

References

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APPENDICES

Appendix A.

Specific Surface Area of CaCO₃

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300

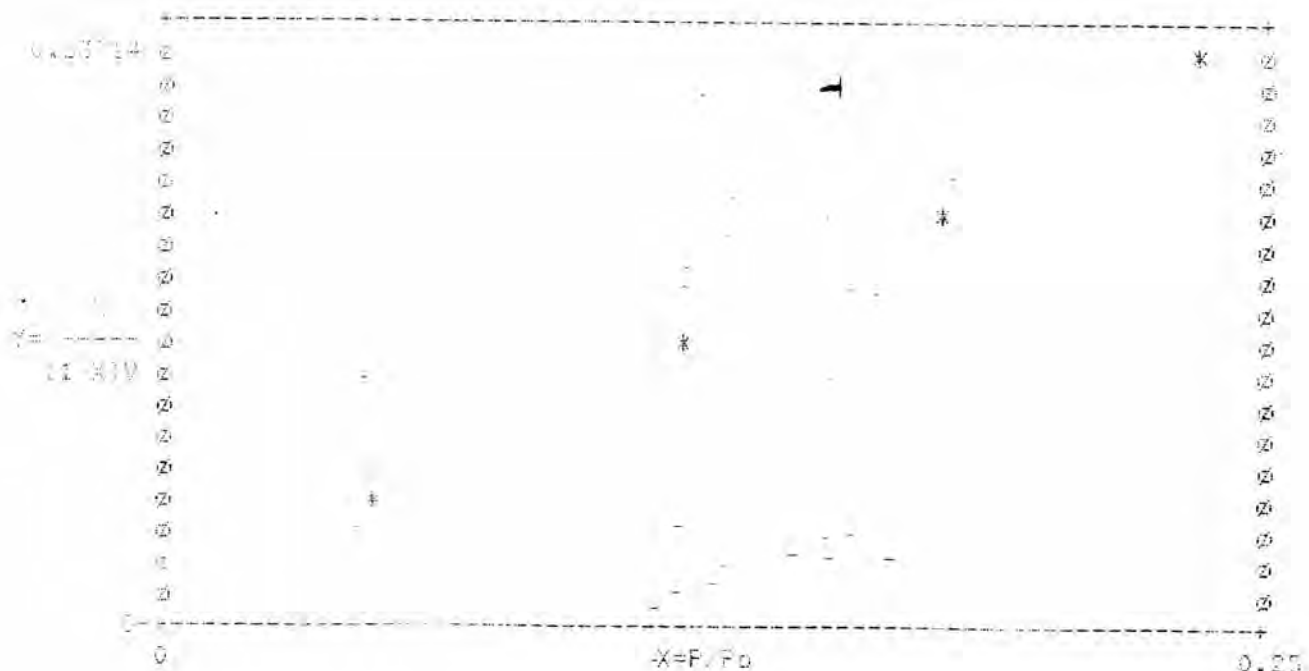
BET SURFACE AREA ANALYSIS
REPORT DATE: 07/31/97

SAMPLE I.D.: calcium carbonate
SAMPLE WEIGHT: 0.0175 g
MOL. CROSS-SECTIONAL AREA: 0.162 nm²
AMBIENT TEMPERATURE: 0.00 C

ADSORBATE: Nitrogen
BAROMETRIC PRESSURE: 760 mmHg
SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/P ₀	Y=X/g(1-X)/VA
5.000	0.18	0.35	0.0490	0.14824
12.000	0.21	0.41	0.1177	0.32867
18.000	0.24	0.46	0.1765	0.46220
24.000	0.25	0.48	0.2354	0.63714

BET SURFACE AREA: 1.667 m²/g
SLOPE: 2.5e93
INTERCEPT: 0.0155
C: 133.53
V_m: 0.36 cm³/g
CORRELATION COEFFICIENT: 0.9998



Appendix B.

Specific Surface Area of dry grinded TTCP for 5 h.

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300

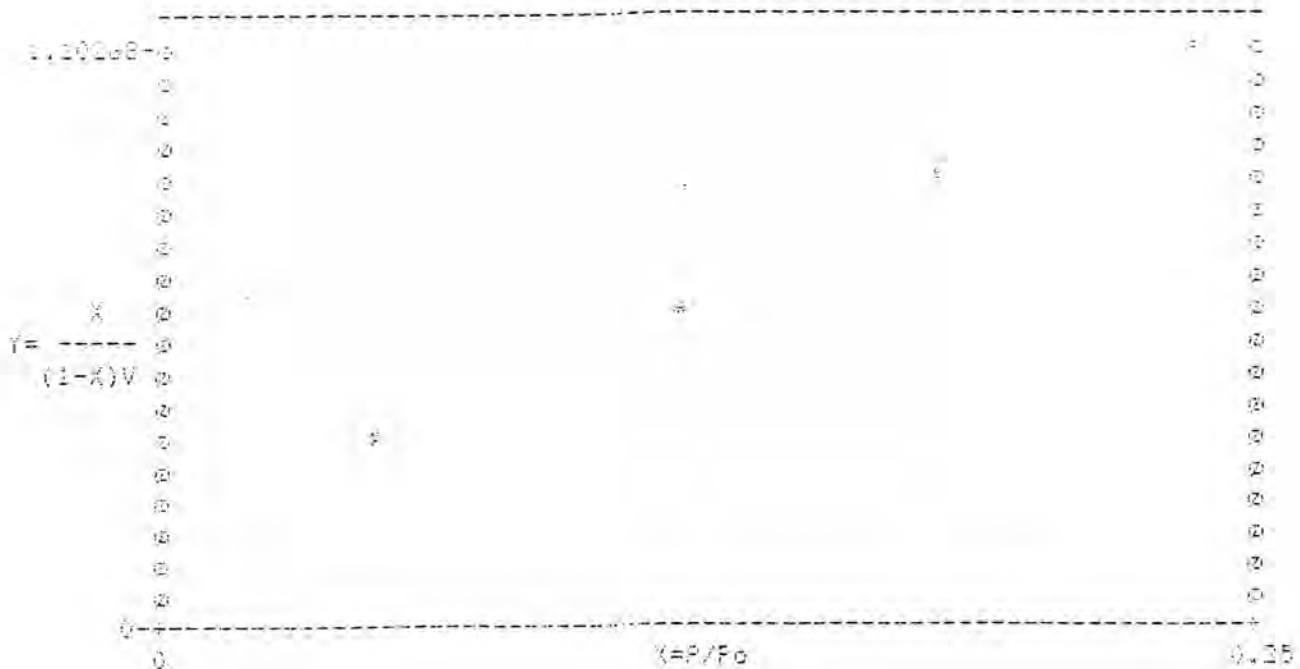
BET SURFACE AREA ANALYSIS
REPORT DATE: 5/28/97

SAMPLE I.D.: TTCP (dry 5 hours)
SAMPLE WEIGHT: 0.2866 g
MOL. CROSS-SECTIONAL AREA: 0.162 nm²
AMBIENT TEMPERATURE: 0.00 C

ADSORBATE: Nitrogen
BAROMETRIC PRESSURE: 760 mmHg
SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA		VOL ADSORBED	X=P/Po	Y=X/E(1-X)V _m
(%)	(VOL)	(cm ³ /g AT STP)		
5.000	0.04	0.14	0.0490	0.36943
11.000	0.06	0.21	0.1177	0.63708
18.000	0.07	0.24	0.1765	0.87762
24.000	0.08	0.28	0.2354	1.10268

BET SURFACE AREA: 1.06 +/- 0.01 m²/g
SLOPE: 3.9497 +/- 0.0325
INTERCEPT: 0.1754 +/- 0.0052
C: 23.52
V_m: 0.24 cm³/g
CORRELATION COEFFICIENT 0.9999



Appendix C.
Specific Surface Area of wet grinded TTCP for 3 h.

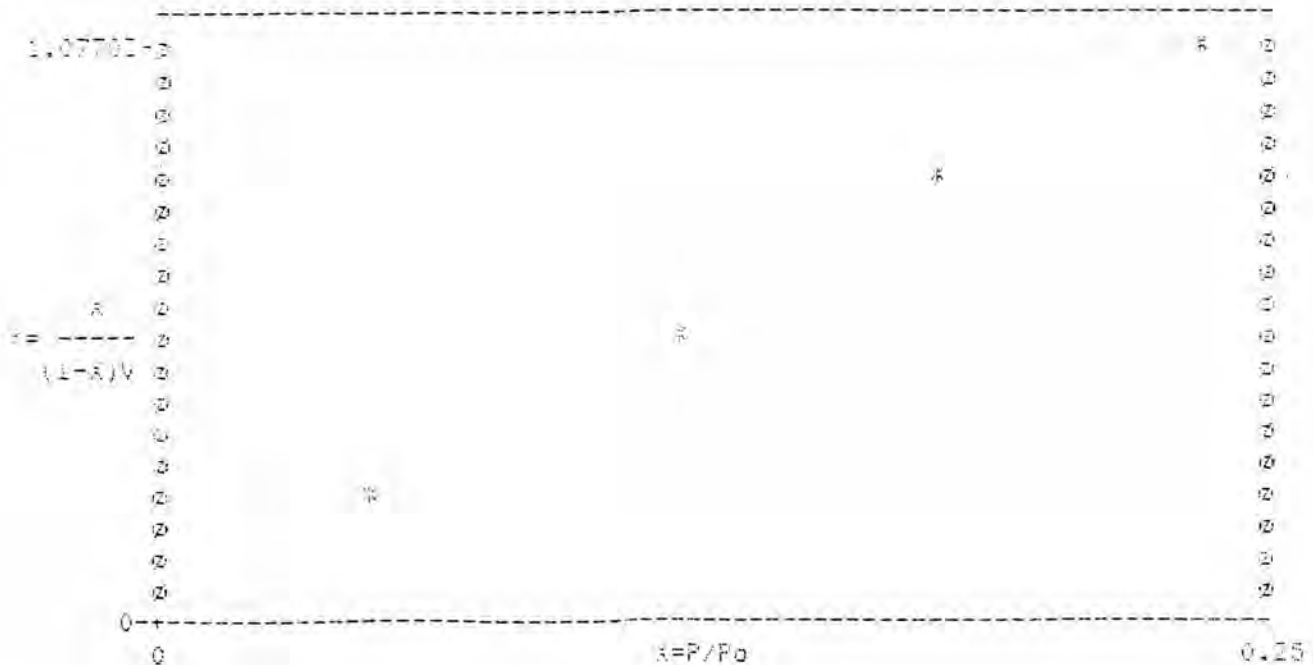
MICROMERITICS INSTRUMENT CORPORATION
 Flowsorb 2300

BET SURFACE AREA ANALYSIS
 REPORT DATE: 04/17/97

SAMPLE I.D.: TTCP (wet 3 hrs.)	ADSORBATE: Nitrogen
SAMPLE WEIGHT: 0.4199 g	BAROMETRIC PRESSURE: 760 mmHg
MOL. CROSS-SECTIONAL AREA: 0.162 nm ²	SATURATION PRESSURE: 775 mmHg
AMBIENT TEMPERATURE: 0.00 C	

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/P ₀	(X/R(1-X))VA
5.000	0.10	0.24	0.0490	0.21650
12.000	0.10	0.24	0.1177	0.36003
18.000	0.11	0.26	0.1765	0.31824
24.000	0.12	0.28	0.2364	1.07703

BET SURFACE AREA: 0.75 +/- 0.02 m²/g
 SLOPE: 4.5027 +/- 0.1115
 INTERCEPT: 0.0022 +/- 0.0178
 C: 2101.85
 V_m: 0.22 cm³/g
 CORRELATION COEFFICIENT 0.9994



Appendix D.
Specific Surface Area of wet grinded TTCP for 5 h.

MICROMERITICS INSTRUMENT CORPORATION
 FlowSorb 2300

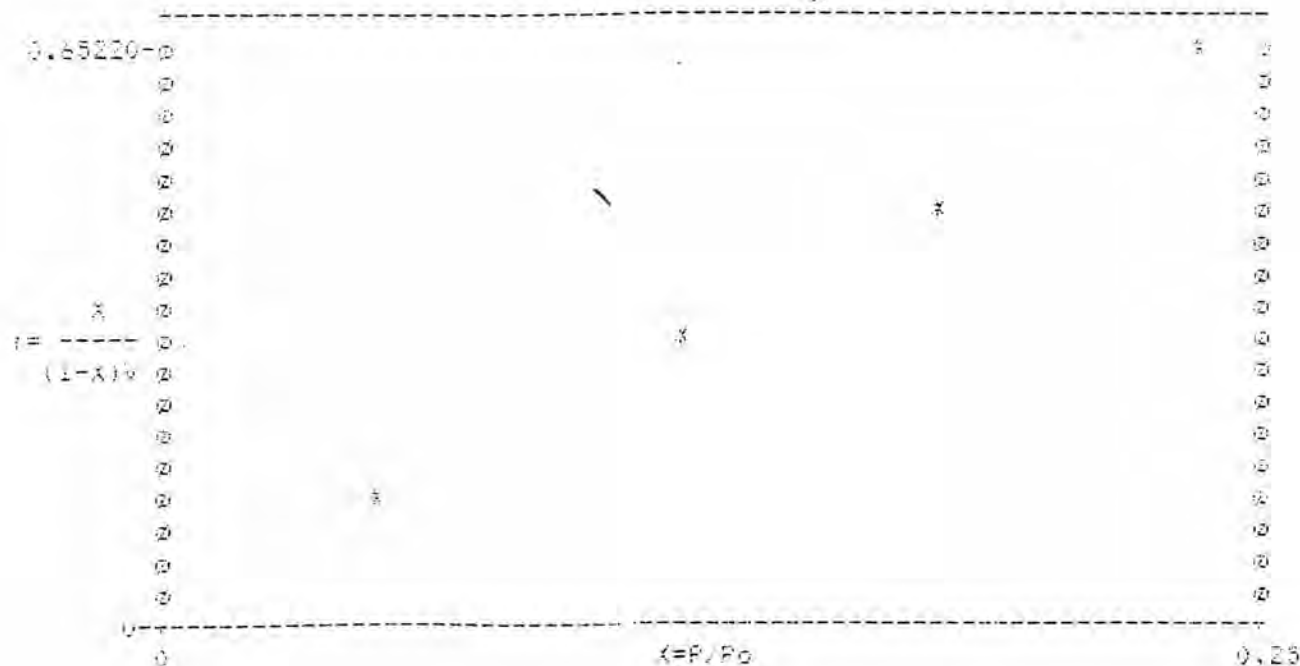
BET SURFACE AREA ANALYSIS
 REPORT DATE: 02/10/97

SAMPLE I.D.: TTCP (wet)
 SAMPLE WEIGHT: 0.3933 g
 MOL. CROSS-SECTIONAL AREA: 0.162 nm²
 AMBIENT TEMPERATURE: 0.00 C

ADSORBATE: Nitrogen
 BAROMETRIC PRESSURE: 760 mmHg
 SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/P ₀	Y=X/B(1-X)VA
5.000	0.19	0.32	0.0490	0.16100
11.000	0.24	0.40	0.1177	0.32971
18.000	0.26	0.44	0.1765	0.48914
24.000	0.28	0.47	0.2354	0.65220

BET SURFACE AREA: 1.63 +/- 0.03 m²/g
 SLOPE: 2.6403 +/- 0.0659
 INTERCEPT: 0.0261 +/- 0.0066
 C: 102.14
 V_m: 0.38 cm³/g
 CORRELATION COEFFICIENT 0.9996



Appendix E.

Specific Surface Area of wet grinded TTCP for 7 h.

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300

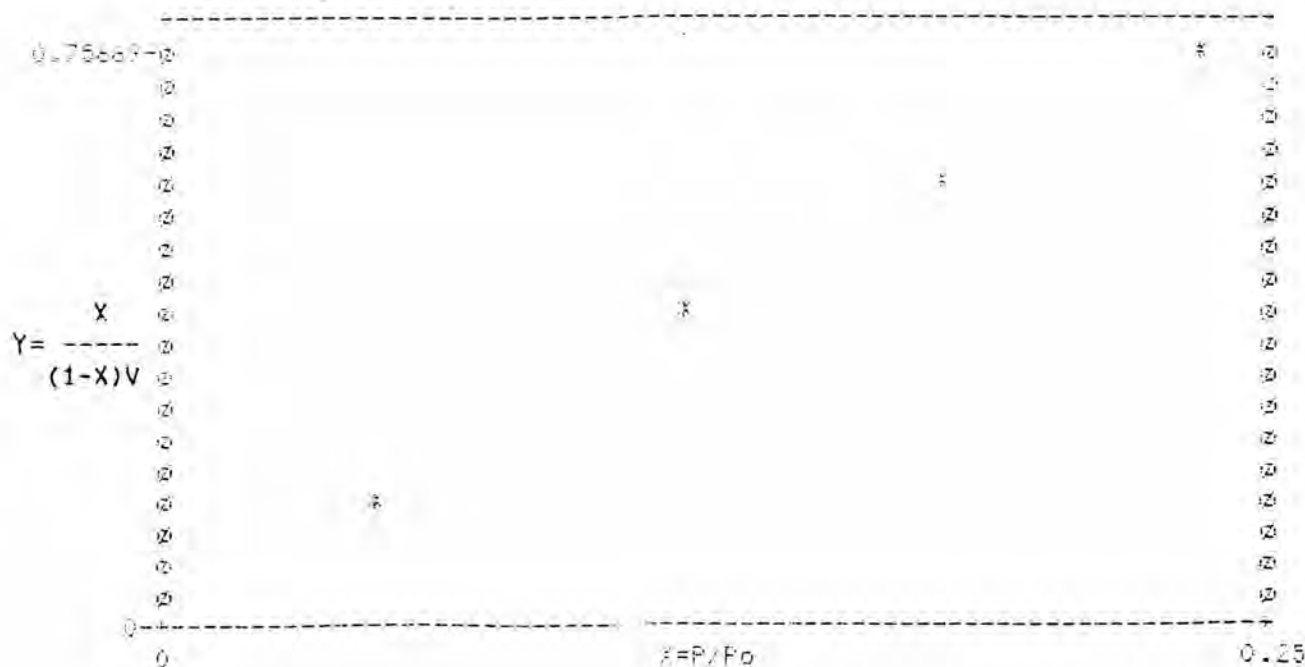
BET SURFACE AREA ANALYSIS
REPORT DATE: 04/17/97

SAMPLE I.D.: TTCP
SAMPLE WEIGHT: 0.4671 g
MOL. CROSS-SECTIONAL AREA: 0.162 nm²
AMBIENT TEMPERATURE: 0.00 C

ADSORBATE: Nitrogen
BAROMETRIC PRESSURE: 760 mmHg
SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	x=P/Po	y=x/R(1-x)V _s
5.000	0.13	0.28	0.0490	0.18525
13.000	0.15	0.32	0.1177	0.41532
18.000	0.17	0.36	0.1765	0.58897
24.000	0.19	0.41	0.2384	0.73669

BET SURFACE AREA: 1.40 +/- 0.04 m²/g
SLOPE: 3.0606 +/- 0.0680
INTERCEPT: 0.0439 +/- 0.0138
C: 70.77
V_m: 0.32 cm³/g
CORRELATION COEFFICIENT 0.9992



Appendix F.

Specific Surface Area of wet grinded TTCP for 8 h.

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300

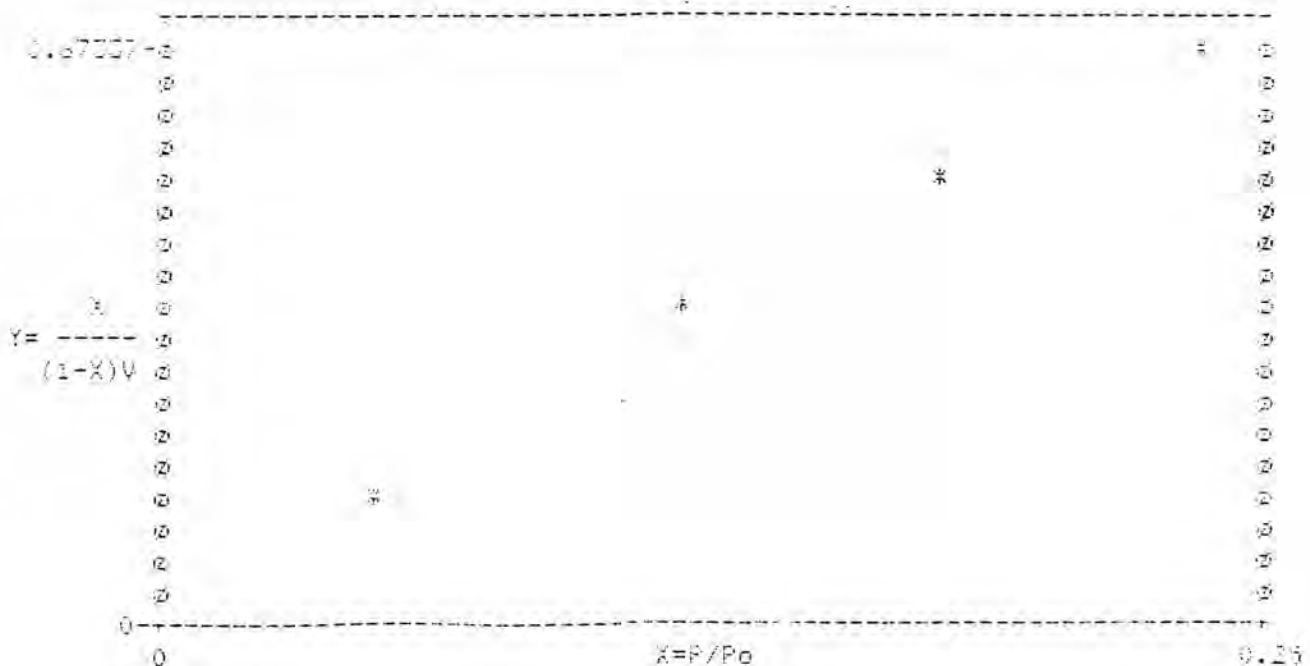
BET SURFACE AREA ANALYSIS
REPORT DATE: 04/17/97

SAMPLE I.D.: TTCP (wet 8 hrs.)
SAMPLE WEIGHT: 0.4813 g
MDL. CROSS-SECTIONAL AREA: 0.163 nm²
AMBIENT TEMPERATURE: 0.00 C

ADSORBATE: Nitrogen
BAROMETRIC PRESSURE: 750 mmHg
SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/Po	Y=X/(1-X)V
8.000	0.17	0.35	0.0490	0.14598
12.000	0.18	0.37	0.1177	0.35582
18.000	0.20	0.42	0.1765	0.51582
24.000	0.22	0.46	0.2354	0.67557

BET SURFACE AREA: 1.53 +/- 0.04 m²/g
SLOPE: 2.8225 +/- 0.0698
INTERCEPT: 0.0147 +/- 0.0112
D: 193.17
Vm: 0.35 cm³/g
CORRELATION COEFFICIENT 0.9994



Appendix G.

Specific Surface Area of α -TCP.

MILWAUKEE STEEL INDUSTRIES CORPORATION
FlowSorb 2200

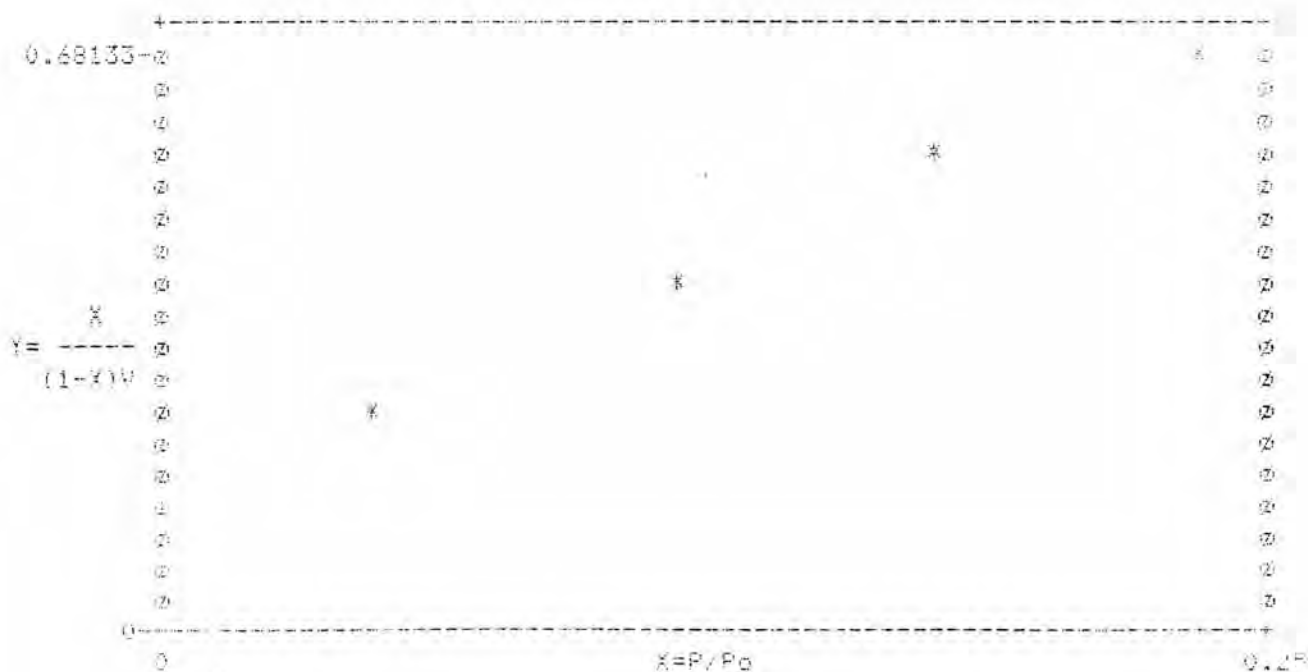
BET SURFACE AREA ANALYSIS
REPORT DATE: 06/17/97

SAMPLE I.D.: α TCP
SAMPLE WEIGHT: 0.3099 g
NOM. CROSS-SECTIONAL AREA: 0.162 cm²
AMBIENT TEMPERATURE: 0.00 C

ADSORBATE: Nitrogen
BAROMETRIC PRESSURE: 760 mmHg
SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL P/P_0 (%)	V/V_0	VOL ADSORBED (cm ³ /g AT STP)	$X=P/P_0$	$Y=X/V(1-X)^2$
8.000	0.08	0.19	0.0490	0.26631
12.000	0.10	0.32	0.1177	0.41332
18.000	0.12	0.39	0.1765	0.55357
24.000	0.14	0.45	0.2354	0.68133

BET SURFACE AREA: 4.32 +/- 0.02 m²/g
SLOPE: 1.2412 +/- 0.0325
INTERCEPT: -1.644 +/- 0.0052
C: 11.51
V_m: 0.42 cm³/g
CORRELATION COEFFICIENT 0.9998



Appendix H. Specific Surface Area of HA.

MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300

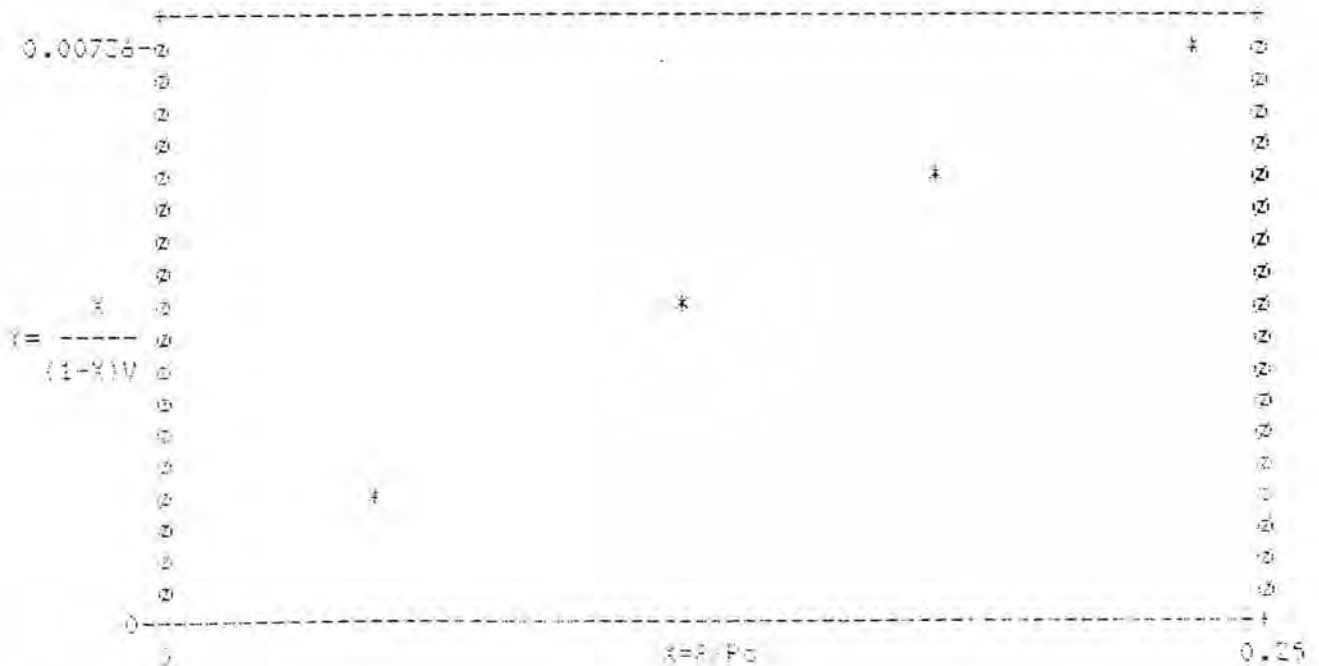
BET SURFACE AREA ANALYSIS
REPORT DATE: 5/30/97

SAMPLE I.D.: HA
 SAMPLE WEIGHT: 0.3256 g
 MOL. CROSS-SECTIONAL AREA: 0.162 nm²
 AMBIENT TEMPERATURE: 0.00 C

ADSORBATE: Nitrogen
 BAROMETRIC PRESSURE: 760 mmHg
 SATURATION PRESSURE: 775 mmHg

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/P ₀	Y=X/R(1-X)V _m
5.000	10.63	32.65	0.0490	0.00158
12.000	11.05	33.94	0.1177	0.00393
13.000	12.38	38.02	0.1725	0.00564
24.000	13.81	41.80	0.2354	0.00738

BET SURFACE AREA: 140.15 +/- 4.29 m²/g
 SLOPE: 0.0309 +/- 0.0009
 INTERCEPT: 0.0002 +/- 0.0001
 D: 196.91
 V_m: 32.20 cm³/g
 CORRELATION COEFFICIENT 0.9991



Appendix I.

Specific Surface Area of CHA.

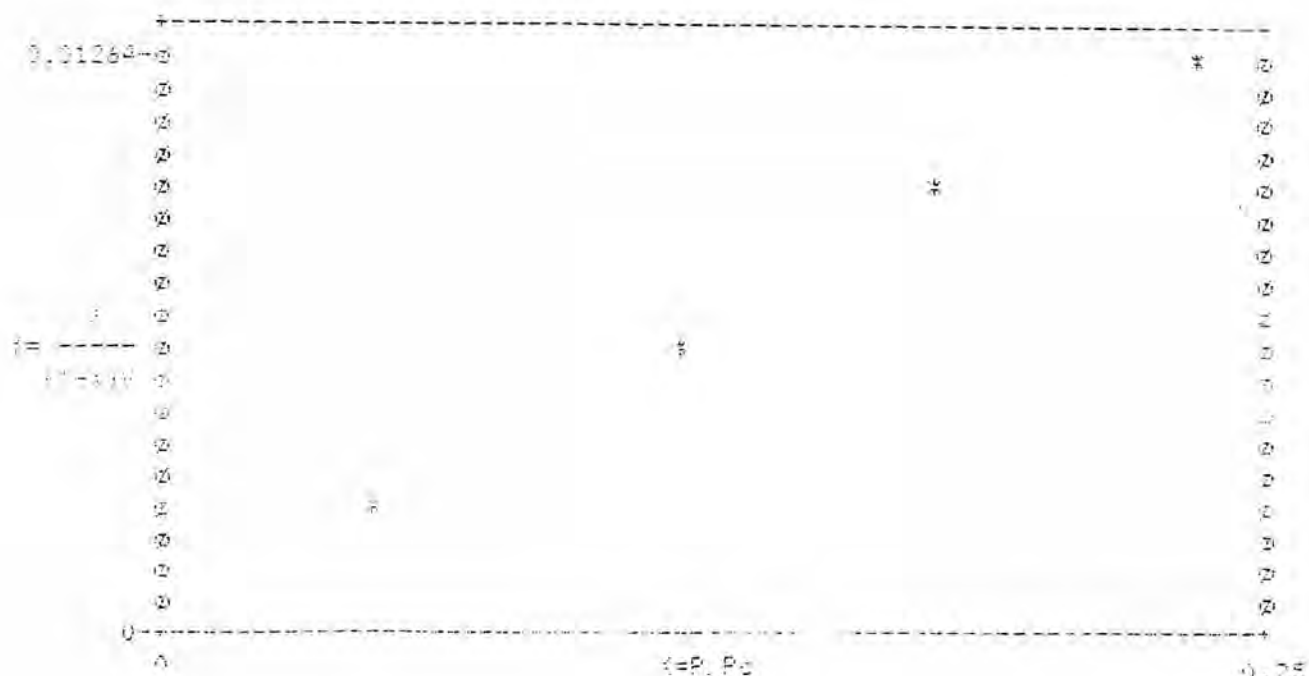
MICROMERITICS INSTRUMENT CORPORATION
FlowSorb 2300

BET SURFACE AREA ANALYSIS
REPORT DATE: 6/4/97

SAMPLE I.D.: CHA	ADSORBATE: Nitrogen
SAMPLE WEIGHT: 0.1014 g	BAROMETRIC PRESSURE: 750 mmHg
MDL. CROSS-SECTIONAL AREA: 0.162 nm ²	SATURATION PRESSURE: 775 mmHg
AMBIENT TEMPERATURE: 0.00 C	

EXPERIMENTAL DATA (%)	(VOL)	VOL ADSORBED (cm ³ /g AT STP)	X=P/P ₀	Y=X/g(1-X)V _g
5.000	1.76	17.36	0.0490	0.00297
12.000	2.08	20.51	0.1177	0.00650
18.000	2.27	22.39	0.1755	0.00958
24.000	2.47	24.36	0.2334	0.01264

BET SURFACE AREA: 83.21 +/- 0.19 m²/g
 SLOPE: 0.0519 +/- 0.0001
 INTERCEPT: 0.0004 +/- 0.0000
 C: 126.49
 V_m: 19.12 cm³/g
 CORRELATION COEFFICIENT 1.0000



Appendix J.

X-ray diffraction card of DCPD.

9-77 MAJOR CORRECTION

d	7.57	4.24	3.05	7.57						
					<chem>CaHPO4 · 2H2O</chem>					★
I/I_1	100	100	75	100	CALCIUM HYDROGEN ORTHOPHOSPHATE HYDRATE					(BAURHITE)
Rad. CuK α_1	λ 1.5405	Filter		Dia. 114.6mm	d Å	I/I $_1$	hkl	d Å	I/I $_1$	hkl
Cut off	50	1/1, PHOTOMETER		(GUINIER CAMERA)	7.57	100	020	2.252	2	240
Ref.	DEVOLFF, TECHN. PHYS. DIENST, DELFT, HOLLAND				4.93	2	11 $\bar{1}$	2.172	20	151
					4.24	100	021	2.140	16	24 $\bar{2}$
					3.00	8	040	2.120	2	042
					3.75	< 1	130	2.100	6	15 $\bar{2}$
					3.63	2	13 $\bar{1}$	2.084	10	31 $\bar{1}$
					3.05	75	111, 041	2.022	4	170, 31 $\bar{2}$
					2.928	50	22 $\bar{1}$	2.001	10	221, 17 $\bar{1}$
					2.855	10	11 $\bar{2}$	1.976	6	26 $\bar{1}$, 11 $\bar{2}$
					2.797	2	200	1.943	2	33 $\bar{1}$
					2.670	4	150	1.899	2	080
					2.648	4	131	1.888	4	11 $\bar{3}$
					2.623	50	220, 15 $\bar{1}$	1.878	14	260
					2.603	30	20 $\bar{2}$	1.858	8	22 $\bar{3}$
					2.554	4	002	1.855	< 1	13 $\bar{2}$
					2.532	2	060	1.819	20	241
					2.520	4	13 $\bar{2}$	1.799	10	062
					2.434	14	24 $\bar{2}$	1.780	4	081, 171+
					2.421	16	022	1.748	2	330
					2.269	4	061			
										PLUS ADDITIONAL LINES
Sys. MONOCLINIC S.G. C2 (5) a 6.363 b 15.19 c 5.815 β 0.4187 γ 0.3828 α β 118.48° γ Z 4 D_x 2.32 Ref. <i>IBID.</i>					Ref. SALE, BONNER, HODGE, <i>IND. ENG. CHEM., ANAL. ED., 17</i> 491 (1945)					
BEEVERS ACTA CRYST <u>11</u> 273-277 (1958) GIVES $a = 5.812$, $b = 15.180$, $c = 5.217$, $\beta = 116.25$ S.G. $C2/a$ $A = 6.359$, $b = 15.180$, $c = 5.208$, $\beta = 118.31$ S.G. $C2/c$ IN THE SETTING USED HERE.										

Appendix K.
X-ray diffraction card of DCPA.

9-80 MAJOR CORRECTION

d	2.96	3.35	3.37	6.74	CaHPO ₄			★		
I/I ₁	100	75	70	14	CALCIUM HYDROGEN PHOSPHATE			(MONETITE)		
Rad. CuKα, λ 1.5405 Filter Dia. 114.6mm (GUINIER CAMERA)					d Å	I/I ₁	hkl	d Å	I/I ₁	hkl
Cut off 50 I/I ₁ PHOTOMETER					6.74	14	010, 110	2.857	4	111
Ref. DEWOLFF, TECHN. PHYS. DIENST, DELFT, HOLLAND					5.43	4	100	2.843	2	012
Svs. TRICLINIC S.G. P1̄ (2)					4.99	4	011	2.763	10	110
a 6.906 b 8.577 c 6.534 AO.3051 CO.7734					4.48	2	011	2.754	20	210
α 93.90° β 91.50° γ 127.63° Z 4 Dx 2.92					4.38	4	101	2.721	35B	200, 100
Ref. MacLENNAN AND BEEVERS, ACTA CRYST. 8 579 (1955)					4.27	4	110	2.581	4	211
EXCEPT CONSTANTS					4.02	4	101	2.532	4	111
ε 1.60 n _w 1.51 η γ 1.63 Sign					3.70	4	111	2.539	16B	211, 021
2V D 2.9 mp Color					3.48	14	111	2.489	2	112
Ref. BALE, DONNER, HODGE, IND. ENG. CHEM., ANAL. ED., 17					3.37	70	020	2.443	2	201
191 (1948)					3.35	75	110, 210	2.411	2	211
DEHYDRATED CaHPO ₄ ·2H ₂ O					3.33	16	210	2.351	4	311, 211
					3.30	10	002	2.305	10	120
					3.13	20	111	2.287	2	310, 211
					3.10	6	011	2.251	16	010
					2.986	2	111, 111	2.242	6	022
					2.958	100	111, 111	2.234	10	310, 111
					2.937	35	101	2.216	4	112
					2.912	4	211	2.199	12	002
					2.881	3	021	2.181	12	002
					SEE FOLLOWING CARD					

Appendix L.
X-ray diffraction card of TTCP.

Calcium Oxide Phosphate ($\text{Ca}_4\text{P}_2\text{O}_9$, TTCP)

d ($^{\circ}\text{A}$)	I/I ₁	hkl	d ($^{\circ}\text{A}$)	I/I ₁	hkl
6.06	4	110	3.053	80	032,013
5.99	5	020	2.995	100	040
5.69	4	101	2.895	30	103,221
4.739	3	002	2.872	45	221
4.558	5	120	2.811	25	113,132
4.405	2	012	2.798	35	023,132
4.122	14	121	2.784	35	113
4.092	20	121	2.763	50	212
3.595	2	102	2.624	40	212
3.897	3	102	2.649	14	141
3.754	4	112	2.642	18	141
3.711	8	022,112	2.607	12	123
3.684	10	031	2.538	6	123
3.511	45	200	2.567	5	222
3.473	20	130	2.545	9	231
3.366	3	210	2.534	16	222,231
3.302	7	201,122	2.476	16	033
3.268	16	131,122	2.387	2	142
3.190	30	211	2.367	7	044,203
3.160	20	211,003	2.322	15	051,014

Appendix M.
X-ray diffraction card of γ -Ca₂P₂O₇
and β -Ca₂P₂O₇

β -Ca ₂ P ₂ O ₇		γ -Ca ₂ P ₂ O ₇	
d (°A)	I	d (°A)	I
4.72	vw	4.47	w
4.43	vw	3.33	s
3.34	s	3.05	vs
3.22	s	2.01	vs
3.09	m	2.67	m
3.01	vs	2.53	m
2.90	m	2.38	m
2.80	m	2.21	s
2.75	m	2.16	m
2.68	w	2.08	m
2.54	m	1.94	vw
2.40	vw	1.85	m
2.33	m	1.80	m
2.27	vw	1.77	m
2.22	m	1.65	m
1.99	m	1.56	m
1.85	w	1.50	m
1.83	w	1.46	m
1.77	m	1.30	m
1.31	m		

Appendix N.
X-ray diffraction card of α -TCP.

9-348 MAJOR CORRECTION

d	2.91	2.62	3.91	12.3	α -Ca ₃ (PO ₄) ₂ ALPHA CALCIUM ORTHOPHOSPHATE	★			
I/I ₁	100	50	40	4					
Rad. CuK α_1	λ 1.5405	Filter	Dis. 114.6mm	d Å	I/I ₁	hkl	d Å	I/I ₁	hkl
Cut off 50	I/I ₁ PHOTOMETER	(GUINIER CAMERA)		12.3	4	110	3.35	8	312
Ref. DEWOLFF, TECHN. PHYS. DIENST, DELFT, HOLLAND				7.31	25	111	3.33	4	421
				6.82	4	021	3.15	4	260
				6.29	10	130	3.12	4	242
				6.12	4	220	3.07	4	440
Sys. ORTHORHOMBIC ^a	S.G.			5.83	10	201	3.05	4	332
a 15.22	b 20.71	c 9.109	A 0.7349 C 0.4398	5.18	12	131,040	3.01	20	510
α	β	γ	Z 16 Dx 2.87	4.55	4	002	2.947	20	113
Ref. IBID.				4.33	4	311	2.919	35	402,023
				4.28	2	240,112	2.905	100	441,170
ϵ a	n w β	J y	Sign	4.17	2	022	2.860	30	511
2V	D 2.814	mp 1720°C	Color	4.00	20	150	2.816	2	203,422
Ref. WACKAY (SEE BELOW)				3.91	40	202	2.786	12	530
				3.88	40	241	2.767	4	171
				3.81	8	400	2.734	< 1	133
				3.73	4	331	2.720	< 1	223
				3.69	40	132	2.665	4	531
				3.66	18	151,222	2.621	50	043,152
				3.51	4	401	2.590	30	080
				3.45	6	060	PLUS ADDITIONAL LINES		

* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY WACKAY,
ACTA CRYST. 2 743 (1953)

SAMPLE OBTAINED BY HEATING β -PHASE AT 1400°C.

Appendix O.
X-ray diffraction card of β -TCP.

9-169 a MAJOR CORRECTION

d	2.38	2.61	3.21	8.15	β -Ca ₃ (PO ₄) ₂ CALCIUM PHOSPHATE (WHITLOCKITE)	★			
I/I ₁	100	65	55	12					
Rad.	λ	Filter	Dis.	d Å	I/I ₁	hkl	d Å	I/I ₁	hkl
Cut off				2.000	3	2.2.12	1.600	4	
Ref.	I/I ₁			1.970	2	410	1.552	12	
				1.946	4	413	1.532	4	
				1.933	20	0.10, 327	1.520	4	
Sys.		S.G.		1.895	16	238	1.505	4	
a ₀	b ₀	c ₀	A	1.879	14	416	1.465	6	
α	β	γ	Z	1.830	12	(0.1.20)	1.440	4	
Ref.			Dx			(3.1.14)	1.429	4	
				1.812	6	3.2.10	1.414	4	
ξ a	n w β	ϵ γ	Sign	1.793	6	502	1.409	6	
2 ν	D	mp	Color	1.781	6	419	1.337	4	
Ref.				1.774	3	054			
				1.753	4	330			
				1.722	25	2.0.20			
				1.711	3				
				1.655	2				
				1.645	4				
				1.637	6				
				1.625	6				
				1.603	6				

SEE PRECEDING CARD

Appendix P.

X-ray diffraction card of HA.

9-432 MAJOR CORRECTION

d	2.81	2.78	2.72	2.17						
					$\text{Ca}_5(\text{PO}_4)_3(\text{OH})$	$\frac{1}{2}(\text{Ca}(\text{OH})_2 \cdot 3\text{Ca}_3(\text{PO}_4)_2)$	★			
I/I ₁	100	60	40	11	CALCIUM PHOSPHATE HYDROXIDE		(HYDROXYL-APATITE)			
Rad. CuK α_1 λ 1.5405 Filter D ₁₆ 114.6mm Cu α_2 50 MI. PHOTOMETER* (GUINIER CAMERA) Ref. DEBOUFF, TECHN. PHYS. DIENST, DELFT, HOLLAND					d Å	I/I ₁	hkl	d Å	I/I ₁	hkl
Syst. HEXAGONAL S.G. P ₆ /m (176) a = 9.418 b _c = 3.884 A C 0.7309 c = 3.884 Z 2 Dx 3.16 Ref. 1810.					5.17	12	100	2.040	2	400
					5.26	8	101	2.000	6	203
					4.72	4	110	1.943	20	222
					4.07	10	200	1.890	16	212
					3.88	10	111	1.871	6	220
					3.51	2	201	1.841	40	213
					3.44	40	002	1.806	20	221
					3.17	12	102	1.780	12	410
					2.08	13	210	1.754	16	402, 303
					2.814	100	211	1.722	20	004, 411
Fe λ 1.936 Filter λ 1.936 Cu α_1 50 MI. PHOTOMETER* (GUINIER CAMERA) Ref. DEBOUFF, TECHN. PHYS. DIENST, DELFT, HOLLAND					2.778	40	112	1.684	4	104
					2.720	40	100	1.644	10	122, 223
					2.631	25	202	1.611	3	212
					2.528	6	301	1.587	4	501, 204
					2.296	8	212	1.542	6	420
					2.262	10	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
					2.065	4	113	1.435	4	510

* — 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).

Appendix Q.
X-ray diffraction card of CaO

1. Calcium oxide (CaO)

Crystal form	Cubes	
Unit cell	Cubic a 4.8105 Å	
Space group	Z	D
$Fm\bar{3}m$	4	3.345
Refractive indices	n 1.837	
Other optical properties	Isotropic	
Main reference [1].		

X-RAY POWDER DATA [1]

d	I	hkl	d	I	hkl
2.778	ms	111	1.203	vw	400
2.405	vvs	200	1.1036	vw	331
1.701	s	220	1.0755	w	420
1.451	w	311	0.9819	w	422
1.390	vw	222	0.9258	vw	511
			0.8504	vw	440
			0.8131	vw	531
			0.8018	vw	600

Appendix R.
X-ray diffraction card of Ca(OH)₂

4. Calcium hydroxide (Ca(OH)₂)

Crystal form	Hexagonal flakes, (0001) cleavage	
Unit cell	Trigonal a 3.593 c 4.909 Å	
Space group	Z	D
$P\bar{3}m1$	1	2.241
Refractive indices	ω 1.573 ϵ 1.545	
Other optical properties	Uniaxial -	
Main references [5].		

X-RAY POWDER DATA [5]

d	I	hkl	d	I	hkl
4.90	vs	0001	1.1432	w	21.1, 10.4
3.3112	m	10.0	1.1275	vw	20.3
2.628	vvs	10.1	1.0599	w	21.2
2.447	vw	0002	1.0366	vw	30.0
1.927	ms	10.2	1.0143	vw	30.1, 11.4
1.796	ms	11.0	0.9551	vw	30.2, 21.3
1.687	m	11.1	0.9369	vw	10.5
1.634	vw	0003	0.8979	vw	22.0
1.557	vw	20.0	0.8838	vw	22.1
1.484	w	20.1	0.8760	vw	30.3
1.449	w	11.2, 10.3	0.8623	vw	31.0, 11.5, 22.2
1.314	vw	20.2	0.8495	vw	31.1, 21.4
1.228	vw	0004	0.8140	vw	0006
1.211	vw	11.3			
1.1762	vw	21.0			

JIS

JAPANESE INDUSTRIAL STANDARD

Dental Zinc Phosphate Cement

☉ JIS T 6602-1985



Translated and Published

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JAPANESE INDUSTRIAL STANDARD

J I S

Dental Zinc Phosphate Cement

T 6602-1985

1. Scope

This Japanese Industrial Standard specifies the dental zinc phosphate cement, hereinafter referred to as the "cement", excluding that for temporary sealing.

Remark: The units and numerical values given in { } in this standard are in accordance with the International System of Units (SI), and are appended for reference.

2. Qualities

The qualities of the cement shall be in accordance with the following:

- (1) The cement consists of powder material and liquid agent. The powder material, of which principal component shall be zinc oxide, and the liquid agent, of which principal component shall be phosphate solution. The powder material shall be uniform in quality and be free from impurities, and the liquid agent shall be colourless and transparent, and neither precipitate nor deposit shall be found.
- (2) The cement shall neither generate gas nor produce undissolved powder lump in kneading.
- (3) The cement, in the case where it has been filled or fitted up, shall not colour the texture of the tooth.
- (4) The cement shall comply with the requirements of Table 1.



Applicable Standard:

JIS Z 8801-Test Sieves

Corresponding International Standard:

ISO 1566-1978 Dental zinc phosphate cement

Table 1. Qualities

Item	Quality	Testing method
Setting time min	4 or over to 8 incl.	3. (3)
Crushing resistant strength MPa ⁽¹⁾ {kgf/cm ² }	68.5 {700} min.	3. (4)
Thickness of film mm	0.03 max.	3. (5)
Collapse rate %	0.2 max.	3. (6)
	No crystal shall be found.	
Arsenic ppm	2 max.	3. (7)

Note ⁽¹⁾ 1 MPa = 1 N/mm²

3. Testing Methods

The tests shall be in accordance with the following.

Furthermore, the tests shall all be carried out in a room of temperature at 23 ± 2°C and relative humidity at 50 ± 10 %.

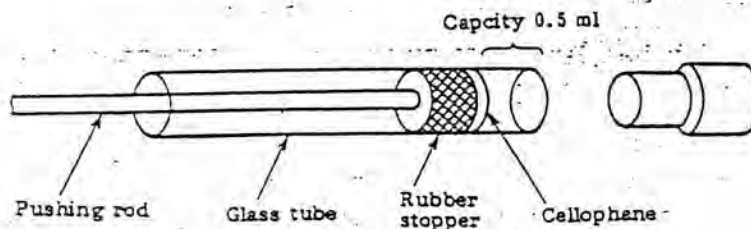
- (1) Kneading Method at the Time of Manufacturing Test Pieces The kneading method in the case of manufacturing test pieces, if it has been designated by the manufacturer, shall be in accordance with the designation. If no designation has been made, take the liquid agent and powder material of equivalent quantity to the standard consistency to be obtained in (2), and knead the split quantity of powder material with the total quantity of the liquid agent. The splitting rate of the powder material, kneading time and order shall be as given in Table 2.

Table 2. Splitting Rate of Powder Material and Kneading Time

Splitting rate of powder material	Kneading time s	Kneading order
1/6	15	↓
1/6	15	
1/3	30	
1/3	30	

- (2) Standard Consistency at the Time of Manufacturing Test Pieces Take 0.5 ml of the liquid agent on a kneading plate, and measure 0.5 ml of cement which has been kneaded with the specified method by adding an appropriate quantity of the powder material, by using the instrument shown in Fig. 1. Next, push out quietly on a glass plate, put another glass plate (approximately 20 g in mass) on this when 3 min has lapsed after the commencement of kneading, place a weight (approximately 100 g in mass) quietly on it so that the total of these becomes 120 ± 0.5 g, remove the weight and upper glass plate when 10 min has lapsed after the commencement of kneading and measure the dimensions of the maximum and the minimum parts between the parallel tangents of the spreaded sample. When the mean value of these has become 29 to 31 mm, consider this to be the standard consistency, and consider the quantity of the powder material in this case as the quantity of powder material to be kneaded with the liquid agent of 0.5 ml.

Fig. 1. Outline Figure for Measuring Device of Sample



- (3) Setting Test Place the acid resistant mould of Fig. 2 on the glass plate of Fig. 3, fill this with the cement of standard consistency, flatten the surface, and when 3 min has lapsed after the commencement of kneading, transfer this into the thermostatic vessel of temperature at 37°C and relative humidity at 100%, drop the Vickers needle (sectional area of needle 1 mm^2) of 300 g in mass quietly on the surface of the test piece, and examine whether any mark of the needle could cause or not. Next, repeat the dropping operations of the Vickers needle quietly on the surface of the test piece until no needle mark can be caused, and consider the time when no needle mark has been left, calculating from the commencement time of kneading, to be the setting time.

Fig. 2. Mould

Unit: mm

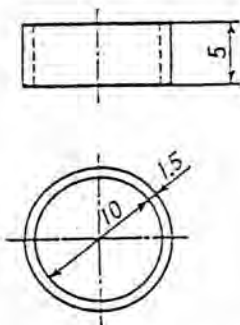
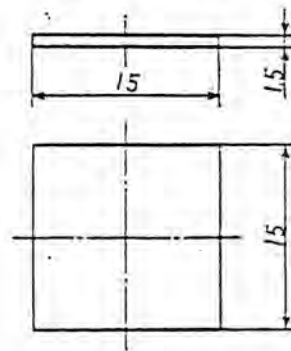


Fig. 3. Glass Plate

Unit: mm

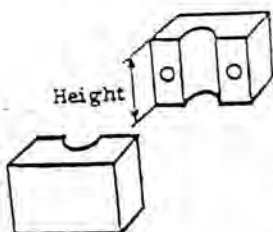


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(4) Crushing Resistant Strength Test. The crushing resistant strength test shall be carried out in accordance with the order of the following respective numbers:

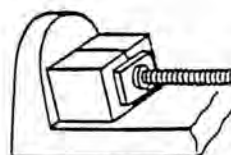
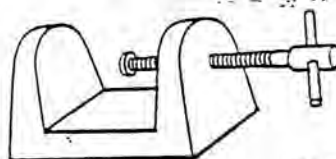
- (a) Place the acid resistant mould of Fig. 4 on an acid resistant plate, and put the cement of standard consistency in this mould.
- (b) Apply another acid resistant plate to it, and force out an excess amount of cement using the pressurizing device of Fig. 5.
- (c) When 3 min has lapsed from the commencement of kneading, transfer this into the thermostatic vessel, as it stands being pressurized, of temperature at 37°C and relative humidity at 100 %.
- (d) When 30 min has lapsed after the commencement of kneading, take out the test piece from the mould, finish the both end faces of the test piece at right angles with the axis and flat, if required, and put into the distilled water at 37°C.
- (e) When 24 h has lapsed after the commencement of kneading, take this out from the distilled water, and carry out the compression test.
- (f) Place the test piece at the centre of the compressing plate of the compression tester and apply pressure gradually, and measure the load when the test piece has been crushed.
- (g) Carry out measurements on the 10 test pieces and consider the mean value of the numerical values, excluding the numerical values of -15 % or under the mean value of the total results, to be the crushing resistant strength. However, when the numerical values of -15 % or under the mean value of the total results are four pieces or over, the retest shall be carried out.

Fig. 4. Mould



Inside diameter of mould: 6 mm
Height of mould: 12 mm

Fig. 5. Outline Figure of Pressurizing Device



- (5) Film Test Interpose a small quantity of cement of the standard consistency between the two pieces of glass plates of approximately 2 cm^2 in area and uniform thickness of 1.5 mm, and when 3 min has lapsed after the commencement of kneading, apply the constant pressure of 147.1 N {15 kgf} to this, and then measure the thickness of the two pieces of glass plate, as interposing the cement, using a measuring instrument having an accuracy of 1/1000 mm, and obtain the difference between this and the thickness of glass plates not interposing the cement.
- (6) Collapse Rate Test The collapse rate test shall be carried out in accordance with the order of the following respective numbers.
- (a) Measure two times the cement of 0.5 ml of the standard consistency, using the instrument given in Fig. 1, and put on a glass plate.
 - (b) Insert the acid resistant fine wires of an appropriate length of known mass into these two test pieces.
 - (c) Lightly press to adhere these by another glass plate to make the diameter of the cement up to approximately 20 mm, and when 3 min has lapsed after the commencement of kneading, transfer into the thermostatic vessel at a temperature of 37°C and at a relative humidity of 100 %.
 - (d) When 1 h has lapsed after the commencement of kneading, peel off the 2 test pieces from the glass plate, immediately put these into the glass bottle with ground stopper, approximately 100 ml in inside capacity, of known mass and apply a stopper, and weigh.
 - (e) Obtain the difference between this mass, the total mass of the bottle and fine wires, and consider this to be the mass of the test pieces.
 - (f) Pour 50 ml of the distilled water into this, suspend the two test pieces with the fine wires in the water, apply the stopper lightly, and hold for 24 h in the thermostatic vessel of temperature at 37°C .
 - (g) Take out the test pieces from the bottle, and examine whether any crystal could have deposited on the surfaces or not.
 - (h) Allow to evaporate the glass bottle by heating on a water bath, and further allow to dry in the thermostatic vessel of temperature at 150°C until the change in mass of the bottle becomes 0.5 mg or under per 24 h.
 - (i) Next, weigh the bottle after this has been cooled in a desiccator.
 - (j) Obtain the mass of evaporation residue by subtracting the mass of the original glass bottle from this value, obtain the percentage of the test pieces to the original mass, and consider this to be the collapse rate.



- (7) Arsenic Content Test After setting, pulverize the cement which has lapsed 24 h in a dry and tightly-closed vessel to the powders which pass through the sieve of 74 μm (200 mesh) specified in JIS Z 8801, weigh 1.0 g of this accurately, add 100 ml of the distilled water, filter after this has been heated for 1 h on a water bath, wash with not more than 20 ml of the warm distilled water, and combine the washing with the filtrate. Consider the liquid that has been condensed to 20 to 15 ml on a water bath to be the test liquid, and carry out test in accordance with the first method of arsenic test method of the general test methods of the Japanese Pharmacopoeia.

4. Designation

The products shall be designated by the name.

Example: Dental zinc phosphate cement

5. Marking

The package of the cement shall be marked with the following information, and be annexed with necessary instructions in use.

- (1) Name
- (2) Distinction of powder material and liquid agent
- (3) Manufacturing number or manufacturing symbol
- (4) Manufacturer's name and address

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Standard Specification for Acrylic Bone Cement¹

This standard is issued under the fixed designation F 451; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers self-curing resins used primarily for the fixation of internal orthopedic prostheses. The mixture may be used in either the predough or dough stage in accordance with the manufacturer's recommendations.

1.2 Units of premeasured powder and liquid are supplied in a form suitable for mixing, which then sets in place.

1.3 While a variety of copolymers and comonomers may be incorporated, the composition of the set cement shall contain poly(methacrylic acid esters) as its main ingredient.

1.4 This specification covers compositional and physical performance and packaging requirements, but does not cover toxicity nor biocompatibility of the cement. The biocompatibility of acrylic bone cement has been reported in the literature (1).²

1.5 *This standard may involve hazardous materials, operations, and equipment. This standard does not purport to address all of 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 Documents

2.1 ASTM Standard:

D 3835 Test Method for Measuring the Rheological Properties of Thermoplastics with a Capillary Rheometer³

2.2 ANSI/ADA Standard:

No. 15 Specification for Acrylic Resin Teeth⁴

3. Definition

3.1 *unit*—one package or vial of premeasured powder component and one package or vial of premeasured liquid component.

4. Physical Requirements

4.1 Liquid:

4.1.1 *Appearance*—The liquid shall be free of extraneous particulate matter or obvious visual contaminants in its container.

4.1.2 *Stability*—The liquid shall not increase in original viscosity to such an extent that its flow time will be increased

by more than 10% when heated 48 h at $60 \pm 2^\circ\text{C}$ in the dark, in a closed container (7.3).

4.1.3 *Sterility*—The liquid, as poured from its container, shall pass the tests described in "Sterility Tests—Liquid and Ointments" (7.4) (2).

4.2 Powder:

4.2.1 *Appearance*—The powder shall be pourable and free of extraneous materials, such as dirt or lint (7.2.2).

4.2.2 *Sterility*—The powder, as poured from its package, shall pass the tests described in "Sterility Tests—Solids" (7.4) (2).

4.3 Powder-Liquid Mixture:

4.3.1 If the mixture is to be used in its predough stage, the material shall conform to the properties given in Table 1.

4.3.2 If the mixture is to be used in its dough stage, the material shall conform to the properties given in Table 1.

4.3.3 If the mixture can be used in either its predough or dough stages, separate units must be tested for compliance with 4.3.1 and 4.3.2.

4.4 *Cured Polymer*—The material after setting shall conform to the properties given in Table 2.

5. Weights and Permissible Variations

5.1 Weight and volume measurements shall be made on the respective powder and liquid components of five units (see 3.1). These units may be subsequently utilized in any of the nonsterile tests of this specification.

5.2 The weights, or volume of the powder and liquid components, or both, shall not deviate by more than 5% from those stated on the package (9.2.2), of each of five units.

5.3 Where a radiopaque material is supplied for addition to the powder at the discretion of the surgeon, the weight or volume percent of the radiopaque material shall not deviate by more than 15% from the value stated on the package (9.2.3).

6. Sampling

6.1 Units of powder and liquid shall be procured to provide sufficient material for all the tests of this specification. The units shall be obtained from regular retail distribution channels. Provided no repeat tests are required, this will amount to between seven and ten units.

6.2 It will only be necessary to maintain sterility in tests described in 7.4. All other tests described in this specification need not be conducted under sterile conditions.

7. Test Methods and Sample Size

7.1 Maintain all equipment, mixing surfaces, and materials at $23 \pm 2^\circ\text{C}$ at least 2 h prior to testing and conduct all tests at $23 \pm 2^\circ\text{C}$ and $50 \pm 10\%$ relative humidity, unless otherwise specified.

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 F04.02 on Resources.

Current edition approved July 25, 1986. Published September 1986. Originally published as F 451 - 76. Last previous edition F 451 - 76 ϵ 1.

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

³ Annual Book of ASTM Standards, Vol 08.03.

⁴ Available from American National Standards Institute, 1430 Broadway, New York, NY 10017.

7.2 *Inspection*—Use visual inspection in determining compliance to the requirements outlined in 4.1.1, 4.2.1, 8.1 and 8.2.

7.2.1 The liquid component of two separate units shall comply with the requirements of 4.1.1 and 8.1.

7.2.2 The powder component of two separate units shall comply with the requirements of 4.2.1 and 8.1.

7.3 *Liquid Component Viscosity*—Record the viscosity change of two separate units (4.1.2) before and after the heating exposure by timing the flow of the liquid level between the 0 and 5 mL marks of a 10 mL measuring pipet. Calculate the percent change as follows:

$$\% \text{ Change} = \frac{t_a - t_b}{t_b} \times 100$$

where:

t_b = flow time before heating, and

t_a = flow time after heating exposure (4.1.2) of $60 \pm 2^\circ\text{C}$ for 48 h in the dark in a closed container.

7.3.1 An alternative method for viscosity may be used if it can be demonstrated to yield similar results. Both shall comply to the less than 10 % change specified (4.1.2).

7.4 The components of the two units shall be tested for sterility in accordance with the test methods described in U.S. Pharmacopoeia, "Sterility Tests" (2).

7.5 *Doughing Time*

7.5.1 *Environment*—All equipment, mixing surfaces, and material (unit size) shall be maintained at $23 \pm 1^\circ\text{C}$ at least 2 h prior to testing and tests shall be conducted at $23 \pm 1^\circ\text{C}$. The relative humidity shall be $50 \pm 10\%$.

7.5.2 Mix all the powder and liquid of a single unit together as directed by the manufacturer's instructions (8.5.2). Start a stop watch at the onset of combining the liquid to the powder and read all subsequent times from this stop watch. Approximately 1.5 min after the onset of mixing, gently probe the mixture with a non-powdered surgically gloved (latex) finger. Take visual notice as to the formation of fibers between the surface of the mix and the finger as it leaves the surface. Repeat this process from that time on at 15-s intervals until the gloved finger separates cleanly. Denote the time at which this is first observed as the doughing time. Mix the mixture between determinations to expose fresh material for each probing.

7.5.3 Determine the doughing time from two separate units.

7.5.4 The two values found shall agree within 30 s of each other, otherwise repeat the test on two additional units. Report the average of all four tests and the range of values.

7.5.5 Report the doughing time to the nearest 15 s as the average of all determinations. Maximum and minimum values of doughing times measured shall not differ by more than $\pm 1\frac{1}{2}$ min from the average.

7.6 *Maximum Temperature*—Within 1 min after doughing time, gently pack approximately 25 g of the dough described in 7.5 into the mold described in Fig. 1. This mold shall be made of polytetrafluoroethylene (PTFE) and be equipped with a No. 24 gage wire thermocouple, or similar device, positioned with its junction in the center of the mold at a height of 3.0 mm in the internal cavity. Immediately seat the plunger with a C-clamp or suitable press to produce the 6.0 mm specimen height. Upon producing plunger seating, remove the excess material and the C-clamp or press for the remainder of the procedure. Continuously record the temperature with respect to time from the onset of mixing the liquid and the powder until cooling is observed. Fig. 2. Report the maximum temperature recorded to the nearest 1°C . This should not exceed the value given in Table 1.

7.6.1 The maximum temperature shall be the average of two separate determinations reported to the nearest 1°C .

7.6.2 If the difference between the maximum temperature for the two determinations is greater than 5.0°C , repeat the test on two additional units and report the average of all four runs to the nearest 1°C . Individual maximum and minimum values for maximum temperature shall not differ by more than $\pm 4^\circ\text{C}$ of the average value of all determinations.

7.7 *Setting Time*—From the continuous time versus temperature recording of 7.6, the setting time (T_{set}) is the time when the temperature of the polymerizing mass is as follows:

$$T_{amp} - (T_{max} - T_{amp})/2$$

where:

T_{max} = maximum temperature, $^\circ\text{C}$, and

T_{amp} = ambient temperature of $23 \pm 1^\circ\text{C}$.

7.7.1 Report the setting time to the nearest 5 s.

7.7.2 Make two separate determinations of the setting time.

7.7.3 The two values should agree within 1 min of each other, otherwise repeat the test on two additional units and report the average of all runs.

7.7.4 Report the setting time to the nearest 15 s as the average of all determinations.

7.8 *Flow Properties and Viscosity Determination*—The manufacturer must specify whether the cement may be used in its pre-dough or dough state, or both. The determination of its usage dictates which of the following tests the cement should comply with. If the mixture is to be utilized in the pre-dough stage, use the extrusion, viscosity test (7.8.1) and Table 1. If the mixture is to be utilized in the dough stage, use the intrusion test (7.8.2) and Table 1. If the mixture is to be used as a dual usage cement, then both the extrusion (7.8.1) and intrusion (7.8.2) tests must be performed.

7.8.1 *Extrusion Viscosity*:

7.8.1.1 *Apparatus*:

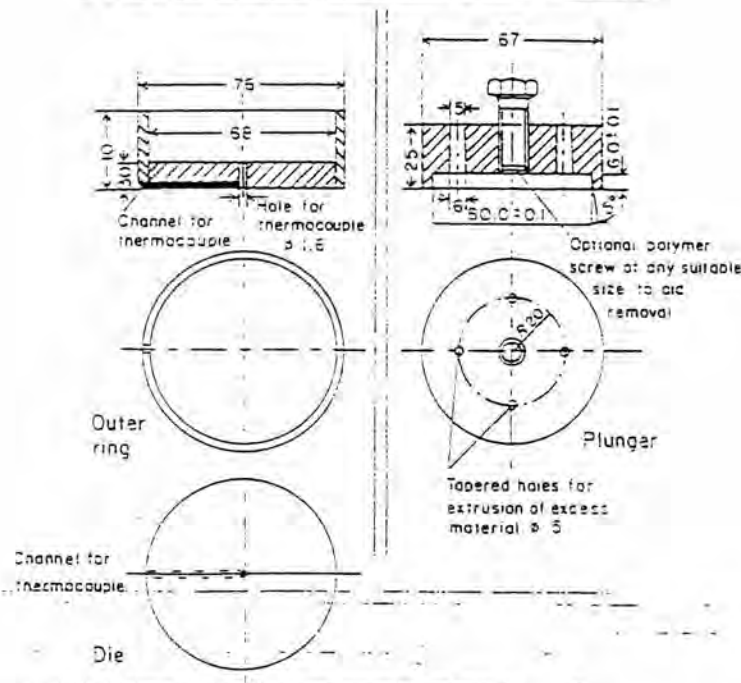
7.8.1.1(a) *Rheometer*—Any capillary rheometer is satisfactory in which acrylic bone cement can be forced from a reservoir through a capillary die and in which temperature.

TABLE 1 Requirements for Powder Liquid Mixture

Property	Extrusion, Viscosity Tests	Dough Usage, Intrusion Tests
Max Dough Time, min.	5.0	5.0
Setting Time Range, min.	5 to 15	5 to 15
Temperature, max., $^\circ\text{C}$	90	90
Intrusion, min., mm	...	2.0

TABLE 2 Requirements for Cured Polymer After Setting

Property	Requirement
Compressive Strength, min., MPa	70
Indentation, max., mm	0.14
Recovery, min., %	60



NOTE—Dimensions in millimetres and ± 0.2 unless otherwise specified. Material for all components: Polytetrafluoroethylene, poly(ethyleneterephthalate), polyoxymethylene, or high density polyethylene.

FIG. 1—Exothermic Heat Mold

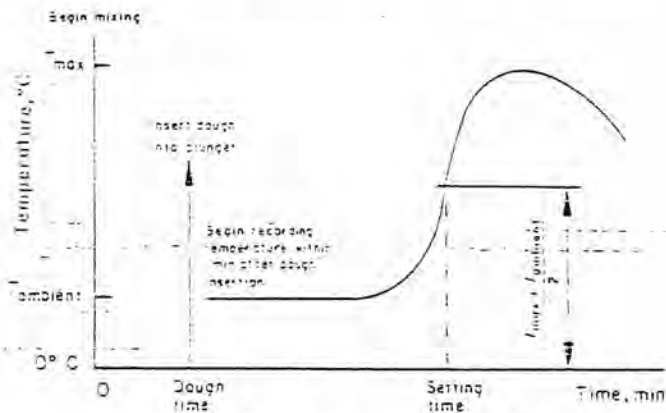


FIG. 2 Continuous Temperature Record

applied force, output rate, and barrel and die dimensions can be controlled and measured accurately. Equipment that provides a constant shear rate has been shown to be equally useful. The capillary die of the rheometer shall have a smooth straight bore that is held within ± 0.0010 mm (± 0.0003 in.) in diameter and shall be held to within ± 0.025 mm (± 0.001 in.) in length. The bore and its finish are critical. It shall have no visible drill or other tool marks and no detectable eccentricity.

7.8.1.1(b) Due to the extreme sensitivity of flow data to the capillary dimensions, it is important that the capillary dimensions are measured with precision and reported. The length to diameter ratio shall normally be between 20 and

40. Larger ratios and ratios less than that suggested require applying large corrections to the data (3, 4). In addition, the ratio of the reservoir diameter to capillary diameter should be between 3 and 15. See Test Method D 3835 for further details of capillary rheometers.

7.8.1.2 Calibration—Perform the test with a certified standard viscosity fluid approximating that expected for bone cement ($50 \text{ N}\cdot\text{s}/\text{m}^2$ to $500 \text{ N}\cdot\text{s}/\text{m}^2$). Determine the viscosity of the standard fluid and the percent error from its specified value. Report this error along with the viscosity of the tested cements.

7.8.1.3 Corrections—Since bone cement is a non-Newtonian fluid, the data may be reported as corrected data.

For example, true shear rates, corrected for non-Newtonian flow behavior and true shear stress corrected for end effects or kinetic energy losses, may be calculated. In such cases, the exact details of the mode of correction must be reported. Some correction factors which may apply are:

- (a) Piston friction.
- (b) Plunger back flow.
- (c) Cement compressibility.
- (d) Barrel back-pressure.
- (e) Capillary entrance effects (Bagley correction) (5).
- (f) Rabinowitsch shear rate correction (6).

7.8.1.4 Procedure:

7.8.1.4.1 Select conditions of temperature and shear stress or shear rate in accordance with expected usage so that the flow rate will fall within desired limits.

7.8.1.4.2 Inspect the rheometer and clean it if necessary. Ensure that previous cleaning procedures and usage have not changed the dimensions or caused scratches or defects in the capillary or apparatus. Make the necessary measurements on the apparatus for future calculations. Prepare the apparatus for running the test.

7.8.1.4.3 Mix one or more complete unit(s) of powder and liquid in the recommended manner. Start a stop watch at the onset of mixing and read all subsequent times from this watch. After complete mixing, transfer the cement to the thermally equilibrated reservoir, and eject any entrapped air or excess bone cement.

7.8.1.4.4 Start the apparatus at a time not greater than 2½ min from the start of mixing and continue operating until the estimated dough time or the viscosity exceeds 500 N·s/m².

7.8.1.4.5 Disassemble the apparatus quickly before the cement sets and clean the apparatus of all remaining cement.

7.8.1.5 Calculations—Perform the calculation for viscosity of the cement at time intervals of 15 s from the start to finish of test run. Use the following equations:

$$\text{Shear Stress, Pa} = \frac{Pr}{2L} = \frac{Fr}{2\pi R^2 L}$$

$$\text{Shear Rate, s}^{-1} = \frac{4Q}{\pi r^2} = \frac{4V}{\pi r^2 t}$$

$$\text{Viscosity, Pa·s} = \frac{P\pi r^2}{8LQ} = \frac{Fr^2 t}{8R^2 L V}$$

where:

- P = pressure by ram, Pa.
- F = force on ram, N.
- r = radius of capillary, m.
- R = radius of barrel, m.
- L = length of capillary, m.
- Q = flow rate, m³/s.
- V = volume extruded, m³, and
- t = extrusion time, s.

7.8.1.5.1 These equations yield true shear rate and true viscosity for Newtonian fluids only; for non-Newtonian fluids, such as bone cement, the apparent shear rate and viscosity are obtained.

7.8.1.6 Report—The report of the flow properties of the cement shall include:

- 7.8.1.6.1 Description of the rheometer used.
- 7.8.1.6.2 Temperature at which the data were obtained.

7.8.1.6.3 The capillary diameter and length to diameter ratio of the capillary.

7.8.1.6.4 The shear rate at which the test was performed.

7.8.1.6.5 Viscosity versus observation time for three tests.

7.8.1.6.6 Statement as to whether any correction factors (7.8.1.3) were applied.

7.8.2 Intrusion:

7.8.2.1 The mold necessary for this test shall be made of PTFE and is shown in Fig. 3. Mix one complete unit of powder and liquid in the recommended manner. When doughing is achieved, gently pack the entire mixture flat into the mold and insert the plunger. One minute after the doughing time, place a load of 49 N (11 lbf) on top of the plunger for one minute. Then remove the weight and allow the mixture to set.

7.8.2.2 Following the set, remove the specimen and measure the average height of the intrusion into all four 1.0-mm diameter holes of the die to the nearest 0.5 mm.

7.8.2.3 Run this test once. If the requirement is not met must be met so in a repeat test.

7.9 Compressive Strength—The test specimens shall be cylinders 12 mm high and 6 mm in diameter. The ends of the specimens shall be flat and smooth and shall be parallel to each other and at right angles to the long axis of the cylinder. An apparatus found convenient for forming these test cylinders is shown in Fig. 4. An apparatus consisting

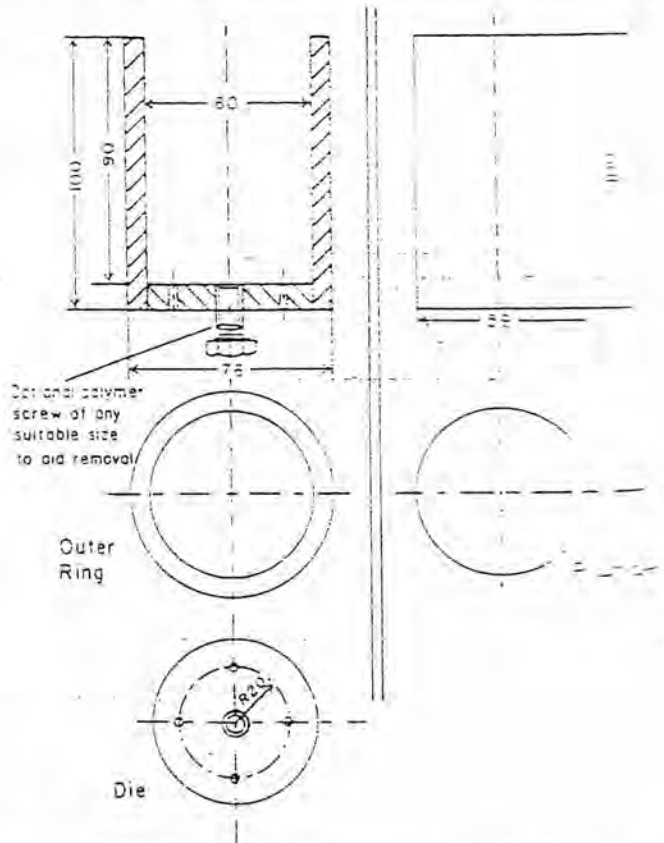


FIG. 3 Intrusion Mold

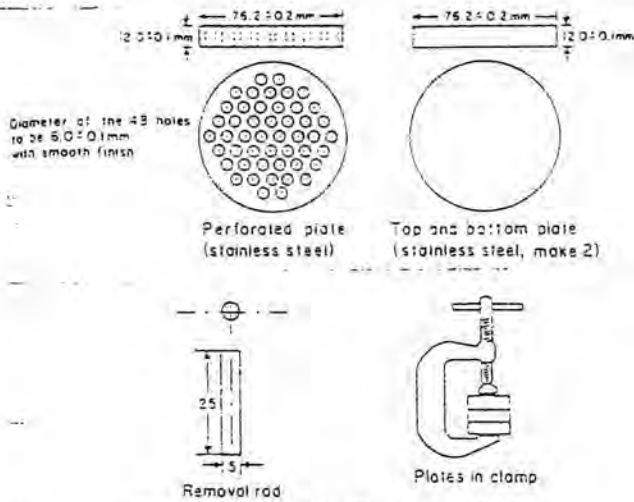


FIG. 4 Compression Specimens Mold

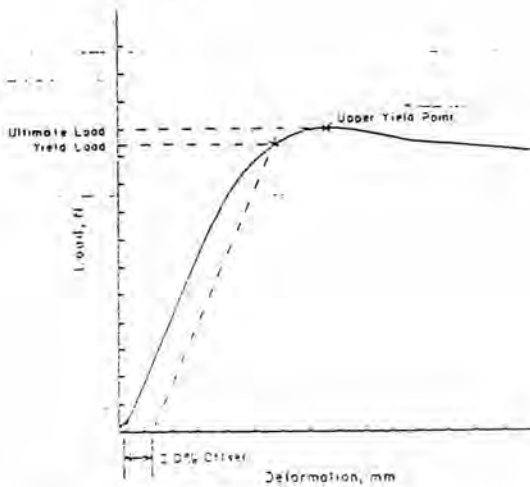


FIG. 5 Failure Load Criteria

more specimen holes may be used as long as adequate spacing between holes is maintained. A mold release agent or silicone spray may be sparingly applied to facilitate specimen removal.

7.9.1 Place the specimen mold on a flat glass or smooth metal plate and slightly overfill using one unit of mixed cement of standard proportions at the commencement of dough time. Press a second flat glass or smooth metal plate on top of the mold. Hold the mold and plates firmly together with a small C-clamp. Then, 1 h later, surface the ends of the cylinder plane at right angles to the axis. The ends of the specimens may be ground flat to the axis by use of a small amount of 240-mesh silicon carbide powder and water. Draw the molds containing the specimens back and forth across the plate coated with the abrasive and water. After surfacing, remove the specimens from the mold.

7.9.2 The time lapse between the start of mixing and the measurement of the compressive strength testing shall be 24 ± 2 h. Storage of the specimens before testing shall be at $23 \pm 2^\circ\text{C}$ and $50 \pm 10\%$ relative humidity. Run specimens on any universal testing machine equipped to record load versus deformation. Employ a deformation cross head speed of 20 or 25.4 mm/min. Test the specimens without the use of any

type of pad between the specimen and the platens of the machine. The failure load shall be the load at the 2.0 % offset (2.0 % proof stress), upper yield point, or at fracture, whichever occurs first (Fig. 5).

7.9.2.1 Calculate the compressive strength as the failure load divided by the calculated cross-sectional area.

7.9.2.2 Report the compressive strength as the average of a minimum of five specimens to the nearest 1 MPa (150 psi).

7.9.3 Repeat the compressive strength test after immersing a minimum of five test specimens in water at 37°C for seven days ± 2 h.

7.9.3.1 After removing the specimens from the mold (7.9.1) place the specimens in water at 37°C . Maintain the samples in the water bath for seven days ± 2 h.

7.9.3.2 Test the specimens in accordance with 7.9.2.

7.9.3.3 Report the compressive strength as the average of a minimum of five specimens to the nearest 1 MPa (145 psi). If the strength is less than 90 % of the value reported in 7.9.2.2, repeat the test. The minimum acceptable strength is 90 % of the value reported in 7.9.2.2 or the value stated in Table 2, whichever is greater.

7.10 Indentation and Recovery (see ANSI/ADA Specification No. 15)—Determine the indentation resistance at 24 ± 2 h after start of mixing with a "Rockwell" type superficial hardness tester. Use the disk made in 7.6, avoiding the region of the thermocouple placement. Remove any flash or other irregularity which would prevent the disk from seating firmly on the hardness tester.

7.10.1 Since some of these "Rockwell" type superficial hardness testers are designed for multiple applications, exercise care to ensure that the instrument used is rigged to apply the loads prescribed below and that the dial gage referred to for measurement of penetration is one having an accuracy of 0.001 mm (0.00004 in.).

7.10.2 Before conducting the test, determine the inherent frame spring factor of the instrument, that is, the amount the frame opens during the application of the major load. The average of three determinations shall be recorded as *A*.

7.10.2.1 Remove the anvil and indenter components.

7.10.2.2 Place a Rockwell "C" calibration block on the anvil seat.

7.10.2.3 Apply the 29.4 N minor load.

7.10.2.4 Apply the 290.4 N major load.

7.10.2.5 Remove the major load and re-zero the instrument.

7.10.2.6 Repeat steps 7.10.2.4 and 7.10.2.5 until the instrument consistently reads zero upon removal of the major load.

7.10.2.7 Then re-apply the major load. The resulting reading, that is, the number of dial divisions through which the needle passes, reflects the inherent frame spring factor, *A*. Repeat three times for an average *A*.

7.10.3 Replace the spot anvil and 12.7-mm ball indenter components and use a Rockwell 30 "Y" block to calibrate the instrument.

7.10.4 Test the specimens in the following manner. The average of three determinations (each at a separate location) shall be made for total indentation and recovery.

7.10.4.1 Apply the minor load.

7.10.4.2 Apply the major load (which includes the minor load) for 10 min.

7.10.4.3 Record the value registered as Reading *B* (indentation plus spring factor).

7.10.4.4 Remove the major load leaving the minor load resting on the specimen.

7.10.4.5 Then, 10 min later, record the value of the number of dial divisions subtracted from 100 as Reading *C* (Recovery Reading).

7.10.4.6 The total indentation under load and recovery shall be determined using the following equations:

$$\text{Total Indentation Under Load (mm)} = B (\text{avg.}) - A (\text{avg.})$$

$$\text{Recovery, \%} = \frac{B (\text{avg.}) - A (\text{avg.}) - C (\text{avg.})}{B (\text{avg.}) - A (\text{avg.})} \times 100$$

7.11 *Precision and Bias*—Since 1976, the original Specification F 451 methodologies have reportedly been routinely utilized by the various manufacturers. With the exception of the viscosity method of 7.8.1, which is based on another accepted ASTM document (Test Method D 3835), each test methodology in Section 7 contains its own statement of reporting acceptable levels of performance, reproducibility, and precision. Therefore, no interlaboratory studies have been performed by the Committee F-4.

8. Packaging

8.1 Materials shall be supplied in properly sealed containers made of materials that shall not contaminate or permit contamination of the contents. The containers shall be packaged so as to prevent damage or leakage during shipping and storage. Materials must be packaged to permit sterile transfer of contents to the sterile field.

8.2 The contents shall be easily accessible, easy to open, and convenient to mix in the operating room. Entire package contents (both powder and liquid) must be mixed to achieve recommended proportions.

9. Labeling

9.1 Labeling on these cements must be in conformance with the Federal Food, Drug, and Cosmetic Act, Code of Federal Regulations, and other pertinent laws and regulations.

9.2 The following minimal information must appear on the container label.

9.2.1 It shall be clearly stated or color coded, or both, if the mixture is intended for usage in the pre-dough, dough, or dual usage state.

9.2.2 The weight or volume, or both, of the liquid and powder components must be stated.

9.2.3 Constituents of the powder and liquid shall be clearly stated in terms of weight or volume percent. This shall include the generic names of polymers, copolymers, chemical initiators, stabilizers, cross-linking agents, and any other ingredients, such as radiopacity agents, gels, fillers, or antibiotics.

9.2.4 A statement that the contents are sterile and that the sterility shall be guaranteed only if the containers are undamaged.

9.2.5 The following warning shall appear on the label: (a) Flammable liquid; (b) Store below 25°C, and (c) Protect from light.

9.2.6 A statement to the effect that federal law restricts this device for sale by or on the order of a physician should be displayed.

9.2.7 The manufacturer and distributor shall be identified.

9.2.8 Each individual component of the package unit must be clearly identified as to batch or lot number.

9.3 The following information shall appear on the product insert labeling accompanying each package.

9.3.1 Adequate and accurate instruction shall be given for handling the components and preparing the cement. Instructions shall include a directive to mix all of the powder with all the liquid of a single unit. Procedures required to mix the materials, along with recommended mixing utensils, shall be given.

9.3.2 Proper technique for administration and recommended procedures for using the cement, including any special precautions, shall be indicated.

9.3.3 Toxic, hazardous, or irritating characteristics associated with the handling and use of the components and cement shall be indicated.

9.3.4 A statement shall be included that states that high temperatures of either the ambient surroundings or material will cause shorter doughing, working, and setting times, while low temperatures of either the ambient surroundings or material will increase doughing, working, and setting times.

9.3.5 The ranges of doughing and setting times as measured at 23 ± 1°C (7.5 and 7.7) shall be clearly stated.

APPENDIX

(Nonmandatory Information)

XI. RATIONALE

X1.1 Acrylic bone cement is a powder-liquid system that is currently sold in the United States for the fixation of internal orthopedic appliances. Because it plays a key role in highly synchronized surgical procedures, such as total joint replacement, its setting characteristics must be known and consistent each time. The material must also have adequate physical properties for placement and function. To these

ends, the standard is addressed.

X1.2 While many of the tests are obvious forms of good manufacturing practice, others may be slightly more subtle and require some elaboration.

X1.2.1 The stability test measures the viscosity after storing the liquid at 60°C. This is an accelerated aging test to ensure that the monomeric component of the bone cement

does not readily polymerize prematurely while stored before use.

X1.2.2 The dough and set times check that the material will be ready for placement at the proper time in the surgical procedure and that it will set neither prematurely nor in a delayed fashion.

X1.2.3 The maximum temperature test makes sure that the mass will not release excessive heat during setting. This could be damaging to the patient's tissue if not properly controlled.

X1.2.4 The dough and setting times and maximum temperature test also evaluate other important parameters. These tests will only respond consistently in the required time and temperature ranges if the powder particle size distribution, the liquid to powder ratio, the complex chemical compositions of both the powder and liquid, and the catalyst amount and distribution have been properly formulated and meted out and strict quality control during all stages of manufacturing is carefully monitored.

X1.2.5 The viscosity tests of the predough material and intrusion evaluations demonstrate that the material will flow into the bony interstices and around prostheses to produce adequate mechanical interlocking upon setting of the material.

X1.2.6 The compressive strength test measures if the set material will be strong enough for clinical applications.

X1.2.7 The indentation and recovery measures the resist-

ance of the surface to an applied load and recovery after release of this load. Multiple measurements of indentation and recovery on various surface locations will verify that the liquid was properly distributed during mixing and pockets of unreacted liquid or low molecular weight polymer are not likely to be present.

X1.3 Further topics presently under consideration for eventual addition to the specification are as follows:

X1.3.1 Other mechanical tests, such as tension, flexion, and fracture toughness, which may be more sensitive to internal porosity and surface defects than the current compression test.

X1.3.2 Statement of biocompatibility of the cement.

X1.3.3 Shortening the time spans of the indentation tests.

X1.3.4 Requiring doughing and setting time data to be furnished with product information.

X1.3.5 Investigating changes in physical properties when leachable additives, such as antibiotics, are purposefully added.

X1.3.6 To define the optimum conditions of the viscosity test methodology so that the performance standards can be established.

X1.4 It should be noted that this document contains both test methodology and performance standards. Currently, in the case of viscosity measurement, only the test methodology has been described. This methodology will serve as a basis for future performance standards.

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