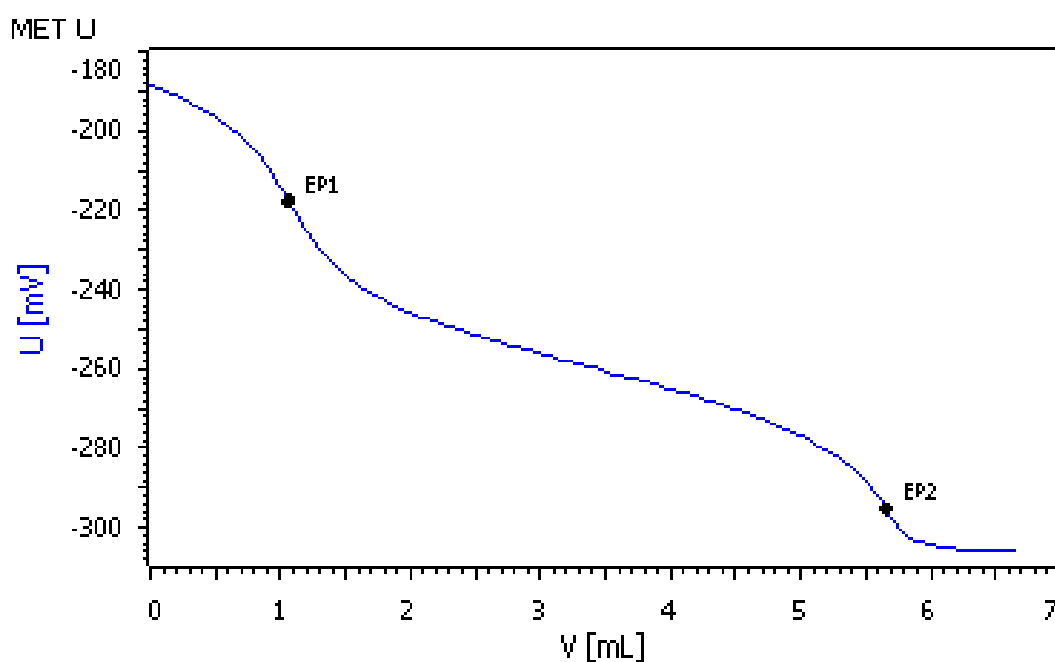


Determination of sulfate in purgative and expectorant salts according to Ph. Eur. 8.0



The composition of purgative and expectorant salts is of major interest as these products are used as medication. The sulfate content is determined by automated, potentiometric titration using EGTA as titrant.

Method description

Sample

Purgative and catarrh expectorant salts

Sample preparation

The salts are dissolved in deionized water and their pH is adjusted 3.5 with hydrochloric acid.

Configuration

| | |
|--|------------|
| 905 Titrand | 2.905.0010 |
| 800 Dosino, 5x | 2.800.0010 |
| 814 USB Sample Processor (1T/1P) | 2.814.0010 |
| 772 Pump Unit | 2.772.0110 |
| Dosing unit 50 mL | 6.3032.250 |
| Dosing unit 20 mL | 6.3032.220 |
| Dosing unit 10 mL, 2x | 6.3032.210 |
| Dosing unit 2 mL | 6.3032.120 |
| 802 Rod stirrer | 2.802.0020 |
| Stirring propeller | 6.1909.050 |
| Sample rack, 16 x 150 mL | 6.2041.320 |
| Titration head, 6x NS 14 and 3x NS 9 openings | 6.1458.010 |
| Sample beakers, glass, 16 x 150 mL | - |
| Combined polymer Ca ISE, Electrolyte $c(\text{NH}_4\text{NO}_3) = 1 \text{ mol/L}$ | 6.0510.100 |
| Ecotrode Plus, Reference electrolyte $c(\text{KCl}) = 3 \text{ mol/L}$ | 6.0262.100 |
| 2x Electrode Cable 2 m / F | 6.2104.030 |

Solutions

| | |
|--------------------------------|---|
| Titration | $c(\text{EGTA}) = 0.05 \text{ mol/L}$, if possible this solution should be bought from a supplier. |
| Barium chloride solution | $c(\text{BaCl}_2) = 0.05 \text{ mol/L}$, if possible this solution should be bought from a supplier. |
| Buffer solution | Ammonia buffer with $\text{pH} = 10$, if possible this solution should be bought from a supplier. |
| CaCl_2 ionic standard | $c(\text{CaCl}_2) = 0.10 \text{ mol/L}$, if possible this solution should be bought from a supplier. |

Analysis

Approx. 5 mL prepared sample solution is pipetted into the titration vessel and placed on the rack. Just before the titration, 50 mL water, 0.5 mL calcium standard and 7.5 mL barium chloride are automatically added to the sample. After a reaction time of 3 min, 5 mL buffer solution is added and the solution is titrated with $c(\text{EGTA}) = 0.05 \text{ mol/L}$ until after the second equivalence point using the Ca ISE.

The blank is determined the same way as the sample, just without sample solution.

Parameters

| | |
|-------------------|-----------|
| Mode | MET U |
| Signal drift | 50 mV/min |
| Pause | 30 s |
| Stirrer speed | 8 |
| Min. waiting time | 5 s |
| Max. waiting time | 26 s |
| Volume increment | 0.1 mL |
| EP criterion | 10 |
| EP recognition | all |

Results

| Sample | Content / % | s(rel) / % |
|--------------------------|-------------|------------|
| Purgative salt | 28.96 | 0.29 |
| catarrh expectorant salt | 30.06 | 0.44 |