



Designation: D 3366 – 95

## Standard Test Method for Color of Maleic Anhydride and Phthalic Anhydride in the Molten State and After Heating (Platinum-Cobalt Scale)<sup>1</sup>

This standard is issued under the fixed designation D 3366; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This test method covers the determination of the visual measurement of the color of maleic and phthalic anhydride melt before and after prolonged heating under specified conditions of time and temperature. Color values are expressed in terms of platinum-cobalt standards. This test method covers the range 0 to 100 color standard numbers.

1.2 The following applies to all specified limits in this standard: for purposes of determining conformance with this standard, an observed value or a calculated value shall be rounded of “to the nearest unit” in the last right-hand digit used in expressing the specification limit, in accordance with the rounding-off method of Practice E 29.

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.* For specific hazard statements see Section 7.

### 2. Referenced Documents

#### 2.1 ASTM Standards:

D 1193 Specification for Reagent Water<sup>2</sup>

D 1209 Test Method for Color of Clear Liquids (Platinum-Cobalt Scale)<sup>3</sup>

D 3438 Practice for Sampling and Handling Naphthalene, Maleic Anhydride, and Phthalic Anhydride<sup>3</sup>

E 29 Practice for Using Significant Digits in Test Data to Determine Conformance With Specifications<sup>4</sup>

#### 2.2 Other Document:

*OSHA Regulations*, 29 CFR, paragraphs 1910.1000 and 1910.1200<sup>5</sup>

### 3. Summary of Test Method

3.1 A freshly melted specimen is filled to mark into a Nessler tube and compared with Platinum-Cobalt color standards.

3.2 After heating for 2 h at a prescribed temperature, the specimen is again compared to the color standards.

### 4. Significance and Use

4.1 The color of maleic anhydride and phthalic anhydride can be an indication of the purity of these materials. High colors normally indicate contamination.

### 5. Apparatus

5.1 *Color Comparison Tubes*—Matched sets of 50-mL Nessler tubes<sup>6</sup> having a total length of about 300 mm. The height of the 50-mL gradation mark shall be within 200 to 250 mm above the exterior bottom of the tube. In a given set the gradation marks shall not vary by more than 3 mm. The use of heat resistant glass is required.

5.2 *Color Comparator*, constructed to permit visual comparison of light transmitted through the 50-mL Nessler tubes in the direction of their longitudinal axes. (The comparator should be constructed so that white light is reflected off a white plate and directed with equal intensity through the tubes, and should be shielded so that no light enters the tubes from the side.)

NOTE 1—For convenience of operation, an electrically heated, insulated comparator tube may be used to prevent the solidification of maleic or phthalic anhydride.

<sup>1</sup> This test method is under the jurisdiction of ASTM Committee D16 on Aromatic Hydrocarbons and Related Chemicals and is the direct responsibility of Subcommittee D16.02 on Oxygenated Aromatics.

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<sup>2</sup> *Annual Book of ASTM Standards*, Vol 11.01.

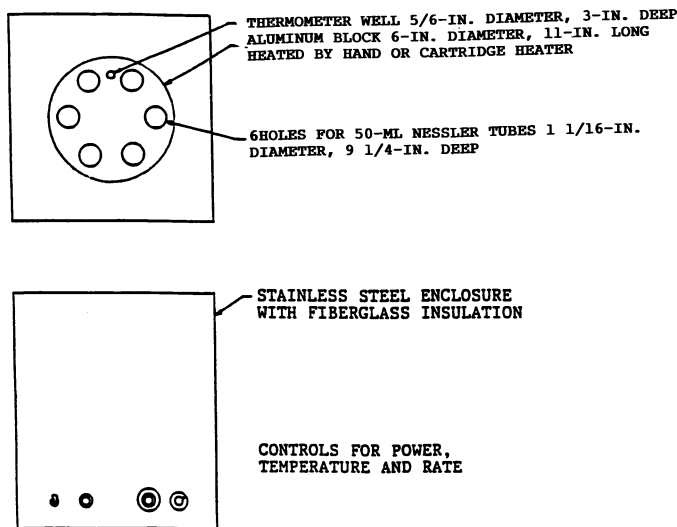
<sup>3</sup> *Annual Book of ASTM Standards*, Vol 06.04.

<sup>4</sup> *Annual Book of ASTM Standards*, Vol 14.02.

<sup>5</sup> Available from Superintendent of Documents, U.S. Government Printing Office, Washington, D.C. 20402.

<sup>6</sup> Kimble No. 45315A-50 or H5315B-50 Nessler tubes have been found satisfactory for this test; available from Fisher Scientific, VWR Scientific and other laboratory supply houses (Fisher Scientific, Corporate Headquarters, 111 Forbes Avenue, Pittsburgh, PA 15219; VWR Scientific, Marketing Department, P.O. Box 13645, Philadelphia, PA 19101).

5.3 *Electric Heating Block*<sup>7</sup>—An electrically heated aluminum block, such as shown in Fig. 1, having the following



**FIG. 1 Heat Stability Block**

operating characteristics:

Maleic anhydride temperature regulation, °C	140 ± 2
Heating time for sample from 60 to 140°C, minutes	25 ± 5
Phthalic anhydride temperature regulation, °C	250 ± 2
Heating time for sample from 150 to 250°C, minutes	25 ± 5
Temperature gradient, sample well to thermometer well, °C max	2

The operating characteristics of the heating block used are critical.

## 6. Reagents

6.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated it is intended that all the reagents should conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>8</sup> 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.

6.2 *Purity of Water*—Unless otherwise indicated reference to water shall be understood to mean reagent water conforming to Specification D 1193, Type II.

6.3 *Cobalt Chloride* (CoCl<sub>2</sub>·6H<sub>2</sub>O).

6.4 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

6.5 *Potassium Chloroplatinate*—(K<sub>2</sub>PtCl<sub>6</sub>).

## 7. Hazards

7.1 Consult current OSHA regulations, supplier's Material

Safety Data Sheets, and local regulations for materials used in this test method.

## 8. Sampling

8.1 Sample in accordance with Practice D 3438.

## 9. Standards

9.1 *Platinum-Cobalt Stock Solution*<sup>9</sup>—Dissolve 1.245 g of K<sub>2</sub>PtCl<sub>6</sub> and 1.000 g of CoCl<sub>2</sub>·6H<sub>2</sub>O in water. Add 100 mL of HCl and dilute to 1 L with water. This solution is defined as color standard No. 500.

9.2 *Platinum Cobalt Standards*<sup>10</sup>—From the stock solution, prepare color standards by diluting the required volumes as given in Table 1, to 50 mL with water in the Nessler tubes.

**TABLE 1 Platinum—Cobalt Color Standards<sup>A</sup>**

Color Standard No.	Stock Solution mL	Color Standard No.	Stock Solution mL
5	0.5	35	3.5
10	1.0	40	4.0
15	1.5	50	5.0
20	2.0	60	6.0
25	2.5	70	7.0
30	3.0	100	10.0

<sup>A</sup> Other color standards may be prepared by proportional dilution in steps of 20 as follows: No. 120, No. 140, No. 160, etc.

When not in use, protect these standards from contamination and evaporation by the use of suitable caps on the tubes.

## 10. Procedure

10.1 Melt an approximately 75-g sample (Note 2) and simultaneously preheat a Nessler tube in an oven, electric heating block, or other similar equipment held at a temperature of 60°C for maleic anhydride and 150°C for phthalic anhydride.

10.2 As soon as the sample is completely liquid, mix by stirring with a clean, dry, glass rod; then quickly fill the preheated Nessler tube to the 50-mL mark with the sample and place in the comparator; immediately compare with the standards and record as the color of the sample in the molten state.

10.3 Place the filled Nessler tube in the heating block which has been regulated at the appropriate temperature:

For maleic anhydride	140 ± 2°C
For phthalic anhydride	250 ± 2°C

Use a small ring of glass wool around the top of the hole to support in a vertical position and to seal off the dead air space between the tube and the heating block. Allow the tube to

<sup>7</sup> A commercial unit designed in accordance with specifications given in this method may be obtained from Petrolab Corporation, 874 Albany-Shaker Road, Latham, New York 12110, Cat. No. 3366. It is identified as "Heater, Color Tubes, PA, ASTM."

<sup>8</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopoeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

<sup>9</sup> The stock solution with color No. 500 may be purchased as such from chemical supply firms. Use of the purchased standards is satisfactory.

<sup>10</sup> The preparation of these platinum-cobalt standards was originally described by Hazen, A. *American Chemical Society Journal*, Vol. 14, 1892, p. 300. The description given in this test method and in ASTM Test Method D 1209, Test for Color of Clear Liquids (Platinum-Cobalt Scale), which appears in the *Annual Book of ASTM Standards*, Vol 06.03, is identical with that given in the *Standard Methods for the Examination of Water and Sewage*, American Public Health Association, Tenth Edition, 1955, p. 88. A description is also given by W. W. Scott in *Standard Methods of Chemical Analysis*, D. Van Nostrand Co., Inc., Sixth Edition, Vol. 2, Part B, p. 2424.



remain in the block for 2 h (which includes 90 min at the test temperature). Protect tubes adequately from contamination during the test period.

NOTE 2—Place a small quantity of glass wool in the bottom of each hole to ensure against breakage of Nessler tubes and thermometers.

10.4 Remove the tube, place in the comparator, and immediately compare with the standard. Record as the color after heating in the molten state.

10.5 In no case apply this test method if the molten sample contains any visible turbidity.

## 11. Report

11.1 Report the following information:

11.1.1 Report (1) color in the molten state, and (2) color in the molten state after heating.

11.1.2 Report as the color, the number of the standard that most nearly matches the sample. In the event that the color lies midway between two standards, report the darker of the two.

11.1.3 If, owing to differences in hue between the sample and the standard, a definite match cannot be attained, report the range over which an apparent match is obtained, and report the sample as “off hue.”

11.1.4 If, owing to large differences in hue between the sample and the standards, no estimate is possible, report the sample as “no match.”

## 12. Precision

12.1 *Intermediate Precision (formerly Repeatability)*—Duplicate results by the same operator should be considered suspect if they differ by more than the following amounts:

	Platinum Cobalt Color	Repeatability
Maleic Anhydride		
Molten color	10 to 20	5
Color after heating	20 to 40	10
Phthalic Anhydride		
Molten Color	15 to 40	5
Color after heating	40 to 70	10

12.2 *Reproducibility*—Duplicate results by each of two laboratories should be considered suspect if they differ by more than the following amounts:

	Platinum Cobalt Color	Reproducibility
Maleic Anhydride		
Molten color	10 to 20	0.3 P <sup>aA</sup>
Color after heating	20 to 40	10
Phthalic Anhydride		
Molten color	15 to 40	0.6 P <sup>aA</sup>
Color after heating	40 to 70	25

<sup>A</sup> P<sup>a</sup> = average platinum-cobalt color.

## 13. Keywords

13.1 color; maleic anhydride; phthalic anhydride

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