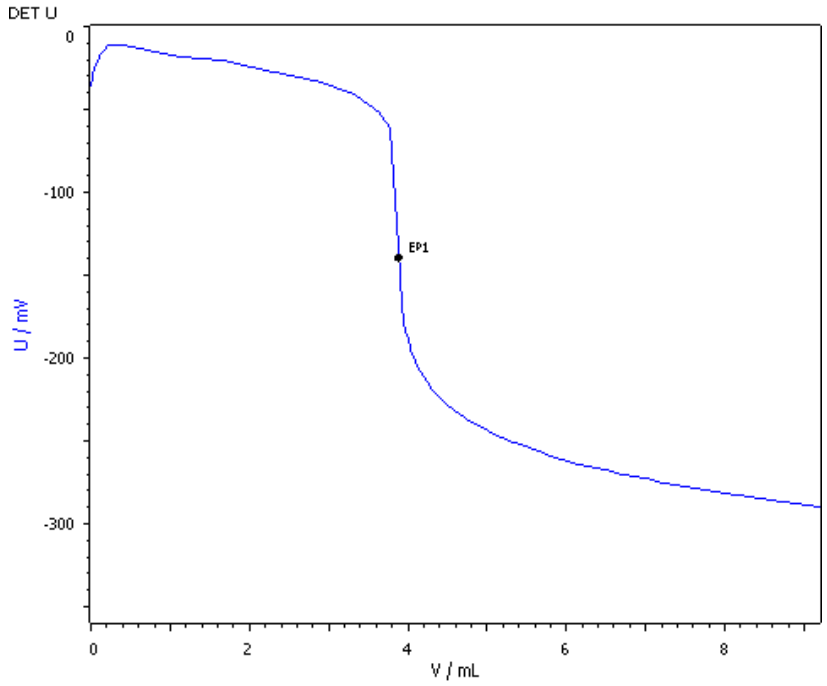
Titration Application Note T–152

Automated determination of mercaptans in refinery products



Mercaptans in refinery products can be determined by potentiometric titration with silver nitrate. This Application Note describes their automatic determination in a gas oil sample.

Metrohm

Method description

Sample

Gas oil

Sample preparation

No sample preparation is required.

Configuration

815 Robotic USB Sample Processor XL (2T/2P)	2.815.0110
786 Swing Head, 2x	2.786.0040
843 Pump Station	2.843.0020
772 Pump Unit	2.772.0110
907 Titrando	2.907.0010
800 Dosino, 4x	2.800.0010
Robotic arm with holder for titration head, right swinging	6.1462.070
Robotic DIS-COVER	6.1462.080
Titration Head, 3x SGJ 14	6.1458.040
Lid for 120 mL sample beaker, 59x	6.9920.164
Sample rack 59 x 120 mL	6.2041.840
Sample beaker 120 mL	6.1459.300
Dosing unit 2 mL	6.3032.120
Dosing unit 20 mL, 2x	6.3032.220
Dosing unit 50 mL	6.3032.250
T pieces, 3x	
Ag Titrode with sulfide coating	6.0430.100S

Solutions

Titrant	$c(AgNO_3)$ = 0.01 mol/L 100 mL $c(AgNO_3)$ = 0.1 mol/L is pipetted into a 1000 mL volumetric flask and mixed with 80 mL deion. H_2O. The flask is then filled up to the mark with isopropanol. The titrant is degassed by passing through a constant stream of nitrogen.
Solvent	2.7 g sodium acetate trihydrate is dissolved in 25 mL deion. H_2O. The solution is then added to 975 mL isopropanol and 4.6 mL glacial acetic acid. The solvent is degassed by passing through a constant stream of nitrogen.

Analysis

The titration beaker is flushed with nitrogen before an appropriate sample amount (see table below) is pipetted into the titration beaker. The titration beaker is again flushed with nitrogen, covered with the DIS-COVER and put on the sample rack. 30 mL solvent is added and the solution is immediately titrated with $c(AgNO_3)$ = 0.01 mol/L until after the first or second equivalence point, depending on the sample.

If the sample contains only H_2S or mercaptans only one equivalence point is found, if the sample contains both two equivalence points are found.

Expected sulfur content / (mg S / kg sample)	Sample size / mL
1–50	50
50–100	25
100–300	10
300–500	5

Parameters

Mode	DET U
Stirring rate	8
Pause	20 s
Signal drift	10 mV/min
Max. waiting time	20 s
Meas. point density	2
Min. increment	50 µL
ERC	30
EP recognition	all

Results

Mean result (n = 8)

RSH (mg S/kg)	s(rel) / %, n = 8
122.14	1.61

Metrohm