

PRO EL20151326

Mobile Phase Prep:

6294-01-01 - Dissolved 7.5252g ammonium dihydrogen phosphate in 3000 mL H<sub>2</sub>O. Adjusted pH of the solution to 7.30 by adding ammonium hydroxide dropwise. Added 1000 mL ACN to the solution. Mixed well by stirring. Prep 11/13/15

- Balance #13 used
- Class A grad cylinders used
- NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> <sup>Drum #16116</sup> ~~H<sub>2</sub>O~~ - 399747, exp 7/31/18
- H<sub>2</sub>O - Elga Flex 2 system, MR = 13.2
- NH<sub>4</sub>OH - 291099, exp 2/26/17
- ACN - 398454, exp 3/31/17

Bromfene solution, ~ 0.07%

6294-01-02 - Transferred 7.06 mg Bromfene Sodium to a class 10 mL volumetric flask

- Added ~ 6 mL ACN, solids did not dissolve
- Added a few drops of H<sub>2</sub>O, solids dissolved
- Dilute solution to volume with H<sub>2</sub>O.

- Bromfene Sodium - 405306, exp 10/6/20

HPLC #1 setup:

Column: Shiseido Capcell Pore C18 AG-120, 4.6 x 150 mm, 5 μm LIMS # 405201

Column Temp: 25°C

Flow Rate: 2 mL/min

Injection volume: 10 μL

DAU: UV @ 266 nm

Mobile phase: 6294-01-01

Method saved as 62940101.m

*[Handwritten signature]* 10/13/15 *[Handwritten signature]*

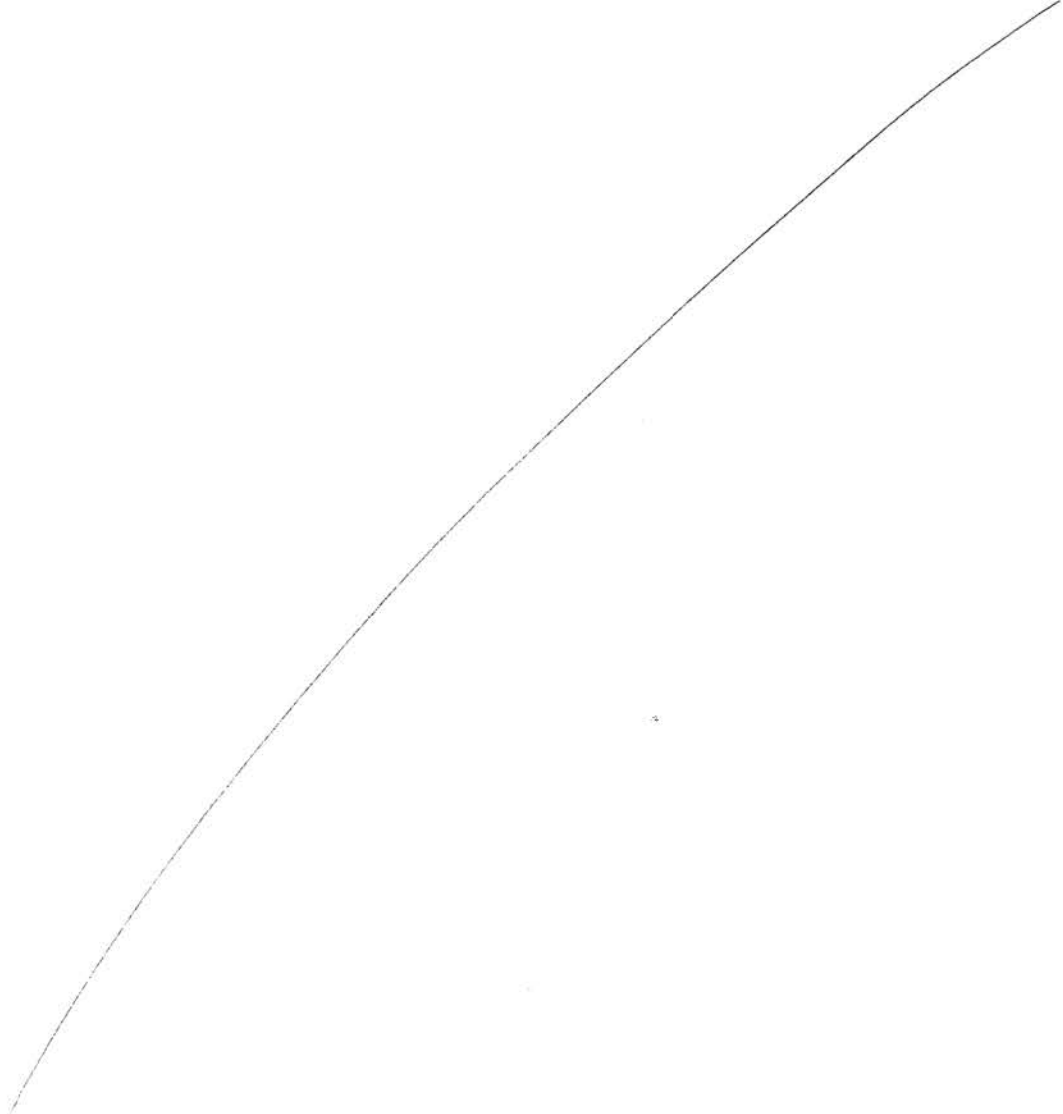
BL 10/14/15  
BL 20151326

6294

Mobile phase prep w/ Triethylamine:

6294-02-01 = Combined 500 mL 6294-01-01 (class A grad cylinder) and 0.5 mL triethylamine (pipet 234542, cal due 1/31/16). Mixed well.  
Exp 11/13/15

Triethylamine - 394339, exp 11/30/17



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10/14/15

*[Handwritten initials]*

IC 10-11  
10/15/15

EL 20151026

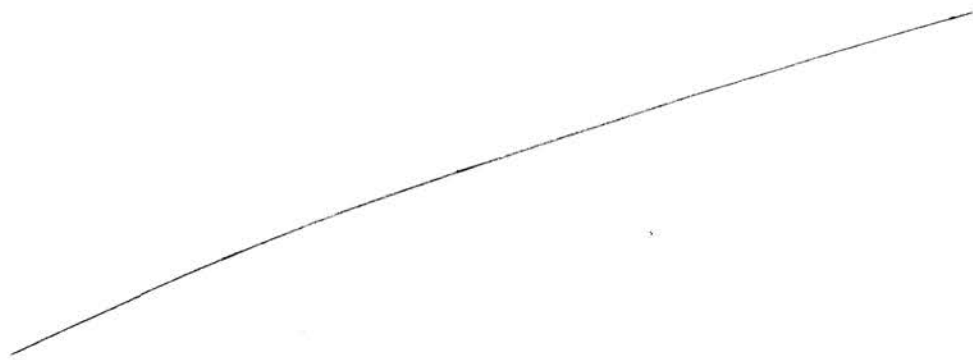
The following samples of Protensa are placed in oven #1 set at 60°C :

- 403472
- 403473
- 403474
- 403475
- 403476
- 403477
- 403478
- 403479
- 403480
- 403481
- 403482
- 403483
- 403484
- 403485
- ~~403486~~ 403488
- 403487 <sup>is</sup>
- ~~403488~~
- ~~403489~~
- 403490

25 ml 10/20/15

} These three bottles are not placed in the oven.

Temperature at start: 60.0°C, thermometer 383675, cal due 2/29/16



*H/M*

10/20/15

*M. G. M.*

10/20/15

BL2015 1326

Placebo solution prep:

6294-01-a1 - Dissolve the following reagents in  $\approx 30$  mL  $H_2O$ :

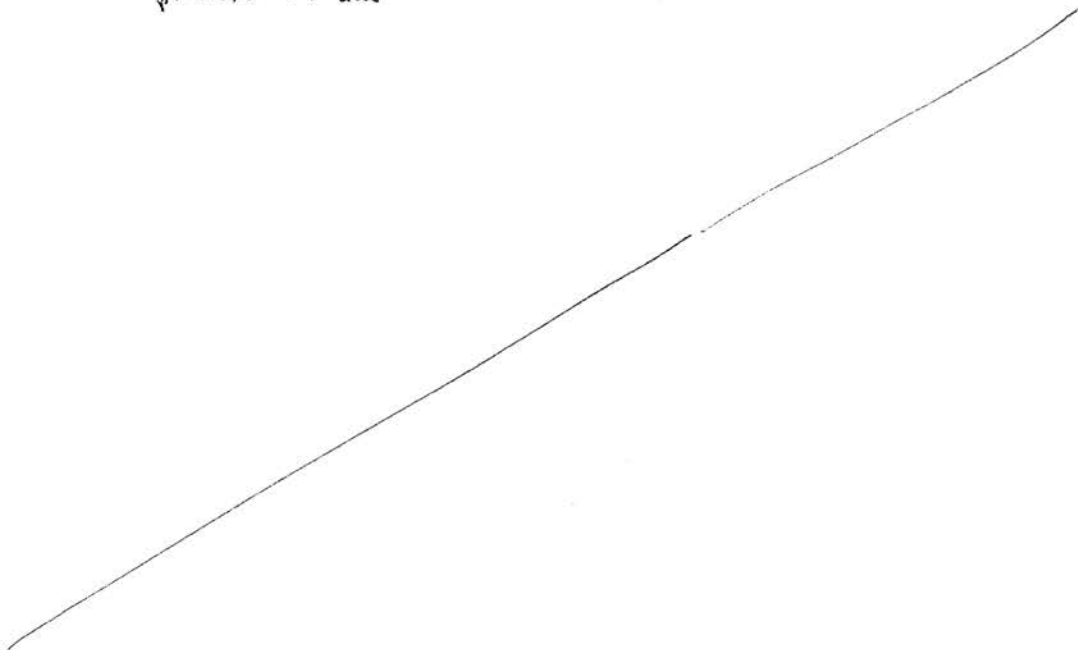
Compound	LTMS	Exp	mass (g) <sup>9 REAM 11/3/15</sup>
Boric Acid	403345	1/31/17	1.3961
Sodium Borate	403344	3/17/17	0.7466
Disodium Edatate	292442	5/18/16	0.0204
Pyridone	403368	1/31/17	0.9923
Sodium Sulfite	403343	12/31/17	0.2010
Tyloxapol	403369	9/16/20	<del>0.0208</del> <sup>0.0208</sup> 11/3/15
Benzalkonium Chloride (575)	403342	4/13/17	0.0056

- pH of this solution = 7.84

- A portion of this solution is syringe filtered through a 0.2  $\mu$ m PVDF membrane and stored in an HPLC vial.

Balance #18 used

pH meter #4 used



MPL

11/3/15

MPL

11/3/15  
11/16/15

6294

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Mobile Phase Prep:

- 6294-05-01 - Dissolved 7.9231g ammonium dihydrose phosphate in 3000 mL H<sub>2</sub>O
- Adjusted pH of solution to 7.30 by adding ammonium hydroxide dropwise
- Added 1000 mL ACN. Mixed thoroughly
- Added 4 mL <sup>from 11/16/15</sup> triethylamine. Mixed thoroughly.  
Exp 12/16/15

Balance #18 used

Class A grad cylinders used

NH<sub>4</sub>H<sub>2</sub>PO<sub>4</sub> <sup>from 11/16/15</sup> ~~11/16/15~~ - 399747, exp 7/31/18

H<sub>2</sub>O - Elga Flex 2 system, MΩ = 18.2

NH<sub>4</sub>OH - 291099, exp 2/26/17

ACN - 395741, exp 1/31/17

Triethylamine - 394339, exp 4/30/17

*[Signature]*

11/16/15

*[Signature]*

11/16/15

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