NATIONAL BUREAU OF STANDARDS REPORT

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REPORT ON DENTAL RESEARCH
AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

January 1 to June 30, 1959

Dental Research Laboratory

This work is a part of the dental research program conducted at the National Bureau of Standards 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 materials and equipment used in restorative dentistry and on natural tooth structures continued at the National Bureau of Standards during the half year ending June 30, 1959.

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 6406	Compressive Properties of Hard Tooth Tissues and Restorative Materials.
NBS Report 6433	Mechanical Evaluation of Some High Speed Handpieces.
NBS Report 6464	Quantitative Analysis of Acrylic Copolymers by Gas Chromatography.
NBS Report 6506	Measurement of the Colors of Dental Silicate Cements.

3. PAPERS PUBLISHED

Determination of Some Compressive Properties of Human Enamel and Dentin. J. W. Stanford, G. C. Paffenbarger, J. W. Kumpula, and W. T. Sweeney. J.A.D.A. 57:487 Oct. 1958.

American Dental Association Specification No. 13 for Self-curing Repair Resins (First revision, approved December 1958, effective January 1, 1959). J.A.D.A. 58:136 March 1959.

Study of the Setting of Plaster. K. D. Jørgensen, A. S. Posner. J. D. Res. 38:491 May-June 1959.

Physical Research. G. C. Paffenbarger, and W. Souder. J.A.D.A. 58:97 June 1959.

Mechanism of Contraction in the Muscle Fibre - ATP System. L. Mandelkern, A. S. Posner, A. F. Diorio, and K. Laki. Proc. Nat. Acad. Sci. 45:814 June 1959.

4. WORK IN PROGRESS

4.1 Human Tooth Enamel and Dentin

(a) Fluorescence Studies.

In the study of the origin and significance of the fluorescence of human teeth, it became necessary to measure the activation and emission spectra of fluorescent extracts of enamel and dentin. Previous attempts to measure these spectra were hampered by the deterioration of sensitive components of the extracts when exposed to high-intensity ultraviolet radiation for thirty minutes as required by the spectroradiometer used. To obviate this difficulty a new rapid-scan spectrophotofluorometer has been purchased and is being calibrated. This instrument is capable of scanning and recording complete fluorescence activation and emission spectra within 10 to 60 seconds.

Study of the fluorescent material produced by salivary bacteria growing on various culture media was complicated by dietary effects. To control this factor, experiments are now being conducted with bacteria isolated from cavities of teeth from nine rats; each rat is fed one of nine different standard diets. This work has been undertaken in cooperation with the Naval Medical Research Institute.

A portable fluorometer for the measurement of the fluorescence intensity of human teeth in the mouth has been constructed and preliminary measurements have been made on volunteers. Clinical evaluation of the instrument is in progress.

(b) Crystallographic Studies.

The relationship between collagen and apatite is being investigated in cooperation with the Biology Department of the Massachusetts Institute of Technology. Samples of fish bone were studied for orientation and size of the apatite crystals using x-ray diffraction techniques.

Low angle x-ray diffraction studies were continued on polyethylene and other fibrous long chain polymers which, like collagen, show a long range periodicity. It has been found that by changing the crystallization conditions of polyethylene samples, the long range spacing changes. A new low angle camera of low resolution has been assembled. It is designed to supplement the low angle camera of high resolution already in use by giving quicker results for preliminary estimates of diffraction effects.

The study of the shrinkage of fibrous proteins, particularly muscle and keratin, and allied polymers has been continued. Wide angle x-ray diffraction techniques are being applied to study the crystallographic changes involved in the shrinkage of these materials by heat or chemical means.

A detailed study was started of the broadened x-ray diffraction maxima by use of a Fourier analysis of the broadened peaks. Data are being taken using the constant-count technique. A program for the IBM 704 electronic computor is being adapted for the necessary calculations. This method will lead to a better understanding of the size and the size distribution of the crystals in various hard tissues.

(c) Physical Property Studies.

The study of the compressive properties of enamel and dentin was completed. A detailed report of the results obtained is appended.

4.2 Metals

(a) Amalgam.

Setting Time.

Repeat determinations of setting time by the punch test of alloys aged for two years were completed. In addition, the effect of specimen thickness on the punch load required for shearing was studied by two methods. These studies led to the conclusion that it is shear stress not punch load which is constant at the carving limit. The previous report on the test method is being revised to include the effect of thickness, and a paper on aging effects is being prepared.

Effect of Alloy-Mercury Ratio on Amalgam.

The effect of the amount of mercury mixed with alloy (mercury/alloy ratio) on the physical properties of the condensed amalgam has been the subject of increasing interest to operative dentistry. A study of this problem has been initiated and data on strength, dimensional changes, and residual mercury are being obtained.

The results on one alloy (Caulk Micro Non-zinc) for different alloy/mercury ratios from 1/1 to 1/10 indicate that the compressive strength is not significatntly effected for relatively large

amounts of mercury in the mix but the residual mercury is higher with the higher ratios.

This study is being extended to include several alloys, as well as dimensional change on setting and the effect of different rates of loading on the strength values.

Effect of Alloy/Hg Ratio on Strength of Amalgam

Alloy/Hg Ratio	Compressive Strength		Residual Hg
	psi	St'd. Dev.	percent
1/1	29,600	700	48.4
1/1.4 (Mfgr. Rec.)	32,200	450	48.8
1/2	32,500	1150	49.8
1/3	32,900	700	50.4
1/4	33,500	1200	50.4
1/6	33,400	850	51.0
1/10	34,500	550	51.6

Tensile Properties.

A study of the tensile properties of amalgam was initiated. Molds to be used in making amalgam specimens and grips to hold the specimens during tensile tests were designed and constructed. Preliminary results gave values of 7,000 to 9,000 psi for the tensile strength of amalgam at one week. This is a property that has not been given sufficient consideration by researchers because of the difficulty of making satisfactory test specimens.

(b) Gold Alloys.

Gold Alloy Analysis.

When the method of analysis previously reported was used quantitatively with known amounts of metals, several difficulties

were encountered which lead to errors too large to be acceptable. Most of those difficulties have been corrected. Large amounts of platinum and/or palladium can now be separated from copper by keeping the Cu in the cupric state as $Cu(OH)_2$, a blue precipitate. The cupric state can be maintained simply by adding several grams of sodium chloride to the solution before precipitating the Cu.

The optimum pH for precipitating Pt and Pd with sodium formate is around 4 or 5. Higher pH's tend to inhibit precipitation. Precipitation is much more rapid on the acid side but the solution must be made alkaline to coagulate the precipitate.

There are still some inconsistencies in the precipitation of Pt. It is suspected that this may be due to the presence of small amounts of organic matter from filter paper.

4.3 Resins

(a) Denture Base Resins.

The clinical and laboratory study of different types of denture base resins and different processing procedures has continued. The dimensions of almost all the 150 artificial dentures reached equilibrium and are now being measured every six months. The single exception are those dentures with an epoxy resin base. Those dentures continue to expand so that an increase of 1% or more has been noted in most of them. The patients have not been aware of these gross changes showing that if they occur gradually the tissues make the necessary adaptation. Tests on the dimensional changes caused by wetting and drying and by release of strain imparted during processing are underway.

Data obtained so far on the thermal expansion of denture base resins show that the coefficients of thermal expansion over the range 15 to 70°C are between 75 and 100 x 10^{-6} per °C for most of the materials when conditioned at 50% relative humidity. A styrene material gave a value of approximately 65 x 10^{-6} and two methyl methacrylate materials containing glass fibers had values near 40 x 10^{-6}. When the specimens were conditioned in water at 68°C the coefficients were increased approximately 20% for most of the resins but a smaller increase was observed for materials containing glass fibers.

(b) Silica-Resin Direct Filling Material.

Additional physical property determinations were made on a silica-resin material formulated for possible use as a tooth restorative material. On indentation resistance tests, the silica-resin material had an indentation of 61 microns and a recovery of 73%. This compares favorably with average values of 104 microns and 73% for average acrylic filling materials. The Knoop surface hardness number was 38. Water sorption was 1.7% by weight and solubility was 0.19%. Color change was slight according to the American Dental Association Specification No. 12 test.

Fifty-five tensile strength specimens of various materials were prepared and tested after seven days in distilled water at 37°C. Four brands of silicate cement ranged from 500 to 1,000 PSI. Two brands of direct filling [poly (methyl methacrylate)] resin were 4,000 to 5,000 PSI. The experimental silica-resin material was 5,000 PSI; this is approximately the same as the value obtained for dentin.

(c) Polymerization Studies.

Further investigations were made to identify a possible molecular complex between dimethyl-aniline (DMA) and oxygen which may inhibit the reaction between dimethylaniline and diphenylpicrylhydrazyl at a pressure as low as one mm of mercury, but which is destroyed at 10^{-6} mm of mercury some evidence for the presence of this complex is the fact that DMA turns yellow in air, but the color disappears when the air is removed or when DMA is saturated with nitrogen. The studies have been temporarily discontinued.

(d) Gas Chromatography.

The quantitative determination of the composition of acrylic copolymers was completed. Ease, speed and accuracy make the modified technique using a heated coil surrounded by carrier gas for pyrolysis of the material most suitable for analysis of many copolymers. Statistical analysis of the results indicates that for most copolymer systems the components can be determined with a precision of 0.5%. Undoubtedly, the reliability of the standard calibration curve can be further improved by increasing the number of analysis of "standard polymers" in the preparation of the calibration curve. A report on this work is appended.

4.4 Color Standards

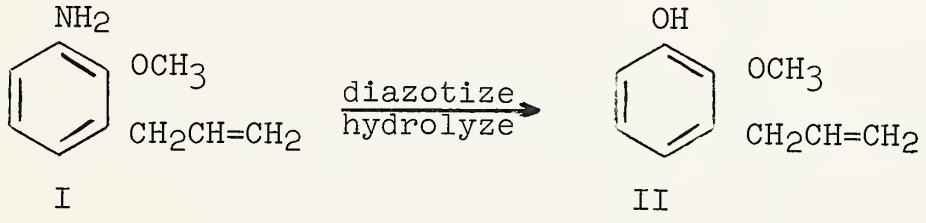
A report on the methods developed for determining and specifying the colors of silicate cements is appended.

4.5 Synthesis of Isomers of Eugenol

Two of the three isomers of eugenol capable of forming chelates have been synthesized in the pure state and the third isomer has been obtained in a mixture.

o-Eugenol was prepared in good yield by conventional Claissen rearrangement.

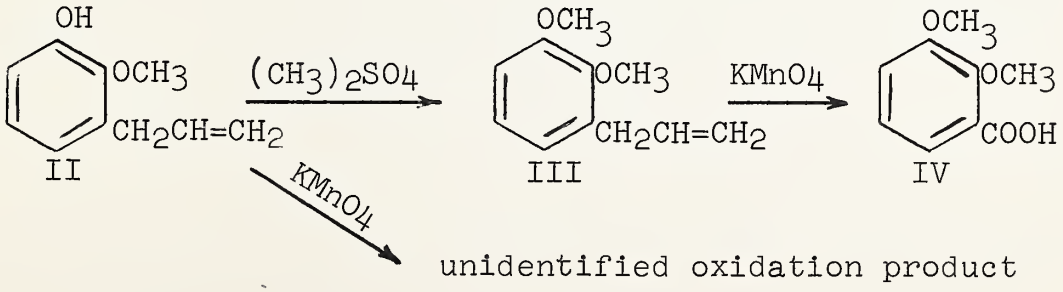
The synthesis of 2-methoxy-3-allylphenol outlined in the previous report was completed successfully. Compound I reported in the last report was diazotized and subsequently hydrolyzed to give II



I II

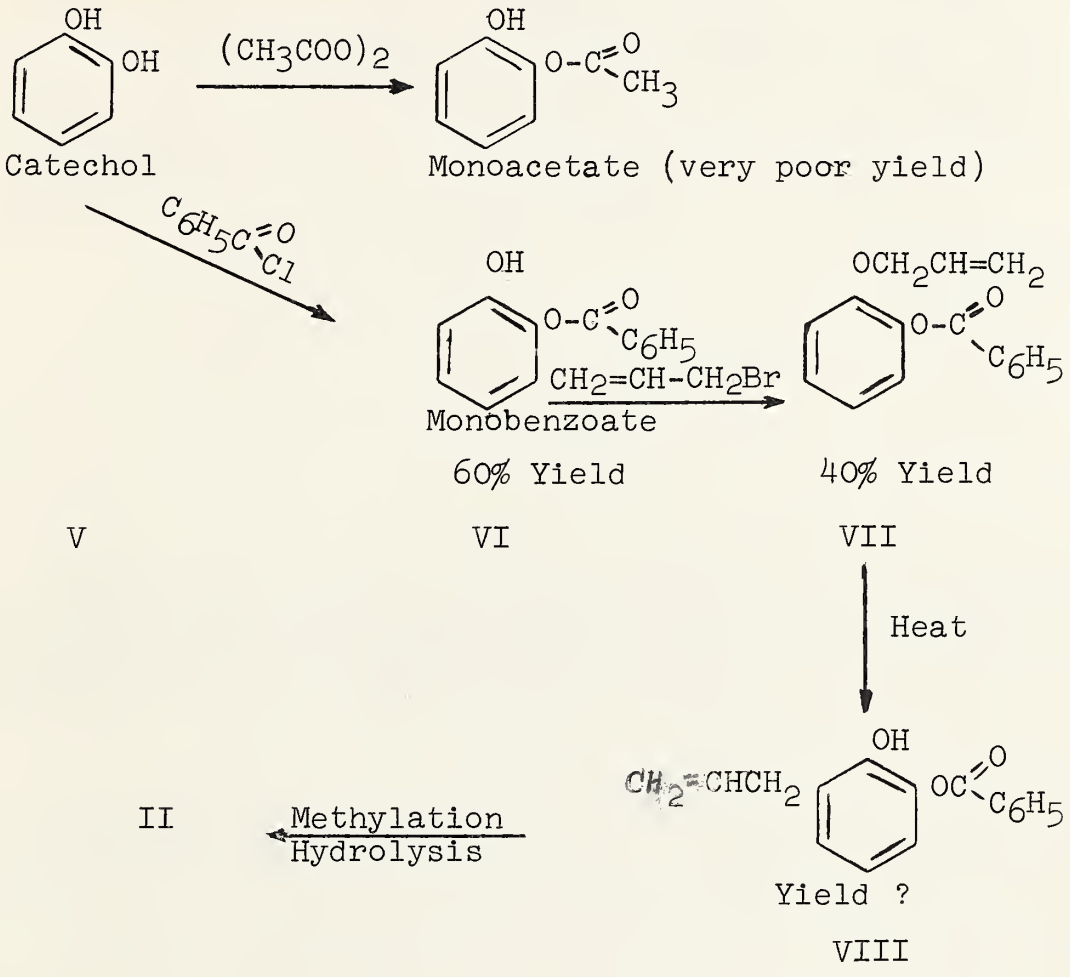
Overall yield was 37% II if a small quantity (5 g) I was used. The yield was reduced to 8% using larger amounts.

Identity of II was established from (1) its infra-red spectrum showing the presence of allyl and absence of vinyl groups (2) analysis for carbon and hydrogen and (3) conversion to 2,3 dimethoxybenzoic acid and mixed melting point with an authentic sample.



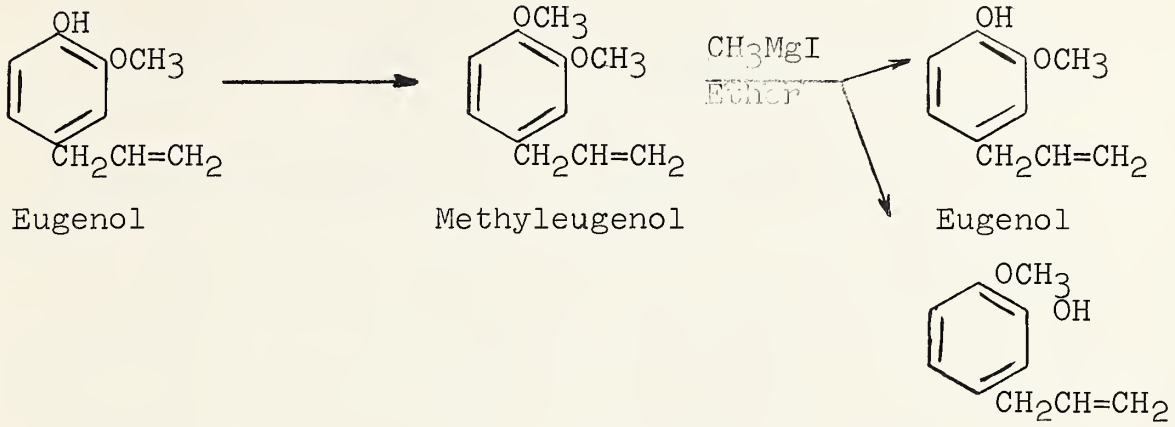
II III IV

The following alternate route to synthesize II was attempted in order to improve the yield and reduce the number of intermediate steps.



Difficulties were encountered in the rearrangement of VII to VIII. In two attempts an unidentified liquid and about 20% benzoic acid were obtained. Hence, some hydrolysis takes place under the conditions employed.

The third isomer chavibetol X was synthesized as reported in the literature:



Eugenol — Methyleugenol — Eugenol

90% Yield — Chavibetol

IX — X

Separation of the resulting eugenol-chavibetol mixture by precipitation of their potassium salts and formation of the benzoyl ester gave an impure chavibetol benzoate which melted 5-10°C below the reported melting point.

4.6 Cutting Instruments

The torque-speed measurements on air turbine handpieces were concluded and this phase of the program has been discontinued. Utilizing the torque-speed data obtained, an exploratory study was undertaken to investigate the relative effects of rotational speed, total power, and instrument design upon cutting efficiency. This work progressed far enough to demonstrate the feasibility of the method. A report on the mechanical evaluation of some high speed handpieces is appended.

4.7 Silicate Cements

Determinations of the setting times resulting from mixing one brand of silicate cement powder with a different brand of liquid were made. Five brands of cement all of which complied with the setting time requirements (3 to 8 minutes) of Federal and American Dental Association specifications were used. As indicated in the table below, 3 of the 25 possible combinations gave excessively long setting times.

Setting Times in Minutes of Combinations of Different Brands of Silicate Cement Powder and Liquid

Powder	Liquid				
	A	B	C	D	E
A	4	4	4	5	7
B	6	4	7	4	11
C	4	3	4	3	5
D	7	4	22	5	30
E	5	4	5	5	6

4.8 Evaluation of Materials

Materials and equipment evaluated for the Federal dental services and the American Dental Association by specification and special test methods included acrylic teeth, amalgams, denture base resins, hydrocolloidal impression materials, inlay casting gold alloys, mercuries, silicate cements, wrought gold wire alloys, zinc phosphate cements, and a mechanical device for removing mercury from amalgam mixes.

For the Director
by

W. T. Sweeney

W. T. Sweeney, Chief
Dental Research Section