# NATIONAL BUREAU OF STANDARDS REPORT

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

Progress Report

July 1 to December 31, 1961

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 a wide variety of dental materials, on dental equipment and on natural tooth structures continued at the National Bureau of Standards during the half year ending Deccember 31, 1961.

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 7407 Studies of Dental Calculus: Organic Portion.

NBS Report 7413 An Improved Zinc Oxide-Eugenol q-Ethoxybenzoic Acid Cement.

NBS Report 7443 Dental Radiographic Film: Standards and Specification.

#### 3. PAPERS PUBLISHED

Low Angle X-ray Diffraction of Crystalline Nonoriented Polyethelene and Its Relation to Crystallization Mechanisms. A. S. Posner, A. F. Dorio and D. E. Roberts. J. Applied Phys. 32:1509 Aug. 1961.

Effect of Mercury-Alloy Ration the Physical Properties of Amalgams. W. T. Sweeney, and C. L. Burns. J.A.D.A. 63:374 Sept. 1961.

Stability of Plastic Impression Trays. J. B. Woelfel and G. C. Paffenbarger. J.A.D.A. 63:705 Nov. 1961.

Improved Starch-Gel Electrophoretic Resolution of Parotid-Fluid Protein. K. C. Hoerman, S. A. Mancewicz and A. F. Forziati. J. Den. Res. 40:1293 Nov.-Dec. 1961.

#### 4. WORK IN PROGRESS

- 4.1 Human Tooth Enamel and Dentin
- (a) Fluorescence Studies.

Study of the relationship between organic content and fluorescence of hard tissues was continued. Serial sections of dog femur were photographed by the fluorescence method. The fluorescence intensity of each section is being determined by comparing the density of the image of the section with the density of the image of a disc of standard fluorescent glass included in each photograph. After extraction with boiling water the sections will be rephotographed by the fluorescence method and analyzed for nitrogen content. The results of these experiments and additional work on sections extracted with ethylene glycol-KOH should provide definite information on the relationship between fluorescence intensity and organic content.

Dentinal peptides, obtained by the partial hydrolysis of dentin matrix, were separated into five fluorescent bands by high voltage electrophoresis. The fluorescence excitation and emission spectra and the ultra violet absorption spectra of these fractions were recorded.

### (b) Dental Calculus Studies.

A detailed report of the studies of the chemical composition of dental calculus is appended.

#### 4.2 Metals

## (a) Amalgam.

The effects of various factors on the compressive strength of amalgam were investigated. A new and simplified specimen packing procedure resulting in higher and more consistent compressive strength values was developed. Data were collected for different cross-head speeds with both the Instron and Tinius Olsen testing machines. Compressive strength values increased with increased rates of loading and there appeared to be better agreement between the two machines at the higher loading rates.

Five amalgam alloys mixed with an S. S. White amalgamator developed maximum compression strength when triturated between 20 and 80 seconds. Using optimum trituration time and various testing temperatures for the five alloys, average 24-hour compressive strengths were about 60,000, 45,000 and 30,000 psi at 23, 40 and 60°C respectively. The average strength of the five widely used brands of amalgam was 15% lower at mouth temperature (37°C) than at room temperature (23°C).

To study the apparent growth of amalgam out of the cavity which is sometimes observed clinically an apparatus was designed to alternately dip amalgam specimens in hot (65°C) and cold (25°C) water. Specimens of two brands of amalgam alloy were condensed in 8 x 8 x 4 mm cavities in 20 x 20 x 10 mm pyrex glass blocks. Specimens cycled 72 times per hour between hot water and cold water containing aluminum oxide particles of 0.3, 1 and 5 u in size showed definite growth out of the cavity after 15,000 cycles. It is thought that the growth results from flow resulting from thermal expansion when the specimens are heated. Space between the amalgam and cavity walls produced when the amalgam is cooled appears to be filled with the aluminum oxide particles, thus resulting in further flow in subsequent cycles.

### (b) Gallium.

The study of gallium alloys, which may be useful as dental restorative materials has been reactivated. Work has centered on palladium-gallium-tin alloys. Equipment is being procured for melting in vacuum or in inert atmospheres and for annealing at high temperatures. An exploratory study of the intermediate phases in some binary alloys of gallium is underway. The knowledge of the structure and composition of these phases is needed in predicting the alloying behavior and physical properties of gallium alloys.

### 4.3 Resins

## (a) Denture Base Resins.

Measurements of the dimensions of clinical dentures made of 12 different types of resins continue into the fourth year. The long continued expansion of the epoxy dentures appears to be about ended. None of the other dentures changed in dimension appreciably after the third month. None of the dentures are warping in service.

## (b) Silica-Resin Direct Filling Material.

Preliminary tests have indicated that shrinkage on hardening of an experimental direct filling silica-resin (the adduct of glycidyl methacrylate and phenol reinforced with vinyl-silane-treated powdered silica) is considerably lower than the shrinkage of the conventional direct filling resins.

## (c) Adhesion Studies.

The search for substances that can be painted into the cavity, that will bond to the tooth structure and to a direct

filling resin and thus provide a tight seal continues. The synthesis of some surface-active comonomers was carried out. One such compound (the addition reaction product of N-phenyl glycine and glycidyl methacrylate) gave significant improvement in the strength of the bond between dentin and a direct filling resin (Kadon) when a 5 per cent ethanol solution of it was applied to the dentin prior to the Kadon. Without the surface-active comonomer, the adhesion of Kadon to dentin after soaking in water 20 hours was 0-10 pounds per square inch: with the surface-active comonomer the adhesion was 200-900 pounds per square inch under the same conditions. ing for five days in water did not significantly lower this bond strength. This surface-active comonomer is believed to have an amphoreric chelate structure which bonds it to the tooth and a methacrylate group which bonds it to the direct filling resin during hardening.

### 4.4 Zinc Oxide-Eugenol Materials

### (a) Zinc Oxide-Eugenol-EBA Cements.

A detailed report of the investigation of zinc oxideeugenol-o-ethoxybenzoic acid cements is appended.

### (b) Eugenol Isomers.

Isoch avibetol and iso-o-eugenol were synthesized. The ultraviolet and near infra-red spectra of eugenol, chavibetol, o-eugenol and 2-methoxy-3-allylphenol as well as of the corresponding propenyl isomers were determined. These spectra yield data with regard to bond interaction in the respective compounds. The acid ionization constants of six of these compounds have been determined by a spectro-photometric method. The validity of the Hammett equation for the ionization equilibrium constant of these vicinal and unsymmetrically trisubstituted benzenes has been investigated. The relative reactivity of these compounds in the setting reaction with zinc oxide is dependent on (1) the presence of ortho substituents which cause steric hindrance and (2) their acid strength. Compounds having larger acid ionization constants will show increased reactivity.

## 4.5 Investment

Data were obtained on the factors affecting the thermal and setting expansion of dental gold alloy investments. The previously reported reduction in thermal expansion with increase in water powder ratio and increase in hygroscopic expansion with increase in water added were verified. It was found that thermal expansion depends on the final water powder

whether the water is included in the initial mix or is added during setting to produce hygroscopic expansion. Thermal expansion was affected very little by variations in heating rate from 4 to 12°C per minute.

#### 4.6 Clinical Studies of Restorative Materials

The clinical comparison of the relative solubilities of certified silicate cements continues. Totals of 88 and 90 paired silicate cement fillings have been placed at the National Bureau of Standards and the National Training School for Boys, respectively. At the National Bureau of Standards, 15 silica-reinforced resin fillings have been paired with conventional direct filling resin and with silicate cement fillings. This in vivo comparison of the solubility of silicate cements is a check on the validity of the in vitro test in distilled water in the American Dental Association, military and international specifications. clinical comparisons of the silica-reinforced and unreinforced resins are to observe the effects of differences in the coefficients of thermal expansion, in moduli of elasticity and in other properties of the filling materials on the incidence of marginal staining, recurrent caries, leakage, abrasion and color stability.

### 4.7 Evaluation of Materials

Materials evaluated for the Federal dental services or the American Dental Association by specification or other methods included acrylic teeth, amalgam alloy, chromiumcobalt casting alloy, gold casting alloy, inlay casting wax, investment and zinc phosphate cement.

For the Director

Ву

W. T. Sweeney, Chief Dental Research Section

