

# NATIONAL BUREAU OF STANDARDS REPORT

7100

REPORT ON DENTAL RESEARCH  
AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

July 1 to December 31, 1960

Dental Research Laboratory



U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS

## THE NATIONAL BUREAU OF STANDARDS

### Functions and Activities

The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. These include the development and maintenance of the national standards of measurement and the provision of means and methods for making measurements consistent with these standards; the determination of physical constants and properties of materials; the development of methods and instruments for testing materials, devices, and structures; advisory services to government agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; and the development of standard practices, codes, and specifications. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services, and various consultation and information services. Research projects are also performed for other government agencies when the work relates to and supplements the basic program of the Bureau or when the Bureau's unique competence is required. The scope of activities is suggested by the listing of divisions and sections on the inside of the back cover.

### Publications

The results of the Bureau's work take the form of either actual equipment and devices or published papers. These papers appear either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau itself publishes three periodicals available from the Government Printing Office: The Journal of Research, published in four separate sections, presents complete scientific and technical papers; the Technical News Bulletin presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: Monographs, Applied Mathematics Series, Handbooks, Miscellaneous Publications, and Technical Notes.

Information on the Bureau's publications can be found in NBS Circular 460, Publications of the National Bureau of Standards (\$1.25) and its Supplement (\$1.50), available from the Superintendent of Documents, Government Printing Office, Washington 25, D.C.

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NBS PROJECT

NBS REPORT

0708-11-07260  
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December 31, 1960

7100

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Progress Report

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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 Dental Sciences Division of the School of Aviation Medicine, USAF; the Navy Dental Corps; and the Veterans Administration.

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U. S. DEPARTMENT OF COMMERCE  
NATIONAL BUREAU OF STANDARDS



REPORT ON DENTAL RESEARCH  
AT THE NATIONAL BUREAU OF STANDARDS

1. INTRODUCTION

Research on various types of dental materials and equipment and on natural tooth structures continued at the National Bureau of Standards during the half year ending December 31, 1960.

Summaries of results obtained on work in progress, a list of reports issued and a list of papers published are given below. Copies of the reports are appended.

2. REPORTS ISSUED

NBS Report 7036 Changes in Dentures During Storage in Water and In Service.  
NBS Report 7067 Alloys of Gallium Having Possible Dental Use.  
NBS Report 7083 Tooth Fluorometer.  
NBS Report 7096 An Investigation of the Constitution of the Mercury-Tin System and Portions of the Mercury-Silver-Tin System.

3. PAPERS PUBLISHED

Comparative Fixation of Calcium and Strontium by Synthetic Hydroxyapatite. R. C. Likens, H. G. McCann, A. S. Posner and D. B. Scott. J. Biolog. Chem. 235:2152, July 1960.

Procedure for the Determination of the Noble Metal Content of Dental Gold Alloys. H. J. Caul, S. J. Clabaugh and M. Susa. J.A.D.A. 61:439, Oct. 1960.

Hydrogen Bonding in Calcium Deficient Hydroxyapatite. A. S. Posner, J. M. Stutman and E. R. Lippincott. Nature 188:486, Nov. 1960.

Crystalline Basic Calcium Orthophosphate (Hydroxyapatite). A. Perloff and A. S. Posner. Chapter IIA-6 in Inorganic Synthesis. Vol. VI. McGraw-Hill, New York 1960.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Studies.

Work on the isolation and identification of the fluorescent components of tooth structure has been interrupted by

the departure of the biochemist. In the meantime, efforts have been concentrated on the perfection of a precise ultra-microtechnique for the quantitative determination of the nitrogen content of very small samples of enamel and dentin in order to test the validity of the assumption that the fluorescence intensity of small areas of tooth sections is related to the nitrogenous organic content.

The simplified fluorometer developed for the measurement of the fluorescence of teeth has been delivered to the staff of the National Institute of Dental Research for determination of the intensity of the fluorescence of teeth in the mouths of individuals of known history and of different oral health. The correlation of fluorescence intensity with DMF index, vitality of the teeth, use of fluoride-containing materials, use of antibiotics and vitamins, and dietary differences will be studied.

(b) Crystallographic Studies.

Work continued on the x-ray diffraction studies of crystal size and strain in synthetic hydroxyapatites and bone and tooth mineral. The crystal size, as related to the rate of crystal growth, was shown to affect the relative incorporation of  $\text{Sr}^{90}$  to  $\text{Ca}^{45}$ . The quickly deposited crystals of hydroxyapatite took up the radioisotopes in the same ratio they were introduced into the precipitating solutions. On the other hand, slowly-grown, larger crystals of synthetic hydroxyapatite contained a lower  $\text{Sr}^{90}/\text{Ca}^{45}$  ratio, thus showing a discrimination against Sr.

The study of the hydrogen bonding in calcium deficient hydroxyapatites and in mineral tissue apatites continued. Infra-red absorption spectrophotometry was utilized to analyze for the hydrogens present as hydrogen bonds between two oxygens of adjacent orthophosphate groups.

X-ray diffraction studies on acellular fish bones were carried out in cooperation with Columbia University College of Physicians and Surgeons. It was shown that acellular bones tend to have high protein (collagen) content as compared to normal bone.

The low angle x-ray diffraction study of the crystallization of polyethylene was finished during this period. A distinct relationship was demonstrated between the long spacing in the polymer (as shown by x-ray diffraction) and the

crystallization temperature.

(c) Dental Calculus Studies.

Samples of subgingival and supergingival in addition to composite samples of dental calculi were decalcified by two different methods. Acid hydrolysates of the residues after decalcification have shown the same amino acid present in the residues of both subgingival and supergingival calculi. Determinations have been carried out by paper chromatography with subsequent determinations of optical densities of eluded ninhydrin reaction products of the various amino acids. Paper chromatography of resin hydrolysates of the residues has also shown the presence of galactose, glucose, mannose and rhamnose present in the carbohydrate fraction. Absorption curves indicate the presence of a dioxypentose also in the carbohydrate.

#### 4.2 METALS

(a) Amalgam.

Constitution of the Silver-Tin-Mercury System.

A detailed report of the work on the silver-tin-mercury system is appended.

Particle Size.

The affect of particle size and shape of dental alloy on the physical properties of dental amalgam has been studied. This was accomplished by using an experimental dental alloy atomized into powdered spherical form and separated into fractions of eight size ranges by sieving and hydraulic elutriation. Physical properties were determined on dental amalgam using individual size ranges, combinations of size ranges and three commercial alloys as controls. Properties investigated by standard methods were compressive strength, dimensional change on setting, flow and residual mercury content. Compressive strength was determined at time intervals of 1 hour, 2 hours, 6 hours, 24 hours, 1 week and 1 month. Flow was determined at 24 hours and at 1 week.

Particle sizes of 15 to 50 microns in diameter gave the most desirable physical properties and compared most favorably to those obtained for the controls. Dimensional change on setting increased as the particle size was increased, with the smallest size range showing 10 microns/cm contraction

and the largest size range showing in excess of 200 microns/cm expansion. The amount of dimensional change within a given size range appeared to be dependent upon the residual mercury content. As each extreme of the size range was approached, the crushing strength decreased and the flow increased. The middle sized ranges gave 24 hour crushing strengths of 50,000 psi using a strain rate of 0.01 inch/min. The smaller particles obtained a high percentage of their total compressive strength within 1-2 hours. The residual mercury content increased as the particle size was decreased, with the smallest size range showing 58% and the largest size range showing 40%.

Theoretical mixtures of 4 size ranges, containing 78-89% of the largest size range, gave a residual mercury content of 30%. The other properties were greatly improved over those determined for the largest size range before the smaller particles were added. The compressive strength was increased 15,000 psi. The setting expansion was reduced to approximately +10 microns/cm and the flow was reduced to 1.5%.

#### Rheological Properties.

An investigation of the rheological properties of dental amalgam was initiated. Because amalgam exhibits considerable viscous flow, values obtained for strength and stress-strain relationships depend greatly on the rate of loading. By means of rheological methods it will be possible to obtain specific information on stress-strain time characteristics of amalgam. Preliminary results indicate that some amalgams flow much more than others under prolonged loading. Results also indicate that the instantaneous modulus of elasticity of amalgam is about twice the maximum modulus calculated from previous stress-strain data.

#### (b) Gold Analysis.

Fire Assay. Experiments with known compositions of Au, Pt and Pd with relatively large ratios (4-1/2 to 1) of Ag show that Au can be separated from Pt and Pd by nitric acid parting. With alloys free of Pt and Pd practical ratios of Ag to Au range between 2-1/2 and 5-1/2 to 1. With ratios above 6:1 the parted metal disintegrates into many small pieces rather than remaining in one piece. With H<sub>2</sub>SO<sub>4</sub> partings the parted metal has remained in one piece for all ratios tried, including 9-1/2:1. Parting procedures including washing which result in accurate determinations must be developed. Indications suggest the possibility of colorimetric methods



for determining the Pd content in the  $\text{HNO}_3$ .

X-ray Fluorescence. Four standard alloys of known composition with variation of each constituent (Au, Pt, Pd, Ag, Cu, and Zn) have been made. Specimens for use in the x-ray spectrometer have been prepared from these alloys. No data have been collected as yet.

(c) Cobalt-Chromium Casting Alloy.

An investigation was made of the combined affect of metal temperature, amount of metal cast, and previous use of the metal upon the mechanical properties of cobalt-chromium alloy. The casting temperature was controlled by a two-color, optical pyrometer and specimens were cast at eleven temperatures between  $2350^\circ\text{F}$  and  $2650^\circ\text{F}$ .

Controlled temperature castings showed better properties than those cast by visual estimate. Small melts showed better properties than large melts. Reused metal (all used and 50% used - 50% new) showed better elongation than new metal. Within the temperature range studied there is evidence of optimum casting temperatures for each type of metal. Repeat tests on additional lots of metal indicate that other unidentified variables are as important as casting temperature in determining properties.

(d) Gallium Alloys.

A detailed report of the results obtained on gallium alloys is appended.

#### 4.3 Resins

(a) Denture Base Resins.

A report of results obtained in the investigation of the dimensional changes of denture base resins is appended.

(b) Silica-Resin Direct Filling Material.

Study was continued on a method for investigating surface characteristics of fine powders of dentin, enamel, bone ash, synthetic apatite, anorganic bone and, for comparison and contrast, silica and ferric oxide. Test tubes containing 5 ml. water, 5 ml. butanol, 0.5% powder and 0.5 and 1.0% of a surface active agent were used to study preferential wetting

of the powder by butanol (organophilic) or water (hydrophilic). All the powders were normally hydrophilic. Sixty surface-active agents were tested each with the 7 powders and a control. In the presence of certain of the agents, the powders became organophilic. This effect was dependent upon the pH, but generally in decreasing order of effectiveness: chelate, anionic, cationic, nonionic, surface active agents. The forces causing the active groups (of these surface active agents) to displace the water adsorbed on the surfaces is postulated to parallel the forces of similar protein side-chain groups. These tests may also lead to the synthesis of "surface active (chelate) monomers" providing a mechanism for obtaining stable adhesion of restorative materials to hard tooth tissues.

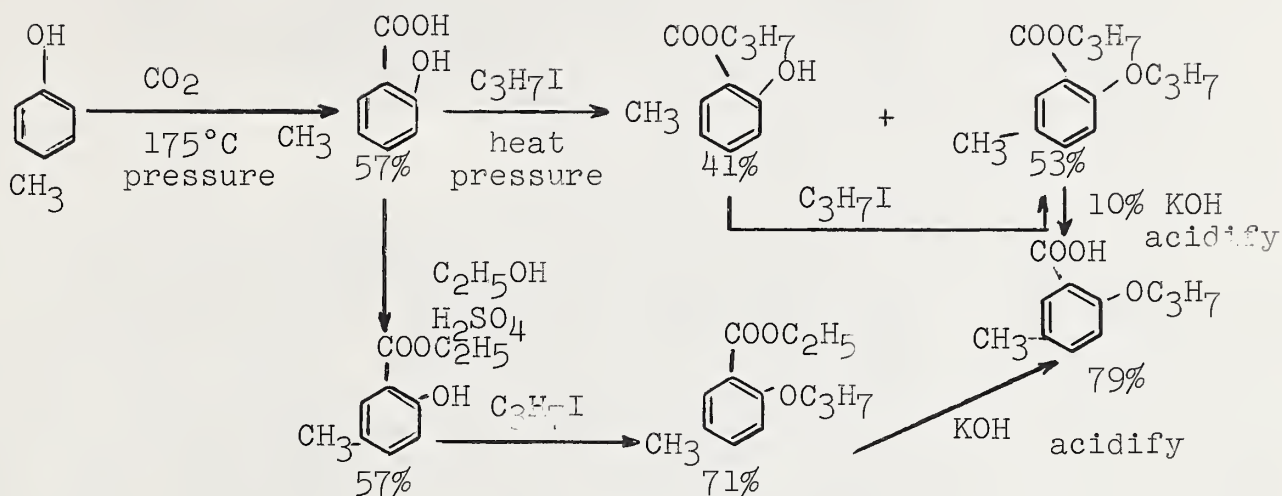
#### (c) Analysis of Pyrolyzates by Gas Chromatography.

Work on this project has been completed. The quantities of nonvolatiles retained in the chromatographic column have been determined. Whereas all pyrolysis products of polymethyl methacrylate are eluted considerable amounts of the products of the degradation of polystyrene and polyethylene below 600°C are retained by the stationary phase.

Pyrolyzates of polymethyl methacrylate depolymerized at temperatures ranging from 400 to 1100°C by the procedure of Madorsky and Straus have been analyzed by gas chromatography. It was found that the composition of the pyrolyzate changes on standing at room temperature. The liquid fraction obtained on degradation in a vacuum below 800°C partially polymerizes on storage forming a nonvolatile, milky-white solid phase. Since the percentage of monomer in the products of the degradation above 800°C is small no polymer formation occurs on storage.

#### 4.4 Synthesis of Chelate Forming Alkoxy Methyl Benzoic Acids

The synthesis of 2-propoxy-5-methylbenzoic acid (PMBA) by two new routes has been accomplished as shown on the next page.



The structure of the new intermediates not previously reported has been ascertained by analysis of their carbon-hydrogen content and their infra-red spectra. The yields, especially of the reactions employed in the second route are very satisfactory. PMBA hardens slowly with zinc oxide and PMBA-eugenol-zinc oxide slurries set within 20 minutes. A mix of standard consistency is obtained with difficulty since the excess zinc oxide is not readily wetted by the liquid. Improvement of the cement by addition of wetting agents may be possible.

#### 4.5 Investment for Chromium-Cobalt Alloys

Properties of investments used for casting chromium-cobalt alloys are being investigated. Preliminary results on dimensional changes of four water-mix investments show setting expansions of near 0.3 to 0.5% and thermal expansions of about 1% between room temperature and 1000°C. These values are considerably below the greater than 2% casting shrinkages generally reported for dental chromium-cobalt alloys.

#### 4.6 Thermal Expansion Apparatus

A digital recording, interferometric, thermal expansion apparatus has been designed and its construction is essentially

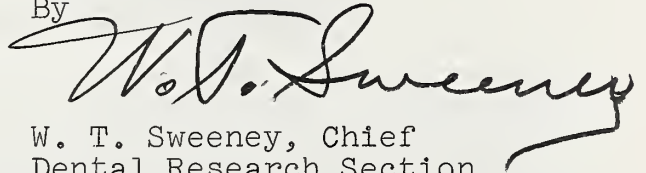
completed. The apparatus controls the heating rate of the specimen and automatically records the change of length, temperature and time of observation. This instrument will be used for determining the coefficient of thermal expansion of small specimens of various types of dental materials.

#### 4.6 Evaluation of Materials

Materials evaluated for the Federal dental services and the American Dental Association by specification or other test methods included amalgam, chromium-cobalt alloy, denture base resin and mercury.

For the Director

By

A handwritten signature in black ink, appearing to read "W. T. Sweeney". The signature is written in a cursive style with a long, sweeping underline that extends to the right.

W. T. Sweeney, Chief  
Dental Research Section