

## Thermo. Titr. Application Note No. H-027

<b>Title:</b>	<b>Determination of organic soda in Bayer aluminate liquors</b>
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<b>Scope:</b>	Determination of total basicity of extractable organic compounds of acidic character in Bayer Process refinery liquors.
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<b>Principle:</b>	<p>An aliquot of Bayer liquor is diluted and then neutralized and acidified with HCl. The acidified solution is extracted with cyclohexanol. This extract is then washed with water and made to volume in a volumetric flask, prior to being dried over anhydrous Na<sub>2</sub>SO<sub>4</sub>. An aliquot of this solution is then made to volume with propan-2-ol in a volumetric flask, and an aliquot of this solution is then titrated with 0.1 mol/L KOH in propan-2-ol to a catalyzed thermometric endpoint, with paraformaldehyde as the catalytic indicator. The result is expressed in terms of the soda equivalent (expressed as Na<sub>2</sub>CO<sub>3</sub> or Na<sub>2</sub>O) of the extractable organic compounds.</p>
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References:

1. M. J. D. Carneiro, M. A. Feres Júnior, and O. E. S. Godinho. Determination of the acidity of oils using paraformaldehyde as a thermometric end-point indicator. *J. Braz. Chem. Soc.* **13** (5) 692-694 (2002)
2. H-028. Standardization of 0.1 mol/L KOH in propan-2-ol

<b>Reagents:</b>	Standard 0.1 mol/L KOH in propan-2-ol Paraformaldehyde Cyclohexanol Concentrated HCl ~33% w/v Anhydrous Na <sub>2</sub> SO <sub>4</sub>
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<b>Method:</b>	Basic Experimental Parameters:	
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor	75
	Stirring speed (802 stirrer)	5
	Delay before start (secs.)	3
	<p>Procedure: 25 mL of spent Bayer liquor is pipetted into a 400mL beaker, and diluted with 125mL D.I. water. Allow 10 minutes for pipette to properly drain, due to the viscous nature of the liquor. Place on a stirrer, and while stirring, carefully neutralize and then acidify with concentrated HCl until the solution is clear. Cool the solution, and transfer to a 250mL separating funnel.</p> <p>Extract this solution with 5 x 25mL aliquots of cyclohexanol, allowing adequate time after each extraction for a full separation of the two phases. Return the collected cyclohexanol extract to a clean separating funnel, and wash with 4 x 100mL amounts of D.I. water. Quantitatively transfer the washed cyclohexanol extract to a 200mL volumetric flask, and make to volume with cyclohexanol. Dry the solution overnight in a sealed container with anhydrous Na<sub>2</sub>SO<sub>4</sub> using a magnetic stirrer to ensure efficient drying.</p> <p>Dilute 25mL of the dried cyclohexanol extract with propan-2-ol to 200mL in a volumetric flask. Allow 10 minutes for the pipette to drain properly due to the viscous nature of the fluid.</p> <p>Pipette a 30mL aliquot into a titration vessel, allowing to drain for a timed 3 minutes due to the viscous nature of the fluid. Add ~0.5g of paraformaldehyde, and titrate to a thermometric endpoint with 0.1 mol/L KOH in propan-2-ol.</p>	

<b>Results:</b>	Replicate analysis (n=6) of alumina refinery spent liquor	
	"Organic Soda" as g/L Na <sub>2</sub> CO <sub>3</sub>	9.92 ± 0.01

**Calculations:**

Legend:

Titre, 0.1 mol/L KOH = T mL

Blank, 0.1 mol/L KOH = B mL

Molarity KOH = M

Original volume of spent liquor = S mL

 Volume to which cyclohexanol extract is made up  
 =  $V_1$  mL

Aliquot of cyclohexanol extract = E mL

 Volume to which aliquot of cyclohexanol extract is made  
 up to with propan-2-ol =  $V_2$  mL

Aliquot of diluted extract taken for titration = A mL

 Formula weight of  $\text{Na}_2\text{CO}_3$  = 105.98844

 "Organic Soda" as g/L  $\text{Na}_2\text{CO}_3$  =

$$\frac{(T-B) \cdot M \cdot F \cdot V_1 \cdot V_2}{(S \cdot E \cdot A \cdot 2)}$$

**Thermometric Titration Plot:**
