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Thermo. Titr. Application Note No. H-013

Title:	Determination of Moisture Content of Ultra-fine Solids

Scope:	Determination of water in moist particulate solids such as cobalt oxyhdroxide.

Principle:	A direct titration of the moisture content of ultra-fine or of high porosity solids may yield inconsistent and low results due to the inability of the titrant to rapidly penetrate moisture-containing interstices. In such cases, a back-titration may be recommended. An excess of 2,2-dimethoxypropane (DMP) is added to a suspension of the moisture-containing solid in a suitable polar solvent such as propan-2-ol to which a small amount of a suitable acid catalyst has been added. After through mixing of the suspension with the excess DMP, this excess is back-titrated with a standard solution of water in propan-2-ol. Water reacts endothermically with DMP to form acetone and methanol.
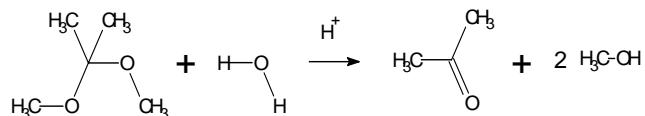
Reagents:	2mol/L 2,2-dimethoxypropane (DMP) in dry (HPLC grade) cyclohexane [DMP data: FW = 104.15, d = 0.847, purity = 98%]. Dilute 250mL DMP to 1000mL with cyclohexane in a volumetric flask
	Methane sulfonic acid
	Propan-2-ol (HPLC grade)
	2 mol/L H_2O in propan-2-ol

Method:	Basic Experimental Parameters:	
	Data rate (per second)	10
	Titrant delivery rate (mL/min.)	2
	No. of exothermic endpoints	1
	Data smoothing factor	75

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Method (Continued):	Weigh rapidly and accurately approximately 0.5 - 1g moist sample (depending on moisture content) into a dry titration vessel. Deliver by bulb pipette 25.00mL dry propan-2-ol, plus 250µL methane sulfonic acid into the vessel.
	Start the titration. The pre-dose part of the back-titration commences. 5.00 mL DMP reagent is dispensed into the titration vessel. A 30-second delay is programmed prior to the back-titration with standard H_2O in propan-2-ol so that all moisture is extracted from the sample and reacted with the DMP. The titration proceeds to an endothermic endpoint.
	The DMP reagent is standardized against the standard water solution by dispensing volumes of DMP ranging from 1-5 mL, and titrating them with 25mL propan-2-ol and 250µL methane sulfonic acid.

Results:	Analysis of very fine moist cobalt oxyhydroxide			
	Sample Mass, g	**DMP Pre-Dose, mL**	**Std. H_2O Back Titre, mL**	**% H_2O**
	0.5102	5.000	1.921	18.9
	0.5067	5.000	1.977	18.7
	0.5030	5.000	2.002	18.6
	0.5180	5.000	1.865	19.0
	0.4906	5.000	2.035	18.9
	0.4934	5.000	2.032	18.8
	0.5306	5.000	1.829	18.8
			Average	**18.8**
			Standard Deviation	**0.1**

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Calculation			
	Data Item	**Symbol**	**Test Data**
	DMP pre-dose vol, mL	P_D	5.000
	DMP molarity	M_D	1.9610
	Std. H_2O soln. molarity	M_W	2.0001
	Std. H_2O back-titre, mL	T_W	2.032
	Titration blank, mL	T_O	0.3001
	Sample mass, g	S	0.4934
	Formula weight H_2O	F_W	18.015

$$\% H_2O = \frac{(((P_D \times M_D) - (M_W(T_W + T_O))) \times F_W \times 100)}{(S \times 1000)}$$

$$\% H_2O = \frac{(((5.000 \times 1.9610) - (2.0001 \times (2.032 + 0.3001))) \times 18.015 \times 100)}{(0.4934 \times 1000)}$$

$= 18.8\%$

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Thermometric Titration Plot:
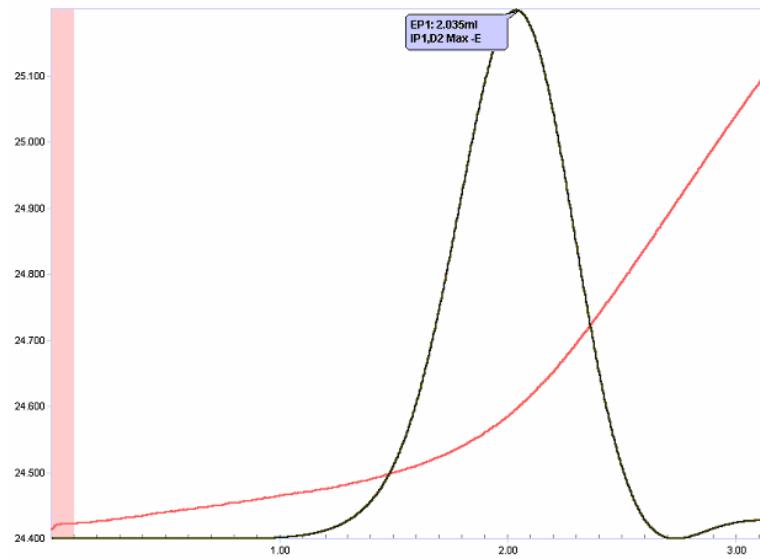
Legend:

Red = solution temperature curve

Black = second derivative curve

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