~~Designation:D4824–94 (Reapproved 1999)~~ Designation: D 4824 – 03 (Reapproved 2008)

Standard Test Method for Determination of Catalyst Acidity by Ammonia Chemisorption[1]

This standard is issued under the fixed designation D 4824; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

INTRODUCTION

This test method involves the measurement of total catalyst acidity by chemisorption of ammonia in a static volumetric system. Acidity is a very important parameter in determining catalyst activity and selectivity in many commercial reactions. Zeolite based catalysts used in the petroleum industry for catalytic cracking are a prime example. This test method describes a simple procedure employing inexpensive equipment that could readily be assembled in most laboratories.

1. Scope

1.1 This test method covers the determination of acidity of catalysts and catalyst carriers by ammonia chemisorption. A volumetric measuring system is used to obtain the amount of chemisorbed ammonia.

~~1.2~~

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:* [2]

D 3766 Terminology Relating to Catalysts and Catalysis

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method

3. Terminology

3.1 *Definitions—~~Consult~~—*See Terminology D 3766.

3.2 *Symbols:*

V_k = calibrated expansion volume, cm^3.
T_1 = temperature of V_k at initial ammonia pressure, K.
T_2 = temperature of V_k at final ammonia pressure, K.
P_1 = initial ammonia pressure, torr.
P_2 = final ammonia pressure, torr.
W_s = ~~weight~~mass of sample, g.
W_1 = tare ~~weight~~ of sample tube, g.
W_2 = sample mass plus tare ~~weight~~ of tube, g.
P_{1T} = initial ammonia pressure corrected to standard temperature, torr.
P_{2T} = final ammonia pressure corrected to standard temperature, torr.

~~[1] This test method is under the jurisdiction of ASTM Committee D-32 on Catalysts and is the direct responsibility of Subcommittee D32.01 on Physical-Chemical Properties.~~

~~Current edition approved Jan. 15, 1994. Published March 1994. Originally published as D4824–88. Last previous edition D4824–93.~~

[1] This test method is under the jurisdiction of ASTM Committee D32 on Catalysts and is the direct responsibility of Subcommittee D32.01 on Physical-Chemical Properties.

Current edition approved April 1, 2008. Published April 2008. Originally approved in 1988. Last previous edition approved in 2003 as D 4824–03.

[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards ~~, Vol 05.03.~~* volume information, refer to the standard's Document Summary page on the ASTM website.

4. Summary of Test Method

4.1 A sample is degassed by heating in a vacuum to remove adsorbed vapors from the surface. The sample is then exposed to an excess of gaseous ammonia and the excess ammonia is removed by freezing it into a trap cooled with liquid nitrogen. The chemisorbed ammonia is calculated as the difference between the volume of ammonia before exposure and the volume recovered in the liquid nitrogen trap.

5. Significance and Use

5.1 This test method can be used to determine the acidity of catalysts and catalyst carriers by ammonia chemisorption for materials specifications, manufacturing control, and research and development in the evaluation of catalysts.

6. Apparatus

6.1 A schematic diagram of one type of apparatus is shown in Fig. 1. It may be constructed of glass or metal and may operate manually or automatically. It has the following features:

6.1.1 *Vacuum System*, capable of attaining and maintaining pressures below 10^{-4} torr (0.01 Pa) in the absence of catalyst.

6.1.2 *Expansion Vessel*, having a volume between 300 to 500 cm^3(V_k) known to the nearest 0.01 cm^3(Note 1). This volume is defined as the volume between stopcocks V_7 and V_8 and includes the pressure gage and ammonia bulb.

NOTE 1—An expansion vessel of smaller volume is recommended for materials of low surface area or smaller sample size.

6.1.3 *Constant Volume Gage*~~capable~~, capable of measuring 0 to 250 torr to the nearest 10^{-1} torr (10 Pa).

6.1.4 *Vacuum Gage*, capable of measuring 0 to 1000 torr to the nearest 10^{-3} torr (0.1 Pa).

6.1.5 ~~*Valve,* (~~*Valve*, (V_8) from ammonia supply to the expansion volume.

6.1.6 *Sample Tubes*, with volume between 5 and 25 cm^3. The sample tube(s) may be connected to the apparatus with standard taper joints, glass-to-glass seals, or compression fittings.

6.1.7 *Dewar Flask(s)*, for immersion of ammonia (freeze-back bulb) in liquid nitrogen.

6.1.8 *Thermometer or Thermocouple* , for measuring the temperature of the expansion volume, V_k. Alternatively, the expansion volume may be thermostatted a few degrees above ambient to obviate the necessity of recording this temperature.

6.1.9 *Heating Mantle or Small Furnace* ~~for~~, for each sample tube.

6.1.10 *Balance*~~with 0.1-mg sensitivity.~~, with 0.1-mg sensitivity.

6.1.11 *Thermometer or Thermocouple* , for measuring the temperature of the sample tube.

6.1.12 *Liquid Nitrogen Cold Trap*~~for vacuum system.~~, for vacuum system.

6.1.13 *Drying Tube*, for drying ammonia gas (for example, a 3A molecular sieve or BaO).

7. Reagents

7.1 *Ammonia Gas*, at least 99.9 % pure.

7.2 *Liquid Nitrogen.*

7.3 *Nitrogen Gas*, at least 99.9 % pure, passed through drying tube.

8. Procedure

8.1 Weigh sample tubes to 0.1 mg and record as W_1. Place approximately 2 g of sample into the sample tube.

8.2 Attach the sample tube to the apparatus.

8.3 Open the sample valves (V_5 or V_6, or both).

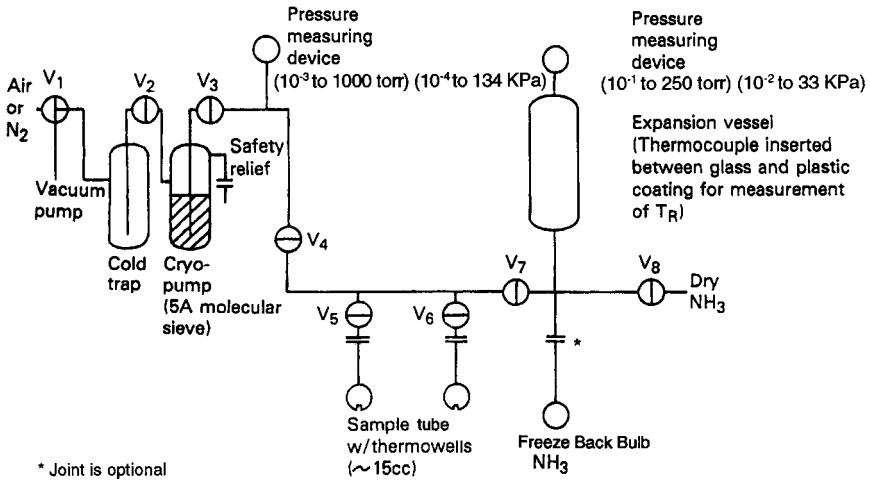

FIG. 1 Apparatus