

1958-1 Dr. Paffenbarger

NATIONAL BUREAU OF STANDARDS REPORT

6152

REPORT ON DENTAL RESEARCH
AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

January 1 to June 30, 1958

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.

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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.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

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NBS 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 Air Force Dental Service, 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 restorative materials and equipment and on natural tooth structure has continued at the National Bureau of Standards during the half year ending June 30, 1958.

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 5937	Dimensional Changes Occurring in Artificial Dentures During and Subsequent to Processing.
NBS Report 5964	Analysis of the Leach Products of Silicate Cements and Studies to Reduce Their Solubility.
NBS Report 6103	Denture Reliners - Direct, Hard, Self-Curing Resin.
NBS Report 6109	A Setting Time Test for Dental Amalgam.
NBS Report 6144	Properties of Dental Gold Alloy Casting Investments.

3. PAPERS PUBLISHED

American Dental Association Specifications for Dental Materials. G. C. Paffenbarger, J. W. Stanford and W. T. Sweeney. American Dental Association. Chicago, Illinois. January 1958.

Physical Properties of Chromium-Cobalt Dental Alloys. Duane F. Taylor, Walter A. Leibfritz and Alfred G. Adler. J.A.D.A. 56:348 March 1958.

Refinement of the Hydroxyapatite Structure. A. S. Posner, Alvin Perloff and A. F. Diorio. Acta Crystallographica 2: 308 April 1958.

Low-angle X-ray Diffraction of Fibrous Polyethylene. Leo Mandelkern, C. R. Worthington and A. S. Posner. Science 127: 1052 May 1958.

Silicate Cement: Proper Clinical Practice. G. C. Paffenbarger and J. W. Stanford. Transactions of the American Dental Society of Europe 1957, page 11.

Scientific Development and Methods in Practice. G. C. Paffenbarger and R. L. Bowen. J.A.D.A. 56:785 June 1958.

Physical Properties of Plastic Teeth. W. T. Sweeney, E. L. Yost and J. G. Fee. J.A.D.A. 56:833 June 1958.

The Reaction of Metal Oxides with X-ethoxybenzoic Acid and Other Chelating Agents. G. M. Brauer, E. E. White and M. G. Moshonas. J. D. Res. 37:547 June 1958.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Studies.

Using chromatographic methods, aspartic acid, hydroxyproline, tyrosine and traces of glycine and serine were found in aqueous bone extracts; histidine, lysine, aspartic acid and traces of glycine, serine hydroxyproline, tyrosine, phenylalamine, leucine and isoleucine were identified in extracts from femur collagen. Ground dentin was extracted with water using a Soxhlet extractor or a dialysis cell. Some amino acids and a peptide were recovered from the extracts and from the non-dialysable contents of the cell, respectively. A mixture of n-butanol and potassium hydroxide was found to be the most effective solvent for extracting fluorescent material from powdered enamel and dentin. A paper chromatogram of the extracted material showed five distinct fluorescent bands. Infrared absorption procedures for identification of the various extracts are being developed.

Preparation of about 3000 individual samples of 14 phosphor reference samples was completed. These are now issued by the National Bureau of Standards as Standard Samples.

(b) Crystallographic Studies.

Low angle x-ray diffraction technics are being used to characterize the role of collagen in the calcification of dentin and bone and to ascertain if there exists a chemical linkage between collagen and apatite in these tissues. Decalcified fish bone was found to retain the collagen low angle diffraction pattern. Subsequent recalcification as shown by electron microscopy and wide angle x-ray diffraction destroyed the low angle pattern of collagen. Low angle x-ray determinations of crystal size and orientation in these tissues will be made.

A study in cooperation with the National Institute of Dental Research, of the difference in uptake, retention and clearance between radiostrontium and radiocalcium in experimental animals showed that when rats were injected with a solution of Ca^{45} + Sr^{89} the $\text{Ca}^{45}/\text{Sr}^{89}$ ratio increased markedly within 24 hours. In vitro studies on synthetic hydroxyapatites tagged with Ca^{45} + Sr^{89} showed that this Sr^{89} discrimination may be explained by the process of recrystallization wherein the crystals constantly perfect gradually rejecting the foreign Sr^{89} ions.

4.2 Metals

(a) Amalgam.

Silver-Tin-Mercury System.

The vapor effusion apparatus designed for study of the Ag-Sn-Hg system was used for preliminary vapor pressure measurements on Hg and 90% Hg - 10% Sn at 37°C. Two orifice sizes were used. The results indicated that, while the use of the 37°C temperature was desirable from the point of view of dental application, experimental procedure would be simplified at higher temperature. Therefore, the water jacket has been replaced by an oil bath and the equipment has been modified to permit thermocouple measurements of cell temperature during runs. The preliminary results show better performance at 65°C.

Setting Time of Dental Amalgam.

A report on the test developed for measuring the setting time of dental amalgam is appended. This method will make it feasible for manufacturers to quantitatively measure the setting time of dental amalgam alloys by a standardized laboratory procedure which correlates with clinical experience.

X-ray Fluorescence Analysis of Amalgam Alloys.

An extensive study of the application of x-ray fluorescence methods to the analysis of amalgam alloys was made. Samples of the amalgam alloy were irradiated in various forms, as received, ground, briquetted with and without an aluminum diluent and in solution. The characteristic x-radiations of the elements produced by this irradiation were resolved by diffraction methods and measured. Comparison of this data with the composition of the alloys as determined by wet analysis indicated that x-ray fluorescence determinations were accurate to 1 or 2%. The method is not satisfactory for

specification tests which require determinations to 0.5% or better. It could be used, however, as a rapid screening test so that wet analysis would be needed only for those alloys with compositions near specification limits.

(b) Gold Alloys.

Casting Procedures.

A study of the effect of casting procedure on the properties of gold alloy castings was initiated. Investigation of the relationship between surface smoothness and the temperature of the alloy when cast is now in progress.

Gold Alloy Analysis.

Work on a method to shorten the time in making analytical determinations of dental gold alloys containing platinum and palladium was continued. The method objective is to determine the noble metals in a lump sum, instead of individually as developed for high precision analysis by R. Gilchrist in 1938. Considerable progress has been made using sodium formate as a precipitant of the noble metals. The reduction to metal is pH sensitive. Some copper does precipitate at the minimum pH. The work is being continued to study such procedures as complexing the copper so it will not precipitate with the noble metals as well as different approaches such as electrogravimetric methods.

4.3 Resins

(a) Denture Reliners

A report on the properties of self-curing hard denture liners is appended.

(b) Denture Base Resins.

A progress report on the investigation of different types of denture base resins and processing technics is appended. Additional clinical and technic dentures are being made, and measurements on the dentures now in service will continue to be made to supplement the information given in the progress report.

(c) Silica-Resin Direct Filling Material.

Vinyl silane treatment of quartz particles used as a filler in resins prepared for possible use as direct filling materials resulted in increased compressive strength of as much as 30 to 60% over the strength of mixtures containing untreated quartz in some instances. Vinyl silane treatment of glass surfaces on which the resin mixtures were placed also increased greatly the adhesion of the resin to glass. In preliminary tests treatment of tooth surfaces with uncatalyzed vinyl trimethoxyethoxysilane reduced adhesion of the filled resin to enamel; 10% undecylenic acid in n-butanol increased adhesion to dentin but not to enamel; high MW polyisocyanate increased the adhesion to enamel; and oleic acid increased the adhesion to dentin.

(d) Polymerization Studies.

Kinetics of the relative reactivities of iodine and p-nitroaniline as compared to diphenylpicrylhydrazyl were studied using solutions containing varying concentrations of the inhibitors.

The decomposition rate of benzoyl peroxide in the presence of dimethylaniline in a high vacuum using diphenylpicrylhydrazyl (DPPH) as a radical scavenger could not be determined accurately since the amine also reacts rapidly with D.P.P.H. Studies have been initiated on the nature of the amine-D.P.P.H. reaction and its dependence on oxygen pressure above it. Dimethylaniline oxide has been synthesized and the spectrum of highly purified samples will be compared with those of pure dimethylaniline and with dimethylaniline mixed with air. Results are expected to show the formation of a dimethylaniline-oxygen complex which has an effect on the reaction with D.P.P.H.

(e) Gas Chromatography.

An extensive study of the quantitative determination of components of methyl methacrylate - ethylene glycol-dimethacrylate copolymers was made. The pyrolysis products of the polymers were chromatographed and their peak height ratios were compared. Standard analysis curves were established that would afford an accuracy of $\pm 1-2\%$ in the 5-25% copolymer range and a maximum of $\pm 5\%$ in the 30-50% copolymer range. The main source of error appeared to be variations of experimental conditions during depolymerization which are difficult to control.

Analysis of commercial copolymers gave results that agreed with the composition given by the manufacturer.

4.4 Color Standards

The determination of acceptable values for the colors of reference standards in water appears to be necessary before color designations for silicate cements can be established. Measurements of the differences between colors of porcelain color standards in air and in water indicated that the water values obtained depend upon the type of color difference meter used. Additional studies are being made in cooperation with the NBS Photometry and Colorimetry Section in an attempt to eliminate as far as possible the instrument dependence of water values assigned to the porcelain standards to be used in the determination of the colors of silicate cements.

4.5 Zinc Oxide Materials

Compressive strength tests on zinc oxide-eugenol cement using reaction molar equivalents of 0.1, 0.15-0.175, 0.2 and 0.3 of eugenol were made. The highest compressive strengths determined were with the 0.15 to 0.175 values. When cements are made with amounts of eugenol less than 0.2 molar equivalents, the combined eugenol is less and compressive strength is higher, 4000 psi or more. To date it is believed that high compressive strength depends on the formation of very small chelate crystals the growth of which is inhibited by the conditions which cause their formation.

4.6 Cutting Instruments

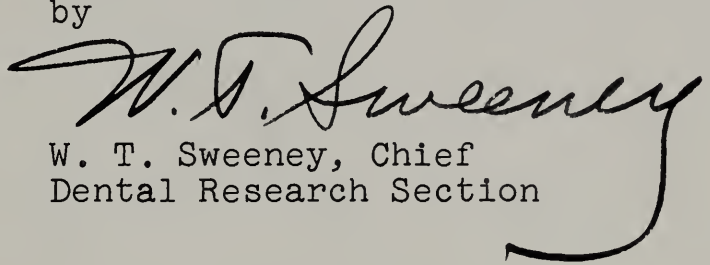
New high speed-low torque measuring apparatus for the laboratory testing of dental handpieces was completed. Auxiliary equipment for the determination of speeds employed under clinical conditions was also designed and constructed.

Several models of air turbine handpieces from various manufacturers were tested in cooperation with the Armed Forces Institute of Pathology and the U. S. Naval Dental School. Torque and speed curves were obtained for each instrument examined. The data thus obtained are currently being analyzed and integrated in an effort to more expressly define the energy-work relationships in modern dental cutting instruments, and to better understand the mechanism of cutting.

4.7 Evaluation of Materials

Materials evaluated for the Federal dental services and the American Dental Association by specification and special test methods included amalgam alloys, chromium-cobalt casting alloys, denture base resins, hydrocolloidal (agar) impression materials, inlay casting gold alloys, inlay casting investments, mercuries, self-curing repair resins and zinc phosphate cements.

For the Director
by

A handwritten signature in cursive script, reading "W. T. Sweeney". The signature is written in black ink and is positioned above the typed name and title.

W. T. Sweeney, Chief
Dental Research Section

