THIRD DECLARATION OF LEONARD J. CHYALL, PH.D.

I, the undersigned, **Dr. Leonard J. Chyall**, U.S. Passport No. 432624896, with a business address of Chyall Pharmaceutical Consulting LLC, 3000 Kent Avenue, Suite D1-105, West Lafayette, Indiana 47906, USA, having been warned that I must state the truth and that I shall be liable to the penalties prescribed by law should I fail to do so, hereby declare in writing as follows:

1. I am the same Leonard J. Chyall who submitted a declaration dated August 3, 2010 (the **"First Chyall Declaration"**) and a declaration dated March 7, 2012 (the **"Second Chyall Declaration"**), in support of the position of Teva Pharmaceutical Industries Ltd. (**"Teva"**) in the proceedings before the Honorable Deputy Registrar of Patents regarding Israel Patent Application No. 172563, filed by Merck & Co. Inc., U.S.A (**"Merck"**).

2. This declaration was prepared in response to the Affidavit of Prof. Jerry L. Atwood submitted on behalf of Merck regarding an experiment that Prof. Atwood conducted in August 2012 (the **"Second Atwood Affidavit"**). I was advised by Teva's counsel that Merck does not rely on paragraphs 3, 6, 7, 8 and 9 of the Second Atwood Affidavit.

3. The fact that I have not commented on any particular point in the Second Atwood Affidavit does not mean that I accept or agree with that point. There is nothing in the Second Atwood Affidavit that causes me to change the views that I expressed in the First and Second Chyall Declarations.

Prof. Atwood's New Experiment

4. In the Second Atwood Affidavit, Prof. Atwood describes a new experiment, in which he claims to have "repeated the procedure for making the 2:1 phosphate salt in isopropanol and water", which was described in Paragraphs 31 and 68 of his First Affidavit, this time with a "filtration and washing" step ("**the New Experiment**"). I shall refer below to the unwashed solids recovered by Prof. Atwood using the procedure described in Paragraphs 31 and 68 of his First Affidavit as "**the Atwood Unwashed Solids,**" and to the solids recovered by Prof. Atwood after his "filtration and washing step" as "**the Atwood Washed Solids**". Based on the elemental analysis of the Atwood Washed Solids, Prof. Atwood claims that his New Experiment shows that "the same 2:1 phosphate salt was obtained in all procedures, with or without Dr. Chyall's suggested work up" (paragraph 5 of Prof. Atwood's Second Affidavit). Prof. Atwood described his alleged "2:1 salt" as having a characteristic X-Ray Powder Diffraction ("**XRPD**") pattern that can be used to identify that alleged salt.

5. I note that Prof. Atwood's New Experiment attempts to rebut (but fails to do so, as explained below) only **one** of the criticisms that I raised in the Second Chyall Declaration with regard to Prof. Atwood's first set of experiments, *i.e.*, Prof. Atwood's failure to filter and wash the solid reaction products that he recovered. The New Experiment does not attempt to address any of the other criticisms I raised in the Second Chyall Declaration with regard to Prof. Atwood's experiments, such as Prof. Atwood's use of irregularly high reactant concentrations and lack of analytical data capable of proving that Prof. Atwood recovered any phosphate salts of sitagliptin other than the expected dihydrogenphosphate salt ("**DHP Salt**"). In

fact, Prof. Atwood's New Experiment is also fraught with these very same flaws, which are not remedied by his washing step.

6. In addition, Prof. Atwood's New Experiment attempts to rebut my criticism of his failure to filter and wash the reaction products with respect to only one of Prof. Atwood's experiments: his "procedure for making the 2:1 phosphate salt in isopropanol and water" (described in paragraphs 31 and 68 of his First Affidavit). Prof. Atwood did not present any experiment to rebut any of my criticisms, including his failure to filter and wash, with regard to any of the other preparations of alleged non-DHP Salts described in the First Atwood Affidavit.

7. There is nothing in the Second Atwood Affidavit that causes me to change the views that I expressed in the First and Second Chyall Declarations, *i.e.*, that the only pharmaceutically suitable stable salt that will result from a reaction of sitagliptin free base and phosphoric acid is the DHP Salt, a salt containing a 1:1 ratio of sitagliptin to phosphoric acid.

Prof. Atwood Used Poor Experimental Techniques

8. Based on my review of Prof. Atwood's Second Affidavit and laboratory notebook pages in which he describes the New Experiment, Prof. Atwood's protocol included at least the following steps that may have resulted in inadequate washing of the recovered solids:

   A Prof. Atwood states that his reaction solution solidified. *See* Atwood Exhibit HH. In my experience, a reaction mixture that has solidified is more likely to contain entrapped impurities, such as unreacted starting materials, than a solid product that precipitated from the reaction solution.

B. Prof. Atwood states that he used a spatula to place his solidified material on a Büchner funnel fitted with an approximately 7 cm diameter piece of filter paper. *See* Atwood Exhibit HH. Prof. Atwood does not indicate whether he crushed or broke up his solidified reaction material before placing the solidified material on the Büchner funnel, a step that would have improved the effectiveness of Prof. Atwood's washing.

C. Prof. Atwood also does not indicate whether he evenly spread his recovered solids on the filter paper to minimize the possibility that washing solvent could pass through the filter with little or no contact with the solids to be washed. Any washing solvent that passed through the filter with little or no contact with the solids to be washed would not effectively wash the solids.

D. Prof. Atwood used filter paper with an approximate diameter of 7 cm (*see* Atwood Exhibit HH), meaning that the Büchner funnel that Prof. Atwood used also likely had a diameter of approximately 7 cm. If Prof. Atwood recovered 100% of his starting materials, he would only have recovered about 1.7 grams of solids. Depending on whether and/or how Prof. Atwood spread his recovered solids on the filter paper, the use of a filter with a 7 cm diameter with such a small amount of solids may leave parts of the filter bare, which would permit washing solvent to pass through the filter with little or no contact with the solids to be washed.

E. Prof. Atwood states that prior to the actual washing, he drew air through his recovered solids and the filter paper for 5 minutes. *See* Atwood Exhibit HH. If no liquid was extracted from the solids by this procedure, then it likely did little more than dry the solids. Drying the solids, including any impurities,

byproducts and/or unreacted sitagliptin base dissolved in the reaction solvent and trapped inside the recovered solids, would make it more difficult to remove those now solidified impurities, byproducts and/or unreacted sitagliptin base through subsequent washing.

F. Prof. Atwood states that he used 3 x 3 mL of isopropanol solvent to wash and filter the solids that he recovered. *See* Atwood Exhibit HH. This is a very small amount of solvent for washing Prof. Atwood's recovered solids when using a Büchner funnel with a diameter of approximately 7 cm. Using too little solvent for washing would result in ineffective removal of impurities, byproducts and unreacted starting materials.

9. Prof. Atwood's laboratory notebook, Exhibit HH of his Second Affidavit, by itself, does not provide enough detail to determine whether one or more of the above steps rendered Prof. Atwood's washing steps inadequate. Therefore, unlike my criticism of Prof. Atwood's previous experiments – which did not include **any** filtration and washing and therefore did not require that I conduct experiments to conclude that Prof. Atwood's assertions regarding the solids that he recovered were unreliable and without scientific merit – I could only prove the misleading nature of Prof. Atwood's New Experiment, which included a "filtration and washing" step, by conducting experiments.

My Experiments Prove That Prof. Atwood's "Filtration And Washing" Was Ineffective

10. I received from Teva a sample container labeled lot no. D6655070112, which I understand to contain Sitagliptin Free Base. The sample was assigned LIMS No. 308390, and I characterized the material using XPRD (see Exhibit A). The XRPD

pattern obtained for the material confirmed that the material was crystalline sitagliptin base as disclosed in PCT Publication No. WO 2009/070314 A2.

11. I first replicated as closely as possible the New Experiment described in the Second Atwood Affidavit. I conducted this replication to ensure that it was possible, based on the procedure described in Prof. Atwood's laboratory notebook, Exhibit HH, to obtain crystalline solids with the characteristic XRPD pattern of Prof. Atwood's alleged "2:1 salt".

12. However, my solution did not solidify overnight like Prof. Atwood's solution. In order to precipitate the reaction product, I cooled the reaction mixture using an ice bath with stirring. I filtered and washed my recovered solids using the same "filtration and washing" protocol that Prof. Atwood used to filter and wash his recovered solids. I analyzed the recovered solids by XRPD. **The recovered solids had the same characteristic XRPD pattern as that of Prof. Atwood's solids (Exhibit B)**, which demonstrates that my use of an ice bath to precipitate solids did not affect the final product and was not a material deviation from Prof. Atwood's procedure. I refer to the solids that I recovered from this experiment as "**the Replicated Atwood Wash Solids**". A detailed description of how I obtained the Replicated Atwood Wash Solids is set forth in my laboratory notebooks, (Exhibit C).

13. I next conducted an experiment to see the effect of progressively more thorough washings than employed by Prof. Atwood's "filtration and washing" protocol. To do so, I again replicated Prof. Atwood's New Experiment, except at double the scale, so as to obtain a sufficient amount of material. This time the solution solidified overnight, like Prof. Atwood's solution. I analyzed the recovered solids by XRPD. The solids recovered on the Büchner funnel after washing once with isopropanol had

the same characteristic XRPD pattern (Exhibit D) as that obtained for Prof. Atwood's solids. My washing procedure, which also included additional washing steps, differed from that used by Prof. Atwood. The differences between Prof. Atwood's "washing and filtering" procedure and my washing and filtering procedure are explained below:

a) **Filter Paper:** I used 45 mm diameter filter paper, which has approximately 59% less surface area than the 70 mm (7 cm) diameter filter paper used by Prof. Atwood. I used a smaller diameter filter and filter paper to give my solids more intimate contact with the washing solvent than there would have been if I had followed Prof. Atwood's washing protocol.

b) **Scale:** I started my experiment at double the scale of Prof. Atwood's New Experiment. Consequently, I started my experiment with approximately twice the solids recovered and filtered by Prof. Atwood. By using twice the solids in my first series of washings, a reduced filter surface area, and as explained below, larger volumes of wash solvent, I ensured that my solids had more intimate contact with the washing solvent than there would have been if I had followed Prof. Atwood's washing protocol.

c) **Volume of Wash Solvent:** Prof. Atwood used 3 mL portions of isopropanol to wash his reaction products that were prepared on a 1.5 g scale. I used at least triple the relative amount of solvent for my washings. For example when I ran the reaction on a 3 g scale, I used 18 mL portions of isopropanol for the washing step.

d) **Number Of Washes:** Prof. Atwood washed his recovered solids three times. I conducted three series of three washes each, as follows:

a. **First Series of Washes** - I washed my recovered solids three times during the first series of washes. I refer to the solids remaining after the third wash as "the 3X Washed Solids."

b. **Second Series of Washes** - I took approximately two-thirds of the 3X Washed Solids and washed them an additional three times in a second series of washes. I refer to the solids remaining after the second series of three washings as "the 6X Washed Solids."

c. **Third Series of Washes** - I took approximately one-half of the 6X Washed Solids and washed them an additional three times in a third series of washes. I refer to the solids remaining after the third series of three washings as "the 9X Washed Solids."

e) **Washing Solvent Volume:**

a. **The 3X Washed Solids:** I conducted my first series of three washes using 18 mL of isopropanol per wash. The reason for this is as follows. Prof. Atwood washed his solids three times with 3 mL of isopropanol. Since I had doubled the scale of Prof. Atwood's New Experiment, I would have needed to use 6 mL of isopropanol per wash to achieve the same wash solvent to solids ratio as Prof. Atwood. However, to ensure that my washing was more thorough than Prof. Atwood's washing, I used 18 mL of isopropanol per wash. This afforded a wash solvent to solids ratio of about three times the ratio that Prof. Atwood used in his New Experiment.

b. **The 6X Washed Solids:** After removing approximately 1/3 of the 3X washed solids from the Büchner funnel (to be used for subsequent analytical characterization) I washed the remaining 2/3 of the material in a second series of three washings with 12 mL of isopropanol in each washing. This afforded, relative to the weight of solids, a wash solvent to solids ratio of about three times the ratio that Prof. Atwood used in his New Experiment. Accordingly, the solids that I recovered after the second series of three washings (*i.e.*, the 6X Washed Solids) were washed a total of six times, using a total of about six times more washing solvent (relative to the weight of solids) than Prof. Atwood used to wash the solids that he recovered.

c. **The 9X Washed Solids:** After removing approximately 1/2 of the 6X washed solids from the Büchner funnel (to be used for subsequent analytical characterization) I washed the remaining 1/2 of the 6X Washed Solids in a third series of three washings with 12 mL of isopropanol in each washing. This afforded, relative to the weight of solids, a wash solvent to solids ratio of about six times the ratio that Prof. Atwood used in his New Experiment. Accordingly, the solids that I recovered after the third series of three washings (*i.e.*, the 9X Washed Solids) were washed a total of nine times, using a total of about 12 times more washing solvent (relative to the weight of solids) than Prof. Atwood used to wash the solids that he recovered.

14. I analyzed the 3X Washed Solids, the 6X Washed Solids and the 9X Washed Solids by XRPD. **The XRPD patterns for the 3X and 6X Washed Solids (Exhibits E and**

F, respectively) were the same as the XRPD pattern for Prof. Atwood's alleged "2:1 salt". The XRPD pattern of the 9X Washed Solids did not match the XRPD pattern of Prof. Atwood's alleged "2:1 salt" (Exhibit G). This indicates that during the third series of washings, the crystal structure that had previously existed, *i.e.*, the crystal structure of Prof. Atwood's alleged "2:1 salt", was destroyed. This demonstrates that Prof. Atwood's alleged "2:1 salt" is not stable.

15. A detailed description of my experiments and analyses are provided in my laboratory notebooks, Exhibit C.

16. The fact that the XRPD patterns of the Replicated Atwood Wash Solids, the 3X Washed Solids and the 6X Washed Solids were the same as Prof. Atwood's XRPD patterns of his unwashed and washed solids, indicates that they all included the same crystalline product.

17. While an XRPD pattern provides information about the crystalline portion of a solid, it does not provide information about the non-crystalline portion of a solid in the case where mixtures are present. The non-crystalline portions of a solid, such as amorphous impurities, byproducts and/or unreacted sitagliptin base in the samples at issue in this proceeding, would show up if at all in an XRPD pattern of substantially crystalline material only as background noise. Therefore, in order for the crystalline product to be a "2:1 salt" irrespective of filtration and washing, as alleged by Prof. Atwood, it must maintain both the same characteristic XRPD pattern and the same relative amounts of the elements after different washing protocols. If, however, the crystalline product maintains the same characteristic XRPD pattern after different washing protocols, but the recovered solids have different ratios of the elements, then this evidences that different washing protocols have different capacities to remove

amorphous impurities from the crystalline product. It also demonstrates that the crystalline product cannot be a "2:1 salt" and must have included amorphous impurities, such as unreacted sitagliptin base, prior to the effective washing.

18. The ratio of nitrogen to phosphorus in the different samples is particularly informative as nitrogen is only present in the "sitagliptin" component of the solids while phosphorus is only present in the "phosphate" component of the solids. As I demonstrate below, both elemental analysis and solution phase NMR spectroscopy show that my more thoroughly washed samples contain relatively less nitrogen with respect to the phosphorus content (*i.e.*, a lower nitrogen to phosphorus molar ratio) than "the Atwood Unwashed Solids" and "the Atwood Washed Solids". This demonstrates that Prof. Atwood's "filtration and washing" step was ineffective, and, accordingly, that the crystalline product cannot be a "2:1 salt".

My Analytical Data Proves That Prof. Atwood's Alleged "Filtration And Washing Step" Was Ineffective

19. I used common analytical techniques to determine the ratio of elements in the Replicated Atwood Wash Product, the 3X Washed Solids and the 6X Washed Solids. I employed elemental analysis to examine the Replicated Atwood Wash Solids, the 3X Washed Solids and the 6X Washed Solids. The ratio of nitrogen to phosphorus in sitagliptin dihydrogenphosphate (N/P ratio) is 2.261. The ratio of nitrogen to phosphorus for a theoretical bis(sitagliptin) phosphate structure is 4.522. For samples that contain between one and two molecules of sitagliptin per molecule of phosphoric acid, the N/P ratio will fall between these two values. The experimentally determined N/P ratio is linearly correlated with the sitagliptin to phosphoric acid ratio in the sample such that division of the experimental N/P ratio by 2.261 will provide the

number of molecules of sitagliptin for each molecule of phosphoric acid (*See* Exhibit H).

20. I also used solution phase carbon-13 Nuclear Magnetic Resonance (^{13}C NMR) spectroscopy to examine the Replicated Atwood Wash Solids and the 6X Washed Solids. Certain ^{13}C NMR resonances of sitagliptin are sensitive to the protonation state of sitagliptin. For NMR solutions of mixtures of sitagliptin and phosphoric acid, the ^{13}C NMR resonances represent an averaged value for protonated and unprotonated sitagliptin due to rapid exchange of the available protons between sitagliptin molecules. The ratio of sitagliptin to phosphoric acid in a sample may be determined by comparing the ^{13}C NMR spectrum for the sample to NMR spectra for reference standards that contain known ratios of sitagliptin to phosphoric acid. By using this methodology I independently confirmed the ratio of sitagliptin to phosphoric acid in my Replicated Atwood Wash Solids and 6X wash solids. Details of how the ^{13}C NMR spectra were obtained and the resulting data are provided in Exhibit I.

21. The molar ratios of sitagliptin to phosphoric acid that I determined based on ^{13}C NMR spectra and elemental analysis data that I obtained, along with the elemental analysis data that Prof. Atwood obtained, are summarized in the table below. The elemental analysis results are in agreement with the ^{13}C NMR results within experimental error. The data evidences that successively more thorough washing of the recovered solids progressively removed more unreacted starting materials, impurities, and/or byproducts and that the crystalline product cannot be a 2:1 salt.

Sample (Source)	Sitagliptin:Phosphoric Acid Molar Ratio	
	determined by elemental analysis	determined by ^{13}C NMR spectroscopy
Atwood Unwashed Solids (First Atwood Affidavit)	2.09:1	-
Atwood Washed Solids (Second Atwood Affidavit)	1.83:1	-
Replicated Atwood Wash Solids (this report)	1.81:1	1.76:1 to 1.79:1
3X Washed Solids (this report)	1.64:1	-
6X Washed Solids (this report)	1.59:1	1.50:1 to 1.55:1

22. The data summarized above clearly demonstrates that as the recovered solids are subjected to progressively more thorough washing, the molar ratio of sitagliptin to phosphoric acid drops from about 1.8:1 (in the Replicated Atwood Wash Product) to less than 1.6:1 (in the 6X washed Solids) without any change in the structure of the crystalline component of the solids. This proves that Prof. Atwood's "filtration and washing" step was ineffective, and that the more thorough washings that I performed removed a significant amount of unreacted starting materials, impurities and/or byproducts that were still present in the product after Prof. Atwood's "filtration and washing" step. Accordingly, Prof. Atwood's New Experiment is misleading, and the conclusions he draws therefrom are incorrect.

23. Had Prof. Atwood more thoroughly washed his solids in his New Experiment, he would have found that his crystalline product has a ratio of less than 1.6 moles of sitagliptin per 1 mole of phosphoric acid. The progressive washing experiments that I conducted provided solids that approach a ratio of 1.5 moles of sitagliptin per 1 mole of phosphoric acid. Such a product cannot be a non-DHP salt. This is because phosphoric acid cannot transfer one half of a proton to a sitagliptin molecule to make a 1.5:1 sitagliptin to phosphoric acid salt.

24. The most likely explanation for my results is that the crystalline product that I recovered is a co-crystal containing a ratio of two molecules of sitagliptin DHP Salt to one molecule of unreacted sitagliptin free base.

25. I conducted additional analyses to examine the products of my experiments, such as indexation, DSC, TGA, ^{1}H NMR, solid-state ^{15}N and ^{13}C NMR, and Karl Fischer analysis. The data obtained in these additional analyses (*see* Exhibits C and J) are supportive of the conclusions presented herein. However, as the analyses described above clearly rebut Prof. Atwood's New Experiment and the conclusions he draws therefrom, I did not find it necessary to rely on these additional analyses.

26. Attached as Exhibit K is a Project Sample Report identifying all of the analyses that I conducted in relation to this declaration. The Project Sample Report identifies the analyses on which I rely as well as analyses on which I do not rely.

Conclusion

27. Upon review of the Second Atwood Affidavit, it is apparent that several aspects of Prof. Atwood's "filtration and washing" step may have rendered it inadequate for removing impurities, byproducts and/or unreacted starting materials in solids that he recovered from his New Experiment.

28. The experiments that I conducted, which are described above, prove that Prof. Atwood's "filtration and washing step" was indeed ineffective in removing impurities, byproducts and/or unreacted starting materials in the solids that he recovered from his New Experiment. Accordingly, Prof. Atwood's conclusion that his New Experiment shows that "the same 2:1 phosphate salt was obtained in all procedures, with or without Dr. Chyall's suggested work up" (paragraph 5), is incorrect.

29. Had Prof. Atwood more thoroughly washed his solids in his New Experiment, he would have found that the Atwood Washed Solids still contained a significant amount of unreacted starting materials, impurities and/or byproducts, and that his crystalline product cannot be a "2:1 salt" of sitagliptin and phosphoric acid, or any non-DHP salt.

30. I therefore remain fully convinced that the only pharmaceutically suitable stable salt that will result from a reaction of sitagliptin free base and phosphoric acid is the DHP Salt, a salt containing a 1:1 ratio of sitagliptin to phosphoric acid.

Date: February 19, 2013

Leonard J. Chyall

Leonard J. Chyall, Ph.D.
President, Chyall Pharmaceutical Consulting LLC

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 1.01 - 39.98 °2θ Step Size: 0.008 °2θ
Collection Time: 1950 s Scan Speed: 1.2°/min Slit: DS: 1/2° SS: null Revolution Time: 1.0 s Mode: Transmission

563271 308390, Compound 184, D6655070112 null short AS extension in place, air 04-Dec-2012 08:37:35

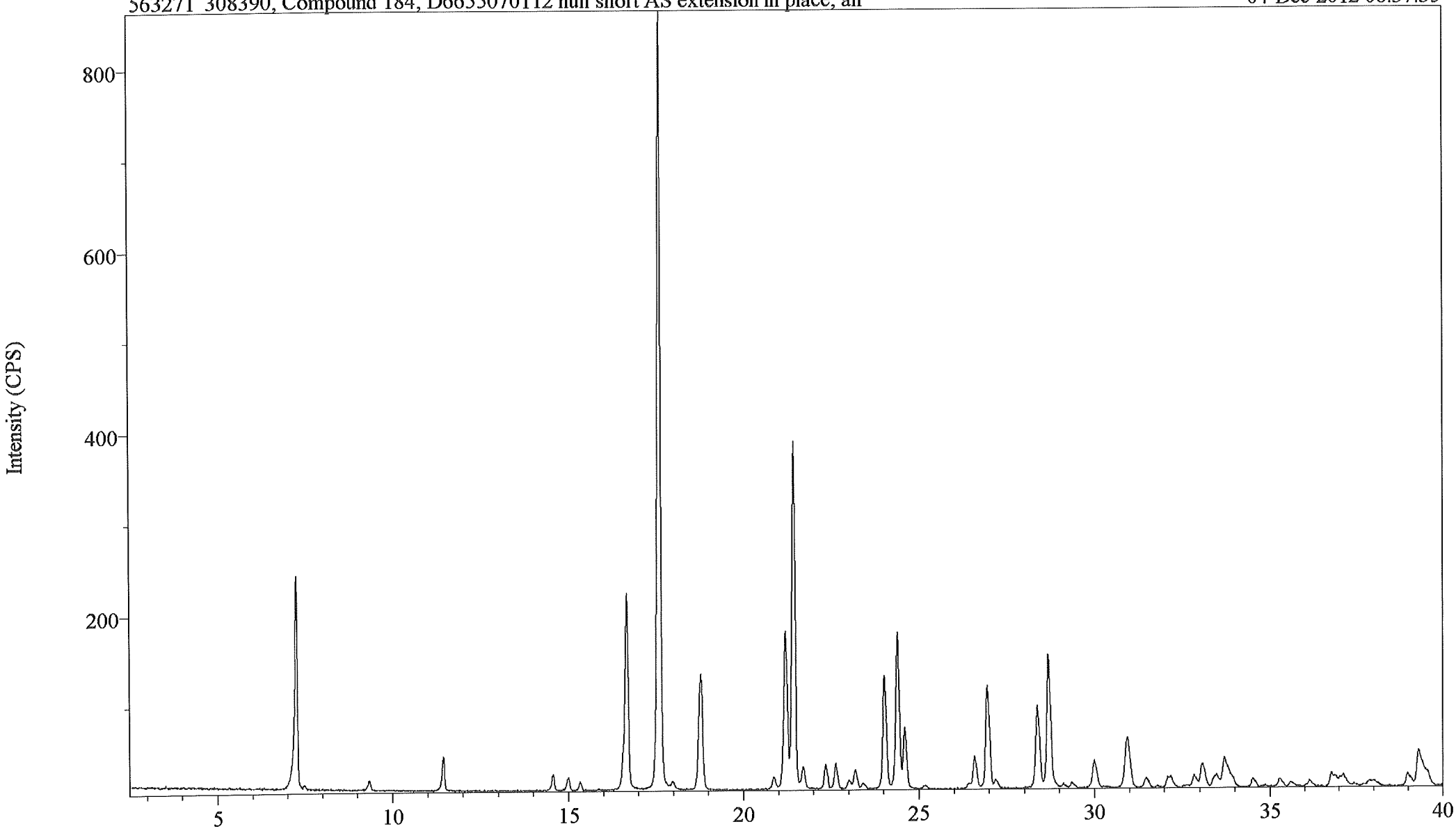


Image by File Monkey v3.2.3

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1850 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

564084 314339, 5135-02-01 Compound 184 spinning

07-Dec-2012 17:25:03

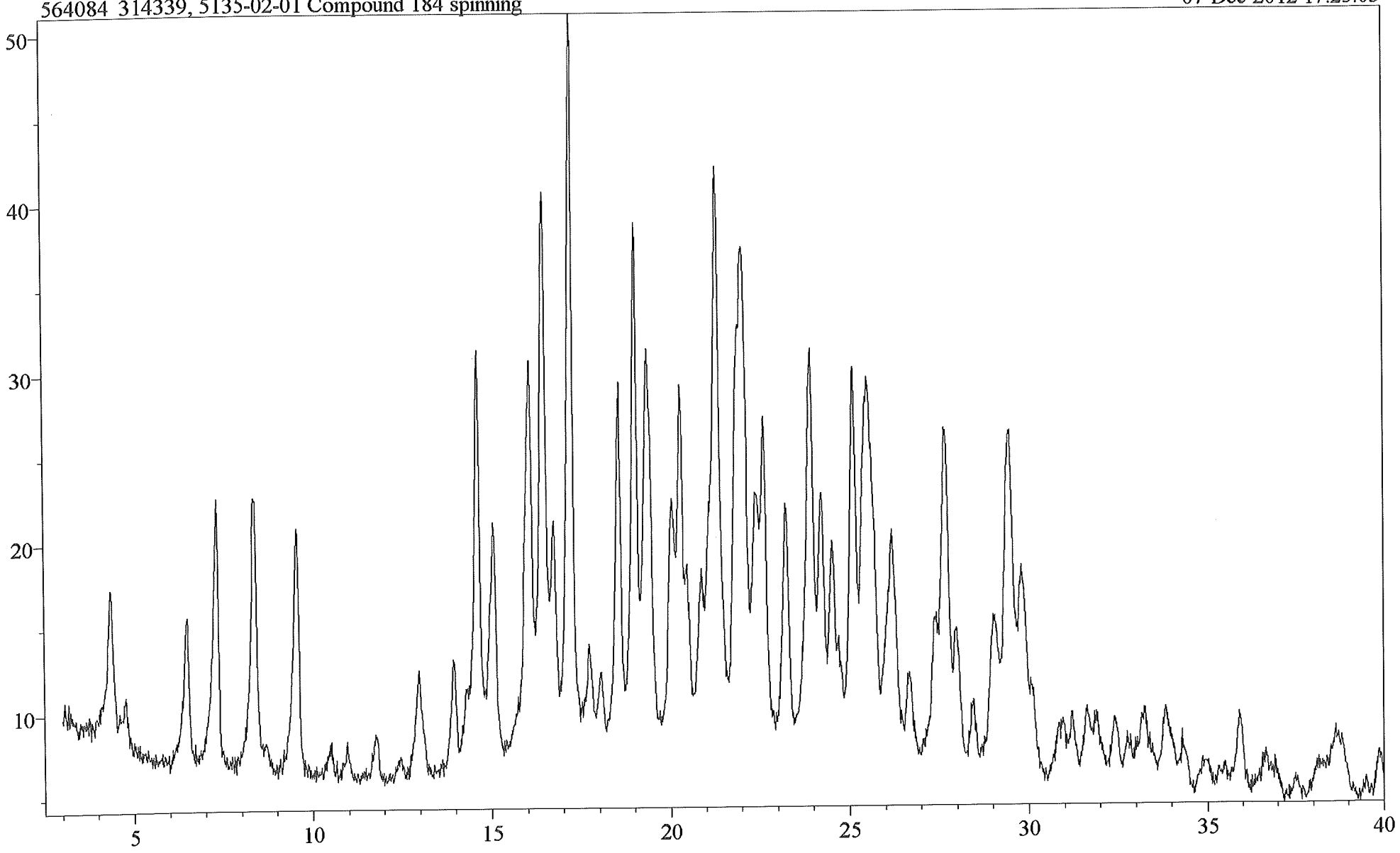


Image by File Monkey v3.2.3

SITAGLIPTIN BASE LIMS 308390
FLUKE THERMOMETER SSCI 1987
WHATMAN FILTER PAPER 42 70mm Lot 7593618
PHOSPHORIC ACID, ≥85% wt % ALDRICH Lot MKBJ4294V LIMS 295670 exp 4/23/17
WATER EMD Lot 52131 LIMS 309745 exp 5/31/13
IPA MALLINCKRODT Lot JI2B15 LIMS 233441 exp 8/7/15
~~pipette SSCI# 1441~~ IE 12/3/2012 KSO SSCI 1143 exp 4/30/13
Dataplate SSCI# 1209 ; 1156
Timer SSCI# 1806 cal due 4/24/13

Continued on Page

Read and Understood By

Karen S. Hurst — Signed — 12/3/2012 — Date
K. James Ely — Signed — 12/14/12 — Date

Seal #4; see 5135-01 equipment; reagents

5135-02-01 Erlenmeyer 25mL empty: 24.2306g
Weighed into flask: 1.5007g LIMS 308390
Added 1x 3200µl IPA LIMS 233441 - solids present
Added 1x 1400µl H_2O LIMS 309745 - solids present
Stirred on data plate, ambient T, using magnetic stirrer - clear solution after ~9min
Added 122.0µl LIMS 295670 (pipette LIMS 262443)
Heated on data plate to 70°C (Fluke #1987), 300rpm
Stirred @ 70°C for 15min (SSCI #1806), 300rpm
Placed on data plate - ambient T, stir, 400rpm
@ 11:17 (wall clock) encapped.
13:20 (wall clock) ↑ stir rate 500rpm
Left to stir overnight

Continued on Page

Read and Understood By

Signed 12/3/12 Date

Signed 12/14/12 Date

Phosphoric acid LIMS 295670 Vial #13

density determination ~~44~~ IE 12/3/2012KSO

Vial placed on bal, tared ; 1 ml LIMS 295670 added

① 1.7637g - discarded
② 1.7614g - discarded
③ 1.7649g - discarded

} AVE 1.7633 g/ml

pipette LIMS 267516

Want 0.215g 85% H_3PO_4 : 0.215g / 1.7633g/ml = 0.1219 ml = 121.9 µl

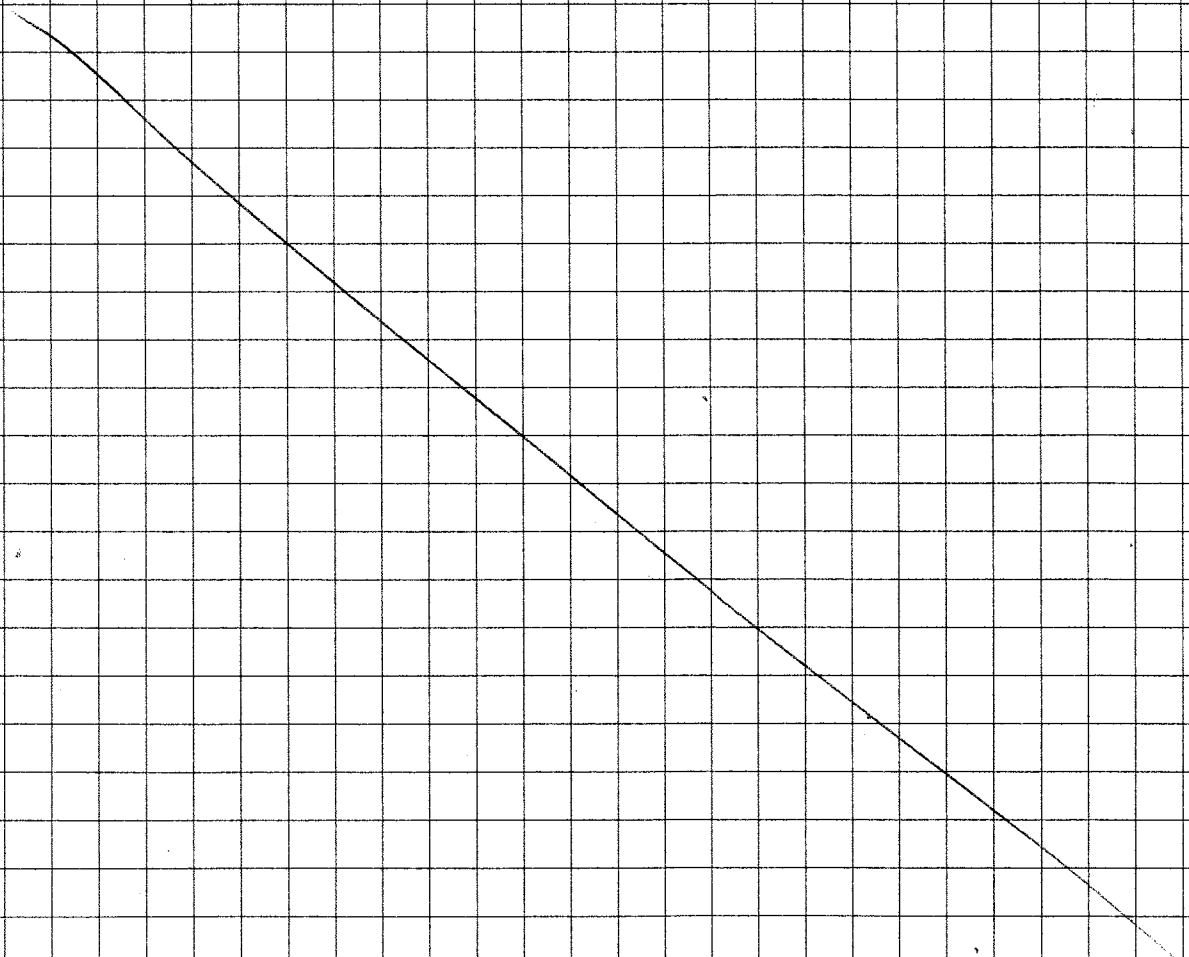
Continued on Page

Read and Understood By

Karen Hurst — Signed — 12/3/12 Date

K. James Ely — Signed — 12/14/12 Date

LIMS 308390 POST XRPD 5135-04-01



Continued on Page

Read and Understood By

Anurag Adhiwan 12/4/12 — Signed / Date

K. James Ely 12/4/12 — Signed / Date

5135-02-01 After 23 hr 28 min (timer SSC# 1806) - Clear, colorless solution.
Placed erlenmeyer flask into ice bath - stirring continued @ 500 rpm @ 10:55 (wall clock)
T_{bath} 1°C (Lims 75262 due 3/2013)
White solids noted after ~ 2 hrs (13:00 wall clock) - continued stirring in ice bath T_{bath} = 0°C (Lims 75262)
15:00 (wall clock) increase in mass of white solids
Let stir in ice bath overnight
see 5135-01 for equipment not listed on this page

Continued on Page

Read and Understood By

Karen L. Sweet — Signed — 12/4/2012 — Date

K. James Ely — Signed — 12/14/12 — Date

5135-06-01 wt 20 mL beaker: 13.9027 g, tared balance (cal #4)
added LIMS 308390 : 1.4997 g
Added 3 x 1000 µL + 1 x 200 µL IPA LIMS 233441
Added 1 x 400 µL + ~~2 x~~ IE 12/4/12 KSG / x 1000 µL H_2O LIMS 309745
Added magnetic stir bar & stirred @ ambient
T on data plate (SSCI # 1156)
pipette LIMS # 7516
Clear solution after ~10 min (timer S/N 12201733)

$$\frac{1.4997\ g}{407.314\ g/mol} = 3.68 \times 10^{-3}\ moles$$

H_3PO_4 needed:

$$\frac{1/2 \cdot 3.68 \times 10^{-3}\ moles \cdot 98\ g/mol}{0.928} = 0.1943\ g\ H_3PO_4$$

~~98 g/mol~~ IE 12/4/2012 KSG

See 5135-01 for equipment & reagents
Into tared 20 mL beaker weighed 0.1943 g LIMS 295670 (cal #13)
Pipetted H_3PO_4 into clear solution dropwise w/ stirring. Used clear solution to rinse beaker containing H_3PO_4 & returned to original beaker w/ free base.
Heated from ambient to 70 °C in 13 min (timer as above) w/ stirring (Fluke SSCI # 1987)
Stirred @ 70 °C for 15 min (timer S/N 12201733)
Removed from heat & continued stirring @ ambient
14:35 (wall clock) – very viscous
16:00 too viscous to stir.
Left uncovered in hood

Continued on Page

Read and Understood By

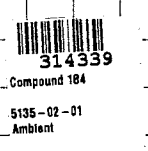
Signed: [signature] Date: 12/4/2012
Signed: R James Date: 12/14/12

5135-02-01 Sample has solids/oil - white solids, not sticking
T bath = 0°C (LIMS 75262) after 21 hr 38 min (timer SSCF 1806)
Scraped solids (from flask using metal spatula)
placed on filter paper (Whatman #42 see 5135-01)
Vacuum filtered for 5 min (timer SSCF 1806) — IE 12/5/2012 KEG
Added 3 ml to rinse solids (1 x 3000 ul IPA)
pipette LIMS 234547
IPA LIMS 233441 see 5135-01
Repeated wash 1 x 3000 µl IPA 2x total wash solvent = 3x3 = 9 ml
Allowed vacuum by aspirator for 10 min (timer SSCF 1806)
after final wash
Vial #4 wt glass jar: 115.8830 g
removed sample from filter paper & placed in jar
jar + sample: 118.2364 g
Covered jar w/ KimWipe held in place w/ rubber band
& placed in VO #11 T = ambient = 23°C LIMS 295940 pump SSCF #1305
vacuum = 30 in Hg (gauge)
Timer SSCF #1806

5135-07-01 Filtrate & combined washes saved in clear vial.
~~pipette~~ IE 12/5/2012 KEG
Wet (damp) sample (118.2364 - 115.8830) g = 2.3534 g

5135-02-01 Timer: 14 hr 8 min - removed from VO #11,
T = 24°C LIMS 295940. White solids
Wt jar + sample: 117.3248 g
Capped jar - Submit XRPD
Sample wt (117.3248 - 115.8830) g = 1.4418 g

Vial #4



Continued on Page

Read and Understood By

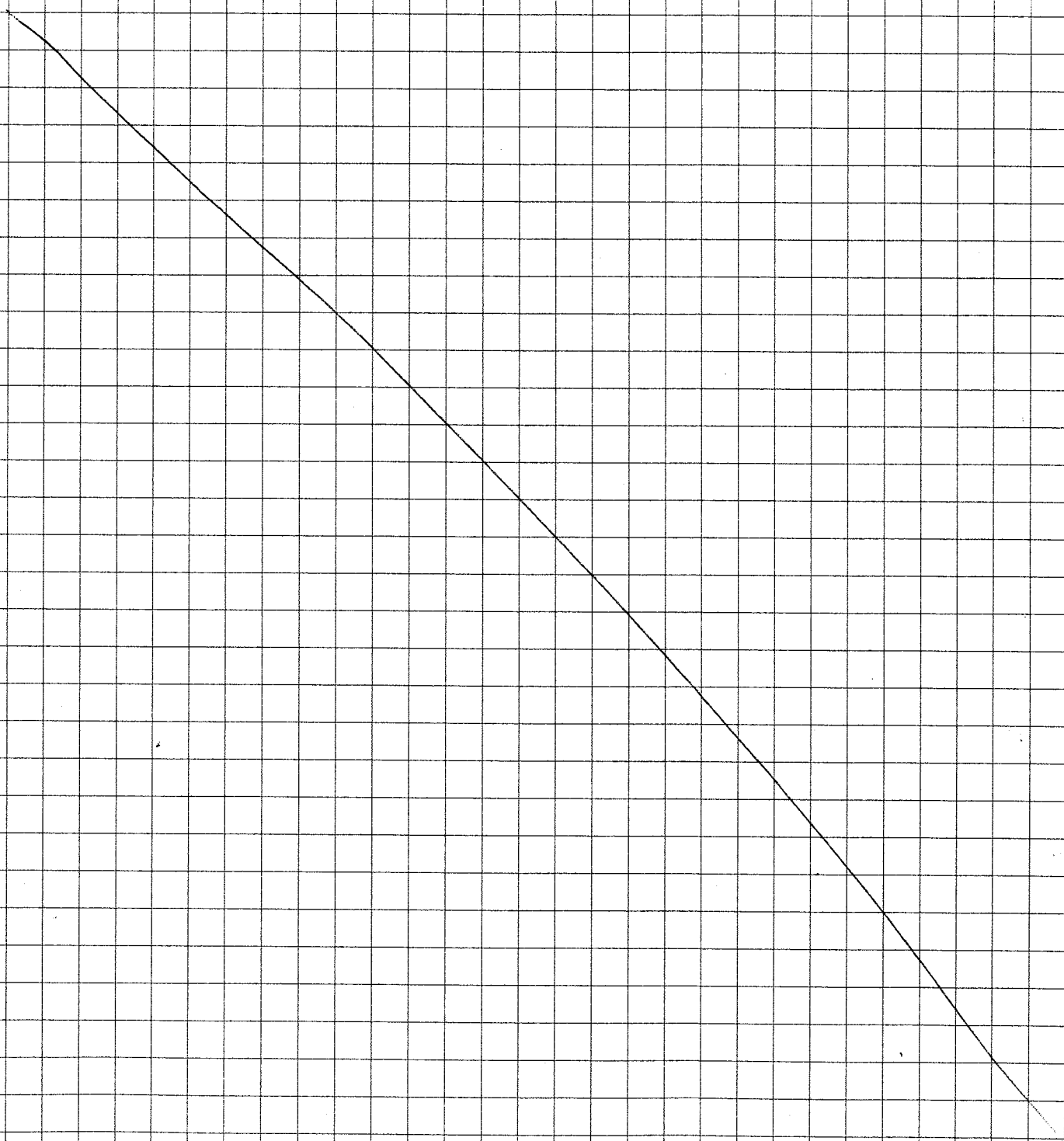
Karen J. Shaw — Signed — 12/5/2012 — Date
K. James — Signed — 12/19/12 — Date

PROJECT ~~EL20110011~~ EL20100011 IE12/5/2012RSC CMPD 184

Notebook No. 5135

Continued From Page

5135-06-01 Sample is clear, viscous & tacky paste. Covered beaker w/ parafilm @ 15:00 (wall clock) & placed in Ref #2.

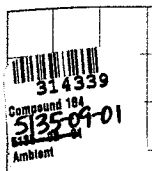


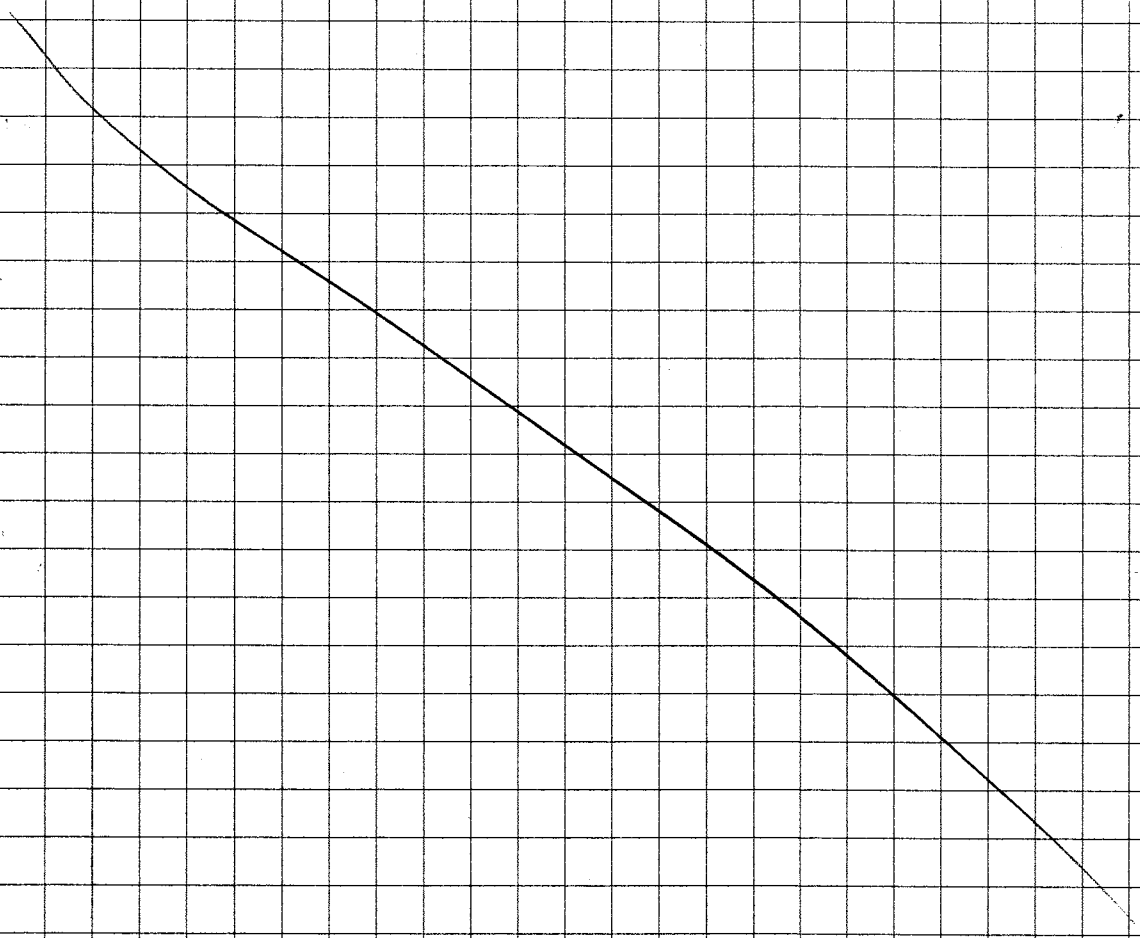
Continued on Page

Read and Understood By

Karen L. Thurmond — Signed — 12/5/2012 — Date

K. James Ely — Signed — 12/19/12 — Date





Continued on Page

Read and Understood By

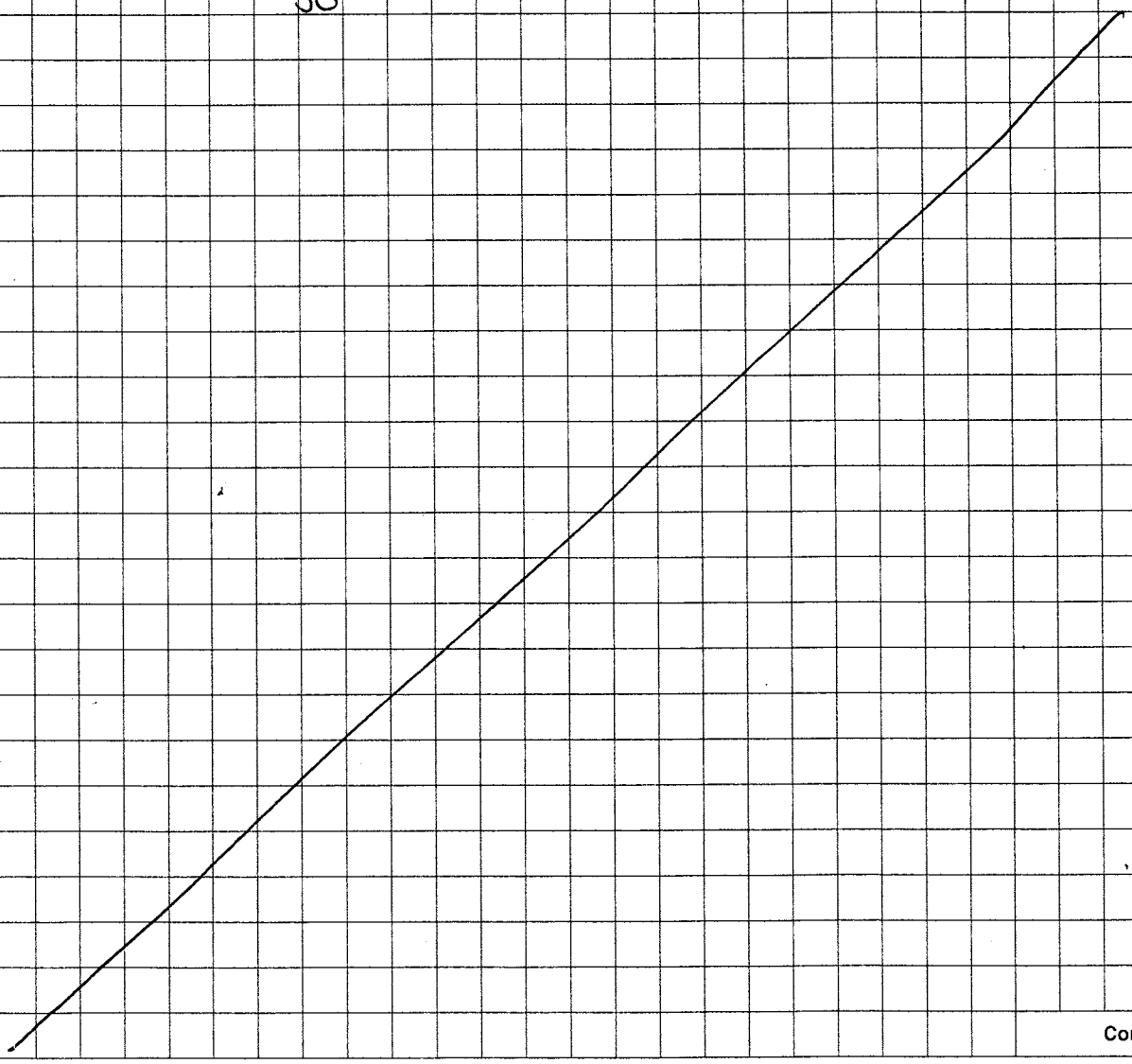
Aurora Atkinson 12/6/12
Signed Date

K. James 12/19/12
Signed Date

Placed a ~ 1/2" layer of molecular sieves (3Å, 1.6mm pellets, Sigma-Aldrich, Batch#MKBA0446, exp 02/13/14) into the bottom of an erlenmeyer flask, ~100mL of anhydrous, methanol (LIMS#309015, exp 09/25/17) added. Swirled sieves w/ methanol and transferred to buchner funnel and vacuum flask. Sieves rinsed w/ ~2 equivalent amounts of methanol.

Sieves transferred to small jar and placed into vac oven #4. Temperature @ ~190°C (Thermometer #75252), vac pulled ~ ~~25~~ 30 in Hg. JE-PEW 12/7/12 5135-10-01

Dye added 12/10/12 PEW 12/10/12



Continued on Page

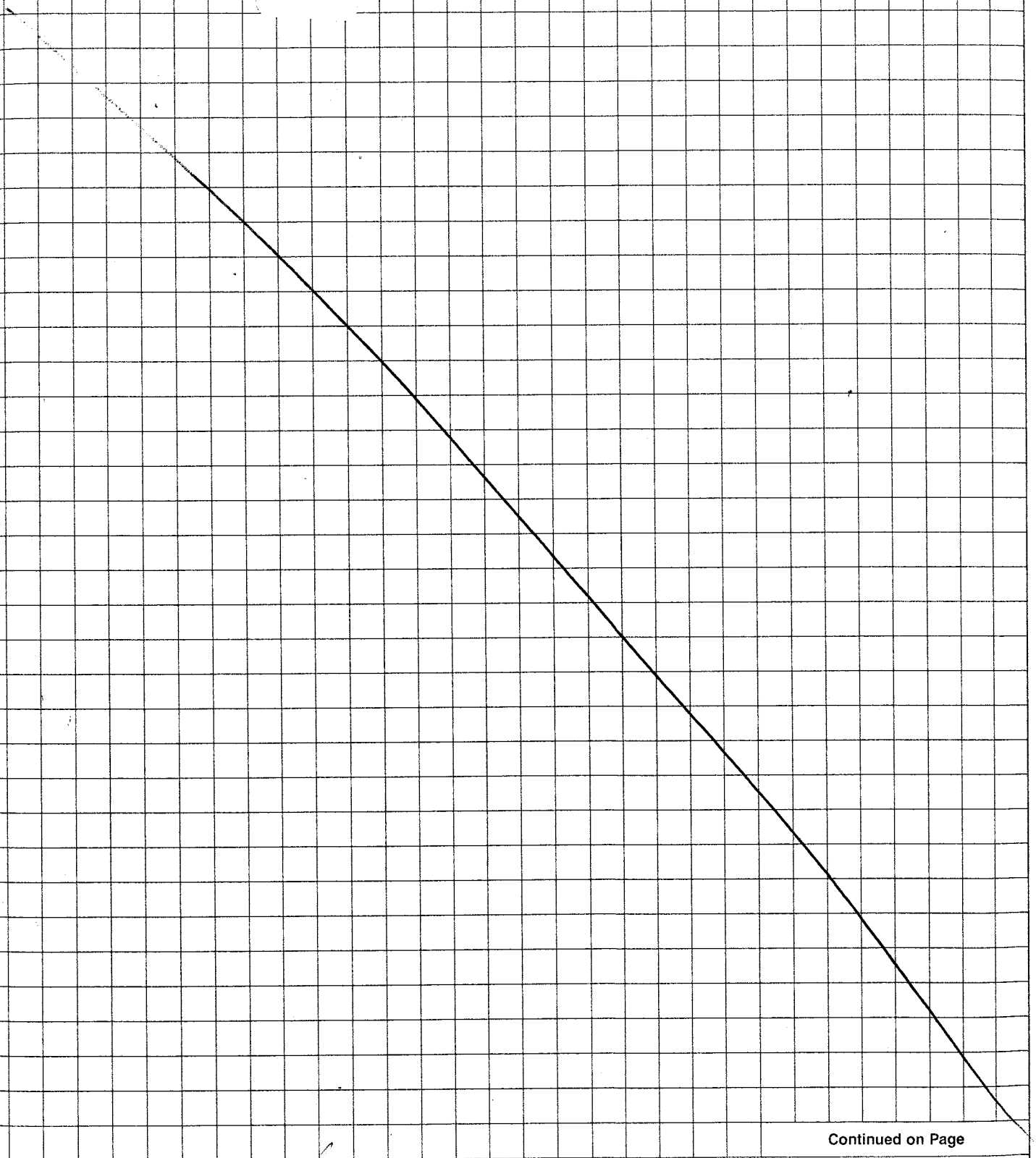
Read and Understood By

Peter E. Dell — Signed — 12/07/12 — Date

K. James Ely — Signed — 12/19/12 — Date

file 564084

314339
Compound 184
5135-11-01
B196-02-01
Ambient



Continued on Page

Read and Understood By

Signed — Date — Signed — Date

12/7/2012 — 12/19/12

5135-12-01 wt 50ml Erlenmeyer : 40.5301g Lot #4
added LIMS 308390 wt : 3.0027g
added (6x1000ul + 1x400ul) IPA
added (1x800ul + 2x1000ul) H_2O
added magnetic stir bar
Stirred on data plate @ ambient T; 300rpm
Clear solution observed after 4min 38sec
Stirred for a total of 7min 45sec
IE 12/10/2012 KSG
Weighed LIMS 295670 into glass vial (tared) : 0.4300g
Transferred H_3PO_4 into Erlenmeyer soln using pipette
rinsed vial & pipette w/ Erlenmeyer contents.
↑T data plate to 70°C; stirring continued @ 300rpm
Solution T = 70°C after 20min
Stirred @ 70°C for 15min
Discontinued heating; stir 500rpm (left on data plate)
Solution reached ambient T (25.2°C) in 40min
Solidification noted after ~2hrs; stir bar moving
but not stirring mixture.
After 4hr 16min - stir bar not moving -
left setting ON - cap (loose) in mouth of flask

12/10/12 KSG

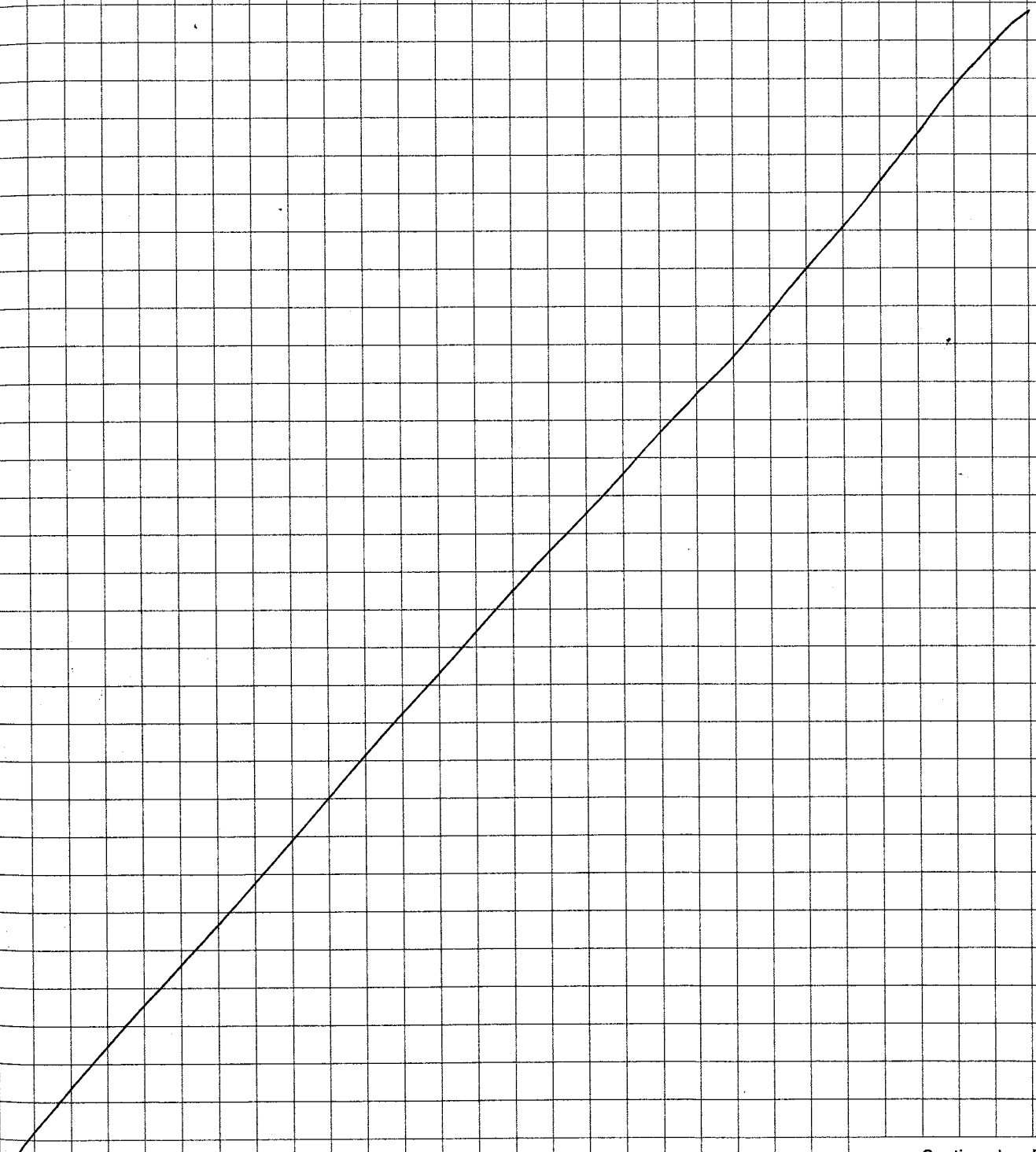
H_3PO_4 see 5135-01 LIMS 295670
Fluke thermometer 1987 due 1/13
timer SSCI # 1806 due 4/24/13
data plate SSCI # 1209
pipette SSCI # 0793 due 4/30/13
H_2O LIMS 309745 see 5135-01
IPA LIMS 233441 see 5135-01

Continued on Page

Read and Understood By

Signed 12/10/12 Date — Signed K. James Ely 12/19/12 Date

While still under vacuum, turned off oven. 5135-10-01 removed from oven #4 and transferred immediately to N_2 - purged glove bag (%RH = 0.9%, Hygrometer ID# 2129, Due 01/18/13)



Continued on Page

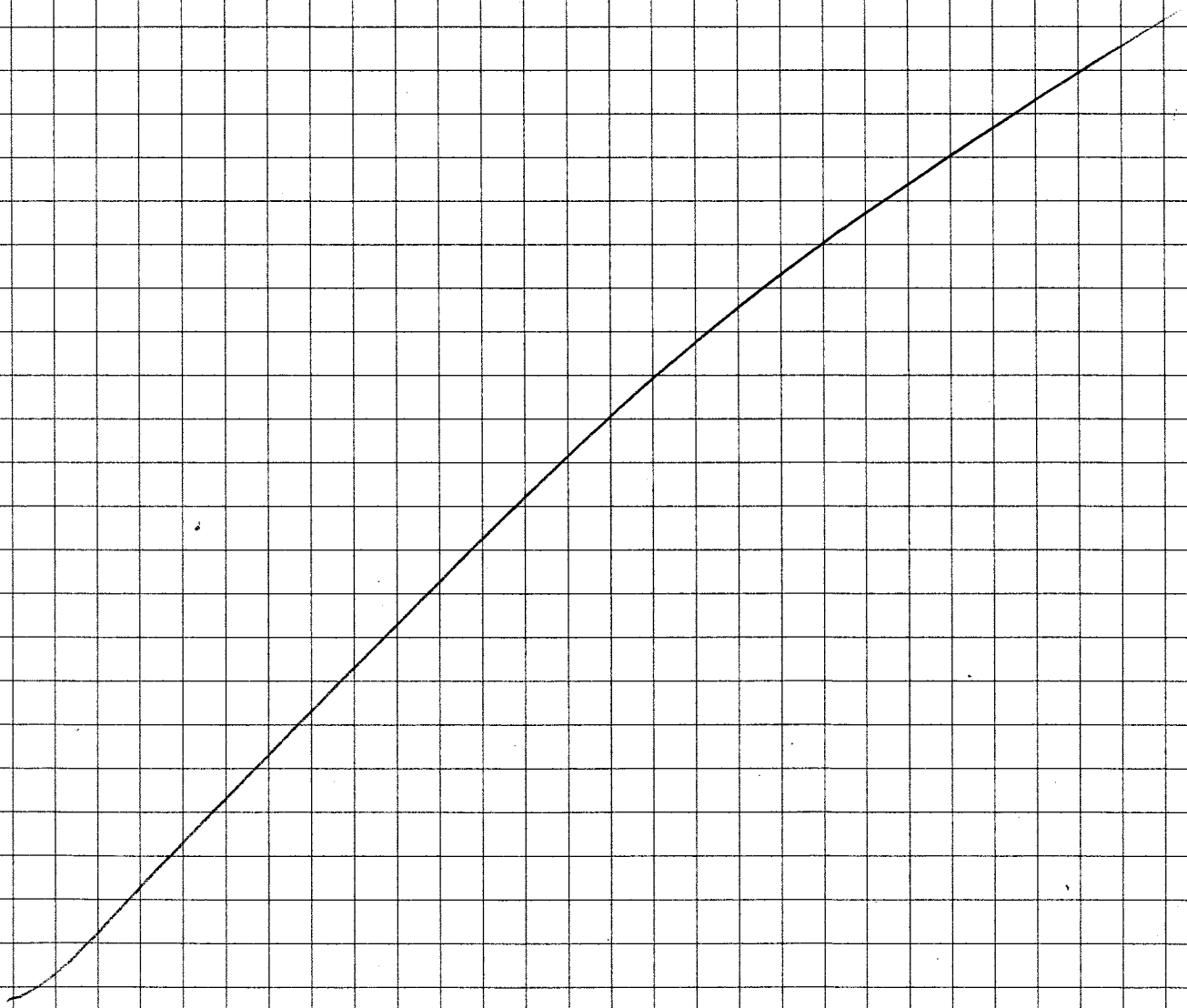
Read and Understood By

Signed	Date	Signed	Date
	12/10/12		12/14/2012

DSC #4 Sample Prep Balance # 16

Sample mass determined by weighing by difference per Method 2, USP<1251>.
Sample mass = Total mass − Tare mass

Sample ID	Filename	Total mass	Tare mass	Sample mass	Pan Type	Pos #
5135-14-01	Ref pan	N/A	52.48 mg	N/A	TOHSLP	R1
314339	564401	55.92 mg	53.14 mg	2.78 mg	TOHSLP	P1



Continued on Page

Read and Understood By

Signed — Date 11 Dec 12

Signed — Date 12/14/12

5135-12-01 (after sitting 22 hrs 05 min solids were scraped from flask & vacuum filtered (Whatman #2 see 5-135-01)
Washed with 1 x 18 mL IPA (filter cake)
Subsampled filter cake & submit XRD (564452)
Filtrate from initial filtrate & 1st wash
⇒ 5135-15-01 Capped & parafilmed
Washed filter cake w/ 1 x 18 mL IPA = 2nd washing
⇒ 5135-15-02 filtrate from 2nd washing placed in glass vial. Capped & parafilmed
Class A graduated cylinder pipette SSCI# 0793 due 4/30/13
Washed filter cake w/ 1 x 18 mL IPA = 3rd washing
Allowed solids to dry on paper while pulling suction.
Subsampled portion of solids & submit XRD
⇒ 5135-15-03
Saved filtrate from 3rd washing in glass vial, capped & parafilmed
⇒ 5135-15-04 3rd washing filtrate
Solids removed from filter paper & placed in glass jar. wt jar: 47.0947 g
+ solids: 49.6491 g
2.5544 g sample mass
Sample ID = 5135-15-05

314705 Compound 184 5135-12-01 Ambient

314731 Compound 184 5135-15-03 Ambient

314760 Compound 184 5135-15-05 Ambient

12/11/2012

Used #4
IPA LIMS 233441 see 5135-01
45 mm filter funnel

Continued on Page

Read and Understood By

Signed 12/11/2012 Date

R. James Signed 12/19/12 Date

5135-16-01 Vial tare wt.: 9.9492 g 10 ml vial (glass)
+ sample : 9.9604 g Lims 314339
sample wt 11.2 mg Lot #4

5135-16-02 IPA/H_2O
(HT 1 x 1000 µl + 1 x 400 µl) IPA + (11 x 1000 µl + 1 x 800 µl) H_2O
into glass vial. Capped & shook to mix

IPA } see 5135-01
H_2O }
pipette SSCI # 0793 exp 4/30/13

5135-16-01 Added 1 x 500 µl 5135-16-02 w/ sonication - solids dissolved.

$\frac{11.2 \text{ mg}}{.5 \text{ ml}} = \geq 22.4 \text{ mg/ml}$

Sample dissolved.

Continued on Page

Read and Understood By

Signed — Date 12/11/2012

Signed — Date 12/19/12

5135-17-01 Weighed 1.6923g 5135-15-05 onto glassine weigh paper.
Vial #4
Transferred solids to filter funnel w/ Whatmans #2 grade filter paper.
While pulling vacuum (water aspiration), washed solids w/ 1x 12ml IPA.
Filtrate 1st wash = 5135-17-02 into glass vial. Capped; parafilmed
Washed solids 2nd time 1x 12ml IPA
Filtrate 2nd wash = 5135-17-03 into glass vial. Capped; parafilmed
Washed solids 3rd time 1x 12ml IPA
Filtrate 3rd wash 5135-17-04 into glass vial, capped; parafilmed.
Dried solids in filter funnel pulling vacuum ~ 45 min
Transferred solids to glass vial:
Vial wt: 21.0494g
+ solids 22.5816g
1.5322g sample 5135-17-01

314783
Compound 184
5135-17-01
Ambient

5135-17-05 Weighed 9.7480g, LIMS 314783 into glass vial
Added 1x 12 ml IPA; Shook vigorously, then poured into filter funnel (Whatmans #2 grade) while pulling vacuum.
5135-17-06 Filtrate from 1st wash - placed in glass vial, capped; parafilmed.
Washed filter cake w/ 1x 12 ml IPA
5135-17-07 Filtrate from 2nd wash - placed in glass vial, capped; parafilmed.
Washed filter cake w/ 1x 12 ml IPA
Filtrate evaporated during filtration.
Left solids on filter w/ vacuum for ~ 1 hr to dry.
Vial (glass) 12.9460g
+ sample: 13.4010g ⇒ 0.4550g sample

314802
Compound 184
5135-17-05
Ambient

Class A graduated cylinder, 10ml
IPA see 5135-01 LIMS 233441
Vial #4, 45mm filter funnel

Continued on Page

Read and Understood By

Signed — Date 12/11/2012 — Signed R. James Ely — Date 12/19/12

5135-18-01 glass vial: 12.9148g lims 314783 Vial #4
added 12/12/12 KSC as written on vial
+ Sample 12.9868g
sample wt: 0.0720g ← added 12/12/12 KSC

Covered vial w/ Kim Wipe held in place w/ rubber band
Placed VO#11 @ ambient T (T= 23°C lims 295940) @ 16:25 (clock)
30 in Hg per gauge; pump SSC#1305

Continued on Page

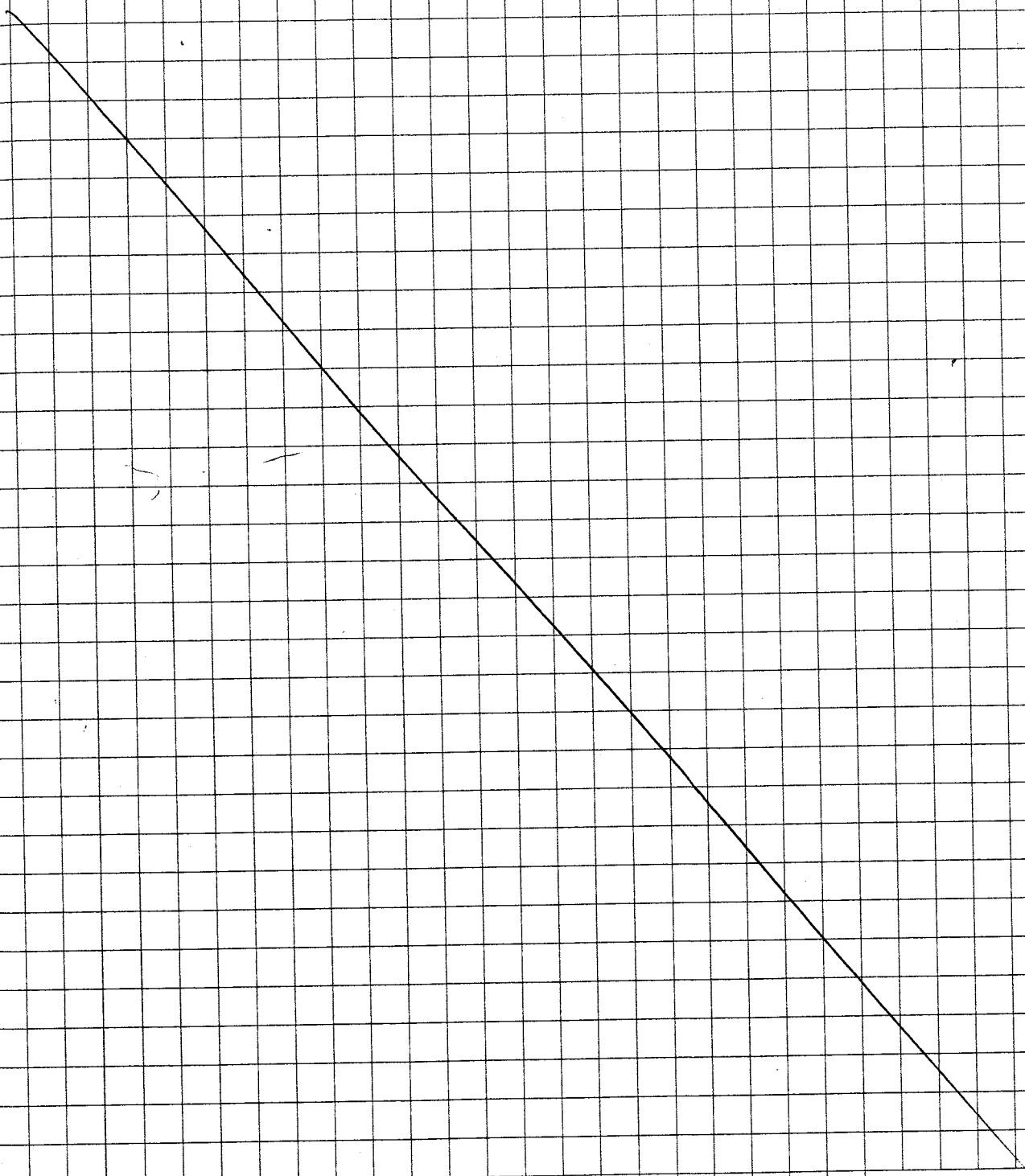
Read and Understood By

Karen Gust — Signed — 12/11/2012 — Date

K. James — Signed — 12/19/12 — Date

5135-18-01 Removed from VO#11 (ambient) T=23°C Lims 295940
30 in Hg per gauge @ 00:25 (wall clock)
Vial + sample: 12.9823g Lot #4
Submit XRPD ; file 564724

314801
Compound 184
5135-18-01
Ambient



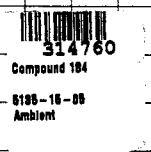
Continued on Page

Read and Understood By

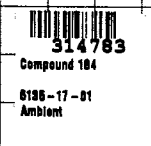
Karen S. Sirk — Signed — 12/12/2012 — Date

F. James Eby — Signed — 12/19/12 — Date

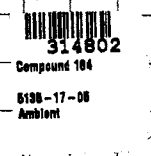
The following post XRPD materials were saved in clear, labeled vials, as indicated:



5135-20-01



5135-20-02



5135.20-03

Continued on Page

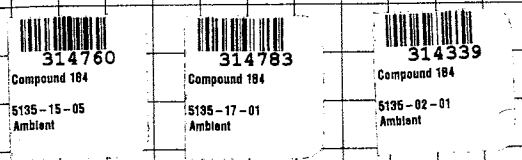
Read and Understood By

Signed	Date	Signed	Date
Laurel Henderson	12 Dec. 2012	R. James Ely	12/19/12

~~Samp~~ IE 12/12/12 KSL

The following samples were subsampled as follows:

LIMS	Notebook	*Subsample wt
314760	5135-15-05	vial: 7.8382g + sample: 8.0489g = 0.2107g
314783	5135-17-01	vial: 7.8695g + sample: 8.0862g = 0.2167g
314339	5135-02-01	vial: 7.7260g + sample: 8.0169g = 0.2909g



Vial #4

* wts of 2dram vials w/ LIMS label

The 3 subsamples were placed in the fume hood; no caps. These ~~samples~~ subsamples were taken 12/12/12 are for elemental analysis. 18:12 (wall clock)

5135-21-01 Weighed LIMS 314802 into glass vial

vial wt: 12.9731g

+ sample: 13.0888g

0.1157g capped vial. Vial #4

5135-17-05 placed uncapped in fume hood.

5135-21-02 Weighed LIMS 314801 into glass vial

vial wt: 5.1315g

+ sample: 5.1527g

0.0212g capped vial Vial #4

5135-18-01 placed uncapped in fume hood.

Continued on Page

Read and Understood By

Karen S. Hirsh — Signed — 12/12/12 Date

R. James Ely — Signed — 12/19/12 Date

Balance # 16, Level

Example calc: spl wt = (spl + pan wt) − pan wt

Weighed the indicated amount of sample into the corresponding DSC pan.

Sample ID	Lic #	pan wt (mg)	spl + pan wt (mg)	spl wt (mg)	Pan Type	Pos #
5135-22-01	Ref. Pan	52.79 mg	52.79 mg	0.00 mg	TZHSLP	R1
314760	564942	52.58 mg	54.17 mg	1.59 mg	TZHSLP	1
314788	564944	52.46 mg	53.93 mg	1.47 mg	TZHSLP	2

Continued on Page

Read and Understood By

Signed — Date 12/12/12

R. James Signed — Date 12/19/12

Balance #16

Example Calc: spl wt = (spl + Pan wt) - Pan wt

Weighed the indicated amount of sample into the corresponding DSC pan.

Sample ID	File #	Pan wt (mg)	Spl + Pan wt (mg)	Spl wt (mg)	Pan Type	Pos #
5135-23-01	Ref. Pan	52.43mg	52.43mg	0.00mg SL 12/12/12 ~~T@HSLP~~	T@HSLP	R1
314760	565125	52.58mg	54.05mg	1.47mg	T@HSLP	1
314783	565124	52.14mg	53.58mg	1.44mg	T@HSLP	2

Continued on Page

Read and Understood By

L L — Signed — 12/13/12 Date

R. James Ely — Signed — 12/19/12 Date

KF-C - STROMBOLI BAL 14

	LIMS 314336	LIMS 314760	LIMS 314783
SCORING			
TARE	19.2816g	19.3812g	19.0016g
TARE + SAMPLE	19.2996g	19.3954g	19.0123g
SAMPLE	0.0180g	0.0142g	0.0107g
RESULT	3.091 %.	6.366 %.	6.244 %
TARGET RANGE = 16-64 mg		8-31mg	8-32mg
ANALYSIS			
SAMPLE 1	35.8mg (P1)	~~36.8mg~~ 31.9mg 12/13/12 IEOM	31.0mg (P3) 31.9mg (P5)
SAMPLE 2	56.6mg (P2)	56.6mg	30.0mg (P4) 31.1mg (P6)
		IEOM 12/13/12	
RESULT 1	*3.120	RESULT 1 6.407 %	RESULT 1 6.382 %.
RESULT 2	3.035	RESULT 2 6.267 %.	RESULT 2 6.430 %.
AVG	3.078 %.	AVG 6.337 %.	AVG 6.406 %.

* See p 25 for calculation

Continued on Page 25

Read and Understood By

Signed — Date 12/13/12

Signed — Date 12/19/12

LIMS314339 RUN #1, COMMUNICATION ERROR. RAW DATA WAS MANUALLY SAVED, BUT SAMPLE MASS INFO WAS NOT TRANSFERRED TO COMPUTER FROM COULOMETER.

MANUALLY CALCULATE H_2O CONTENT.

FROM FILE 108_20121213_1348_1.PDF

TOTAL WATER = 1204.90 µg
ANALYSIS TIME = 252 sec (4.2 MIN)
DRIFT = 5 µg/min
BLANK = 67 µg

$$1204.90\,\mu g - 67\,\mu g - \left(\frac{5\,\mu g}{MIN} \times 4.2\,MIN\right) = 1116.9\,\mu g$$

$$\frac{1116.9\,\mu g}{*35800\,\mu g} \times 100 = \boxed{3.120\%}$$

* 35.8 mg from page 24

SEE FILE CORRECTION 565225

SAVED CORRECTED DATA AS 108_20121213_1348.PDF

Continued on Page

Read and Understood By

Aurora Atkinson — Signed — 12/13/12 — Date
R. James Slay — Signed — 12/19/12 — Date

PROJECT EL20100011

Notebook No. 5135

Continued From Page

Subsamples of LIMS 314760, 314783, 314339 listed on 5135-21 capped, parafilmed, shipped for elemental analysis. Capped @ 11:00Am.

Continued on Page

* work completed on 12/13/2012 per LIMS entries, notebook page not signed & dated until 12/14/2012 KL

Read and Understood By

Karen S. Lusk — Signed — *12/14/2012 — Date

K. James Ely — Signed — 12/19/12 — Date

Continued from 5135-21 :

5135-18-01 removed from fumehood, capped; submitted XRD 2 days in fume hood uncapped
⇒ new sample ID 5135-27-01

315083 Compound 184 5135-27-01 Ambient

17 clarification 12/14/2012

5135-A-05 removed from fume hood, capped; submitted XRD 2 days in fume hood uncapped
⇒ new sample ID 5135-27-02

315084 Compound 184 5135-27-02 Ambient

RH ~20% Control Company RH pen LIMS 311402 due 8/31/13

315084 Compound 184 5135-27-02 Ambient

315083 Compound 184 5135-27-01 Ambient

post XRPD sample returned to original vial - all sample was utilized for testing

5135-27-03
post XRD

Continued on Page

Read and Understood By

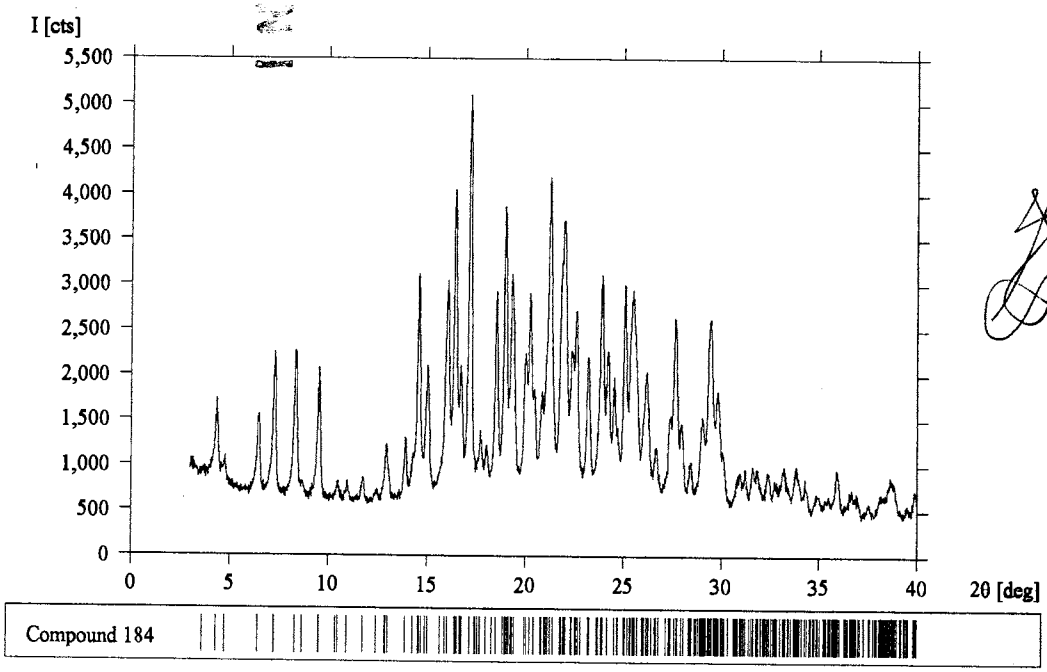
Signed 12/14/2012 Date

Signed 12/19/12 Date

XRPD file 564084 was indexed using Dicvol04 [1]

[1] A. Boultif ; D. Louër ; J. Appl. Cryst. Cryst., 37(3), 724-731, 2004

Indexing results for XRPD file 564084 collected with Cu-Kα radiation.



	Compound 184
Bravais Type	Primitive Monoclinic
a [Å]	21.319
b [Å]	6.202
c [Å]	25.387
α [deg]	90
β [deg]	107.50
γ [deg]	90
Volume [Å³/cell]	3,201.3
Chiral Contents?	Chiral
Extinction Symbol	P 1 21 1
Space Group(s)	$P2_1$ (4)
Source	Manual Input

5735-28
18 December 2012

Continued on Page

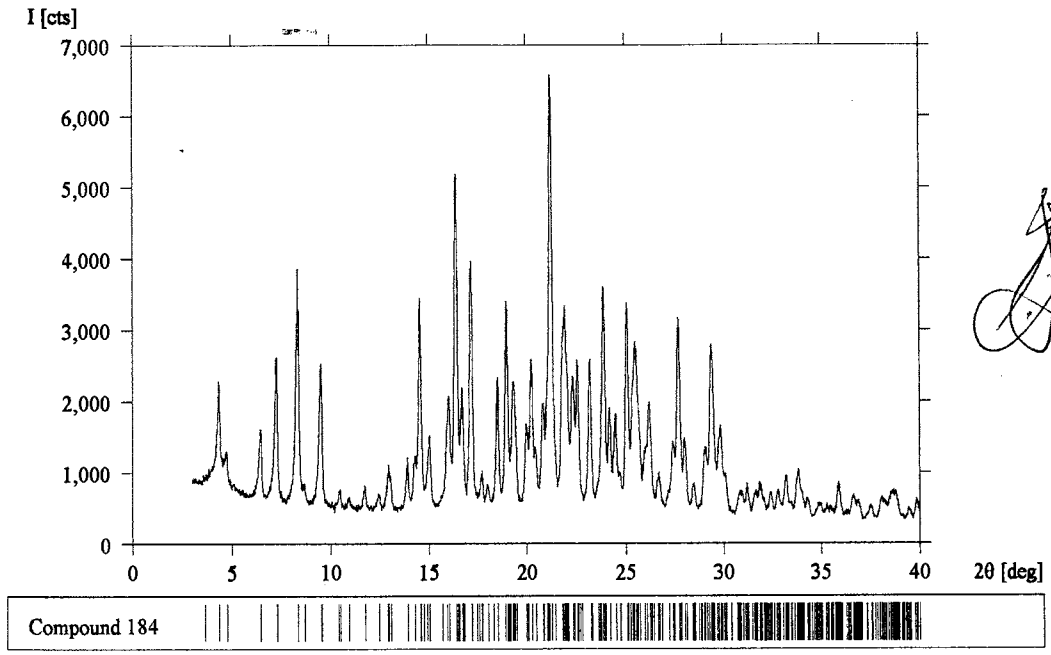
Read and Understood By

Signed — 18 December 2012 — Date

Signed — 18 Dec 2012 — Date

XRPD file 564765 was indexed using DicvolO4 [1]
[1] A. Boultif ; D. Louër ; J. Appl. Crystallogr., 37(5), 724-731, 2004

Indexing results for XRPD file 564765 collected with Cu-Kα radiation.



	Compound 184
Bravais Type	Primitive Monoclinic
a [Å]	21.325
b [Å]	6.205
c [Å]	25.432
α [deg]	90
β [deg]	107.57
γ [deg]	90
Volume [Å³/cell]	3,208.2
Chiral Contents?	Chiral
Extinction Symbol	P 1 21 1
Space Group(s)	$P2_1$ (4)
Source	Manual Input

5135-29
18 December 2012

Continued on Page

Read and Understood By

Signed | 18 December 2012 Date | Signed | 18 Dec 2012 Date

Lims 314783 (5135-17-01) was weighed as follows: Vial #4

Sample ID	wt vial	vial + sample	sample wt
5135-30-01	4.6796g	4.6903g	10.7mg
5135-30-02	4.6153g	4.6656g	50.3mg
5135-30-03	13.0020g	13.3235g	321.5mg

5135-30-03 Covered uncapped vial w/ Kim Wipe held in place w/ rubberer band. Placed vial in VO #11, ambient T (T=23°C Lims 25940)
@ 09:50Am Timer SSCI #1806. P=30in Hg (oven gauge); Pump SSCI #1305.
@ 17:50 Sample removed (from VO #11 (ambient T=23°C)
P=30in Hg prior to removing sample (Timer = 8hr 1min)
wt vial + sample: 13.3039g Vial #4
Vial + sample left in fume hood, uncapped

Continued on Page

Read and Understood By

Signed — Date 12/19/12 — Signed — Date 12/21/12

Weighed the following subsamples (Vial #4)

Original	subsample ID	vial wt	+ sample	sample wt
LIMS 308389	5135-31-01	4.6383 g	4.6886 g	50.3 mg
LIMS 308390	5135-31-02	4.6274 g	4.6775 g	50.1 mg

Samples submitted ^{13}C solution NMR.

Continued on Page

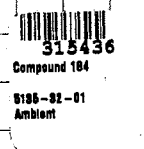
Read and Understood By

Karen S. Austin — Signed — 12/19/2012 — Date

K. James Ely — Signed — 12/21/12 — Date

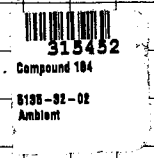
The following mixtures were made. Vial #16, 2dram vial

Sample ID	Vial wt	LIMS 308390 *	LIMS 308389 *
5135-32-01	7.84192g	35.70mg	44.33mg



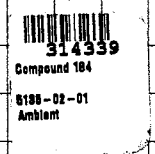
* Weights are actual weights in vial. Vial was tared on balance; LIMS 308390 weighed into vial. LIMS 308389 was IE 12/20/12 KSG
weighed on to glassine paper then transferred to tared vial containing LIMS 308390; weight recorded.

Sample ID	vial wt	+ LIMS 308396	wt LIMS 308390
5135-32-02	~~7.79397g~~ IE 12/20/12 KSG 7.79403g	7.81703g	= 23.0mg



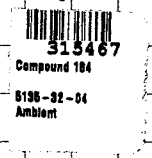
balance tared w/ vial & LIMS 308390 before weighing LIMS 308389 = wt 57.03mg

5135-32-03 Weighed 80.02 LIMS 314339 Sample 5135-02-01 into 2dram vial. Submit ^{13}C NMR solution



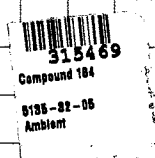
Sample ID	vial wt	+ LIMS 308390	
5135-32-04	~~7846.54mg~~ IE 12/20/12 KSG 7734.94mg	= 19.51mg LIMS 308390.	
	7715.43mg		

Tared vial; added LIMS 308389 (pre-weighed on glassine paper). Amount 308389 in vial: 60.51mg



Sample ID	vial wt	+ LIMS 308390	
5135-32-05	7806.19mg	7832.18mg	= 25.99mg LIMS 308390, tared vial

Added 53.98mg LIMS 308389



Vial #16
All samples submitted ^{13}C NMR solution.

Continued on Page

Read and Understood By

Warren A. Sun — Signed — 12/20/12 — Date

K. James Ely — Signed — 12/21/12 — Date

5135-30-03 Vial + sample: 13.3235g (vial #4; 10:35 Am (wall clock)
subsampled portion for elemental analysis:
5135-33-01 vial = 7.6710g + sample = 7.8684g
sample wt: 197.4mg
Vial capped & parafilmed for shipping
Remainder of sample submitted XRPD, DSC, KF, TGA

315535
Compound 184
5135-30-03
Ambient

Continued on Page

Read and Understood By

Karen Hurst — Signed — 12/21/2012 — Date
R. James Ely — Signed — 12/21/12 — Date

LIMS 315535
SCOPING RUN ~ 11.9mg RESULT = 6.563%

TARGET SAMPLE MASS = 8 - 30mg

LIMS 315535

	TARE	TARE + SAMPLE	SAMPLE	RESULT
RUN 1	19.2075g	19.2197g (Weight 12/12/12)	12.2mg	6.383%
RUN 2	19.3378g	19.3482g	10.4mg	6.919%

AVG = $\left(\frac{6.383 + 6.919}{2}\right)$ = 6.651%

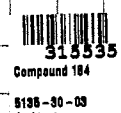
Continued on Page

Read and Understood By

Courtney Williams 12/21/12
Signed Date

K. James Ely 12/21/12
Signed Date

The measured material was stored in a clean, labeled vial, as indicated



5135-35-01

EMPTY

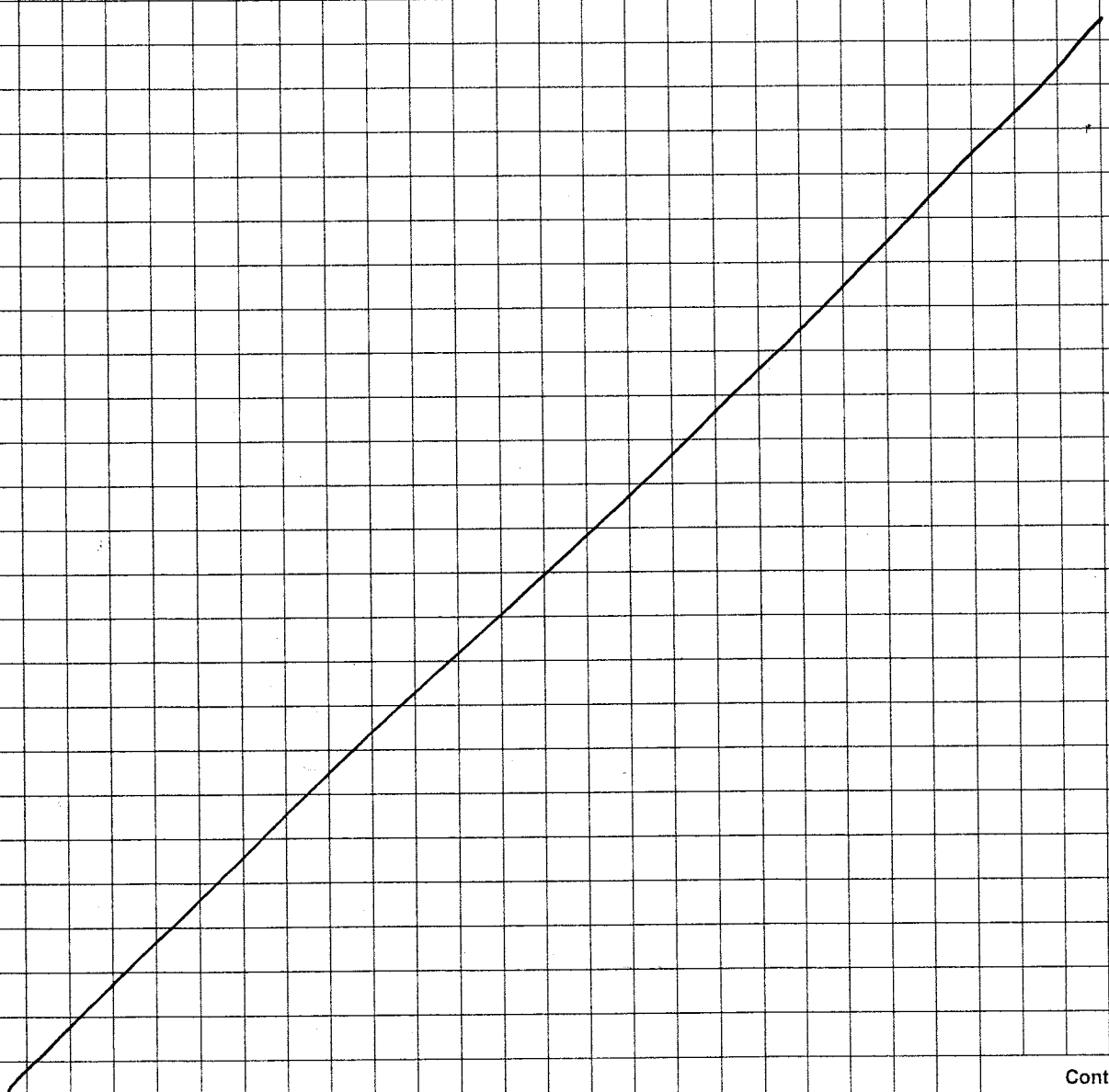
Continued on Page

Read and Understood By

Signed	Date	Signed	Date
Camille Westrom	21 Dec 2012	K. James Ely	12/21/12

Packed the following samples into separate 4mm zirconia rotors using SSNMR packing tools. Rotors stored in separate Eppendorf tubes until analysis by ~~FF~~ REVISED 12/11/12 15N CPMAS SSNMR

LIMS#	Sample ID	Rotor #
308389	5143-01-01	RSN40044
308390	5143-01-02	RSN40033



Continued on Page

Read and Understood By

Signed 12/11/12 Date

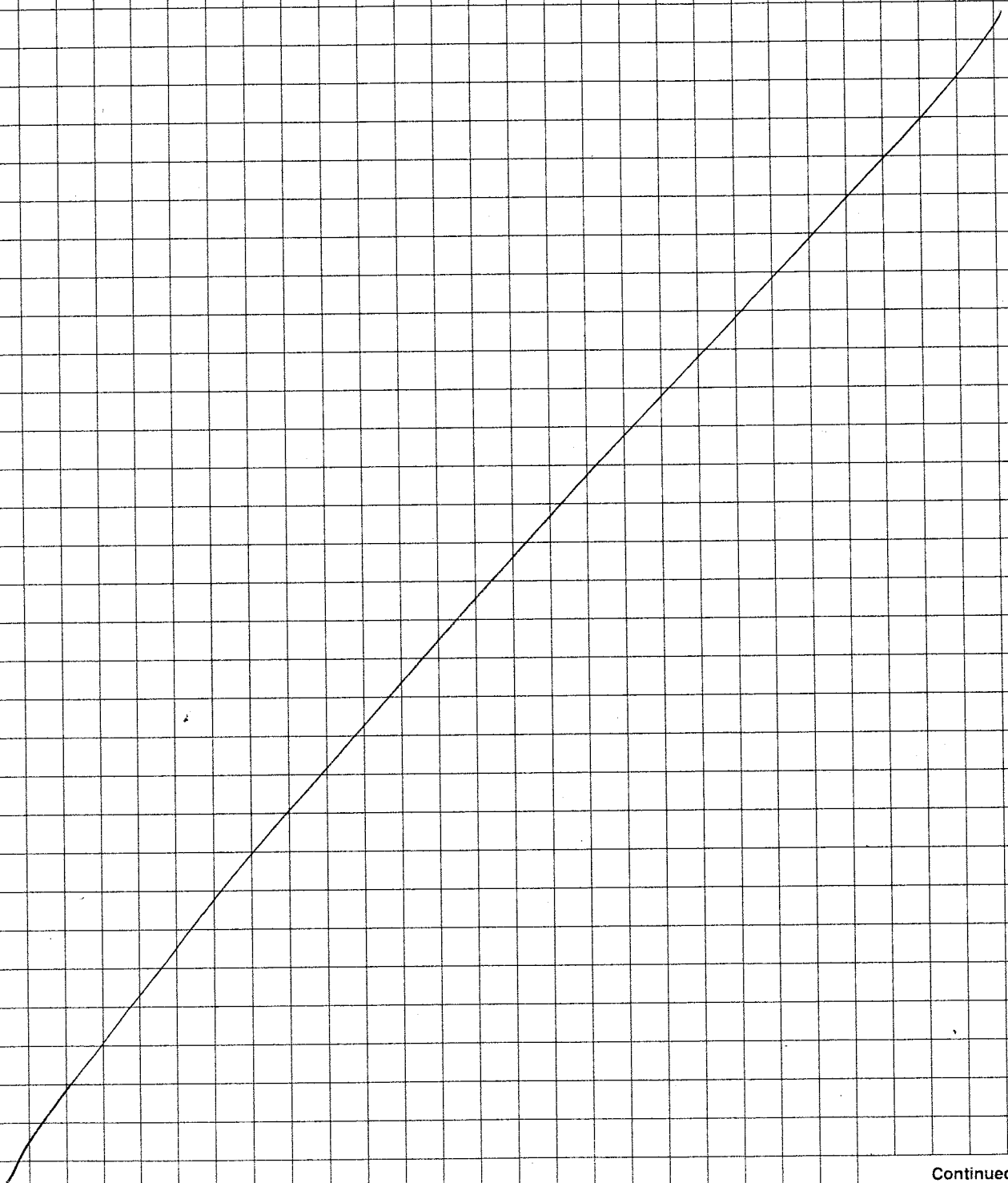
Signed ID 1/3/13 2013 1/3/~~2012~~ Date

PROJECT EL20100011

Notebook No. 5143

Continued From Page

Packed LIMS# 314339 into ~~Rotor~~ IE.PEW 12/12/12 4mm zirconia rotor (# 27J1012) using SSNMR packing tools. Rotor stored in an Eppendorf tube until analysis. 5143-02-01



Continued on Page

Read and Understood By

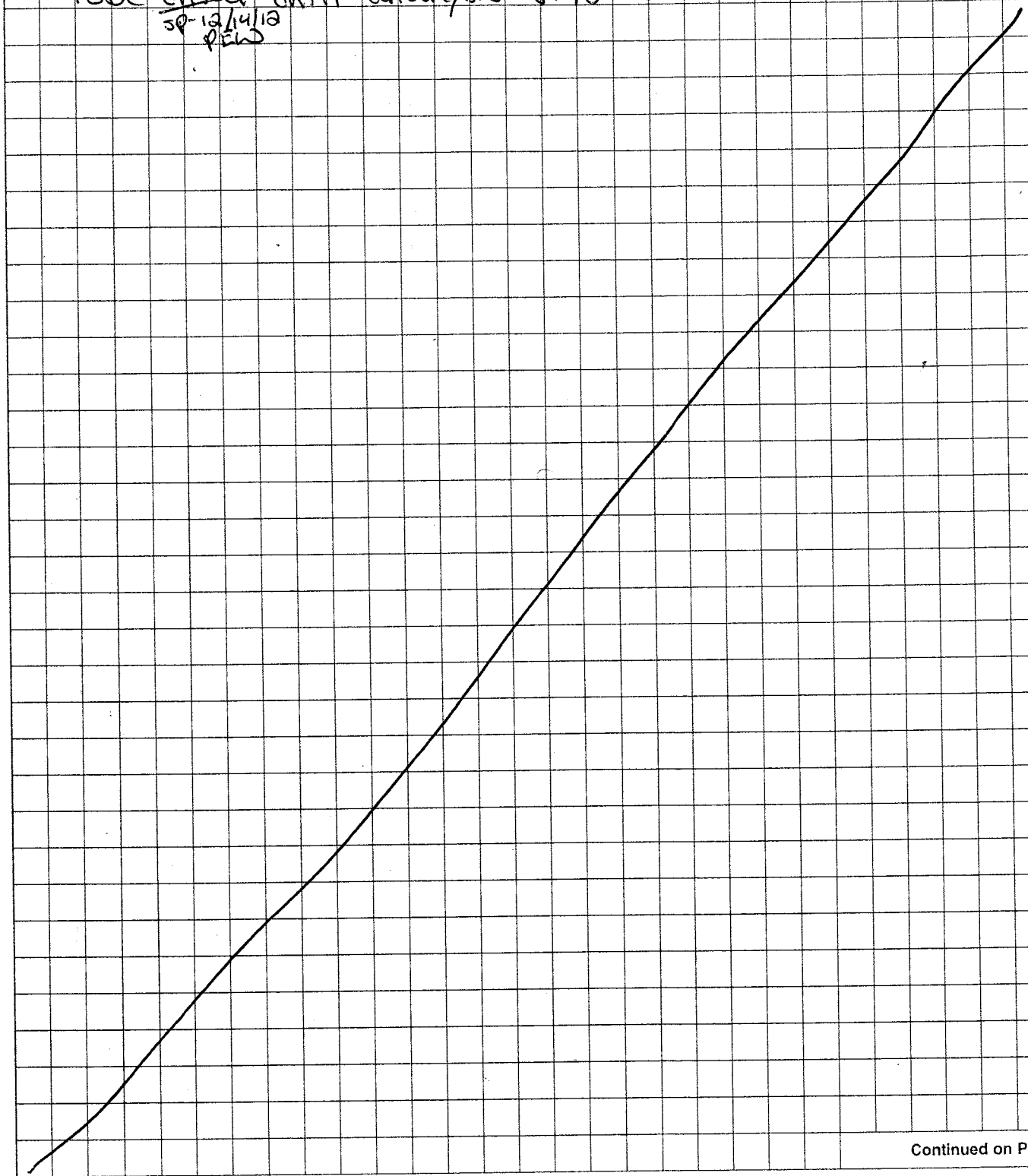
Signed

12/12/12
Date

Signed

ID 1/3/13 KSG
1/3/2013
Date

Packed LIMS# 314783 into 4mm Zirconia rotor (#26K1017) using SSNMR packing tools. Rotor stored in an Eppendorf tube ~~until~~ SP-12/14/12 PEW until analysis- 5143-03-01



Continued on Page

Signed — 12/14/12 Date

Read and Understood By

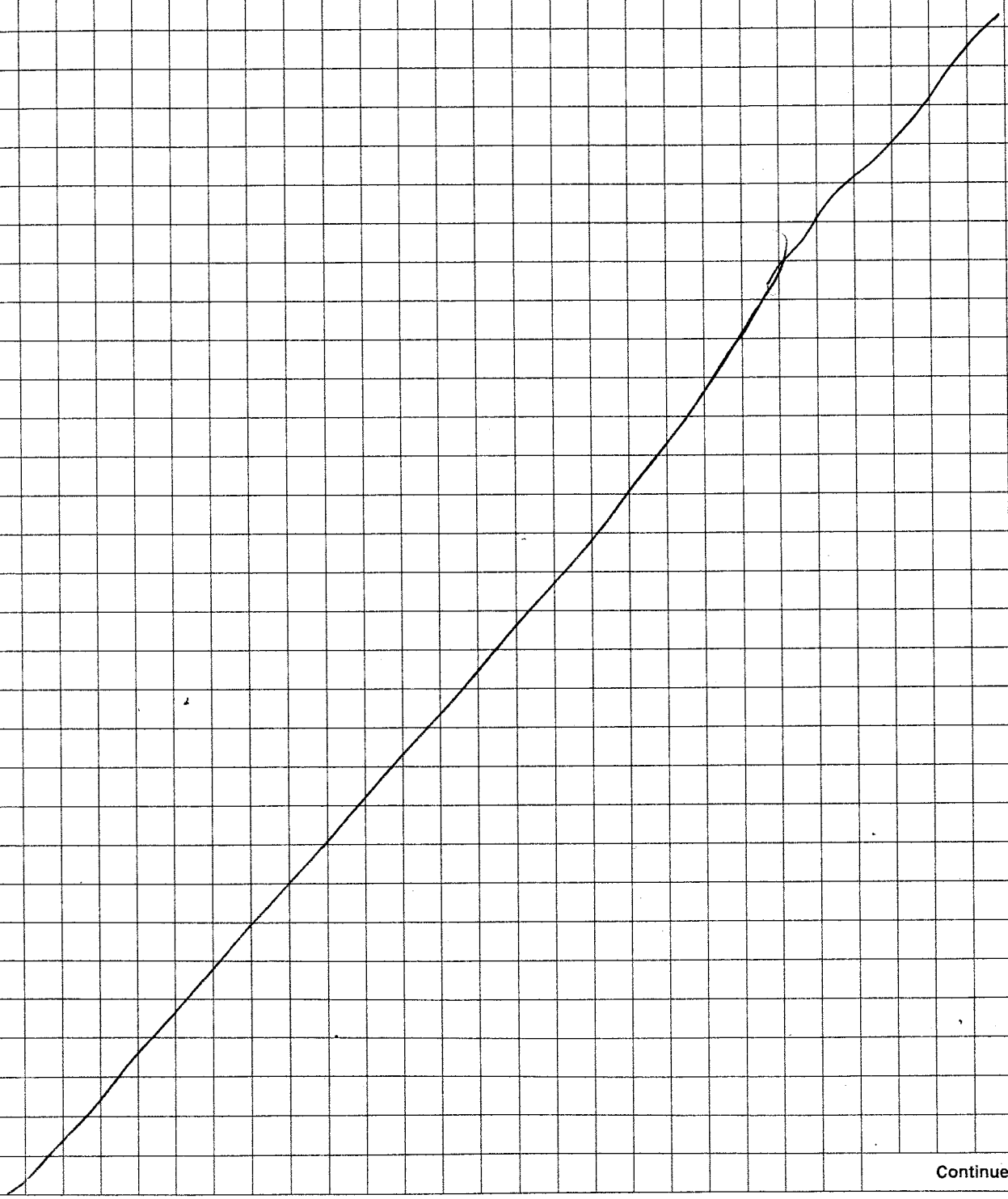
Signed — ID 1/3/13 KG 1/3/2013 Date

PROJECT EL-20100011

Notebook No. 5143

Continued From Page -

5143-04-01- Inside of an N_2-purged glovebag (%RH = 0.5%, Hygrometer ID# 2129, Due 01/18/13) Transferred a portion of LIMS# 314549 (DMSO-d_6, 99.9%D) to an NMR tube (Norell, 507-HP).



Continued on Page

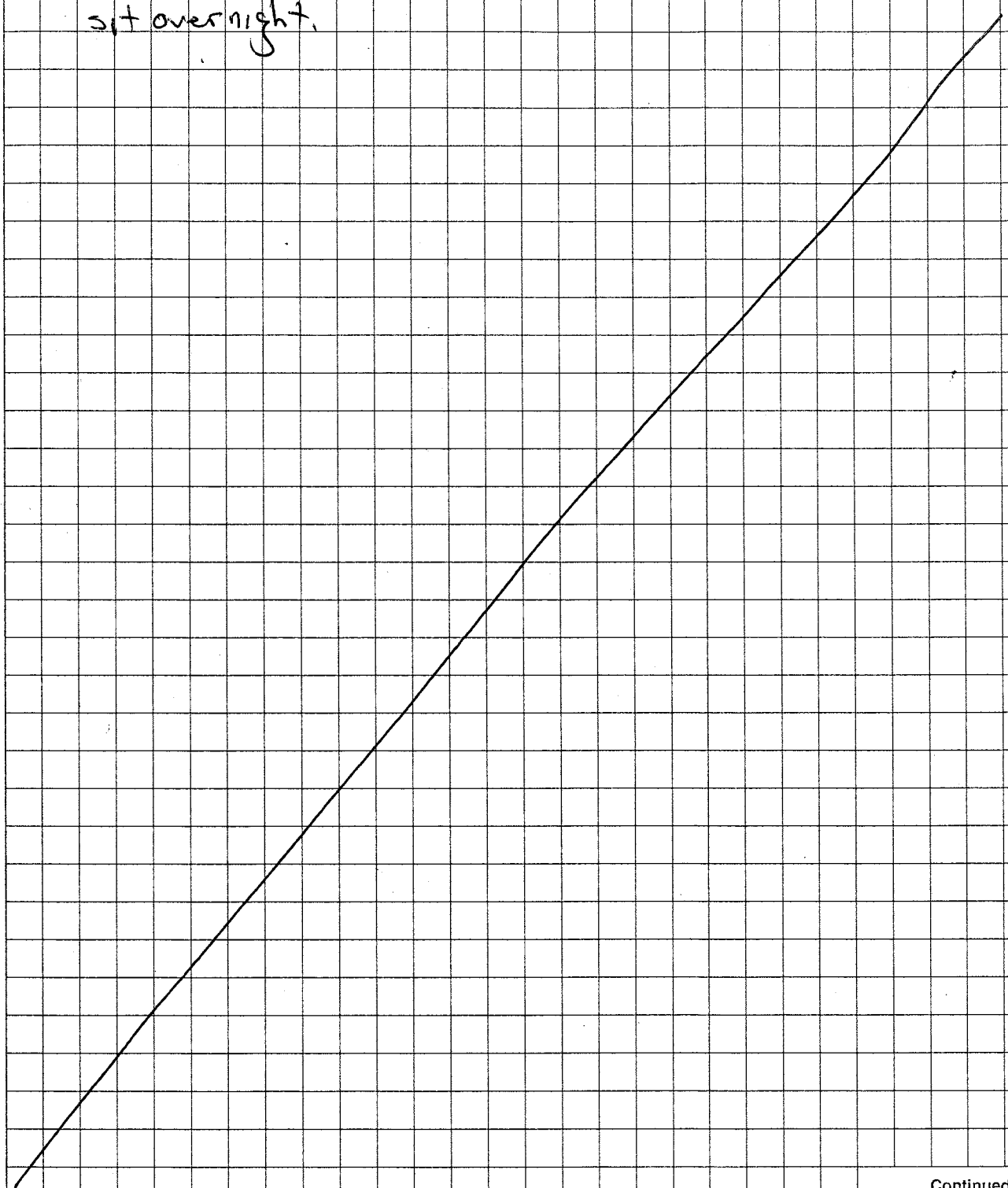
Read and Understood By

Signed — Date — Signed — Date

12/18/12

1/3/13 ID 1/3/13

Inside of an N_2-purged glovebag (~0.5% RH, Hygrometer ID# 2129, Due 01/18/13). Added molecular sieves (5135-10-01) to LIMS # 314549 (~1/8 of the volume). Allow to sit overnight.



Continued on Page

Read and Understood By

Signed 12/18/12 Date

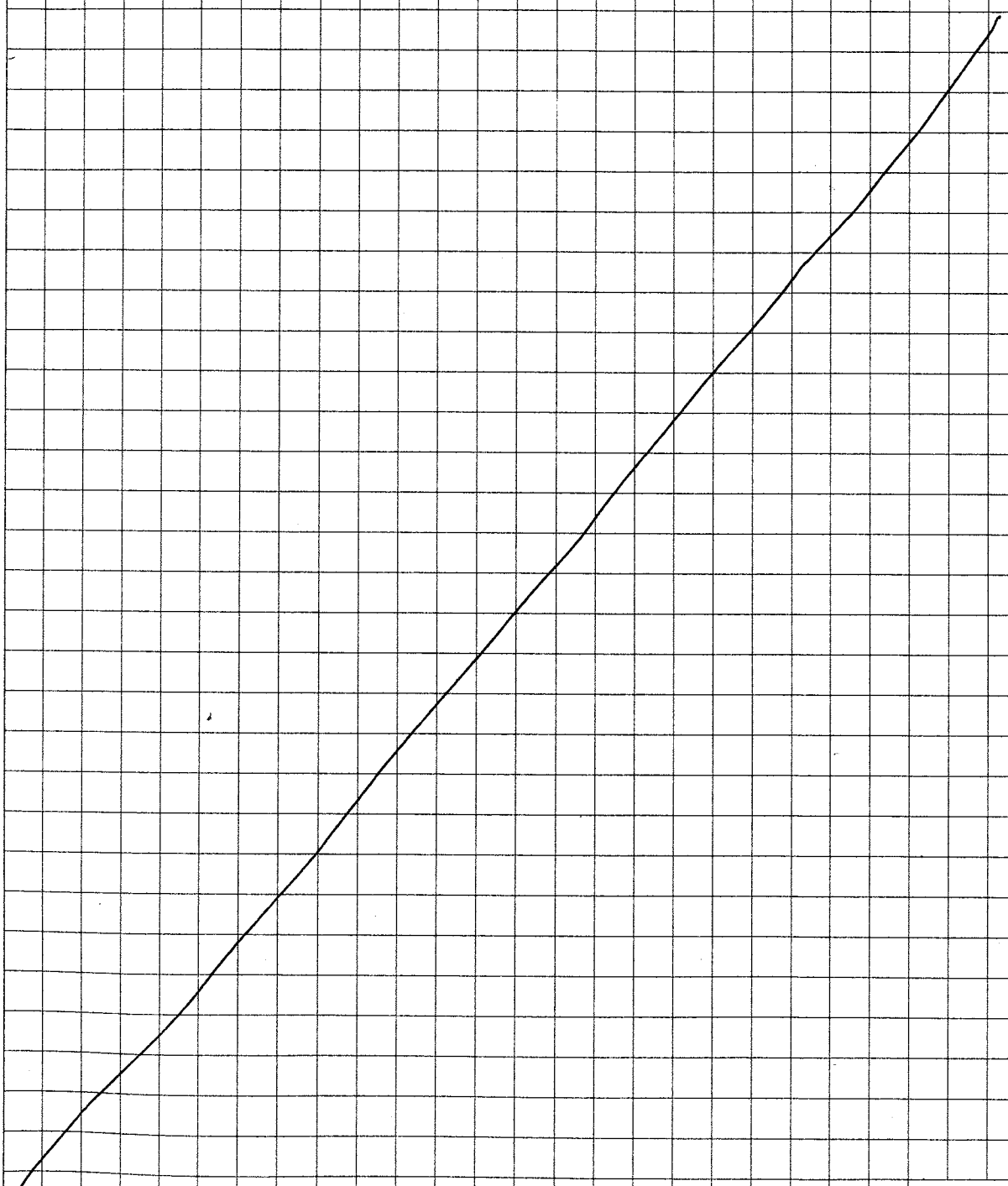
Signed 1/3/2013 Date

PROJECT ~~E230~~ IE-PEW 12/19/12 EL20100011

Notebook No. 5143

Continued From Page

Inside of an N_2-purged glovebag (~0.4% RH, Hygrometer ID# 2129, Due 01/18/13) transferred 600 mL of Lot# 314549 (over sieves, 5135-10-01) using Pipet # 88317 (Due 04/30/13) to an NMR tube (Norell, 507-HP) - 5143-06-01



Continued on Page

Signed

IE-PEW 12/19/12
12/19/12
Date

Read and Understood By

Signed

1/4/2013
Date

To the samples below added 0.6 mL of DMSO-d_6 (LIMS#271458① oversieves) using 1 mL graduated Class A pipet. Soln transferred to separate NMR tubes (Norell, 507-HP). All work performed in an N_2-purged glovebag (~0.5%RH, Hygrometer ID # 2129, Due 01/18/13).

Sample ID	NMR Sample ID
5135-30-01	5143-07-01
5135-30-02	5143-07-02

① Exp 11/30/17 - PEW 12/19/12

Continued on Page

Read and Understood By

Signed — Date 12/19/12 — Signed — Date 1/4/2013

Inside of an N_2-purged glove bag (~0.4% RH, Hygrometer ID# 2129, Due 01/18/13) transferred 600 µL of DMSO-d_6 (LIMS# 271458, over sieves, exp 11/30/17) using Pipet # 88317 (Due 04/30/13). Soln transferred to an NMR tube (Norell, 507-HP). Sample kept in glove bag until analysis.

LIMS #	Original Sample ID	NMR Sample ID
308389	5135-31-01	5143-08-01
308390	5135-31-02	5143-08-02

Continued on Page

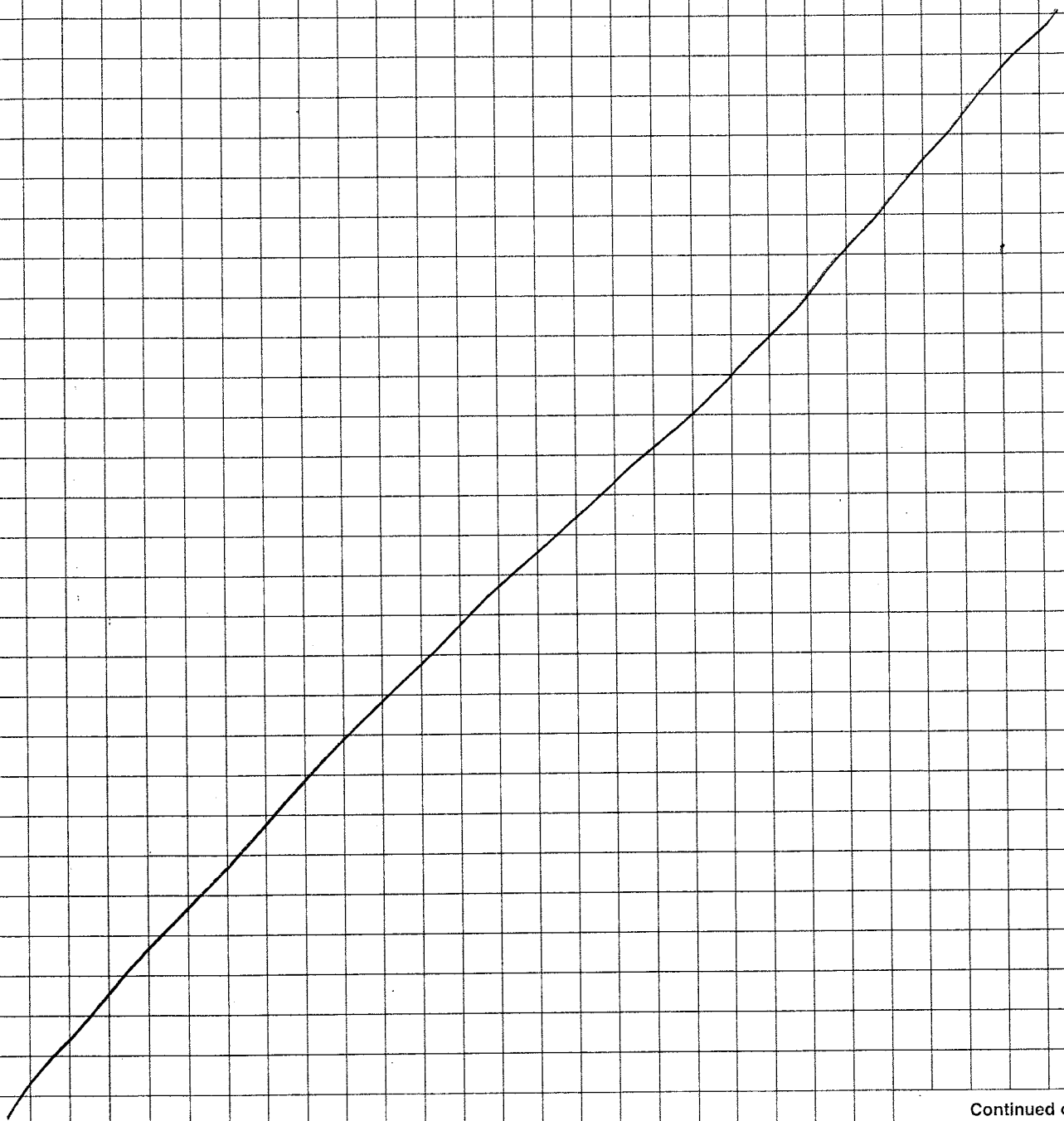
Read and Understood By

Signed — Date: 12/19/12

Signed — Date: 1/4/2013

Inside of an N_2-purged glovebag (~0.5% RH, Hygrometer, ID# 2129, Due 01/18/13) transferred 1000 µL of DMSO-d6 (LIMS# 271458, over sieves, exp 11/30/17) using Pipet# 88317 (Due 04/30/13)①, Soln transferred to an NMR tube (Norell 1,507-HP) - 5143-09-01

① Dissolved entire portion of LIMS# 315436 - PEW 12/20/12.



Continued on Page

Signed 12/20/12 Date

Read and Understood By

Signed 1/4/2013 Date

Inside of an N_2-purged glove bag (~0.5% RH, Hygrometer ID # 2129, Due 01/18/13) dissolved the entire portion of the following samples w/ 1000 mL DMSO-d6 (LIMS# 271458, over sieves, exp 11/30/17) using Dipet # 88317 (Due 04/30/13). Soln transferred to separate NMR tubes (Worcell, 507-HP)

LIMS #	SampleID
315452	5143-10-01
314539	5143-10-02
315467	5143-10-03
315469	5143-10-04

Continued on Page

Read and Understood By

Signed — Date 12/20/12

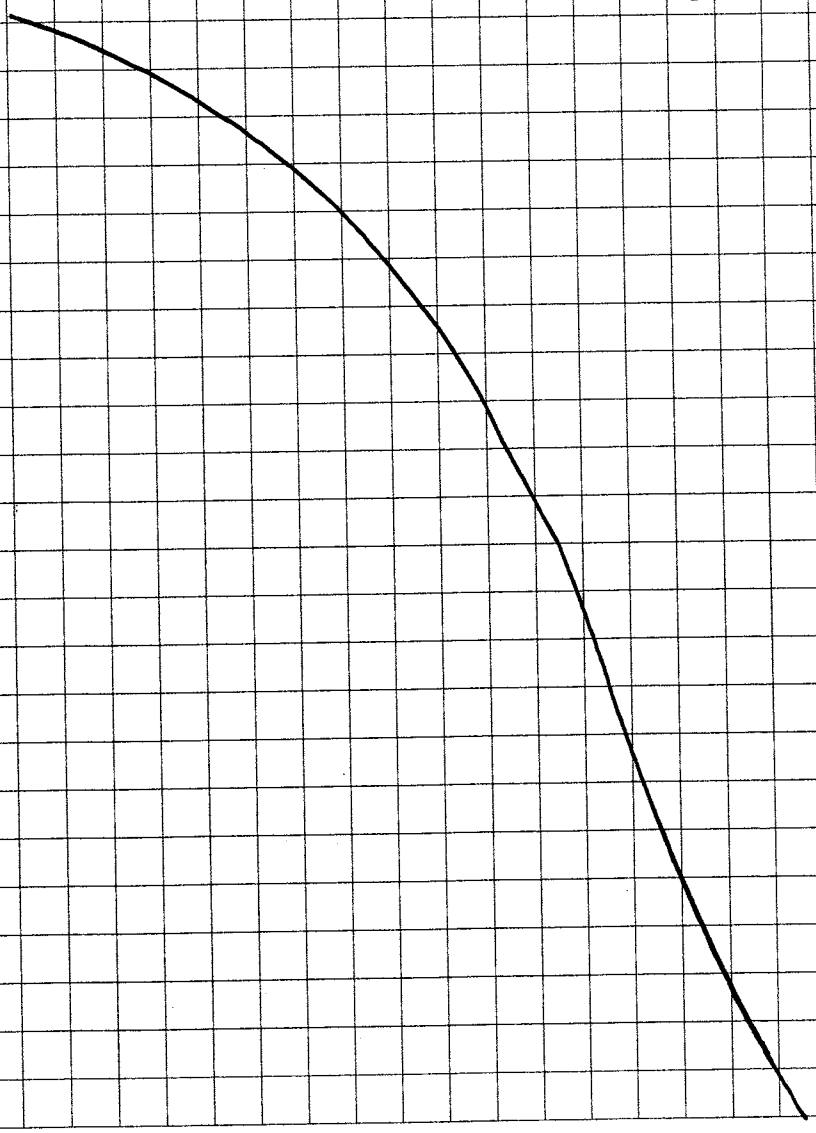
Signed — Date 1/4/2013

Balance #16, Level

Example Calc: Spl wt = (Spl + Pan wt) - Pan wt

Weighed the indicated amount of sample into the corresponding DSC pan.

Sample ID	File #	Pan wt (mg)	Spl + Pan wt (mg)	Spl wt (mg)	Pan Type	Post
5143-11-01	Ref. Pan	53.00 mg	53.00 mg	0.00 mg	TZHSLP	R1
315535	566460	53.14 mg	54.57 mg	1.43 mg	TZHSLP	1



Continued on Page

Read and Understood By

Signed — 12/21/12 Date

Signed — 1/4/2013 Date

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1846 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

564452 314705, 5135-12-01 Compound 184 spun 11-Dec-2012 10:52:06

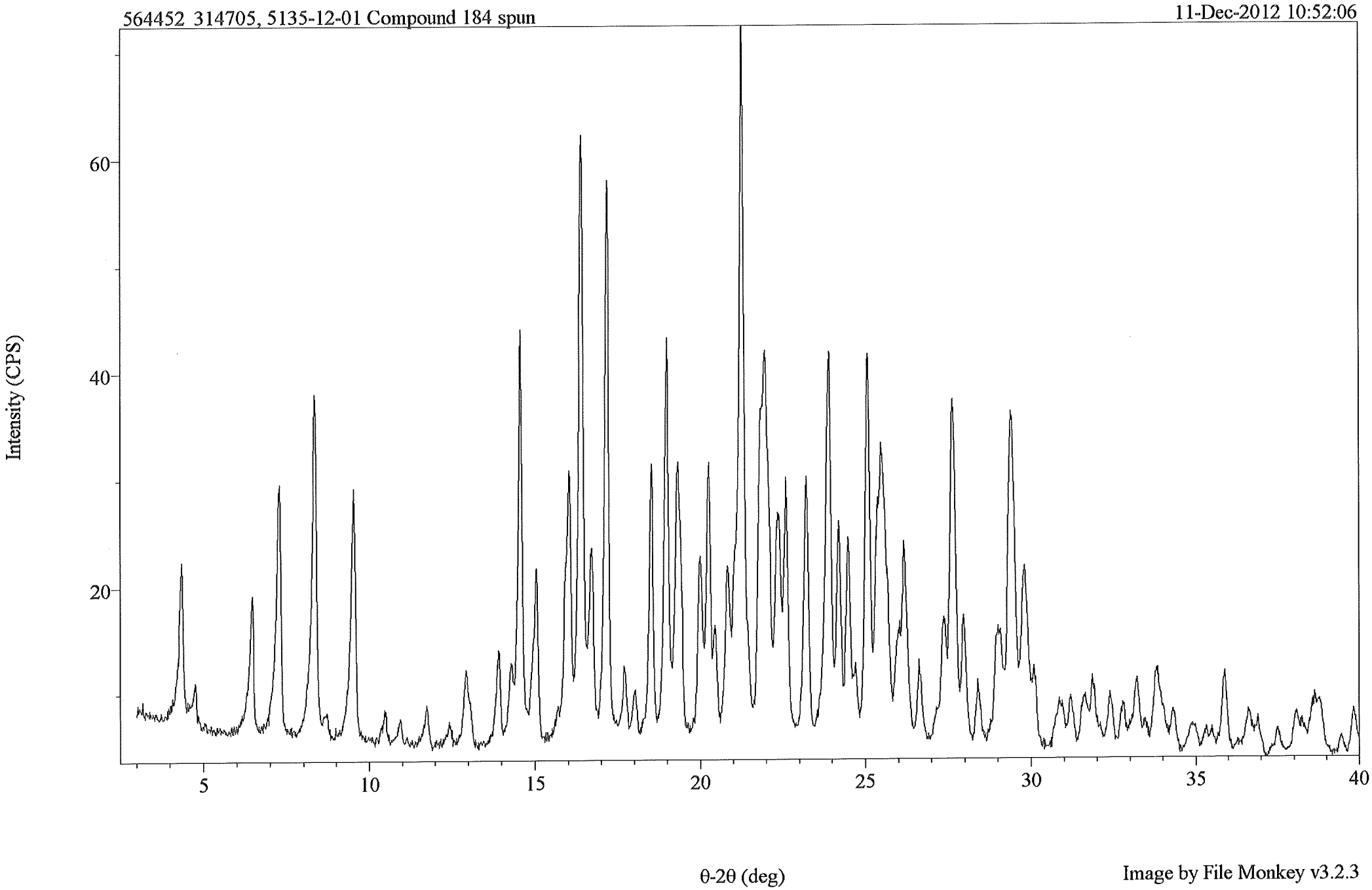


Image by File Monkey v3.2.3

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1846 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

12-Dec-2012 11:31:28

564767 314760, 5135-15-05 Compound 184 spun

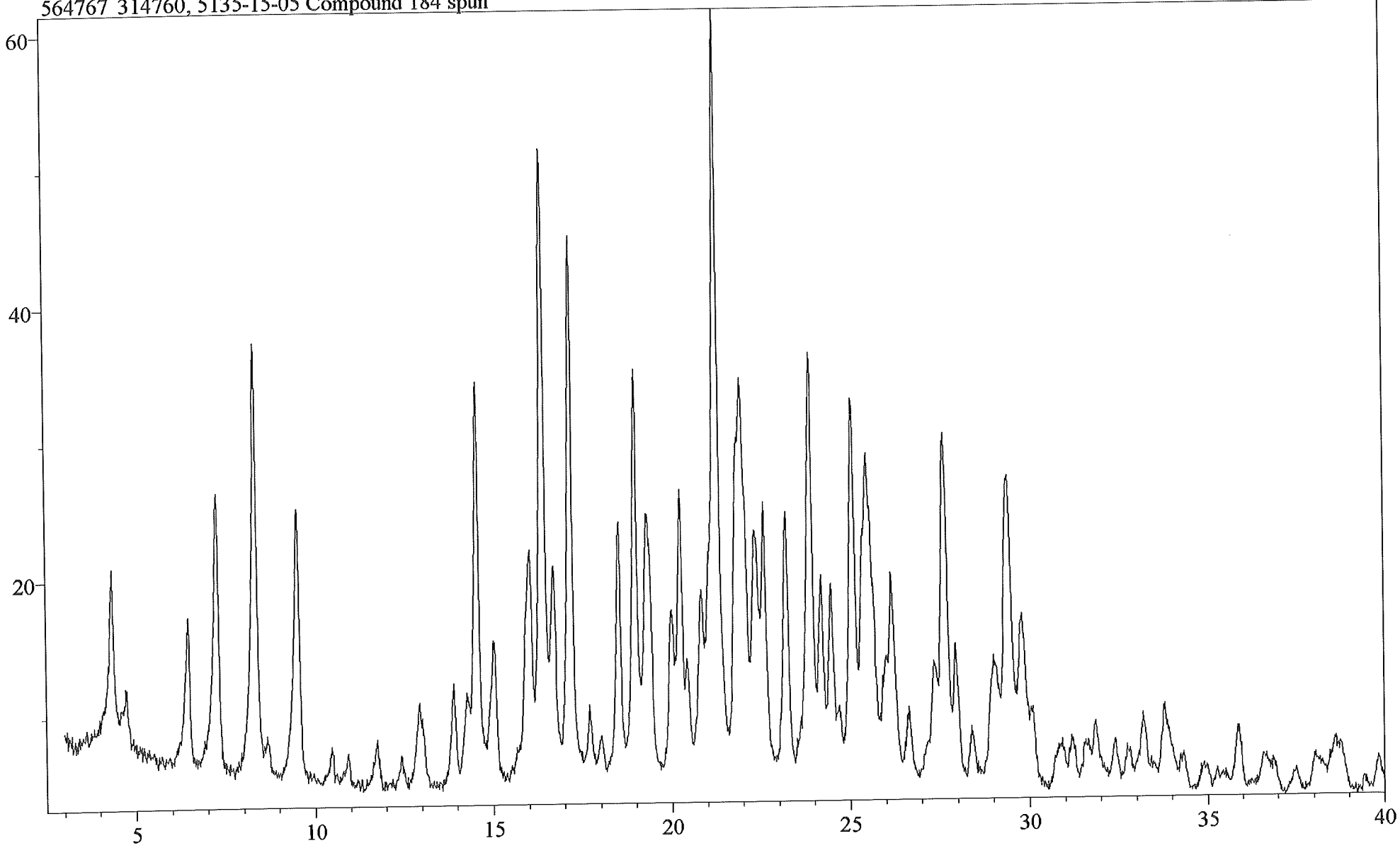


Image by File Monkey v3.2.3

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1851 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

564765 314783, 5135-17-01 Compound 184 spun

12-Dec-2012 12:19:41

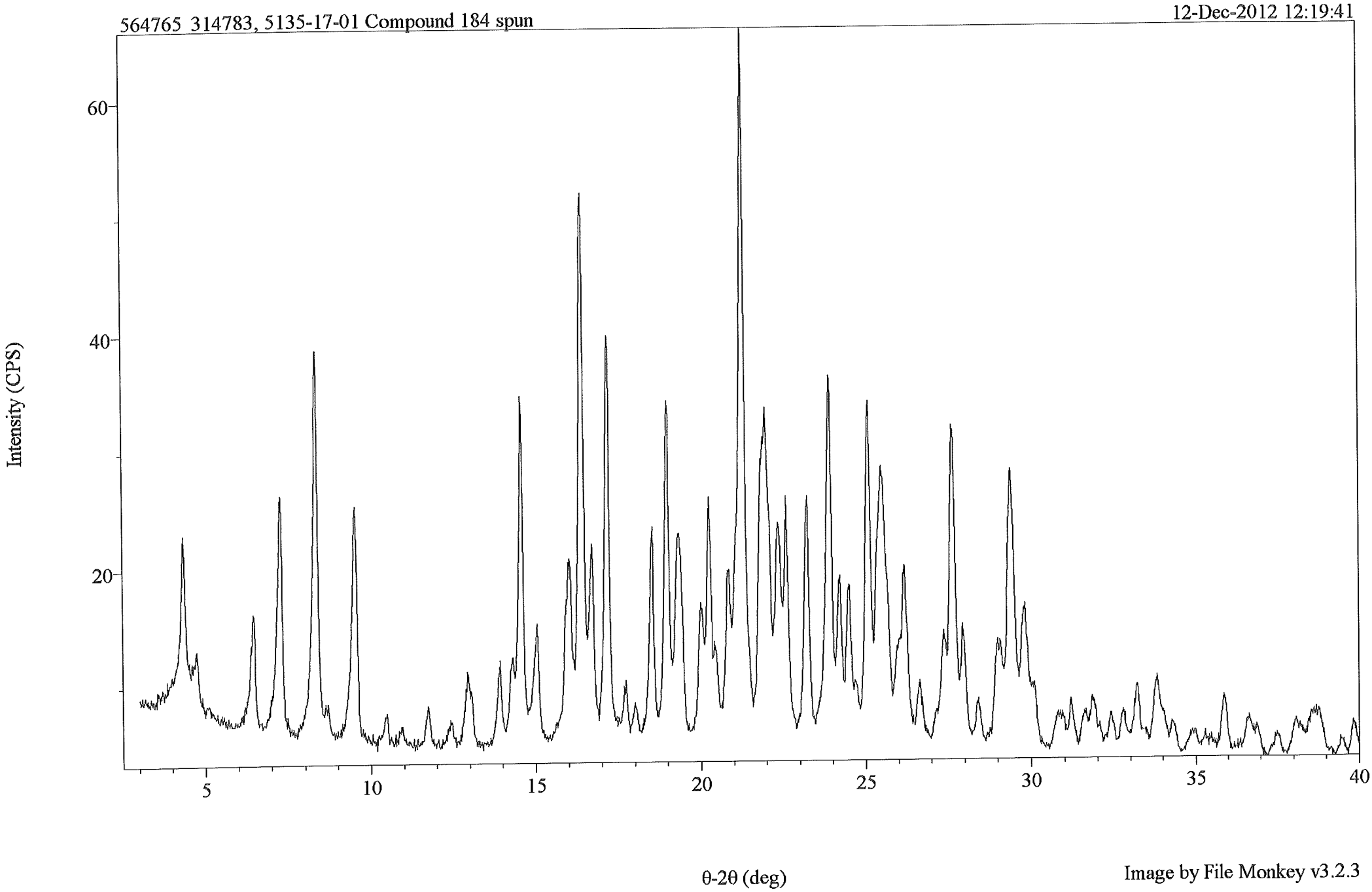
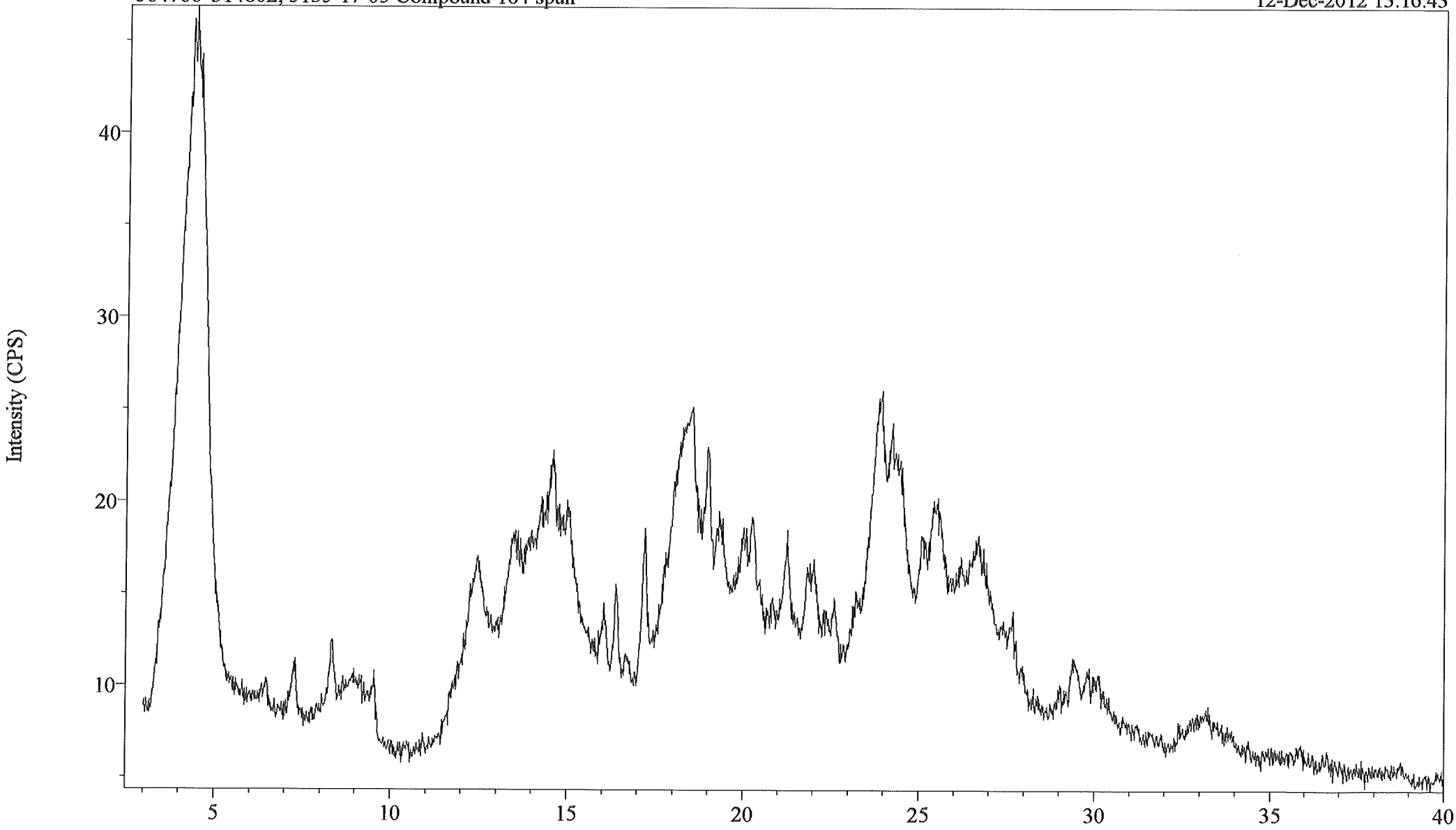


Image by File Monkey v3.2.3

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1851 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

564766 314802, 5135-17-05 Compound 184 spun

12-Dec-2012 13:16:43



Laboratory Report

Report prepared for:
Karen Gushurst
Sr. Research Investigator
Aptuit WLF (SSCI)
3065 Kent Ave
West Lafayette, IN USA 47906
Phone: 765-463-0112 ext. 3231
Fax: 765-463-4722
Email: karen.gushurst@aptuit.com

Report prepared by:
Daniel R Longnecker

Purchase Order:
APLWF-3498

For further assistance, contact:
Daniel R Longnecker
Technical Manager
PO Box 51610
Knoxville, TN 37950 -1610
877-449-8797 ext. 1855
danlongnecker@galbraith.com

Sample: LIMS 314760
Lab ID: 2012-S-7525 **Received:** 2012-12-14

Analysis	Method	Result	Basis	Sample Amount Used	Date (Time)
C : Carbon					
	GLI Procedure ME-12	36.56 %	As Received	1.768 mg	2012-12-17
	GLI Procedure ME-12	36.16 %	As Received	1.704 mg	2012-12-17
H : Hydrogen					
	GLI Procedure ME-12	4.21 %	As Received	1.768 mg	2012-12-17
	GLI Procedure ME-12	4.06 %	As Received	1.704 mg	2012-12-17
N : Nitrogen					
	GLI Procedure ME-12	13.92 %	As Received	1.768 mg	2012-12-17
	GLI Procedure ME-12	13.78 %	As Received	1.704 mg	2012-12-17
P : Phosphorus					
	GLI Procedure ME-70	3.72 %	As Received	45.37 mg	2012-12-16
	GLI Procedure ME-70	3.77 %	As Received	44.21 mg	2012-12-16
	GLI Procedure ME-70 (matrix spike) [1]	95 % Recovery	As Received	46.04 mg	2012-12-16

1. The matrix spike analysis was performed to satisfy method requirements. There is no additional charge for the matrix spike result.

Sample: LIMS 314783
Lab ID: 2012-S-7526 **Received:** 2012-12-14

Analysis	Method	Result	Basis	Sample Amount Used	Date (Time)
C : Carbon					
	GLI Procedure ME-12	36.05 %	As Received	1.585 mg	2012-12-17
	GLI Procedure ME-12	36.28 %	As Received	1.800 mg	2012-12-17
H : Hydrogen					
	GLI Procedure ME-12	4.19 %	As Received	1.585 mg	2012-12-17
	GLI Procedure ME-12	4.13 %	As Received	1.800 mg	2012-12-17
N : Nitrogen					
	GLI Procedure ME-12	13.72 %	As Received	1.585 mg	2012-12-17

	GLI Procedure ME-12	13.80 %	As Received	1.800 mg	2012-12-17
P : Phosphorus					
	GLI Procedure ME-70	3.85 %	As Received	44.36 mg	2012-12-16
	GLI Procedure ME-70	3.81 %	As Received	47.89 mg	2012-12-16

Sample: LIMS 314339
Lab ID: 2012-S-7527 **Received:** 2012-12-14

Analysis	Method	Result	Basis	Sample Amount Used	Date (Time)
C : Carbon					
	GLI Procedure ME-12	37.33 %	As Received	1.739 mg	2012-12-17
	GLI Procedure ME-12	37.67 %	As Received	1.988 mg	2012-12-17
H : Hydrogen					
	GLI Procedure ME-12	4.12 %	As Received	1.739 mg	2012-12-17
	GLI Procedure ME-12	4.11 %	As Received	1.988 mg	2012-12-17
N : Nitrogen					
	GLI Procedure ME-12	14.26 %	As Received	1.739 mg	2012-12-17
	GLI Procedure ME-12	14.27 %	As Received	1.988 mg	2012-12-17
P : Phosphorus					
	GLI Procedure ME-70	3.50 %	As Received	62.82 mg	2012-12-16
	GLI Procedure ME-70	3.48 %	As Received	61.53 mg	2012-12-16

For all samples on this report:

2. These analyses were performed in general compliance with the Laboratory sections of Current Good Manufacturing Practices for bulk pharmaceuticals as defined in 21 CFR 210 and 211, with the following exception:
The analytical methods used for the determination of carbon, hydrogen, nitrogen, and phosphorus have been validated to reference materials, but have not been validated for your sample.

Signatures:

Created By:	daniel.r.longnecker	2012-12-17T21:44:18.37-05:00
Published By:	tammy.saylor	2012-12-17T22:30:00.887-05:00
Inspected By:	tammy.saylor	2012-12-17T22:29:47.813-05:00

Physical signatures are on file.
"Published By" signature indicates authorized release of data.
"Inspected By" signature indicates QA review and approval.

Laboratory Report

Report prepared for:
Karen Gushurst
Sr. Research Investigator
Aptuit WLF (SSCI)
3065 Kent Ave.
West Lafayette, IN USA 47906
Phone: 765-463-0112
Fax: 765-463-4722
Email: karen.gushurst@aptuit.com

Report prepared by:
Daniel R Longnecker

Purchase Order:

For further assistance, contact:
Daniel R Longnecker
Technical Manager
PO Box 51610
Knoxville, TN 37950 -1610
877-449-8797 ext. 1855
danlongnecker@galbraith.com

Sample: LIMS 315535
Lab ID: 2012-S-8147 **Received:** 2012-12-26

Analysis	Method	Result	Basis	Sample Amount Used	Date (Time)
C : Carbon					
	GLI Procedure ME-12	37.17 %	As Received	2.368 mg	2012-12-27
	GLI Procedure ME-12	36.33 %	As Received	2.065 mg	2012-12-27
H : Hydrogen					
	GLI Procedure ME-12	4.06 %	As Received	2.368 mg	2012-12-27
	GLI Procedure ME-12	3.94 %	As Received	2.065 mg	2012-12-27
N : Nitrogen					
	GLI Procedure ME-12	13.92 %	As Received	2.368 mg	2012-12-27
	GLI Procedure ME-12	13.80 %	As Received	2.065 mg	2012-12-27
P : Phosphorus					
	GLI Procedure ME-70	4.26 %	As Received	22.92 mg	2012-12-27
	GLI Procedure ME-70	4.24 %	As Received	21.00 mg	2012-12-27
	GLI Procedure ME-70 (matrix spike) [1]	95 % Recovery	As Received	20.19 mg	2012-12-27

1. The matrix spike analysis was performed to satisfy method requirements. There is no additional charge for the matrix spike result.

For all samples on this report:

2. These analyses were performed in general compliance with the Laboratory sections of Current Good Manufacturing Practices for bulk pharmaceuticals as defined in 21 CFR 210 and 211, with the following exception:
The analytical methods used for the determination of carbon, hydrogen, nitrogen, and phosphorus have been validated to reference materials, but have not been validated for your sample.

3. Duplicate analyses were performed as part of our internal Quality Control Program. There is no additional charge for duplicate values.

The precision among the carbon replicates is less than that normally observed for the method used. Additional analyses may be necessary to fully understand the precision associated with your matrix. Please contact a member of our technical staff for further information.

Signatures:

Published By:	sandie.a.jones	2012-12-29T00:05:59.647-05:00
Inspected By:	sandie.a.jones	2012-12-29T00:05:53.61-05:00
Created By:	daniel.r.longnecker	2012-12-28T18:40:27.76-05:00

Physical signatures are on file.
"Published By" signature indicates authorized release of data.
"Inspected By" signature indicates QA review and approval.

NMR Assay for Sitagliptin Base - Sitagliptin Phosphate Mixtures

Table 1. Sitagliptin-Phosphoric Acid ^{13}C NMR Assay – Std Curve Sample Preparation

Sample ID	Sitagliptin Base (mg)	Sitagliptin DHP (mg)	Phosphoric acid (mmol)	Sitagliptin (mmol)	Sitagliptin: H_3PO_4 ratio	H_3PO_4 mol fraction
5135-31-01	-	50.30	0.09954	0.09954	1.00	0.5
5135-32-04	19.51	60.51	0.11975	0.16765	1.40	0.41667
5135-32-02	23.00	57.03	0.11286	0.16933	1.50	0.39995
5135-32-05	25.99	53.98	0.10682	0.17063	1.60	0.38501
5135-32-01	35.70	44.33	0.08773	0.17537	2.00	0.33344

Table 2. ^{13}C NMR Chemical Shifts for the Four Resonances used in the Assay

Sample ID	H_3PO_4 mol fraction	Resonance 1 (ppm)	Resonance 2 (ppm)	Resonance 3 (ppm)	Resonance 4 (ppm)	NMR File
5135-31-01	0.5	169.239	169.110	31.873	31.691	566184
5135-32-04	0.41667	169.619	169.444	32.837	32.495	566295
5135-32-02	0.39995	169.702	169.520	33.064	32.700	566260
5135-32-05	0.38501	169.740	169.566	33.201	32.829	566297
5135-32-01	0.33344	169.945	169.771	33.747	33.360	566240
Linear Regression Analysis						
Slope		-4.26013	-3.97204	-11.31637	-10.02244	
Y - Intercept		171.38293	171.09887	37.55031	36.69426	
Correlation(R)		0.99785	0.99972	0.99923	0.99969	

Table 3. Calculated Sitagliptin:Phosphoric Acid Ratios (Sample Analysis)

Sample Description	Replicated Atwood Wash Solids	6X Washed Solids
Sample ID	5135-02-01	5135-17-01
NMR File	566281	565811
^{13}C Resonance 1 (ppm)	169.854	169.710
Interpolated Mol Fraction	0.35889	0.39269
Sitagliptin:H_3PO_4 Ratio	**1.79**	**1.55**
^{13}C Resonance 2 (ppm)	169.672	169.528
Interpolated Mol Fraction	0.35923	0.39548
Sitagliptin:H_3PO_4 Ratio	**1.78**	**1.53**
^{13}C Resonance 3 (ppm)	33.444	33.041
Interpolated Mol Fraction	0.36286	0.39848
Sitagliptin:H_3PO_4 Ratio	**1.76**	**1.51**
^{13}C Resonance 4 (ppm)	33.064	32.692
Interpolated Mol Fraction	0.36221	0.39933
Sitagliptin:H_3PO_4 Ratio	**1.76**	**1.50**

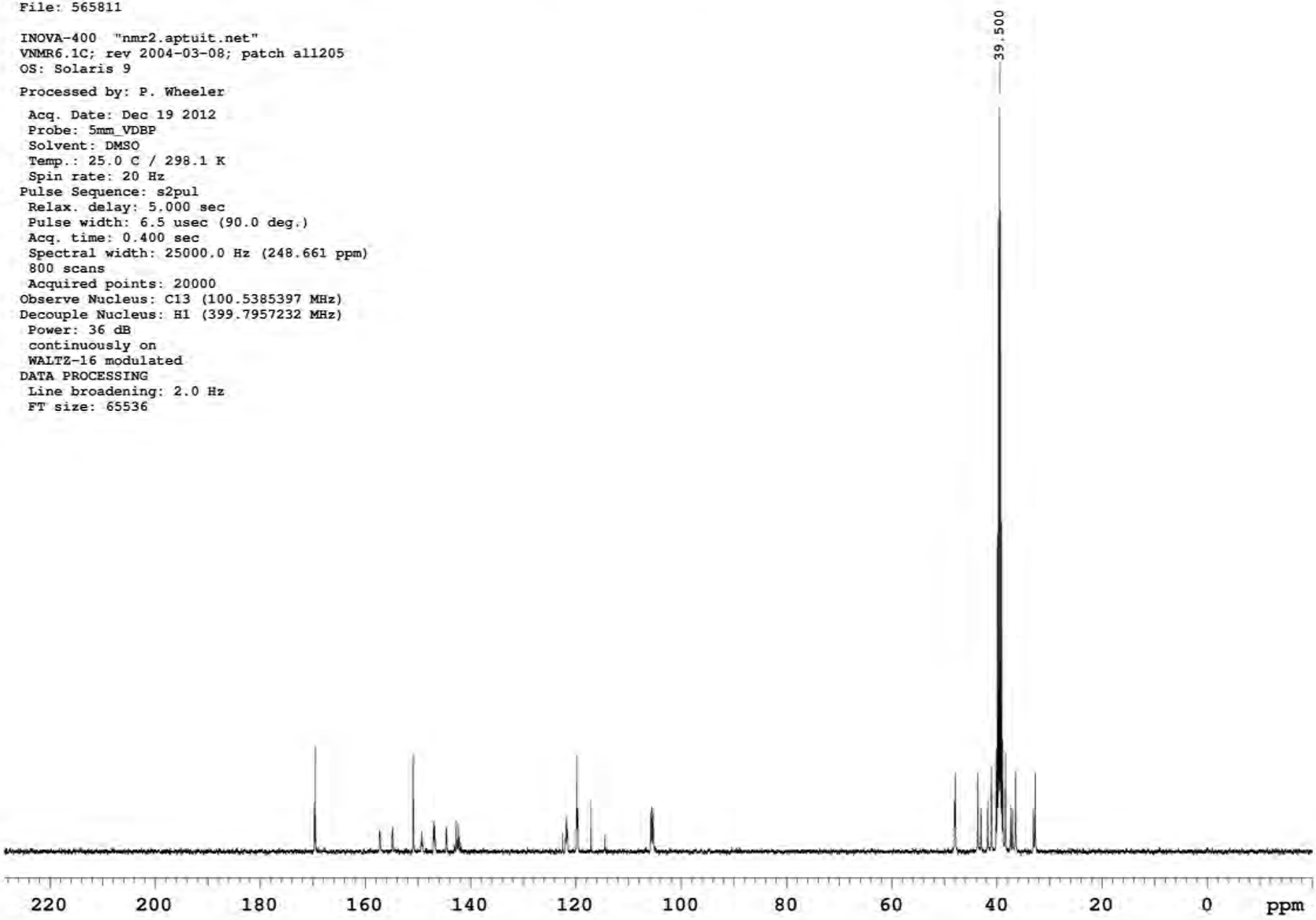
314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 565811

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch a11205
OS: Solaris 9

Processed by: P. Wheeler

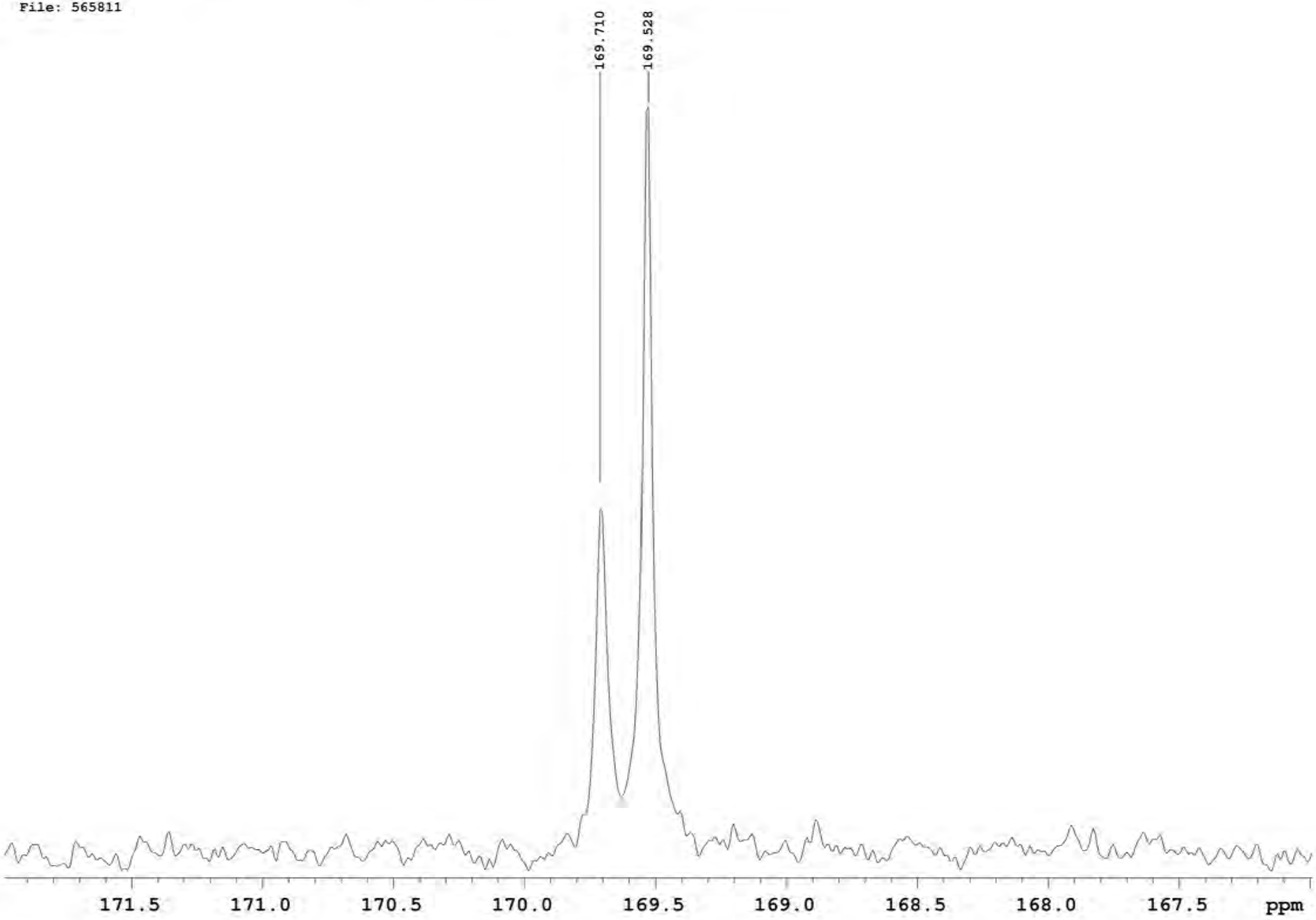
Acq. Date: Dec 19 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 565811-1

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

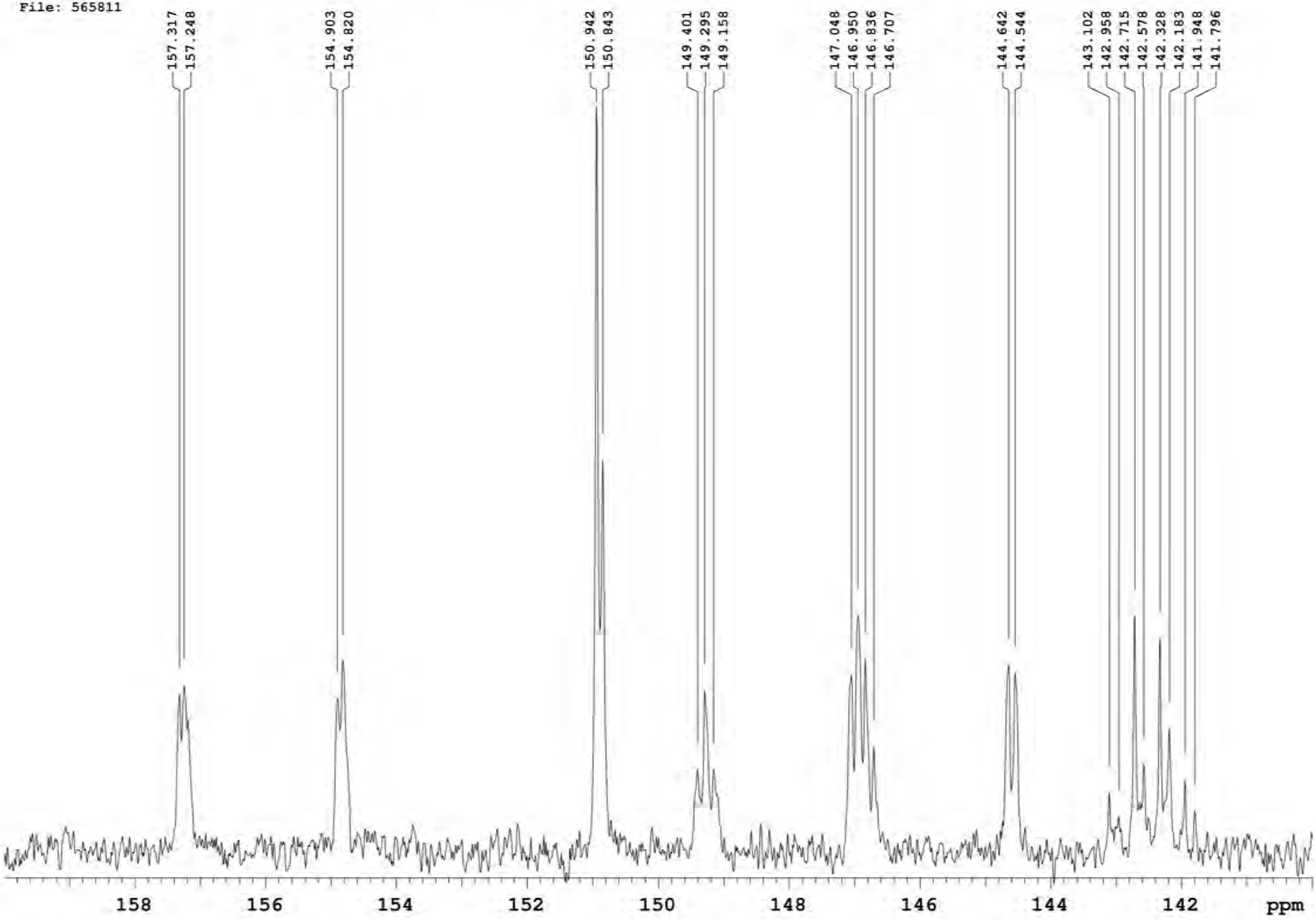
File: 565811



Plot file: 565811-2

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

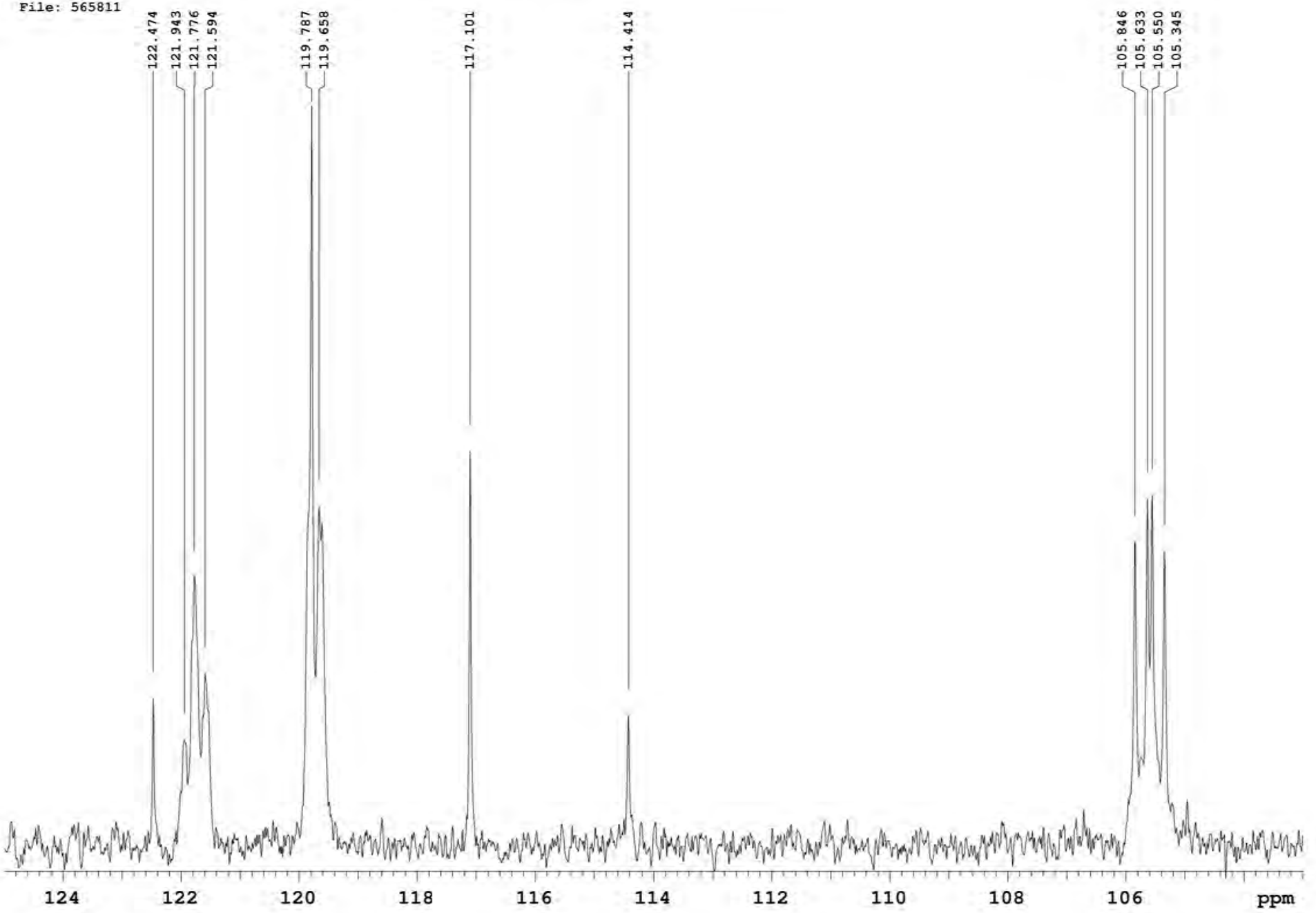
File: 565811



Plot file: 565811-3

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

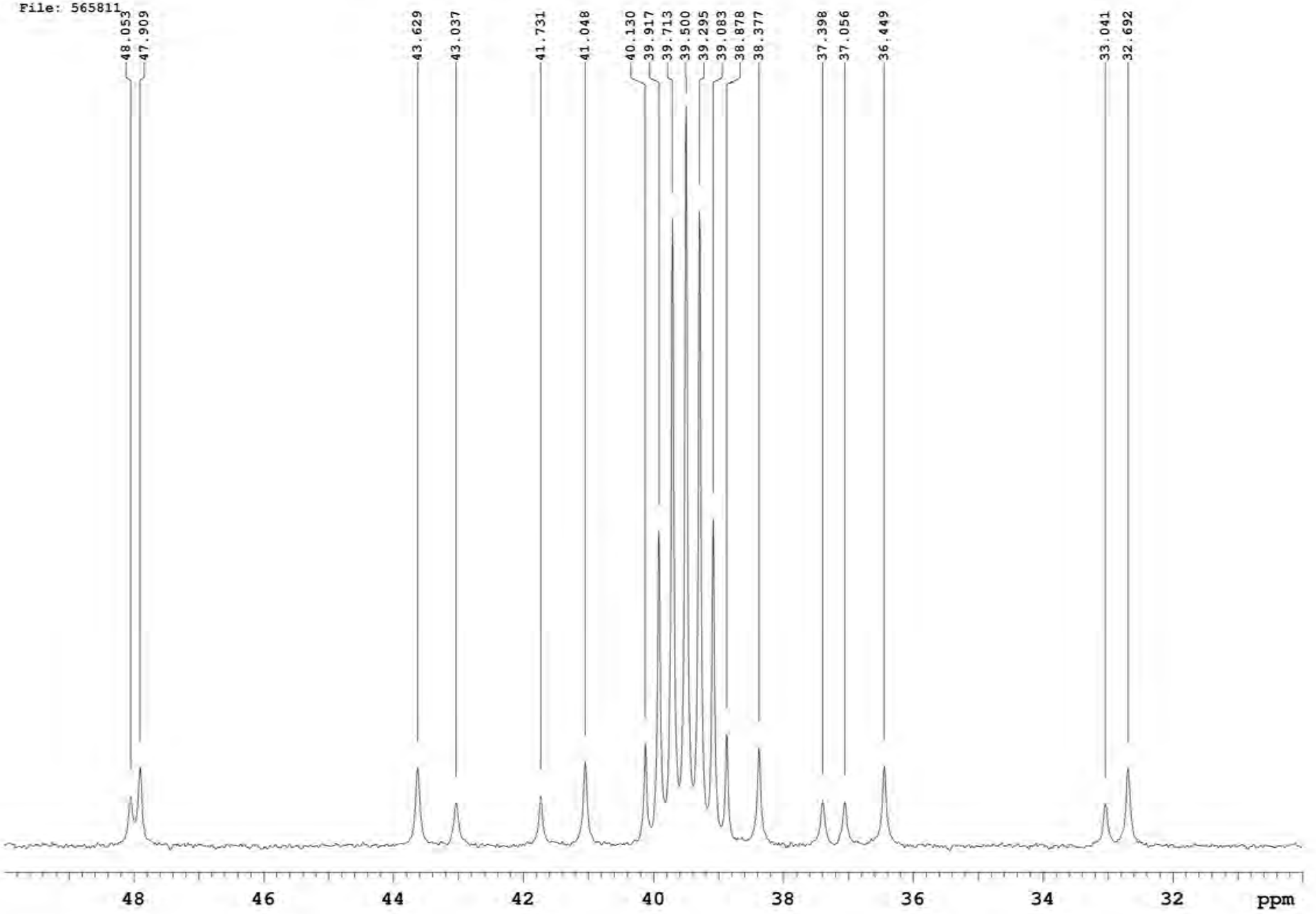
File: 565811



Plot file: 565811-4

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 565811



Plot file: 565811-5

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 565811

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 565811-1_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 565811

INDEX	FREQUENCY	PPM	HEIGHT
1	17060.610	169.710	65.2
2	17042.300	169.528	141.8

Plot file: 565811-2_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 565811

INDEX	FREQUENCY	PPM	HEIGHT
1	15814.730	157.317	29.9
2	15807.864	157.248	31.6
3	15572.115	154.903	29.1
4	15563.723	154.820	36.4
5	15173.861	150.942	141.8
6	15163.943	150.843	74.6
7	15018.984	149.401	15.7
8	15008.303	149.295	30.6
9	14994.570	149.158	15.6
10	14782.473	147.048	33.7
11	14772.555	146.950	44.9
12	14761.111	146.836	36.8
13	14748.141	146.707	19.9
14	14540.621	144.642	35.5
15	14530.703	144.544	34.0
16	14385.745	143.102	11.2
17	14371.249	142.958	6.7
18	14346.835	142.715	44.8
19	14333.102	142.578	16.6
20	14307.925	142.328	40.6
21	14293.429	142.183	23.5
22	14269.778	141.948	13.5
23	14254.519	141.796	7.8

Plot file: 565811-3_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 565811

INDEX	FREQUENCY	PPM	HEIGHT
1	12312.075	122.474	28.2
2	12258.669	121.943	20.2
3	12241.885	121.776	51.5
4	12223.574	121.594	32.9
5	12041.995	119.787	141.8
6	12029.025	119.658	64.9
7	11771.914	117.101	75.6
8	11501.833	114.414	24.8
9	10640.475	105.846	58.1
10	10619.113	105.633	66.4
11	10610.720	105.550	67.0
12	10590.121	105.345	56.3

Plot file: 565811-4_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 565811

INDEX	FREQUENCY	PPM	HEIGHT
1	4830.691	48.053	9.6
2	4816.195	47.909	15.0
3	4385.897	43.629	15.1
4	4326.388	43.037	8.3
5	4195.162	41.731	9.7
6	4126.498	41.048	16.2
7	4034.182	40.130	19.6
8	4012.820	39.917	60.5
9	3992.220	39.713	120.2
10	3970.858	39.500	141.8
11	3950.259	39.295	121.7
12	3928.896	39.083	62.7
13	3908.297	38.878	21.4
14	3857.943	38.377	18.8
15	3759.524	37.398	8.9
16	3725.192	37.056	8.5
17	3664.156	36.449	15.3
18	3321.597	33.041	8.1
19	3286.501	32.692	14.9

Plot file: 565811-5_peaks

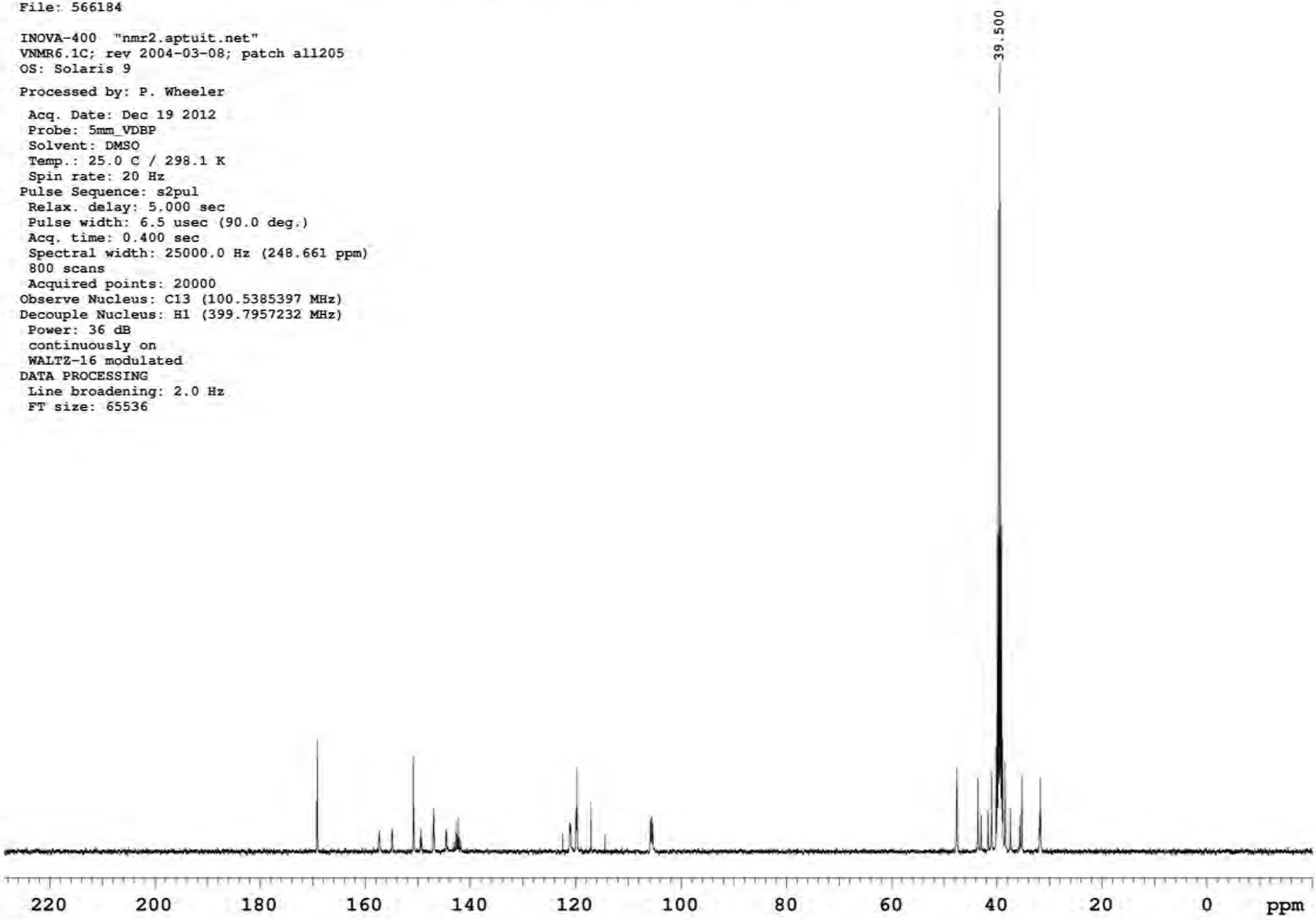
308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566184

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch a11205
OS: Solaris 9

Processed by: P. Wheeler

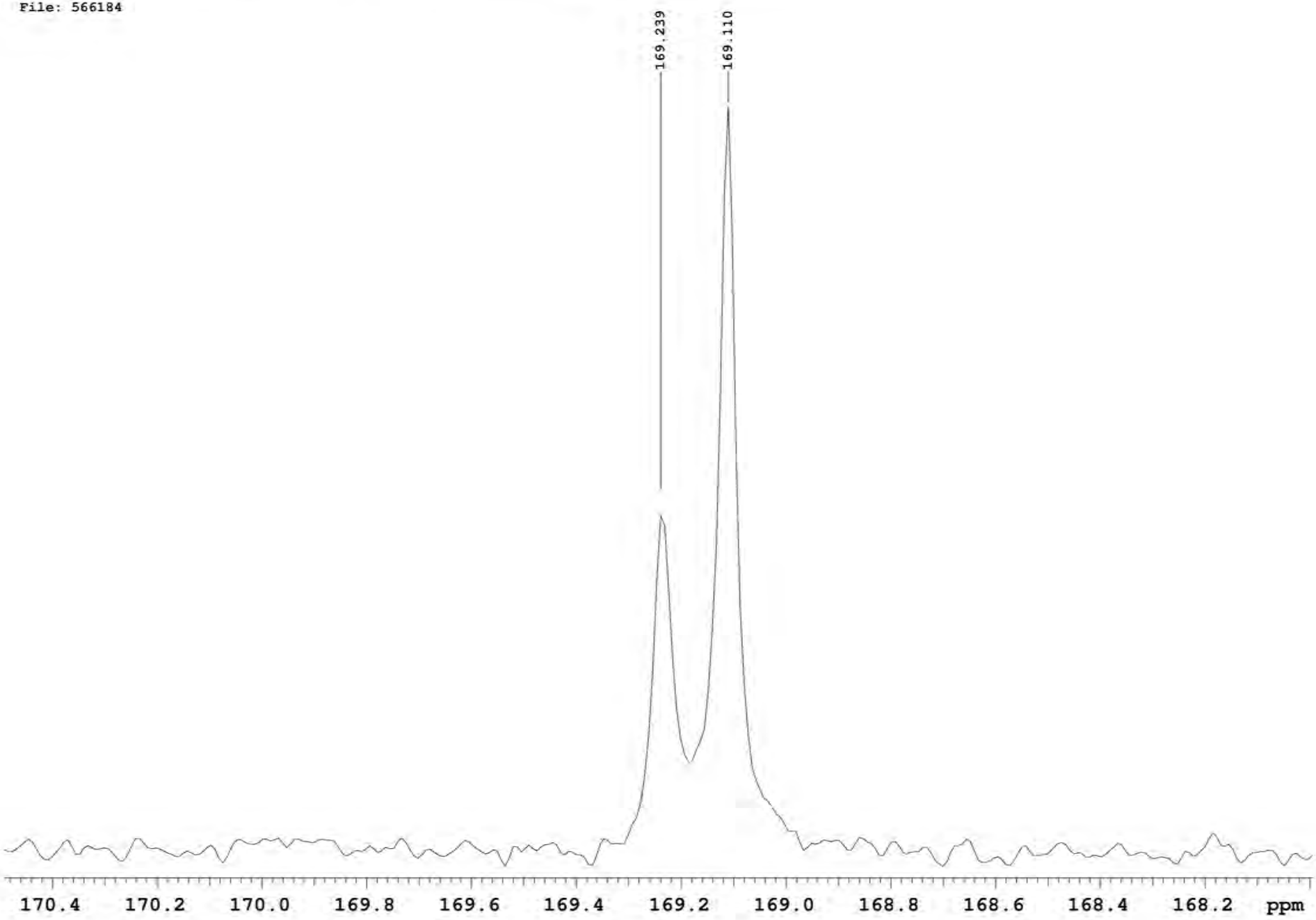
Acq. Date: Dec 19 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 566184-1

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

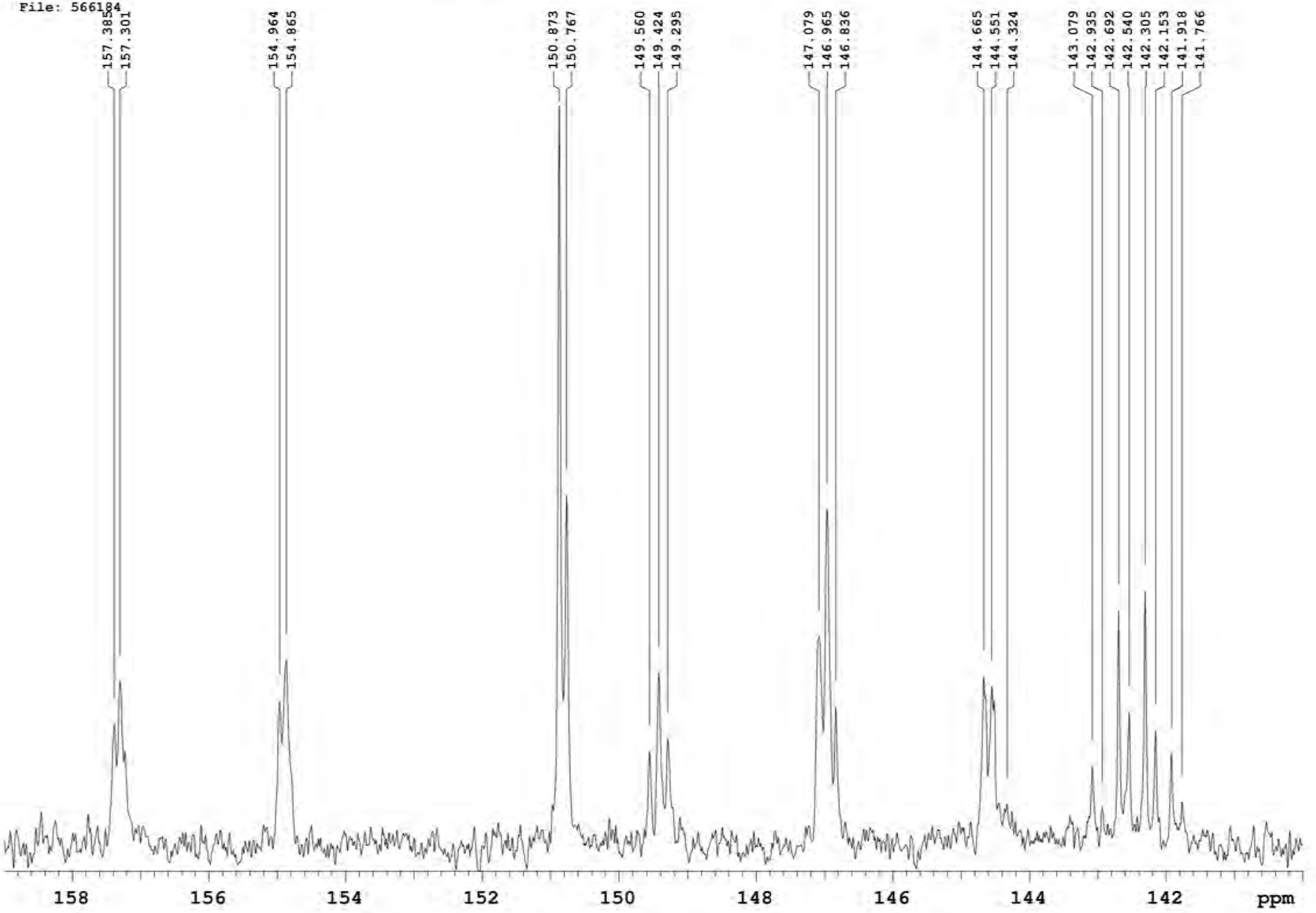
File: 566184



Plot file: 566184-2

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

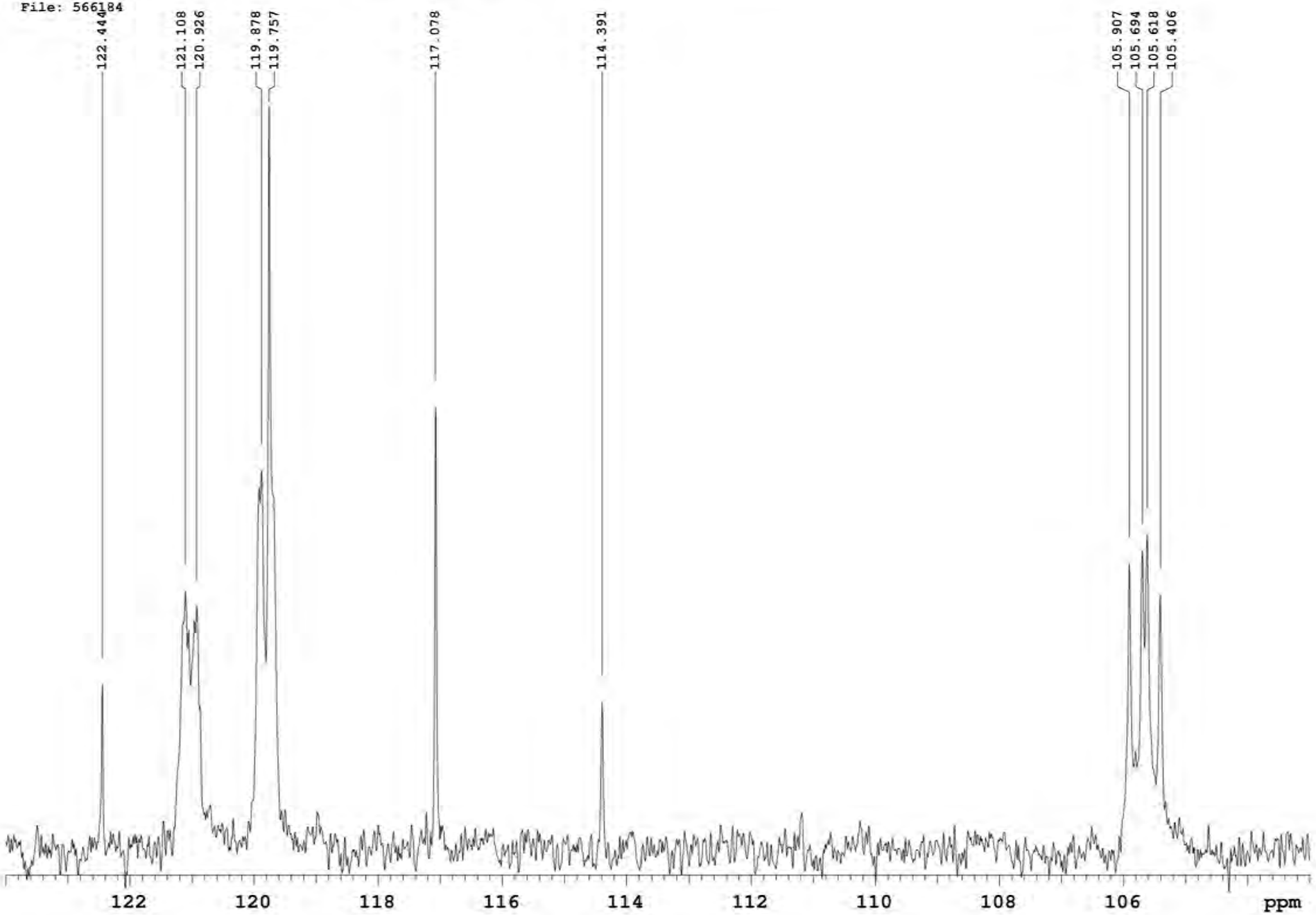
File: 566184



Plot file: 566184-3

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

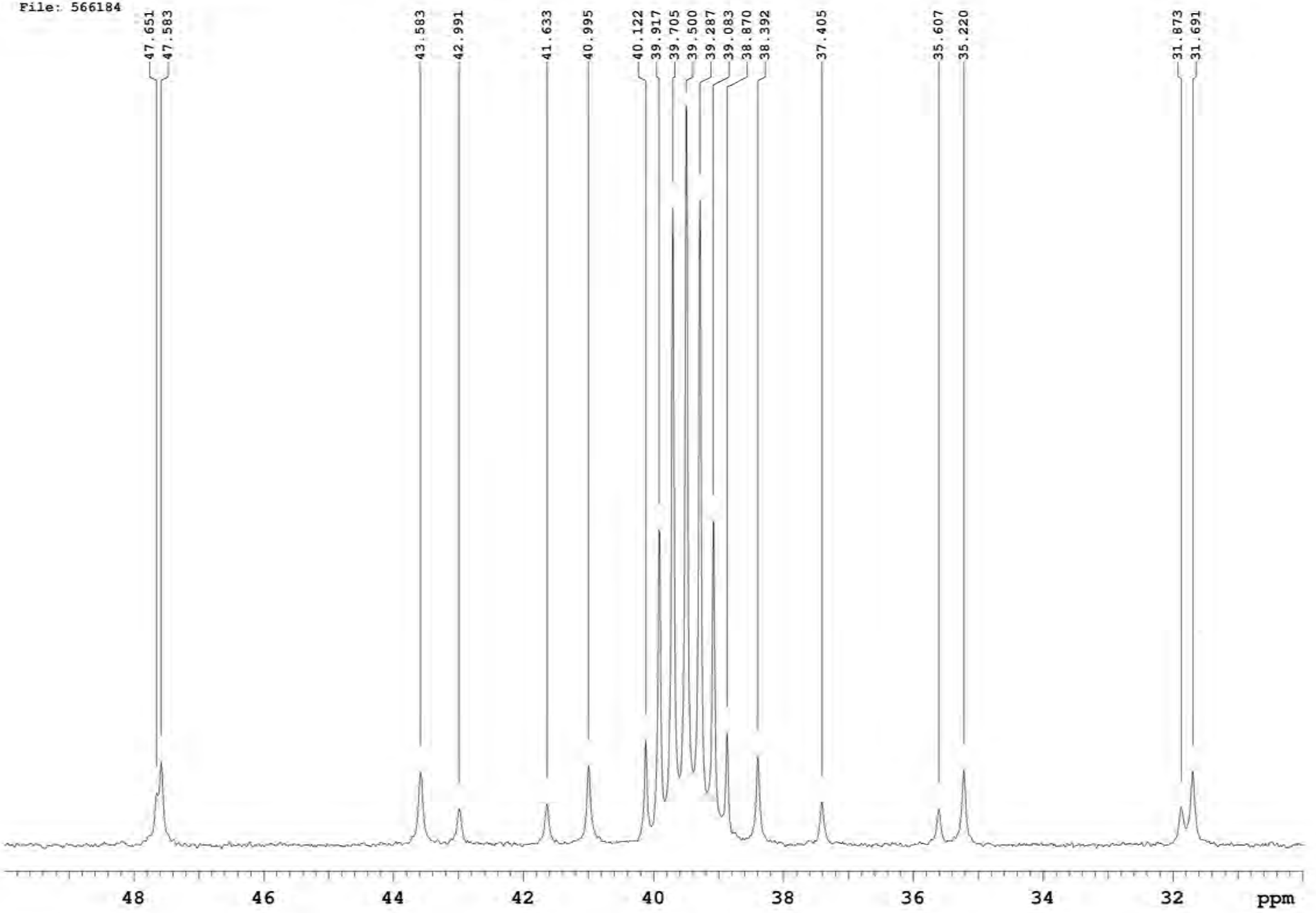
File: 566184



Plot file: 566184-4

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566184



Plot file: 566184-5

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566184

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 566184-1_peaks

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566184

INDEX	FREQUENCY	PPM	HEIGHT
1	17013.308	169.239	63.9
2	17000.338	169.110	141.8

Plot file: 566184-2_peaks

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566184

INDEX	FREQUENCY	PPM	HEIGHT
1	15821.597	157.385	23.2
2	15813.204	157.301	31.4
3	15578.219	154.964	27.6
4	15568.301	154.865	35.5
5	15166.995	150.873	141.8
6	15156.314	150.767	67.0
7	15035.006	149.560	18.0
8	15021.273	149.424	33.0
9	15008.303	149.295	20.4
10	14785.525	147.079	40.0
11	14774.081	146.965	64.3
12	14761.111	146.836	26.5
13	14542.910	144.665	32.3
14	14531.466	144.551	30.5
15	14508.578	144.324	7.8
16	14383.456	143.079	15.2
17	14368.960	142.935	7.3
18	14344.546	142.692	45.0
19	14329.287	142.540	25.5
20	14305.636	142.305	48.7
21	14290.377	142.153	22.0
22	14266.726	141.918	17.6
23	14251.467	141.766	8.4

Plot file: 566184-3_peaks

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566184

INDEX	FREQUENCY	PPM	HEIGHT
1	12309.023	122.444	31.5
2	12174.746	121.108	49.2
3	12156.436	120.926	46.4
4	12051.150	119.878	72.1
5	12038.943	119.757	141.8
6	11769.625	117.078	84.3
7	11499.545	114.391	28.0
8	10646.578	105.907	54.4
9	10625.216	105.694	56.9
10	10617.587	105.618	60.0
11	10596.224	105.406	48.5

Plot file: 566184-4_peaks

308389, Compound 184, Lot LB-1017, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566184

INDEX	FREQUENCY	PPM	HEIGHT
1	4790.255	47.651	10.0
2	4783.389	47.583	16.0
3	4381.320	43.583	14.0
4	4321.810	42.991	6.9
5	4185.244	41.633	7.9
6	4121.157	40.995	15.3
7	4033.419	40.122	20.1
8	4012.820	39.917	60.5
9	3991.458	39.705	122.2
10	3970.858	39.500	141.8
11	3949.496	39.287	123.6
12	3928.897	39.083	62.1
13	3907.534	38.870	21.5
14	3859.469	38.392	17.0
15	3760.287	37.405	8.2
16	3579.470	35.607	7.0
17	3540.560	35.220	14.4
18	3204.104	31.873	7.2
19	3185.794	31.691	14.1

Plot file: 566184-5_peaks

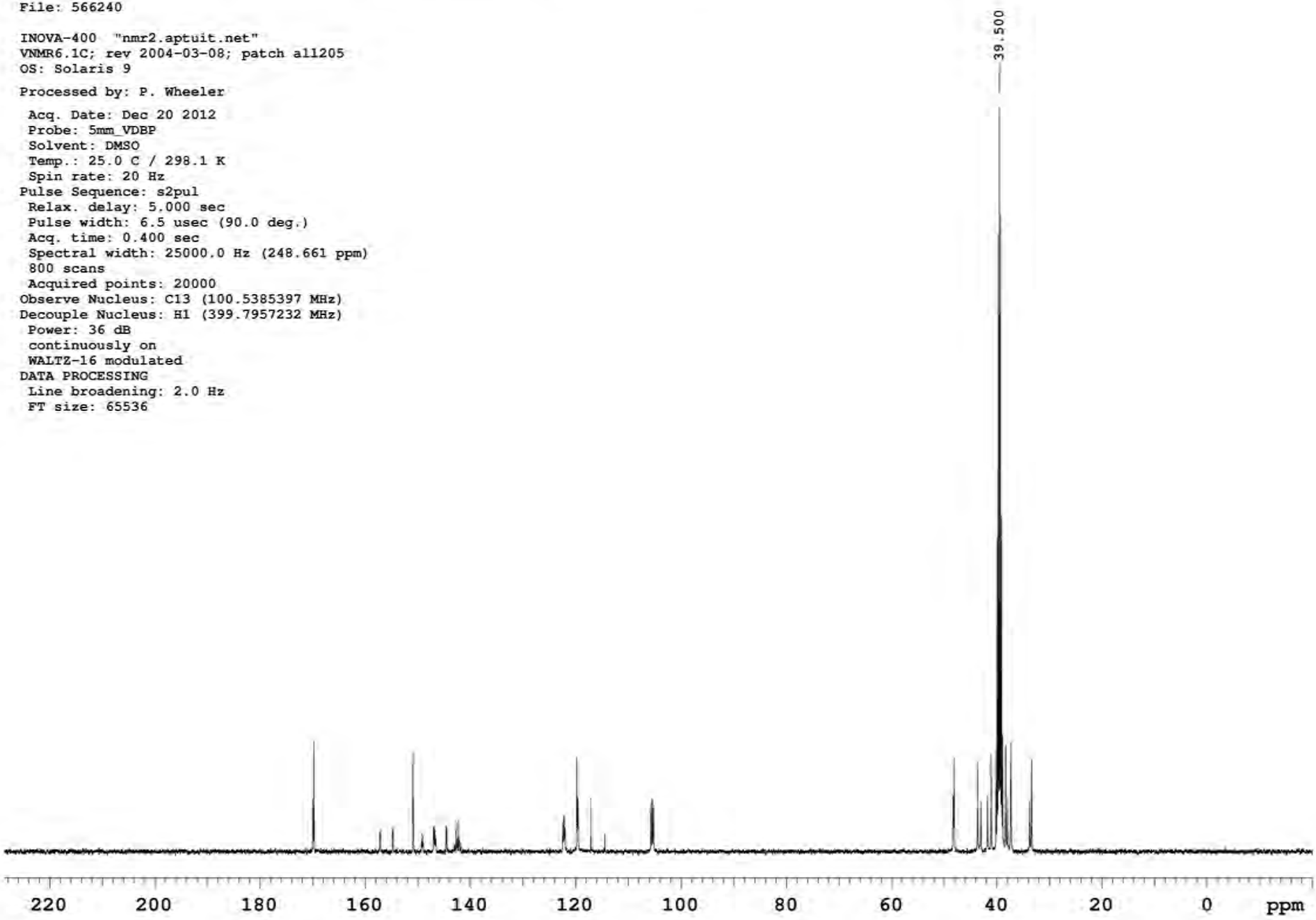
315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566240

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch a11205
OS: Solaris 9

Processed by: P. Wheeler

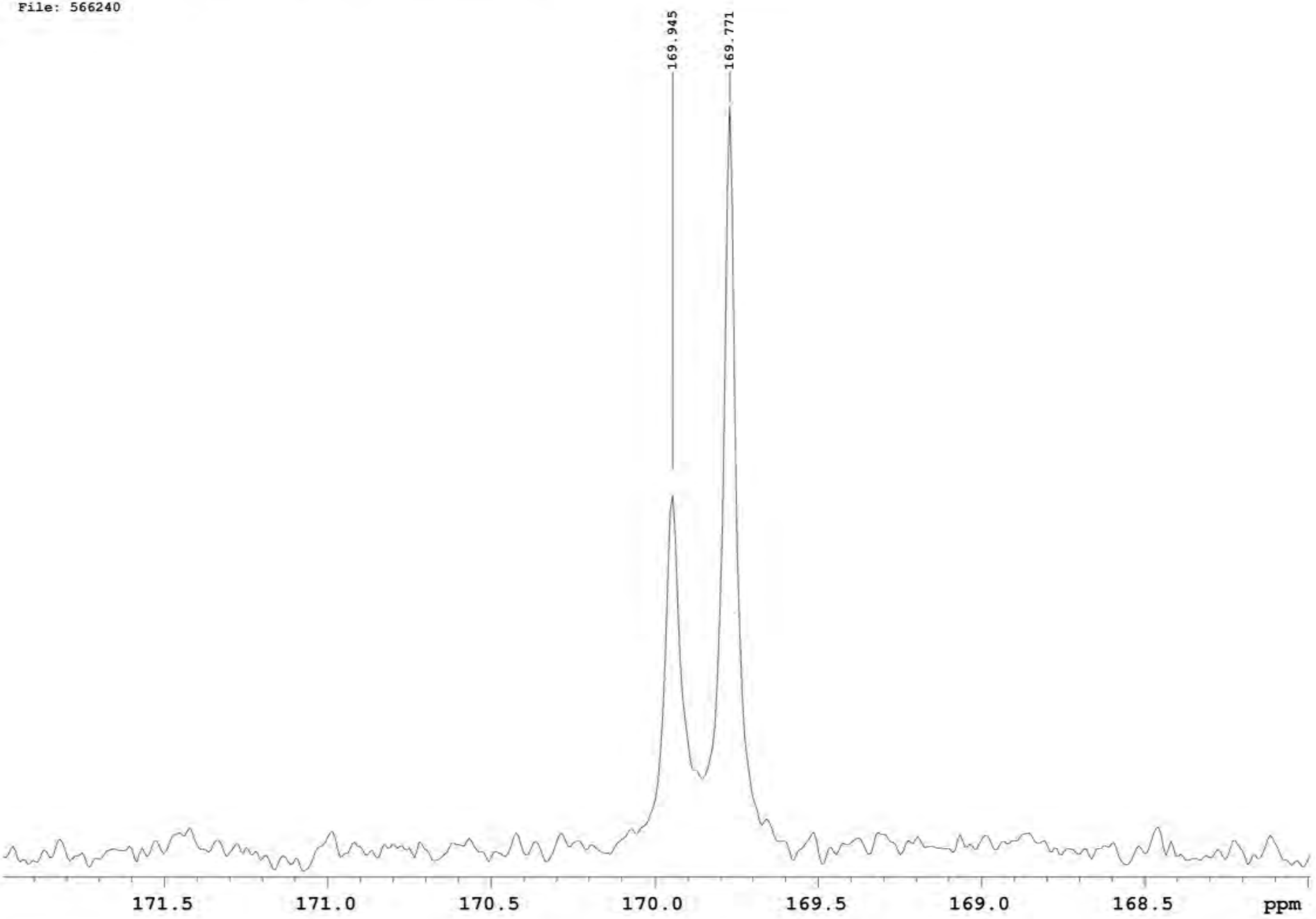
Acq. Date: Dec 20 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 566240-1

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

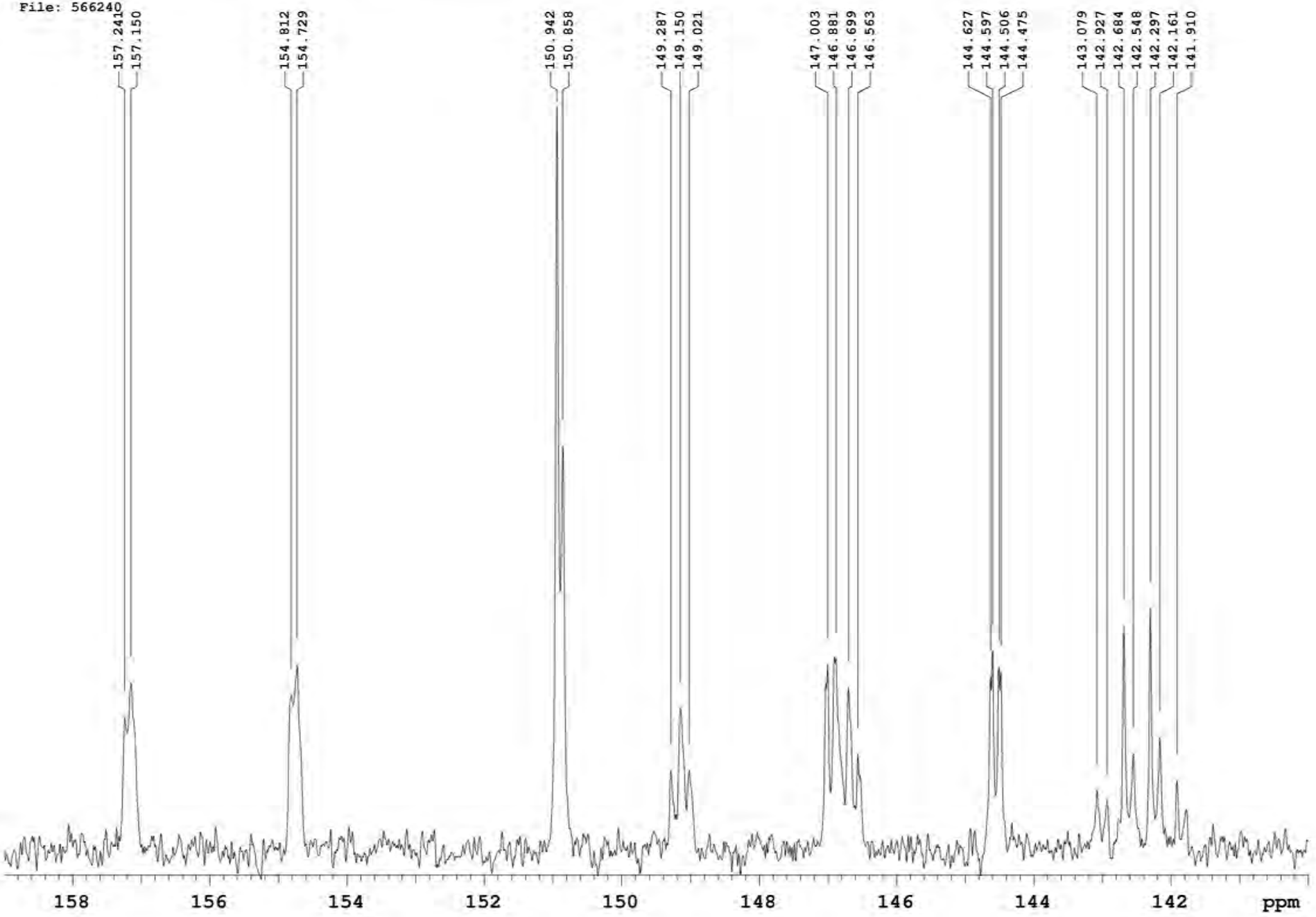
File: 566240



Plot file: 566240-2

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

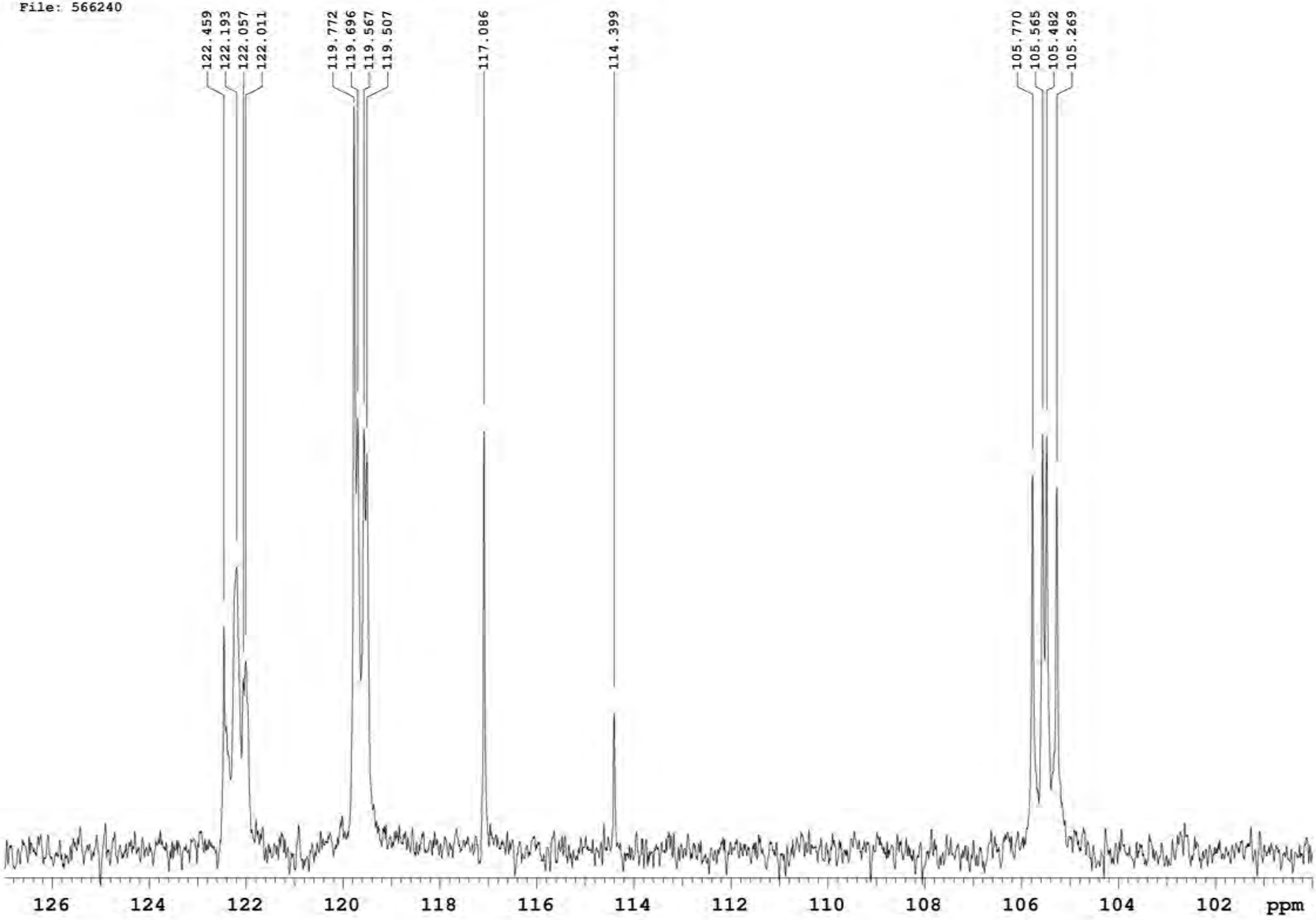
File: 566240



Plot file: 566240-3

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

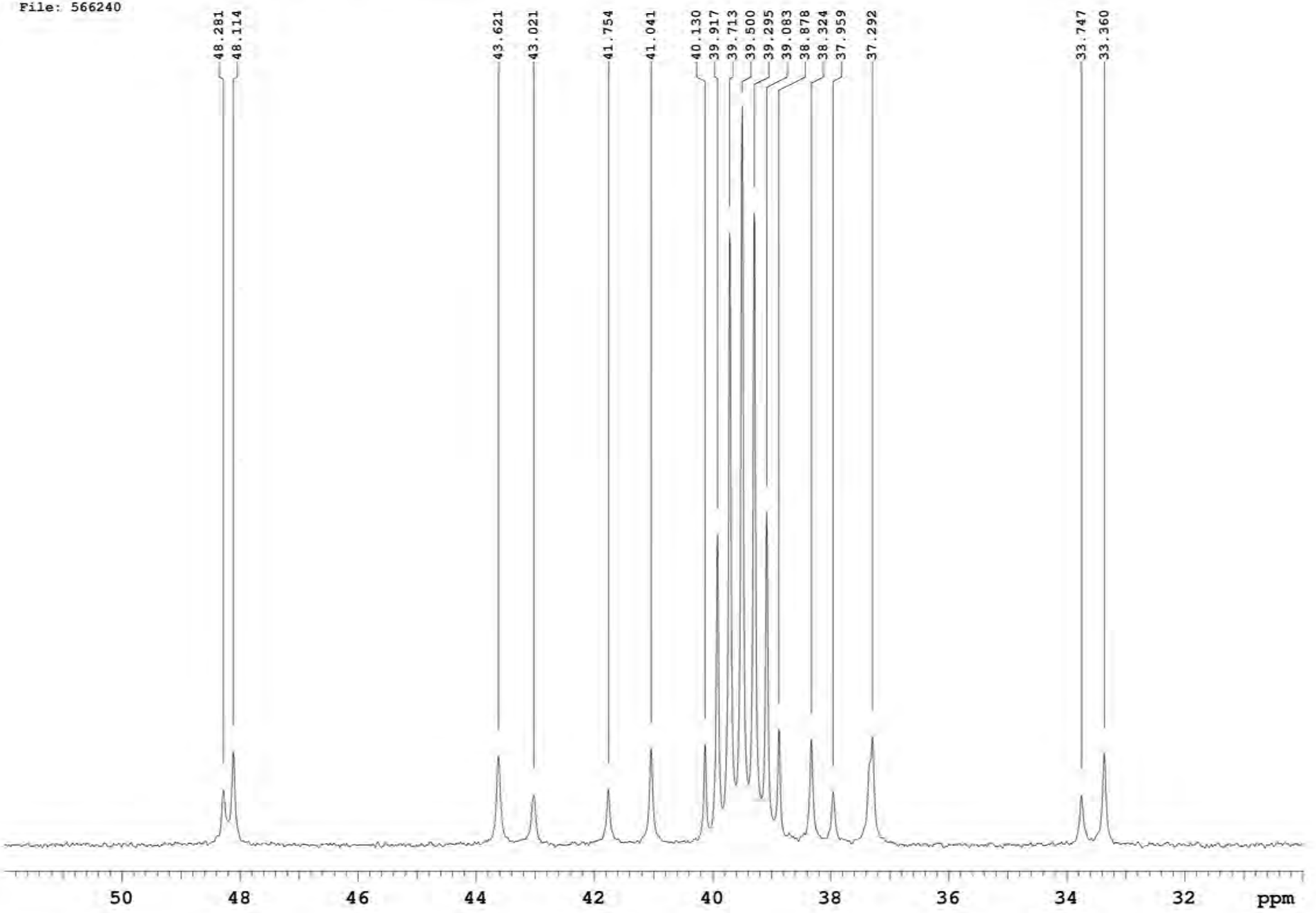
File: 566240



Plot file: 566240-4

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566240



Plot file: 566240-5

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566240

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 566240-1_peaks

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566240

INDEX	FREQUENCY	PPM	HEIGHT
1	17084.262	169.945	67.7
2	17066.714	169.771	141.8

Plot file: 566240-2_peaks

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566240

INDEX	FREQUENCY	PPM	HEIGHT
1	15807.101	157.241	25.1
2	15797.946	157.150	31.7
3	15562.960	154.812	29.4
4	15554.568	154.729	35.2
5	15173.861	150.942	141.8
6	15165.469	150.858	76.9
7	15007.540	149.287	15.0
8	14993.807	149.150	26.8
9	14980.837	149.021	15.0
10	14777.896	147.003	35.2
11	14765.689	146.881	36.3
12	14747.378	146.699	30.9
13	14733.645	146.563	18.1
14	14539.096	144.627	32.9
15	14536.044	144.597	37.8
16	14526.889	144.506	34.8
17	14523.837	144.475	33.8
18	14383.456	143.079	11.2
19	14368.197	142.927	9.3
20	14343.783	142.684	42.6
21	14330.050	142.548	18.2
22	14304.873	142.297	45.9
23	14291.140	142.161	21.3
24	14265.963	141.910	13.0

Plot file: 566240-3_peaks

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566240

INDEX	FREQUENCY	PPM	HEIGHT
1	12310.549	122.459	42.9
2	12283.847	122.193	54.1
3	12270.114	122.057	32.9
4	12265.536	122.011	36.2
5	12040.469	119.772	141.8
6	12032.839	119.696	82.5
7	12019.869	119.567	80.3
8	12013.766	119.507	75.7
9	11770.388	117.086	80.0
10	11500.308	114.399	26.3
11	10632.846	105.770	71.6
12	10612.246	105.565	79.3
13	10603.854	105.482	79.1
14	10582.492	105.269	69.4

Plot file: 566240-4_peaks

315436, 5135-32-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566240

INDEX	FREQUENCY	PPM	HEIGHT
1	4853.579	48.281	10.6
2	4836.795	48.114	17.9
3	4385.134	43.621	17.1
4	4324.862	43.021	9.7
5	4197.451	41.754	10.6
6	4125.735	41.041	18.5
7	4034.182	40.130	19.2
8	4012.820	39.917	59.6
9	3992.221	39.713	117.3
10	3970.858	39.500	141.8
11	3950.259	39.295	121.2
12	3928.897	39.083	63.8
13	3908.297	38.878	22.2
14	3852.603	38.324	20.3
15	3815.982	37.959	10.3
16	3748.843	37.292	20.7
17	3392.550	33.747	9.6
18	3353.640	33.360	17.5

Plot file: 566240-5_peaks

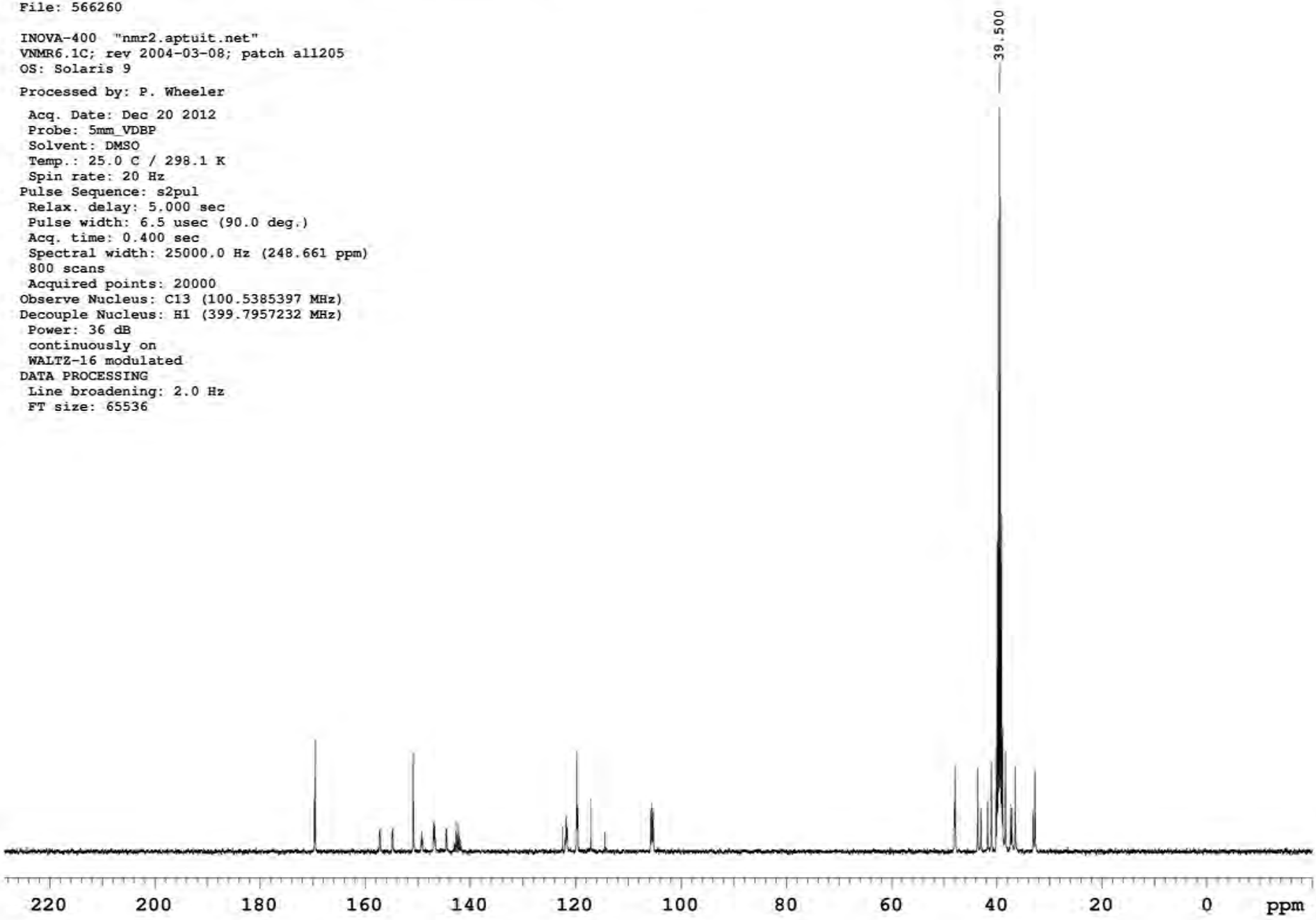
315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566260

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch a11205
OS: Solaris 9

Processed by: P. Wheeler

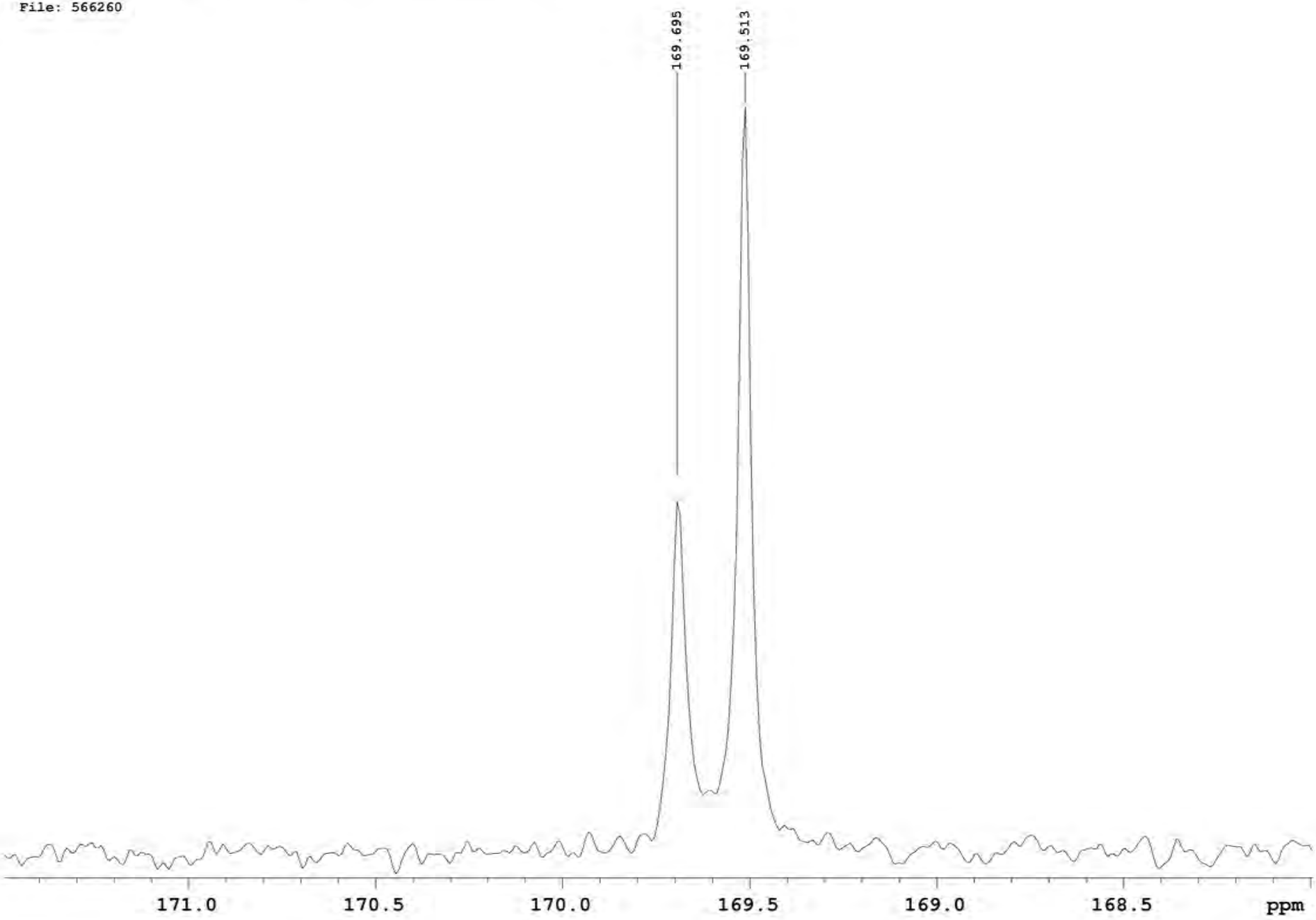
Acq. Date: Dec 20 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 566260-1

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

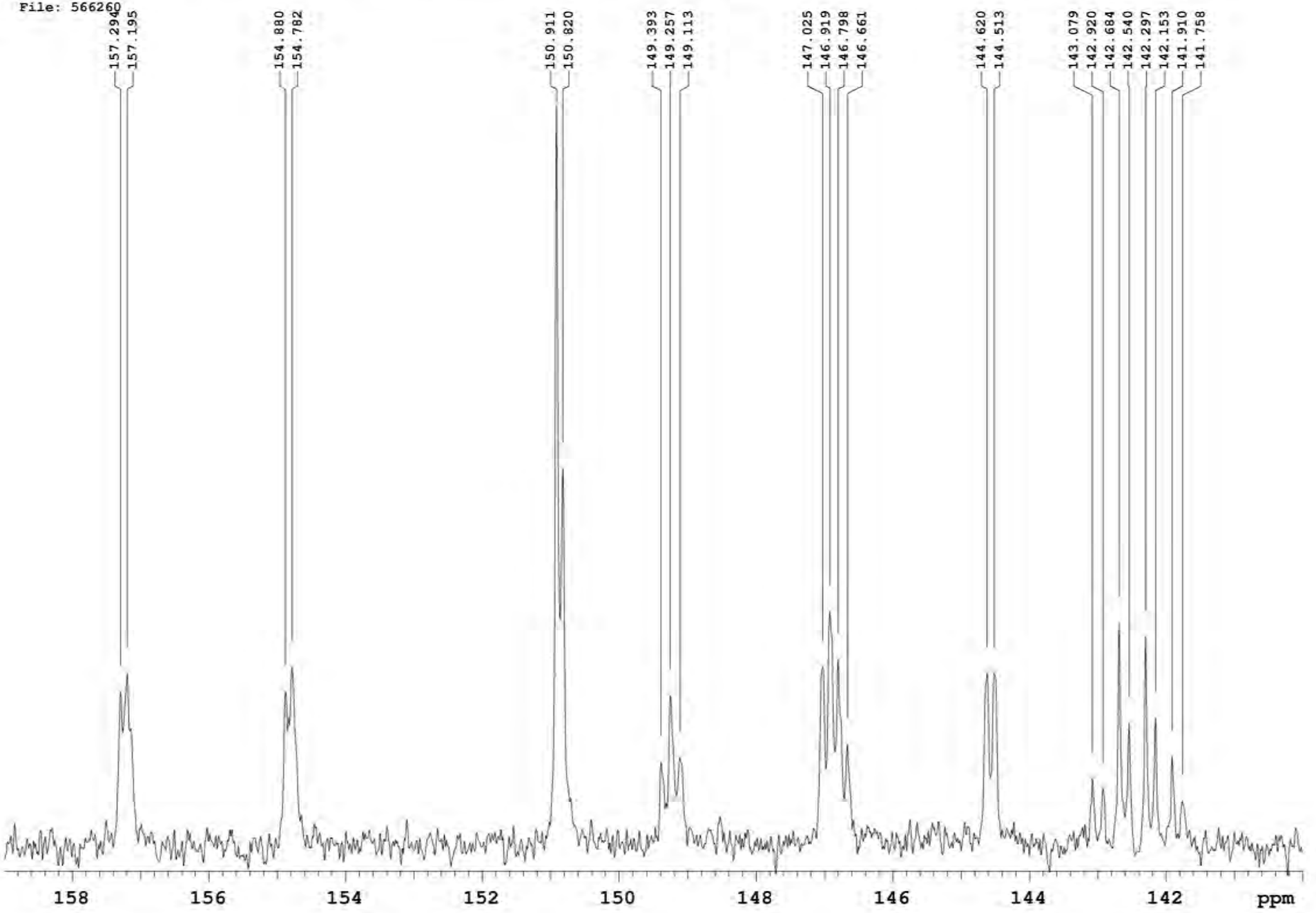
File: 566260



Plot file: 566260-2

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

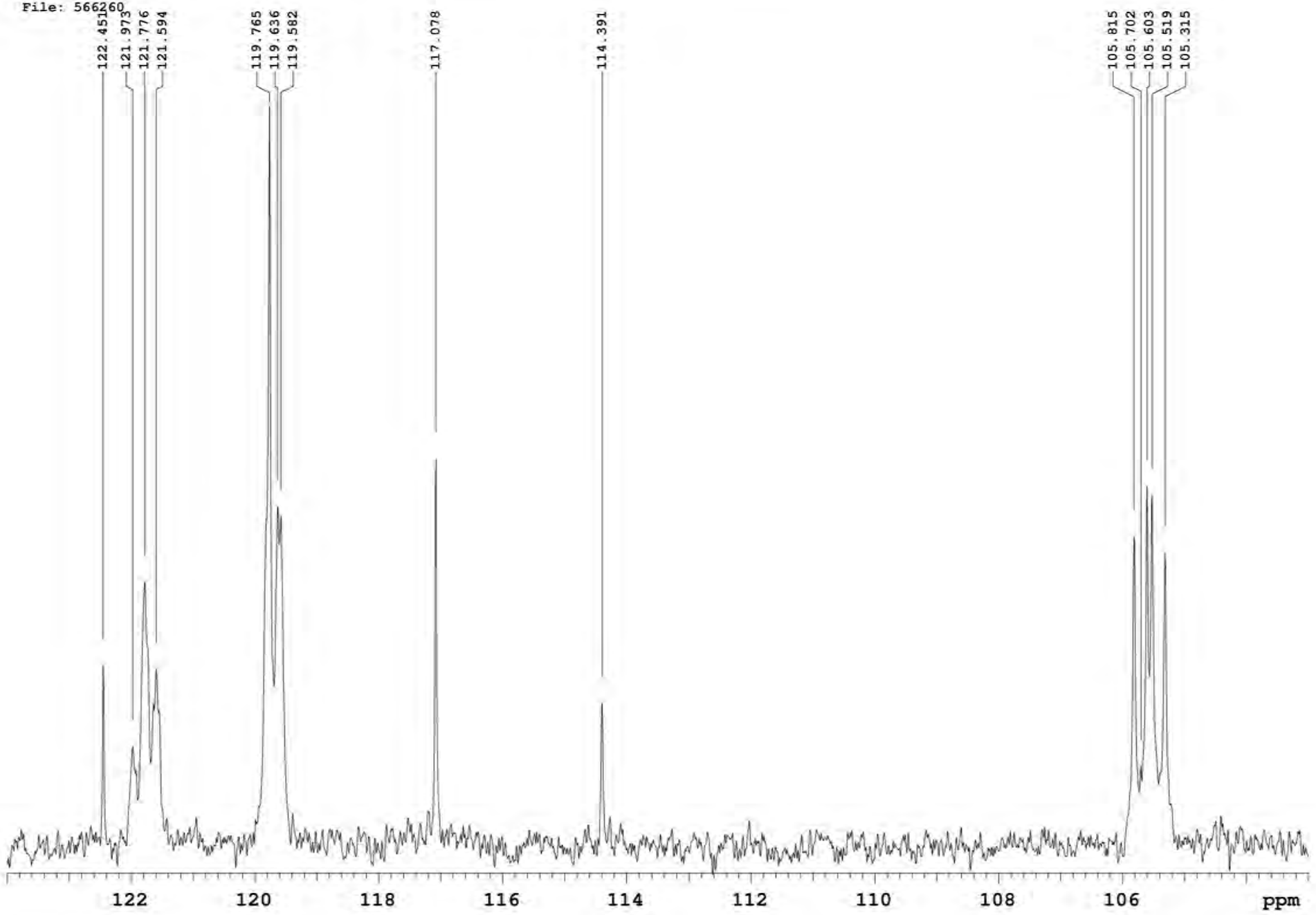
File: 566260



Plot file: 566260-3

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

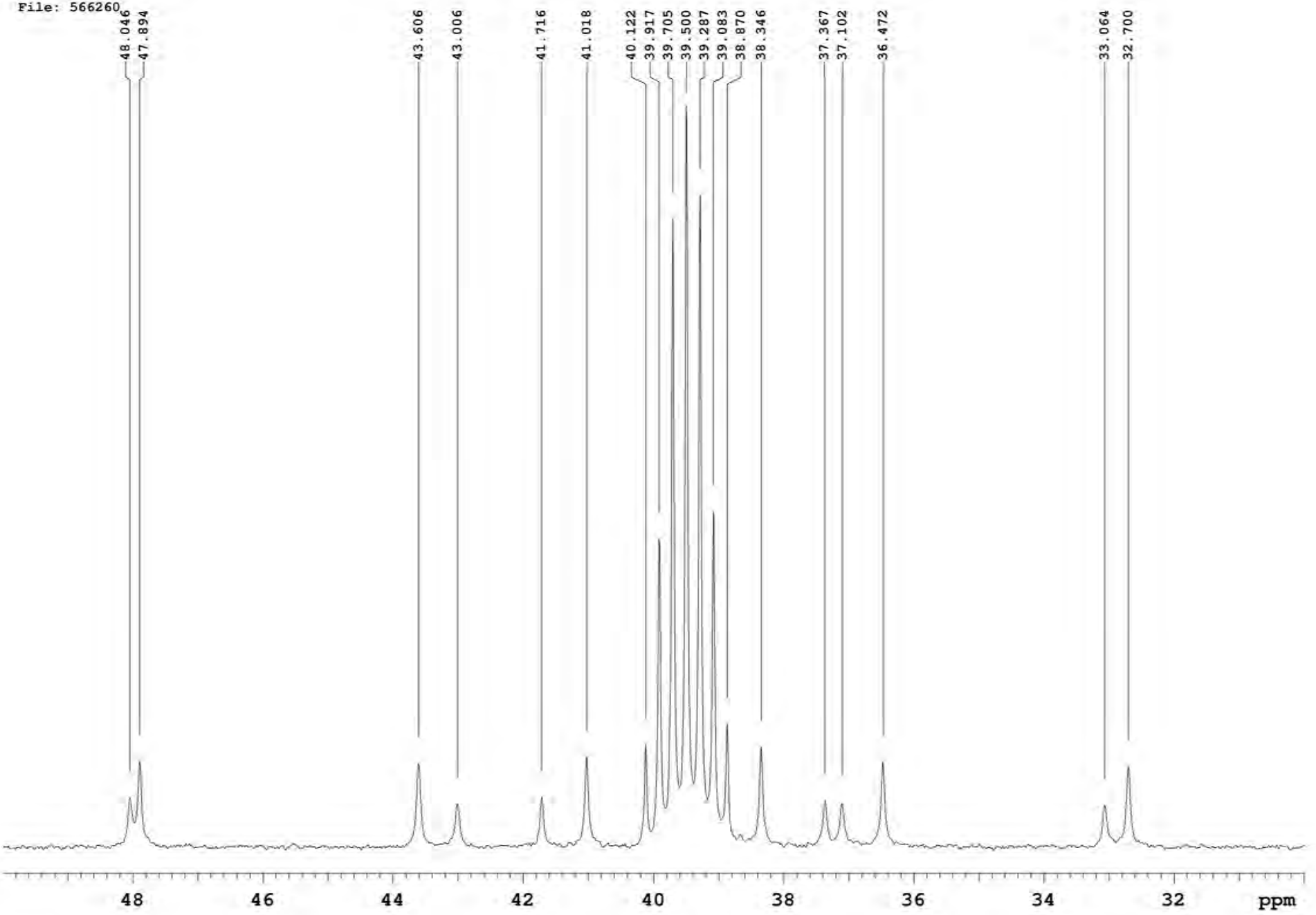
File: 566260



Plot file: 566260-4

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566260



Plot file: 566260-5

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566260

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 566260-1_peaks

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566260

INDEX	FREQUENCY	PPM	HEIGHT
1	17059.085	169.695	66.6
2	17040.774	169.513	141.8

Plot file: 566260-2_peaks

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566260

INDEX	FREQUENCY	PPM	HEIGHT
1	15812.441	157.294	29.4
2	15802.523	157.195	32.9
3	15569.827	154.880	29.5
4	15559.909	154.782	34.1
5	15170.809	150.911	141.8
6	15161.654	150.820	72.2
7	15018.222	149.393	16.0
8	15004.489	149.257	28.7
9	14989.993	149.113	16.7
10	14780.184	147.025	34.2
11	14769.503	146.919	44.8
12	14757.296	146.798	35.6
13	14743.563	146.661	19.3
14	14538.333	144.620	33.0
15	14527.651	144.513	32.9
16	14383.456	143.079	12.6
17	14367.434	142.920	10.8
18	14343.783	142.684	42.7
19	14329.287	142.540	23.4
20	14304.873	142.297	40.0
21	14290.377	142.153	24.4
22	14265.963	141.910	17.0
23	14250.704	141.758	8.5

Plot file: 566260-3_peaks

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566260

INDEX	FREQUENCY	PPM	HEIGHT
1	12309.786	122.451	34.8
2	12261.721	121.973	19.3
3	12241.885	121.776	50.6
4	12223.574	121.594	34.0
5	12039.706	119.765	141.8
6	12026.736	119.636	65.1
7	12021.395	119.582	63.3
8	11769.625	117.078	74.3
9	11499.545	114.391	27.5
10	10637.423	105.815	59.4
11	10625.979	105.702	15.5
12	10616.061	105.603	69.0
13	10607.669	105.519	67.3
14	10587.069	105.315	56.3

Plot file: 566260-4_peaks

315452, 5135-32-02, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566260

INDEX	FREQUENCY	PPM	HEIGHT
1	4829.928	48.046	9.5
2	4814.669	47.894	16.4
3	4383.608	43.606	16.0
4	4323.336	43.006	8.2
5	4193.637	41.716	9.5
6	4123.446	41.018	17.1
7	4033.419	40.122	19.6
8	4012.820	39.917	58.9
9	3991.458	39.705	120.2
10	3970.858	39.500	141.8
11	3949.496	39.287	124.6
12	3928.897	39.083	64.2
13	3907.534	38.870	23.6
14	3854.891	38.346	19.2
15	3756.472	37.367	9.0
16	3729.769	37.102	8.6
17	3666.445	36.472	16.2
18	3323.886	33.064	8.0
19	3287.264	32.700	15.4

Plot file: 566260-5_peaks

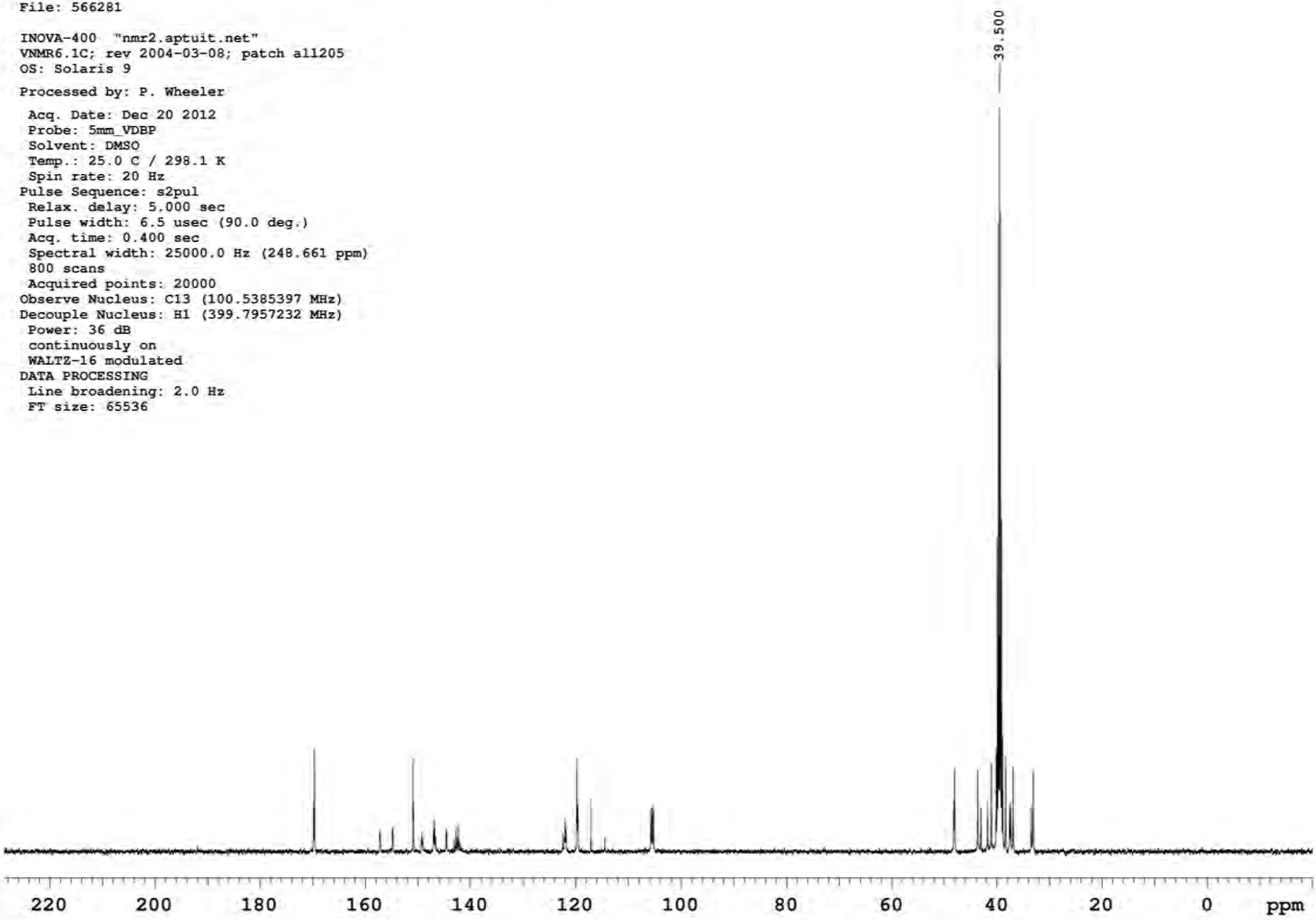
314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566281

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch a11205
OS: Solaris 9

Processed by: P. Wheeler

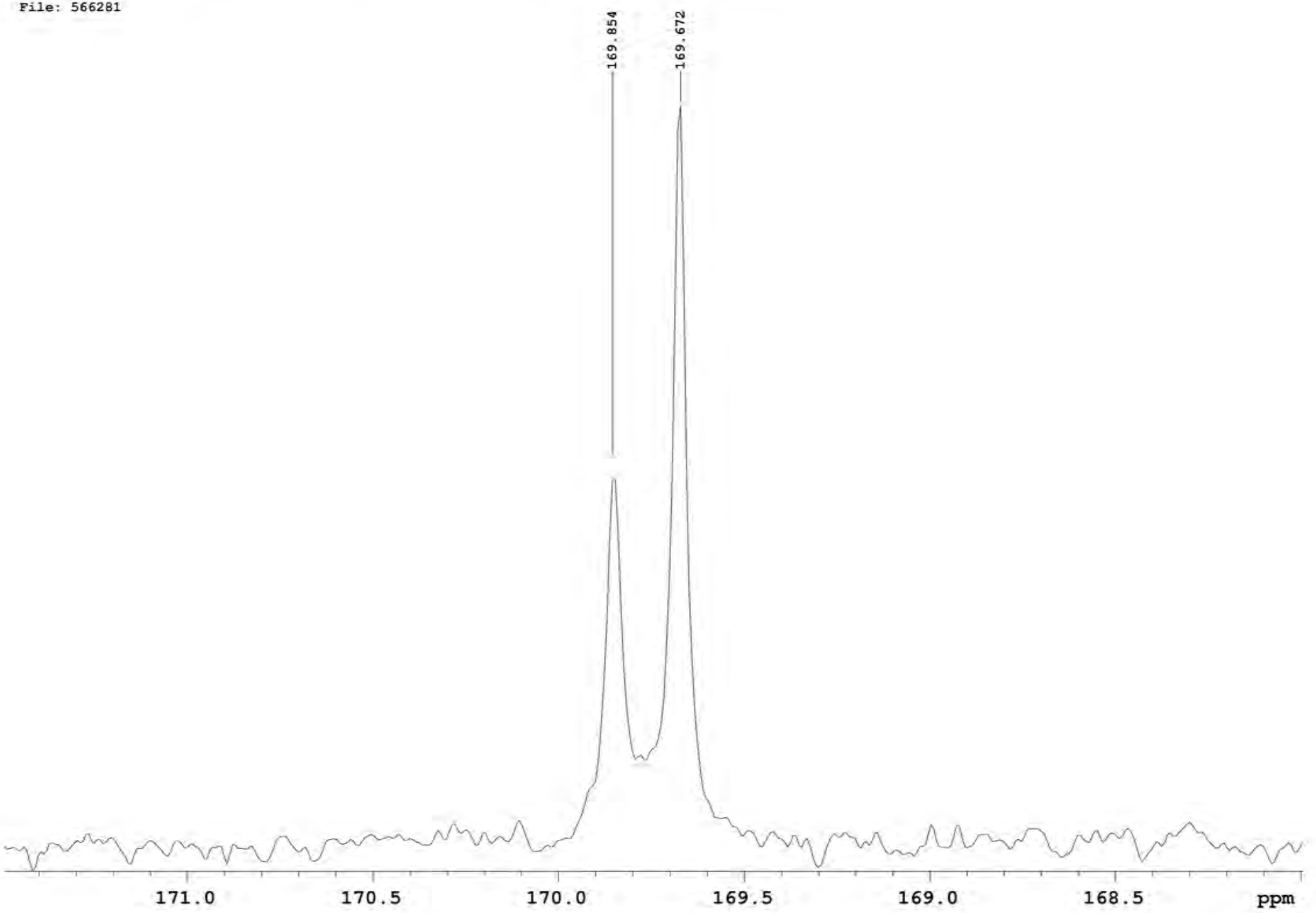
Acq. Date: Dec 20 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 566281-1

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

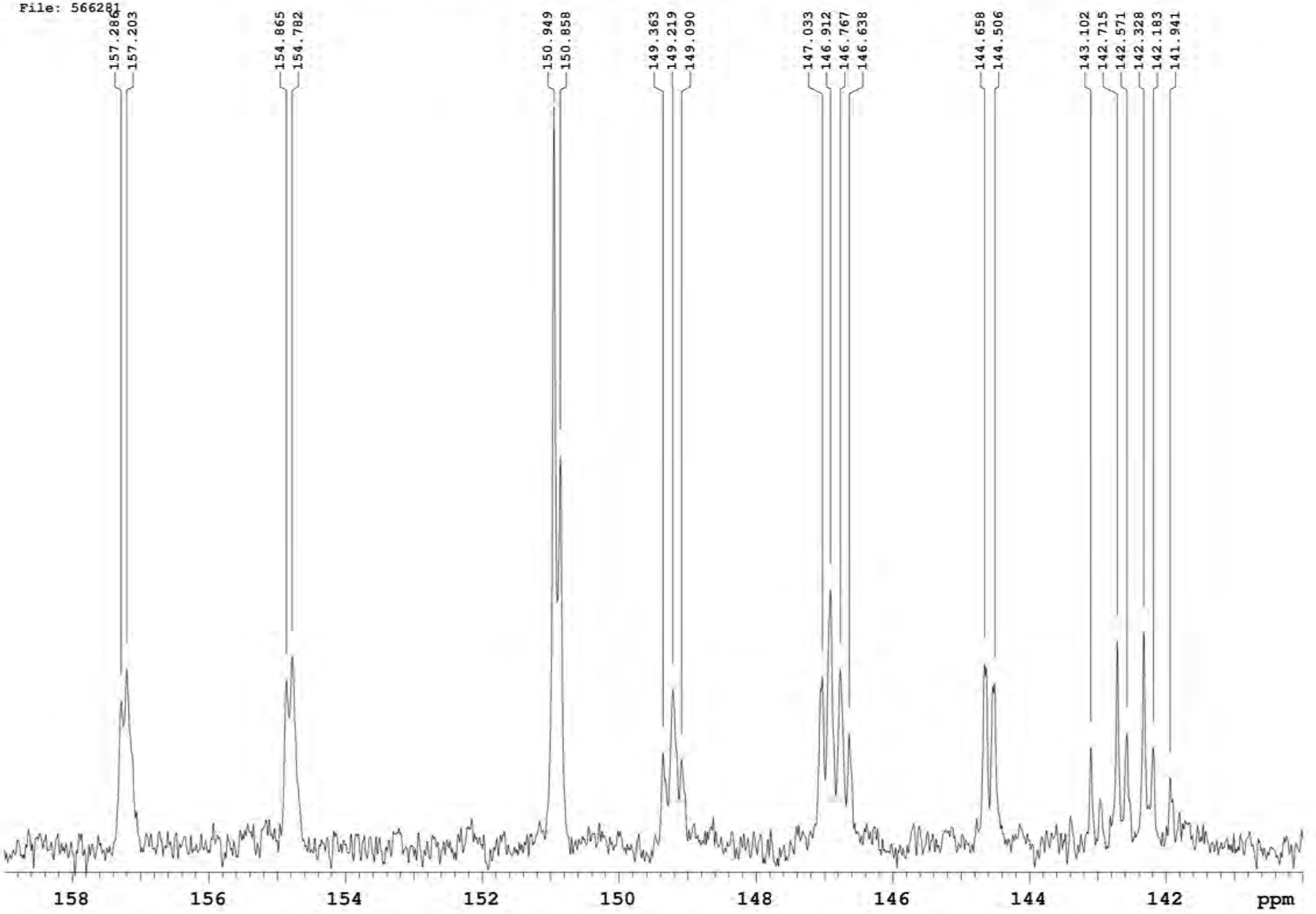
File: 566281



Plot file: 566281-2

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

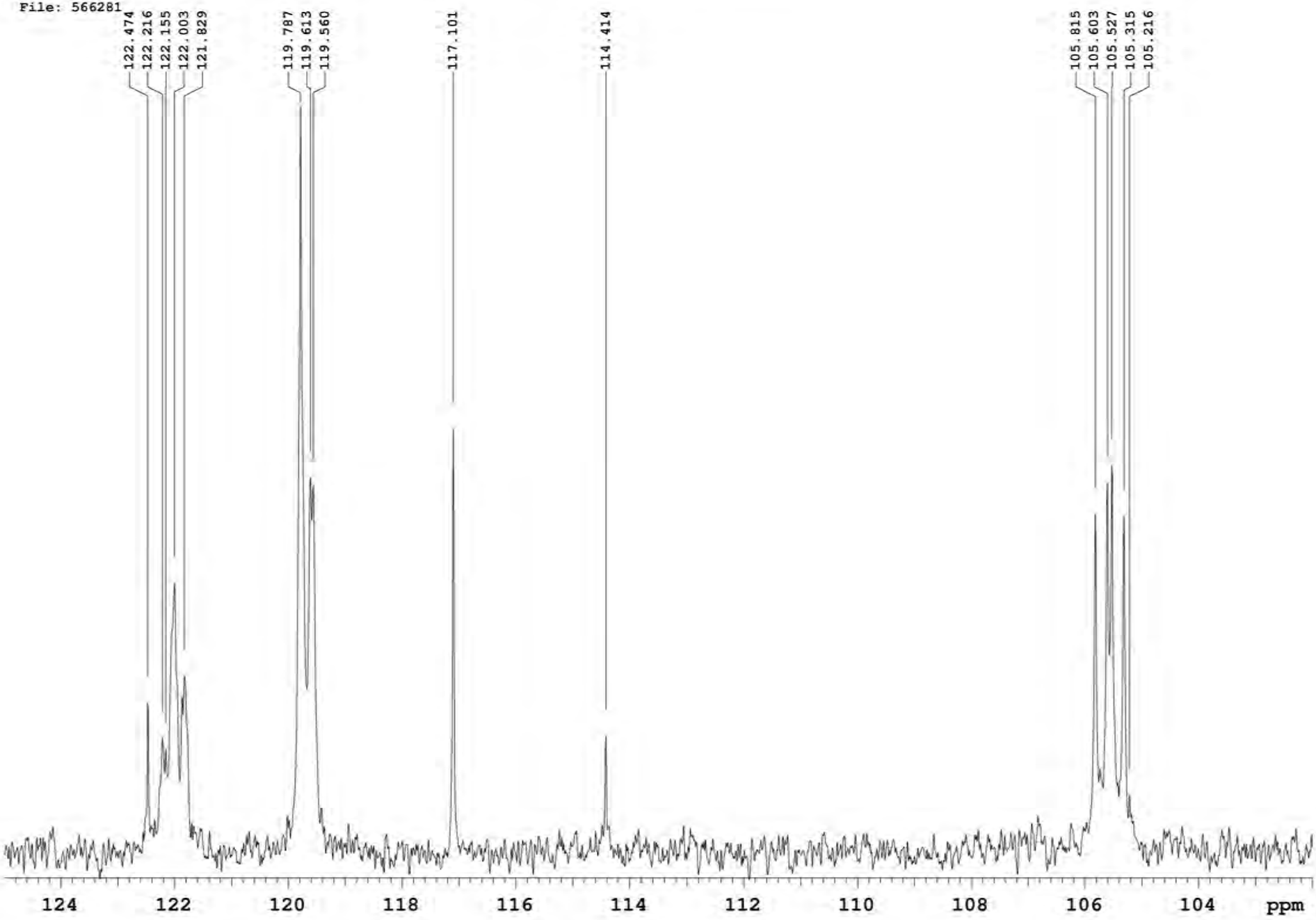
File: 566281



Plot file: 566281-3

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

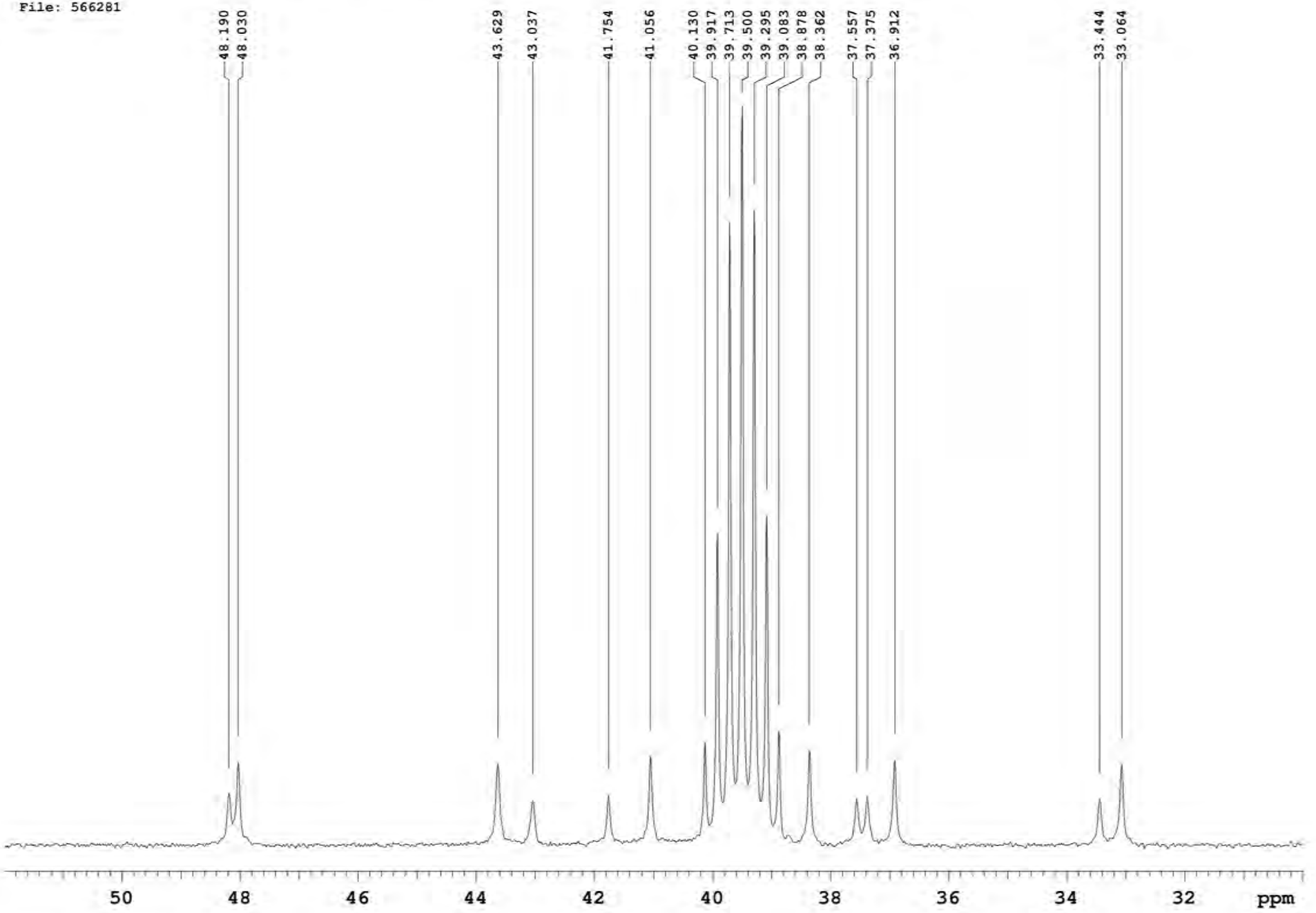
File: 566281



Plot file: 566281-4

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566281



Plot file: 566281-5

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566281

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 566281-1_peaks

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566281

INDEX	FREQUENCY	PPM	HEIGHT
1	17075.106	169.854	70.0
2	17056.796	169.672	141.8

Plot file: 566281-2_peaks

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566281

INDEX	FREQUENCY	PPM	HEIGHT
1	15811.678	157.286	27.8
2	15803.286	157.203	34.0
3	15568.301	154.865	31.9
4	15559.908	154.782	36.4
5	15174.624	150.949	141.8
6	15165.469	150.858	74.8
7	15015.170	149.363	17.9
8	15000.674	149.219	30.0
9	14987.704	149.090	16.6
10	14780.947	147.033	32.5
11	14768.740	146.912	49.1
12	14754.244	146.767	33.9
13	14741.274	146.638	21.5
14	14542.147	144.658	35.0
15	14526.888	144.506	31.1
16	14385.745	143.102	18.9
17	14346.835	142.715	39.3
18	14332.339	142.571	21.6
19	14307.925	142.328	41.0
20	14293.429	142.183	18.8
21	14269.015	141.941	13.1

Plot file: 566281-3_peaks

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566281

INDEX	FREQUENCY	PPM	HEIGHT
1	12312.075	122.474	28.3
2	12286.135	122.216	21.7
3	12280.032	122.155	19.6
4	12264.773	122.003	51.1
5	12247.225	121.829	33.4
6	12041.995	119.787	141.8
7	12024.447	119.613	71.1
8	12019.106	119.560	69.8
9	11771.914	117.101	80.4
10	11501.833	114.414	22.0
11	10637.423	105.815	64.2
12	10616.061	105.603	70.1
13	10608.431	105.527	73.4
14	10587.069	105.315	63.8
15	10577.151	105.216	10.7

Plot file: 566281-4_peaks

314339, 5135-02-01, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566281

INDEX	FREQUENCY	PPM	HEIGHT
1	4844.424	48.190	9.9
2	4828.402	48.030	15.9
3	4385.897	43.629	15.6
4	4326.388	43.037	8.6
5	4197.451	41.754	9.6
6	4127.261	41.056	16.9
7	4034.182	40.130	19.7
8	4012.820	39.917	59.8
9	3992.220	39.713	119.3
10	3970.858	39.500	141.8
11	3950.259	39.295	121.7
12	3928.896	39.083	63.1
13	3908.297	38.878	21.9
14	3856.417	38.362	18.1
15	3775.546	37.557	9.0
16	3757.235	37.375	9.4
17	3710.696	36.912	16.2
18	3362.032	33.444	8.9
19	3323.885	33.064	15.4

Plot file: 566281-5_peaks

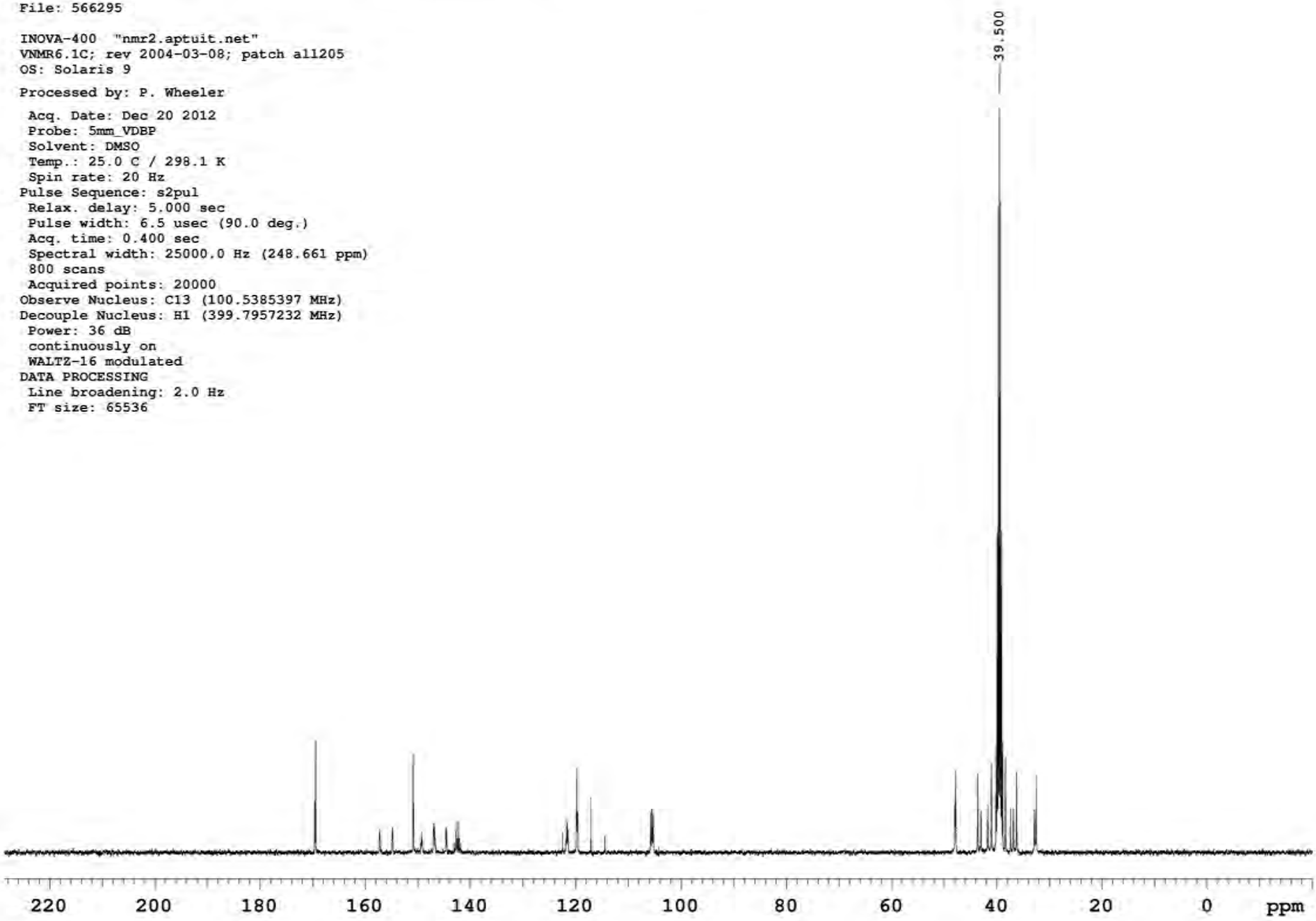
315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566295

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch a11205
OS: Solaris 9

Processed by: P. Wheeler

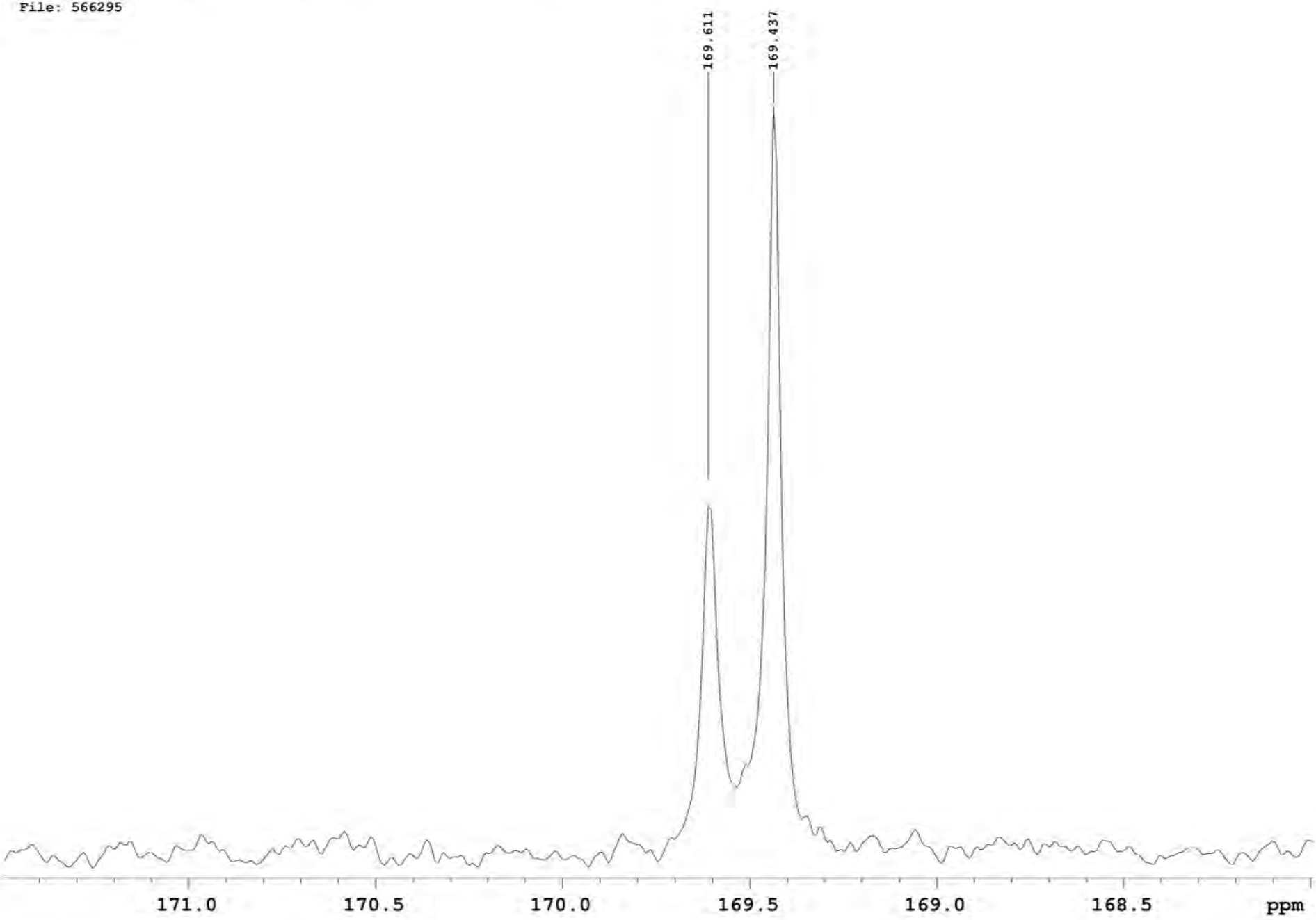
Acq. Date: Dec 20 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 566295-1

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

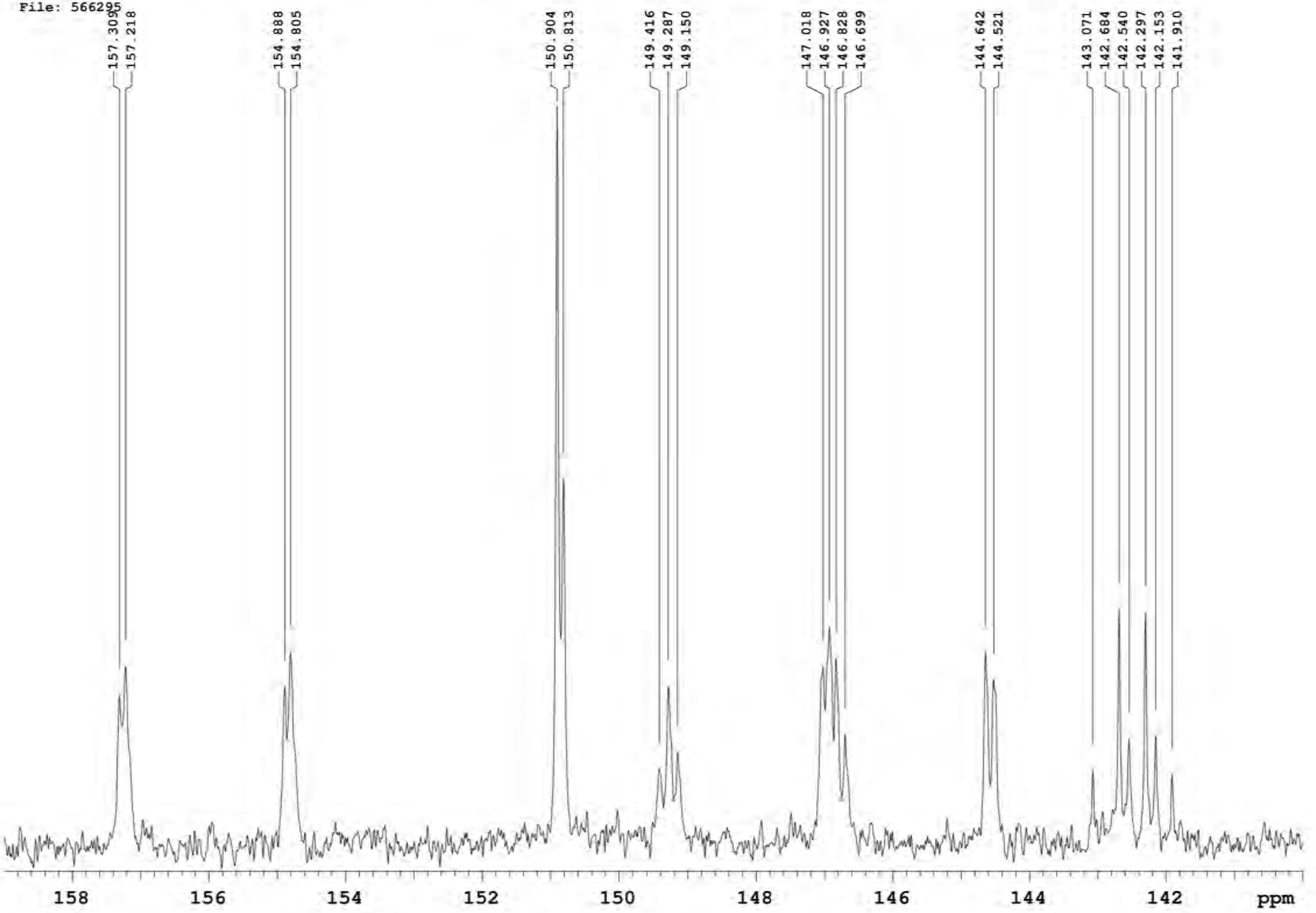
File: 566295



Plot file: 566295-2

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

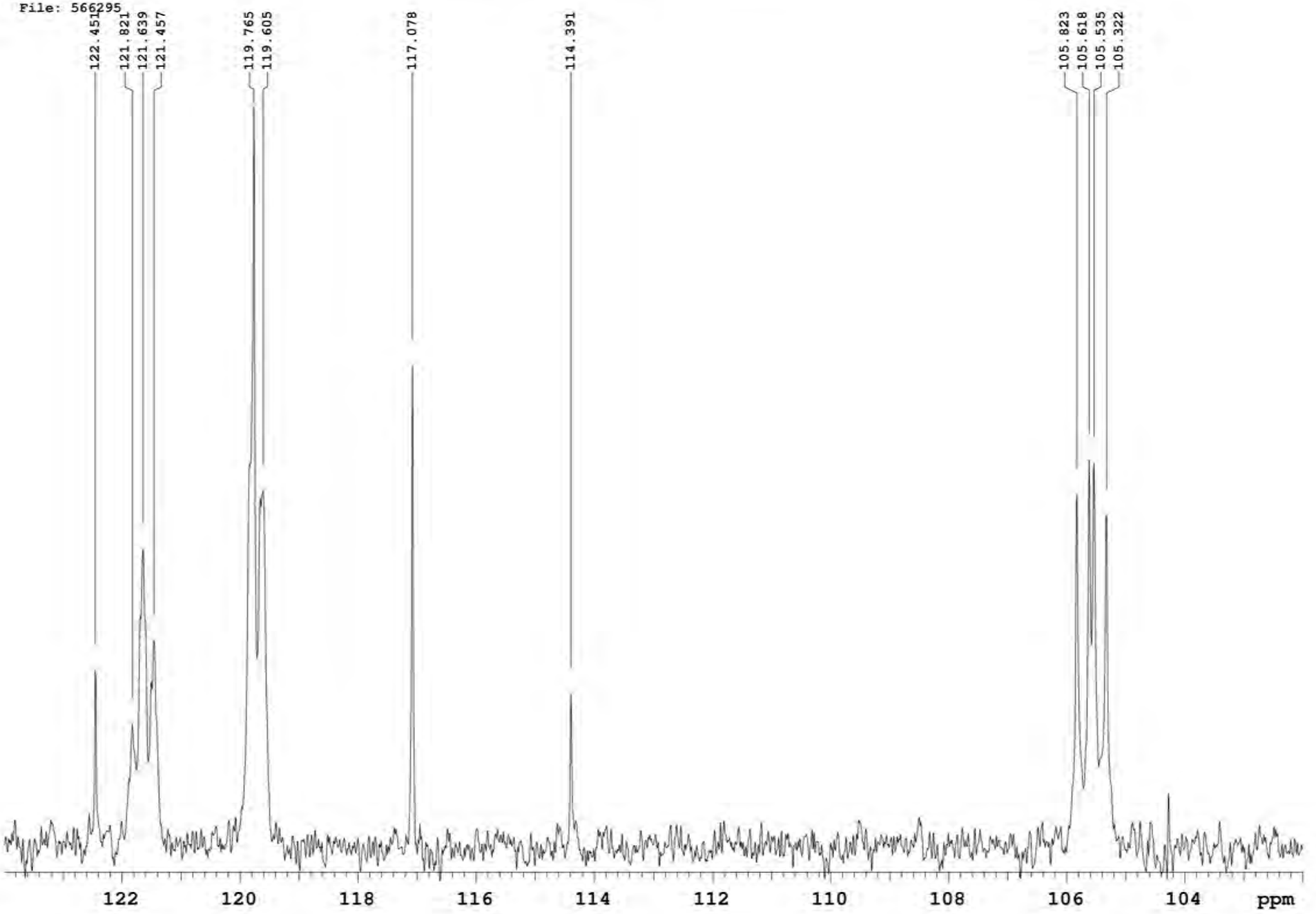
File: 566295



Plot file: 566295-3

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

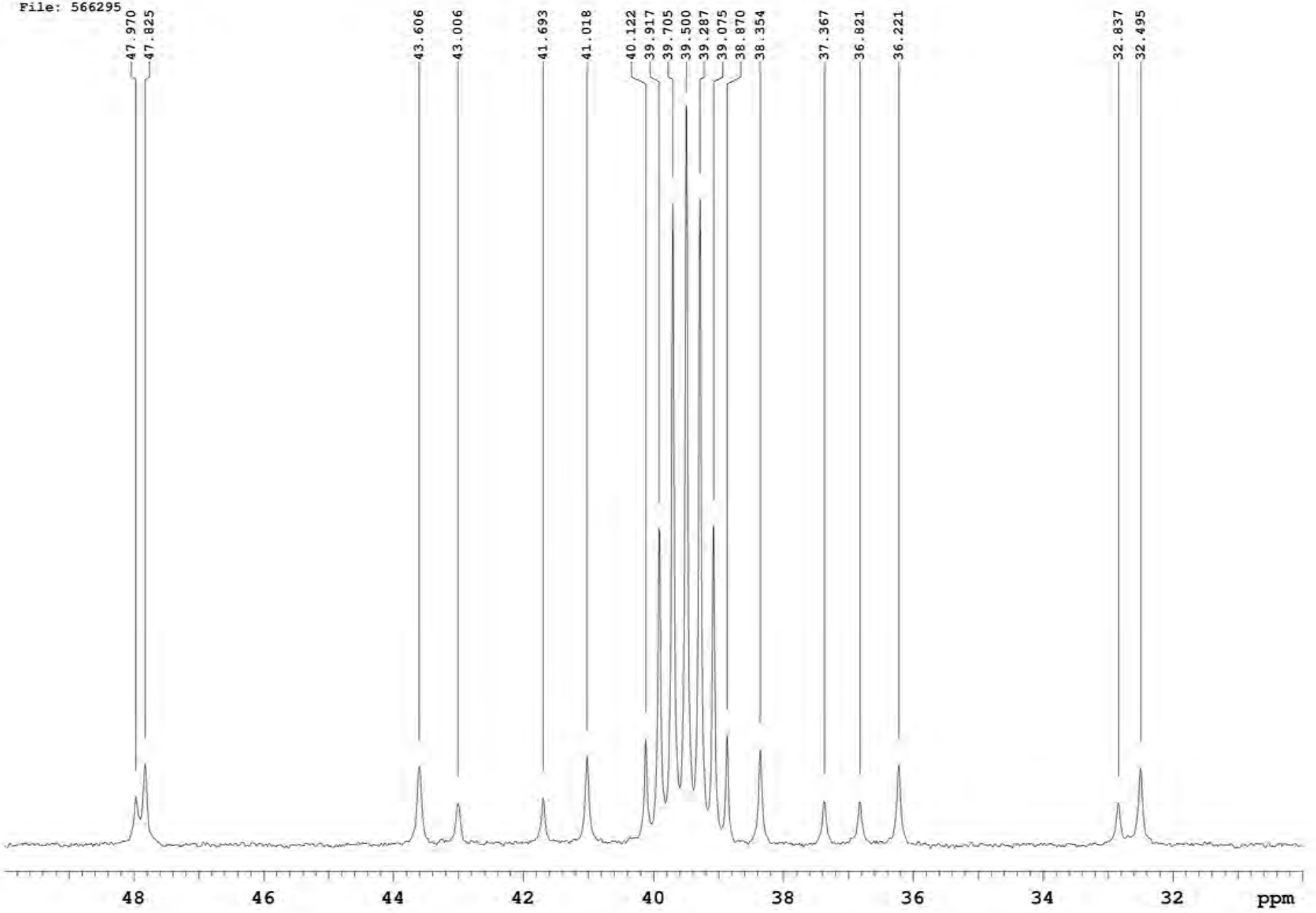
File: 566295



Plot file: 566295-4

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566295



Plot file: 566295-5

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566295

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 566295-1_peaks

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566295

INDEX	FREQUENCY	PPM	HEIGHT
1	17050.692	169.611	65.8
2	17033.145	169.437	141.8

Plot file: 566295-2_peaks

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566295

INDEX	FREQUENCY	PPM	HEIGHT
1	15813.967	157.309	28.9
2	15804.812	157.218	34.2
3	15570.590	154.888	30.5
4	15562.197	154.805	37.1
5	15170.046	150.904	141.8
6	15160.891	150.813	70.2
7	15020.510	149.416	14.6
8	15007.540	149.287	30.4
9	14993.807	149.150	17.8
10	14779.421	147.018	34.1
11	14770.266	146.927	41.9
12	14760.348	146.828	35.8
13	14747.378	146.699	21.3
14	14540.621	144.642	37.1
15	14528.414	144.521	31.7
16	14382.693	143.071	14.3
17	14343.783	142.684	45.3
18	14329.287	142.540	20.5
19	14304.873	142.297	44.6
20	14290.377	142.153	20.9
21	14265.963	141.910	13.6

Plot file: 566295-3_peaks

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566295

INDEX	FREQUENCY	PPM	HEIGHT
1	12309.786	122.451	33.7
2	12246.462	121.821	23.4
3	12228.152	121.639	56.9
4	12209.841	121.457	39.4
5	12039.706	119.765	141.8
6	12023.684	119.605	68.1
7	11769.625	117.078	92.1
8	11499.545	114.391	29.1
9	10638.186	105.823	67.3
10	10617.587	105.618	73.9
11	10609.194	105.535	73.4
12	10587.832	105.322	63.3

Plot file: 566295-4_peaks

315467, 5135-32-04, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566295

INDEX	FREQUENCY	PPM	HEIGHT
1	4822.299	47.970	9.3
2	4807.803	47.825	15.6
3	4383.608	43.606	15.1
4	4323.336	43.006	8.0
5	4191.348	41.693	9.1
6	4123.446	41.018	17.0
7	4033.419	40.122	20.4
8	4012.820	39.917	60.8
9	3991.458	39.705	123.0
10	3970.858	39.500	141.8
11	3949.496	39.287	123.8
12	3928.134	39.075	61.2
13	3907.534	38.870	21.1
14	3855.654	38.354	18.2
15	3756.472	37.367	8.6
16	3701.541	36.821	8.5
17	3641.268	36.221	15.4
18	3300.997	32.837	8.1
19	3266.665	32.495	14.8

Plot file: 566295-5_peaks

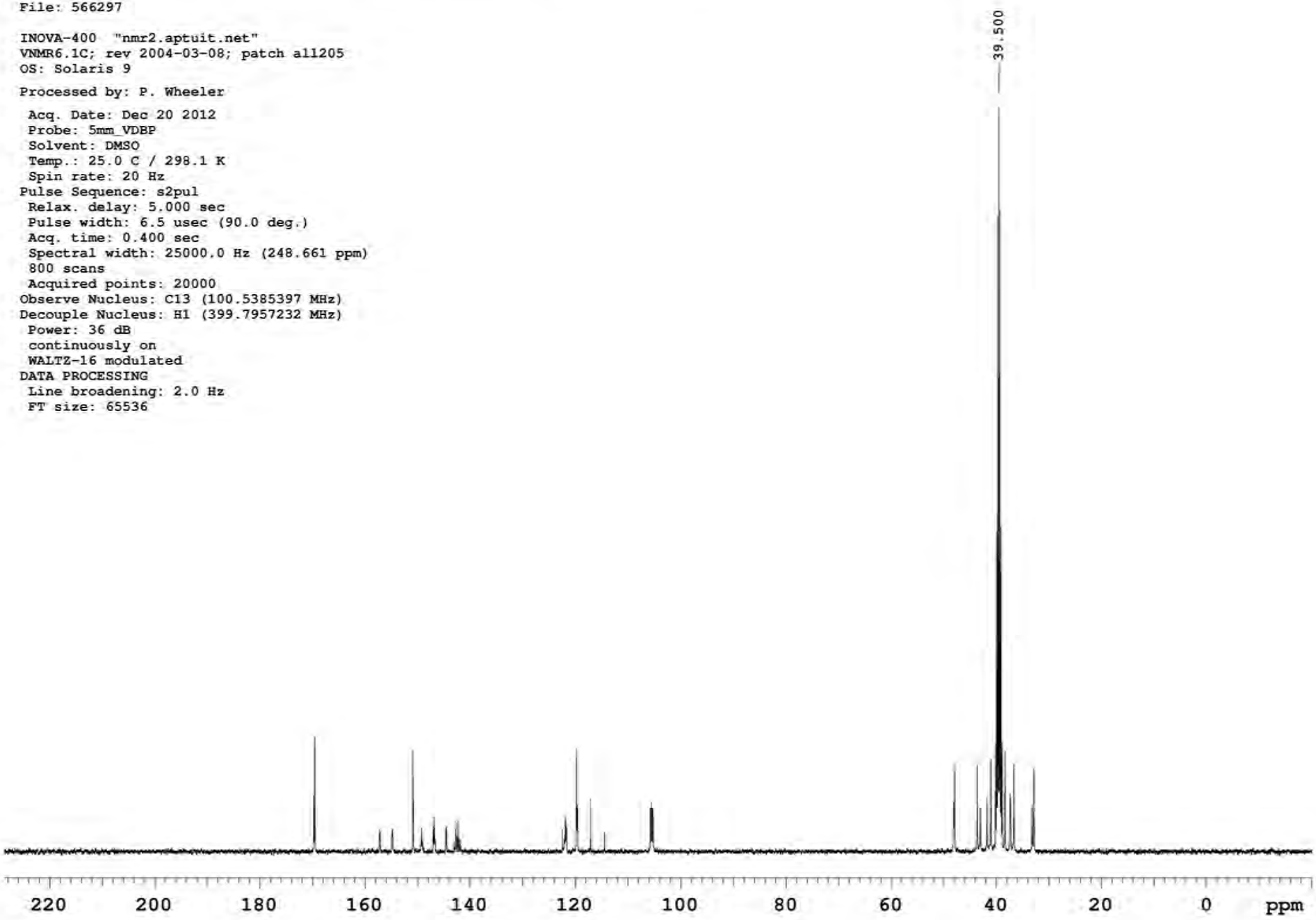
315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566297

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

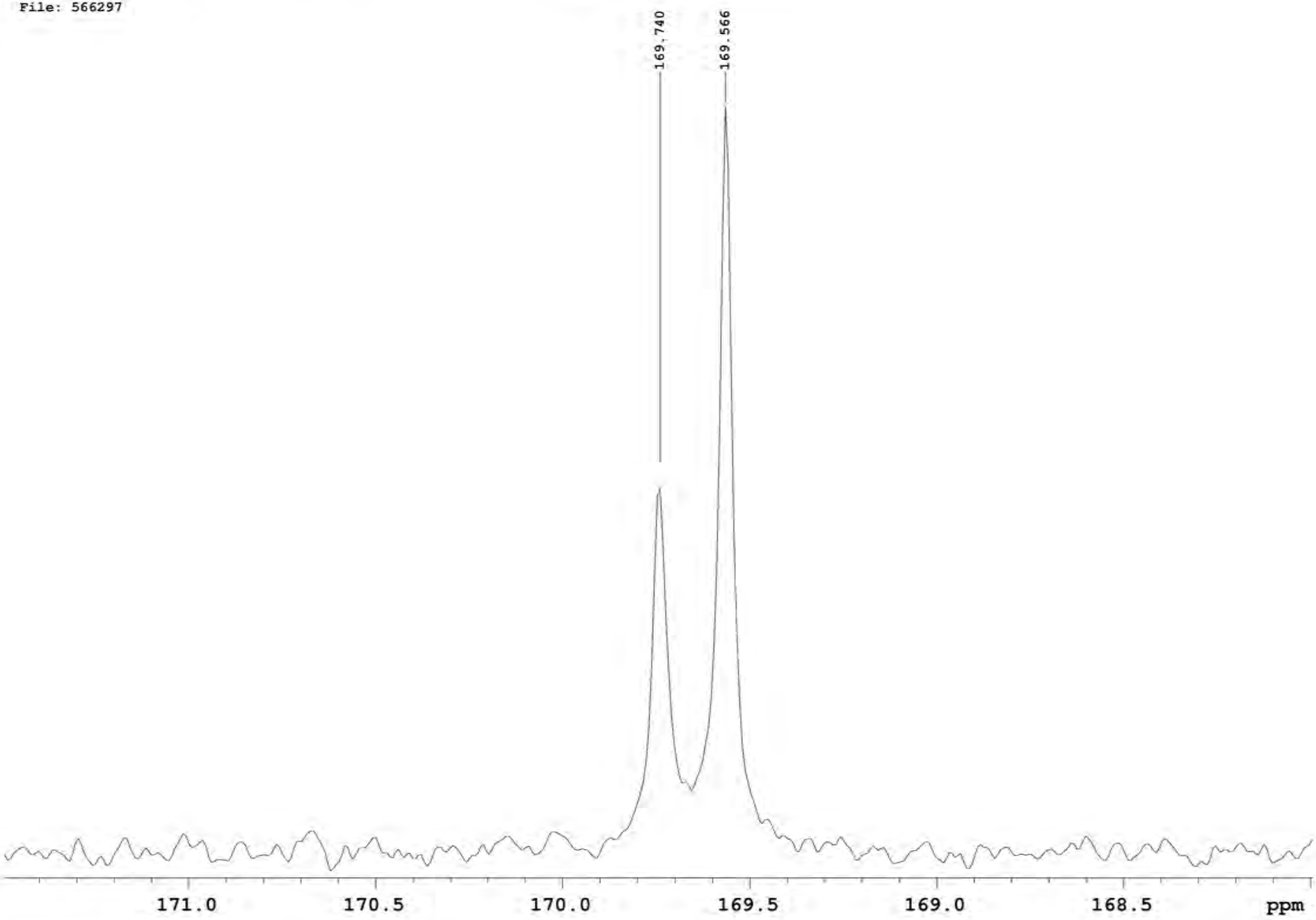
Acq. Date: Dec 20 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 566297-1

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

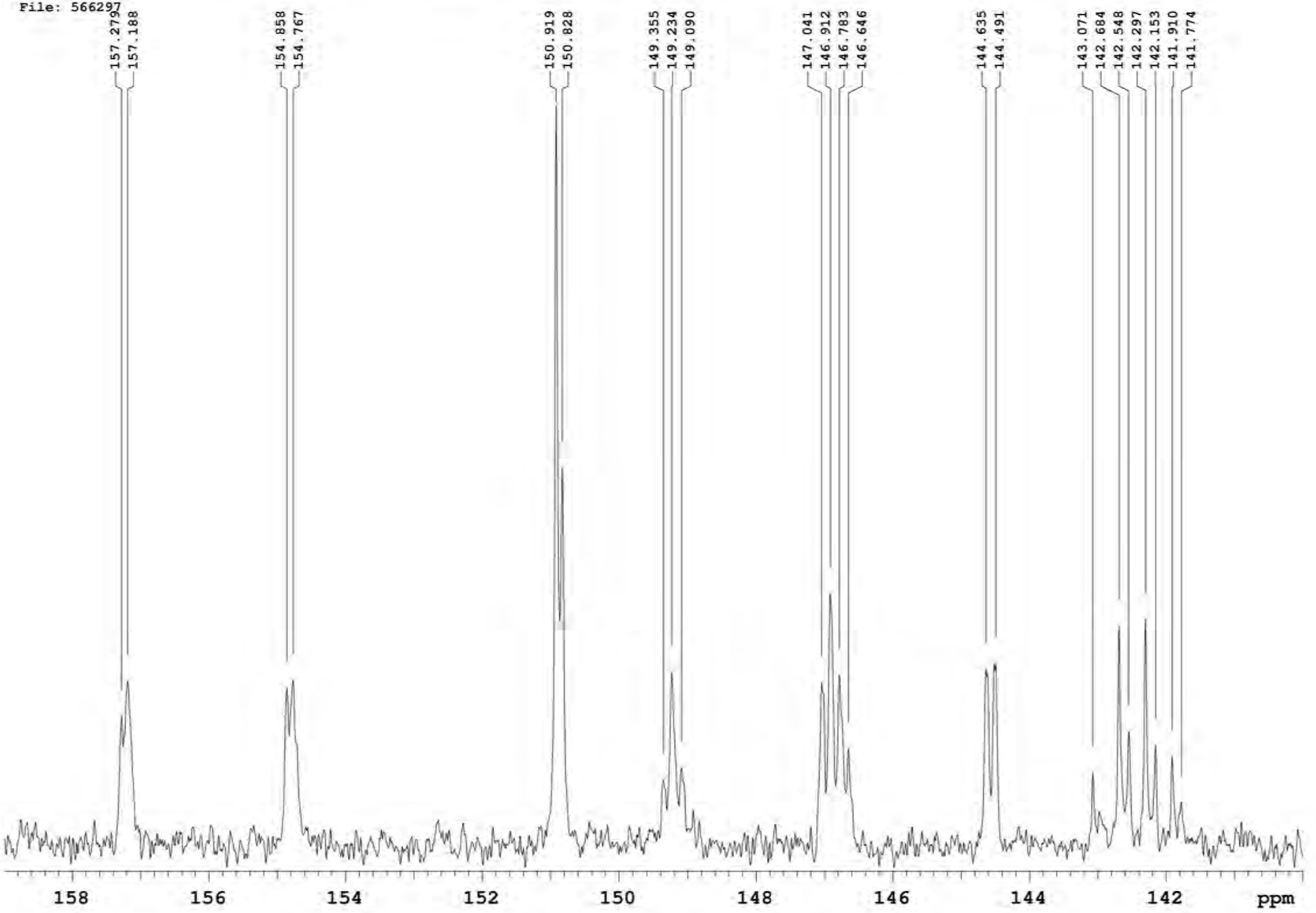
File: 566297



Plot file: 566297-2

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

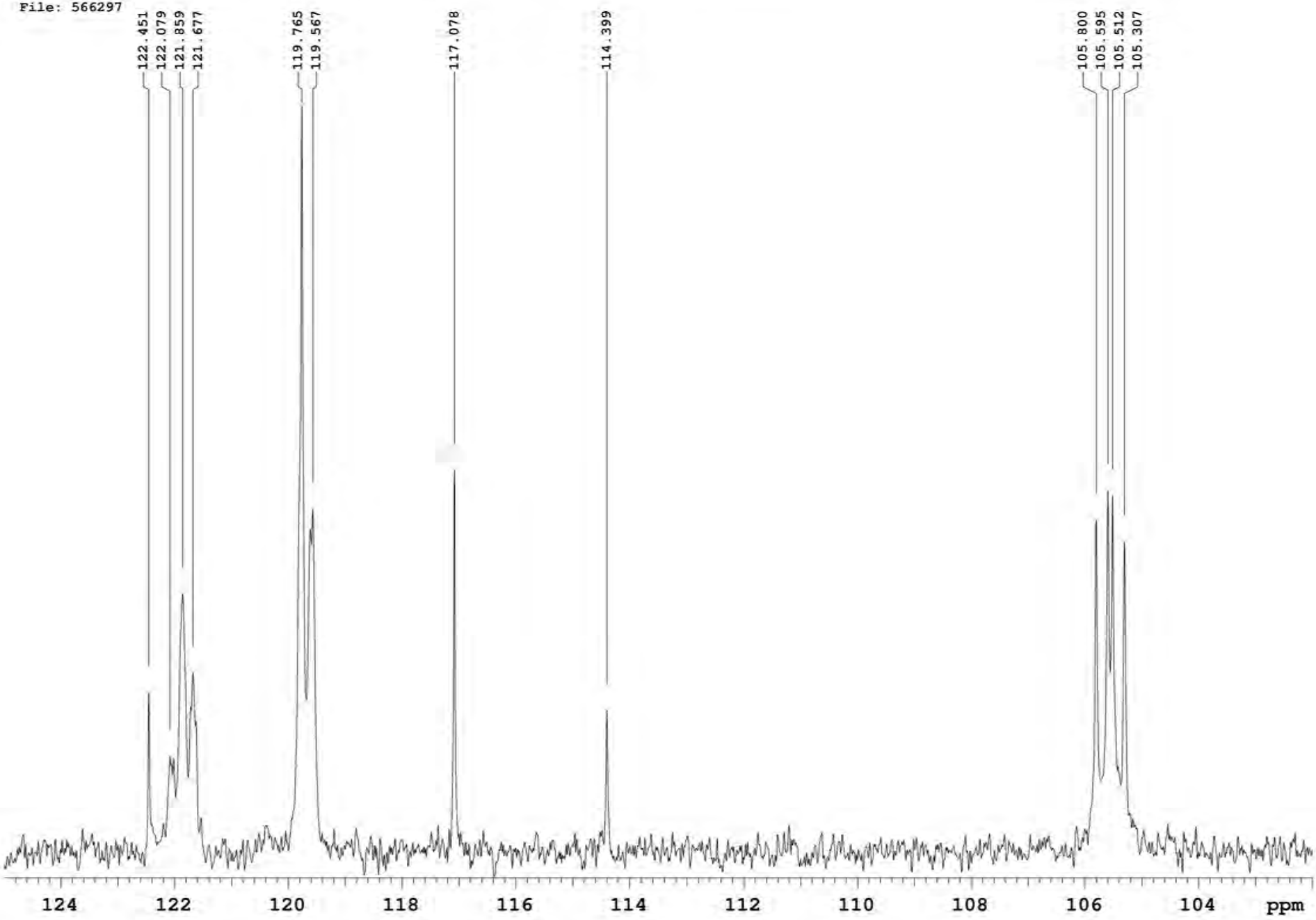
File: 566297



Plot file: 566297-3

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

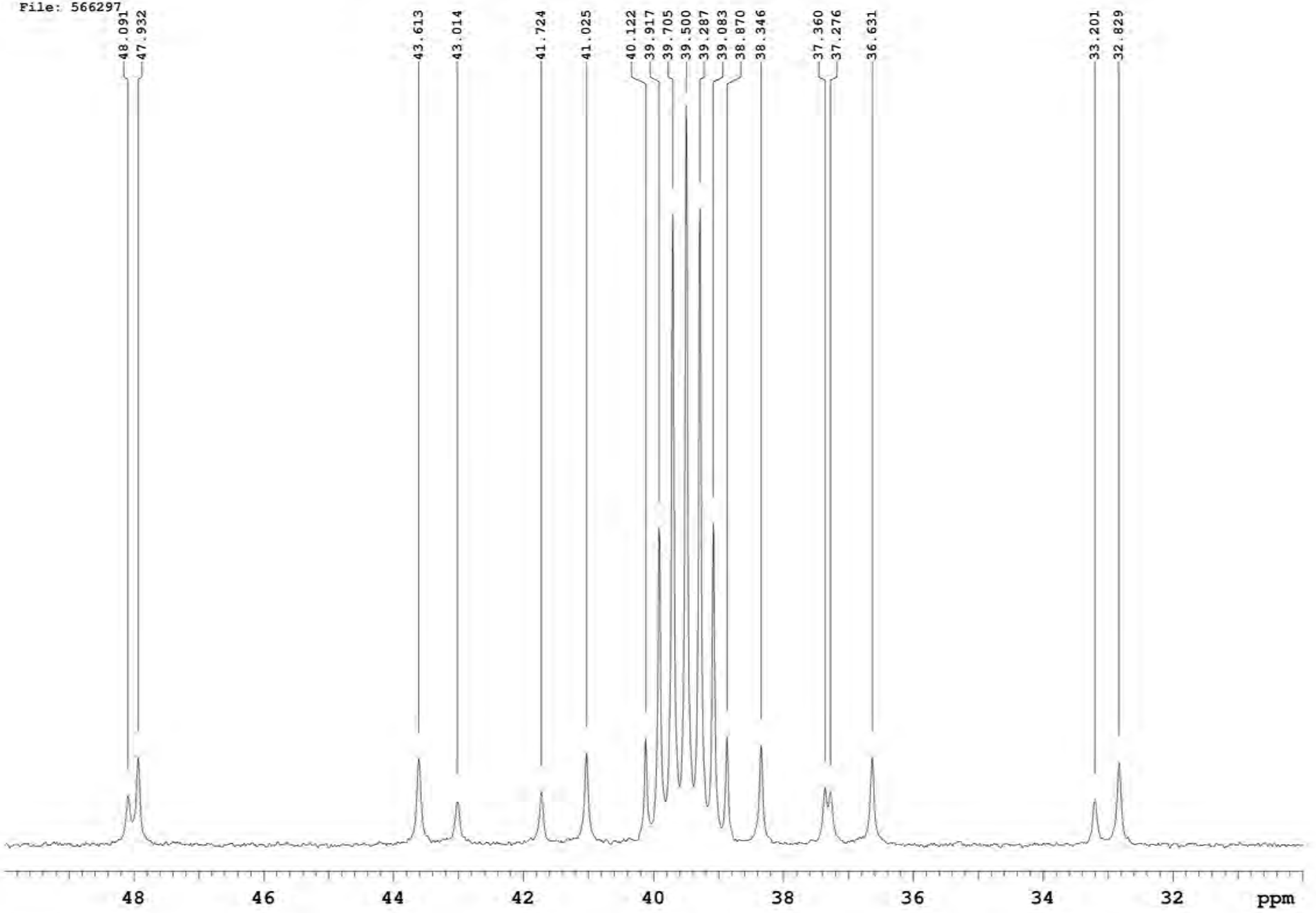
File: 566297



Plot file: 566297-4

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566297



Plot file: 566297-5

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566297

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 566297-1_peaks

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566297

INDEX	FREQUENCY	PPM	HEIGHT
1	17063.662	169.740	69.1
2	17046.115	169.566	141.8

Plot file: 566297-2_peaks

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566297

INDEX	FREQUENCY	PPM	HEIGHT
1	15810.916	157.279	24.7
2	15801.760	157.188	31.5
3	15567.538	154.858	30.1
4	15558.383	154.767	31.7
5	15171.572	150.919	141.8
6	15162.417	150.828	72.4
7	15014.407	149.355	12.5
8	15002.200	149.234	33.2
9	14987.704	149.090	14.8
10	14781.710	147.041	31.2
11	14768.740	146.912	48.2
12	14755.770	146.783	32.6
13	14742.037	146.646	18.5
14	14539.858	144.635	33.8
15	14525.363	144.491	34.9
16	14382.693	143.071	13.9
17	14343.783	142.684	42.1
18	14330.050	142.548	21.7
19	14304.873	142.297	43.3
20	14290.377	142.153	19.2
21	14265.963	141.910	17.0
22	14252.230	141.774	8.3

Plot file: 566297-3_peaks

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566297

INDEX	FREQUENCY	PPM	HEIGHT
1	12309.786	122.451	30.2
2	12272.402	122.079	18.1
3	12250.277	121.859	49.0
4	12231.967	121.677	34.1
5	12039.706	119.765	141.8
6	12019.869	119.567	65.2
7	11769.625	117.078	72.6
8	11500.308	114.399	26.9
9	10635.897	105.800	63.1
10	10615.298	105.595	68.7
11	10606.906	105.512	67.8
12	10586.306	105.307	58.9

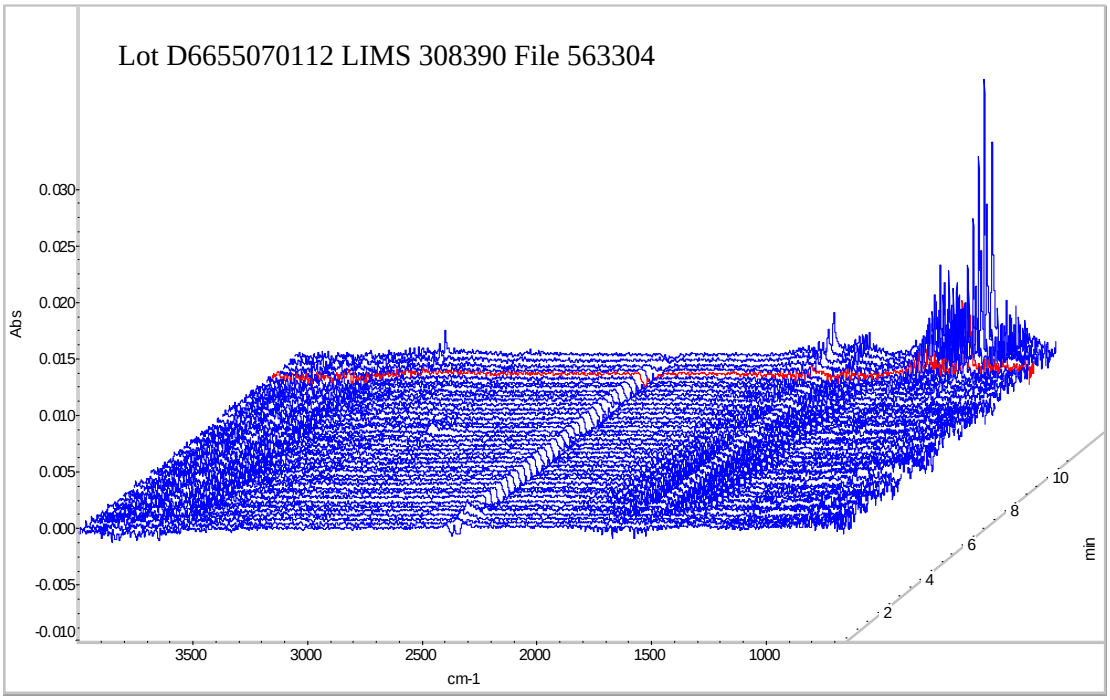
Plot file: 566297-4_peaks

315469, 5135-32-05, Compound 184, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566297

INDEX	FREQUENCY	PPM	HEIGHT
1	4834.506	48.091	9.5
2	4818.484	47.932	16.7
3	4384.371	43.613	16.5
4	4324.099	43.014	8.5
5	4194.399	41.724	10.2
6	4124.209	41.025	17.7
7	4033.419	40.122	20.5
8	4012.820	39.917	60.8
9	3991.458	39.705	120.9
10	3970.858	39.500	141.8
11	3949.496	39.287	122.0
12	3928.897	39.083	61.7
13	3907.534	38.870	20.7
14	3854.891	38.346	19.1
15	3755.709	37.360	11.1
16	3747.317	37.276	10.3
17	3682.467	36.631	16.8
18	3337.618	33.201	8.8
19	3300.234	32.829	15.9

Plot file: 566297-5_peaks


Lot D6655070112 LIMS 308390 File 563304
Abs
0.030
0.025
0.020
0.015
0.010
0.005
0.000
-0.005
-0.010
3500
3000
2500
2000
1500
1000
cm-1
2
4
6
8
10
min

COLLECTION AND PROCESSING INFORMATION

Title: 308390, D6655070112, Compound 184
Collected: Tue Dec 04 14:10:31 2012 (GMT-05:00)
Filename: I:\Aptuit Consulting\EL20100011\IR\563304.srs
Custom info 1:
Custom info 2:

DATA COLLECTION INFORMATION
Number of sample scans: 16
Sampling interval: 16.12 sec
Resolution: 4.000
Levels of zero filling: 0
Number of scan points: 8480
Number of FFT points: 8192
Laser frequency: 15798.3 cm-1
Interferogram peak position: 4096
Apodization: Happ-Genzel
Number of background scans: 16
Background gain: 2.0

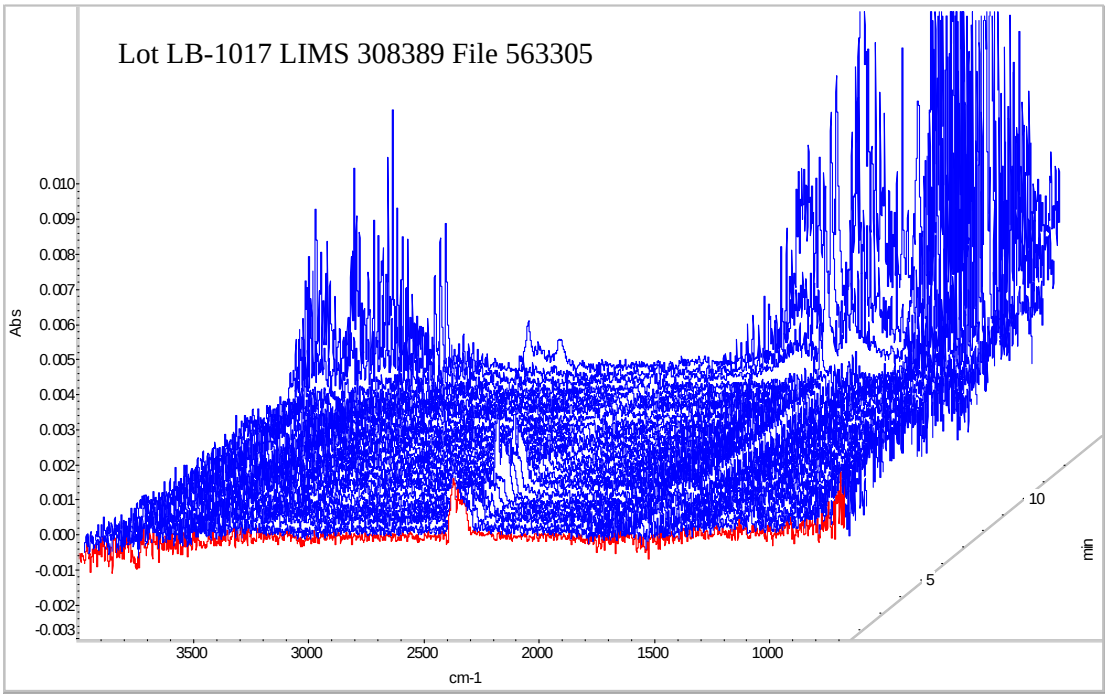
DATA DESCRIPTION
Number of points: 1738
X-axis: Wavenumbers (cm-1)
Y-axis: Absorbance
First X value: 649.9040
Last X value: 3999.7058
Data spacing: 1.928498

SPECTROMETER DESCRIPTION
Spectrometer: Magna System 560
Source: IR
Detector: MCT/A
Beamsplitter: KBr
Sample spacing: 2.0000
Digitizer bits: 20
Mirror velocity: 0.6329
Aperture: 100.00
Sample gain: 2.0
High pass filter: 200.0000
Low pass filter: 11000.0000

DATA PROCESSING HISTORY
Collect Series on Tue Dec 04 14:10:31 2012 (GMT-05:00)
Series title: 308390, D6655070112, Compound 184
Data collection type:TGA/IR
Total collection time: 12.63
Final format: Absorbance
Resolution: 4.000 from 649.9040 to 3999.7058

CURRENT DIGITAL SIGNATURE STATUS
Not currently signed.

SERIES DESCRIPTION
Minimum value: 0.2687
Maximum value: 12.6255
Step size: 0.2686
Number of spectra: 47
Gram-Schmidt offset: 10
Gram-Schmidt interferogram points: 100


Lot LB-1017 LIMS 308389 File 563305
Abs
0.010
0.009
0.008
0.007
0.006
0.005
0.004
0.003
0.002
0.001
0.000
-0.001
-0.002
-0.003
3500
3000
2500
2000
1500
1000
cm-1
5
10
min

COLLECTION AND PROCESSING INFORMATION

Title: 308389, LB-1017, Compound 184
Collected: Tue Dec 04 13:18:39 2012 (GMT-05:00)
Filename: I:\Aptuit Consulting\EL20100011\IR\563305.srs
Custom info 1:
Custom info 2:

DATA COLLECTION INFORMATION
Number of sample scans: 16
Sampling interval: 16.12 sec
Resolution: 4.000
Levels of zero filling: 0
Number of scan points: 8480
Number of FFT points: 8192
Laser frequency: 15798.3 cm-1
Interferogram peak position: 4096
Apodization: Happ-Genzel
Number of background scans: 16
Background gain: 2.0

DATA DESCRIPTION
Number of points: 1738
X-axis: Wavenumbers (cm-1)
Y-axis: Absorbance
First X value: 649.9040
Last X value: 3999.7058
Data spacing: 1.928498

SPECTROMETER DESCRIPTION
Spectrometer: Magna System 560
Source: IR
Detector: MCT/A
Beamsplitter: KBr
Sample spacing: 2.0000
Digitizer bits: 20
Mirror velocity: 0.6329
Aperture: 100.00
Sample gain: 2.0
High pass filter: 200.0000
Low pass filter: 11000.0000

DATA PROCESSING HISTORY
Collect Series on Tue Dec 04 13:18:39 2012 (GMT-05:00)
Series title: 308389, LB-1017, Compound 184
Data collection type:TGA/IR
Total collection time: 12.63
Final format: Absorbance
Resolution: 4.000 from 649.9040 to 3999.7058

CURRENT DIGITAL SIGNATURE STATUS
Not currently signed.

SERIES DESCRIPTION
Minimum value: 0.2687
Maximum value: 12.6273
Step size: 0.2687
Number of spectra: 47
Gram-Schmidt offset: 10
Gram-Schmidt interferogram points: 100

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 1:48 PM**

Sample data

No.	Note / ID	Start time	Sample size
1	5135-02-01, Compound 184, p2	12/13/2012 1:48 PM	-18251776.000000 g

Results

No.	Note / ID	Start time	Sample size and results
1	5135-02-01, Compound 184, p2	12/13/2012 1:48 PM	-18251776.0 g R1 = 0.000 %

Series note

Raw data

Sample

No.	1			
Identification				
Note	5135-02-01, Compound 184, p2			
Titration stand	Internal stand			
Mass	m = -18251776.000000 g			
Stirrer speed	30 %			
Mix time	120 s			
Blank	BLANK = 67 µg			
Drift	DRIFT = 5 µg/min			
Temperature (Stromboli)	170 oC			

KF determination

Consumption	EP	CEQ1 =	12916.528 mC	
		Q1 =	1204.90 µg water	
Duration		TIME =	252 s	(2517 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 0.000 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:00:50 PM**

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 2:03 PM**

Sample data

No.	Note / ID	Start time	Sample size
1	5135-02-01, Compound 184, p2		
	314339	12/13/2012 2:03 PM	0.0566 g

Results

No.	Note / ID	Start time	Sample size and results		
1	5135-02-01, Compound 184, p2				
	314339	12/13/2012 2:03 PM		0.0566	g
			R1 =	3.035	%

Series note

Raw data

Sample

No.	1
Identification	314339
Note	5135-02-01, Compound 184, p2
Titration stand	Internal stand
Mass	m = 0.0566 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 67 µg
Drift	DRIFT = 5 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	19377.150 mC	
		Q1 =	1807.57 µg water	
Duration		TIME =	274 s	(2740 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 3.035 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:07:42 PM**

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 2:03 PM**

Sample data

No.	Note / ID	Start time	Sample size
2	5135-15-05, Compound 184, p3		
	314760	12/13/2012 2:09 PM	0.0310 g

Results

No.	Note / ID	Start time	Sample size and results		
2	5135-15-05, Compound 184, p3				
	314760	12/13/2012 2:09 PM		0.0310	g
			R1 =	6.407	%

Series note

Raw data

Sample

No.	2
Identification	314760
Note	5135-15-05, Compound 184, p3
Titration stand	Internal stand
Mass	m = 0.0310 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 67 µg
Drift	DRIFT = 5 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	22259.008 mC	
		Q1 =	2076.40 µg water	
Duration		TIME =	280 s	(2802 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 6.407 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:07:42 PM**

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 2:03 PM**

Sample data

No.	Note / ID	Start time	Sample size
3	5135-15-05, Compound 184, p4		
	314760	12/13/2012 2:14 PM	0.0300 g

Results

No.	Note / ID	Start time	Sample size and results		
3	5135-15-05, Compound 184, p4				
	314760	12/13/2012 2:14 PM		0.0300	g
			R1 =	6.267	%

Series note

Raw data

Sample

No.	3
Identification	314760
Note	5135-15-05, Compound 184, p4
Titration stand	Internal stand
Mass	m = 0.0300 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 67 µg
Drift	DRIFT = 5 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	21110.146 mC	
		Q1 =	1969.23 µg water	
Duration		TIME =	267 s	(2669 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 6.267 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:07:42 PM**

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 2:03 PM**

Sample data

No.	Note / ID	Start time	Sample size
4	5135-17-01, Compound 184, p5		
	314783	12/13/2012 2:19 PM	0.0319 g

Results

No.	Note / ID	Start time	Sample size and results		
4	5135-17-01, Compound 184, p5				
	314783	12/13/2012 2:19 PM		0.0319	g
			R1 =	6.382	%

Series note

Raw data

Sample

No.	4
Identification	314783
Note	5135-17-01, Compound 184, p5
Titration stand	Internal stand
Mass	m = 0.0319 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 67 µg
Drift	DRIFT = 5 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	22787.718 mC	
		Q1 =	2125.72 µg water	
Duration		TIME =	276 s	(2756 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 6.382 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:07:42 PM**

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 2:03 PM**

Sample data

No.	Note / ID	Start time	Sample size
5	5135-17-01, Compound 184, p6		
	314783	12/13/2012 2:24 PM	0.0311 g

Results

No.	Note / ID	Start time	Sample size and results		
5	5135-17-01, Compound 184, p6				
	314783	12/13/2012 2:24 PM		0.0311	g
			R1 =	6.430	%

Series note

Raw data

Sample

No.	5
Identification	314783
Note	5135-17-01, Compound 184, p6
Titration stand	Internal stand
Mass	m = 0.0311 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 67 µg
Drift	DRIFT = 5 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	22396.760 mC	
		Q1 =	2089.25 µg water	
Duration		TIME =	272 s	(2719 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 6.430 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:07:42 PM**

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 1:48 PM**

Sample data

No.	Note / ID	Start time	Sample size
1	5135-02-01, Compound 184, p2	12/13/2012 1:48 PM	*0.0358 g

Results

No.	Note / ID	Start time	Sample size and results		
1	5135-02-01, Compound 184, p2				
		12/13/2012 1:48 PM		*0.0358	g
			R1 =	*3.120	%

Series note

Statistics

Rx	Name	n	Mean value	Unit	s	srel [%]
R1		1	*3.120	%		

Raw data

Sample

No.	1
Identification	
Note	5135-02-01, Compound 184, p2
Titration stand	Internal stand
Mass	m = *0.0358 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 67 µg
Drift	DRIFT = 5 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	12916.528 mC	
		Q1 =	1204.90 µg water	
Duration		TIME =	252 s	(2517 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = *3.120 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:00:50 PM**
Last modified: **Aaron Atkinson (ATKINSON), 12/13/2012 2:23:38 PM**

Method: **108** **Stromboli** **11/12/2012 11:02 AM**
Start time: **12/13/2012 2:03 PM**

Sample data

No.	Note / ID	Start time	Sample size
1	5135-02-01, Compound 184, p2		
	314339	12/13/2012 2:03 PM	0.0566 g

Results

No.	Note / ID	Start time	Sample size and results		
1	5135-02-01, Compound 184, p2				
	314339	12/13/2012 2:03 PM		0.0566	g
			R1 =	3.035	%

Series note

Raw data

Sample

No.	1
Identification	314339
Note	5135-02-01, Compound 184, p2
Titration stand	Internal stand
Mass	m = 0.0566 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 67 µg
Drift	DRIFT = 5 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	19377.150 mC	
		Q1 =	1807.57 µg water	
Duration		TIME =	274 s	(2740 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 3.035 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/13/2012 2:07:42 PM**

Method: **108** **Stromboli** **12/21/2012 11:56 AM**
Start time: **12/21/2012 11:58 AM**

Sample data

No.	Note / ID	Start time	Sample size
1	Compound 184, 5135-30-03, p1		
	315535	12/21/2012 11:58 AM	0.0122 g

Results

No.	Note / ID	Start time	Sample size and results		
1	Compound 184, 5135-30-03, p1				
	315535	12/21/2012 11:58 AM		0.0122	g
			R1 =	6.383	%

Series note

Raw data

Sample

No.	1
Identification	315535
Note	Compound 184, 5135-30-03, p1
Titration stand	Internal stand
Mass	m = 0.0122 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 104 µg
Drift	DRIFT = 3 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	9605.763 mC	
		Q1 =	896.06 µg water	
Duration		TIME =	267 s	(2669 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 6.383 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/21/2012 12:03:17 PM**

Method: **108** **Stromboli** **12/21/2012 11:56 AM**
Start time: **12/21/2012 11:58 AM**

Sample data

No.	Note / ID	Start time	Sample size
2	Compound 184, 5135-30-03, p2		
	315535	12/21/2012 12:03 PM	0.0104 g

Results

No.	Note / ID	Start time	Sample size and results		
2	Compound 184, 5135-30-03, p2				
	315535	12/21/2012 12:03 PM		0.0104	g
			R1 =	6.919	%

Series note

Raw data

Sample

No.	2
Identification	315535
Note	Compound 184, 5135-30-03, p2
Titration stand	Internal stand
Mass	m = 0.0104 g
Stirrer speed	30 %
Mix time	120 s
Blank	BLANK = 104 µg
Drift	DRIFT = 3 µg/min
Temperature (Stromboli)	170 oC

KF determination

Consumption	EP	CEQ1 =	8959.454 mC	
		Q1 =	835.77 µg water	
Duration		TIME =	245 s	(2445 [0.1s])
Termination condition	Delay			

Calculation

Result	R1 = 6.919 %
Formula	R1 = (f1/(C1*m))*(Q-(COMP+BLANK))
Constant	C1 = 10000
Factor	f1 = 1.0000

Created: **Aaron Atkinson (ATKINSON), 12/21/2012 12:03:17 PM**

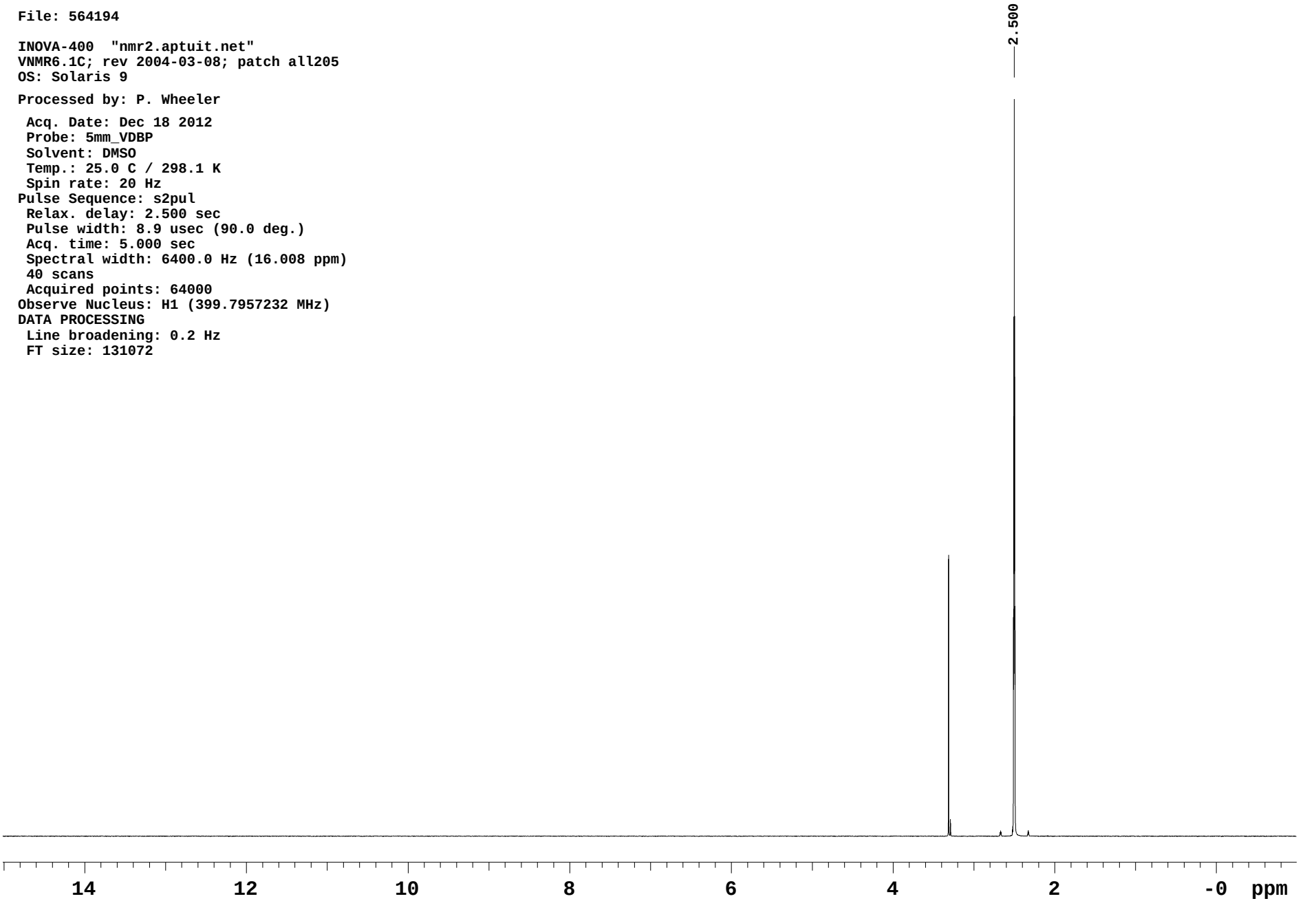
314549, Dimethyl Sulfoxide-D6, Lot 12I-403, as-received, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C
Solvent Certification

File: 564194

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

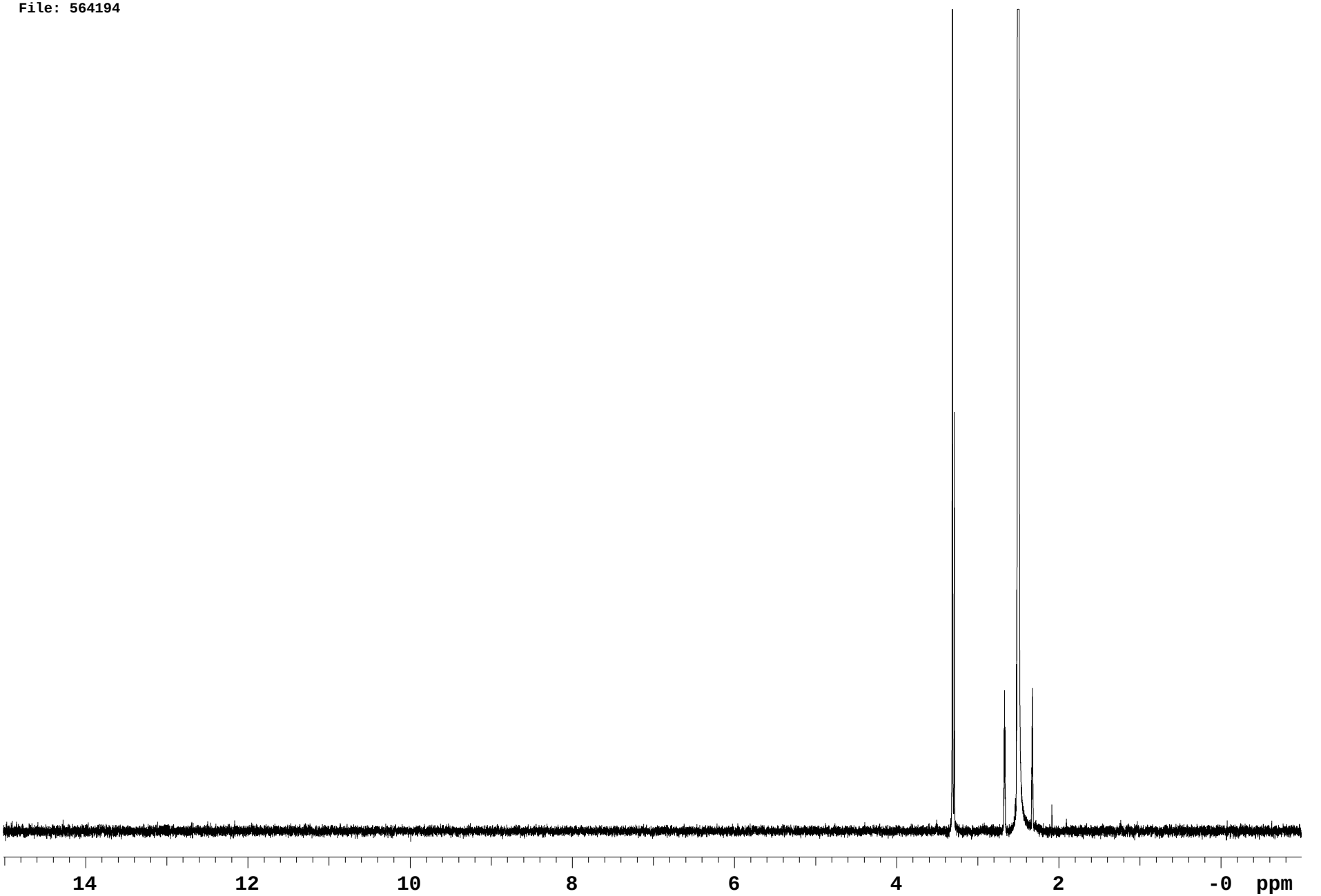
Acq. Date: Dec 18 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 2.500 sec
Pulse width: 8.9 usec (90.0 deg.)
Acq. time: 5.000 sec
Spectral width: 6400.0 Hz (16.008 ppm)
40 scans
Acquired points: 64000
Observe Nucleus: H1 (399.7957232 MHz)
DATA PROCESSING
Line broadening: 0.2 Hz
FT size: 131072



Plot file: 564194-1

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, as-received, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C
Solvent Certification

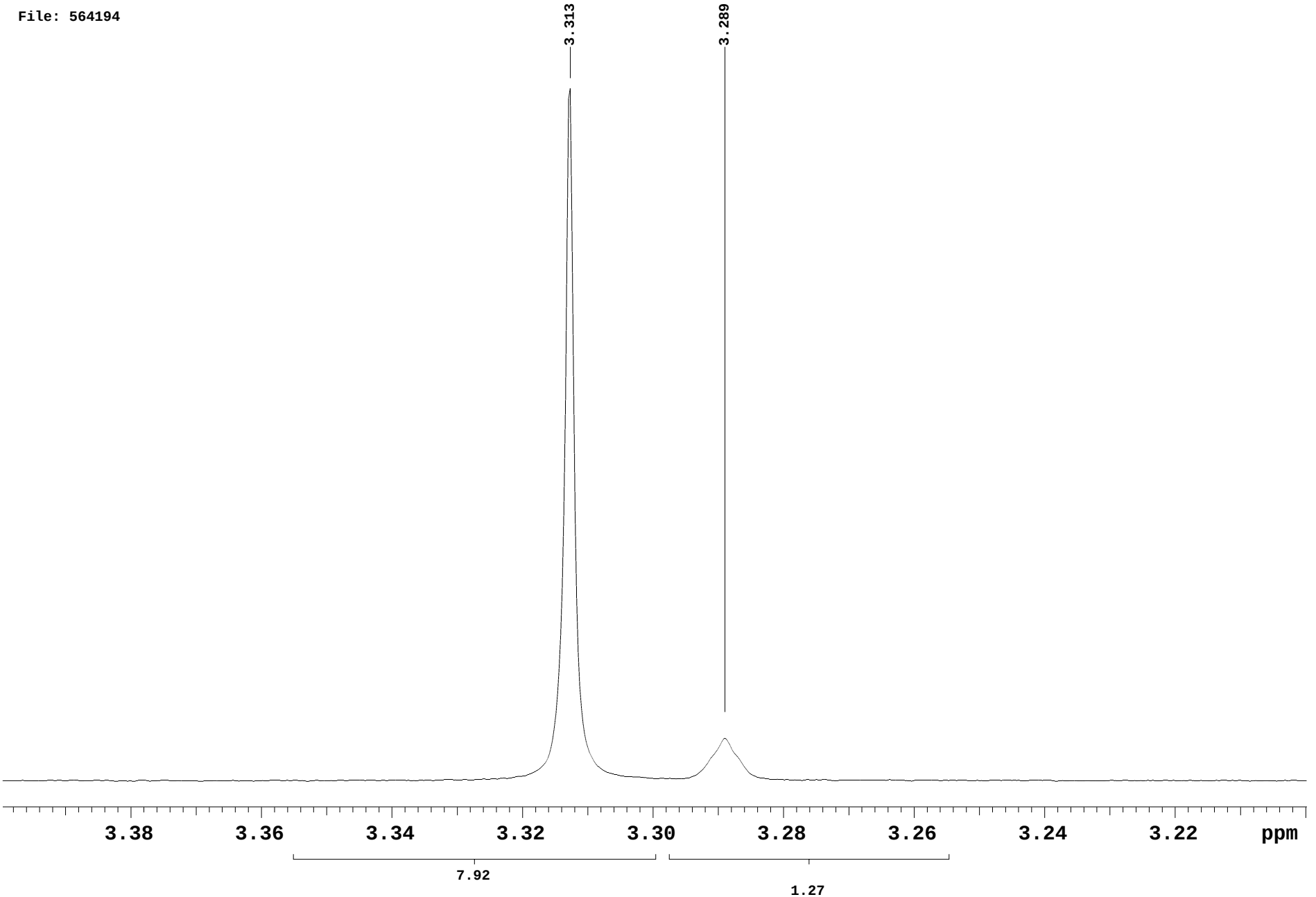
File: 564194



Plot file: 564194-2

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, as-received, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C
Solvent Certification

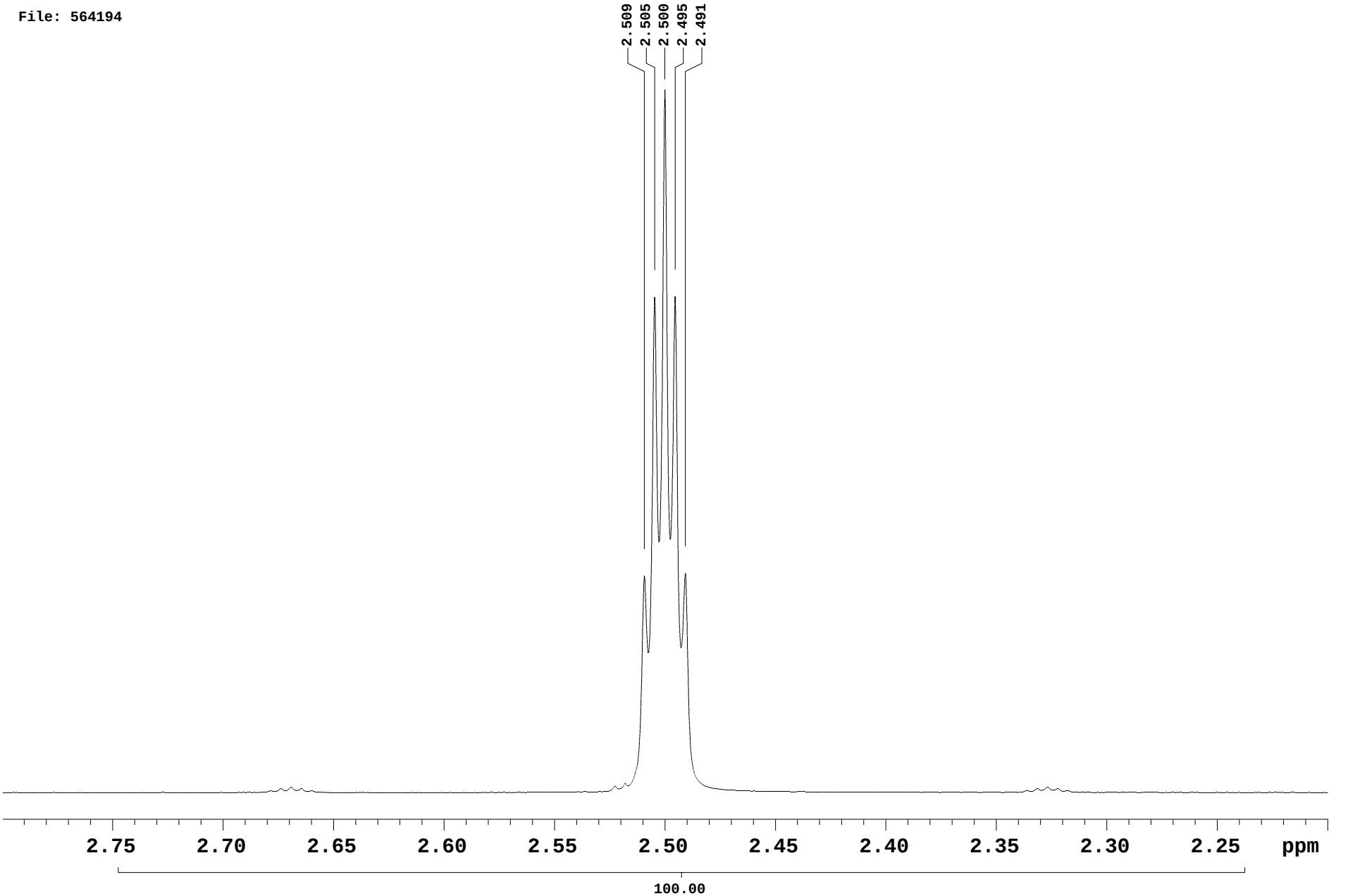
File: 564194



Plot file: 564194-3

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, as-received, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C
Solvent Certification

File: 564194



Plot file: 564194-4

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, as-received, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C
Solvent Certification

File: 564194

INDEX	FREQUENCY	PPM	HEIGHT
1	999.482	2.500	141.8

Plot file: 564194-1_peaks

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, as-received, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C
Solvent Certification

File: 564194

INDEX	FREQUENCY	PPM	HEIGHT
1	1324.385	3.313	132.0
2	1314.912	3.289	8.1

Plot file: 564194-3_peaks

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, as-received, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C
Solvent Certification

File: 564194

INDEX	FREQUENCY	PPM	HEIGHT
1	1003.193	2.509	40.7
2	1001.338	2.505	93.1
3	999.482	2.500	132.0
4	997.627	2.495	93.2
5	995.771	2.491	41.2

Plot file: 564194-4_peaks

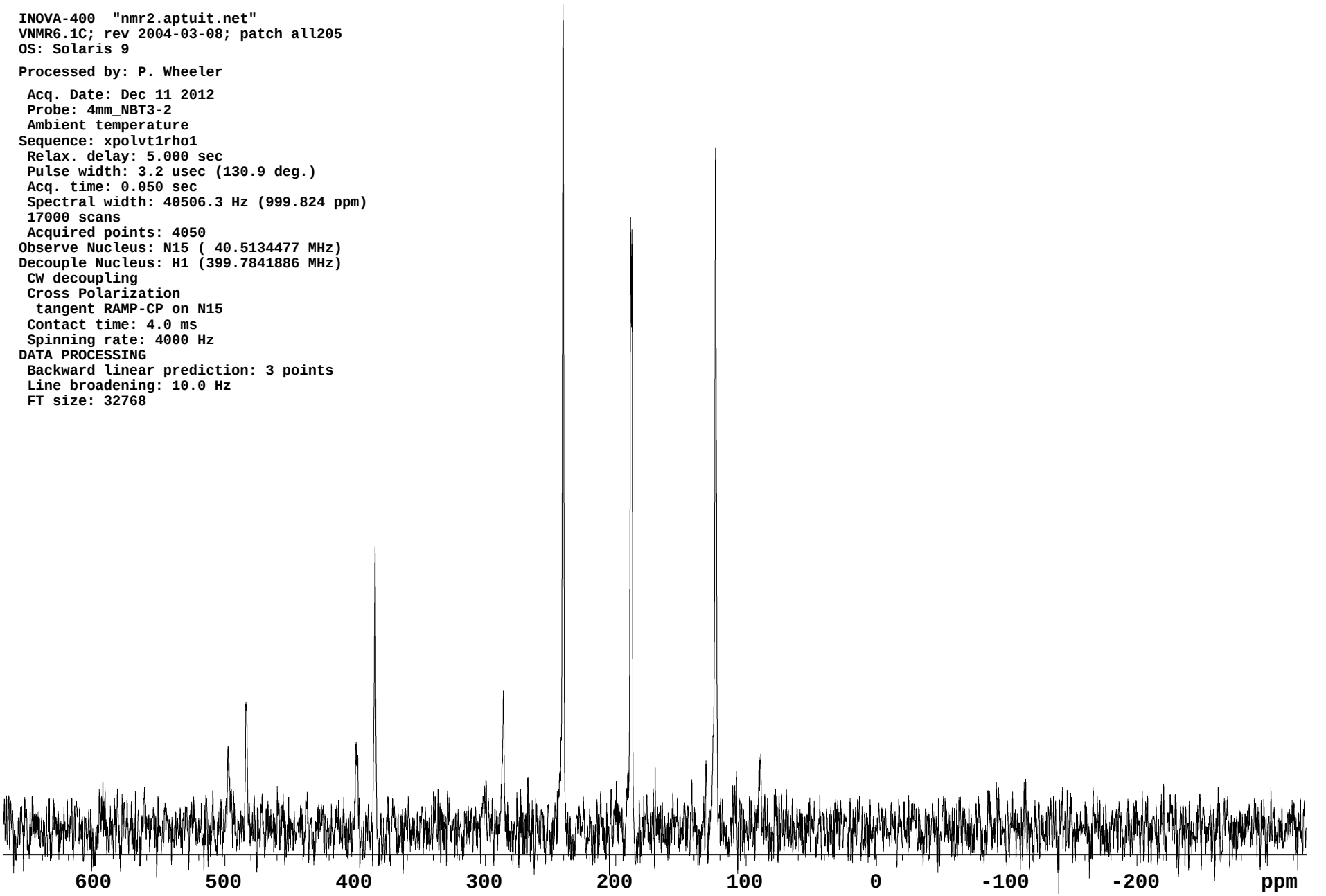
308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm
4 co-added FIDs

File: 564392

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

 Acq. Date: Dec 11 2012
 Probe: 4mm_NBT3-2
 Ambient temperature
Sequence: xpolvt1rho1
 Relax. delay: 5.000 sec
 Pulse width: 3.2 usec (130.9 deg.)
 Acq. time: 0.050 sec
 Spectral width: 40506.3 Hz (999.824 ppm)
 17000 scans
 Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
 CW decoupling
 Cross Polarization
  tangent RAMP-CP on N15
 Contact time: 4.0 ms
 Spinning rate: 4000 Hz
DATA PROCESSING
 Backward linear prediction: 3 points
 Line broadening: 10.0 Hz
 FT size: 32768



Plot file: 564392-1

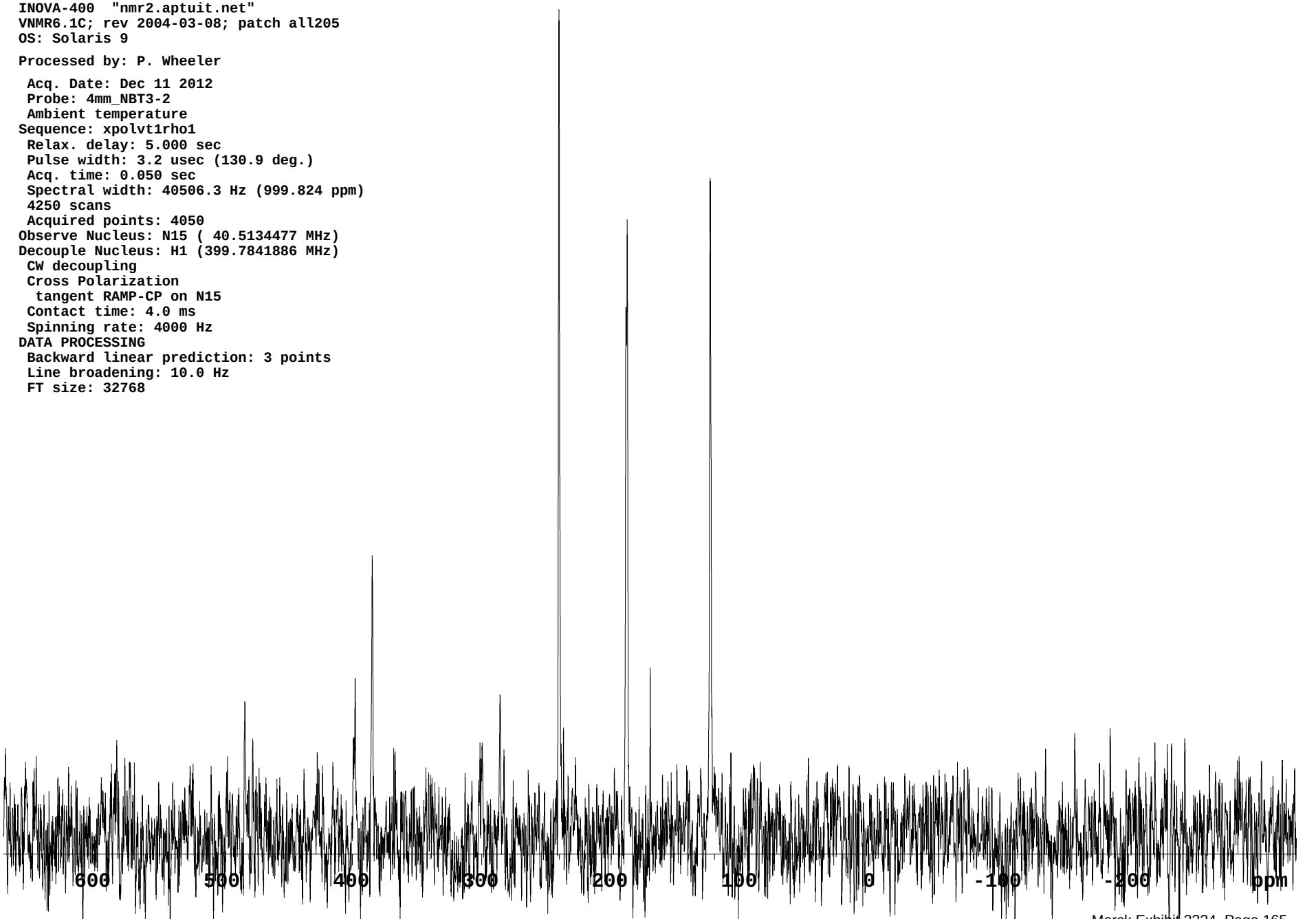
308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-1

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

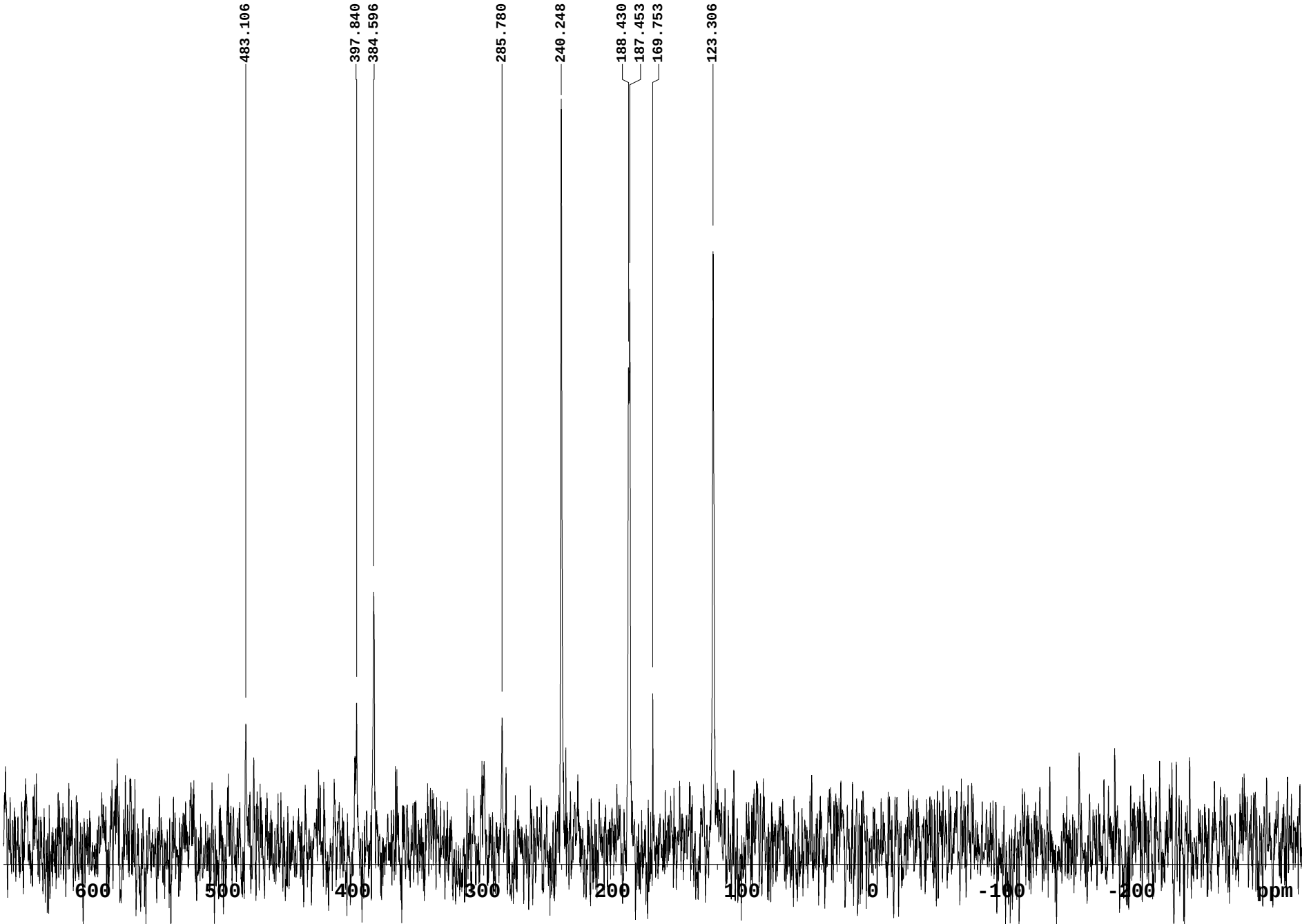
Acq. Date: Dec 11 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564392-1a

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

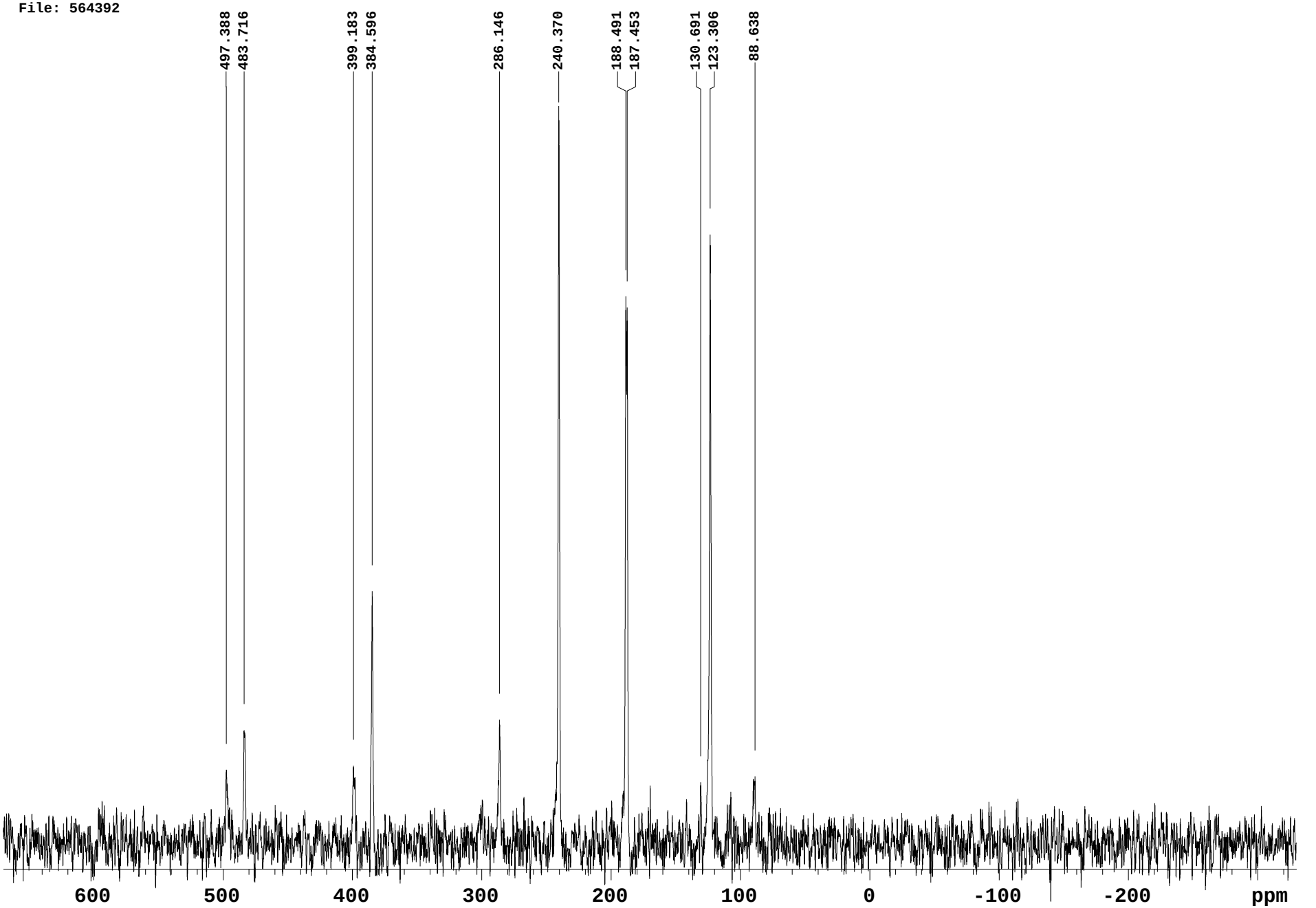
File: 564392-1



Plot file: 564392-1b

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm
4 co-added FIDs

File: 564392



Plot file: 564392-2

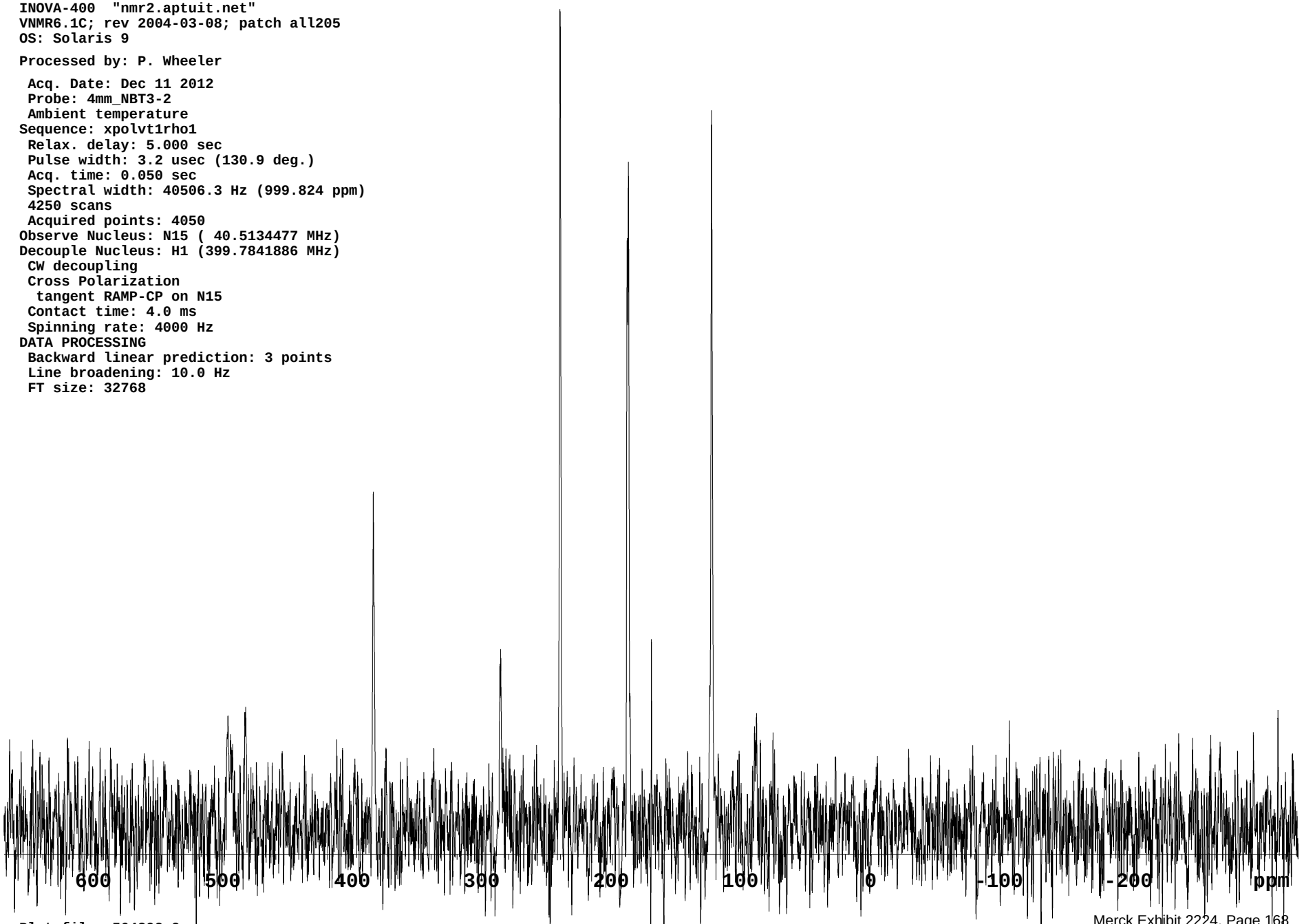
308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-2

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

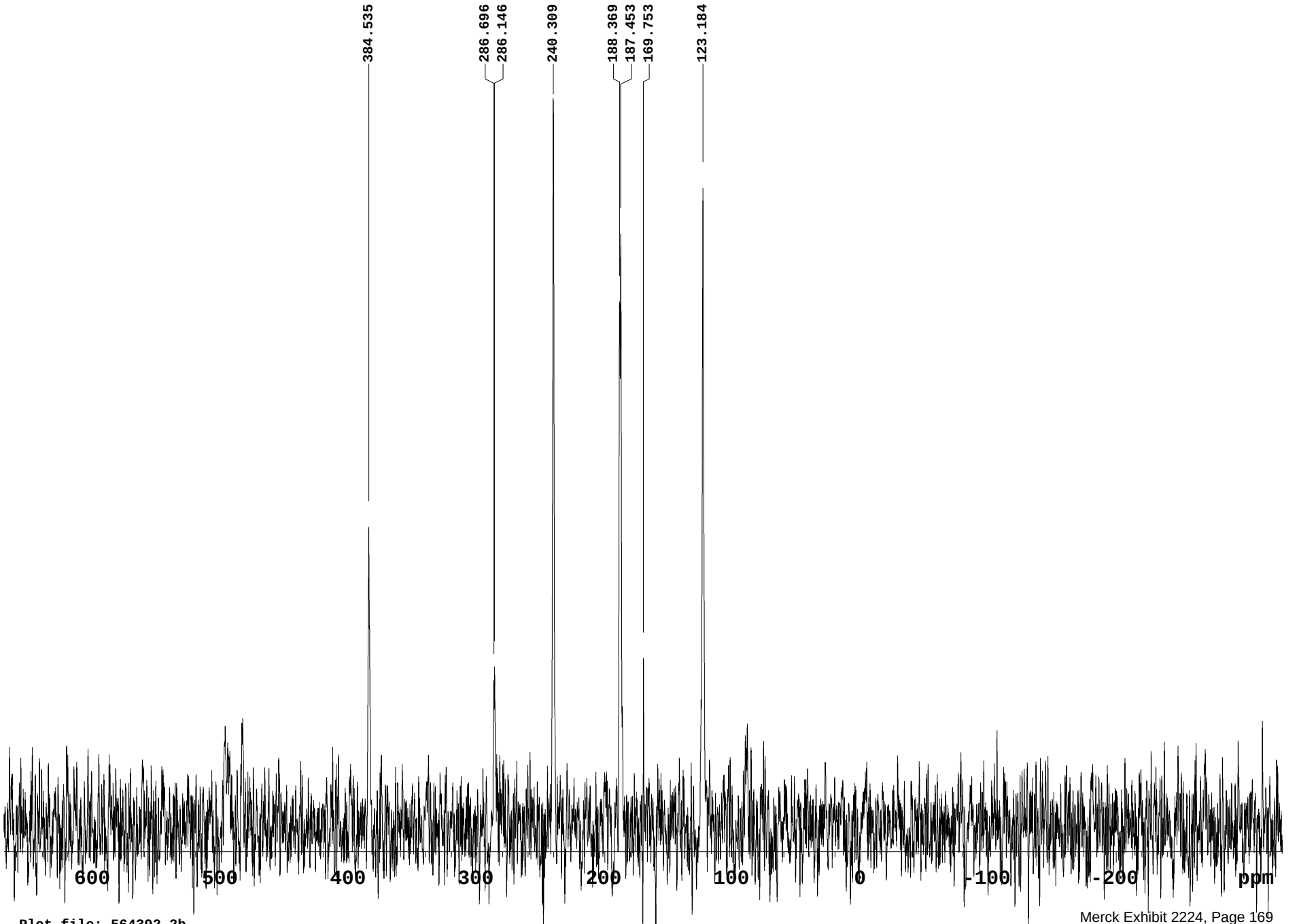
Acq. Date: Dec 11 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564392-2a

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-2



Plot file: 564392-2b

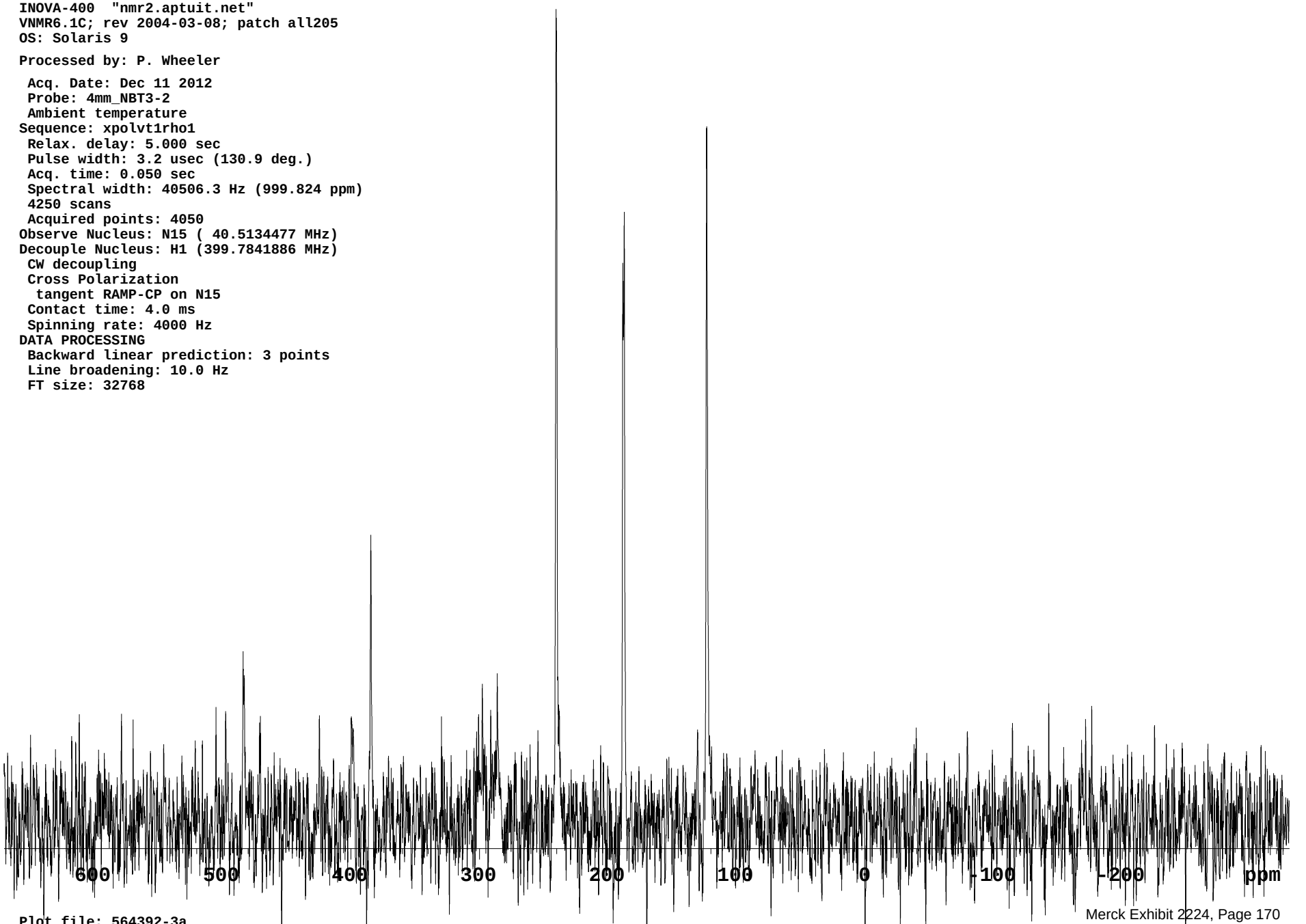
308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-3

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

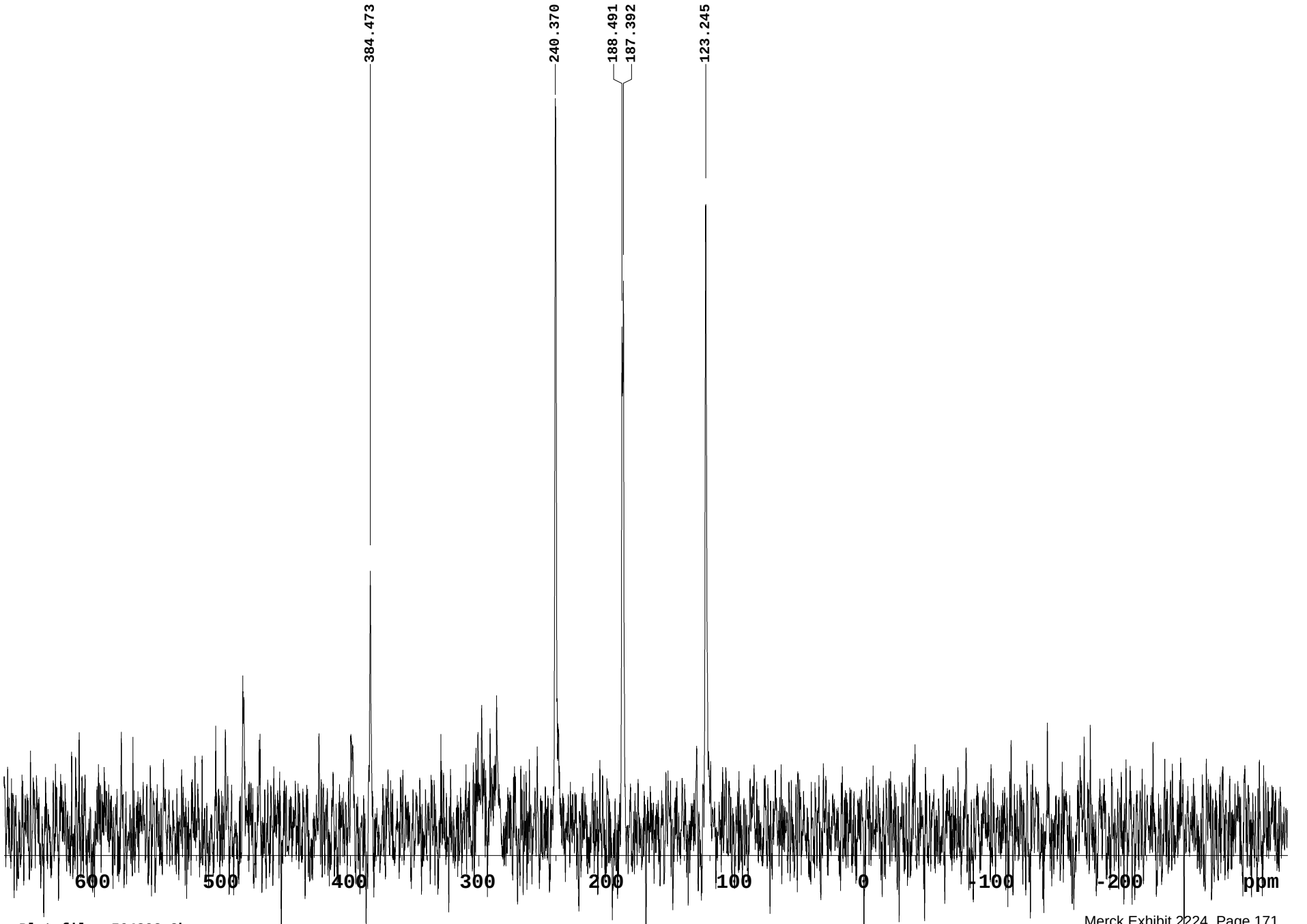
Acq. Date: Dec 11 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564392-3a

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-3



Plot file: 564392-3b

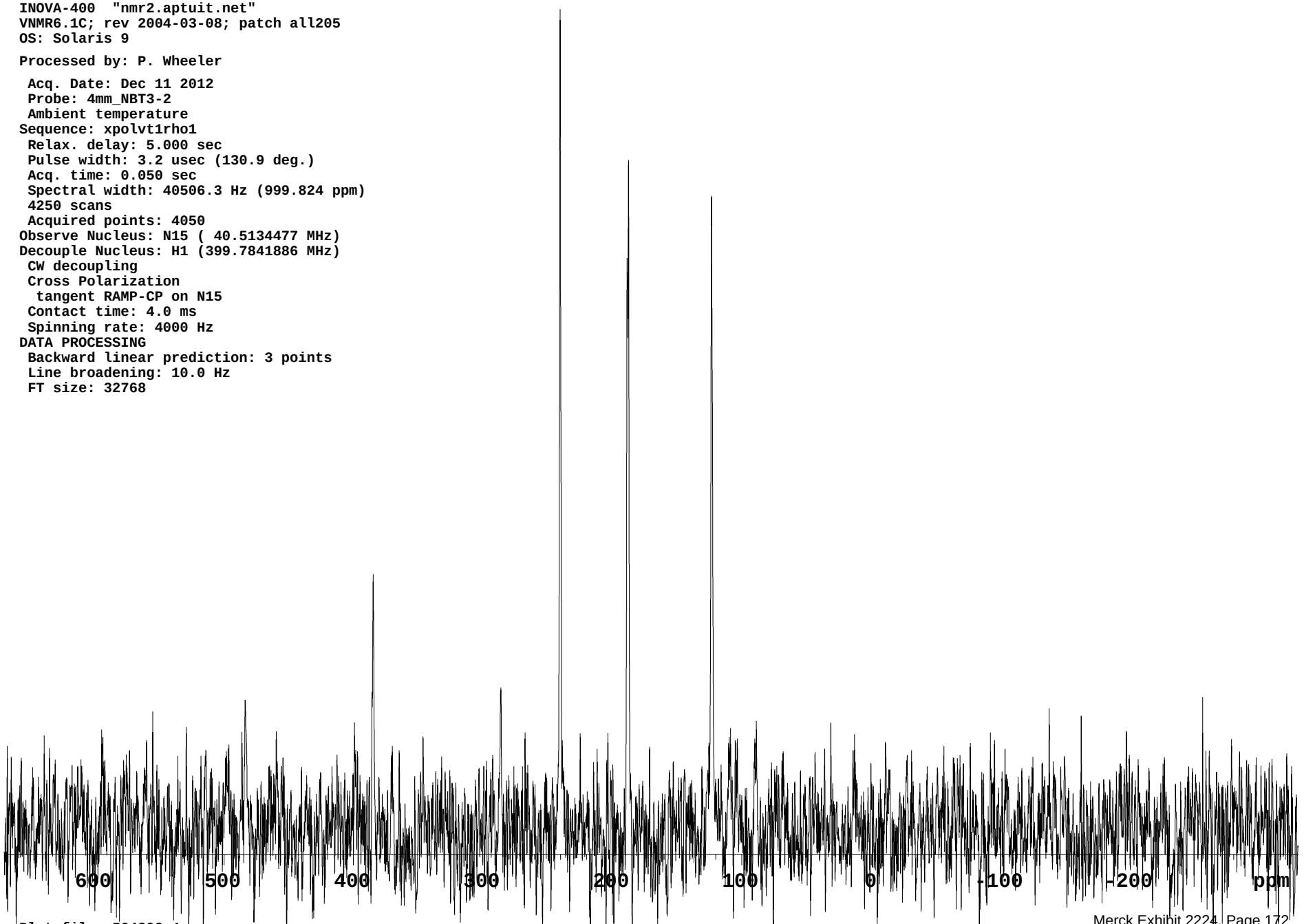
308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-4

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

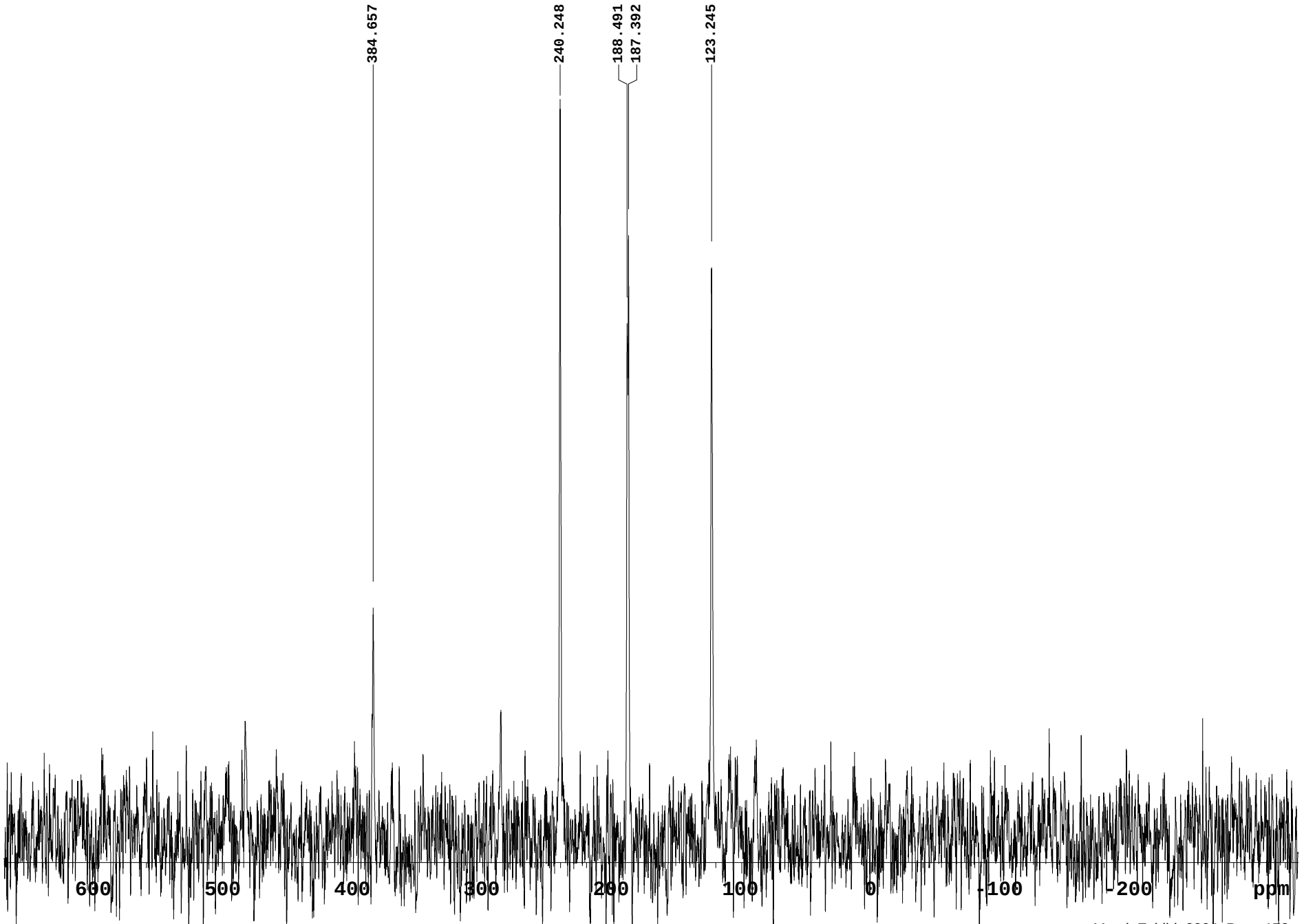
Acq. Date: Dec 11 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564392-4a

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-4



Plot file: 564392-4b

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-1

INDEX	FREQUENCY	PPM	HEIGHT
1	19568.954	483.106	22.0
2	16115.137	397.840	26.0
3	15578.646	384.596	47.2
4	11575.976	285.780	23.1
5	9731.632	240.248	141.8
6	7632.641	188.430	90.2
7	7593.084	187.453	105.3
8	6876.114	169.753	27.8
9	4994.686	123.306	112.5

Plot file: 564392-1b_peaks

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm
4 co-added FIDs

File: 564392

INDEX	FREQUENCY	PPM	HEIGHT
1	20147.475	497.388	14.1
2	19593.677	483.716	21.8
3	16169.528	399.183	14.9
4	15578.646	384.596	48.5
5	11590.809	286.146	23.8
6	9736.577	240.370	141.8
7	7635.113	188.491	105.2
8	7593.084	187.453	103.0
9	5293.835	130.691	11.7
10	4994.686	123.306	117.0
11	3590.414	88.638	12.9

Plot file: 564392-2_peaks

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-2

INDEX	FREQUENCY	PPM	HEIGHT
1	15576.173	384.535	58.2
2	11613.060	286.696	28.5
3	11590.809	286.146	31.0
4	9734.104	240.309	141.8
5	7630.169	188.369	102.1
6	7593.084	187.453	115.3
7	6876.114	169.753	32.7
8	4989.741	123.184	124.2

Plot file: 564392-2b_peaks

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-3

INDEX	FREQUENCY	PPM	HEIGHT
1	15573.701	384.473	50.2
2	9736.577	240.370	141.8
3	7635.113	188.491	97.5
4	7590.612	187.392	106.4
5	4992.214	123.245	121.3

Plot file: 564392-3b_peaks

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564392-4

INDEX	FREQUENCY	PPM	HEIGHT
1	15581.118	384.657	44.0
2	9731.632	240.248	141.8
3	7635.113	188.491	98.6
4	7590.612	187.392	115.6
5	4992.214	123.245	109.4

Plot file: 564392-4b_peaks

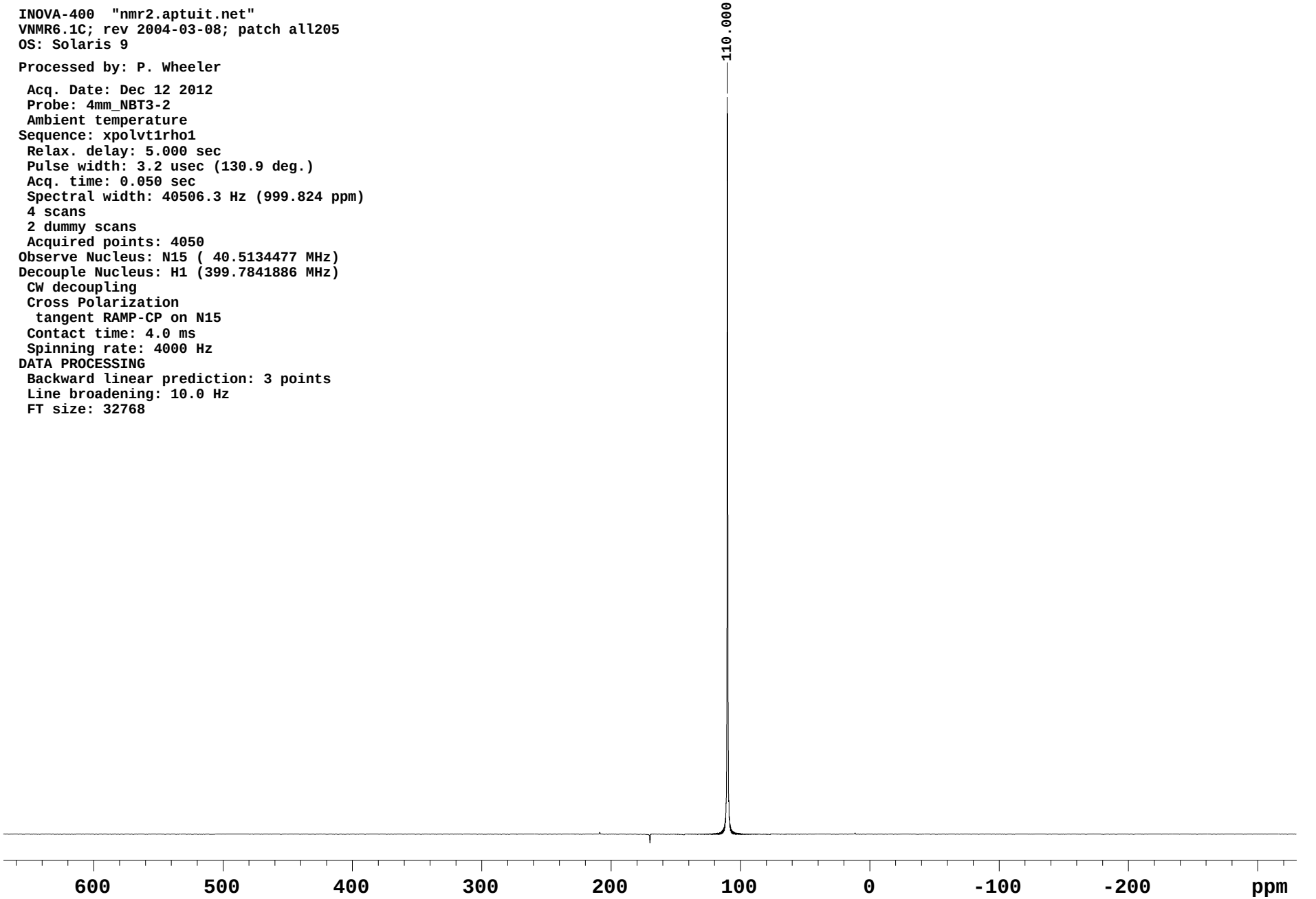
72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17

File: 564393

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564393-1

72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17

File: 564393

INDEX	FREQUENCY	PPM	HEIGHT
1	4455.722	110.000	141.8

Plot file: 564393-1_peaks

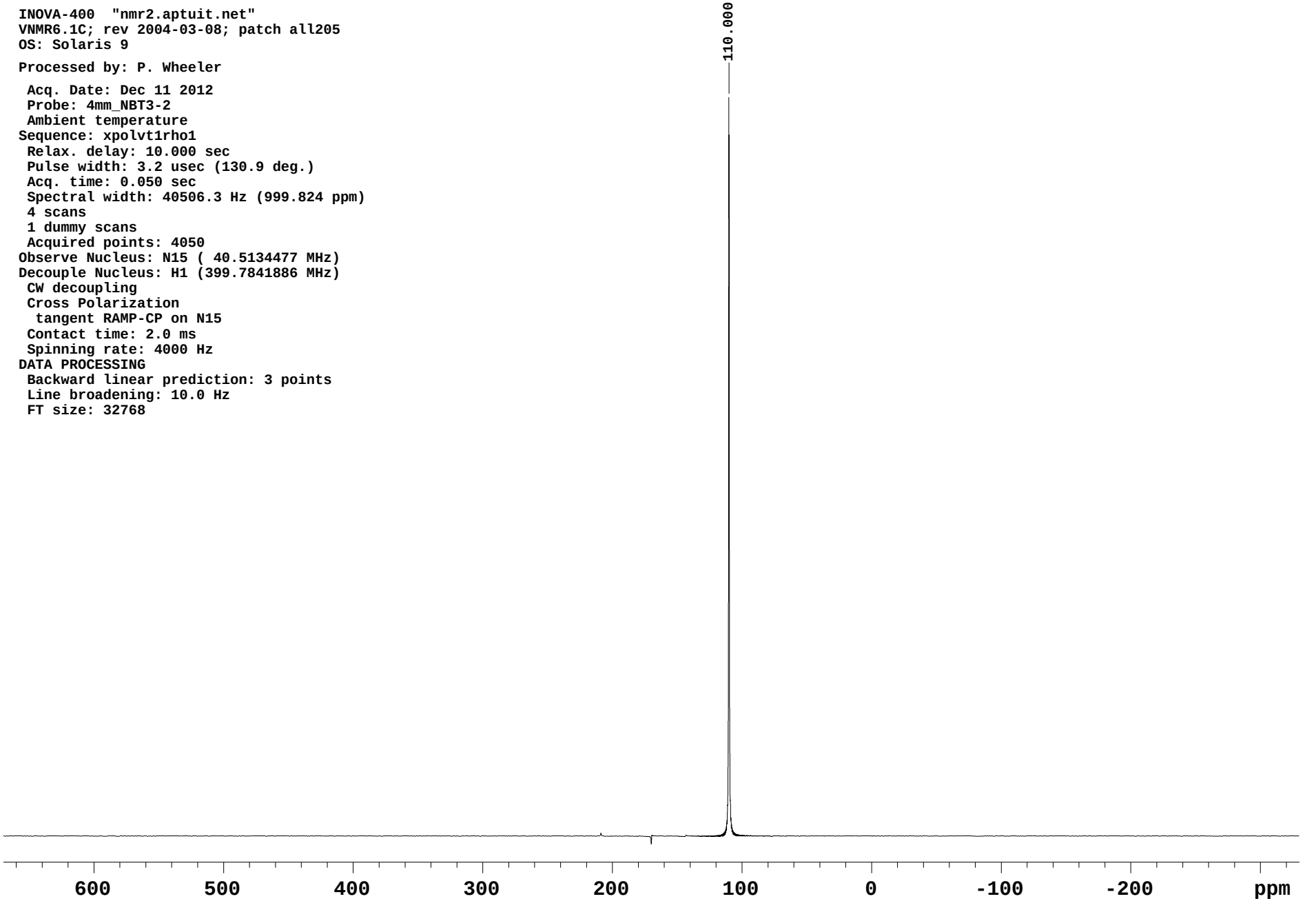
72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17

File: 564428

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 11 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 10.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4 scans
1 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 2.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564428-1

72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17

File: 564428

INDEX	FREQUENCY	PPM	HEIGHT
1	4455.722	110.000	141.8

Plot file: 564428-1_peaks

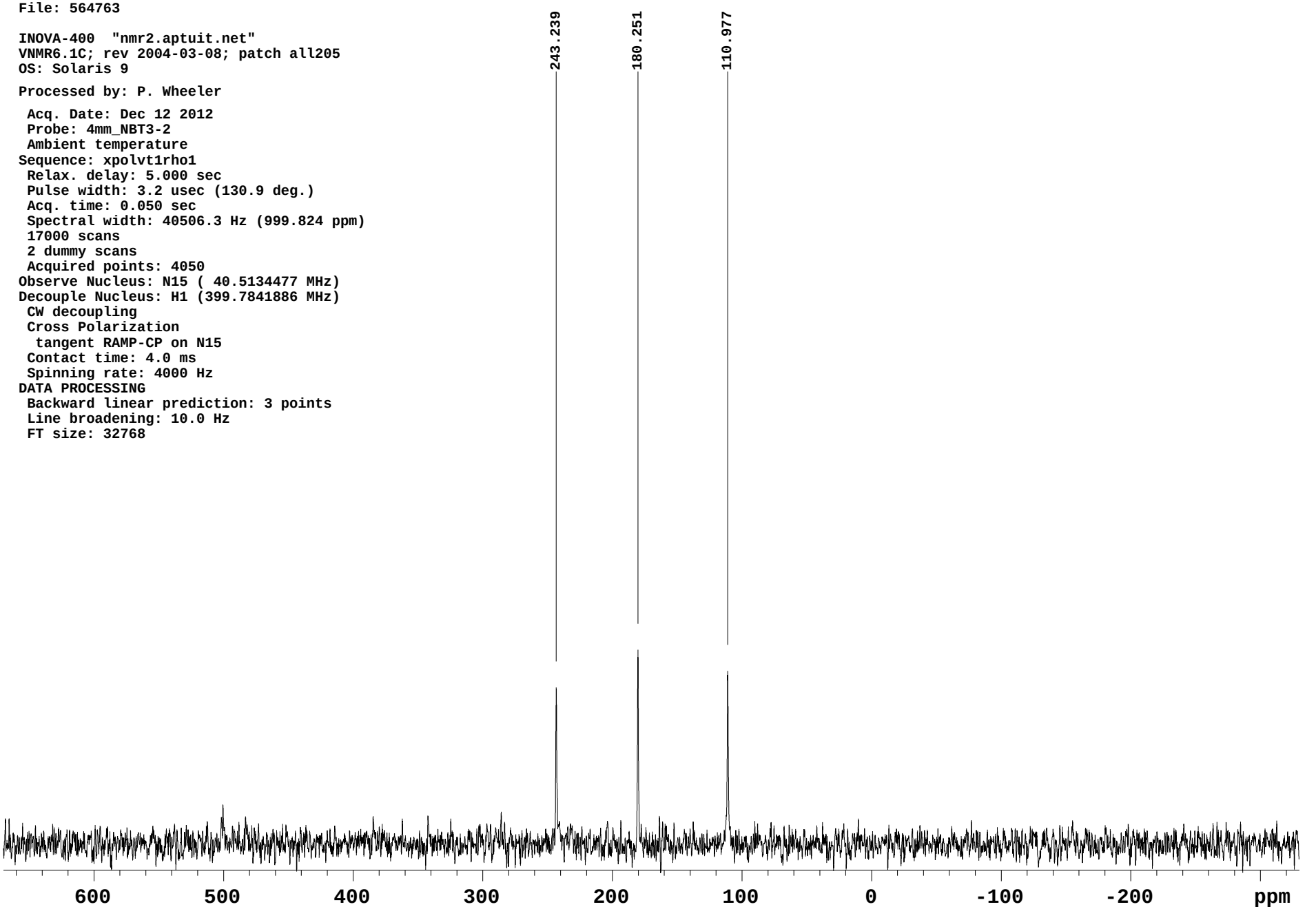
308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm
4 coadded fids

File: 564763

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
17000 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564763-1

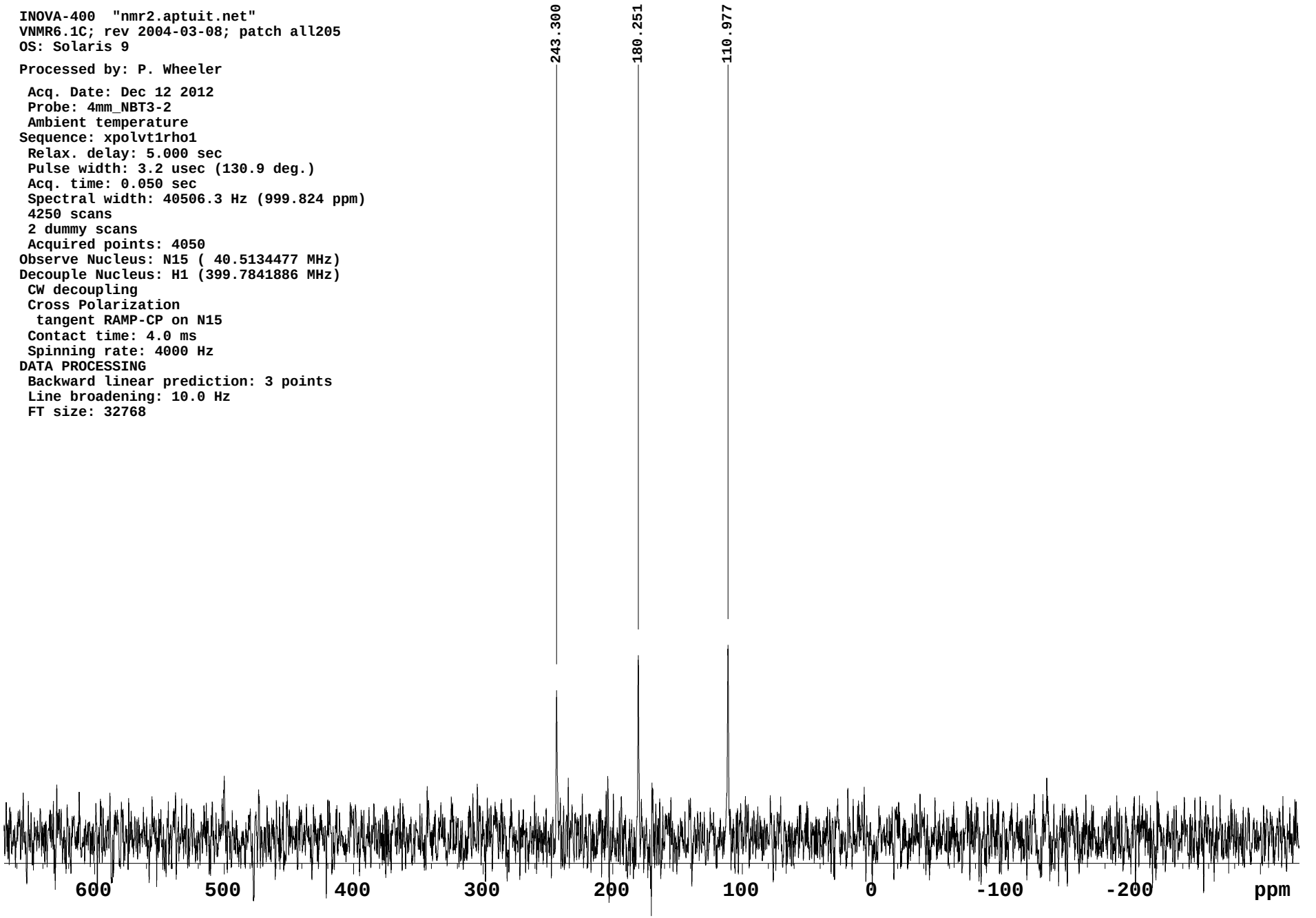
308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-1

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564763-1a

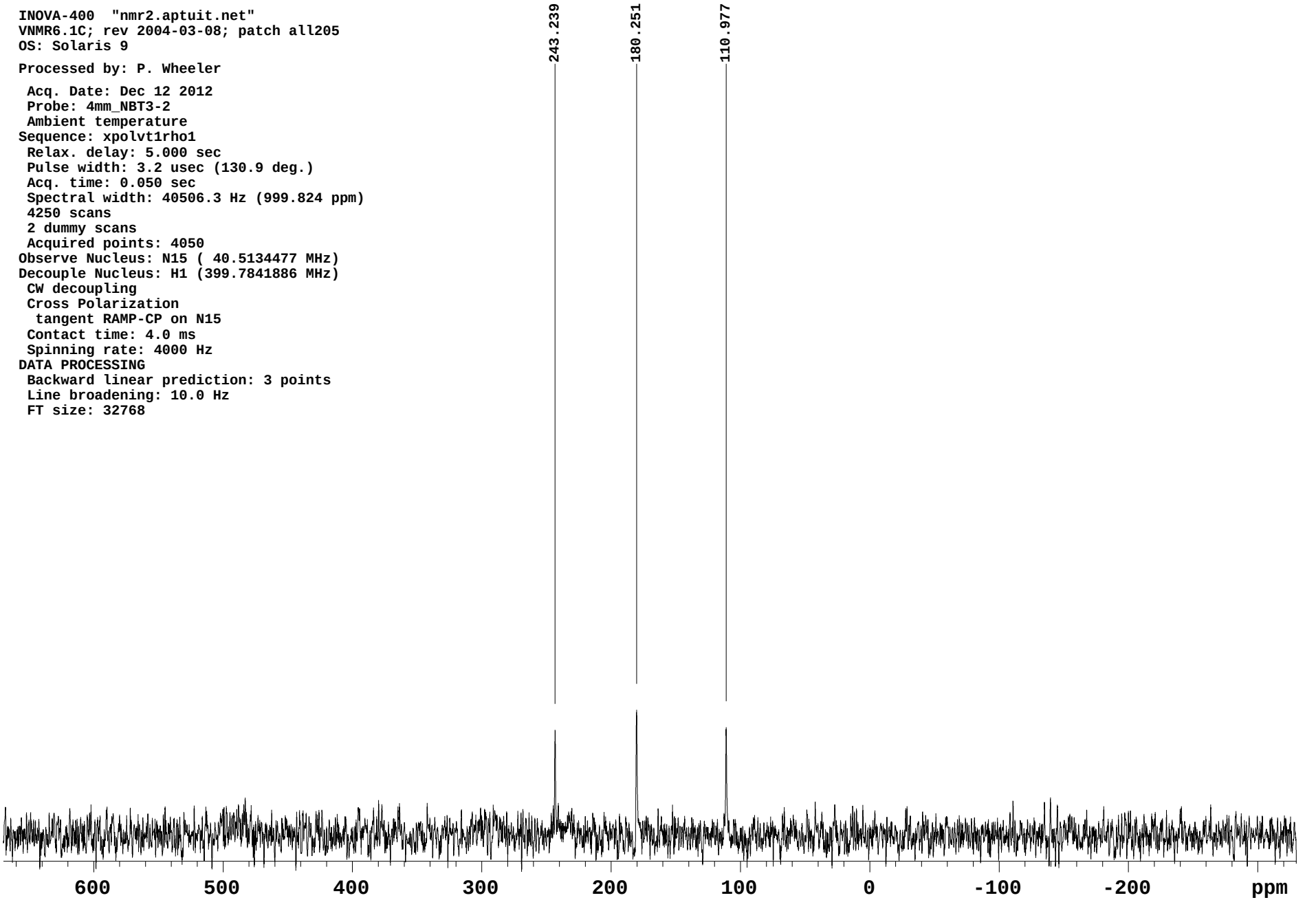
308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-2

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564763-2a

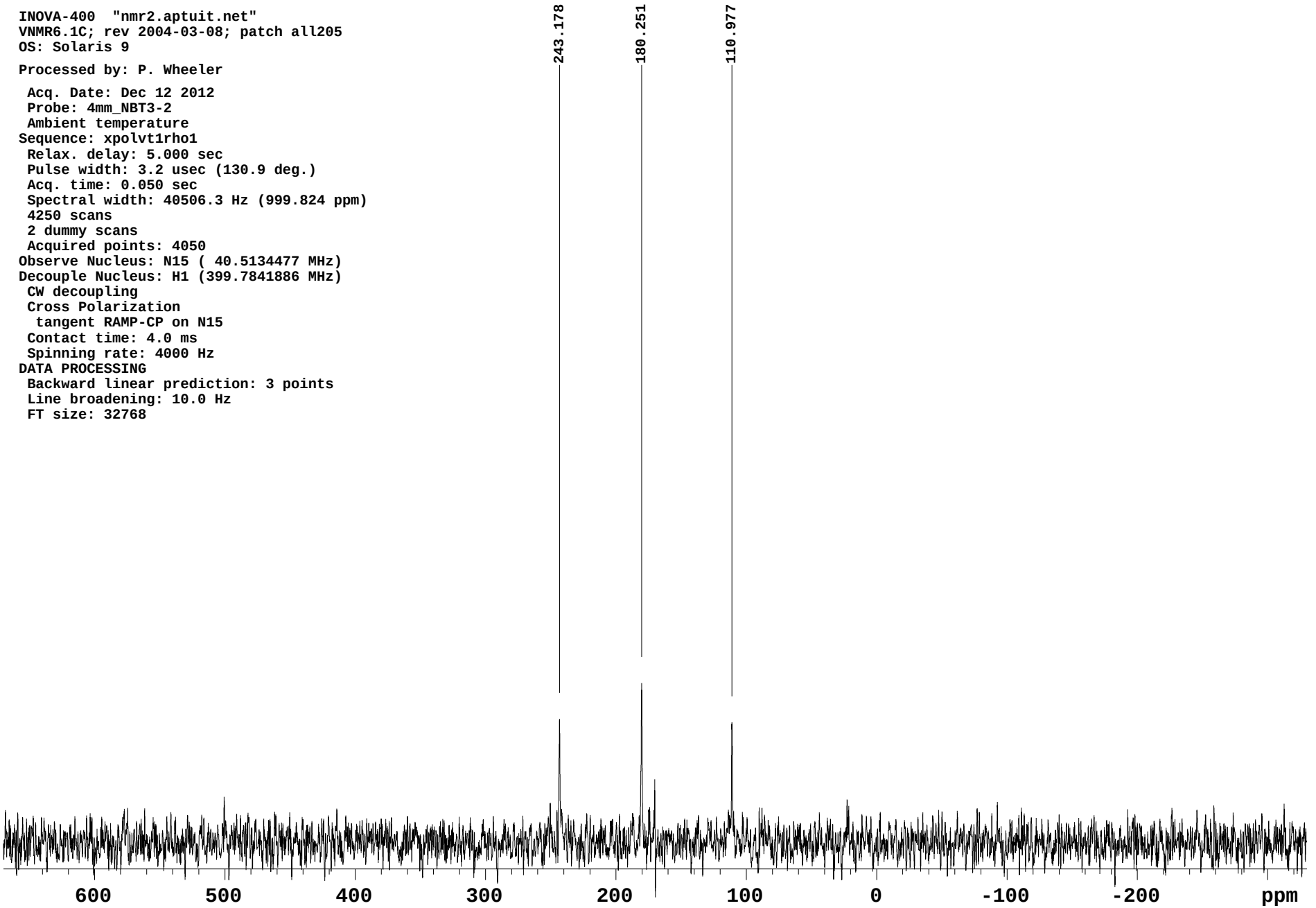
308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-3

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564763-3a

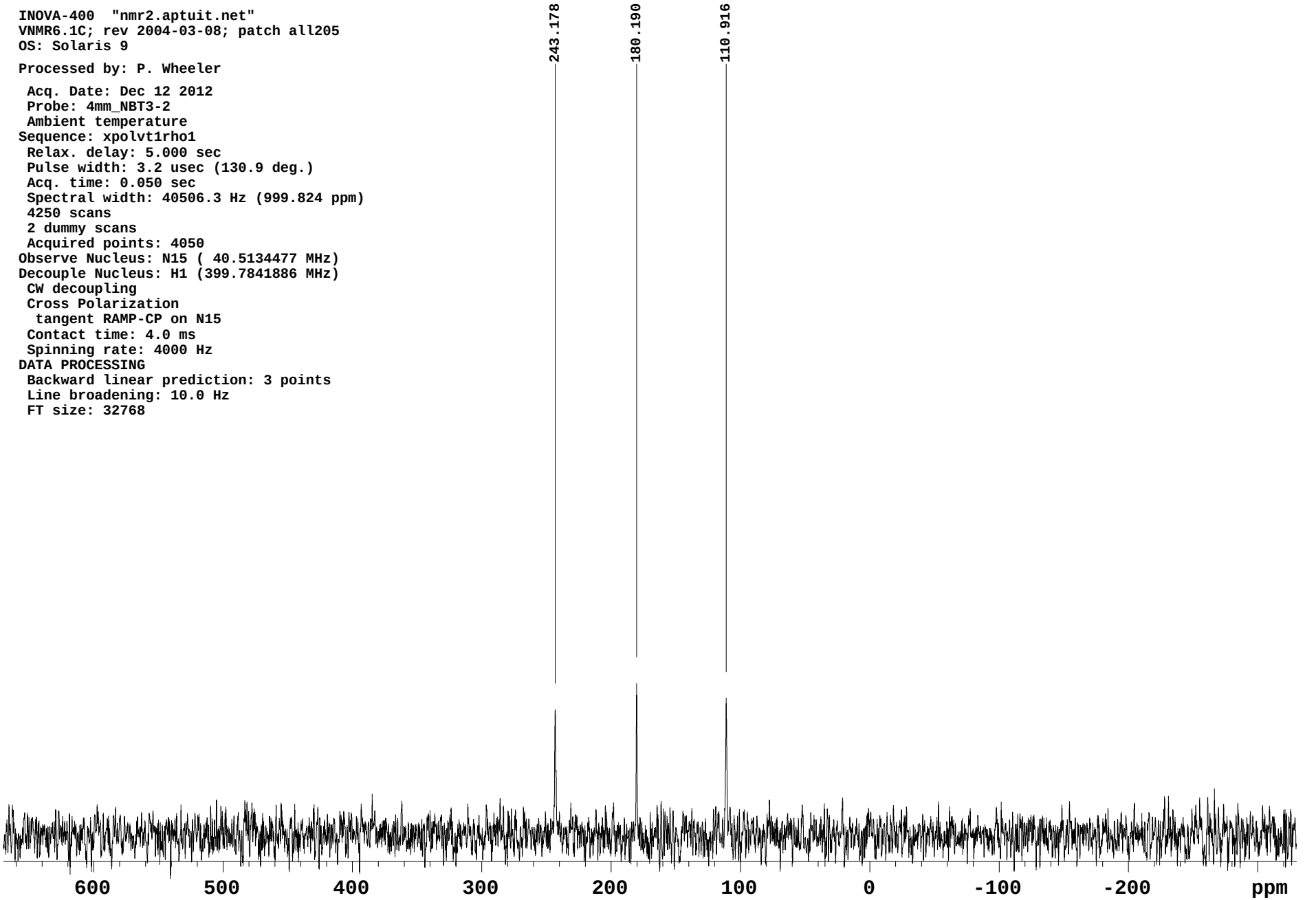
308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-4

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564763-4a

308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm
4 coadded fids

File: 564763

INDEX	FREQUENCY	PPM	HEIGHT
1	9852.775	243.239	30.1
2	7301.351	180.251	37.3
3	4495.279	110.977	33.3

Plot file: 564763-1_peaks

308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-1

INDEX	FREQUENCY	PPM	HEIGHT
1	9855.248	243.300	28.2
2	7301.351	180.251	35.0
3	4495.279	110.977	37.0

Plot file: 564763-1a_peaks

308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-2

INDEX	FREQUENCY	PPM	HEIGHT
1	9852.775	243.239	20.3
2	7301.351	180.251	24.1
3	4495.279	110.977	20.8

Plot file: 564763-2a_peaks

308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-3

INDEX	FREQUENCY	PPM	HEIGHT
1	9850.303	243.178	23.6
2	7301.351	180.251	30.5
3	4495.279	110.977	23.0

Plot file: 564763-3a_peaks

308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564763-4

INDEX	FREQUENCY	PPM	HEIGHT
1	9850.303	243.178	24.2
2	7298.879	180.190	29.3
3	4492.807	110.916	26.4

Plot file: 564763-4a_peaks

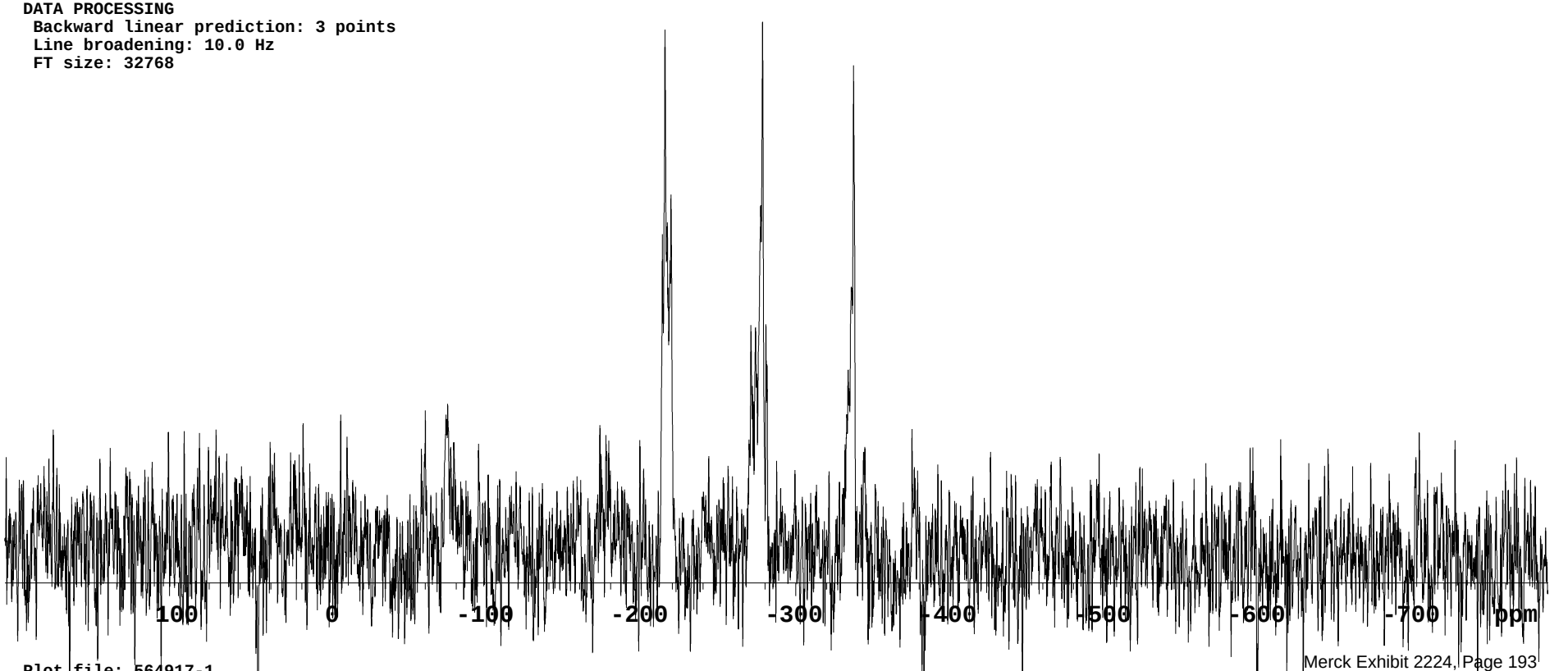
314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 564917

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

 Acq. Date: Dec 13 2012
 Probe: 4mm_NBT3-2
 Ambient temperature
Sequence: xpolvt1rho1
 Relax. delay: 5.000 sec
 Pulse width: 3.2 usec (130.9 deg.)
 Acq. time: 0.050 sec
 Spectral width: 40506.3 Hz (999.824 ppm)
 17000 scans
 2 dummy scans
 Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
 CW decoupling
 Cross Polarization
  tangent RAMP-CP on N15
 Contact time: 4.0 ms
 Spinning rate: 4000 Hz
DATA PROCESSING
 Backward linear prediction: 3 points
 Line broadening: 10.0 Hz
 FT size: 32768



Plot file: 564917-1

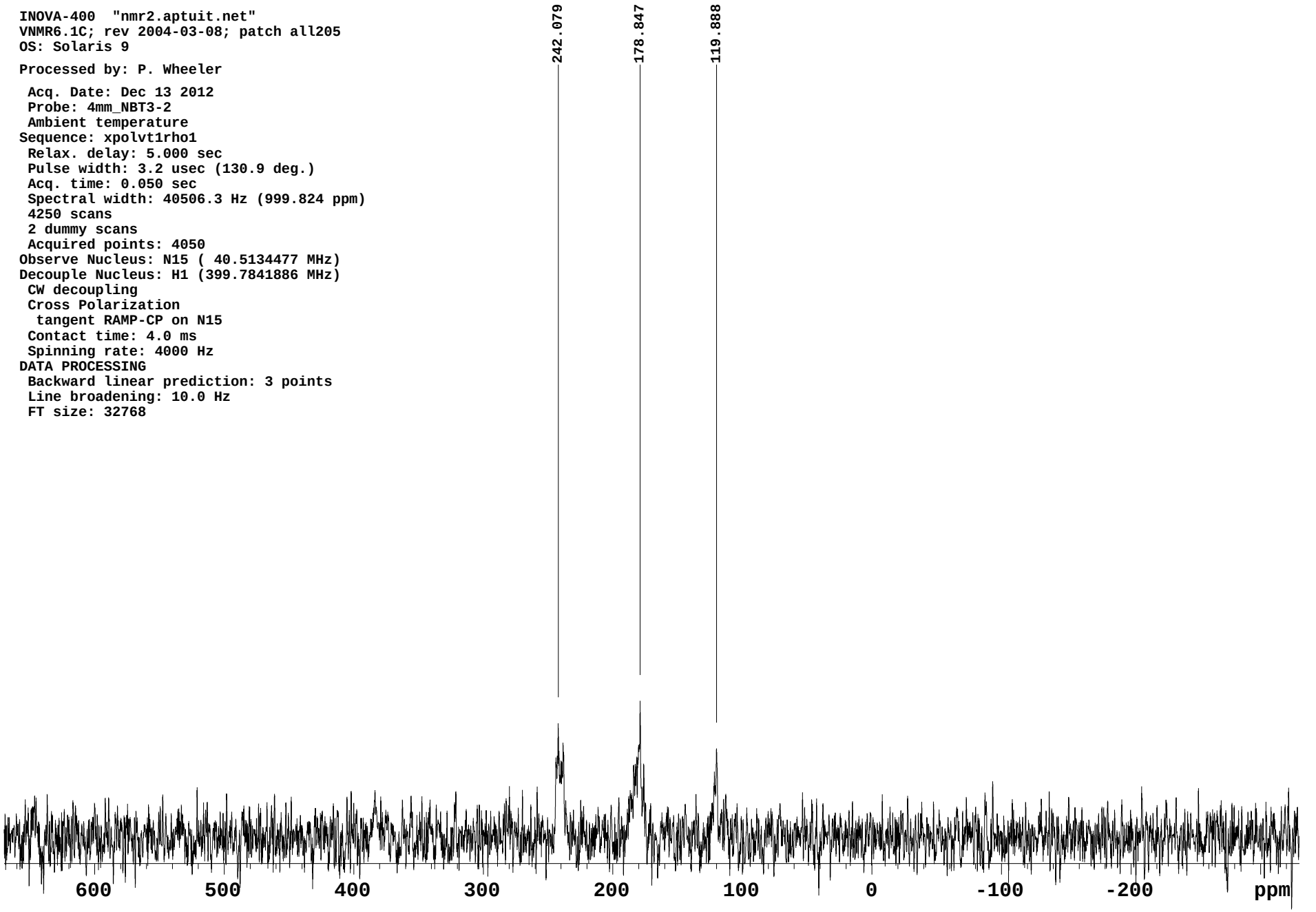
314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564917-1

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

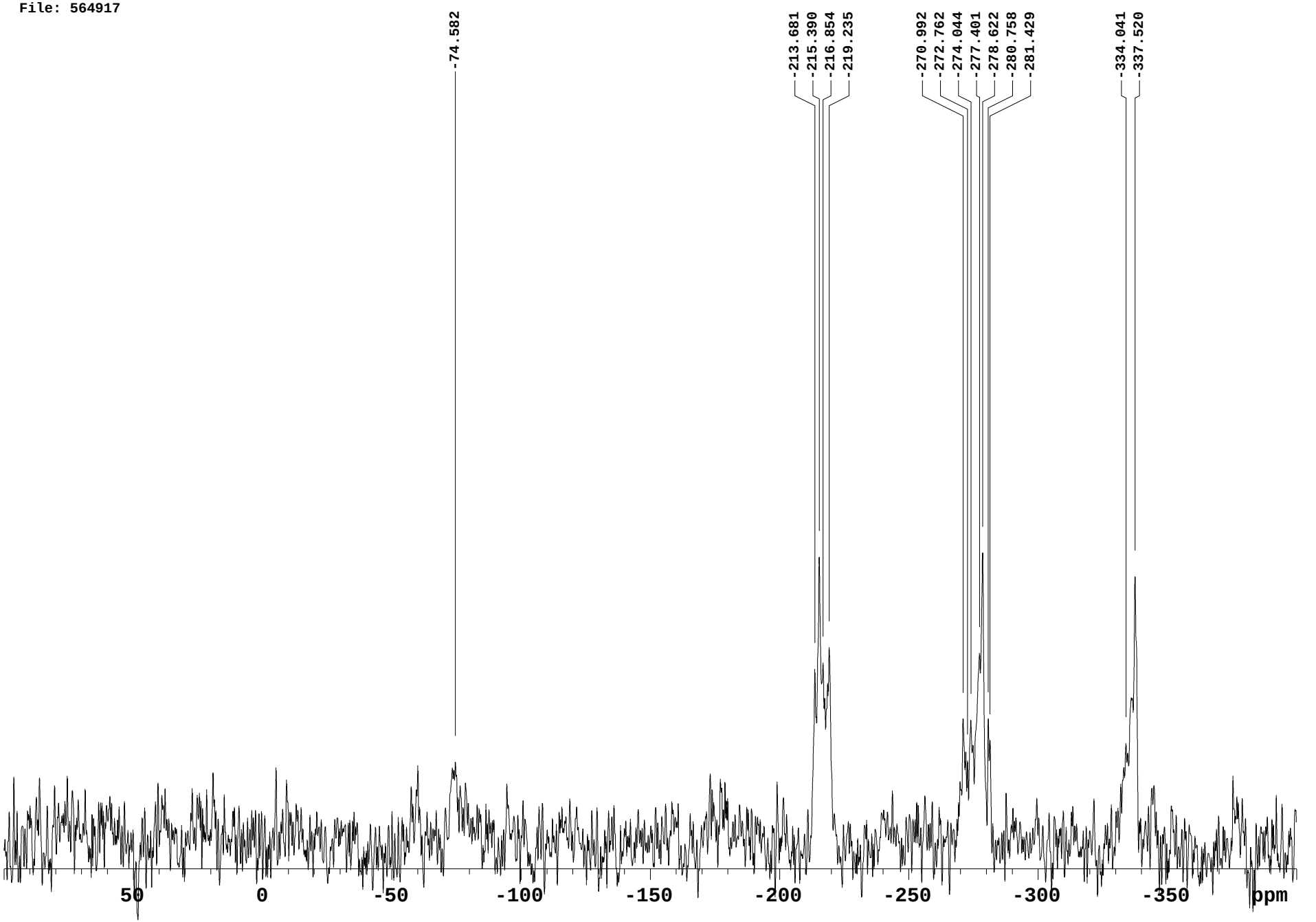
Acq. Date: Dec 13 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564917-1a

314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 564917



Plot file: 564917-2

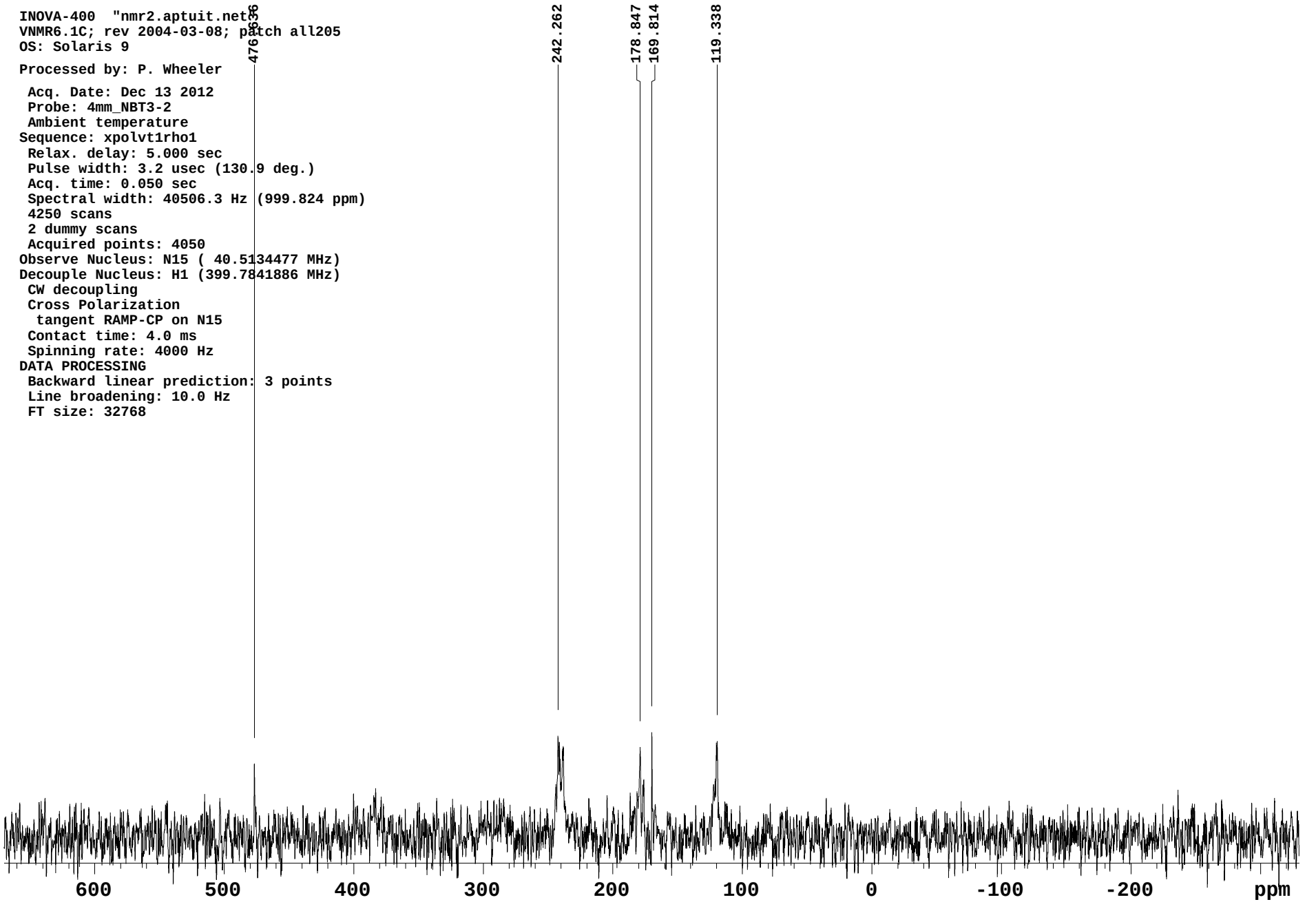
314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564917-2

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 13 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564917-2a

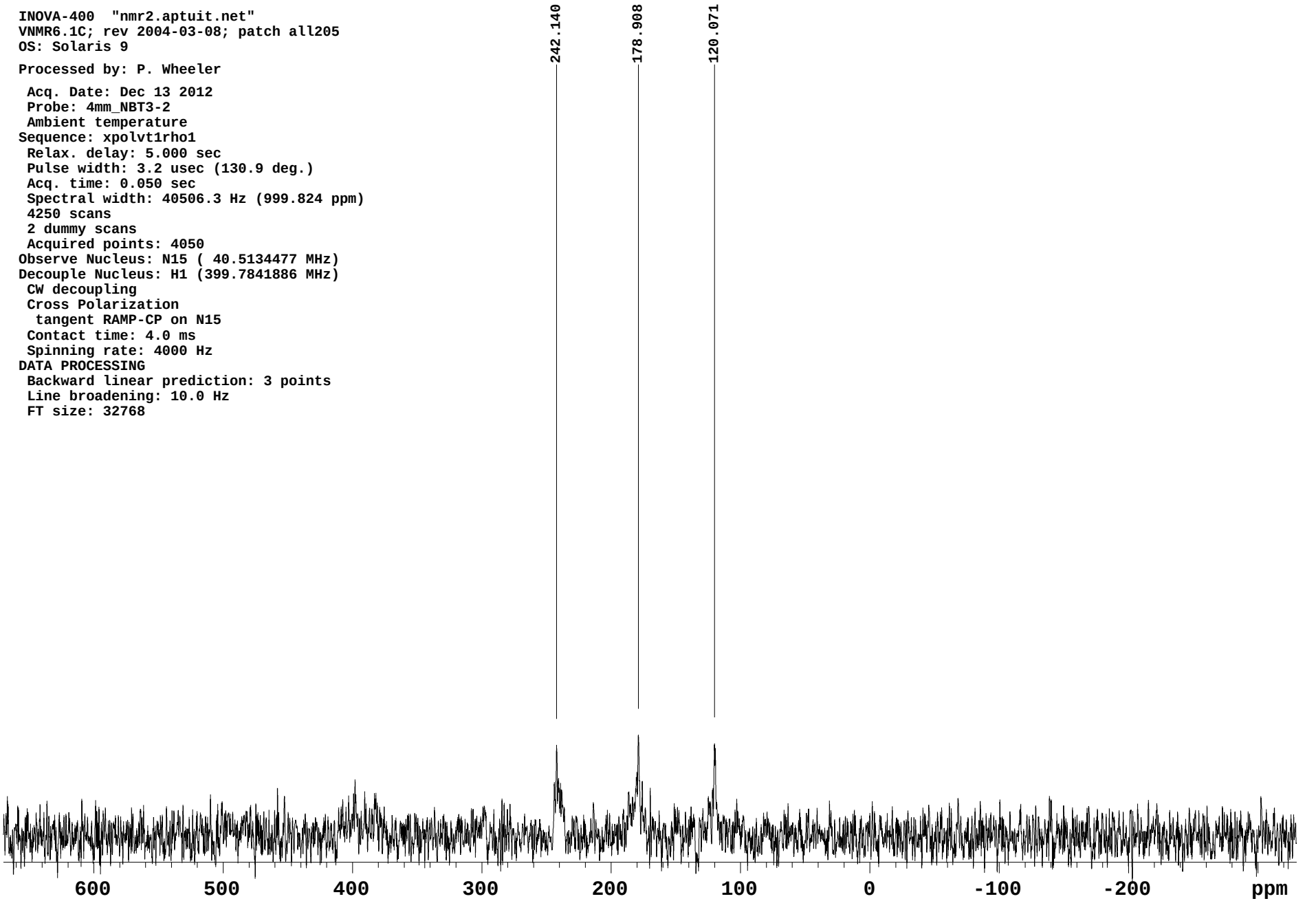
314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564917-3

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 13 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4250 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564917-3a

314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564917-1

INDEX	FREQUENCY	PPM	HEIGHT
1	9805.801	242.079	21.9
2	7244.488	178.847	26.2
3	4856.236	119.888	17.1
4	-13127.348	-324.080	-13.8

Plot file: 564917-1a_peaks

314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 564917

INDEX	FREQUENCY	PPM	HEIGHT
1	-3021.073	-74.582	15.6
2	-8655.468	-213.681	33.5
3	-8724.693	-215.390	55.0
4	-8784.028	-216.854	34.7
5	-8880.448	-219.235	37.6
6	-10976.967	-270.992	23.9
7	-11048.664	-272.762	15.8
8	-11100.583	-274.044	23.7
9	-11236.560	-277.401	36.5
10	-11286.006	-278.622	55.8
11	-11372.537	-280.758	24.0
12	-11399.732	-281.429	19.8
13	-13530.863	-334.041	19.2
14	-13671.785	-337.520	51.2

Plot file: 564917-2_peaks

314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564917-2

INDEX	FREQUENCY	PPM	HEIGHT
1	19306.889	476.636	14.1
2	9813.218	242.262	19.5
3	7244.488	178.847	17.3
4	6878.586	169.814	20.2
5	4833.986	119.338	18.5

Plot file: 564917-2a_peaks

314339, 5135-02-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at 110 ppm

File: 564917-3

INDEX	FREQUENCY	PPM	HEIGHT
1	9808.273	242.140	17.6
2	7246.960	178.908	19.5
3	4863.653	120.071	17.9

Plot file: 564917-3a_peaks

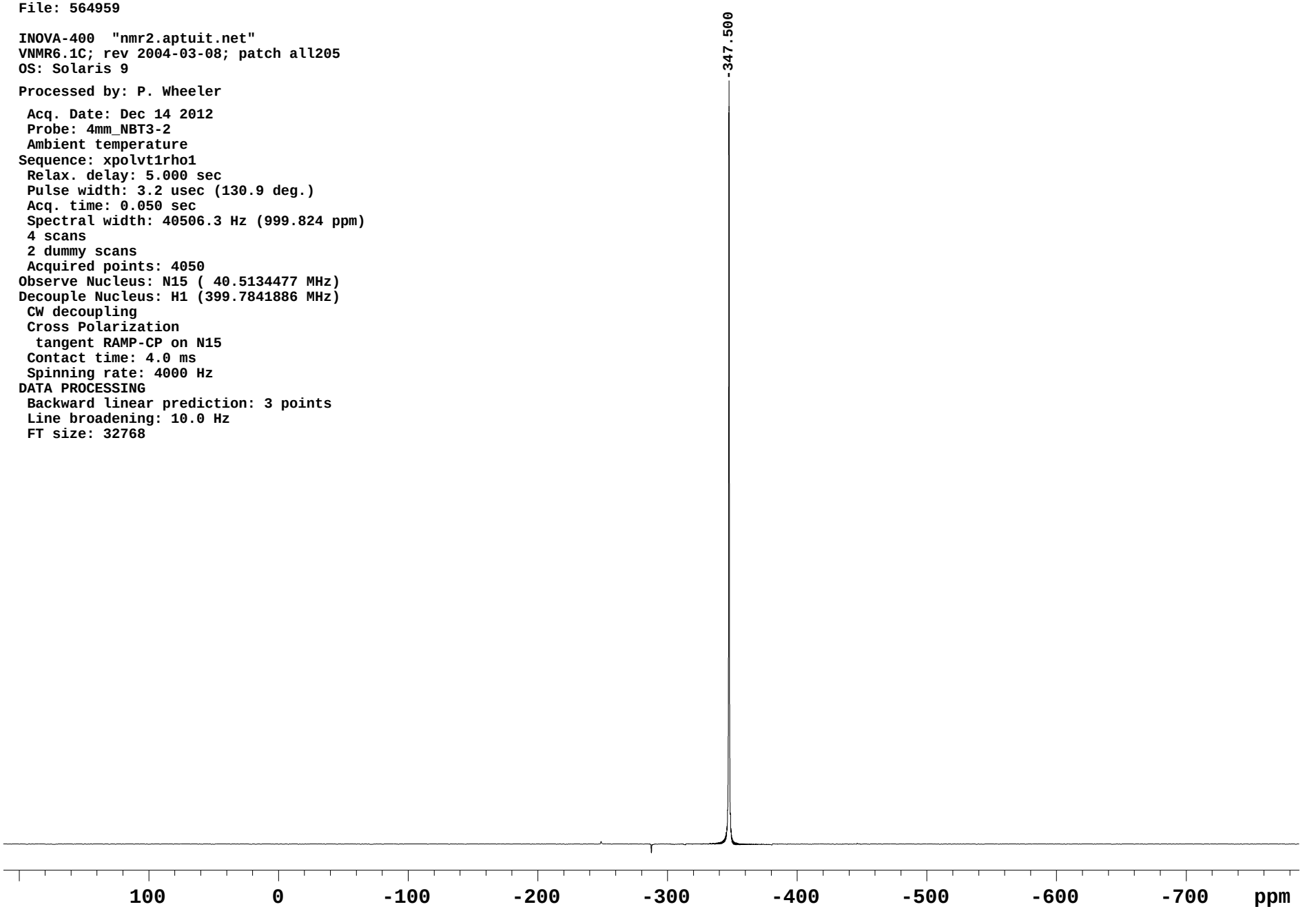
72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17
Referenced to -347.5 ppm vs. nitromethane

File: 564959

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 14 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 564959-1

72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17
Referenced to -347.5 ppm vs. nitromethane

File: 564959

INDEX	FREQUENCY	PPM	HEIGHT
1	-14082.473	-347.500	141.8

Plot file: 564959-1_peaks

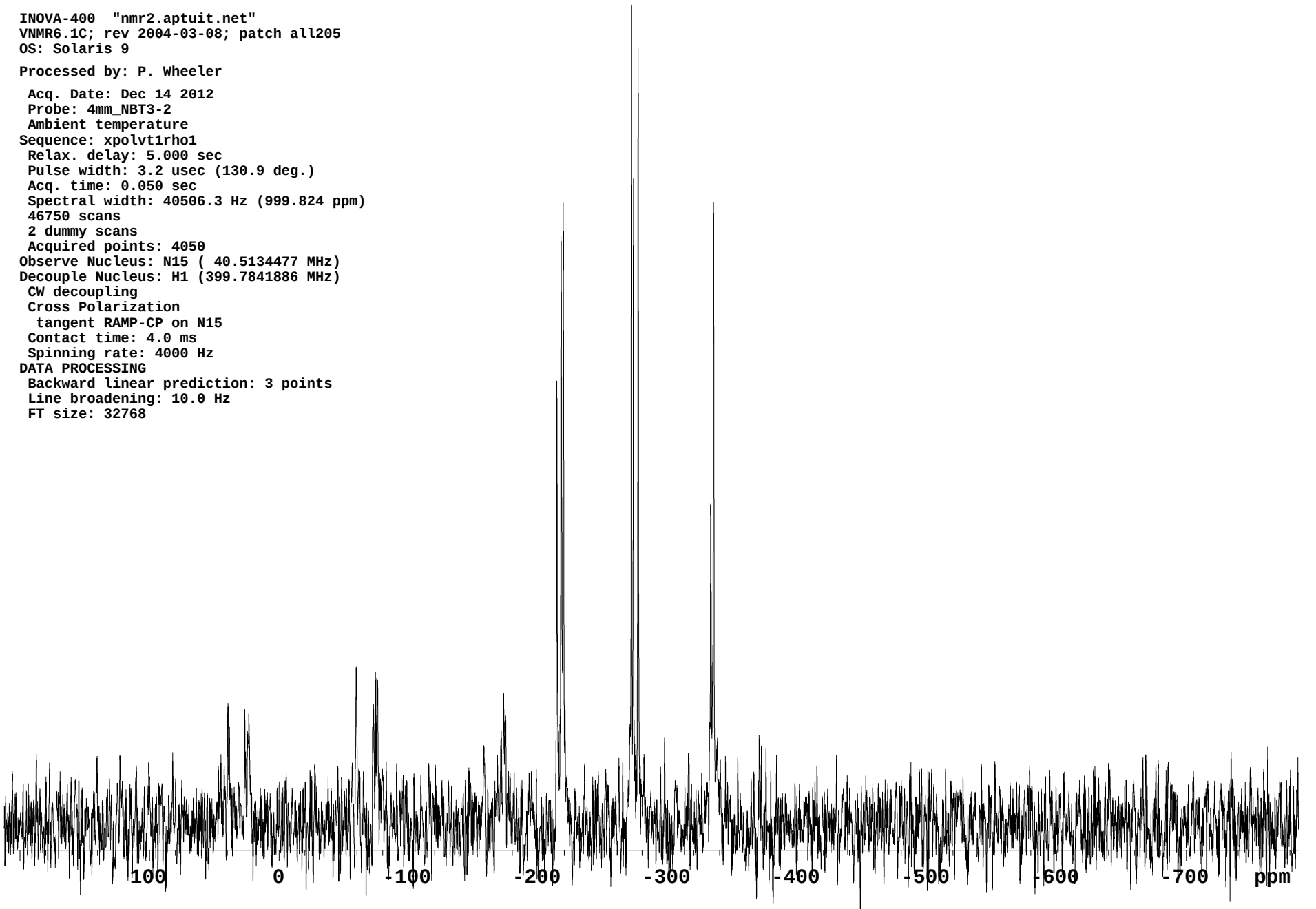
314783, 5135-17-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
11 co-added FIDs

File: 565268

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

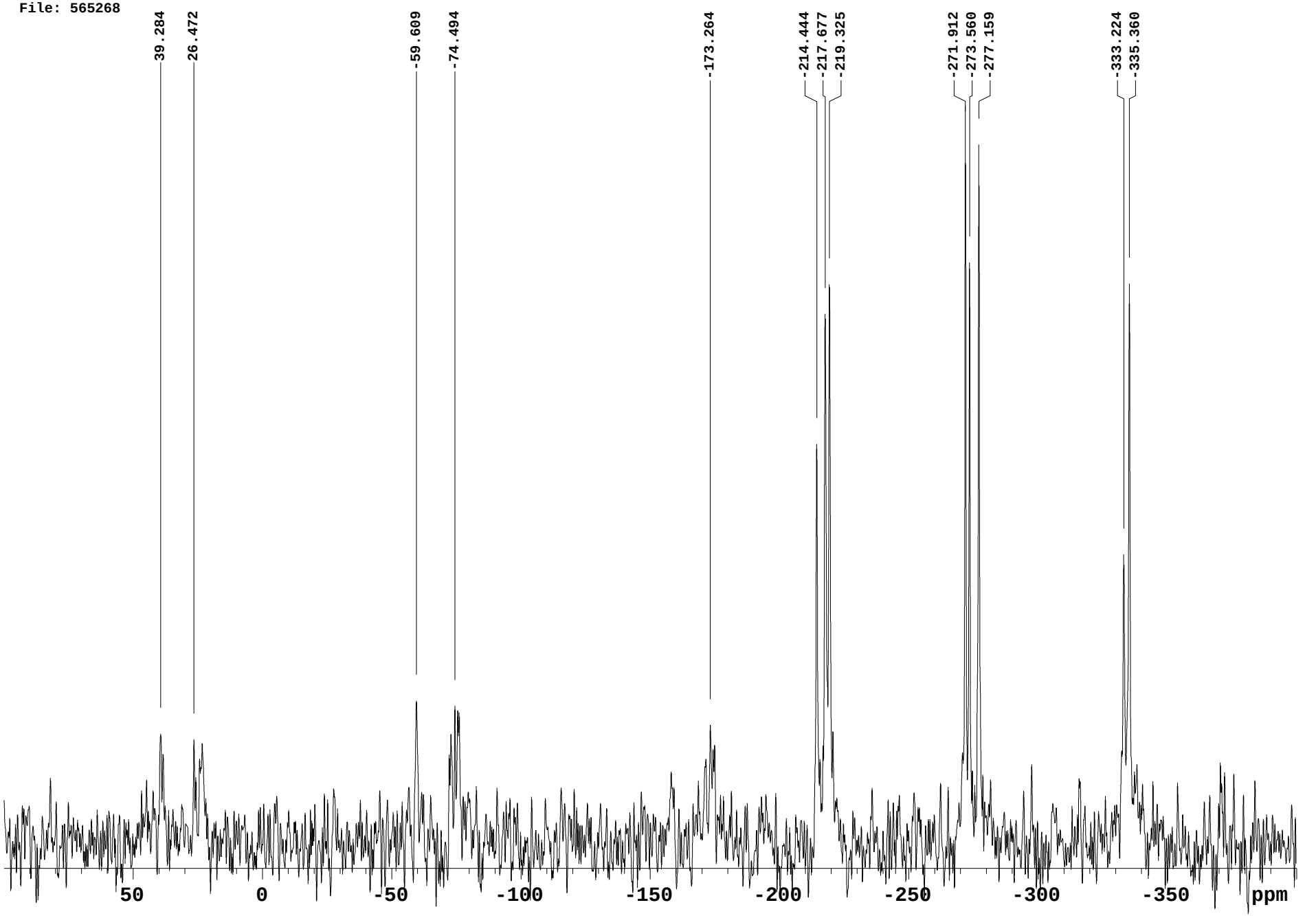
 Acq. Date: Dec 14 2012
 Probe: 4mm_NBT3-2
 Ambient temperature
Sequence: xpolvt1rho1
 Relax. delay: 5.000 sec
 Pulse width: 3.2 usec (130.9 deg.)
 Acq. time: 0.050 sec
 Spectral width: 40506.3 Hz (999.824 ppm)
 46750 scans
 2 dummy scans
 Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
 CW decoupling
 Cross Polarization
  tangent RAMP-CP on N15
 Contact time: 4.0 ms
 Spinning rate: 4000 Hz
DATA PROCESSING
 Backward linear prediction: 3 points
 Line broadening: 10.0 Hz
 FT size: 32768



Plot file: 565268-1

314783, 5135-17-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
11 co-added FIDs

File: 565268



Plot file: 565268-2

314783, 5135-17-01, Compound 184, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
11 co-added FIDs

File: 565268

INDEX	FREQUENCY	PPM	HEIGHT
1	1591.973	39.284	20.9
2	1072.788	26.472	19.8
3	-2415.642	-59.609	27.3
4	-3018.885	-74.494	26.3
5	-7021.555	-173.264	22.6
6	-8690.365	-214.444	76.7
7	-8821.397	-217.677	101.7
8	-8888.149	-219.325	107.4
9	-11019.281	-271.912	141.8
10	-11086.033	-273.560	111.6
11	-11231.899	-277.159	134.3
12	-13503.952	-333.224	55.4
13	-13590.483	-335.360	107.6

Plot file: 565268-2_peaks

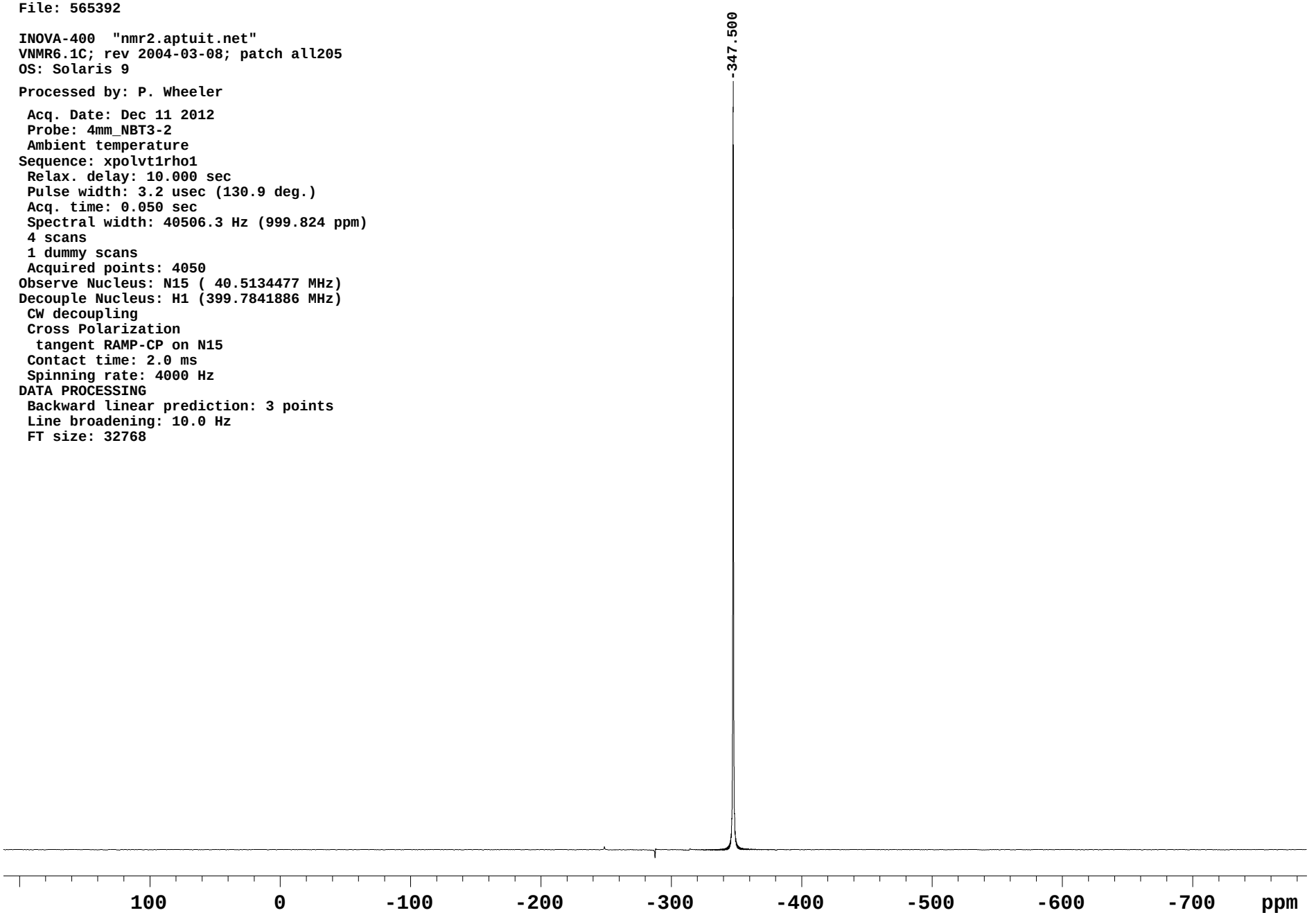
72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17
Referenced to -347.5 ppm vs. nitromethane

File: 565392

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 11 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 10.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4 scans
1 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 2.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 565392-1

72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17
Referenced to -347.5 ppm vs. nitromethane

File: 565392

INDEX	FREQUENCY	PPM	HEIGHT
1	-14076.052	-347.500	141.8

Plot file: 565392-1_peaks

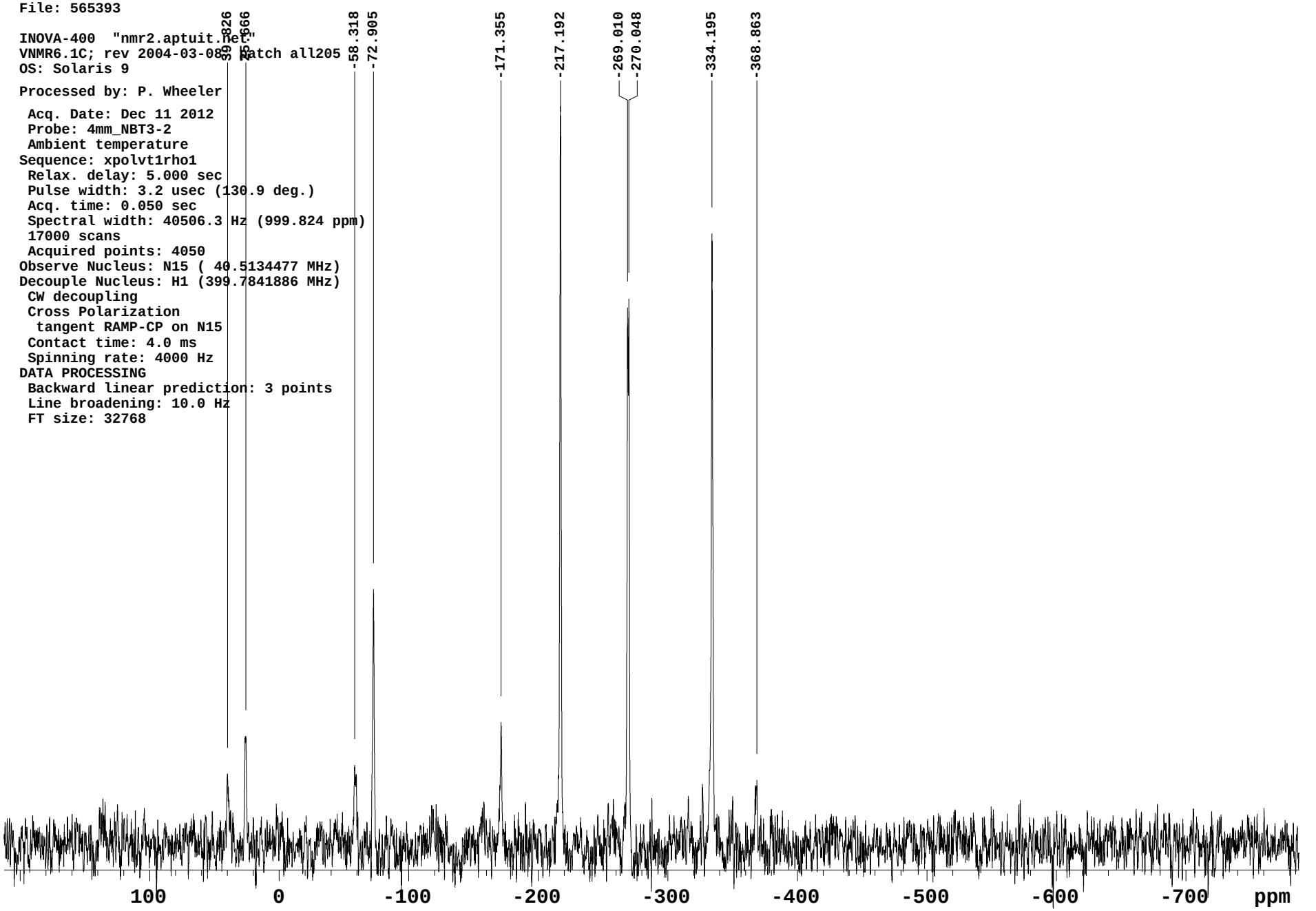
308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 565393

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08 match all205
OS: Solaris 9

Processed by: P. Wheeler

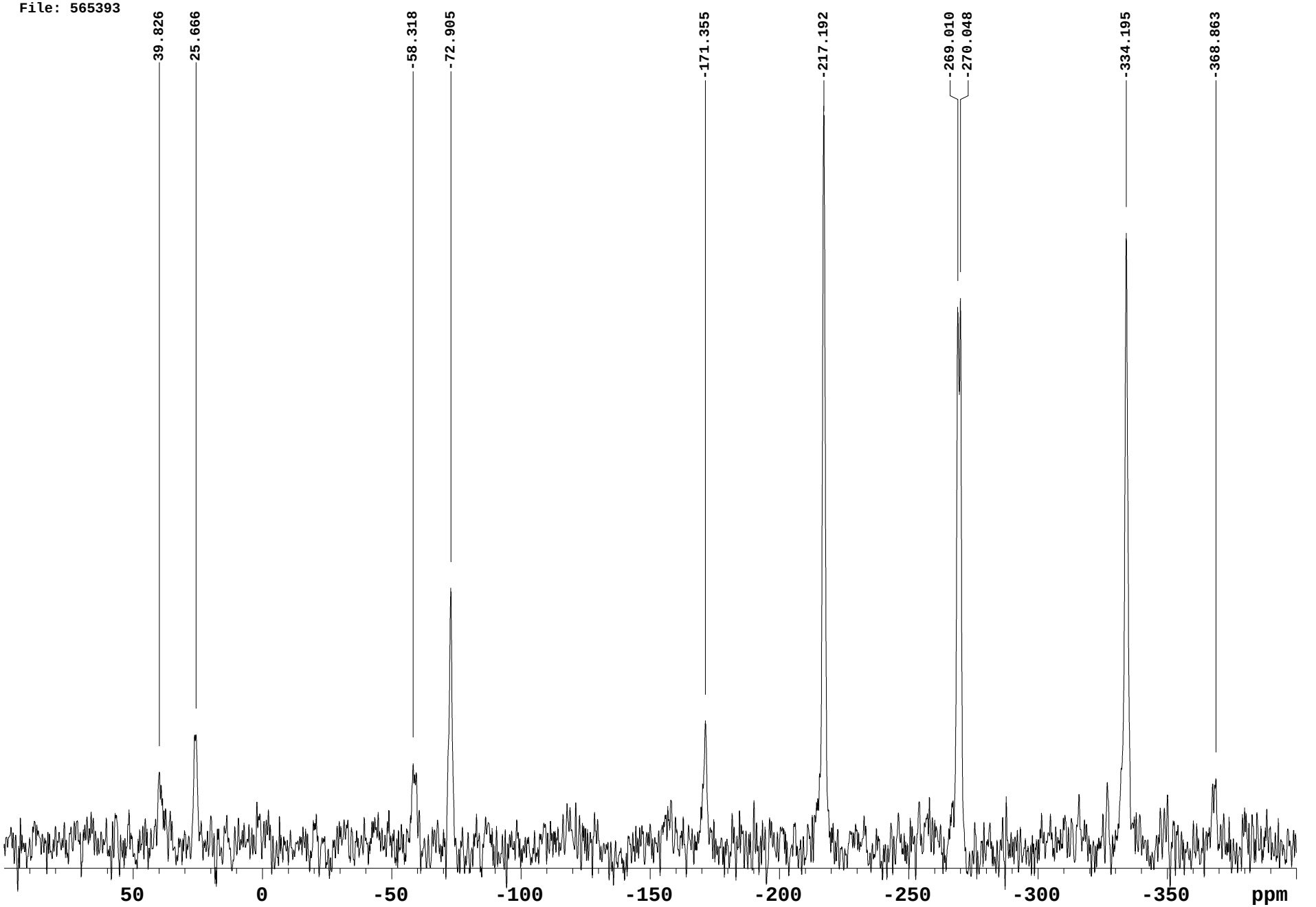
Acq. Date: Dec 11 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
17000 scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 565393-1

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 565393



Plot file: 565393-2

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 565393

INDEX	FREQUENCY	PPM	HEIGHT
1	1613.208	39.826	13.5
2	1039.632	25.666	20.8
3	-2362.266	-58.318	15.2
4	-2953.148	-72.905	49.0
5	-6940.985	-171.355	23.4
6	-8797.690	-217.192	141.8
7	-10896.681	-269.010	103.1
8	-10938.710	-270.048	104.7
9	-13537.108	-334.195	117.3
10	-14941.380	-368.863	12.3
11	-24207.599	-597.622	-12.3

Plot file: 565393-1_peaks

308389, Compound 184, Lot LB-1017, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 565393

INDEX	FREQUENCY	PPM	HEIGHT
1	1613.208	39.826	13.5
2	1039.632	25.666	20.8
3	-2362.266	-58.318	15.2
4	-2953.148	-72.905	49.0
5	-6940.985	-171.355	23.4
6	-8797.690	-217.192	141.8
7	-10896.681	-269.010	103.1
8	-10938.710	-270.048	104.7
9	-13537.108	-334.195	117.3
10	-14941.380	-368.863	12.3

Plot file: 565393-2_peaks

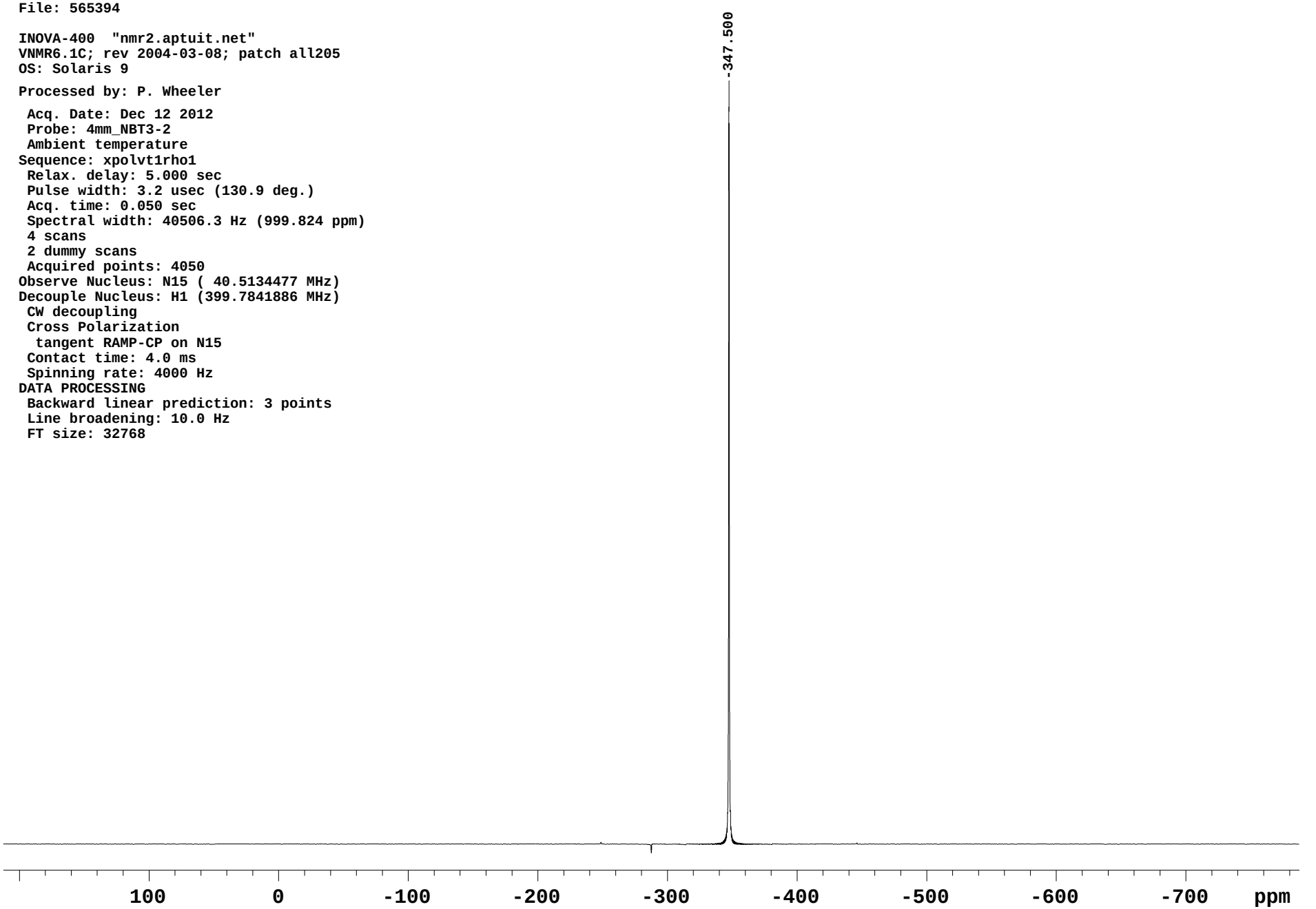
72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17
Referenced to -347.5 ppm vs. nitromethane

File: 565394

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
4 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 565394-1

72084, 15N-Glycine, Lot PR-15054, 15N CPMAS SSNMR, Exp. Date: 12/10/17
Referenced to -347.5 ppm vs. nitromethane

File: 565394

INDEX	FREQUENCY	PPM	HEIGHT
1	-14076.022	-347.500	141.6

Plot file: 565394-1_peaks

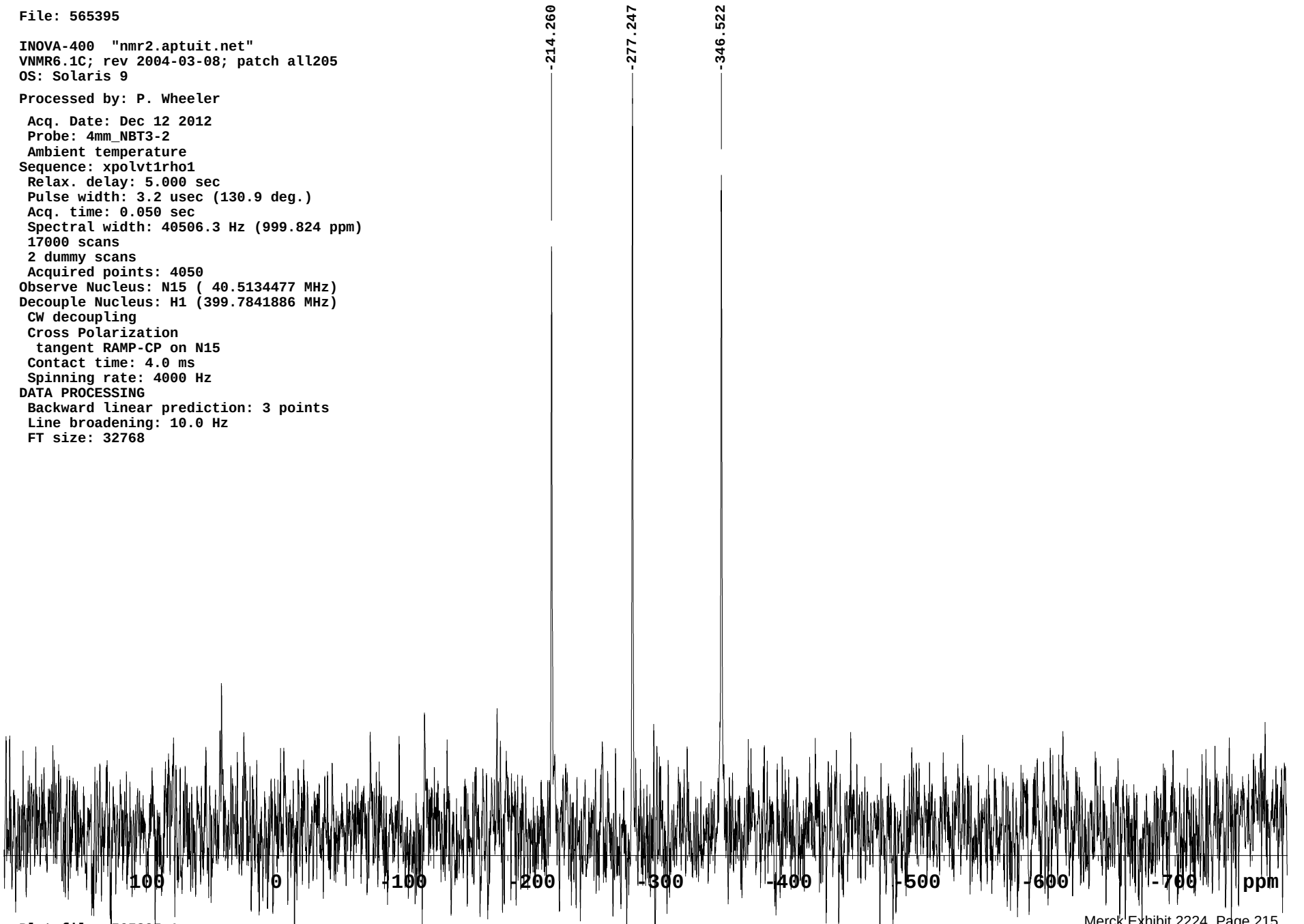
308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 565395

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 12 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 3.2 usec (130.9 deg.)
Acq. time: 0.050 sec
Spectral width: 40506.3 Hz (999.824 ppm)
17000 scans
2 dummy scans
Acquired points: 4050
Observe Nucleus: N15 (40.5134477 MHz)
Decouple Nucleus: H1 (399.7841886 MHz)
CW decoupling
Cross Polarization
tangent RAMP-CP on N15
Contact time: 4.0 ms
Spinning rate: 4000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 32768



Plot file: 565395-1

308390, Compound 184, Lot D6655070112, 15N CPMAS NMR, externally referenced to 15N-glycine at -347.5 ppm
4 co-added FIDs

File: 565395

INDEX	FREQUENCY	PPM	HEIGHT
1	-8678.919	-214.260	113.1
2	-11230.343	-277.247	141.7
3	-14036.415	-346.522	126.9

Plot file: 565395-1_peaks

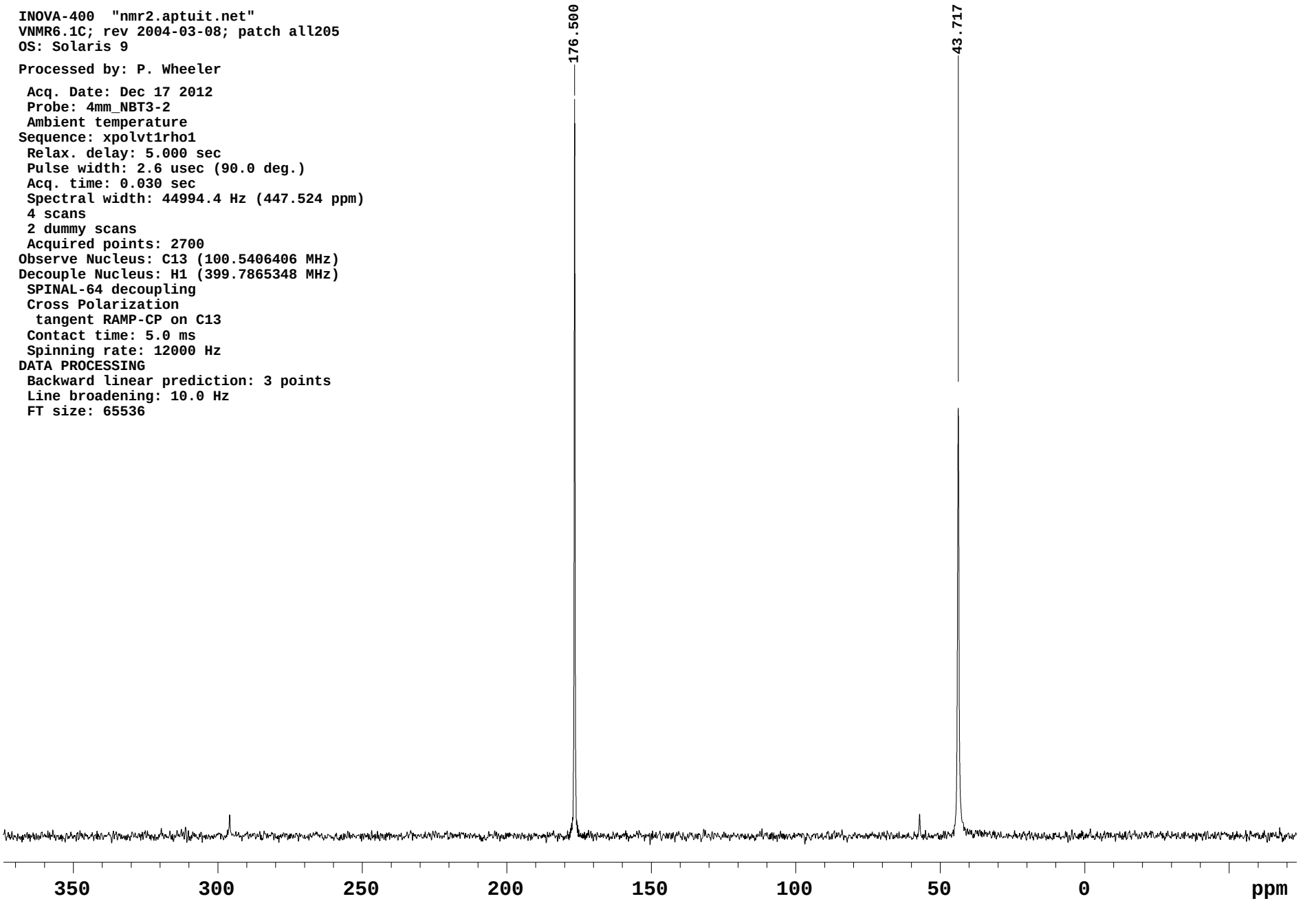
72083, Glycine 98%, Lot 11903CN, 13C CPMAS SSNMR, Exp. Date: 12/23/14

File: 565563

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

Acq. Date: Dec 17 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 5.000 sec
Pulse width: 2.6 usec (90.0 deg.)
Acq. time: 0.030 sec
Spectral width: 44994.4 Hz (447.524 ppm)
4 scans
2 dummy scans
Acquired points: 2700
Observe Nucleus: C13 (100.5406406 MHz)
Decouple Nucleus: H1 (399.7865348 MHz)
SPINAL-64 decoupling
Cross Polarization
tangent RAMP-CP on C13
Contact time: 5.0 ms
Spinning rate: 12000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 65536



Plot file: 565563-1

72083, Glycine 98%, Lot 11903CN, 13C CPMAS SSNMR, Exp. Date: 12/23/14

File: 565563

INDEX	FREQUENCY	PPM	HEIGHT
1	17742.754	176.500	141.8
2	4394.660	43.717	82.4

Plot file: 565563-1_peaks

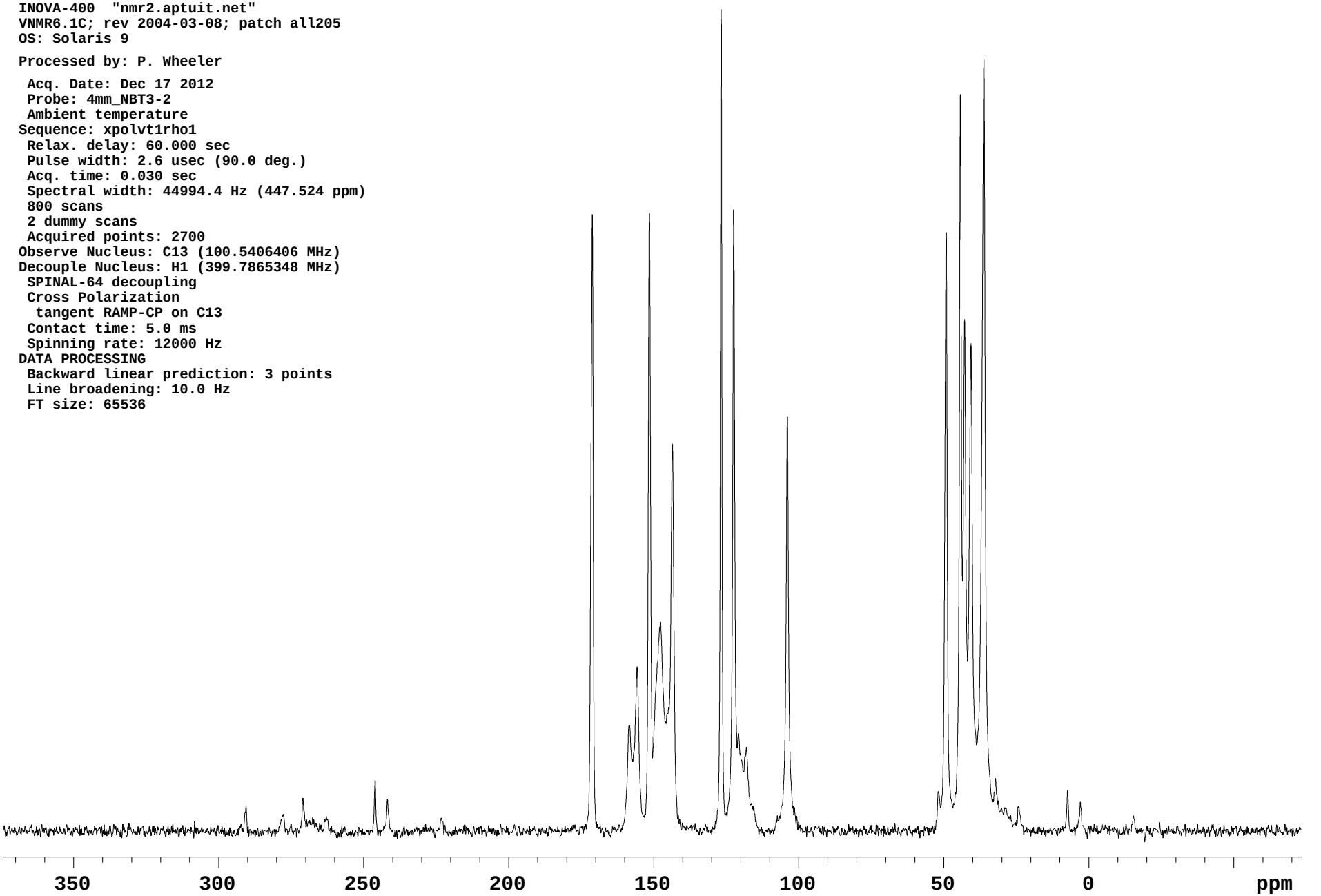
308390, Compound 184, Lot D6655070112, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565564

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

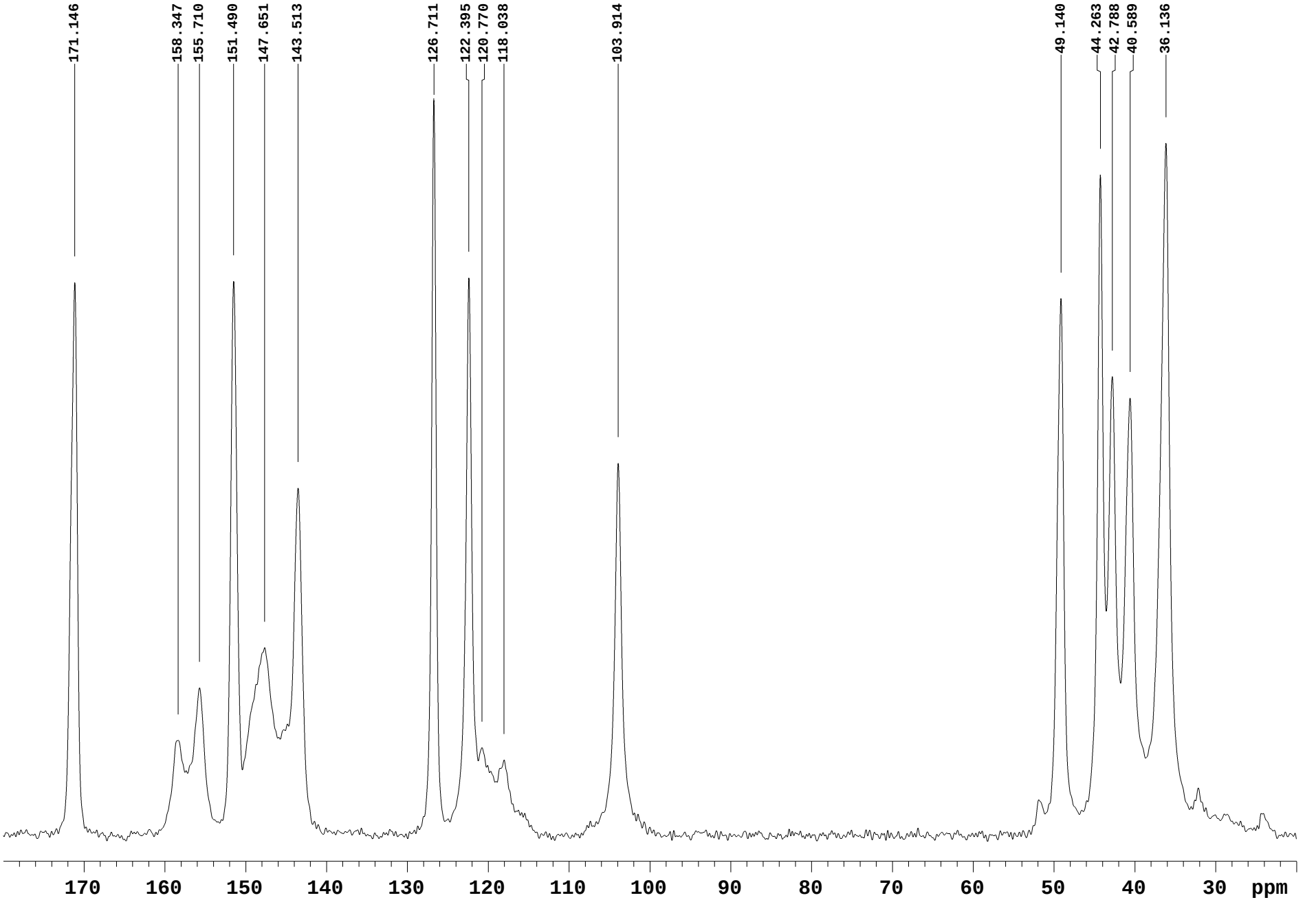
Acq. Date: Dec 17 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 60.000 sec
Pulse width: 2.6 usec (90.0 deg.)
Acq. time: 0.030 sec
Spectral width: 44994.4 Hz (447.524 ppm)
800 scans
2 dummy scans
Acquired points: 2700
Observe Nucleus: C13 (100.5406406 MHz)
Decouple Nucleus: H1 (399.7865348 MHz)
SPINAL-64 decoupling
Cross Polarization
tangent RAMP-CP on C13
Contact time: 5.0 ms
Spinning rate: 12000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 65536



Plot file: 565564-1

308390, Compound 184, Lot D6655070112, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565564



Plot file: 565564-2

308390, Compound 184, Lot D6655070112, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565564

INDEX	FREQUENCY	PPM	HEIGHT
1	17204.491	171.146	106.4
2	15917.878	158.347	18.3
3	15652.866	155.710	28.4
4	15228.572	151.490	106.6
5	14842.726	147.651	36.1
6	14426.671	143.513	66.8
7	12737.734	126.711	141.8
8	12303.828	122.395	107.3
9	12140.427	120.770	16.9
10	11865.803	118.038	14.5
11	10445.998	103.914	71.6
12	4939.789	49.140	103.3
13	4449.585	44.263	127.1
14	4301.288	42.788	88.3
15	4080.216	40.589	84.1
16	3632.579	36.136	133.1

Plot file: 565564-2_peaks

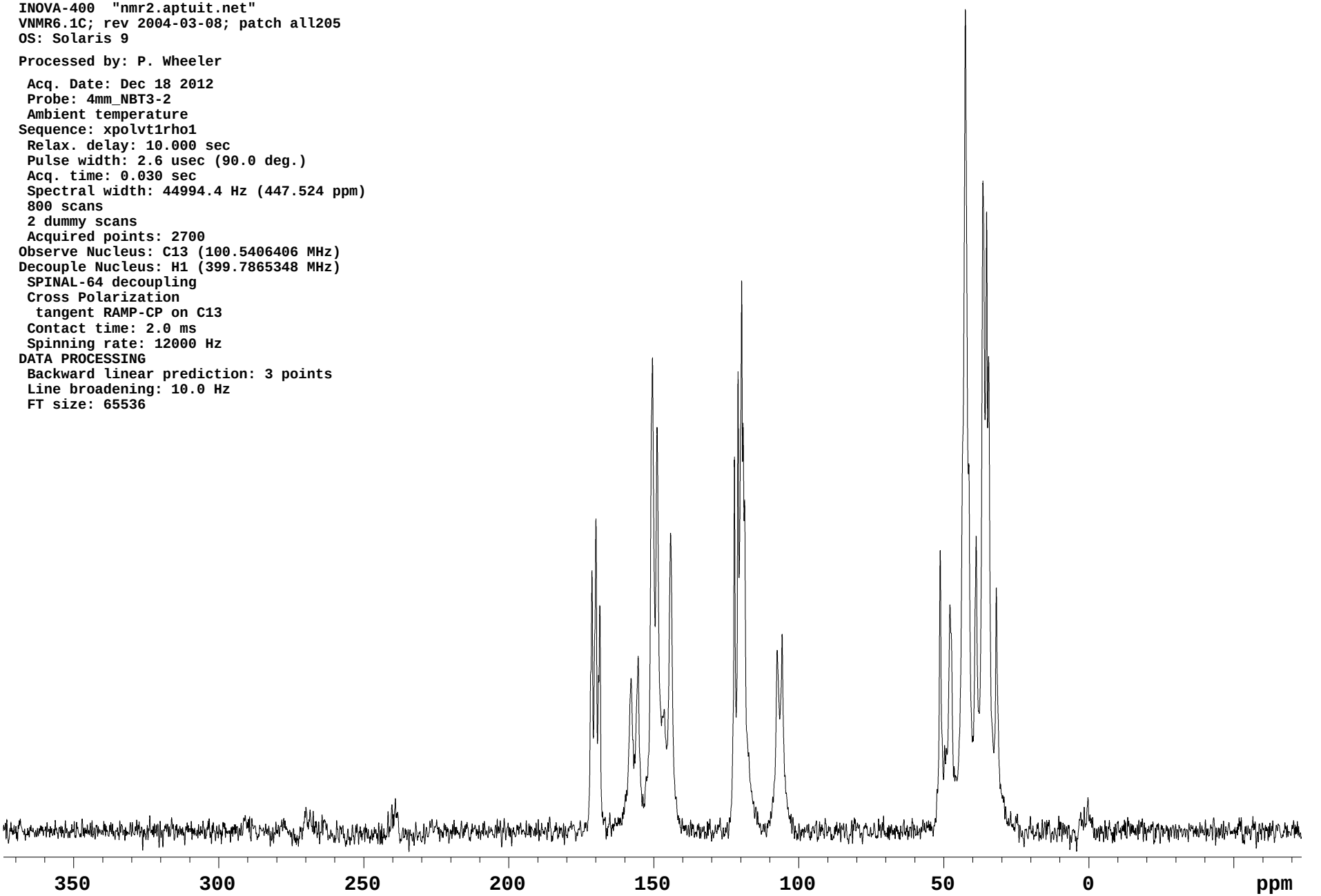
314783, 5135-17-01, Compound 184, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565565

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

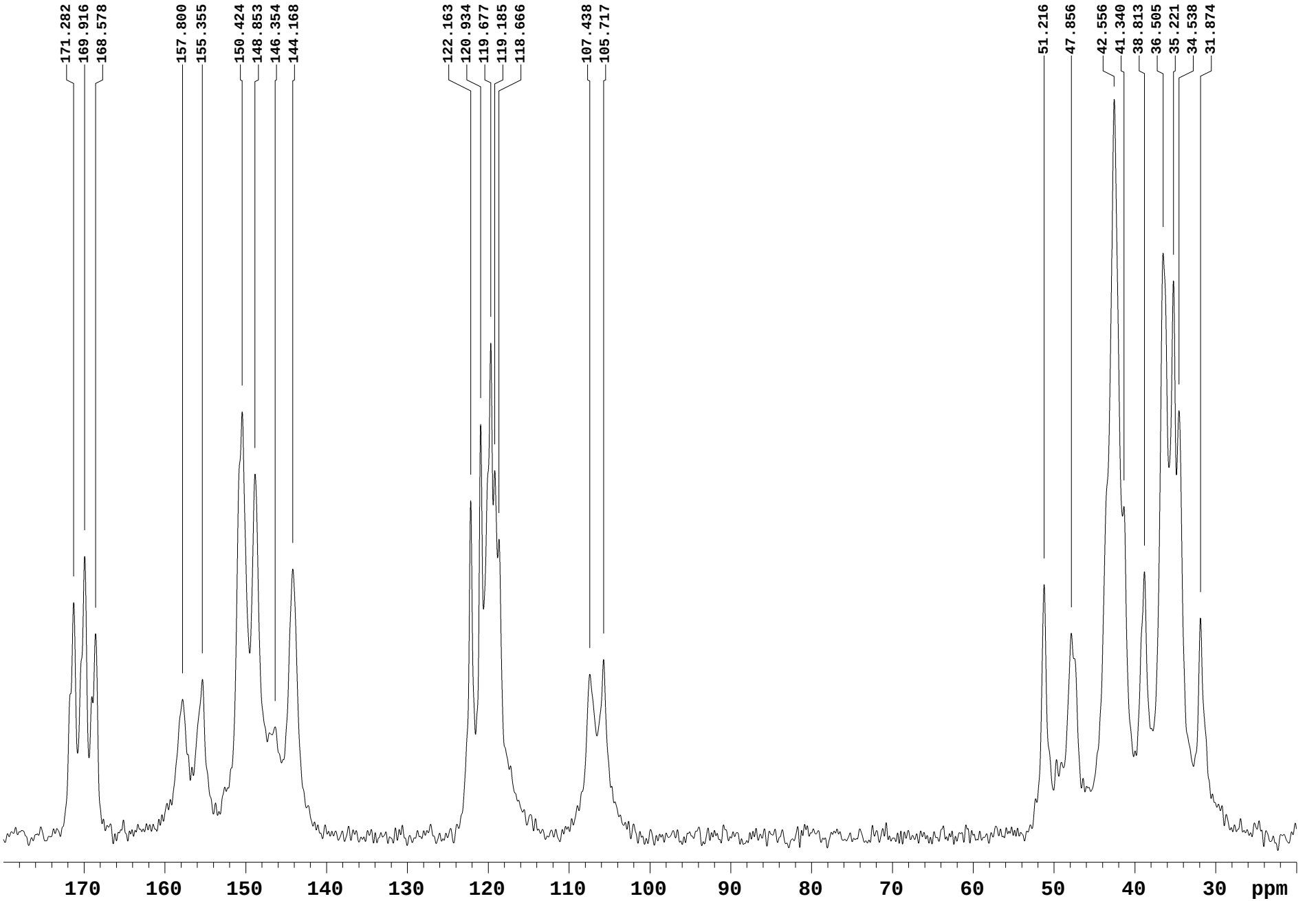
Acq. Date: Dec 18 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 10.000 sec
Pulse width: 2.6 usec (90.0 deg.)
Acq. time: 0.030 sec
Spectral width: 44994.4 Hz (447.524 ppm)
800 scans
2 dummy scans
Acquired points: 2700
Observe Nucleus: C13 (100.5406406 MHz)
Decouple Nucleus: H1 (399.7865348 MHz)
SPINAL-64 decoupling
Cross Polarization
tangent RAMP-CP on C13
Contact time: 2.0 ms
Spinning rate: 12000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 65536



Plot file: 565565-1

314783, 5135-17-01, Compound 184, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565565



Plot file: 565565-2

314783, 5135-17-01, Compound 184, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565565

INDEX	FREQUENCY	PPM	HEIGHT
1	17218.222	171.282	45.0
2	17080.910	169.916	53.9
3	16946.345	168.578	38.9
4	15862.953	157.800	26.4
5	15617.165	155.355	30.2
6	15121.469	150.424	81.7
7	14963.560	148.853	69.7
8	14712.279	146.354	21.0
9	14492.580	144.168	51.4
10	12280.485	122.163	64.6
11	12156.904	120.934	79.3
12	12030.577	119.677	94.9
13	11981.145	119.185	70.4
14	11928.966	118.666	57.2
15	10800.262	107.438	31.2
16	10627.249	105.717	34.0
17	5148.503	51.216	48.4
18	4810.715	47.856	39.1
19	4277.945	42.556	141.8
20	4155.738	41.340	63.4
21	3901.710	38.813	50.9
22	3669.653	36.505	112.2
23	3540.580	35.221	106.8
24	3471.924	34.538	81.9
25	3204.166	31.874	42.0

Plot file: 565565-2_peaks

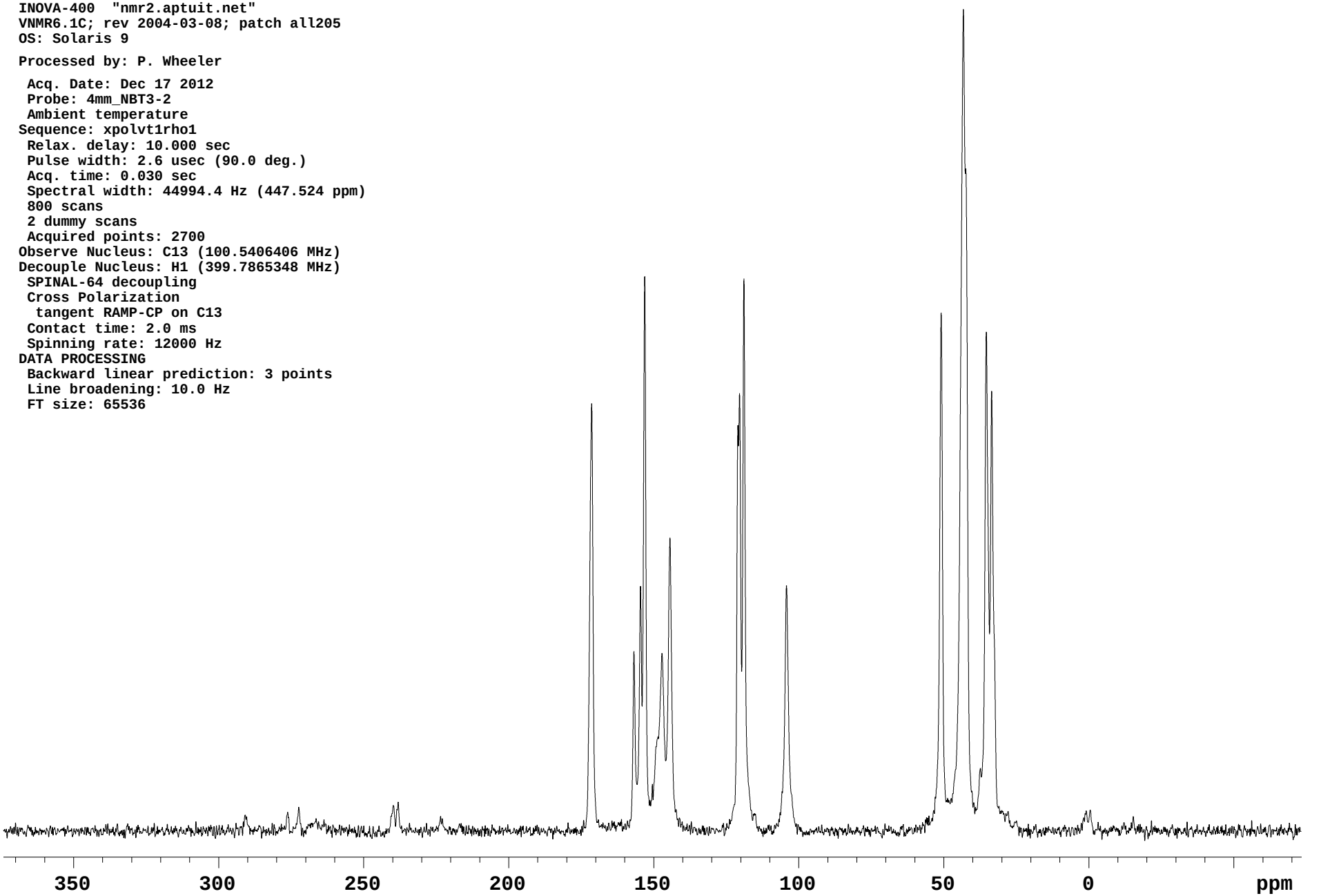
308389, Compound 184, Lot LB-1017, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565613

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

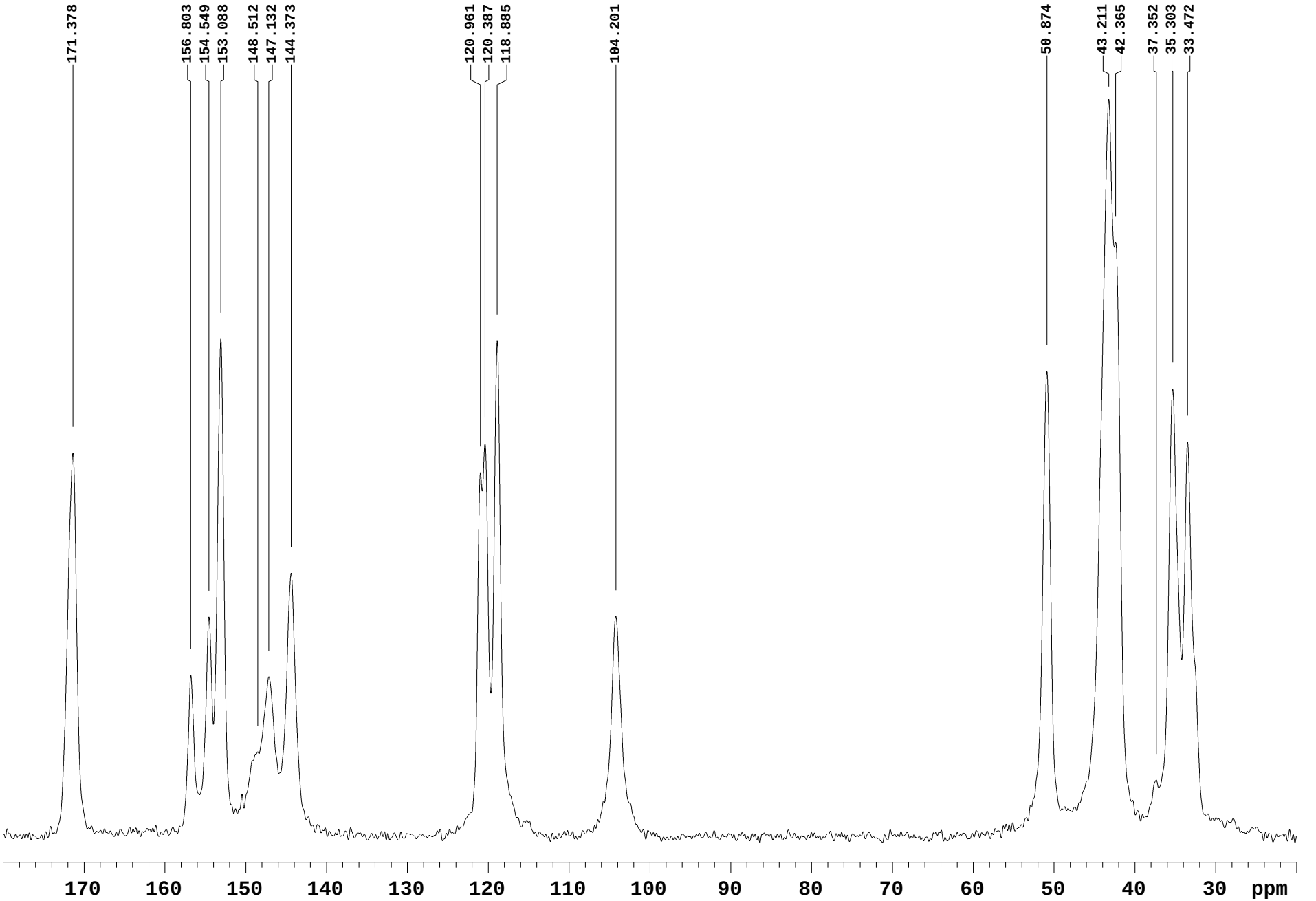
Acq. Date: Dec 17 2012
Probe: 4mm_NBT3-2
Ambient temperature
Sequence: xpolvt1rho1
Relax. delay: 10.000 sec
Pulse width: 2.6 usec (90.0 deg.)
Acq. time: 0.030 sec
Spectral width: 44994.4 Hz (447.524 ppm)
800 scans
2 dummy scans
Acquired points: 2700
Observe Nucleus: C13 (100.5406406 MHz)
Decouple Nucleus: H1 (399.7865348 MHz)
SPINAL-64 decoupling
Cross Polarization
tangent RAMP-CP on C13
Contact time: 2.0 ms
Spinning rate: 12000 Hz
DATA PROCESSING
Backward linear prediction: 3 points
Line broadening: 10.0 Hz
FT size: 65536



Plot file: 565613-1

308389, Compound 184, Lot LB-1017, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

File: 565613



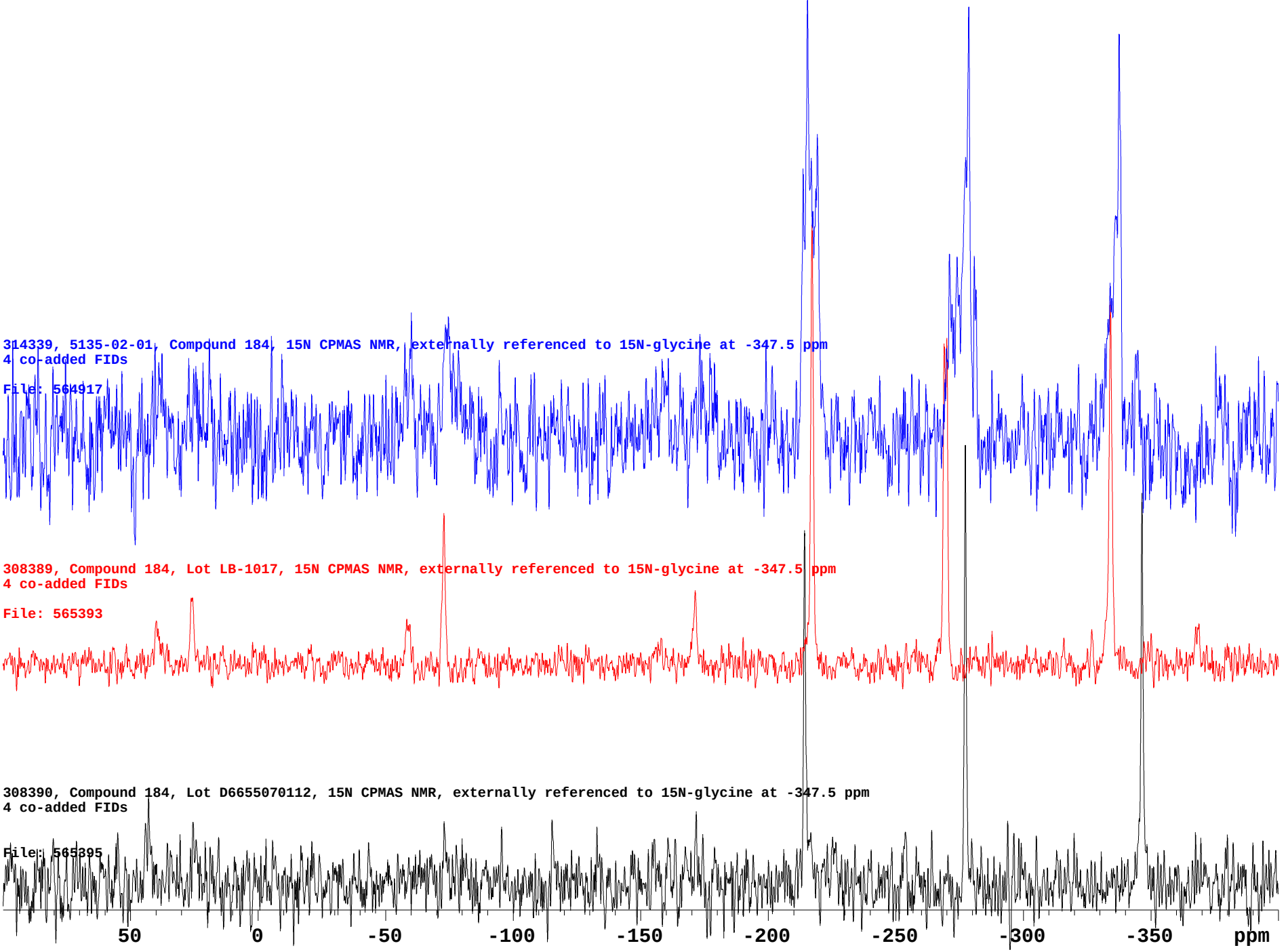
Plot file: 565613-2

308389, Compound 184, Lot LB-1017, 13C CPMAS NMR, externally referenced to glycine at 176.5 ppm

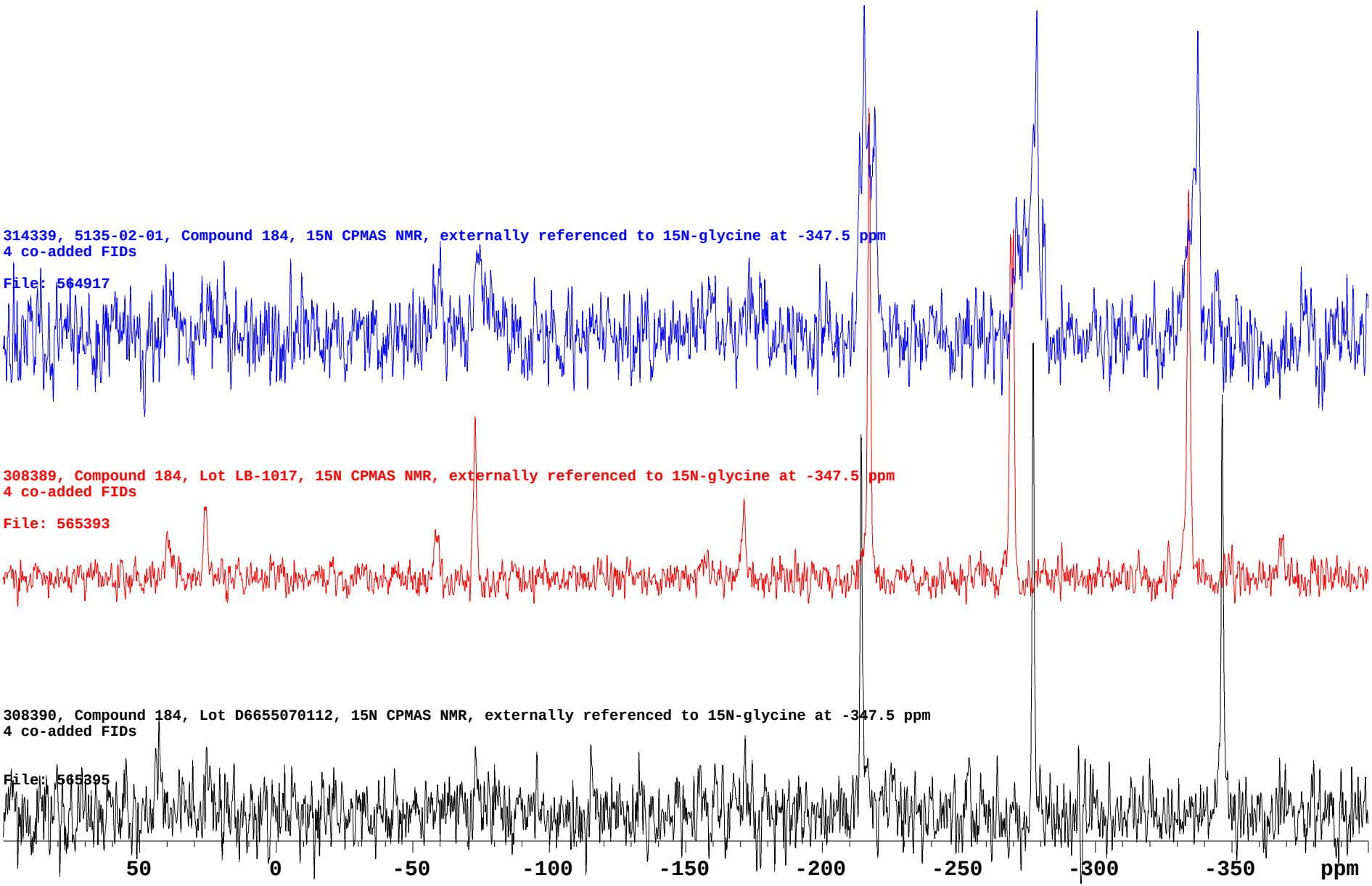
File: 565613

INDEX	FREQUENCY	PPM	HEIGHT
1	17227.834	171.378	73.7
2	15762.716	156.803	31.0
3	15536.151	154.549	42.2
4	15389.227	153.088	95.7
5	14929.232	148.512	16.3
6	14790.547	147.132	30.7
7	14513.177	144.373	50.6
8	12159.651	120.961	70.0
9	12101.979	120.387	75.5
10	11950.936	118.885	95.3
11	10474.833	104.201	42.3
12	5114.175	50.874	89.4
13	4343.855	43.211	141.8
14	4258.722	42.365	114.2
15	3754.787	37.352	10.8
16	3548.819	35.303	86.1
17	3364.821	33.472	75.9

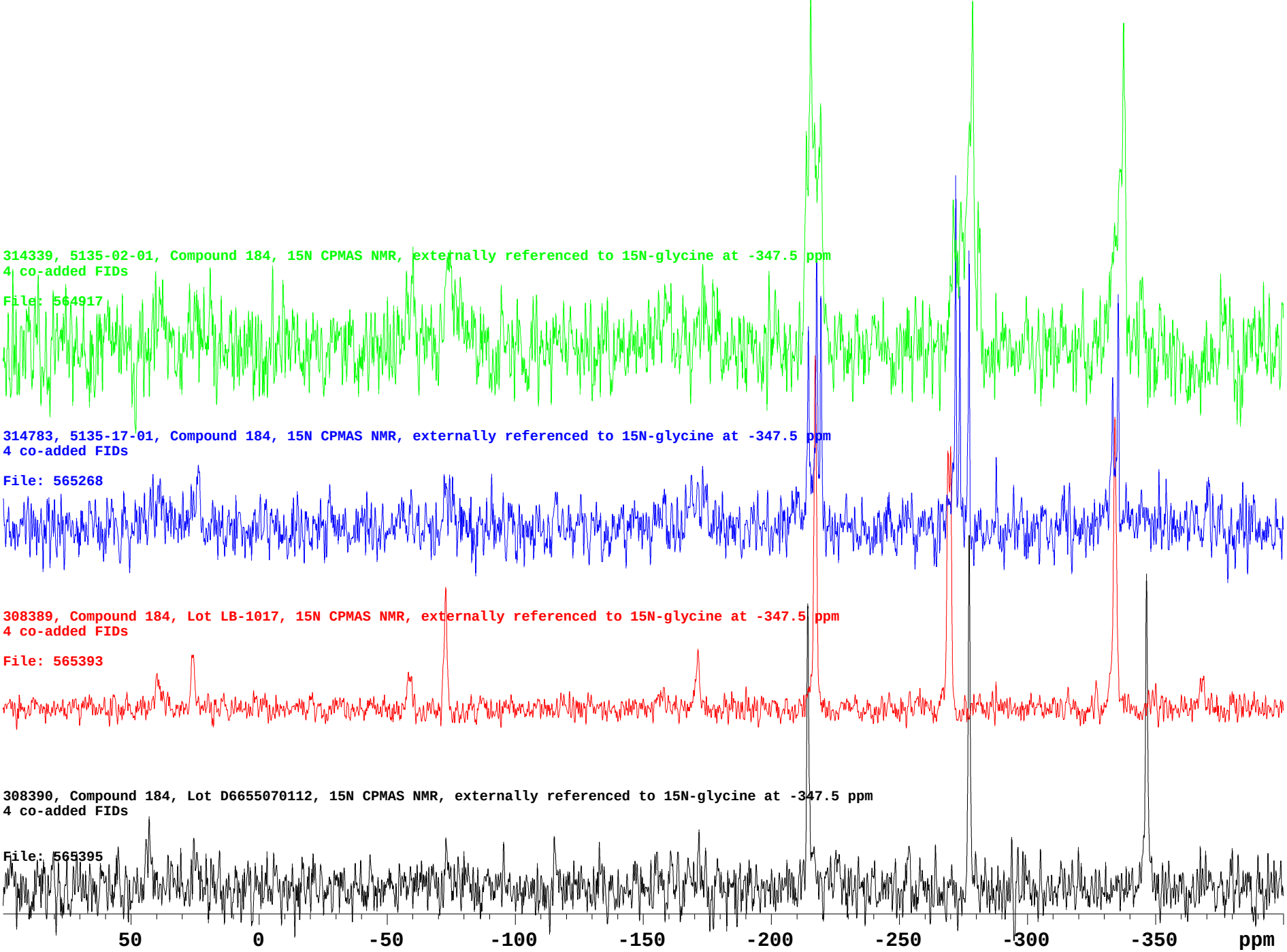
Plot file: 565613-2_peaks



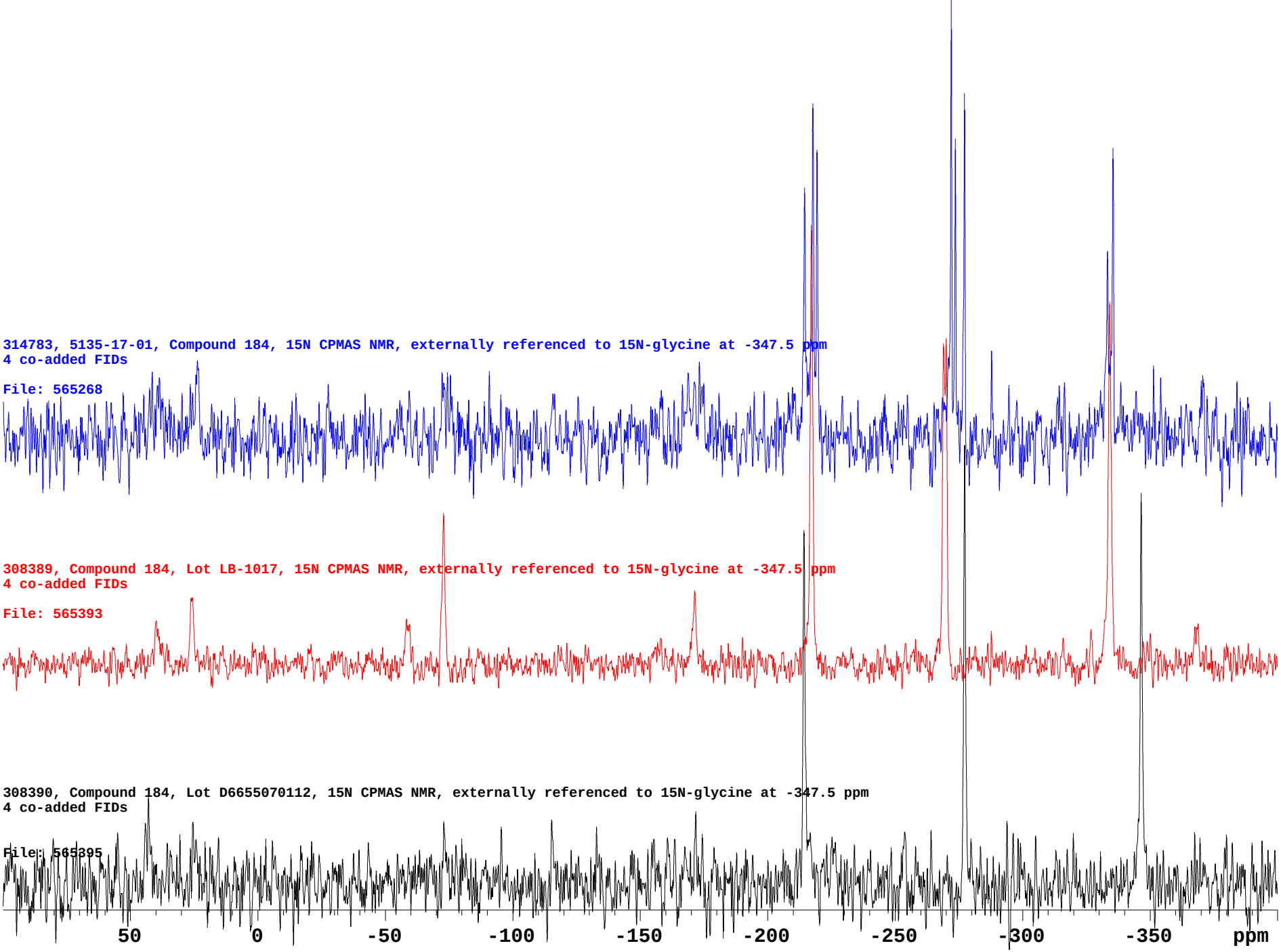
Plot file: overlay1



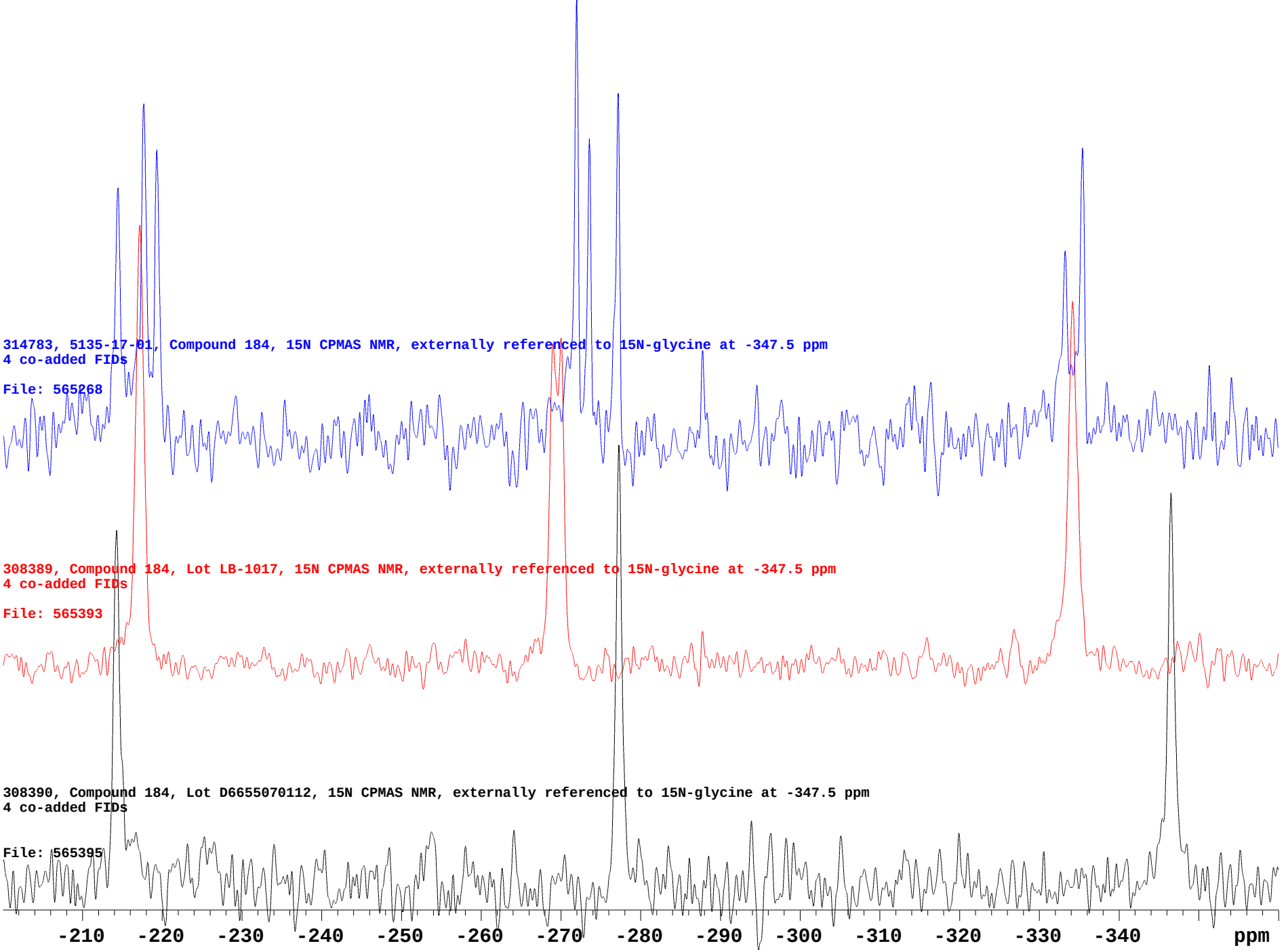
Plot file: overlay2



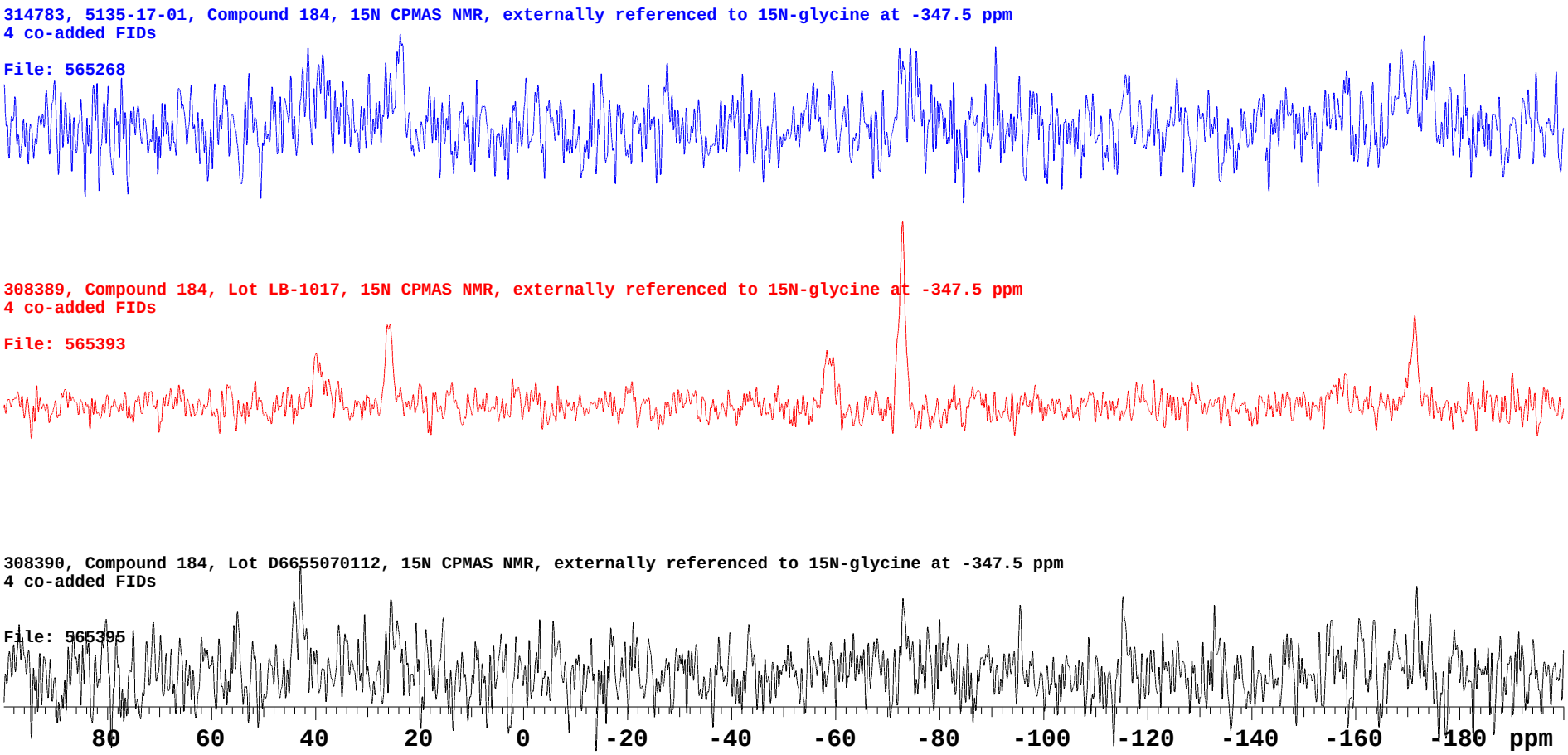
Plot file: overlay3



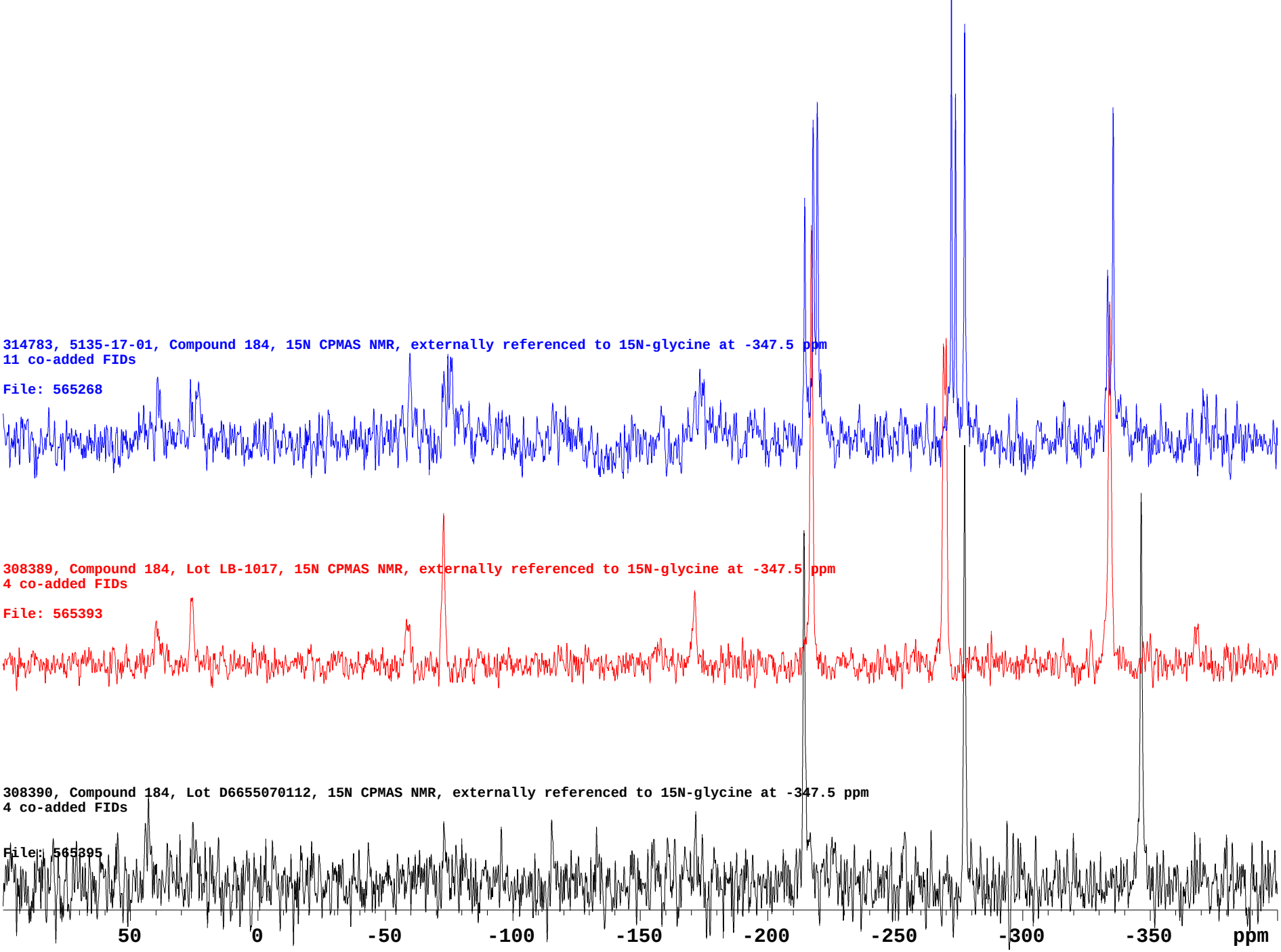
Plot file: overlay4



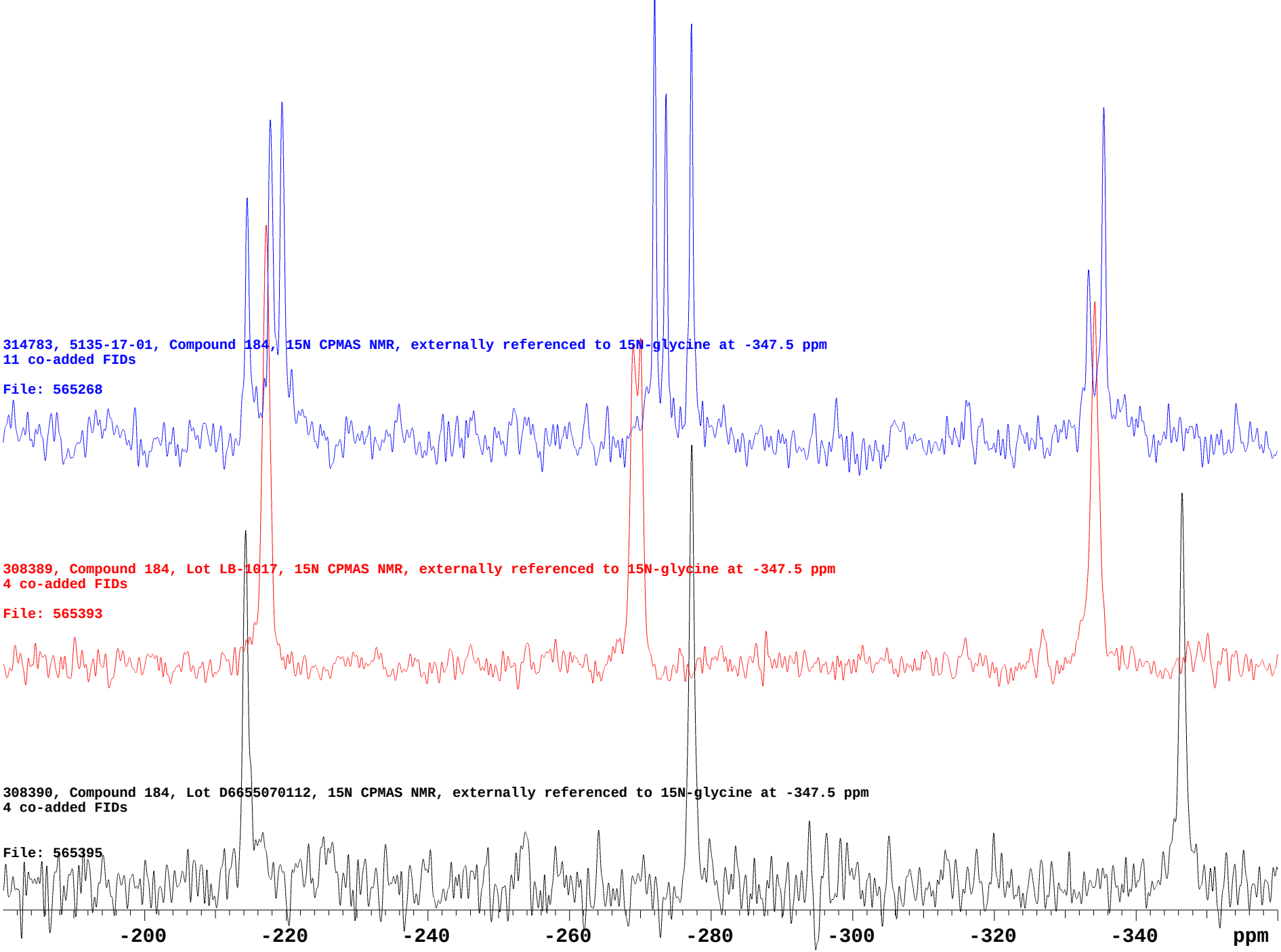
Plot file: overlay5



Plot file: overlay6



Plot file: overlay7



Plot file: overlay8

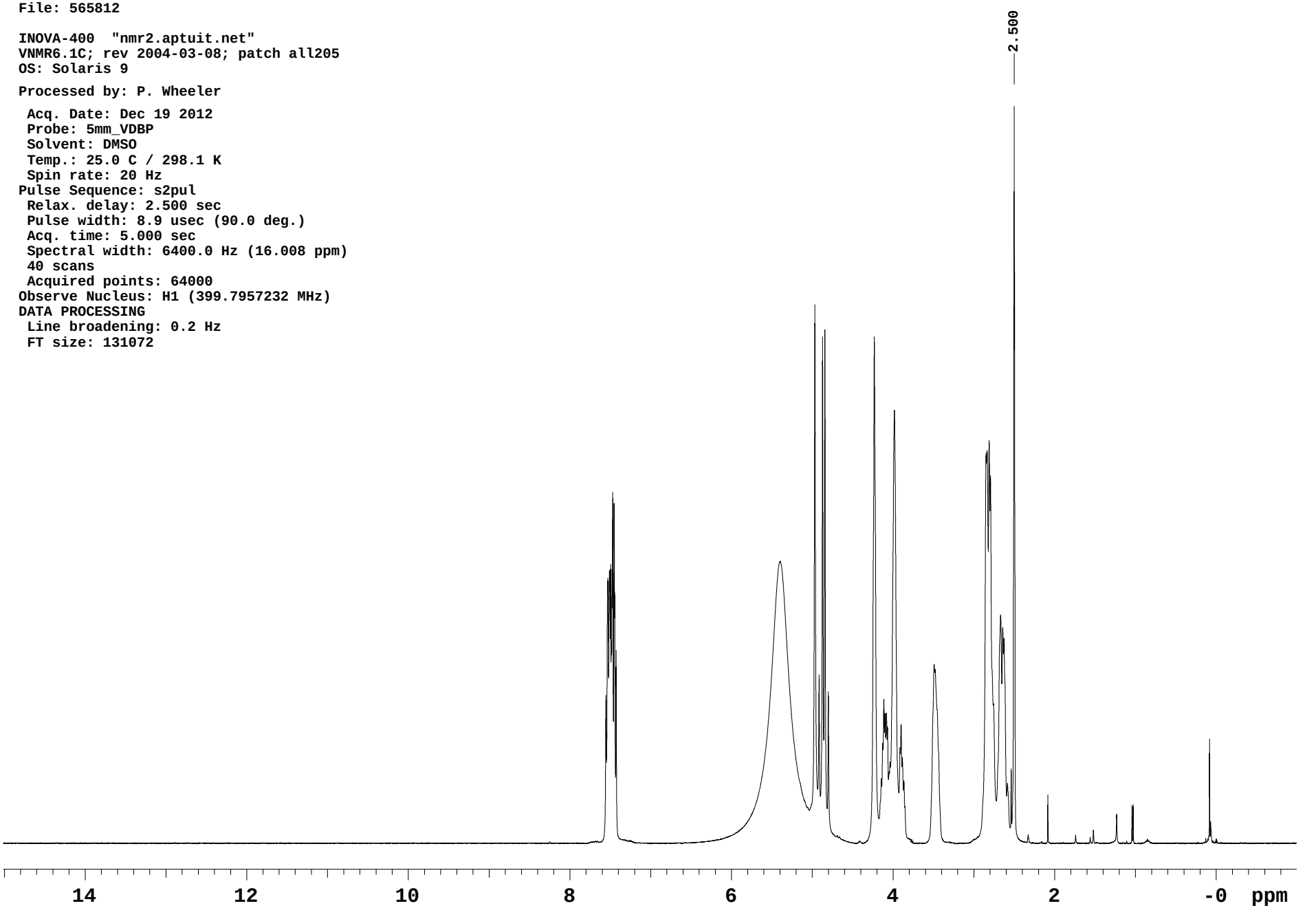
314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

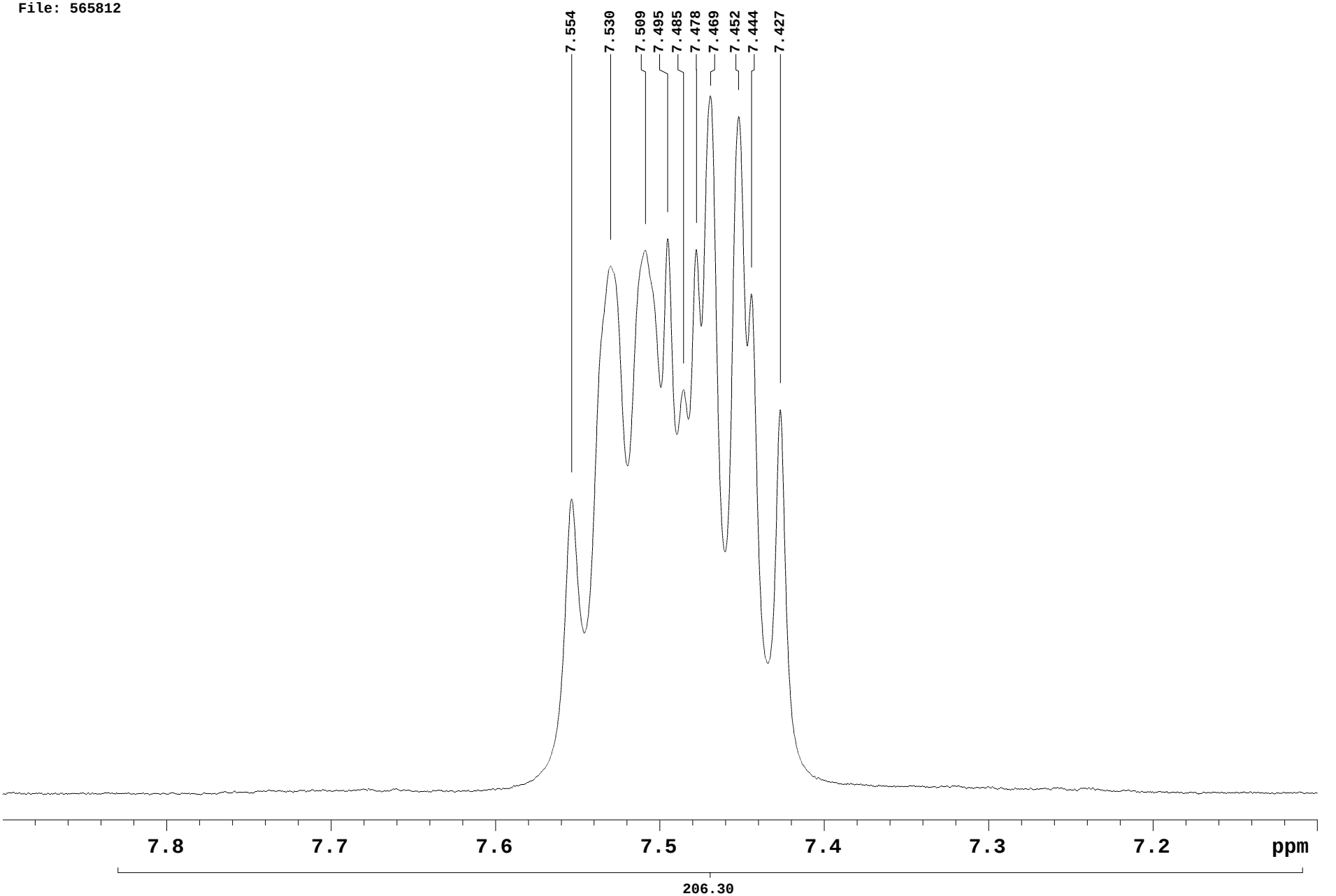
Acq. Date: Dec 19 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 2.500 sec
Pulse width: 8.9 usec (90.0 deg.)
Acq. time: 5.000 sec
Spectral width: 6400.0 Hz (16.008 ppm)
40 scans
Acquired points: 64000
Observe Nucleus: H1 (399.7957232 MHz)
DATA PROCESSING
Line broadening: 0.2 Hz
FT size: 131072



Plot file: 565812-1

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

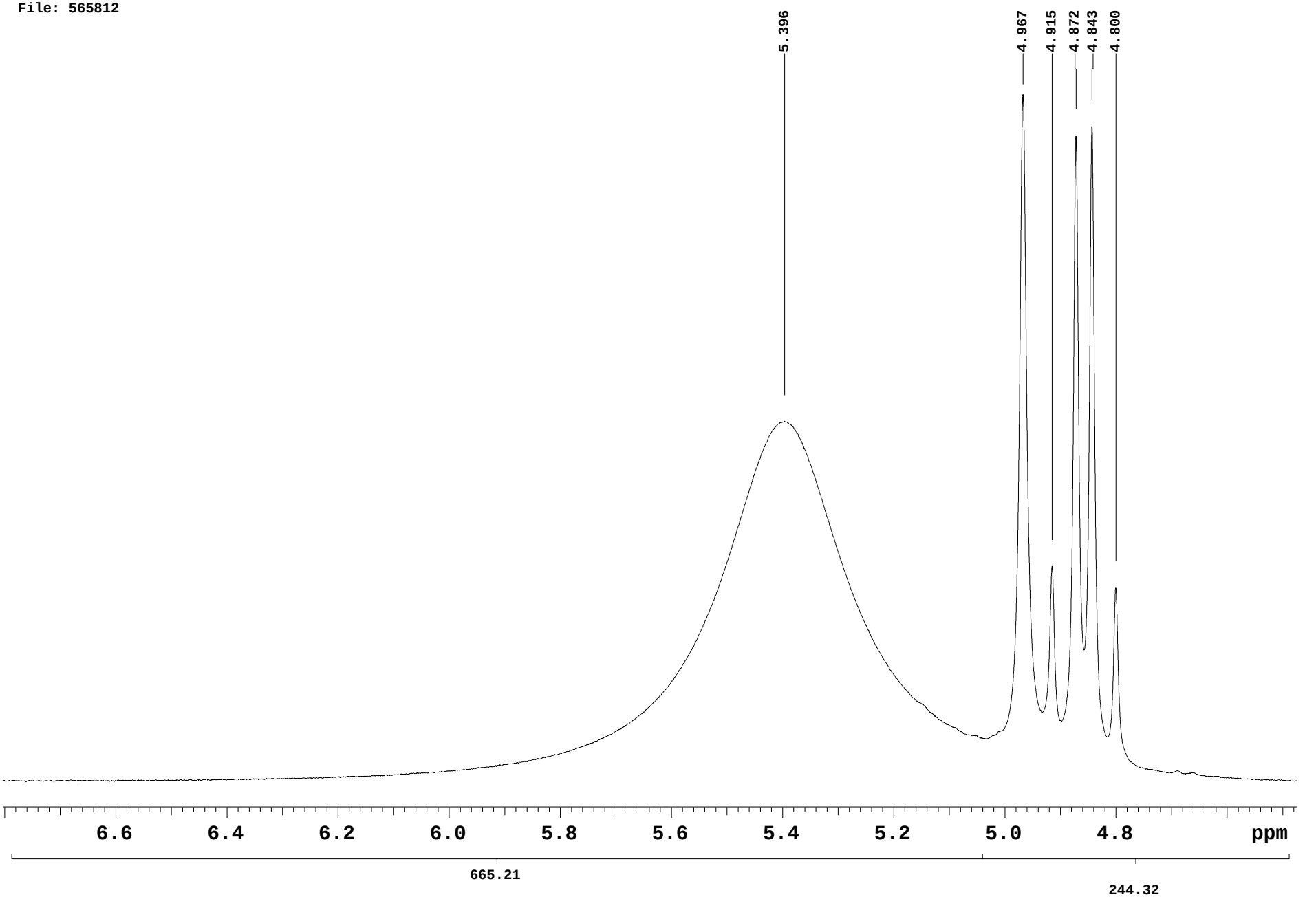
File: 565812



Plot file: 565812-2

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

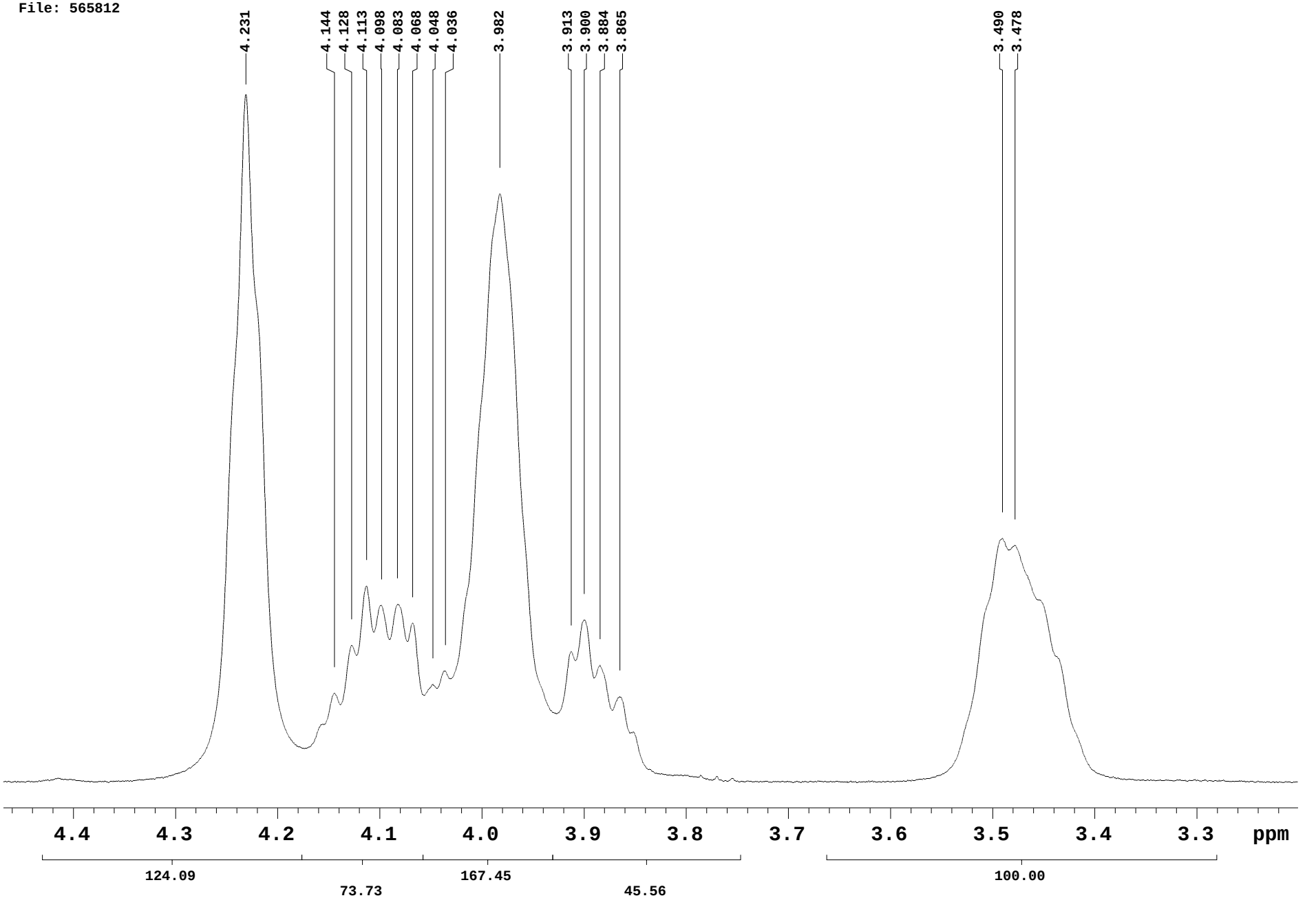
File: 565812



Plot file: 565812-3

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

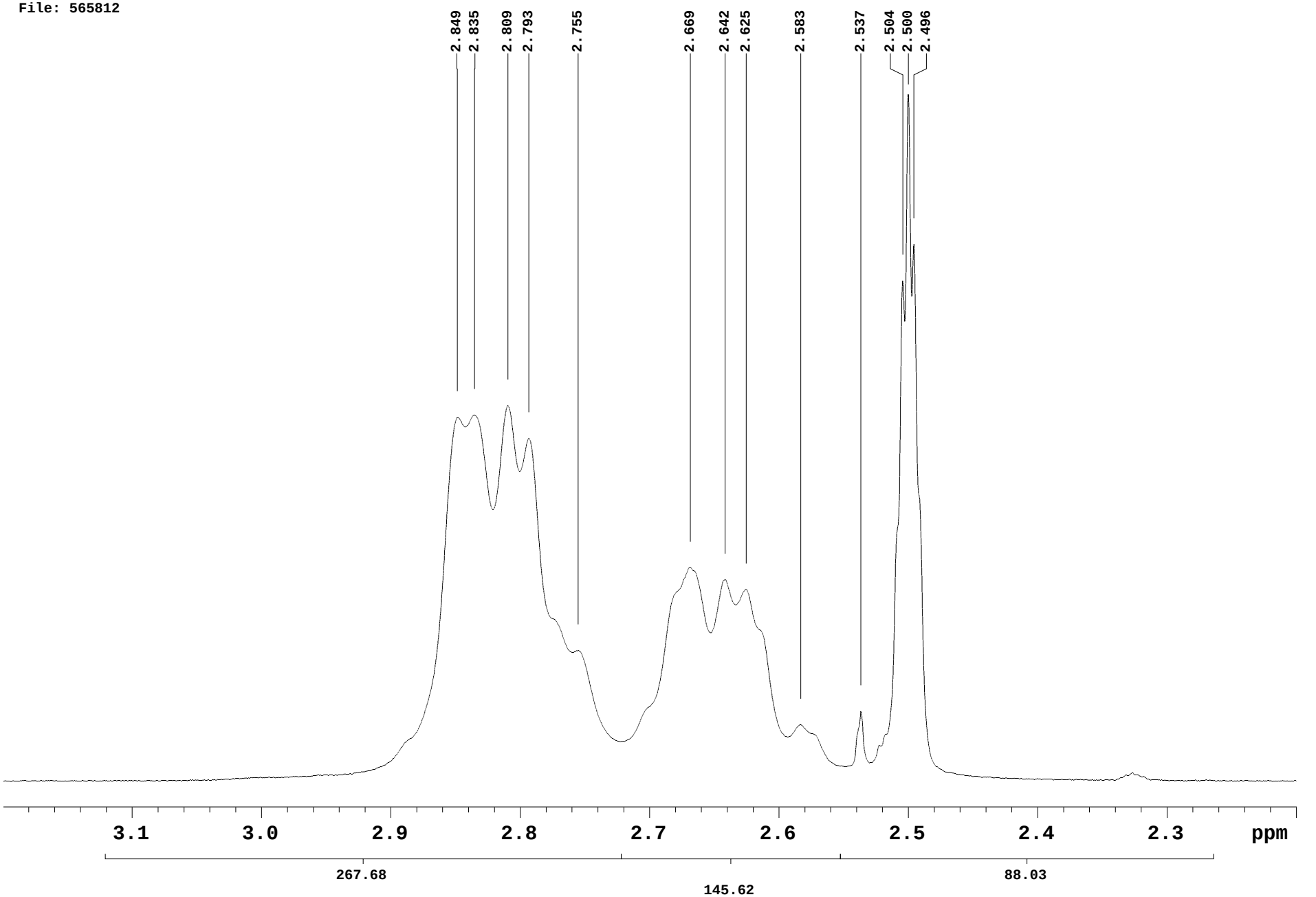
File: 565812



Plot file: 565812-4

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

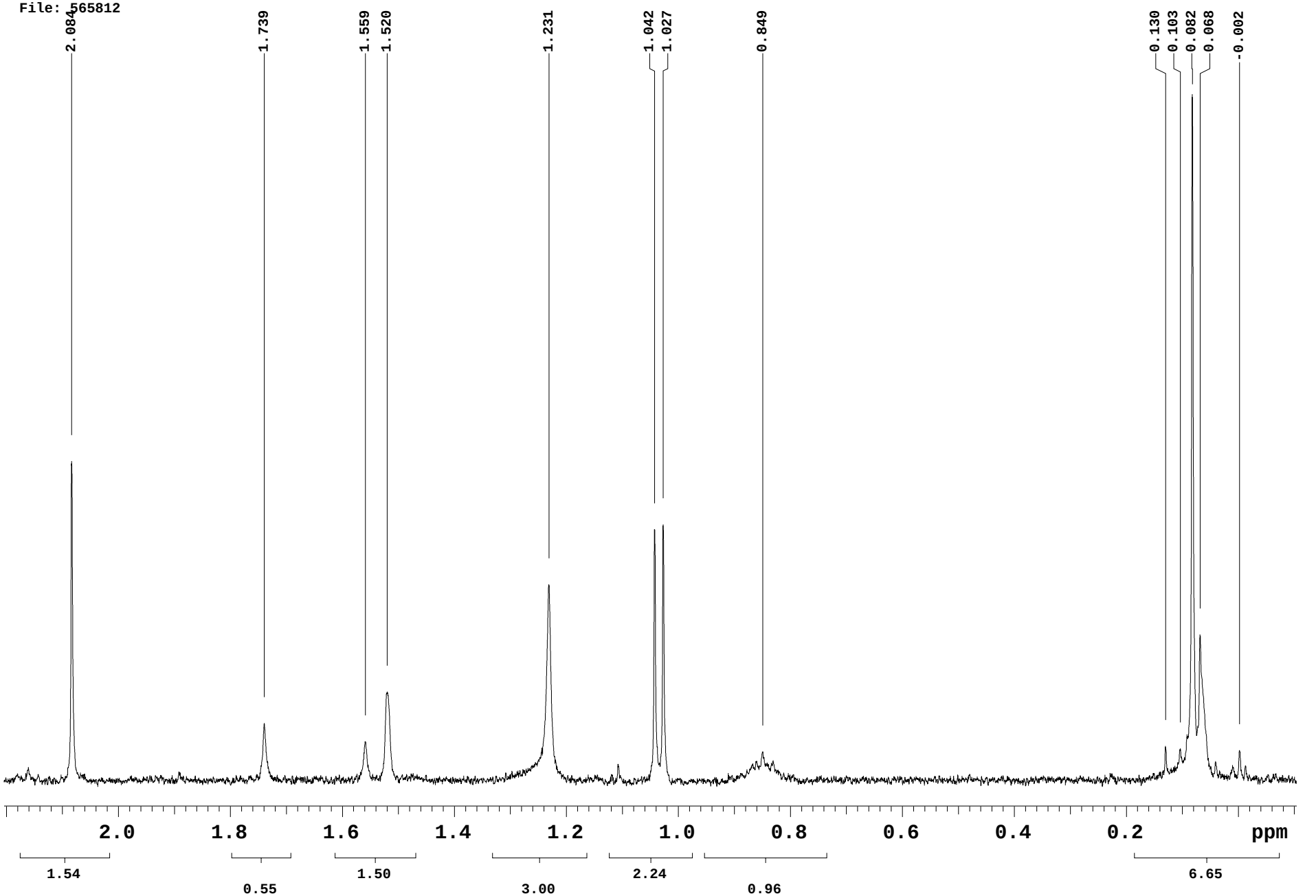
File: 565812



Plot file: 565812-5

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812



Plot file: 565812-6

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812

INDEX	FREQUENCY	PPM	HEIGHT
1	999.482	2.500	141.8

Plot file: 565812-1_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812

INDEX	FREQUENCY	PPM	HEIGHT
1	3019.892	7.554	55.8
2	3010.420	7.530	99.8
3	3001.924	7.509	102.8
4	2996.553	7.495	105.0
5	2992.646	7.485	76.4
6	2989.521	7.478	102.9
7	2986.103	7.469	132.0
8	2979.267	7.452	128.1
9	2976.142	7.444	94.5
10	2969.111	7.427	72.6

Plot file: 565812-2_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812

INDEX	FREQUENCY	PPM	HEIGHT
1	2157.392	5.396	69.2
2	1985.810	4.967	132.0
3	1964.912	4.915	41.3
4	1947.724	4.872	124.1
5	1936.201	4.843	125.9
6	1919.014	4.800	37.2

Plot file: 565812-3_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812

INDEX	FREQUENCY	PPM	HEIGHT
1	1691.572	4.231	132.0
2	1656.904	4.144	17.0
3	1650.166	4.128	26.2
4	1644.307	4.113	37.6
5	1638.447	4.098	33.9
6	1632.197	4.083	34.1
7	1626.240	4.068	30.5
8	1618.330	4.048	18.7
9	1613.447	4.036	21.3
10	1592.158	3.982	112.9
11	1564.228	3.913	25.0
12	1559.150	3.900	31.1
13	1552.900	3.884	22.4
14	1545.185	3.865	16.4
15	1395.381	3.490	46.7
16	1390.498	3.478	45.4

Plot file: 565812-4_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812

INDEX	FREQUENCY	PPM	HEIGHT
1	1138.838	2.849	69.9
2	1133.564	2.835	70.4
3	1123.213	2.809	72.2
4	1116.767	2.793	65.9
5	1101.533	2.755	25.1
6	1066.865	2.669	41.0
7	1056.123	2.642	38.7
8	1049.580	2.625	36.8
9	1032.685	2.583	10.8
10	1014.131	2.537	13.4
11	1001.142	2.504	96.2
12	999.482	2.500	132.0
13	997.724	2.496	103.2

Plot file: 565812-5_peaks

314783, 5135-17-01, Compound 184, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565812

INDEX	FREQUENCY	PPM	HEIGHT
1	832.978	2.084	61.4
2	695.381	1.739	11.0
3	623.115	1.559	7.5
4	607.588	1.520	17.0
5	492.158	1.231	37.7
6	416.767	1.042	48.3
7	410.615	1.027	49.2
8	339.424	0.849	5.5
9	51.924	0.130	6.5
10	41.279	0.103	6.1
11	32.783	0.082	132.0
12	27.217	0.068	28.0
13	-0.908	-0.002	5.7

Plot file: 565812-6_peaks

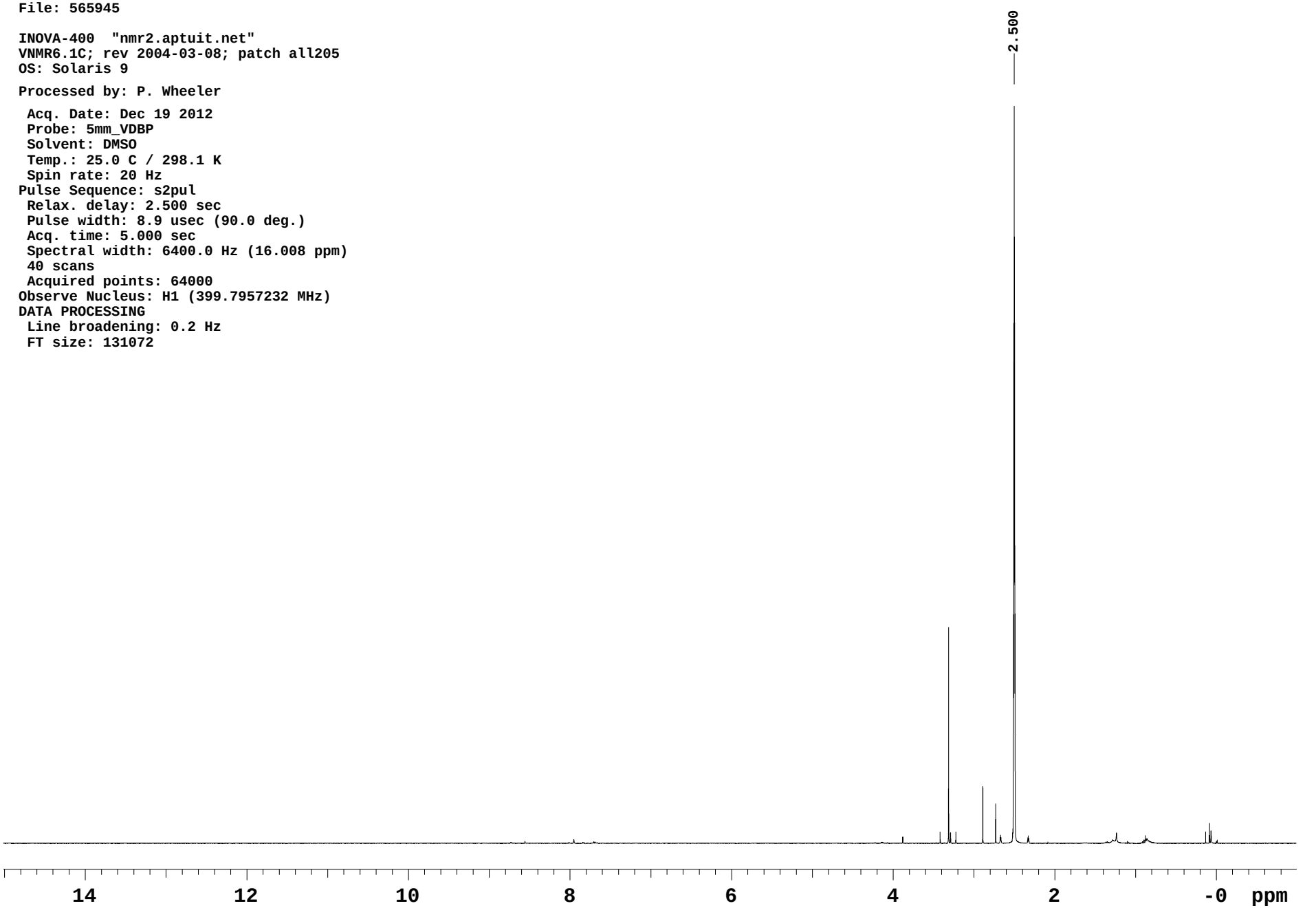
314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565945

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

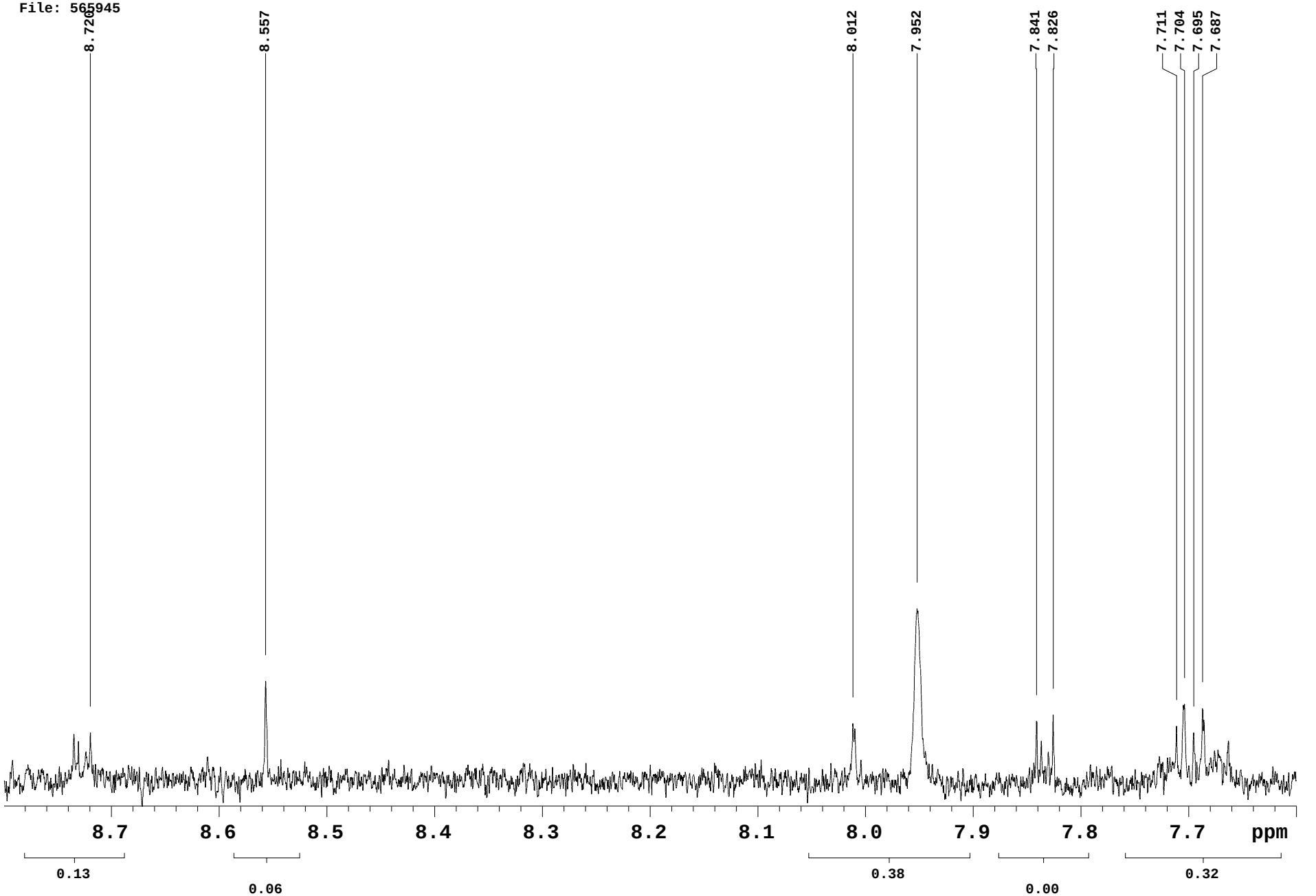
Acq. Date: Dec 19 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 2.500 sec
Pulse width: 8.9 usec (90.0 deg.)
Acq. time: 5.000 sec
Spectral width: 6400.0 Hz (16.008 ppm)
40 scans
Acquired points: 64000
Observe Nucleus: H1 (399.7957232 MHz)
DATA PROCESSING
Line broadening: 0.2 Hz
FT size: 131072



Plot file: 565945-1

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

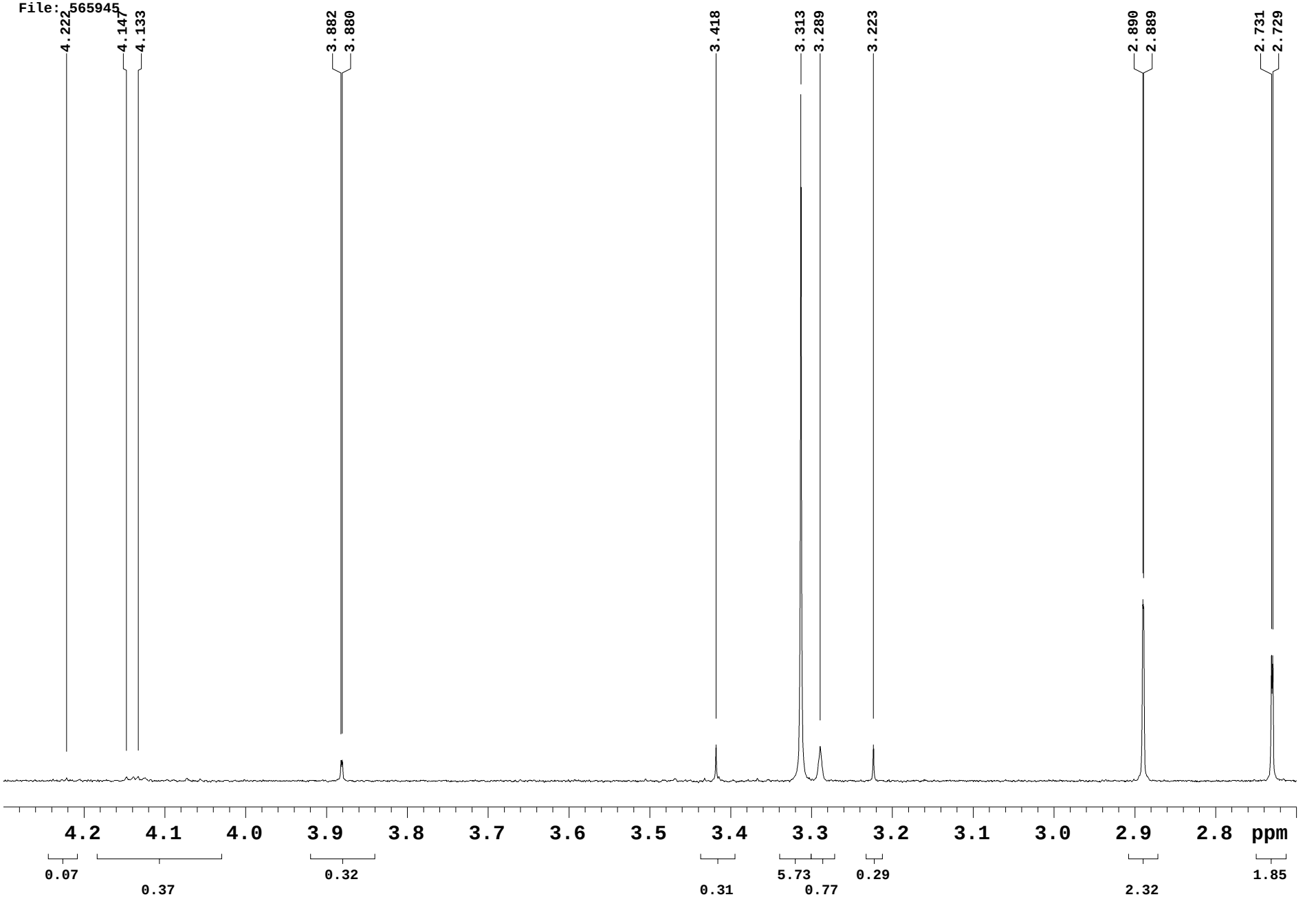
File: 565945



Plot file: 565945-2

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

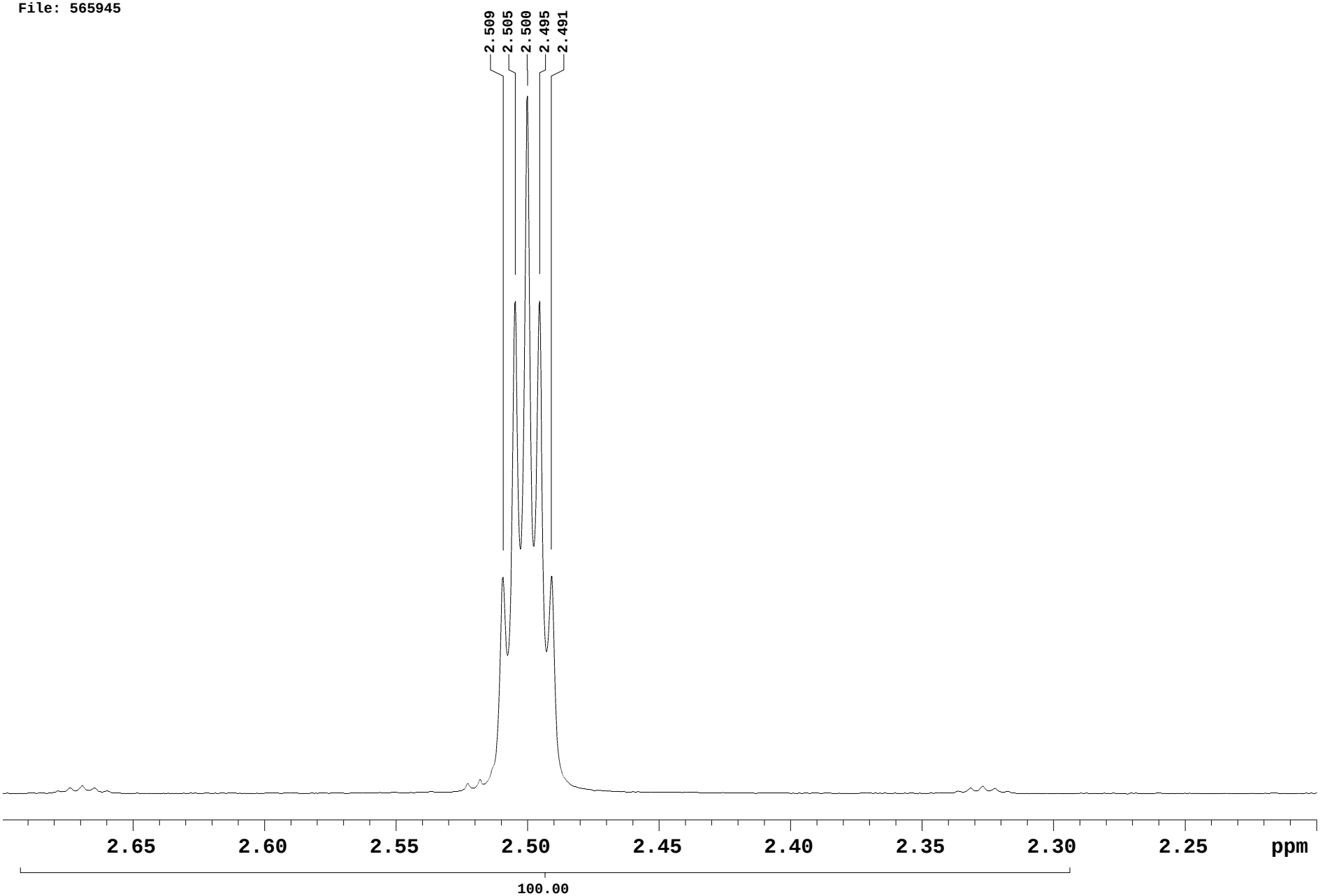
File: 565945



Plot file: 565945-3

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

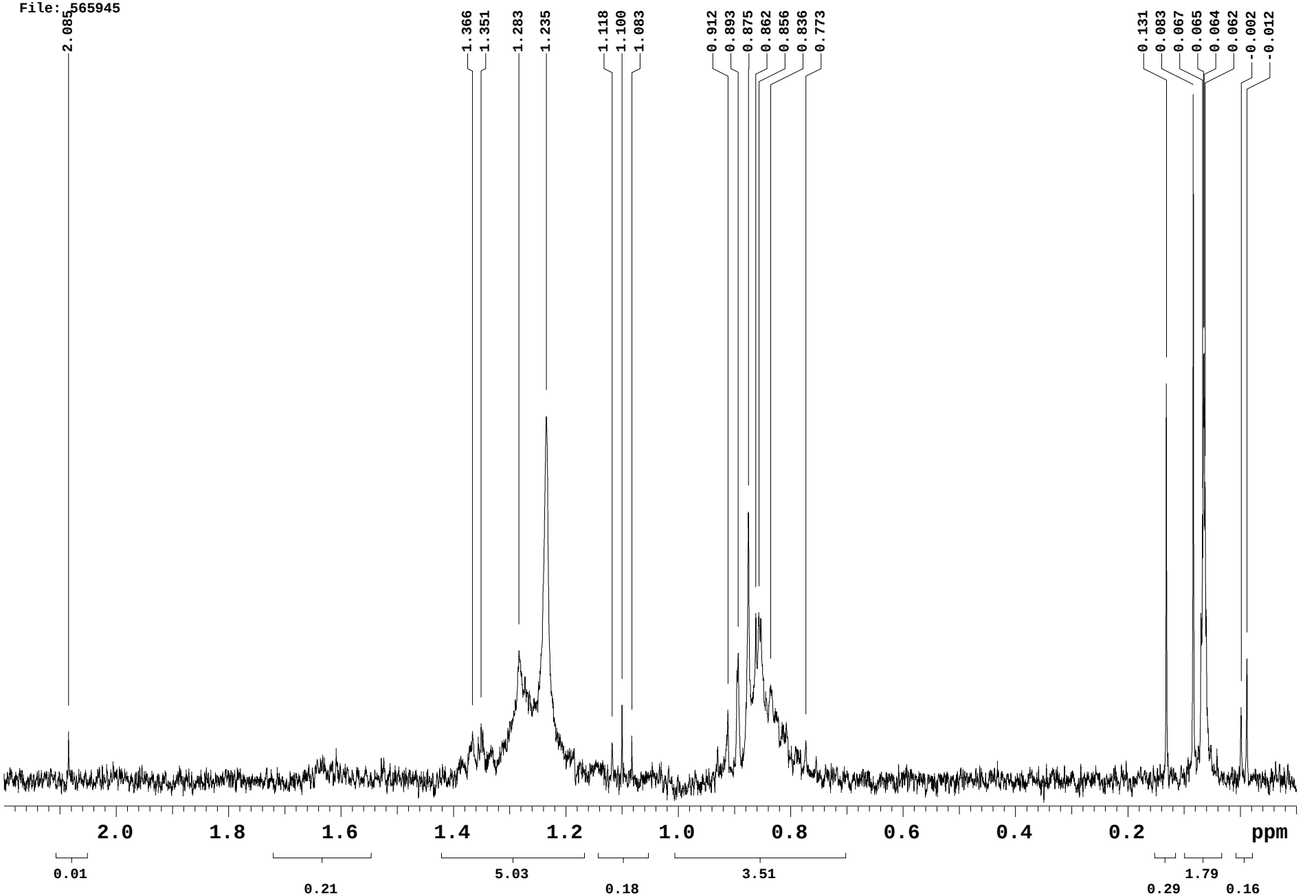
File: 565945



Plot file: 565945-4

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm
25 C

File: 565945



Plot file: 565945-5

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C

File: 565945

INDEX	FREQUENCY	PPM	HEIGHT
1	999.482	2.500	141.8

Plot file: 565945-1_peaks

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C

File: 565945

INDEX	FREQUENCY	PPM	HEIGHT
1	3486.006	8.720	9.1
2	3420.967	8.557	19.1
3	3202.998	8.012	10.9
4	3179.170	7.952	33.0
5	3134.834	7.841	11.4
6	3128.682	7.826	12.6
7	3082.881	7.711	10.4
8	3079.951	7.704	14.7
9	3076.533	7.695	9.1
10	3073.213	7.687	13.8

Plot file: 565945-2_peaks

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C

File: 565945

INDEX	FREQUENCY	PPM	HEIGHT
1	1687.764	4.222	0.6
2	1658.076	4.147	0.8
3	1652.217	4.133	0.9
4	1552.021	3.882	4.0
5	1551.338	3.880	4.1
6	1366.474	3.418	7.0
7	1324.482	3.313	132.0
8	1315.010	3.289	6.7
9	1288.642	3.223	7.0
10	1155.342	2.890	34.9
11	1155.049	2.889	34.0
12	1091.670	2.731	24.2
13	1091.084	2.729	24.1

Plot file: 565945-3_peaks

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C

File: 565945

INDEX	FREQUENCY	PPM	HEIGHT
1	1003.193	2.509	41.0
2	1001.338	2.505	93.1
3	999.482	2.500	132.0
4	997.627	2.495	93.3
5	995.869	2.491	41.1

Plot file: 565945-4_peaks

314549, Dimethyl Sulfoxide-D6, Lot 12I-403, over sieves, in DMSO-d6, 1H NMR, referenced to solvent at 2.5 ppm 25 C

File: 565945

INDEX	FREQUENCY	PPM	HEIGHT
1	833.467	2.085	9.4
2	546.064	1.366	9.4
3	540.010	1.351	10.9
4	513.057	1.283	25.0
5	493.623	1.235	70.1
6	446.846	1.118	7.2
7	439.717	1.100	14.5
8	432.783	1.083	8.6
9	364.424	0.912	13.5
10	357.099	0.893	24.5
11	349.873	0.875	51.7
12	344.599	0.862	32.1
13	342.353	0.856	32.3
14	334.053	0.836	18.4
15	308.955	0.773	7.7
16	52.510	0.131	76.3
17	33.369	0.083	132.0
18	26.826	0.067	51.1
19	26.142	0.065	82.0
20	25.654	0.064	82.2
21	24.971	0.062	57.4
22	-0.713	-0.002	14.1
23	-4.815	-0.012	23.4

Plot file: 565945-5_peaks

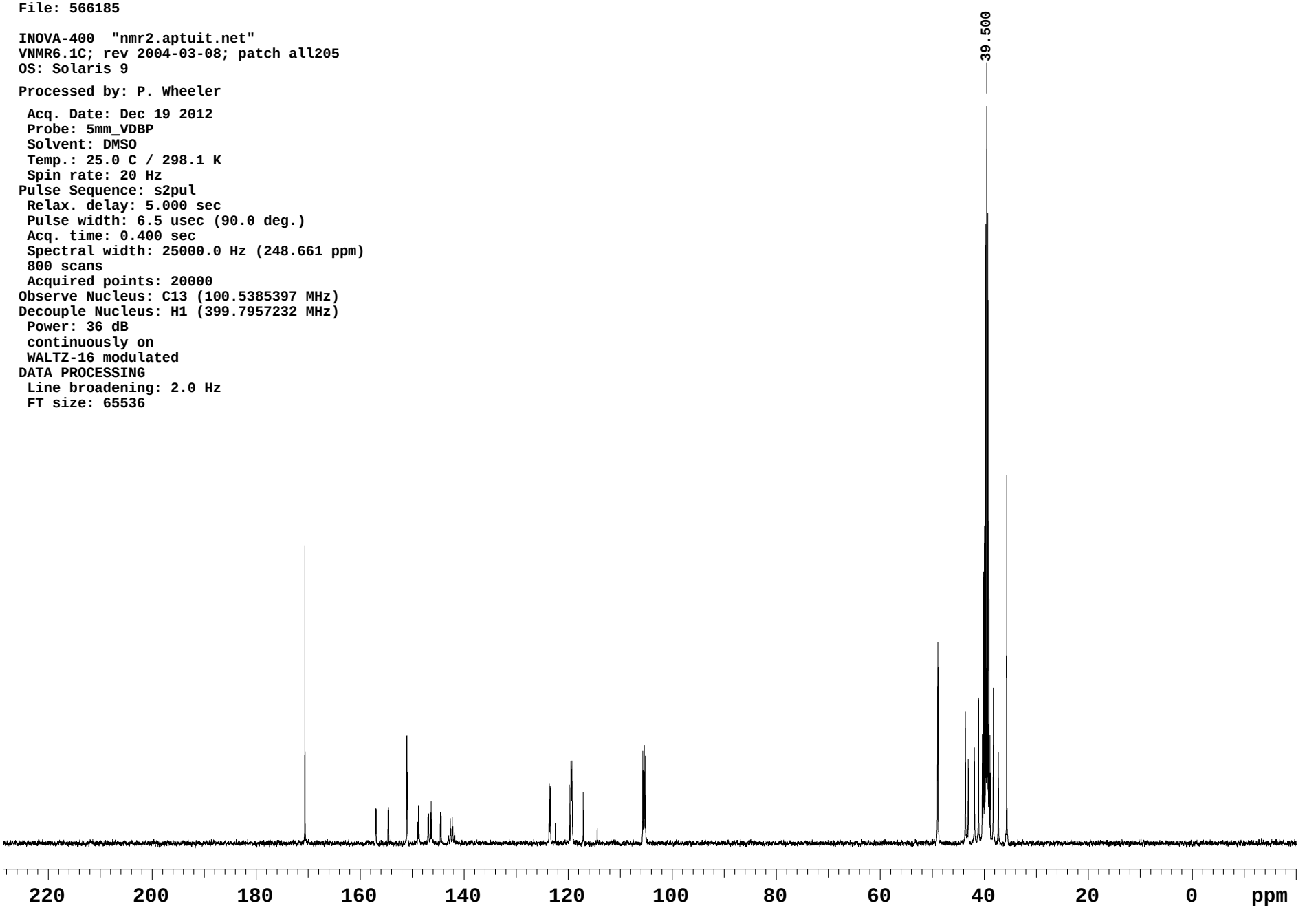
308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566185

INOVA-400 "nmr2.aptuit.net"
VNMR6.1C; rev 2004-03-08; patch all205
OS: Solaris 9

Processed by: P. Wheeler

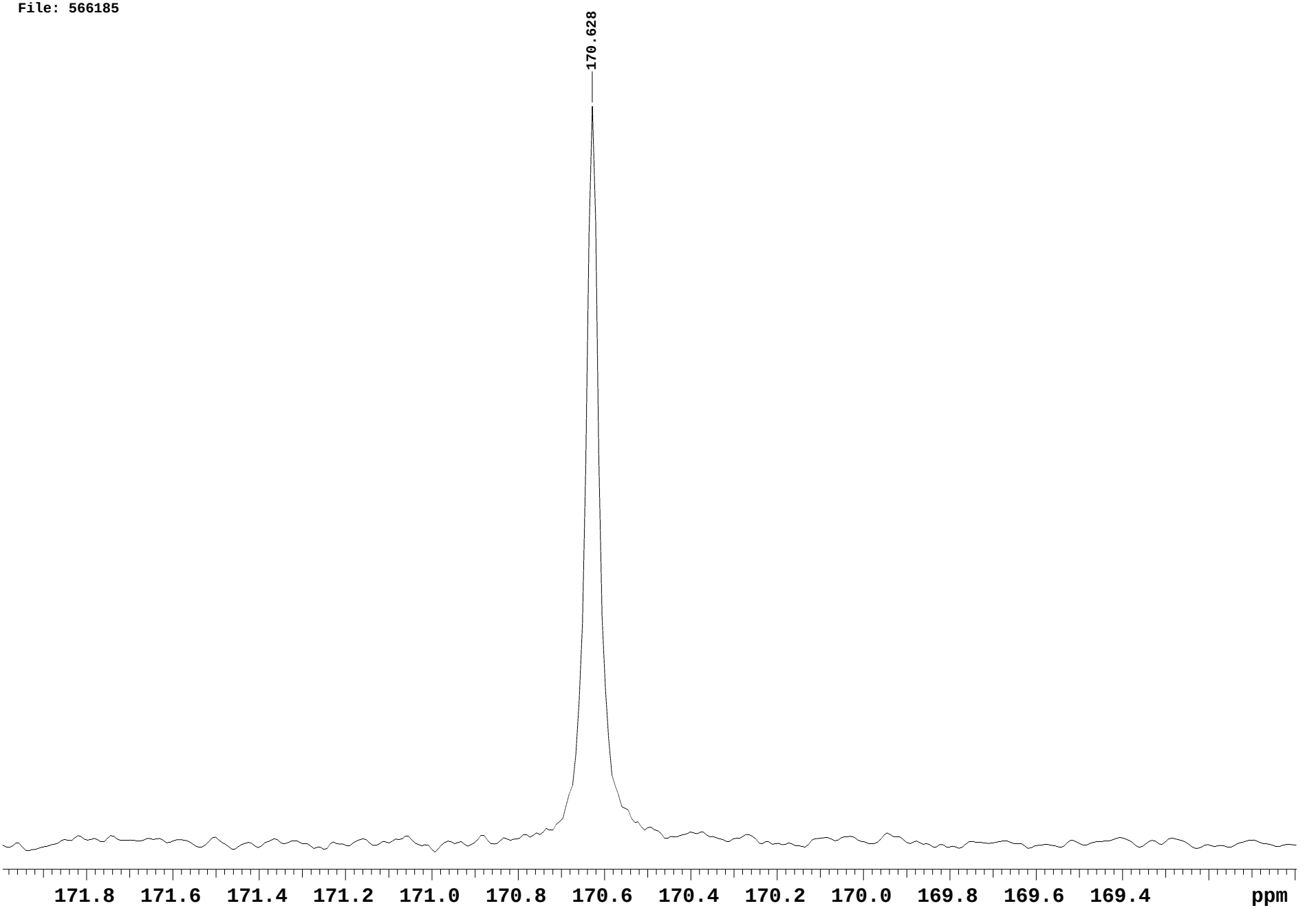
Acq. Date: Dec 19 2012
Probe: 5mm_VDBP
Solvent: DMSO
Temp.: 25.0 C / 298.1 K
Spin rate: 20 Hz
Pulse Sequence: s2pul
Relax. delay: 5.000 sec
Pulse width: 6.5 usec (90.0 deg.)
Acq. time: 0.400 sec
Spectral width: 25000.0 Hz (248.661 ppm)
800 scans
Acquired points: 20000
Observe Nucleus: C13 (100.5385397 MHz)
Decouple Nucleus: H1 (399.7957232 MHz)
Power: 36 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening: 2.0 Hz
FT size: 65536



Plot file: 566185-1

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

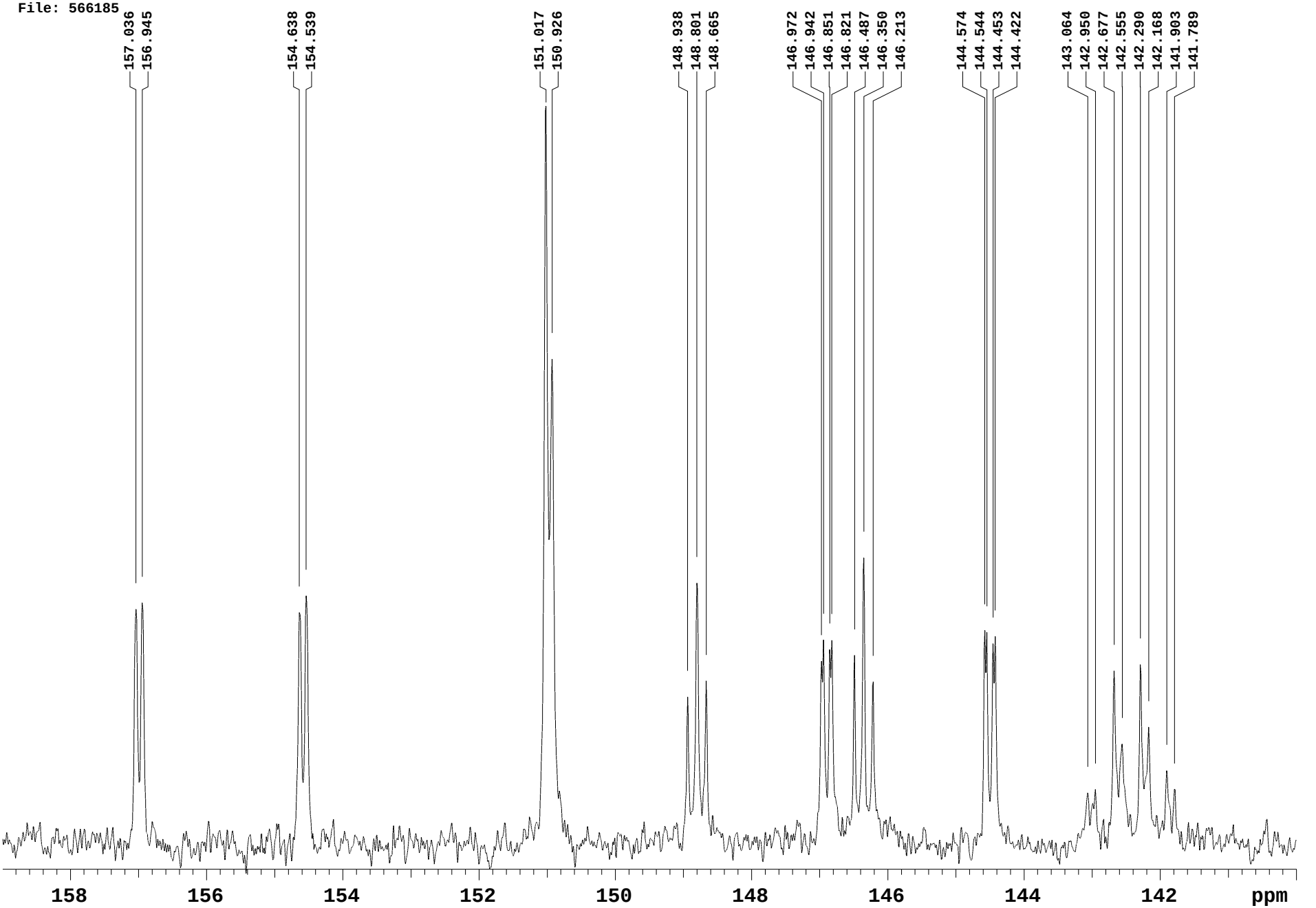
File: 566185



Plot file: 566185-2

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

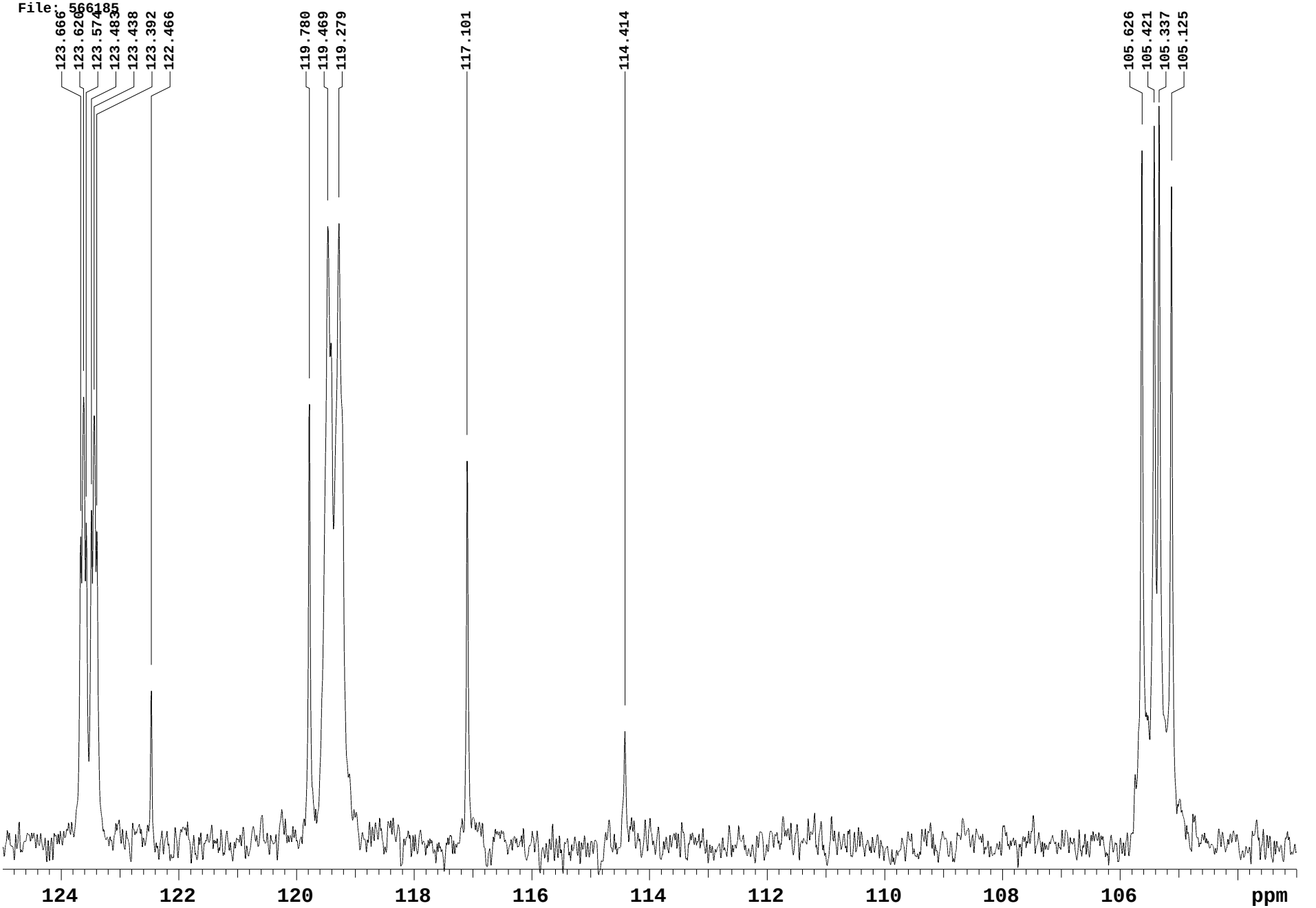
File: 566185



Plot file: 566185-3

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

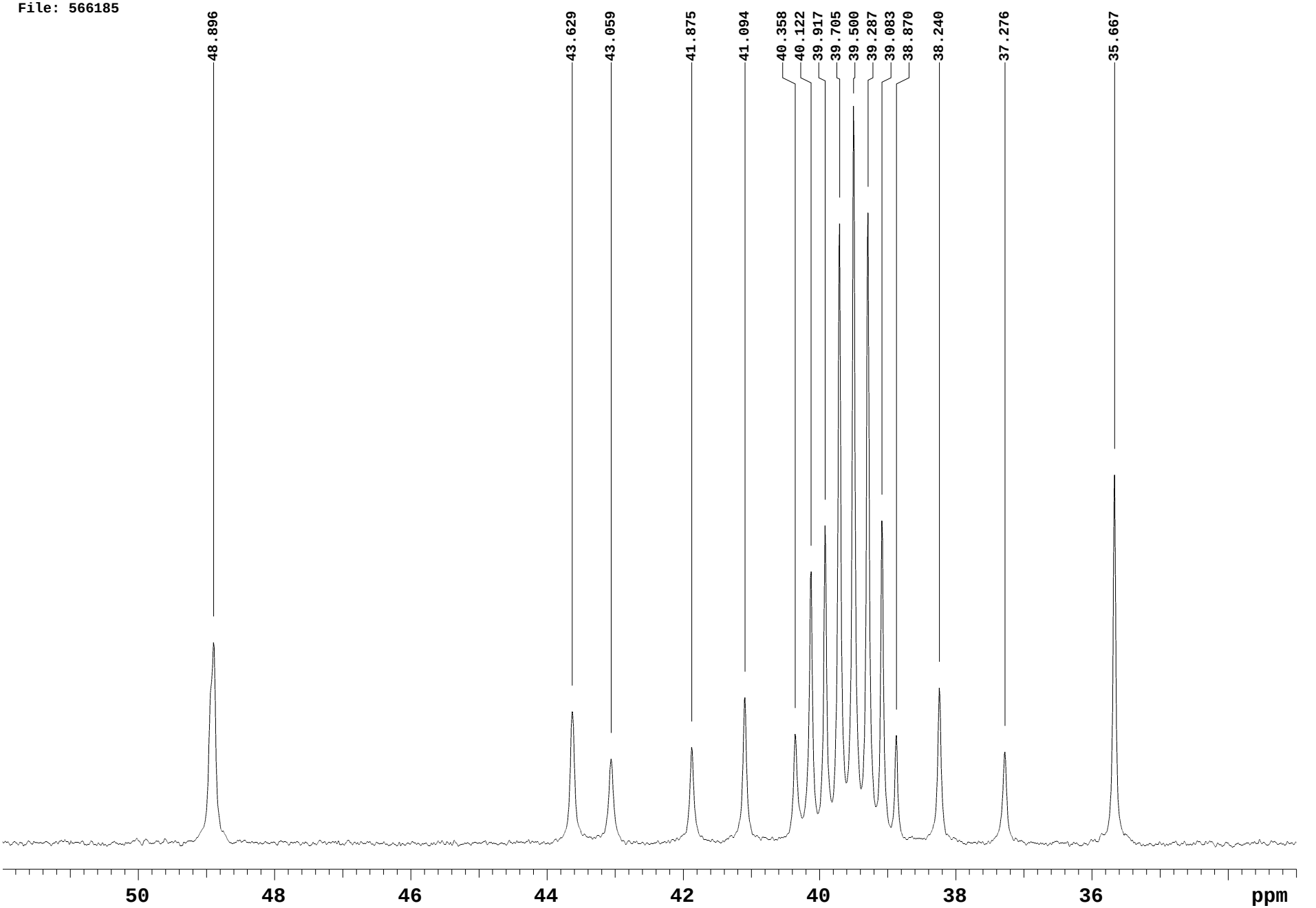
File: 566185



Plot file: 566185-4

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm
25C

File: 566185



Plot file: 566185-5

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm 25C

File: 566185

INDEX	FREQUENCY	PPM	HEIGHT
1	3970.858	39.500	141.8

Plot file: 566185-1_peaks

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm 25C

File: 566185

INDEX	FREQUENCY	PPM	HEIGHT
1	17152.926	170.628	141.8

Plot file: 566185-2_peaks

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm 25C

File: 566185

INDEX	FREQUENCY	PPM	HEIGHT
1	15786.502	157.036	45.0
2	15777.346	156.945	46.2
3	15545.413	154.638	44.4
4	15535.495	154.539	47.6
5	15181.491	151.017	141.8
6	15172.335	150.926	93.1
7	14972.445	148.938	28.2
8	14958.712	148.801	50.1
9	14944.979	148.665	31.2
10	14774.844	146.972	35.0
11	14771.792	146.942	39.1
12	14762.637	146.851	37.3
13	14759.585	146.821	39.1
14	14726.016	146.487	36.1
15	14712.283	146.350	54.9
16	14698.550	146.213	31.1
17	14533.755	144.574	41.0
18	14530.703	144.544	40.6
19	14521.548	144.453	38.4
20	14518.496	144.422	39.8
21	14381.930	143.064	9.7
22	14370.486	142.950	10.4
23	14343.020	142.677	33.2
24	14330.813	142.555	19.1
25	14304.110	142.290	34.4
26	14291.903	142.168	22.3
27	14265.200	141.903	14.0
28	14253.756	141.789	10.4

Plot file: 566185-3_peaks

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm 25C

File: 566185

INDEX	FREQUENCY	PPM	HEIGHT
1	12431.857	123.666	58.9
2	12427.279	123.620	85.8
3	12422.702	123.574	61.7
4	12413.546	123.483	64.0
5	12408.969	123.438	82.2
6	12404.391	123.392	59.9
7	12311.312	122.466	29.3
8	12041.232	119.780	84.4
9	12009.951	119.469	118.6
10	11990.878	119.279	119.2
11	11771.914	117.101	73.5
12	11501.834	114.414	21.5
13	10618.350	105.626	133.2
14	10597.750	105.421	137.9
15	10589.358	105.337	141.7
16	10567.996	105.125	126.3

Plot file: 566185-4_peaks

308390, Compound 184, Lot D6655070112, in DMSO-d6, 13C NMR, referenced to solvent at 39.5 ppm 25C

File: 566185

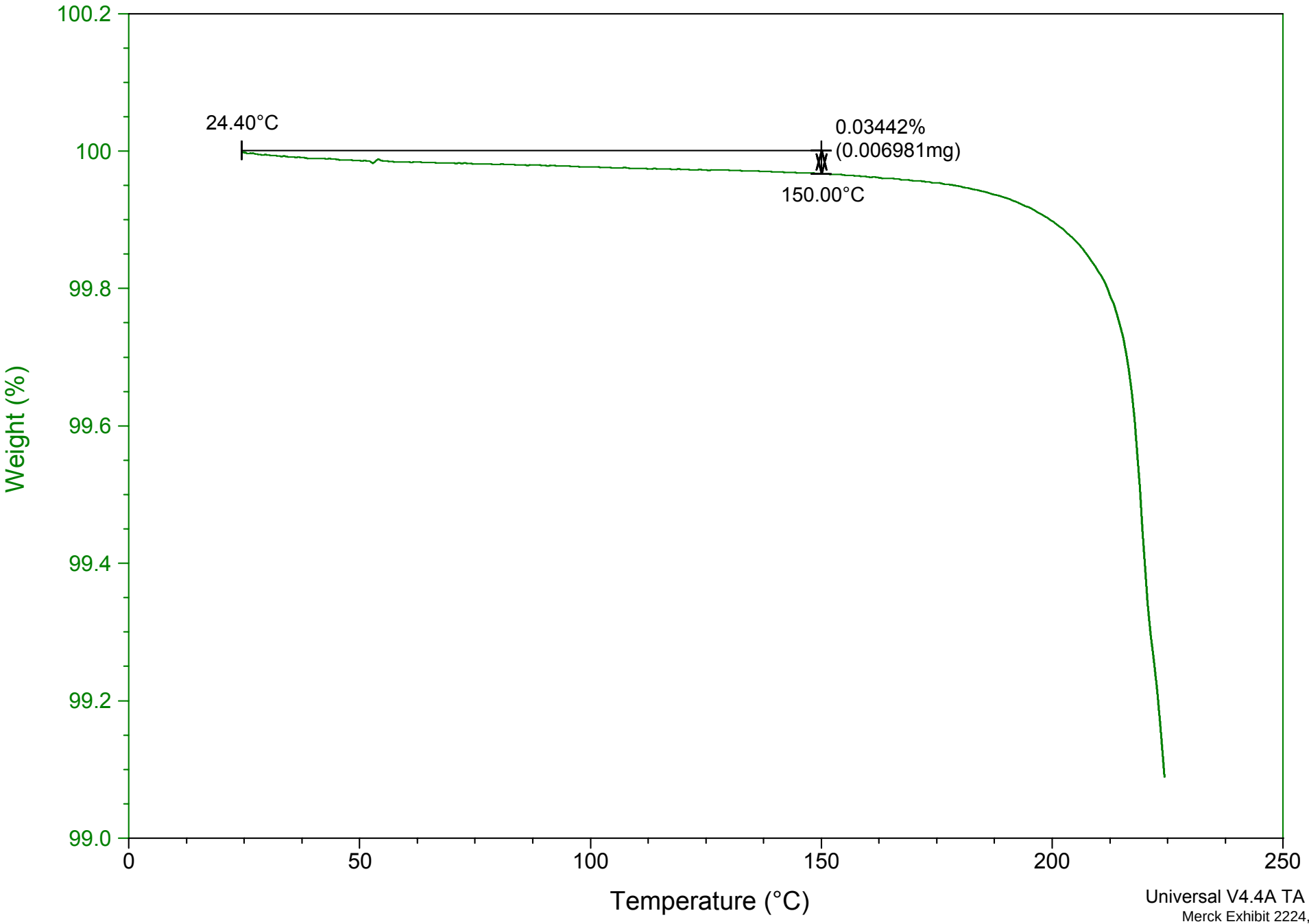
INDEX	FREQUENCY	PPM	HEIGHT
1	4915.377	48.896	38.6
2	4385.897	43.629	25.3
3	4328.677	43.059	16.2
4	4209.658	41.875	18.5
5	4131.076	41.094	28.0
6	4057.070	40.358	21.0
7	4033.419	40.122	52.2
8	4012.820	39.917	61.1
9	3991.458	39.705	119.1
10	3970.858	39.500	141.7
11	3949.496	39.287	121.2
12	3928.897	39.083	62.0
13	3907.534	38.870	20.7
14	3844.210	38.240	29.9
15	3747.317	37.276	17.6
16	3585.574	35.667	70.8

Plot file: 566185-5_peaks

Sample: Compound 184
Size: 20.2800 mg
Method: 00-250-20
Comment: 308389, LB-1017, 20 °C /min

TGA

File: J:...\TGA\563182.tga
Operator: KEL
Run Date: 04-Dec-2012 13:18
Instrument: 2050 TGA V5.4A

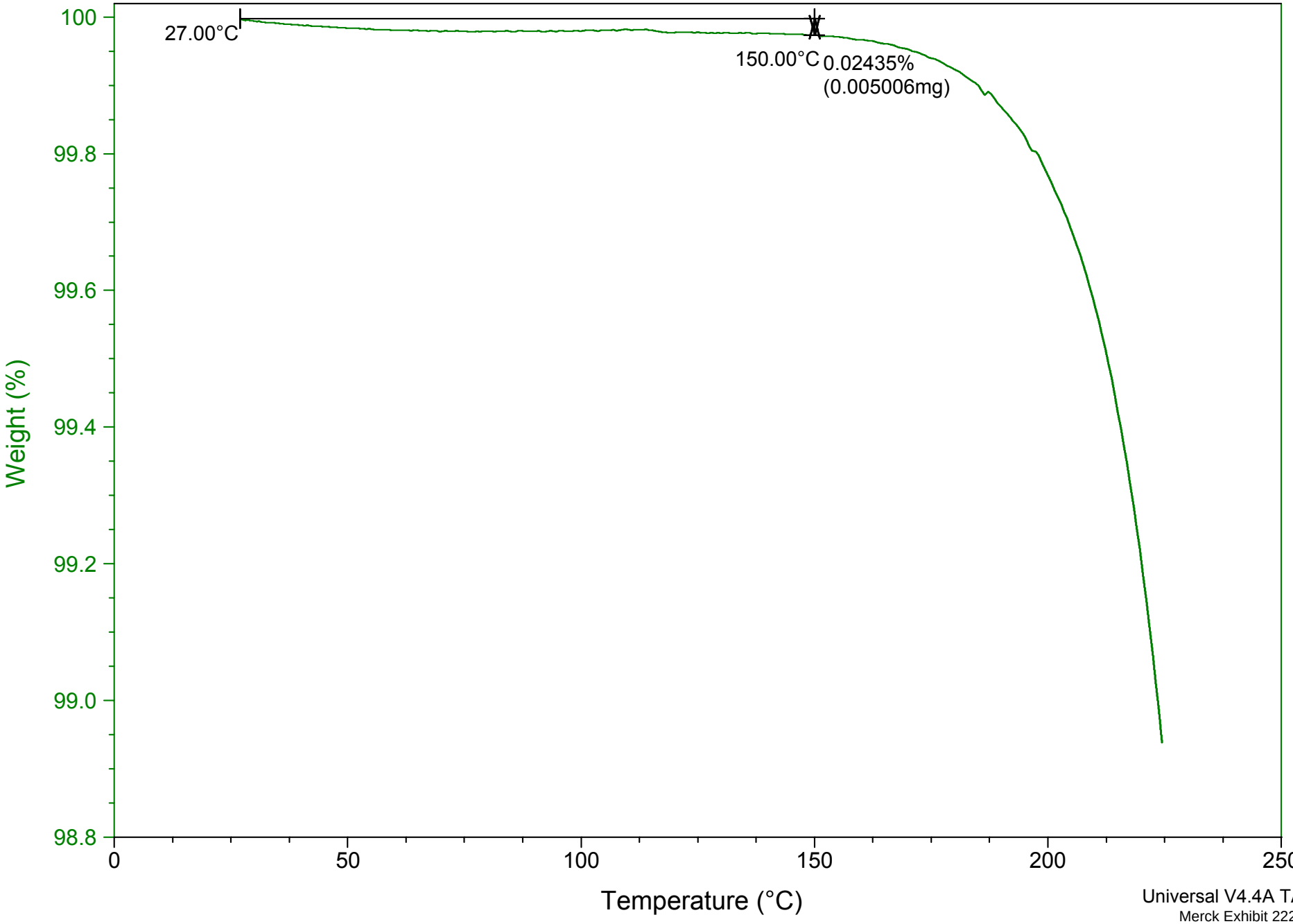


Universal V4.4A TA Instruments

Sample: Compound 184
Size: 20.5580 mg
Method: 00-250-20
Comment: 308390, D6655070112, 20 °C /min

TGA

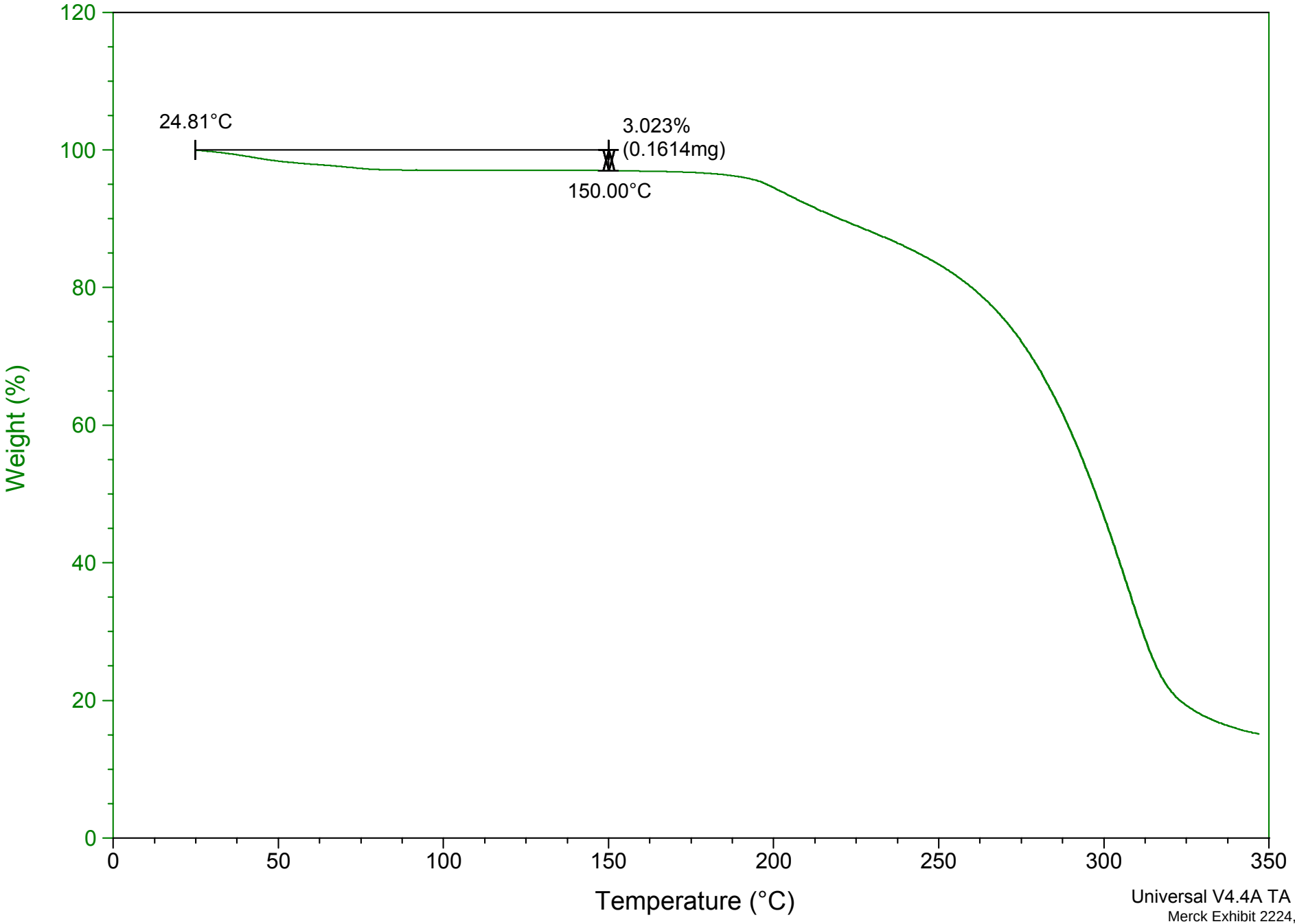
File: J:...\TGA\563183.tga
Operator: KEL
Run Date: 04-Dec-2012 14:10
Instrument: 2050 TGA V5.4A



Sample: Compound 184
Size: 5.3390 mg
Method: 00-350-10
Comment: 314339, 5135-02-01, 10°C/min, P1

TGA

File: J:...\TGA\564400.tga
Operator: DMP
Run Date: 11-Dec-2012 09:58
Instrument: AutoTGA 2950 V5.4A

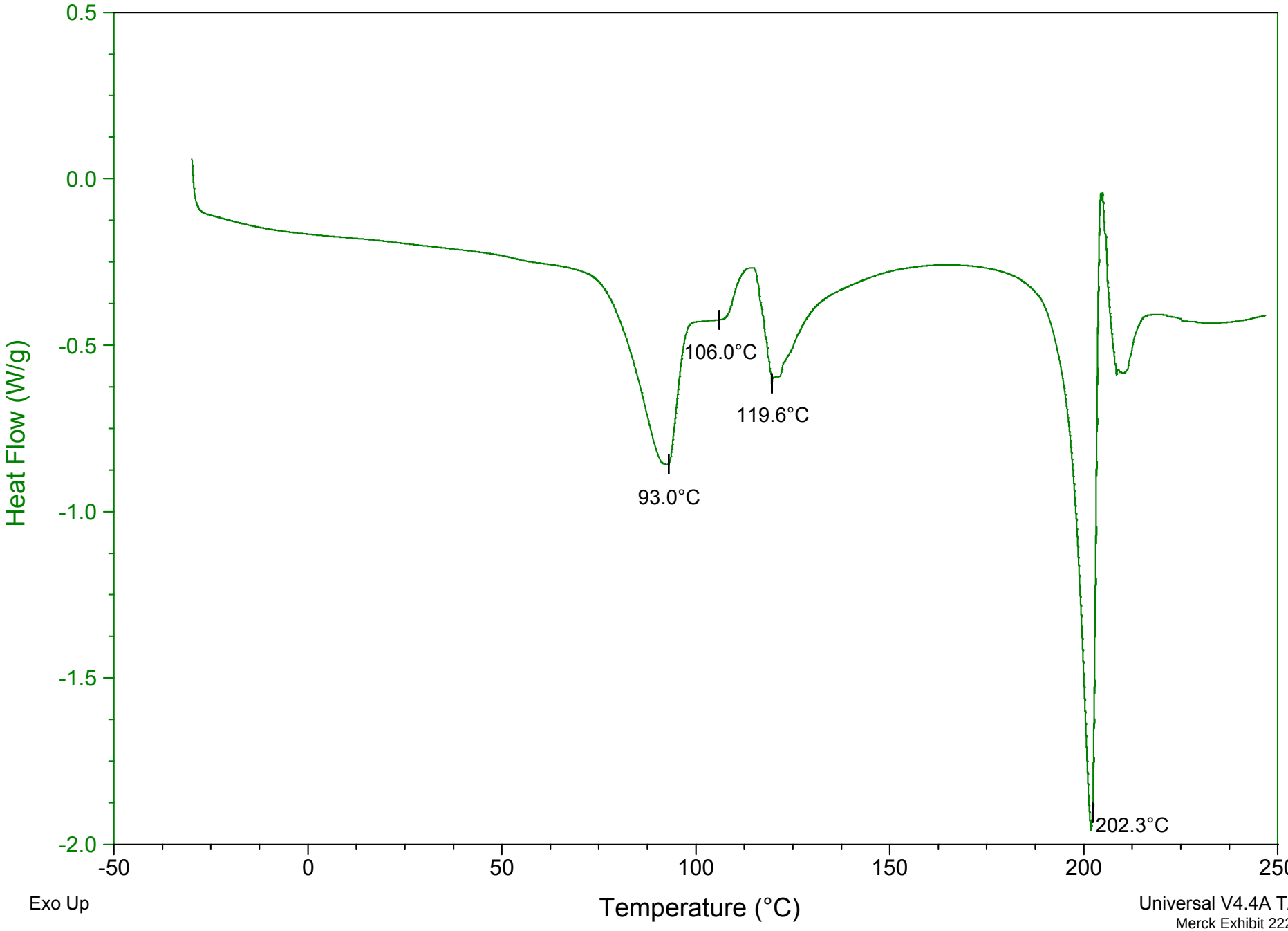


Universal V4.4A TA Instruments

Sample: Compound 184
Size: 2.7800 mg
Method: (-30)-250-10
Comment: 314339, 5135-02-01, 10°C/min,T0HSLP, R1, P1

DSC

File: J:...\DSC\564401.dsc
Operator: DMP
Run Date: 11-Dec-2012 09:48
Instrument: DSC Q2000 V23.10 Build 79

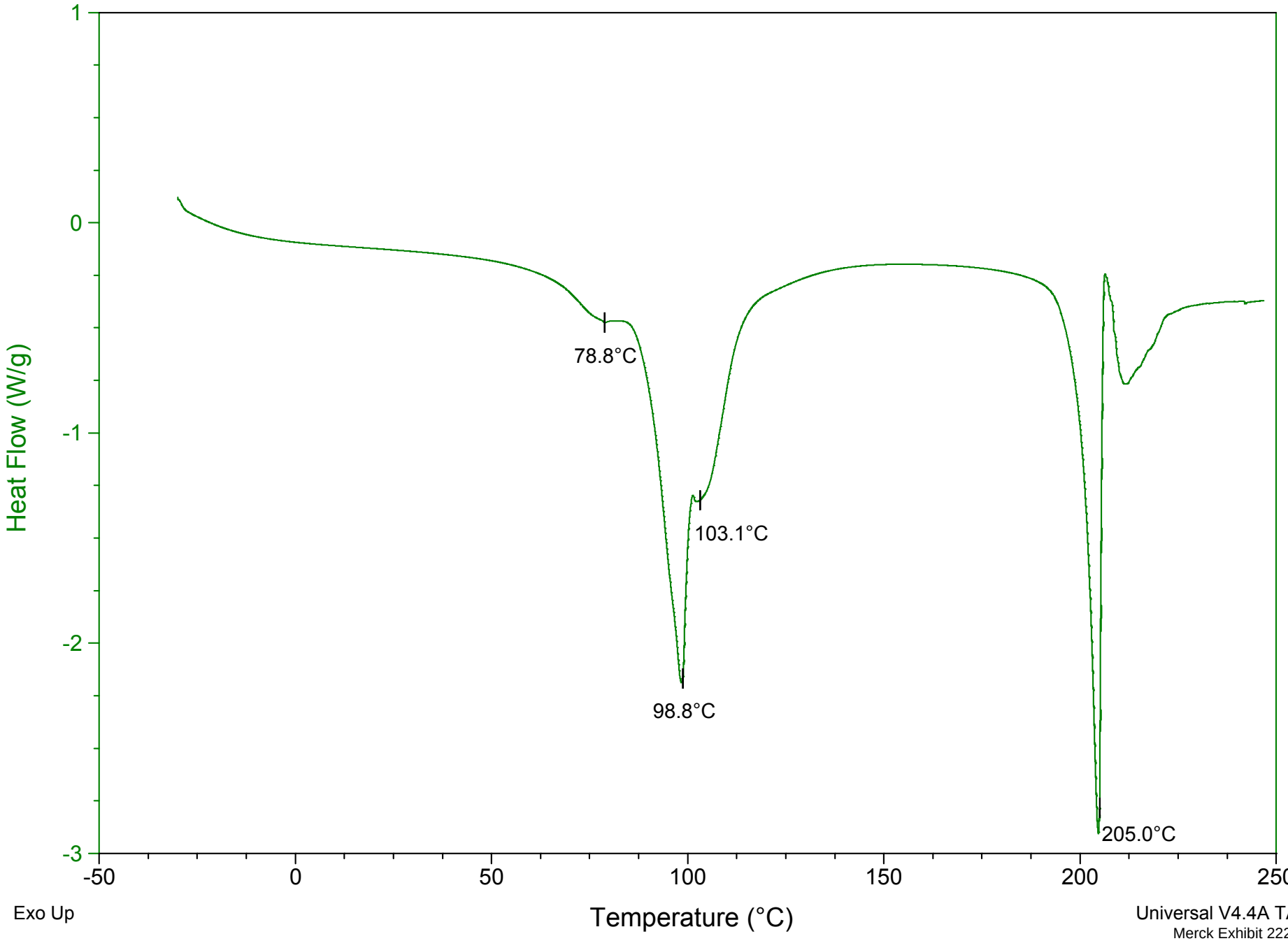


Universal V4.4A TA Instruments

Sample: Compound 184
Size: 1.5900 mg
Method: (-30)-250-10
Comment: 314760, 5135-15-05, 10°C/min, T0HSLP, R1, P1

DSC

File: J:...\DSC\564942.dsc
Operator: KEL
Run Date: 12-Dec-2012 16:27
Instrument: DSC Q2000 V23.10 Build 79



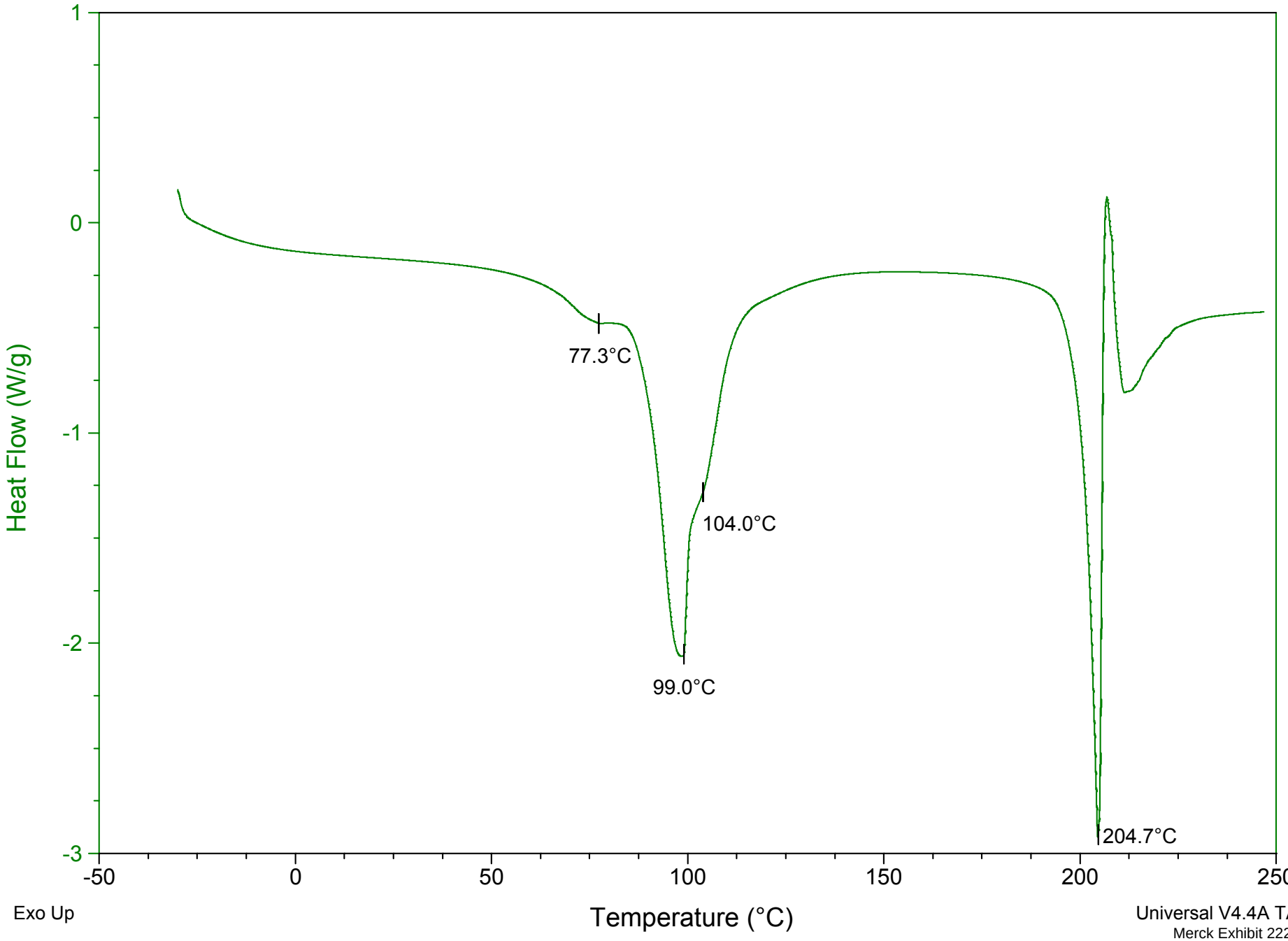
Exo Up

Universal V4.4A TA Instruments

Sample: Compound 184
Size: 1.4700 mg
Method: (-30)-250-10
Comment: 314783, 5135-17-01, 10°C/min, T0HSLP, R1, P2

DSC

File: J:...\DSC\564944.dsc
Operator: KEL
Run Date: 12-Dec-2012 17:06
Instrument: DSC Q2000 V23.10 Build 79



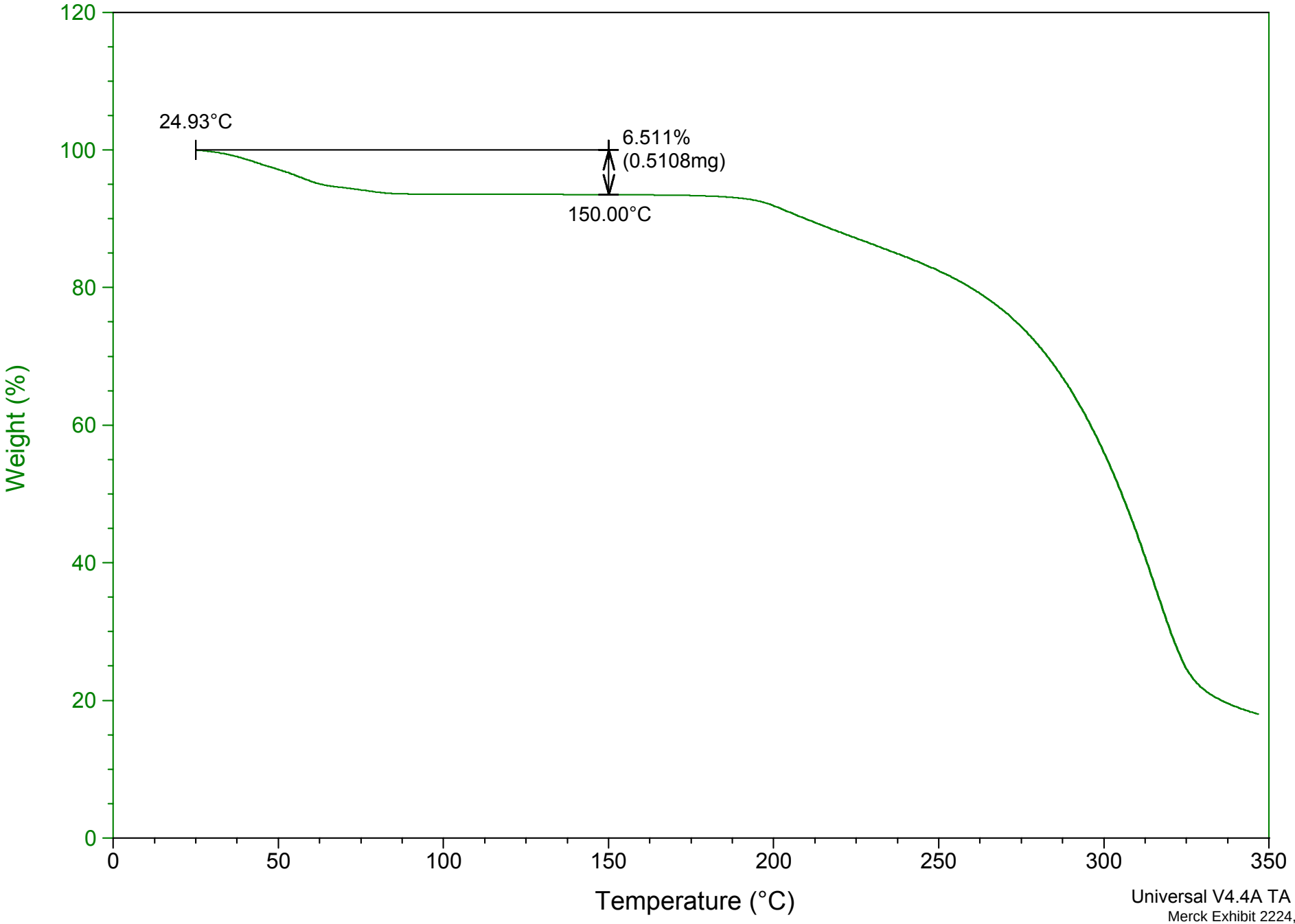
Exo Up

Universal V4.4A TA Instruments

Sample: Compound 184
Size: 7.8450 mg
Method: 00-350-10
Comment: 314760, 5135-15-05, 10°C/min, P1

TGA

File: J:...\TGA\564945.tga
Operator: KEL
Run Date: 12-Dec-2012 15:54
Instrument: AutoTGA 2950 V5.4A

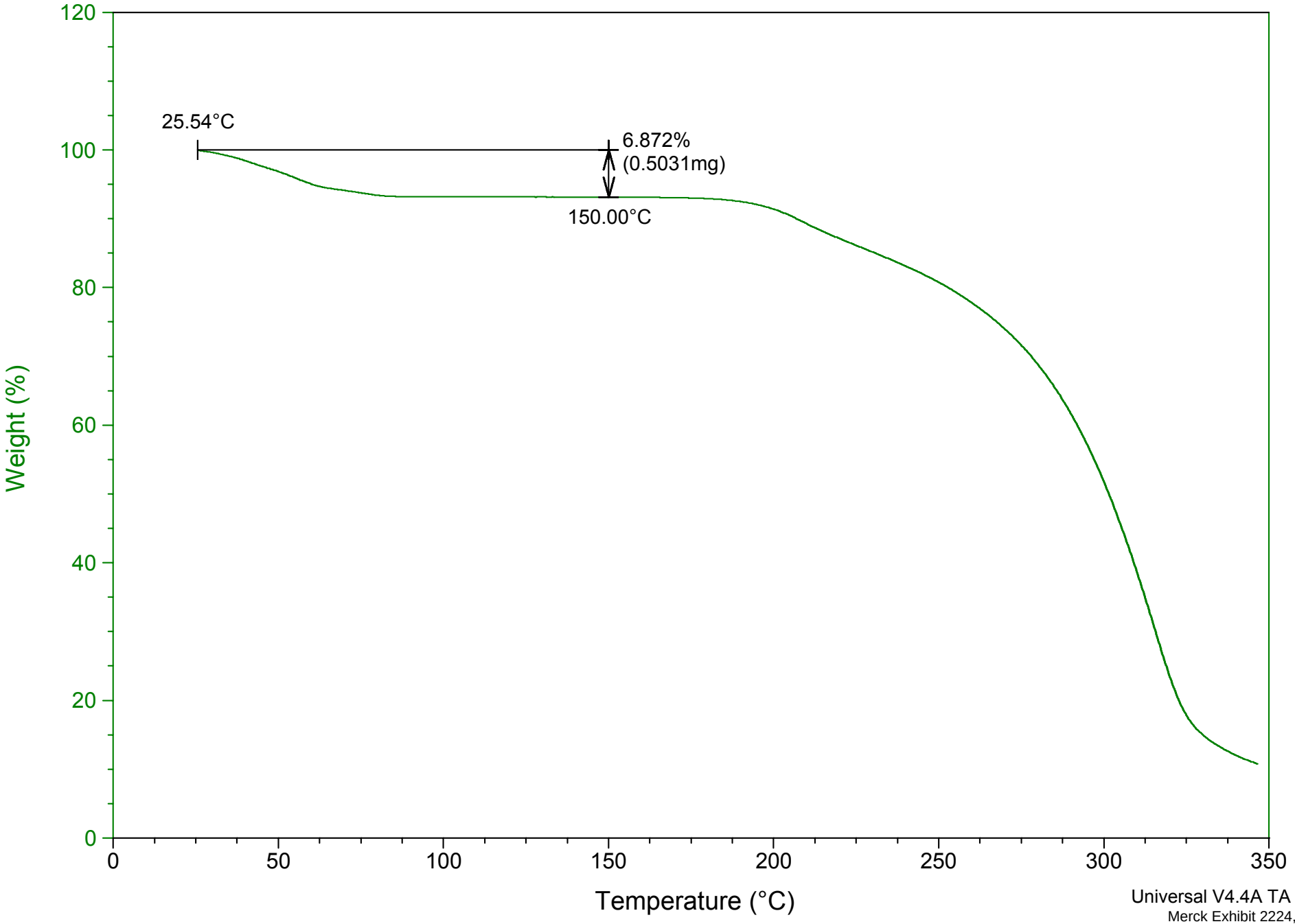


Universal V4.4A TA Instruments

Sample: Compound 184
Size: 7.3210 mg
Method: 00-350-10
Comment: 314783, 5135-17-01, 10°C/min, P2

TGA

File: J:...\TGA\564946.tga
Operator: KEL
Run Date: 12-Dec-2012 16:55
Instrument: AutoTGA 2950 V5.4A

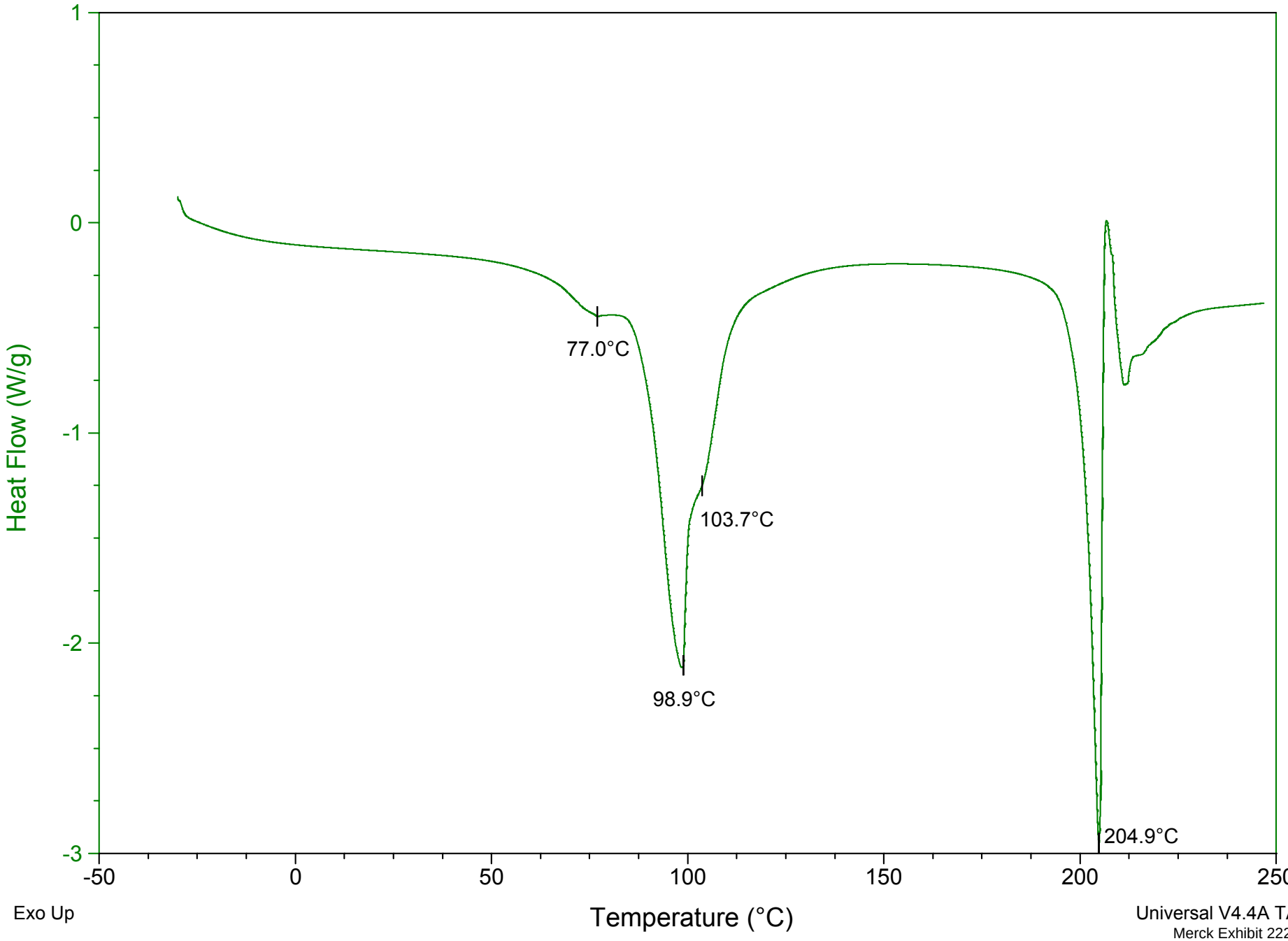


Universal V4.4A TA Instruments

Sample: Compound 184
Size: 1.4400 mg
Method: (-30)-250-10
Comment: 314783, 5135-17-01, 10°C/min, T0HSLP, R1, P2

DSC

File: J:...\DSC\565124.dsc
Operator: KEL
Run Date: 13-Dec-2012 13:29
Instrument: DSC Q2000 V23.10 Build 79

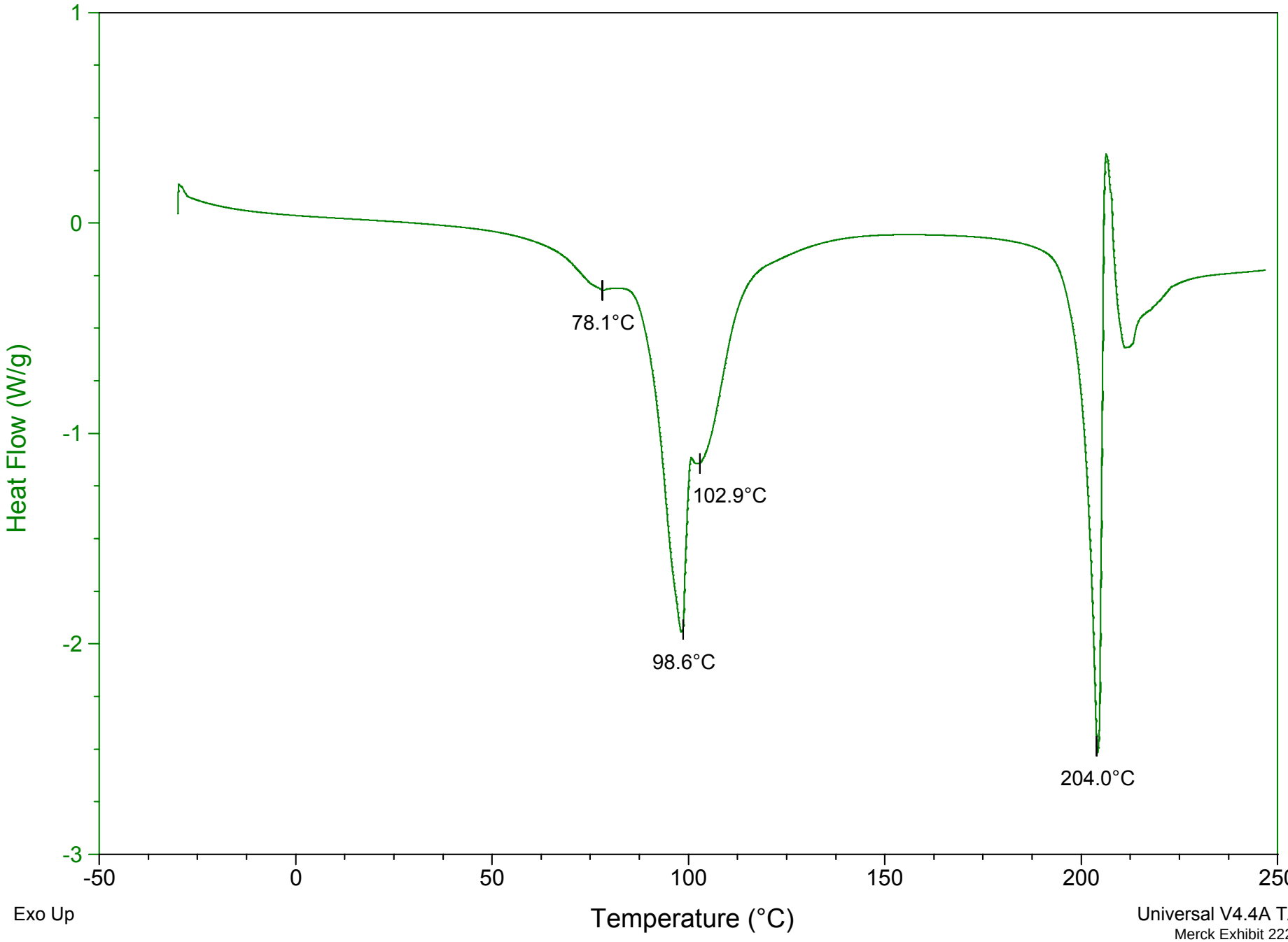


Universal V4.4A TA Instruments

Sample: Compound 184
Size: 1.4700 mg
Method: (-30)-250-10
Comment: 314760, 5135-15-05, 10°C/min, T0HSLP, R1, P1

DSC

File: J:...\DSC\565125.dsc
Operator: KEL
Run Date: 13-Dec-2012 12:51
Instrument: DSC Q2000 V23.10 Build 79



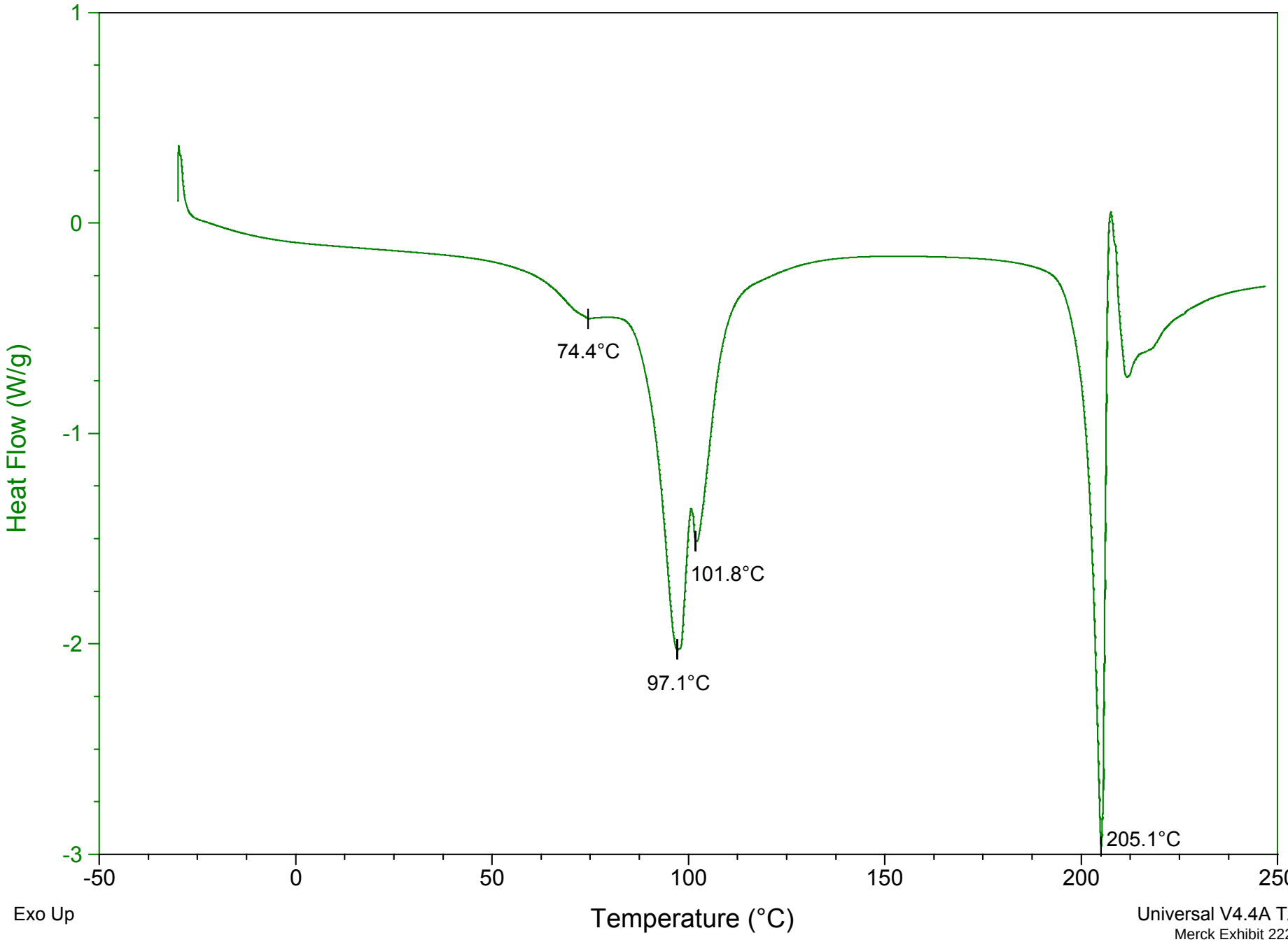
Exo Up

Universal V4.4A TA Instruments

Sample: Compound 184
Size: 1.4300 mg
Method: (-30)-250-10
Comment: 315535, 5135-30-03, 10°C/min, T0HSLP, R1, P1

DSC

File: J:...\DSC\566460.dsc
Operator: KEL
Run Date: 21-Dec-2012 12:25
Instrument: DSC Q2000 V23.10 Build 79



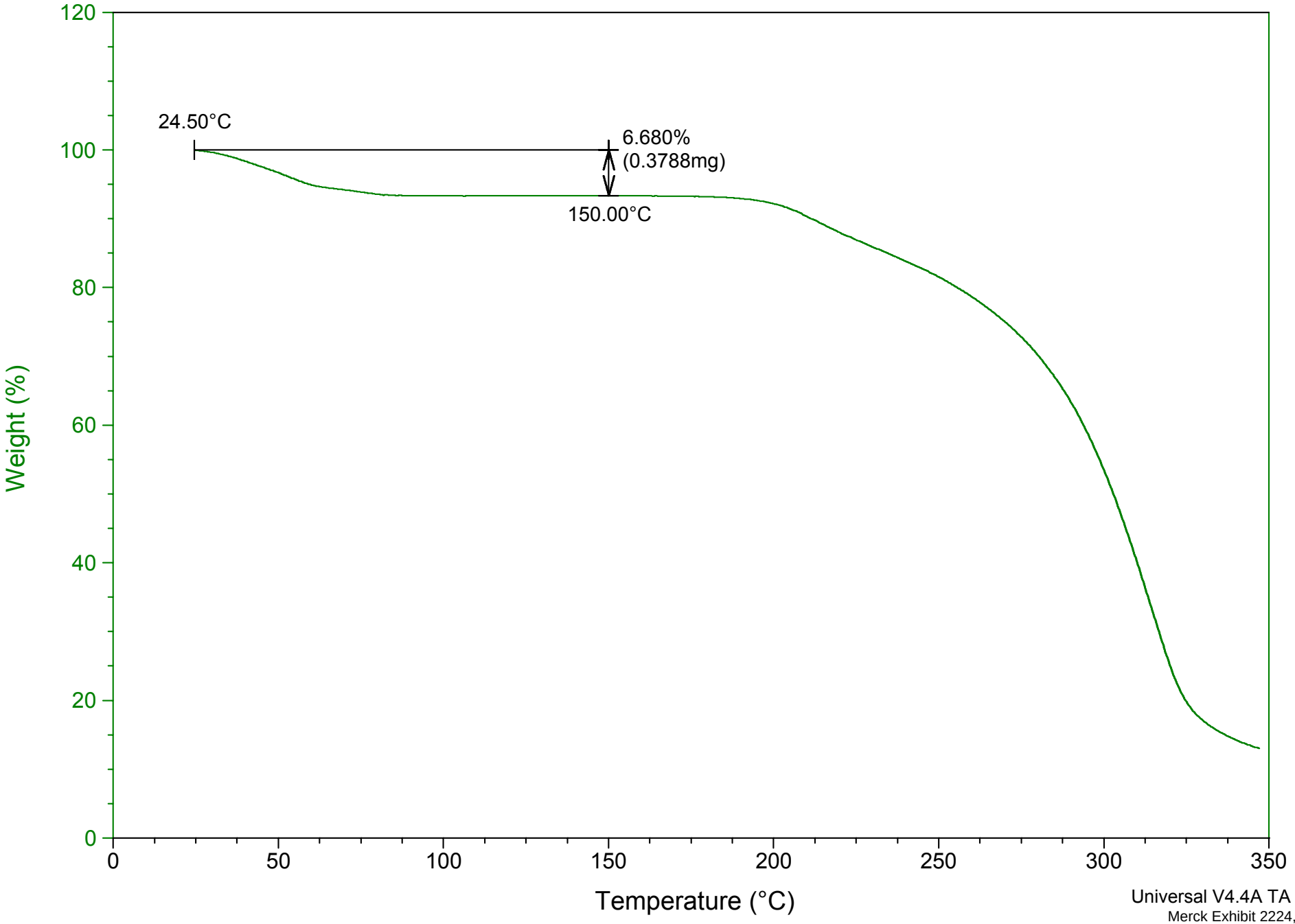
Exo Up

Universal V4.4A TA Instruments

Sample: Compound 184
Size: 5.6710 mg
Method: 00-350-10
Comment: 315535, 5135-30-03, 10°C/min, P1

TGA

File: I:...\EL20100011\TGA\566461.tga
Operator: KEL
Run Date: 21-Dec-2012 12:06
Instrument: AutoTGA 2950 V5.4A



Universal V4.4A TA Instruments

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 1.01 - 39.98 °2θ Step Size: 0.008 °2θ
Collection Time: 1950 s Scan Speed: 1.2°/min Slit: DS: 1/2° SS: null Revolution Time: 1.0 s Mode: Transmission

563246 308390, Compound 184, D6655070112 null short AS extension in place, air 04-Dec-2012 08:37:35

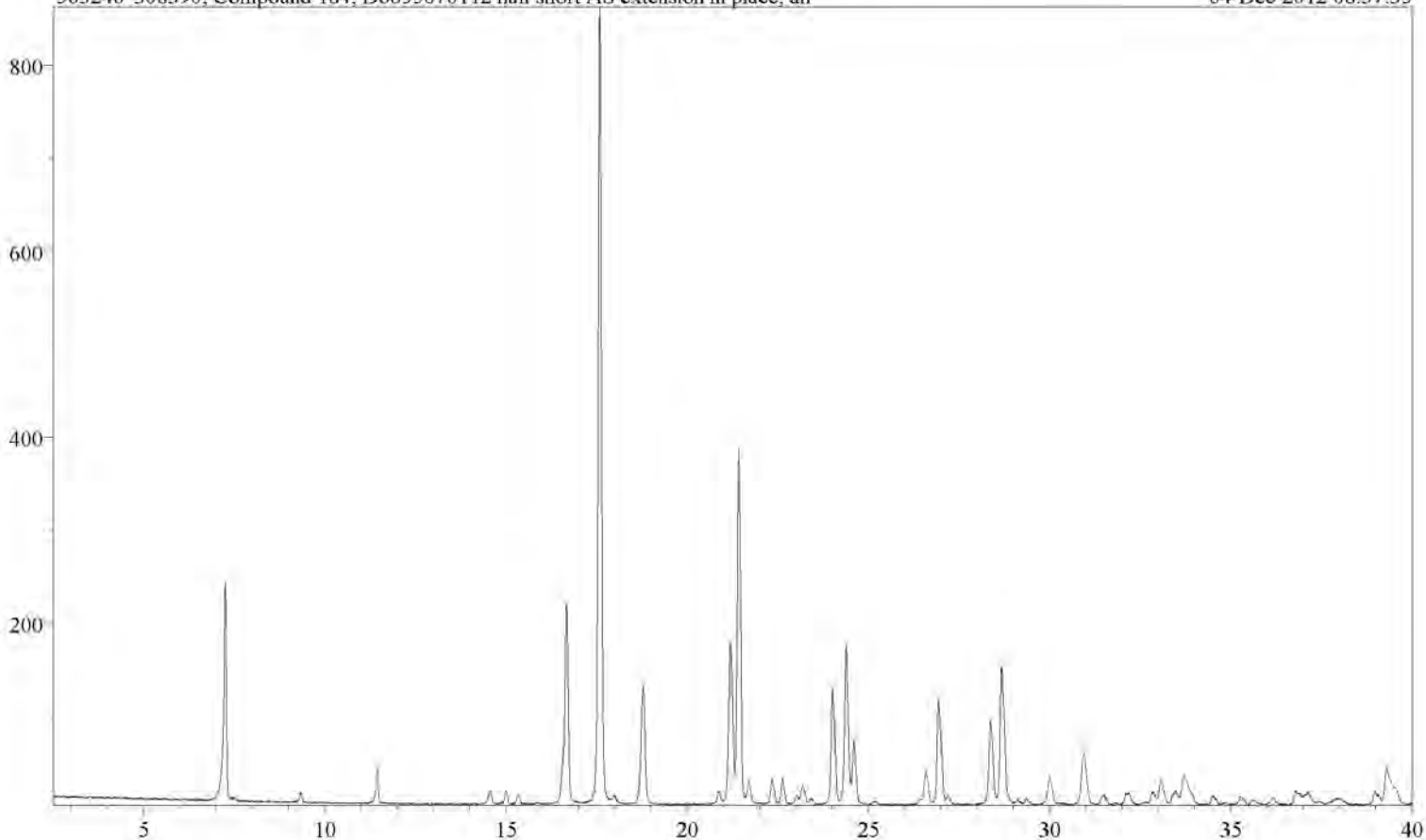
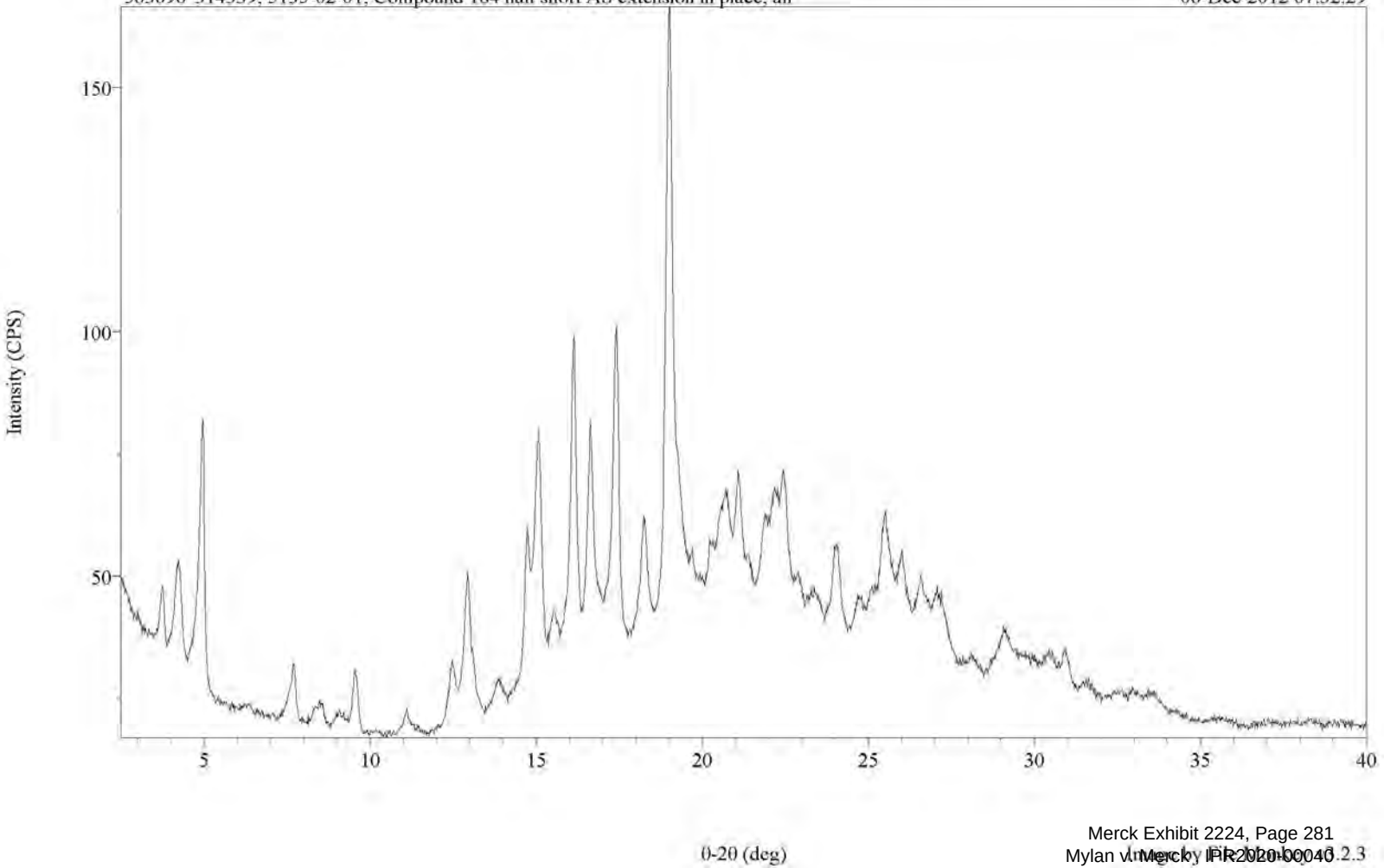
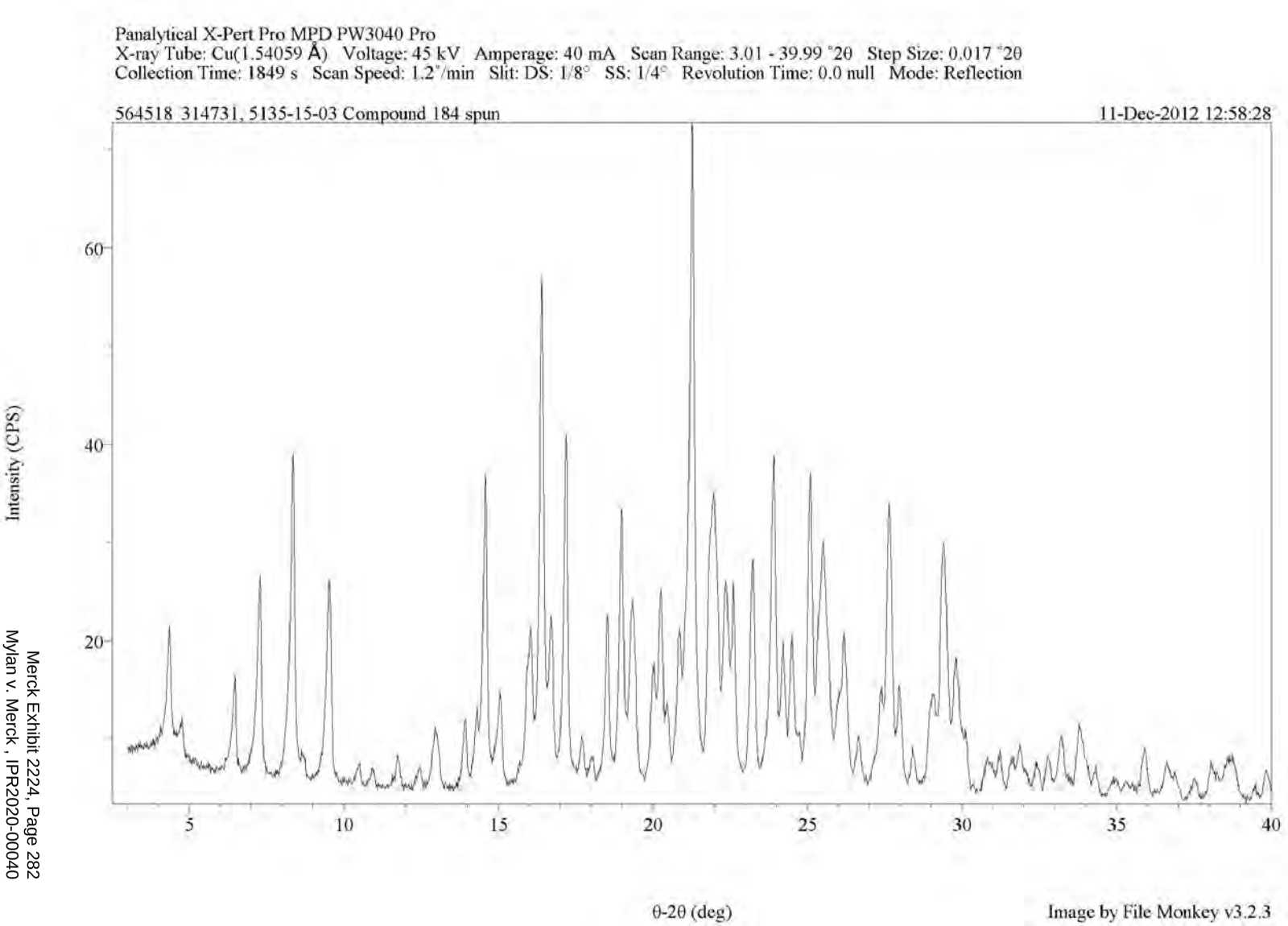


Image by File Monkey v3.2.3

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 1.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1939 s Scan Speed: 1.2°/min Slit: DS: 1/2° SS: null Revolution Time: 1.0 s Mode: Transmission

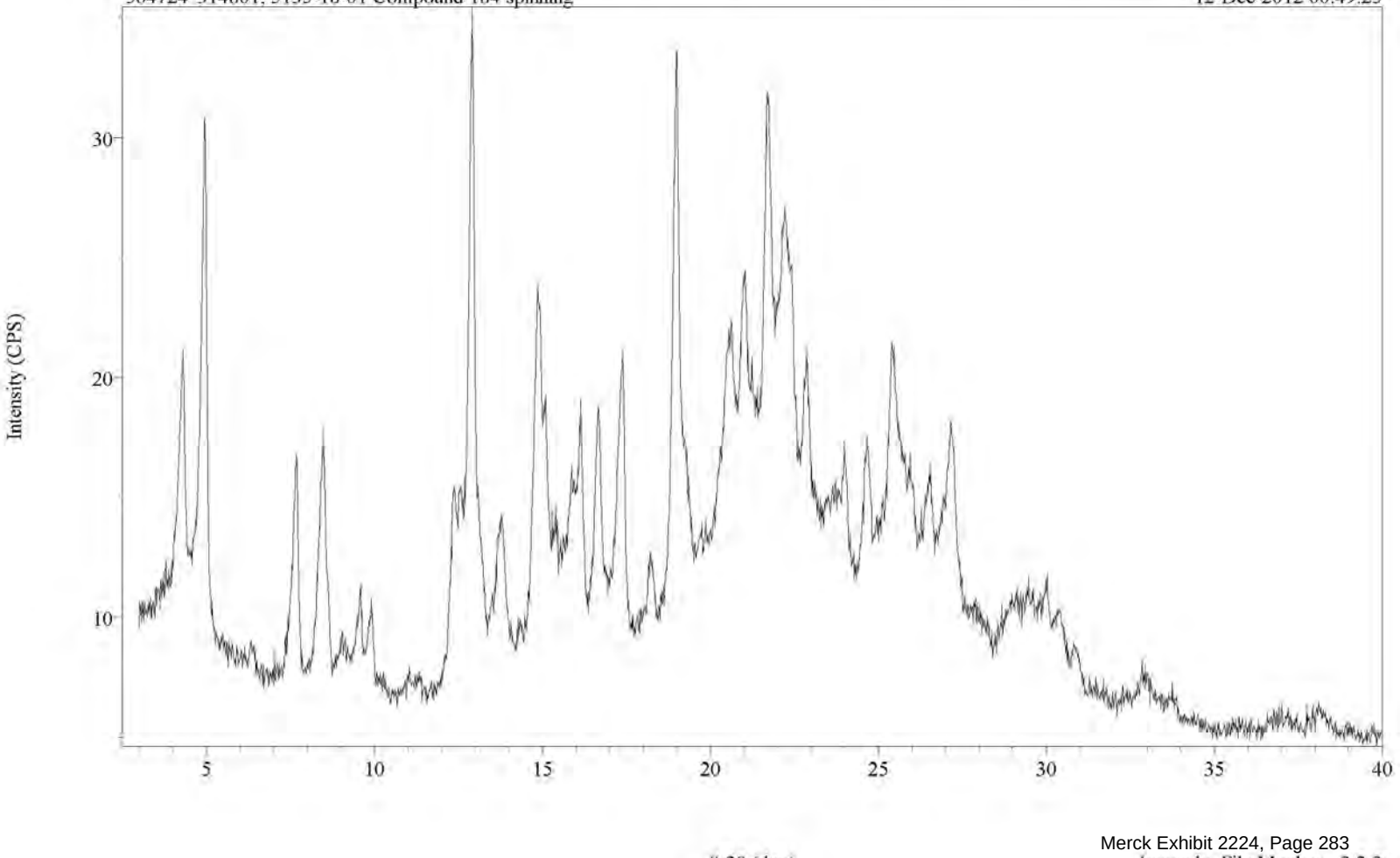
563696 314339, 5135-02-01, Compound 184 null short AS extension in place, air 06-Dec-2012 07:32:29





Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1849 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

564724 314801, 5135-18-01 Compound 184 spinning 12-Dec-2012 00:49:25



Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1850 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

565362_315083, 5135-27-01 Compound 184 spinning 14-Dec-2012 12:51:23

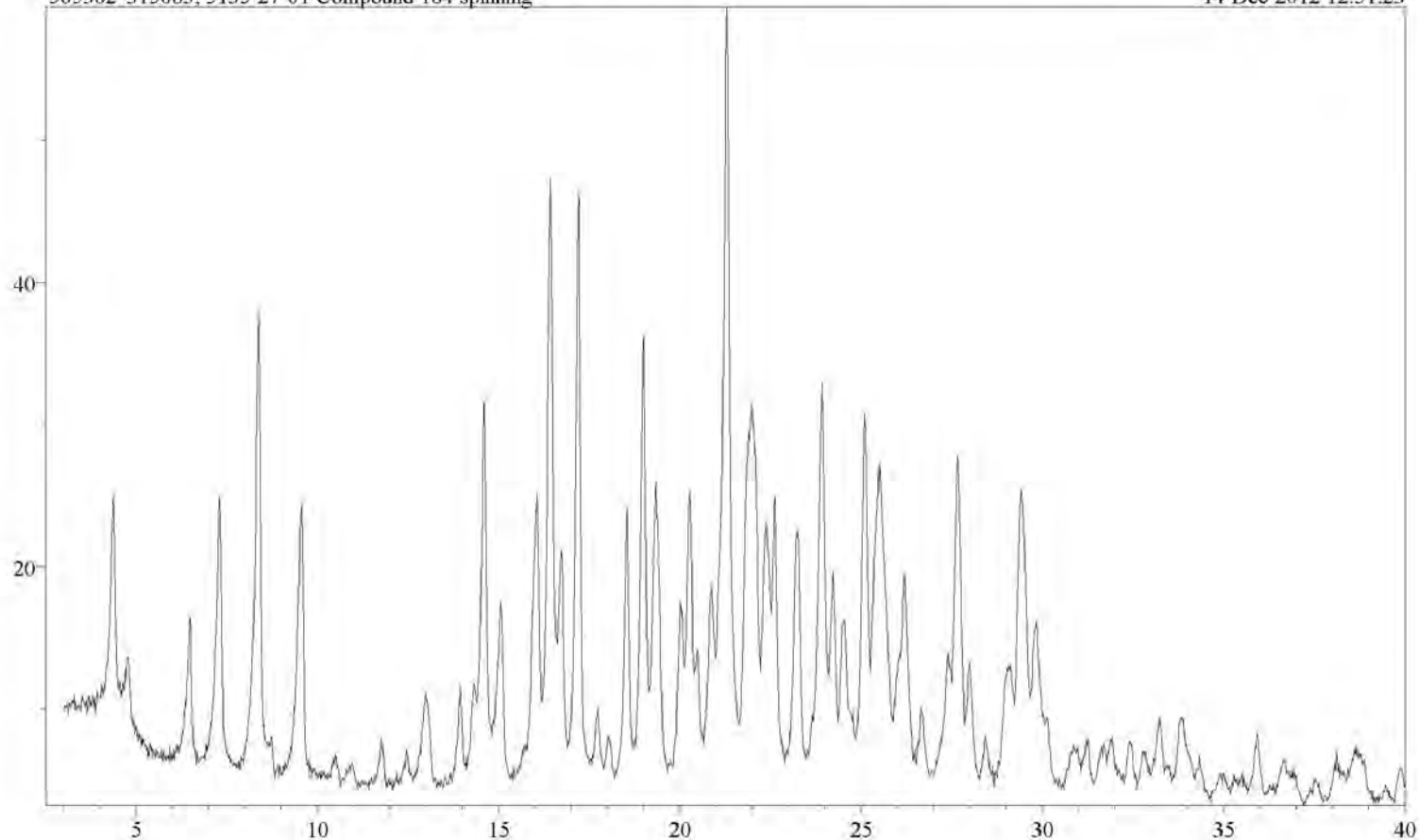
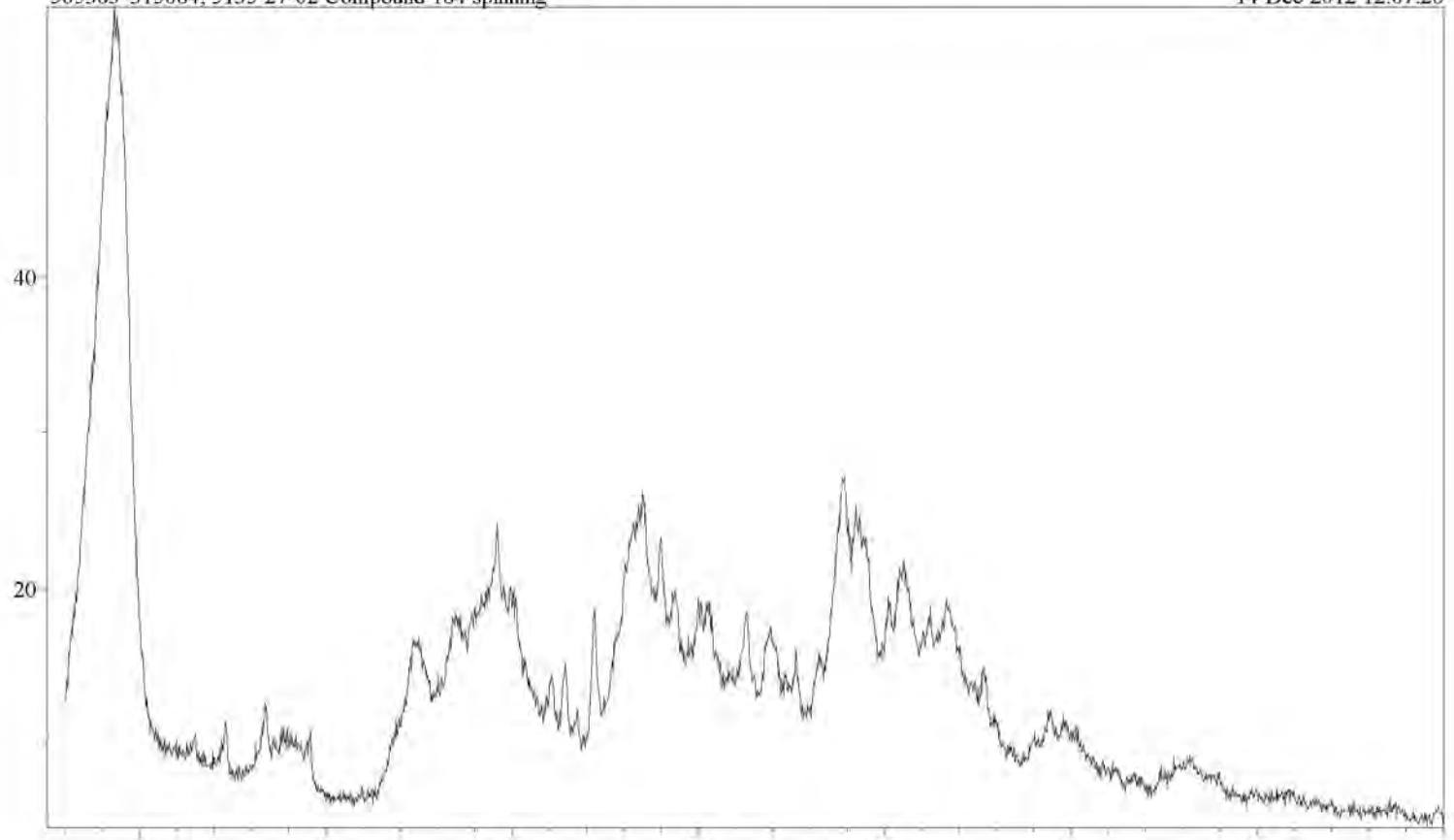


Image by File Monkey v3.2.3

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1847 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

565363 315084, 5135-27-02 Compound 184 spinning

14-Dec-2012 12:07:26



Intensity (CPS)

θ-2θ (deg)

Image by File Monkey v3.2.3

Panalytical X-Pert Pro MPD PW3040 Pro
X-ray Tube: Cu(1.54059 Å) Voltage: 45 kV Amperage: 40 mA Scan Range: 3.01 - 39.99 °2θ Step Size: 0.017 °2θ
Collection Time: 1851 s Scan Speed: 1.2°/min Slit: DS: 1/8° SS: 1/4° Revolution Time: 0.0 null Mode: Reflection

566458 315535, 5135-30-03 Compound 184 spun 21-Dec-2012 13:17:55

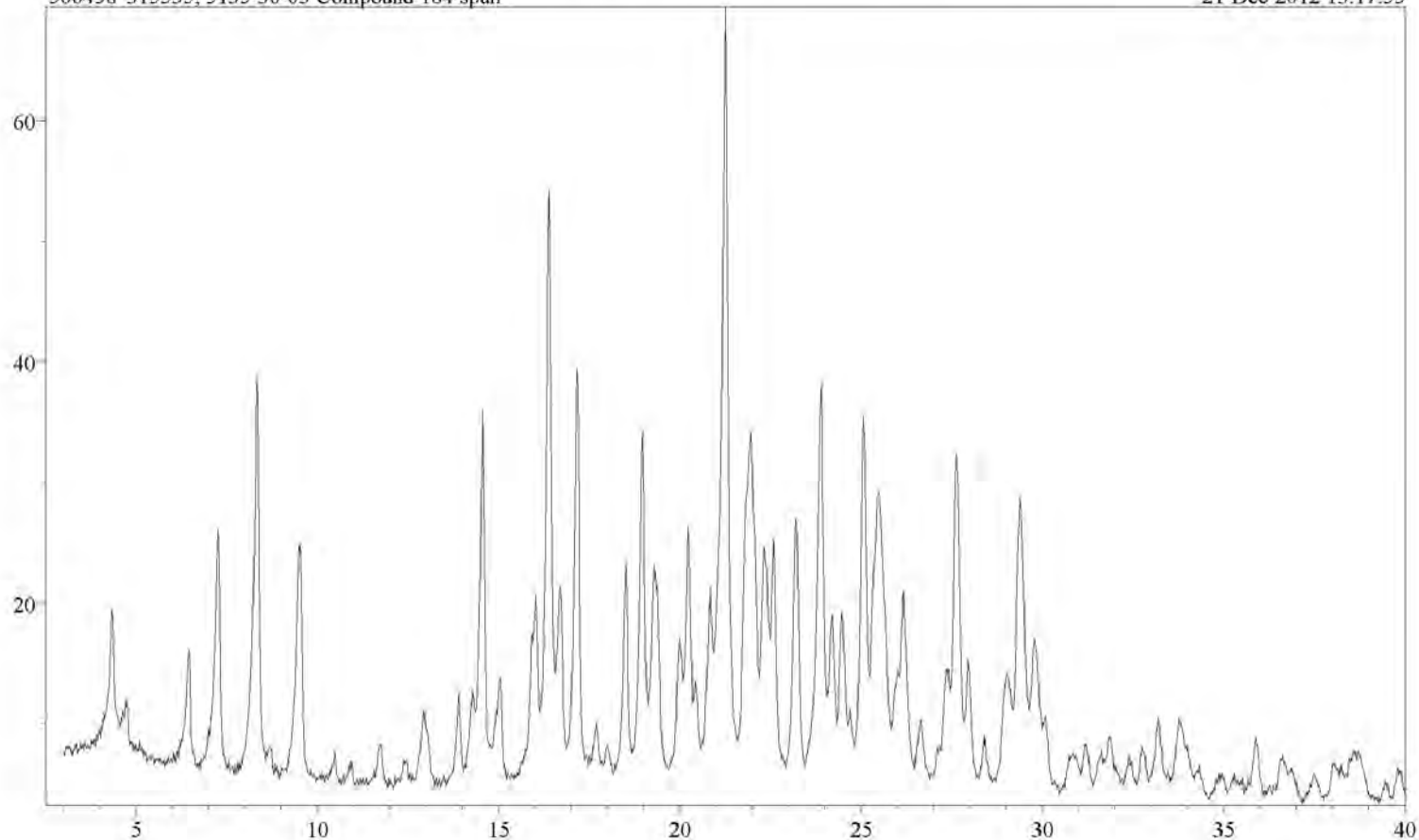


Image by File Monkey v3.2.3

Project ID: EL20100011

308389	Compound 184	LB-1017	Ambient	Legal sample	NC_Urgent	Completed

09/21/2012 12:46:31
AMARCOV: Sitagliptin
CAS# 790712-60-6
Received sample from ASSIA, part of Teva group, Israel
12/13/2012 15:50:06
KGUSHURST: Phosphate salt

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
IR (B) bench	TGIR_IRB_STD_MCTA	2129	563305	KLEACH	NC_Urge	Completed	12/4/12	KLEACH	12/04/2012 13:14:52 KLEACH: B-screen installed He carrier gasTransfer line/Cell = 250°C/250°C TGA collected in file 563182	
NMR #2	Q_NMR_liquids_C13_1D	1154	566184	PWHEELER	NC_Urge	Completed	12/19/12	PWHEELER	12/19/2012 18:40:01 KGUSHURST: 50.3mg see 5135-31 12/19/2012 19:17:14 PWHEELER: see ntbk# 5143-08-01 for sample prep	
NMR #2	Q_NMR_solids_C13_CPMA	1169	565563	PWHEELER	NC_Urge	Done	12/17/12	PTISHMACK	12/17/2012 13:31:54 PWHEELER: glycine chemical shift reference	
NMR #2	Q_NMR_solids_C13_CPMA	1169	565613	PWHEELER	NC_Urge	Done	12/17/12	PTISHMACK	AUD: 12/17/2012 13:31:03 PWHEELER: sample analysis 12/17/2012 13:45:59 PWHEELER: Rotor# RSN40044, see ntbk# 5143-01-01 for sample prep	
NMR #2	Q_NMR_solids_N15_CPMA	1175	564392	PWHEELER	NC_Urge	Rejected	12/11/12	KGUSHURST	12/11/2012 09:29:48 PWHEELER: Rotor# RSN40044, see ntbk# 5143-01-01 for sample prep AUD: 12/14/2012 1241 PWHEELER: File Correction. New corrected filename: 565393	FC
NMR #2	Q_NMR_solids_N15_CPMA	1175	564428	PWHEELER	NC_Urge	Rejected	12/11/12	KGUSHURST	AUD: 12/11/2012 09:18:55 PWHEELER: N15 glycine shift reference AUD: 12/14/2012 1241 PWHEELER: File Correction. New corrected filename: 565392	FC
NMR #2	Q_NMR_solids_N15_CPMA	1175	564438	PWHEELER	NC_Urge	Done	12/11/12	PTISHMACK	AUD: 12/11/2012 09:56:36 PWHEELER: glycine analysis	
NMR #2	Q_NMR_solids_N15_CPMA	1175	565392	PWHEELER	NC_Urge	Done	12/14/12	PTISHMACK		
NMR #2	Q_NMR_solids_N15_CPMA	1175	565393	PWHEELER	NC_Urge	Done	12/14/12	PTISHMACK		
SUBCONTRACTORS	Data_Processing	1381	565690	PWHEELER	NC_Urge	Completed	12/17/12	PWHEELER	12/17/2012 16:34:47 PWHEELER: securing overlaid plots	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
308389		Compound 184	LB-1017	Ambient	Legal sample		NC_Urgent	Completed	09/21/2012 12:46:31 AMARCOV: Sitagliptin CAS# 790712-60-6 Received sample from ASSIA, part of Teva group, Israel 12/13/2012 15:50:06 KGUSHURST: Phosphate salt

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
TGA #4	TGIR_TG4_STD_20	352	563182	KLEACH	NC_Urge	Completed	12/4/12	KGUSHURST	12/04/2012 13:16:07 KLEACH: manually loaded, no autosamlper He purge Pt pans IR collected in file 563305 12/04/2012 13:50:02 KLEACH: After analysis, the sample was clear.	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
308390		Compound 184	D6655070112	Ambient	Legal sample		NC_Urgent	Completed	09/21/2012 12:46:55 AMARCOV: Weight(Gross) Sitagliptin CAS# 790712-60-6 Received sample from ASSIA, part of Teva group, Israel 12/13/2012 15:49:47 KGUSHURST: Free base

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
IR (B) bench	TGIR_IRB_STD_MCTA	212	563304	KLEACH	NC_Urge	Completed	12/4/12	KLEACH	12/04/2012 14:07:37 KLEACH: B-screen installed He carrier gasTransfer line/Cell = 250°C/250°C TGA collected in file 563183	
NMR #2	Q_NMR_liquids_C13_1D	1154	566185	PWHEELER	NC_Urge	Completed	12/19/12	PWHEELER	12/19/2012 18:40:26 KGUSHURST: 50.1mg see 5135-31 12/19/2012 19:17:21 PWHEELER: see ntbk# 5143-08-02 for sample prep 12/19/2012 20:44:51 PWHEELER: see ntbk# 5143-08-02 for sample prep	
NMR #2	Q_NMR_solids_C13_CPMA	116	565564	PWHEELER	NC_Urge	Done	12/17/12	PTISHMACK	12/17/2012 16:24:59 PWHEELER: Rotor# RSN40033, see ntbk# 5143-01-02 for sample prep	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
308390		Compound 184	D6655070112	Ambient	Legal sample		NC_Urgent	Completed	09/21/2012 12:46:55 AMARCOV: Weight(Gross) Sitagliptin CAS# 790712-60-6 Received sample from ASSIA, part of Teva group, Israel 12/13/2012 15:49:47 KGUSHURST: Free base

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_solids_N15_CPMA	1175	564393	PWHEELER	NC_Urge	Rejected	12/12/12	KGUSHURST	12/12/2012 09:00:31 PWHEELER: N15 glycine chemical shift reference AUD: 12/14/2012 1241 PWHEELER: File Correction. New corrected filename: 565394	FC
NMR #2	Q_NMR_solids_N15_CPMA	1175	564763	PWHEELER	NC_Urge	Rejected	12/12/12	KGUSHURST	AUD: 12/12/2012 09:01:19 PWHEELER: sample analysis 12/12/2012 10:47:10 PWHEELER: Rotor# RSN40033, see ntbk# 5143-01-02 for sample prep AUD: 12/14/2012 1241 PWHEELER: File Correction. New corrected filename: 565395	FC
NMR #2	Q_NMR_solids_N15_CPMA	1175	565394	PWHEELER	NC_Urge	Done	12/14/12	PTISHMACK		
NMR #2	Q_NMR_solids_N15_CPMA	1175	565395	PWHEELER	NC_Urge	Done	12/14/12	PTISHMACK		
TGA #4	TGIR_TG4_STD_20	352	563183	KLEACH	NC_Urge	Completed	12/4/12	KGUSHURST	12/04/2012 14:02:17 KLEACH: Manually loaded, no autosampler Pt pans He purge IR collected in file 563304 12/04/2012 14:33:02 KLEACH: Sample was clear after analysis.	
XR #10	XR10_TSH	1956	563246	AATKINSON	NC_Urge	Rejected	12/4/12	KGUSHURST	12/04/2012 08:29:20 AATKINSON: Prepared by sandwiching specimen between two Etnom films. 12/04/2012 09:12:00 AATKINSON: Using 0.02/0.02 soller slits. Forgot to update software from 0.04 to 0.02 for ASi. 12/04/2012 09:13:01 AATKINSON: See 5135-04 for post AUD: 12/04/2012 0919 AATKINSON: File Correction. New corrected filename: 563271	Other See analyst's task comments.
XR #10	XR10_TSH	1956	563271	AATKINSON	NC_Urge	Done	12/4/12	PTISHMACK	12/04/2012 09:20:23 AATKINSON: Correcting ASi	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314339	5135-02-01	Compound 184		Ambient	Legal sample		NC_Urgent	Completed	12/13/2012 14:33:09 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:53 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
DSC #4	Q_DSC_STD_10	172	564401	DPOWELL	NC_Urge	Done	12/11/12	KGUSHURST	12/10/2012 17:17:17 KGUSHURST: T0HSLP. Ensure that data is archived in I:\Aptuit Consulting 12/11/2012 09:45:25 DPOWELL: autosampler enabled refer to legal NB 5135-14 ref pan = 5135-14-01, R1	
KF-C	Q_KFC_STD_STROMBOLI	2202	564954	AATKINSON	NC_Urge	Rejected	12/13/12	KGUSHURST	12/12/2012 15:54:01 KGUSHURST: Expected weight loss is ~3%. Weight loss should be complete by 80 °C. 12/12/2012 16:08:34 KGUSHURST: Analyze in duplicate. 12/12/2012 17:11:22 KGUSHURST: See analyst prior to analyzing sample, please. 12/13/2012 13:08:10 AATKINSON: Bal 14. Fluka, Hydranal Coulomate AG Oven, LIMS 262121, lot SZBA3200, exp 10/15. Nitrogen flow ~175 mL/min See file 565063 for drift and blank. Samples prepared in glove box purged with nitrogen. 12/13/2012 14:14:21 AATKINSON: See 5135-24 and 25. AUD: 12/13/2012 1424 AATKINSON: File Correction. New corrected filename: 565225	Other see task comments
KF-C	Q_KFC_STD_STROMBOLI	2202	565225	AATKINSON	NC_Urge	Done	12/13/12	PTISHMACK	12/13/2012 14:25:13 AATKINSON: See 5135-25. Recalculation of initial run due to communication error.	
NMR #2	Q_NMR_liquids_C13_1D	1154	566281	PWHEELER	NC_Urge	Completed	12/20/12	KGUSHURST	12/20/2012 13:20:20 PWHEELER: see ntbk# 5143-10-02 for sample prep	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314339	5135-02-01	Compound 184		Ambient	Legal sample		NC_Urgent	Completed	12/13/2012 14:33:09 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:53 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_solids_N15_CPMA	1175	564394	PWHEELER	NC_Urge	Completed	12/13/12	PTISHMACK	12/10/2012 17:16:29 KGUSHURST: No initial equilibration. Ensure that data is archived in I:\Aptuit Consulting 12/12/2012 14:58:32 PWHEELER: N15 glycine chemical shift reference AUD: 12/12/2012 14:58:50 PWHEELER: sample analysis 12/13/2012 12:50:20 PWHEELER: Rotor# 27J1012, see ntbk# 5143-02-01 for sample prep	
NMR #2	Q_NMR_solids_N15_CPMA	1175	564917	PWHEELER	NC_Urge	Done	12/13/12	PTISHMACK		
SUBCONTRACTORS	ELEM_SUB_GALBR	407	564989	KGUSHURST	NC_Urge	Completed	12/21/12	KGUSHURST	12/13/2012 15:03:40 KGUSHURST: Subsample sent to Galbraith Labs. FedEx tracking number 474131093241. See 5135-21, -26. 12/21/2012 14:13:02 KGUSHURST: PO# APWLF-3498	
TGA #1	TG1_STD_10	247	564400	DPOWELL	NC_Urge	Completed	12/11/12	KGUSHURST	12/11/2012 09:51:58 DPOWELL: autosampler enabled sample in P1	
XR #10	Q_XR_PAN_TSH	2288	563696	AATKINSON	NC_Urge	Completed	12/6/12	KGUSHURST	12/06/2012 07:28:57 AATKINSON: Prepared by sandwiching specimen between two Etnom films. 12/06/2012 08:07:10 AATKINSON: See 5135-09 for post	
XR #9	XR9_BRAGG_BRENTANO	1671	564084	KGUSHURST	NC_Urge	Done	12/7/12	PTISHMACK	12/07/2012 17:31:46 KGUSHURST: Sample packed into well of holder 'ZBH 0.2mm well B', gently crushed larger particles with the back side of a spatula. Sample was flush with rim of well and leveled with a smooth glass slide then placed into PW1813/32 holder for measurement. 12/07/2012 17:49:43 KGUSHURST: Well is filled with sample. 12/07/2012 17:59:50 KGUSHURST: Post XRPD sample placed in clean vial - see 5135-11.	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314549		Dimethyl Sulfoxide-D6	12I-403	Light Sensitive	Flammable		NC_Urgent	Completed	12/07/2012 14:31:27 CGILMAN: (D, 99.9%) DLM-10-100 CAS#: 2206-27-1

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_liquids_H1_1D	1153	564194	PWHEELER	NC_Urge	Done	12/18/12	PTISHMACK	12/10/2012 10:28:43 PWHEELER: solvent certification as-received 12/18/2012 15:08:27 PWHEELER: see ntbk# 5143-04-01 for sample prep 12/19/2012 14:52:23 PSWEENEY: Form 138 #94716	
NMR #2	Q_NMR_liquids_H1_1D	1153	565945	PWHEELER	NC_Urge	Completed	12/19/12	PWHEELER	AUD: 12/18/2012 16:31:54 PWHEELER: Reanalysis with sieves 12/19/2012 08:54:40 PWHEELER: see ntbk# 5143-06-01 for sample prep	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314705	5135-12-01	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	Q_XR9_Bragg_Brentano	1712	564452	CGENDRON	NC_Urge	Completed	12/11/12	KGUSHURST	12/11/2012 11:00:38 CGENDRON: The entire damp sample was packed in ZBH 0.2 mm well A, the well was nearly full and leveled with a SS spatula then leveled with a smooth glass slide, placed in a PW1813/32 for easurement. The sample was still damp at the start of the measurement. 12/11/2012 11:26:13 CGENDRON: The measured material was returned to the original vial. 12/11/2012 11:57:18 CGENDRON: "easurement" should be measurement.	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314731	5135-15-03	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314731	5135-15-03	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	Q_XR9_Bragg_Brentano	1712	564518	CGENDRON	NC_Urge	Completed	12/11/12	KGUSHURST	12/11/2012 13:00:57 CGENDRON: The entire sample was packed in ZBH 0.2 mm well B, the well was nearly full and leveled with a smooth glass slide, placed in a PW1813/32 for measurement. 12/11/2012 13:30:26 CGENDRON: The measured material was returned to the original vial.	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314760	5135-15-05	Compound 184		Ambient	Legal sample		NC_Urgent	Completed	12/13/2012 14:33:11 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:55 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
DSC #4	Q_DSC_STD_10	172	564942	KLEACH	NC_Urge	Rejected	12/12/12	KGUSHURST	12/12/2012 15:38:44 KGUSHURST: T0HSLP. Save data to D\Legal Data 12/12/2012 16:24:53 KLEACH: Autosampler enabled refer to NB 5135-22 for sample prep. ref. pan: 5135-22-01, R1	Other See task comments
DSC #4	Q_DSC_STD_10	172	565125	KLEACH	NC_Urge	Completed	12/13/12	KGUSHURST	AUD: 12/13/2012 10:57:20 KGUSHURST: File 564942 contains incorrect temperature table. 12/13/2012 12:45:41 KLEACH: Autosampler enabled refer to NB 5135-23 for sample prep. ref. pan: 5135-23-01, R1	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314760	5135-15-05	Compound 184		Ambient	Legal sample		NC_Urgent	Completed	12/13/2012 14:33:11 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:55 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
KF-C	Q_KFC_STD_STROMBOLI	220	564955	AATKINSON	NC_Urge	Completed	12/13/12	KGUSHURST	12/12/2012 15:54:10 KGUSHURST: Expected weight loss is ~3%. Weight loss should be complete by 80 °C. 12/12/2012 16:08:43 KGUSHURST: Analyze in duplicate. 12/12/2012 17:11:34 KGUSHURST: See analyst prior to analyzing sample, please. 12/13/2012 13:08:13 AATKINSON: Bal 14. Fluka, Hydranal Coulomate AG Oven, LIMS 262121, lot SZBA3200, exp 10/15. Nitrogen flow ~175 mL/min See file 565063 for drift and blank. Samples prepared in glove box purged with nitrogen.	
SUBCONTRACTORS	ELEM_SUB_GALBR	407	564987	KGUSHURST	NC_Urge	Completed	12/21/12	KGUSHURST	12/13/2012 14:14:23 AATKINSON: See 5135-24 and 25. 12/13/2012 15:03:50 KGUSHURST: Subsample sent to Galbraith Labs. FedEx tracking number 474131093241. See 5135-21, -26. 12/21/2012 14:11:58 KGUSHURST: PO# APWLF-3498	
TGA #1	TG1_STD_10	247	564945	KLEACH	NC_Urge	Completed	12/12/12	KGUSHURST	12/12/2012 15:42:32 KGUSHURST: No initial equilibration. Please save data to D:\LegalData 12/12/2012 15:52:23 KLEACH: Autosamlper enabled Al pans	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314760	5135-15-05	Compound 184		Ambient	Legal sample		NC_Urgent	Completed	12/13/2012 14:33:11 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:55 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	Q_XR9_Bragg_Brentano	1712	564767	CGENDRON	NC_Urge	Completed	12/12/12	KGUSHURST	12/12/2012 09:12:23 KGUSHURST: Indexable quality patterns, please. 12/12/2012 11:34:08 CGENDRON: The entire sample was broken up and mixed with a SS spatula. A specimen was packed in ZBH 0.2 mm well A, full and leveled with a clean glass slide, placed in a PW1813/32 for measurement. 12/12/2012 12:12:24 CGENDRON: The measured material was saved in a clean, labeled vial; see 5135-20.	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314783	5135-17-01	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	12/13/2012 14:33:13 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:57 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
DSC #4	Q_DSC_STD_10	172	564944	KLEACH	NC_Urge	Rejected	12/12/12	KGUSHURST	12/12/2012 15:39:08 KGUSHURST: T0HSLP. Save data to D\Legal Data 12/12/2012 16:24:57 KLEACH: Autosampler enabled refer to NB 5135-22 for sample prep. ref. pan: 5135-22-01, R1	Other File contains incorrect temperature table

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314783	5135-17-01	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	12/13/2012 14:33:13 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:57 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
DSC #4	Q_DSC_STD_10	172	565124	KLEACH	NC_Urge	Completed	12/13/12	KGUSHURST	AUD: 12/13/2012 10:56:13 KGUSHURST: file 564944 contains incorrect temperature table 12/13/2012 10:56:40 KGUSHURST: T0HSLP. Save data to D\Legal Data 12/13/2012 12:45:46 KLEACH: Autosampler enabled refer to NB 5135-23 for sample prep. ref. pan: 5135-23-01, R1	
KF-C	Q_KFC_STD_STROMBOLI	220	564956	AATKINSON	NC_Urge	Completed	12/13/12	KGUSHURST	12/12/2012 15:54:17 KGUSHURST: Expected weight loss is ~3%. Weight loss should be complete by 80 °C. 12/12/2012 16:08:27 KGUSHURST: Analyze in duplicate. 12/12/2012 17:11:42 KGUSHURST: See analyst prior to analyzing sample, please. 12/13/2012 13:08:16 AATKINSON: Bal 14. Fluka, Hydranal Coulomate AG Oven, LIMS 262121, lot SZBA3200, exp 10/15. Nitrogen flow ~175 mL/min See file 565063 for drift and blank. Samples prepared in glove box purged with nitrogen.	
NMR #2	Q_NMR_liquids_H1_1D	115	565812	PWHEELER	NC_Urge	Completed	12/19/12	PTISHMACK	12/13/2012 14:14:25 AATKINSON: See 5135-24 and 25. 12/18/2012 10:28:25 KGUSHURST: use approximately 10mg in dry DMSO-d6 (similar as in 4063-30). 12/18/2012 10:57:34 KGUSHURST: see files 399864 and 400883 12/19/2012 09:52:49 KGUSHURST: 10.7mg see 5135-30 12/19/2012 10:33:25 PWHEELER: see ntbk# 5143-07-01 for sample prep	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314783	5135-17-01	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	12/13/2012 14:33:13 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:57 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_liquids_C13_1D	1154	565811	PWHEELER	NC_Urge	Completed	12/19/12	PTISHMACK	12/18/2012 10:27:15 KGUSHURST: use approximately 50mg in dry DMSO-d6 12/18/2012 10:28:37 KGUSHURST: similar to 4063-62 12/18/2012 11:03:05 KGUSHURST: see files 409909, 409910 12/19/2012 09:53:02 KGUSHURST: 50.3mg see 5135-30 12/19/2012 12:07:44 PWHEELER: see ntbk# 5143-07-02 for sample prep	
NMR #2	Q_NMR_solids_C13_CPMA	1169	565565	PWHEELER	NC_Urge	Done	12/18/12	PTISHMACK	12/18/2012 08:43:54 PWHEELER: Rotor# 26K1017, see ntbk# 5143-03-01 for sample prep	
NMR #2	Q_NMR_solids_N15_CPMA	1175	564959	PWHEELER	NC_Urge	Done	12/14/12	PTISHMACK	12/13/2012 16:17:41 PWHEELER: N15 glycine chemical shift reference AUD: 12/13/2012 16:17:26 PWHEELER: sample analysis 12/14/2012 14:24:30 PWHEELER: Rotor# 26K1017, see ntbk# 5143-03-01 for sample prep	
NMR #2	Q_NMR_solids_N15_CPMA	1175	565268	PWHEELER	NC_Urge	Done	12/14/12	PTISHMACK		
SUBCONTRACTORS	ELEM_SUB_GALBR	407	564988	KGUSHURST	NC_Urge	Completed	12/21/12	KGUSHURST	12/13/2012 15:03:57 KGUSHURST: Subsample sent to Galbraith Labs. FedEx tracking number 474131093241. See 5135-21, -26. 12/21/2012 14:12:14 KGUSHURST: PO# APWLF-3498	
TGA #1	TG1_STD_10	247	564946	KLEACH	NC_Urge	Completed	12/12/12	KGUSHURST	12/12/2012 15:42:37 KGUSHURST: No initial equilibration. Please save data to D:\LegalData 12/12/2012 15:52:26 KLEACH: Autosamlper enabled Al pans	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314783	5135-17-01	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	12/13/2012 14:33:13 CGILMAN: Sent subsample to Galbraith FedEX 474131093241 12/17/2012 16:13:57 CGILMAN: APWLF-3498

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	Q_XR9_Bragg_Brentano	1712	564765	CGENDRON	NC_Urge	Completed	12/12/12	KGUSHURST	12/12/2012 09:11:15 KGUSHURST: Indexable quality patterns, please. 12/12/2012 12:14:22 CGENDRON: The entire sample was broken up and mixed with a SS spatula. A specimen was packed in ZBH 0.2 mm well B, full and leveled with a clean glass slide, placed in a PW1813/32 for measurement. 12/12/2012 12:51:33 CGENDRON: The measured material was saved in a clean, labeled vial; see 5135-20.	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314801	5135-18-01	Compound 184		Ambient	Fume Hood 3		NC_Urgent	Done	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	XR9_BRAGG_BRENTANO	1671	564724	KGUSHURST	NC_Urge	Done	12/12/12	PTISHMACK	12/12/2012 00:51:47 KGUSHURST: The entire sample was packed into Si holder ZBH 0.2mm well B. Well is full and sample leveled to rim of holder using smooth glass slide. 12/12/2012 01:21:20 KGUSHURST: Sample returned to original vial post XRPD analysis.	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314802	5135-17-05	Compound 184		Ambient	Fume Hood 3		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
314802	5135-17-05	Compound 184		Ambient	Fume Hood 3		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	Q_XR9_Bragg_Brentano	1712	564766	CGENDRON	NC_Urge	Completed	12/12/12	KGUSHURST	12/12/2012 09:11:50 KGUSHURST: Indexable quality patterns, please. 12/12/2012 12:53:22 CGENDRON: The entire sample was broken up and mixed with a SS spatula. A specimen was packed in ZBH 0.2 mm well A, full and leveled with a clean glass slide, placed in a PW1813/32 for measurement. 12/12/2012 13:48:28 CGENDRON: The measured material was saved in a clean, labeled vial; see 5135-20.	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315083	5135-27-01	Compound 184		Ambient	Analytical Bin		NC_Urgent	Done	12/14/2012 10:43:11 KGUSHURST: 5135-18-01 left open in fume hood, 2 days

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	XR9_BRAGG_BRENTANO	1671	565362	KGUSHURST	NC_Urge	Done	12/14/12	PTISHMACK	12/14/2012 10:44:16 KGUSHURST: Use Compound 184 program 12/14/2012 10:44:49 KGUSHURST: Use Compound 184 program; save to D:\Legal Data 12/14/2012 10:51:27 KGUSHURST: And same configuration as 564765 12/14/2012 12:58:13 KGUSHURST: Sample packed into 0.2mm well of Si ZBH well B. Full and leveled to rim of well with smooth glass slide. Well placed in PW1813/32 holder. 12/14/2012 13:33:28 KGUSHURST: Post XRPD sample returned to original vial (all of sample was utilized for analysis).	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments

Project ID: EL20100011

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315084	5135-27-02	Compound 184		Ambient	Analytical Bin		NC_Urgent	Done	12/14/2012 10:43:44 KGUSHURST: 5135-17-05 left open in fume hood, 2 days

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	XR9_BRAGG_BRENTANO	1671	565363	KGUSHURST	NC_Urge	Done	12/14/12	PTISHMACK	12/14/2012 10:44:43 KGUSHURST: Use Compound 184 program; save to D:\Legal Data 12/14/2012 10:51:45 KGUSHURST: And same configuration as 564765 12/14/2012 12:06:13 KGUSHURST: Sample packed into 0.2mm well of Si ZBH well A. Full and leveled to rim of well with smooth glass slide. Well placed in PW1813/32 holder. 12/14/2012 12:48:38 KGUSHURST: Post XRPD sample saved in clean vial. 5135-27-03	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315436	5135-32-01	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_liquids_C13_1D	1154	566240	PWHEELER	NC_Urge	Completed	12/20/12	PTISHMACK	12/20/2012 10:25:36 PWHEELER: see ntbk# 5143-09-01 for sample prep	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315452	5135-32-02	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_liquids_C13_1D	1154	566260	PWHEELER	NC_Urge	Completed	12/20/12	PTISHMACK	12/20/2012 11:57:36 PWHEELER: see ntbk# 5143-10-01 for sample prep	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315467	5135-32-04	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_liquids_C13_1D	1154	566295	PWHEELER	NC_Urge	Completed	12/20/12	PTISHMACK	12/20/2012 14:53:16 PWHEELER: see ntbk# 5143-10-03 for sample prep	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315469	5135-32-05	Compound 184		Ambient	Analytical Bin		NC_Urgent	Completed	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
NMR #2	Q_NMR_liquids_C13_1D	1154	566297	PWHEELER	NC_Urge	Completed	12/20/12	PTISHMACK	12/20/2012 16:05:18 PWHEELER: see ntbk# 5143-10-04 for sample prep	

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315535	5135-30-03	Compound 184		Ambient	Analytical Bin		NC_Urgent	Logged	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
DSC #4	Q_DSC_STD_10	172	566460	KLEACH	NC_Urge	Completed	12/21/12	KGUSHURST	12/21/2012 11:29:38 KGUSHURST: TOHSLP analyze similar to file 565124 save data in D:\LegalData 12/21/2012 12:21:35 KLEACH: Autosampler enabled refer to NB 5143-11 for sample prep. ref. pan: 5143-11-01, R1	
KF-C	Q_KFC_STD_STROMBOLI	2202	566459	AATKINSON	NC_Urge	Completed	12/21/12	KGUSHURST	12/21/2012 11:27:56 KGUSHURST: sample contains ~6% water. Analyze in duplicate and similarly to file 564956. Sample needs XRPD analysis (please save some sample). 12/21/2012 11:42:29 AATKINSON: Bal 17. Fluka, Hydranal Coulomat AG Oven, LIMS 262121, exp 10/15. See file 566428 for drift. Sample and blank prepared at ambient. 12/21/2012 11:45:27 AATKINSON: See 5135-34	
SUBCONTRACTORS	ELEM_SUB_GALBR	407	566493	KGUSHURST	NC_Urge	Received		KGUSHURST		
TGA #1	TG1_STD_10	247	566461	KLEACH	NC_Urge	Completed	12/21/12	KGUSHURST	12/21/2012 11:30:27 KGUSHURST: no initial equilibration please save data in D:\LegalData 12/21/2012 12:03:55 KLEACH: Autosampler enabled Al pans	

Project ID: EL20100011

Sample Information

Lims No	Notebook #	Compound	Lot #	Storage	Retain Location	Hazard Code	Priority	Status	Sample Comments
315535	5135-30-03	Compound 184		Ambient	Analytical Bin		NC_Urgent	Logged	

Tests assigned:

Instrument	Test Name	Test Code	Filename	Assign To	Priority	Status	Start Date	Approver	Task Comment	Reason for rejection
XR #9	Q_XR9_Bragg_Brentano	1712	566458	CGENDRON	NC_Urge	Completed	12/21/12	KGUSHURST	12/21/2012 11:47:20 KGUSHURST: Use Compound 184 program and 0.2mm well ZBH holder; same configuration as file 564765 (SSi=.02, SSd=.04, 1/8 DS, 1/4 ASi, 0.0167, 100 sec, spin). 12/21/2012 13:19:35 CGENDRON: A specimen was packed in ZBH 0.2mm well B, full and leveled with a smooth glass slide, placed in aPW1813/32 holder for measurement. 12/21/2012 13:49:51 CGENDRON: The measured material was saved; see 5135-35.	