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APPENDICES

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Appendix A

X-Ray Diffraction card of Hydroxyapatite (Ca₅(PO₄)₃OH)

9-432 MAJOR CORRECTION

d	2.81	2.78	2.72	2.17	Ca ₅ (PO ₄) ₃ (OH) 1/2 [Ca(OH) ₂ ·3Ca ₃ (PO ₄) ₂]		★			
I%	100	60	40	11	CALCIUM PHOSPHATE HYDROXIDE		(HYDROXYL-APATITE)			
Rad. CuKα, λ 1.5405	Fiber	Dia. 114.8mm			d Å	I%	hkl	d Å	I%	hkl
Calc. 50	1/2, PHOTOMETER*	(GUINIER CAMERA)			2.17	12	100	2.040	2	400
Ref. DEVLORFF, Techn. Phys. Dienst, DELFT, HOLLAND					2.24	6	101	2.000	4	202
					4.72	4	110	1.943	20	222
					4.07	10	200	1.890	14	212
					3.88	19	111	1.871	6	220
Syn. HEXAGONAL	S.G. P6 ₃ /m (176)				3.51	1	201	1.841	40	212
a: 1.418 Å	c: 1.284 Å	A	C: 0.7309		3.44	40	002	1.804	20	221
Ref. 1879.		Z: 2	D: 3.14		3.27	12	102	1.780	12	410
					3.08	12	210	1.754	14	402, 301
					2.814	100	211	1.722	20	004, 411
Sp. D	mp	Color	Sign		2.778	40	112	1.684	4	104
Ref.					2.720	40	300	1.644	16	222, 222
					2.631	23	202	1.611	3	212
					2.528	6	201	1.587	4	101, 204
					2.294	2	212	1.542	6	420
					2.282	20	210	1.520	6	221
					2.228	2	221	1.503	10	214, 421
					2.148	16	311	1.474	12	102
					2.124	4	202	1.445	4	210
					2.063	2	113			

* -- ARE PEAK VALUES FROM A PATTERN WHICH SHOWS
SLIGHT BROADENING OF PEAK APPEARANCES.
SAMPLE OBTAINED FOLLOWING THE PROCEDURE INDICATED BY
METZGER S.P., IND. ENG. CHEM. ANAL. Ed. 20 154 (1938).

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Appendix B

X-Ray Diffraction card
of
Alpha Tricalcium Phosphate
(α $\text{Ca}_3(\text{PO}_4)_2$)

9-348 MAJOR CORRECTION

d	2.91	3.62	3.91	12.3	α - $\text{Ca}_3(\text{PO}_4)_2$	★					
I%	100	90	40	4	ALPHA CALCIUM ORTHOPHOSPHATE						
Rad. Colls. λ 1.5405 Filter CuK α 114.6mm Cut off 50 I/L PHOTOMETER (GUINIER CAMERA) Ref. DEKALF, Techn. Phys. Dienst, DELFT, HOLLAND						d Å	I%	hkl	d Å	I%	hkl
Syn. Orthorhombic ^a S.G. a 18.22 b 20.71 c 9.107 A 0.7349 C 0.4398 β γ Z 16 D_2 2.87 Ref. 1819.						12.3	4	110	3.39	8	312
a b c β γ Z D_2 Ref. MARRAT (SEE BELOW)						7.31	25	111	3.33	4	421
						6.82	4	021	3.15	4	240
^a STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						6.29	10	130	3.12	4	242
						6.12	4	220	3.07	4	440
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						5.83	10	201	3.05	4	332
						5.18	12	131,040	3.01	20	810
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						4.55	4	002	2.947	20	112
						4.23	4	211	2.919	35	402,020
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						4.28	2	240,112	2.905	100	441,170
						4.17	2	022	2.840	30	811
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						4.00	20	150	2.814	2	203,422
						3.91	40	202	2.784	12	830
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						3.88	40	241	2.787	4	172
						3.81	8	400	2.734	<1	122
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						3.72	4	231	2.720	<1	222
						3.69	40	132	2.645	4	831
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						3.44	18	131,222	2.621	30	042,232
						3.31	4	401	2.590	20	080
* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MARRAT, Acta Cryst. 2 942 (1953) SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.						3.45	8	040	PLUS ADDITIONAL LINES		

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X-Ray Diffraction Standard Data
of
Beta Tricalcium Phosphate
(β $\text{Ca}_3(\text{PO}_4)_2$)

Indices (hkl)	d(\AA)	I/I_0	Indices (hkl)	d(\AA)	I/I_0
012	8.15	12	220	2.607	65
104	6.49	16	0.1.14	2.562	6
006	6.22	6	223	2.553	8
110	5.21	20	2.1.10	2.520	12
113	4.80	2	131	2.499	6
202	4.39	8	1.2.11, 226	2.407	10
018	4.15	4	315	2.375	6
024	4.06	16	1.0.16	2.263	10
116	4.00	4	1.1.15	2.249	4
1.0.10	3.45	25	042	2.241	2
211	3.40	4	404	2.195	14
.122	3.36	10	3.0.12	2.165	12
199, 208	3.25	8	1.2.14	2.103	4
214	3.21	55	0.2.16+	2.076	8
0.0.12, 125	3.11	2	321	2.063	4
300	3.01	16	232	2.061	6
3.2.10, 217	2.680	100	048	2.033	10
128	2.757	20	.324	2.023	6
306	2.710	10	3.1.11	2.017	4
1.1.12	2.674	8	Plus Additional lines		

X-Ray Diffraction card
of
Magnesium Oxide
(MgO)

4-0829 MAJOR CORRECTION

d	2.11	1.49	1.22	2.431	MgO					
4-0829										
4-0629	100	52	12	10	MAGNESIUM OXIDE	(PERICLASE)				
Rad. CuK α_1	λ 1.5405	Filter III		d Å	1/1 ₁	hkl	d Å	1/1 ₁	hkl	
Dia.	Cut off	Coll.		2.431	10	111				
1/1 ₁	G. C. DIFFRACTOMETER	decorr. abs.?		2.106	100	200				
Ref.	SWANSON AND TATGE, JC FEL. REPORTS, 1949			1.489	52	220				
				1.270	4	311				
				1.216	12	222				
Syn. Cubic		S.G. O _h ⁶ - Fm3m		1.0533	5	400				
a ₀ 4.213	b ₀	A	C	0.9665	2	331				
a	β	Z 4		.9415	17	420				
Ref. Ibid.				.8600	15	422				
				.8109	3	511				
ρ	2.581 mp	Color	Sign							
2V										
Ref. Ibid.										
HIGH PURITY PHOSPHOR SAMPLE FROM RCA HEATED AT 1900°C FOR 5 HRS. AT 26°C TO REPLACE 1-1235, 2-1207, 3-0998										

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Standard Test Methods for Rheological Properties of Non-Newtonian Materials by Rotational (Brookfield) Viscometer¹

This standard is issued under the fixed designation D 2196; 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 to replace Method 4287 of Federal Test Method Standard No. 141A. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

¹ NOTE—Section 24 was added editorially in August 1991.

1. Scope

1.1 These test methods cover the determination of the apparent viscosity and the shear thinning and thixotropic properties of non-Newtonian materials in the shear rate range from 0.1 to 50 s⁻¹.

1.2 This standard does not purport to address the safety problems 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 Document

- 2.1 *ASTM Standard:*
E 1 Specification for ASTM Thermometers²

3. Summary of Test Methods

3.1 Test Method A consists of determining the apparent viscosity of coatings and related materials by measuring the torque on a spindle rotating at a constant speed in the material.

3.2 Test Methods B and C consist of determining the shear thinning and thixotropic (time-dependent) rheological properties of the materials.³ The viscosities of these materials are determined at a series of prescribed speeds of a rotational-type viscometer. The agitation of the material immediately preceding the viscosity measurements is carefully controlled.

4. Significance and Use

4.1 Test Method A is used for determining the apparent viscosity at a given rotational speed, although viscosities at two or more speeds better characterize a non-Newtonian material than does the single viscosity measurement.

4.2 With Test Methods B and C, the extent of shear

thinning is indicated by the drop in viscosity with increasing viscometer speed. The degree of thixotropy is indicated by comparison of viscosities at increasing and decreasing viscometer speeds (Test Method B), viscosity recovery (Test Method B), or viscosities before and after high shear (combination of Test Methods B and C). The high-shear treatment in Test Method C approximates shearing during paint application. The viscosity behavior measured after high shear is indicative of the characteristics of the paint soon after application.

5. Apparatus

5.1 Rotational-type viscometers having at least four speeds, such as:

5.1.1 *Brookfield Viscometer*,⁴ Model LVF, having four rotational speeds, or Model LYT having eight rotational speeds, with set of four spindles; or

5.1.2 *Brookfield Viscometer*, Model RVF, having four rotational speeds, or Model RVT having eight rotational speeds, with set of seven spindles.

5.2 *Thermometer*—ASTM thermometer having a range from 20 to 70°C and conforming to the requirements for Thermometer 49C as prescribed in Specification E 1.

5.3 *Containers*, round 1-pt (0.5-L) can, 3 $\frac{3}{8}$ in. (85 mm) in diameter, or 1-qt (1-L) can, 4 in. (100 mm) in diameter.

5.4 *Shaker*,⁵ or equivalent machine capable of vigorously shaking the test specimen.

6. Materials

6.1 *Standard Oils*,⁶ calibrated in absolute viscosity, millipascal seconds.

7. Calibration of Apparatus

7.1 Select at least two standard oils of viscosities differing by at least 5 P (0.5 Pa·s) within the viscosity range of the material being measured and in the range of the viscometer. Condition the oils as closely as possible to 25.0°C (or other

¹ These test methods are under the jurisdiction of ASTM Committee D-1 on Paint and Related Coatings, Materials, and Applications and are the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

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² *Annual Book of ASTM Standards*, Vol 14.03.

³ Pierce, P. E., "Measurement of Rheology of Thixotropic Organic Coatings and Resins with the Brookfield Viscometer," *Journal of Paint Technology*, Vol 43, No. 557, 1971, pp. 35-43.

⁴ Brookfield viscometers are available from the Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

⁵ A reciprocating shaker may be obtained from the Red Devil Tools, 2400 Vauxhall Rd., Union, NJ 07083.

⁶ Absolute viscosity standards are available in 1-pt samples from The Cannon Instrument Co., P.O. Box 16, State College, PA 16801, or Brookfield Engineering Laboratories, Inc., 240 Cushing St., Stoughton, MA 02072.

agreed-upon temperature) for 1 h in a 1-pt (0.5-L) can, 3 3/8 in. (85 mm) in diameter. Measure the viscosities of each oil as described in Test Method B (Section 13) taking readings only at increasing speeds (13.7). Make certain that the spindle is centered in the container prior to taking measurements.

NOTE 1—The Brookfield LV and RV series viscometers are equipped with a spindle guard leg. The spindle/speed multiplying factors (Table 1) are designed for use with the guard leg in place except for the following conditions: RV series when the factors are the same with or without the guard leg for spindles No. 3 through 7; or LV series when the factors are the same with or without the guard leg for spindles No. 3 and 4.

7.1.1 Calibration in a 1-pt (0.5-L) can is always possible with the LV series viscometer with the guard leg attached. Calibration of the RV series viscometer in the 1-pt can must be done with spindles No. 3 through 7 without the guard leg. If the No. 1 or No. 2 spindles are to be used, calibration is carried out in the 1-qt (1-L) can with the guard leg attached.

7.2 Combining the tolerance of the viscometer ($\pm 1\%$, equal to the spindle/speed factor) and the tolerance of the temperature control (typically $\pm 0.5^\circ\text{C}$ at 25°C) it is reasonable to assume that a viscometer is calibrated if the calculated viscosities are within $\pm 5\%$ of the stated values (see Table 2 for examples of the considerable change in viscosity with temperature exhibited by standard oils). If measurements are not made at 25°C , then the stated viscosities should be corrected to the temperature at which they are measured. If the viscosities determined in 7.1 differ from the stated values of the viscosity standard by more than 5% , calculate new factors for each spindle/speed combination as follows:

$$f = V/s \quad (1)$$

where:

f = new factor for converting scale reading to viscosity, cP (mPa·s),

V = viscosity of standard oil, mPa·s, and

s = scale reading of the viscometer.

TABLE 1 Factors for Converting Brookfield Dial Readings to Centipoises (Millipascal Seconds)

NOTE—M = 1000.

Speed, rpm	RV Series Factors Spindles						
	1	2	3	4	5	6	7
0.5	200	600	2000	4000	8000	20M	60M
1	100	400	1000	2000	4000	10M	40M
2	50	200	500	1000	2000	5M	20M
2.5	40	160	400	800	1600	4M	16M
4	25	100	250	500	1000	2.5M	10M
5	20	80	200	400	800	2M	8M
10	10	40	100	200	400	1M	4M
20	5	20	50	100	200	500	2M
50	2	8	20	40	60	200	800
100	1	4	10	20	40	100	400

Speed, rpm	LV Series Factors Spindles			
	1	2	3	4
0.3	200	1000	4000	20M
0.6	100	500	2000	10M
1.5	40	200	800	4M
3.0	20	100	400	2M
6	10	50	200	1M
12	5	25	100	500
30	2	10	40	200
60	1	5	20	100

7.3 Prepare a table of new factors similar to that furnished with the viscometer (Table 1) for the spindle/speed combinations worked out in 7.2. Spindle/speed factors vary inversely with speed.

8. Preparation of Specimen

8.1 Fill a 1-pt or 1-qt can with sample to within 1 in. (25 mm) of the top with the sample and bring it as close as possible to a temperature of 25°C or other agreed-upon temperature prior to test.

8.2 Vigorously shake the specimen on the shaker or equivalent for 10 min, remove it from the shaker, and allow it to stand undisturbed for 60 min at 25°C prior to testing (Note 2). Start the test no later than 65 min after removing the can from the shaker. Do not transfer the specimen from the container in which it was shaken.

NOTE 2—Shake time may be reduced if necessary, or as agreed upon between the purchaser and manufacturer, but, in any case, should not be less than 3 min.

TEST METHOD A—APPARENT VISCOSITY

9. Procedure

9.1 Make all measurements as close as possible to 25°C , or other agreed-upon temperature.

9.2 Place the instrument on the adjustable stand. Lower the viscometer to a level that will immerse the spindle to the proper depth. Level the instrument using the attached spirit level.

9.3 Tilt the selected spindle (Note 3), insert it into one side of the center of the surface of the material, and attach the spindle to the instrument as follows: Firmly hold the upper shaft coupling with thumb and forefinger; screw left-hand thread spindle coupling securely to the upper shaft coupling being very careful when connecting to avoid undue side pressure which might affect alignment. Avoid rotating the dial so that pointer touches the stops at either extreme of the scale.

NOTE 3—Select the spindle/speed combination that will give a minimum scale reading of 10 but preferably in the middle or upper portion of the scale. The speed and spindle to be used may differ from this by agreement between user and producer.

9.4 Lower the viscometer until the groove (immersion mark) on the shaft just touches the material. Adjust the viscometer level if necessary. Move the container slowly in a horizontal plane until the spindle is located in approximately the center of the container so that the test will be run in a region undisturbed by the lowering of the spindle.

9.5 Turn on the viscometer. Adjust the viscometer to the rpm selected (Note 3) for the material under test. Allow the viscometer to run until the pointer has stabilized (Note 4). After the pointer has stabilized, depress the clutch and switch

TABLE 2 Viscosity Variation of Cannon Viscosity Standards About the 25°C Temperature Point

Cannon Viscosity Standard	Viscosity at 25°C , cP (mPa·s)	Viscosity Change With $+1^\circ\text{C}$ at 25°C , cP (mPa·s)
S-600	1 400	87.7 (6.26 %)
S-2000	4 900	332 (6.77 %)
S-8000	20 000	1462.3 (7.31 %)

off the motor so that when it stops, the pointer will be in view (Note 5).

NOTE 4—In thixotropic paints, the pointer does not always stabilize. On occasion it reaches a peak and then gradually declines as the structure is broken down. In these cases, the time of rotation or number of revolutions prior to reading the viscometer should be agreed to between user and manufacturer.

NOTE 5—Always release the clutch while the spindle is still immersed so that the pointer will float, rather than snap back to zero.

10. Calculation

10.1 Calculate the apparent viscosity at each speed, as follows:

$$V = fs$$

where:

V = viscosity of sample in centipoises, mPa·s,

f = scale factor furnished with instrument (see Table 1), and

s = scale reading of viscometer.

11. Report

11.1 Report the following information:

11.1.1 The Brookfield viscometer model and spindle,

11.1.2 The viscosity at the spindle/speed utilized,

11.1.3 The specimen temperature in degrees celsius, and

11.1.4 The shake time and rest period, if other than specified.

12. Precision and Bias

12.1 *Precision*—See Section 23 for precision, including that for measurement at a single speed.

12.2 *Bias*—No statement of bias is possible with this test method.

TEST METHOD B—VISCOSITY UNDER CHANGING SPEED CONDITIONS, DEGREE OF SHEAR THINNING AND THIXOTROPY

13. Procedure

13.1 Make all measurements with the Brookfield viscometer as close as possible to 25°C, or other agreed upon temperature.

13.2 Adjust the instrument and attach the spindle as in 9.2 through 9.4.

13.3 Set the viscometer at the slowest rotational speed (Notes 5 and 6). Start the viscometer and record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

NOTE 6—When the eight speed viscometers (RVT and LVT) are used, lower or higher speeds than that permitted by the four speed viscometers may be used upon agreement between producer and user.

13.4 Increase the viscometer speed stepwise and record the scale reading after ten revolutions (or equivalent time for each spindle/speed combination) at each speed. After an observation has been made at the top speed, decrease the speed in steps to the slowest speed, recording the scale reading after ten revolutions (or equivalent time) at each speed.

NOTE 7—It is preferable to change speed when the motor is running.

13.5 After the last reading has been taken at the slowest speed, shut off the viscometer and allow it and the specimen to stand undisturbed for an agreed-upon rest period. At the

end of the rest period, start the viscometer at the slowest speed and record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

14. Calculations and Interpretation of Results

14.1 Calculate the apparent viscosity at each speed as shown in Section 9.

14.2 If desired, determine the degree of shear thinning by the following method:

14.2.1 *Shear Thinning Index* (sometimes erroneously called the thixotropic index)—Divide the apparent viscosity at a low rotational speed by the viscosity at a speed ten times higher. Typical speed combinations are 2 and 20 rpm, 5 and 50 rpm, 6 and 60 rpm but selection is subject to agreement between producer and user. The resultant viscosity ratio is an index of the degree of shear thinning over that range of rotational speed with higher ratios indicating greater shear thinning.

14.2.2 A regular or log-log plot of viscosity versus viscometer speed in rpm may also be useful in characterizing the shear-thinning behavior of the material. Such plots may be used for making comparisons between paints or other materials.

14.3 If desired, estimate the degree of thixotropy (under conditions of *limited* shearing-out of structure) by one of the following methods:

14.3.1 Calculate the ratio of the slowest speed viscosity taken with increasing speed to that with decreasing speed. The higher the ratio, the greater the thixotropy.

14.3.2 Calculate the ratio of the slowest speed viscosity taken after the rest period to that before the rest period. The higher the ratio, the greater the thixotropy.

15. Report

15.1 Report the following information:

15.1.1 The Brookfield viscometer and spindle,

15.1.2 The viscosities at increasing and decreasing spindle speeds,

15.1.3 The rest period time and the viscosity at the end of that time,

15.1.4 The specimen temperature in degrees celsius, and

15.1.5 The shake time if other than that specified.

15.2 *Optional Reporting*:

15.2.1 *Degree of Shear Thinning*—Shear thinning index and speeds over which it was measured (14.2).

15.2.2 *Estimated Degree of Thixotropy* (under conditions of shearing-out of structure)—Ratio of the lowest speed viscosities, for both increasing and decreasing speeds; or ratio of the lowest speed viscosities before and after the rest period, and speed at which they were measured (14.3).

16. Precision and Bias

16.1 *Precision*—See Section 23 for precision, including that for measurement of the shear thinning index (ratio of viscosity at 5 r/min to that at 50 r/min). It has not been possible to devise a method for determining precision for viscosities at increasing and decreasing speeds other than as individual measurements. No attempt was made to determine the precision of the measurement of the degree of thixotropy because this parameter is dependent on the material, the time of the test, and other variables.

16.2 *Bias*—No statement of bias is possible with this test method.

TEST METHOD C—VISCOSITY AND SHEAR THINNING OF A SHEARED MATERIAL

17. Apparatus

17.1 High-speed laboratory stirrer with speeds of at least 2000 rpm and equipped with a 2-in. (50-mm) diameter circular dispersion blade.⁷

18. Preparation of Specimen

18.1 Insert the 2-in. (50-mm) blade into the center of the can (4.3) so that the blade is about 1 in. (25 mm) from the bottom. Run the mixer at 2000 rpm (Note 8) for 1 min.

NOTE 8—Materials may be sheared at other speeds using other size blades upon agreement between producer and user.

19. Procedure

19.1 Immediately insert the same spindle used in Test Method B into the sheared material in the same manner as in Section 9.

19.2 Start the viscometer and adjust to the highest speed used in Test Method B (13.5). Record the scale reading after ten revolutions (or other agreed-upon number of revolutions).

19.3 Decrease the viscometer speed (Note 7) step-wise and record the scale readings at each speed down to the lowest speed used in Test Method B, recording the scale reading after ten revolutions at each speed (or other agreed-upon number of revolutions).

20. Calculations and Interpretation of Results

20.1 As in Test Method B, calculate the viscosities at each decreasing speed.

20.2 If desired, calculate the degree of shear thinning by the method given in Test Method B, 14.2. The measured viscosity behavior after shearing is essentially that of the paint immediately after application (disregarding changes in solids).

20.3 If desired, estimate the degree of thixotropy (under conditions of *complete* shearing-out of structure) by calculating the ratio of the lowest speed viscosities before and after shear. The lowest speed before-shear viscosity is taken from Test Method B, 14.1, at the lowest increasing speed. The lowest speed after-shear viscosity is taken from 20.1. The higher the ratio, the greater the thixotropy.

21. Report

21.1 Report the following information:

- 21.1.1 The Brookfield viscometer model and spindle,
- 21.1.2 The viscosities at decreasing spindle speeds,
- 21.1.3 The specimen temperature in degrees celsius, and
- 21.1.4 The speed of the high-speed mixer, size of blade, and time of mixing if different from method.

21.2 *Optional Reporting*:

21.2.1 *Degree of Shear Thinning*—Shear thinning index and speed over which it was measured (14.2).

21.2.2 *Estimated Thixotropy*—Ratio of lowest speed viscosities before and after shear and the speed at which they were measured.

22. Precision and Bias

22.1 *Precision*—The precision for individual viscosity measurements is the same as for Test Method A in Section 23. No attempt has been made to determine the precision of the shear thinning index or degree of thixotropy for Test Method C for the reasons given in 16.1.

22.2 *Bias*—No statement of bias is possible with this test method.

23. Summary of Precision

23.1 In an interlaboratory study of Test Methods A and B, eight operators in six laboratories measured on two days the viscosities of four architectural paints comprising a latex flat, a latex semi-gloss, a water-reducible gloss enamel, and an alkyd semi-gloss, that covered a reasonable range in viscosities and were shear thinning. Measurements at increasing speeds of 5, 10, 20, and 50 r/min (equivalent to eight operators testing 16 samples) were used to obtain the precision of Test Method A. The within-laboratory coefficient of variation for Test Method A (single speed) was found to be 2.49 % with 121 degrees of freedom and for Test Method B (Shear Thinning Index) 3.3 % with 31 degrees of freedom. The corresponding between-laboratories coefficients are 7.68 % with 105 degrees of freedom and 7.63 % with 27 degrees of freedom. Based on these coefficients the following criteria should be used for judging the acceptability of results at the 95 % confidence level:

23.1.1 *Repeatability*—Two results obtained by the same operator at different times should be considered suspect if they differ by more than 7 % relative for single speed viscosity and 9.5 % relative for shear thinning index.

23.1.2 *Reproducibility*—Two results obtained by operators in different laboratories should be considered suspect if they differ by more than 21.6 and 22.1 % relative, respectively, for the same two test methods.

24. Keywords

24.1 Brookfield viscometer, non-Newtonian; rheological properties; rheology; rotational; shear thinning; thixotropic; thixotropy; viscometer; viscosity

⁷ Cowles or Shear type mixer/dispenser.

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Standard Test Methods for Apparent Porosity, Water Absorption, Apparent Specific Gravity, and Bulk Density of Burned Refractory Brick and Shapes by Boiling Water¹

This standard is issued under the fixed designation C 20; 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. Consult the DoD Index of Specifications and Standards for the specific year of issue which has been adopted by the Department of Defense.

1. Scope

1.1 These test methods cover the determination of the following properties of burned refractory brick:

- 1.1.1 Apparent porosity,
- 1.1.2 Water absorption,
- 1.1.3 Apparent specific gravity, and
- 1.1.4 Bulk density.

1.2 These test methods are not applicable to refractories attacked by water.

1.3 The values stated in inch-pound units are to be regarded as the standard. The values given in parentheses are for information only.

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.*

2. Referenced Document

2.1 ASTM Standard:

- C 134 Test Methods for Size and Bulk Density of Refractory Brick and Insulating Firebrick²
- E 691 Practice for Conducting an Interlaboratory Study to Determine the Precision of a Test Method³

3. Significance and Use

3.1 Apparent porosity, water absorption, apparent specific gravity, and bulk density are primary properties of burned refractory brick and shapes. These properties are widely used in the evaluation and comparison of product quality and as part of the criteria for selection and use of refractory products in a variety of industrial applications. These test methods are used for determining any or all of these properties.

3.2 These test methods are primary standard methods which are suitable for use in quality control, research and development, establishing criteria for and evaluating compli-

ance with specifications, and providing data for design purposes.

3.3 Fundamental assumptions inherent in these test methods are that the test specimens are not attacked by water, the test specimens conform to the requirements for size, configuration, and original faces, the open pores of the test specimens are fully impregnated with water during the boiling treatment, and the blotting of the saturated test specimens is performed as specified in a consistent and uniform manner to avoid withdrawing water from the pores. Deviation from any of these assumptions adversely affects the test results.

3.4 Certain precautions must be exercised in interpreting and using results from these test methods. All four property values are interrelated by at least two of the three base data values generated during testing. Thus, an error in any base data value will cause an error in at least three of the property values for a given test specimen. Certain of the properties, that is, apparent specific gravity and bulk density, are functions of other factors such as product composition, compositional variability within the same product, imperious porosity, and total porosity. Generalizations on or comparisons of property values should only be judiciously made between like products tested by these test methods or with full recognition of potentially inherent differences between the products being compared or the test method used.

4. Test Specimens

4.1 When testing 9-in. (228-mm) straight brick, use a quarter-brick specimen by halving the brick along a plane parallel to the 9 by 2-1/2 or 3-in. (228 by 64 or 76-mm) face and along a plane parallel to the 4-1/2 by 2-1/2 or 3-in. (114 by 64 or 76-mm) face. Four of the surfaces of the resultant quarter-brick specimen include part of the original molded faces.

4.2 When testing other refractory shapes, cut, drill, or break from each shape a specimen having volume of approximately 25 to 30 in.³ (410 to 490 cm³). The specimen shall include interior and exterior portions of the shape.

4.3 Remove all loosely adhering particles from each specimen.

5. Procedure

5.1 *Dry Weight, D*—Dry the test specimens to constant weight by heating to 220 to 230°F (105 to 110°C) and

¹ These test methods are under the jurisdiction of ASTM Committee C-8 on Refractories and are the direct responsibility of Subcommittee C08.03 on Physical Tests and Properties.

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² Annual Book of ASTM Standards, Vol 15.01.

³ Annual Book of ASTM Standards, Vol 14.02.

determine the dry weight, D , in grams to the nearest 0.1 g.

5.2 The drying procedure may be omitted only when the test specimens are known to be dry, as may be the case with samples taken directly from kilns.

5.3 The drying of the specimens to constant weight and the determination of their dry weight may be done either before or after the saturation operation (Section 6). Usually, the dry weight is determined before saturation. However, if the specimens are friable or evidence indicates that particles have broken loose during the saturating operation, dry and weigh the specimens after the suspended weight, S , and the saturated weight, W , have been determined, as described in Sections 7 and 8. Use this second dry weight in all appropriate calculations.

6. Saturation

6.1 Place the test specimens in water and boil for 2 h. During the boiling period, keep them entirely covered with water, and allow no contact with the heated bottom of the container.

6.2 After the boiling period, cool the test specimens to room temperature while still completely covered with water. After boiling keep the specimens immersed in water for a minimum of 12 h before weighing.

7. Suspended Weight, S

7.1 Determine the weight, S , of each test specimen after boiling and while suspended in water in grams to the nearest 0.1 g.

7.2 This weighing is usually accomplished by suspending the specimen in a loop or halter of AWG Gage 22 (0.643-mm) copper wire hung from one arm of the balance. The balance shall be previously counter-balanced with the wire in place and immersed in water to the same depth as is used when the refractory specimens are in place.

8. Saturated Weight, W

8.1 After determining the suspended weight, blot each specimen lightly with a moistened smooth linen or cotton cloth to remove all drops of water from the surface and determine the saturated weight, W , in grams by weighing in air to the nearest 0.1 g. Perform the blotting operation by rolling the specimen lightly on the wet cloth, which has previously been saturated with water, and then press only enough to remove such water as will drip from the cloth. Excessive blotting will induce error by withdrawing water from the pores of the specimen.

9. Exterior Volume, V

9.1 Obtain the volume, V , of the test specimens in cubic centimetres by subtracting the suspended weight from the saturated weight, both in grams, as follows:

$$V, \text{ cm}^3 = W - S \quad (1)$$

NOTE 1—This assumes that 1 cm³ of water weighs 1 g. This is true within about 3 parts in 1000 for water at room temperature.

10. Volumes of Open Pores and Impervious Portions

10.1 Calculate the volume of open pores and the volume of the impervious portions of the specimen as follows:

$$\text{Volume of open pores, cm}^3 = W' - D \quad (2)$$

$$\text{Volume of impervious portion, cm}^3 = D - S \quad (3)$$

11. Apparent Porosity, P

11.1 The apparent porosity expresses as a percentage the relationship of the volume of the open pores in the specimen to its exterior volume. Calculate P as follows:

$$P, \% = [(W' - D)/V] \times 100 \quad (4)$$

12. Water Absorption, A

12.1 The water absorption, A , expresses as a percentage the relationship of the weight of water absorbed to the weight of the dry specimen. Calculate A as follows:

$$A, \% = [(W' - D)/D] \times 100 \quad (5)$$

13. Apparent Specific Gravity, T

13.1 Calculate the apparent specific gravity, T , of that portion of the test specimen which is impervious to boiling water as follows:

$$T = D/(D - S) \quad (6)$$

14. Bulk Density, B

14.1 The bulk density, B , of a specimen in grams per cubic centimetre is the quotient of its dry weight divided by the exterior volume, including pores. Calculate B as follows:

$$B, \text{ g/cm}^3 = D/V \quad (7)$$

14.2 This test method of determining bulk density is useful for checking bulk density values obtained by direct measurement of Test Methods C 134.

NOTE 2—While it is more accurate than the direct measurement method, and generally gives higher values (by about 0.02 to 0.04), the direct measurement method is better suited for plant and field testing, since it is a less involved technique. The present method is preferable for specimens that are branded deeply or irregular in contour.

15. Report

15.1 For each property, report the individual values obtained.

15.2 Report apparent porosity and water absorption results to one decimal place, and apparent specific gravity and bulk density results to two decimal places.

NOTE 3—When values are reported for water absorption but not for porosity, it is suggested that the report shall also give the results for bulk density. This makes it possible to calculate the corresponding apparent porosity values as follows:

$$P, \% = A \times B \quad (8)$$

16. Precision and Bias

16.1 *Interlaboratory Test Data*—An interlaboratory round-robin test was conducted between six laboratories on three different types of refractories. The same four specimens of each material were sent from laboratory to laboratory, thereby eliminating sample variation. Each laboratory conducted two separate tests using two different operators, Operators A and B. The components of variance expressed as standard deviation and relative standard deviation (coefficient of variation) for absorption, apparent porosity, bulk density, and apparent specific gravity (ASG) were as given in Table 1.

TABLE 1 Interlaboratory Test Data

		Material			Grand Average
		A	B	C	
Absorption avg, %	X_i	12.37	6.67	4.83	...
Standard deviation within	S_r	0.1102	0.0776	0.075	0.0876
Standard deviation between	S_L	0.0866	0.0562	0.0182	0.0537
Relative standard deviation, %	V_r	0.89	1.16	1.55	1.20
	V_L	0.70	0.84	0.38	0.64
Apparent porosity, avg, %	X_i	22.24	14.44	11.22	...
	S_r	0.1693	0.1559	0.1696	0.1716
	S_L	0.0000	0.0000	0.0000	0.0000
	V_r	0.85	1.08	1.51	1.15
	V_L	0.00	0.00	0.00	0.00
Bulk density, avg	X_i	1.799	2.172	2.327	...
	S_r	0.00189	0.00458	0.00206	0.00284
	S_L	0.00274	0.00296	0.00267	0.00279
	V_r	0.105	0.211	0.089	0.135
	V_L	0.152	0.136	0.115	0.134
Apparent specific gravity, avg	X_i	2.314	2.539	2.621	...
	S_r	0.00577	0.00634	0.00495	0.00569
	S_L	0.00442	0.00468	0.00438	0.00449
	V_r	0.249	0.250	0.189	0.229
	V_L	0.191	0.184	0.167	0.181

TABLE 2 Precision and Relative Precision

Test Property	Precision		Relative Precision	
	Repeatability, i_r	Reproducibility i_m	% i_r	% i_m
Absorption, %	0.25	0.29	3.40	3.9
Apparent porosity, %	0.49	0.49	3.25	3.25
Bulk density	0.008	0.011	0.38	0.54
Apparent specific gravity	0.016	0.021	0.65	0.83

NOTE 4—All statistical calculations are in accordance with Practice E 691.

16.2 *Precision*—For the components of variation given in 16.1, a test result on any one sample should be considered significantly different at a confidence level of 95 %, if the repeatability used for reproducibility exceeds the precision data given in Table 2.

16.3 *Bias*—No justifiable statement on bias is possible since the true physical property values of refractories cannot be established by an accepted reference material.

17. Keywords

17.1 apparent porosity; apparent specific gravity; bulk density; refractory shapes; water absorption; water boil

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Standard Specification for Composition of Ceramic Hydroxylapatite for Surgical Implants¹

This standard is issued under the fixed designation F 1185; 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 NOTE—Keywords were added and Section 3 editorially corrected in March 1993.

1. Scope

1.1 This specification covers material requirements for ceramic hydroxylapatite intended for surgical implants. For a material to be called ceramic hydroxylapatite, it must conform to this specification. (See Appendix X1.)

1.2 The biological response to ceramic hydroxylapatite in soft tissue and bone has been characterized by a history of clinical use (1, 2, 3)² and by laboratory studies (4, 5, 6).

1.3 This specification specifically excludes hydroxylapatite coatings, non-ceramic hydroxylapatite, ceramic-glasses, tribasic calcium phosphate, whitlockite, and alpha- and beta-tricalcium phosphate. (See Specification F 1088.)

2. Referenced Documents

2.1 ASTM Standard:

F 1088 Specification for Beta-Tricalcium Phosphate for Surgical Implantation³

2.2 Code of Federal Regulations:⁴
Title 21, Part 820.

2.3 National Formulary:⁵

Tribasic Calcium Phosphate

2.4 United States Pharmacopeia:⁶

Identification Tests for Calcium and Phosphate <191>

Lead <251>

Mercury <261>

Arsenic <211>

Heavy Metals <231> Method 1

2.5 U. S. Geological Survey Method:⁷

Cadmium

3. Terminology

3.1 Descriptions of Terms Specific to This Standard:

¹ This specification is under the jurisdiction of ASTM Committee F-4 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.13 on Ceramic Materials.

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² The boldface numbers in parentheses refer to the list of references at the end of this specification.

³ Annual Book of ASTM Standards, Vol 13.01.

⁴ Available from U.S. Government Printing Office, Washington, DC 20402.

⁵ National Formulary XVI. Available from U.S. Pharmacopeia Convention, Inc., 12601 Twinbrook Parkway, Rockville, MD 20852.

⁶ United States Pharmacopeia XXI. Available from U.S. Pharmacopeia Convention, Inc., 12601 Twinbrook Parkway, Rockville, MD 20852.

⁷ Crook, J. G., Felichte, F. E., and Briggs, P. H., "Determination of Elements in National Bureau of Standards Geological Reference Materials SRM 278 Obsidian and SRM 688 Basalt by Inductively Coupled Argon Plasma—Atomic Emission Spectrometry," *Geostandards Newsletter*, Vol 7, 1983, pp. 335-340.

3.1.1 *calcining*—the heat treatment of a ceramic precursor for the purpose of eliminating volatile constituents. Calcining is also accompanied by some surface area and bulk volume reductions. Increases in mechanical properties are not usually significant.

3.1.2 *ceramic hydroxylapatite*—hydroxylapatite which has been fired at sintering temperatures. Firing time is mass dependent, and should be sufficiently long to cause significant densification and formation of a biologically stable form.

3.1.3 *hydroxylapatite*—the chemical substance having the empirical formula $\text{Ca}_5(\text{PO}_4)_3\text{OH}$.⁸

3.1.4 *sintering*—an integration of time and temperature of a ceramic precursor which develops a coherent body with useful properties. Sintering is a non-melting process accompanied by significant surface area and bulk volume reductions (densification), grain growth, and increases in mechanical properties.

4. Chemical Requirements

4.1 Elemental analysis for calcium and phosphorus will be consistent with the expected stoichiometry of hydroxylapatite.

4.2 A quantitative X-ray diffraction analysis shall indicate a minimum hydroxylapatite content of 95 % (7). Analysis of relative peak intensities shall be consistent with published data.⁹

4.3 The concentration of trace elements in the hydroxylapatite shall be limited as follows:

Element	ppm, max
As	3
Cd	5
Hg	5
Pb	30
total heavy metals (as lead)	50

For referee purposes, methods in 2.4 and 2.5 shall be used.

4.4 The maximum allowable limit of all heavy metals determined as lead will be 50 ppm as described in 2.4 or

⁸ Chemical Abstracts Service Registry Number [1306-06-5].

⁹ The Joint Committee on Powdered Diffraction Standards has established a Powder Diffraction File. The Committee operates on an international basis and cooperates closely with the Data Commission of the International Union of Crystallography and ASTM (American Society for Testing and Materials). Hydroxylapatite data can be found on file card number 9-432 and is available from the Joint Committee on Powder Diffraction Standards, 1600 Park Lane, Swarthmore, PA 19081.

4.5 It is recommended that all metals or oxides not detected as lead present in concentrations equal to or greater than 0.1 % be listed on the package insert.

5. Test Specimen Fabrication

5.1 Prepare test specimens from the same batch of material and by the same processes as those employed in

fabricating the ceramic implant device.

6. Quality Program Requirements

6.1 The manufacturer shall conform to Good Manufacturing Practices (2.2) or its equivalent.

APPENDIX

(Nonmandatory Information)

XI. RATIONALE

XI.1 Ceramic hydroxylapatite is commercially available as a synthetic bone-grafting material. As with any implant material, the bioresponse is critically dependent upon the material properties. To achieve reliable biocompatibility these must be known and consistent. This material standard provides specifications for a biocompatible grade of hydroxylapatite. Trace element content and leachability, physical form, and size must be within established biocompatibility standards.

XI.2 X-ray powder diffraction analysis provides better differentiation between hydroxylapatite and several commonly occurring second phases than traditional wet chemical methods.

XI.3 It is recognized that a separate performance standard may be necessary for each end-use product. For this reason, physical and mechanical properties were not specified. A source of general test methods for ceramics may be found in Ref (8).

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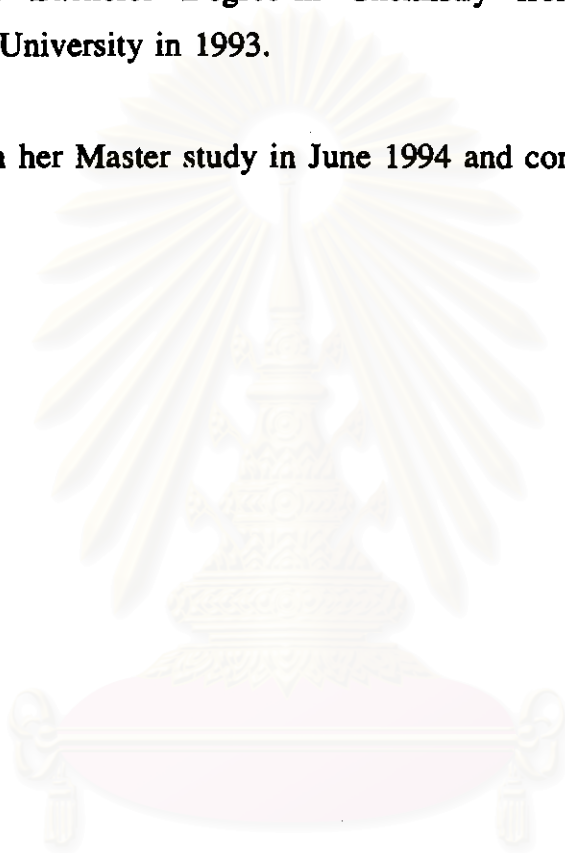
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