

Thermo. Titr. Application Note No. H-024

Title:	Determination of Tar Acids (Phenolics) in Tar Products
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Scope:	Determination of tar acids in coal tar products. This procedure may also be applied to the determination of a range of weakly acidic organic compounds such as carboxylic acids, hydroxy acids, phenols, phenolic acids, keto-enols, imides and aromatic nitro compounds ¹ .
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Principle:	A sample is dissolved in acetone, and titrated with standard KOH in anhydrous methanol or isopropanol. At the endpoint, excess hydroxyl ions react catalytically and strongly exothermically with acetone to form diacetone alcohol. The procedure is essentially an automated version of the S.T.P.T.C. method ² .
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Reagents:	Standard 0.5mol/L KOH in anhydrous methanol or isopropanol. Anhydrous acetone (<0.2% H ₂ O), A. R.
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Method:	<p>Basic Experimental Parameters:</p> <table> <tr> <td>Data rate (per second)</td> <td>10</td> </tr> <tr> <td>Titrant delivery rate (mL/min.)</td> <td>0.5</td> </tr> <tr> <td>No. of exothermic endpoints</td> <td>1</td> </tr> <tr> <td>Data smoothing factor</td> <td>50</td> </tr> </table> <p>Procedure: Weigh accurately approximately 2-5g coal tar product into a clean, dry titration vessel. Solid coal tar products should be melted and mixed, before dispensing an appropriate amount into the titration beaker and allowing to cool. Add a spinning, and 25mL anhydrous acetone. Cover with a watch glass, and mix slowly on a magnetic stirrer until thoroughly dissolved. Titrate with 0.5mol KOH in methanol to an exothermic endpoint.</p>	Data rate (per second)	10	Titrant delivery rate (mL/min.)	0.5	No. of exothermic endpoints	1	Data smoothing factor	50
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Results:	Percentage phenol in coal tar products
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<p>1. Coal tar solvent. Mean = 2.53±0.004% (n=4)</p> <p>2. Creosote. Mean = 0.70±0.003</p>
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Calculation:

$$\% w / w \text{ Tar Acids} = \frac{((\text{titre, mL} - \text{blank, mL}) \times M \text{ KOH} \times FW \text{ phenol} \times 100)}{(\text{sample mass, g} \times 1000)}$$

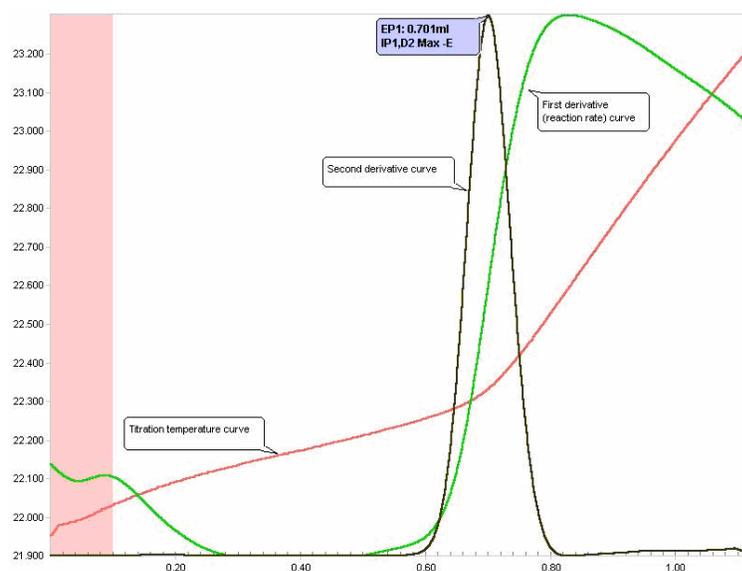
$$\% w / w \text{ Tar Acids} = \frac{((1.311 - 0.024) \times 0.5 \times 94.113 \times 100)}{(2.3926 \times 1000)}$$

$$= 2.53\%$$

References:

1. Vaughan, G. A. *Thermometric and Enthalpimetric Titrimetry*. Van Nostrand Reinhold Co. Ltd (1973)
2. S.T.P.T.C. "Standard methods for testing tars and products", 6th Ed., S.T.P.T.C. (1967)

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Green = first derivative curve

Black = second derivative curve