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APPENDICES

Appendix A

X - Ray Diffaction Card

of

Hydroxyapatite

($Ca_5(PO_4)_3OH$)

9-432 MAJOR CORRECTION

d	2.81	2.78	2.72	8.17
I/I$_1$	100	60	50	11

$Ca_5(PO_4)_3(OH)$ $1/2[Ca(OH)_2 \cdot 3Ca_3(PO_4)_2]$ ★

CALCIUM PHOSPHATE HYDROXIDE (HYDROXYL-APATITE)

Rad. CuKα$_1$ λ 1.5405 Filter Dia. 114.6mm
Cut off 50 I/I$_1$ PHOTOMETER* (GUINIER CAMERA)
Ref. DE WOLFF, TECHN. PHYS. DIENST, DELFT, HOLLAND

Sys. HEXAGONAL S.G. $P6_3/m$ (176)
a$_o$ 9.413 b$_o$ c$_o$ 6.884 A C 0.7309
α β γ Z 2 D$_x$ 3.16
Ref. IBID.

ε ω ε Sign
2V D 3.08 mp Color
Ref.

* □□$_1$ 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).

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
8.17	12	100	2.040	2	400
5.26	6	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	320
3.51	2	201	1.841	40	213
3.44	40	002	1.806	20	321
3.17	12	102	1.780	12	410
3.08	18	210	1.754	16	402,303
2.814	100	211	1.722	20	004,411
2.778	60	112	1.684	4	104
2.720	50	300	1.644	10	322,223
2.631	25	202	1.611	3	312
2.528	6	301	1.587	4	501,204
2.296	8	212	1.542	6	420
2.262	20	310	1.530	6	331
2.228	2	221	1.509	10	214,421
2.143	10	311	1.474	12	502
2.124	4	302	1.465	4	510
2.065	3	113	PLUS ADDITIONAL LINES		

Appendix B

X - Ray Diffaction Card

of

Alpha Tricalcium Phosphate

(α - $Ca_3(PO_4)_2$)

9-348 MAJOR CORRECTION

d	2.91	2.62	3.91	12.3
I/I$_1$	100	50	40	4

α-$Ca_3(PO_4)_2$

ALPHA CALCIUM ORTHOPHOSPHATE

Rad. $CuK\alpha_1$ λ 1.5405 Filter Dia. 114.6mm
Cut off 50 I/I$_1$ PHOTOMETER (GUINIER CAMERA)
Ref. DEWOLFF, TECHN. PHYS. DIENST, DELFT, HOLLAND

Sys. ORTHORHOMBIC* S.G.
a 15.22 b 20.71 c 9.109 A 0.7349 C 0.4398
α β γ Z 16 Dx 2.87
Ref. IBID.

ε α ω β γ Sign
2V D 2.814 mp 1720°C Color
Ref. MACKAY (SEE BELOW)

* STATED TO BE MONOCLINIC PSEUDORHOMBIC BY MACKAY, ACTA CRYST. 4 743 (1953)
SAMPLE OBTAINED BY HEATING β-PHASE AT 1400°C.

d Å	I/I$_1$	hkl	d Å	I/I$_1$	hkl
12.3	4	110	3.35	8	312
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
5.83	10	201	3.05	4	332
5.18	12	131,040	3.01	20	510
4.55	4	002	2.947	20	113
4.33	4	311	2.919	35	402,023
4.28	2	240,112	2.905	100	441,170
4.17	2	022	2.860	30	511
4.00	30	150	2.816	2	203,422
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	043,252
3.51	4	401	2.590	30	080
3.45	6	060	PLUS ADDITIONAL LINES		

X - Ray Diffaction Standard Data

of

Beta Tricalcium Phosphate

(β - $Ca_3(PO_4)_2$)

Indices (hkl)	d(A°)	I/I_o	Indices (hkl)	d(A°)	I/I_o
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		

Designation: C 373 – 72 (Reapproved 1982)

Appendix C

Standard Test Method for WATER ABSORPTION, BULK DENSITY, APPARENT POROSITY, AND APPARENT SPECIFIC GRAVITY OF FIRED WHITEWARE PRODUCTS[1]

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 saturated mass, M, to the nearest 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 cm^3 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 buik 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.

Designation: F 981 – 91

Standard Practice for Assessment of Compatibility of Biomaterials (Nonporous) for Surgical Implants with Respect to Effect of Materials on Muscle and Bone[1]

This standard is issued under the fixed designation F 981; 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 practice provides a series of experimental protocols for biological assays of tissue reaction to nonporous, nonabsorbable biomaterials for surgical implants. It assesses the effects of the material on animal tissue in which it is implanted. The experimental protocol is not designed to provide a comprehensive assessment of the systemic toxicity, carcinogenicity, teratogenicity, or mutagenicity of the material. It applies only to materials with projected applications in human subjects where the materials will reside in bone or soft tissue in excess of 30 days and will remain unabsorbed. Applications in other organ systems or tissues may be inappropriate and are therefore excluded. Control materials will consist of any one of the metal alloys in Specifications F 67, F 75, F 90, F 136, F 138, or F 562, high purity dense aluminum oxide as described in Specification F 603 or ultra high molecular weight polyethylene as stated in Specification F 648 or USP polyethylene negative control.

1.2 This document is a combination of Practice F 361 – 80 and Practice F 469 – 78. The purpose, basic procedure, and method of evaluation of each type of material are similar; therefore, they have been combined.

1.3 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

F 67 Specification for Unalloyed Titanium for Surgical Implant Applications[2]

F 75 Specification for Cast Cobalt-Chromium-Molybdenum Alloy for Surgical Implant Applications[2]

F 86 Practice for Surface Preparation and Marking of Metallic Surgical Implants[2]

F 90 Specification for Wrought Cobalt-Chromium-Tungsten-Nickel Alloy for Surgical Implant Applications[2]

F 136 Specification for Wrought Titanium 6Al-4V ELI Alloy for Surgical Implant Applications[2]

F 138 Specification for Stainless Steel Bars and Wire for Surgical Implants (Special Quality)[2]

F 361 Practice for Assessment of Compatibility of Metallic Materials for Surgical Implants with Respect to Effect of Materials on Tissue[3]

F 469 Practice for Assessment of Compatibility of Nonporous Polymeric Materials for Surgical Implants with Regard to Effect of Materials on Tissue[4]

F 562 Specification for Wrought Cobalt-Nickel Chromium-Molybdenum Alloys for Surgical Implant Application[2]

F 603 Specification for High Purity Dense Aluminum Oxide for Surgical Implant Application[2]

F 648 Specification for Ultra-High-Molecular-Weight Polyethylene Powder and Fabricated Form for Surgical Implants[2]

F 763 Practice for Short-Term Screening of Implant Materials[2]

3. Summary of Practice

3.1 This practice describes the preparation of implants, the number of implants and test hosts, test sites, exposure schedule, implant sterilization techniques, and methods of implant retrieval and tissue examination of each test site. Histological criteria for evaluating tissue reaction are provided.

4. Significance and Use

4.1 This practice covers a test protocol for comparing the local tissue response evoked by biomaterials, from which medical implantable devices might ultimately be fabricated, with the local tissue response elicited by control materials currently accepted for the fabrication of surgical devices. Currently accepted materials are the metals (and metal alloys (see Section 2)), dense aluminum oxide and polyethylene which are standardized on the basis of acceptable long-term experience. The controls consistently produce cellular reaction and scar to a degree that has been found to be acceptable to the host.

5. Test Hosts and Sites

5.1 Rats (acceptable strains such as Fischer 344), New Zealand rabbits, and dogs may be used as test hosts for soft tissue implants response. It is suggested that the rats be age and sex matched. Rabbits and dogs may be used as test hosts for bone implants.

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[1] This practice 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 Apr. 15, 1991. Published August 1991. Originally published as F 981 – 86. Last previous edition F 981 – 87.

[2] *Annual Book of ASTM Standards*, Vol 13.01.

[3] Discontinued—See *1986 Annual Book of ASTM Standards*, Vol 13.01.

[4] Discontinued—See *1987 Annual Book of ASTM Standards*, Vol 13.01.

5.2 The sacro-spinalis, paralumbar, gluteal muscles, and the femur or tibia can serve as the test site for implants. However, the same site must be used for test and material implants in all the animal species.

5.3 Table 1 contains a suggested minimum number of study animals and a suggested schedule for the necropsy of animals.

6. Implant Specimens

6.1 *Fabrication*—Each implant shall be made in a cylindrical shape with hemispherical ends (see 6.2 and 6.3 for sizes). If the ends are not hemispherical, this shall be reported. Each implant shall be fabricated, finished, and its surface cleaned in a manner appropriate for its projected application in human subjects in accordance with Practice F 86.

6.2 Reference metallic specimens shall be fabricated in accordance with 6.1 from materials such as the metal alloys in Specifications F 67, F 75, F 90, F 136, F 138, or F 562, ceramic in F 603 or polymers such as in Specification F 648 on polyethylene USP Negative Control Plastic.

6.3 *Suggested Sizes and Shapes of Implants for Insertion in Muscle:*

6.3.1 *For Rats*—1 mm diameter by 2 cm long cylindrical implants.

6.3.2 *For Rabbits*—1 mm diameter by 10 to 15 mm long cylindrical implants.

6.3.3 *For Dogs*—6 mm diameter by 18 mm long cylindrical implants.

6.3.4 If fabrication problems prevent preparing specimens mm in diameter, alternative specimen sizes are 2 mm diameter by 6 mm long for rats and 4 mm diameter by 12 mm long for rabbits. If these alternate dimensions are used, such should be reported in accordance with 8.1.

6.4 *Sizes and Shapes of Implants for Insertion in Bone:*

6.4.1 *For Rabbits*—2 mm diameter by 6 mm long cylindrical implants.

6.4.2 *For Dogs*—4 mm diameter by 12 mm long cylindrical implants.

6.4.3 If the length of the bone implants needs to be less than that designated because of anatomical constraints, such should be reported in accordance with 8.1.

6.5 *Number of Test and Control Implants:*

6.5.1 In each rat, due to size, there shall be two implants; one each test and control material implant.

6.5.2 In each rabbit, due to size, there shall be six implants; four test materials and two control material implants.

6.5.3 In each dog, there shall be twelve implants; eight test materials and four control material implants.

6.6 *Conditioning:*

6.6.1 Remove all surface contaminants with appropriate agents and rinse all test and control implants in distilled water prior to sterilization. It is recommended that the implant materials be processed and cleaned in the same way the final product will be.

6.6.2 Clean, package, and sterilize all implants in the same way as used for human implantation.

6.6.3 After final preparation and sterilization, handle the test and control implants with great care to ensure that they are not scratched, damaged, or contaminated in any way prior to insertion.

6.6.4 Report all details of conditioning in accordance with 8.1.

6.7 *Implantation Period*—Insert all implants into each animal at the same surgical session so that implantation periods run concurrently. The implantation period is 52 weeks for rats and rabbits; 104 weeks for dogs, with interim sacrifices at 12, 26, and 52 weeks (see 7.4).

TABLE 1 Intervals of Sacrifice

Necropsy Periods (Weeks After Insertion of Implants)	Numbers of Animals to be Necropsied		
	Rat	Rabbit	Dog
12 weeks	4	4	2
26 weeks	4	4	2
52 weeks	4	4	2
104 weeks	—	—	2

7. Procedure

7.1 *Implantation (Muscle):*

7.1.1 Place material implants in the paravertebral muscles of the adult rat, rabbits, and dogs in such a manner that they are directly in contact with muscle tissue.

7.1.2 Introduce material implants in dogs by the technique of making an implantation site in the muscle by using a hemostat to separate the muscle fibers. Then insert the implant using plastic-tipped forceps or any tool that is nonabrasive to avoid damage to the implant. Do not insert more than twelve implant materials in each dog.

7.1.3 Introduce material implants in rabbits and rats using sterile technique. Sterile disposable Luer-lock needles may be used to implant the material implants into the paravertebral muscles along the spine. In rats insert a negative control implant on one side of the spine and a test material implant on the other side. In rabbits implant one negative control material on each side of the spine and implant two test materials on each side of the spine. If larger diameter specimens are used in accordance with 6.3.4, an alternative implantation technique is that described in 7.1.2.

7.2 *Implantation (Femur)*—Expose the lateral cortex of each rabbit femur and drill three holes 1/16 in. (1.6 mm) through the lateral cortex using the technique and instrument appropriate for the procedure. For dogs, make the holes 1/8 in. (3.2 mm) in diameter; make six holes in each femur. Into each one of these holes, insert one of the implants by finger pressure. Then close the wound.

NOTE 1—Caution should be taken to minimize the motion of the implant in the tissue on the desired result.

7.3 *Postoperative Care:*

7.3.1 Care for the animals in accordance with accepted standards as outlined in the *Guide for the Care and Use of Laboratory Animals* (1).[3]

7.3.2 Carefully observe each animal during the period of assay and report any abnormal findings.

7.3.3 Infection or injury of the test implant site may invalidate the results. The decision to replace the animal so that the total number of retrieved implants will be as represented in the schedule shall be dependent upon the design of the study.

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

7.3.4 If an animal dies prior to the expected date of sacrifice, necropsy it in accordance with the procedure in 7.4 to determine the cause of death. Replacement of the animal to the study shall be dependent upon the design of the study. Include the animal in the assay of data if the cause of death is related to the procedure or test material.

7.4 *Sacrifice and Implant Retrieval:*

7.4.1 Euthanatize animals by a humane method at the intervals listed in Table 1.

NOTE 2—The necropsy periods start at 12 weeks because it is assumed that acceptable implant data has been received for earlier periods such as 1, 4, and 8 weeks from short term implant testing.

7.4.2 At necropsy, record any gross abnormalities of color or consistency observed in the tissue surrounding the implant. Remove each implant with an intact envelope of surrounding tissue. Include in the tissue sample a minimum of a 4-mm thick layer of tissue surrounding the implant. If less than a 4-mm thick layer of tissue is removed, report in accordance with 8.1.

7.5 *Postmortem Observations*—Necropsy all animals that are sacrificed for the purposes of the assay or die during the assay period in accordance with standard laboratory practice. Establish the status of the health of the experimental animal during the period of the assay. Report as described in Section 8.

7.6 *Histological Procedure:*

7.6.1 *Tissue Sample Preparation*—Prepare two blocks from each implantation site.

7.6.1.1 Process the excised tissue block containing either a test implant or control implant for histopathological examination and such other studies as are appropriate. Cut the sample midway from end to end into appropriate size for each study. Record the gross appearance of the implant and the tissue.

7.6.1.2 If special stains are deemed necessary, prepare additional tissue blocks or slides, or both, and make appropriate observations.

7.7 *Histopathological Observations*—Compare the amount of tissue reaction adjacent to the test implant to that adjacent to a similar location on the control implant with respect to thickness of scar, presence of inflammatory or other cell types, presence of particles, and such other indications of interaction of tissue and material as might occur with the actual material under test. A suggested method for the evaluation of tissue response after implantation is Turner et al. (4).

7.7.1 *Suggested Method for Tissue Response Evaluation:*

7.7.1.1 A suggested format with cellular elements to be evaluated and a scoring range of 0 to 3 using the criteria shown in Table 2.

7.7.1.2 The scoring system of 0 to 3 is based upon the number of elements in high power field (470×) average of five fields.

Number of Elements	Score
0	0
1–5	0.5
6–15	1
16–25	2
26 or more	3

7.7.1.3 The degree of necrosis score is determined using the same range of 0 to 3, as follows:

Degree	Score
Not present	0
Minimal present	0.5
Mild degree of involvement	1
Moderate degree of involvement	2
Marked degree of involvement	3

7.7.1.4 An overall toxicity rating of test samples may be given using a rating range of 0 to 4, as follows:

Rating	Score
Nontoxic	0
Very slight toxic reaction	1
Mild toxic reaction	2
Moderate toxic reaction	3
Marked toxic reaction	4

7.7.1.4.1 Pathologists may choose to use the scoring system of comparing the negative control to the test material as an aid in their evaluation. The overall toxicity of the test material as compared to the negative control is to be evaluated independently for all time periods.

8. Report

8.1 The report shall include the following information:

8.1.1 All details of implant characterization, fabrication, conditioning (including cleaning, handling, and sterilization techniques employed).

8.1.2 Procedures for implantation and implant retrieval.

8.1.3 Details of any special procedure (such as unusual or unique diet fed to test animals).

8.1.4 The observations of each control and test implant as well as the gross appearance of the surrounding tissue in which the implants were implanted.

8.1.5 The observation of each histopathological examination and the pathologist's evaluation as to toxicity of test material provided.

TABLE 2 Suggested Evaluation Format and Scoring Range

Animal Number						
Duration of Implant (weeks)						
Sample Description						
Gross Response						
Histopath-Number						
Score		0	.5	1	2	3
Necrosis						
Degeneration						
Inflammation						
	Polymorphonuclear Leukocytes					
	Lymphocytes					
	Eosinophils					
	Plasma Cells					
	Macrophages					
Fibrosis						
Giant Cells						
Foreign Body Debris						
Fatty Infiltration						
Relative Size of Involved Area in mm						
Histopathologic Toxicity Rating						

APPENDIX

(Nonmandatory Information)

X1. RATIONALE FOR PRACTICE F 981

X1.1 This practice is based on the research techniques utilized by Cohen (5). and by Laing, Ferguson, and Hodge (6, 7) in the early 1960's. These studies involved the implantation of metal cylinders in paravertebral muscle of rabbits. The biological reaction to the cylinders was described as the thickness of the fibrous membrane or capsule formed adjacent to the implant. The thickness of the capsule and the presence of inflammatory cells was used as a measure of the degree of adverse reaction to the test material.

X1.2 As first published in 1972, Practice F 361 was a test for the biological response to metallic materials. The scope had been expanded beyond that of the published reports to include bone as well as muscle as an implant test site. To avoid species specific reactions, the method called for the use of rats and dogs as well as rabbits. Cylindrical test specimens with rounded ends were used to avoid biological reactions associated with sharp corners or other variations in specimen shape.

X1.3 In 1978, Practice F 469 was published as a parallel document for the test of polymeric materials. In that the methods are essentially the same, the scope of F 361 has been expanded to include the testing of specimens made of metallic, polymeric or ceramic materials. thereby including and superseding F 469.

X1.4 Porous or porous coated materials are specifically excluded since the response to such materials includes ingrowth of tissue into the pores. As a result. the method of tissue fixation and sectioning. and the evaluation scheme are substantially different.

X1.5 Stainless steel. cobalt chromium. and titanium alloys are used as reference materials since the biological response to these materials has been well characterized by their extensive use in research. The response to these materials is not defined as compatible, but rather the response is used as a reference against which reactions to other materials is compared.

X1.6 This practice is a modification of the original Practice F 361 in that it only involves long term test periods. The short term response to materials is to be evaluated using Practice F 763.

X1.7 This practice was revised in 1987 to allow for alternative specimen dimensions for rats and rabbits for muscle implantation. The original specimen dimensions were intended to be implanted through a needle, which was a change from F 361 and F 469. The alternate dimensions restore those specified since 1972 which some members felt were more appropriate for some material types.

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Designation: F 1185 – 88

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Specification for Composition of Ceramic Hydroxylapatite for Surgical Implants[1]

This standard is issued under the fixed designation F 1185; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

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

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

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

2. Referenced Documents

2.1 *ASTM Standard:*
F 1088 Specification for Beta-Tricalcium Phosphate for Surgical Implantation[3]
2.2 *Code of Federal Regulations:*[4]
Title 21, Part 820.
2.3 *National Formulary*[5]
Tribasic Calcium Phosphate
2.4 *United States Pharmacopeia:*[6]
Identification Tests for Calcium and Phosphate <191>
Lead <251>
Mercury <261>
Arsenic <211>
Heavy Metals <231> Method I
2.5 *U. S. Geological Survey Method:*[7]
Cadmium

3. Descriptions of Terms Specific to This Standard

3.1 *hydroxylapatite*—the chemical substance having the empirical formula $Ca_5(PO_4)_3OH$.[8]

3.2 *ceramic hydroxylapatite*—hydroxylapatite which h been fired at sintering temperatures. Firing time is ma dependent, and should be sufficiently long to cause signi cant densification and formation of a biologically stab form.

3.3 *sintering*—an integration of time and temperature o ceramic precursor which develops a coherent body wi useful properties. Sintering is a non-melting process accon panied by significant surface area and bulk volume redu tions (densification), grain growth, and increases in mecha ical properties.

3.4 *calcining*—the heat treatment of a ceramic precurs for the purpose of eliminating volatile constituents. Ca cining is also accompanied by some surface area and bu volume reductions. Increases in mechanical properties a not usually significant.

4. Chemical Requirements

4.1 Elemental analysis for calcium and phosphorus will b consistent with the expected stoichiometry of hy droxylapatite.

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

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

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

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

4.4 The maximum allowable limit of all heavy metal determined as lead will be 50 ppm as described in 2.4 o equivalent. Sample preparation will be identical to that fo tribasic calcium phosphate as specified in the Nationa Formulary (2.2) except that approximately 1 g of materia will be dissolved in approximately 30 mL of 5 % HCl an boiled.

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[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 Oct. 31, 1988. Published December 1988.

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

[3] *Annual Book of ASTM Standards*, Vol 13.01.

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

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

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

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

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

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

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

5. Test Specimen Fabrication

5.1 Prepare test specimens from the same batch of material and by the same processes as those employed in fabricating the ceramic implant device.

6. Quality Program Requirements

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

APPENDIX

(Nonmandatory Information)

X1. RATIONALE

X1.1 Ceramic hydroxylapatite is commercially available as a synthetic bone-grafting material. As with any implant material, the bioresponse is critically dependent upon the material properties. To achieve reliable biocompatibility these must be known and consistent. This material standard provides specifications for a biocompatible grade of hydroxylapatite. Trace element content and leachability, physical form, and size must be within established biocompatibility standards.

X1.2 X-ray powder diffraction analysis provides better differentiation between hydroxylapatite and several commonly occurring second phases than traditional wet chemical methods.

X1.3 It is recognized that a separate performance standard may be necessary for each end-use product. For this reason, physical and mechanical properties were not specified. A source of general test methods for ceramics may be found in Ref (8).

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(5) Drobeck, H. P., Rothstein, S. S., Gumaer, K. I., Sherer, A. D., and Slighter, R. G., "Histologic Observation of Soft Tissue Responses to Implanted; Multifaceted Particles and Discs of Hydroxylapatite," *Journal of Oral and Maxillofacial Surgery*, Vol 42, 1984, pp. 143-149.

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(8) *Annual Book of ASTM Standards*, Vol 15.02.

Determination of Elements in National Bureau of Standards' Geological Reference Materials SRM 278 Obsidian and SRM 688 Basalt by Inductively Coupled Argon Plasma-Atomic Emission Spectrometry

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Two new geologic reference materials, issued by the National Bureau of Standards as standard reference materials, have been analyzed by a precise, accurate, and rapid method of analysis for their element content. The described procedure uses a multi-acid, low temperature digestion in a closed teflon vessel, followed by the simultaneous determination of 50 elements by inductively coupled argon plasma-optical emission spectrometry. A preconcentration procedure was applied to a separate digestion for the accurate and precise determination of the rare earth elements. Average concentrations of 39 elements for SRM 278 obsidian and 36 elements for SRM 688 basalt are reported. The results for most elements are in good agreement with the certified values and those reported in the literature. Data for as many as 12 elements are reported for these samples for the first time.

Inductively coupled argon plasma-optical emission spectrometry (ICAP-AES), both simultaneous and sequential, is becoming an established, multielement analytical technique for the determination of the element content of geological materials, e.g., Church (1), Crock and Lichte (2) and McLaren et al. (3). The ICAP-AES determination of the elemental content of geological materials following a closed vessel acid digestion has been shown to have good precision and relative freedom from matrix interferences which is also demonstrated in this study by the analysis of two standards recently released by the National Bureau of Standards, SRM 688 basalt and SRM 278 obsidian. Elements reported here include some that show a comparison with literature values and certified values, as well as some reported for the first time for these two samples.

Some of the inherent limitations in spectroscopic analysis, such as spectral line overlap from other elements present and background shifts due to matrix variations, are minimized by proper background correction and mathematical interelement corrections. A preconcentration and separation procedure (2) was used for the determination of the rare earth elements (REE) at or near their chondritic abundance levels.

EXPERIMENTAL

Instrumentation

All ICAP-AES measurements were performed on a 63-channel ICAP emission spectrometer, Jarrell-Ash* model 1160 Atom Comp, with some in-house modifications. These modifications include a Matheson mass flow controller, model 8249, for controlling the sample and plasma gas flow rates, a Gilson Minipulse II peristaltic pump to deliver the sample to a modified Babington nebulizer (4), an autoprofiler, a water saturation system for the nebulizer argon gas flow, and a Perkin-Elmer model 100 autosampler. A Digital Equipment Corporation PDP 11-34 minicomputer controls the ICAP emission spectrometer and is interfaced to a Hewlett-Packard 1000 computer for data reporting. The operating conditions and parameters are listed in Table 1. The wavelengths at which spectral measurements were made are given in Table 2.

Digestion Procedure

The digestion procedure used for all elements except the rare earth elements was one modified from that described by Patchett and Tatsumoto (5). This procedure is based on a low-temperature sealed-container digestion of the sample carried out in a 30 mL teflon bottle. The procedure used is as follows:

* The use of trade names is for descriptive purposes only and does not constitute endorsement by the U.S. Geological Survey.

Geostandards Newsletter, *Vol. 7, N° 2, Octobre 1983, p. 335 à 340*

Table 1. Operating conditions and parameters

Power	1250 W
Argon flow rate	18 L/min, coolant 0.50 L/min, sample
Sample pump rate to nebulizer	0.70 mL/min
Observation height	14.5 mm above load coil
Reciprocal linear dispersion	0.54 mm/mm
Nebulizer	Modified Babington
Optics	1:3 magnification at entrance slit
Slits	25 μm x 33 mm, entrance 50 μm x 33 mm, exit

Table 2. Wavelengths used for analysis

Element	Wavelength (nm)	Element	Wavelength (nm)
Ag	328.0	Mo	202.0
Al	309.2	Na	588.9
As	189.0	Nb	309.4
Au	242.7	Nd	430.3
B	249.7	Ni	231.6
Ba	455.4	P	213.6
Be	313.0	Pb	220.3
Bi	223.0	Pr	422.2
Ca	317.9	Pt	203.6
Cd	226.5	Sb	217.5
Ce	418.6	Sc	424.6
Co	228.6	Se	196.0
Cr	267.7	Si	251.6
Cu	324.7	Sm	442.4
Dy	340.7	Sn	189.9
Er	369.2	Sr	421.6
Eu	381.9	Ta	240.0
Fe	259.9	Tb	367.6
Fe	271.4	Te	238.6
Ga	294.3	Th	401.9
Gd	303.2	Ti	334.9
Ge	265.1	Tl	190.3
Hf	241.6	Tm	313.1
Ho	345.6	U	409.0
K	766.4	V	292.4
La	398.8	W	207.9
Li	670.7	Y	321.6
Lu	261.5	Yb	328.9
Mg	285.2	Zn	213.8
Mn	257.6	Zr	339.1

Solutions Required:

Hydrochloric Acid: J.T. Baker "Instra-Analyzed" for trace element analysis grade, 37%.

Nitric Acid: J.T. Baker "Instra-Analyzed" for trace element analysis grade, 70%.

Hydrofluoric Acid: J.T. Baker Reagent Grade, 48%.

Perchloric Acid: G. Frederick Smith Co., "Double Distilled", 70%.

Aqua Regia: 3 parts Hydrochloric Acid: 1 part Nitric Acid.

Lu internal standard, 500 mg/L: Dissolve in hydrochloric acid a sufficient amount (depends on manufacturer's assay) of 99.999% Lu_2O_3 (available from Spex Industries, Inc., Metuchen, N.J.) to make 100 mL of a 10,000 mg/L solution. Make the 500 mg/L solution by serial dilution with 30% (v/v) hydrochloric acid.

Weight 1.000 g of the −80 mesh sample into a 30 mL thick-walled teflon bottle (available from Savillex Corp., Minnetonka, Minn.). Wrap the bottle's threads with teflon tape to insure a good seal. Add to the sample 500 μL of the Lu internal standard solution. Add 5 mL nitric acid and 5 mL hydrofluoric acid. Heat gently on a hotplate until dry. Remove from the hot plate, cool, and slowly add 3 mL hydrochloric acid and 2 mL nitric acid and allow the reaction to subside, about 15 min. Add 2 mL hydrofluoric acid. Cap the bottle tightly and place in an aluminum heating block (24 in x 12 in x 1 in, with 50 holes drilled to slightly more than the outside diameter of each bottle) on a hotplate preset at 100°C. Heat for 30 min. in the heating block. Remove, cool to room temperature, and carefully remove the caps. Add 0.2 mL perchloric acid and return to the heating block with the lids removed. Evaporate the solution to dryness at 160°C, usually overnight. Remove, cool to room temperature, and add 1 mL of aqua regia. Place the bottles, uncovered, on a steam bath for 15 min. Cool and transfer the solution to an empty pre-weighed 2 oz. polypropylene bottle and dilute to 50.00 g final solution weight with 1% (v/v) nitric acid.

The rare earth elements and yttrium were determined by an auxiliary procedure given in Crock and Lichte (2). This procedure involves an acid digestion of the sample followed by cation and anion exchange separations. The detection limits are improved at least one order of magnitude over the detection limits obtained by the previous procedure.

RESULTS AND DISCUSSION

Detection limits for granitic materials using the described digestion procedure are given in Table 3. Detection limits were defined as three times the standard deviation of the background noise or blank, whichever was larger. Due to spectral interferences from other elements present, these detection limits may vary in other geologic matrices.

Table 3. Elements and detection limits reported for ICP-AES quantitative analysis (1) from an acid digestion

Element	ICP-AES Detection Limit (ug/g)	Element	ICP-AES Detection Limit (ug/g)
Al	500	Mn	4
Fe	500	Mo	2
Mg	500	Nb	4
Ca	500	Ni	2
Na	1000	Pb	4
K	1000	Sb	10
Ti	100	Sc	2
P	100	Se	10
		Sn	4
		Sr	2
Ag	2	Th	4
As	10	U	80
Au	8	V	1
Ba	1	Y (2)	2
Be	0.1	Yb (2)	1
Bi	10	Zn	2
Cd	2	Pr (2)	10
Ce (2)	4	Nd (2)	4
Co	1	Sm (2)	50
Cr	1	Eu (2)	2
Cu	5	Gd (2)	10
Ga	4	Tb (2)	20
Ge	10	Dy (2)	4
La (2)	2	Ho (2)	4
Li	2	Er (2)	4

(1) For granites and a dilution factor of 50 (1.000 g sample dissolved in 50.00 g solution). Elements and/or detection limits may change for non-silicates or highly mineralized samples.

(2) The detection limits used in reporting the REE and Y in Tables 4 and 5 are listed in Crock and Lichte (2). The detection limits given here are those which result when omitting the separation/preconcentration procedure. Also Lu and Tm are not reported in the multi-element analysis, but are reported following the REE separation/preconcentration procedure.

The average result and standard deviation of the results from three bottles of each standard rock taken through the closed vessel digestion three separate times are given in Tables 4 and 5. The REE and Y values as reported in these tables are for ICAP-AES analysis after preconcentration and separation according to the method of Crock and Lichte (2). Due to numerous spectral interferences, the detection limits without this separation-preconcentration procedure are larger for the REE and Y (see Table 3). For SRM 278 obsidian, 39 elements were determined to be above the detection limit and 11 were below the limit of detection. In like manner, for SRM 688 basalt 36 elements were determined to be above the detection limits and 14 were below.

In most cases where the results were at least five times above the detection limits, relative standard deviations ranged from less than 1 percent to about 10 percent. It can be concluded that the ICAP-AES simultaneous determination of trace, minor, and major elements can be precise following an acid digestion of a geologic material.

Even though the ICAP-AES technique can detect most elements, the digestion procedure and subsequent dissolution of the salts are not appropriate for all elements. Silicon and boron are volatilized as fluorides by the hydrofluoric acid and some resistant minerals, such as zircon, may not be totally dissolved, resulting in low values for some elements. However, precise and accurate zirconium results were obtained for both SRM 278 obsidian and SRM 688 basalt in this work, possibly due to the very fine grained structure of both SRM's, which facilitates dissolution. This is not the case with all geological materials, especially granites. Therefore, zirconium is not usually reported for geological materials following an acid digestion, and therefore no detection limit is given in Table 3. Other elements may also give low or imprecise results if they are present in resistant minerals. An example of this is seen in SRM 688 with its chromium content. The chromium is probably present as a rutile-structure mineral, such as chromite or ilmenite, and will have a limited solubility. This limited solubility is shown by imprecise data and a mean chromium value which is lower than the expected value. Several of the elements listed in Table 2 such as platinum, tungsten and the REE, whose analytical lines are routinely looked for in the ICP-AES procedure, generally have concentrations below our limits of detection for most silicate rocks. Because of the importance of rare earths for petrogenetic modeling (7), we have developed a preconcentration procedure, using an ion exchange separation procedure, and have lowered their limits of detection by more than an order of magnitude from those listed in Table 3.

A closed vessel is more effective than an open vessel in dissolving many types of samples because of the increased pressure at a given temperature. A closed vessel also improves refluxing and therefore requires smaller acid volumes, which reduces analyte concentrations in blank. This digestion procedure coupled with the powers of a simultaneous ICAP-AES analysis proves to be a rapid, accurate, and precise method to analyze geological materials.

Table 4. Elemental concentrations* of NBS SRM 278 obsidian as determined by ICAP-AES

	This Study (A)	NBS (B)	Literature (C)
Al (%)	7.78 ±0.08	7.49 ±0.08	7.8 ±0.2
Ba (ppm)	928 ±9	1140	1080 ±60
Be (ppm)	2.4 ±0.1		
Ca (%)	0.72 ±0.01	0.703 ±0.001	0.68 ±0.11
Ce (ppm)	61 ±1	62.2	59.4 ±6.8
Co (ppm)	2 ±1	1.5	1.85 ±0.18
Cr (ppm)	5 ±0.5	6.1	6.34 ±0.93
Cu (ppm)	<5	5.9 ±0.2	
Dy (ppm)	6.8 ±0.4		
Er (ppm	4.1 ±0.3		
Eu (ppm)	0.77 ±0.03	0.84	0.76 ±0.06
Fe (%)	1.47 ±0.01	1.43 ±0.01	1.52 ±0.05
Ga (ppm)	22 ±4		
Gd (ppm)	6.1 ±0.3	5.3	5.34 ±0.08
Ho (ppm)	1.5 ±0.1		
K (%)	3.34 ±0.03	3.45 ±0.02	3.44 ±0.0
La (ppm)	31.0 ±0.7		35.4 ±2.5
Li (ppm)	47 ±1		
Lu (ppm)	0.71 ±0.01	0.73	0.84 ±0.05
Mg (%)	0.143 ±0.002	0.139	
Mn (ppm)	373 ±3	400 ±20	400 ±50
Mo (ppm)	2 ±1		
Na (%)	3.44 ±0.02	3.63 ±0.04	3.46 ±0.26
Nb (ppm)	12.7 ±0.9		
Nd (ppm)	28.6 ±0.9		28.2 ±1.0
Ni (ppm)	4 ±2	3.6 ±0.3	
P (%)	0.017 ±0.001	0.016 ±0.002	
Pb (ppm)	18 ±3	16.4 ±0.2	
Pr (ppm)	8.5 ±0.8		
Sc (ppm)	6 ±0.5	5.1	5.24 ±0.14
Sm (ppm)	6.8 ±0.6	5.7	5.66 ±0.10
Sr (ppm)	60 ±3	63.5 ±0.1	
Th (ppm)	13 ±3	12.4 ±0.3	12.8 ±0.3
Ti (%)	0.148 ±0.001	0.147 ±0.004	0.145 ±0.009
Tm (ppm)	0.50 ±0.1		
U (ppm)	<30	4.58 ±0.04	4.8 ±0.4
V (ppm)	8 ±1		
Y (ppm)	38.3 ±4		
Yb (ppm)	4.68 ±0.05	4.5	4.54 ±0.86
Zn (ppm)	47.8 ±0.4	55	54.0 ±2.5
Zr (ppm)	290 ±2		285 ±16

*Ag, As, Au, Bi, Cd, Sb, Se, Sn and Tb are not listed because they were less than the detection limits and data was not reported by NBS or reference (6).

(A) Mean of 9 determinations with ± standard deviation.

(B) Certificate of Analysis, Standard Reference Material SRM 278 Obsidian; Office of Standard Reference Material, National Bureau of Standards, U.S. Department of Commerce, Washington, D.C. (oxides converted to elemental content).

(C) Graham et al. (6).

Table 5. Elemental concentrations* of NBS SRM 688 basalt as determined by ICAP-AES

	This Study (A)	NBS (B)	Literature (C)
Al (%)	9.04 ±0.05	9.19 ±0.05	9.1 ±0.2
Ba (ppm)	178 ±2	200	197 ±33
Be (ppm)	0.20 ±0.05		
Ca (%)	8.82 ±0.02	8.70	7.9 ±0.3
Ce (ppm)	11.3 ±0.4	13.3	10.1 ±3.9
Co (ppm)	47 ±1	49.7	47.5 ±1.5
Cr (ppm)	260 ±20	332 ±9	328 ±15
Cu (ppm)	90 ±1	96	
Dy (ppm)	3.8 ±0.2		
Er (ppm)	1.9 ±0.1		
Eu (ppm)	1.01 ±0.02	1.07	0.92 ±0.05
Fe (%)	7.34 ±0.03	7.24 ±0.03	7.23 ±0.19
Ga (ppm)	17 ±7		
Gd (ppm)	3.6 ±0.3		2.82 ±0.08
Ho (ppm)	0.80 ±0.05		
K (%)	0.162 ±0.003	0.155 ±0.006	0.17 ±0.01
La (ppm)	5.3 ±0.1		7.54 ±0.93
Li (ppm)	7 ±1		
Lu (ppm)	0.33 ±0.01	0.34	0.34 ±0.06
Mg (%)	5.08 ±0.02	5.07	5.7 ±0.4
Mn (ppm)	1240 ±20	1290 ±20	1150 ±70
Na (ppm)	1.63 ±0.05	1.60 ±0.02	1.39 ±0.12
Nb (ppm)	5 ±1		
Nd (ppm)	10.4 ±0.5		9.95 ±1.08
Ni (ppm)	143 ±2	150	
P (%)	0.056 ±0.002	0.058 ±0.002	
Pb (ppm)	<4	3.3 ±0.2	
Pr (ppm)	2.4 ±0.6		
Sc (ppm)	43.3 ±0.5	38.1	36.1 ±0.9
Sm (ppm)	2.9 ±0.7	2.79	2.31 ±0.08
Sr (ppm)	170 ±.10	169	
Th (ppm)	<4	0.33 ±0.02	
Ti (%)	0.739 ±0.009	0.70 ±0.01	0.72 ±0.02
U (ppm)	<80	0.37	
V (ppm)	248 ±1	250	235 ±25
Y (ppm)	19.5 ±0.1		
Yb (ppm)	2.20 ±0.03	2.09	1.86 ±0.27
Zn (ppm)	79 ±1	58	
Zr (ppm)	63 ±4		58.6 ±8.7

*Ag, As, Au, Bi, Cd, Mo, Sb, Se, Sn, Tb and Tm are not listed because they were less than the detection limits and data was not reported by NBS or reference (6).

(A) Mean of 9 determinations with ± standard deviation.

(B) Certificate of Analysis, Standard Reference Material SRM 688 Basalt; Office of Standard Reference Materials, National Bureau of Standards, U.S. Department of Commerce, Washington, D.C. (oxides converted to elemental content).

(C) Graham et al. (6).

RESUME

Deux nouveaux échantillons géochimiques de référence distribués par le "National Bureau of Standards" ont été analysés par une méthode précise, juste et rapide. La procédure décrite est basée sur la décomposition de l'échantillon avec une dissolution multi-acide à basse température dans un bécher en teflon fermé; ensuite, 50 éléments ont été dosés par spectrométrie d'émission à plasma induit (argon). Une procédure de préconcentration a été appliquée à un aliquot de la solution afin de doser les éléments de terres rares avec précision. Pour l'échantillon d'obsidian (SRM 278), 39 éléments ont pu être déterminés tandis que pour le basalte (SRM 688) 36 éléments ont pu être dosés. Les résultats sont en général en accord avec les valeurs certifiées et celles publiées. Des données pour 12 éléments sont présentées pour la première fois.

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