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NATIONAL BUREAU OF STANDARDS REPORT

3960

REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

July 1 to December 31, 1954

Dental Research Laboratory

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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

NATIONAL BUREAU OF STANDARDS
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THE NATIONAL BUREAU OF STANDARDS

The scope of activities of the National Bureau of Standards is suggested in the following listing of the divisions and sections engaged in technical work. In general, each section is engaged in specialized research, development, and engineering in the field indicated by its title. A brief description of the activities, and of the resultant reports and publications, appears on the inside of the back cover of this report.

Electricity. Resistance and Reactance Measurements. Electrical Instruments. Magnetic Measurements. Electrochemistry.

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

Heat and Power. Temperature Measurements. Thermodynamics. Cryogenic Physics. Engines and Lubrication. Engine Fuels. Cryogenic Engineering.

Atomic and Radiation Physics. Spectroscopy. Radiometry. Mass Spectrometry. Solid State Physics. Electron Physics. Atomic Physics. Neutron Measurements. Infrared Spectroscopy. Nuclear Physics. Radioactivity. X-Ray. Betatron. Nucleonic Instrumentation. Radiological Equipment. Atomic Energy Commission Radiation Instruments Branch.

Chemistry. Organic Coatings. Surface Chemistry. Organic Chemistry. Analytical Chemistry. Inorganic Chemistry. Electrodeposition. Gas Chemistry. Physical Chemistry. Thermochemistry. Spectrochemistry. Pure Substances.

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

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

Metallurgy. Thermal Metallurgy. Chemical Metallurgy. Mechanical Metallurgy. Corrosion.

Mineral Products. Porcelain and Pottery. Glass. Refractories. Enameled Metals. Concreting Materials. Constitution and Microstructure.

Building Technology. Structural Engineering. Fire Protection. Heating and Air Conditioning. Floor, Roof, and Wall Coverings. Codes and Specifications.

Applied Mathematics. Numerical Analysis. Computation. Statistical Engineering.

Electronics. Engineering Electronics. Electron Tubes. Electronic Computers. Electronic Instrumentation. Process Technology.

Radio Propagation. Upper Atmosphere Research. Ionospheric Research. Regular Propagation Services. Frequency Utilization Research. Tropospheric Propagation Research. High Frequency Standards. Microwave Standards.

●Office of Basic Instrumentation ●Office of Weights and Measures.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT

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January 31, 1955

NBS REPORT

3960

REPORT ON DENTAL RESEARCH AT THE NATIONAL BUREAU OF STANDARDS

Progress Report

July 1 to December 31, 1954

Dental Research Laboratory

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.

(Task 7756-41 BV No.55-14)

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

REPORT ON DENTAL RESEARCH
AT THE NATIONAL BUREAU OF STANDARDS

1. INTRODUCTION

Investigations of materials and equipment used in dentistry and of natural tooth structures have been carried on in the Dental Research Section of the National Bureau of Standards during the half year ending December 31, 1954. Summaries of results obtained on work now in progress are given below. Copies of reports issued on phases of the work completed during this period are also included.

2. REPORTS ISSUED

NBS Report 3890	The Setting Reaction of Zinc Oxide and Eugenol.
NBS Report 3919	Revision of American Dental Association Specification No. 3 for Dental Impression Compound.
NBS Report 3955	Some Factors Influencing the Cutting Characteristics of Rotating Dental Cutting Instruments.
NBS Report 39~~66~~56	Use of Ionization Chambers in Obtaining Data on Radiation at Points Within the Head During Dental X-ray Exposure.

3. WORK IN PROGRESS

3.1 Structure of Human Tooth Enamel and Dentin

The depolarization fluorometer constructed for use in obtaining data on the molecular weight of fluorescing materials has been tested using fluorescein solutions and has provided data giving a value for the molecular weight of fluorescein within 10% of the formula weight. Experiments on extracts of enamel and dentin are now in progress but decay times must also be determined before weight values can be determined. In the meantime methods of improving the precision of the fluorometer are being investigated. By substitution of an ammonium dihydrogen phosphate crystal light modulator for the rotating polaroid discs now used, greatly increased precision may be obtained.

The crystallographic studies of tooth-like materials have been resumed. Synthetic calcium phosphates have been prepared for X-ray diffraction studies. Methods of preparing large crystals of non-stoichiometric apatites are being studied.

3.2 Determination of Contours of Dental Impressions, Models and Dentures

The method of measuring the contours of impressions, models and dentures using an instrument of the pantographic type has been further developed. Recent improvements of the instrument include the addition of an electronic device for determining the point of contact with elastic impression materials and a 100% humidity chamber to maintain the dimensional stability of agar and alginate impression materials during measurement. A statistical analysis of results obtained indicates that the standard deviation for the measurements is approximately 0.002 inch.

3.3 Low-Temperature Alloys

Attempts have been made to correlate the physical properties of gallium-copper-tin alloys with the microstructure of the original copper-tin alloy. However, much difficulty has been encountered in revealing the microstructure as the alloys are unusual and technics for use on them are not well defined.

Preliminary experiments indicate that an alloy made by combining gallium with a copper alloy containing 0.25% beryllium may have physical and chemical properties useful for dental applications.

Arrangements were made with the School of Aviation Medicine, Randolph Field, Texas, for determination of biologic response to an alloy of gallium and speculum metal (68T Cu - 32% Sn).

3.4 Resins

(a) Polymerization of Resins.

Setting times and physical behavior of polymers cured from monomer-polymer mixtures containing a series of tertiary amines in varying amine-benzoyl peroxide concentrations were determined by means of penetrometer tests and Rockwell superficial hardness measurements. Polymers cured with dimethyl-p-toluidine which is used in most commercial products gave satisfactory properties. Only a few other accelerators gave desirable polymerization products.

Another series of amine salts of p-toluene sulfinic acid were prepared. Three solid salts of aliphatic amines not previously reported in the literature were characterized by elementary analysis. Four solids gave sharp melting points but the elementary analysis deviated considerably from the theoretical values. Difficulties were encountered in purifying liquid salts of aliphatic and heterocyclic amine salts. Most of these salts are soluble in methyl methacrylate and act as polymerization accelerators. For aliphatic amine salts increased chain length reduces the accelerating efficiency. Salts of heterocyclic amines proved to be poor accelerators. Polymers cured with the N-methyl aniline salt of p-toluene sulfinic acid gave somewhat lower

indentation values as compared to salts prepared from p-toluene sulfinic acid. N-ethyl-bis-[p-tolysulfon-methyl]-amine which has been reported to be an excellent polymerization accelerator was prepared.

(b) Water Sorption of Acrylic Resins.

The depolymerization of methyl methacrylate polymers in boiling water was investigated. No degradation could be observed for low molecular weight sample (M.W. = 45,000 to 100,000). Specimens of 1,615,000 molecular weight showed a decrease in molecular weight to 800,000 on prolonged storage in water.

(c) Dental Resins.

Additonal data were obtained on the physical and chemical properties of dental resin cements and filling materials. Knoop hardness values of these materials ranged from 7 to 23 or from considerably below to slightly above the top value (approximately 20) usually reported for polymethyl methacrylate. Radio-opacity measurements indicated that some of the materials are less opaque than dentin and may therefore present a problem in dental X-ray diagnosis. Observation of the adhesion of the cements to abraded brass surfaces showed that none of the materials adhered after immersion for two weeks in water. Volumetric shrinkage of these materials on polymerization was found to vary from 5 to 12%.

Dimensional changes during storage in water were measured on resin cement specimens 0.5 mm thick by 50 mm in diameter. The specimens were kept at 80°C in air over night and then dried at room temperature until constant weight was established before immersion in water. Peak linear expansions ranging from 0.2 to 0.7% were obtained for most materials in approximately 3 days. After this period shrinkage was observed.

A study of the color of heat-cured dental resins has been started as part of a general study of methods for specifying the colors of dental materials. Measurements are being made with a Gardner automatic color difference meter. Methods are now being developed for preparation of uniform and reproducible specimens. Preliminary results show that some color differences of dental resins can be correlated with small differences in the monomer-polymer ratio used in making the specimens.

Work has been started on a study of the effects of pressure, temperature and time of cure on the molecular weight of polymethyl methacrylate made from a monomer-polymer mix. Preliminary investigations were made using a pressure of 200 lb per square inch and a temperature of 100°C. Viscosity measurements made on specimens cured for 2, 4 and 6 hours indicate that the molecular weights decrease after the 2-hour period.

3.5 Chromium-Cobalt Alloys

A small vacuum and controlled atmosphere furnace has been constructed for melting point determination of chromium-cobalt alloys and is now under test. Chemical compositions of the alloys are being determined. Data on these two properties will be used with the previously reported data on tensile properties and hardness in the preparation of a specification for the dental chromium-cobalt alloys.

3.6 High-Speed Handpieces

Methods for evaluation of "high-speed" handpieces are being developed. A testing bench has been constructed which will make it possible to test simultaneously five or six handpieces running at a constant speed under conditions approximating the normal use of the instruments in the mouth. Data will be obtained on heat rise, lateral play, run-out and backlash after various periods of operation.

3.7 Panographic X-ray Investigation

The construction of a prototype X-ray machine for taking panoramic dental X-ray exposures is almost completed. The machine is in process of assembly, and should be ready for trials in about 30 days. It is anticipated that a series of 100 or more patients will be radiographed with the device.

Measurement of the amount of ionizing radiation produced at points in and about the human head during panoramic dental radiography has been completed. These results would indicate that the panoramic device possesses a marked advantage over conventional dental radiography in that the amount of ionizing radiation to the patient during full-mouth examination is reduced by a factor of 50 in the areas receiving the heavier dosages.

3.8 X-ray Film Survey

The effect of aging on dental X-ray film is now being investigated. Density of film exposed and processed under standard conditions after 1, 3, 6 and 9 months storage will be determined. This work will bring to a close the X-ray film survey.

3.9 Motion Picture

Practically all of the photography for a motion picture on the testing of dental materials has been completed.

3.10 Porcelain Restorative Materials

Mixtures of high and low fusing porcelain powders were made in an attempt to reduce the large shrinkage which occurs in the firing of porcelain restorations. Although the reduction in shrinkage was very slight the method appeared to distribute the shrinkage more uniformly throughout the specimen. Investigations of the solubility of various types of porcelains are now being made.

3.11 Setting Time of Dental Amalgam

Work on the development of a simple test for the setting time of dental amalgam is in progress. Most promising results have been obtained from a system in which a thin disk of amalgam is placed in a die through which a punch can pass and penetrate the amalgam. The time at which the amalgam first supports the punch is considered to be the setting time.

3.12 Evaluation of Materials

Materials evaluated for the Federal Dental Services and the American Dental Association by specification or special test methods included amalgam alloys, chromium-cobalt alloys, denture base resins, gold casting alloys, handpieces, hydrocolloidal impression materials, inlay waxes, investments, mercuries, wrought gold wire and X-ray film.

For the Director,
by
W. T. Sweeney
W. T. Sweeney, Chief
Dental Research Section

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT **NBS REPORT**

0708-10-3824 January 11, 1955 3890

Progress Report

THE SETTING REACTION OF ZINC OXIDE AND EUGENOL

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** Chemist, Dental Research Section.
\# Chief, Dental Research Section.
° Research Associate, American Dental Association Research Fellowship, Dental Research Section.

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

The data in this report was presented in a thesis by Henry I. Copeland, Jr., in partial fulfillment of the requirements of the Graduate School, Georgetown University, Washington, D. C. for the degree of Master of Science.

(Task 7756-41 BV No. 55-14)

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U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

THE SETTING REACTION OF ZINC OXIDE AND EUGENOL

Abstract

Zinc oxide-eugenol mixtures have been used for a number of years in dentistry as temporary filling materials, for surgical cements and as impression pastes. In the present study the mechanism of setting of these mixtures was investigated. The setting rate is dependent on the water of hydration and particle size of the zinc oxide. X-ray diffraction data and electron micrographs give evidence that a crystalline compound is formed. Chemical analysis and molecular weight determinations show that this compound corresponds to the empirical formula $(C_{10}H_{11}O_2)_2Zn$, zinc eugenolate. Infrared data as well as the non-reactivity of meta and para substituted phenols as compared to the ortho isomers suggest that the zinc eugenolate is a chelate. The hardened mass resulting from mixes of zinc oxide and eugenol consists of zinc oxide embedded in a matrix of long sheath-like crystals of zinc eugenolate. The excess eugenol is sorbed by both the zinc eugenolate and the zinc oxide.

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1. INTRODUCTION

The zinc oxide-eugenol materials have been developed for utilization in a number of dental applications. They are most widely employed as palliative and anti-bacterial agents in the treatment of carious lesions [1]. With modifiers, these materials are used as impression pastes and as surgical cements.

Generally, the use of zinc oxide and eugenol depends on their tendency to form a hardened coherent mass when mixed. Unexplained variations in the behavior of the mixtures are important to the clinician who uses these materials to treat dental conditions. Of special concern are variations in setting rate, hardness, and strength of the set mass.

The mechanism of the setting process of zinc oxide and eugenol is unknown. Factors which have been reported to produce variations in the physical behavior are the type of zinc oxide used, the amount of water present (both in

the zinc oxide and atmosphere) and the ambient temperature. The following theories have been advanced in regard to the setting process: 1. The setting process is a purely physical one in which the constituents retain their individuality [2]. 2. A chemical reaction occurs and a compound resembling zinc phenolate is formed [3]. 3. The setting process is a combination of physical and chemical reactions [3].

The object of this investigation was to study the setting of zinc oxide and eugenol mixtures in order to more nearly define physical and chemical processes involved.

2. MATERIALS AND METHODS

Materials.

The zinc oxide (reagent grade) used had a particle size of around 3,000 Angstroms and contained less than 2% water. It was white. An experimental type of zinc oxide (Merck Hyperfine*) was also used. According to the manufacturer's specifications it had a particle size of 125-250 Angstroms and contained approximately 5% water. This zinc oxide was reddish in color.

The eugenol was USP grade. All other chemicals used were reagent grade.

Experiments.

Hardening.

Mixes of zinc oxide (reagent grade) were made with guaiacol, methyl guaiacol, eugenol, phenol, veratrole, safrol, anethole, m-methoxy phenol and thymol. The mixtures were made with a stellite spatula on a glass slab at room temperature. It was necessary to melt the phenol and thymol prior to mixing. The maximum amount of zinc oxide that could be wetted by the liquids was incorporated.

Samples of the zinc oxides were dehydrated by heating at 110°C for 24 hours. The dehydrated zinc oxides were mixed with eugenol in the same manner.

Heat of Reaction.

The change in temperature caused by the reaction was determined by means of a calibrated iron-constantan thermocouple connected to a Brown recording potentiometer. Zinc oxide (Hyperfine)-eugenol mixtures were used in this experiment.

* The authors wish to thank J. B. Conn of Merck and Co., Inc., for supplying the Hyperfine zinc oxide.

Analysis for Reaction Products.

Mixtures of 0.5 mole zinc oxide (reagent grade) and 0.1 mole eugenol were prepared. The hardened mass was pulverized by grinding in a mortar to a fine powder. The particles were extracted with chloroform for 30 hours in a Soxhlet extractor. The residue left in the filter thimble was vacuum dried and weighed. The chloroform was distilled off from the extract and the residue distilled over at 87 - 101°C and 2 mm Hg pressure.

Equimolar mixtures of zinc oxide (Hyperfine) and eugenol were prepared and extracted as above. The residue in the filter thimble was dried to constant weight at 110°C and 1-2 mm Hg pressure. The extract was filtered through a weighed Gooch crucible and the residue washed with three 10 ml portions each of chloroform, methanol and ether and dried to constant weight. The low boiling solvents used for extraction and washing were distilled off from the filtrate and the weight, refractive index and boiling point of the remaining liquid were determined.

The experiments were repeated using methanol, benzene, and chlorinated hydrocarbons other than chloroform as the extraction solvent.

Measurement of X-ray Diffraction.

The x-ray diffraction measurements were made by the section on Constitution and Microstructure at the National Bureau of Standards. The instrument used was a North American - Phillips Diffractometer utilizing a Geiger counter and a recording potentiometer. All measurements were made through 90 degrees. Patterns were obtained of zinc oxide (reagent grade), zinc oxide (Hyperfine), eugenol, mixtures of eugenol with each of the two zinc oxides and a mixture of zinc oxide (Hyperfine) with guaiacol. Patterns were also obtained of the purified reaction product and a zinc oxide-water mixture.

Elementary Analysis.

The purified reaction product was analyzed for carbon, hydrogen and zinc content using micro techniques.* The dry combustion method was used for the carbon and hydrogen determinations while the zinc was determined as the sulfate by digestion in the presence of sulfuric acid.

Infrared studies.

Zinc 8-quinolinolate was prepared according to Ztzsche [4]. Zinc guaiacolate was obtained on mixing guaiacol

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* Elementary analyses were performed by R. A. Paulson of the National Bureau of Standards.

with zinc oxide. The unreacted guaiacol was removed by repeated washing with cold chloroform. The infrared absorption spectra of 8-quinolinol, zinc 8-quinolinolate, eugenol, zinc eugenolate, guaiacol and zinc guaiacolate were measured. The solid samples were incorporated in potassium bromide pellets. All samples were examined with a Perkins-Elmer, Model 21 Spectrophotometer.*

Electron Micrographs.

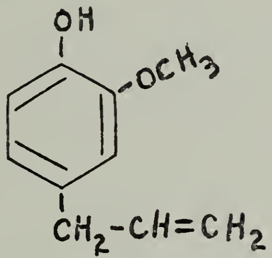
Electron micrographs of the Hyperfine zinc oxide-eugenol mixture and the purified reaction product were made at 10,000 and 4,000 diameter magnifications respectively.**

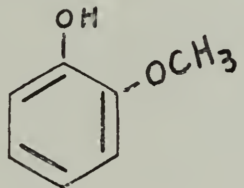
Water Sorption Study.

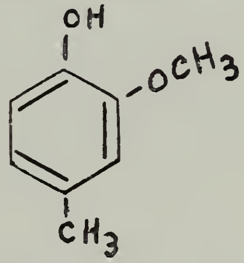
Dried zinc eugenolate samples were placed in a desiccator over a saturated potassium nitrate solution (93.3% relative humidity at 30°C).

3. RESULTS AND DISCUSSION

The mixtures of zinc oxide (reagent grade) with eugenol, guaiacol or methyl guaiacol set into a hardened mass.

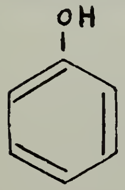


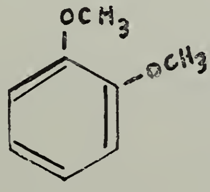


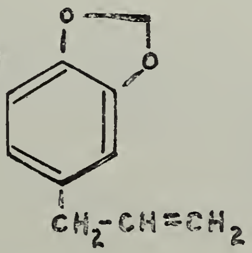


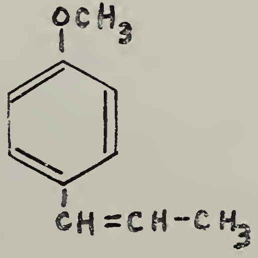
These aromatic oils have a methoxy group ortho to a phenolic hydroxy group. Guaiacol has no substituent group in the para position. Methyl guaiacol has a methyl group as a para substituent. Eugenol has a para beta allyl group.

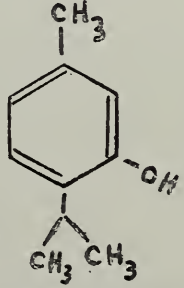
Mixtures of zinc oxide with phenol itself, veratrole, safrol, anethole, thymol, and m-methoxy phenol, did not harden.

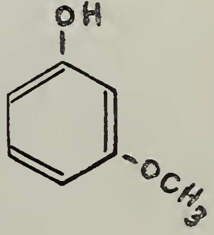












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* Infrared absorption spectra were made by J. E. Stewart and G. Wyman of the National Bureau of Standards.

** Electron micrographs were made with F. A. Heckman of the National Bureau of Standards.

These compounds do not have a methoxy group ortho to a hydroxy group.

From the above considerations, it appears that the para groups are not involved in the setting reaction, and the ortho methoxy group is essential for the hardening to take place.

The dehydrated zinc oxides did not form a hardened mass when mixed with eugenol. The untreated reagent grade zinc oxide (less than 2% water) required more than 24 hours to harden when mixed with eugenol. The Hyperfine zinc oxide-eugenol mixture hardened in less than 15 minutes. This zinc oxide had the greater water content (5% according to manufacturer) and also had a much larger surface area. In all experiments, the setting rate of either of the zinc oxides mixed with eugenol, was more rapid in warm and humid weather than when the relative humidity was low.

The hardening process was found to be exothermic in nature. The temperature rise was directly related to the rate of hardening and to the ultimate hardness of the product; e.g., rapidly setting mixtures gave a harder product and a greater temperature rise.

On extraction with chloroform the reagent grade zinc oxide-eugenol mixture yielded approximately 80% eugenol. The residue in the filter thimble showed a 10% increase in weight over the original weight of the zinc oxide, and analysis indicated that some eugenol was still present in the residue. An insoluble film deposited in the extractor flask suggested some chemical change had taken place.

Since the zinc oxide (Hyperfine) - eugenol mixture had demonstrated faster setting and increased exothermic activity, the extractions were repeated using this mixture. Table 1 gives the composition of a typical mixture of Hyperfine zinc oxide and eugenol.

Table 1. Composition of a Zinc Oxide-Eugenol Mixture.

A. Before Setting:

Zinc oxide (containing 1.3% water)	9.60 g	0.117 moles
Weight of dry zinc oxide	9.48 g.	
Eugenol	19.26 g.	0.117 moles
Material in mixture	28.74 g.	

B. After Setting:

Residue on extraction	15.5480 g.	
Residue in extract (other than eugenol) ..	1.1909 g.	
Eugenol recovered	11.6676 g.	. 65.2%
Material accounted for	28.4065 g.	. 98.9%

After the chloroform was removed from the extract, the residue soon formed an oily paste. This paste was composed of a white crystalline material and eugenol.

X-ray diffraction measurements were made to determine changes in crystal structure which might have occurred during setting. Both reagent grade zinc oxide and the Hyperfine zinc oxide gave the characteristic pattern indicating principal spacings of approximately 2.9, 2.6, and 2.5 Angstroms. The small particle size of the Hyperfine zinc oxide caused broadening of the bases of the peaks in its pattern. Since there were no other differences in the diffraction pattern, the manufacturer's suggestion that the reddish color of the Hyperfine was due to its small particle size appears reasonable. Eugenol showed no peaks, being non-crystalline.

The mixture of reagent grade zinc oxide with eugenol showed peaks indicating the presence of a new crystal form with spacing of approximately 17.0, 8.8, and 7.0 Angstroms. The zinc oxide (Hyperfine) mixed with eugenol demonstrated the presence of the same crystal form with approximately the same spacing. The peaks were much higher in the pattern of the latter mixture which indicated the presence of a much greater quantity of the new crystal form. Zinc oxide mixed with guaiacol demonstrated the presence of a new crystal form with spacings of 12.0, 9.3, and 8.7 Angstroms. The zinc oxide-water mixture gave only the characteristic zinc oxide pattern.

The diffraction pattern of the purified reaction product showed only the presence of the new crystal form although as little as 2% of zinc oxide could have been detected by the diffractometer used.

Representative x-ray diffraction patterns are shown in Figures 1 and 2. The peaks between 30 and 82 degrees have been identified as belonging to zinc oxide [5]. The peaks occurring between 6 and 26 degrees are the result of chemical reaction. The reaction product is identified as zinc eugenolate in pattern No. 1 of Figure 2.

The anlaysis of the compound formed by the reaction of Hyperfine zinc oxide and eugenol is given in Table 2. As a compound resembling zinc phenolate had been suggested[3] as a possible reaction product, the composition of zinc eugenolate was calculated from the empirical formula and compared with the values obtained for the new crystalline compound. The comparison indicated that the formula for the compound would be that of zinc eugenolate, $(C_{10}H_{11}O_2)_2Zn$.

Table 2. Analysis of Reaction Product

	CALC.	FOUND
Carbon	61.34%	59.6%*
Hydrogen	5.61%	5.7%
Zinc	16.72%	17.1%*
Oxygen (by difference)	16.33%	17.6%*
Molecular weight**	391	435

*The low carbon as well as the high zinc and oxygen percentages would be explained by the presence of a small amount of zinc oxide in the purified product.

The molecular weight was determined cryoscopically in acetamide (molar freezing point depression Kf equals 36.3°C).

Some physical properties of the zinc eugenolate are given in Table 3.

Table 3. Some Physical Properties of Zinc Eugenolate

M.P. (Dissociation Temperature)	245°C.
Crystal Form (Electron micrograph*, Fig. 4)	Monoclinic
Refractive Index (n_D^{25})	1.69
Density	1.6 g/ml
Solubility	v.sl.s. hot chloroform; sol. in ammonium hydroxide, pyridine, hot acetamide, and hot butyrolactone.
Fluorescence** (excited by 3650 A° radiation)	White

*Figure 3 shows an electron micrograph of a zinc oxide-eugenol mixture.

**The presence of zinc eugenolate may be readily detected by means of the characteristic white fluorescence emitted by this compound when excited by ultraviolet radiation. When similiarly excited, zinc oxide and eugenol fluoresce bright yellow and dark brown, respectively. A mixture of zinc oxide and eugenol fluoresces deep red.

Zinc eugenolate was found to be insoluble in all common organic solvents including anisole, mesityl oxide, α - chloronaphthalene, dioxanone, diethyl carbamate, ethylene carbonate, diethyl phosphite, dimethyl amido phosphate, tributyl amine and dimethyl formamide.

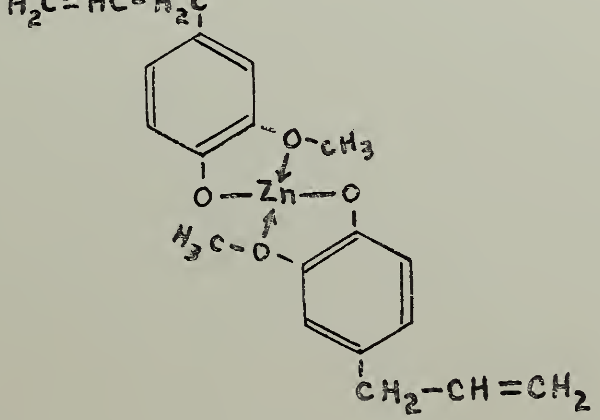
Infrared absorption spectra were studied in an attempt to gain more information on the molecular structure of zinc eugenolate. Zinc 8-quinolinolate was used for comparison because an x-ray analysis of its structure had been reported [6]. For further comparison, guaiacol and the zinc oxide - guaiacol reaction product were used.

Interesting changes in infrared absorption occurred, as shown in Figure 5. In the 7.1 to 7.4 micron area, the absorption due to the hydroxy aromatic group of 8-quinolinol, eugenol, and guaiacol was not evident in their zinc derivatives. The disappearance of this band is due to the replacement of the phenolic hydrogen by zinc. At 7.5 microns, absorption was observed in all three zinc compounds whereas none was present in the original hydroxy aromatics. This absorption has been attributed to the appearance of bond bending strain.

An absorption band at 6.08 microns due to the vinyl group was present in both eugenol and its zinc derivative spectra. This was evidence that the para substituent did not take part in the reaction.

The apparent inability of the setting reaction to take place in the absence of the o-methoxy group, the insolubility of the reaction product, and the infrared absorption changes suggest that zinc eugenolate is related to the metallo-pyrocatechol complexes or chelates[7].

Zinc is a bivalent element with a common coordination number of four. As such, zinc is a metal likely to form a chelate. Eugenol, guaiacol and methyl guaiacol have a replaceable hydrogen and a nearby donor in the oxygen of the o-methoxy group. The chelate formed would have a proportion of two eugenol molecules to one zinc. It may be represented as follows:



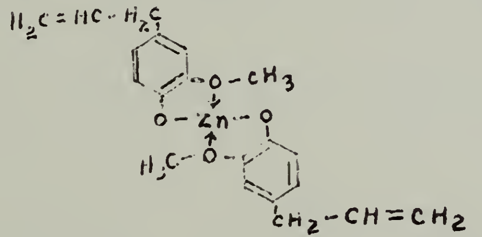
SUMMARY

1. The setting of mixtures of zinc oxide and eugenol involves both a physical and a chemical process.

2. X-ray diffraction data and electron photomicrographs give evidence that a crystalline compound is formed by the chemical reaction.

3. Chemical analysis and molecular weight determinations show that this crystalline compound corresponds to the empirical formula $(C_{10}H_{11}O_2)_2Zn$, zinc eugenolate.

4. Infrared absorption data suggest that zinc eugenolate is a chelate compound with the following structure:



5. The hardened mass resulting from mixes of zinc oxide and eugenol consists of zinc oxide embedded in a matrix of long, sheath-like crystals of zinc eugenolate. The excess eugenol is sorbed by both the zinc eugenolate and the zinc oxide.

The authors wish to specially acknowledge the generous assistance given by H. Swanson and G. Ugrinic of the Microstructure Section, National Bureau of Standards, in performing x-ray diffraction measurements.

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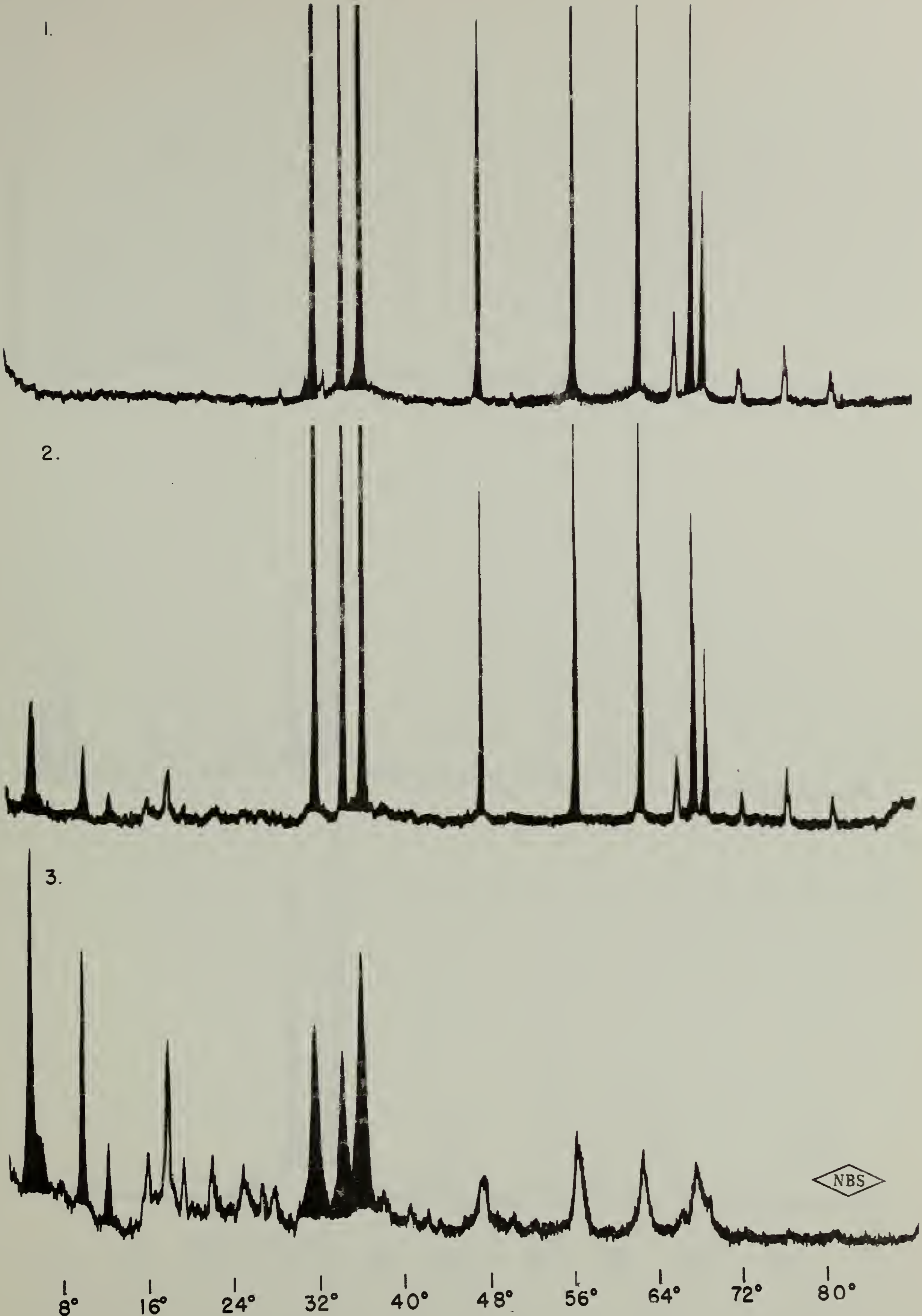

Figure 1. X-ray Diffraction Patterns. 1. ZnO (reag.). 2. ZnO (reag.)-Eug. mixture. 3. ZnO (Hpf.)-Eug. mixture.

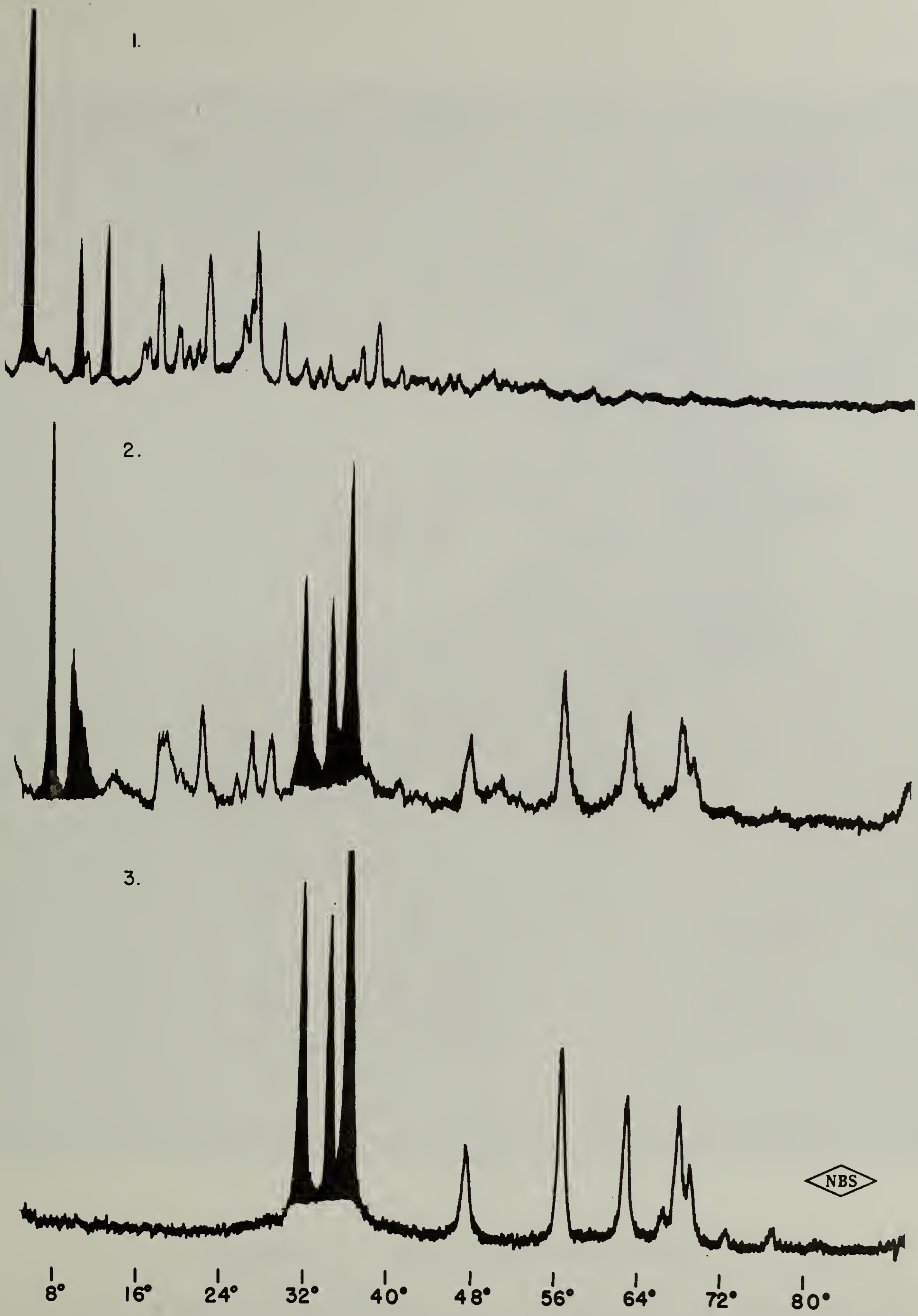

Figure 2. X-ray Diffraction Patterns. 1. Zinc eugenolate.
2. ZnO-Guaiacol mixture. 3. ZnO-Water mixture.

Figure 3. ELECTRON MICROGRAPH

An electron micrograph of a zinc oxide-eugenol mixture showing the small, rounded particles of zinc oxide and the thin, elongated crystals of zinc eugenolate. The sheaf-like aggregation is typical of the unextracted hardened mass.

Figure 4. ELECTRON MICROGRAPH

An electron micrograph of zinc eugenolate, palladium shadowed 4:1, showing the crystal habit typical of the extracted reaction product. The small, rounded particles seen in the background and on the surfaces of the large, crystalline formations are probably zinc oxide.

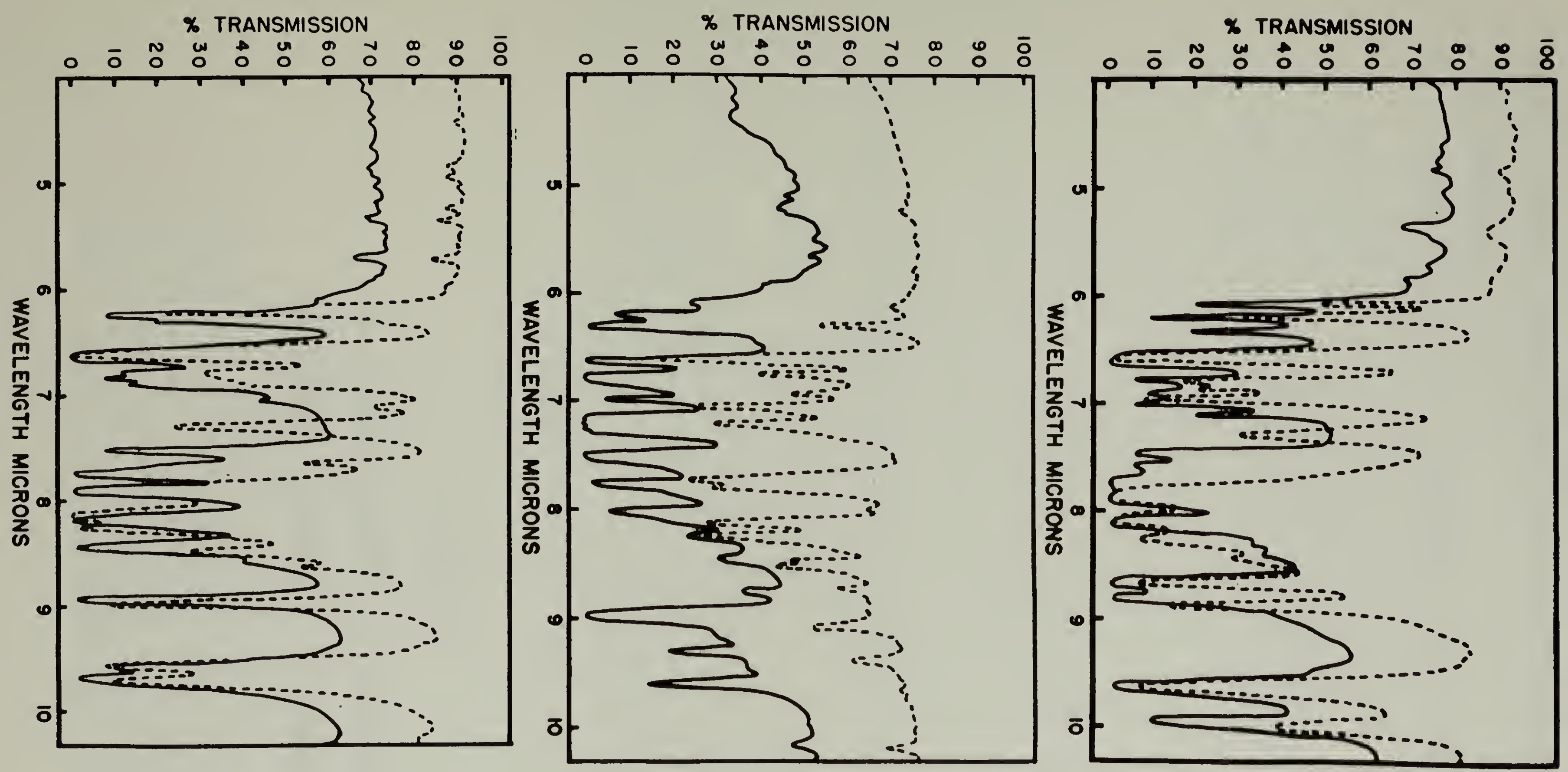

Figure 5. INFRA-RED ABSORPTION SPECTRA

In each case the broken line is the hydroxy aromatic and the solid line is the zinc derivative. TOP. Eugenol and zinc eugenolate. MIDDLE. 8-quinolinol and zinc quinolinolate. BOTTOM. Guaiacol and zinc guaiacolate.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT		NBS REPORT
0708-20-3824	December 31, 1954	3919

Progress Report

A Revision of AMERICAN DENTAL ASSOCIATION SPECIFICATION NO. 3 FOR DENTAL IMPRESSION COMPOUND

by

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

(Task 7756-41 BV No. 55-14)

NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

A Revision of
AMERICAN DENTAL ASSOCIATION
SPECIFICATION NO. 3 FOR DENTAL IMPRESSION COMPOUND

Abstract

A revision of American Dental Association Specification No. 3 for Dental Impression Compound is proposed in order to simplify testing procedures and to obtain better reproducibility of flow test results. The revision is based on experience derived from a recent survey of certified dental impression compounds and a data exchange program with three of the manufacturers of certified compounds. Two major changes were made in the detail requirements. The requirement for flow at the 40.0°C (104.0°F) flow testing temperature was deleted. Also, the requirement for flow at the 50.0°C (122.0°F) flow testing temperature for tray compound was replaced with a 45.0°C (113.0°F) testing temperature. Precise methods for preparation and testing the flow specimen are incorporated in the revised specification.

Foreward

The American Dental Association revises its specifications for dental materials whenever new or improved test methods and materials become available. To promote these revisions, the Council on Dental Research of the American Dental Association seeks the help of the Dental Materials Group of the International Association for Dental Research. The aid given by the Specifications Committee of this Group is reflected in the following current revision of American Dental Association

Specification No. 3 for Dental Impression Compound and in the background work involved in the revision. The authors wish to express their appreciation for the cooperation given them in this revision by the following manufacturers: Mizzy, Inc., The Ransom and Randolph Co., The S. S. White Dental Manufacturing Co.

1. INTRODUCTION

Since 1931 when the A.D.A. specification for impression compound[1] was first formulated, there have been no significant changes in the composition of the compounds. The first revision[2] of the specification and this, the second revision, were occasioned primarily by refinements in the test procedure for determining flow. In fact, with the exception of the requirement for flow, the detail requirements of the specification remain unchanged. The format of the specification has been changed to conform more nearly to the form[3] for Federal specifications.

When products on the List of Certified Dental Materials[4] are surveyed for compliance with the respective specifications and are found not to comply, an exchange of test data is made between the Dental Research Section at the National Bureau of Standards and the particular manufacturer. Such was the case with one manufacturer when impression compounds were retested. The exchange of data showed that differences existed between results obtained at the National Bureau of Standards and those obtained in the manufacturer's laboratory. These differences were thought to be due to one or more of the following factors: (1) differences in lots or batches of compound; (2) differences in methods of preparing specimens; and (3) differences in test procedures.

An extensive data exchange program, enlarged to include three manufacturers, was therefore set up to determine whether or not the foregoing factors were responsible for the differences. Also an independent investigation of the effect of variations in the preparation of specimens and in test procedures was begun in the Dental Research Section at the National Bureau of Standards.

2. EXPERIMENTAL PROCEDURE AND RESULTS

2.1 Exchange program.

The data exchange program was set up as outlined in Table 1. This scheme presents a method of sorting the variables with a minimum number of tests. For Result A the impression compound was supplied by the manufacturer and the specimens were prepared and tested by him. Result A differed from Result B only in that Result B was obtained from tests in the laboratories of the National Bureau of Standards. Therefore, any difference between Results A and B should seemingly be caused by the test procedure. Likewise, Result A should differ from Result C and Result B should differ from Result D only because of differences in preparing the specimens. The entire test procedure was duplicated on impression compound obtained in the open market (Results E, F, G and H in Table 1).

The results obtained by following the outline in Table 1 with three manufacturers, X, Y and Z, are shown in Tables 2 through 5. In the case of manufacturer Z, the upper half of the outlined plan (Table 1) or Results A through D were obtained on a different lot of material than that available at the National Bureau of Standards. These results appear in Table 3.

Manufacturer Y also did not have the same lot available for testing. The greatest difference in flow results between laboratories appeared in this part of the exchange program, the data of which are in Table 4. An additional exchange of specimens and data was made with manufacturer Y for Results A through D; however in this case, revised methods for preparation and testing the flow specimens were used. These data are given in Table 5.

In Table 2 where the results of interchange of data with manufacturer X are given, there is fair agreement for results obtained at 37.0°C (98.6°F). A flow of one percent amounts to only 60 microns on a 6 mm high specimen. Thus the difference between the highest values (Result F), 3.8 percent, and the lowest (Results A or H), 1.9 percent, represents a difference in flow of 114 microns and the average deviation on Results A through H was only ± 1.2 percent or ± 72 microns. The agreement at 45.0°C (113.0°F) is almost as good, the average deviation being ± 1.7 percent (± 102 microns). At 40.0°C (104.0°F) the critical flow temperature, the poor agreement between Results E and G and between F and H indicates that differences in preparing the specimens was largely the cause.

Examination of Table 3 (Results E, G and H) showing the exchange of data with manufacturer Z, indicates that the agreement is good. Result F at 40.0°C (104.0°F) is most out of line. When Result E is compared with Result F and Result G with Result H, one sees that the test method used at the National Bureau of Standards usually gave higher values than those from the manufacturer. Also, the specimens made at the National

Bureau of Standards gave higher values for Result F than those made by the manufacturer when tested by both groups (Result F compared with Result H). However, when Result E is compared with Result G the values of flow are reversed. It would seem that the method of preparing the specimens was involved also.

By reference to Results E through H in Table 3, it will be noted that the average flow of 9.8 percent for this compound at 40.0°C (104.0°F) prevented it from complying with the American Dental Association specification[2] which requires a minimum flow of 20 percent. Moreover, the compound would not pass the impression test in the current specification.[2] Since the manufacturer had none of the batch that was available at the Bureau, and as the sample at the Bureau was too small to permit complete testing by both laboratories, the exchange was made using a batch of compound furnished by the manufacturer (upper half of Table 3, Results A through D). A comparison of these data with those of Results E through H indicates a difference at 37.0°C and 40.0°C between the two batches. These data show fair agreement and much of the discussion of Results E through H applies. The data also show that this batch complies with the A.D.A. specification[2].

The foregoing results indicate that the method of preparation of flow specimens should be described more specifically and that test procedures should be revised to minimize differences between laboratories. In section 4.3.1.1 of the proposed revision of the specification (which follows) detailed directions for heating the compound, forming the specimens and removal of the specimens from the mold have been included.

2.2 Investigation of test procedures.

The most important factors in the flow test procedure appear to be: 1) the age of the specimen at the time of testing; 2) slight temperature variations at the critical 40.0°C (104.0°F) flow temperature; and 3) the chilling of the top of the specimen by the metal platen of the apparatus.

The effect of the age of a specimen upon its flow at 37.0°C (98.6°F) under the conditions of test (4.3.1.2)* is shown in Figure 1. The specimens were stored in air at 20-25°C (68-77°F). Tests conducted on specimens ½ hour, 2 hours, 6 hours, 24 hours, and 1 week old showed that the flow of specimens 24 hours old or older did not vary significantly. As the data in Figure 1 are typical of the currently available compounds, there seems to be no reason for extending the age of the specimens used in the test for flow beyond the 24 hour value in the present specification[2]. It may be that the decrease in flow with time is caused by the release of internal strain as the specimens age. This was suggested by data obtained several years ago when the flow of specimens molded from a compound softened in the conventional manner was compared with the flow of specimens made by pouring the melted compound into the mold. It appeared that the incorporation of strain in the molded specimens resulted in greater flow.

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* Numbers refer to sections of the revised specification hereinafter printed.

As it was not possible to control how much strain was placed in a specimen, the flow values on specimens made from softened compound were not in reasonable agreement.

The effect of slight temperature variations upon the amount of flow at the critical 40.0°C (104.0°F) flow temperature is shown by the steepness of the slope of the flow curves in Figure 2. Here it will be noted that practically 70 percent flow occurred between 38.0°C (100.4°F) and 42.0°C (107.6°F) and at or near 40.0°C (104.0°F) a change of 0.1°C (0.18°F) in the temperature at which the compound was tested could make approximately a 2 percent difference in the flow values.

Therefore, it was decided to delete the requirement specifying a determination for the flow at 40.0°C (104.0°F). Furthermore, there apparently is no reason why the requirement of flow at 37.0°C (98.6°F) and at 45.0°C (113.0°F) will not be sufficient to define a satisfactory compound.

The third major consideration was the chilling of the top of the flow specimen by the metal platen of the test apparatus (C in Figure 3-3). The truncated cone-shaped specimen resulting from the flow test (A in Fig. 3) indicates that the temperature of the top of the specimen was not as high as the temperature of the bottom. A symmetrical barrel-shaped flow specimen (B in Fig. 3) shows the effect of testing a specimen which was apparently nearly uniform in temperature throughout. To produce this type of flow specimen it was necessary to reduce the conduction of heat from the specimen through the platen and its shaft. A hard rubber shaft (B in Fig. 3-3) having a low thermal

conduction was, therefore, substituted for a metallic shaft having a high thermal conduction. Thus the temperature of the platen, which is submerged in a constant temperature bath, is maintained sufficiently close to the temperature of the bath to cause the formation of symmetrically shaped specimens during the test for flow.

In addition to the change in shaft, the diameter of the platen (C in Fig. 3-3) was increased from ½ inch to 2 inches. This was necessary since the compound at 45.0°C (113.0°F) flowed out under the ½ inch platen and up its side.

2.3 Results obtained with revised procedures.

To determine the effectiveness of the new specimen preparation and test procedure, an additional series of tests were made in cooperation with manufacturer Y using the methods outlined in paragraphs 4.3.1.1 and 4.3.1.2 of the proposed revision of the specification. A comparison of the results shown in Tables 4 and 5 indicates that with the revised procedure better uniformity results both in specimen preparation and in test procedure in every instance with the exception of the data showing differences due to preparation of specimens for the 37°C (98.6°F) flow tests. At this temperature the effect of specimen preparation was not large under either the original or revised procedure. It is possible that the better agreement shown in Table 5 as compared to Table 4 may be affected by the fact that material of different batches was used for the two series of tests. The general agreement between the average values shown in Table 4 and in Table 5 indicates, however, that the difference between the two batches of material is small.

American Dental Association
SPECIFICATION NO. 3 FOR DENTAL IMPRESSION COMPOUND

Second Revision
(To be effective July 1, 1955)

1. SCOPE AND CLASSIFICATION

1.1 Scope. This specification is for dental impression compound, sometimes called modeling compound. This compound is thermally reversible, rigid when cold and plastic when heated a few degrees above mouth temperature.

1.2 Classification.

1.2.1 Types. Dental impression compound covered by this specification shall be of the following types as specified:

Type I. Impression Compound

Type II. Tray Compound

2. APPLICABLE SPECIFICATIONS

2.1 Specification. There are no other specifications applicable to this specification. (Copies of American Dental Association specifications may be obtained free upon application to the American Dental Association Research Fellowship, National Bureau of Standards, Washington 25, D. C.)

3. REQUIREMENTS

3.1 Material.

3.1.1 Type I. The material shall be suitable for taking impressions in the oral cavity.

3.1.2 Type II. The material shall be suitable for forming trays to be used in the oral cavity.

3.2 General. The compound shall have the following properties:

3.2.1 Homogeneity throughout.

3.2.2 A smooth, glossy surface after flaming.

3.2.3 Firm, smooth margins after trimming with a knife at room temperature.

3.3 Detail

3.3.1 Flow. Type I Compound

3.3.1.1 The flow at 37.0°C (98.6°F) shall not be more than 6.0 percent.

3.3.1.2 The flow at 45.0°C (113.0°F) shall not be less than 85.0 percent.

3.3.2 Flow. Type II Compound

3.3.2.1 The flow at 37.0°C (98.6°F) shall not be more than 2.0 percent.

3.3.2.2 The flow at 45.0°C (113.0°F) shall not be less than 70.0 percent, nor more than 85.0 percent.

3.3.3 Impression Test

3.3.3.1 Type I compound shall record accurately the surface detail of the test block (Fig. 1-3) referred to in 4.3.2.2 at 45.0°C (113.0°F).

3.3.4 Manufacturer's Instructions. Instructions, including method of softening, working temperature, and a curve or data showing the shrinkage of the compound from 40°C (104°F) to 20°C (68°F) shall be supplied with each package.

4. SAMPLING, INSPECTION AND TESTING PROCEDURES

4.1 Sampling. Three packages or approximately one and one-half pounds of compound shall be procured at retail by a member of the American Dental Association. This sample shall be forwarded in the original unopened package or packages to the American Dental Association Research Fellowship at the National Bureau of Standards, Washington 25, D. C.

4.2 Inspection. Visual inspection shall be used in determining compliance with the requirements outlined in 3.1, 3.2, 3.3.4, 5.1 and 5.2.

4.3 Physical Tests.

4.3.1 Flow

4.3.1.1 Preparation of specimens. A cake or cakes of compound shall be broken into pieces and placed in an iron crucible of 50 ml capacity. The crucible shall be placed 130 mm (5.1 in.) below a 250 watt infrared lamp bulb. The stirred compound shall be heated to a temperature of 75 ± 5°C (167 ± 9°F) (checked with a thermometer) until melted throughout and then shall be poured into a mold (Fig. 2-3). The mold shall be a flat, stainless steel metal plate 6.0 mm (0.236 in.) thick, containing four holes 10.0 mm (0.394 in.) in diameter. The mold shall be preheated to a temperature of 55 ± 5°C (131 ± 9°F) and placed on a smooth glass slab /152 mm long, 76 mm wide, 19 mm thick (6 in., 3 in., and 0.75 in.)/ preheated to the same temperature. After the mold has been over-filled with material, a smooth flat tinfoil-covered glass plate preheated to 55 ± 5°C (131 ± 9°F) shall be placed on

top of the mold. A load of 9,000 g (20 lb) shall be applied to the top of the tinfoiled glass plate for ten minutes. Then the mold shall be placed in water at a temperature of 10°C (50°F) for ten minutes. The upper tinfoiled plate shall then be removed and the excess material shall be trimmed away. This can be accomplished by using a metal scraper with the top surface of the mold as a guide. The mold may then be removed from the glass slab by gently tapping the side of the mold. The specimens shall be removed from the mold by chilling in water for ten minutes at 10°C (50°F) and shall be stored at room temperature 20-25°C (68-77°F) for 24 hours before testing.

4.3.1.2 Method of test. The original length of the specimen shall be determined using a metric micrometer caliper. Four measurements shall be made around the circumference and one measurement shall be made on the center of the specimen. The measurements shall be recorded to the nearest 0.005 mm. The specimen and flow testing instrument (Fig. 3-3) shall be placed in a water bath and held at the testing temperature for twenty minutes prior to testing. The flow testing instrument as illustrated consists of the following: A- a weight, B- a non-conducting shaft, and C- a brass platen. The total weight in air of these three components is 2,000 g (4.41 lb). The weight A shall be separated a minimum of 76 mm (3 in.) from the brass platen on the shaft. This shaft shall be of hard rubber or of a similar poor thermal conductor to avoid loss in heat due to conduction. The diameter of the brass platen which touches the specimen shall not be less than 51 mm (2 in.). The thickness of the brass platen shall not exceed 6.5 mm (0.25 in.).

The temperature in the bath shall be controlled to within ± 0.1°C (± 0.2°F) of the required temperature. (A calibrated thermometer shall be used for determining the temperature.) A thin sheet of waterproof cellophane shall be placed between the instrument and each end of the specimen. The bottom of the specimen shall be 51 mm (2 in.) below the surface of the water in the bath. A constant axial load of 2,000 g (4.4 lb) shall then be applied to the specimen for ten minutes, after which the specimen shall be removed and cooled in air to room temperature. The cellophane shall be stripped off and the final length shall be determined as is the original length. The flow, as evidenced by the change in length, shall be reported as percentage of the original length. The value for flow at any temperature shall be the average value for two specimens and shall be reported to the nearest 0.1 percent.

4.3.2 Impression test.

4.3.2.1 Preparation of impression specimen. The impression specimen shall be a disk 40 mm (1.57 in.) in diameter and between 4 mm (0.16 in.) and 7 mm (0.28 in.) in thickness. The cake of compound shall be warmed in water and the disk cut. If the cake is thinner than 4 mm (0.16 in.), two cakes shall be flamed and pressed together before cutting the disk.

4.3.2.2 Method of test. A water bath shall be adjusted to 45.0 ± 0.1°C (113.0 ± 0.2°F). A flat plate (to support the specimen while it is coming to the required temperature), a test block (Fig. 1-3) and a brass weight, having a base 50 mm (1.97 in.) in diameter and weighing 1,000 ± 5 g (2.2 ± .01 lb) in air,

shall be placed in the bath and allowed to come to the temperature of the bath. [The water level in the bath shall be 30 ± 5 mm (1.2 ± 0.2 in.) above the top surface of the test block.]

The impression specimen shall then be placed in the water bath on the flat plate. Twenty minutes later, the specimen shall be centered on the test block, a sheet of waterproof cellophane shall be placed over the specimen, and the weight shall be placed on the specimen. Ten minutes later, the weight shall be removed, the test block and specimen shall be taken from the bath and chilled at a temperature of 10°C (50°F), and the specimen shall be removed from the test block.

The impression shall be examined to determine if the ridges corresponding to the large cross grooves (B, Fig.1-3) of the test block are sharp, and the ridges corresponding to the fine grooves (C, Fig. 1-3) are complete and visible to the unaided eye for at least 30 mm (1.2 in.) of their length.

5. PREPARATION FOR DELIVERY

5.1 Packaging. The material shall be packaged in accordance with accepted commercial practice.

5.2 Marking.

5.2.1 Lot numbers. Each container shall be marked with a serial number or a combination of letters and numbers which refer to the manufacturer's records for the particular lot or batch of compound.

5.2.2 Date of manufacture. The date of manufacture (year and month) shall be given on the container either as a separate item or as a part of the lot number (5.2.1).

5.2.3 Net weight. The minimum net weight of the contents shall be indicated on all containers.

SUMMARY AND CONCLUSIONS

The American Dental Association specification for dental impression compound was revised with the assistance of the Specifications Committee of the Dental Materials Group of the International Association for Dental Research, with the aid of three manufacturers, and with the information derived from a recent survey of the certified brands of dental impression compounds. Two major changes were made in the detail requirements. First, since the 40.0°C (104.0°F) flow testing temperature appeared to present a critical point in testing, the requirement for flow at this temperature was deleted. Second, the requirement for flow at 50.0°C (122.0°F) for tray compound was replaced with a requirement for flow at 45.0°C (113.0°F). Therefore, in the revised specification both impression compound and tray compound will be tested at the same temperatures in determining values of percent flow. After examination of possible variables introduced in preparation and testing of flow specimens and after examination of results of the data exchange program, more precise methods for preparation and testing of specimens for flow were incorporated in the revised specification.

1. Taylor, N. O.; Sweeney, W. T.; and Paffenbarger, Geo. C. A specification for dental impression compound. J.A.D.A. 18:53 (Jan.) 1931.
2. Sweeney, W. T. and Beall, John R. American Dental Association specification no. 3 for dental impression compounds. First revision 1939. J.A.D.A. 27:713 (May) 1940.
3. Outline of form and instructions for the preparation of Federal specifications (21 October 1947). Superintendent of Documents, U. S. Government Printing Office, Washington 25, D. C.
4. List of certified dental materials revised to November 15, 1953. J.A.D.A. 47:693 (Dec.) 1953.

Table 1

Plan for exchanging data on impression compounds

Source of Material	Specimens made by	Specimens tested by	Result*
From the manufacturer	Mfr.	Mfr.	A
		NBS	B
	NBS	Mfr.	C
		NBS	D
Obtained on the open market by the A.D.A. Research Fellowship at the National Bureau of Standards	Mfr.	Mfr.	E
		NBS	F
	NBS	Mfr.	G
		NBS	H

* The value reported for each of the results is the average of two specimens as required by the specification.

Table 2

Data exchanged on impression compound with manufacturer X

Result	Percent Flow at 37°C (98.6°F)	40°C (104°F)	45°C (113°F)
A	1.9	30.4	----
B	2.2	24.0	93.8
C	3.3	26.4	90.7
D	3.2	30.6	95.6
E	3.4	25.4	91.7
F	3.8	30.4	95.7
G	2.7	16.2	90.7
H	1.9	19.0	93.3
Average	2.8 ± 1.2	25.3 ± 4.1	93.1 ± 1.7

Table 3

Data exchanged on impression compound with manufacturer Z

Result	Percent flow at 37°C (98.6°F)	40°C (104°F)	45°C (113°F)
A)	3.2	29.4	86.1
B) *	3.6	33.0	95.8
C)	2.1	23.5	89.2
D)	3.2	30.0	94.7
Average	3.0 ± 0.5	29.0 ± 2.7	91.4 ± 3.8
E)	0.6	7.8	86.4
F) #	1.2	14.2	92.6
G)	0.8	8.2	88.2
H)	0.6	8.8	91.5
Average	0.8 ± 0.2	9.8 ± 2.2	89.7 ± 2.4

* Data exchanged with manufacturer Z on impression compound furnished by him and not the same lot or batch as reported in Results E through H.

Data exchanged with manufacturer Z on impression compound obtained in open market for survey.

Table 4

Data exchanged with manufacturer Y on impression compound obtained in the open market

Result	Percent flow at 37°C (98.6°F)	40°C (104°F)	45°C (113°F)
E	2.1	44.0	87.2
F	3.2	34.7	90.4
G	1.0	21.0	87.0
H	4.6	55.2	96.0
Average	2.7 ± 1.2	38.7 ± 10.9	90.2 ± 3.0

Table 5

Data exchanged with manufacturer Y using revised methods on impression compound furnished by him and not the same lot or batch as reported in Table 4

Result	Percent flow at 37°C (98.6°F)	40°C (104°F)	45°C (113°F)
A	3.8	47.1	90.5
B	4.6	53.4	92.3
C	2.2	36.4	90.4
D	3.1	45.1	92.6
Average	3.4 ± 0.8	45.5 ± 4.9	91.4 ± 1.0

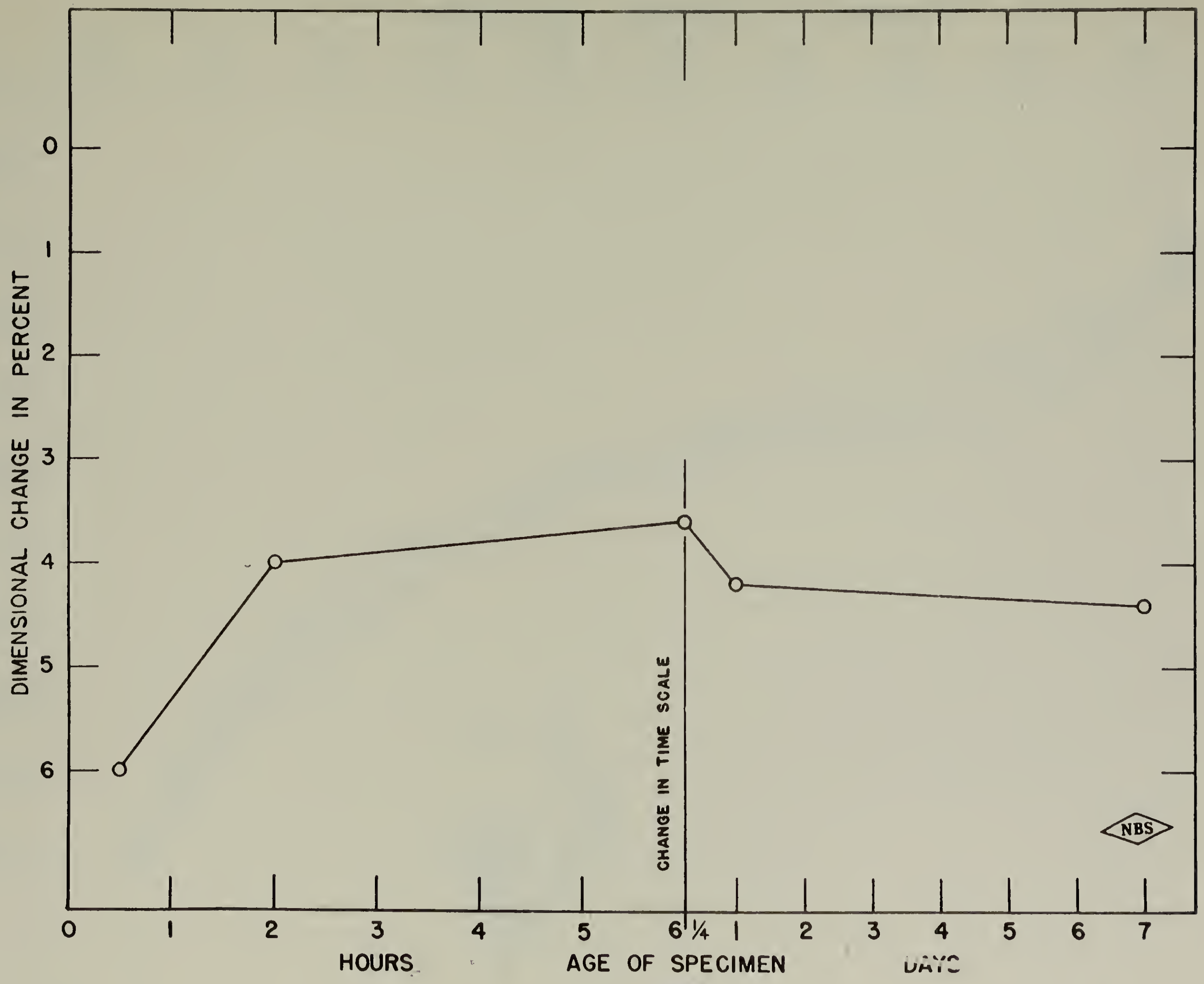


FIGURE I

DECREASE IN FLOW OF IMPRESSION COMPOUND WITH INCREASE IN AGE OF SPECIMEN
TESTED AT 37.0°C (98.6°F)

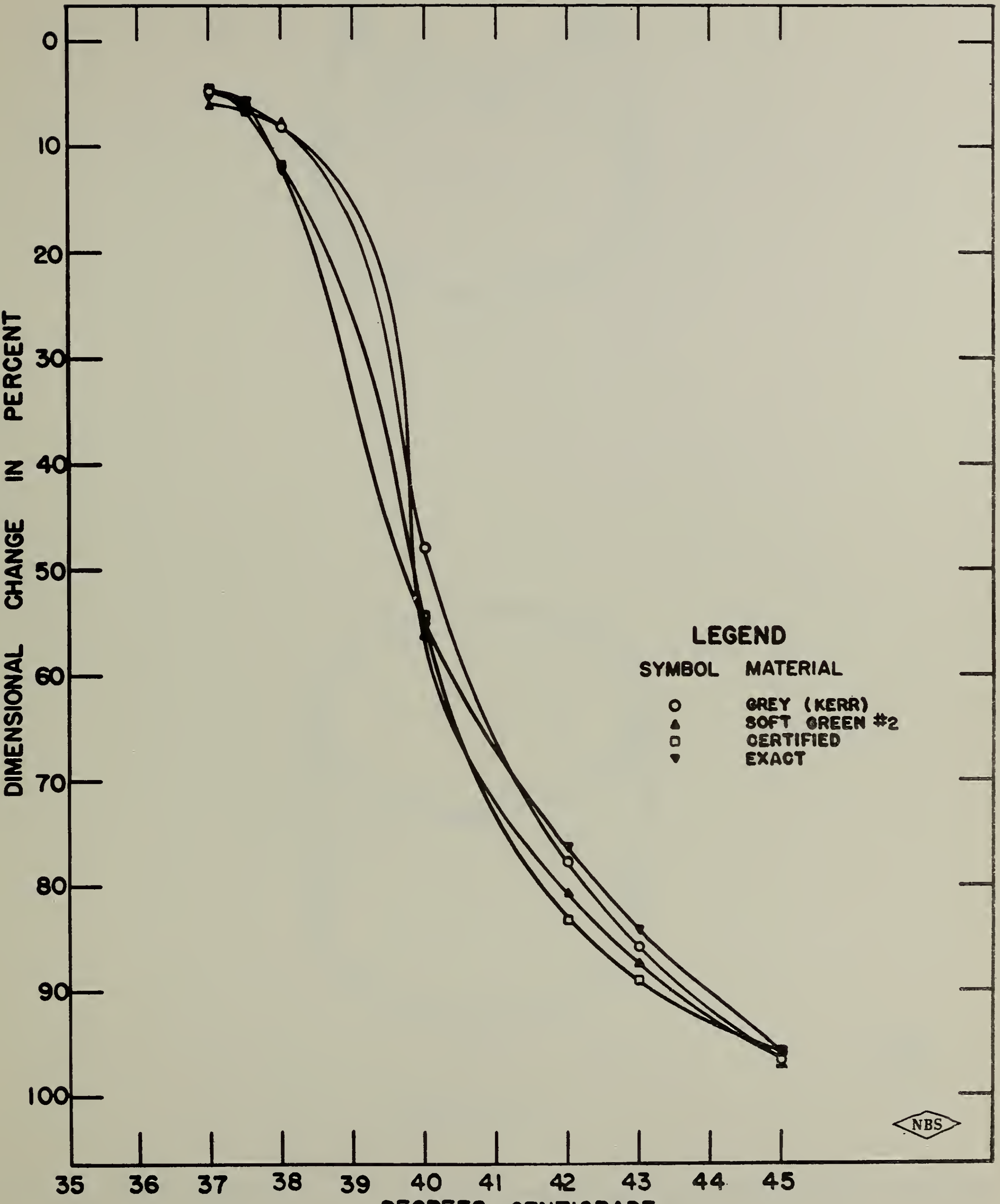


FIGURE 2

FLOW OF IMPRESSION COMPOUNDS

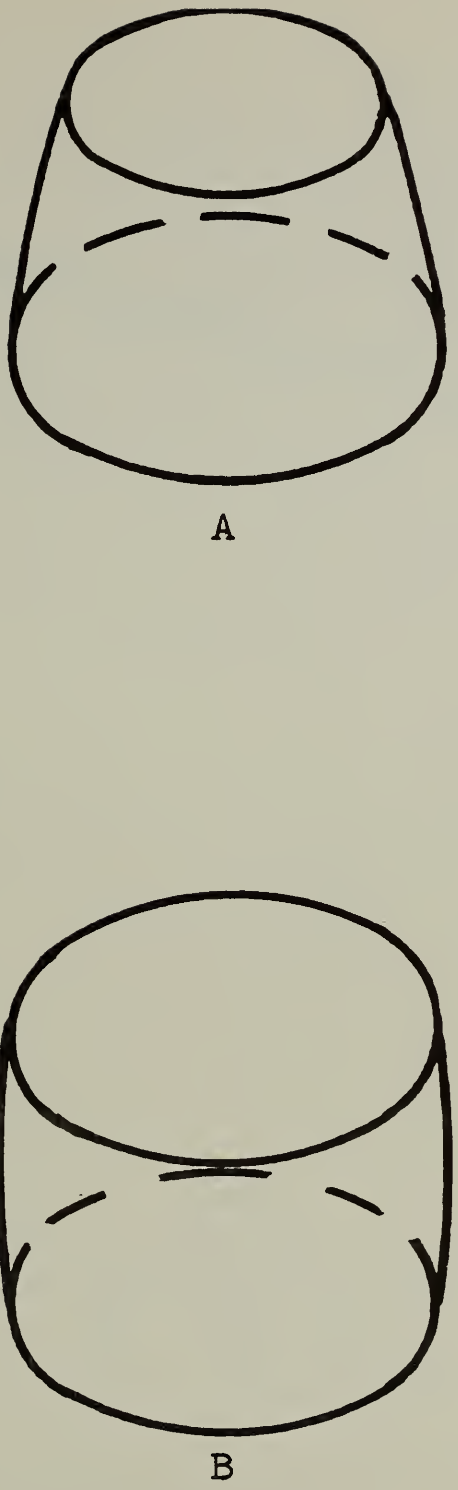
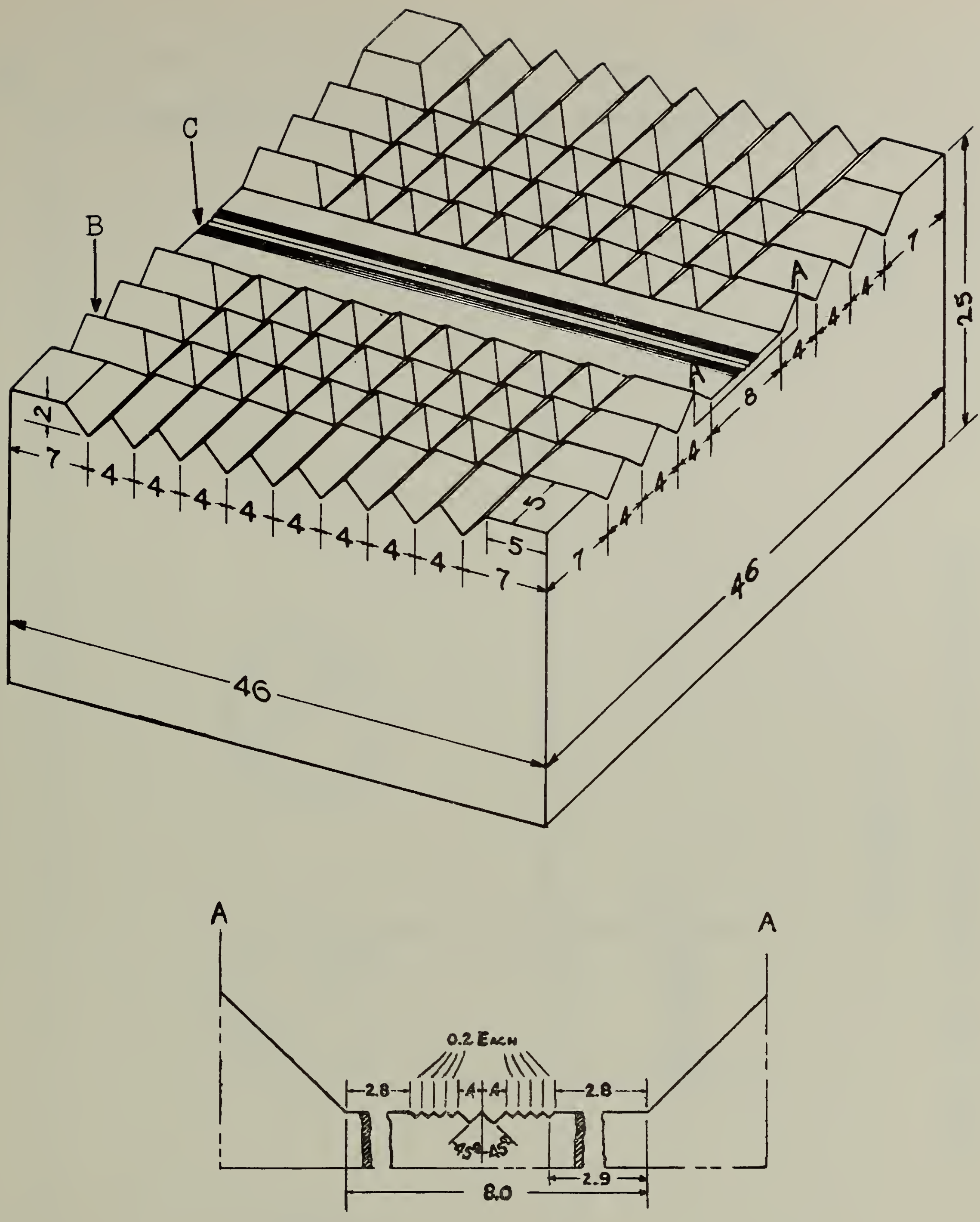


FIGURE 3

Flow Specimens. A. Truncated-shaped specimen formed using metal shaft.

B. Barrel-shaped specimen formed using non-conducting shaft.



ALL DIMENSIONS IN MILLIMETERS.
ALL GROOVES, 90° INCLUDED ANGLE, SIDES AT 45° TO SURFACE OF BLOCK. ALL DIMENSIONS TO BE HELD TO ±0.02 MM.

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FIGURE 1-3

Test Block for Dental Impression Compounds

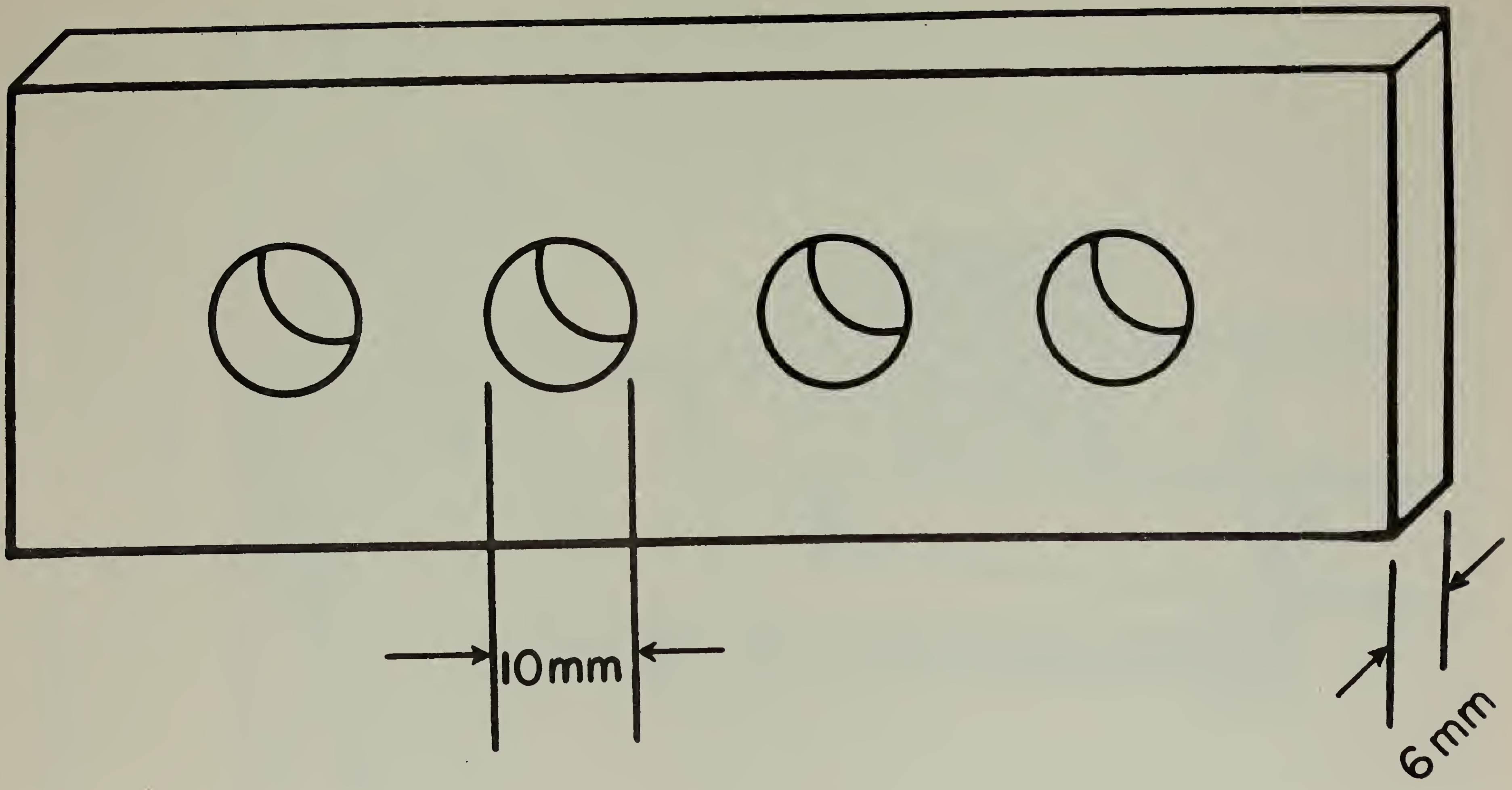

FIGURE 2-3

Mold for Forming Flow Specimens

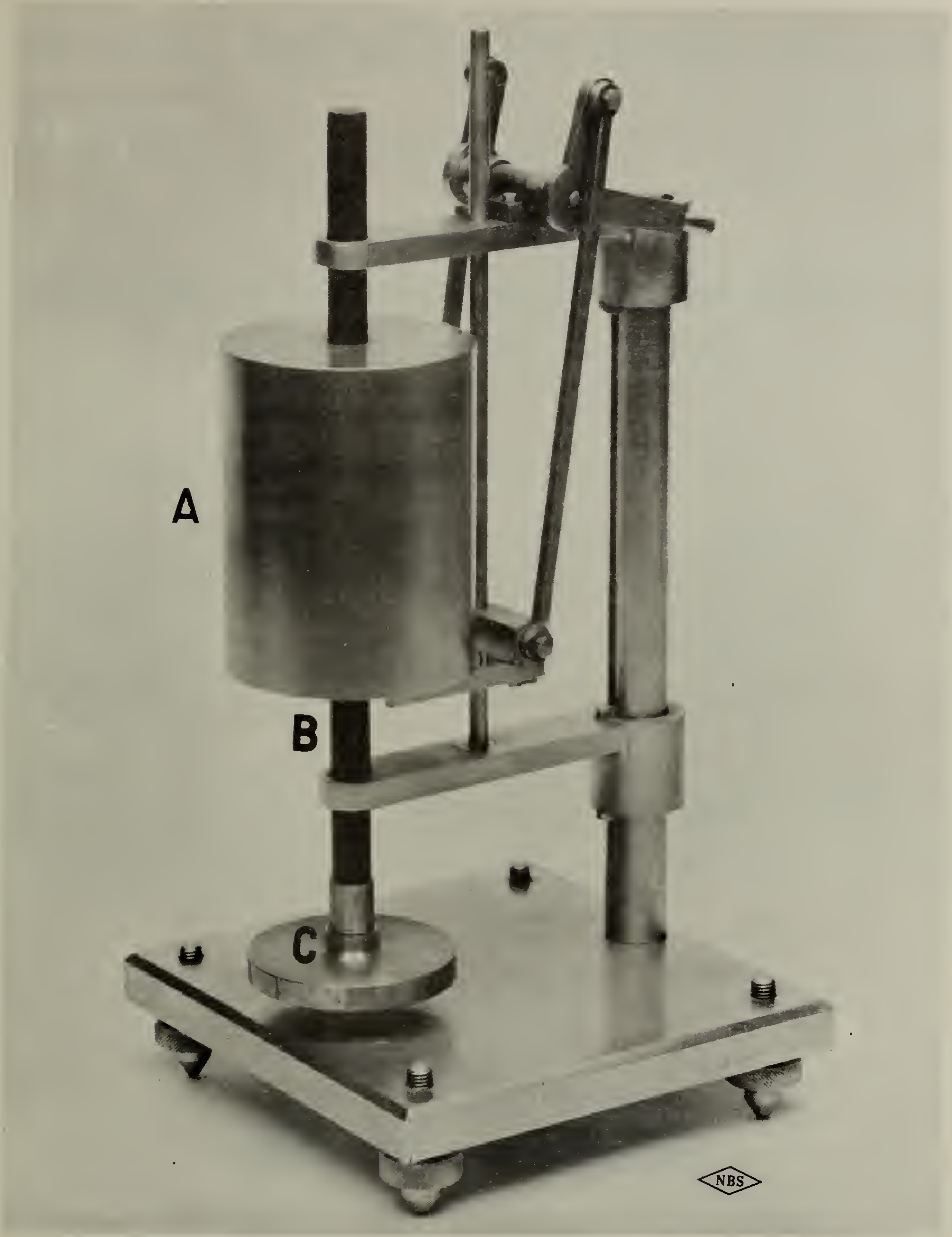

FIGURE 3-3

Flow Testing Instrument. A. Weight

B. Non-conducting shaft

C. Platen

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Progress Report

SOME FACTORS INFLUENCING THE CUTTING CHARACTERISTICS OF ROTATING DENTAL CUTTING INSTRUMENTS

by

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(Task 7756-41 BV No. 55-14)

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

SOME FACTORS INFLUENCING THE CUTTING CHARACTERISTICS OF ROTATING DENTAL CUTTING INSTRUMENTS

Abstract

The demand for more efficient rotating dental cutting instruments has made it desirable to investigate the factors that influence cutting. This report includes the results on steel burs, carbide burs, and diamond instruments. The data obtained under controlled tests compare the cutting rates of these tools on human enamel at speeds of 5,000 and 10,000 rpm under a load of 20 grams. The rates of enamel cutting were slightly improved at high speed with carbide burs. Diamond instruments have much higher cutting rates and cutting efficiency than the metal burs in cutting enamel.

1. INTRODUCTION

The Dental Research Laboratory of the National Bureau of Standards has been conducting research on methods of evaluating rotating dental cutting instruments for some time. Since 1950, when the U. S. Air Force began active support of the program, the following characteristics of conventional dental burs and abrasive instruments have been examined: (1) temperatures developed in rotating dental burs (2) vibration produced by dental burs and diamond abrasive instruments, and (3) dynamic studies, using high-speed motion picture photography showing for the first time the behavior of these instru-

ments as they cut tooth structures.

The purpose of these studies has been two-fold. Primary interest has focussed on obtaining the most efficient operation of rotary cutting instruments consistent with producing minimum trauma from temperature rise and vibration on vital structures. Secondly, it is hoped that information obtained from these studies may be used in formulating specifications for burs and abrasive instruments for the Armed Services.

Peyton, Vaughn, and Henry [1, 2 and 3] have conducted investigations at the University of Michigan on relationships between temperature rise and cutting rates of dental burs and their rotational speeds. A study conducted at the National Bureau of Standards also dealt with temperatures developed in rotating dental burs [4]. This study revealed that temperatures ranging from 140°F to 600°F may be reached at the site of operation, depending upon the speed of rotation, operating pressure, and the material being cut. Such temperatures would contribute to the discomfort of dental operative procedures and would damage vital tissues.

It is concluded from these laboratory studies that cooling sprays or streams of water should be employed in clinical practice, even when instruments are operating at low rotational speeds.

Vibration caused by rotating instruments, while possibly not as traumatic to vital structures as heat, annoys the patient and contributes to his dislike of dental operations. Furthermore, a chattering instrument cannot be controlled by the operator as he strives to carry out the finer details of cavity preparation.

Walsh and Symmons [5] have made a study of vibration and its effect on dental patients. They find that vibrational frequencies between 100 cps (cycles per second) and 200 cps produce the most unfavorable response when applied to the teeth. Frequencies above 1000 cps are generally beyond the upper range of average patient perception (Figures 1 and 2). Unfortunately the lower frequencies in the range 100 to 200 cps are dominant in eccentric burs rotated at speeds of 6000 to 10,000 rpm, speeds now in use by many practitioners.

2. EXPERIMENTAL PROCEDURE

Experimental apparatus used in our studies of vibration produced in rotating instruments consisted of the following: a hard steel vane supported so that it rested against the periphery of the bur or abrasive wheel being studied, and an electronic micrometer of the mutual inductance type energized by the 4.8 megacycle output of a low-powered RF generator. The pickup was held close to the steel vane and reacted to minute movements of this vane as it was caused to shift in a plane normal to the long axis of the pickup from irregularities in the rotating instrument (Figure 3).

Instruments were rotated in a precision chuck of the draw-collet type, whose eccentricity was negligible; thus, any displacement of the steel vane was caused by instrument eccentricity or surface irregularity. Output of the vibration pickup was applied to the Y-axis deflection plates of an oscilloscope on whose screen the wave forms were studied or photographed.

When observing the pattern of vibrations produced by dental burs and abrasive wheels with no consideration of the effects of

handpiece looseness or irregularity, we found that the principal cause of vibration was bur eccentricity. The amplitude of this vibration was directly proportional to the degree of eccentricity (Figures 4 and 5).

A photograph of the oscilloscope trace produced by a true-running 8-bladed bur, rotating at 10,000 rpm, shows eight low-amplitude peaks per revolution at a frequency of 1330 cps (Figure 6). A similar bur, if eccentric and rotating at the same speed, produces a fundamental frequency of 166 cps, with amplitude proportional to the amount of eccentricity. The 1330 cps wave is still seen, modulating the lower frequency, but at an amplitude that is low in comparison to that of the fundamental (Figure 7).

3. DISCUSSION

Thus, we see that a true-running bur at 10,000 rpm produces vibration in a frequency range well above that which produces unpleasant reaction in the patient [5], while the eccentric bur produces vibration in the range of maximum unpleasantness. Application to clinical practice of the information obtained in this study will, of necessity, await improvements in the manufacture of rotating instruments. Closer tolerances in fabrication and more symmetrical shaping of the cutting heads of burs and wheels, should, to a great degree, remove the cause of annoying chatter and vibration in the range of frequencies most distressing to the patient.

We cannot, however, ignore the effects of poor handpiece performance. A true-running instrument will not perform properly in a handpiece that does not operate smoothly. To date, our studies

at the National Bureau of Standards have not included dental handpieces. We plan to undertake such studies in the near future.

Data on relative cutting abilities of steel and carbide burs in a synthetic dentin substitute were obtained in earlier experiments [4]. Preliminary results of our more recent work reveal that they behave similarly in human dentin. There is little difference between cutting rates of steel and carbide burs and they both cut faster as their speeds of rotation increased.

Further results of these more recent studies of steel and carbide burs when cutting human tooth enamel are shown in Tables 1 and 2. Diamond abrasive instrument data are shown in Table 3.

These data reveal little difference in the amount of enamel cut by steel burs of two different makes rotating at 5000 rpm and cutting in specimens taken from a single molar tooth (Table 1). Carbide burs of two different makes, cutting specimens from the same tooth under the same conditions, show little difference in cutting rate (Table 2).

When speed of rotation was 10,000 rpm and specimens from another molar tooth were cut, little difference in rate of cut was noted between two different makes of steel burs. Two different makes of carbide burs also cut at about the same rate at this speed.

When cutting enamel at 5000 rpm, the rate of cut was substantially the same for carbide burs as for steel burs. At 10,000 rpm, the rate of cut of carbide burs was twice that of steel burs in the same tooth specimen.

No effort has been made to account for these differences between carbide and steel bur cutting rates.

The enamel from one tooth was consumed during the evaluations at 5000 rpm. Another was required for obtaining data at 10,000 rpm. Since a possible variation in hardness of enamel between the two teeth used must be considered, no valid comparison of the cutting efficiencies of these burs at the two speeds can be made.

Diamond abrasive instruments, under the same experimental conditions, removed 25 to 30 or more milligrams of enamel per 100 surface feet of operation (Table 3). Burs of steel and carbide removed approximately 0.5 to 2.0 milligrams of enamel per 100 SF (Table 2). The cutting efficiency of the diamond instruments tested did not increase as the speed of rotation was raised from 5000 rpm to 10,000 rpm. Cutting rates, however, were approximately doubled at the higher speed.

This information is of clinical significance to us in pointing out the importance of choosing the most appropriate cutting instrument for the operation being performed. The efficient accomplishment of operative procedures with a minimum of trauma to vital structures, using conventional burs and stones, requires careful selection and proper employment of these instruments. Further improvements in design and closer control in production should provide us with smoother running and more efficient rotating cutting instruments.

4. SUMMARY

Vibration in a frequency range most distressing to patients is produced by eccentricity in rotating dental instruments. High speed (10,000 rpm) rotation of dental instruments is advantageous

in that vibration produced by bur blades is raised in frequency to a point above the range of maximum unpleasantness as defined by Walsh and Symmons. Also at this higher speed diamond instruments cut more rapidly. Carbide burs cut at twice the rate of steel burs at the rotational speed of 10,000 rpm. Cutting rates of both steel and carbide burs are improved at the higher speed when cutting dentin. Diamond abrasive instruments are many times more efficient than steel or carbide burs when cutting tooth enamel.

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Table 1

Cutting Rate in Human Enamel
With Steel Dental Burs (#559)
at 200 g load

Sample	Mfgr.	No. Blades	Cutting Rate	Cutting Efficiency
			mg/min	mg/100 SF
		Speed	5,000 rpm*	
1	A	6	1.04	1.33
2	"	"	0.82	1.05
3	"	"	0.66	0.85
4	C	"	1.25	1.60
5	"	"	1.64	2.10
6	"	"	1.54	1.98
		Speed	10,000 rpm**	
7	A	"	0.74	0.48
8	"	"	0.96	0.61
9	"	"	0.48	0.31
10	C	"	1.04	0.66
11	"	"	0.76	0.49
12	"	"	0.62	0.40

* 78 surface ft. per min. All cuts in specimens from tooth #1, cutting for 5 min.

** 156 surface ft. per min. All cuts in specimens from tooth #3, cutting for 5 min.

Table 2

Cutting Rate in Human Enamel with Carbide Burs (#559) at 200 g load

Sample	Mfgr.	No. Blades	Cutting Rate	Cutting Efficiency
			mg/min	mg/100 SF
Speed 5,000 rpm*				
1	A	6	1.17	1.50
2	"	"	0.82	1.05
3	"	"	1.63	2.09
4	B	8	0.72	0.92
5	"	"	1.04	1.33
6***	"	"	4.19	5.37
Speed 10,000 rpm**				
7	A	6	1.10	0.70
8	"	"	1.28	0.82
9	"	"	1.63	1.04
10	B	8	1.34	0.86
11	"	"	1.25	0.80
12	"	"	2.01	1.29

* 78 surface feet per minute. All cuts in specimens from tooth #1, cutting for 10 minutes.

** 156 surface feet per minute. All cuts in specimens from tooth #3, cutting for 10 minutes.

*** A bur blade fractured; resulting eccentric instrument cut at higher rate but produced severe vibration.

Table 3

Cutting Rates of Diamond Abrasive Wheels Approximately 1/4" in Diameter in Human Enamel at 200 g Load

Sample	Mfgr.	Cutting Rate mg/min	Cutting Efficiency mg/100 SF
		Speed 5,000 rpm*	
1	A	125.7	27.94
2	"	125.7	27.93
3	"	130.9	29.09
4	"	155.1	34.46
5	"	116.1	25.80
6	B	119.2	26.49
7	"	138.4	30.75
8	"	113.9	25.31
9	"	115.3	25.62
10	"	104.8	23.29
		Speed 10,000 rpm*	
11	A	214.9	23.87
12	"	179.7	19.96
13	"	244.6	27.18
14	"	280.3	31.14
15	"	202.1	22.45
16	B	262.6	29.18
17	"	323.3	35.92
18	"	234.3	26.03
19	"	225.9	25.10
20	"	321.8	35.75

* Surface speed adjusted according to diameter of instrument to 450 SFM and 900 SFM, respectively, corresponding to approximately 5000 and 10,000 rpm. Multiple tooth specimens were used in these experiments. The enamel was removed from approximately 200 teeth while obtaining the data. Total cutting time for each instrument was 10 minutes.

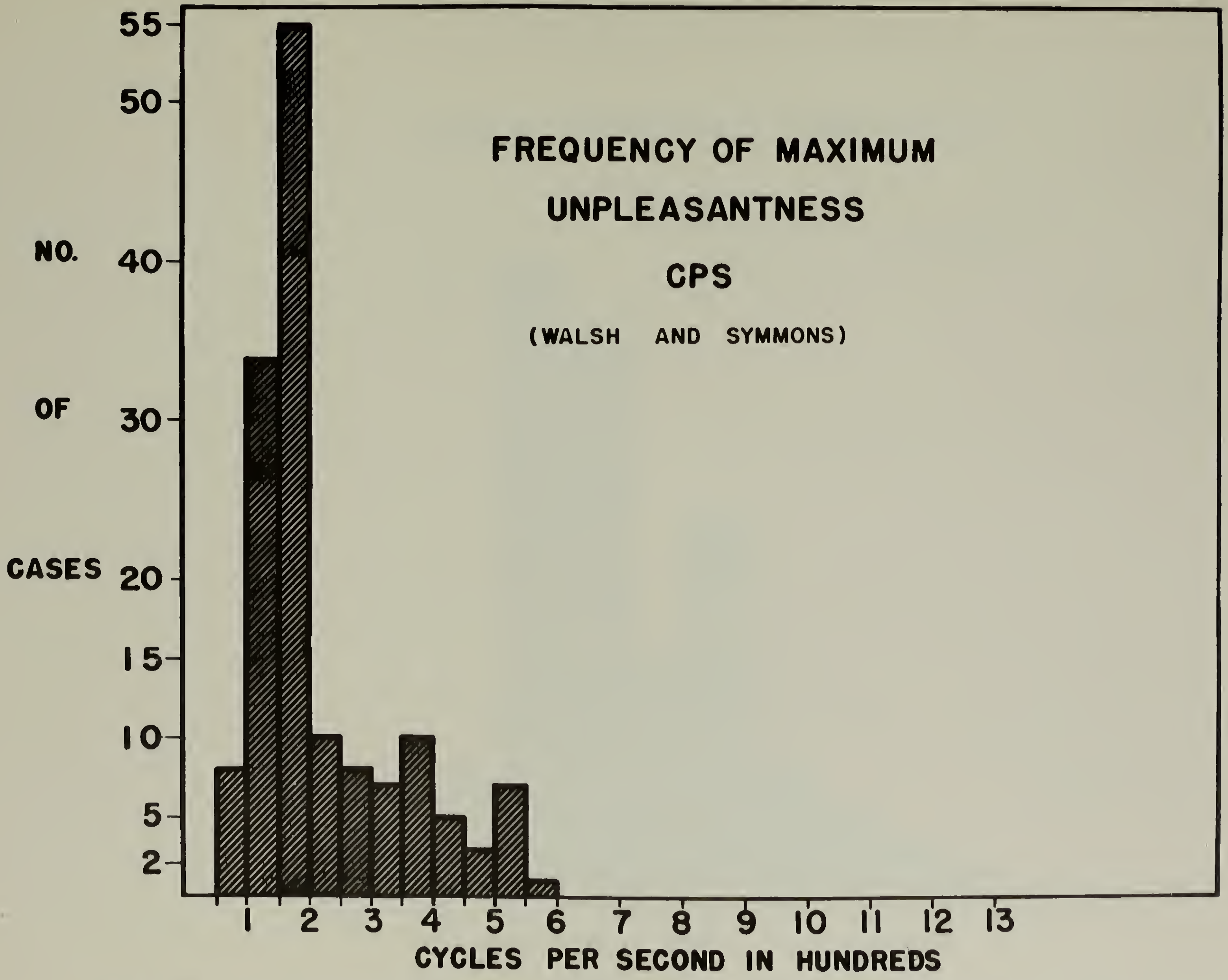

Figure 1. Graph showing frequencies found to produce most unpleasant reaction when applied to teeth. (Walsh and Symmons).

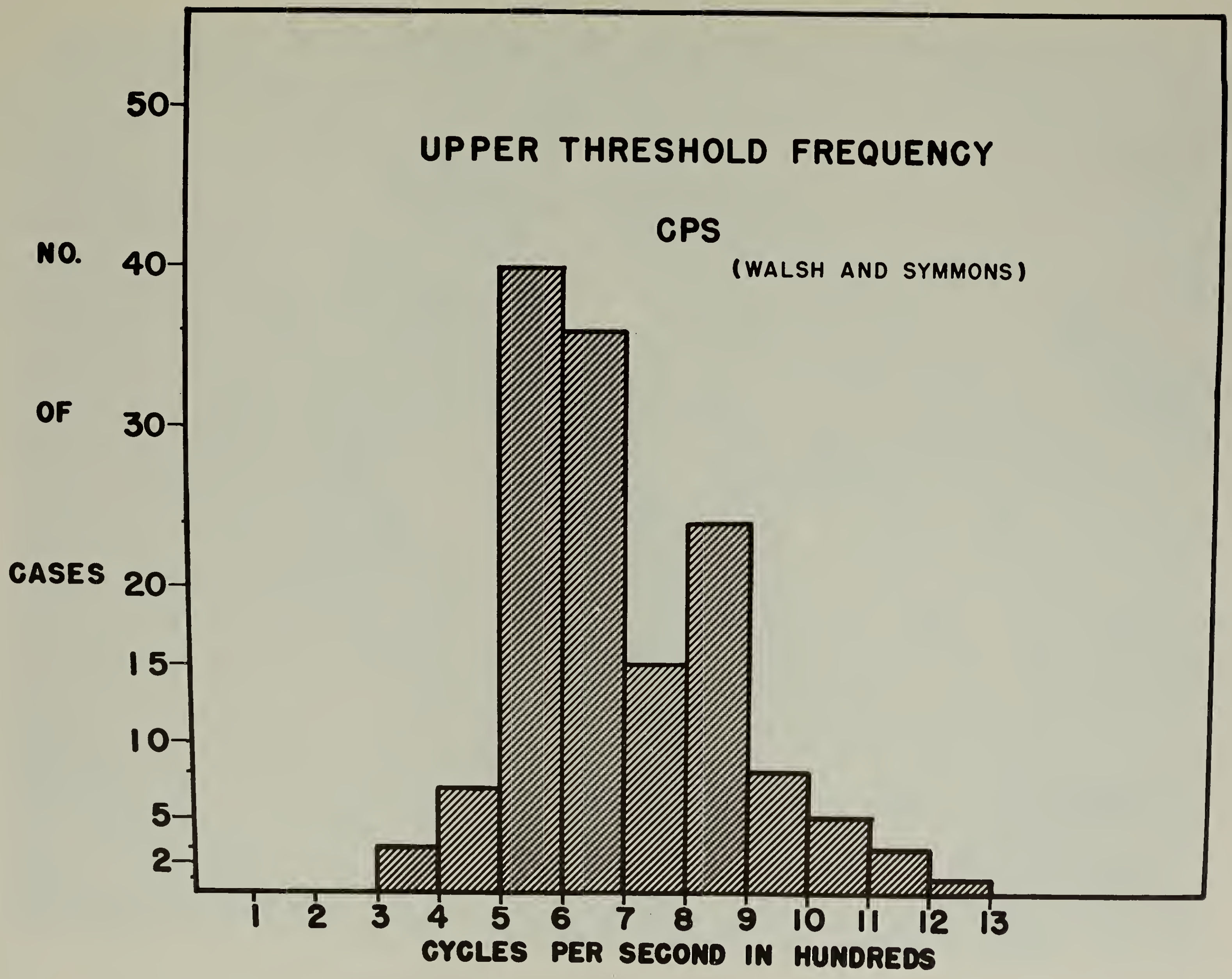

Figure 2. Graph showing upper limits of vibration perception when applied to teeth. (Walsh and Symmons).

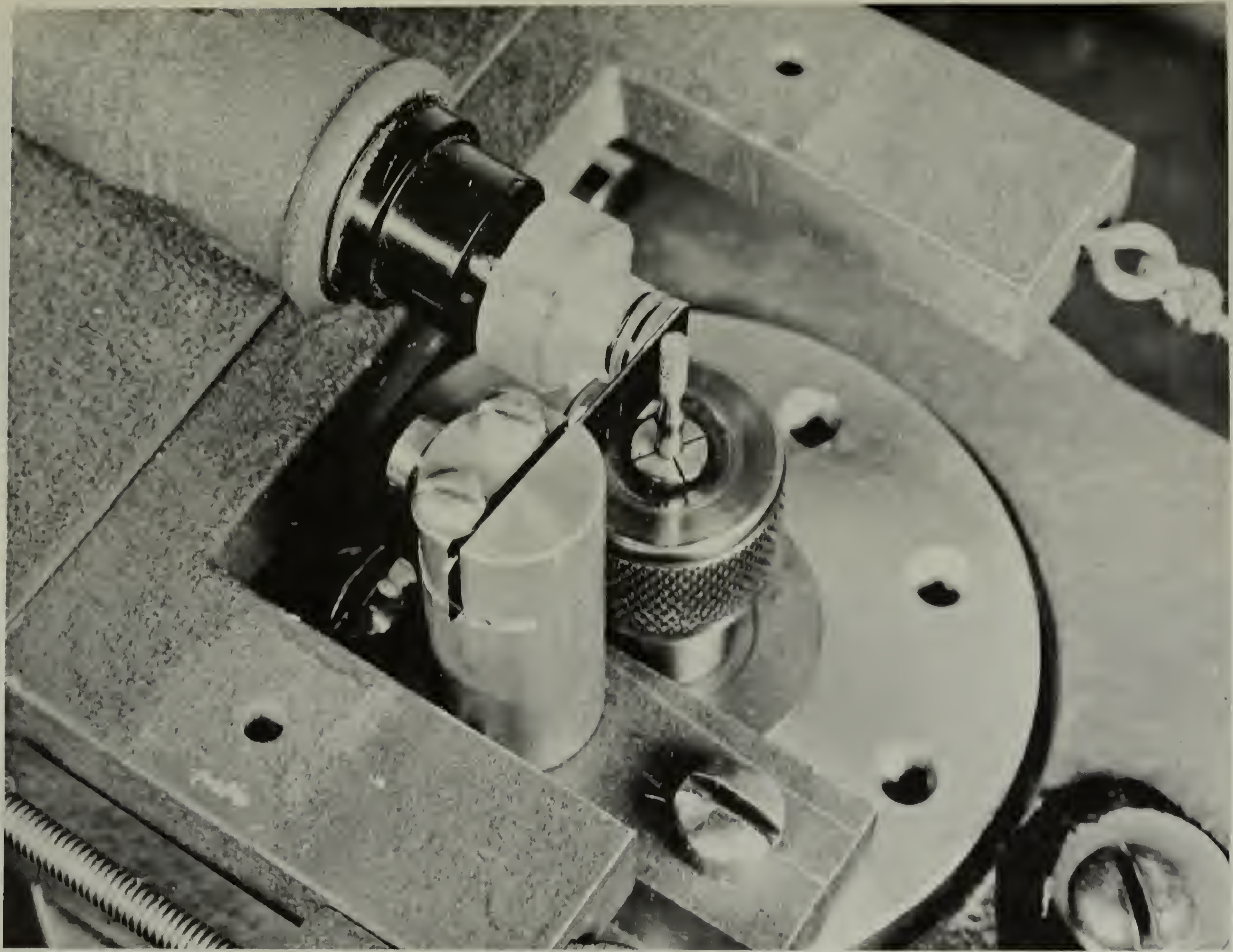

Figure 3. Electronic micrometer and steel vane arranged to pick up vibration of rotating instrument.

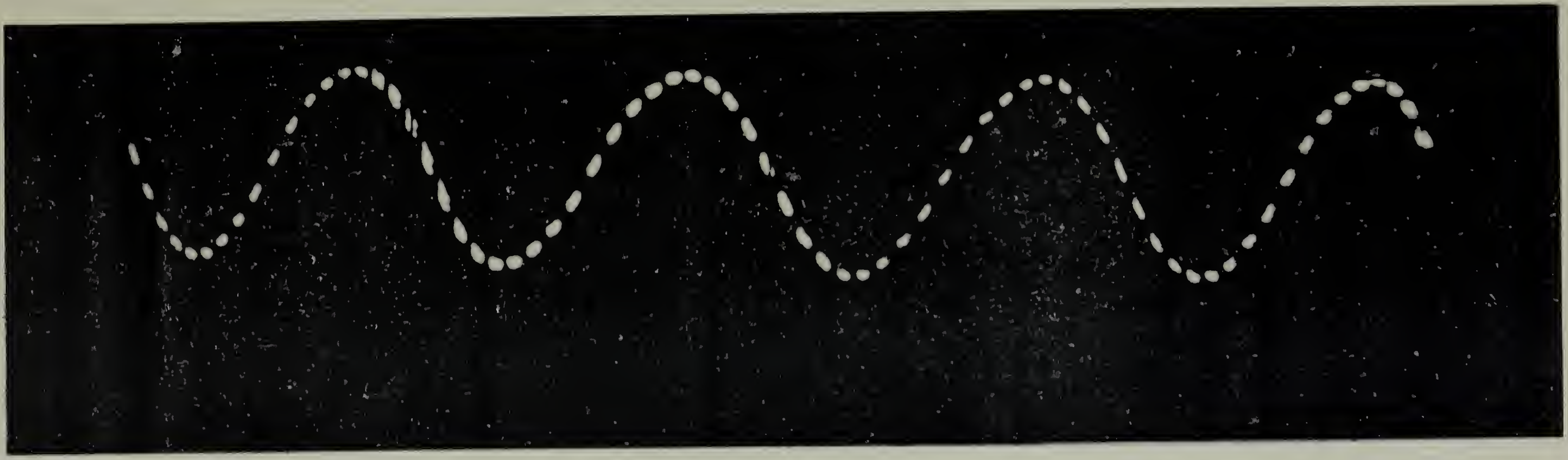

Figure 4. Trace from rotating dental instrument with eccentricity of .007 in.

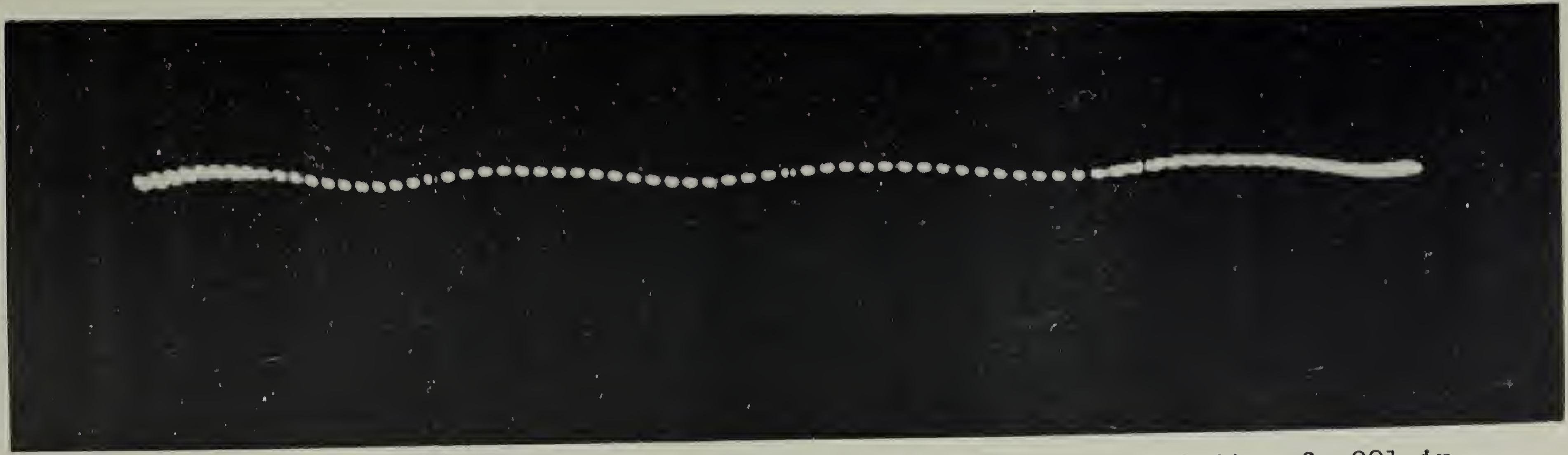

Figure 5. Trace from rotating dental instrument with eccentricity of .001 in.

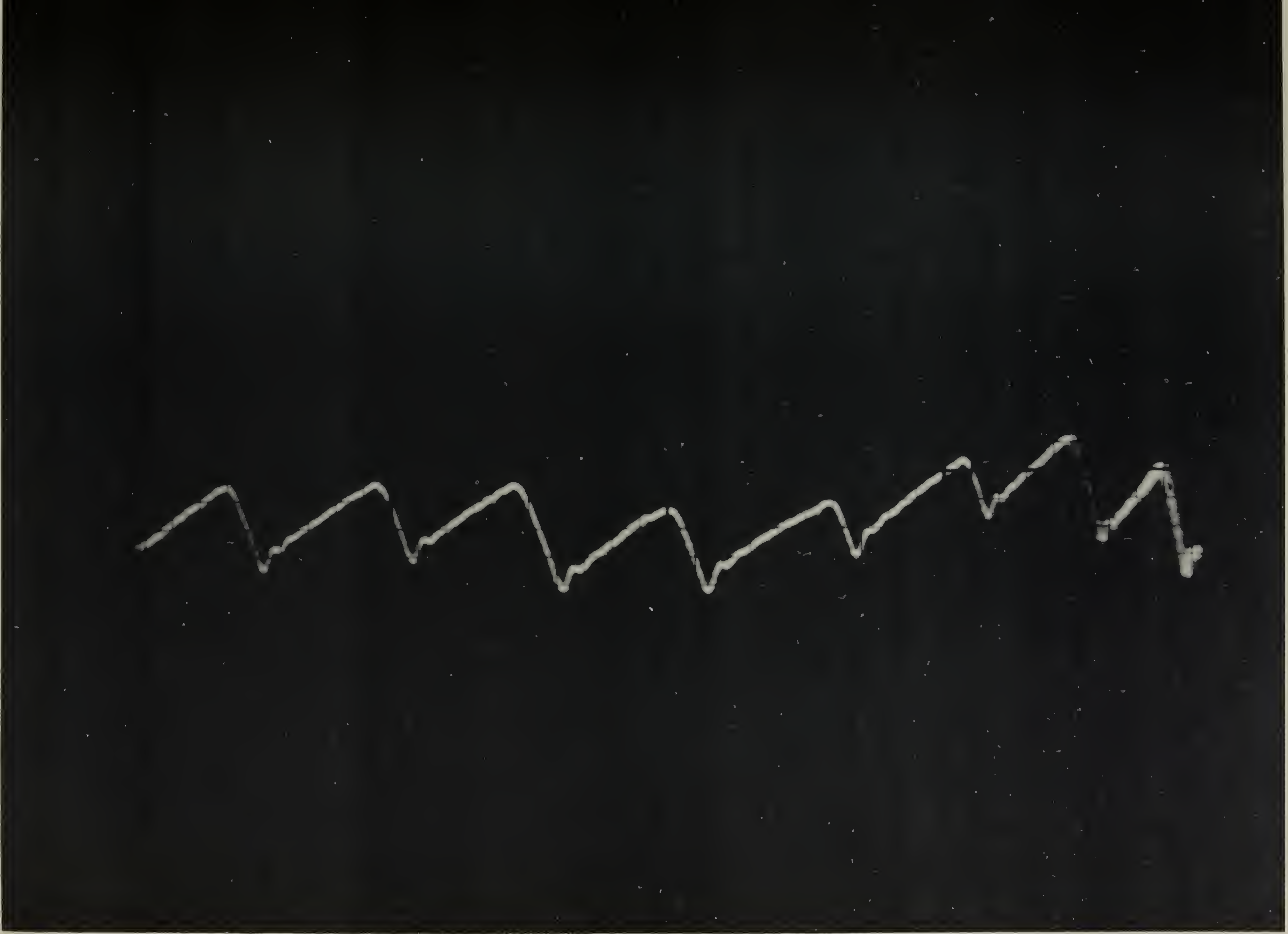

Figure 6. Trace produced by 8-bladed true-running dental bur. Frequency of vibration 1330 c.p.s. at 10,000 rpm.

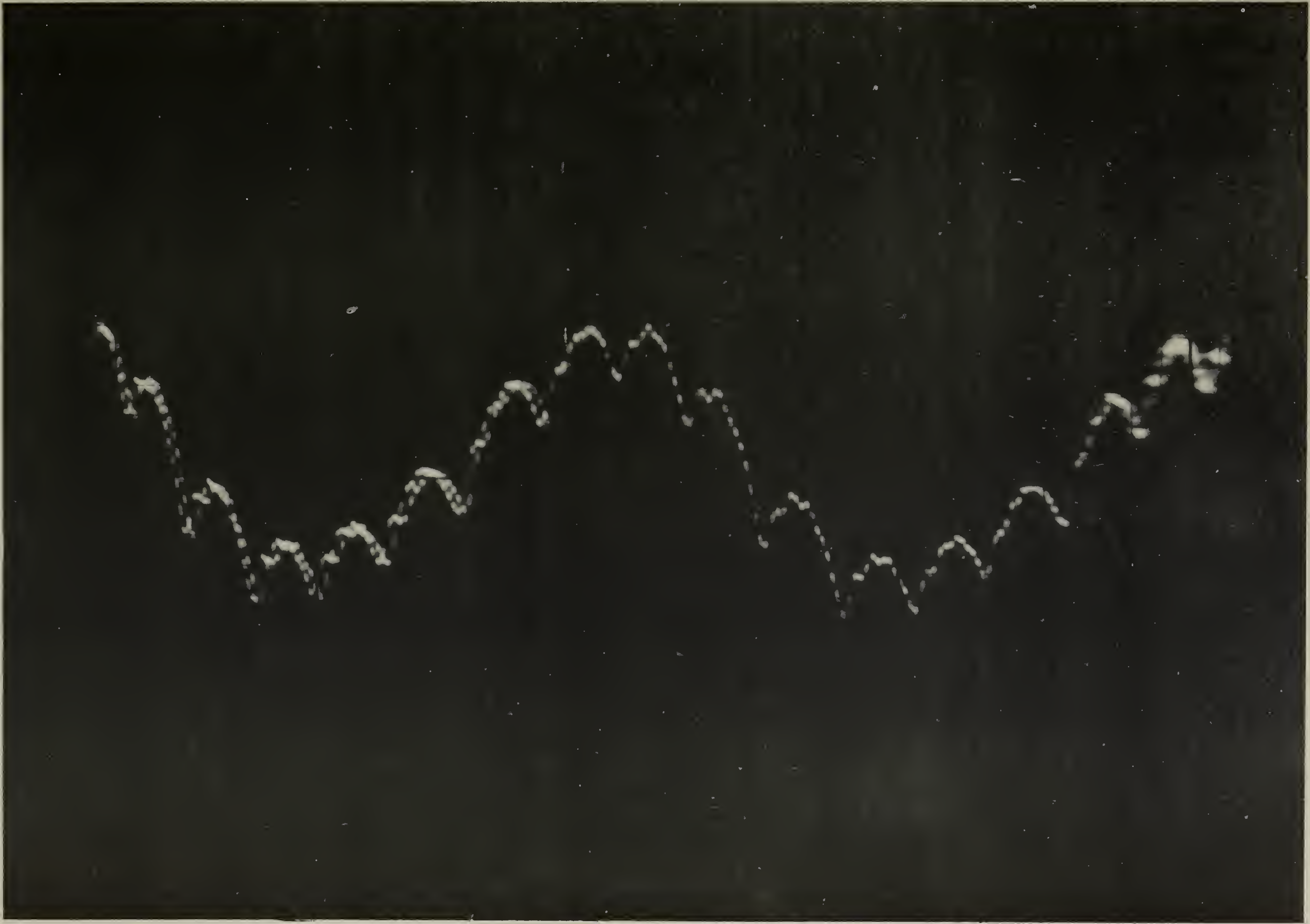

Figure 7. Trace produced by 8-bladed eccentric dental bur. Frequency of high amplitude 166 c.p.s. and modulating component 1330 c.p.s. of low amplitude. Bur rotated at 10,000 rpm.

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Progress Report

USE OF IONIZATION CHAMBERS IN OBTAINING DATA ON RADIATION AT POINTS WITHIN THE HEAD DURING DENTAL X-RAY EXPOSURE

by

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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 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

USE OF IONIZATION CHAMBERS IN OBTAINING DATA ON RADIATION AT POINTS WITHIN THE HEAD DURING DENTAL X-RAY EXPOSURE

Abstract

A study to determine levels of ionizing radiation at various points in and about the human head during dental radiography was undertaken. A phantom of tissue-equivalent wax was built around an adult human skull, and measurements were made using small Sievert ionization chambers placed at the skin and within the skull at ten different locations.

Radiation absorbed during full-mouth intra-oral dental radiography using conventional 8" skin-target distance, 65 KV and 10 ma with 1.5 mm of aluminum filtration, and exposing 14 films, was measured. Dosage at the skin of the cheek amounted to 20-25R and various lower values were obtained at other points within the head.

A comparison of these radiation levels was made with measurements taken at the same points in the head when using a panoramic extra-oral radiographic technic. Maximum dosage in this technic occurred in the lymphatic region of the neck and a reading of .42R was recorded there during full-mouth exposure. Levels within the skull ranged downward from this value.

1. INTRODUCTION

A project at the Dental Research Laboratory, National Bureau of Standards, sponsored by the U. S. Air Force Dental Service, is concerned with the development of a device for making extra-oral full-mouth panoramic dental X-ray exposures. In connection with this project, it was desired to determine levels of radiation produced at various points in and about the head using the panoramic device. For purposes of comparison, measurements were also desired that would indicate radiation levels existing during full-mouth exposure using conventional dental X-ray technic.

2. APPARATUS EMPLOYED

Since the panoramic method employs a narrow beam of X-rays in making exposure, an ionization chamber of small volume was required. Such a device was found in the Sievert ionization chamber [1,2], which has an air volume slightly in excess of 30 mm, and a physical size which was easily accommodated in the specimens to be irradiated. The Sievert chamber (Fig. 1 and 2), is a condenser-type R meter, having a wide range of sensitivity dependent upon the charge applied, and can be relied upon to produce measurements accurate to within ± 10% at the energies used in this experiment. All voltage measurements after charging and after exposure to radiation were made using the Kiethley Electrometer. Using calibration curves previously prepared for each chamber, and corrections for atmospheric pressure and humidity, radiation levels to which the chambers had been exposed were calculated.

The X-ray source was a standard radiographic unit, operated at 65 KV and 10 ma, having $\frac{1}{2}$ mm Al. equivalent inherent filtration,

to which was added 1 mm Al. The quality of the radiation was 18-20 KEV, calculated from its absorption coefficient.

A wax phantom head was constructed about an adult human skull. The wax was of tissue-equivalent density to X-rays and consisted of a mixture of 50% paraffin and 50% beeswax by weight to which was added .5% by weight of rosin [3]. The phantom was constructed to permit access to points within the head for the placement of the ionization chambers (Fig. 3 and 4).

3. EXPERIMENTAL METHOD

Ten points were chosen at which to measure radiation levels. Several of these were chosen because they are located in areas rich in lymphoid or glandular tissue. Points at which overlap or intersecting planes of radiation might be expected were also included. These points were: (1) at the skin of the neck when using the panoramic exposure and at the skin of the cheek when using conventional exposure; (2) in the region of the sella tursica; (3) in the region of the parotid gland; (4) in the sublingual region; (5) in the center of the base of the tongue; (6) in the cornea of the eye; (7) in the region of the thyroid gland; (8) in the deep cervical lymphatic region; (9) in a lower bicuspid tooth socket; and (10) at a point just posterior and medial to the lower third molar tooth. The last-mentioned location was included since the axis about which the beam of X-rays was rotated during panoramic exposure passed through this point. It was desired to determine if any excessive concentration of radiation occurred at this center of rotation.

Rate of delivery from the filtered X-ray source was approximately 558 mr/sec in air at 12" from the target when using the slit or narrow

beam in the panoramic technic, and about 647 mr/sec in the air at the same distance when using a round cone of rays for the conventional intra-oral technic. Delivery rates were measured using the Sievert chambers.

Skin-target distance was 8" when employing the standard intra-oral 14 film radiographic method and varied from 8" to 12.5" depending on the position of the X-ray source at any given time in its cycle of motion about the head during radiography by the panoramic method. Total exposure time was 25 sec. with the panoramic technic and 40.5 sec. with the intra-oral method.

In each full-mouth exposure included in this experiment, the conditions that would exist during actual clinical practice were adhered to. Standard lead-backed dental films (Eastman Radiatized) were positioned in the phantom for each single exposure by the conventional technic. One series of 14 exposures was found to be sufficient to produce radiation levels at all measuring points falling within the range of the ionization chambers. It was necessary to repeat the exposure ten times when using the panoramic method in order to obtain levels of radiation that could be reliably measured, using the same chambers.

Total skin-surface area exposed to radiation at any point in the exposure cycle using the panoramic method, amounted to less than one square inch. During exposure by conventional methods, approximately 12 square inches of skin area were irradiated during each individual film exposure and overlapping of irradiated areas is unavoidable during a series of films required for full-mouth radiography.

4. RESULTS

Levels of radiation measured at the points selected are graphically displayed in Figure 5. It will be noted that under the conditions of this experiment, the highest level of ionizing radiation during full-mouth radiographic exposure using the panoramic technic existed in the cervical lymphatic region of the neck and amounted to .42R. The highest levels of radiation when using conventional intra-oral radiographic technic, existed at the skin of the cheek and in the region of the thyroid gland, where 23 and 27 R, respectively, were recorded.

5. SUMMARY

Levels of ionizing radiation produced at points in and about a wax phantom of the human head, were measured during full-mouth dental radiography by a panoramic method and by a conventional 14-film intra-oral technic. The panoramic method was found to produce radiation levels approximately 1/50th as intense as those recorded at comparable points when using conventional dental radiographic technic.

6. ACKNOWLEDGEMENT

The authors wish to express their appreciation to the personnel of the Naval Medical Research Institute, Bethesda, Maryland, for their cooperation in furnishing the ionization chambers and in charging and measuring voltages on the chambers during this experiment. Lt. James W. Duckworth and Lt. (JG) Robert Sharp rendered most valuable service in performing the above mentioned technical procedures.

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3. Hasterlik, R. J., Argonne Cancer Research Hospital, Chicago, Illinois, (Personal communication).

Figure 1. Sievert ionization chamber, showing small size.

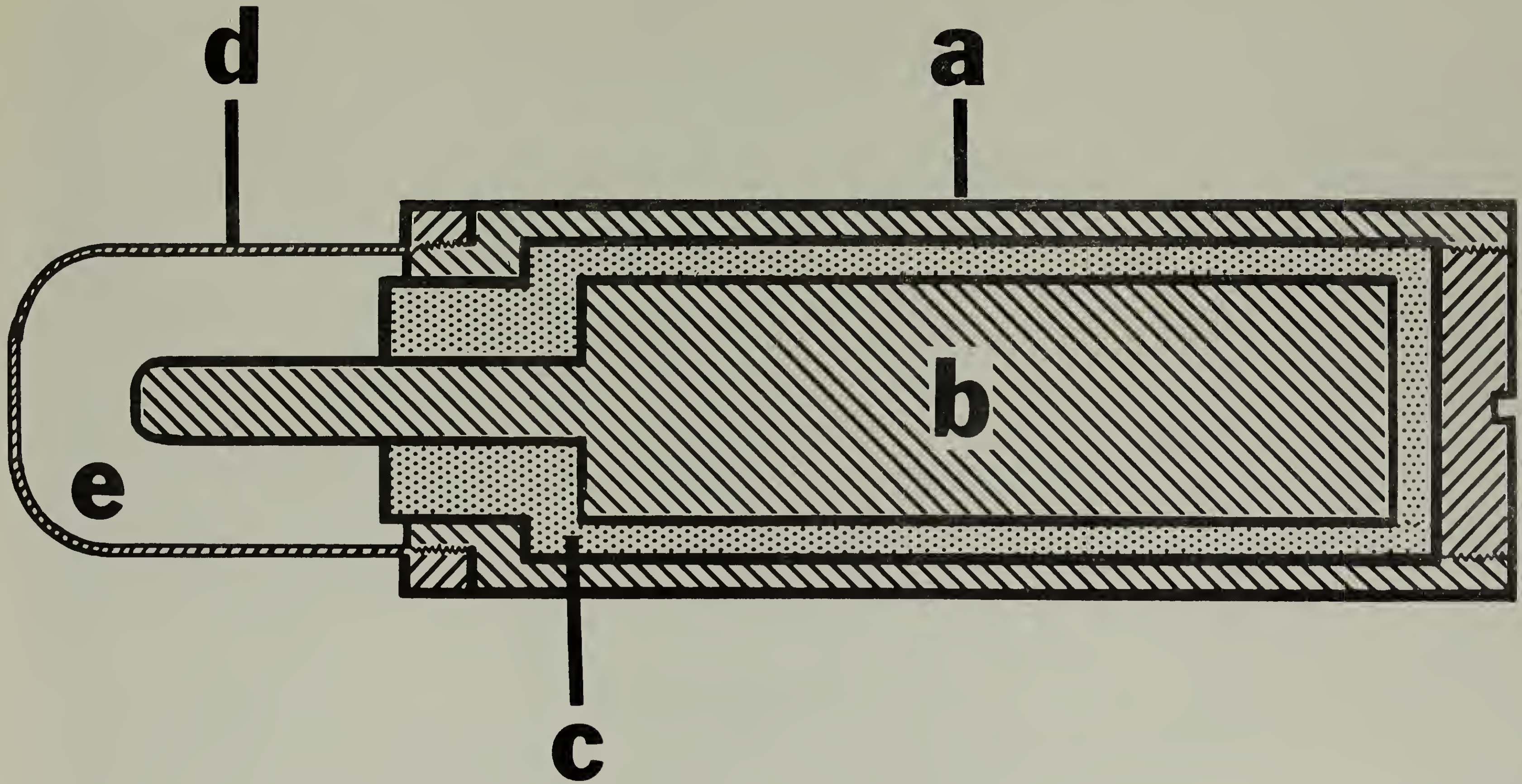

Figure 2. Sievert ionization chamber.
(a) Outer shell.
(b) Inner condenser element with antenna.
(c) Dielectric.
(d) Thin-walled cap.
(e) Air chamber.

Figure 3. Wax phantom showing placement of ionization chamber at skin of cheek and recessed location for measurements in region of neck lymphatics.

Figure 4. Wax phantom disarticulated for placement of ionization chambers at internal points.

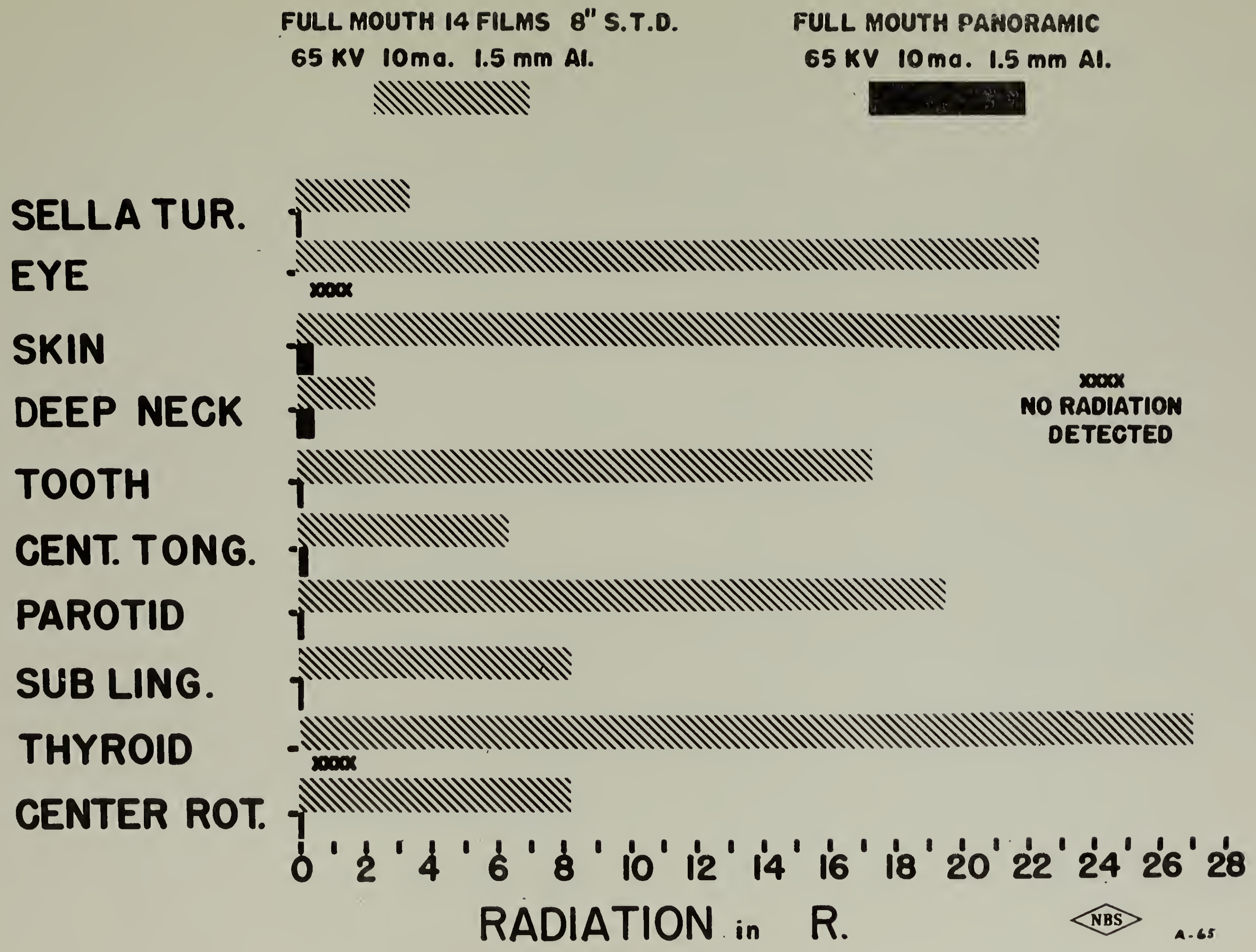

Figure 5. Chart showing levels of radiation measured at 10 selected points in and about the human head phantom, using a conventional and panoramic full-mouth radiographic technic.

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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 front cover.

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