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G. Paffenbarger

NATIONAL BUREAU OF STANDARDS REPORT

6329

REPORT ON DENTAL RESEARCH
AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

July 1 to December 31, 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.

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.

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.

NBS

U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

1. INTRODUCTION

Research on materials and equipment used in restorative dentistry and on natural tooth structures has continued at the National Bureau of Standards during the half year ending December 31, 1958.

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

2. REPORTS ISSUED

NBS Report 6308	Analysis of Methyl Methacrylate Copolymers by Gas Chromatography.
NBS Report 6317	Microstructure of the Human Tooth.
NBS Report 6326	Evaluation of Dental Cutting Procedure: Method and Apparatus.
NBS Report 6327	A Method of Evaluating the Clinical Effect of Warping a Denture: A Case Report.
NBS Report 6333	Development of a Silica-Resin Direct Filling Material.

3. PAPERS PUBLISHED

The Program of Training and Graduate Instructions in Dental Materials at the National Bureau of Standards. W. T. Sweeney. J. D. Ed. 22:217 May 1958.

Change in A.D.A. Specification No. 12 for Denture Base Resin. J.A.D.A. 56:910 June 1958.

Co-Report: Developments in Instruments of Materials and Instruments, Dental. W. T. Sweeney. Int. Dent. J. 8:238 June 1958.

Effect of Calcium Treatment on Solubility and Calcium Uptake of Synthetic Hydroxyapatite and Rat Molar Enamel. R. C. Likens, A. S. Posner, and A. C. Steere. J.A.D.A. 57:335 September 1958.

Dimensional Stability of Denture Base Resins. W. E. Mowery, C. L. Burns, George Dickson, and W. T. Sweeney. J.A.D.A 57:345 September 1958.

Changes in American Dental Association Specification Nos. 7 and 14. J.A.D.A. 57:545 October 1958.

Changes in Specification No. 15. J.A.D.A. 57:577 October 1958.

Zinc Phosphate and Silicate Cements. G. C. Paffenbarger and J. W. Stanford. The Dental Clinics of North America. p. 561 November 1958. W. B. Saunders, Co., Philadelphia, Pa.

Acrylic Resins in Prosthetic Dentistry. W. T. Sweeney. The Dental Clinics of North America. p. 593 November 1958.

Cobalt-Gallium Dental Alloys. Denton L. Smith and Harold J. Caul. U. S. Patent No. 2,864,695, Issued Dec. 16, 1958.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Studies.

Reference infrared absorption spectra for pure samples of amino acids were established for the spectrophotometer used in this study. Comparison with the absorption curves obtained from chromatographic eluates showed that these eluates are mixtures requiring further separation.

Attempts to determine the fluorescence emission spectrum of five fluorescent bands obtained on a paper chromatogram of dentin extracts were unsuccessful due to the instability of the fluorescent material toward ultraviolet radiation. Instruments which would decrease the time of exposure of the fluorescent material to ultraviolet radiation and thereby permit measurement of the emission spectra are being considered.

To investigate the possible sources of the fluorescent material in teeth, the products of lactobacillus, and proteolytic, gram-positive and gram-negative bacteria grown on various culture media are being studied. A fluorescent material has been obtained in preliminary experiments.

(b) Crystallographic Studies.

The octocalcium phosphate study was continued with the thermal decomposition of this compound resulting in $CaHPO_4$ and hydroxyapatite. This process was followed by infrared absorption spectroscopy and x-ray diffraction.

A study of the possible existence of $(OH)_4$ groups substituted for $(PO)_4$ groups in calcified tissue was started.

Hydrogarnet, an example of a compound containing tetrahedrally coordinated cations in which these cations are partially or entirely missing, was prepared but so far no large crystals have been successfully prepared.

A study of polyethylene which may be of assistance in the determination of the structure of collagen was undertaken. Low angle and wide angle x-ray diffraction data were collected on samples of polyethylene crystallized at different temperatures. The low angle spacings change markedly with temperature of crystallization while the wide angle spacings do not change appreciably.

(c) Physical Property Studies.

Data were obtained on compressive strength, elastic limit and modulus of elasticity of approximately 200 cylindrical specimens of hard tooth tissue. A study is being made of the relationships between these mechanical properties and the position and orientation of the specimens in the tooth, tooth vitality and fluoride content of drinking water in the area in which the tooth developed.

4.2 Metals

(a) Amalgam.

The studies on amalgam setting time have been continued with emphasis upon the effects of shelf ageing of the alloy and specimen size upon the shear strength of amalgam. The determinations in progress of alloy naturally aged for two years correlate well with earlier results on artificially aged alloys. Two separate effects of specimen thickness have been observed. An analysis of over 800 tests indicates no effect of random variations in the thickness of specimens of the same nominal dimensions upon shear strength and thus on setting time. It was found, however, that large variations ($>$ 50%) in the amount of material used to form the specimens did show a direct correlation with indicated shear strength.

(b) Gold Alloys.

Casting Procedures.

Data relating the degree of surface roughness to nominal temperatures of the mold when removed from the oven after burnout and that of the molten gold alloy when cast have been recorded. One hundred sixty inlay dental gold castings have been cast with mold burnout temperatures varying from 800°F to 1500°F and alloy

casting temperatures varying from 1700°F to 2200°F. Extremely high mold and metal temperatures increase surface roughness. The effect of high temperatures seems to be an additative one. At burnout temperatures below 1300°F and metal casting temperatures below2000°F, surface roughness appears to be constant.

Gold Alloy Analysis.

A method of determining the noble metal content of dental gold alloys has been developed which is a modification of the Gilchrist method. After the removal of silver, base metals and gold the remaining solution containing only the platinum metals as complex nitrites is evaporated to dryness with a small amount of sulfuric acid and the nitrites and nitrates are converted to sulfates. The platinum metals are then precipitated from a slightly alkaline solution with sodium formate. Coagulation of the platinum metals occurs nicely at pH's above 7 and the problem of colloidal precipitates is overcome.

At the present time the method is being carefully checked using known amounts of metals. This method will considerably reduce the time necessary to analyze this type of alloy.

(c) Chromium Cobalt Alloys (Effect of Remelting).

A comparison of some properties of chromium cobalt dental castings made from new metal and from a 50-50 mixture of new and reused (scrap) metal was made. Properties determined were tensile strength, yield strength, percent elongation and modulus of elasticity. On the basis of results obtained on 14 castings of new metal and 18 of the 50-50 mixture there appears to be no significant difference between the tensile properties of castings made from new metal and from the mixture. Although these comparisons are based on castings made of metal which had been resued only once and although some properties such as corrosion resistance were not determined, the results are sufficiently encouraging to warrant a clinical evaluation of the effect of reusing chromium cobalt alloy.

More work is in process to evaluate the effect of several remelts on the physical properties of the castings.

4.3 Resins

(a) Denture Base Resins.

The clinical and laboratory investigation of different types of denture base resins and processing technics was continued. Additional measurements of dimensional changes were made on both the clinical and technic dentures. Tests on the ten resins under study showed that only one failed to comply with the water sorption and solubility requirements of American

Dental Association Specification No. 12. Coefficients of thermal expansion of the denture base resins is being determined under both dry and wet conditions.

(b) Silica-Resin Direct Filling Material.

A report on the results obtained so far in the development of a silica-resin direct filling material is appended.

(c) Polymerization Studies.

In the study of the kinetics of the decomposition of benzoyl peroxide, it was found that dimethyl-aniline (D.M.A.) and diphenylpicrylhydrazyl (D.P.P.H.) react with each other. In air the reaction is reasonably slow. The kinetics of the decomposition of benzoyl peroxide with dimethylaniline in air have already been studied and found to be complex. At pressures of about one mm of mercury, the reaction of D.M.A. with D.P.P.H. is much slower, but it is very sensitive to the pressure and it was difficult to get reproducible results. At pressures of about 10^{-6} mm. of mercury the D.M.A. - D.P.P.H. reaction is very rapid and kinetic studies were impossible on the benzoyl peroxide decomposition.

It is believed that a complex between D.M.A. and oxygen may explain the dependence of the D.M.A.-D.P.P.H. reaction on the pressure of air above it. Pure D.M.A. was prepared and compared to the material which had been exposed to oxygen in the ultraviolet, visible and infrared regions of the spectrum. The only significant difference was found in the "yellow" region at about 400 mμ, and this was not measurable quantitatively.

It was also found impossible to separate the oxygen complex by absorption chromatography by iodometric titration. Samples were studied by paramagnetic resonance to detect the presence of free radical species, but this also failed to show significant differences. It is thought that the best way to establish the mechanism of this reaction would be the separation and purification of products obtained on reacting larger quantities of D.M.A. and D.P.P.H. in appropriate solvents.

(d) Gas Chromatography.

A modified procedure for the quantitative determination of components of copolymers was developed. The inlet system of the chromatograph was redesigned so that the decomposition products of the sample which is pyrolyzed on a hot wire are carried directly into the column by the helium gas. Using this technique the components of methyl methacrylate-ethyl methacrylate copolymers could be quantitatively determined within 1%. A

progress report on the previous work is appended.

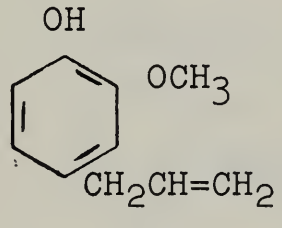
4.4 Color Standards

Tristimulus color values were assigned to two "duplicate" series of six porcelain color standards by comparison in air with a group of reflectance standards maintained in the NBS Photometry and Colorimetry Section. The porcelain standards are being used for measurement in water of the colors of silicate cement specimens representing most of the shades commercially available. Specimens are being measured after ageing for periods of one day up to several weeks to obtain data on change of color with age. Arrangements have been made to have a series of specimens measured in manufacturers' laboratories (L. D. Caulk Co. and S. S. White Mfg. Co.) to check the reliability of the procedure used.

4.5 Zinc Oxide Materials

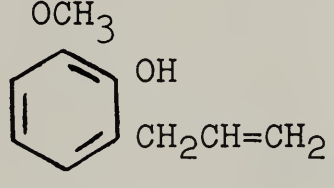
Synthesis of Isomers and Chelates of Eugenol.

A study to correlate structure and reactivity of position isomers of eugenol capable of forming metal chelates was initiated. Only one of the three possible position isomers of eugenol (o-eugenol) is readily available



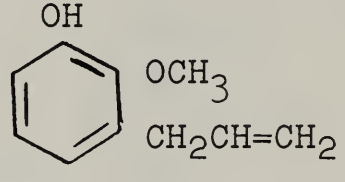
eugenol

I



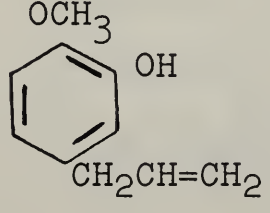
o-eugenol

II



2-methoxy-3-allylphenol

III

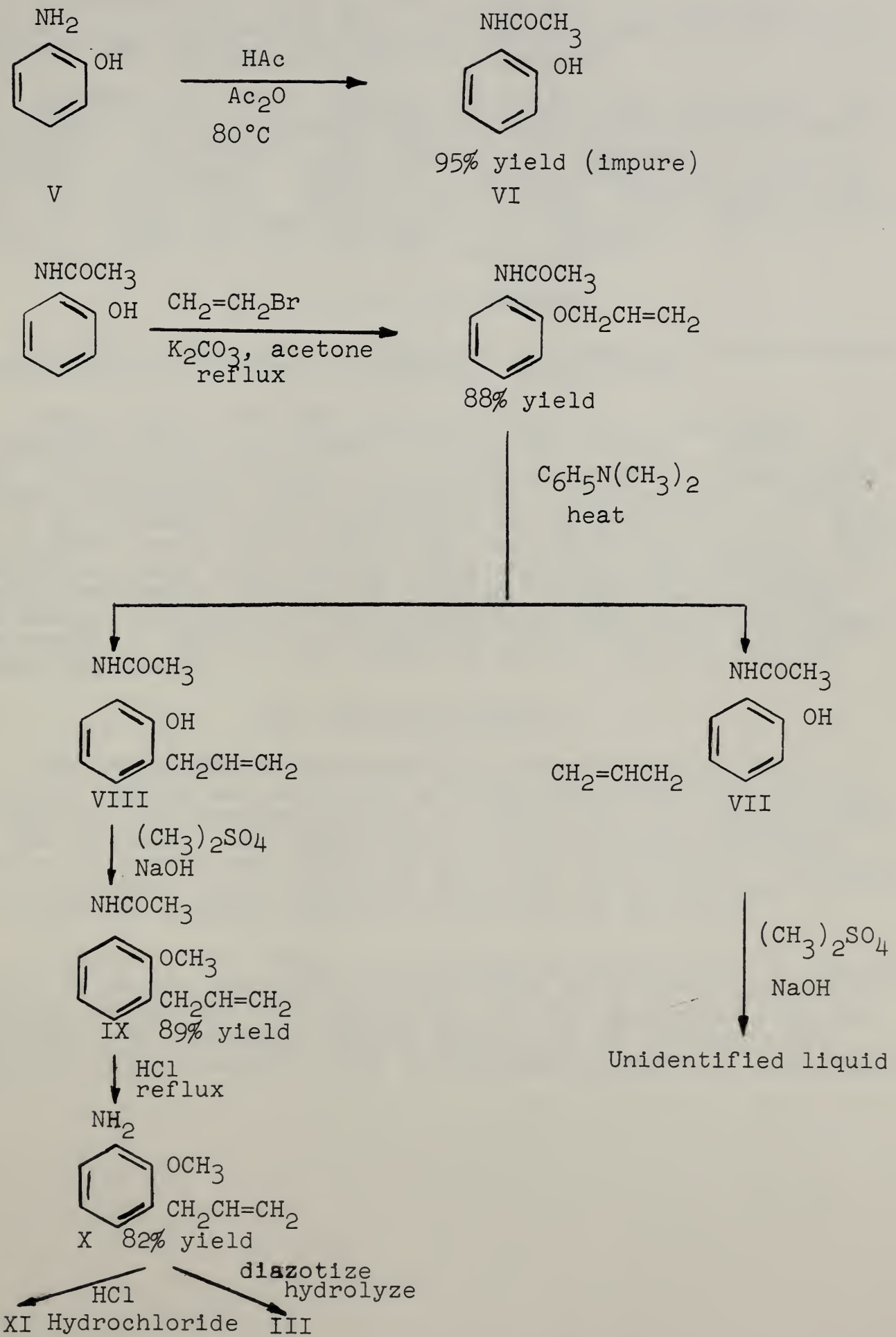


IV

2-methoxy-5-allylphenol

Compounds III and IV are now being synthesized. 2-methoxy-3-allylphenol III has not been reported in the literature and an attempted synthesis through the following reaction steps is

now being conducted.



Compounds IX, X and XI which have not been previously reported in the literature were identified by analysis for carbon, hydrogen and nitrogen and infra-red analysis.

Synthesis of metal chelates of eugenol and isoeugenol was attempted in order to study the physical properties of these compounds. The eugenol was reacted with the respective metal acetates in methanol. Under the reaction conditions used the zinc and cupric metal chelates were formed. The chelate formation took place on reacting eugenol or isoeugenol and with calcium, nickel or lead acetate, respectively.

4.6 Cutting Instruments

A report describing instruments developed for speed-torque characteristics of dental handpieces is appended.

Speeds up to 400,000 rpm and torques up to 400 gram-centimeters have been rather extensively studied. Results show that whereas speed ratios of 1 to 100 are common between handpieces of different types (conventional, multiplying, turbine, etc.) the greatest power ratios are of the order of 1 to 5 with the higher powers developed by the low speed instruments. Comparison of power with cutting rates indicates that the high speed instruments utilize for cutting a higher percentage of the energy delivered to the cutting tool than do the low speed instruments. More of the energy is lost as heat in the low speed instruments.

4.7 Silicate Cements

The investigation of solubility and disintegration of silicate cements was completed on measuring solubility and disintegration of specimens stored in water that was changed after 24 hour periods. The solubility of both silicate specimens treated with R-23 and untreated specimens decreases on continued water exposure. This indicates that some of the soluble constituents are largely leached out during the first day.

A study of the effect on the physical properties of silicate cement of mixing the powder of one brand with the liquid of a different brand has been initiated. It has generally been recommended that different brands should not be combined, but specific data to support this recommendation have not been available.

4.8 Evaluation of Materials

Materials evaluated for the Federal dental services and the American Dental Association by specification and special test methods included amalgam alloys, denture base resins, chromium-cobalt casting alloys, hydrocolloidal impression materials, inlay casting investments, inlay casting gold alloys, mercuries, silicate cements and wrought gold wire alloys.

For the Director
by

W. T. Sweeney, Chief
Dental Research Section