SI Analytics
a xylem brand

Mercaptan sulphur in Gasoline and Kerosene Aviation Turbine and Distillate Fuels according to ASTM D 3227

Application

Use

Determination of mercaptan sulphur in a range from 3 mg/kg – 100 mg/kg.
.

Appliances

- Titrator: TL 7000/TL 7750 M1
- Basic device
- Magnetic stirrer TM 235
- 10 mL Exchange unit WA 10, with amber glass bottle for the titrant, complete

Electrodes

- Electrode: AgS 62 RG or AG 1100 (sulphidized) + A 1180/H 1180 glass electrode
- Electrode cable: L 1 A or L 1 A + L 1 N

Reagents

- Titrant: AgNO3 0.01 mol/l in isopropanol (IPA)
- Titer determination: with KI or NaCl solution 0.1 mo/l
- Solvent: Sodium acetate trihydrate solution in IPA

Description

Preparation and standardization of the K-I and alcoholic $AgNO_3$ solutions

Dissolve 17 g (weigh to 0.01g) of **KI** in 100 ml of water in a 1 L volumetric flask and dilute to 1 L. Calculate the exact molarity. It is also possible to use NaCl instead of KI. Commercial available 0.1 mol/L solutions for NaCl can be also used.

Use a standard ampoule **0.1 m $AgNO_3$** and fill it up with IPA (99%) in a 1 L volumetric flask.

Add 6 drops of conc. HNO3 to 100 ml of water in a 250 ml tall form beaker. Remove oxides of nitrogen by boiling for 5 min. Cool to ambient temperature. Pipette 5 ml of a 0.1 m KI solution into the beaker and titrate with the 0.1 m $AgNO_3$.to an inflection point.

The **0.01 m $AgNO_3$** should be prepared daily by dilution of the 0.1 m standard. Calculate the exact molarity.

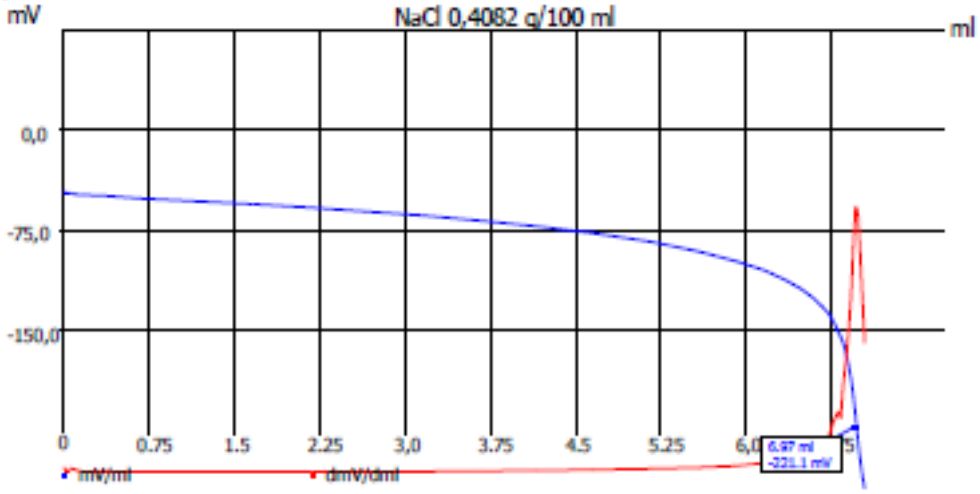
Application

Method for titer determination

Page 1

GLP documentation

Titration graph



Method data

Method name:	AgNO3 0,01 M in IPA	Titration duration:	7 m 7 s
End date:	25.03.15	End time:	17:31:48

Titration data

Sample ID:	NaCl 0,4082 g/100 ml	Pattern:	1.000 ml
Start mV:	-46.6 mV	End mV:	-267.9 mV
EQ:	6.970 ml / -221.1 mV	Titer:	0.01002 mol/l
Mean value:	---	RSD:	---

Calculation formula

Titer:	(V*F2)/((EQ1-B)*M*F1) -> WA	Mol (M):	1.00000
Pattern (V):	1.000 ml (m)	Factor 2 (F2):	69.8458
Blank value (B):	0.0000 ml	Factor 1 (F1):	1000.0000
Statistics:	1 from 3		

Application

Page 2

Method data overall view

Method name:	AgNO3 0,01 M in IPA	Created at:	03/25/15 17:24:34
Method type:	Automatic titration	Last modification:	03/25/15 17:24:34
Measured value:	mV	Damping settings:	strong
Titration mode:	Dynamic	Documentation:	GLP
Dynamic:	User-defined:	Max. step size:	0.5000 ml
		Slope max ml:	15.00 mV/min
		Min. step size:	0.0200 ml
		Slope min ml:	230.00 mV/min
Measuring speed / drift:	User-defined:	minimum holding time:	05 s
		maximum holding time:	15 s
		Measuring time:	04 s
		Drift:	05 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
End value:	Off		
EQ:	On (1)		
Slope value:	User-defined	Value:	400

Dosing parameter

Dosing speed:	100.00 %	Filling speed:	30 s
Maximum dosing volume:	10.00 ml		

Unit values

Unit size:	10ml
Unit ID:	10035433
Reagent:	AgNO3 in IPA
Batch ID:	no entry
Concentration [mol/l]:	0.01000
Determined at:	03/25/15 22:33:43
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	05/04/12
Last modification:	03/25/15 15:33:54

Preparation of the Solvent

Dissolve 2.7 g of sodium acetate trihydrate in 20 ml oxygen free water and pour into 975 ml 2-propanol (IPA). Add 4.6 ml of glacial acetic acid. Remove dissolved oxygen with a rapid stream of nitrogen for 10 min each day prior to use. Keep protected from the atmosphere.

Preparation of the $CdSO_4$ solution

Dissolve 150 g of CdSO4 ($3CdSO_4$ * 8 H_2O) in water. Add 10 ml of H2SO4 (1:5) and dilute to 1L with water

Connection of the electrode

The AgS 62 RG is directly connected to pH/mV socket with cable L 1 A.

Option:
The A 1180 is connected with the cable L 1 A to pH/mV socket". The Ag 1100 (sulphidized) is connected with cable L 1 N to the reference socket.

Titration

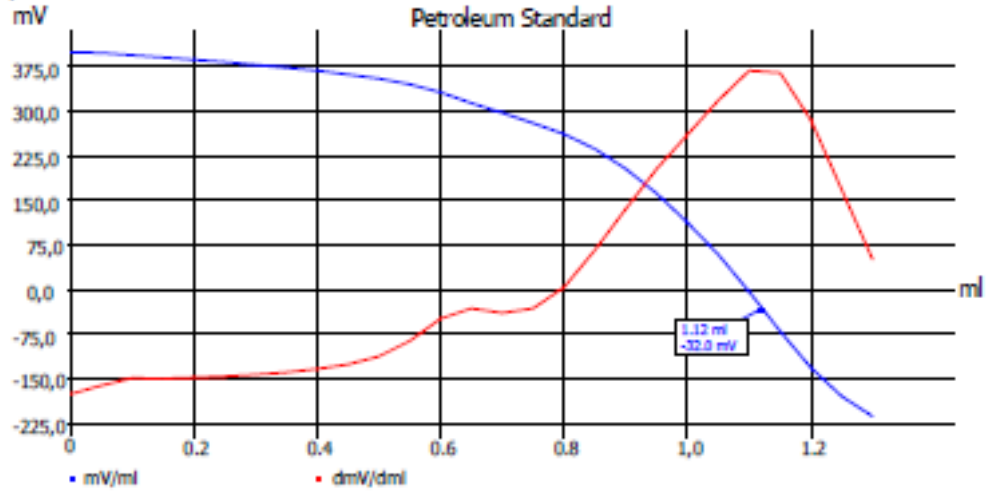
Preparation of the sample: Removal of Hydrogen Sulfide

Test the sample quality for Hydrogen sulfide (H2S) by shaking 5 ml of the sample with 5 ml of the acid CdSO4 solution. If no precipitate appears, proceed with the analysis of the sample described below. If a yellow precitipate appears, remove the H2S in the following matter: Place a quantity of the sample (i.e 200 ml) , three, two, four times that required for the analysis, in a separatory funnel containing a volume of the acid CDSO4 solution equal to one half of the sample (i.e. 100 ml) and shake vigorously. Draw off and discard the aqueous phase, and wash the sample with three 25-30 ml portions of water, withdrawing the water after each washing. Repeat the extraction with CdSO4 until all of the H2S has been removed.

Measure with a pipet or weigh 20 to 30 ml of the original or treated sample into a 150 ml titration beaker (titration vessel) containing 70 ml of the solvent mixture. Immediately immerse the electrodes and buret tip into the sample. Titrate with the 0.01 m AgNO3 with the attached titration parameters. After the titration the electrodes should be rinsed with alcohol and then with water.

GLP documentation

Titration graph



Method data

Method name:	R-SH without H2S	Titration duration:	6 m 40 s
End date:	25.03.15	End time:	19:09:14

Titration data

Sample ID:	Petroleum Standard	Pattern:	20.000 ml
Start mV:	400.7 mV	End mV:	-213.9 mV
EQ:	1.121 ml / -32.0 mV	R-SH:	21.9 ppm

Calculation formula

R-SH:	(EQ1-B)*T*M*F1/(V*F2)	Mol (M):	32.06000
Blank value (B):	0.0000 ml	Titre (T):	0.00998 (a)
Factor 1 (F1):	1000.0000	Pattern (V):	20.000 ml (m)
Factor 2 (F2):	0.8200	Statistics:	Off

Application

Result page 2:

Method data overall view

Method name:	R-SH without H2S	Created at:	03/25/15 19:02:24
Method type:	Automatic titration	Last modification:	03/25/15 19:02:24
Measured value:	mV	Damping settings:	strong
Titration mode:	Linear	Documentation:	GLP
Linear steps:	0.050 ml		
Measuring speed / drift:	User-defined:	minimum holding time:	05 s
		maximum holding time:	15 s
		Measuring time:	04 s
		Drift:	05 mV/min
Initial waiting time:	0 s		
Titration direction:	Decrease		
Pretitration:	Off		
End value:	Off		
EQ:	On (1)		
Slope value:	User-defined	Value:	500

Dosing parameter

Dosing speed:	100.00 % (20.00 ml/min)	Filling speed:	30 s
Maximum dosing volume:	10.00 ml		

Unit values

Unit size:	10ml
Unit ID:	10035433
Reagent:	AgNO3 in IPA
Batch ID:	no entry
Concentration [mol/l]:	0.00998
Determined at:	03/25/15 18:22:34
Expire date:	--
Opened/compounded:	--
Test according ISO 8655:	05/04/12
Last modification:	03/25/15 18:22:40

Application

Notes

If you have any questions on the application, you can feel free to contact us..

SI Analytics GmbH
Hattenbergstr. 10
55122 Mainz
Germany

Phone: +49 (0) 6131 / 66 – 5062
+49 (0) 6131 / 66 – 5118
Fax: +49 (0) 6131 / 66 – 5001
E-Mail: titration@si-analytics.com
Homepage: www.si-analytics.com