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Progress Report

on

Specification for Hydrocolloidal Impression Material-Alginate Type

by

Masayoshi Ohashi*
George C. Paffenbarger **
John W. Stanford**

* Guest worker, Nihon University (Tokyo, Japan)
Dental Research Section, National Bureau of Standards
Washington, D. C.

** Research Associate, American Dental Association, Dental Research Section, National Bureau of Standards, Washington, D. C.

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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

SPECIFICATION FOR HYDROCOLLOIDAL IMPRESSION MATERIAL-ALGINATE TYPE

Abstract

A new specification formulated for the delineation of a satisfactory dental elastic impression material based upon alginates embodies fast setting and normal setting types. Requirements were judged by testing sixteen materials in current use. Generally none of the alginate materials imparted as good a surface to the gypsum cast into them as did the agar materials. The compressive properties of all of the materials degenerated when the sample was aged for one week at 60° C.

1. INTRODUCTION

The Dental Research Section of the National Bureau of Standards maintains a close liaison with the Specification Committee of the Dental Materials Group of the International Association for Dental Research as this committee is the chief consultant group for the Council on Dental Research regarding the revision and formulation of the specifications for dental materials of the American Dental Association.

The subcommittee of the Specifications Committee of the Dental Materials Group (IADR) which was responsible for formulating this proposed specification for alginate impression material consisted of:

Ralph W. Phillips, Chairman (1956-61), Indiana University
George C. Paffenbarger, Chairman (1961-63), Dental Research Section, National Bureau of Standards
E. W. Skinner, Northwestern University
A. B. Godber, The S. S. White Dental Mfg. Co.
James Cresson, The L. D. Caulk Co.
Alex Dunlop, The Kerr Dental Mfg. Co.
R. L. Bowen, Secretary, Dental Research Section, National Bureau of Standards

Much of the testing of dental materials for determining the adequacy of the testing procedures given in a tentative or proposed specification is done in the Dental Research Section of the National Bureau of Standards. As one example of this the alginate impression materials listed in Table 1 were subjected to the tests outlined in the specification given in the appendix except where modified. The data resulting from these tests are presented in Table 2.

2. DISCUSSION OF SPECIFICATION TESTS AND DATA

3.2.3* and 4.3.5 - Fineness. The data in Table 2 show that three of the alginate powders had lumps in them that would not pass a No. 20 sieve. These tests were all conducted as outlined in the second draft of the proposed specification for alginate impression material. This

* Section of the specification for alginate impression material given in the appendix

method specified that the powder shall be brushed through the sieve with as little abrasion as possible. The third draft and fourth draft of the specification require vibration of the sieve for 2 minutes. Comparison testing indicated there was no difference in the results obtained by the two methods.

3.2.4 and 4.3.6 - Compatability with Gypsum. The test for compatability with gypsum was designed to avoid the use of commerical dental gypsums to which chemicals have been added to modify the time of setting and the setting expansion. In other words it was necessary to prescribe a standard unmodified gypsum for the test in 3.2.4 without the use of a trade name. This was resolved by adjusting, if needed, to 10 ± 3 minutes, the time of setting of a commerical gypsum (see 6.1) free of additives by using set gypsum (calcium sulfate dihydrate-$CaSO_4 \cdot 2H_2O$) as the accelerator. It was thought necessary to do this because it is commom knowledge that some combinations of dental alginate impression materials and dental gypsums result in a very poor surface on the gypsum cast.

Hosoda and Fusayama [1] show that certain combinations of impression and model materials give superior surfaces to the gypsum casts. They attribute this condition to the presence of additives in dental gypsums. Using X-ray diffraction technics Smith and Fairhurst [2] showed that potassium sulfate when used in gypsum caused the formation of syngenite [$K_2Ca(SO_4)_2.H_2O$] on gypsum surfaces cast in alginate impressions but did not determine its effect. This work was reported as a research annotation and as the authors state was only a preliminary study. More work needs to be done to find out the specific cause of poor surfaces on gypsum casts poured in alginate impressions.

All of the alginate impression materials listed in Table 1 gave poorer gypsum surfaces than the agar based impression materials which were tested during the revision of the specification for the agar type material.

The data in Table 2 for 3.2.4 and 4.3.6 show that better reproduction of the lines on the block (Figure 1) was usually obtained when the impression was shaken to remove the exudate (footnote 5 in Table 2) than when the surface of the impression was blotted (footnote 4 in Table 2).

Dr. Floyd A. Peyton [3] stated that his coworkers at the University of Michigan found that dusting the test block (Figure 1) with talcum and then blowing away the excess powder prevented the alginate and other impression materials from adhering to the brass and at the same time improved the detail reproduction of the lines (Figure 1). The experience in this laboratory confirms this observation so this suggestion has been incorporated into 4.3.6 of the fourth draft of the alginate specification (see appendix).

3.2.6 and 4.3.7 - Setting Time. Mr. J. Cresson of the L. D. Caulk Co. [4] described a simple and effective setting time test that this laboratory found most useful and reliable. The data in Table 2 show that the setting time correlates well with the two types (1.2.1) which are designated on a basis of time of setting. Therefore, this test has been incorporated in the fourth draft of the specification (see appendix).

3.2.7 and 4.3.8 - Working Time. This test delineated the rubber base impression materials adequately [5] and was found to be as useful with the alginate-containing materials.

3.2.8 and 4.3.9 - Impression of the Metal Model. All of the alginates could be withdrawn from the model (Figure 2) without rupturing. There were no fins except where the material adhered to the metal.

It is impossible to judge when the surface of a gypsum cast is chalky and when it is not by simple visual inspection. All of the surfaces were chalky to some extent when rubbed with the fingers. The surfaces of gypsum casts made in alginate impressions were never as good as those cast in the agar impressions.

Eventually a test should be devised that would give quantitative values. Probably the scratch test suggested by Hosoda et al [6] would be a good beginning.

3.2.9 - Uniformity. All of the materials complied with 3.2.9 except material M which had little white granules present in it after mixing.

3.2.10 and 4.3.10.3 - Permanent Deformation Caused by Fixed Strain. The current American Dental Association Specification No. 11 for Hydrocolloidal Impression Material -

Agar Type [7] specifies that, "The set shall not exceed 3.0 percent when stresses of 100,1000, 0 and 100 grams per square centimeter are applied for 1 minute intervals in the foregoing sequence." Cresson [8] pointed out that it would be preferable to specify a fixed strain rather than a fixed stress because the elastic impression materials undergo strain when slipping over undercut surfaces and that it would require varying amounts of stress to strain the different materials a fixed amount. Cresson suggested that all specimens be given a strain of 12 percent in determining the permanent set. It is believed that this test method (4.3.10.3) will rate the ability of the impression materials to take accurate impressions of undercut surfaces. It is argued by some that the abandonment of the test for permanent deformation based on a stress test would permit the use of impression materials which might even be deformed by the weight of the gypsum slurry poured into the impression. However, the limitation on strain in compression (3.2.12) eliminates such materials.

Data were obtained on permanent set caused by a fixed strain (see Table 2) as outlined in 3.2.10 and 4.3.10.3 of the alginate specification (appendix) and on permanent set caused by fixed stress [7] (see Table 2). The permanent deformation caused by the fixed stress [7] is always higher than that caused by the fixed strain but the relationship between the two values varies among the alginate materials. The higher permanent deformation obtained with the fixed stress method can probably be attributed to the greater time in compression (60 rather than 30 seconds) and for most materials the greater strain associated with the fixed stress method. Material J with the highest value of permanent deformation, 6.6%, is also the material having the highest value for strain in compression 19.8% (see Table 2). An examination of the gypsum case made in the impression of alginate J showed no distortion. The diameter of the shaft of post B, Figure 2, of the gypsum cast made in alginate J impression was 4.54 mm. The metal post was 4.51 mm so there was only a 30 micron enlargement.

3.2.11 and 4.3.10.4 - Compressive Strength. The values in Table 2 were determined on a machine operated at constant rate of strain. The specification prescribes a constant rate of stress. An attempt was made to compress the specimens in the constant rate of strain machine at an averate rate of stress by noting on trial specimens the load at rupture and the time required to attain it. Values

derived under such conditions may be lower than those obtained on machines operated at constant stress rates but preliminary tests on Material A showed no difference.

In most instances a higher value for compressive strength is obtained on the higher rates of loading.

After the tests were completed it was decided to prescribe specimens 8 instead of 6 minutes old. This will raise all of the values in the table different amounts depending on the rate of hardening of each material during the 2 minute interval between specimens 6 and 8 minutes old.

3.2.12 and 4.3.10.5 - Strain in Compression. The values in Table 2 for strain in compression were obtained on the same specimens used in the test for permanent deformation at a fixed stress as specified in the current American Dental Association Specification No. 11 for Hydrocolloidal Impression Material-Agar Type [7] except that the specimens were six minutes old when the test began. The revised requirement in the specification now prescribes a specimen 10 minutes old that has not been subjected to any previous loading.

These values are in general much higher than those obtained on the agar base materials. Generally lower values than those reported would be obtained if the specimens were tested at 10 minutes as now prescribed rather than at 6 minutes.

3.2.13 and 4.3.10.6 - Deterioration. Since introduction of the alginate impression materials during World War II the deterioration during storage has been a problem. Such is still true. A comparison of the compressive strengths of the materials held in a sealed container at 60° C and 100 percent relative humidity for one week with the compressive strengths of the unaged materials (Table 2) shows that in every instance the compressive strength on the aged material was reduced. Sometimes the reduction amounted to as much as 94 percent (Material P) or to as little as 14 percent (Material H) on the values obtained at the lower rates of loading.

Both the strain in compression and the permanent deformation are increased on specimens made from aged materials (4.3.10.6).

3.3 - Instructions for Use. It is necessary to have the powder/water ratio expressed in grams of powder to milliliters of water because it is not possible to obtain accurate values by using the proportioners that accompany the packages. The chief source of trouble is that the amount of powder that a scoop will hold depends upon the method of packing the powder in the scoop.

3. SUMMARY

Sixteen currently used hydrocolloidal impression materials-alginate type were measured in a variety of physical tests that have pertinence relating to their dental use. Using these tests as a basis, a proposed new specification was formulated.

Suggestions relating to the specification are most welcome.

REFERENCES

1. Hosoda, H. and Fusayama, T. Surface reproduction of elastic impressions. J. D. Res. 38:932, September-October, 1959.

2. Smith, D. C. and Fairhurst, C. W. Effect of hydrocolloid impression materials on the surface of dental stone. J. D. Res. 41:1103, September-October, 1962.

3. Personal communication.

4. Personal communication.

5. Miller, W. A. C., Hansen, W. C., Dickson, G. and Sweeney, W. T. Physical properties of synthetic-rubber-base dental impression materials. J.A.D.A. 60:211, February, 1960.

6. Hosoda, H., Otani, H., Hirano, T. and Fusayama, T. Measurement and reinforcement of the superficial hardness of indirect stone models. J. D. Res. 41:752, July-August, 1962.

7. American Dental Association Specification No. 11 for hydrocolloidal impression material-agar type, Guide to Dental Materials. p. 61, 1962-1963.

8. Cresson, J. Suggested revisions for testing dental elastic impression materials. J. D. Res. 28:573, December, 1949.

APPENDIX

January 1, 1963

FOURTH DRAFT OF A PROPOSED

AMERICAN DENTAL ASSOCIATION SPECIFICATION NO. ___

for

DENTAL HYDROCOLLOIDAL IMPRESSION MATERIAL -- ALGINATE TYPE

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification is for dental impression material in powder form containing an alginate as the gel-forming ingredient.

1.2 Classification

1.2.1 Types. Alginate hydrocolloidal impression material shall be of the following types.

Type I - Fast Setting (3.2.6)

Type II - Normal Setting (3.2.6)

2. APPLICABLE SPECIFICATION

2.1 Specifications. The following specifications form a part of this specification. Federal Specification RR-S-366b-(3) Sieves; Standard for testing purposes, July 27, 1953 and American Dental Association Specification No. 2 for Casting Investment for Dental Gold Alloy. (Copies of American Dental Association specifications may be obtained upon application to the Council on Dental Research, American Dental Association, 222 East Superior Street, Chicago 11, Illinois, or to the American Dental Association Research Division, National Bureau of Standards, Washington 25, D. C.)

3. REQUIREMENTS

3.1 General requirements. The material shall be uniform and free of foreign materials. When used in accordance with instructions (3.3) accompanying the package, the material shall form a smooth plastic mass suitable for taking impressions in the mouth.

3.2 Special requirements.

3.2.1 Odor and flavor. The material shall not have an unpleasant odor or flavor.

3.2.2 Irritation. The manufacturer shall furnish a statement at the time of certification reporting that the material does not normally irritate oral tissues and does not contain poisonous ingredients in sufficient concentration to be harmful to human beings when used as directed, or in the event of accidental ingestion of 10 milliliters.

3.2.3 Fineness. All of the material, when tested as in 4.3.5, shall pass a number 20 sieve. The sieve shall conform to Federal Specification RR-S-366b (3) Sieves; Standard for testing purposes.

3.2.4 Compatibility with gypsum. The material shall impart a smooth surface to and shall separate cleanly from a cast made from unmodified alpha calcium sulfate hemihydrate to which has been added, if necessary, sufficient calcium sulfate dihydrate to adjust the time of setting to 10 ± 3 minutes. The method for determining the time of setting shall be that given in American Dental Association Specification No. 2 for Casting Investment for Dental Gold Alloy. A gypsum cast poured against the material in accordance with 4.3.6 shall reproduce a line 0.075 mm (0.003 in) wide (Figure 1). The gypsum shall itself be capable of reproducing the line 0.050 mm (0.002 in) wide when allowed to harden against the block (Figure 1).

3.2.5 Mixing time. The time of mixing (as stated in the manufacturer's directions) to obtain a smooth workable consistency shall be not more than one minute.

3.2.6 Setting time. The setting time, when tested as in 4.3.7, shall be not less than 60 seconds, nor more than 120 seconds for Type I, Fast Setting; and shall be not less than 120 seconds, nor more than 4 minutes and 30 seconds for Type II, Normal Setting.

3.2.7 Working time. The working time when tested as in 4.3.8 shall be not less than 1 minute and 15 seconds for Type I, Fast Setting; and shall be not less than 2 minutes for Type II, Normal Setting.

3.2.8 Impression of the metal model. The material shall be sufficiently elastic to take an impression of the metal model (Figure 2) without rupturing.

3.2.9 Uniformity. After mixing, the ingredients shall not segregate. The mixed material shall be homogeneous, shall have a smooth surface and shall be free of lumps and granules.

3.2.10 Permanent deformation. The permanent deformation shall be not more than 3.0 percent after 12 percent strain is applied for 30 seconds when tested as in 4.3.10.3.

3.2.11 Compressive strength. The compressive strength shall be not less than 3,500 Gm/cm^2 (50 lb/in^2) when tested as in 4.3.10.4.

3.2.12 Strain in compression. The strain shall be not less than 4.0 nor more than 20.0 percent between a stress of 100 Gm/cm^2 (1.42 lb/in^2) and a stress of 1,000 Gm/cm^2 (14.2 lb/in^2) when tested as in 4.3.10.5.

3.2.13 Deterioration. The compressive strength of the material, when tested as in 4.3.10.4, after storage as described in 4.3.10.6, shall be not less than 2,600 Gm/cm^2 (37 lb/in^2).

3.3 Instructions for use. Adequate and accurate instructions for manipulation shall accompany each package. These instructions shall include: (1) the powder-water ratio in grams of powder and milliliters of water, (2) the time and method of mixing, (3) the temperature of the water and the material, (4) time in the mouth for proper set and (5) any special treatment of the impression, such as the use of a fixative solution, in the interval

between withdrawal from the mouth and preparation of the gypsum cast.

4. SAMPLING, INSPECTION AND TESTING PROCEDURES

4.1 Sampling. A number of packages (sufficient to provide a total sample of at least 1 kilogram or 2 pounds) shall be procured at retail by a member of the American Dental Association. This sample shall be forwarded in the original unopened package or packages to the American Dental Association Research Division, National Bureau of Standards, Washington 25, D. C.

4.2 Inspection. Visual inspection shall be used in determining compliance with requirements outlined in 3.1, 3.2.2, 3.2.5, 3.2.8, 3.2.9, 3.3, 5.1, 5.2, 5.3.1 and 5.3.2.

4.3 Testing procedures.

4.3.1 Preparation of specimens. The mix shall be prepared in accordance with the directions which accompany the package (3.3).

4.3.2 Standard testing conditions. All physical tests shall be made under uniform atmospheric conditions of 23 ± 2.0° C. (73.4 ± 3.6° F.) and 50 ± 10 percent relative humidity. Equipment and material shall be conditioned in the testing room for not less than 10 hours prior to making the tests.

4.3.3 Odor and flavor. The pleasantness or unpleasantness of the odor and flavor shall be determined by smelling and tasting the material.

4.3.4 Irritation. The material, prepared according to the manufacturer's instructions and used as an impression material, shall not cause visible evidence of irritation of the oral mucosa.

4.3.5 Fineness. The container of alginate shall be tumbled or rotated to insure homogeneity of the contents. Ten to twelve grams of the alginate powder as received from the manufacturer shall be placed on a number 20 sieve, the top and bottom shall be replaced, and the assembly shall be vibrated for 2 minutes. On completion of the test, the sieve shall be carefully examined for residue remaining on the upper surface of the wire or

occluding the sieve openings.

4.3.6 Compatibility with gypsum. A ring of the type specified in 4.3.10.1 shall be positioned on a stainless steel test block similar to that shown in Figure 1 so that the intersection of a cross-line and a 0.025 mm (0.001 in) wide line is in the center of the ring. (The stainless steel test blocks may be lightly dusted with talcum powder and the excess talcum powder blown off, if the impression material adheres to the blocks.) The ring shall be slightly overfilled with the material. A flat plate shall be placed on top and the excess material shall be squeezed out. Two minutes after the start of mix, the entire assembly shall be placed in a water bath maintained at 37 ± 1° C. (98.6 ± 1.8° F.). Six minutes after the start of mix, the assembly shall be removed from the bath. The ring with the impression material shall be separated immediately from the plate. The impression shall be shaken by hand to remove the excess exudate. A gypsum slurry (3.2.4), under gentle vibration, shall be poured against the impression within 2 minutes from the time the impression is separated from the test block.

The calcined gypsum shall be unmodified alpha calcium sulfate hemihydrate (3.2.4) which is made by calcining gypsum under steam in a closed container. This type of calcined gypsum usually requires about 30 milliliters of water in 100 grams of powder to produce a mix of workable consistency. The gypsum when tested directly against the block (Figure 1) shall reproduce the line 0.050 mm (0.002 in) wide. The poured impression shall be placed in an air bath at 23 ± 2.0° C. (73.4 ± 3.6° F.) and 100 percent relative humidity for 30 minutes. The gypsum cast shall be removed and examined, without magnification, under low-angle illumination with a microscope lamp. The reproduction of the 0.075 mm (0.003 in) line shall be satisfactory if it is continuous for the full width of the ring.

4.3.7 Setting time. A metal ring 3 centimeters inside diameter and 16 millimeters high shall be placed on a flat plate and over-filled with the mixed impression material. The excess impression material shall be struck off level with the top of the ring with the spatula used in mixing. Immediately thereafter the flat end of a polished rod of poly(methyl methacrylate),

6 millimeters in diameter and 10 centimeters long, shall be placed in contact with the exposed surface of the impression material and immediately withdrawn. This contact shall be repeated at 10-second intervals until the end of the rod cleanly separates from the impression material. The time of setting shall be the number of minutes and seconds elapsed from the starting of the mix to the time when the impression material does not adhere to the end of the rod. The average of two tests shall be reported to the nearest 10 seconds.

4.3.8 Working time. A penetrometer, using a cylindrical point 4 millimeters in diameter and a load of 50 grams, and having an indicator sensitive to 0.002 mm (0.0001 in) shall be used for this test. A smooth flat plate shall be placed under the point and a fiducial reading made. A ring 16 m llimeters high and approximately 3 centimeters inside diameter, shall be placed upon the flat plate beneath the point and filled with material mixed in accordance with the manufacturer's instructions. The top surface shall be leveled. One and one quarter minutes from the start of the mix for Type I, Fast Setting, or two minutes from the start of the mix for Type II, Normal Setting, the point shall be placed in contact with the top surface of the material and released. The indicator reading shall be made 10 seconds after release. The difference between the two readings shall be not more than 0.25 mm (0.010 in). All readings shall be made to the nearest 0.02 mm (0.001 in).

4.3.9 Impression of the metal model. The directions which accompany the package (3.3) shall be followed in taking impressions of the metal model (Figure 2) except that the pins and floor of the metal model may be covered with a layer of impression material before the tray, filled with impression material, is pressed into place over the posts of the metal model. (The recesses at the bottom of Posts B, C and D in Figure 2 are optional.) Two minutes from the start of mix, the assembled model and tray shall be placed in a water bath maintained at 37 ± 1° C. (98.6 ± 1.8° F.). Six minutes from the start of mix, the assembly shall be removed from the water and the impression separated from the model with a quick pull. The impression shall not rupture during withdrawal from the metal model. A gypsum

slurry, as specified in 4.3.6 and under gentle vibration, shall be poured immediately into the impression to form the cast. After the dental stone has set for 30 minutes the impression material shall be removed. The surface of the gypsum cast shall be smooth and nonchalky and there shall be no evidence of fins caused by ruptures of the impression. During or after removal from the cast, the impression shall be carefully sectioned to reveal ruptures closed so tightly that they are not otherwise apparent on visual inspection.

4.3.10 Properties in compression.

4.3.10.1 Preparation of specimens. Test specimens shall be made by placing a ring 3 centimeters inside diameter and 16 millimeters high on a flat glass or metal plate and by filling the ring slightly more than one-half full with alginate material mixed according to the manufacturer's instructions. A metal mold 12.7 mm (1/2 in) inside diameter, 25.4 mm (1 in) outside diameter and 19 mm (3/4 in) high shall be placed immediately inside the ring and shall be forced into the material until the mold touches the plate and the material has exuded onto the top of the mold. A flat glass or metal plate shall be pressed on the top of the mold to remove excess material. Two minutes after the start of mix, the mold and its accompanying plates shall be placed in a water bath maintained at 37 ± 1° C. (98.6 ± 1.8° F.). Five minutes and thirty seconds after the start of mix, the mold and plates shall be removed from the water bath.

4.3.10.2 Test conditions. The specimens shall be tested at 23 ± 2° C. (73.4 ± 3.6° F.). During the test the specimen shall be protected by a loosely wrapped moistened cloth gauze to prevent excessive moisture losses.

4.3.10.3 Permanent deformation caused by fixed strain. Six minutes from the start of mix, a specimen, prepared as specified in 4.3.10.1, shall be placed in a suitable instrument consisting essentially of a dial indicator graduated in 0.02 mm (0.001 in), mounted to a stable base and equipped with a screw, positioned in such a manner that sufficient force can be applied to the specimen to produce the required amount of strain (Figure 3). Six

minutes after the start of mix, a lightweight plate shall be placed on top of the specimen and the foot of the dial indicator shall contact the plate. The weight of the plate and the force exerted by the indicator shall be 50 ± 5 grams. The dial indicator shall be read 30 seconds after its foot contacts the plate. This value shall be reading A. The foot of the indicator shall be lowered 2.3 mm (0.090 in) by the screw for 30 seconds, then released and the specimen allowed to rest under no load (except that of the lightweight plate) for 30 seconds. Then the dial indicator shall be lowered onto the plate for 30 seconds and a second reading taken. This value shall be reading B. The difference between readings A and B, divided by the original length of the specimen and multiplied by 100, shall be recorded as the percent permanent deformation. The average permanent deformation of three specimens shall be not more than 3.0 percent.

4.3.10.4 Compressive strength. Eight minutes from the start of mix, a specimen prepared as specified in 4.3.10.1 shall be placed in a suitable machine, accurate to 50 grams, and tested for compressive strength. A piece of bond paper shall be placed under and over the specimen in the machine. The specimens shall be loaded continuously and as uniformly as possible to give an average rate of 10 ± 2 kg (22 ± 4.4 lb) per minute until fracture. The maximum load at fracture shall be recorded to the nearest 50 grams. The maximum load shall be divided by the cross-section area of the mold and reported in grams per square centimeter. The average strength of three specimens shall be not less than 3,500 Gm/cm^2 (50 lb/in^2).

4.5.10.5 Strain in compression. Ten minutes from the start of mix, the specimen prepared as specified in 4.3.10.1 shall be placed in a suitable instrument (Figure 4) and shall be subjected to a load calculated to produce a stress of 100 grams per square centimeter. Thirty seconds later the dial indicator, graduated in 0.02 mm (0.001 in), shall be read. This value shall be reading A. Sixty seconds after application of a stress of 100 grams per square centimeter, an additional load calculated to produce a total stress on the specimen of 1,000 grams per square centimeter shall be gradually applied during an interval of ten seconds. Thirty seconds after

initiation of the stress of 1,000 grams per square centimeter a reading of the dial indicator shall be taken. This value shall be reading B. The difference between readings A and B divided by the original length of the specimen (the original length of the specimen shall be considered as the height of the mold used in forming it), times 100, shall be recorded as the percentage of strain between the stresses of 100 and 1,000 grams per square centimeter. The average strain of three specimens shall be not less than 4.0 nor more than 20.0 percent.

4.3.10.6 Deterioration. Sufficient packages of material to make three specimens (4.3.10.1) shall be stored in original packages for one week at 60 ± 1° C. (140 ± 1.8° F.) and 100 percent relative humidity. At the end of the storage period three specimens shall be made and tested in accordance with 4.3.10.4. The average compressive strength shall be not less than 2,600 Gm/cm^2 (37 lb/in^2).

5. PREPARATION FOR DELIVERY

5.1 Packaging. The material shall be supplied in sealed, airtight containers made of materials which shall not contaminate or permit contamination of the contents.

5.2 Instructions for use. Instructions for use shall accompany each package (3.3).

5.3 Marking

5.3.1 Lot numbers. Each container of material shall be marked with a serial number or a combination of letters and numbers which shall refer to the manufacturer's records for the particular lot or batch.

5.3.2 Date of manufacture. The date of manufacture (year and month) shall be given on the container either as a separate item or as a part of the lot number (5.3.1).

5.3.3 Volume after mixing. The minimum volume after mixing, according to the manufacturer's directions (3.3), shall be given in legible type on the bulk container to the nearest 10 milliliters, and on a container holding an individual impression "unit"

to the nearest 1 milliliter. When the term "unit" is used as a designation on the package, or if the material is supplied in individual "unit" packages, a "unit" shall represent a volume of not less than 56 milliliters when mixed according to the manufacturer's directions. The value shall be reported as the average of tests on three individual impression "units" and shall be recorded to the nearest milliliter.

6. NOTES

6.1 Source of unmodified alpha calcium sulfate hemihydrate. One trade brand of unmodified alpha calcium sulfate hemihydrate is "Hydrocal B-Base" made by the U. S. Gypsum Company, 300 West Adams Street, Chicago 6, Illinois.

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TABLE I

List of Hydrocolloidal Impression Materials Tested

Materials	Manufacturer
Coe Alginate	Coe Laboratories, Inc.
Coe Alginate (Fast Setting)	Coe Laboratories, Inc.
Coe Loid	Coe Laboratories, Inc.
Easy-Mix	Baker Dental Division Englehard Industries, Inc.
Elastic Impression Cream	Dental Perfection Company
Formula 55	The William Getz Corporation
Jeltrate	The L. D. Caulk Company
Jeltrate (Fast Set)	The L. D. Caulk Company
Jeltrate (Heavy Body)	The L. D. Caulk Company
Jeltrate (Inlay)	The L. D. Caulk Company
Kalginate	Lee Smith Company
Key to Alginates	Dental Perfection Company
Kromopan	F. H. Wright Dental Company
Technicol	The G. C. Chemical Mfg. Company
Whip-Mix Alginate	Whip-Mix Corporation
Zelex	The L. D. Caulk Company

TABLE II

Summary of Test Data Obtained by Specification Methods in the Attached Fourth Draft of the Proposed American Dental Association Specification No. for Hydrocolloid Impression Material-Alginate Type (see footnotes for modification, page 23)

Section of Specification	Fineness 3.2.3 4.3.5	Compatibility with Gypsum 3.2.4 4.3.6		Setting Time 3.2.6 4.3.7
Material		(4)	(5)	Min Sec
A	S(2)	Line D	Line C	3:00
B(1)	S	Line D	Line A	2:00
C	S	Line D	Line C	3:10
D	S	Line D	Line A	4:20
E	S	Line E	Line D(6)	2:30
F	US(3)	Line C	Line A	2:40
G(1)	US	Line C	Line B	1:10
H	US	Line E	Line A(7)	3:20
I	S	Line C	Line C	3:00
J	S	Line D	Line B	2:20
K	S	Line D	Line D	3:10
L	S	Line C	Line C	3:00
M(1)	S	Line D	Line C	2:00
N	S	Line C and D	Line C	2:00
O	S	Line D	Line D	3:00
P	S	Line C	Line C	3:00
Requirement in Fourth Draft of Specification (Appendix)	All material shall pass a No. 20 sieve	Must reproduce line C (0.076 mm wide) Fig. 1		Type I Fast Setting Min. 1 min. 0 sec. Max. 2 min. 0 sec. Type II Normal Setting Min. 2 min. 0 sec. Max. 4 min. 30 sec.

TABLE II (Con't.)

Section of Specification	Working Time 3.2.7 4.3.8		Permanent Deformation 3.2.10 4.3.10.3	
Material	mm	in	Fixed strain %	Fixed stress %
A	0.10	0.004	2.1	4.6
B(1)	0.10	0.004	2.0	2.9
C	0.13	0.005	1.9	3.0
D	0.08	0.003	2.6	4.3
E	0.05	0.002	1.7	4.8
F	0.05	0.002	2.3	3.8
G(1)	0.05	0.002	2.3	3.2
H	0.05	0.002	2.4	3.8
I	0.05	0.002	2.0	3.9
J	0.05	0.002	2.8	6.6
K	0.08	0.003	1.5	4.3
L	0.05	0.002	1.9	4.3
M(1)	3.69(8)	0.145	1.5	3.6
N	0.10	0.004	2.9	3.8
O	0.13	0.005	1.8	3.3
P	0.13	0.005	2.2	3.7
	Not more than 0.25 mm between readings at the end of 2 min. (Type II Normal Setting), or 1 1/4 min. (Type I Fast Setting)		3.0% on 6 min. old specimen	

TABLE II (Con't.)

Section of Specification	Compressive Strength 3.2.11 4.3.10.4(9)				Strain in Compression 3.2.12(10) 4.3.10.5
Material	Gm/cm^2	Kg/min.	Gm/cm^2	Kg/min.	%
A	5300	8.5	5410	20.5	15.5
B(1)	5150	8.8	5650	24.9	13.7
C	6370	9.8	6340	25.4	14.1
D	2580	6.2	2710	14.3	15.2
E	2780	6.5	2970	15.8	17.3
F	6620	12.1	6750	30.8	13.3
G(1)	6740	12.4	7430	29.8	12.1
H	6160	13.8	6720	33.0	11.3
I	9430	14.5	9840	34.6	17.6
J	6540	9.1	8300	22.8	19.8
K	10930	15.9	12900	44.2	17.7
L	10030	13.6	11580	40.1	17.9
M(1)	7160	11.9	7700	32.9	15.9
N	6250	13.0	6690	33.9	11.2
O	5860	11.6	5940	28.4	14.3
P	4520	9.2	4810	23.9	14.3
	3500 Gm/cm^2(10 ± 2 Kg/min) on 8 minute old specimen				Min - 4% Maximum - 20% on 10 minute old specimen

TABLE II (Con't.)

Section of Specification	Deterioration-Compressive Strength			
	3.2.13		4.3.10.6(11)	
Material	Gm/cm^2	Kg/min.	Gm/cm^2	Kg/min.
A	1920	5.2	2080	13.5
B(1)	2350	6.3	2450	15.2
C	2690	6.4	2930	17.1
D	1530	4.4	1550	9.3
E	2010	5.3	2440	13.6
F	830	2.6	840	6.3
G(1)	2560	6.3	2650	17.4
H	5290	14.1	4790	30.4
I	6180	10.7	6370	26.5
J	3070	5.6	3420	14.5
K	4330	8.5	5200	21.6
L	5550	9.6	6220	24.9
M(1)	3790	8.1	3990	18.7
N	4010	10.7	4280	29.7
O	3500	9.1	3710	22.4
P	270	1.3	260	3.0

2600 Gm/cm^2 (10 ± 2 kg/min.)

Footnotes

1. Type I - Fast Setting

2. S = Satisfactory

3. US = Unsatisfactory

4. Impression was blotted with absorbent paper to remove surface exudate prior to pouring cast without vibrating the gypsum slurry.

5. Impression was shaken to remove surface exudate prior to pouring the cast with vibration of the gypsum slurry.

6. Line A is discernible when the impression is soaked in a fixing solution for 10 minutes as recommended by the manufacturer.

7. Immersed in primer solution for 1 1/2 minutes as in manufacturer's directions.

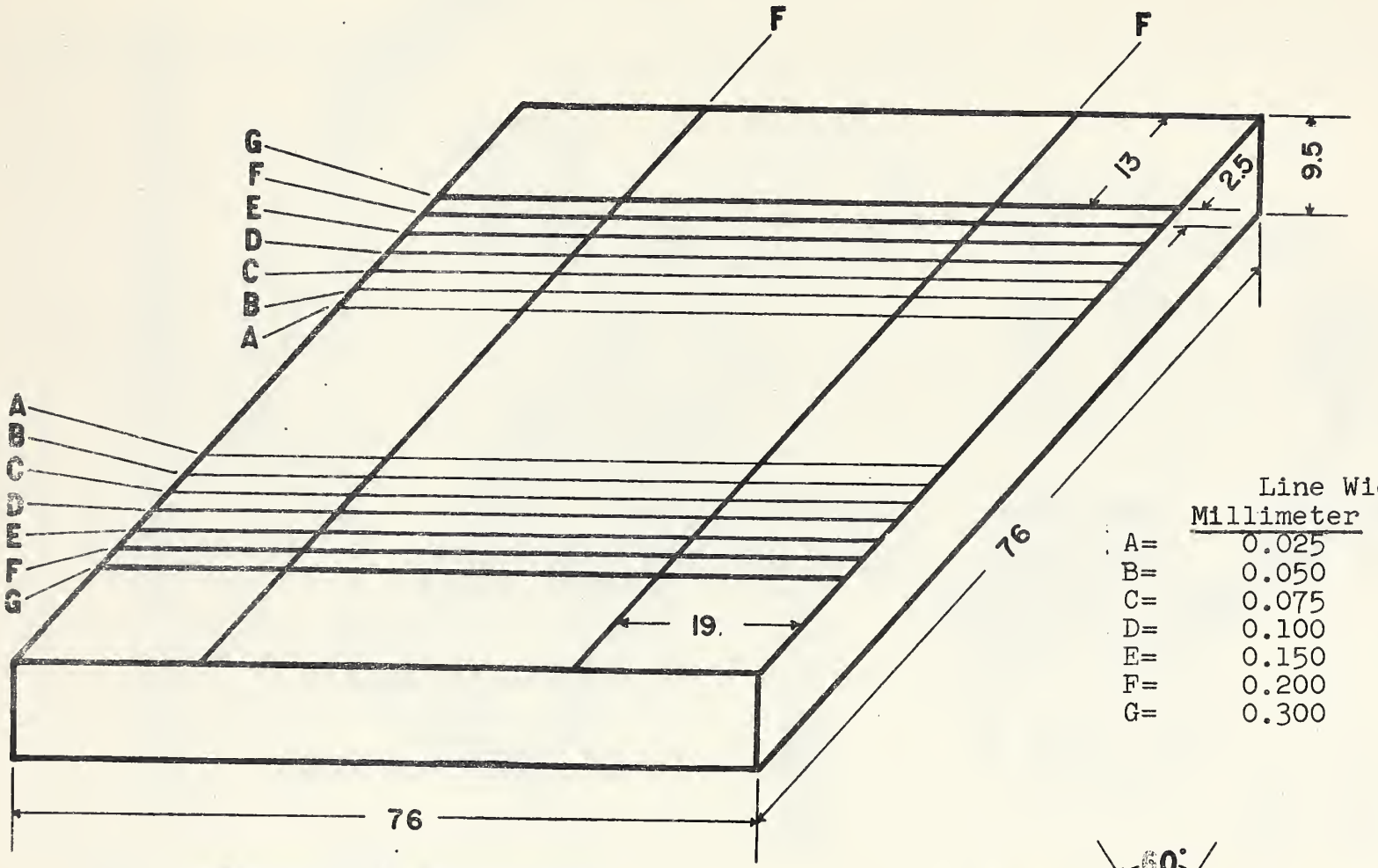
8. Material M was not labled Fast Setting but complied with Type I Fast Setting and did not comply with Type II Normal Setting. The reading when tested as a Type I Fast Setting was 0.18 mm (0.007 in).

9. The values for compressive strength were on six- not eight-minute-old specimens as the proposed specification now calls for; hence the values given should be lower than those values derived on specimens eight minutes old. Average rate of loading is given.

10. The values for strain in compression were on six- not ten-minute-old specimens as the proposed specification now calls for; hence the values given should be higher than those values derived on specimens ten minutes old.

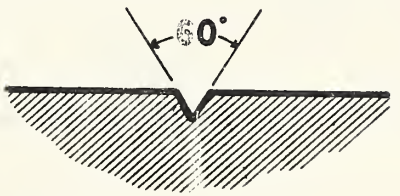
11. The values for compressive strength of the deteriorated material were on six-not eight-minute-old specimens as the proposed specification now calls for; hence the values given should be lower than those values derived on specimens eight minutes old. The average rate of loading is given.

BLOCK FOR DETAIL REPRODUCTION



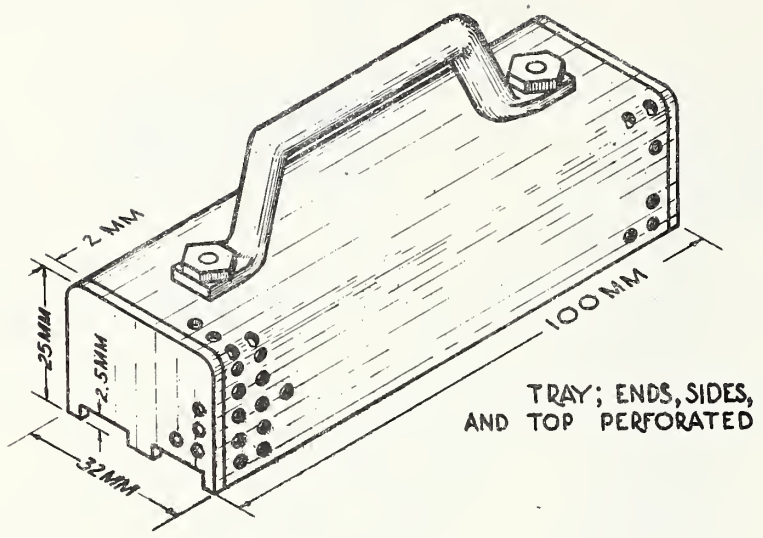
ALL DIMENSIONS IN MILLIMETERS

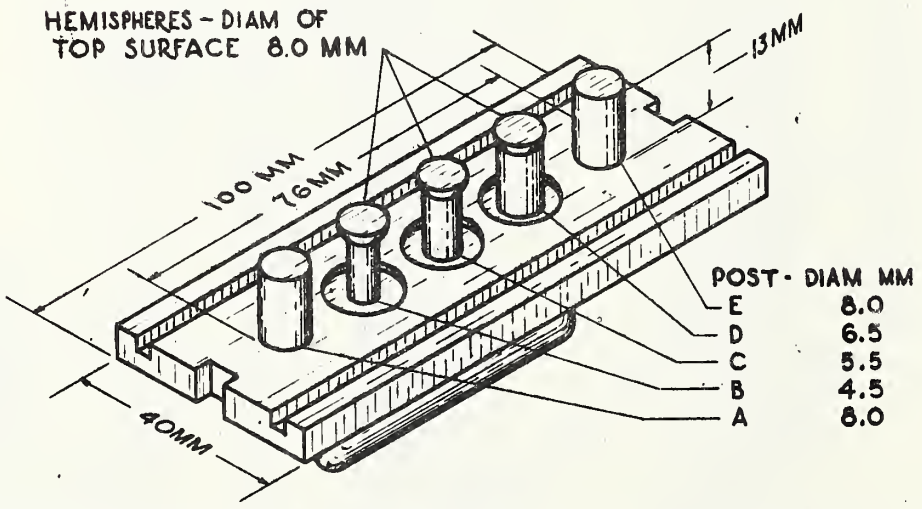
	Line Width Millimeter	(Inch)
A=	0.025	(0.001)
B=	0.050	(0.002)
C=	0.075	(0.003)
D=	0.100	(0.004)
E=	0.150	(0.006)
F=	0.200	(0.008)
G=	0.300	(0.012)



CROSS SECTION OF RULED LINES

Figure 1.





Recesses at Bottom of Posts B, C, and D are optional

MASTER METAL MODEL
FOR TESTING HYDROCOLLOID IMPRESSION MATERIALS
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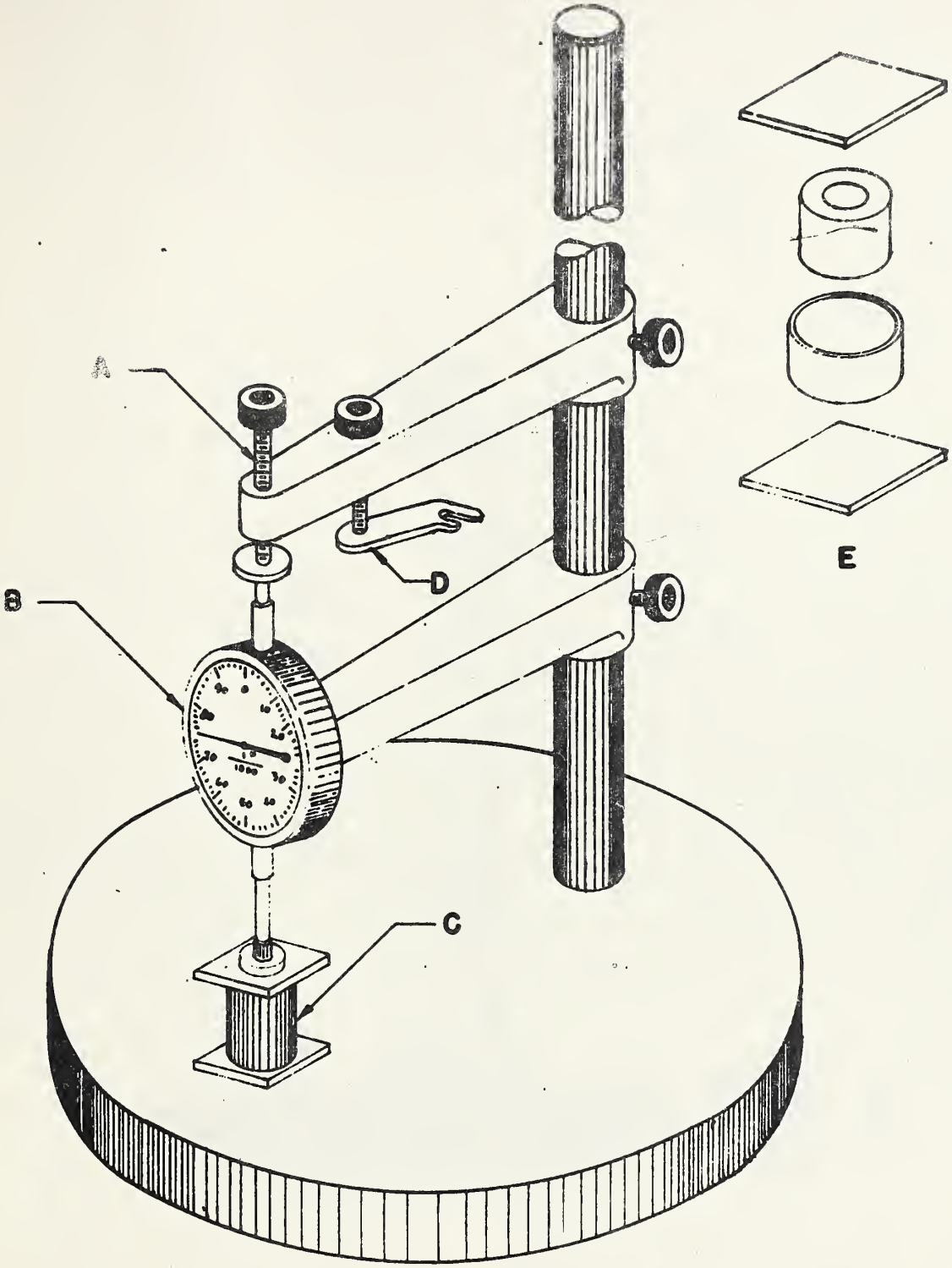
Figure 2.



INSTRUMENT FOR MEASURING PERCENT OF SET

Figure 3

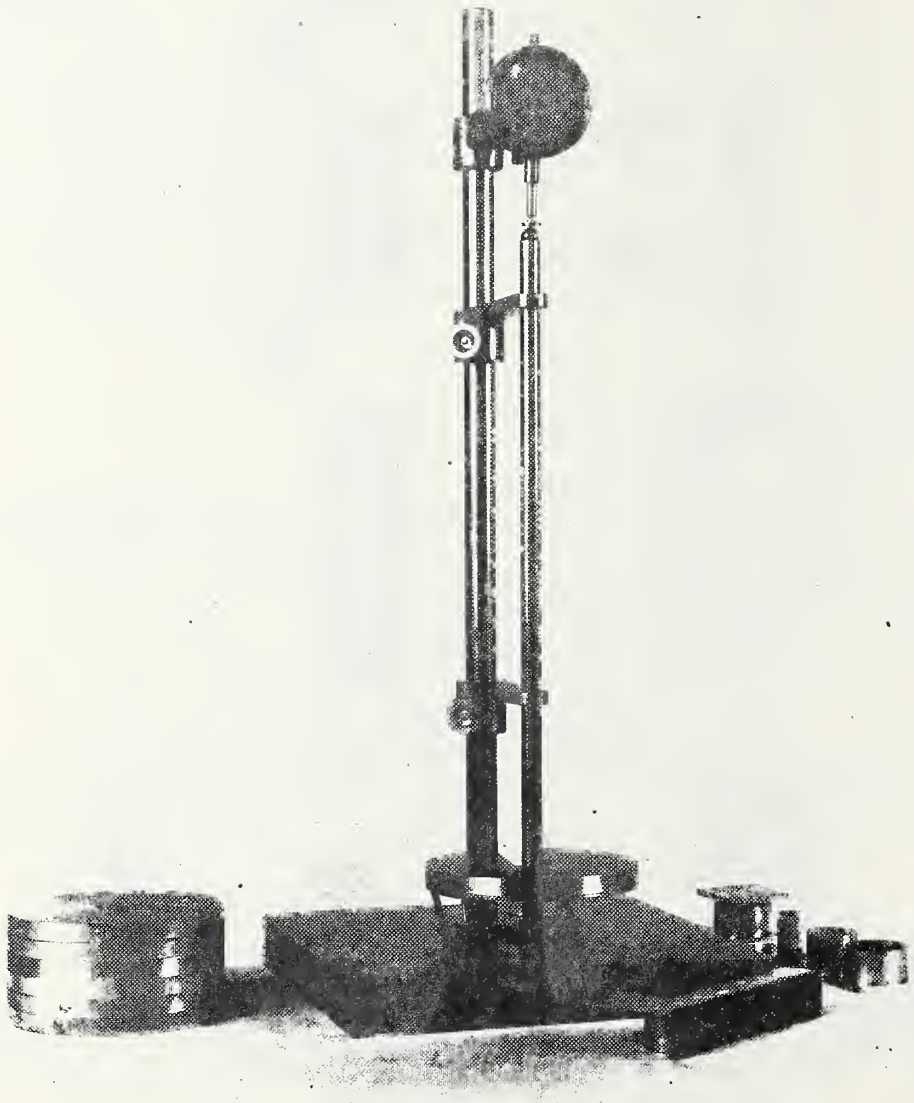


Figure 4