NATIONAL BUREAU OF STANDARDS REPORT

5802

REPORT ON DENTAL RESEARCH
AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

July 1 to December 31, 1957

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. A major portion of the Bureau's work is performed for other Government Agencies, particularly the Department of Defense and the Atomic Energy Commission. The scope of activities is suggested by the listing of divisions and sections on the inside of the back cover.

Reports and Publications

The results of the Bureau's work take the form of either actual equipment and devices or published papers and reports. Reports are issued to the sponsoring agency of a particular project or program. Published 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 monthly periodicals, available from the Government Printing Office: The Journal of Research, which presents complete papers reporting technical investigations; the Technical News Bulletin, which presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions, which provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: The Applied Mathematics Series, Circulars, Handbooks, Building Materials and Structures Reports, and Miscellaneous Publications.

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 ($0.75), available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C.

Inquiries regarding the Bureau's reports should be addressed to the Office of Technical Information, National Bureau of Standards, Washington 25, D. C.

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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 Air Force Dental Service, the Navy Dental Corps, and the Veterans Administration.

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 restorative materials and equipment and on natural tooth structures has continued at the National Bureau of Standards during the half year ending December 31, 1957.

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

2. REPORTS ISSUED

NBS Report 5754	Proposed Specification for Impression Material; Synthetic Rubber Base, Dental.
NBS Report 5757	Investigation of Selected Waxes for Possible Use in Dentistry.
NBS Report 5758	Denture Base Reliners for Self Use.
NBS Report 5759	Effect of Calcium Treatment on Solubility and Calcium Uptake of Synthetic Hydroxyapatite and Rat Molar Enamel.
NBS Report 5791	Refinement of Hydroxyapatite Structure.

3. PAPERS PUBLISHED

The Structure of Bone Mineral. Aaron S. Posner. Clinical Orthopaedics 9:5, 1957.

Calcification in Lysine Deficiency. R. C. Likens, L. Bavetta and A. S. Posner. Archives of Biochemistry and Biophysics. 70:601, 1957.

Microstructure of the Human Tooth. F. L. Losee, W. H. Jennings, M. E. Lawson and A. F. Forziati. J. D. Res. 36:911 December 1957.

Characteristics of Ultrasonic Vibrations. William T. Sweeney, J.A.D.A. 55:819 December 1957.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Studies.

Sections of teeth from individuals who have lived since birth in areas where the water is rich in fluoride ions were photographed by the fluorescence method. These sections will

be analyzed for nitrogen and fluoride content through the cooperation of Georgetown University and the relationship between fluorescence and composition will be studied.

Chromatographic and infrared absorption studies of enamel and dentin constituents have been initiated in cooperation with the Naval Medical Research Institute.

(b) Crystallographic Studies.

Construction of a new low-angle diffraction camera was completed and studies of the particle size of tooth and bone mineral were made. Preliminary results show that certain bones which take up comparatively large amounts of fluoride in vivo appear to contain a higher percentage of finely divided material than do bone samples which take up less fluoride.

Work on the structure of lead apatite $Pb_{10}(PO_4)_6(OH)_2$ was continued. A study of octocalcium phosphate, a synthetic apatite of very low Ca/P ratio (8 to 6) was started. This material which resembles apatite in some respects is believed to be the first appearance of calcification in the deposition of calcified tissue in the body.

(c) Physical Property Studies.

A large number of specimens of dentin and enamel were made for determination of compressive strength, elastic limits and modulus of elasticity. Data for dentin show a strength of 30 to 55 x 10^3 psi and moduli of elasticity from 1.4 to 2.0 x 10^6 psi.

4.2 Metals

(a) Amalgam

Silver-Tin-Mercury System

Construction of the vacuum apparatus for measurement of mercury vapor pressures over alloys of the Ag-Sn-Hg system has been completed. Tests indicate that it will maintain pressures in the order of 10^{-3} μ of Hg for periods of up to 200 hours which is adequate for all contemplated tests. Specimens of various composition have been prepared and are being annealed preparatory to testing. The initial tests will be of tin-mercury binary alloys in order to obtain a comparison with the diffusion results obtained earlier.

Setting Time of Dental Amalgams

Previous tests of the setting time of dental amalgam had indicated that many of the commercial alloys exhibited an aging effect apparently independent of strain release. An additional series of tests were made to confirm these findings and to extend the period of natural aging covered by the investigation. Tests were made by different operators and the results were found to be independent of this factor. Other tests were made to determine the effect of normal variation in the proposed technique for determining setting time upon the results, and in all cases such variation was found to have no significant effect. This research clearly indicates that certain alloys are affected by shelf life a property not heretofore considered of practical clinical importance.

4.3 Resins

(a) Denture Reliners.

The physical properties of commercial self-curing hard denture reliners have been investigated. The materials have adequate flow and consistency. Peak curing temperatures vary from 30° to 53°C for resins cured at 23°C and from 59° to 79°C for those polymerized at 37°C. Water sorption is the same as that of denture base resins, but water solubility is considerably higher. Most brands show poor color stability. Some materials still show considerable flow at the time suggested in the manufacturer's instructions for the removal of the relined denture from the mouth. Details of the final impression made by the reliner are sometimes poor. Knoop hardness, and indentation and recovery values are somewhat lower than those of denture base resins due to the large amount of plasticizer in the reliner resin. The plasticizers in the monomeric and polymeric components of the reliners were determined quantitatively after identification by infrared absorption. A clinical study of relined dentures showed no significant warpage or dimensional change.

(b) Denture Base Resin.

Investigation of ten different denture base resins and processing methods was undertaken to determine if any particular type of resin or curing procedure would produce dentures with greater dimensional accuracy and/or stability without sacrificing the other desirable qualities in complete denture fabrication. Complete upper and lower technique dentures of both the thick and thin type were made in each of the denture base resins as controls. Clinical dentures are to be made

in each resin to determine patient reaction, dimensional change in service compared to technique dentures of the same material, and degree of clinical success achieved.

To date, 36 technique dentures have been completed, and 14 clinical dentures inserted. Results of the transverse deflection test indicate that polystyrene is significantly stiffer and stronger than the mean of the group, and the self-curing resin chosen was more flexible and weaker than the average. The range of dimensional shrinkage of the clinical and technique dentures has been -1.1% to 0.2% from molar to molar. Most of this shrinkage occurs upon removal of the denture from its master cast after processing. Subsequent linear changes are comparatively small, generally positive or showing a small expansion after prolonged immersion in water or clinical use. Greater dimensional changes (negative) were observed in lower dentures as opposed to upper dentures, thin dentures as opposed to thick dentures (upper and lower). With few exceptions, the dimensional changes occurring on the tissue side (across the buccal flanges) and the dimensional changes occurring on the polished surface (second molar to second molar) were very similar when compared on a percentage basis, indicating the majority of the change to be predominately shrinkage and expansion rather than one of warpage.

The self-curing resin exhibited the greatest linear expansion, the vinyl-acrylic copolymer exhibited the least linear expansion over a three-month period. The epoxy denture resin apparently had the least over-all linear change but was not color stable. The lower dentures made with the heat-cured resin containing glass fibers showed the greatest linear shrinkage. To date, all clinical dentures have demonstrated equal success, both from the viewpoint of the investigator and from the satisfaction of the patient.

(c) Silica-Resin Direct Filling Material.

An addition product of bisphenol A and glycidyl methacrylate (GMA) was prepared and thinned with a reactive diluent, tetraethylene glycol dimethacrylate. It was activated with 1% dimethyl-para-toluidene (DMPT). When mixed with clear fused quartz powder containing 1% benzoyl peroxide catalyst, it hardened in one to three minutes forming a hard, tooth-colored solid. The compressive strength when tested according to A.D.A. Specification No. 9 was 10,700 psi. When the same resin was mixed with fused quartz powder that had been given a vinyl silane surface treatment, the compressive strength was 16,000 psi. The visual opacity test indicated approximately 35% when tested as described in A.D.A. Specification No. 9.

The solubility and disintegration was 0.25%. X-ray opacity could be adjusted to match tooth structure (without perceptibly affecting the color or visual opacity) by using 20% to 30% of 325 mesh clear "flint" glass. Maximum compressive strength of 18,500 psi was obtained at about 5% BP and 80% treated quartz powder.

Another synthesis was made using 1.5 equivalents of GMA per equivalent of bisphenol A. Addition occurred in about four hours at 80°C with 0.5% DMPT as catalyst in nitrogen atmosphere. After synthesis another 0.5% DMPT was added as accelerator for room temperature polymerization and compressive strengths of approximately 22,000 psi were obtained.

Syntheses made using a nitrogen atmosphere produced a very light-colored resin. An air atmosphere produced an amber colored, darker but less viscous resin. Bisphenol A appears to darken in the presence of alkali, molecular oxygen and heat. Molecular oxygen appears to inhibit polymerization of methacrylate compounds. A synthesis using Epon 828, methacrylic acid, DMPT as catalyst and air as an inhibitor for the methacrylate group did not result in a darkened resin.

(d) Polymerization Studies.

Apparatus to study the benzoyl peroxide decomposition in the presence of amine in a vacuum up to 10^{-6} mm has been set-up. Rate of free radical formation of a few thoroughly degassed samples was studied.

Theoretical curves for the decomposition in benzene of diphenylpierylhydrazyl in its competitive scavenging reaction with iodine for isobutyronitrile radicals was calculated. The calculated values are in fair agreement with the experimental results.

(e) Gas Chromatography.

Investigations of the usefulness of gas chromatographic methods for the qualitative identification and quantitative determination of copolymers were continued. The constituents of a series of linear and cross linked acrylic copolymers have been identified by chromatographic analysis of their liquid pyrolysis products. Trace copolymeric components as low as 0.1% have been detected.

The composition of polymer mixtures can be determined within 1%. The chromatograms of the pyrolysis products of polymer mixtures and copolymers of known composition were com-

pared. Quantitative determination of the copolymeric constituents is most conveniently obtained from the peak height ratios and is accurate within a few percent.

For the analysis of solvent mixtures the bracketing method has proved satisfactory. Areas under the peaks are used to obtain quantitative data.

4.4 Elastic Impression Materials

(a) Alginate Materials.

A tentative draft of a revised specification for alginate impression materials had been drawn up as the result of work in the first six months of 1957. Much of this work had, however, been directed toward measurements which were not included in the final specification draft.

In order to confirm the suitability of the limits specified, additional samples of six popular commercial alginate impression materials were obtained and tested in accordance with the specification. Except for one minor revision in requirements and a few wording changes in procedure the specification was found to be suitable as written. The specification itself and the results of both sets of tests are being readied for publication.

(b) Synthetic Rubber Base Materials.

Additional data on new batches of rubber base impression materials were obtained and minor revisions were made in the tentative specification for these materials. The specification is appended as a separate report.

4.5 Color Standards

Procedures and equipment for obtaining more accurate measurements of the colors of silicate cements were developed. A simplified measuring circuit for determining tristimulus color values with the Gardner Color Difference Meter was designed and constructed. Necessary requirements for a new set of porcelain color standards for use with silicate cements were defined and the standards procured and calibrated. An improved specimen cell was constructed. Data were obtained on the differences in colors of standards produced by a change from an air to a water medium. The effect of different temperatures on the rate of color change of silicate cements was investigated.

4.6 Zinc Oxide Materials

Additional information has been obtained on the setting reaction and physical properties of zinc oxide-eugenol materials. Previous data had shown that these materials form a chelate containing two moles of eugenol and one of zinc oxide. Materials mixed in these proportions contained up to 97% combined eugenol but were very weak apparently because practically no zinc oxide became entangled during chelation. To determine the amount of combined eugenol that produces the highest compressive strength, cements with 0.1, 0.2 and 0.3 mole equivalents have been prepared. With 0.3 mole equivalent of eugenol specimens were strong enough and expanded sufficiently to split the thin walled glass cylinders in which they were formed.

4.7 Cutting Instruments

The test apparatus constructed to measure simultaneously the torque and speed was used to determine the characteristics of dental handpieces. With this apparatus light reflected from a sectored disc into a photo-cell, frequency meter system is used to measure the rotational speed of the handpiece and dental engine. Eddy currents induced in the disc by rotation through the gap of a pendulum-mounted electro-magnet apply a braking torque which is controlled by variation of the electric current to the magnet. The torque is measured in terms of the magnet displacement.

Representative samples of conventional, multiplying, and turbine handpieces were studied. The measured speeds ranged up to 200,000 rpm and the measured torques up to 300 gm-cm. As was to be anticipated, handpieces delivering the highest speeds exhibited the lowest torques. The results, for those handpieces having maximum speeds below 60,000 rpm (2/3 of those tested) are believed to be of good accuracy. In the higher speed ranges, however, air drag and dynamic imbalance of the large (2" diameter) disc introduced inaccuracies.

As a result a modified apparatus specifically designed for high-speed, low-torque handpieces is being constructed in which a small cylindrical permanent magnet is used in place of the disc. An external A.C. field of variable frequency is used to drive this magnet as the armature of a synchronous motor thus providing both torque and controlled speed. Preliminary tests have indicated the feasibility of this method and the working equipment is under construction.

4.8 Gypsum Materials

(a) Investment.

Physical properties of a number of gold alloy investments were determined to obtain data for revision of the Federal and American Dental Association specifications for these materials. The revised specifications will include requirements for hygroscopic inlay investment and thermally expanding partial denture investment in addition to the thermally expanding inlay investment listed in present specifications. Data obtained demonstrate that inlay thermal and inlay hygroscopic investments can be grouped together and tested within approximately the same consistency (powder-water ratio) limits used in present specifications. New consistency requirements are being developed to approximate the dryer mix normally used with partial denture investments.

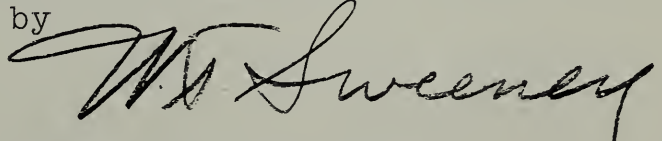
(b) Gypsum Setting Reactions.

Dilatometric studies coupled with x-ray diffraction, index of refraction and density measurements were made on the setting process of gypsum. The effect of adding K_2SO_4, gelatine, borax and other salts on setting kinetics and crystal formation were investigated. Crystal shape and size showed some relationship with dimensional changes on setting.

4.9 Evaluation of Materials

Materials evaluated for the Federal dental services and the American Dental Association by specification and special test methods included denture base resins, inlay casting gold alloys, mercuries, silicate cements, wrought gold wire alloys and zinc phosphate cements.

For the Director
by



W. T. Sweeney, Chief
Dental Research Section

U. S. DEPARTMENT OF COMMERCE
Sinclair Weeks, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

THE NATIONAL BUREAU OF STANDARDS

The scope of activities of the National Bureau of Standards at its headquarters in Washington, D. C., and its major laboratories in Boulder, Colo., is suggested in the following listing of the divisions and sections engaged in technical work. In general, each section carries out specialized research, development, and engineering in the field indicated by its title. A brief description of the activities, and of the resultant publications, appears on the inside front cover.

WASHINGTON, D. C.

Electricity and Electronics. Resistance and Reactance. Electron Devices. Electrical Instruments. Magnetic Measurements. Dielectrics. Engineering Electronics. Electronic Instrumentation. Electrochemistry.

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

Heat. Temperature Physics. Thermodynamics. Cryogenic Physics. Rheology. Engine Fuels. Free Radicals Research.

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

Chemistry. Organic Coatings. Surface Chemistry. Organic Chemistry. Analytical Chemistry. Inorganic Chemistry. Electrodeposition. Molecular Structure and Properties of Gases. Physical Chemistry. Thermochemistry. Spectrochemistry. Pure Substances.

Mechanics. Sound. Mechanical Instruments. Fluid Mechanics. Engineering Mechanics. Mass and Scale. Capacity, Density, and Fluid Meters. Combustion Controls.

Organic and Fibrous Materials. Rubber. Textiles. Paper. Leather. Testing and Specifications. Polymer Structure. Plastics. Dental Research.

Metallurgy. Thermal Metallurgy. Chemical Metallurgy. Mechanical Metallurgy. Corrosion. Metal Physics.

Mineral Products. Engineering Ceramics. Glass. Refractories. Enameled Metals. Concreting Materials. Constitution and Microstructure.

Building Technology. Structural Engineering. Fire Protection. Air Conditioning, Heating, and Refrigeration. Floor, Roof, and Wall Coverings. Codes and Safety Standards. Heat Transfer.

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

Data Processing Systems. SEAC Engineering Group. Components and Techniques. Digital Circuitry. Digital Systems. Analog Systems. Application Engineering.

• 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. Sun-Earth Relationships. VHF Research.

Radio Propagation Engineering. Data Reduction Instrumentation. Modulation Systems. Navigation Systems. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Radio Systems Application Engineering. Radio Meteorology.

Radio Standards. High Frequency Electrical Standards. Radio Broadcast Service. High Frequency Impedance Standards. Calibration Center. Microwave Physics. Microwave Circuit Standards.


NBS