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## Standard Test Method for Thermogravimetric Analysis of Hydraulic Cement<sup>1</sup>

This standard is issued under the fixed designation C1872; 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 reappraisal. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reappraisal.

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<sup>ε1</sup> NOTE—Added research report footnote to Section 14 editorially in May 2013.

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### 1. Scope

1.1 This test method provides a technique incorporating a thermogravimetric analyzer to determine the mass changes of hydraulic cement upon heating in an inert gas environment. The data can be used to determine the abundance of some mineralogical components in hydraulic cement powders.

1.2 *Units*—The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.3 This test method is applicable to hydraulic cement powders.

1.4 *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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.5 **Warning**—Fresh hydraulic cementitious mixtures are caustic and may cause burns to skin and tissue upon prolonged exposure. The use of gloves, protective clothing, and eye protection is recommended. Wash contact area with copious amounts of water after contact. Wash eyes for a minimum of 15 min. Avoid exposure of the body to clothing saturated with the liquid phase of the unhardened material. Remove contaminated clothing immediately after exposure.

1.6 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

2.1 *ASTM Standards:*

[C114 Test Methods for Chemical Analysis of Hydraulic Cement](#)

[C219 Terminology Relating to Hydraulic Cement](#)

[C670 Practice for Preparing Precision and Bias Statements for Test Methods for Construction Materials](#)

[E473 Terminology Relating to Thermal Analysis and Rheology](#)

[E691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method](#)

[E1131 Test Method for Compositional Analysis by Thermogravimetry](#)

[E1142 Terminology Relating to Thermophysical Properties](#)

[E1582 Test Method for Temperature Calibration of Thermogravimetric Analyzers](#)

[E1868 Test Methods for Loss-On-Drying by Thermogravimetry](#)

[E2040 Test Method for Mass Scale Calibration of Thermogravimetric Analyzers](#)

[E2402 Test Method for Mass Loss and Residue Measurement Validation of Thermogravimetric Analyzers](#)

### 3. Terminology

3.1 *Definitions:*

3.1.1 Technical terms used in this guide are defined in Terminologies [C219](#), [E473](#), [E1142](#).

### 4. Summary of Test Method

4.1 Thermogravimetric analysis of cement is performed by continuously monitoring mass changes of a hydraulic cement powder specimen, in an environment with a controlled atmosphere as the test temperature is increased at a constant rate. Mass loss

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over specific temperature ranges and in a specific atmosphere can be used to supplement the compositional analysis of the cement by providing estimates of the mass fraction of certain mineral constituents.

## 5. Significance and Use

5.1 This test method is intended for use in acquiring, analyzing, and reporting thermogravimetric data obtained from hydraulic cement powders.

5.2 This test method can be used to determine the calcium carbonate content of cements with interground limestone if the calcium carbonate content of the limestone is known.

5.3 This test method can be used to determine the calcium hydroxide content of hydraulic cement powder.

5.4 This test method can be used to determine the mass loss upon heating hydraulic cement powders within a specific temperature range.

5.5 This test method can be used for qualitative and quantitative characterization, under certain conditions, of various sulfate mineral components including calcium sulfate dihydrate, calcium sulfate hemihydrate, and syngenite.

5.6 Different kinds of thermogravimetric analyzers are available with different configurations and controllers. Therefore, the parameters described should be considered as guidelines. They may be altered to conform to the instrument manufacturer's instructions, provided the changes are noted in the report.

## 6. Interferences

6.1 This test method depends upon distinctive thermal stability ranges of the determined components as a principle of the test. Materials that have no well-defined thermally stable range, or that have thermal stability ranges that are the same as other components in the cement, may create interferences. Particular examples include the following:

6.1.1 Calcium silicate hydrate gel (C-S-H) releases physically and chemically bound water over a continuous and broad temperature range, typically 100 to 600°C, and may be observed even in nominally unhydrated cement powders that have been stored in humid conditions. In specimens of partially hydrated cement paste, the continuous release of water by C-S-H upon heating can interfere with the measurement of mass loss by other decomposing minerals within that temperature range, such as hydrated calcium sulfates and portlandite.

6.1.2 In unhydrated cement powders, the conversion of gypsum (calcium sulfate dihydrate) to calcium sulfate hemihydrate occurs over a temperature range, 100 to 200°C, that is close to that of the subsequent decomposition of hemihydrate to anhydrite (calcium sulfate). The two sources of mass loss are difficult to distinguish when using an open sample container, but can be separated better by covering the container with a lid having a narrow slit or hole for escape of vapors.

6.2 Heating rate can influence the temperature at which the decomposition of a component is detected, as well as the temperature at which that component reaches its maximum rate of decomposition, identified readily by a peak in the first derivative of the thermogravimetric data with respect to temperature (see Fig. 1). A difference in heating rate of 5°C/min can change the peak temperature by as much as 50°C. However, heating rates between 5 and 15°C/min have a negligible influence on the total mass loss accompanying a given decomposition event.

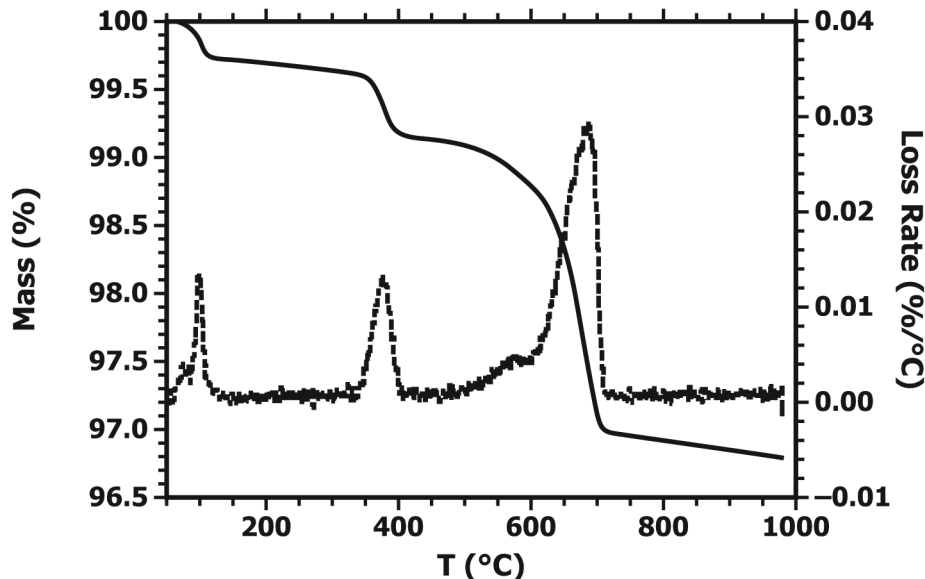


FIG. 1 Sample Thermogravimetric Curve for Unhydrated Portland Cement

## 7. Apparatus

7.1 Thermogravimetric analyzer with a microbalance, temperature controller, and data collection device, and gas flow control device all complying with the requirements of Test Method E1131, *Compositional Analysis by Thermogravimetry*.

7.1.1 Containers (pans, lids, and so forth), for holding the specimen must be dimensionally stable and chemically inert with respect to cementitious components within the temperature limits of this method.

7.1.1.1 Aluminum oxide is suitably inert with respect to hydraulic cement to be used for containers and lids at temperatures exceeding 600°C or when the lid need not be sealed to the pan.

7.1.1.2 Aluminum is a suitable material for measurements of the calcium sulfate hydrates and other components at temperatures less than 600°C, which require the pan and lid to be sealed with only a small slit or pinhole to allow escape of vapors.

7.1.1.3 Platinum pans can be used for analyses from ambient temperature up to 1200°C

7.1.1.4 Pans and lids can be obtained from the manufacturer of the thermogravimetric analyzer or from a vendor recommended by the manufacturer.

## 8. Reagents and Materials

8.1 An inert compressed gas is required for this method. High-purity nitrogen gas (99.99 % N<sub>2</sub>) is sufficient. High-purity argon or helium are also acceptable.

## 9. Sampling, Test Specimens, and Test Units

9.1 Cement powders are normally analyzed as soon as possible after being received because they will tend to absorb moisture and react with carbon dioxide over time. Measuring an as-received sample provides a baseline against which subsequent measurements can be compared to assess the degree of aging.

9.2 Typical sample masses will depend on the instrument being used and on the specific minerals of interest. Depending on the instrument being used, measurement of calcium hydroxide, syngenite, and calcium carbonate typically require about 50 mg of cement powder, while determination of calcium sulfate hydrate content in cement often require about 100 mg of powder. Refer to the manufacturer's instructions for guidance on the optimum mass of sample.

## 10. Calibration

10.1 Calibrate the mass signal from the apparatus according to Test Method E2040.

10.2 Calibrate the temperature signal from the apparatus according to Test Method E1582.

## 11. Procedure

11.1 Turn on the flow of the inert gas and establish its flow rate.

NOTE 1—The appropriate flow rate depends on the type of instrument being used. For example, instruments with small horizontal tube furnaces typically operate with a gas flow rate of 50 mL/min. Refer to the manufacturer's instructions for guidance on the appropriate flow rate.

11.2 Open the apparatus and load the reference and empty sample pans. Close the apparatus.

11.3 Zero the mass signal with the reference and sample pans in place.

11.4 Open the apparatus to expose the specimen holder.

11.5 Prepare the specimen as outlined in Section 9 and carefully place it in the specimen holder, if this was not already done as part of the sample conditions described in Section 9.

11.6 Close the apparatus.

11.7 Record the initial mass after any sample conditioning already described.

11.8 Initiate the user specified temperature program and collect the data of mass and mass change versus time and temperature. Table 1 provides typical measurement parameters. Consult the manufacturer's instructions to determine optimum parameters for a specific thermogravimetric analyzer.

11.8.1 The mass loss profile shall be expressed in absolute mass units of milligrams or grams. Expanded scale operation may be useful over selected temperature ranges.

**TABLE 1 Suggested Compositional Analysis Parameters**

Specimen	Sample Size, mg	Flow Rate mL/min <sup>A</sup>	Purge Time min	Temperature °C		Heating Rate °C/min
				Initial	Maximum	
Unhydrated Cement	50	50	10	25	1000	10
Calcium Sulfate Hydrates or Syngenite	100	50	10	25	500	10

<sup>A</sup> May differ depending on instrument design.

NOTE 2—Some instrument software may default to a normalized mass percentage scale based on the original sample mass. Absolute mass loss profiles can be recovered from a percentage scale if the original mass is known.

11.8.2 If only one or two components of the compositional analysis are desired, specific, more limited temperature ranges may be used. Similarly, several heating rates may be used during analysis in those regions of greater or lesser interest.

11.9 The analysis is complete when a state of constant mass is obtained at the maximum temperature of interest.

11.10 Calculate and report the sample composition (see Sections 12 and 13).

## 12. Calculation and Interpretation of Results

12.1 Table 2 shows the typical temperature ranges for decomposition reactions in an open pan for common components of unhydrated cement. The temperature ranges reflect averages of those reported in several studies, and are dependent on heating rate. In general, lower rates of heating will cause decomposition onset temperatures to be lower.

### 12.2 Quantitative Determination of Individual Components:

12.2.1 The mass percentage of any solid component listed in Table 2 can be determined from the mass lost by that component upon its thermal decomposition.

12.2.2 Each mineral component thermally decomposes over a characteristic temperature range. Approximate minimum temperature  $X$  and maximum temperature  $Y$  for each component are indicated in Table 2.

12.2.3 For some components such as calcium hydroxide, calcium sulfate hemihydrate, or syngenite, mass loss occurs over a relatively narrow temperature range. Other components that may be present due to significant prehydration effects, such as C-S-H gel, lose mass over a wide temperature range and therefore contribute to uncertainty in attributing a mass loss value to a particular mineral. Normally the steps are interpreted as a change in mass before and after the effect, so the corresponding “baseline” is horizontal. Sometimes relatively sharp decomposition steps occur during the decomposition process. This baseline drift, possibly due to mass loss by minerals such as C-S-H gel, may be accommodated by constructing tangent lines to the baseline at the onset and termination of the main mass loss signal, marking the point at which the tangent lines depart from the curve as the onset and terminating temperatures  $T_i$  and  $T_f$ , respectively, and assigning the mass difference  $\Delta m = m(T_i) - m(T_f)$  as the mass loss due to the component in question. Calculating and plotting the derivative of the mass loss with respect to temperature may be helpful both in identifying reaction onset/completion temperatures and in delineating the baseline drift.

12.2.4 The mass percentage of a particular component with a narrow decomposition temperature range may be calculated on an original mass basis, assuming that the component is stoichiometric and pure, by using the following equation:

$$P = \frac{M\Delta m}{Cm_o} \times 100 \quad (1)$$

where:

$P$  = mass percent of component in the original powder,

$M$  = molar mass of pure stoichiometric component in Table 2 (g/mol),

$\Delta m$  = vertical distance between upper and lower tangent lines, as shown in Fig. 2 (g),

$C$  = mass loss per mole of pure stoichiometric component in Table 2 due to thermal decomposition (g/mol), and

$m_o$  = original mass of sample (g).

12.2.5 Determinations of calcium sulfate dihydrate and calcium sulfate hemihydrate are complicated by the fact that the dihydrate form often first decomposes to the hemihydrate form before decomposing fully to anhydrite. Therefore, the hemihydrate mass loss signal generally has contributions both from original hemihydrate and from any hemihydrate formed by decomposition of dihydrate; an example is shown in Fig. 2.

Referring to Fig. 2, calculations of calcium sulfate dihydrate and hemihydrate can be made according to the following formulas, each of which is based on Eq 1 and the values in Table 2, assuming that the components are pure and stoichiometric:

**TABLE 2 Mass and Mass Loss Per Mole of Pure, Stoichiometric Cementitious Components, With Typical Decomposition Temperature Ranges**

Component	$M$ g/mol <sup>A</sup>	$C$ g/mol <sup>A</sup>	Temperature °C	
			Min (X)	Max (Y)
CaSO <sub>4</sub> · 0.5 H <sub>2</sub> O (Bassanite)	145.1	9	90	200
CaSO <sub>4</sub> · 2 H <sub>2</sub> O (Gypsum)	172.1	27	100	200
Mg(OH) <sub>2</sub> (Brucite)	58.3	18	230	330
K <sub>2</sub> Ca(SO <sub>4</sub> ) <sub>2</sub> · H <sub>2</sub> O (Syngenite)	328.4	18	250	300
Ca(OH) <sub>2</sub> (Portlandite)	74.1	18	380	600
MgCO <sub>3</sub> (Magnesite)	84.3	44	550	800
CaCO <sub>3</sub> (Calcite)	100.1	44	600	1000
Ca <sub>5</sub> (SiO <sub>4</sub> ) <sub>2</sub> CO <sub>3</sub> (Spurrite)	444.6	44	700	850

<sup>A</sup> Values of  $M$  and  $C$  assume pure mineral components.