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*Guo Paffenbarger*

# **NATIONAL BUREAU OF STANDARDS REPORT**

4796

REPORT ON DENTAL RESEARCH  
AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

January 1 to June 30, 1956

Dental Research Laboratory



**U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS**

# U. S. DEPARTMENT OF COMMERCE

Sinclair Weeks, *Secretary*

## NATIONAL BUREAU OF STANDARDS

A. V. Astin, *Director*



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**Optics and Metrology.** Photometry and Colorimetry. Optical Instruments. Photographic Technology. Length. Engineering Metrology.

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**Atomic and Radiation Physics.** Spectroscopy. Radiometry. Mass Spectrometry. Solid State Physics. Electron Physics. Atomic Physics. Nuclear Physics. Radioactivity. X-rays. Betatron. Nucleonic Instrumentation. Radiological Equipment. AEC Radiation Instruments.

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**Metallurgy.** Thermal Metallurgy. Chemical Metallurgy. Mechanical Metallurgy. Corrosion.

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**Applied Mathematics.** Numerical Analysis. Computation. Statistical Engineering. Mathematical Physics.

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● Office of Basic Instrumentation

● Office of Weights and Measures

### BOULDER, COLORADO

**Cryogenic Engineering.** Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Gas Liquefaction.

**Radio Propagation Physics.** Upper Atmosphere Research. Ionospheric Research. Regular Propagation Services.

**Radio Propagation Engineering.** Frequency Utilization Research. Tropospheric Propagation Research.

**Radio Standards.** High Frequency Standards Branch: High Frequency Electrical Standards. Radio Broadcast Service. High Frequency Impedance Standards. Microwave Standards Branch: Extreme High Frequency and Noise. Microwave Frequency and Spectroscopy. Microwave Circuit Standards.

# NATIONAL BUREAU OF STANDARDS REPORT

## NBS PROJECT

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0708-20-3824  
0708-20-3857

## NBS REPORT

July 24, 1956

4796

### REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

#### Progress Report

January 1 to June 30, 1956

Dental Research Laboratory

The dental research program at the National Bureau of Standards is carried on in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Service, the Navy Dental Corps, and the Veterans Administration.

(Task 7756-41 BV No. 55-14)



## U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

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REPORT ON DENTAL RESEARCH  
AT THE NATIONAL BUREAU OF STANDARDS

1. INTRODUCTION

Research on materials and equipment used in dentistry and studies of natural tooth structures have been continued in the Dental Research Section of the National Bureau of Standards during the half year ending June 30, 1956. Lists of reports issued and papers published and summaries of work in progress during this period are given below. Copies of the reports are appended.

2. REPORTS ISSUED

NBS Report 4585	A Proposed Specification for Dental Chromium-Cobalt Casting Alloys.
NBS Report 4635	A Panoramic Dental X-ray Machine.
NBS Report 4660	A Simple Technic for Making Dental Porcelain Jacket Crowns.
NBS Report 4702	Some Changes in the Properties of Agar-Agar Type Duplicating Material and Agar-Agar on Heating.
NBS Report 4703	Some Physical Properties of Gallium-Copper-Tin Alloys.

3. PAPERS PUBLISHED

Sulfinic Acid Derivatives as Accelerators in the Polymerization of Methyl Methacrylate. G. M. Brauer and F. R. Burns. J. Polymer Sci. 19:311 Feb. 1956.

Research on Dental Materials. Ralph W. Phillips and George C. Paffenbarger. J.A.D.A. 52:426 April 1956.

Resin Cements and Posterior Type Direct Filling Resins. P. J. Schouboe, George C. Paffenbarger and W. T. Sweeney. J.A.D.A. 52:584 May 1956.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Investigation.

Studies on tooth sections of the relationship between fluorescence intensity and organic content showed that the fluorescence intensity decreased as either the organic or mineral content was removed, the decrease being more marked upon removal of organic material. In neither case was the fluorescence extinguished. Experiments are being continued on powdered enamel and dentin to secure more homogeneous specimens.





The comparison of artificial and natural caries carried out in cooperation with the National Naval Medical Research Institute has been completed. Study of the artificially produced caries and natural caries by ordinary light, polarized light, and ultraviolet technics revealed marked similarities including the following: (1) gross appearance of a white spot lacking fluorescence, (2) alteration of fluorescence and birefringence in the lesion areas as seen in thin sections, (3) accentuation of the enamel rods, their cross striations and the Retzius lines, and (4) serration at the degradation front.

(b) Crystallographic Investigation.

Detailed x-ray diffraction structure analyses were performed on a non-stoichiometric lead apatite and a perfect lead apatite,  $\text{Pb}_{10}(\text{PO}_4)_6(\text{OH})_2$ , to obtain information on the non-stoichiometric apatite thought to be present in tooth structure. It was found that the pure lead apatite was isomorphous with other apatites. In the case of the non-stoichiometric salt, the best fit for the x-ray diffraction data was found when it was assumed that cations were missing statistically from the "columnar" positions in the apatite structure. These results were confirmed with index of refraction studies.

A complete simple crystal x-ray diffraction analysis of hydroxyapatite was started. This is a long process necessitating months of data collection and calculation. The results should give an exact determination of the arrangement in space of the calcium, phosphorous and oxygen ions in the structures of hydroxyapatite,  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ .

Studies of lysine-deficient rat bone were made in cooperation with workers at the National Institute of Dental Research. Crystallographic results showed the bone apatite particle size of the epiphyses and shanks of the tibias of normal and lysine-deficient bone to be about 300A. This is in contrast to the expectation from in vivo calcification results that the growing ends of the bones would have smaller crystals.

(c) Mechanical Properties.

Values for modulus of elasticity, elastic limit and compressive strength of enamel and dentin have been determined. Because of the very small size of the specimens of tooth structure it has been necessary to apply to the values obtained empirical corrections based on data obtained for small specimens of materials of known properties. Corrected values are given in the following table.





<u>Structure</u>	<u>Physical Properties</u>		
	Modulus of Elasticity	Elastic Limit	Compressive Strength
	psi	psi	psi
Enamel			
Tips of cusps	8.2x10 <sup>6</sup>	34,000	40,000
Sides of teeth	6.3	21,000	28,000
Occlusal surface	4.0	15,000	18,000
Dentin	4.1	25,000	50,000

#### 4.2 Metals

##### (a) Gallium alloys.

Detailed information on the investigation of gallium-copper-tin alloys is given in the appended report. Additional data on these alloys indicate that they may not be equal to dental amalgam in corrosion resistance. Alloys believed to be more resistant to corrosion have been made of gallium with gold-tin and with palladium-tin and are being studied.

Apparatus has been designed and constructed for the determination of the strength of gallium and amalgam alloys at mouth and higher temperatures.

##### (b) Amalgam.

The studies of the Sn-Hg binary system by diffusion techniques have been continued and have provided additional evidence for the composition of the  $\delta$  phase in a previously blank region of the diagram. Electrode potential measurements and diffusion tests utilizing single crystals of tin have been started to confirm these findings and to eliminate the possibility of alternative explanations of the data.

A test for the setting time of dental amalgam has been developed. The setting of amalgam is a complex but continuous metallurgical process passing through a number of as yet poorly defined states. Any setting time test must therefore involve an arbitrary selection of a limiting value for some property. This limiting value should correlate well with some clinical characteristic. For the test developed, the clinical characteristic selected is the "limit of carvability," the amalgam consistency at which the average dentist feels that further carving embodies an excessive risk of fracture. A punch test approximating pure shear loading was selected to measure this property. Amalgam specimens approximately 0.70 inch in diameter and 0.028 inch thick are formed in a die under a load of 2000 psi. The





load required to punch 1/4 inch holes in the centers of the specimens at various times after the start of mixing is determined. The results of a large series of hand carving tests made on similar specimens indicates that the clinical limit of carvability is approximated closely by that time at which the load required for punching is 25.5 pounds. Additional studies of factors which may affect the test such as the rate of loading of the punch are in progress.

#### 4.3 Resins

Investigations of the relative inhibiting efficiency of typical inhibitors towards cyanopropyl radicals was resumed. Measurements of the "scavenging power" of inhibitors in air were completed. Difficulties were encountered in measuring the rate of free radical formation from azo-bis-isobutyronitrile (AIBN) with 2,2 diphenyl -1- picrylhydrazyl (DPPH) in a vacuum since the reaction is greatly influenced by the presence of traces of oxygen. The apparent rate of free radical formation from AIBN is greatly decreased in a vacuum probably due to the non-stoichiometric reaction between cyanopropyl and DPPH radical. Preliminary results indicate that the relative inhibiting efficiency of inhibitors as compared to DPPH is not altered greatly in a vacuum. Additional studies of the rate of free radical formation of the benzoyl peroxide-dimethylaniline were made in a large number of solvents. Both the kinetics of free radical formation and stoichiometry of the reaction between the free radicals formed and the DPPH vary in different solvents.

Epoxy resins are being explored for possible use as direct filling resins since some of their properties, particularly that of adhesiveness, should be advantageous. Hardening time of two epoxy resins was measured using organic and inorganic acid hardening agents, various plasticizers and fillers. Combinations giving practical room temperature hardening times were tested for water sorption, solubility and compressive strength. Without fillers one resin, hardened with phosphoric acid, had higher water sorption and lower apparent solubility than polymethyl methacrylate. Another combination containing approximately 60% powdered fused quartz and 37% epoxy resin hardened with 3% phosphoric acid had a compressive strength of approximately 16,000 pounds per square inch.

#### 4.4 Elastic Impression Materials

Additional properties of rubber base impression materials, both thiokol and silicone types, have been determined. Temperature rise on setting of these materials varied from approximately 3° to 5°C. Shrinkage of the thiokol materials over a 24-hour period was 0.03% to 0.2% and that of the silicone material was about 0.5%. Other properties of the silicone material appeared to be equal or superior to those of the thiokol products. One of





the greatest <sup>dis</sup>advantages of the rubber base materials is the difficulty in obtaining uniform mixes of sufficient quantity for large impressions.

A detailed report on the deterioration of agar-agar duplicating materials is appended.

#### 4.5 Thermal Expansion

The determination of the coefficient of thermal expansion of various dental materials has been initiated. An interferograph previously developed at the National Bureau of Standards is being employed. Dental porcelains used in the fabrication of jacket crowns and self-curing and heat-curing denture base resins of both the gel and powder-liquid types have been studied. Linear thermal expansion coefficients obtained for the various denture base resins are approximately  $90$  to  $100 \times 10^{-6}$  per  $^{\circ}\text{C}$  from room temperature to  $85^{\circ}\text{C}$ .

#### 4.6 Panoramic X-ray Machine

A report on the panoramic x-ray machine is attached. The machine is now being tested clinically by the Air Force.

#### 4.7 Color Standards

Fundamental exploratory investigation into the colors of various types of dental materials, especially silicate cements, has demonstrated the need for standard specifications of color. The random numbering of shades now used and the irregular spacing of these shades on the ICI (International Commission on Illumination) diagram have further emphasized the advantages of using this generally accepted method of chromaticity designation. In this study, a Gardner Automatic Color Difference Meter has been used for all color measurements. These have included not only the determination of the color of various shades of cement, but also the change of color with different powder-liquid ratios and the change of color with age. In addition to the work currently in progress, a study of other dental materials such as denture base resins and plastic and porcelain teeth is anticipated. The color determination of a wide range of natural teeth is also planned.

#### 4.8 Zinc Oxide Materials

Investigation of the reaction of zinc oxide with typical chelating agents which are liquids at room temperature were studied. Cements which show some adhesive properties and good crushing strength and which exert no inhibiting effects on the setting of acrylic restorations were obtained from zinc oxide and O-ethoxybenzoic acid. On substitution of barium oxide, lead oxide, mercuric oxides, cadmium dioxide, magnesium oxide, calcium hydroxide and mercuric chloride for zinc oxide hard cements are also formed.





Reagent grade zinc oxide-ethoxybenzoic acid mixtures harden within 25 minutes. The reaction is speeded considerably on incorporation of magnesium oxide. The water solubility and physical properties of the products are under active investigation.

#### 4.9 Rotating Cutting Instruments

An instrument for determining the developed torque in dental handpieces has been designed and built. The instrument is based upon the phenomenon of eddy currents produced when a non-magnetic conductor passes through a non-uniform magnetic field. In this instrument a thin aluminum disk is rotated through a magnetic field parallel to its axis of rotation. The eddy currents produced result in a torque which resists the rotation of the disk. This disk is attached to a dental handpiece; therefore, the torque is directly transmitted to the handpiece.

The source of the magnetic field is a D-shaped magnet in which the two ends are separated by a 1 mm slot. The field is varied by means of a current-carrying coil wrapped around the core of the magnet. The magnet is balanced on two supports so that the torque acting upon the magnet tends to rotate it. The amount of elongation of a calibrated spring used to return the magnet to its equilibrium position may be related directly to the torque produced. The speed at which the handpiece is rotating is also to be determined. A frequency meter has been constructed for this purpose.

#### 4.10 Dental Materials Syllabus

The preparation of a documented syllabus of the dental materials field is in progress. The chemical and physical characteristics of dental materials and their relation to clinical dentistry are covered. The syllabus is primarily designed for presenting the science of dental materials to military dental officers. It may be used as a teaching outline or for a ready reference review guide. Documentation is accomplished through the inclusion of selected references to significant contributions published in American literature.

#### 4.11 Evaluation of Materials

Materials and equipment evaluated for the Federal dental services and the American Dental Association by specification or special test methods included amalgam, denture base resin, denture base repair resin, impression compound, inlay casting gold alloy, inlay casting wax and an oxygen regulator.

For the Director  
by

W. T. Sweeney, Chief  
Dental Research Section



# NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

0708-20-3824

March 28, 1956

4585

## Progress Report

### A PROPOSED SPECIFICATION FOR DENTAL CHROMIUM-COBALT CASTING ALLOYS

by

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This work is a part of the dental research program conducted at the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Service, the Navy Dental Corps and the Veterans Administration.

(Task 7756-41 BV No. 55-14)



**U. S. DEPARTMENT OF COMMERCE**  
**NATIONAL BUREAU OF STANDARDS**

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# A PROPOSED SPECIFICATION FOR DENTAL CHROMIUM-COBALT CASTING ALLOYS

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## Abstract

The widespread use of chromium-cobalt casting alloys in the construction of partial dentures has presented a need for a specification for alloys of this type. The major requirements of the specification proposed herein are a composition of not less than 85% chromium, cobalt and nickel, yield strength of 60,000 psi, tensile strength of 90,000 psi, elongation of 1.5%, and hardness of 50 (Rockwell 30N). The alloys are divided into two types, one with a minimum fusion temperature of 2400°F and one with a maximum fusion temperature of 2400°F.

Chromium-cobalt base alloys were introduced for dental use in 1929 [1], and since that time have shown gradual and steady gains in popularity. While the desirable physical properties of these materials and their relatively low cost have promoted their use, the necessity of using specialized equipment and techniques has restricted their application almost entirely to commercial dental laboratories. In recent years, as dentists have come to rely more upon the services of commercial laboratories and of auxiliary personnel in general, the chrome-cobalt alloys have nearly preempted the partial denture field.





The increasing popularity of these materials has prompted the appearance on the market of many new products of this type which exhibit wide variations in their suitability for dental use. As the number of available products has multiplied, a growing need has been felt by the dental profession and the laboratories for a standard or specification as an aid in the selection of a material. The interest of the Federal dental services has been particularly pronounced since such a specification would facilitate their purchase of these alloys.

Much of the research on the properties of these materials has been directed toward properties which are of interest for their industrial use, such as creep resistance, but which have little pertinence for their dental application. Several groups of investigators have worked with the dental alloys in the last twenty years, but few results have found their way into the literature, and as yet there is no accepted standard for these materials.

The accompanying specification is submitted as representing an initial approach to this problem, and has been approved by the Specifications Committee of the Dental Materials Group of the International Association for Dental Research. The values proposed in this specification are based upon the work done by Paffenbarger, Caul, and Dickson [1], and more recently by Taylor, Leibfritz, and Adler [2] at the National Bureau of Standards, and upon that done at the University of Michigan by Bush, Ingersoll, Mahler and Peyton [3]. The tensile test specimen design and spruing arrangement recommended are essentially those developed



by the Michigan group.

As far as possible the following principles were observed in drafting this specification:

1. The values specified should be selected to eliminate materials having properties inferior to those readily obtainable in currently available products, without at the same time excluding products of known clinical acceptability.
2. The specification should limit the variation in composition and properties to types within which acceptable products may be used interchangeably without major alterations in equipment or technique.
3. The specification should parallel as far as possible the existing specifications for other materials used for the same purpose [4].
4. The test methods specified should be selected to simplify testing and reduce the required interpretation of the observations as far as possible without sacrificing accuracy or thoroughness.

Certain of the individual requirements of the specification made on this basis would seem to merit further discussion.

This specification divides the chromium-cobalt base dental casting alloys into two types on the basis of their liquidus temperatures. The dividing temperature has been taken as 2400°F on the basis of advice from the manufacturers of investments for these materials that alloys melting above 2400°F cannot be cast with routine success into gypsum-bonded investments. Since the techniques required for the water-mixed, gypsum-bonded investments.





differ greatly from those employed for materials utilizing ethyl silicate or some other high temperature binder, this seemed to constitute a logical basis for a division into separate types.

The composition requirement that the alloy contain no less than 85% by weight of chromium, cobalt, and nickel serves two purposes. It defines the general group of alloys considered by the specification somewhat more closely than does the title, and at the same time it is an attempt to compensate for the lack of a satisfactory test for corrosion resistance under oral conditions. In the absence of such a test it is felt that this composition requirement gives some assurance of corrosion resistance without unduly imposing penalties in the form of reduced mechanical properties. All widely used, clinically successful alloys meet this requirement without difficulty.

The specimen design used for the determination of tensile properties was selected as approximating dental dimensions, and, on the basis of the results of Bush et al, as showing greater uniformity of properties and freedom from casting failure than other designs. The chrome-cobalt alloys are particularly hard to cast in the rod form used for testing dental golds.

The numerical values required for the tensile properties correspond approximately to those of Federal Specification QQ-G-540 for dental casting golds, type IV. The requirements for yield strength and elongation are set slightly lower than those for golds since some base metal alloys of known clinical suitability are unable to meet those requirements. The hardness test



requirement is in terms of a Rockwell superficial hardness scale (30N), rather than the Brinell test employed for golds, because the diamond indenter used in the Rockwell test is not subject to the deformation that occurs in the ball indenter of the Brinell tester when used on these hard materials.





# BIBLIOGRAPHY

1. Paffenbarger, George C., Caul, H. J. and Dickson, George. Base metal alloys for oral restorations. J.A.D.A. 30, 852-862 June 1943.
2. Taylor, Duane F., Leibfritz, Walter A., and Adler, Alfred G. Physical properties of chromium cobalt dental alloys. (In preparation).
3. Bush, S. H., Ingersoll, C. E, and Peyton, F. A. Navy contract N6-Onr-232 Progress Reports, School of Dentistry, University of Michigan.
4. Federal Specification QQ-G-540 for Gold; Casting, Inlay, Dental.



PROPOSED SPECIFICATION FOR  
DENTAL CHROMIUM-COBALT CASTING ALLOYS

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification covers chromium-cobalt casting alloys of the type used in removable partial denture prostheses.

1.2 Types. The chromium-cobalt alloys covered by this specification shall be of the following types:

Type I. High fusing.

Type II. Low fusing.

2. APPLICABLE SPECIFICATIONS

2.1 Specification. There are no other specifications applicable to this specification.

3. REQUIREMENTS

3.1 Color. The color of the alloy shall be the color specified by the purchaser.

3.2 Composition. The alloy shall contain a total of not less than 85% by weight of chromium, cobalt and nickel.

3.3 Physical properties. The requirements for physical properties for each type are listed in Table 1.

Table 1

REQUIREMENTS FOR PHYSICAL PROPERTIES

Type	Fusion tempera- ture	Yield strength	Tensile strength	Elongation (one inch gage length)	Hardness Rockwell 30N
	°F	psi	psi	%	
		min.	min.	min.	min.
I	2400 (min.)	60,000	90,000	1.5	50
II	2400 (max.)	60,000	90,000	1.5	50





3.4 Manufacturer's directions. Instructions for use shall accompany each package. The instructions shall include casting temperature of the alloy, burnout temperature for the investment, a description of necessary auxiliary materials and equipment, and any special precautions for satisfactory use.

#### 4. SAMPLING, INSPECTION, AND TEST PROCEDURES

4.1 Sampling. The manufacturer shall provide a set of six specimens of the type described in 4.4.1, cast from each of the lots or batches of alloy included in the delivery. The specimens shall be submitted in the "as cast" condition and with sprues and buttons attached. An additional six ounces of material shall be chosen at random from each lot or batch of material included in the delivery.

4.2 Inspection. Compliance with the requirements outlined in 3.1, 3.4 and 5, shall be determined by inspection.

4.3 Test procedures.

4.3.1 Composition. At the option of the purchaser, certification by the manufacturer that the alloy contains not less than 85% of chromium, cobalt, and nickel may be considered to satisfy the composition requirement. Otherwise the composition shall be determined by wet analysis\*. The determined values for metallic constituents shall be recorded as the nearest 0.5 percent. When a determined value falls midway between a half and a whole number, the whole number shall be recorded.

\* Analyses for chromium, nickel and cobalt shall be made by procedures given in J. Research NBS 53,353 (1954), ASTM Methods of Chemical Analysis of Metals (1950) Chemical Analysis of Iron and Steel, by Lundell, Hoffman and Bright, or by methods demonstrated to be of equal accuracy.



4.3.2 Fusion temperature. The fusion temperature shall be the liquidus temperature as determined by cooling curves. The manufacturer shall determine and certify the fusion temperature. The purchaser may, at his option, accept such certification as satisfying the fusion temperature requirement.

4.3.3 Physical properties.

4.3.3.1 Preparation of specimens. Physical properties other than fusion temperature shall be determined on cast specimens tested in the "as cast" condition without subsequent heat treatment. The specimens shall be of the type and dimensions indicated in Figure 1. They shall be cast in accordance with the manufacturer's directions for partial dentures except that where those directions conflict with the provisions of this specification, the specification shall be followed. It is suggested that two, three or four such patterns be symmetrically arranged around a central sprue, and be provided with gates, risers, and vents in a manner similar to that indicated in Figure 2. If so desired, a continuous ring of adequate cross-section may be substituted for individual risers.

4.3.3.2 Compliance. An alloy shall be considered to meet the requirements for tensile properties and hardness when all of the recorded values for three or more of a set of six specimens meet the requirements of Table 1.





4.3.3.3 Yield Strength. The yield strength shall be determined using an averaging extensometer and a one-inch gage length. An initial load calculated to produce a stress of 5000 pounds per square inch shall be applied to the specimen and the strain gage read. The load shall then be increased to produce a stress of 60,000 pounds per square inch and a second reading taken. The load shall be applied at a rate of not less than 50 nor more than 100 pounds per minute. The maximum strain permitted between the limits specified shall be 0.0025 inch per inch. The determined value of strain shall be recorded to the nearest 0.0001 inch per inch. When the determined value falls midway between two numbers, the even number shall be recorded.

4.3.3.4 Tensile strength. After the loading for the yield strength (4.3.3.3) has been completed and the strain gages have been removed, additional load shall be applied to the specimen until rupture occurs. The determined value for tensile strength shall be recorded to the nearest 1000 pounds per square inch. When the determined value falls midway between two numbers, the even number shall be recorded.

4.3.3.5 Elongation. The elongation shall be determined on the specimens used in the tests for yield strength and tensile strength. Elongation shall be measured on a one-inch gage length. Castings that break outside of the gage marks shall not be repulled, but the elongation between the marks shall



be recorded if it equals or exceeds the minimum specified value. The determined value shall be recorded to the nearest 0.1 percent. When the determined value falls midway between two numbers the even number shall be recorded.

4.4.6 Hardness. The Rockwell 30 N number shall be determined on each tensile specimen. After fracture the tensile specimens shall have a flat surface wet ground on their rod portions. Five indentations shall be made on the flat surface of each specimen no nearer than one-eighth inch from the fractured end or from the tapered end. The high and the low readings shall be discarded and the remaining three shall be averaged. The average hardness number of each specimen shall be recorded to the nearest whole number. When the determined value falls midway between two numbers, the even number shall be recorded.

## 5. PREPARATION FOR DELIVERY

- 5.1 Packaging. The alloy shall be packaged in accordance with acceptable commercial practice.
- 5.2 Marking. Each package shall be marked with the name of the manufacturer or contractor, name of the material, type and minimum net weight of contents, together with a serial number or combination of letters and numbers which shall refer to the manufacturer's records for the particular lot or batch of alloy. The date of manufacture shall be indicated either as a part of the serial number or as a separate item.
- 5.3 Instructions for use. Instructions for use shall accompany each package (3.4).





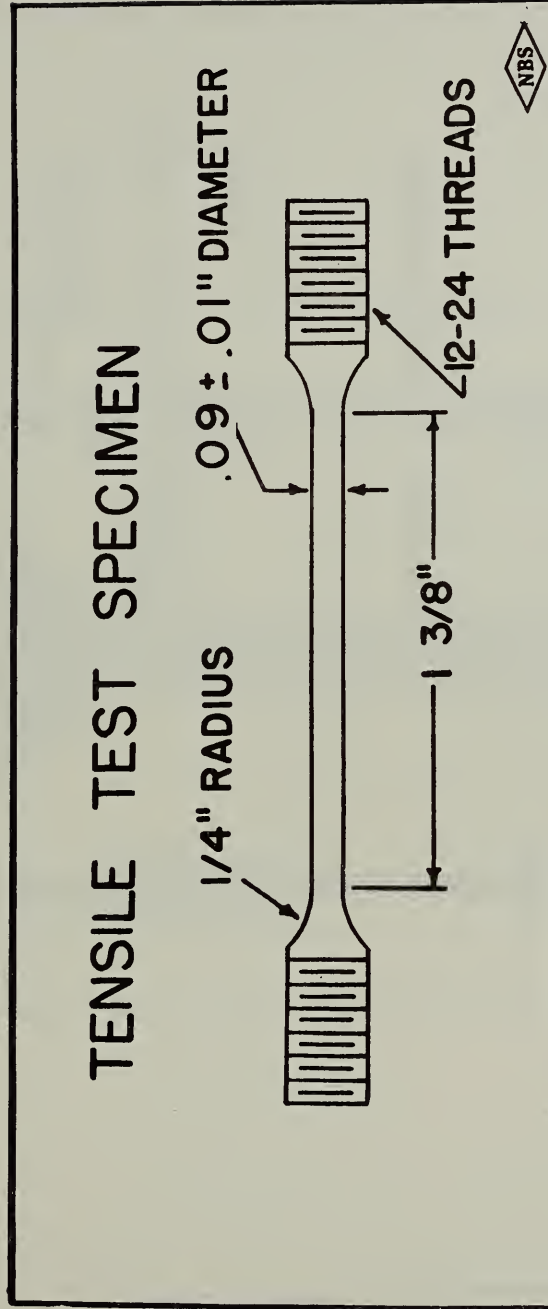


FIGURE 1



# MOLD DESIGN FOR TENSILE SPECIMENS

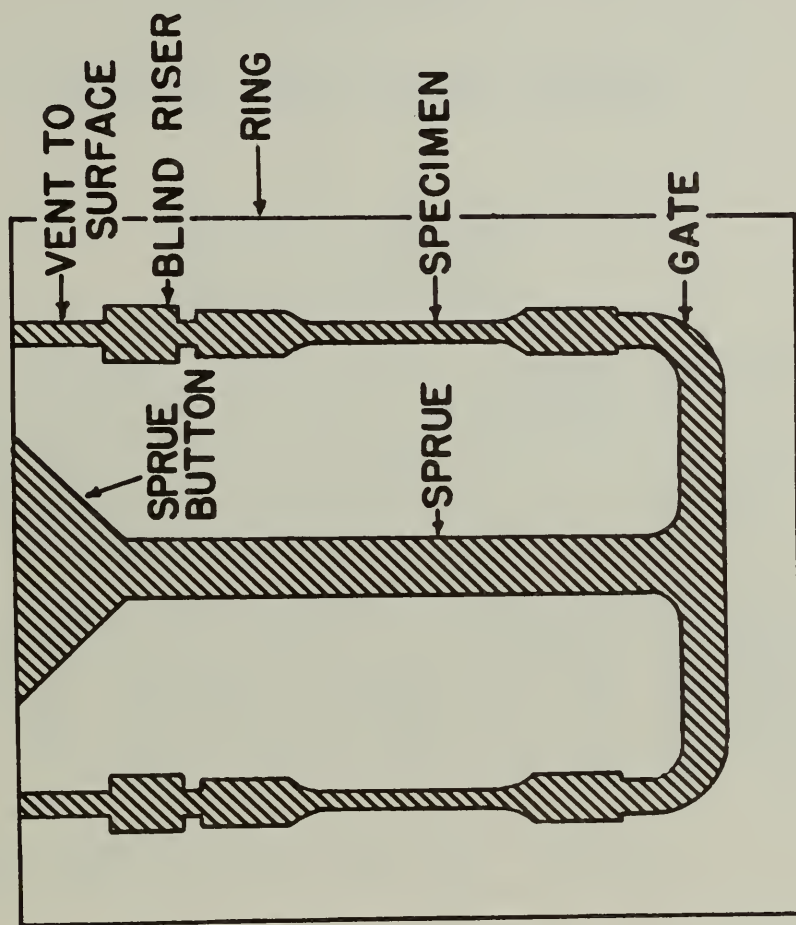


FIGURE 2





# NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

NBS REPORT

0708-20-3857

May 2, 1956

4635

## Progress Report

### A PANORAMIC DENTAL X-RAY MACHINE

by

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- \*\* Laboratory Mechanic, Dental Research Section, National Bureau of Standards.
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This work is a part of the dental research program conducted at the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Service, the Navy Dental Corps and the Veterans Administration.

(USAF SAM Research Task No. 7756-42)



NBS

U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS

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general dental health level of the subject and information on dental treatment requirements during the Service life of the individual and to aid in establishing identification records.

Present methods, in which intraoral films are used, require the placement and exposure of 14 or more films. This creates a time-consuming problem in processing and handling and fails to show pathological conditions lying outside the field covered by the small films.

Several methods for producing full-mouth dental x-rays on a single film, both intraorally and extraorally, have been explored. Smathers [1], Nelsen [2], Zulauf [3], and Paatero [4], [5], and [6] have produced such radiograms using machines of various designs.

An extraoral method, termed curved-surface laminography, was selected for the present development because of the simplicity of the technic and the fact that fitting and placement of a bulky lead-backed film in the patient's mouth would not be required. It was also decided that rotation of the x-ray source and film holder would be employed rather than rotation of the patient and chair as described by Paatero [6] thus further simplifying the apparatus.

## 2. METHOD

The principles of curved-surface laminography have been discussed by Paatero [4,5] and by Duhamel [7,8]. The essential condition for this type of radiography is that the shadow or image of the object which passes through a beam of x-rays must fall on a film which is moving at the same velocity as is the image. Paatero has shown that the synchronization of image and film in dental radiography can be obtained by rotating in opposite directions





the subject and a film curved to the shape of the dental arch. In the panoramic machine described in this report, synchronization of image and film is obtained by moving a flat film by means of a cable passing around a cam shaped to simulate the curve of the dental arch.

The present form of the panoramic machine was adopted after preliminary studies were made to determine geometric relationships and factors influencing projection of images of the desired mouth structures. In one device, a light beam was employed rather than x-rays, and transparent plastic models of the dental arches containing opaquely-outlined tooth silhouettes were used. Photographic printing paper was substituted for film. Information gained in the above experiment was applied to the design of another panoramic device using an x-ray source and a human skull. This model did not provide for movement of the x-ray source due to the bulk and weight of the x-ray tube head. The subject (skull) was rotated and the film-holding device rotated to achieve the same geometric pattern as had been obtained in the light beam model. Many radiograms were made with this device, using different x-ray exposures, different rates of motion, different positions of subject, film and x-ray source, and various widths of x-ray beam. Information gained was used in the design of the machine which followed and which represents the final development achieved in this project to date.

In the final form of the machine, the x-ray source and film holder are rotated around the subject so that the axis of rotation is just medial to the angle of the mandible on the side opposite that to be radiographed. The rate of linear motion of the x-ray beam as it sweeps the dental arch varies with the changes in



curvature of this arch. Molar and bicuspid teeth for example, lie on the circumference of a circle whose radius is slightly shorter than that of a circle whose circumference passes through the incisor and cuspid teeth, the center of both circles being located at the center of rotation previously described. Therefore, the linear rate of motion of the film past the vertical slit in the face of the film holder must be slightly increased when projecting the anterior teeth as compared to the linear rate when projecting the posterior teeth. This variable rate is provided by the cam of the film-drive mechanism. Curvature of an average dental arch is simulated in each of the four lobes of the cam and its contour is proportioned so that the proper rate of movement of the film past the film-housing vertical slit is maintained. The result is a projection of those dental structures lying in the curved plane described by the dental arch, while structures lying outside or inside this plane are projected in such distorted and diffuse shadows that they do not interfere with the visualization of the structures that it is desired to radiograph. Since the divergence of the x-ray beam produces a slight magnification in the image, the overall rate of the film movement dictated by the size of the cam must be slightly greater than the rate of linear motion of the x-ray beam along the line of the dental arch.

The method by which the panoramic image is obtained is shown schematically in Figure 1. The diagram shows the arrangement for exposing one-half of the arch. After this half is exposed, the subject is moved laterally so that the axis of rotation is back of





the molars on the opposite side of the arch and the second half of the arch is then exposed. In practice the subject and cam remain in a fixed position during the exposure while the source and film holder are rotated around them. In principle this is identical to holding the source and film holder fixed and rotating the subject as shown in the figure. From this figure it can be seen that a clear image of T can be obtained only if the film moves so that at all times point I is on the extension of a straight line from the source S through T as T moves through the x-ray beam (from  $T_1$  to  $T_2$  to  $T_3$ ). This condition will be met if

$$\frac{FI}{SF} = \frac{r_T \sin \phi}{SR + r_T \cos \phi} \quad (1)$$

where  $\phi$  is equal to or less than  $\theta$ . Since  $\phi$  is small it can be assumed that  $\cos \phi = 1$  and  $\sin \phi = \phi$ . Equation (1) therefore can be written

$$\frac{FI}{SF} = \frac{r_T \phi}{SR + r_T} \quad (2)$$

Using this relation, the dimensions of the cam required to control the movement of the film can be determined. The angular velocity  $\omega$  of a point T moving through the x-ray beam from  $\phi = \theta$  to  $\phi = 0$  in time  $t$  is  $\omega = \frac{\phi}{t}$  (3)

and the linear velocity is

$$\omega r_T = \frac{r_T \phi}{t} \quad (4)$$

assuming that the linear velocity  $V_I$  of the image across the slit is constant, from equation (2):





$$V_I = \frac{FI}{t} = \frac{SF r_T \phi}{(SR + r_T)t} \quad (5)$$

Substituting from equation (3)

$$V_I = \frac{SF \omega r_T}{SR + r_T} \quad (6)$$

If the velocity  $V_F$  of the film is controlled by a cable around a cam also moving with angular velocity  $\omega$ , the linear velocity of the film is  $V_F = \omega r_C$  where  $r_C$  is the effective radius of the cam when T is passing through the x-ray beam.

For image formation  $V_F = V_I$  or

$$\omega r_C = \frac{SF}{SR + r_T} \omega r_T \quad (7)$$

$$r_C = \frac{SF}{SR + r_T} r_T \quad (8)$$

This equation gives the required effective radius  $r_C$  at any point on the cam to provide a sharp image of any point T on the subject at a distance  $r_T$  from the axis of rotation. In the design of the actual cam, the geometry of the machine and of the cam which affect the point of contact of the cable and the cam must also be taken into account.



A point at a distance  $r_T + \delta$  from the axis of rotation will project a line rather than a point on the film as it passes through the x-ray beam. The length  $\Delta$  of this line which determines the sharpness of the image can be calculated by comparing the relative positions of the film and image when the image of point  $T_\delta$  is at F and when the image of this point is at the edge of the slit.

When the image  $I_\delta$  of point  $T_\delta$  is at the edge of the slit:

$$\frac{(r_T + \delta) \sin \varphi}{SR + (r_T + \delta) \cos \varphi} = \tan \alpha \quad (9)$$

Considering  $\cos \varphi = 1$  and  $\sin \varphi = \varphi$ , since  $\varphi$  is small:

$$\varphi = \frac{(SR + r_T + \delta) \tan \alpha}{r_T + \delta} \quad (10)$$

The distance  $FI_\delta$ , when  $I_\delta$  is at the edge of the slit, can be written  $FI_\delta = SF \tan \alpha$  (11)

Since  $I_\delta$  and I will both fall on F when  $\varphi = 0$  and since the movement of the film is synchronized with the movement of I, the length  $\Delta$  of the projection of  $I_\delta$  on the film is equal to the distance between  $I_\delta$  and I when  $I_\delta$  is at the edge of the slit. From equation (2)

$$FI = \frac{SF r_T \varphi}{SR + r_T \varphi} \quad (12)$$

substituting from equation (10)

$$FI = \frac{SF r_T (SR + r_T + \delta) \tan \alpha}{(SR + r_T) (r_T + \delta)}$$

$$\Delta = FI_\delta - FI = \frac{SF \tan \alpha (SR + r_T)(r_T + \delta) - SF r_T (SR + r_T + \delta) \tan \alpha}{(SR + r_T) (r_T + \delta)}$$





$$\Delta = \frac{SF \cdot SR \cdot \delta \cdot \tan \alpha}{(SR + r_T)(r_T + \delta)} \quad (13)$$

From this equation it can be seen that the sharpness of an image at a distance  $\delta$  from  $r_T$  is directly proportional to  $\tan \alpha$ . Since  $\alpha$  is determined by the slit width, the importance of the slit width in determining the sharpness of the image is obvious.

The length on the film of the image of a point at a distance  $r_T + \delta$  from the center of rotation can be calculated by substituting numerical values in equation (13). The approximate values for the present panoramic x-ray machine are as follows:

SF	425 mm
SR	298 mm
RF	127 mm
$r_T$	50-60 mm
FI	3.75 mm
tan	0.00882

Substituting in equation 13 and assuming  $r_T = 55$  mm, values for  $\Delta$  can be determined. Since the equation gives the value for image movement from the center to the edge of the slit, the total length of the image is equal to  $2 \Delta$ . Calculated values are given below:

$\delta$	$2 \Delta$
1 mm	0.11 mm
5 mm	0.53 mm
10 mm	0.97 mm
-10 mm	-1.41 mm

### 3. PROTOTYPE MACHINE

#### 3.1 Construction of Prototype Machine

As now constituted, the panoramic dental x-ray machine is shown in Figure 2. A Fischer Model ANM bedside radiographic machine base, controls, and self-contained tube-head transformer



combination were incorporated in the new device. The x-ray source and film-holding mechanism are suspended from a movable arm which pivots about an axis secured to a large fixed horizontal support arm extending out from the main support column.

Immediately below the axis of rotation is a four-lobed cam which, with its cable drive, serves to move the film inside the holder at the proper rate to secure exposure of the desired structures.

A reversible motor with reduction gearing is coupled through a magnetic clutch to a pulley and belt system to drive the movable arm about the support axis. Movement is controlled by mechanical stops permitting about 200° of total rotation and a reversing-switch automatic-stop device which automatically stops the machine at the end of one half-arch exposure and reverses polarity to the motor as the machine is moved by hand to the opposite side for the second half-arch exposure.

Regular medical x-ray film is placed in a cassette with intensifying screens and inserted into the film carriage in the film-holding element of the machine. The carriage is constructed so as to roll on a horizontal track inside the film holder to such an extent that the entire film can pass a vertical slit in the center of the front face of the film-holder housing. This face, which is toward the x-ray source, is lined with lead and only those x-rays passing through the vertical slit can reach the moving film. Fogging due to secondary radiation is reduced in this manner.





X-rays emerging from the source carried at the end of the movable arm opposite the film holder, are confined by means of a lead slit so that a narrow vertical beam is projected. This beam passes directly through the subject and falls on the opening in the face of the film holder. Objects in the beam path project shadows on the film in proportion to their relative radiopacities as is the case in all x-ray exposures.

### 3.2 Operation of the Machine

The subject is seated and his head is positioned by means of a chin rest so that the axis of rotation of the machine falls at a point just medial to the angle of the mandible on the side opposite that to be radiographed. The head is also tilted forward so that basal skull shadows will not fall in the area of the teeth but will be projected higher on the film where they will not obscure detail in the dental structures.

The movable arm is swung to the extreme limit of its travel so that the film holder is now near the teeth and jaws of the side to be radiographed. The machine is energized and the x-ray beam sweeps about the rear of the head from about the posterior border of the ramus of the mandible on the side opposite that being radiographed to a point just lateral to the vertebrae of the neck. At the same time the film holder has moved from its position at the start of the exposure, near the molar tooth region, to a position near the incisor teeth of the side of the dental arch being radiographed. Following this, the patient's head is moved to orient the opposite side of the arch, the machine is swung manually about its axis to reposition the x-ray tube and film holder, the machine





is again energized and the second half-arch is exposed.

Time required totals 40 seconds for the two exposures and it is anticipated that once the technic for using the machine is perfected, an experienced operator should be able to take full-mouth radiographs in three to five minutes.

Adequate density has been obtained in adult human dental radiographs using x-ray tube voltage and current of 65KV at 10 ma. Patient radiation at various points in and about the head is reduced in this method of radiography due to the fact that a narrow beam of x-ray is used and repeated exposure of the same area does not take place as it does in conventional dental radiography [9].

#### 4. RADIOGRAPHIC RESULTS

Radiograms of the entire dentition as well as the body and ramus of the mandible and the bony structure of the mandible and maxillary regions are produced (Figure 3). The presence of impacted or unerrupted teeth or retained roots is shown. The general status of the alveolar bone is revealed. Fillings, bridges, etc., are shown as are caries in cases where penetration of the enamel has occurred. Information recorded is believed sufficient to establish many facts of value in mass surveys and for specialized applications in prosthetic and surgical diagnosis. If further detailed spot films are required, these can be made as needed. The panoramic machine is not designed to supplant the conventional technic where the recording of extreme detail is necessary.



## 5. SUMMARY

A panoramic dental x-ray machine for use in mass surveys and examinations has been developed and a prototype constructed. This device produces full-mouth dental radiograms on a single 5" x 10" film using an extraoral technic. Patients are positioned either seated or standing, beneath the moving arm of the machine, which carries an x-ray source and film holder. Rotation of this arm about its axis sweeps the x-ray source and film about the patient's head, producing a projection of the desired tooth images on the moving film. Adequate detail is secured to obtain a diagnosis of general mouth conditions. The panoramic dental x-ray device is not designed to supplant the conventional dental x-ray machine in cases where extreme sharpness and detail must be obtained.





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Figure 1. Schematic diagram of panoramic x-ray machine.



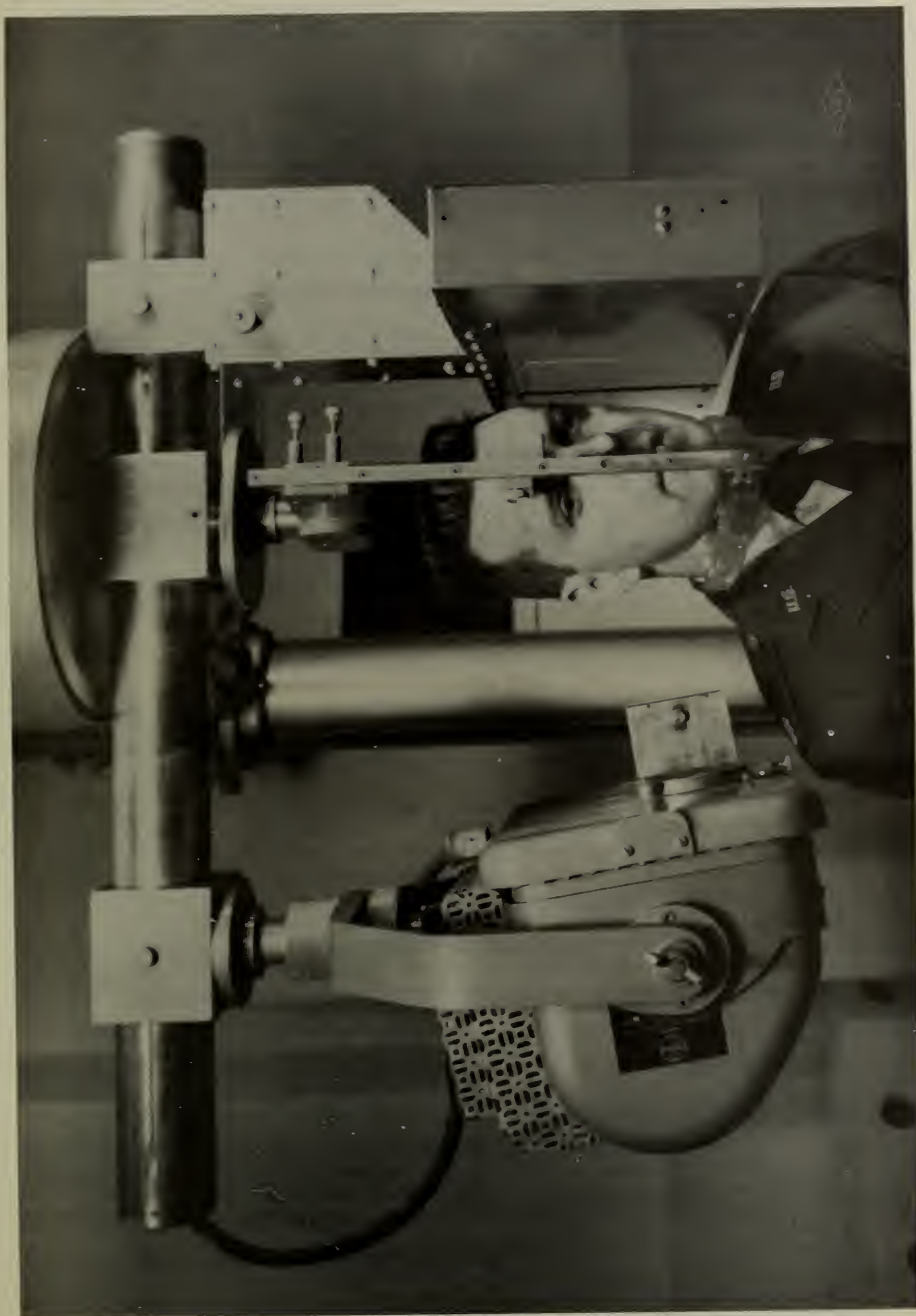


Figure 2. Panoramic x-ray machine.





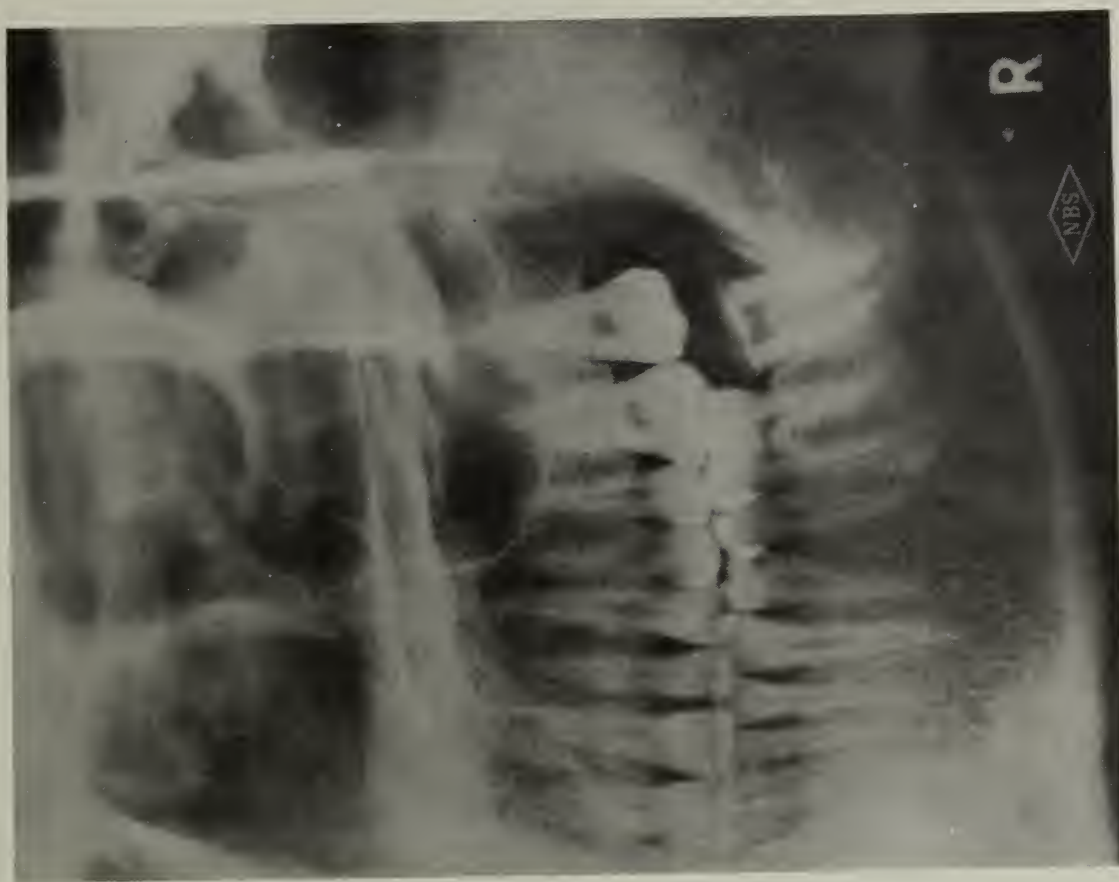
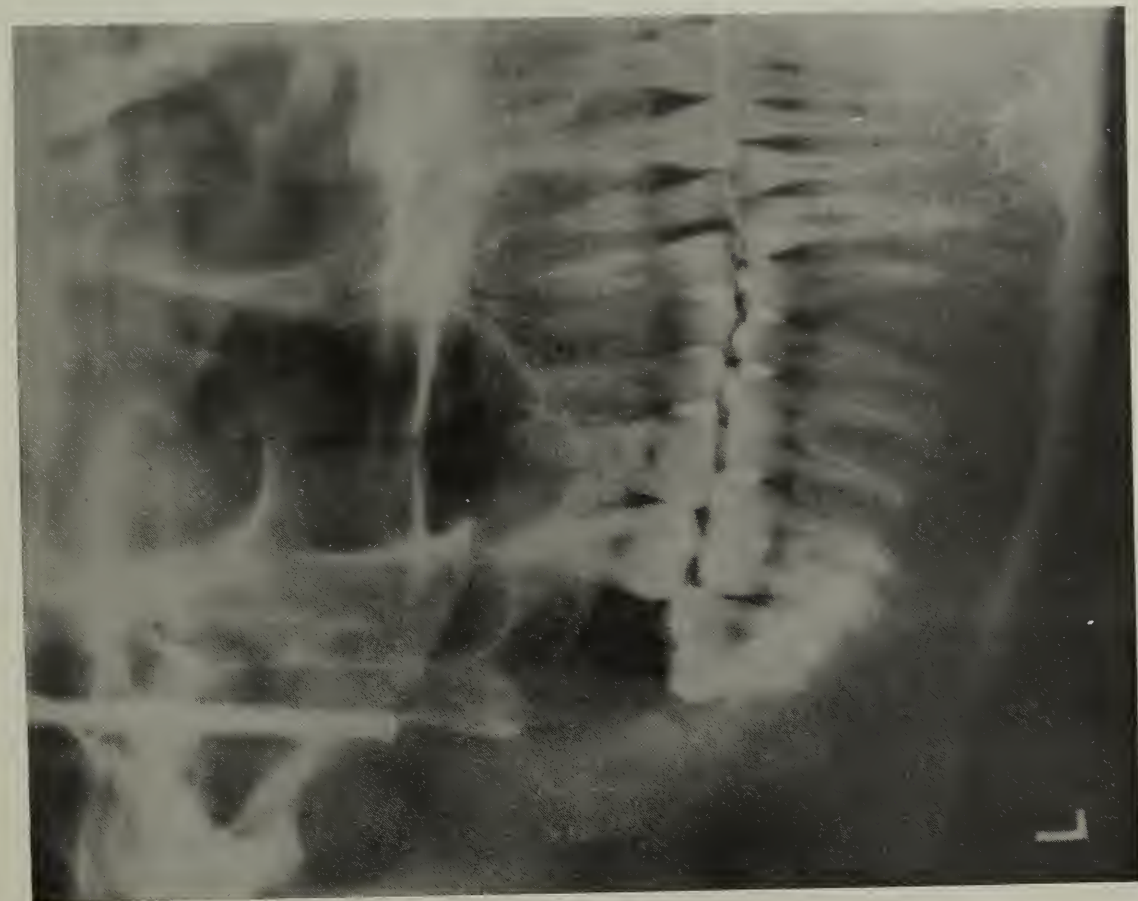


Figure 3. Reproduction of full-mouth survey (of skull).



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### Progress Report

#### A SIMPLE TECHNIC FOR MAKING DENTAL PORCELAIN JACKET CROWNS

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# A SIMPLE TECHNIC FOR MAKING DENTAL PORCELAIN JACKET CROWNS

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## Abstract

A rapid, precise, and comparatively simple technic for fabricating fused porcelain jacket crowns is described. The essential features of the technic are the employment of a prefabricated fused porcelain veneer and of two porcelains having widely different fusion temperatures. The matching of the anatomy, the color, and the translucency are largely predetermined by those of the porcelain veneer, which can be tried in the patient's mouth when the preparation of the tooth is completed. Shrinkage on fusion has been greatly reduced and the number of fusions has been cut from four or five needed in customary technics to two.

## 1. INTRODUCTION

The idea that dentistry is both an art and a science is peculiarly applicable to the use of fused porcelain as a restorative material. The making of a porcelain jacket crown probably requires a greater degree of artistic skill than that needed for any other dental restoration. This artistic skill must be combined with a thorough knowledge of the physical properties of the porcelain to produce a beautiful result. These facts account for the infrequent use of the fused porcelain jacket crown in places where it should be the first choice unquestionably. Frequently, the acrylic resins, which have not proved satisfactory, are substituted for fused porcelain in jacket crown restorations.

The principal reasons why porcelain jacket crowns are not as widely used as they should be are: (1) the difficulty in producing the correct color and translucency in different parts of the jacket crown so that it will be indistinguishable from the adjacent and opposing natural or artificial teeth; (2) the skill needed to mold a crown of the correct size and shape that will harmonize with the other teeth of the patient; (3) the experience and skill required in fusing the porcelain; (4) the comparatively long time required to make a porcelain jacket crown by the current classical technic, which was invented and perfected by Charles H. Land [1] at the beginning of the twentieth century.

It is the purpose of this report to present a technic that will minimize the foregoing difficulties.





## 2. OUTLINE OF TECHNIC

An artificial porcelain tooth of proper size, shape and color is ground to an approximate fit of the tooth preparation in such a way as to serve as a labial and proximal veneer for the crown. This procedure is almost identical to that proposed by Edward B. Spalding [2] in 1904 after Land had showed him the fused porcelain crown. Spalding powdered porcelain teeth and used this to fill in the parts of the crown not supplied by the veneer. Thus, the porcelain used in completing the crown fused at the same temperature as the veneer and so required the utmost skill in handling. In the technic here presented, a layer of a mix of powdered porcelain teeth and a low-fusing porcelain is first applied, then a layer of low-fusing porcelain is placed over this first layer for the first bake, and low-fusing porcelain is used for the second bake.

## 3. DESCRIPTION OF TECHNIC

### 3.1 Preparation of the Crown for the First Fusion

Preparation of the tooth. The reader is referred to current textbooks on crown and bridge and porcelain work for classical shoulder preparation.

Impression of prepared tooth. The impression of the prepared tooth is the common one used in the indirect method, employing the festooned copper band filled with an impression compound or a rubber base impression material. The impression is boxed in and an artificial stone die prepared in it.

Baseplate wax impression of prepared tooth. A triangular piece of baseplate wax is warmed, folded to form a cone, pressed over the preparation, trimmed to relieve the proximal and incisal surfaces, and scored to cause it to withdraw with the subsequent impression. This wax covering is shown in position in Figure 1.

Impression of adjacent teeth. A hydrocolloidal or rubber base impression is then taken of the wax covered preparation and the adjacent teeth. On withdrawal, the baseplate wax impression stays in the hydrocolloid or rubber impression.

Making model. The greased artificial stone die (the replica of the tooth preparation) is seated into the baseplate wax impression of the preparation and sealed with hot wax. Then the model is poured.

Matrix on which porcelain is fused. Because a low-fusing porcelain is employed [the one used by the authors is "Apco" fusing at 1024°C (1875°F)], the matrix is prepared on the





artificial stone die with gold foil, 0.001 inch thick.

Selection and preparation of artificial tooth. A porcelain tooth of approximately the correct shape, size, and color is selected. The pins are cut off with a silicon carbide disk and the lingual surface hollowed out by diamond stones (Figure 2) to fit the labial and proximal surfaces of the preparation. It may be necessary to remove some porcelain from the incisal, cervical, or proximal surfaces so that the veneer is in proper alignment and approximately fits the die. A lingual view of the veneer at this stage is shown in Figure 3. It is good practice to try the veneer in the mouth now so that both the dentist and patient can see the approximate color and shape of the labial face of the crown as it will appear when completed.

Preparation of powdered porcelain teeth. Four or five light-colored porcelain teeth of any of the current brands are crushed in a diamond mortar and are screened on standard sieves. The portions that (1) pass through No. 40 sieve but are retained on the No. 70 sieve and (2) pass through the No. 70 sieve but are retained on the No. 100 sieve are mixed in equal portions. This size distribution, selected by trial and error, gives a low shrinkage on fusion when mixed with low-fusing porcelain, yet presents a surface after fusion that is smooth and glazed.

Placing the porcelain for the first bake. A color of the low-fusing porcelain which approximates that of the veneer is selected. An exact match is not necessary. A dry mix of equal portions of the low-fusing porcelain and the powdered porcelain teeth is made on the slab.

A thin layer of the low-fusing porcelain-water mixture is painted on the labial surface of the preparation with a heavier amount on the labial shoulder.

A thin layer of the aqueous mixture of low-fusing porcelain and powdered porcelain teeth is applied to the lingual surface of the veneer, with a heavier amount in the incisal area.

The veneer facing is then placed in position on the die, and the porcelain is condensed by the classic method of vibration with a serrated instrument (Figure 4) and by blotting the excess water brought to the surface.

The die is now placed in the model, and the alignment of the veneer is checked. If incorrect, the condensed porcelain is touched with a wet brush to facilitate moving the veneer to proper alignment. The porcelain is again dried by





blotting. The die is removed from the model, placed in the inlay holder, and held in position with the fingers.

The mixture of powdered porcelain teeth, low-fusing porcelain, and water is now painted on the lingual surface, the largest bulk being placed on the marginal ridges, lingual shoulder, and the cingulum. Water is brought to the surface by careful vibration with the serrated instrument and is removed by blotting. Crumbs of porcelain are removed with a No. 5 red sable hair brush (Figure 5).

Low-fusing porcelain only is now applied to the proximal and incisal portions of the crown, making certain that the porcelain overlaps the veneer. The porcelain is again condensed by carefully vibrating and blotting, and loose crumbs are removed with the large brush as previously described. The proximal surfaces are gently burnished with the flat side of the spatula and again brushed. The gingival margin is cleaned with a small, wet No. 00 red sable hair brush (Figure 6). The crown is now ready for the first fusion.

### 3.2 First Fusion of the Crown

The furnace (Figure 7) can be regulated so that it will be at 593° to 649°C (1100° to 1200°F) when the application of the porcelain for the first bake is completed. The crown is removed, placed on the firing tray, and set on top of the muffle to dry (A in Figure 7). The temperature of the furnace is then advanced to 760°C (1400°F) in three to four minutes. At this temperature the muffle door is opened, and the tray with the crown is shifted from the top of the muffle to the shelf (B in Figure 7) just below the opening of the muffle and left there from one to two minutes. By this time the temperature of the furnace has drifted to 593° to 649°C (1100° to 1200°F). (These time-temperature relationships will, of course, vary with the type of furnace used. The authors employed an S.S.White No. 3) The tray is now placed in the muffle so that the crown will be beneath the thermocouple junction. The door is returned to the shelf below the muffle opening, leaving a space between the door and the muffle (C in Figure 7). This space is gradually closed until the furnace reaches 1400°F. This requires three to four minutes. The furnace is now raised to 982°C (1800°F) in 55°C (100°F) intervals, spaced approximately three minutes apart. When 982°C (1800°F) is reached, the temperature is immediately reduced over a period of five to six minutes to 816°C (1500°F), then the firing tray and crown are removed and placed under a beaker until the crown can be handled (D in Figure 7).





### 3.3 Preparation of the Crown for Second Fusion

The cooled crown is seated on the die, which is then inserted into the model to check for alignment and contact points (Figure 8). Sometimes it is necessary to relieve the contact point by grinding so that the crown may be seated properly.

The die and crown are again mounted in the holder, and the crown is moved up and down slightly to make certain that the matrix is not stuck to the die.

The lingual anatomy is completed by painting the low-fusing porcelain-water mixture on the marginal ridges, the cingulum, and the central lingual area. The porcelain is again condensed by gentle vibration with the serrated instrument (Figure 4). If the vibration is too vigorous, the contours of the marginal ridges and cingulum may be lost as the porcelain flows. After burnishing and cleaning with the large dry brush (Figure 5) and a small wet brush (Figure 6), the crown is ready for the second fusion.

### 3.4 Second Fusion of the Crown

The schedule of the second fusion follows that of the first fusion except that the final temperature is 1024°C (1875°F) instead of 982°C (1800°F).

A schematic description of the firing schedule is given in Table 1.

### 3.5 Preparation of the Crown for Cementing

When the crown is cool enough to handle, water placed in the matrix aids in its removal. The gold matrix is teased from the crown with cotton tweezers (Figure 9).

Any overhanging fin of porcelain at the gingival margin is removed carefully with a small round silicon carbide mounted stone. The crown is now ready for cementation.

Zinc phosphate cement is mixed on a chilled slab to the heaviest cementing consistency. An exact match of the color of the cement and the color of the crown is not necessary. The cement is placed in the crown and not on the preparation. The patient is instructed to hold the crown in position as the cement is hardening. The excess hardened cement is carefully removed from the gingiva.

The points of contact between the porcelain jacket crown and the opposing teeth at rest and in motion are checked with articulating paper. High spots are removed from the porcelain by grinding. Even if there are no high spots, the glaze on the





lingual surface of the porcelain crown should be removed in those areas where contact with the opposing teeth is anticipated. The natural teeth are abraded by the ground surface of the porcelain, and thus occlusal trauma is prevented.

#### 4. DISCUSSION OF THE TECHNIC

##### 4.1 Simplification of Color Matching

The use of a fused porcelain veneer formed from a stock porcelain tooth simplifies the matching of color and translucency of adjacent teeth. This color match can be checked directly in the mouth with the porcelain veneer. The color of the veneer does not change on firing because subsequent fusions are made at temperature well below the fusion temperature of the veneer.

The selection of any of the light colors of powdered teeth is satisfactory, and only an approximate match is necessary between the color of the low-fusing porcelain and the veneer and between the color of the zinc phosphate cement and the crown. With the classical technic the matching and the reproduction of the colors and opacities are difficult to achieve.

##### 4.2 Reduction in Carving of Anatomy

There is no change in the contour of the veneer during the firings, as its fusion temperature is much higher [ $1288^{\circ}\text{C}$  ( $2350^{\circ}\text{F}$ )] than that of the low-fusing porcelain [ $1024^{\circ}\text{C}$  ( $1875^{\circ}\text{F}$ )] used on the lingual surface of the crown. Thus, the labial, proximal, and incisal areas are largely established by the veneer. In contrast, the carving and the making of the porcelain jacket crown with high-fusing porcelain alone is so difficult that only a small number of porcelain experts attempt it.

##### 4.3 Reduction in Firing Shrinkage

The large firing shrinkage of either high- or low-fusing porcelain is a great handicap to the dental porcelain worker. By this technic the firing shrinkage of the porcelain is greatly reduced. The large bulk of the crown is the veneer in which there is no shrinkage within the heating range used during the firing of the crown [room temperature to  $1024^{\circ}\text{C}$  ( $1875^{\circ}\text{F}$ )]. Also, the fusion shrinkage of the mixture of low-fusing porcelain and the powdered porcelain teeth is less than the fusion shrinkage of either low- or high-fusing porcelain (Table 2) and amounts to a reduction of 29 percent of that portion of the crown in which this mixture is used.





These determinations of the shrinkage on fusion of several porcelains (Table 2) were made on cylinders approximately 10 mm high and 6 mm in diameter, by hydrostatic weighing. The porcelain-water mixture was compacted into the mold in much the same manner as described in making the jacket crown. The heating schedule of all of the porcelains in Table 1 was the same as that used in fusing the crowns, except that the manufacturer's directions were used in firing the high-fusing porcelains.

Other investigators have reported firing shrinkages as low as 24 percent and as high as 48 percent by volume [3,4,5]. Variations among the data could be attributed to the size and shape of the specimens, the method of condensation, and the time-temperature schedule of fusing.

## 5. PROPERTIES OF THE MATERIALS USED

### 5.1 Thermal Expansion

The thermal expansion of a low- and of a high-fusing porcelain of powdered porcelain teeth and of a mixture of low-fusing porcelain and powdered porcelain teeth was determined over the range of 30 to 400°C (86 to 752°F) by an automatically recording interferometric method [6]. The data (Table 3 and Figure 10) show that there are no important differences in the thermal expansions of the various porcelains used in fabricating the crown. Certainly no significant strain should be set up in the crown during cooling that could be attributed to the small differences in the coefficients of thermal expansion among the porcelains used.

The data in Table 3 also show that the thermal expansion of the fused porcelain jacket crown approaches closely that of the tooth. This approximate matching of the thermal expansion of a restorative material with that of the tooth is of great clinical importance. As previously demonstrated, it has a direct bearing on the fluid exchange at the margins of dental restorations that occurs with a change of temperature in the mouth when hot and cold foods are eaten [8].

### 5.2 Compressive Strength

The cylinders used in determining the volumetric shrinkage on fusing were trued and used for determining the compressive strength. The walls of the cylinders were paralleled, and the ends were made plane and at right angles to the long axis of the cylinder by grinding the spinning cylinder with a revolving diamond-coated wheel. The resultant specimens were  $7 \pm 1.5$  mm in height and  $4 \pm 0.7$  mm in diameter, depending on the amount of truing necessary. The load was applied at the rate of 500





pounds per minute. The data (Table 4) show that there are no appreciable differences among the compressive strengths of the low- and the high-fusing porcelain and the mixture of low-fusing porcelain (80%) and the powdered porcelain tooth (20%). The mixture of low-fusing porcelain (50%) and of powdered porcelain teeth (50%) has about one-fourth the strength of the other porcelains in Table 4. This low compressive strength of the 50-50 mixture was thought at first to rule out such a mixture in fabricating the porcelain jacket crown. However, repeated cross-sectioning of fused crowns showed no difference in the appearance of the veneer, the mixture of low-fusing porcelain (50%) and the powdered porcelain teeth (50%), and the low-fusing porcelain zones. There was no stratification visible, as the different layers had all blended together. The low-fusing porcelain layer in the crown had evidently permeated the layer of the mixture, which no longer remained a 50-50 mixture and was probably closer to the 80-20 mixture. An inspection of cross-sections of specimens of the 50-50 mixture used in determining its compressive strength showed that insufficient low-fusing porcelain was available for fusing the particles of the powdered porcelain teeth together under the conditions of fusing.

Just as the compressive strengths of the high- and low-fusing porcelains were approximately the same (Table 4), so were the strengths in transverse bending of similar porcelains reported by Fitzgerald [9]. The correlation of the brand-names with the data of Fitzgerald is given by Moldal [10].

### 5.3 Solubility

The solubility of the various porcelains in a four percent solution of acetic acid is given in Table 5. Each porcelain was fired at the temperatures given in the table and was then powdered in the diamond mortar. Only that portion which would pass a No. 200 sieve was used. A two-gram sample of this powdered fused porcelain was boiled for 30 minutes in 200 ml of a four percent solution of acetic acid. This solution-powdered porcelain mixture was passed through a tared porcelain filter, which was then placed in a furnace at 150°C (292°F) for one hour, cooled in a desiccator, and weighed. The test is patterned closely after that of Koenig [11] who employed it to test frits and glazes used on enamelware for the household.

There are slight differences in the solubility of the different porcelains listed in Table 5. No literature was found showing that any of the porcelains tested would disintegrate or etch in the mouth. The senior author has not observed the etching of any of the porcelains in Table 4 over a period of ten years of clinical experience.





## 6. SUMMARY

A rapid, precise, and comparatively simple method for fabricating fused porcelain jacket crowns is described. Physical properties of the materials used are reported. The technic can easily be mastered by dentists and laboratory technicians after a few trials.



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TABLE 1

Time-Temperature Scheme for Fusing Porcelains

Step	Schedule	First Fusion	
		Time Min.	Temperature of Furnace °C      °F
1	Begin application of porcelain and turn furnace on.	0	Room Temperature °C      °F
2	Complete application of porcelain (Fig. 6) and place crown on top of muffle.	10	595 to 650      1100 to 1200
3	Leave crown on top of muffle.	3 to 4	760      1400
4	Remove muffle door and shift tray from top of muffle to shelf below door.	1 to 2	595 to 650      1100 to 1200
5	Place tray in muffle so that crown will be under junction of thermocouple. Return door to shelf, but have it ajar at first. Gradually close opening.	3 to 4	760      1400
6	Raise temperature of furnace in 55°C (100°F) intervals spaced about 3 minutes apart.	12	980      1800
7	Reduce temperature immediately.	5 to 6	815      1500
8	Remove crown and place under glass beaker to cool.		Room Temperature
Second Fusion			

The second fusion follows the pattern of the first except that the final temperature is carried to 1025°C (1875°F) instead of 980°C (1800°F).



TABLE 2

Firing Shrinkage of Fused Dental Porcelains

Brand Name	Type of Porcelain	Firing * Shrinkage-	Standard ** Deviation.
Apco	Low-fusing 1024°C (1875°F)	Percent by Volume 38	1.9
Apco 50% Powdered Trubyte (New Hue)Teeth 50%	Mixture of low- and (1024°C)(1875°F) High-fusing 1288°C (2350°F)	27	1.0
	Low-fusing 927°C (1700°F)	38	0.8
S.S.White	High-fusing 1338°C (2440°F)	37	1.9

\* Average of ten specimens.

\*\* S.D. = 
$$\sqrt{\frac{(x - x_1)^2}{n - 1}}$$





TABLE 3

Thermal Expansion of Fused Dental Porcelains and Human Teeth

Porcelain	Coefficient (30 - 400°C) (86 - 752°F)
	$\times 10^{-6} *$
Apco - Low-fusing (1024°C) (1875°F)	7.8
Apco (1024°C) (1875°F) 80% Powdered Trubyte-New Hue teeth (1288°C) (2350°F) 20%	7.9
Powdered Trubyte - New Hue teeth (1288°C) (2350°F)	7.9
S.S.White high-fusing (1338°C) (2440°F)	6.4
Teeth (7)	(20 - 50°C) (68 - 122°F)
Tooth (root)	8.3
Tooth (across crown)	11.4
Tooth (root and crown)	6.4 8.7 8.3

\* Average of duplicate tests.



TABLE 4

Compressive Strength  
of  
Fused Dental Porcelains

Porcelain	Compressive Strength	
	lb/in. <sup>2</sup> *	Standard ** Deviation.
Apco low-fusing (1024°C) (1875°F)	48,000	11,000
Apco low-fusing (1024°C) (1875°F) 80% Powdered Trubyte-New Hue teeth (1288°C) (2350°F) 20%	39,000	10,000
Apco low-fusing (1024°C) (1875°F) 50% Powdered Trubyte-New Hue teeth (1288°C) (2350°F) 50%	12,000	1,000
S.S.White high-fusing (1338°C) (2440°F)	48,000	3,000

\* Average of five specimens.

$$** \text{ S.D. } = \sqrt{\frac{(\sum x - x_i)^2}{n - 1}}$$





TABLE 5

Solubility of Fused Dental Porcelains  
in  
Four Percent Solution of Acetic Acid

Porcelain	Weight Loss *
	percent
Apco low-fusing (1024°C) (1875°F)	0.3
S.S.White low-fusing (927°C) (1700°F)	0.2
S.S.White high-fusing (1338°C) (2440°F)	0.1
Apco low-fusing (1024°C) (1875°F) 50% Powdered Trubyte New-Hue teeth (1288°C) (2350°F) 50%	0.2

\* Average of duplicate tests.



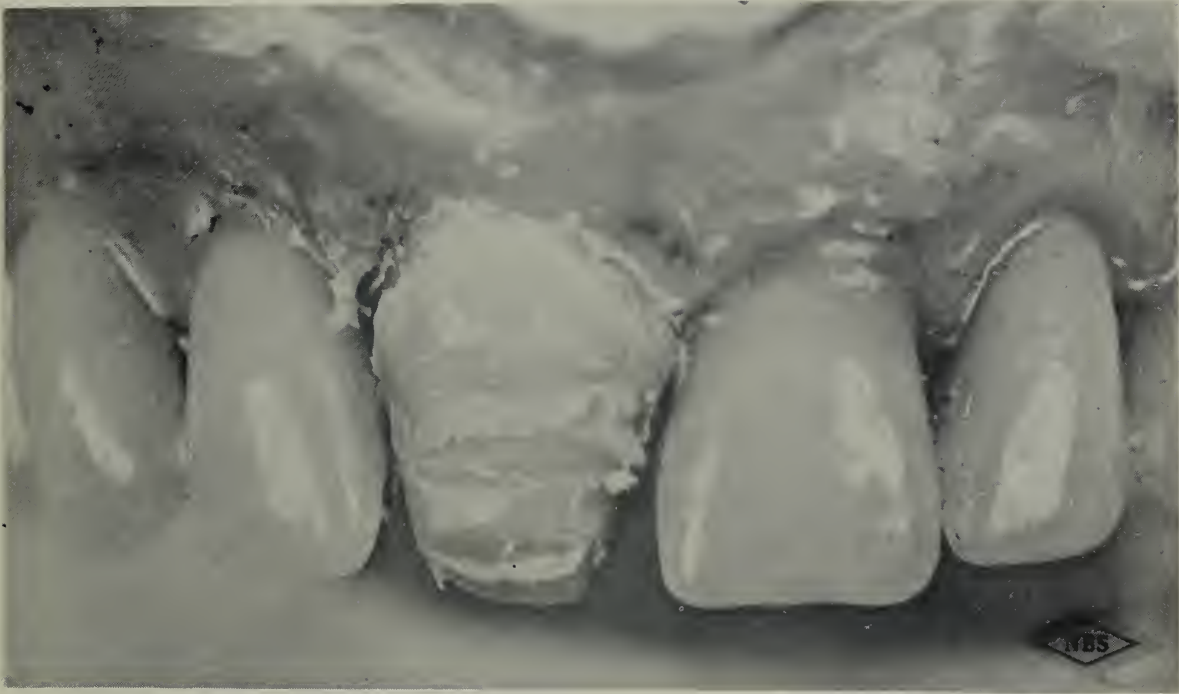


Figure 1. Baseplate wax impression of preparation in place prior to taking the impression of the front section of the upper arch.







Figure 2. Finishing lingual surface of porcelain tooth with diamond cylinder.





Figure 3. Lingual aspect of porcelain veneer on the model.





Figure 4. Vibration of porcelain-water mixture with a serrated instrument to jar porcelain powder particles into close apposition and to bring excess water to the surface.







Figure 5. A No. 5 sable hair brush is used  
to remove loose crumbs of porcelain  
from the surface.





Figure 6. The gingival margin is freed of crumbs of porcelain with a wet No. 00 red sable hair brush. The crown is now ready for the first fusion.





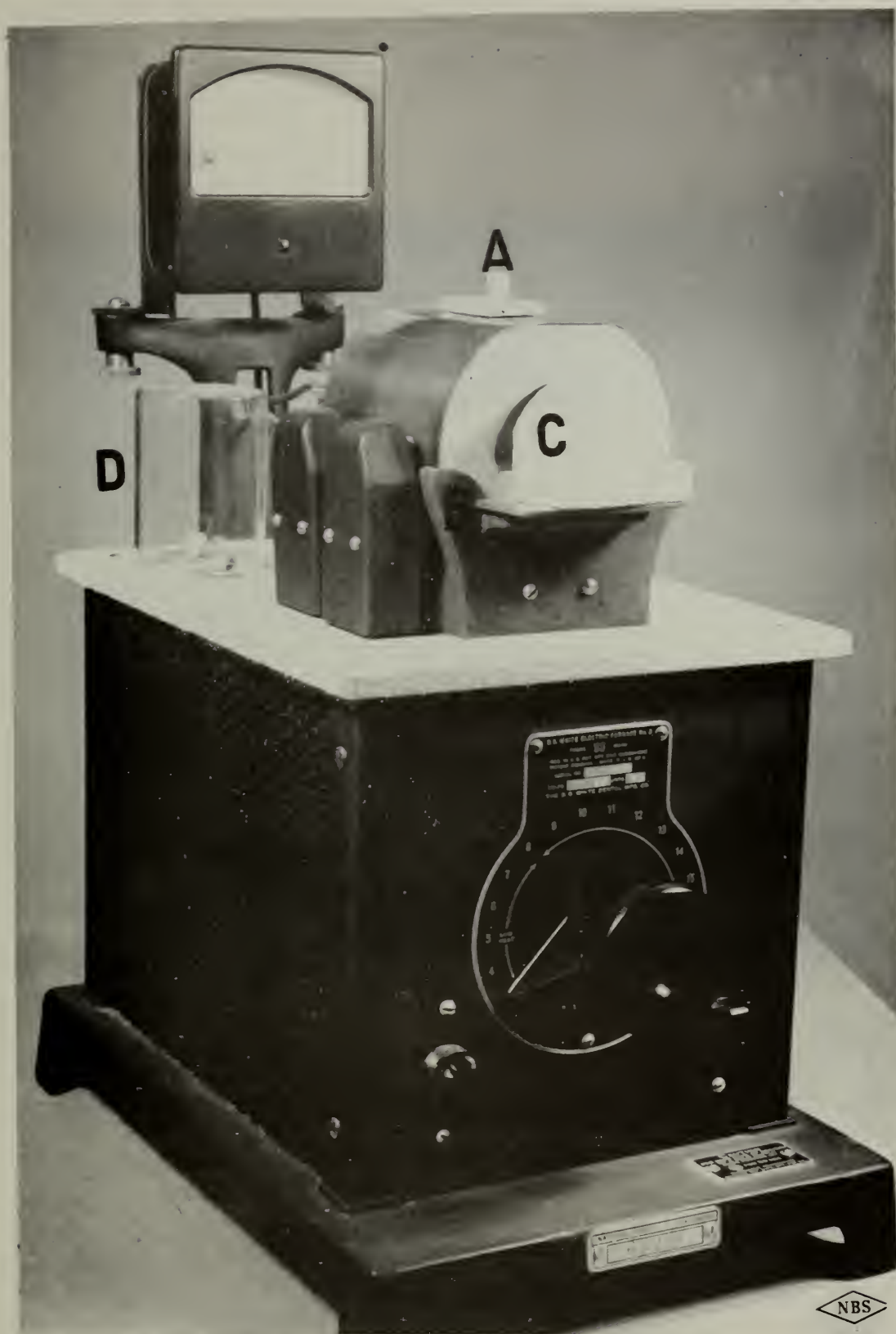


Figure 7. Furnace used in fusing the crown. A is initial position of crown on top of muffle. (Steps 2 and 3 in Table 1). B is shelf where crown is placed in Step 4, Table 1. C shows initial position of the door in Step 5, Table 1. D shows fused crown cooling under a glass beaker.



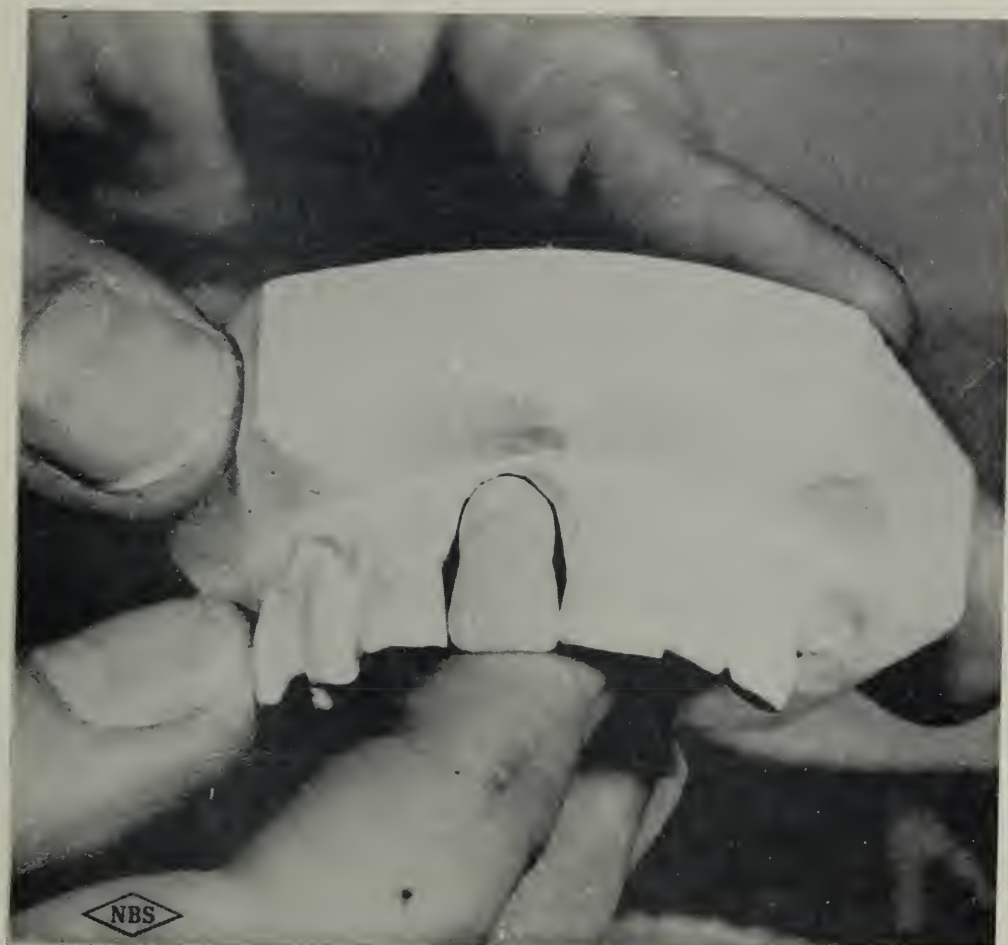


Figure 8. Checking the crown for alignment and contact points after the first fusion has been completed.





Figure 9. Method of peeling matrix out of crown.





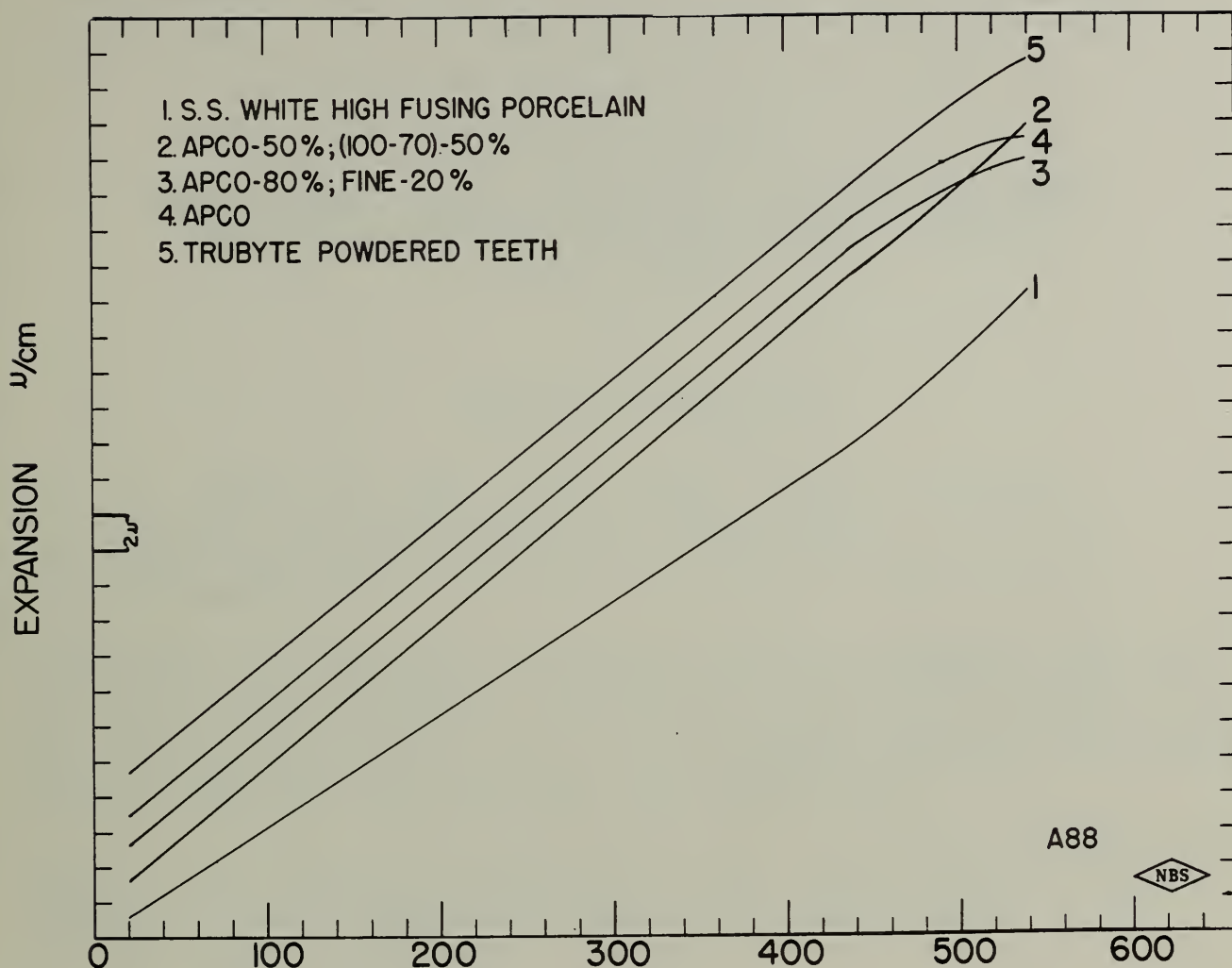


Figure 10. Thermal expansion of dental porcelains.

- Curve 1. S.S.White high-fusing porcelain ( $1338^{\circ}C$ ) ( $2440^{\circ}F$ ).
- Curve 2. Apco low-fusing porcelain ( $1024^{\circ}C$ ) ( $1875^{\circ}F$ ) 50% and powdered Trubyte New Hue teeth ( $1228^{\circ}C$ ) ( $2350^{\circ}F$ ) 50%. The particle size of the powdered teeth consisted of equal portions that (1) pass through a No. 40 sieve but are retained on No. 70 sieve and (2) that pass through No. 70 but are retained on a No. 100 sieve.
- Curve 3. Apco low-fusing porcelain ( $1024^{\circ}C$ ) ( $1875^{\circ}F$ ) 80% and powdered Trubyte New Hue teeth ( $1288^{\circ}C$ ) ( $2350^{\circ}F$ ) 20%. The powder size of the powdered teeth would pass a No. 70 but be retained on a No. 100 sieve.
- Curve 4. Apco low-fusing porcelain ( $1024^{\circ}C$ ) ( $1875^{\circ}F$ ).
- Curve 5. Powdered Trubyte New Hue teeth ( $1288^{\circ}C$ ) ( $2350^{\circ}F$ ).



# NATIONAL BUREAU OF STANDARDS REPORT

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## Progress Report

### SOME CHANGES IN THE PROPERTIES OF AGAR-AGAR TYPE DUPLICATING MATERIAL AND AGAR-AGAR ON HEATING AND STORAGE

by

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This work is a part of the dental research program conducted at the National Bureau of Standards in cooperation with the Council on Dental Research of the American Dental Association, the Army Dental Corps, the Air Force Dental Service, the Navy Dental Corps and the Veterans Administration.

The data in this report were presented in a thesis by Peter M. Margetis in partial fulfillment of the requirements of the Graduate School, Georgetown University, Washington, D. C. for the degree of Master of Science.

(Task 7756-41 BV No. 55-14)



**U. S. DEPARTMENT OF COMMERCE**  
**NATIONAL BUREAU OF STANDARDS**

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# SOME CHANGES IN THE PROPERTIES OF AGAR-AGAR TYPE DUPLICATING MATERIAL AND AGAR-AGAR ON HEATING AND STORAGE

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## Abstract

The effects of heating-gelation cycles and storage at constant temperature on the viscosity, pH, and the time required to fracture specimens under a constant load were observed on agar-agar and a commercial agar type duplicating material. The viscosity of the duplicating material stored at 52°C increased, whereas the viscosity of the reheated duplicating material showed a downward trend. The crushing time decreased in all specimens. Specimens maintained at 52°C fractured under a constant load in a shorter time than did specimens subjected to the heating-gelation cycle. Little correlation between changes in pH and changes in viscosity was found. The effects of both storage at constant temperature and of the heating-gelation cycle on viscosity and time to fracture were found to be reversible. It is suggested that the change in the physical properties of the duplicating material are due to variations in intra- and inter-molecular hydrogen bonding.



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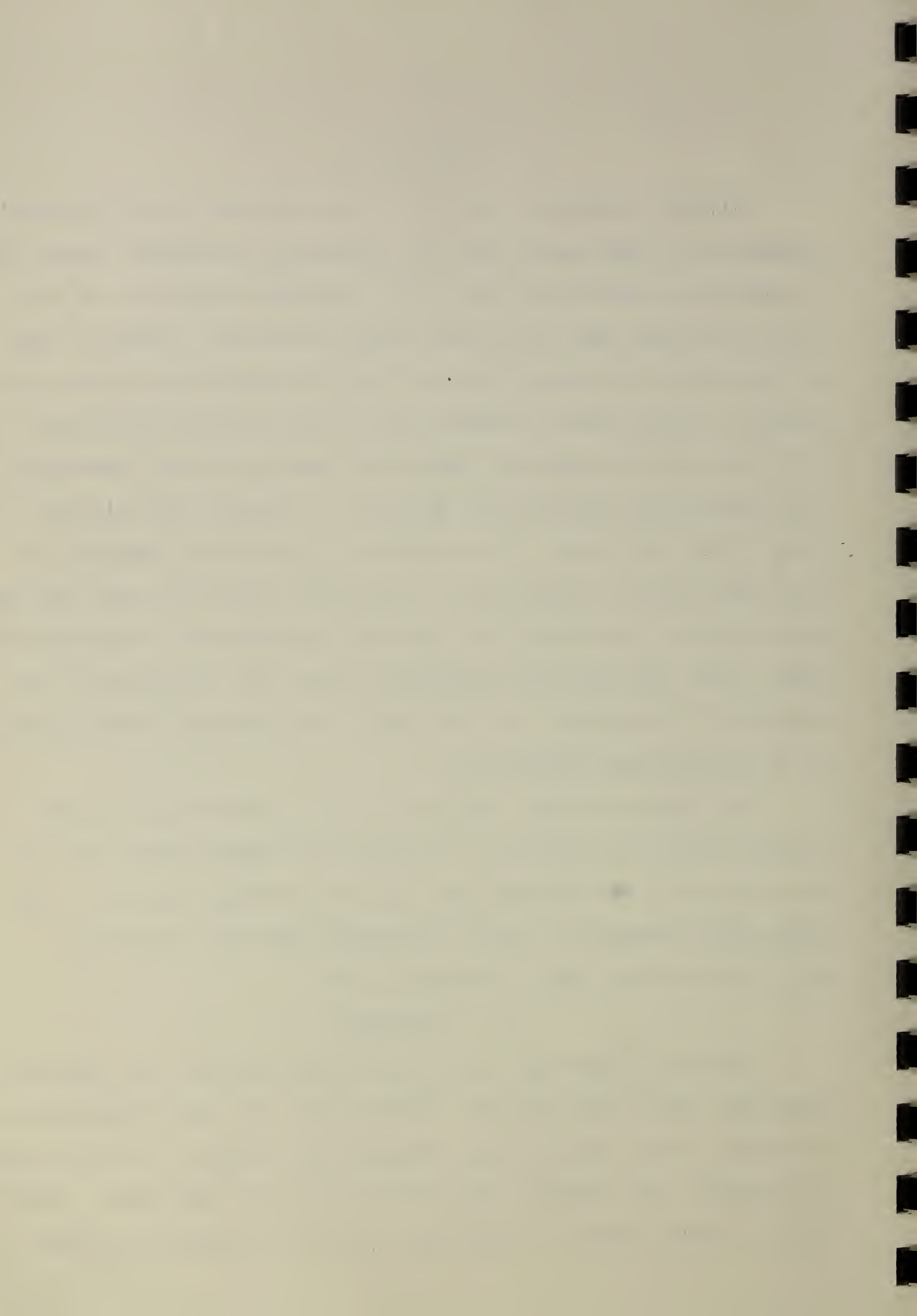
## 1. INTRODUCTION

Although extensive data have been published on the physical properties of agar-agar type oral impression materials, there is a shortage of published data on the physical properties of the reversible agar-agar type duplicating compounds. Though it may be true that the compositions of these two materials differ primarily in their water content, their uses are quite different. It is the general practice today when making an oral impression with reversible hydrocolloid material to discard the material after it is used once. The reversible duplicating compound on the other hand is reused and is repeatedly cycled between the gel and sol state, and stored at elevated temperatures for periods of time. When the desirable properties have been degenerated, the material is discarded. On this basis the problem of deterioration is of considerable importance.

The object of these experiments was to observe, and more clearly define the effects of heating and storage upon some of the properties of the agar-agar type duplicating compound. The properties measured in these experiments were pH, viscosity, and crushing time under a constant load.

## 2. PROCEDURE

Samples of agar-agar type duplicating compound and powdered agar-agar (USP) were procured commercially for these experiments. Specimens of the duplicating compound were prepared in the following manner: the compound was ground into small particles, mixed with an equal weight of distilled water, at a temperature near





100°C, in a container which was then immersed in boiling water for one hour. The resulting suspension was poured into mason jars, sealed and immediately placed in a water bath which was kept at 52°C. Four separate runs were made. In three of the runs (1, 3 and 4) the specimens were divided into two groups. One group was allowed to remain in the 52°C water bath for periods extending up to 32 days. The second group was removed from the water bath each afternoon and allowed to gel overnight. The following morning the specimens of this second group were immersed in boiling water for one hour and then replaced in the water bath. This heating and gelation cycle was repeated daily, exclusive of week-ends, for periods extending up to 32 days.

Samples of agar-agar were prepared by adding the powdered material to distilled water. A concentration of 3% was used in runs 1 and 3 and 5% in runs 2 and 4. These mixtures were placed in jars and kept in boiling water for one hour and then poured into mason jars and sealed. The jars were immediately placed in a water bath at 52°C. In three of the runs (1, 3 and 4) the samples were divided into two groups and treated in the same manner as the two groups of commercial agar-agar type duplicating material.

Changes in viscosity were measured with a Brookfield rotational viscometer. Readings were taken at four rates of shear, corresponding to 2, 4, 10 and 20 revolutions per minute. Changes in pH were recorded by the use of a Beckman model G pH meter equipped with external electrodes. By the use of the external electrodes it was possible to measure the pH of the specimens





while they were immersed in the constant temperature bath. Viscosity changes were measured at 52°C. All data for both pH and viscosity were made upon the same specimens at different ages. Crushing time was measured with a compression tester under a constant load of 300 grams per square centimeter. The time, in seconds, was recorded from the application of the 300 gram load until the specimen completely fractured. The groups were stored overnight in the 52°C water bath before the first readings were made. Hence, the reboiled\* specimens had been reboiled once before the initial readings were made. All measurements were made in a room with a constant temperature of 22°C and a relative humidity of 55 percent.

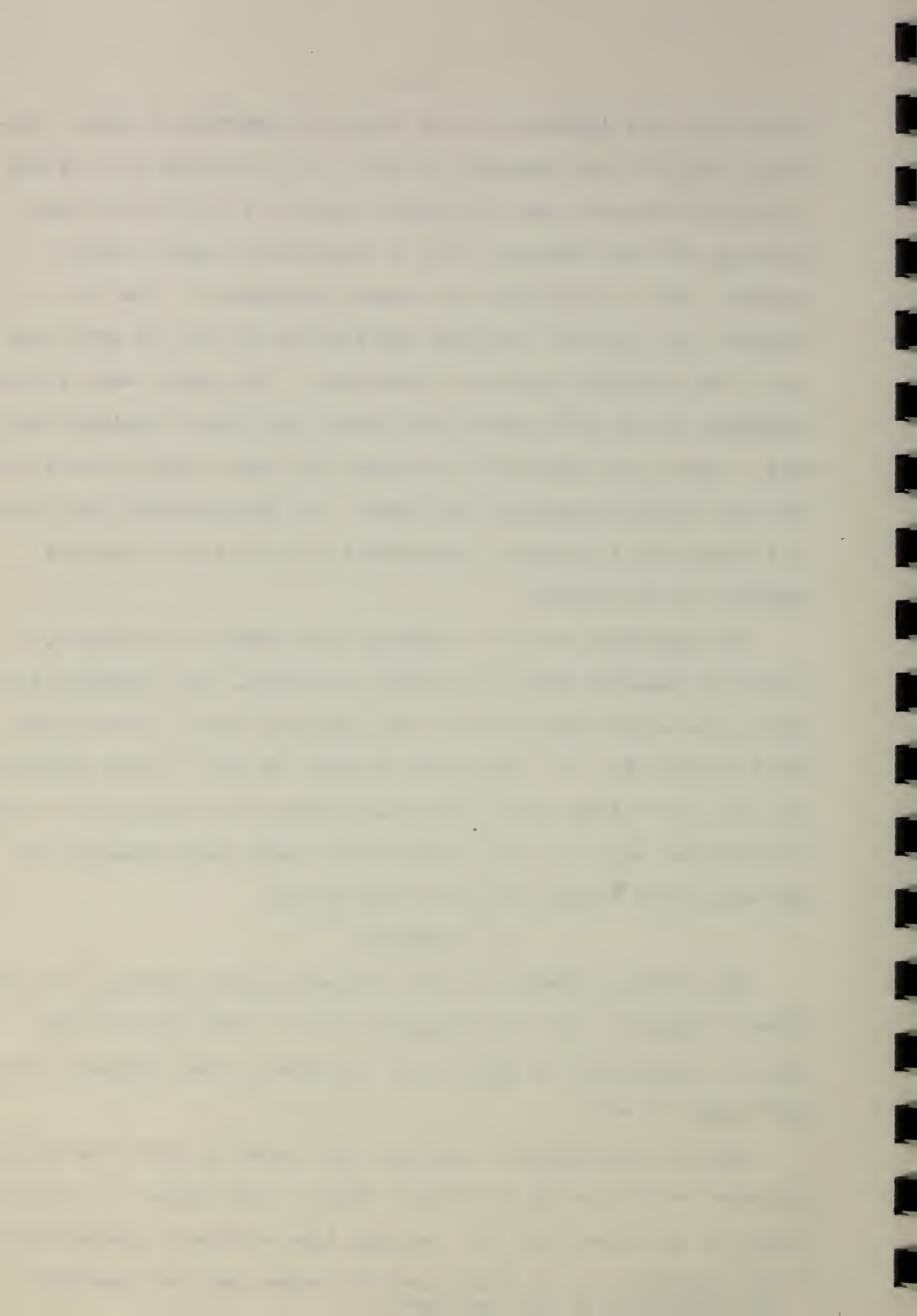
The specimens for the crushing time tests, cylinders 0.5 inches in diameter and 0.75 inches in length, were prepared according to the method described in the American Dental Association Specification No. 11. They were allowed to gel in room temperature air for thirty minutes and then removed and placed in a beaker of distilled water at room temperature where they remained for another thirty minutes before being tested.

### 3. RESULTS

The results obtained on pH, viscosity and crushing time are given in Tables 1 to 5 and Figures 1 to 4. The figures show typical curves and are taken from the data of one specimen from each group in run 4.

When the duplicating compound was stored at 52°C the viscosity increased while the pH decreased, Table 1 and Figure 1. In the figure it is shown that the crushing time decreased greatly and

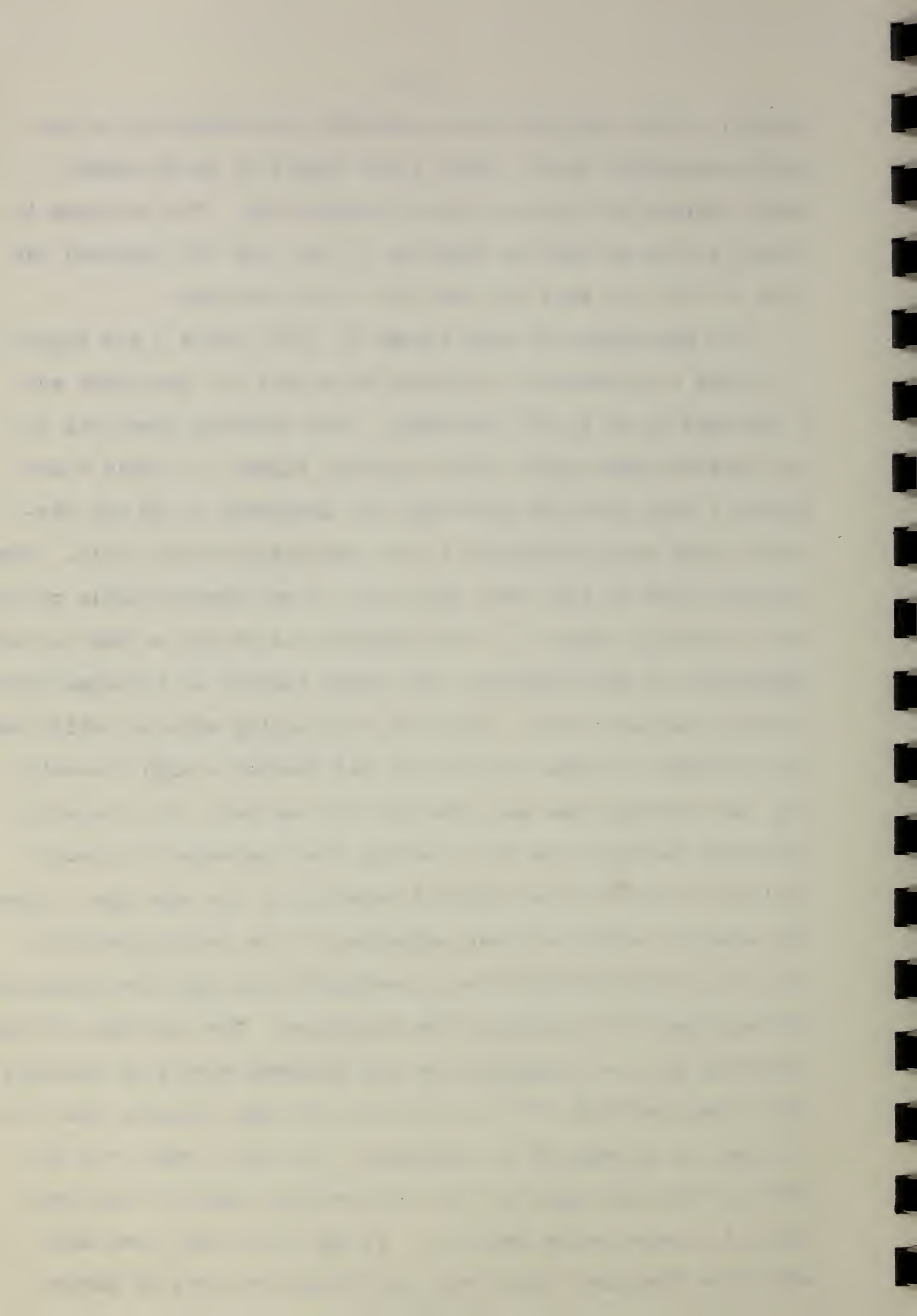
\* By "reboiled" it is meant that the mason jars are immersed in boiling water for one hour.





rapidly. When the duplicating material was subjected to the heating-gelation cycle, Table 2 and Figure 2, pH decreased while changes in viscosity were inconsistent. The decrease in crushing time was not as rapid as it was when the compound was held at 52°C nor were the same low values reached.

The agar-agar sol when stored at 52°C, Table 3 and Figure 3, showed a decrease in viscosity in almost all specimens and a decrease in pH in all specimens. The crushing time fell to an indeterminable point quite rapidly, Figure 3. Table 4 and Figure 4 show that the agar-agar sol decreased in pH and viscosity upon being subjected to the heating-gelation cycle. The crushing time in this case also fell to an indeterminable point quite rapidly, Figure 4. The reduction of pH may be due to the hydrolysis of the sulphuric acid ester radical of the agar-agar, forming sulphuric acid. When the duplicating material which had been allowed to stand at 52°C and had reached a high viscosity and low crushing time was reboiled for one hour, the viscosity decreased markedly and the crushing time increased to a point similar to that of the reboiled material of the same age. When the material which had been subjected to the heating-gelation cycle was stored at 52°C for a period of five days the viscosity increased and the crushing time decreased. The condition of high viscosity and low crushing time was reversed merely by boiling, while the condition of low viscosity and high crushing time was reversed by storage of the material. As can be seen from the tables, both agar-agar and the duplicating compound displayed typical non-Newtonian behavior. It was also found that both materials displayed thixotropy, although the curve of torque vs rpm was not linear for decreasing rpm.





As can be seen in Table 3 the mean deviation is relatively high in runs 2 and 4. This is due to one specimen in each run which did not have the general trend in viscosity as did the other specimens in the run. The pH and viscosity values for each run are given in Table 5 which shows the average values for all the specimens of the run, the average eliminating the specimen whose viscosity is anomolous and the data for this specimen alone at each age period. As can be seen, the effect on the mean deviation is slight initially, but becomes more pronounced towards the end of the run.

That some chemical change had occurred was indicated by the change in pH. To determine the nature of this change, x-ray diffraction and infrared absorption spectra were made. The x-ray patterns of duplicating material which had been stored at 52°C, and that which had been subjected to the heating-gelation cycle exhibited no significant difference, Figure 5. The patterns were typical x-ray scattering curves of an amorphous material showing no tendency of the gels to form crystals or ordered regions. Agar-agar, 5%, gave similar patterns. Infrared absorption spectra were obtained from potassium bromide pellets containing finely divided samples of dessicated agar type duplicating material which had been stored at 52°C and samples of the material which had been subjected to the daily heating and gelation cycle. No significant differences were found, Figure 6.

Some samples of duplicating material were stored at 52°C and left undisturbed, no measurements being performed upon them. In all cases complete gelation occurred between the sixth and ninth day of storage.



#### 4. DISCUSSION

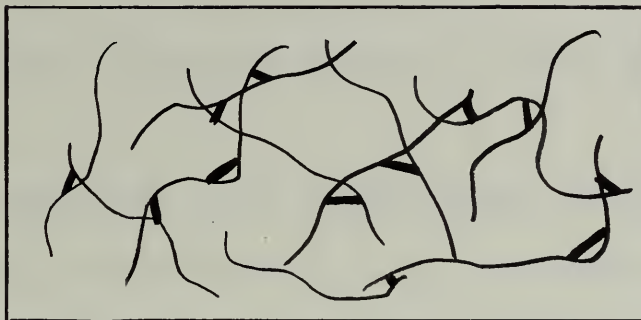
As shown in Figure 1, when agar-agar type duplicating compound was stored at 52°C the viscosity increased while the crushing time decreased to a point where it would support a load of 300 grams per square centimeter for only two seconds. The increase in viscosity, exclusive of that due to pH change, may be attributed to bonding of the agar-agar molecule at first by secondary attractive forces at widely separated points followed by more bonding by nonlocalized secondary attractive forces as the material is allowed to age [1]. Although some of the increase in the viscosity observed in the stored duplicating material is due to an electroviscous effect caused by the decrease in pH, there are three phenomena which indicate that the agglomeration of agar-agar molecules contributes to this increase. Firstly, the fact that complete gelation occurs in the duplicating material after a few days of undisturbed storage at 52°C. Secondly, the increase found in the viscosity of some of the samples of agar-agar sol, even though the pH is changing in a direction which should cause a decrease in viscosity. Finally, the fact that the duplicating material reboiled for one hour displays no correlation with the change in pH. Had the material been reboiled for longer periods of time than one hour, the viscosity would probably have decreased continuously due to a more complete breakdown of the hydrogen bonds.

The decrease in crushing time as the material ages at 52°C could also be explained on the basis of increased hydrogen bonding with age. If a specimen were prepared from the material when

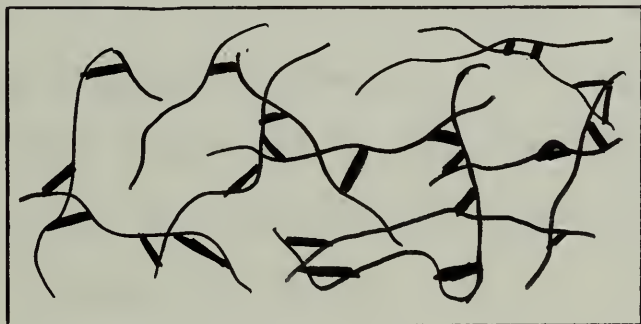




it had been stored only for two or three days, the bonding would appear schematically as follows:



But if a sample for crushing were prepared from the same material which was somewhat older; schematically, the hydrogen bonding would appear in this manner:



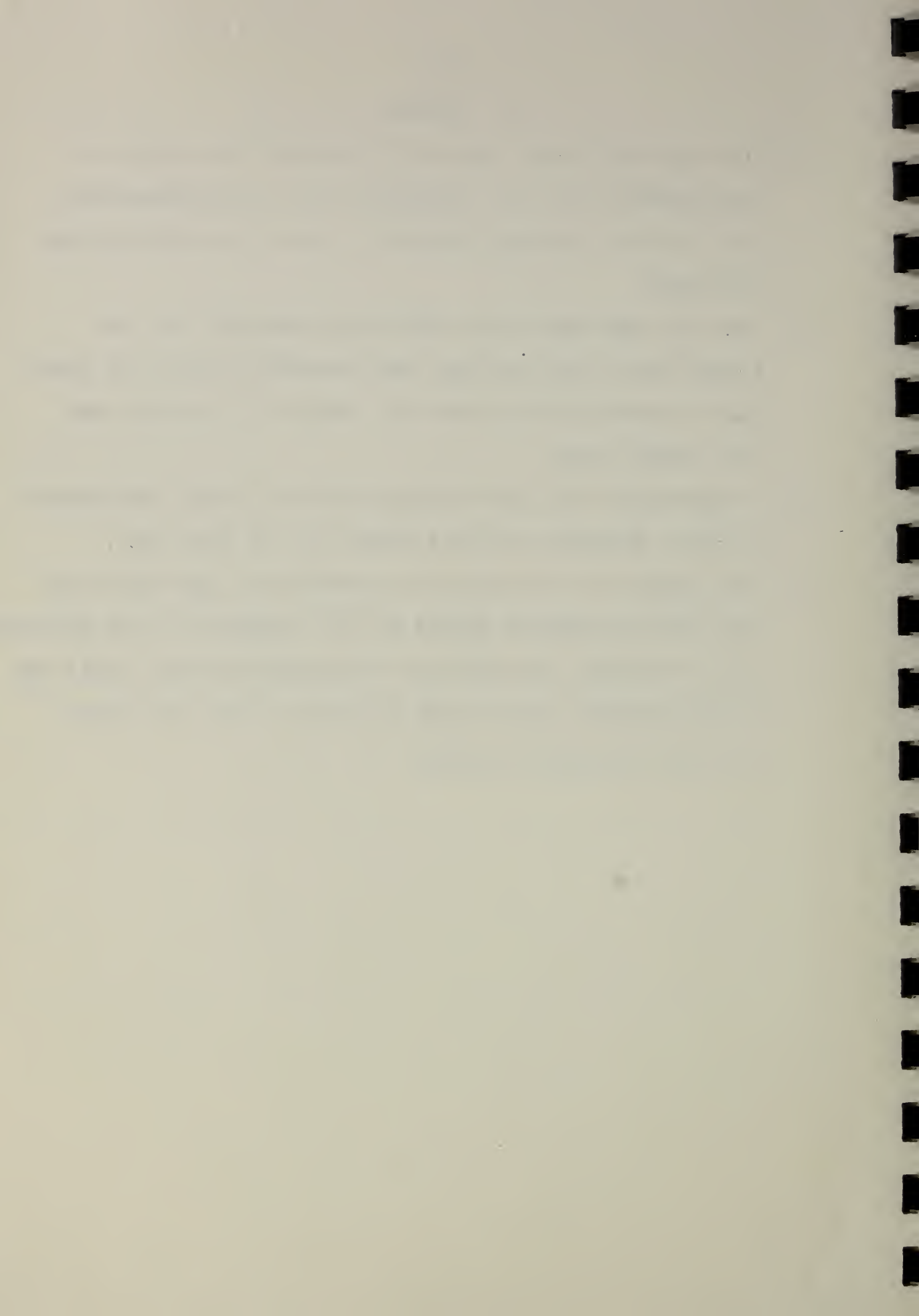
The few bonds in sample 1, permit deformation so that many bonds must be severed simultaneously to rupture the specimen. In sample 2, the many bonds stiffen the specimen and little deformation is possible. Consequently, one bond at a time may be broken resulting in a low crushing time. Further measurements on the mechanical properties of agar gels are required to substantiate this hypothesis.





## 5. SUMMARY

1. When agar-agar type duplicating material was stored at approximately 52°C as recommended by the manufacturers, the viscosity increased markedly while the crushing time decreased.
2. When the agar-agar type duplicating material was re-boiled daily the crushing time decreased slowly but there was no correlation between the change in viscosity and the change in pH.
3. In agar-agar type duplicating material stored undisturbed at 52°C, gelation occurred within six to nine days.
4. The changes in the physical properties of agar-agar type duplicating compound stored at 52°C compared to the material which has been subjected to the heating-gelation cycle may be explained by postulating increased intra- and inter-molecular hydrogen bonding.



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Table 1

pH AND VISCOSITY OF DUPLICATING  
MATERIAL STORED AT 52°C

Run	Age in Days	No. of Spec.	pH	Viscosity in Poise			
				2 rpm	4 rpm	10 rpm	20 rpm
1	1	3	8.07±0.01	122 ± 4	72.7± 0.8	40.3± 0.2	25.8 ± 0.2
	3	3	8.17±0.08	192 ± 12	113 ± 8	63.4± 3.7	41.1 ± 2.2
	6	3	8.03±0.02	271 ± 9	166 ± 6	90.2± 2.8	62.3 ± 1.1
	10	3	7.81±0.05	374 ± 9	218 ± 8	126 ± 4	76.8 ± 4.1
2	1	5	8.08±0.03	44.8± 2.2	26.7± 1.2	14.9± 0.8	9.78± 0.57
	4	5	7.80±0.07	50.3± 1.1	30.6± 0.5	17.2± 0.5	11.6 ± 0.2
	7	5	7.79±0.08	70.2± 2.0	43.3± 1.1	24.1± 0.7	15.9 ± 0.5
	10	5	7.72±0.07	83.2± 3.3	51.6± 2.0	28.8± 1.0	19.4 ± 1.5
	14	5	7.77±0.04	110 ± 6	66.8± 3.1	36.2± 1.4	25.5 ± 0.9
3	1	2	8.17±0.01	35.6± 1.6	23.2± 1.3	13.7± 0.9	9.69± 0.63
	4	2	8.00±0.00	64.8± 3.4	40.5± 1.6	22.5± 0.8	15.0 ± 0.3
	11	2	7.88±0.01	245 ± 15	145 ± 6	76.4± 2.0	47.9 ± 0.5
	18	2	7.57±0.00	484 ± 14	280 ± 8	142 ± 6	88.2 ± 2.5
4	1	3	8.00±0.02	74 ± 3	43 ± 1	23.7± 0.5	15.6 ± 0.5
	4	3	7.92±0.02	122 ± 12	75 ± 8	41.1± 4.5	26.4 ± 2.9
	8	3	7.75±0.02	224 ± 26	137 ± 17	73.9± 9.4	47.3 ± 5.5
	11	3	7.72±0.02	282 ± 57	173 ± 26	89.5± 18.0	56.7 ± 10.9
	17	3	7.56±0.01	422 ± 108	249 ± 65	128 ± 33	79.0 ± 19.7
	23	3	7.30±0.03	647 ± 211	372 ± 124	187 ± 65	116 ± 38
	32	3	7.20±0.03	814 ± 232	507 ± 164	267 ± 91	162 ± 54



Table 2

pH AND VISCOSITY OF DUPLICATING  
MATERIAL REBOILED DAILY

Run	Age in Days	No. of Spec.	pH	Viscosity in Poise			
				2 rpm	4 rpm	10 rpm	20 rpm
1	1	3	8.06±0.00	127 ± 1	75.0± 0.7	41.0±0.8	26.0 ±0.5
	3	3	8.01±0.07	57 ± 2	35 ± 2	20.0±1.6	13.3 ±1.2
	6	3	7.86±0.01	57 ± 2	32 ± 2	18.5±1.3	12.3 ±0.6
	10	3	7.65±0.04	59 ±21	36 ±12	22.1±6.0	16.6 ±3.1
3	1	2	8.04±0.02	24.6± 0.8	17.5± 0.0	11.0±0.5	7.97±0.49
	4	2	7.78±0.00	65.8± 7.4	38.2± 3.4	21.6±2.4	14.1 ±1.4
	11	2	7.52±0.01	98 ± 4	56.0± 2.0	29.1±1.1	18.4 ±0.2
	18	2	7.42±0.01	93 ± 0	55.0± 0.5	28.8±0.8	18.2 ±0.6
4	1	3	7.92±0.01	61 ±10	36 ± 5	19.8±1.9	13.0 ±0.6
	4	3	7.73±0.02	100 ± 5	60 ± 2	29.2±1.4	17.8 ±1.0
	8	3	7.64±0.01	69 ± 6	40 ± 4	21.4±2.4	13.8 ±1.5
	11	3	7.43±0.01	127 ±19	72 ±11	38.1±4.3	23.9 ±2.0
	17	3	7.34±0.01	55 ± 8	35 ± 5	19.1±2.5	12.6 ±1.3
	23	2	7.24±0.01	35 ± 1	22 ± 0	12.6±0.1	8.9 ±0.7
	32	2	7.30±0.00	70 ±25	40 ±13	20.5±6.0	13.2 ±3.5





Table 3

## pH AND VISCOSITY OF AGAR-AGAR STORED AT 52°C

Run	Age in Days	No. of Spec.	pH	Viscosity in Poise			
				2 rpm	4 rpm	10 rpm	20 rpm
1	1	3	5.51±0.07	21.8 ± 0.3	12.6 ± 0.2	6.55±0.07	4.19±0.11
	3	3	5.96±0.01	17.2 ± 0.4	10.5 ± 0.2	5.74±0.09	3.83±0.04
	6	3	5.80±0.01	12.0 ± 0.5	7.07 ± 0.21	3.75±0.13	2.46±0.08
	10	3	5.70±0.05	9.2 ± 0.7	5.4 ± 0.3	2.81±0.19	1.83±0.14
2	1	5	5.76±0.05	117 ± 16	64.1 ± 5.9	31.3 ± 3.2	19.2 ± 1.1
	4	5	5.81±0.02	109 ± 12	61.1 ± 6.1	29.6 ± 2.4	18.3 ± 1.1
	7	5	5.98±0.04	107 ± 10	60.9 ± 5.3	30.1 ± 2.8	18.5 ± 1.5
	10	5	5.74±0.03	82 ± 21	46.0 ± 11.0	23.1 ± 4.9	14.3 ± 3.0
	14	5	6.00±0.09	82 ± 18	45.0 ± 8.6	22.5 ± 6.7	13.9 ± 3.3
3	1	2	5.81±0.02	1.1 ± 0.1	0.95 ± 0.05	0.70 ± 0.02	0.64 ± 0.01
	4	2	5.78±0.04	0.40 ± 0.00	0.40 ± 0.05	0.38 ± 0.03	0.36 ± 0.03
	11	2	6.34±0.20	0.3 ± 0.1	0.3 ± 0.0	0.30 ± 0.02	0.33 ± 0.05
	18	2	6.26±0.02	0.4 ± 0.2	0.3 ± 0.1	0.20 ± 0.05	0.26 ± 0.04
4	1	3	5.65±0.04	32.3 ± 4.6	20.9 ± 2.1	11.2 ± 1.2	7.44 ± 0.68
	3	3	5.18±0.10	28.5 ± 8.1	17.8 ± 5.1	9.67 ± 2.22	6.46 ± 1.64
	7	3	5.19±0.04	19.1 ± 8.6	11.2 ± 5.1	5.7 ± 2.6	3.59 ± 1.65
	10	3	5.20±0.03	13 ± 7	7.7 ± 4.0	3.8 ± 2.0	2.3 ± 1.2
	16	3	5.13±0.04	13 ± 8	7.2 ± 4.2	3.5 ± 1.7	2.08 ± 1.20
	22	2	5.03±0.00	19.5 ± 10.7	10.8 ± 5.9	5.2 ± 2.8	3.1 ± 1.6
	31	2	5.11±0.07	25.1 ± 17.1	13.9 ± 9.4	6.6 ± 4.3	3.4 ± 1.6





Table 4

pH AND VISCOSITY OF AGAR-AGAR  
REBOILED DAILY

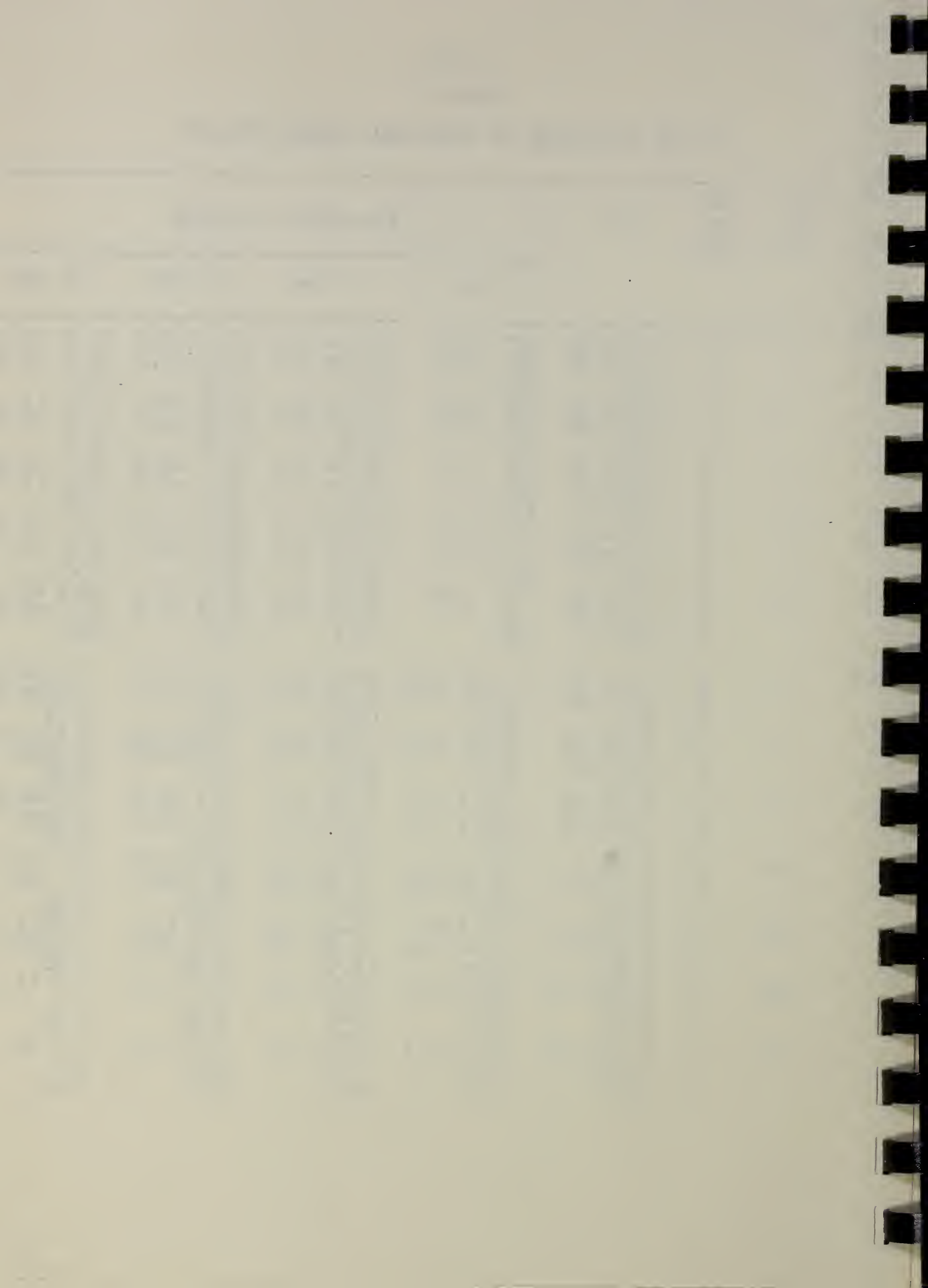
Run	Age in Days	No. of Spec.	pH	Viscosity in Poise			
				2 rpm	4 rpm	10 rpm	20 rpm
4	1	3	6.21±0.02	6.5 ±0.6	4.6 ±0.4	2.9 ±0.3	2.26±0.12
	3	3	5.99±0.02	1.7 ±0.4	1.5 ±0.2	1.2 ±0.1	1.1 ±0.1
	7	3	5.99±0.10	3.7 ±1.8	2.6 ±1.4	1.7 ±0.6	1.4 ±0.4
	10	3	5.83±0.07	0.8 ±0.2	0.7 ±0.1	0.61±0.14	0.57±0.11
	16	3	5.58±0.08	0.6 ±0.1	0.4 ±0.1	0.37±0.12	0.40±0.07
	22	3	5.53±0.09	0.3 ±0.1	0.3 ±0.0	0.2 ±0.0	0.29±0.02
	31	3	5.14±0.12	0.1 ±0.1	0.2 ±0.1	0.2 ±0.0	0.1 ±0.00
1	1	3	5.45±0.01	19.8 ±1.6	11.9 ±0.4	6.58±0.08	4.45±0.07
	3	3	5.42±0.01	3.63±0.56	2.27±0.21	1.47±0.17	1.10±0.15
	6	3	5.34±0.06	2.2 ±0.4	1.5 ±0.2	0.92±0.09	0.69±0.07
	10	3	5.28±0.09	0.3 ±0.1	0.2 ±0.0	0.20±0.03	0.23±0.01
3	1	2	6.43±0.00	0.4 ±0.1	0.4 ±0.1	0.49±0.01	0.50±0.01
	4	2	6.19±0.00	0.4 ±0.2	0.4 ±0.2	0.38±0.05	0.42±0.05
	11	2	5.91±0.04	0.2 ±0.0	0.2 ±0.0	0.22±0.02	0.30±0.05
	18	2	5.72±0.01	0.2 ±0.1	0.2 ±0.1	0.20±0.01	0.20±0.01



Table 5

pH AND VISCOSITY OF AGAR-AGAR STORED AT 52°C

Run	Age in Days	No. of Spec.	pH	Viscosity in Poise			
				2 rpm	4 rpm	10 rpm	20 rpm
2	1	5	5.76±.05	117 ±16	64.1± 5.9	31.3 ±3.2	19.2 ±1.1
		4	5.73±.04	120 ±18	64.9± 6.7	31.5 ±3.7	19.2 ±1.6
		1	5.84	106	61.0	30.2	19.2
	4	5	5.81±.02	109 ±12	61.1± 6.1	29.6 ±2.4	18.3 ±1.1
		4	5.81±.02	107 ±13	60.5± 6.8	29.3 ±2.5	18.2 ±1.4
		1	5.82	116	63.5	31.0	18.6
	7	5	5.98±.04	107 ±10	60.9± 5.3	30.1 ±2.8	18.5 ±1.5
		4	5.99±.04	106 ±11	60.1± 5.2	29.7 ±2.9	18.2 ±1.4
		1	5.96	112	64.0	32.0	19.8
	10	5	5.74±.03	82 ±21	46.0±11.0	23.1 ±4.9	14.3 ±3.0
		4	5.73±.04	72 ±11	40.4± 5.6	20.4 ±2.1	12.6 ±1.1
		1	5.77	121	68.5	33.9	21.3
	14	5	6.00±.09	82 ±18	45.0± 8.6	22.5 ±6.7	13.9 ±3.3
		4	5.94±.03	70 ± 4	38.4± 2.3	19.2 ±1.3	11.8 ±0.7
		1	6.22	128	71.5	35.7	22.0
4	1	3	5.65±.04	32.3± 4.6	20.9± 2.1	11.2 ±1.2	7.44±0.68
		2	5.64±.05	33.8± 4.8	22.0± 2.1	11.7 ±1.3	7.78±0.68
		1	5.67	29.2	18.8	10.1	6.76
	3	3	5.18±.10	28.5± 8.1	17.8± 5.1	9.67±2.22	6.46±1.64
		2	5.26±.02	22.4± 1.6	14.0± 0.5	7.76±0.16	5.23±0.31
		1	5.04	40.6	25.4	13.5	8.92
	7	3	5.19±.04	19.1± 8.6	11.2± 5.1	5.7 ±2.6	3.59±1.65
		2	5.16±.02	12.7± 1.1	7.3± 0.7	3.8 ±0.3	2.35±0.15
		1	5.25	32.0	18.9	9.6	6.06
	10	3	5.20±.03	13. ± 7.	7.7± 4.0	3.8 ±2.0	2.3 ±1.2
		2	5.19±.03	8.2± 0.8	4.7± 0.5	2.3 ±0.3	1.4 ±0.3
		1	5.22	23.8	13.7	6.76	4.14
	16	3	5.13±.04	13. ± 8	7.2± 4.2	3.5 ±1.7	2.08±1.20
		2	5.14±.01	7.0± 2.0	4.0± 1.0	1.9 ±0.6	1.18±0.36
		1	5.03	24.0	13.5	6.52	3.88
	22	2	5.03±.00	19.5±10.7	10.8± 5.9	5.2 ±2.8	3.1 ±1.6
		1	5.03	8.8	4.9	2.4	1.5
		1	5.03	30.2	16.7	7.96	4.72
	31	2	5.11±.07	25.1±17.1	13.9± 9.4	6.6 ±4.3	3.4 ±1.6
		1	5.18	8.0	4.5	2.3	1.4
		1	5.04	42.2	23.3	11.0	6.36





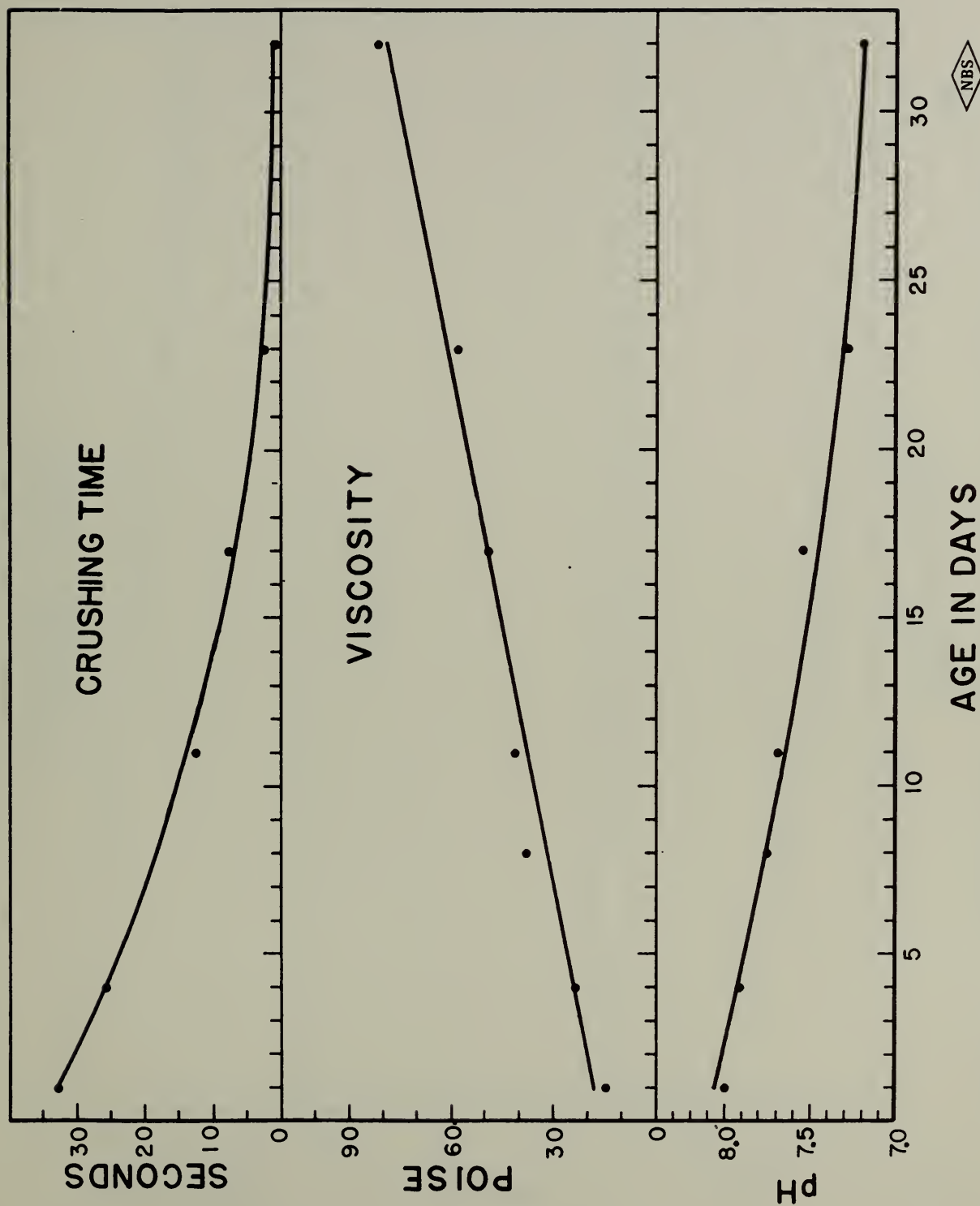


Figure 1. Crushing time, viscosity and pH as a function of time stored of commercial agar-agar type duplicating compound stored at 52°C.



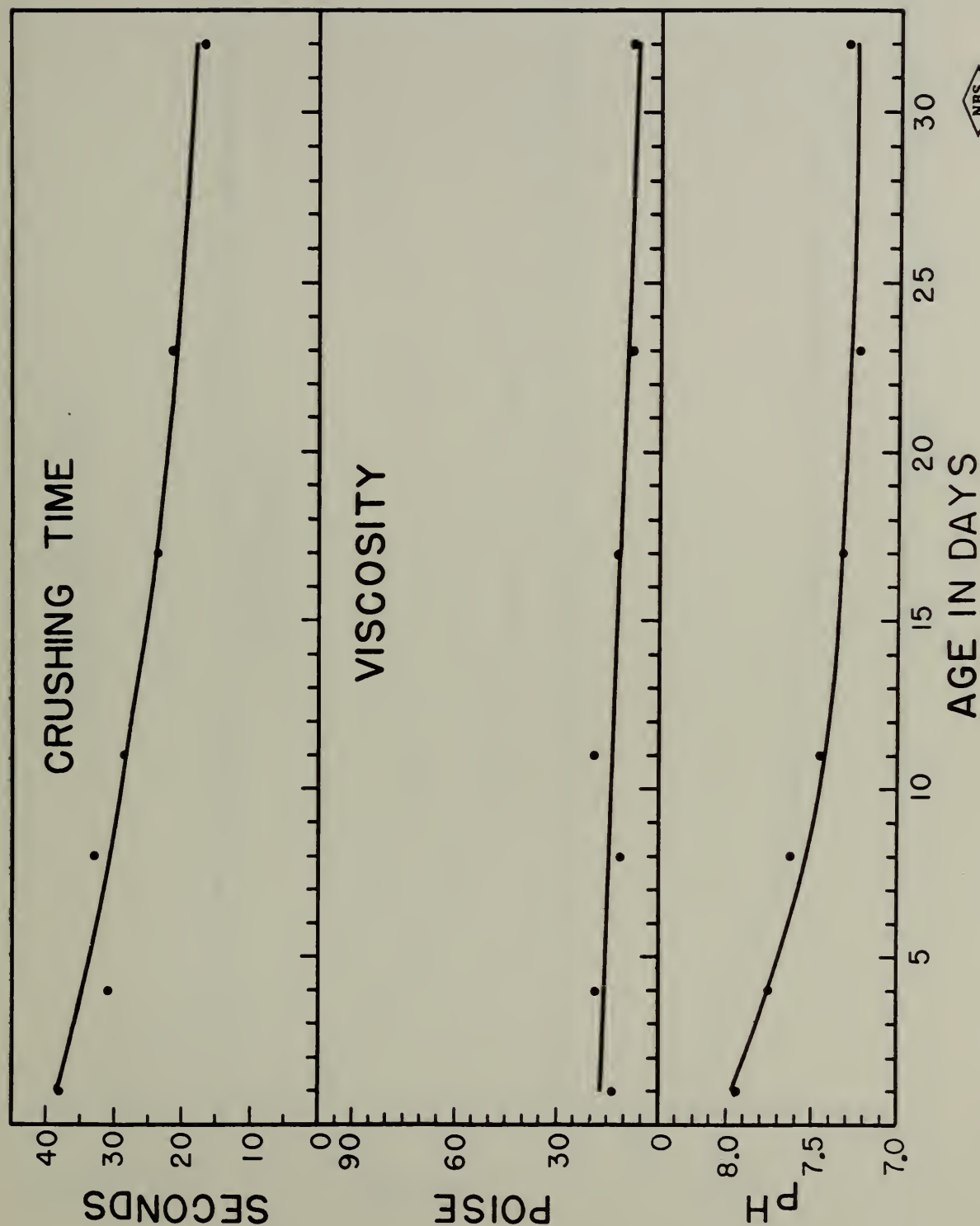


Figure 2. Crushing time, viscosity and pH as a function of time of re-boiled commercial agar-agar type duplicating compound.



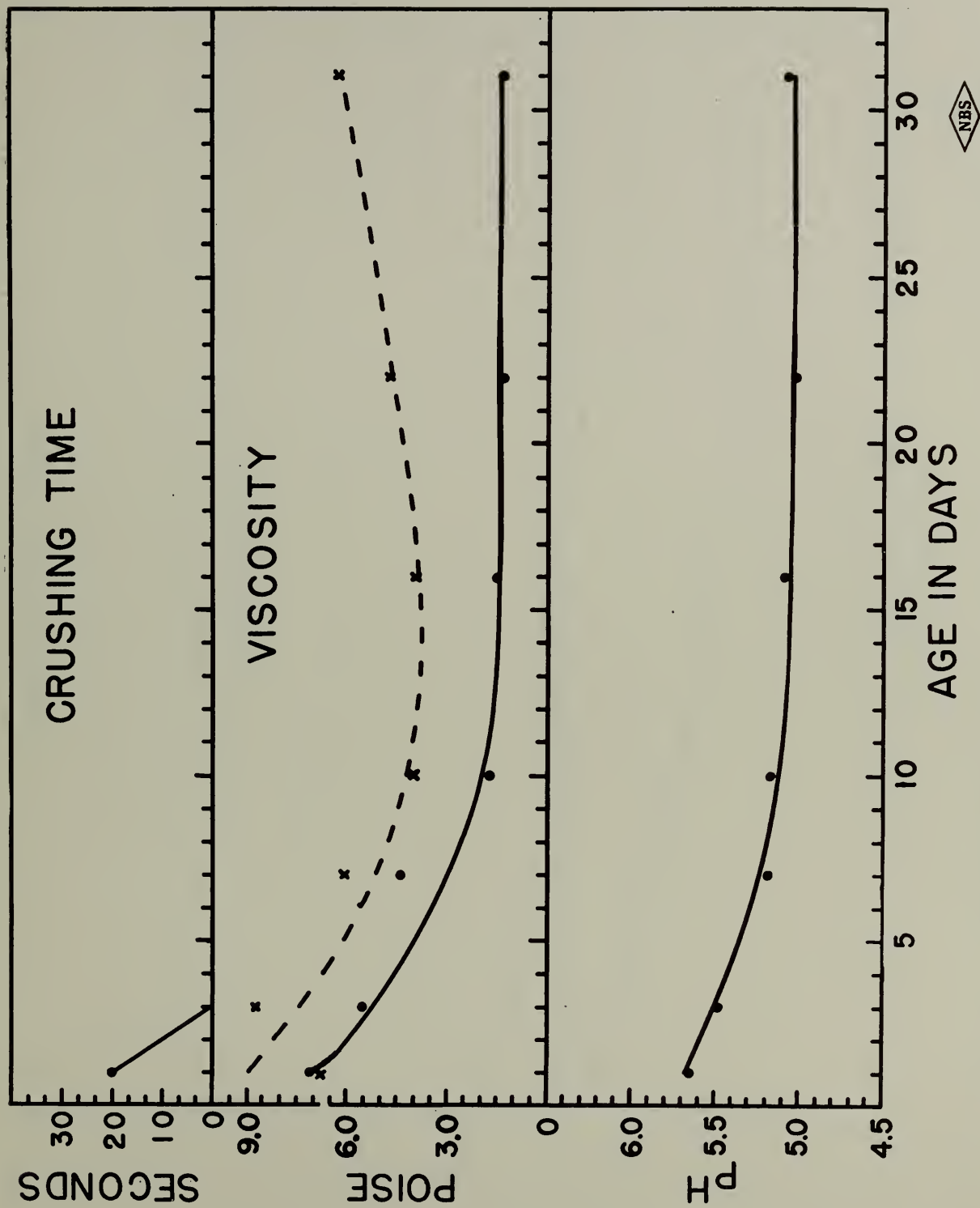


Figure 3. Crushing time, viscosity and pH as a function of time stored of 5% agar-agar.



Date	Description	Amount
1900	Jan 1	100.00
1900	Feb 1	100.00
1900	Mar 1	100.00
1900	Apr 1	100.00
1900	May 1	100.00
1900	Jun 1	100.00
1900	Jul 1	100.00
1900	Aug 1	100.00
1900	Sep 1	100.00
1900	Oct 1	100.00
1900	Nov 1	100.00
1900	Dec 1	100.00
1900	Total	1200.00

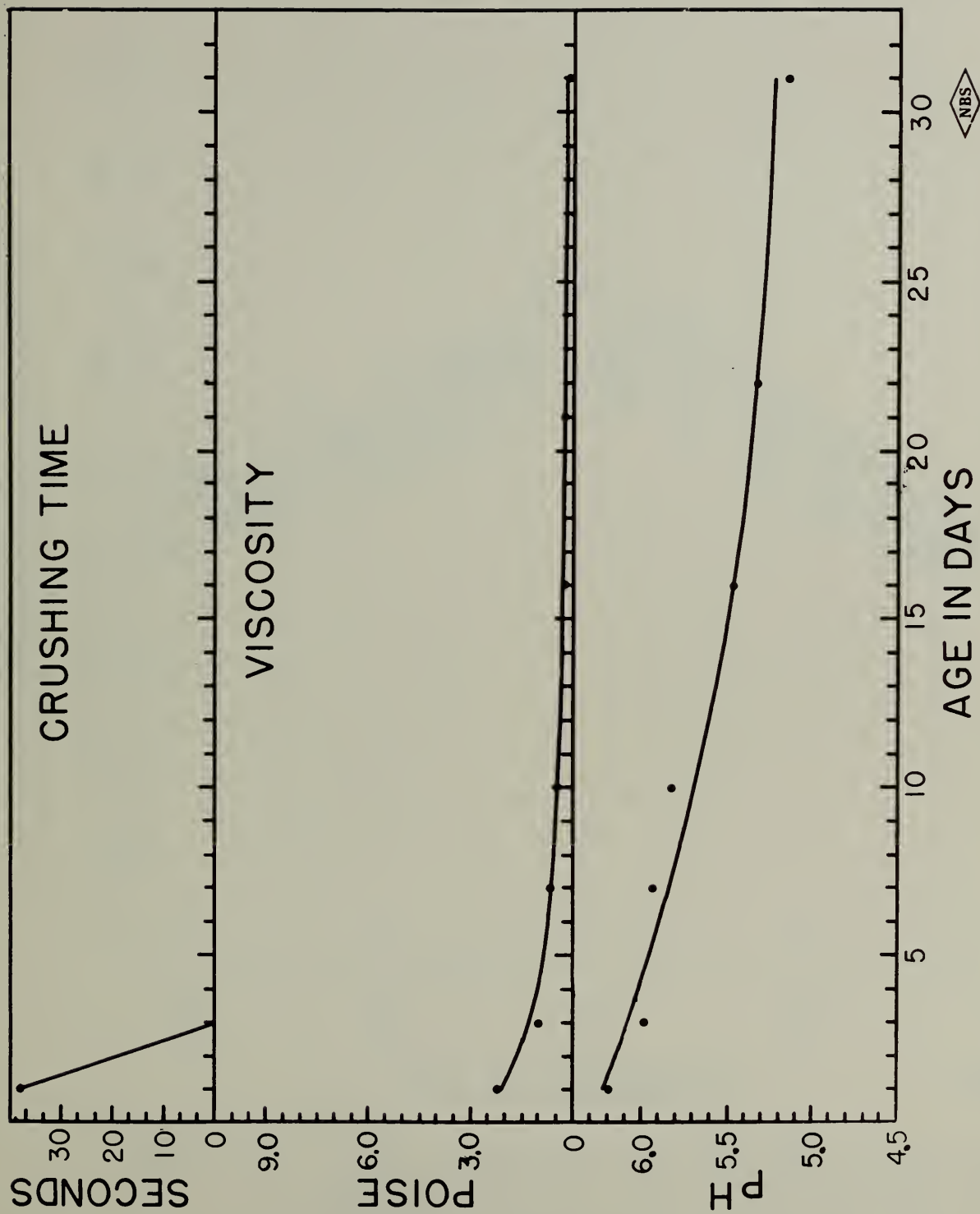


Figure 4. Crushing time, viscosity and pH as a function of time of reboiled 5% agar-agar.



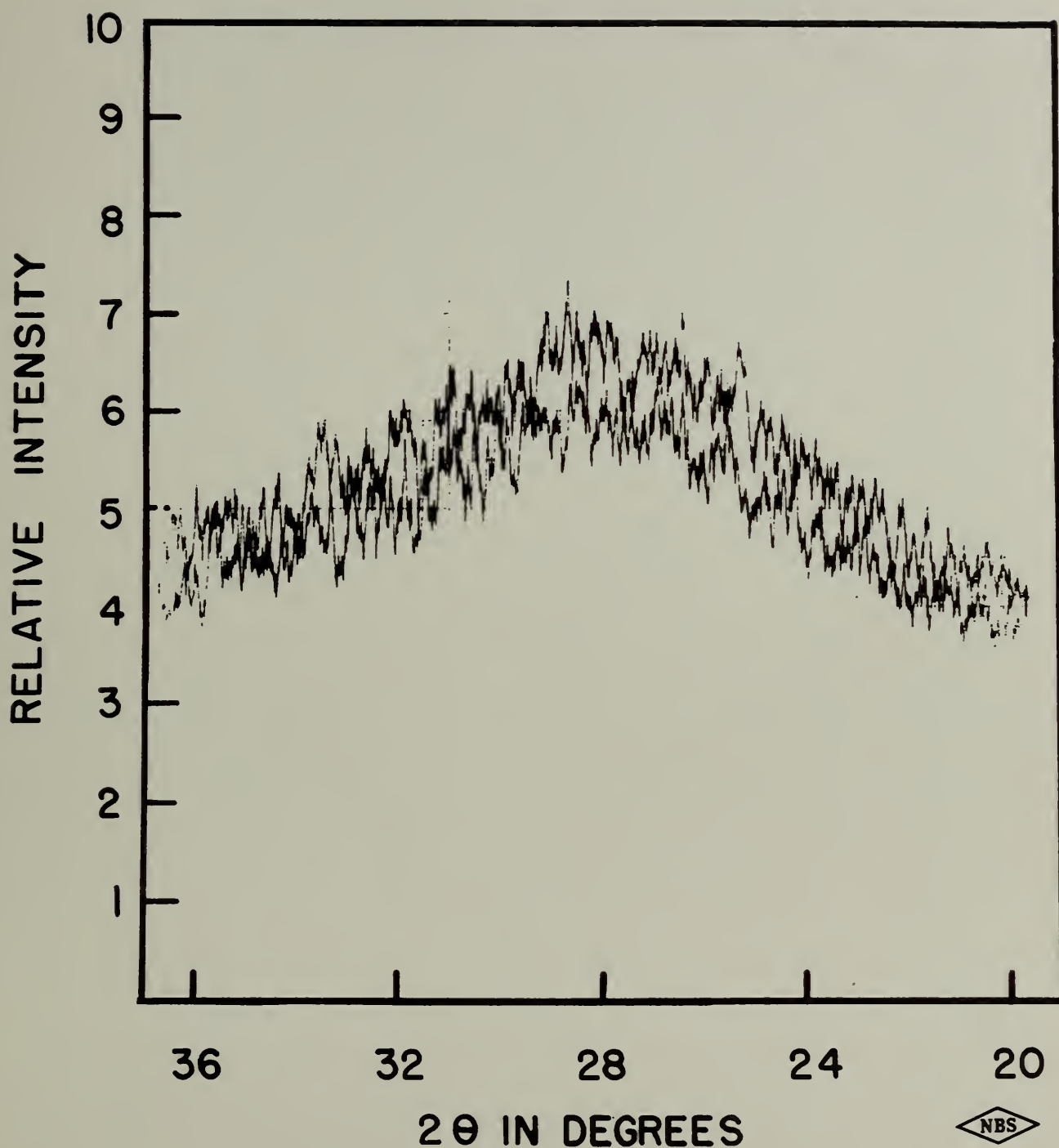


Figure 5. X-ray diffraction of agar-agar type duplicating compound. The top pattern is the reheated material and the bottom pattern is the material stored at 52°C.





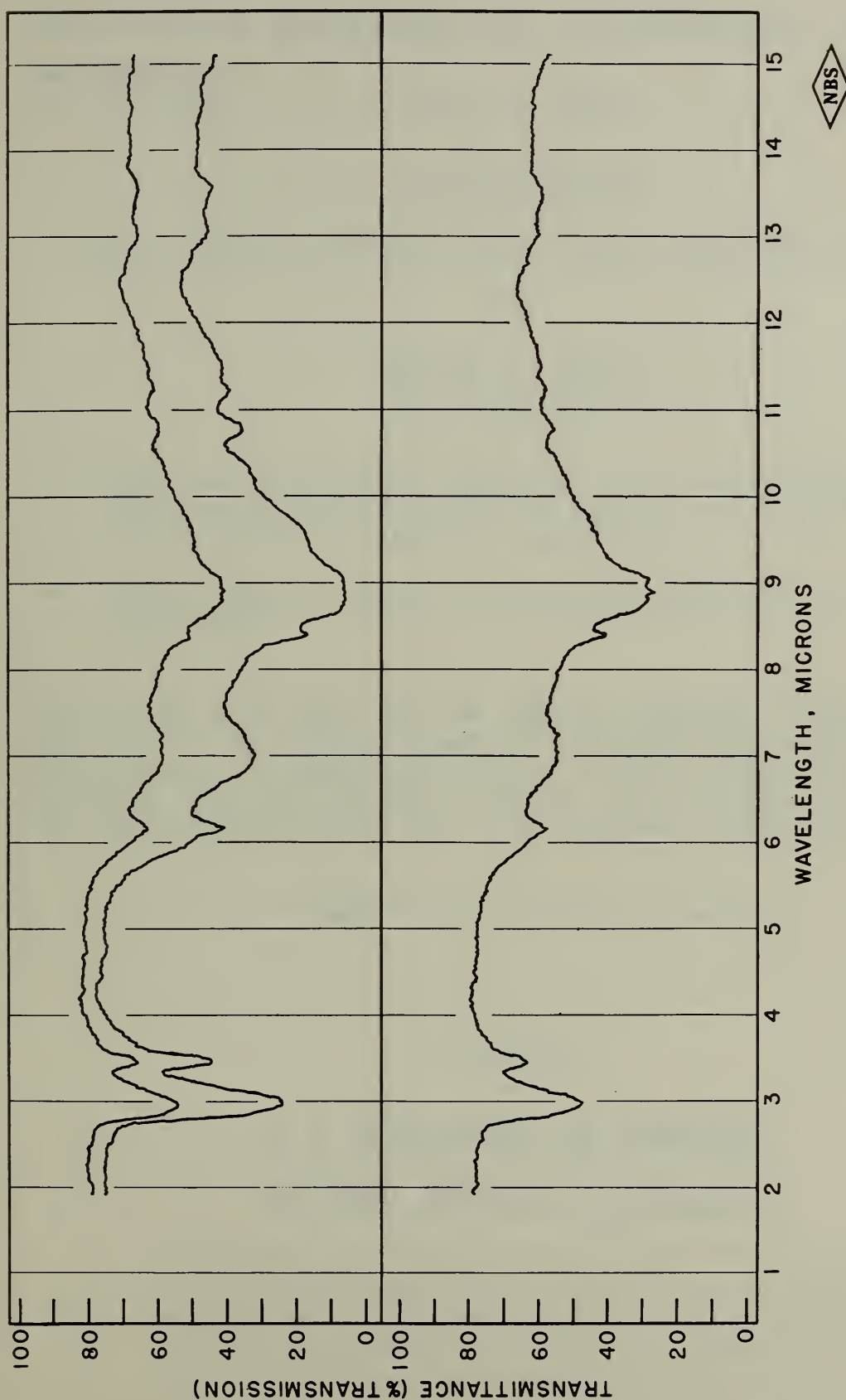


Figure 6. Curves in upper graph are the spectra of commercial agar-agar type duplicating compound stored at 52°C but made with different KBr pellet concentrations. The lower graph is the spectrum of the reheated commercial agar-agar type duplicating material.



# NATIONAL BUREAU OF STANDARDS REPORT

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## Progress Report

### SOME PHYSICAL PROPERTIES OF GALLIUM-COPPER-TIN ALLOYS

by

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(Task 7756-41 BV No. 55-14)



**U. S. DEPARTMENT OF COMMERCE**  
**NATIONAL BUREAU OF STANDARDS**

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# SOME PHYSICAL PROPERTIES OF GALLIUM-COPPER-TIN ALLOYS

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## Abstract

Some physical properties of gallium-copper-tin alloys were investigated. These alloys, formed by the combination of a liquid alloy of gallium and tin with copper powder and powdered alloys of copper and tin, are mixed, packed, and hardened in much the same manner as dental amalgam. Values were determined for the dimensional changes during hardening; compressive strength at one hour, one day, and one week; secant modulus of elasticity in compression; and hardness. The material formed by triturating the liquid gallium-tin alloy (36%) with powdered copper (64%) and the liquid gallium-tin alloy (30-35%) with powdered alloy (62% copper-38% tin) had physical properties that compared favorably with the corresponding properties of dental amalgam. Studies of corrosion resistance in the mouth, biological assay, and clinical evaluation of these alloys must be completed before they can be recommended for dentistry.

## 1. INTRODUCTION

In a previous investigation [1] it was found that alloys of the gallium-copper-tin system had physical properties that might be utilized for oral restorations. These alloys are silvery in appearance. Liquid gallium combines with copper powder and powdered copper-tin alloys to form products which will harden at room or mouth temperature in a manner similar to the silver amalgam now used in dentistry. One of the difficulties in using pure gallium as the liquid metal component of a filling alloy was the necessity of preheating the mixing device before trituration in order to melt the gallium as its melting point,  $29.78^{\circ}\text{C}$  ( $85.6^{\circ}\text{F}$ ) [2] is slightly above ordinary room temperature. An alloy of 89% gallium and 11% tin which melted at about  $20^{\circ}\text{C}$  ( $68^{\circ}\text{F}$ ) was used in this investigation as the liquid component to make alloys which harden at or below body temperature. Although these gallium-copper-tin materials are referred to as alloys in this paper, they may metallurgically be closer to the "cemented" materials in which the powder particles are held together by limited alloying action with the liquid component. The dimensional change during setting, compressive strength, the secant modulus of elasticity, and hardness of these alloys were determined because knowledge of these properties is necessary in evaluating filling materials.



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### DEPARTMENTS

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## 2. MATERIALS AND METHODS

Copper powder\* and powdered bronze\*\* (90% copper-10% tin) were obtained commercially. Copper-tin alloys of other compositions were made from reagent grade tin and high purity bulk copper. The latter alloys, which comprise several of the phases of the copper-tin system, were as follows: (1) 77% copper-23% tin; (2) 73% copper-27% tin; (3) 68% copper-32% tin; (4) 62% copper-38% tin; and (5) 41% copper-59% tin. Ingots of approximately one-half pound were prepared by induction melting the carefully weighed components in a graphite crucible. The melt was manually stirred and then allowed to cool in the crucible. The composition of the alloy was assumed to be that of the weighed components. Alloys containing 77%, 73%, and 41% copper were machined into particles on a lathe. The 77% and 73% copper alloys were quite tough which made comminution by this method difficult. The brittle alloys containing 68% and 62% copper were powdered in a diamond mortar. Only that portion of the alloy powder that passed a No. 325 sieve of the U. S. Standard Sieve series was used. All the graded powders were boiled in distilled water for 15 minutes, washed and decanted twice with alcohol and once with ether and dried on a steam bath. This procedure served to clean and dry the powders. It was reported by Kroll [3] in 1932 that an alloy of 89% gallium and 11% tin formed a eutectic melting at 15°C (59°F). Also, in 1932, Puschin [4] reported the composition of the eutectic as 92% gallium and 8% tin and having a melting point of about 20°C (68°F). No attempt was made to resolve the discrepancy of the eutectic composition in this investigation.

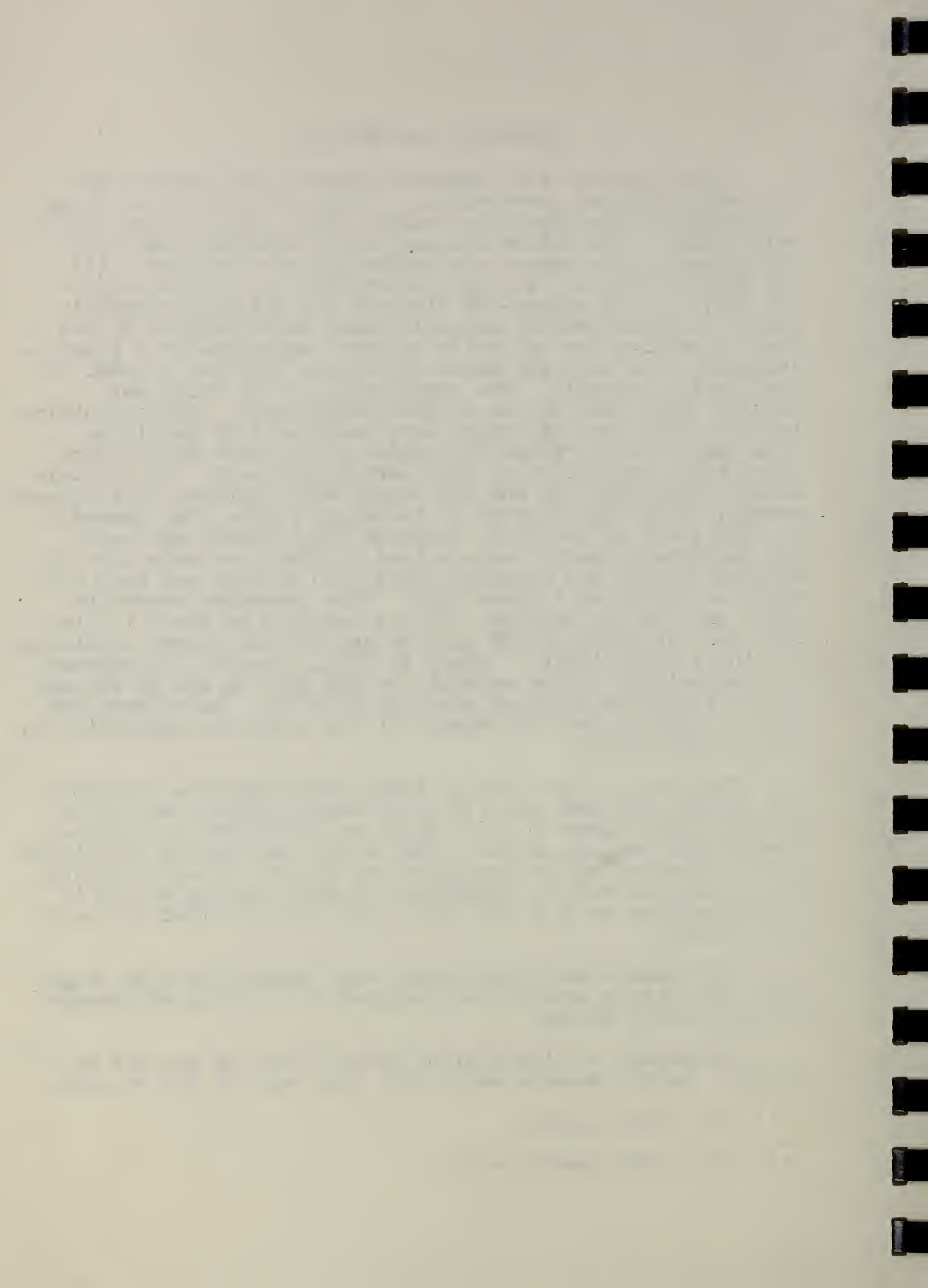
The matrix alloy used in these investigations contained 89% gallium (99.9+%) and 11% tin (reagent grade), and had a melting point close to 20°C (68°F) as determined by repeated heating and cooling curves. This alloy was used as the liquid metal component and is designated as the gallium-tin eutectic. This alloy was made by immersing a weighed amount of tin in liquid gallium at 37°C (98.6°F). The tin dissolved in two or three hours.

The powders and liquid alloy were proportioned by weighing. The liquid eutectic was weighed in a Teflon cup because it did not wet Teflon.

The amount of liquid alloy varied from 30% and 40% by weight. Lesser amounts made a mix that was too dry to pack,

\* 99% minimum purity.

\*\* Meets ASTM specification.





and a larger amount of gallium-tin eutectic in the mix made it too wet to pack easily.

The gallium-tin eutectic was triturated with the powdered copper-tin alloys in a glass mortar fitted with a stainless steel pestle under a constant load of three pounds. Trituration was at the rate of 140 revolutions per minute of the pestle, for periods of from 30 to 60 seconds, depending on the size of the mix. The mixes were packed in steel molds using heavy hand pressure on Sweeney-type pluggers having face diameters of 3.3 and 4.3 mm. No liquid metal was expressed at any time in making the specimens.

### 3. RESULTS

#### 3.1 Dimensional Changes During Hardening

The dimensional changes during hardening of the alloys were determined with the dental interferometer, using the standard size (5 mm diameter, 10 mm long) specimen. The specimen required a mix of two grams which was triturated 140 revolutions in 60 seconds. A fiducial reading was taken 15 minutes after the start of the mix. The dimensional changes of each alloy were determined at 37°C (98.6°F) and 21.1°C (70°F) for seven days the results of which are given in Table 1.

Two compositions of these materials, namely, 73% copper and 77% copper, the remainder being tin, when combined with the gallium-tin eutectic resulted in alloys having setting expansions of from 29 to 46  $\mu$ /cm\* at 37°C (98.6°F). Further investigations of these two alloys were discontinued because of the high expansion.

The setting expansion of the 90% copper-10% tin alloy when combined with the gallium-tin eutectic was 26  $\mu$ /cm at 37°C (98.6°F). This may be an excessive expansion. Other compositions expanded as much as 16  $\mu$ /cm at 37°C (98.6°F) which is slightly above the specification [5] limits of 3 to 13  $\mu$ /cm for silver amalgam usually tested at 20-25°C (68-77°F). However, expansions of 16 microns per centimeter might not be excessive for dental application.

Alloys made by the combination of the gallium-tin eutectic with copper; with alloys of 68% copper-32% tin; and with 62% copper-38% tin, mixed and packed easily. The amount of

\* One micron per centimeter equals  $1 \times 10^{-4}$  inches per inch.





expansion seems to be dependent on the temperature at which the alloy hardens although the difference is small. The rate at which the expansion occurs is more rapid at 37°C (98.6°F) than at 21.1°C (70°F) (Fig. 1). This increase in the rate and amount of expansion at the higher temperature may indicate a change in solution rate of the powder particles in the liquid component.

The alloy containing 41% copper-59% tin, when combined with the gallium-tin eutectic, was the only alloy of this system that was found to contract. The working properties were poor, however, and the observed contraction may be due to the near completion of the hardening reaction prior to the fiducial reading. This alloy was difficult to pack as it was very dry at the completion of trituration.

### 3.2 Compressive Strength

The compressive strengths of alloys of the gallium-copper-tin system were determined on specimens 4 mm in diameter and 8 mm long. Specimens of this size required a one-gram mix which was trituated 70 revolutions in 30 seconds. The ends of the specimens were faced in a lathe and polished on 3/0 polishing paper. For the specimens aged one hour, the polishing paper only was used. Specimens were stored at 37°C (98.6°F) and tested at 21.1°C (70°F) at a rate of load application of 80 to 110 pounds per minute. The results of the compressive strength determinations for specimens stored for one hour, one day, and one week are summarized in Table 1. Each value represents an average of a minimum of five specimens. The seven-day compressive strength formed by the combination of the gallium-tin eutectic with the 41% copper-59% tin alloy was approximately 28,000 psi; that of all the other alloys ranged from 43,000 to 55,200 psi. Because of the low strength and poor working qualities of the high tin alloy no other mechanical properties of alloys of this composition were determined. The highest strength (55,200 psi) was obtained using an alloy of 62% copper-38% tin combined with 30% gallium-tin eutectic. The standard deviation of 1,800 psi was low, indicating good uniformity of specimens. The lower strength of 43,000 psi was obtained when the gallium-tin eutectic (38%) was combined with 62% bronze powder (90% copper-10% tin). The standard deviation of 6,000 psi for this alloy was also the greatest of all determinations indicating lower uniformity of specimens.

The one-hour strength and one-day strength give a measure of the rate at which the material hardens. A high one-hour strength is desirable for dental application since the patient is less apt to damage a restoration that gains strength





rapidly. The one-hour strength varied from a low of 2,200 psi when gallium-tin eutectic (40%) was combined with 60% bronze powder (90% copper-10% tin) to a high of 9,300 psi when gallium-tin eutectic (30%) was combined with 70% powdered alloy containing 62% copper-38% tin.

From the standpoint of compressive strength, any of the gallium-copper-tin alloys investigated are satisfactory after seven days. The compressive strengths at one hour of the alloys formed by the combination of the gallium-tin eutectic (30-33%) with powdered metal of 62% copper-38% tin were equal to or better than the one-hour strength of dental amalgam. The one-hour strength of the combination of 64% copper powder and gallium-tin eutectic (36%) also had a one-hour strength that approached that of dental amalgam. The one-day strength, when compared with the seven-day strength, is also a measure of the rate of hardening. The alloy formed by the combination of the gallium-tin eutectic with a powder containing 62% copper-38% tin has a one-day strength that approaches its seven-day strength. This would indicate that the setting reaction of this alloy is nearly completed at the end of the first 24 hours. From the differences between the one-day and seven-day strengths of the other alloys investigated, it would follow that hardening of these alloys occurs over a period of time greater than 24 hours.

### 3.3 Hardness

The baby Brinell hardness numbers, determined on the dimensional change specimens which had a minimum age of seven days, are also summarized in Table 1. The baby Brinell hardness as reported by Taylor [6] for 43 different dental amalgams averaged 54, and varied from 28 to 76. The hardness of the gallium-copper-tin alloys is, in all cases, greater than that of silver amalgams. For some compositions they are very much harder than amalgam, and attain values as high as 121, which is within the range of the specified limits [7] of 90-140 for a type III (hard) inlay casting-gold alloy.

### 3.4 Modulus of Elasticity in Compression

The modulus of elasticity is an index to the resistance of a material to deformation (within the elastic range) when a stress is applied, and is thus a measure of the stiffness. For this determination, cylinders 4 mm in diameter and 12 mm long were used. Trituration was 105 revolutions in 45 seconds for the gallium-copper-tin alloys using a mix weighing  $1\frac{1}{2}$  grams. The manufacturer's directions were followed when dental amalgam





was mixed. Tuckerman optical strain gages [8] were employed to measure the amount of deformation under loading. The gage length was set at approximately  $1/4$  inch and the gages were adapted to the specimen as shown in Figure 2.

When such a short gage length is used it must be accurately known, otherwise a large error will be introduced when converting the deformation to unit strain. To determine the distance between the knife edges of each gage, impressions of the knife edges were made on carbon paper and while the gage was still in contact with the carbon paper, a reading was taken through the auto-collimator. The length between the impressions was measured to  $\pm 0.001$  inch using a measuring microscope. Knowing the measured gage length for one reading of the auto-collimator it is possible to calculate the gage length for any reading. Results, attained by repeated observations, indicated the effective gage length was known to about 0.4%.

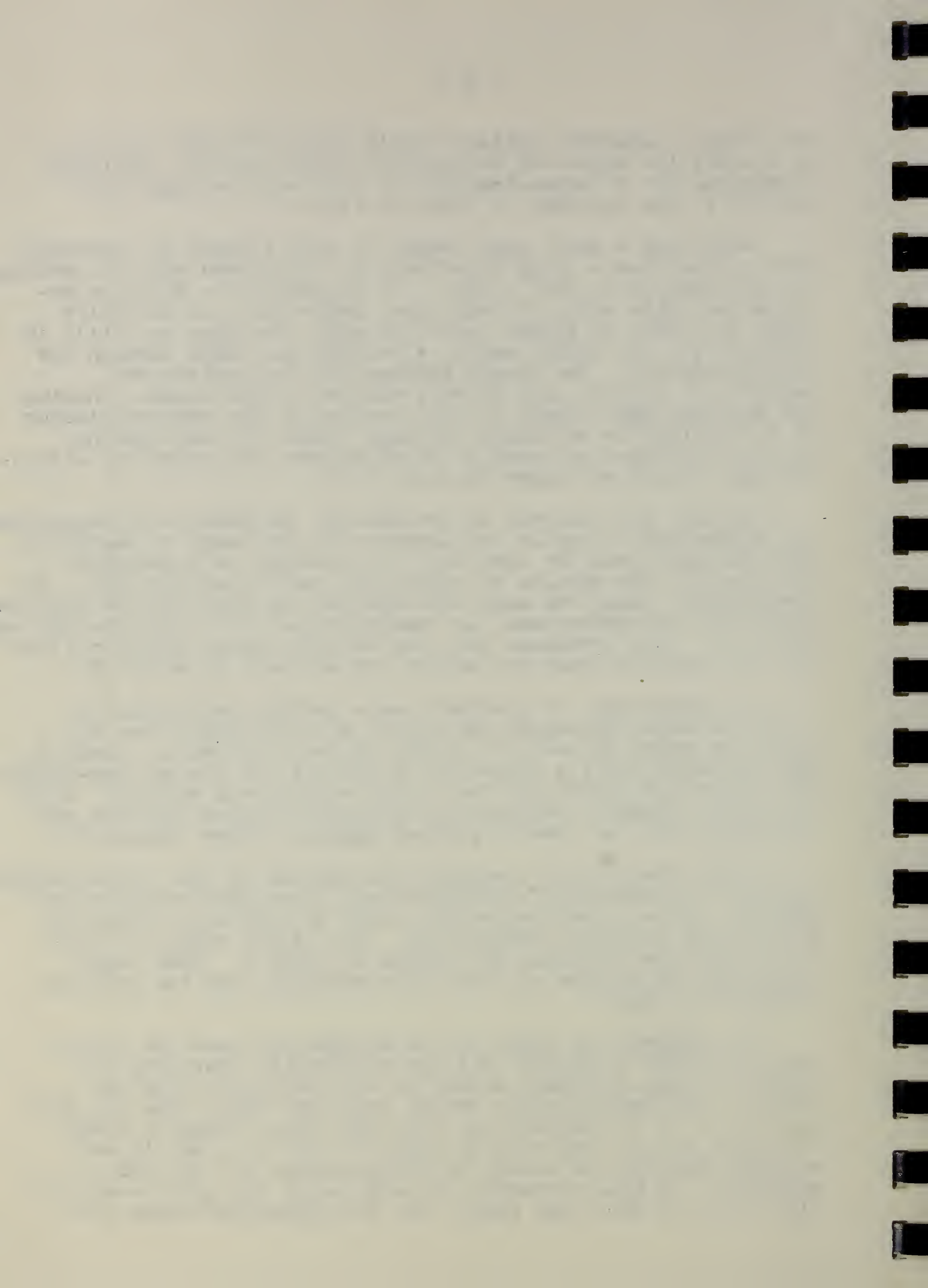
To test the accuracy of the method, the modulus of magnesium was determined. Tensile and compressive specimens of magnesium were machined from the same bar. In tension, on a one-inch gage length, the modulus of elasticity was 6.5 million psi. In compression, using the same size specimen as used for the gallium-copper-tin determinations, an average value of 6.3 million psi was obtained. This agreement with the accepted value of 6.5 million psi [9] indicated that the method was accurate to about 3%.

The seven-day old specimens were loaded continuously at a rate of about 80 pounds per minute with readings taken on alternate gages at every 20 pounds of load increment. Loading was continued until a stress of 30,000 psi for the gallium-copper-tin alloys and 25,000 psi for dental amalgam was applied. From the auto-collimator readings the strain in inches per inch was calculated and the results plotted against stress (Figure 3).

Since there is no straight line portion of the stress-strain diagram, the modulus of elasticity was calculated as the secant modulus. This is defined as the ratio of stress to strain at a specified stress or strain [10]. The secant moduli of elasticity at three stresses are listed in Table 1. Each value represents an average of three determinations for the gallium-copper-tin alloys.

For amalgam the value of 3.2 million psi over the stress range of 1,000 to 25,000 psi was obtained by averaging the data from nine specimens packed by two operators using two alloys. This method gives values that are higher than the average of 1.4 to 1.8 million psi over the stress range of 5,000 to 25,000 psi, as reported by Taylor et al [11], and is more accurate because the strain of the specimens was determined directly. In some instances the modulus of the gallium-copper-tin alloys is more than double the corresponding stress level





for amalgam. The highest values for the modulus were obtained from the alloy formed by the combination of the gallium-tin eutectic with powdered alloys of 68% copper-32% tin, and 62% copper-38% tin. Reducing the amount of the gallium-tin eutectic from 33 to 30% when combined with 62% copper-38% tin caused a reduction in the modulus from 8.2 million psi to 5.8 million psi between 1,000 and 20,000 psi. This may indicate that the modulus is dependent on the gallium-tin eutectic content and the effect of this variation on cementing action should be investigated further.

#### 4. CONCLUSIONS

The liquid eutectic of 89% gallium-11% tin will alloy with copper powder and powdered alloys of copper and tin. These gallium-copper-tin alloys change in dimensions very little during hardening. Hardness and strength are equal to and, in some instances, greater than the hardness and strength of the conventional silver amalgam used for dental fillings.

Specifically, there are two gallium-copper-tin alloys that exhibit good mechanical and physical properties from a dental restoration viewpoint. Pure copper powder (64%) when triturerated with the gallium-tin eutectic (36%) had a setting expansion of 12 to 15  $\mu$ /cm, a compressive strength of 4,600 psi after one hour and 46,500 psi after seven days, a baby Brinell hardness of 67 and a secant modulus of elasticity of 6.2 million psi when calculated between 1,000 and 20,000 psi. Dental amalgam had a modulus of 3.5 million psi when calculated over the same stress range.

The second alloy exhibiting good physical properties was made by combining 30 to 35% of the gallium-tin eutectic with 65 to 70% of an alloy containing 62% copper and 38% tin. These alloys had a setting expansion of 10 to 16  $\mu$ /cm, compressive strengths of 5,400 to 9,300 psi at the end of one hour and 48,400 to 55,200 psi after seven days, a baby Brinell hardness of 105-116, and a secant modulus of elasticity as much as 8.2 million over the stress range of 1,000 to 20,000 psi. For dental restorations these physical and mechanical properties are superior to the corresponding properties of dental amalgam.

The results of this investigation indicate that gallium-copper-tin alloys of the type studied show promise for use as an oral restorative material. Further studies involving corrosion resistance, biological assay, and clinical evaluation must be completed before these alloys can be recommended for general clinical use.





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Table 1

PHYSICAL PROPERTIES OF GALLIUM-COPPER-TIN ALLOYS  
AND DENTAL AMALGAM

Composition (Ga-Cu-Sn Alloys)		Linear Dimension- <sup>(1)</sup> al Change During Hardening (7 days)		Baby Brinell <sup>(4)</sup> Hardness at, 7 or More Days	
Liquid Eutectic (89% Ga- 11% Sn)	Powder	21.1°C (70°F)	37°C (98.6°F)	No.	$\sigma$
%	%	$\mu$ /cm	$\mu$ /cm		
36	64 (100%Cu)	+15 <sup>(6)</sup>	+12	67	5
37	63 (100%Cu)	+14 <sup>(6)</sup>	+ 9 <sup>(6)</sup>	66	8
38	62 (100%Cu)	--	--	64	3
38	62 (90%Cu+10%Sn)	+19 <sup>(6)</sup>	+26 <sup>(6)</sup>	63	5
40	60 (90%Cu+10%Sn)	--	--	--	--
36	64 (77%Cu+23%Sn)	+32	+46	--	--
36	64 (73%Cu+27%Sn)	+29	+44	--	--
33	67 (68%Cu+32%Sn)	+ 3	+ 8	121	24
30	70 (62%Cu+38%Sn)	+ 9	+15	114	15
33	67 (62%Cu+38%Sn)	+10	+16	116	15
35	65 (62%Cu+38%Sn)	--	--	105	29
30-32	68-70 (41%Cu+59%Sn)	- 8 <sup>(6)</sup>	0 <sup>(6)</sup>	--	--
(Dental Amalgam)		<sup>(10)</sup>			
Liquid (Hg)	Powder	20°C (68°F) to 25°C (77°)	37°C (98.6°F)	No.	$\sigma$
%	%	$\mu$ /cm	$\mu$ /cm		
45-55	45-55 <sup>(9)</sup> { Ag-65% min. Cu- 6% max. Zn- 2% max. Sn-25% min. }	+3 to +13 <sup>(11)</sup>	-2 <sup>(12)</sup>	54 <sup>(9)</sup>	--

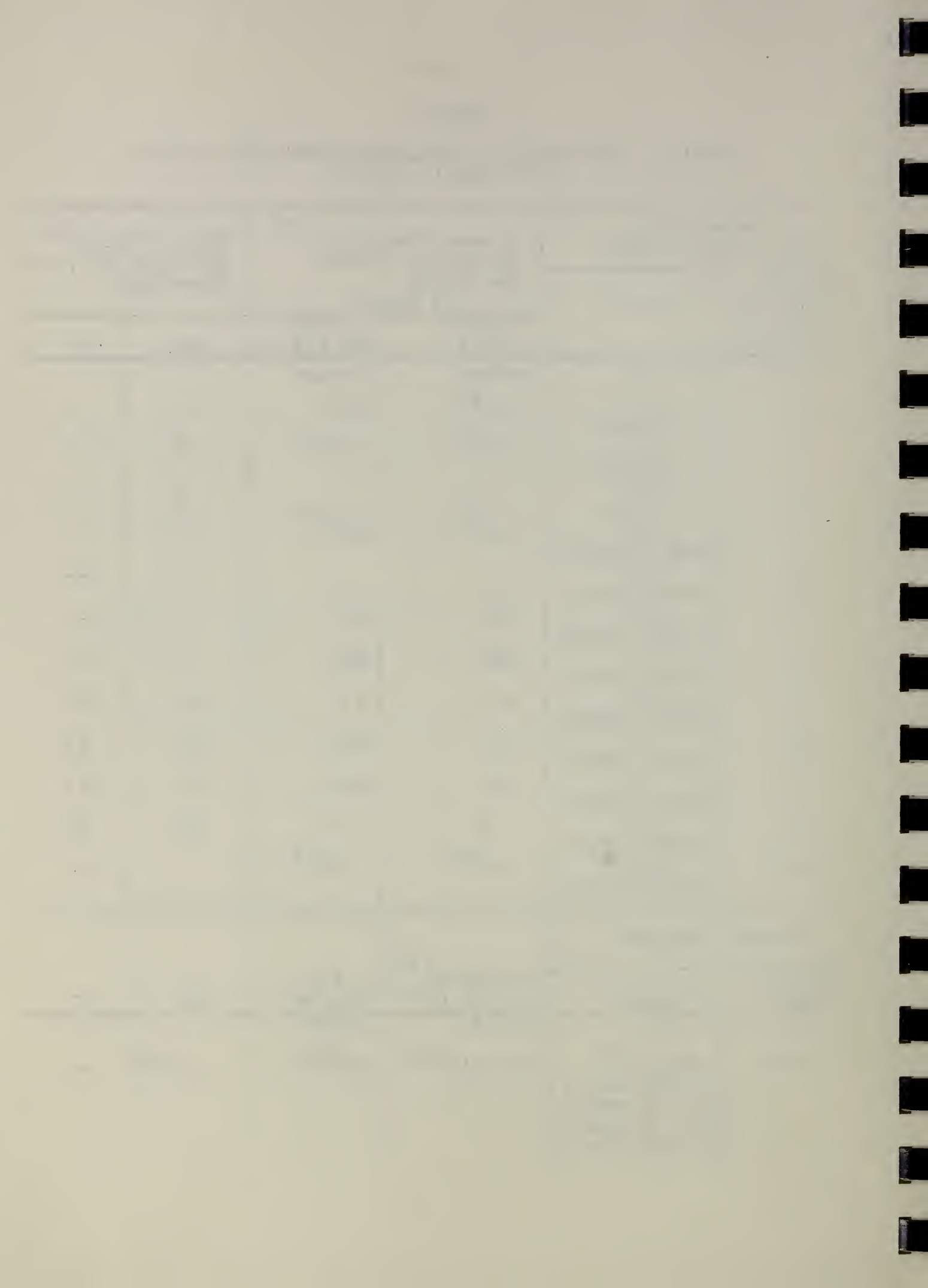


Table 1 (Cont'd)

Composition (Ga-Cu-Sn Alloys)		Compressive Strength <sup>(2)(3)</sup> at					
Liquid Eutectic (89% Ga- 11% Sn)	Powder	1 hour	$\sigma$ (7)	1 day	$\sigma$	7 days	$\sigma$
%	%	psi		psi		psi	
36	64 (100%Cu)	4,600	1,400	27,500	3,400	46,500	5,800
37	63 (100%Cu)	2,900	800	32,600	11,100	48,700	3,400
38	62 (100%Cu)	3,500	600	30,200	6,400	44,700	5,200
38	62 (90%Cu+10%Sn)	2,600	800	41,000	3,200	43,000	6,000
40	60 (90%Cu+10%Sn)	2,200	400	26,900	12,900	--	--
36	64 (77%Cu+23%Sn)	--	--	--	--	--	--
36	64 (73%Cu+27%Sn)	--	--	--	--	--	--
33	67 (68%Cu+32%Sn)	2,800	400	32,400	12,100	50,000	2,200
30	70 (62%Cu+38%Sn)	9,300	1,000	53,000	2,700	55,200	1,800
33	67 (62%Cu+38%Sn)	6,600	1,200	49,300	2,400	54,400	400
35	65 (62%Cu+38%Sn)	5,400	900	43,400	5,000	48,400	2,200
30-32	68-70 (41%Cu+59%Sn)	--	--	--	--	28,100	3,200
(Dental Amalgam)		Compressive Strength <sup>(8)</sup> at					
Liquid (Hg)	Powder	1 hour	1 day		7 days		
%	% <sup>(9)</sup>	psi	psi		psi		
45-55	45-55 (Ag-65% min.) (Cu- 6% max.) (Zn- 2% max.) (Sn-25% min.)	8,000	36,000		41,500		





Table 1 (Cont'd)

Composition (Ga-Cu-Sn Alloys)		Secant Modulus of Elasticity in Compression <sup>(2)(5)</sup> from Stress of 1,000 psi to					
Liquid Eutectic (89% Ga- 11% Sn)	Powder	10,000	$\sigma$	20,000	$\sigma$	25,000	$\sigma$
%	%	psi		psi		psi	
36	64 (100%Cu)	$7.9 \times 10^6$	$0.7 \times 10^6$	$6.2 \times 10^6$	$0.4 \times 10^6$	$5.3 \times 10^6$	$0.4 \times 10^6$
37	63 (100%Cu)	--	--	--	--	--	--
38	62 (100%Cu)	--	--	--	--	--	--
38	62 (90%Cu+10%Sn)	--	--	--	--	--	--
40	60 (90%Cu+10%Sn)	$6.8 \times 10^6$	$0.7 \times 10^6$	$4.8 \times 10^6$	$0.4 \times 10^6$	$4.0 \times 10^6$	$0.6 \times 10^6$
36	64 (77%Cu+23%Sn)	--	--	--	--	--	--
36	64 (73%Cu+27%Sn)	--	--	--	--	--	--
33	67 (68%Cu+32%Sn)	$10.4 \times 10^6$	$0.4 \times 10^6$	$8.5 \times 10^6$	$0.1 \times 10^6$	$7.8 \times 10^6$	$0.1 \times 10^6$
30	70 (62%Cu+38%Sn)	$7.5 \times 10^6$	$0.7 \times 10^6$	$5.8 \times 10^6$	$0.7 \times 10^6$	$5.0 \times 10^6$	$0.7 \times 10^6$
33	67 (62%Cu+38%Sn)	$10.2 \times 10^6$	$0.3 \times 10^6$	$8.2 \times 10^6$	$0.3 \times 10^6$	$7.2 \times 10^6$	$0.3 \times 10^6$
35	65 (62%Cu+38%Sn)	--	--	--	--	--	--
30-32	68-70 (41%Cu+59%Sn)	--	--	--	--	--	--
(Dental Amalgam)		Secant Modulus of Elasticity in Compression <sup>(10)</sup> from stress of 1,000 psi to					
Liquid (Hg)	Powder	10,000	$\sigma$	20,000	$\sigma$	25,000	$\sigma$
%	%	psi		psi		psi	
45-55	45-55 <sup>(9)</sup> (Ag-65% min.) (Cu- 6% max.) (Zn- 2% max.) (Sn-25% min.)	$4.4 \times 10^6$	$0.6 \times 10^6$	$3.5 \times 10^6$	$0.3 \times 10^6$	$3.2 \times 10^6$	$0.3 \times 10^6$



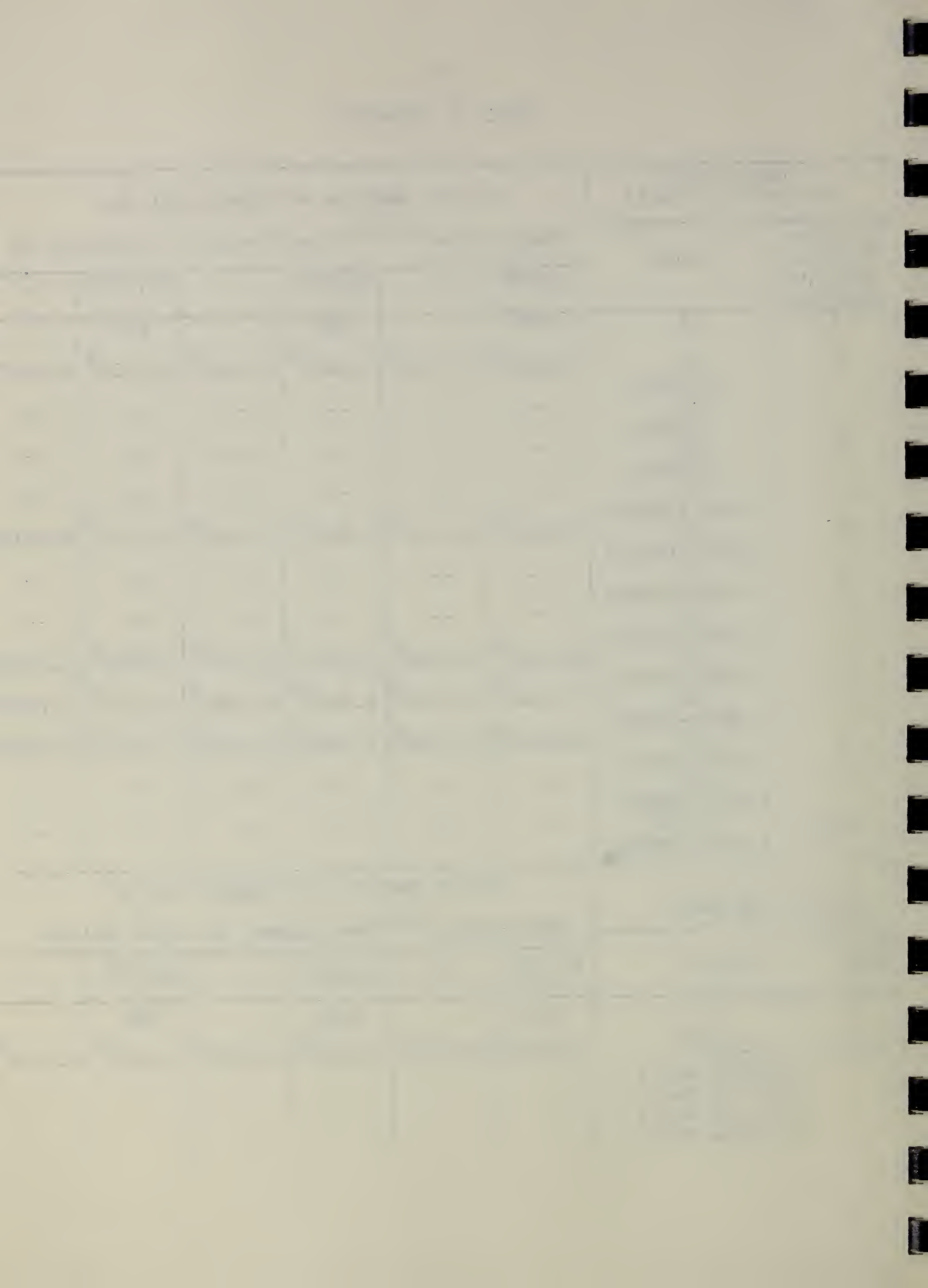


Table 1 (Cont'd)

FOOTNOTES

- (1) Specimens made at 21.1°C (70°F) and tested at indicated temperature.
- (2) Specimens made at 21.1°C (70°F), stored at 37°C (98.6°F), and tested at 21.1°C (70°F).
- (3) Each result represents the average of five determinations.
- (4) Each result represents the average of eight determinations.
- (5) Each result represents an average of three determinations. All specimens aged seven days.
- (6) Single values, others in these two columns are averages of duplicates.
- (7)  $\sigma$  indicates standard deviation calculated by the formula

$$\sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}}$$

- (8) Average hand packed values from "Dental amalgam: the effect of mechanical condensation on some physical properties." Ryge, G.; Dickson, G., Smith, D. L., and Schoonover, I.C. J.A.D.A. 45:269 Sept. 1952.
- (9) Computed from results in "A specification for dental amalgam alloys: A report to the Scientific Foundation and Research Commission of the American Dental Association." Taylor, N. O. J.A.D.A. 27:112 Jan. 1930.
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1. The first part of the paper discusses the importance of the study of the history of the United States. It is argued that a knowledge of the past is essential for a full understanding of the present and for the development of a sound policy for the future.

2. The second part of the paper discusses the role of the government in the development of the United States. It is argued that the government has played a crucial role in the development of the country, and that its actions have been guided by a set of principles that have been passed down from generation to generation.

3. The third part of the paper discusses the role of the individual in the development of the United States. It is argued that the individual has played a crucial role in the development of the country, and that his actions have been guided by a set of principles that have been passed down from generation to generation.

4. The fourth part of the paper discusses the role of the community in the development of the United States. It is argued that the community has played a crucial role in the development of the country, and that its actions have been guided by a set of principles that have been passed down from generation to generation.

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6. The sixth part of the paper discusses the role of the world in the development of the United States. It is argued that the world has played a crucial role in the development of the country, and that its actions have been guided by a set of principles that have been passed down from generation to generation.

7. The seventh part of the paper discusses the role of the future in the development of the United States. It is argued that the future has played a crucial role in the development of the country, and that its actions have been guided by a set of principles that have been passed down from generation to generation.

8. The eighth part of the paper discusses the role of the past in the development of the United States. It is argued that the past has played a crucial role in the development of the country, and that its actions have been guided by a set of principles that have been passed down from generation to generation.

9. The ninth part of the paper discusses the role of the present in the development of the United States. It is argued that the present has played a crucial role in the development of the country, and that its actions have been guided by a set of principles that have been passed down from generation to generation.

10. The tenth part of the paper discusses the role of the future in the development of the United States. It is argued that the future has played a crucial role in the development of the country, and that its actions have been guided by a set of principles that have been passed down from generation to generation.

# DIMENSIONAL CHANGE DURING HARDENING

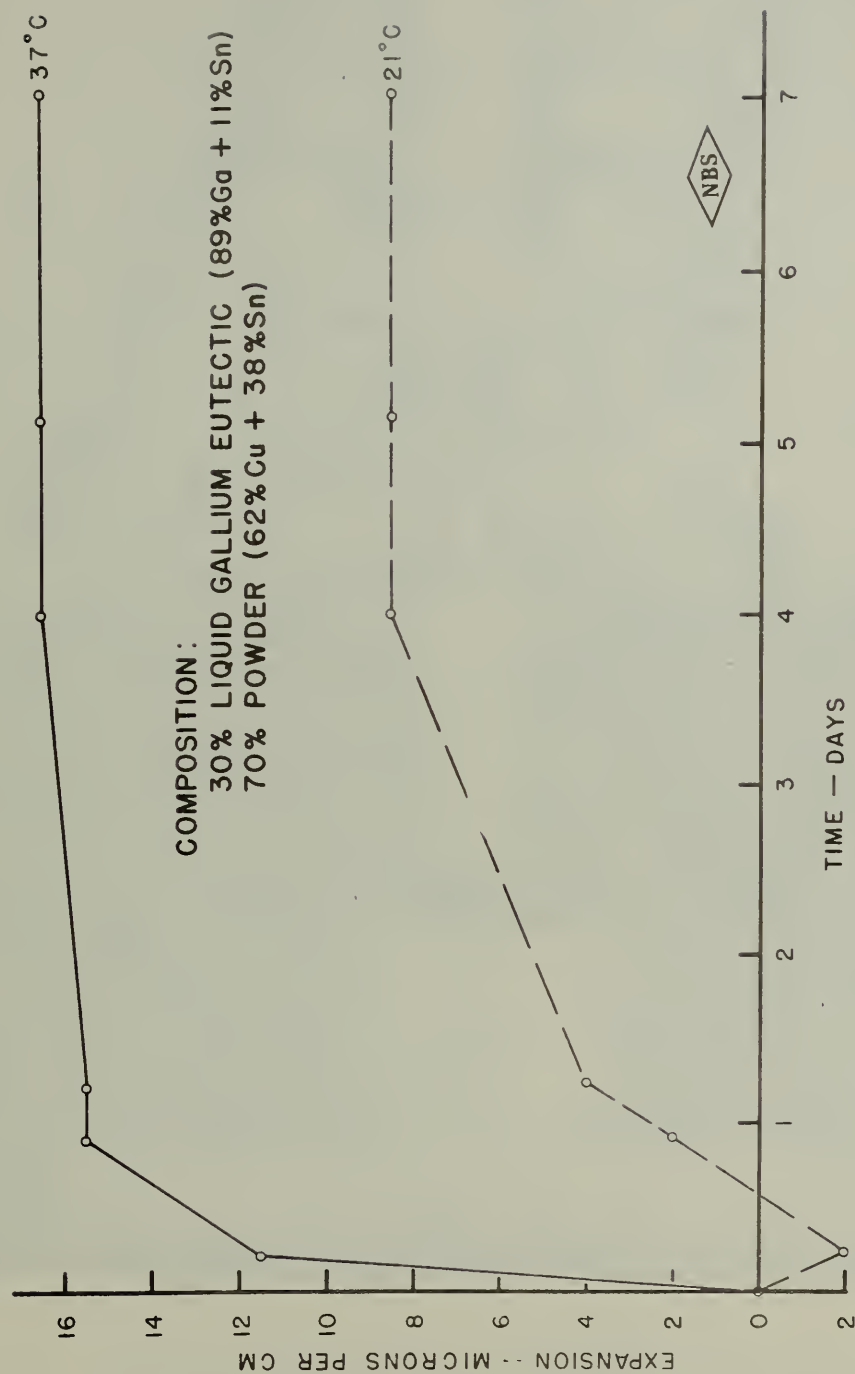


Figure 1. The effect of temperature on the rate of expansion of a gallium-copper-tin alloy.





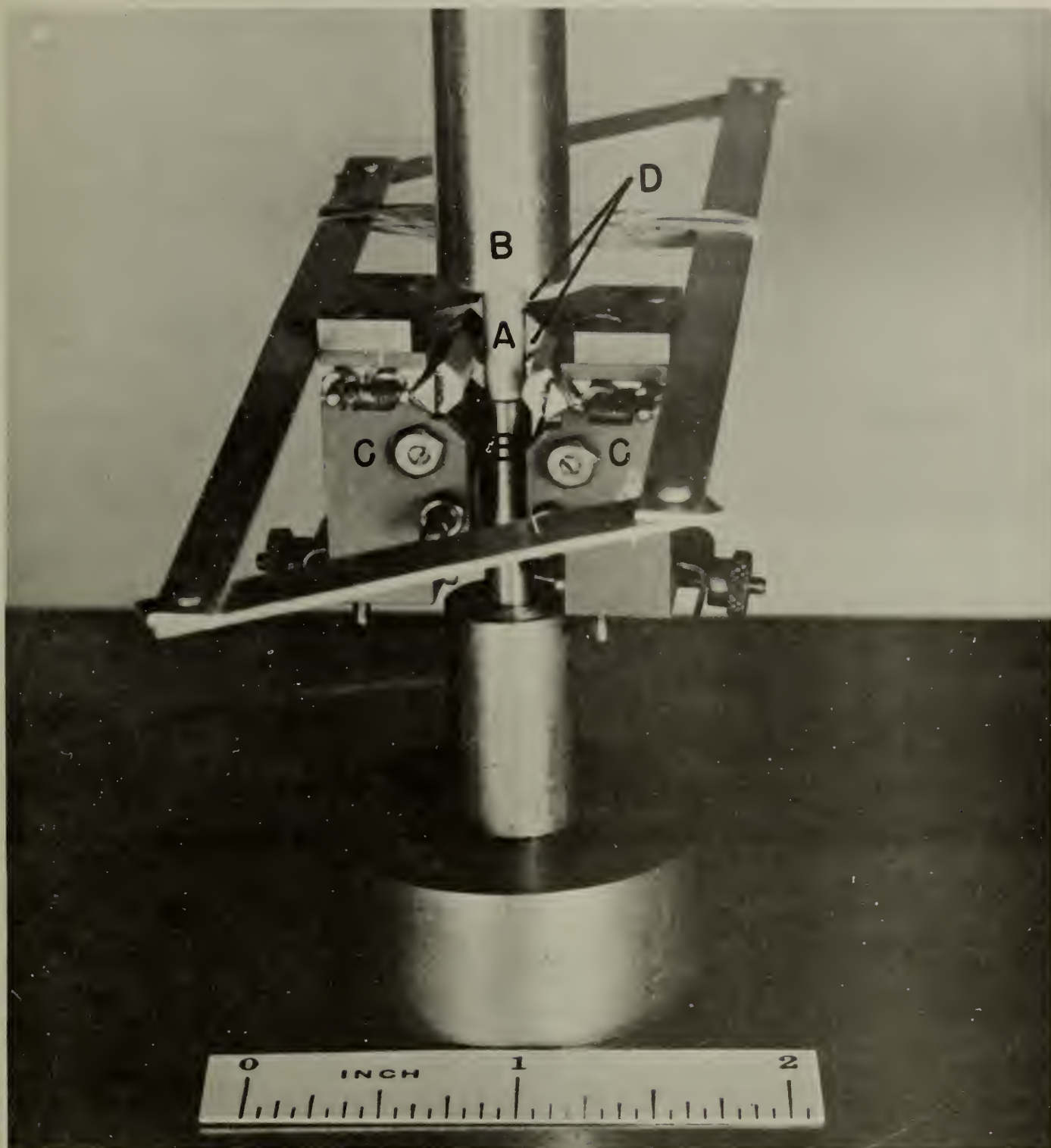


Figure 2. The arrangement of specimen and Tuckerman optical strain gages for determining stress-strain data in compression from which the moduli of elasticity were calculated.



- A = Specimen
- B = Platens
- C = Tuckerman optical strain gages
- D = Knife edges



# STRESS-STRAIN DIAGRAM

GA-CU-SN ALLOYS & AMALGAM

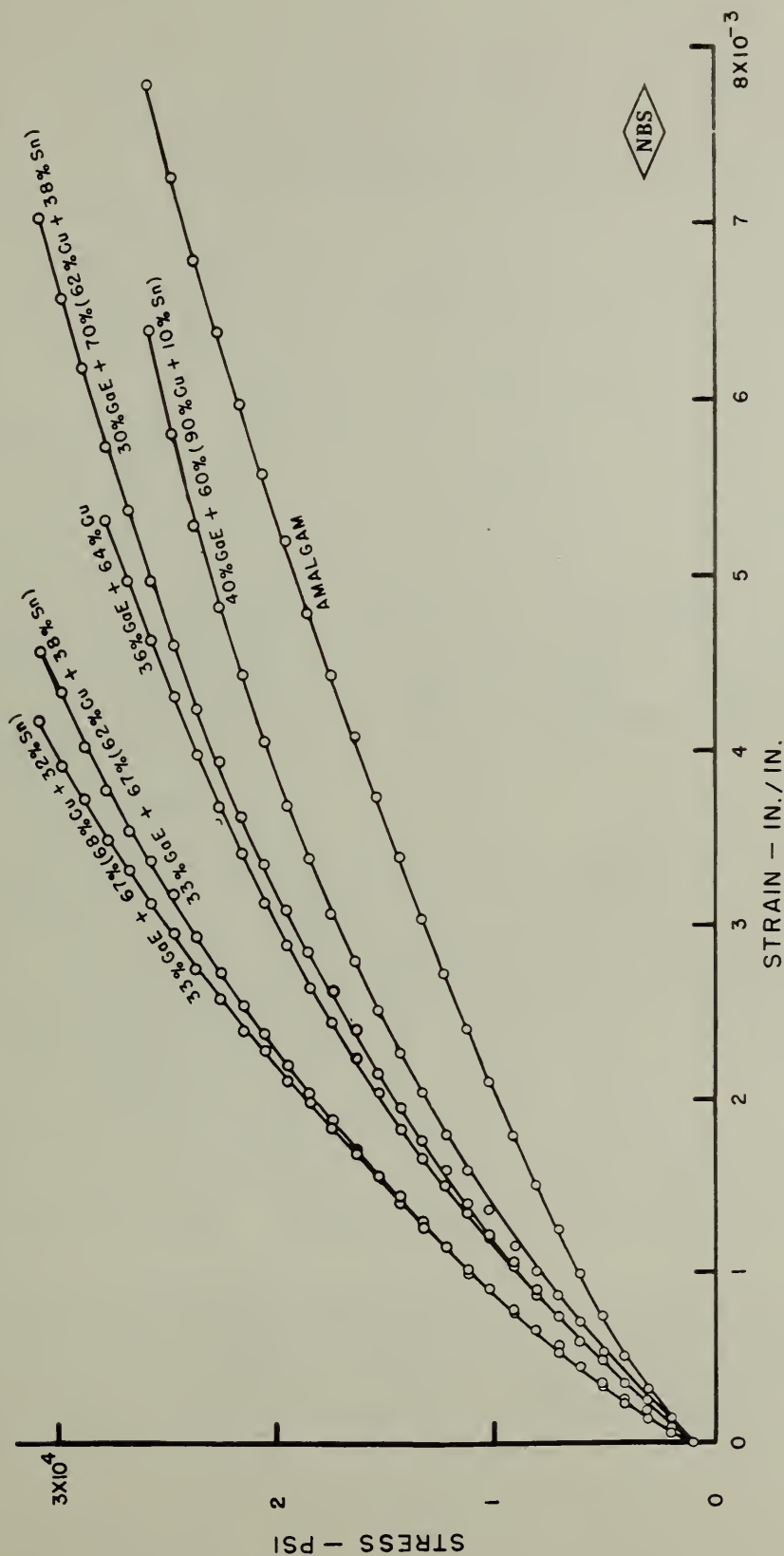


Figure 3. Average stress-strain data for seven-day-old specimens of gallium-copper-tin alloys and dental amalgam. All specimens were made at  $21.1^{\circ}\text{C}$  ( $70^{\circ}\text{F}$ ), stored at  $37^{\circ}\text{C}$  ( $98.6^{\circ}\text{F}$ ), and tested at  $21.1^{\circ}\text{C}$  ( $70^{\circ}\text{F}$ ).

GaE = Gallium-tin eutectic (89% gallium + 11% tin).



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