



Designation: D 3037 – 93

Standard Test Methods for Carbon Black—Surface Area By Nitrogen Adsorption¹

This standard is issued under the fixed designation D 3037; 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.

This standard has been approved for use by agencies of the Department of Defense.

1. Scope

1.1 These test methods cover the determination of the surface area of carbon blacks. The test methods appear in the following order:

	Sections
Test Method A—Surface Area by Ni-Count-1 Apparatus	9-14
Test Method B—Surface Area by Model 2200 Automatic Surface Area Analyzer	15-20
Test Method C—Surface Area by Continuous Flow Chromatography	21-27
Test Method D—Surface Area by Monosorb Surface Area Analyzer	28-35

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 Goal values for the standard reference blacks were determined by using Test Methods D 4820.

1.4 *This standard does not purport to address all of the safety problems, 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.* For specific precautionary statement, see Note 3.

2. Referenced Documents

2.1 ASTM Standards:

D 1799 Practice for Carbon Black—Sampling Packaged Shipments²

D 1900 Practice for Carbon Black—Sampling Bulk Shipments²

D 3324 Practice for Carbon Black—Improving Test Reproducibility Using ASTM Reference Blacks²

¹ These test methods are under the jurisdiction of ASTM Committee D-24 on Carbon Black and are the direct responsibility of Subcommittee D24.21 on Adsorptive Properties of Carbon Black.

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² Annual Book of ASTM Standards, Vol 09.01.

D 4483 Practice for Determining Precision for Test Method Standards in the Rubber and Carbon Black Industries²

D 4820 Test Methods for Carbon Black—Surface Area by Multipoint B.E.T. Nitrogen Adsorption²

3. Significance and Use

3.1 These test methods are used to measure the surface area of carbon black that is available to the nitrogen molecule.

4. Reagents

4.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.³ Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

4.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

5. ASTM D-24 Standard Reference Blacks

5.1 The apparatus shall be checked initially for correct calibration by testing the ASTM D-24 Standard Reference Blacks. These blacks shall be tested regularly, along with sample blacks, as described in Test Methods A through D. The standard value and range for each of the standard blacks, when tested by these test methods, is given in Practice D 3324.

5.1.1 In Test Methods A through D, all samples and standard blacks shall be dried for 1 h at 125°C before weighing. Cool and keep blacks in a well-maintained desiccator before taking

³ “Reagent Chemicals, American Chemical Society Specifications,” Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see “Reagent Chemicals and Standards,” by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the “United States Pharmacopeia.”

the weight. This precaution will minimize inaccuracies due to adsorbed water and other materials.

6. Sampling

6.1 Samples shall be taken in accordance with Practice D 1799 or D 1900.

7. Precision and Bias⁴

7.1 This precision and bias has been prepared in accordance with Practice D 4483. Refer to this practice for terminology and other statistical details.

7.2 The precision results in this precision and bias give an estimate of the precision as described below. The precision parameters should not be used for acceptance/rejection testing of any group of materials without documentation stating that they are applicable to those particular materials and the specific testing protocols that include this test method.

7.3 A Type 1 interlaboratory precision program was conducted. Both repeatability and reproducibility represent short term testing conditions. Six laboratories tested the six standard reference blacks (A4 to F4) twice on each of two different days. Therefore, $p = 6$, $q = 6$, and $n = 4$. A test result is the value obtained from a single determination. Acceptable difference values were not measured.

7.4 The results of the precision calculations are given in Table 1 with the materials arranged in ascending mean level order of the surface area by nitrogen adsorption.

7.5 The precision for the pooled values for surface area by nitrogen adsorption may be expressed as follows:

7.5.1 *Repeatability*—The repeatability, (r), of the nitrogen surface area adsorption has been established as 1.62 %. Two single test results (or determinations) that differ by more than 1.62 % must be considered suspect and dictates that some appropriate investigative action be taken.

7.5.2 *Reproducibility*—The reproducibility, (R), of the nitrogen surface area adsorption has been established as 4.59 %. Two single test results (or determinations) produced in separate laboratories that differ by more than 4.59 % must be considered as suspect and dictates that appropriate investigation or technical/commercial actions be taken.

7.6 *Bias*—In test method terminology, bias is the difference between an average test value and the reference (true) test property value. Reference values do not exist for these test methods since the value or level of the test property is exclusively defined by these test methods. Bias, therefore, cannot be determined.

8. Keywords

8.1 carbon black; continuous flow chromatography; surface area analyzer; surface area by nitrogen absorption

TEST METHOD A—SURFACE AREA BY NI-COUNT-1 APPARATUS

9. Apparatus

9.1 *Ni-Count-1 Specific Surface Area Apparatus*⁵ (Fig. 1, Fig. 2, and Fig. 3), or an equivalent one-point adsorption apparatus. (See Fig. 4 for installation diagram.)

9.2 *Heater and Voltage Control Device*, to maintain a temperature of $300 \pm 10^\circ\text{C}$ for degassing the samples. The heater (Fig. 1) is furnished with the Ni-Count-1 apparatus.

9.3 *Vacuum Pump*, capable of ultimate pressure of 1.3 Pa (1×10^{-2} mm Hg abs).

9.4 *Dewar Flask*, 265-cm³ capacity, approximately 145 mm high (supplied with the Ni-Count-1 apparatus).

9.5 *Nitrogen Vapor Pressure Thermometer*, a part of the Ni-Count-1 apparatus (Fig. 2).

9.6 *Sample Tubes* (Fig. 5).

⁴ Supporting data are available from ASTM Headquarters. Request RR: D24-1021.

⁵ The Ni-Count-1 is developed by Phillips Petroleum Co. and is available from Chandler Engineering Co., 7707 E. 38th St., Tulsa, OK.

TABLE 1 Precision Values—Type 1^A

NOTE 1—Symbols are defined as follows:

Sr = within laboratory standard deviation,
 r = repeatability, measurement units
 (r) = repeatability, %
 SR = between laboratory standard deviation,
 R = reproducibility, measurement units
 (R) = reproducibility, %

Material	Mean Level, 10 ³ m ² /kg (m ² /g)	Within Laboratories ^B			Between Laboratories ^B		
		Sr	r	(r)	SR	R	(R)
D-4 (N 762)	24.31	0.123	0.349	1.452	0.343	0.971	4.046
E-4 (N 660)	34.85	0.270	0.764	2.193	0.579	1.638	4.700
F-4 (N 683)	38.62	0.205	0.580	1.502	0.707	2.000	5.180
B-4 (N 330)	75.40	0.529	1.498	1.997	0.770	2.180	2.906
A-4 (N 326)	77.83	0.263	0.744	0.956	0.586	1.660	2.133
C-4 (N 121)	126.61	0.547	1.549	1.223	2.091	5.917	4.673
Pooled or Aver- aged Values	62.94	0.36	1.02	1.62	1.02	2.89	4.59

^A This is short-term precision (days) where: $p = 6$, $q = 6$, and $n = 4$.

^B Report repeatability and reproducibility in percent.

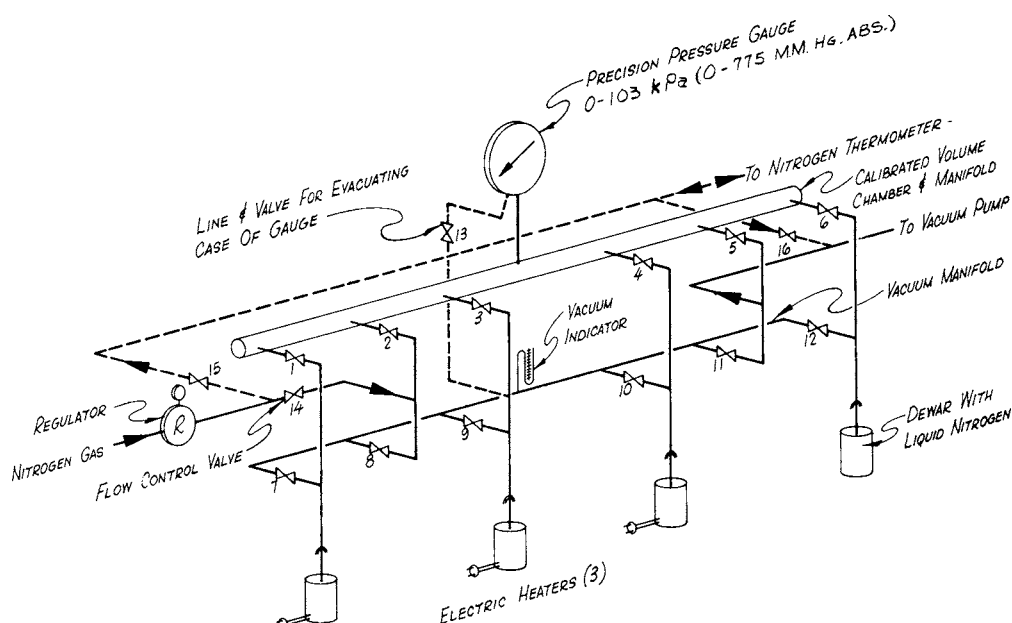


FIG. 1 Ni-Count-1 Apparatus with Provision for Four Samples

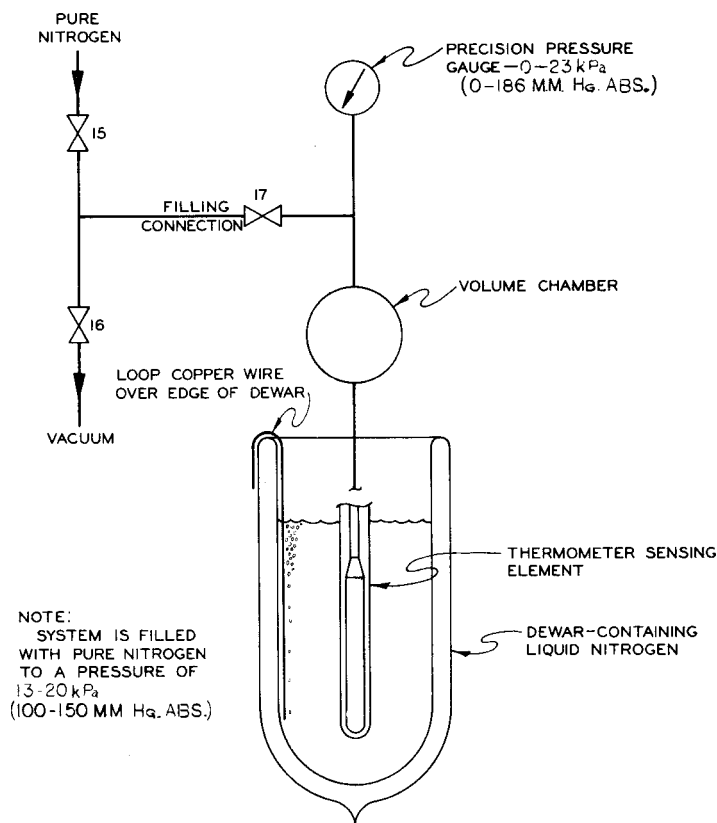


FIG. 2 Nitrogen Thermometer

9.7 *Stopcock Grease*,⁶ or polychlorotrifluoroethylene lubricant, supplied with Ni-Count-1 apparatus.

9.8 *Analytical Balance*, with 0.1-mg sensitivity.

9.9 *Fine Glass Wool*.

9.10 *Convection Oven*, capable of maintaining $125 \pm 5^\circ\text{C}$.

9.11 *Desiccator*.

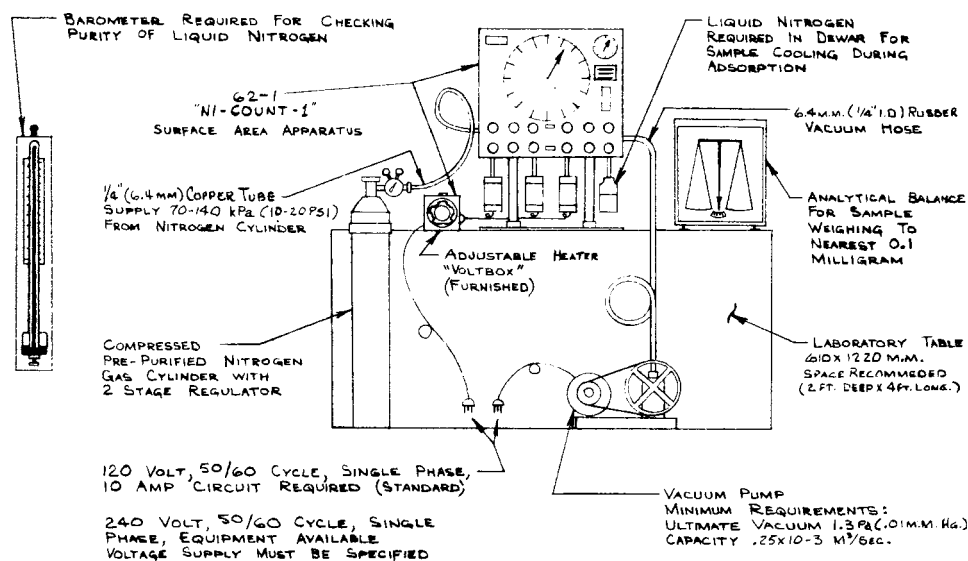
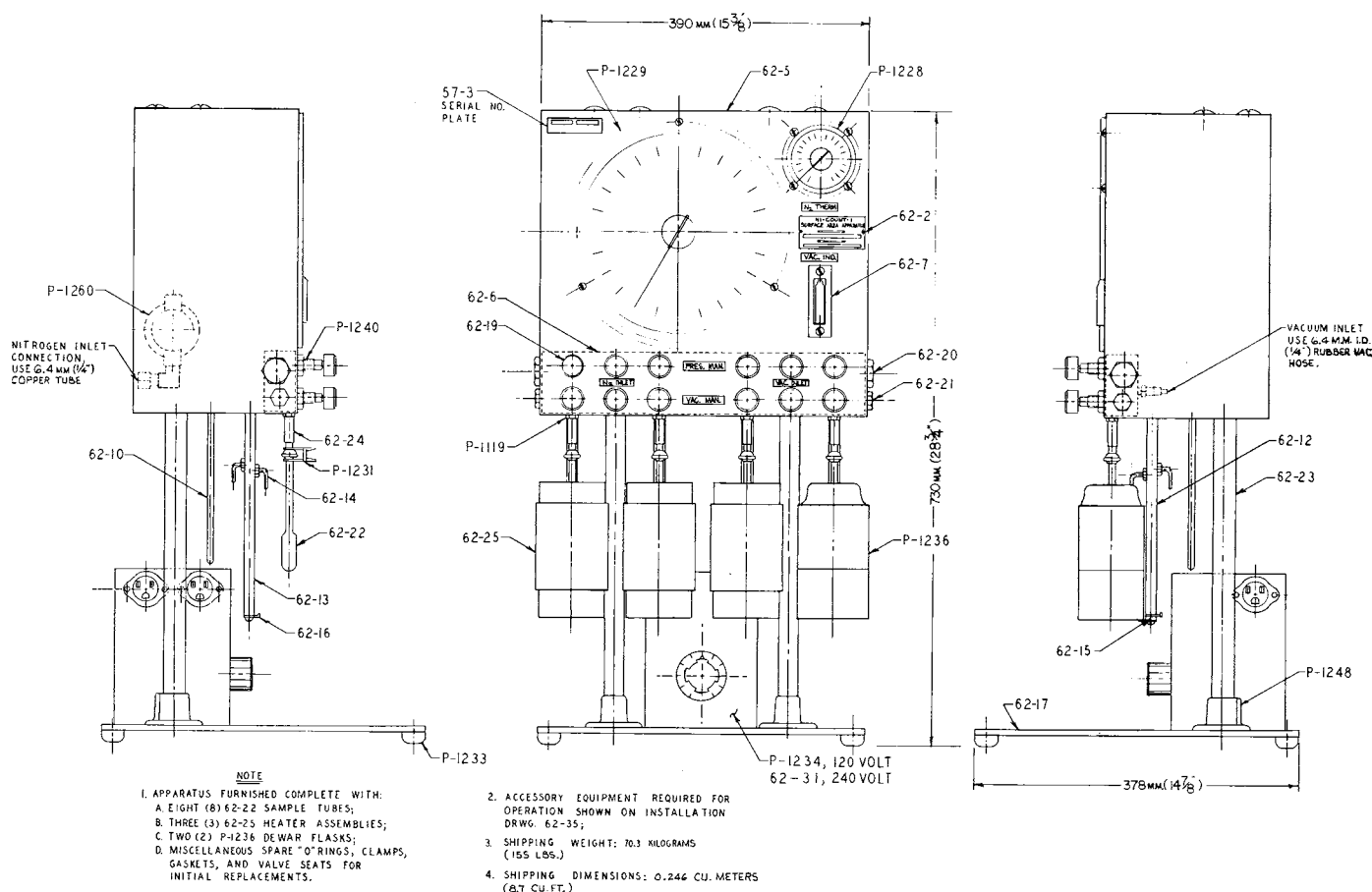
9.12 Supply of small flat-bottom vials for drying blacks.

10. Reagents

10.1 *Liquid Nitrogen*, approximately 300 g or 3 samples.

⁶ Spectro-Vac Stopcock Grease, Type 2, available from Robert R. Austin, Ph.D., P. O. Box 5374, Pasadena, CA 91107, has been found satisfactory.

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10.2 Nitrogen Gas, cylinder or other source of prepurified nitrogen gas.

11. Preparation and Calibration of Apparatus

11.1 The all metal Ni-Count-1 apparatus has an adjusted internal volume of 139.5 cm³. This internal volume includes all

lines to the sample valve, and the volume in the bellows of the pressure gage so positioned that the gage indicates 66.7 kPa (500 mm Hg abs pressure). The tables of surface area versus pressure furnished with the Ni-Count-1 will yield accurate specific surface areas, if the internal volume of the instrument has been accurately adjusted at the factory.