

REFERENCE

- Allen, T. Particle Size Measurement. Chapman and Hall Ltd., 1981. pp. 196-197.
- Aoki, H. Science and Material Applications of Hydroxyapatite. Tokyo, Takayama Press System Center Co, Inc., 1991.
- _____, Okayama, S. and Akao, M. Effect of Strontium Content on the mechanical properties of Bone and Sintered Hydroxyapatite. In W. Bonfield, G.W.Hastings and K.E.Tanner (eds.), Bioceramics Volume 4 Proceedings of the 4th International Symposium on Ceramics in Medicine, pp. 87-90.
- Boretos, J.W. Ceramics in Clinical Cave. Am.Ceram.Bull. 8(1985): 1098-1100.
- Brubaker, B.D. and Russel, R., JR. Residual Stress Development in Laminar Ceramics. Am.Ceram.Ball. 12(1967): 1194-1197.
- Callis, P.D., Donaldson, K. and McCord, J.F. Early Cellular Responses to Calcium phosphate Ceramics. Clinical Materials. 3(1988): 183-190.
- Cullity, B.D. Elements of X-ray diffraction. Addison-Wesley Publishing Company, Inc., 1967. pp. 177-184.

- Duke, D.A., McGles, J.E., JR., Macdowell, J.F. and Bopp, H.F. Strengthening Glass-Ceramics by Application of Compressive Glazes. J.Am.Ceram.Soc. 2 (1968): 98-102.
- Groot, K.D. Calcium Phosphate Bioceramics. In J.P.Paul, J.D.S. Gaylor, J.M.Courtney and T.Gilchrist(eds.), Biomaterials in Artificial Organs (Proceedings of a seminar on biomaterials), pp. 301-311. Glasgow, 1983.
- Hench, L.L. Bioceramics: From Concept to Clinic. J.Am.Ceram.Soc. 7 (1991): 1487-1510.
- Jarcho, M. The Future of Hydroxyapatite Ceramics. In H.Oonishi, H.Aoki and Sawai (eds.), Bioceramics (Proceedings of 1st International Bioceramics Symposium), pp. 57-61. Japan, 1988.
- _____, et al. Hydroxyapatite Synthesis and Characterization in Dense Polycrystalline Form. J.Mat.Sci. 11(1976) : 2027-2035.
- Kingery, W.D., Bowen, H.K. and Uhlmann, D.R. Introduction to Ceramics(2nd ed). Singapore, John Willey & Sons (SEA) Pte.Ltd., 1991. PP. 1-20.
- Kirchner, H.P. Strengthening of Ceramics : Treatments, Test, and Design Applications. New York, Marcel Dekker, Inc., 1979. pp. 203-204.
- Kondo, K., et al. Preparation of High-Strength Apatite Ceramics. J.Am.Ceram.Soc. 11(1984): c-222-c-223.

Lavernia, C. and Schoenung, J.M. Calcium Phosphate
Ceramics as Bone Substitues. Am.Ceram.Bull.
1(1991): 95-100.

Lorprayoon, C. Synthesis of Calciumhydroxyapatite and
Tricalcium phosphate from Bone Ash. Adv.Mats.
(1989): 329-335.

McColm, I.J. and Clark, N.J. Forming, Shaping and
Working of High-Performance Ceramics. London,
Blackie and Son Ltd., 1988.

Monma, H. and Nagai, M. Calcium Orthophosphates. In
T. Kanazawa (ed.), Inorganic Phosphate Materials,
pp. 87-91. Kodansha Ltd., 1989.

Newesely, H. and Osborn, J.F. Structural and Textural
Implications of Calcium Phosphates in Ceramics.
In G.W. Hastings and D.F. Williams (eds.),
Mechanical Properties of Biomaterials, pp. 457-
464. John Wiley & Son Ltd., 1980.

Nordstrom, E.G. and Karlsson, K.H. Slip-Cast Apatite
Ceramics by Application of Compressive Glazes.
J.Am.Ceram.Soc. 2(1968): 98-102.

Parmelee, C.W. Ceramics Glazes. Cahners Publishing
Company, Inc., 1973.

- Ray, C., et al. Apatite Chemistry in Biomaterial Preparation, Shaping and Biological Behaviour. In W.Bonfield, G.W. Hastings and K.E. Tanner (eds.), Bioceramics Volume 4 Proceedings of the 4th International Symposium on Ceramics in Medicine, pp. 57-64. London, 1991.
- Reed, J.S. Introduction to the Principles of Ceramic Processing. John Wiley & Sons (SEA) Pte. Ltd., 1989.
- Richerson, D.W. Modern Ceramic Engineering. New York, Marcel Dekker, Inc., 1982. pp.147-301.
- Santos, J.D., Morrey, S., Hastings, G.W. and Monteiro, F.J. The production and Characterisation of a Hydroxyapatite Ceramic Material. In W.Bonfield, G.W.Hasting and K.E. Tanner(eds.), Bioceramics Volume 4 Proceedings of the 4th International Symposium on Ceramics in Medicine, pp. 87-90. London, 1991.
- Somasundaram, P. Theories of Grinding. In G.Y.Onoda, Jr. and L.L.Hench (eds.), Ceramic Processing Before firing, pp. 105-123. John Wiley & Sons, Inc., 1978.
- Sombuthawee, C. Phase of Cattle Bones at Elevated Temperatures. "Rachadapiseksompoj" Research Fund, Chulalongkorn University, 1985.

- _____, Monroe, E.A. and Rausch, J.D. Strengthening of Calcium Hydroxyapatite by Ion Exchange. Biomater. Med. Devices and Artif. Org. 4(1981): 244-245.
- Taylor, J.R. and Bull, A.C. Ceramic Glaze Technology. Pergamon Press, 1986. pp. 75-96.
- Ueno, Y., Shima, Y. and Akiyama, T. Development of a New Biomaterial as a Bone Substitute ; True Bone Ceramic. In P.Vincezini (ed.), High Tech Ceramics, pp. 369-378. Elsevier Science Publishers, 1987.

APPENDICES

Appendix A

X-Ray Diffraction Card
of Hydroxyapatite

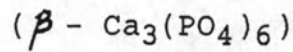
9-432 MAJOR CORRECTION

d	2.81	2.78	2.72	8.17	$\text{Ca}_5(\text{PO}_4)_3(\text{OH})$ $\frac{1}{2}[\text{Ca}(\text{OH})_2 \cdot 3\text{Ca}_3(\text{PO}_4)_2]$	* CALCIUM PHOSPHATE HYDROXIDE (HYDROXYL-APATITE)				
I/I ₁	100	60	60	11						
Rad. $\text{CuK}\alpha_1$ λ 1.5405 Filter Dia. 114.6mm Cut off 50 I/I ₁ PHOTOMETER* (GUINIER CAMERA) Ref. DEWOLFF, TECHN. PHYS. DIENST, DELFT, HOLLAND					d Å	I/I ₁	hkl	d Å	I/I ₁	hkl
Sys. HEXAGONAL S.G. $\text{P6}_3/\text{m}$ (176) a 9.418 b c 6.884 A C 0.7309 Ref. IBID. Y Z 2 D _z 3.16					8.17	12	100	2.040	2	400
					5.26	6	101	2.000	6	200
fe n w s f y Color Sign 2V D 3.08 mp Ref.					4.72	4	110	1.943	30	222
					4.07	10	200	1.890	16	312
* I/I ₁ ARE PEAK VALUES FROM A PATTERN WHICH SHOWS SLIGHT BROADENING OF PRISM REFLECTIONS. SAMPLE OBTAINED FOLLOWING THE PROCEDURE INDICATED BY HODGE C.S., IND. ENG. CHEM. ANAL. ED. 10 156 (1938).					3.88	10	111	1.871	6	320
					3.51	2	201	1.841	40	213
PLUS ADDITIONAL LINES					3.43	40	002	1.806	20	321
					3.17	12	102	1.780	12	410
2.814 100 211 1.754 16 402,303 1.722 20 004,411					3.08	18	210	1.754	16	410
					2.778	60	112	1.694	4	104
2.720 60 300 1.644 10 322,223 2.631 25 202 1.611 8 313 2.528 6 301 1.58 4 501,204 2.296 8 212 1.542 6 420 2.262 20 310 1.520 6 331 2.228 2 221 1.503 10 214,421 2.148 10 311 1.474 12 502 2.124 4 302 1.465 4 510					2.814	100	211	1.722	20	004,411
					2.778	60	112	1.694	4	104
2.720 60 300 1.644 10 322,223 2.631 25 202 1.611 8 313 2.528 6 301 1.58 4 501,204 2.296 8 212 1.542 6 420 2.262 20 310 1.520 6 331 2.228 2 221 1.503 10 214,421 2.148 10 311 1.474 12 502 2.124 4 302 1.465 4 510					2.720	60	300	1.644	10	322,223
					2.631	25	202	1.611	8	313
2.528 6 301 1.58 4 501,204 2.296 8 212 1.542 6 420 2.262 20 310 1.520 6 331 2.228 2 221 1.503 10 214,421 2.148 10 311 1.474 12 502 2.124 4 302 1.465 4 510					2.528	6	301	1.58	4	501,204
					2.296	8	212	1.542	6	420
2.262 20 310 1.520 6 331 2.228 2 221 1.503 10 214,421 2.148 10 311 1.474 12 502 2.124 4 302 1.465 4 510					2.262	20	310	1.520	6	331
					2.228	2	221	1.503	10	214,421
2.148 10 311 1.474 12 502 2.124 4 302 1.465 4 510					2.148	10	311	1.474	12	502
					2.124	4	302	1.465	4	510
2.065 d 113					2.065	d	113	1.465	4	510

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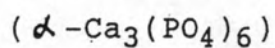
Appendix B**X-Ray Diffraction Standard Data
of Some Calcium Phosphates**

Beta Tricalcium Phosphate



Indices (hkl)	d(A°)	I_{I_0}	Indices (hkl)	d(A°)	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
0.2.10, 217	2.880	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		

Alpha Tricalcium Phosphate



Indices (hkl)	d(A°)	$\frac{I}{I_0}$	Indices (hkl)	d(A°)	$\frac{I}{I_0}$
110	12.3	4	312	3.35	8
111	7.31	25	421	3.33	4
021	6.82	4	260	3.15	4
130	6.29	10	242	3.12	4
220	6.12	4	440	3.07	4
201	5.83	10	332	3.05	4
131, 040	5.18	12	510	3.01	20
002	4.55	4	113	2.947	20
311	4.33	4	402, 023	2.919	35
240, 112	4.28	2	441, 170	2.905	100
022	4.17	2	511	2.860	30
150	4.00	20	203, 422	2.816	2
202	3.91	40	530	2.786	12
241	3.88	40	171	2.767	4
400	3.81	8	133	2.734	1
331	3.73	4	223	2.720	1
132	3.69	40	531	2.665	4
151, 222	3.66	18	043, 352	2.621	50
401	3.51	4	080	2.590	30
060	3.45	6	Plus Additional lines.		

Hilgenstockite ($\text{Ca}_4\text{P}_2\text{O}_9$)

Indices (hkl)	d(\AA°)	I_{10}	Indices (hkl)	d(\AA°)	I_{10}
110	6.06	4	032, 013	3.053	80
020	5.99	5	040	2.995	100
$\bar{1}01$	5.69	4	$\bar{1}03, \bar{2}21$	2.895	30
002	4.739	3	221	2.872	45
120	4.558	5	$\bar{1}13, \bar{1}32$	2.811	25
012	4.405	2	023, 132	2.789	35
$\bar{1}21$	4.122	14	113	2.784	35
121	4.092	20	$\bar{2}12$	2.763	50
$\bar{1}02$	3.959	2	212	2.724	40
102	3.897	3	$\bar{1}41$	2.649	14
$\bar{1}12$	3.754	4	141	2.642	18
022, 112	3.711	8	$\bar{1}23$	2.607	12
031	3.684	10	123	2.583	6
200	3.511	45	$\bar{2}22$	2.567	5
130	3.473	20	$\bar{2}31$	2.545	9
210	3.366	3	222, 231	2.534	16
$\bar{2}01, \bar{1}22$	3.302	7	033	2.476	16
$\bar{1}31, 122$	3.268	16	$\bar{1}42$	2.387	2
211	3.190	30	004, $\bar{2}03$	2.367	7
211, 003	3.160	20	051, 014	2.322	15

Plus Additional Lines

Appendix C

American Atandard Test Method
for Tap Density of Powders of Refractory Metals
and Compounds by Tap-Pak Volumerer.

Standard Test Method for
TAP DENSITY OF POWDERS OF REFRACTORY METALS
AND COMPOUNDS BY TAP-PAK VOLUMETER¹

This standard is issued under the fixed designation B 527; 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 (ϵ) indicates an editorial change since the last revision or reappraisal.

1. Scope

1.1 This method covers determination of the tap density (packed density) of refractory metal powders and compounds by means of the Tap-Pak Volumeter.²

1.2 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

2. Apparatus

2.1 *Graduated Cylinder*,³ calibrated to contain 25 mL at 20°C, internal diameter 15 mm, height 180 mm and weight approximately 60 g.

2.2 *Holder*—A cylinder holder weighing 1 lb (454 g).

2.3 *Tapping Device*, consisting of a baseplate with single-phase a-c condenser motor, with worm drive, reduction ratio 15 to 1, cam shaft speed 250 rpm, tapping stroke travel 1/8 in. (3.17 mm).

2.4 *Counter*—A four-digit adjustable counter, which can be preset to deliver numbers of taps between 1 and 9999.

2.5 *Balance*, having a capacity of at least 100 g and a sensitivity of 0.1 g.

3. Significance and Use

3.1 This test provides a guide for evaluation of the tapped density physical characteristic of powders. The degree of correlation between the results of this test and the quality of powders in use will vary with each particular application and has not been fully determined.

4. Test Specimen

4.1 The test specimen shall be 50 g except as noted in 4.2.

4.2 For refractory metal and compound powders too voluminous to fit into the 25-mL graduated cylinder, reduce sample size to 20 g or 10 g, as necessary, and follow the standard procedure.

5. Procedure

5.1 Weigh 50 g of the test specimen to an accuracy of ± 0.1 g.

5.2 Pour the test specimen carefully into the graduated cylinder, using a funnel. To ensure proper level, rotate the funnel while pouring the test specimen.

5.3 Preset the counter for 3000 taps.

5.4 Start tapping device.

5.5 Read the tapped volume, V , in millilitres, by calculating the mean value between the highest and the lowest point at the tapped volume.

6. Calculation and Report

6.1 Calculate tap density in grams per cubic centimetre to the nearest tenth by dividing 50 g (10 or 20 g for samples as noted in 4.2) by the tapped volume, V , read in millilitres as follows:

$$\text{Tap density, g/cm}^3 = 50 \text{ g} / V$$

¹ This method is under the jurisdiction of ASTM Committee B-9 on Metal Powders and Metal Powder Products. Current edition approved July 31, 1981. Published September 1981. Originally published as B 527 - 70. Last previous edition B 527 - 70 (1976).

² Tap-Pak Volumeter Model No. JEL ST2 manufactured by J. Engelsmann A.G. of Ludwigshafen a. Rh. West Germany. Available through Shandon Scientific Company, Sewickley, Pennsylvania.

³ Example: Corning, No. 3046, Pyrex Brand.

where

V = tapped volume, mL.

7. Report

7.1 Report the average of three tests.

8. Precision and Accuracy

8.1 Precision and accuracy cannot be stated at this time as the standard covers a broad range of powders and to establish such numbers is not warranted.

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, 1916 Race St., Philadelphia, Pa. 19103.

Appendix D

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



Designation: D 2196 - 86 (Reapproved 1991)¹

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 LVT 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, 3³/₈ 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 and Materials and are the direct responsibility of Subcommittee D01.24 on Physical Properties of Liquid Paints and Paint Materials.

Current edition approved Aug. 29, 1986. Published October 1986. Originally published as D 2196 - 63 T. Last previous edition D 2196 - 81.

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, 3³/₈ 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	500	2000	4000	8000	20M	80M
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	80	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 (6.25%)
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:

⁷ Cowles or Shar type mixer/disperser.

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

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Appendix E

American Standard Test Method
for Water Absorption, Bulk Density, Apparent Porosity
and Apparent Specific Gravity of Fired Whiteware Products

Designation: C 373 - 72 (Reapproved 1982)

This standard is issued under the fixed designation C 373; 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 method covers procedures for determining water absorption, bulk density, apparent porosity, and apparent specific gravity of fired unglazed whiteware products.

2. Apparatus and Materials

2.1 *Balance*, of adequate capacity, suitable to weigh accurately to 0.01 g.

2.2 *Oven*, capable of maintaining a temperature of 150 ± 5 C (302 ± 9 F).

2.3 *Wire Loop, Halter, or Basket*, capable of supporting specimens under water for making suspended mass measurements.

2.4 *Container*—A glass beaker or similar container of such size and shape that the sample, when suspended from the balance by the wire loop, specified in 2.3, is completely immersed in water with the sample and the wire loop being completely free of contact with any part of the container.

2.5 *Pan*, in which the specimens may be boiled.

2.6 *Distilled Water*.

3. Test Specimens

3.1 At least 5 representative test specimens shall be selected. The specimens shall be unglazed and shall have as much of the surface freshly fractured as is practical. Sharp edges or corners shall be removed. The specimens shall contain no cracks. The individual test specimens shall weigh at least 50 g.

4. Procedure

4.1 Dry the test specimens to constant

mass (Note) by heating in an oven at 150 C (302 F), followed by cooling in a desiccator. Determine the dry mass, D , to the nearest 0.01 g.

NOTE—The drying of the specimens to constant mass and the determination of their masses may be done either before or after the specimens have been impregnated with water. Usually the dry mass is determined before impregnation. However, if the specimens are friable or evidence indicates that particles have broken loose during the impregnation, the specimens shall be dried and weighed after the suspended mass and the saturated mass have been determined, in accordance with 4.3 and 4.4. In this case, the second dry mass shall be used in all appropriate calculations.

4.2 Place the specimens in a pan of distilled water and boil for 5 h, taking care that the specimens are covered with water at all times. Use setter pins or some similar device to separate the specimens from the bottom and sides of the pan and from each other. After the 5-h boil, allow the specimens to soak for an additional 24 h.

4.3 After impregnation of the test specimens, determine to the nearest 0.01 g the mass, S , of each specimen while suspended in water. Perform the weighing by placing the specimen in a wire loop, halter, or basket that is suspended from one arm of the balance. Before actually weighing, counterbalance the scale with the loop, halter, or basket in place and immerse in water to the same depth as is used when the specimens are in place. If it is

¹ This method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products. Current edition approved Aug. 29, 1972. Published October 1972. Originally published as C 373 - 55 T. Last previous edition C 373 - 56 (1970).

desired to determine only the percentage of water absorption, omit the suspended mass operation.

4.4 After the determination of the suspended mass or after impregnation, if the suspended mass is not determined, blot each specimen lightly with a moistened, lint-free linen or cotton cloth to remove all excess water from the surface, and determine the mass to 0.01 g.

Perform the blotting operation by rolling the specimen lightly on the wet cloth, which shall previously have been saturated with water and then pressed only enough to remove such water as will drip from the cloth. Excessive blotting will introduce error by withdrawing water from the pores of the specimen. Make the weighing immediately after blotting, the whole operation being completed as quickly as possible to minimize errors due to evaporation of water from the specimen.

5. Calculations

5.1. In the following calculations, the assumption is made that 1 cm³ of water weighs 1 g. This is true within about 3 parts in 1000 for water at room temperature.

5.1.1 Calculate the exterior volume, V , in cubic centimetres, as follows:

$$V = M - S$$

5.1.2 Calculate the volumes of open pores and impervious portions in cubic centimetres as follows:

$$\text{Volume of open pores, cm}^3 = M - D$$

$$\text{Volume of impervious portions, cm}^3 = D - S$$

5.1.3 The apparent porosity, P , expresses, as a percentage, the relationship of the volume of the open pores of the specimen to

its exterior volume. Calculate the apparent porosity as follows:

$$P = [(M - D)/V] \times 100$$

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

$$A = [(M - D)/D] \times 100$$

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

$$T = D/(D - S)$$

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

$$B = D/V$$

6. Report

6.1 For each property, report the average of the values obtained with at least 5 specimens, and also the individual values. Where there are pronounced differences among the individual values, another lot of 5 specimens shall be tested and in addition to individual values the average of all 10 determinations shall be reported.

7. Precision and Accuracy

7.1 This method is accurate to ± 0.2 percent water absorption in interlaboratory testing when the average value recorded by all laboratories is assumed to be the true water absorption. The precision is approximately ± 0.1 percent water absorption on measurements made by a single experienced operator.

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