

Thermo. Titr. Application Note No. H-083

Title: Determination of surface basicity in Zeolites and other materials with a high specific surface area

Scope: Determination of total basic surface active sites in natural and synthetic surface-active materials.

Principle: A weighed sample of surface-active material is suspended in dry toluene or cyclohexane, and reacted with a known volume of standard methane sulfonic acid in dry 2-propanol. The excess base is back-titrated with standard n-butylamine in cyclohexane or toluene.

Reagents: *0.1 mol/L n-butylamine solution:* Dispense 10mL 99.5% n-butylamine (e.g., Aldrich cat. no. 471305, FW=73.08914) into a dry 1000mL volumetric flask, and make to volume with dry cyclohexane. Protect the reagent with a freshly filled soda-lime guard tube.

0.1 mol/L methanesulfonic acid, CH₃SO₃H. Weigh accurately approximately 9.6g 99.5+% methane sulfonic acid (e.g., Aldrich cat. no. 471356, FW=95.98812) into a dry 1000mL volumetric flask, and make to volume with dry 2-propanol. Protect the reagent with a freshly filled molecular sieve 3A guard tube.

Method: *Basic Experimental Parameters:*

Titration delivery rate (mL/min.)	4
No. of endothermic endpoints	1
Data smoothing factor	88
Stirrer speed	15

Basic Procedure: Weigh approximately 0.5 - 2 g of sample (depending on the nature of the sample) into a clean, freshly oven-dried 100mL glass vessel provided with a stopper. Immediately pipette in 75mL toluene, and then accurately dispense (by pipette or Dosino) in 15mL of 0.1mol/L methane sulfonic acid. Add a dry magnetic spin bar, and stir on a magnetic stirrer for 30 minutes. Take off the stirrer, and allow any fine solids to settle. Pipette 30 mL of clear supernatant solution into a clean, dry titration vessel. Back-titrate with 0.1 mol/L n-butylamine solution to an exothermic endpoint.

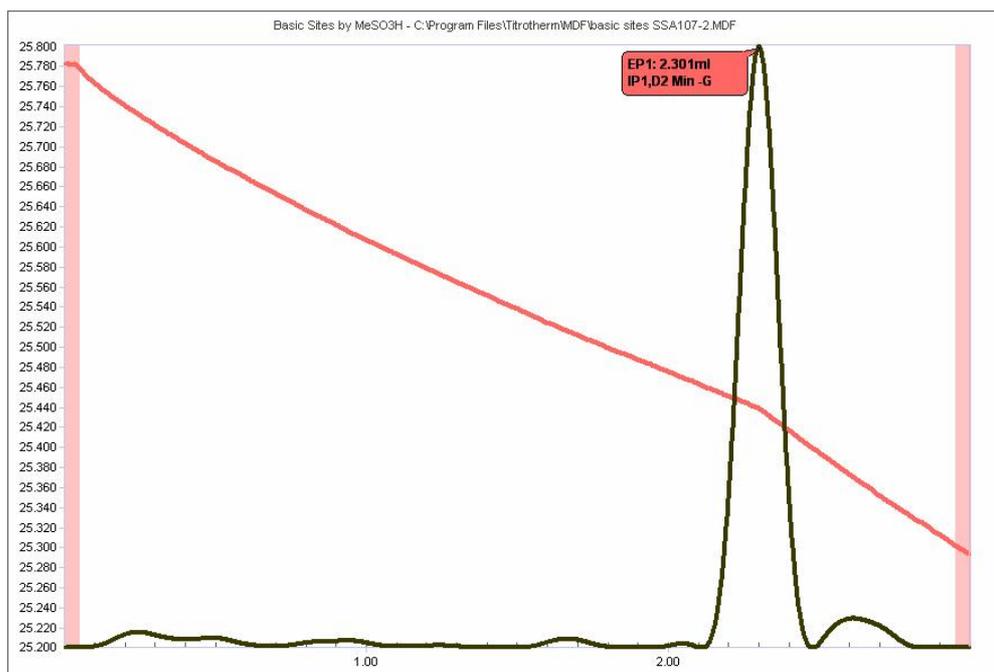
Blank determination: Accurately dispense 5mL of 0.1mol/L methane sulfonic acid solution into a clean, freshly dried titration vessel. Add 25mL of dry cyclohexane, and titrate to an exothermic endpoint. Perform this determination at least in triplicate, and use the mean mL value .

Results: All samples were dried at 200°C for 4 hours immediately prior to analysis.

Sample	Basic Sites, mmol/g
Zeolite (unknown provenance)	0.156±0.001 (n=5)
Silica gel (from drying sachet)	0.016±0.003 (n=5)
Smelter grade alumina, 35m ² /g	0.263, 0.265
Smelter grade alumina, 107m ² /g	0.555±0.002 (n=5)

Calculation: basic sites, mmol/g = $\frac{((\text{blank} - \text{titre}) \times \text{mol/L CH}_3(\text{CH}_2)_3\text{NH}_2)}{\text{sample mass, g}}$

Thermometric Titration Plot:



Legend:

Red = solution temperature curve

Black = second derivative curve