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NATIONAL BUREAU OF STANDARDS REPORT

6696

REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

July 1 to December 31, 1959

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.

NATIONAL BUREAU OF STANDARDS 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.

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 and equipment and on natural tooth structures continued at the National Bureau of Standards during the half year ending December 31, 1959.

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 6660	Revision of American Dental Association Specification No. 4 for Dental Inlay Casting Wax.
NBS Report 6667	Surface Roughness of Dental Gold Castings.
NBS Report 6690	Thermal Expansion of Denture Base Resins.
NBS Report 6691	Effect of Mercury-Alloy Ratio on the Physical Properties of Amalgam.

3. PAPERS PUBLISHED

Comparative Metabolism of Calcium and Strontium in the Rat. R. C. Likens, A. S. Posner, M. L. Kunde and D. L. Craven. Arch. Biochem. Biophys. 83:472 Aug. 1959.

Method of Evaluating the Clinical Effect of Warping a Denture: Report of a Case. J. B. Woelfel and G. C. Paffenbarger. J.A.D.A. 59:250 Aug. 1959.

Denture Reliners - Direct, Hard, Self-Curing Resin. G. M. Brauer, Eli E. White, Jr., Claire L. Burns and J. B. Woelfel. J.A.D.A. 59:270 Aug. 1959.

Dimensional Changes in Systems of Fibrous Macromolecules: Polyethlene. L. Mandelkern, D. E. Roberts, A. F. Diorio and A. S. Posner. J. Am. Chem. Soc. 81:4149 Aug. 20, 1959.

A Discussion of Federal Specification GG-X-620 and L-F-310 for Dental X-ray Apparatus and Dental X-ray Film. G. C. Paffenbarger, A. F. Forziati and M. P. Kumpula. J.A.D.A. 59:473 Sept. 1959.

Some Clinical Applications of Research Findings in Dental Materials. G. C. Paffenbarger. Ohio Dental J. 33:218 Sept. 1959.

Dimensional Changes Occurring in Artificial Dentures. J. B. Woelfel and G. C. Paffenbarger. Internatl. Dental J. 9:451 Dec. 1959.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Studies.

Isolation and Identification of the Fluorescent Components of Tooth Structure.

Organic material has been extracted from dentin by means of (a) hot water; (b) 0.1 NHCl acid; and (c) ethylene glycol containing 3% KOH. The extracts were filtered, treated with ether to remove lipids, and then chromatographically fractionated on a Dowex 1-X-2 column. The fraction showing the strongest fluorescence was subjected to acid hydrolysis at elevated temperatures and pressures. Subsequent paper chromatographic seperation of the hydrolysate, using a butanol/acetic acid/water system, yielded six nonfluorescent spots characteristic of six amino acids. However, the spot at the point of application showed considerable fluorescence. The nonmigration of the fluorescent material suggests the presence of a fluorophore group incorporated in the polypeptide. Attempts to release the fluorescent material by treating the protein complexes with enzymes are being made.

Tooth Fluorometer.

Clinical tests showed that the alignment of the fluorometer, with the tooth under study, was critical and that two operators were required to make the necessary measurements. An improved model has been designed and built. The new instrument is equipped with pivots and swivels so as to facilitate positioning. By incorporating automatic controls and a strip chart recorder, one investigator can now satisfactorily operate the fluorometer.

(b) Crystallographic Studies.

Low angle x-ray diffraction studies on long chain polymers were carried out during this period. Work was done on collagen and other proteins as well as synthetic polymers such as polyethylene which resemble proteins in certain ways. A new camera for medium resolution of low angle diffraction patterns was designed, built and assembled. One of the major aims of this study is to elucidate the relationship between collagen and apatite in calcified tissue.

A project on the Fourier analysis of broadened x-ray lines was begun. This is intended to determine whether the x-ray diffraction line broadening found in biological apatite patterns is due to small crystal size, strain on the atomic scale or some combination of both of these reasons. This Fourier method has been programmed for the IBM No. 704 electronic computer.

The work on the shrinkage of highly crystalline, oriented polymers (polyethylene, rubber, collagen, keratin, and muscle) is being continued. Special x-ray diffraction cameras have been designed and built to facilitate this study.

A cooperative study on the comparative metabolism of Ca and Sr in calcified tissue has been carried out in cooperation with the National Institute of Dental Research.

(c) Studies of Dental Calculus.

Organic Portion.

Existing methods of analyses for lipids, proteins and carbohydrates were reviewed. Samples of calculus were collected to form a composite sample to evaluate methods of separation of the inorganic from the organic portion.

The following solvents have been used: (a) boiling water, (b) ethylene glycol with KOH, and (c) ethylenediamine tetraacetic acid. Initial qualitative chemical and infrared spectroscopic studies have revealed peptide linkages in the organic residue indicating the presence of protein. Thus far, infrared spectra show only CH_3-, $>CH_2$, and $>C=O$ bonds present in the lipid fraction. This would indicate the presence of a neutral fat. Hydrolyses of the protein fractions for detection of the amino acids present have been initiated.

4.2 Metals

(a) Amalgam.

Tensile Properties.

Tensile strength and modulus of elasticity were determined on several brands of amalgam alloy. Specimens were dumbbell shaped with a straight gage length approximately 0.3 inch long and 0.1 by 0.1 inch cross section. Since the stress-strain curves did not have a straight line portion, secant moduli were calculated. Results were as follows for seven day old specimens.

Brand	Modulus from 450 psi to 3000 psi	Modulus from 450 psi to 5100 psi	Tensile Strength
Aristalloy	4.6×10^6 psi	3.0×10^6 psi	8,300 psi
Caulk Regular	3.6	2.6	7,100
Caulk Micro Pellet	3.2	2.2	7,000

Variations in strain rate from head speeds of 0.0003 to 0.05 inches per minute did not produce significant differences in tensile strength of seven day old specimens.

Tensile strength was found to vary with age of specimens as shown below.

Brand	Tensile Strength in PSI			
	5 hours	1 day	7 days	14 days
Aristalloy	--	6000	8000	--
Caulk Regular	--	6700	7000	8100
Caulk Micro Pellets	4300	5800	6800	7200

(b) Gold Alloys

Gold Alloy Analysis.

The procedure for determination of Pt metals was revised. Difficulties were encountered with about one quarter of the 16 wrought gold wires being analyzed. Troubles were with slow, incomplete and premature precipitation, lack of coagulation and in washing the precipitate.

These difficulties have been overcome and an accurate method has been developed. Revisions in the procedure consist of:

1. Nearly enough sulfuric acid is added to convert the solution to sulfates. After evaporation to dryness an excess of H_2SO_4 is added and sample is heated until the white fumes of H_2SO_4 appear. After cooling and washing beaker sides with water the sample is reheated to H_2SO_4 fumes. Thus all salts are converted to sulfates free of nitrates and

nitrites and precipitation will not be slow or incomplete.

2. Next, the salts are dissolved by heating with diluted HCl (1 + 99) and then silica is removed by filtering. Diluted HCl prevents premature hydrolytic precipitation.

3. The Pt metals are precipitated with sodium formate at a pH of 5. The precipitate is coagulated by heating several hours (usually overnight) and at the same time the solution becomes slightly alkaline.

4. The precipitate is filtered and washed with a 1% ammonium formate solution (pH = 8 1/2). No trouble in washing the Pt metals precipitate has occurred in reanalyzing 8 gold wires.

5. A reagent blank must be used because of the large amounts of sodium salts and because of the high Phs.

Quantitative results with known amounts of platinum have been excellent with errors of 0.0 to 0.2 milligrams occurring.

At present the procedure in its entirety is being checked against known amounts with mixtures of Ag, Au, Pd, Pt and Cu. The analysis of 8 more wrought gold wires is being completed to further check out the procedure.

4.3 Resins

(a) Denture Base Resins

Additional data were obtained on the dimensional changes of different types of denture bases. The dentures of 43 patients after about two years of service did not fit as well as when first inserted. As the dentures changed little in dimension after insertion, except for a few cases, the lack of fit was attributed to tissue changes. The retention of dentures that had changed in dimension was as good as the retention of dentures having little or no change. The dentures made of an epoxy resin continued to enlarge in all directions - some as much as 1 1/2 percent linearily and the porcelain teeth were loosening.

Thin upper and lower dentures having 12 different resin bases were: (a) dried 22 days, (b) placed in water at 37°C for 22 days, and (c) heated in water for 30 minutes at 10 degree intervals from 50 to 100°C. The weight loss in (a) ranged from 0.38% (vinyl-acrylic resin) to 1.29% (cold-curing acrylic resin); the weight gain in (b) from 0.35% (vinyl-acrylic resin) to 1.20% (polymethyl methacrylate). The gain in weight from (b) through (c) ranged from 0.34% (cold-curing acrylic resin)

to 0.14% (polystyrene). On procedures (a) and (b) the dimensional changes generally followed the weight changes with the exception that vulcanite expanded on drying and shrank on wetting. In (c) the largest distortions occurred in the dentures made of polystyrene; the least in cold curing acrylic resin. The temperature at which the major part of the strain was relieved ran from 50°C (glass fiber-filled acrylic resin) to 90°C (polystyrene).

(b) Denture Reliners.

Results of consistency, temperature rise and hardness tests of hard reliners conducted according to the proposed specification for these materials gave unsatisfactory reproducibility for different observers. Evaluation of the techniques used indicated that the experimental conditions outlined in the specification were not specific enough for uniform application by different investigators. The wording of the specification has therefore been clarified.

(c) Silica-Resin Direct Filling Material.

For comparison with the properties of the experimental silica-resin direct filling material, tensile strength tests were made on a zinc phosphate cement (400 psi), human dentin (6,000 to 8,000 psi), human enamel (1,000 to 2,000 psi), bovine dentin (8,000 psi) and bovine enamel (3,000 psi). The silica-resin tensile strength depended upon the treatment of the silica powder: when control powder having no vinyl silane treatment was used, the material had an average tensile strength of 1,500 psi, while the best vinyl silane treatment gave 4,600 psi for the material.

The density of the synthetic monomer was between 1.11 and 1.16 g/ml depending on the amount of reactive diluent in it. The refractive index at 26°C was between 1.526 and 1.539. The apparent number average molecular weight was about 400.

(d) Gas Chromatography.

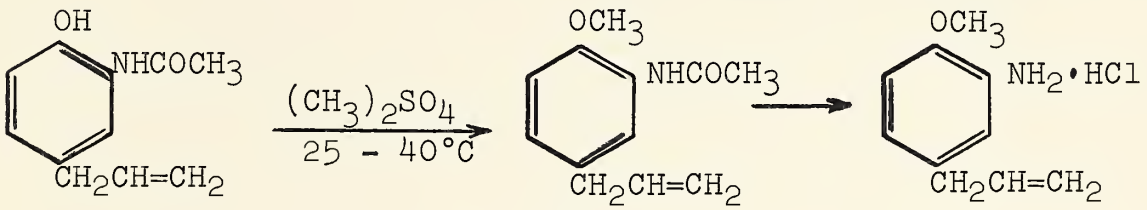
A study of the effect of temperature on the pyrolysis of polymers using gas chromatographic analysis of the products has been initiated. A Vycor pyrolysis boat surrounded by a heating element and containing a thermocouple has been incorporated in the chromatographic inlet system. Pyrolysis of poly(methyl methacrylate) has been followed between 350° and 850°C. It degrades nearly exclusively into monomer at temperatures ranging from 350° to 550°C. At higher temperatures eight products, some of them not identified so far, were obtained.

Degradation of polystyrene yielded six products which were identified as styrene, ethylene and ethane, toluene, benzene and ethylbenzene. At 400°C 97% styrene is formed whereas at 1000°C 67% (by wt. of product) is made up of monomer. Comparison of the yields of products obtained with those of Madorsky and co-workers shows a higher percentage of styrene and smaller quantities of secondary decomposition products. Since under the conditions used in the present experiments the pyrolysis products were more rapidly removed from the hot zone a decrease in secondary reactions would be expected.

4.4 Synthesis of Isomers of Eugenol

A large scale synthesis of 2-methoxy-3-allyphenol was attempted. The yields obtained previously for the first 4 steps could be duplicated or improved. Difficulties were encountered in preparing 2-methoxy-3-allyaniline. On purification a portion of the product polymerized and it appears that isomerization to 2-methoxy-3-propenylaniline took place.

An alternate route to synthesize chavibetol is being explored. The 2-acetamido-4 allylphenol (I) obtained in low yield on synthesis of the 2-acetamido-6 allyphenol by the Claissen rearrangement was methylated to 2-methoxy-5-allylacetanilide (II)



84%

I II III

An impure amine hydrochloride III has also been obtained.

4.5 Investment for Chromium-Cobalt Alloys

An investigation of the properties of investments used with dental chromium-cobalt casting alloys has been started. Preliminary tests are being made by methods described in the revised specification for gold alloy investments. Data obtained will be used to develop test procedures and specification requirements for the chromium cobalt alloy investments.

4.6 Evaluation of Materials

Materials evaluated for the Federal dental services and the American Dental Association by specification and special test methods included amalgam, denture base resin, inlay casting gold alloy, inlay casting investment, inlay casting wax, mercury, plastic teeth, self-curing repair resin and wrought gold wire alloy.

For the Director
By

W. T. Sweeney, Chief
Dental Research Section

USCOMM-NBS-DC