Designation: D 4842 – 90 (Reapproved 2001)

Standard Test Method for Determining the Resistance of Solid Wastes to Freezing and Thawing[1]

This standard is issued under the fixed designation D 4842; 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 procedures for determining material losses produced by repeated freezing and thawing of solid waste specimens. It also covers the visual observation of the disintegration of solid specimens.

1.2 This test method intends that the material used in the procedure be physically, chemically, and biologically representative, hence it does not address problems as a result of the inhomogeneity of specimens.

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:*

C 305 Practice for Mechanical Mixing of Hydraulic Cement Pastes and Mortars of Plastic Consistency[2]

D 2216 Test Method for Laboratory Determination of Water (Moisture) Content of Soil and Rock[3]

3. Significance and Use

3.1 This test method is intended for the evaluation of the freezing and thawing resistance of monolithic, solid, solidified/stabilized wastes under the testing conditions of this test method.

3.2 This test method may be used for the comparison of freezing and thawing resistance of wastes.

3.3 Data tabulated in the charts shown in Figs. 1-3 may be used to observe irregularities caused by inhomogeneity of specimens or comparison of mass loss-cycle relations of different wastes, or both, as well as to measure method-related weight losses such as matrix dissolution.

4. Apparatus

4.1 *Disposable Molds*, 44-mm inside diameter by 74-mm in length.

4.2 *Balance or Scale*, with a capacity at least 50 % greater than the weight of the specimen and beaker, and a sensitivity of 0.01 g.

4.3 *Drying Oven*, a thermostatically controlled drying oven capable of maintaining a temperature of 60 ± 2°C; to be used for drying moisture specimen and for the solids content determination.

4.4 *Freezing Cabinet*, capable of maintaining − 20 ± 3°C.

4.5 *Refrigerator*, capable of maintaining + 4 ± 3°C.

4.6 *Moisture Chamber*, a suitably covered container capable of maintaining a temperature of 20 ± 3°C and maintain 95 % relative humidity, for preconditioning and thawing specimens.

4.7 *Beakers*, 400-mL size (narrow type), to store sample and to collect particulates.

4.8 *Tongs*, to handle samples.

5. Sample Preparation

5.1 *Specimen Size*— 44-mm diameter by 74-mm in length.

5.1.1 Specimens may be cut to size from larger samples.

5.1.2 Specimens can also be molded in disposable plastic molds. When molding specimens refer to Practice C 305 (see 2.1).

NOTE 1—Practice C 305 refers to pastes and mortars. Molding materials with different consistency may require modifications and may result in different precision.

5.2 Condition samples that are not molded for this test in the moisture chamber for a period of seven days.

5.2.1 Samples molded for this test have to be cured in the moisture chamber for a period of 28 days.

6. Procedure

6.1 Select one specimen for moisture content determination.

6.2 Determine moisture content of specimen in accordance with Test Method D 2216 but revised to use a temperature of 60° ± 3°C (see 2.1).

6.3 Select three specimens for testing and three for control and mark them respectively.

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[1] This test method is under the jurisdiction of ASTM Committee D34 on Waste Management and is the direct responsibility of Subcommittee D34.01.06 on Analytical Methods.

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[2] *Annual Book of ASTM Standards*, Vol 04.01.

[3] *Annual Book of ASTM Standards*, Vol 04.08.

TABLE 1 Control Group

Laboratory:
Technician:

Sample name:
Sample Id No.:
Test Start Date:

Sample 1 / Cycle No.	1					2					3				
Date	$T_{l,c,1}$	$B_{l,c,1}$	$W_{l,c,1}$	$M_{c,1}$	$R_{l,c,1}$	$T_{l,c,2}$	$B_{l,c,2}$	$W_{l,c,2}$	$M_{c,2}$	$R_{l,c,2}$	$T_{l,c,3}$	$B_{l,c,3}$	$W_{l,c,3}$	$M_{c,3}$	$R_{l,c,3}$
1.															
2.															
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															
11.															
12.															

TABLE 2 Sample Group

Laboratory:
Technician:

Sample name:
Sample Id No.
Test Start Date:

Sample 1 / Cycle No.	1					2					3				
Date	$T_{l,s,1}$	$B_{l,s,1}$	$W_{l,s,1}$	$M_{s,1}$	$R_{l,s,1}$	$T_{l,s,2}$	$B_{l,s,2}$	$W_{l,s,2}$	$M_{s,2}$	$R_{l,s,2}$	$T_{l,s,3}$	$B_{l,s,3}$	$W_{l,s,3}$	$M_{s,3}$	$R_{l,s,3}$
1.															
2.															
3.															
4.															
5.															
6.															
7.															
8.															
9.															
10.															
11.															
12.															

6.4 Weigh specimens (to the nearest 0.01 g).

6.5 Place each specimen into a tared beaker, dried in accordance with Test Method D 2216, and weighed to the nearest 0.01 g. Cover the beaker with a watch glass or plastic wrap.

6.6 Place the three beakers with testing specimens in a freezing cabinet. Maintain temperature at − 20 ± 3°C for 24 h.

6.7 Store the three beakers with the control specimens in the moisture chamber at 20°C for 24 h.

6.8 Remove the specimens from the freezing cabinet and the moisture chamber.

6.8.1 To the frozen specimens add 240 mL of distilled chilled water. This water shall be at a temperature of 4 ± 3°C.

6.8.2 To the control specimens, add 240 mL of room temperature water. This water shall be at a temperature of 20 ± 3°C.

6.8.3 Place a watch glass or plastic wrap on the beakers and store the water covered specimens at 20 ± 3°C for 23 h.

6.9 Using tongs, transfer each specimen to another dry

TABLE 3 Relative Weight Loss

Laboratory:
Technician:

Sample name:
Sample Id No.:
Test Start Date:

Cycle No.	$\bar{R}_{l,s}$	$\bar{R}_{l,c}$	$\bar{C}_l$	$\bar{S}_l$	Observations
1.					
2.					
3.					
4.					
5.					
6.					
7.					
8.					
9.					
10.					
11.					
12.					
$\bar{S}$					

beaker. This second set of beakers shall be prepared in accordance with 6.5.

NOTE 2—Excessive tong pressure may result in premature failure or damage to specimen.

6.10 Remove any loosely attached particulates by spraying distilled water from a wash bottle to the surface of specimen (10 to 20 mL distilled water). Let water drain into the beaker of origin.

6.11 Conduct visual observation on the specimens' physical deterioration including: cracking, fracturing, integrity, and surface roughness.

6.12 Determine the specimens' weight loss: the mass of the solid residue in beakers by evaporating water at 60 ± 3°C in drying oven.

6.13 Correct the average relative mass loss of samples using the average relative mass loss of control specimens.

6.14 Repeat the procedures in 6.5-6.10 eleven additional times, for a total of 12 cycles.

6.15 Terminate experiment of all specimens if the corrected cumulative mass loss of any of the specimens exceeds 30 % (failure), and note the number of cycles survived.

7. Calculation

7.1 Calculate the dry mass of specimens as follows:

$$M_s = 1 - \frac{w}{100} M_{sw} \text{ g}$$

where:
M_s = oven dry mass of specimen in g,
M_{sw} = initial mass of specimen in g, and
w = moisture content, %.

It is assumed that the moisture contents of specimens are identical. Oven dry masses of sample and control specimens are calculated on that basis.

7.2 Calculate corrected mass loss of specimens after each cycle. Express mass loss in percent of initial calculated oven-dry mass. Calculate average cumulated, corrected mass loss of specimens after each cycle as follows:

$$W_{i,s,j} = T_{i,s,j} - B_{i,s,j} \text{ g} \quad (1)$$

where:
$W_{i,s,j}$ = mass loss of sample j during cycle i, in g,
$T_{i,s,j}$ = oven-dry mass of beaker and residue of sample j after cycle i, in g, and
$B_{i,s,j}$ = oven-dry mass of beaker for sample j before cycle i, in g.

$$W_{i,c,j} = T_{i,c,j} - B_{i,c,j} \text{ g} \quad (2)$$

where:
$W_{i,c,j}$ = mass loss of control j during cycle i, in g,
$T_{i,c,j}$ = oven-dry mass of beaker and residue of control j after cycle i, in g, and
$B_{i,c,j}$ = oven dry mass of beaker for control j before cycle i, in g.

$$R_{i,s,j} = \frac{W_{i,s,j}}{M_{s,j}} \%$$

where:
$R_{i,s,j}$ = relative mass loss of sample j during cycle i, %,
$W_{i,s,j}$ = mass loss of sample j during cycle i, in g, and
$M_{s,j}$ = oven-dry mass of specimen j, in g.

$$R_{i,c,j} = \frac{W_{i,c,j}}{M_{c,j}} \% \quad (4)$$

where:
$R_{i,c,j}$ = relative mass loss of j control during cycle i, %,
$W_{i,c,j}$ = mass loss of control j during cycle i, in g, and
$M_{c,j}$ = oven-dry mass of control j, in g.

$$\bar{R}_{i,s} = \frac{\sum_{j=1-3} R_{i,s,j}}{3} \% \quad (5)$$

where:
$\bar{R}_{i,s}$ = average relative mass loss of samples ($j = 1 - 3$) during cycle i, %, and
$R_{i,s,j}$ = relative mass loss of sample j during cycle i, %.

$$\bar{R}_{i,c} = \frac{\sum_{j=1-3} R_{i,c,j}}{3} \% \quad (6)$$

where:
$\bar{R}_{i,c}$ = average relative mass loss of control ($j = 1 - 3$) during cycle i, %, and
$R_{i,c,j}$ = relative mass loss of control j during cycle i, %.

$$\bar{C}_i = \bar{R}_{i,s} - \bar{R}_{i,c} \% \quad (7)$$

where:
$\bar{C}_i$ = average corrected relative mass loss of samples ($j = 1 - 3$) during cycle i, %,
$\bar{R}_{i,s}$ = average relative mass loss of samples ($j = 1 - 3$) during cycle i, %, and
$\bar{R}_{i,c}$ = average relative mass loss of control ($j = 1 - 3$) during cycle i, %.

$$\bar{S}_i = \sum_{i=1-i} \bar{C}_i \% \quad (8)$$

where:
$\bar{S}_i$ = average cumulated, corrected relative mass loss of samples after i cycles %, and
$\bar{C}_i$ = average corrected relative mass loss of samples ($j = 1 - 3$) during cycle i, %.

$$\bar{S} = \sum_{i=1-12} \bar{C}_i \% \quad (9)$$

where:
$\bar{S}$ = average cumulated, corrected relative mass loss of samples after 12 cycles, %, and
$\bar{C}_i$ = average corrected relative mass loss of samples ($j = 1 - 3$) during cycle i, %.

8. Report

8.1 Report the following information:

8.1.1 Moisture content of specimens.

8.1.2 Average cumulative, corrected relative mass loss after 12 cycles, ($\bar{S}_i$).

8.1.3 Number of cycles survived if the specimens did not survive 12 cycles of testing.

8.1.4 Results of visual observation after each cycle (physical deterioration).

9. Precision and Bias [4]

9.1 *Precision*:

9.1.1 The precision of this test method, in terms of standard deviation, was determined in an interlaboratory experiment involving four laboratories, two types of specimens and respective controls. Duplicates of specimens and controls were measured in each laboratory.

9.1.2 The precision of this test method can be expressed as follows:

Sample Code	Mean ($\bar{X}$)	Standard Deviation (S)
LFP	0.09	0.07
CFP	1.99	1.20

9.1.3 The precision of this test method may be dependent on the level of the properties measured.

9.2 *Bias*:

Since there is no accepted reference material suitable determining the bias for the procedure in this test method, no statement on bias is being made.

[4] Supporting data are available from ASTM Headquarters. Request R.R. 1003.