xylem

Application Note

Mercaptan Sulfur in Gasoline and Kerosene Aviation Turbine and Distillate Fuels

USING ASTM D 3227

Introduction

For the determination of mercaptan sulfur in a range from 3 mg/kg – 100 mg/kg.

Apparatus

- TitroLine® 7000 or higher
- Magnetic stirrer (TM 235)
- 10 mL Exchange unit WA 10, with amber glass bottle for the titrant

Electrode and Electrolyte

- **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

- **Solvent:** Sodium acetate trihydrate solution in IPA
- **Standardization:** KI or NaCl solution 0.1 mo/L
- **Titrant:** $AgNO_3$ 0.01 mol/L in isopropanol (IPA)

YSI

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Procedure

Preparation and standardization of the KI and alcoholic $AgNO_3$ solutions

Dissolve 17 g (weigh to 0.01g) of **KI** in100 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. HNO_3 to 100 ml of water in a 250 ml tall form beaker. Remove oxides of nitrogen by boiling for 5 minutes. 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.

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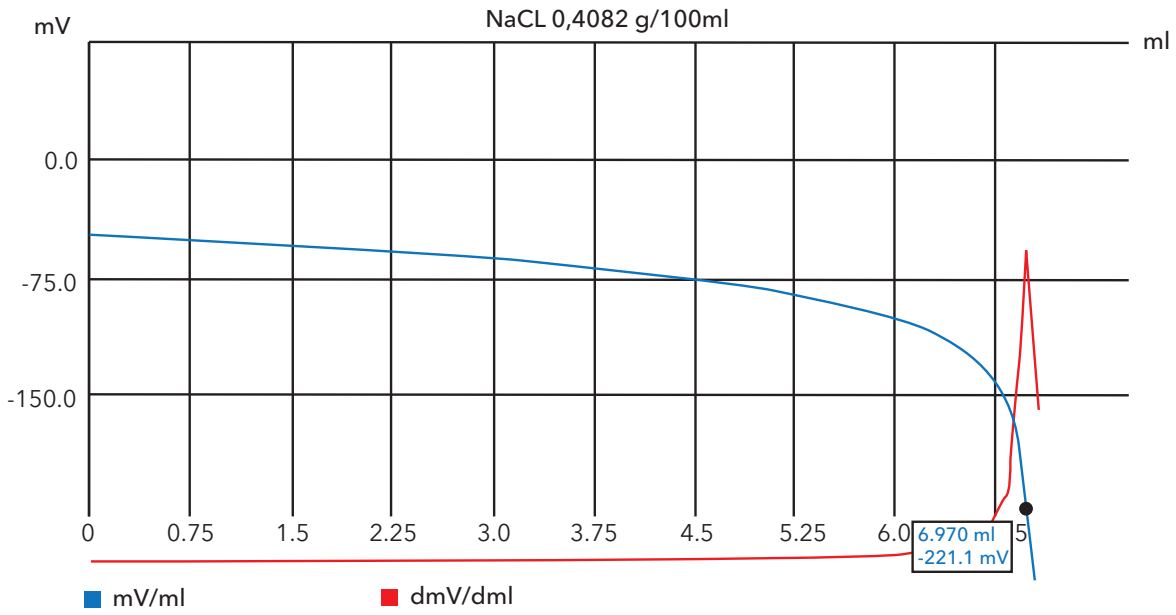
Standardization (Page 1)

GLP documentation

Titration graph



Method data

Method name:	AgN03 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		

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Method Data

Method data overall view

Method name:	AgN03 0.01 M in IPA	Created at:	03/25/15 17:24:34
Method type:	Automatic titration	Last modification:	03/25/15 17:34:34
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 ml/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:	10 ml
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

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Preparation of the Solvent

Dissolve 2.7 g of sodium acetate trihydrate in 20 ml oxygen-free water and pour into 975 ml of 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 $CdSO_4$ ($3CdSO_4 * 8 H_2O$) in water. Add 10 ml of H_2SO_4 (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 (H_2S) by shaking 5 ml of the sample with 5 ml of the acid $CdSO_4$ solution. If no precipitate appears, proceed with the analysis of the sample described below. If a yellow precipitate appears, remove the H_2S in the following manner: Place approximately 2-3 times the amount of sample needed for analysis (ie 200 ml), into a separatory funnel containing a volume of $CDSO_4$ equal to half of the sample volume (ie 100 ml) and shake vigorously. Remove the aqueous phase and wash the sample with three 25-30 ml portions of water, removing the aqueous layer after each wash. Repeat the extraction with $CdSO_4$ until all of the H_2S 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 containing 70 ml of the solvent mixture. Immediately immerse the electrodes and burette tip into the sample. Titrate with the 0.01 m $AgNO_3$ with the attached titration parameters. After the titration, the electrodes should be rinsed with alcohol and then with water.

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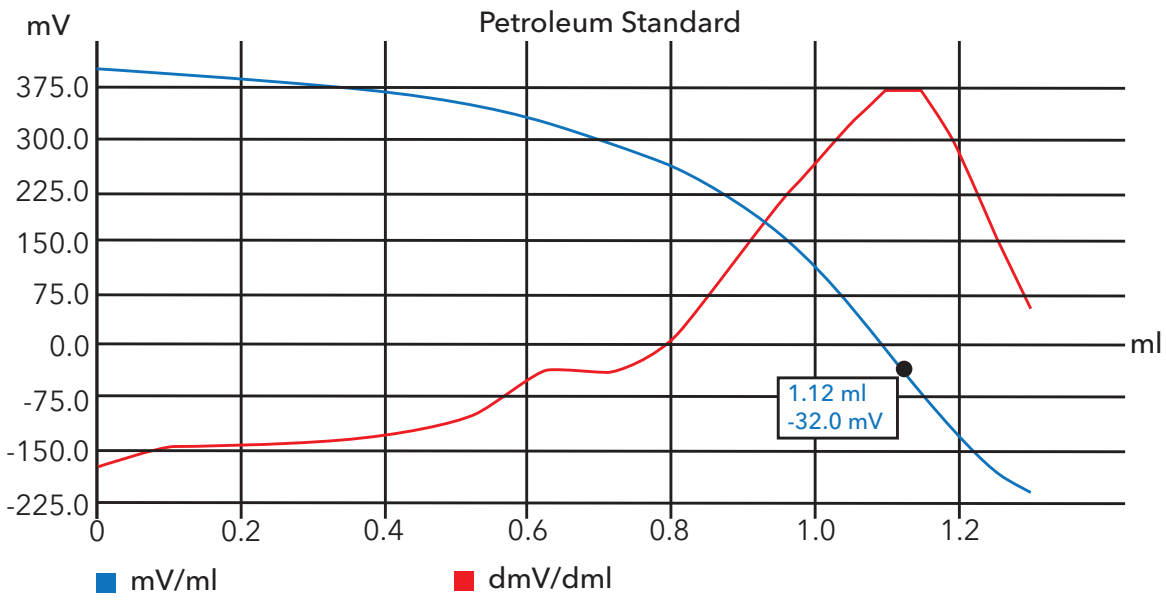
Result (Page 1)

GLP documentation

Titration graph



Method data

Method name:	R-SH without H_2S	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	R-SH:	21.9 ppm

Calculation formula

R-SH:	(EQ1-B)*T*M*F1/(V*F2)	Mol (M):	32.06000
Blank value (V):	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

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Method Data

Method data overall view

Method name:	R-SH without H_2S	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:	10 ml
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

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Literature

ASTM D3327

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