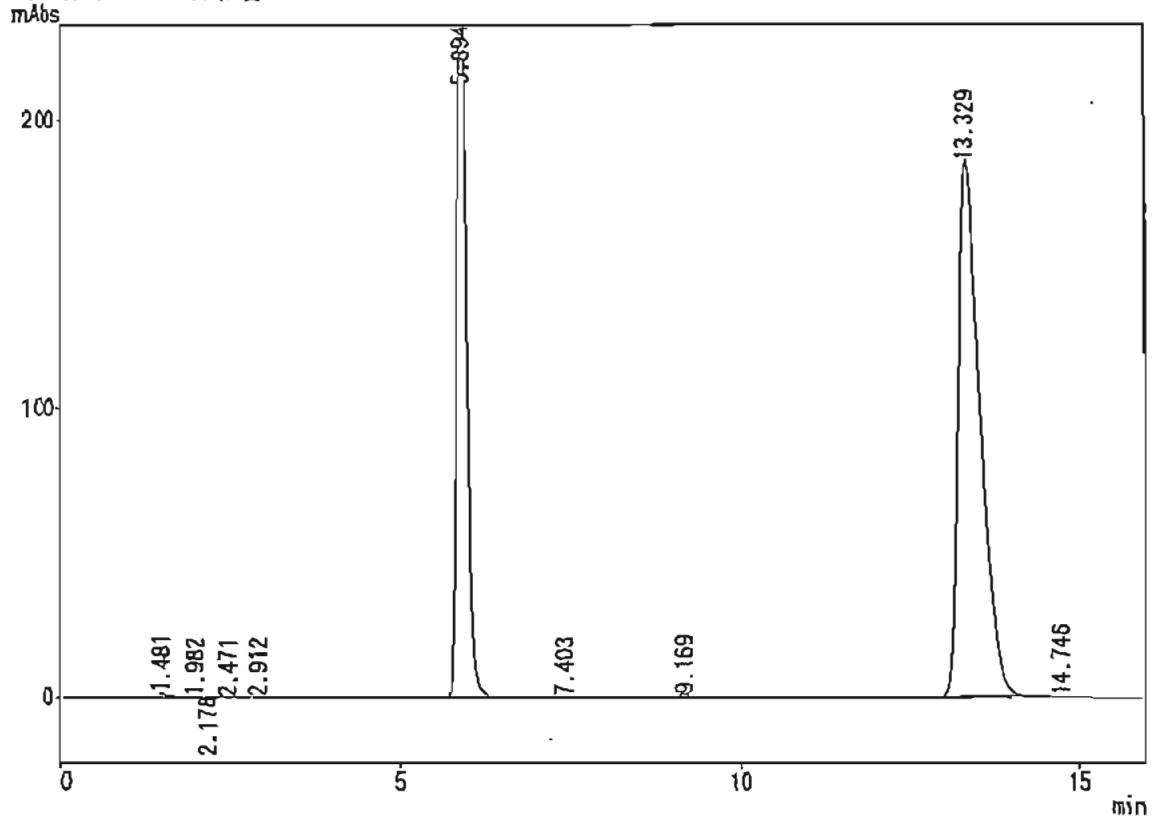


*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.480	15436	1525	V		0.2145	
2	1.976	3117	265	V		0.0433	
3	2.183	1577	207	V		0.0219	
4	2.473	2311	172	V		0.0321	
5	2.911	3517	381	V		0.0489	
6	5.889	2845412	306190			39.5437	
7	7.392	7708	652			0.1071	
8	9.160	24302	1787	V		0.3377	
9	13.301	4285770	186873	S		59.5609	
10	14.717	6465	343	TV		0.0898	
		7195813	498396			100.0000	

サンプル : A03
 ID : 40°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレーター名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロリグラム *** ファイル名:AH3N22.C03



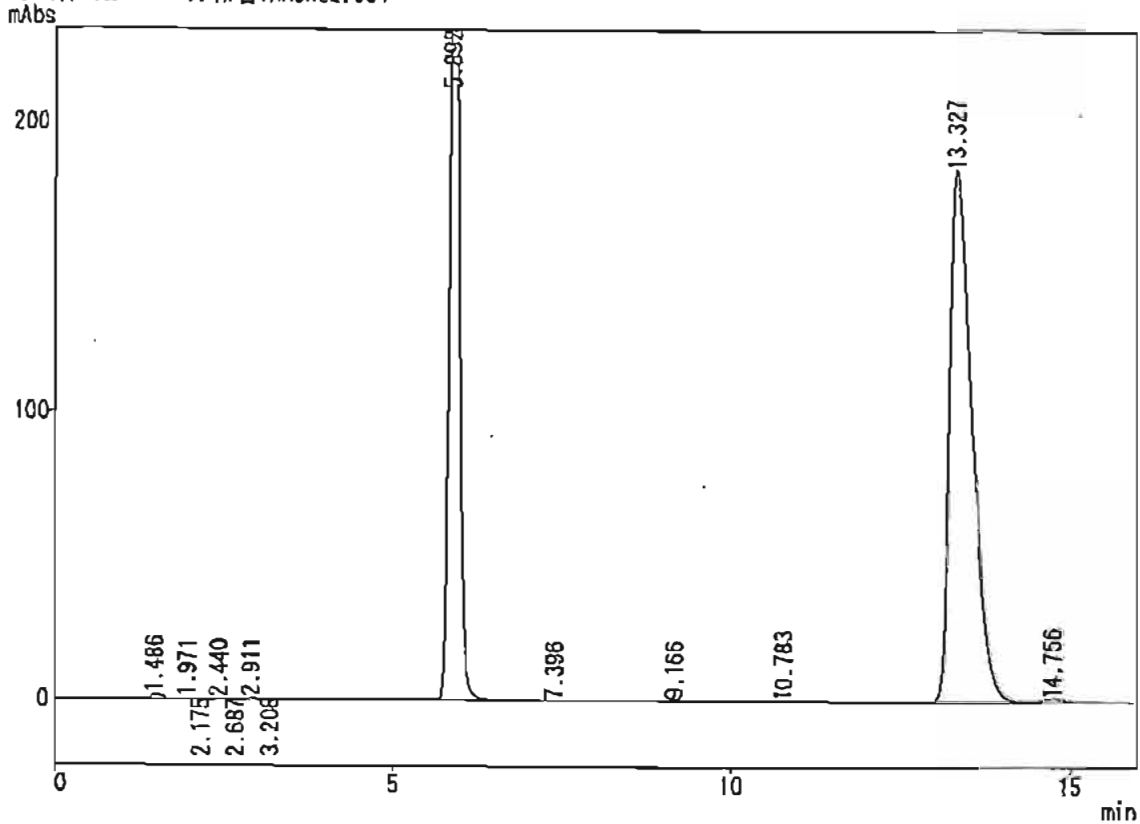
*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.481	15393	1548	V		0.2170	
2	1.982	2416	228	V		0.0341	
3	2.178	1298	176	V		0.0183	
4	2.471	1399	134	V		0.0197	
5	2.912	2357	341	V		0.0332	
6	5.894	2814027	302517			39.8685	
7	7.403	7828	679			0.1104	
8	9.169	18125	1346	V		0.2555	
9	13.329	4224937	185639			59.5577	
10	14.746	6080	339			0.0857	
		7093860	492845			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=33 テータ=AH3N22.004 03/04/22 13:25:46

サンプル : BF
 ID : 40°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名: 澤 嗣郎
 メソッド名 : IAHRT028.MET

*** クロマトグラム *** ファイル名:AH3N22.C04

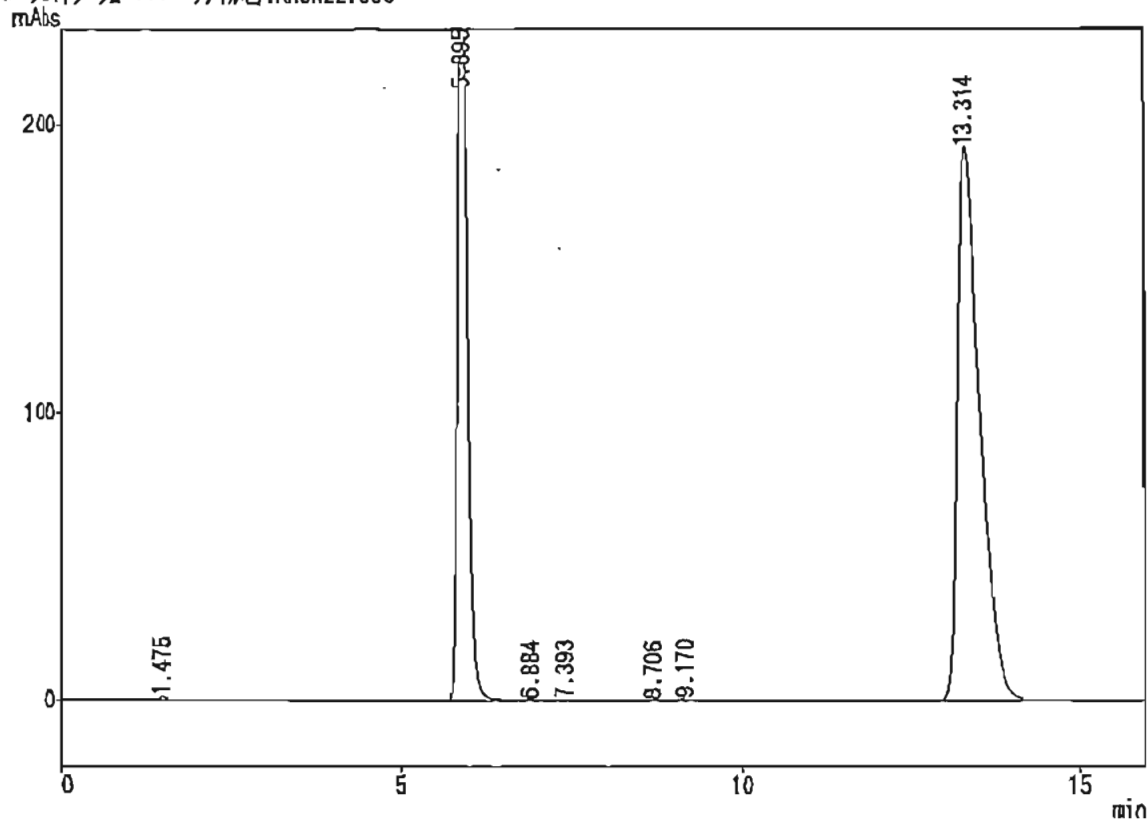


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.486	23939	1819	V		0.3343	
2	1.971	4231	505	V		0.0591	
3	2.175	1414	211	V		0.0197	
4	2.440	4969	444	V		0.0894	
5	2.687	1271	169	V		0.0177	
6	2.911	7124	917	V		0.0995	
7	3.208	1311	127	V		0.0183	
8	5.892	2848573	304476	S		39.7811	
9	7.396	2201	197	T		0.0307	
10	9.166	9418	711	V		0.1315	
11	10.783	2921	191	V		0.0408	
12	13.327	4222388	184975			58.9868	
13	14.756	30858	1228	V		0.4309	
		7160615	495969			100.0000	

サンプル : A01
 ID : 25°C-4M
 タイプ : 未知試料
 検出器 : SPD-10A シングル
 オペレーター名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH3N22.C05



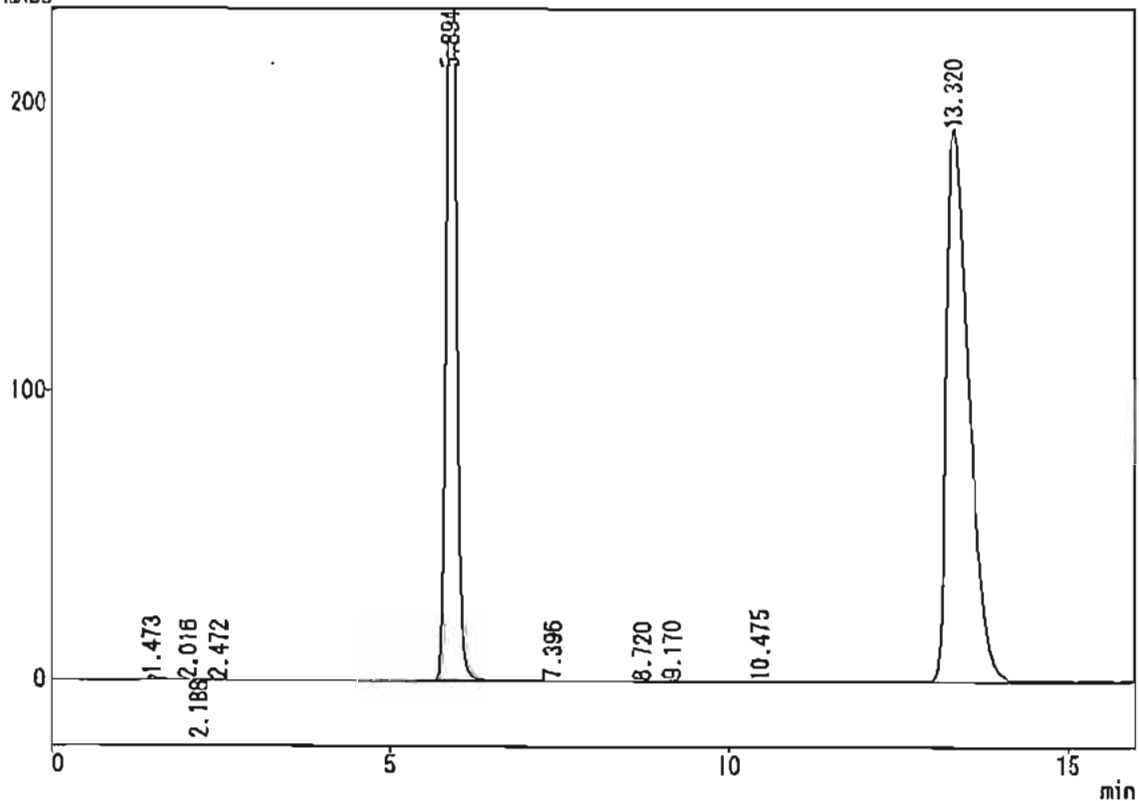
*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.475	11771	1337	SV		0.1610	
2	5.895	2839972	305128			38.8489	
3	6.884	12107	373	V		0.1656	
4	7.393	8430	471	V		0.0880	
5	8.706	1673	126			0.0229	
6	9.170	10537	794	V		0.1441	
7	13.314	4427810	192151			60.5695	
		7310300	500380			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 レポート番号=35 データ=AH3N22.D06 03/04/22 14:00:42

サンプル : A03
 ID : 25°C-4M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレーター名 : 澤 嗣郎
 サンプル名 : !AHR1028.MET

*** カロリグラム *** ファイル名: AH3N22.C06
 mAbs



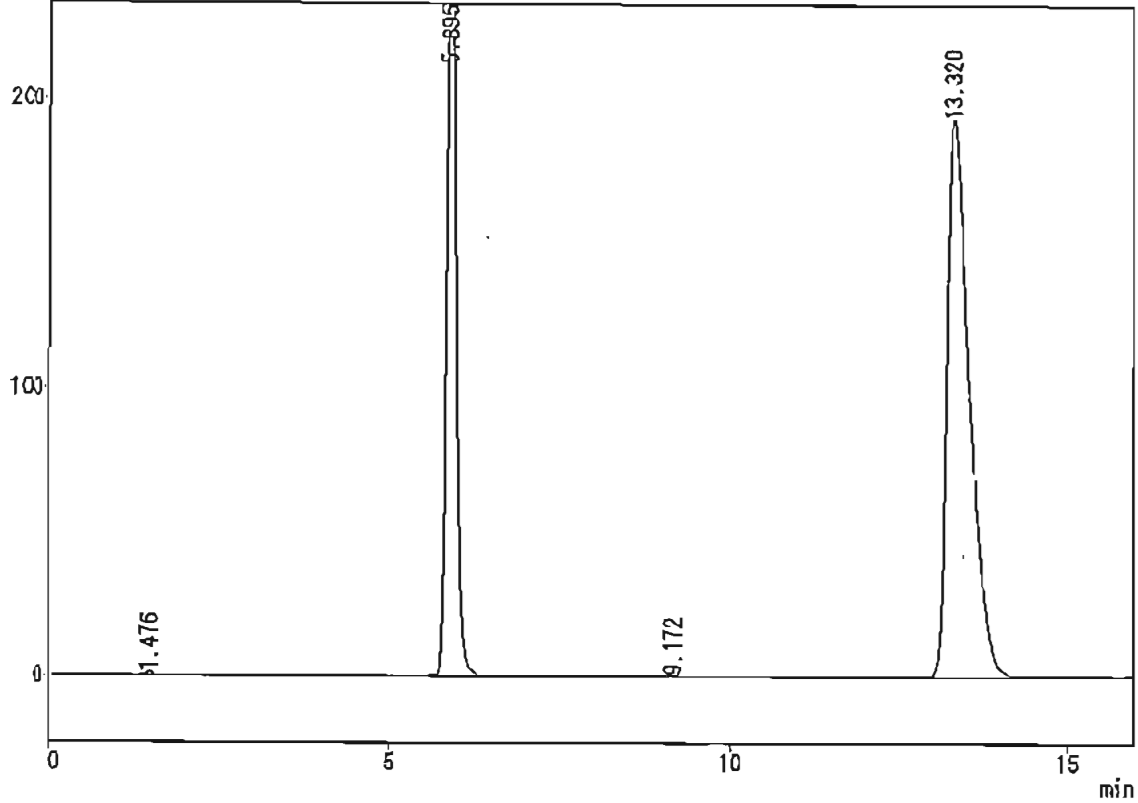
*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.473	11713	1383	SV		0.1609	
2	2.016	1258	117	T		0.0173	
3	2.188	1084	103	TV		0.0146	
4	2.472	1051	82	TV		0.0144	
5	5.894	2840521	305624			39.0137	
6	7.396	4561	380			0.0626	
7	8.720	1972	144			0.0271	
8	9.170	7400	553	V		0.1016	
9	10.475	1409	84			0.0193	
10	13.320	4409879	191505			60.6684	
		7280828	499875			100.0000	

CLASS-LC10 Ver.=1.62 システム番号=1 Ch=1 ピーク番号=36 データ=AH3N22.007 03/04/22 14:18:12

サンプル : BF
ID : 25°C-4M
タイプ : 未知試料
検出器 : SPD-10A シンガム
オペレーター名 : 澤 嗣郎
メソッド名 : !AHR1028.MET

*** クロマトグラム *** ファイル名:AH3N22.C07
mAbs

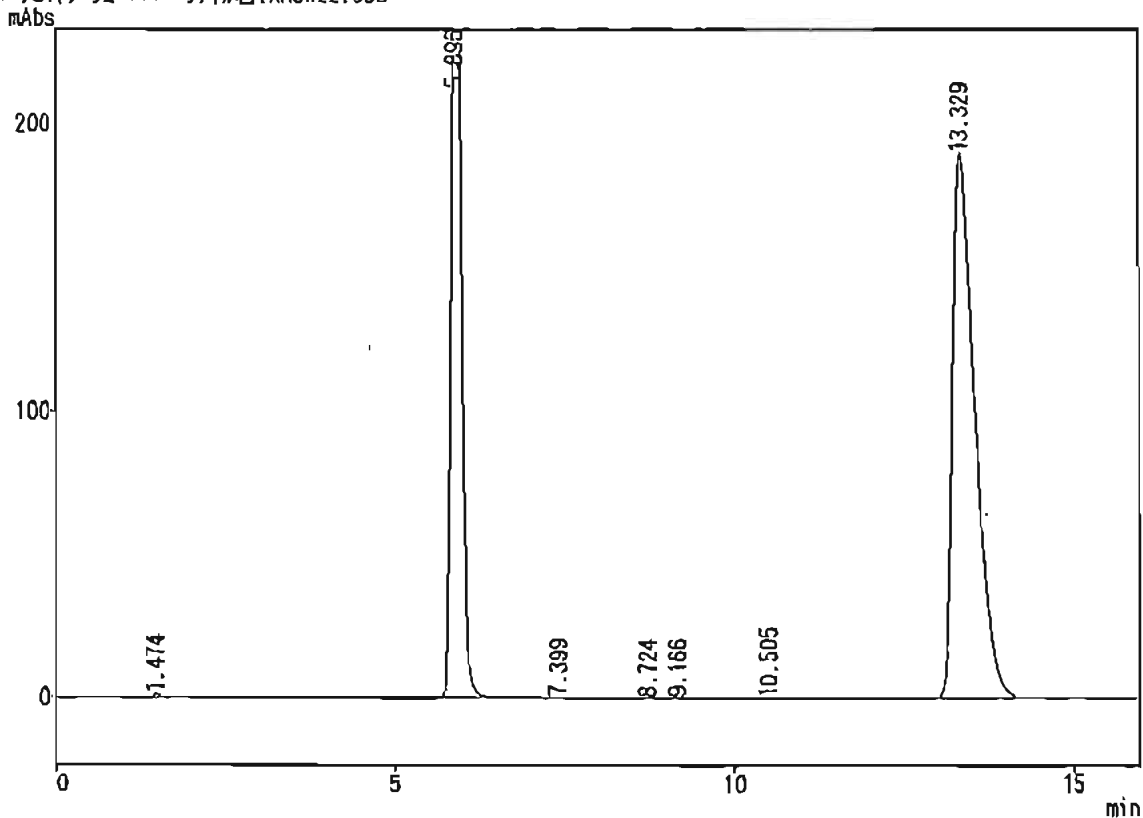


*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.476	14738	1308	V		0.2013	
2	5.895	2832712	304412			38.6819	
3	9.172	5195	395			0.0709	
4	13.320	4470457	193753	S		51.0459	
		7323102	499870			100.0000	

サンプル : A01
 ID : 25°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シンクル
 オペレータ名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** クロマトグラム *** ファイル名: AH3N22.C08

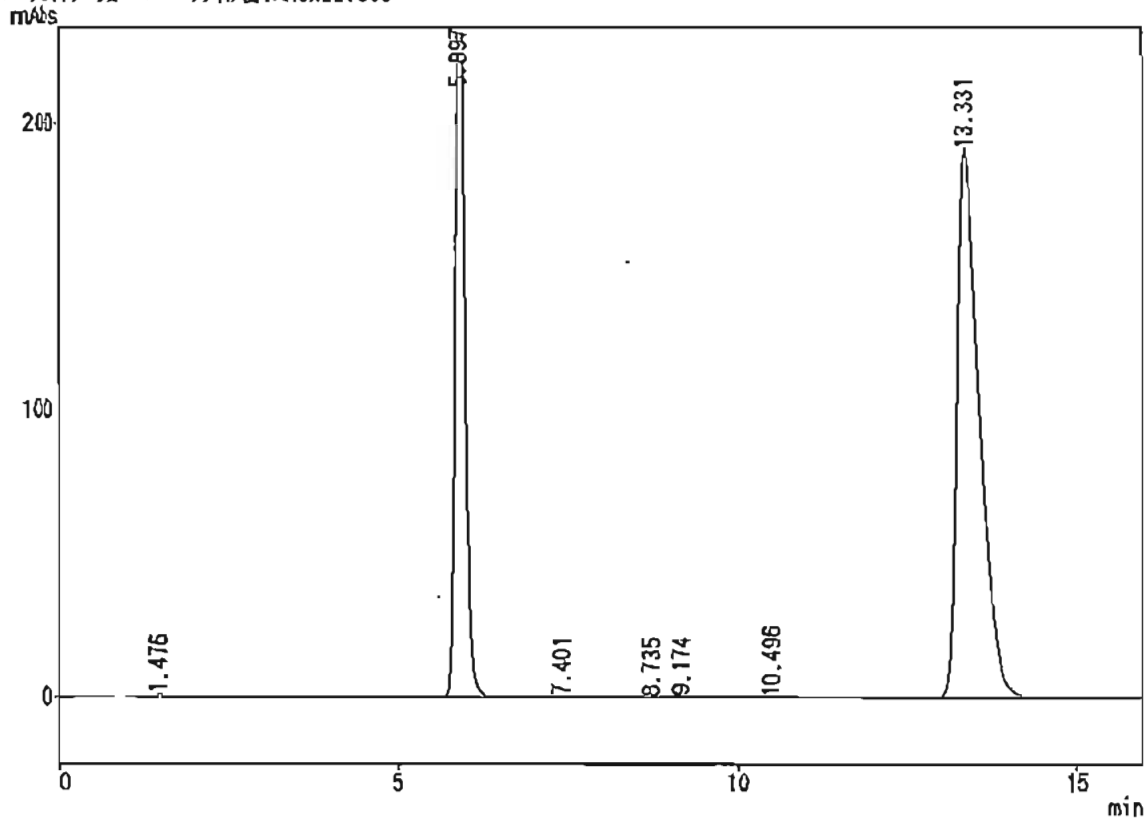


*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.474	10357	1271	V		0.1436	
2	5.896	2823021	303876			39.1311	
3	7.399	5142	430			0.0713	
4	8.724	1643	122			0.0228	
5	9.166	4570	348	V		0.0633	
6	10.505	1338	94			0.0185	
7	13.329	4368198	189924			60.5494	
		7214269	496062			100.0000	

サンプル : A03
 ID : 25°C-6M
 タイプ : 未知試料
 検出器 : SPD-10A シンプル
 オペレーター名 : 澤 嗣郎
 メソッド名 : !AHR1028.MET

*** カロリフラ *** ファイル名:AH3N22.C09



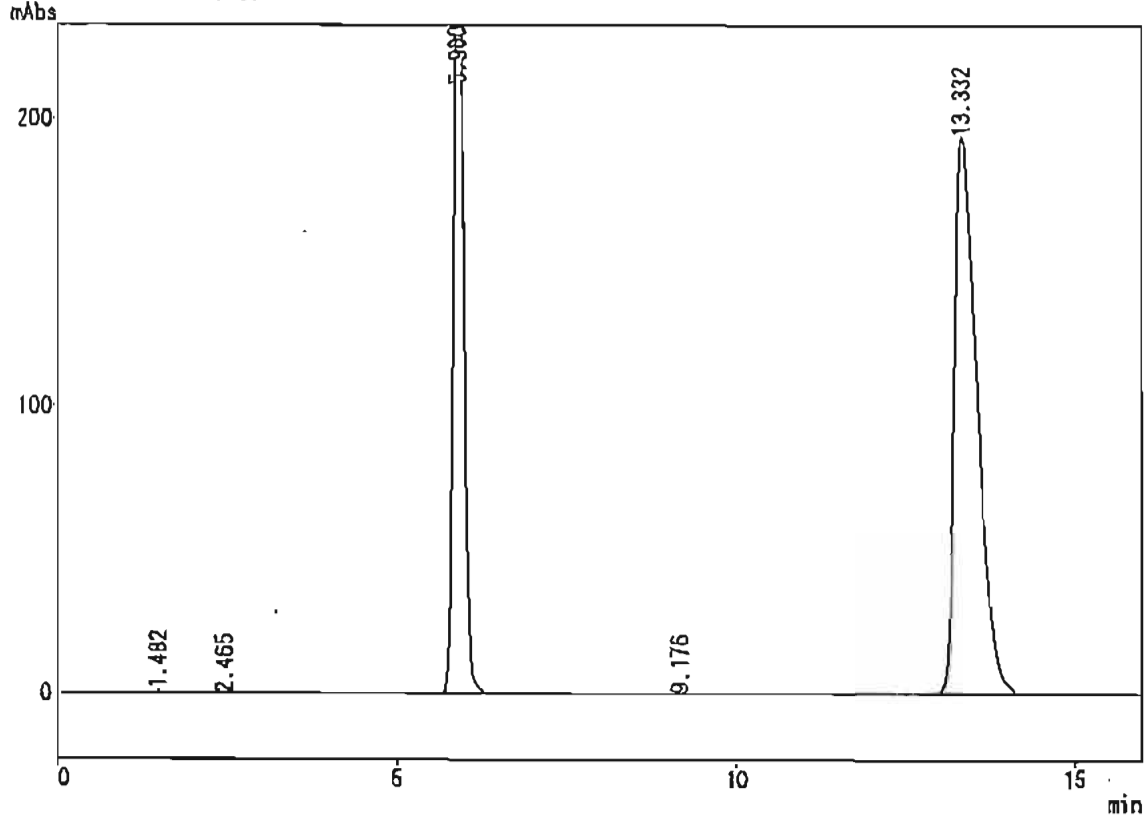
*** ヒートマップ ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.476	9893	1142	V		0.1363	
2	5.897	2826921	304114			38.9537	
3	7.401	5145	438			0.0709	
4	8.735	1792	134			0.0247	
5	9.174	7142	534	V		0.0984	
6	10.496	1668	109			0.0230	
7	13.331	4404577	191086			60.6930	
		7257137	497556			100.0000	

CLASS=LC10 Ver.=1.62 システム番号=1 Ch=1 検出器番号=39 データ=AH3N22.D10 03/04/22 15:10:38

サンプル : 8F
ID : 25°C-6M
タイプ : 未知試料
検出器 : SPD-10A シンクル
オペレーター名 : 澤 嗣郎
メソッド名 : 1AHR102B.MET

*** クロマトグラム *** ファイル名:AH3N22.C10

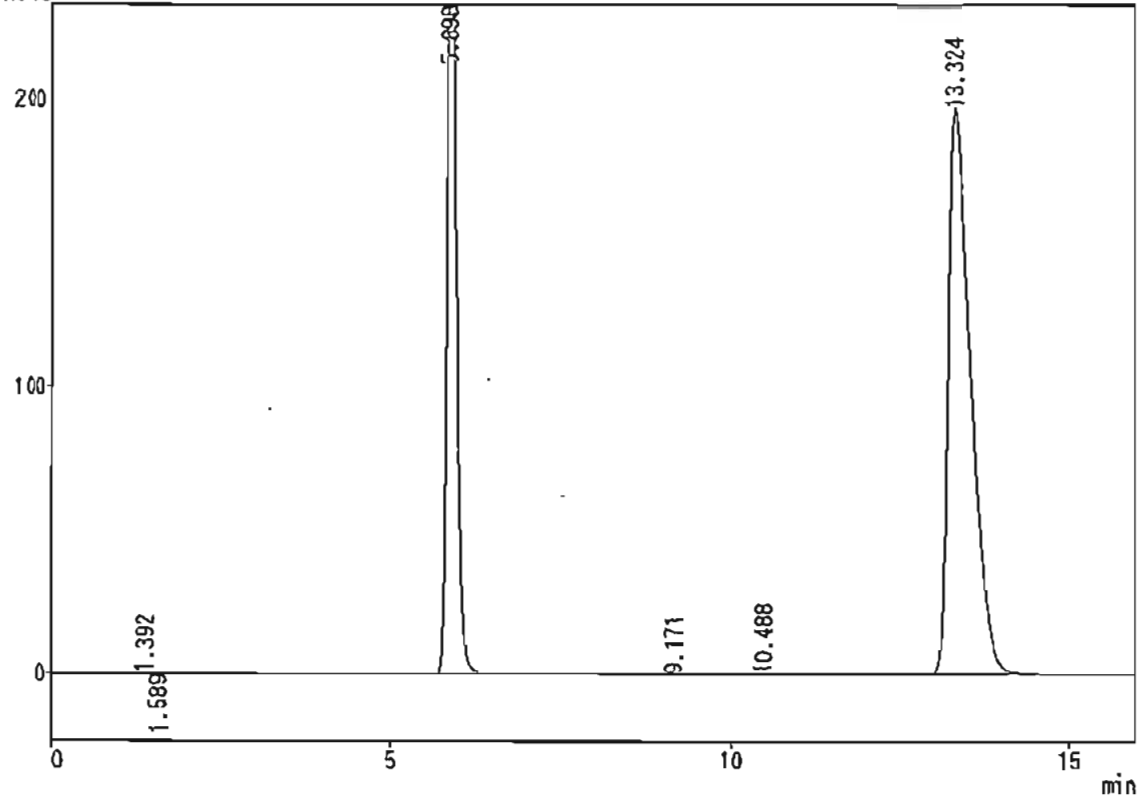


*** ヒストグラム ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.482	15601	1277	SV		0.2119	
2	2.465	1935	183	V		0.0263	
3	5.900	2852405	305281			38.7508	
4	9.176	4107	311			0.0558	
5	13.332	4486855	194369			60.9552	
		7360903	601420			100.0000	

サンプル : STD
 ID :
 タイプ : 未知試料
 検出器 : SPD-10A シンチ
 オペレーター名 : 澤 嗣郎
 メソッド名 : IHR1028.MET

*** カロリグラム *** ファイル名:AH3N22.C11
 mAbs



*** ピークレポート ***

PKNO	TIME	AREA	HEIGHT	MK	IDNO	CONC	NAME
1	1.392	1803	131	V		0.0244	
2	1.589	1967	149	V		0.0266	
3	5.898	2834325	305722			38.2981	
4	9.171	2866	202	V		0.0360	
5	10.488	2142	135			0.0289	
6	13.324	4557788	197363			61.5860	
		7400689	503701			100.0000	