Designation: D 6580 – 00

Standard Test Method for The Determination of Metallic Zinc Content in Both Zinc Dust Pigment and in Cured Films of Zinc-Rich Coatings[1]

This standard is issued under the fixed designation D 6580; 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.

1. Scope

1.1 This test method covers the determination by differential scanning calorimetry of the metallic zinc content of both zinc-dust pigment, and of dried films of zinc-rich coatings. This test method is applicable to both inorganic and organic zinc-rich coatings

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:*

D 521 Test Methods for Chemical Analysis of Zinc-Dust (Metallic Zinc Powder)[2]

E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method[3]

3. Summary of Test Method

3.1 Samples of either zinc-dust pigment or of cured films of zinc-rich coatings are ground in a mortar and pestle, then carefully weighed into standard differential scanning calorimetry (DSC) sample pans. The pans are then crimped shut, and analyzed in a differential scanning calorimeter in a single dynamic heating step, ranging from 370 to 435°C at 10°C per min, under a nitrogen purge. The percent metallic zinc in the sample is determined by measuring the energy associated with the endothermic peak near 419°C caused by the melting of the metallic zinc, and comparing this value to the heat of fusion of pure zinc.

4. Significance and Use

4.1 This test method is useful for determining the amount of metallic zinc in zinc dust pigment, and also in dried films of both inorganic and organic zinc-rich coatings. Test Methods D 521 is an appropriate method for analyzing zinc dust, but has shortcomings when applied to samples of cured coatings.

5. Interferences

5.1 An increase or decrease in heating rate from those specified may slightly alter the results. However, the variation would be expected to be minimal, so long as the zinc reference standard and the samples are subjected to the same heating rate.

5.2 Daily calibration of the calorimeter with high purity zinc foil results in improved results. Reagent grade zinc granules or zinc powder are of insufficient purity to properly calibrate the instrument. Furthermore, the high purity zinc foil should only be used one time as a calibration standard. **Warning:** Using the same piece of foil more than once can result in inaccurate results, due to oxidation of the zinc at the high temperatures in the calorimeter, coupled with the alloying effects of zinc with the aluminum sample pans.

5.3 Important steps in achieving accurate and reproducible results are very gentle tapping of the pan in order to distribute the sample evenly over the bottom of the pan, and careful placement of the pan lid to avoid expulsion of the fine powder during crimping.

NOTE 1—Round-robin testing has shown no evidence that pyrolysis of the binder interferes with the measurement of the heat of fusion. Either pyrolysis does not occur, occurs during stabilization of the instrument prior to the scan, or is negligible due to the small amount of binder present in such coatings. If there is reason to suspect interference from the binder, the analyst may wish to test a blank sample of binder (with no zinc pigment) to ensure that there is no effect on heat flow measurements.

6. Apparatus

6.1 *Differential Scanning Calorimeter*, either of the heat flux or power compensation type, capable of heating rates up to at least 10 ± 1°C/min and of automatic recording of difference in heat input between the sample and a reference material, to the required sensitivity and precision.

6.2 *Sample Pans*—Aluminum or other metal pans of high thermal conductivity are appropriate. A method or instrument for crimping the pans shut is also necessary.

6.3 *Reference Material*—High purity zinc foil suitable for calibration of DSC instruments can be obtained from several instrument manufacturers.

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[2] *Annual Book of ASTM Standards*, Vol 06.03.

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

6.4 *Nitrogen*, or other inert gas supply, for blanketing the sample.

6.5 *Microbalance*, capable of measuring to at least 10 µg.

6.6 *Mortar and Pestle*—An agate mortar and pestle, of diameter approximately 29 mm (1½ in.) has been found satisfactory.

7. Calibration

7.1 The instrument should be calibrated for heat flow as described in the manufacturer's instruction manual. In addition to, or as part of the manufacturer's standard calibration procedure, the instrument should be calibrated using high-purity zinc-foil reference material. Approximately 3 to 6 mg of reference material shall be weighed into a standard aluminum pan using a microbalance capable of weighing to at least 10 µg. The pan shall then be crimped using an aluminum lid, and the reference sample analyzed in a single dynamic step ranging from 370 to 435°C at 10°C per min, under a nitrogen purge.

7.2 When analyzed as just described, the reference sample of zinc foil will give rise to an endothermic peak due to the fusion of zinc near 419°C. The area under the endothermic transition, in joules per gram, should be measured either electronically or manually, and should be very close to that for pure zinc (108 J/g).

8. Procedure

8.1 In the case of pigment samples, after thoroughly agitating the container in which the sample is contained, weigh 3 to 6 mg, to at least the nearest 10 µg, into a standard aluminum pan, and crimp the pan shut. In the case of dried coating films, it is recommended that a razor blade or razor-blade type knife be used to remove the coating from an area measuring at least 12.7 mm by 12.7 mm (½ by ½ in.) in size. This coating should then be ground briefly, and approximately 3 to 6 mg weighed into a standard aluminum pan, which should then be crimped shut.

8.2 The sample should be analyzed in a singe dynamic step ranging from 370 to 435°C at 10°C per min, under a nitrogen purge. The area under the endothermic transition corresponding to the melting (fusion) of zinc, in joules per gram, should then be determined in the same fashion as described for the zinc foil reference standard. Triplicate analyses should be performed, and the results should be averaged to obtain the average heat of fusion.

9. Calculation

9.1 Calculate the percent metallic zinc in the sample as follows:

$$\text{Percent zinc metal} = (X/108) \cdot 100, \quad (1)$$

where:

X = measured heat of fusion of sample, in joules/gram.

10. Report

10.1 Report the following information:

10.1.1 Complete identification and description of the material tested, including source and manufacturer's code, if known,

10.1.2 Description of instrument used for the test, and

10.1.3 Description of calibration procedure.

11. Precision and Bias

11.1 *Precision*—An interlaboratory evaluation of this test method with three laboratories and four materials resulted in the following statistics, from Practice E 691, as shown in Table 1, where *Sr* and *r* are the within laboratory standard deviation and repeatability and *SR* and *R* are the multi-laboratory standard deviation and reproducibility: Materials I and II represent two inorganic zinc rich coatings, while III and IV represent two organic zinc rich coatings.

11.2 Based on this, the following criteria should be used for judging the acceptability of results:

11.2.1 *Repeatability (Single Analyst)*—The standard deviation for a single determination has been estimated to be 2.8 % relative. The 95 % limit for the difference between two such runs is 7.8 % relative.

11.2.2 *Reproducibility (Multilaboratory)*—The coefficient of variation of results (each the average of three determinations), obtained by analysts in different laboratories has been estimated to be 5.0 % relative. The 95 % limit for the difference between two such averages is 13.9 % relative.

11.2.3 *Bias*—Bias cannot be determined, as there are no standard materials available.

12. Keywords

12.1 differential scanning calorimetry; metallic zinc; zinc dust pigment; zinc-rich primers

TABLE 1 Summary of Interlaboratory Precision Data

Materials	Average	*Sr*	*SR*	*r*	*R*
I	84.1000	1.9156	3.9497	5.3636	11.0591
II	71.8264	1.7122	4.8757	4.7943	13.6520
III	82.5222	2.0552	2.6453	5.7546	7.4068
IV	69.3944	2.7797	3.5989	7.7831	10.0768