

PRO EL20151326

Mobile Phase Prep:

6294-01-01 - Dissolved 7.5252g ammonium dihydrogen phosphate in 3000 mL H₂O. Adjusted pH of the solution to 7.30 by adding ammonium hydroxide Impure. Added 1000 mL ACN to the solution. Mixed well by stirring. Prep 11/13/15

Balance #13 used

Class A grad cylinders used

NH₄H₂PO₄ ^{Primo 11/6/16} ~~H₂O~~ - 399747, exp 7/31/18

H₂O - Elga Flex 2 system, M2 = 13.2

NH₄OH - 291079, exp 2/26/17

ACN - 398454, exp 3/31/17

Bromfene solution, ~ 0.07%

6294-01-22 - 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 H₂O, solids dissolved

- Dilute solution to volume with H₂O.

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

HPLC #1 setup:

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

Column Temp: 25°C

Flow Rate: 2 mL/min

Injection volume: 10 µL

DAU: 4V @ 266 nm

Mobile phase: 6294-01-01

Method saved as 62940101.m

[Signature]

10/13/15

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10/13/15

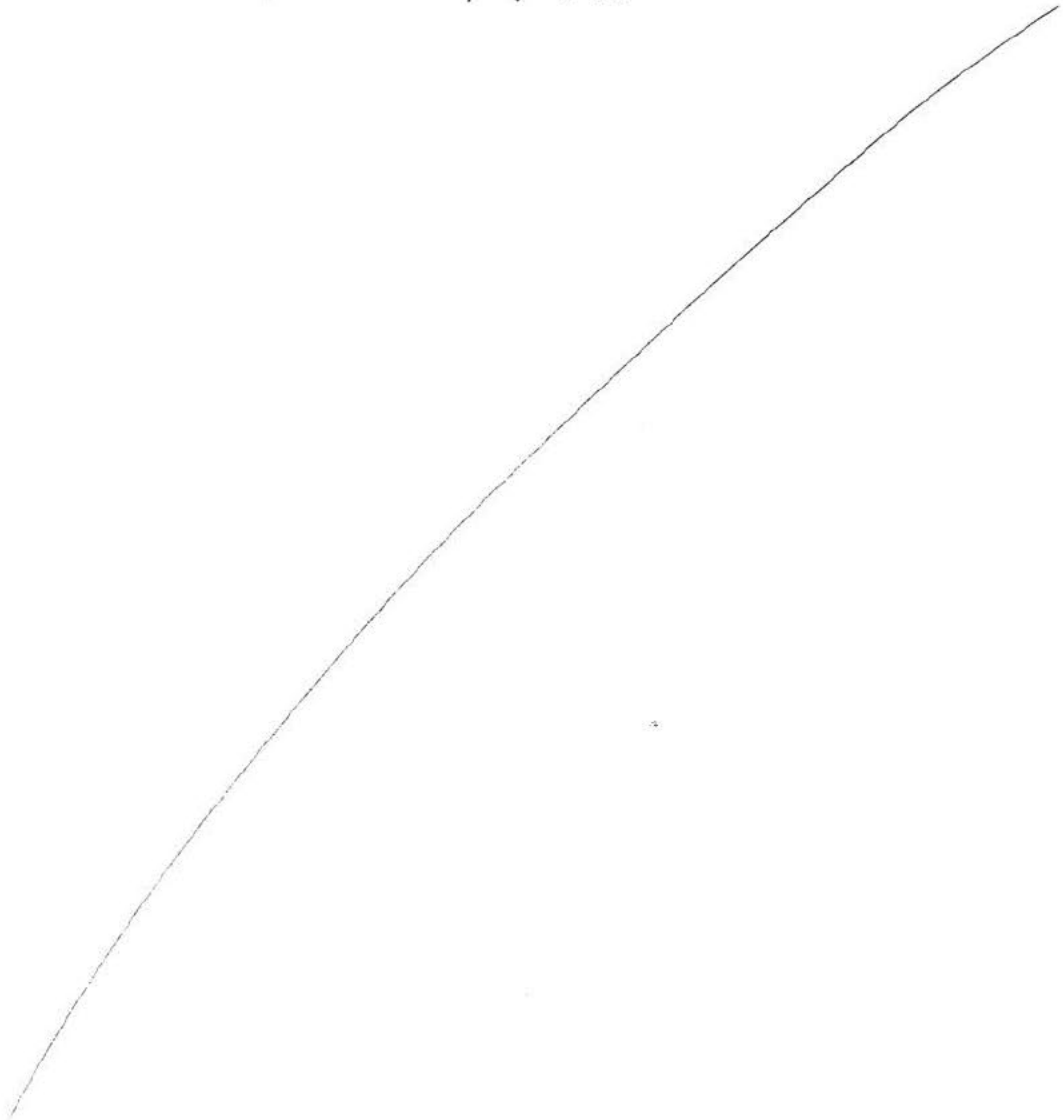
10/14/15
EL20151326

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 - 344339, exp 11/30/17



[Handwritten signature]

10/14/15

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11/15/15
10

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

403472

403473

403474

403475

403476

403477

403478

403479

403480

403481

403482

403483

403484

403485

25 at 10/20/15

~~403486~~ 403488

403487

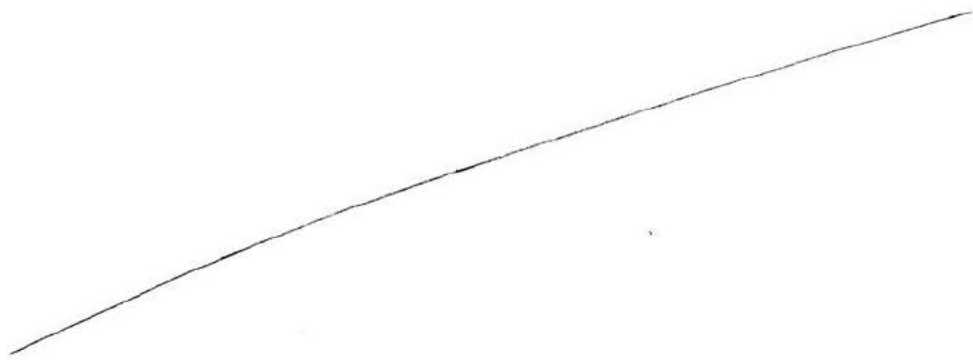
~~403488~~

~~403489~~

403490

} These three bottles are not placed in the oven.

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



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10/20/15

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10/20/15

BL2015 1326

Placebo solution prep:

6294-01-a1 - Dissolve the following reagents in ≈ 30 mL H₂O:

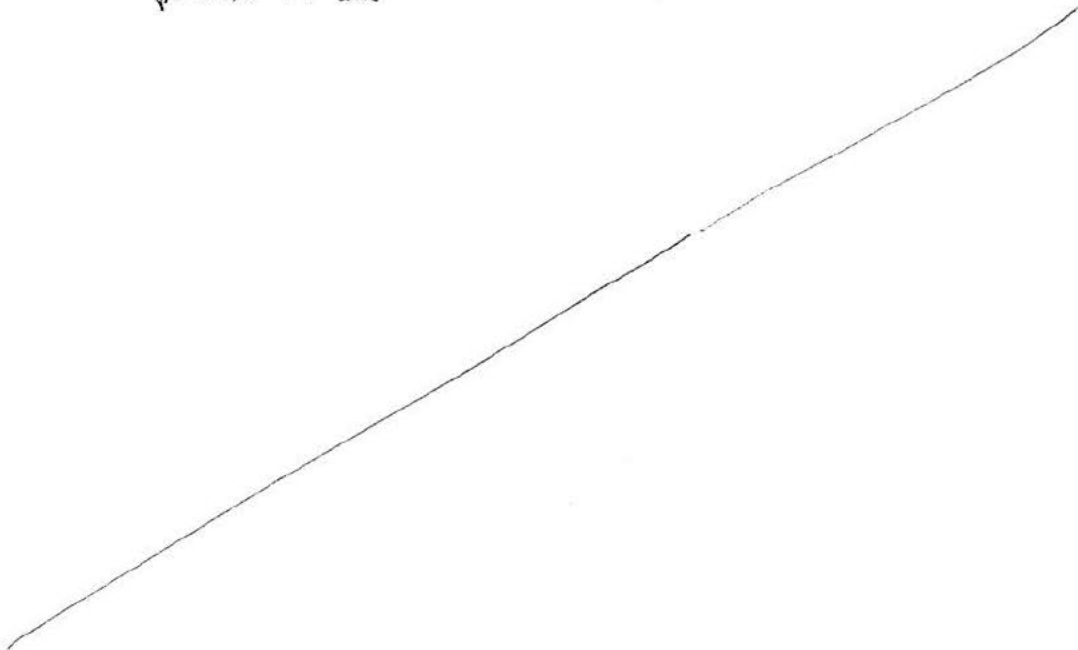
| Compound | LTMS | Exp | mass (g) ^{22.04 11/3/15} |
|--------------------------------|--------|----------|---|
| Boric Acid | 403345 | 1/31/17 | 1.3961 |
| Sodium Borate | 403344 | 3/17/17 | 0.7466 |
| Disodium Edatate | 292442 | 5/18/16 | 0.0204 |
| Paribone | 403368 | 1/31/17 | 0.9923 |
| Sodium Sulfite | 403343 | 12/31/17 | 0.2010 |
| Tyloxapol | 403369 | 9/16/20 | 0.0208 ^{22.04 11/3/15} 0.0208 |
| Benzalkonium Chloride (S27) | 403342 | 4/13/17 | 0.0086 |

- pH of this solution = 7.84

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

Balance #18 used

pH meter #4 used



M/R

11/3/15

M/R

 11/3/15
 11/3/15
 11/3/15

6294

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Mobile Phase Prep:

- 6294-05-01 - Dissolved 7.9231g ammonium dihydrose phosphate in 3000 mL H₂O
 - Adjusted pH of solution to 7.30 by adding ammonium hydroxide dropwise
 - Added 1000 mL ACN. Mixed thoroughly
 - Added 4 mL triethylamine. Mixed thoroughly.
- Rep 12/16/15 ^{JEK 11/16/15}

Balance #18 used

Class A grad cylinders used

NH₄H₂PO₄ ~~11/16/15~~ ^{JEK 11/16/15} - 395747, exp 7/31/18

H₂O - Elga Flex 2 system, MΩ = 18.2

NH₄OH - 291099, exp 2/26/17

ACN - 395741, exp 1/31/17

Triethylamine - 394339, exp 11/30/17

MJR

11/16/15

JEK

11/16/15

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