# NATIONAL BUREAU OF STANDARDS REPORT

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REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

January 1 to June 30, 1962

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 dental materials, dental equipment and natural tooth structures continued at the National Bureau of Standards during the half year ending June 30, 1962.

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

#### 2. Reports Issued

- NBS Report 7547 Metallurgical Considerations in the Design of Plastic Gallium Alloys Which Harden at Room Temperature.
- NBS Report 7564 Marginal Defects in Amalgam Fillings as a Result of Thermal Expansion and Flow.
- NBS Report 7574 Some Factors Affecting the Dimensional Changes of Gold-Alloy Investments.
- NBS Report 7708 Effect of Rate of Loading, Time of Trituration and Test Temperature on the Compressive Strength Values of Dental Amalgam.

3. Papers Published

Properties of Silico-Phosphate Cements. J. N. Anderson and G. C. Paffenbarger. Dent. Prog. 2:72 Jan. 1962.

Tensile Strength and Modulus of Elasticity of Tooth Structure and Several Restorative Materials. R. L. Bowen and M. S. Rodriguez. J.A.D.A. 64:373 March 1962.

Research and the Saving of Teeth. G. C. Paffenbarger. J. Pros. Dent. 12:369 March-April 1962.

Revision of American Dental Association Specification No. 7 for Dental Wrought Gold Wire Alloy. J.A.D.A. 64:439 March 1962.

Guide to Dental Materials, 1962-1963. Fifth Edition, American Dental Association. Chicago, Illinois March 1962.

Characteristics of Some Air-Turbine Handpieces. D. F. Taylor, R. R. Perkins and J. W. Kumpula. J.A.D.A. 64:794 June 1962.

4. Work in Progress

4.1 Human Tooth Enamel and Dentin

(a) Heat of Immersion of the Components of Teeth.

To obtain more fundamental knowledge of the chemical groups that adhere to tooth structure, a program has been started to determine initially the heats of immersion of apatites, dentin and enamel in dilute aqueous solutions. From the results obtained with well characterized samples it should be possible to determine what groupings react with or are absorbed or adsorbed by tooth structure as well as the magnitude of the bond. A calorimeter has been built and is being calibrated to measure heats of reaction of the order of 1 cal. within ± 3%.

(b) Dental Calculus: Organic Portion.

Since previous work indicated that salivary mucoid might be involved in the formation of calculus, further characterizations of the protein fraction in addition to attempts to detect any organic acids present in the calculus has been made using electrophoretic technics. While the presence of a glyco or mucoprotein has been confirmed by electrophoretic technics the results have also indicated the possible presence of other proteins.

(c) Fluorescence Studies.

Studies of the fluorescence of calcified tissues were continued with emphasis on the relationship between fluorescence intensity and organic content. The portable fluorometer was redesigned to facilitate operation in a clinic. Also use of a coaxial optical fiber light conductor is planned to carry the ultraviolet radiation to the tooth surface and to return visible fluorescence back to the photoelectric cell.

4.2 Metals

(a) Amalgam

Investigation of the rheological behavior of dental amalgam was continued. During this phase of the study a more extensive knowledge of its phenomenological behavior was obtained. It has been demonstrated that amalgam exhibits at least four rheological phenomena, (1) elastic behavior, (2) transient creep, (3) steady state creep and (4) strain hardening. Successful application of rheological theory to these phenomena has been made in order to show their relation to stress-strain behavior. A more general investigation is being made to establish the usefulness of rheological theory in giving a more complete understanding of the mechanical properties of all types of dental materials and the inter-relationship of all their mechanical properties. A detailed report on the effects of rate of loading, temperature and specimen preparation on the values obtained for compressive strength of amalgam is appended.

#### (b) Gallium

A report on various gallium alloys which may have applications as restorative materials is appended. Properties of palladium-gallium-tin alloys were found to be dependent to a considerable extent upon the variation of particle size in the metal powder and the consequent variation in total surface area available for reaction with the liquid gallium-tin alloy. A study of the variation of these properties as a function of particle size will be conducted using spherical powders.

# (c) Gold

Further investigation of the use of x-ray fluorescence for rapid analysis of dental gold alloys continues to indicate that the method is feasible. It will be necessary to prepare a series of standards covering the composition ranges of dental gold alloys for routine analysis. The method will also be investigated for use in analyzing dental amalgam alloys.

## 4.3 Resins

(a) Analysis of Polymers by Gas Chromatography.

Methods to analyze the pyrolysis products of linear and branched polyethylenes were studied using a variety of columns which have been proposed for the determination of  $C_1$  to  $C_5$  hydrocarbons. Difficulites were encountered since the chromatograms contained overlapping peaks that are not suitable for the quantitative estimation of the components of the pyrolysis mixture. The use of longer columns as well as combinations of two or more columns is now being investigated.

(b) Silica-Reinforced Direct Filling Resin.

The particle-size distribution of the reinforcing powder has been shown to affect the silica-to-resin ratio obtainable in specimens of reinforced resins. Reinforcing powders were prepared giving specimens with as low as 14 percent resin by weight (approximately 25 percent by volume). Other properties of these highly reinforced materials are being determined.

(c) Adhesion Studies.

The addition reaction product of N-phenyl glycine and glycidyl methacrylate (NPG-GMA) was further studied in its capacity to couple together a methacrylate polymer and calcified substrates. Average adhesion values in psi for surfaces of dentin, enamel and fluorapatite surfaces were respectively, (1) for controls with no NPG-GMA: 0, 240 and 910, (2) with NPG-GMA applied: 510, 690 and 1,900; and (3) with NPG-GMA applied after the surfaces were cleaned with a 10% solution of EDTA at pH 7: 1,100, 770 and 1,100. These data show that collagen is not an essential part of the bonding mechanism of NPG-GMA.

# 4.4 Zinc Oxide-Eugenol Materials

(a) Zinc Oxide-Eugenol-EBA Cements.

The effect of various additives, fillers and ion exchange resins on zinc oxide-eugenol-EBA-cements has been studied. Cements showing greatly improved physical properties such as compressive strength and negligible water solubility and disintegration under laboratory conditions have been developed. The most desirable cements consist of 74% zinc oxide, 6% hydrogenated rosin and 20% of dried fused quartz particles. These products have maximum 24 hour compressive strength of 12,400 psi. Studies conducted at the U. S. Army Institute for Dental Research indicate that these materials possess excellent tissue tolerance, much superior to zinc phosphate cements. Controlled clinical studies to evaluate the usefulness of the products as more permanent temporary filling materials have been initiated.

Investigations of the heat stability of these cements were undertaken. Results indicate that very little decomposition occurs in air below 110°C. Preliminary results on the products formed in the setting reaction of EBA-zinc oxide and EBA-eugenol-zinc oxide mixes have been obtained and should prove helpful in establishing the setting mechanism.

(b) Eugenol Isomers.

The ionization constants of a number of phenolic compounds related to eugenol have been determined. Methods to synthesize m-allylphenol are being explored. The mechanism of the Claisen rearrangement of allyl-2-benzoyloxyphenyl ether has been established by identifying l-allyl-2,3-dihydroxybenzene as a major reaction product.

The usefulness of the spectrophotometric techniques for the determination of ionization constants of conjugated aliphatic carboxylic acids has been briefly investigated employing acrylic and methacrylic acids as model compounds,

# 4.5 Investment

A report on factors affecting the dimensional changes of gold alloy casting investments is appended.

Measurements of the thermal expansion of investments for chromium-cobalt casting alloys were continued. Variations in heating rate were found to affect the thermal expansion obtained at the recommended casting temperature. Although this effect was not the same for all investments a heating rate of 10°C per minute produced greater thermal expansion values than did slower rates for most materials. 4.6 Evaluation of Materials

Materials evaluated for the Federal dental services or the American Dental Association by specification or other methods included agar impression material, alginate impression material, amalgam alloy, denture base resin, gold casting alloy, inlay wax, investment, mercury, plastic teeth, repair resin and silicate cement.

For the Director

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W. T. Sweeney, Chief Dental Research Section