



Designation: D 4824 – 94 (Reapproved 1999)

Standard Test Method for Determination of Catalyst Acidity by Ammonia Chemisorption¹

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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 *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:*

D 3766 Terminology Relating to Catalysts and Catalysis²

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

3. Terminology

3.1 *Definitions*—Consult 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 of sample, g.

W_1 = tare weight of sample tube, g.

W_2 = sample 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.

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 cm³ (V_k) known to the nearest 0.01 cm³ (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.

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.

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² *Annual Book of ASTM Standards*, Vol 05.03.

³ *Annual Book of ASTM Standards*, Vol 14.02.