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MAGNETIC HEAD/TAPE INTERFACE STUDY FOR SATELLITE TAPE RECORDERS

Technical Report
Volume III

IITRI Project No. E6134
Contract No. NAS5-11622

Goddard Space Flight Center
Greenbelt, Maryland

REPRODUCED BY
NATIONAL TECHNICAL
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SPRINGFIELD, VA 22161

IIT RESEARCH INSTITUTE

Technical Report

Volume III

Test Methods and Procedures

for

Magnetic Head/Tape Interface Study for
Satellite Tape Recorders
(1 December 1968 - 31 December 1970)

Contract No. NAS5-11622

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Greenbelt, Maryland

February 1971

NOTICE

THIS REPORT CONTAINS THE RESULTS OF TESTS DESIGNED TO ISOLATE THE EFFECTS OF HEAD, TAPE, AND OPERATIONAL PARAMETERS ON THE PERFORMANCE OF SATELLITE TAPE RECORDERS. BECAUSE OF CERTAIN UNIQUE REQUIREMENTS FOR SATELLITE RECORDERS, THE RESULTS ARE NOT DIRECTLY APPLICABLE TO CONVENTIONAL, GROUND BASED, COMMERCIAL OR HOME RECORDING SYSTEMS. THE INTENT OF THE PROGRAM WAS TO DEVELOP SOUND ENGINEERING TECHNIQUES FOR SELECTING AND USING TAPE IN SATELLITE APPLICATIONS IRRESPECTIVE OF MANUFACTURER, BRAND NAME, CATALOG NUMBER, OR THE APPLICATION FOR WHICH THE TAPES WERE DESIGNED. THE TEST RESULTS REPORTED SHALL BE CONSIDERED VALID FOR THE INDIVIDUAL REELS OF TAPE EVALUATED. NO PORTION OF THE TEXT OR DATA SHOULD BE INTERPRETED AS AN ENDORSEMENT OR CRITICISM BY IITRI OR NASA OF ANY COMMERCIAL PRODUCT OR MANUFACTURING ORGANIZATION.

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TEST METHODS AND PROCEDURES

SECTION I

INTRODUCTION

This is Volume III of a three volume Technical Report on IIT Research Institute Project No. E6134, entitled "Magnetic Head/Tape Interface Study for Satellite Tape Recorders." The objective of this program was to evaluate between the various physical and chemical factors involved in the interaction between heads andtape on unattended, satellite tape recorders, and to establish relationships between these parameters and the ultimate recorder performance. To achieve this goal, an extensive experimental and analytical program was carried out to investigate the effects of the factors involved. The results of these investigations are reported in Volume II. Those parameters found to be particularly significant in the determination of final performance were utilized in the form of guidelines for the selection and use of tape and heads for satellite recorders. These guidelines, along with the appropriate test procedures, are reported in Volume I.

This volume is a discussion of the methods and procedures involved in the characterization of tapes and in the tests leading to the conclusions drawn in Volume II. Section II of the volume describes an extensive chemical analysis conducted to characterize the binder formulations of a number of tape types. This analysis was required to identify the types of polymers used inthe fabrication of magnetic tape, as well as the quantities of additives incorporated. Such information was not found to be generally available to most satellite recorder manufacturers.

Section III of the report is a discussion of the tape types selected for the evaluation of tape related parameters throughout the testprogram. In addition to the chemical

analysis, certain other physical and mechanical properties of these tapes were measured. The procedures and results of these tests are reported. Section IV is a description of the two types of tape transports used throughout the test program.

The final section is a summary of the types of tests used to determine the adhesion and debris performance of the heads and tapes investigated. Test procedures are discussed, and sample results are presented.

SECTION II

CHEMICAL INVESTIGATIONS OF MAGNETIC TAPE BINDER-SYSTEMS

A. Introduction

Using instrumentation magnetic tape for data acquisition imposes a number of requirements on the properties of organic resin binders, used as vehicles for magnetic powders, such as acicular gamma iron oxide or chromium dioxide. In order to impart the desired magnetic properties to the tape, one important prerequisite is the capability of the binder to accommodate a relatively large proportion (70% by weight) of magnetic pigment. At the same time, keeping up the dispersed pigment in a well bonded state to minimize the probability of the removal of magnetic oxide from the tape coating is necessary. Requiring satisfactory bond conditions between the pigment and resin in tape coatings makes the application of organic polymers with functional groups potentially capable of interacting with selective sites on the pigment surface desirable. Thus polar resins, such as polyurethanes, epoxies, and the less polar polyvinyl acetates, have found use in binder formulations of magnetic tapes.

The benefits gained from resins having a more pronounced polar nature, particularly those that can be cross-linked during curing, relate also to the relative thermal stability of these resins. The temperature resistance of magnetic tape binders is important as locally heated areas at the tape surface during traverse across recording heads could contribute to the thermal degradation of the binder. This degradation could result in the loss of mechanical integrity of the pigment-binder system and lead to adhesion and "stickion" problems at the head/tape interface.

In instrumentation tapes intended for use in satellite recorders the requirement of binder stability becomes more critical than in ordinary magnetic tape applications. The reason is that the degradative changes which might occur during the remotely-controlled use of the tape could lead to operational problems, resulting in irreversible failure of the recording system. The difficulty of selecting an instrumentation magnetic tape for satellite recorders from commercially available tapes is a result of the

1. Limited demand of tapes for space applications,

2. Relative adequacy of the performance of instrumentation tapes under normal (terrestrial) conditions of use, and

3. Highly competitive and proprietary nature of the manufacturing phase of magnetic tape, which restricts dissemination of technical information by manufacturers.

This limited exhange of data between fabricator and tape user makes it difficult to adapt an available magnetic tape for satellite recorder use by eliminating those ingredients in the coating formulation that might adversely affect the performance of the tape in space. Accordingly, efforts to select an instrumentation tape for satellite recorders from a series of those commercially available must rely on data derived from a comparative study of mechanical, electrical, and chemical properties of the tapes considered. Chemical characterization studies of coating systems are, in many instances, hampered by the relative intractability of cross-linked binders and the inapplicability of conventional solution analysis methods. Thus, selective preparative and instrumentation techniques must be utilized to obtain the desired information on the nature of organic constituents in magnetic tape coatings.

The chemical investigations in this study paralleled the mechanical and electrical tests and were conducted with due

consideration to the critical performance requirements of magnetic instrumentation tape for satellite recorders. These requirements related to the desirability of minimizing head/tape interaction, preserving the initial sliding conditions between head and tape over extended periods of tape use, and ensuring the integrity of the binder pigment system to minimize oxide shedding.

B. Physical-Chemical Aspects of Interfacial Head/Tape Problems

During the traverse of magnetic tape across the surface of recording heads problems might arise. These problems are closely related to physical-chemical effects insofar as friction and head/tape adhesion depend, to a great extent, on the inherent properties of the binder and the changes that occur in the binder during exposure to the environment and conditions of use. Thus, a polymer with a preponderance of hydroxyl (OH) or amino (NH_2) groups may exhibit a greater tendency toward interaction (friction, adhesion) with active sites on metal surfaces than a polymer devoid of such groups. This relationship becomes apparent when the static coefficients of friction for nylon 6.6 and steel ($\mu_s = 0.54$) and high density polyethylene and steel ($\mu_s = 0.35$) are compared under equivalent test conditions.[1] However, when a polymer undergoes surface changes as a result of oxidation or degradation-promoting conditions, its frictional behavior is also bound to change.

It has been shown that the adhesion of polyethylene to polar substrates (cellophane) can greatly be enhanced if the hydrocarbon film is subjected to surface oxidation.[2] Brief exposure of polyethylene to an oxidizing environment at 60°C increased the adhesive bond between the surface-modified plastic and the polar substrate from a very low value to 800 kg/cm^2. This increase in adhesion resulted in a sharp increase in the infrared absorption band intensities that are characteristic

of C-O and OH groups for the surface oxidized polymer. Because chemical relationships on interfacial conditions at solid-solid interfaces are important, further elaboration of these relationships is desirable for a better understanding of the magnetic head/tape problem in satellite recorders.

1. Frictional Effects in Polymers

It is well known that the kinetic energy of a body set to motion, when in contact with another body, diminishes with time because of friction. The loss of kinetic energy, dissipated to a great extent as heat, is a result of interfacial forces that act in a direction tangential to the dynamic force. Various theories have been advanced on the nature of friction for surfaces in sliding contact. One, proposed by Schnurrman,[3,4] is based on the concept of clusters of electrical charges of opposite polarity on adjoining surfaces. The electrostatic force field thus developed is believed to resist slippage of contacting surfaces until a tangential force sufficient to separate the charges to the breakdown potential is applied. When the local attraction at the interface is overcome, relative movement of the adjoining surfaces occurs in a macroslip process.

Another theory of friction, based on molecular concepts,[5] ascribes the presence of restrictive forces between surfaces in sliding to interactions of molecules at a separation distance near the minimum of the potential energy curve. The spatial distribution of these effectively interacting entities is such that the majority of them approaches the field of attraction, while a remainder assumes positions in the region of mutual repulsion. The energy expended in severing molecular associations at the interface is represented in the frictional resistance. The load carrying capacity of the substrate is attributed to the restoring force developed as a result of negative interaction of those molecules which enter into the repulsion field.

For high-molecular-weight organic materials, a welding-shearing theory of friction, proposed by Bowden and Tabor,[6] has gained wide acceptance. This theory explains the mechanism of friction in macroscopic terms, without essentially disagreeing with the previously advanced concepts. It postulates the dependence of frictional resistance to sliding on two main factors: (1) the force required to shear off irregularities at points of contact between two surfaces, and (2) the energy expended in plowing grooves in one material by the asperities of the other.

The overall frictional force, F, is thus represented by the sum of the shearing and plowing contribution

$$F = A\,s + P \qquad (1)$$

where the plowing force, P, for a spherical asperity of radius, r, can be given by:

$$P = 1/12\ (C^3\ p'/r) \qquad (2)$$

where

A = area supporting the load

s = shear strength of the asperities

C = chord length of the segment of the asperity engaged in plowing

p' = yield pressure of the material

Equations (1) and (2) suggest that the plowing term could dominate the frictional behavior of relatively soft and deformable plastics for which the depth of indentation by asperities (chord length) would become significant. This condition was experimentally verified in studies on unplasticized and dioctyl phthalate-containing polyvinyl chloride.[7] Differences in

frictional behavior of polymers treated with softening agents strongly suggest the interrelationship between surface and bulk mechanical properties of plastics. The possibility of such relationship was substantiated in studies on polyethylene that demonstrated the apparent equivalence of the coefficient of kinetic friction and the ratio of shear strength and yield pressure for this polymer.[8]

It is apparent from the foregoing considerations that a plasticized polymer coating or binder system that would become appreciably plasto-elastic at elevated temperatures could develop excessive frictional forces during sliding. For this reason, the pigment vehicle for magnetic coatings of instrumentation tape is usually chosen in order to have good thermal stability, thus minimize viscous effects in locally heated areas. Such effects would enhance friction through plowing or increase the interaction between surfaces in intimate contact, according to the molecular concepts of the friction theory.

2. Adhesion

The adhesion of polymers, such as represented in organic binder systems of magnetic coatings, to solids is often considered a result of van der Waals interactions. However, other forces, notably ion-dipole,[9] ion-ion[10], and hydrogen bond[11,12] have been responsible for the adhesion of resins to metal and other surfaces. Poor contact on a molecular level minimizes the interaction (adhesion) of an organic compound with a solid surface by London forces, since the dispersion force field has an extremely limited range. (It is proportional to the seventh power of distance.)

The requirement for intimate contact between adherend and adhesive is less stringent for systems in which the interactions are of the dipole-dipole or electrostatic nature. Such interactions are believed to take place at metal surfaces. It has

been emphasized that good adhesive bonding of hydroxy- or carboxy-functional resins to metals is accomplished through interaction of these groups with the oxygen on the metal.[13] The interaction of hydroxyl group-containing surfaces with carboxy-functional compounds was postulated to take place through hydrogen bonding which was considered a special case of electrostatic interaction (interaction of proton donor and acceptor) in studies of the adhesion of polymethacrylic acid and methacrylamide derivatives to glass surfaces.[14] Although the formation of primary chemical bonds between substrate and polymer is not considered necessary for the formation of strong adhesive joints, the existence of such bonds has been demonstrated in desorption studies of vinyltrichlorosilane-treated glass surfaces.[15] Also, bonding of sulfur-containing natural rubber to copper is believed to proceed through formation of a chemical linkage (electrovalent bond) at the metal/rubber interface.

Although polymers can adhere to metal surfaces by any of the interactions suggested in the preceding discussion, catastrophic seizure of magnetic tape to a head surface may occur through an intermediate layer of organic material that could be deposited on it by the tape while in prolonged head/tape contact. A weld-type adhesive bond would then result at the head/tape interface. Polymer-to-polymer adhesion, responsible for this type of interaction, would most likely take place through a diffusion mechanism. The diffusion theory postulates the dependence of adhesion on the mobility of ends of macromolecules and the resultant interdiffusion and mechanical interlocking of sections of polymer molecules in adjoining surface layers, when these layers are brought into close contact.[16]

To accomplish this, the polymer must be maintained at a temperature above the glass transition point, where microbrownian motion is possible. The glass transition temperature requirement for polymer-polymer adhesion is well known in practice

where it has long been established that polymer films cannot be effectively sealed below a critical "fusion" temperature, regardless of the pressure applied. Since for low-molecular-weight compounds, the number of free chain ends is relatively high, the diffusion-related adhesion of these compounds is also high. Also, adhesion of low-molecular-weight fractions is favored by better orientation of molecular segments in the direction of diffusion, as suggested by Barrer.[17] These relationships have been confirmed in experimental studies which indicated that the adhesion of polyisobutylene[18] and polyisoprene[19] increased with decreasing molecular weight of these polymers.

Specific interactions of polymers at reactive sites on solid surfaces with formation of adhesive bonds have been postulated in studies of the adhesion of diisocyanates to metals.[20] Here, the formation of a chemical bond between free isocyanate and hydrated metal oxide groups was considered responsible for the good adhesion of polymerized hexamethylene diisocyanate to steel, light metal alloys, porcelain and other substrates. In epoxide polymers, the ethylene oxide of hydroxyl functionality is credited with the adhesive capacity of these polymers in metal or ceramic bonding applications. Considering the above relationships in the perspective of the potential adhesive type failure of magnetic tape that remains in sliding contact with the surface of a recording head, the following conditions are favorable in reducing the susceptibility of a binder to seizure at the head/tape interface.

- The number of free functional groups in the cured binder should be minimized to reduce the probability of its interaction with metal surfaces. These groups may include polar functionalities such as iscyanate (-N=C=O), hydroxyl (-OH), carboxyl (-COOH), and primary or secondary amino groups ($-NH_2$, -NH) that could be present in incompletely reacted polyurethane, polyester, epoxy, or polyamide binder systems.

- Preferentially, well-polymerized, cross-linked binders should be employed to reduce the number of free polymer chain ends that could enhance diffusion-dependent adhesion.
- Thermally stable polymers should be used to minimize the degradation of the binder in sliding friction and thereby reduce the danger of formation of low-molecular-weight fragments that could promote adhesion at the head/tape interface.
- Binder additives that are inherently adhesive acquire adhesive properties or interfere with the polymerization of the binder should be eliminated.
- Only those lubricants that minimize head/tape interaction without affecting the structural integrity of the polymer (when used in acceptable amounts) should be employed in the binder.

The above factors are undoubtedly given consideration by the manufacturers in the formulation of magnetic coatings for instrumentation tape. However, under normal conditions of use, where no significant power or load restrictions are imposed on the tape transport, excessive friction and localized adhesive bonding at the head/tape interface will produce less serious problems than in unattended satellite recorders. It is in those special applications of instrumentation tape where manufacturers, having well developed coating formulations for commercial tapes, may tend to modify these formulations for other applications by introducing larger quantities of a certain additive (e.g., lubricant) without thoroughly evaluating the effects of this modification on other tape properties (coating integrity, completeness of binder polymerization, thermal stability). Consequently, the evaluation of this special tape becomes the responsibility of the user who, having no information about the coating ingredients in the furnished product (unless the formulation has been developed by him or according to his exact specifications), must find the task of determining the chemical and mechanical properties of tape challenging.

C. Experimental Methods Used in Investigations of Magnetic Tape Coatings

1. General Approach

In accordance with the relationships discussed in the preceding section, the potential malfunction of commercially available magnetic tapes in satellite recorders, as considered from a chemical point of view specifically relating to frictional and adhesive properties of the tape, was studied by selected analytical methods. These methods were used to discriminate between different coating systems of magnetic tapes in a manner that would permit correlation of the measured properties with the performance ratings of the tapes used. The properties investigated included the following:

- Content of organic and inorganic constituents in magnetic coating, with a view toward possible insufficiency of the binder to accommodate excessive amounts of pigment.
- Integrity of the magnetic coating as related to the binder solubility and extractibility of additives by selected solvents.
- Presence of adhesion- and friction-promoting functional groups in the binder, as ascertained by infrared spectrophotometric methods.
- Thermal stability of the binder, determined by thermogravimetric and differential thermal analytical methods.
- Presence of volatile or volatilizable constituents in the magnetic coating as indicated by gas chromatographic measurements.
- Sensitivity of magnetic coatings to moisture.
- Presence of corrosion-promoting constituents in coating that could induce changes in recording head surfaces thereby enhancing head/tape interaction.

These and other investigations of the chemical properties of magnetic tape coatings were performed as a result of the electromechanical studies on different tape types considered in this program. In those instances where the selected performance criteria indicated unusual tape behavior in loop or reel-to-reel tests, the tapes in question were studied more intensively with regard to binder properties (e.g., relative amounts of lubricant in binder). Those tapes considered inapplicable on the basis of performance data or others that appeared chemically equivalent were not completely evaluated according to the scheme presented above. Thus, a reasonable degree of flexibility was maintained in chemical characterization studies. This permitted adjustment of the effort according to the needs arising during this investigation.

2. Initial Studies, Procedures and Applications

The utilization of different polymers as binders for magnetic tape coatings, e.g., polyester-based polyurethanes[21] which represent a binder type used quite extensively in instrumentation tapes, polyamide-polyesters,[22] vinyl chloridevinyl acetate resins,[23] and the application of binder additives such as hydrocarbons[24] or stearic acid[25] lubricants and antistatic agents,[26] magnifies the problems of component characterization in a heterogeneous system, such as represented by magnetic tape.

One of the major problems encountered in the analytical procedures of characterizing the constituents in magnetic tape is the presence of some components in small amounts in comparison with the bulk quantities of the main binder polymer, substrate, and inorganic pigment. Consequently, the separation of the magnetic coating (binder, binder additives, magnetic oxide) from the tape substrate (in most instances, polyethylene therephthalate) is necessary before the organic ingredients in the coating can be isolated and identified through selective extraction methods.

a. Separation of Coating from Tape Substrate.

Initial attempts to separate the magnetic coating from the substrate by stripping with adhesive tape, crushing in liquid nitrogen and scraping with a scalpel proved unsuccessful because of the introduction of contaminants (from adhesive), failure of the coating to embrittle or detach from the substrate at low temperatures (liquid nitrogen), and the inefficiency of mechanical removal of the coating (100 mg/hr). Consequently, a more convenient method for the separation of the coating from the underlying film substrate was employed. This method involved immersion of the intact tape (240 feet) in 3l of 5% aqueous sodium hydroxide (NaOH) solution that was maintained at 50°C. After one hour (or less) the pigment-binder system was detached from the substrate by sliding the tape between the fingers of a latex-rubber-gloved hand (the glove was presoaked in 5% NaOH). The released coating was washed in cold water until the rinse appeared neutral to litmus paper. The 5% NaOH solution and the wash water were then neutralized with hydrochloric acid (HCl).

After the bulk of water was evaporated from the neutralized system, organic matter was extracted from the saturated salt solution with ether, to determine whether substantial amounts of organic matter were removed in the treatment of tape with NaOH solution. Infrared spectroscopic measurements, made on the ether extract of a commercial instrumentation tape (3M 888), indicated the presence of small amounts of hydrolyzed aromatic polyester that originated from the substrate or binder as suggested by C=O (1735 cm^{-1}), =C-O (1285 cm^{-1}) and CH_2O (1125 cm^{-1}) absorption bands. However, the bulk of binder and organic additives were retained by the detached coating; and since the extraction losses in 5% NaOH did not appear too significant, the removal of the magnetic coating from the substrate in alkaline solution was adopted for all tape types investigated.

b. Extraction of Organic Constituents from Tape Coating. A Soxhlet extraction procedure was used to remove the organic constitutents from the magnetic tape coating that was detached from the substrate by the previously described separation method. In order to determine the effectiveness of the extraction process, a series of solvents ranging in solubility parameter values (partition eluent strength) from 7.3 (n-hexane) to 23.4 (water) was used. The amounts of organic material extracted from the coating of a commercial instrumentation tape (3M 888) by different solvents over a period of 24 hours are shown in Table I.

The extraction measurements indicated the relative paucity of nonpolar (hexane-extractable) and highly polar, water-extractable constituents, since the bulk of organic material that could be recovered from the coating was eluted with solvents of intermediate partition eluent strength. Therefore, chloroform and mixtures of chloroform and methanol were used to isolate the less and more polar binder components in solubility and separation studies of magnetic tape coatings, as described in Section II.D.

c. Saponification of Esters in Magnetic Tape Coating. The preliminary infrared spectroscopic evidence of the polyester-urethane nature of several binder systems which was also corroborated by the color test in the reaction of the coating with p-diaminobenzaldehyde[27] suggested saponifying the binder to insolate the coating ingredients better. The reaction was performed by a method closely paralleling the procedure used in thin layer chromatographic (TLC) analyses of polyurethanes.[28] A 1-g sample of the magnetic coating was placed in a round-bottom flask in 25 ml 1N sodium methoxide solution in methanol. After refluxing for five hours, the residue containing (in addition to the insoluble iron oxide pigment and carbon) sodium salts or dicarboxylic acids (if present) was filtered off. The

Table I

AMOUNT OF MATERIAL EXTRACTED FROM TAPE COATING
BY DIFFERENT SOLVENTS

Solvent	Solubility Parameter	Amount Extracted percent/weight of coating	Remarks
n-hexane	7.3	0.66	
Cyclohexane	8.2	1.21	
Toluene	8.9	4.36	
Chloroform	9.3	4.67	
Acetone	9.9	4.54	
1, 2 dichloroethane	9.9	4.30	
Tetrahydrofuran		4.66	
Dioxane	10	6.13	
Methanol	14.4	4.14	
M-cresol		(14.5)	Part of coating lost in transfer
Dimethyl formamide	12.1	(15.6)	Part of coating lost in transfer
Water	23.4	1.12	

residue was then treated with a 25% aqueous solution of ammonia to dissolve any possible salts of adipic, phthalic or other dicarboxylic acids. The filtrate of the saponified products was acidified with hydrochloric acid, and an attempt was made to separate diamines from glycols by chloroform extraction. The isolation of saponification products is shown diagrammatically in Fig. 1.

In an attempt to identify the isolated fractions, infrared spectrograms were obtained for some of the residues and extracts as indicated in the diagram (IR #1 through #5). Although hydroxyl- and amino-functionalities were suggested in compounds of the saponification filtrate, indicating the glycol and diamino-origin of the absorption bands, a clear identification of the only partially separated binder constituents, or rather their derivatives, by infrared methods was not possible. In an effort to achieve better separation of the binder constituents, thin layer chromatographic methods were employed in a subsequent study.

d. Thin Layer Chromatographic Studies. To obtain information about the heterogeneity of the compounds isolated from the saponified products of the magnetic coating, these compounds were subjected to TLC analysis. Plates were prepared by dispersing 25 g of Silica G (5 to 15 μ) in 50 ml distilled water, spreading the dispersion with a doctor blade at 0.36 mm clearance and drying the plates first in air for 24 hours and then in an oven for 30 minutes at 120°C. The separated products, recovered in fractions 1, 3, and 4 (Fig. 1) were deposited and subsequently eluted from the plates with a solvent system containing 35 p/v n-propyl alcohol (PrOH), 15 p/v ethyl alcohol (EtOH) and 50 p/v ether (EtO). After the plate was fixed by spraying with a solution containing 95 ml 0.2% ml 0.2% ninhydrin in water and 5 ml 1% acetic acid, fractions 3 and 4 revealed the presence of 3 and 4 components, respectively. Fraction 1

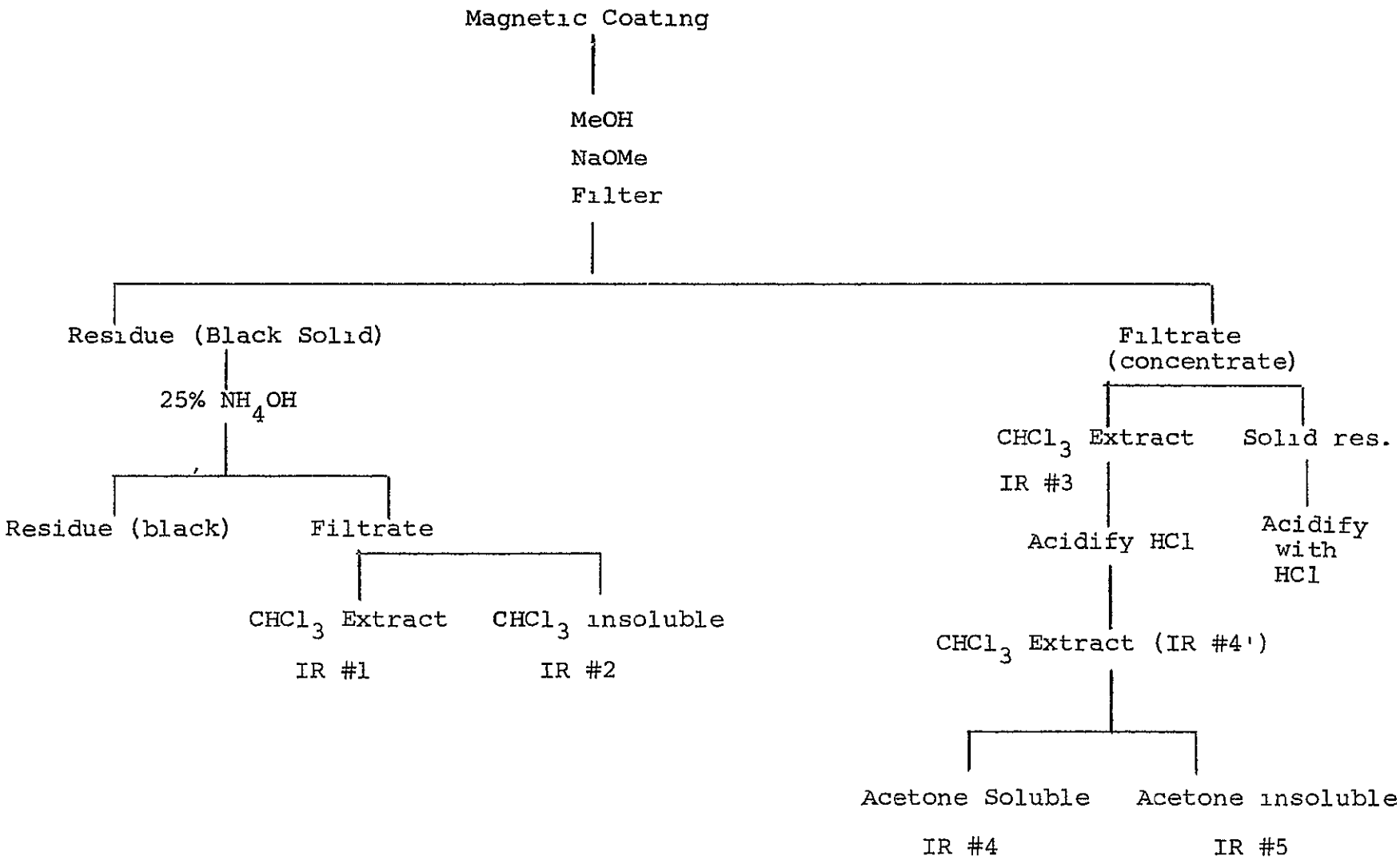


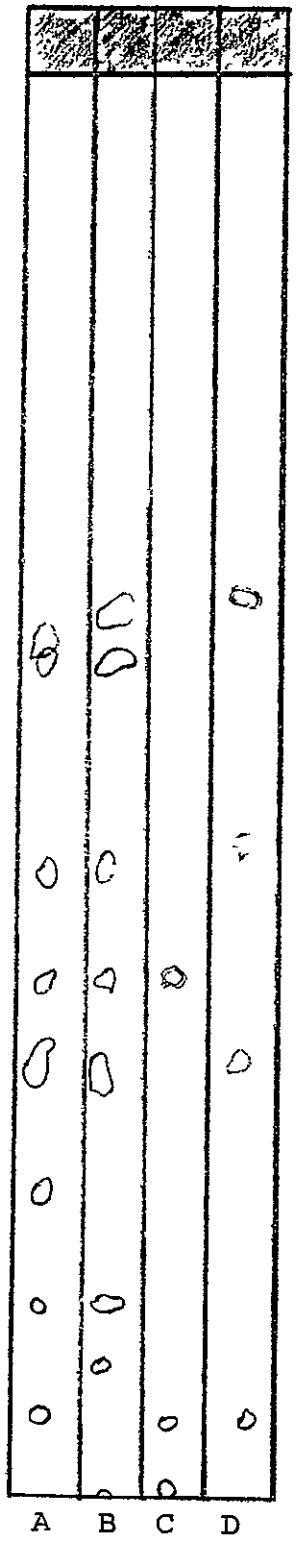
Fig. 1 SEPARATION OF CONSTITUENTS IN SAPONIFIED COATING

exhibited a fast-migrating constituent that was not well resolved. Obviously, the products obtained in the saponification process represented by no means single constituents, which accounted for the difficulty of identifying the "isolated" compounds by infrared methods.

The separation of binder ingredients in a magnetic coating of instrumentation tape (3M 888) by TCL methods was subsequently performed on residues obtained in Soxhlet extracts with methanol (MeOH), chloroform ($CHCl_3$), dioxane and toluene. From the several elution systems employed, including benzene:acetic acid: MeOH (45:4:8), $CHCl_3$:acetone (96:4) and $CHCl_3$:MeOH in different proportions, the 90:10 $CHCl_3$:MeOH solvent produced best separation. The results shown in Fig. 2 indicate the extractibility of more than six components with dioxane or $CHCl_3$ solvents. The volatility of $CHCl_3$ in comparison with dioxane, its intermediate partition eluent strength, and good solvent action qualified chloroform as an extraction solvent for the isolation of binder ingredients in magnetic tape coatings.

With regard to the identification of TLC-isolated binder constituents, it was soon realized that infrared methods proved inadequate because of the relatively small amounts of compounds available for analysis in each of the separated components. Consequently, a preparative technique, based on liquid column chromatographic separation methods, was used to obtain larger quantities of binder components for instrumental analysis.

e. Isolation of Binder Components by Liquid Layer Chromatographic Methods. The need of a larger quantity of compounds isolated from the coating for analysis made it necessary to use LCC methods for the preparation of samples. A chromatographic separation column was prepared by introducing 3 g of desiccant-activated 200-325 mesh-screened silica gel



Soxhlet Extracts:

A Chloroform
B Dioxane
C Toluene
D Methanol

Fig. 2 ELUTION OF BINDER CONSTITUTENTS FROM MAGNETIC COATING BY TLC METHOD

(Davison Chemical Division, W. R. Grace and Company) into a 70 cm-long 6 mm-diameter Pyrex tube. Specimens of 100 g, obtained by evaporating the solvent from the $CHCl_3$ extract of the magnetic coating, were placed in a well divided state at the top of the column. Eluent, consisting of $CHCl_3$ and MeOH in proportions of 100:0, 99:1, 98:2. 95:5, 90:10, 75:25, 50:50 and 0:100, was introduced into the column in 10-ml portions. The 10-ml fractions were collected separately at least four times for each $CHCl_3$:MeOH mixture, thus providing 35 to 40 specimens that represented organic material recovered from the column by solvents of increasing polarity. After evaporation of the liquid from the fractions in a stream of dry nitrogen at 25°C, the organic residue was weighed in each collected vial, and if necessary, several consecutively eluted samples were combined to provide enough material for infrared spectroscopic identification. The results obtained are described in Section II.D.

f. Infrared Spectroscopic Measurements. Initial attempts to identify organic material at the tape surface by the multiple internal reflection (MIR) technique, without resorting to the cumbersome method of isolating and separating the organic matter from tape coatings, proved unsatisfactory. An excessive amount of infrared radiation was lost by scattering from the microscopically rough tape surface. This, combined with the small quantities of organic material in the coating in comparison with the pigment constituent, resulted in a very poor infrared spectrum. Much better spectra were obtained by applying the organic material from solution to sodium chloride (NaCl) windows, or by using the potassium bromide (KBr) pellet technique, particularly for specimens that were insoluble or those that formed powder residues after solvent evaporation. The KBr pellet method was also used for identification of organic materials that were transferred from the tape to the

head surface in loop tests conducted over an extended period of time. The very thin layer of "varnish" was removed from the brass head by polishing it with KBr powder, and the collected abrasive was molded into a pellet for infrared analysis. Unfortunately, the amount of material thus recovered was insufficient to permit analysis of the sample beyond identifying its organic origin from the symmetric and asymmetric stretching modes of the $-CH_2-$ group at 2850 cm^{-1} and 2930 cm^{-1}, respectively.

A much thicker deposit of organic matter on a head surface, transferred from an instrumentation tape (3M 888) to brass during prolonged contact in a reel-to-reel test, was identical (except for some signs of oxidation) with the coating extract obtained from this tape. The infrared measurements for identifying this deposit were performed on a film of the organic material that was cast from $CHCl_3$ solution on NaCl flats.

In preliminary investigations of coating extracts obtained from several magnetic tapes of one commercial origin (3M Company), the use of a polyester-modified urethane binder was strongly suggested. The pertinent infrared absorption bands appeared at 3300 cm^{-1} (N-H stretching frequency), 1540 cm^{-1} (Amide II band), 1510 cm^{-1} (N-H bending mode), 1730 cm^{-1} (C=O stretching frequency) and 1225 cm^{-1} (=C-O stretching mode). An infrared spectrogram of a $CHCl_3$ extract of a computer tape coating (3M 777), shown in Fig. 3, exemplifies the polyester-urethane nature of this tape. A spectrogram obtained for a commercial polyurethane prepolymer (Spenkel P49-60-CX, Spencer Kellogg Company) shows the similarity between this compound (Fig. 4) and the tape extract. However, the missing isocyanate absorption band at 2230 cm^{-1} in the urethane binder of the magnetic tape (which is present in the prepolymer) suggests the completeness of the reaction of residual isocyanate groups in the binder, as far as infrared spectroscopic measurements are capable of discerning. This is very different in a polyisocyanate, where the NC=O-

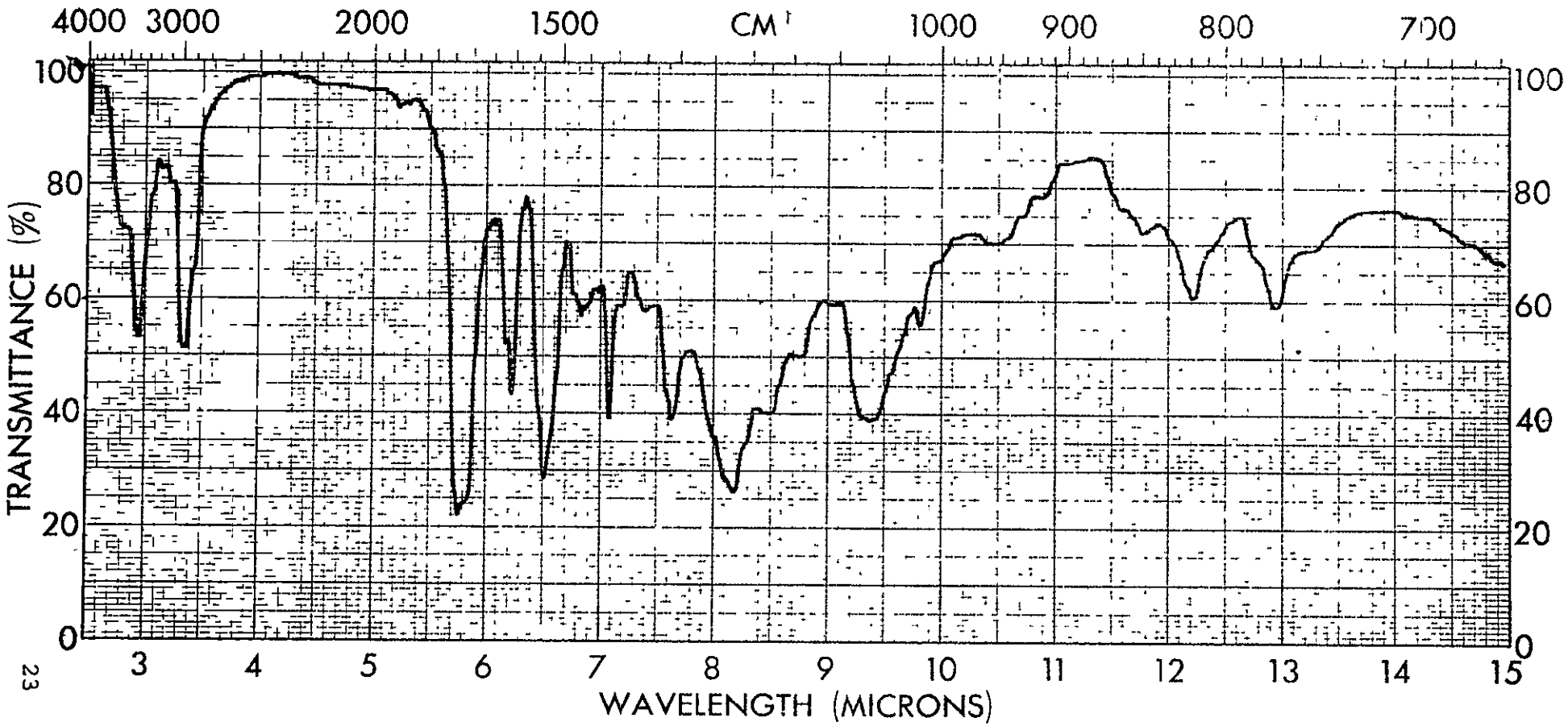


Fig. 3 INFRARED SPECTRUM OF A POLYURETHANE-BASED MAGNETIC TAPE BINDER

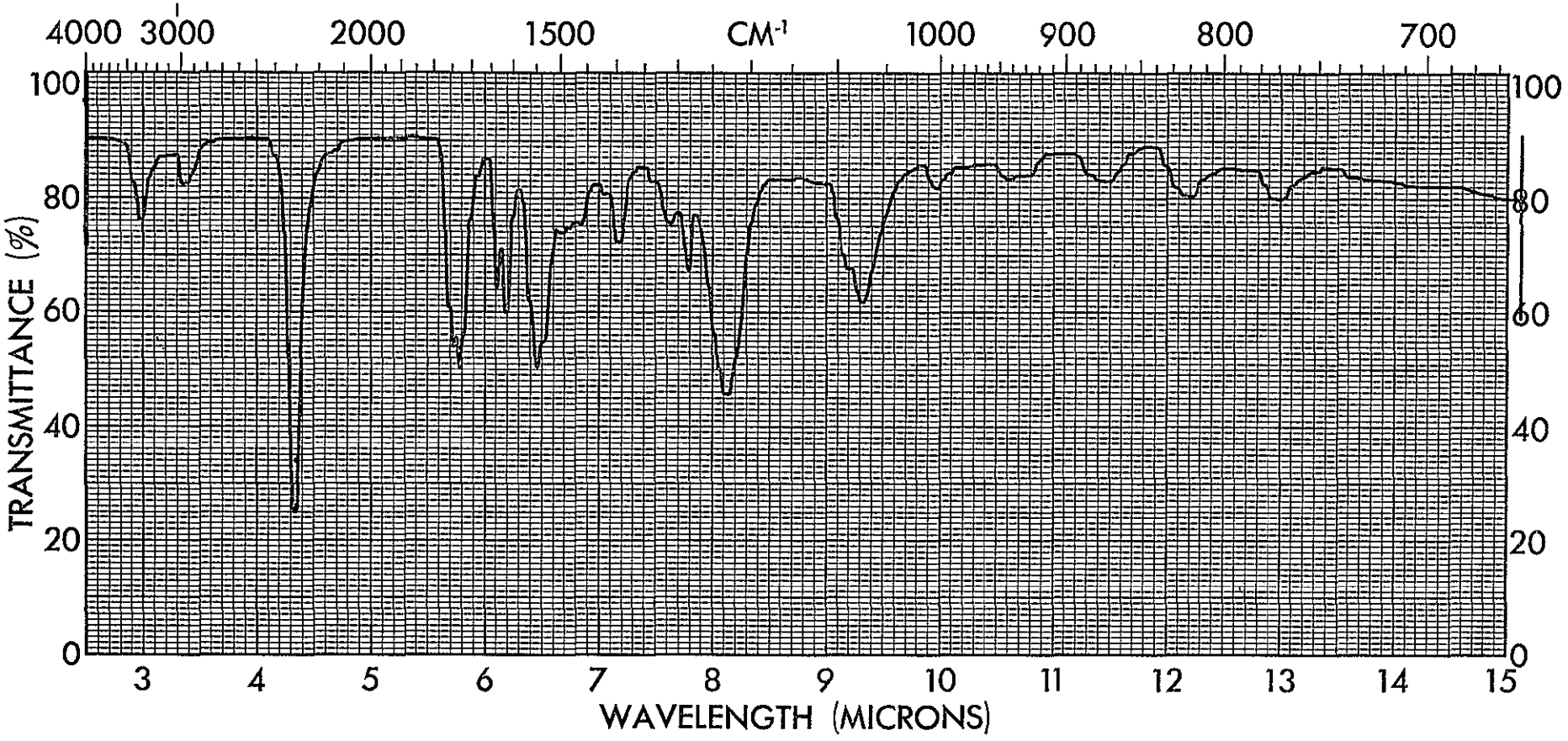


Fig. 4 INFRARED SPECTRUM OF A COMMERCIAL POLYURETHANE PREPOLYMER

associated band frequency is quite prominent in a commercial product (Mondur MRS, Mobay Chemical Company) as shown in Fig. 5. The undesirable effects of residual polar groups in magnetic tape binders with regard to potential adhesion have been discusses previously. Thus, the absence of free isocyanate functionalities in urethane-based binders for magnetic tape coatings appears important. The question as to whether infrared methods are capable of detecting residual isocyanates in trace amounts (less than 0.5%) has led to the use of microanalytical techniques in studies conducted elsewhere.[29]

In the preliminary infrared studies, a small sodium chloride spectrophotometer (Perkin-Elmer 137) was used for the general characterization of tape extracts. Later, a grating spectrometer (Perkin-Elmer 621) was employed to resolve better the absorption bands of relatively similar binder systems.

g. Thermoanalytical Investigations. In an effort to determine the applicability of thermoanalytical investigations of tape coatings, thermogravimetric (TGA) and differential thermoanalytical (DTA) measurements were performed on an instrumentation tape (3M 888). Three types of specimens, ranging in size from 75 to 200 mg (to account for differences in the amount of organic matter) were employed: (1) intact tape, (2) coating detached from substrate, and (3) substrate detached from coating. The DTA and TGA experiments on samples subjected to an oxygen (O_2) and argon (Ar) atmosphere were programmed at a heating rate of 6°C/minute for a temperature range of 25 to 600°C.

Figure 6 shows the much greater susceptibility of the magnetic tape coating to thermal degradation in oxygen in comparison with argon. The thermograms obtained for intact tape are not representative of the behavior of the binder since the response of the organic constituents in the magnetic coating is very obscured by the (polyester) substrate. Thus,

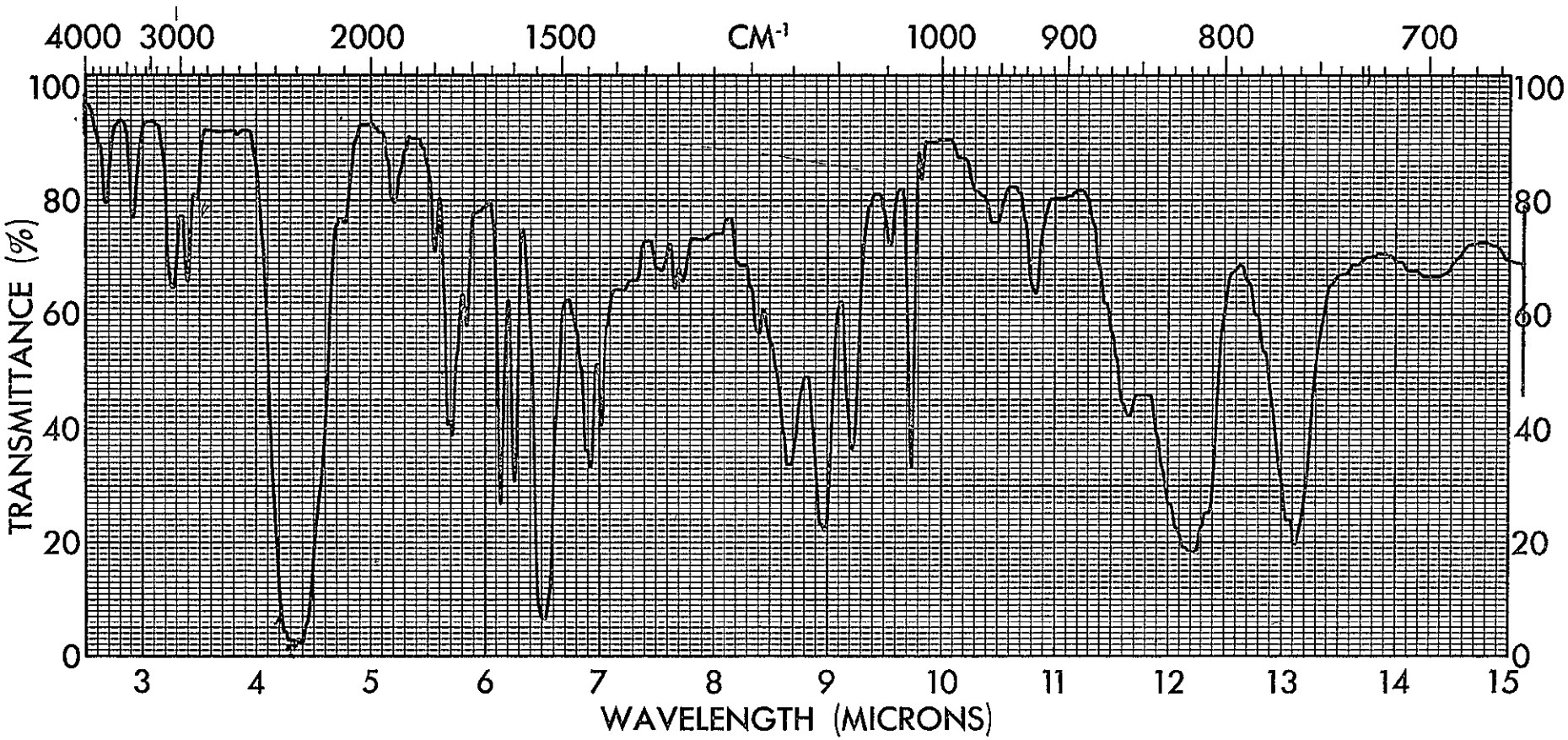


Fig. 5 INFRARED SPECTRUM OF A COMMERCIAL POLYISOCYANATE

more meaningful thermoanalytical information for magnetic tape would be obtained from TGA and DTA measurements performed on the detached coating than from the intact tape. However, even for the coatings, data may be misleading in those instances where the binder contained carbon as a conductivity-increasing additive. Here, the oxidation of finely divided carbon may produce weight losses, suggesting the thermal instability of the binder, as indicated in Fig. 6 for the TGA-grams of the coating in comparison with the polyester substrate. The apparent inconsistency of the thermograms obtained in O_2 and Ar where, in an inert environment, the binder exhibits better thermal stability than the substrate, points to carbon as the cause of observed effects. The DTA-grams obtained for the coating and substrate in oxygen indicate early exotherms at 220 and 300°C, respectively. Other, hear evolving reactions in the presence of O_2 take place at 450°C and 590°C for the magnetic coating, while for the substrate, the corresponding exotherm peaks appear at 480 and 570°C. Much less pronounced changes were observed in tapes that were heated in argon. Here, endotherms at 260 and 480°C, indicative of a possible phase transition and polymer decomposition, respectively, indicate the relatively good thermal stability of the magnetic tape in an inert environment. Thus, the results of these tests suggested a preference for operation in an oxygen free atmosphere. This conclusion was not confirmed in later adhesion tests (Volume II), wherein frictional performance appeared to be independent of atmosphere.

Although DTA and TGA measurements appeared useful for gaining general information about the thermal behavior of magnetic tape, the method was not sensitive enough (possibly because of equipment limitations and the variability of specimens with regard to size and physical state) to differentiate between two polyurethane coatings of different origin. For this reason, thermoanalytical measurements were not performed on the entire series of tapes investigated in this study.

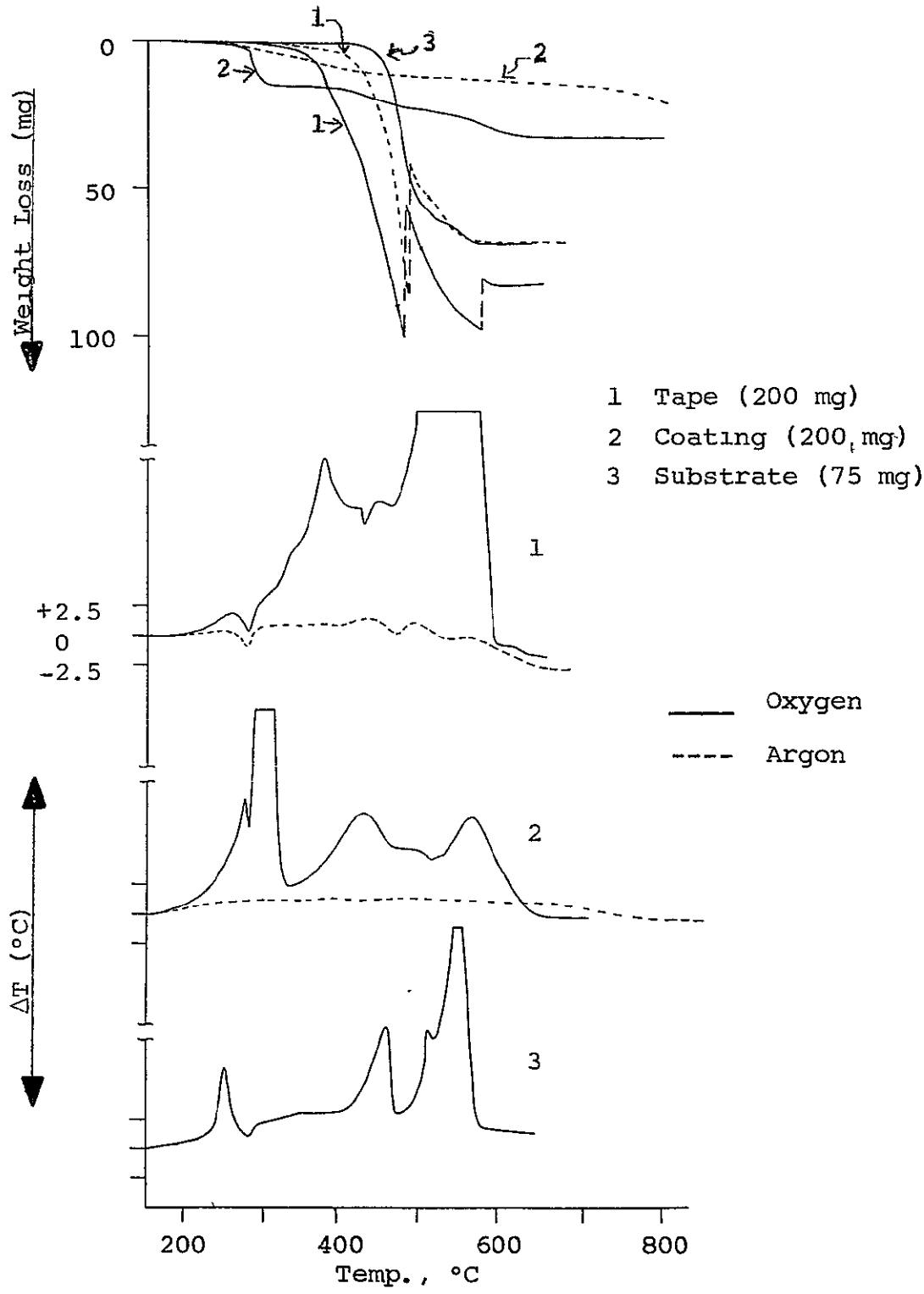


Fig. 6 TGA AND DTA MEASUREMENTS ON MAGNETIC TAPE

D. Experimental Results and Discussion

1. Characterization of Magnetic Tape Coatings

The magnetic tapes considered in this study included a series of instrumentation tapes (3M 888, 3M 951, 3M 551, 3M 871, Memorex 63L), some computer tapes (3M 777, Memorex Quantum, and DuPont Crolyn), and video tapes (3M 351 and Memorex 79L). One special tape (RCA 617) not marketed commercially was included.

In an effort to determine the relative amounts of organic and inorganic material in the tapes investigated, and thereby gain some information about the possible dependence of pigment shedding and head/tape adhesion, gravimetric measurements were made on the detached coating before and after combustion in a muffler furnace. The results, shown in Table II and presented graphically in Fig. 7, indicate the 70.4% to 83.6% range of the inorganic residue content in magnetic coatings. The high values of 83.2% and 83.6% obtained for two instrumentation tapes (RCA 617 and RCA 6050) may not be representative of the quality of magnetic oxide in the tape coatings since later studies indicated the presence of a significant amount of silicone lubricant (which would be oxidized to residual silica during combustion) in the binder of these tapes.

In general, it appears that instrumentation tape coatings have a greater pigment content than computer tapes. This might be related to the greater coating thickness of computer tapes and the desirability of reducing the susceptibility of these coatings to oxide shedding by increasing the binder content. The amounts of combustible organic material in the coatings of magnetic tapes in relation to the total weight of coatings are presented in Fig. 8. The greater proportion of organic binder in a computer (3M 777) and video tape (Memorex 79L) could suggest a lesser tendency of these tapes to lose pigment although this condition was not consistently demonstrated for the tapes investigated.

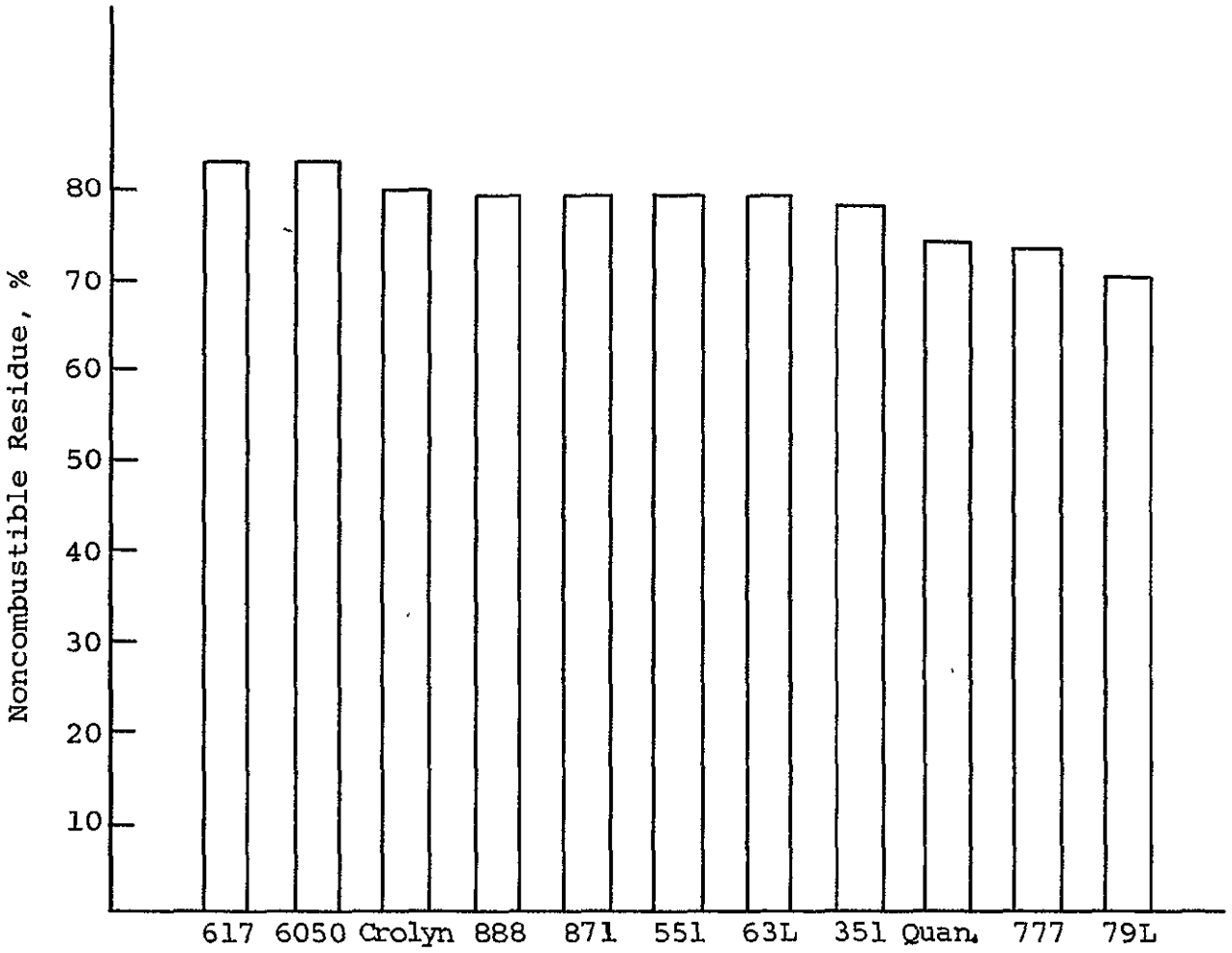


Fig. 7 NONCOMBUSTIBLE INORGANIC RESIDUE IN TAPE COATINGS

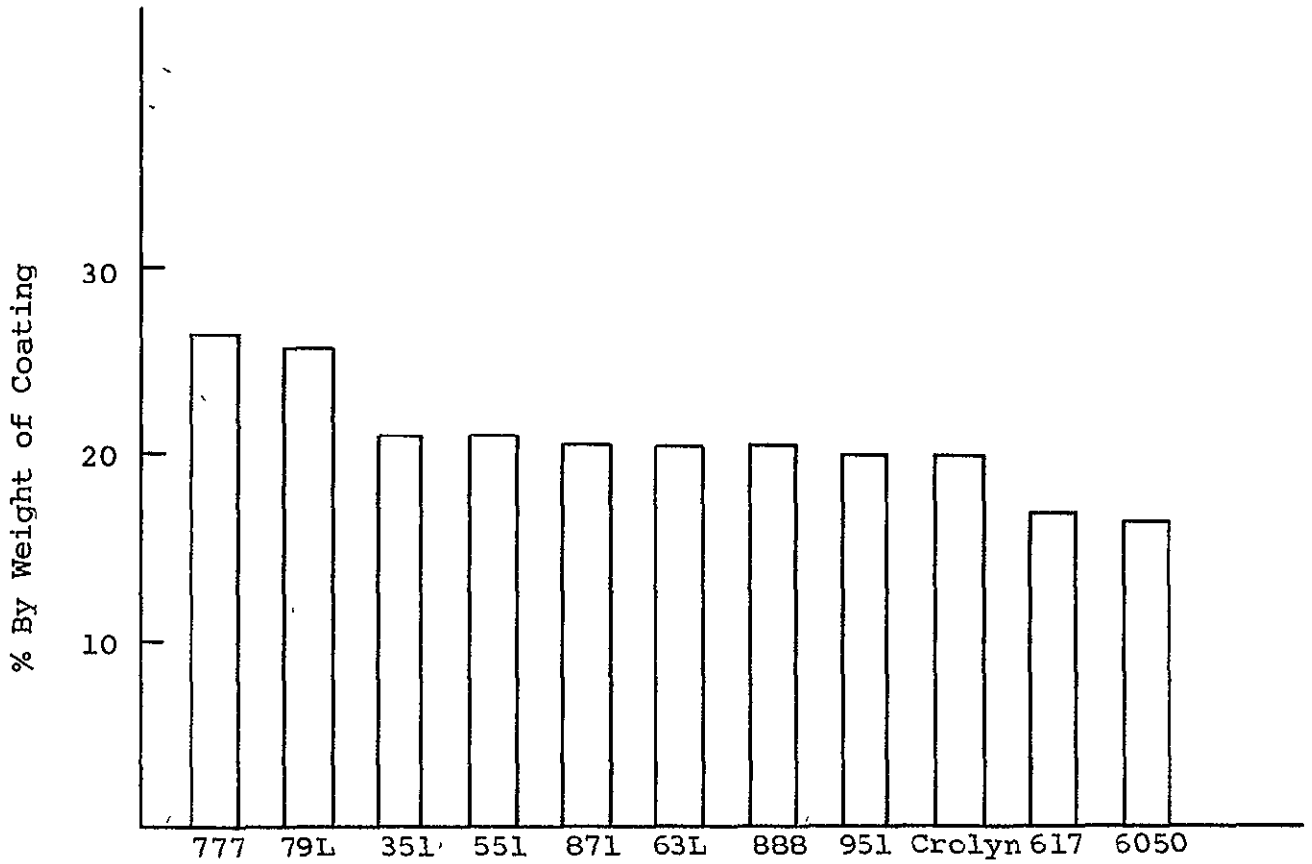


Fig. 8 ORGANIC MATERIAL IN COATINGS

Table II

AMOUNTS OF COATING, INORGANIC RESIDUE, AND CARBON-CONTAINING MATERIAL IN MAGNETIC TAPES*

Tape Identification	Amount of Material, Percent by Weight: Inorganic Residue Relative to Weight of Coating	Coating (Binder & Oxide) Relative to Weight of Tape	Organic Material Relative to Weight of: Coating	Tape	Unit Weight of Material g/ft: Tape	Coating
3M 888	79.6	26.2	20.4	5.3	0.172	0.0451
3M 951		27.6	19.9	5.4	0.169	0.0468
3M 351	78.6	26.7	21.4	5.7	0.162	0.0410
3M 777	73.5	33.7	26.5	8.9	0.301	0.1014
3M 551	78.5	24.7	21.1	5.2	0.165	0.0408
3M 871	79.5	31.7	20.5	6.5	0.182	0.0578
Memorex 63L	79.3	25.4	20.7	5.3	0.174	0.0442
Memorex 79L	70.4	26.9	29.6	8.0	0.163	0.0400
Memorex Quantum	74.1	33.8	25.9	8.8	0.290	0.0983
Crolyn	79.9	16.7	20.1	3.4	0.236	0.0394
RCA 617	83.2	42.5	16.8	7.1	0.214	0.0908
RCA 765	–	34.9	–	–	0.190	–
RCA 6050	83.6	38.9	16.4	6.4	0.205	0.0799

*Coating includes magnetic pigment, organic binder and graphite lubricant. Carbon-containing matter includes all combustible material.

In exploratory emission spectrographic investigations of elemental tape constituents, which were not continued because of the apparent insensitivity of the method due to large amounts of iron-oxide that obscured the spectrum, the (later disproved) presence of chloride appeared to be indicated for one instrumentation tape binder (3M 888). In order to confirm this finding and obtain parallel information about other tapes investigated, the coatings of these tapes were mechanically removed from the substrate (to prevent contamination by chloride-containing chemicals); they were subsequently subjected to flame treatment on a copper plate holder, according to the Beilstein method. The validity of the Beilstein test was checked in two instances where positive results were obtained, by precipitating silver chloride from hot nitric acid extract of the coating in a reaction with silver nitrate. The results of the chloride tests on coatings of magnetic tapes are shown in Table III.

The occurrence of chlorine-(Cl) containing compounds in magnetic tape coatings may derive from different sources, ranging from a binder additive or co-reactant such as vinylidene chloride[30] to residual amounts of polymerization retarders, such as p-nitro benzoyl chloride.[31] From the viewpoint of head/tape relationships, the presence of chloride-containing binder constituents may not be desirable, particularly in those instances where the decomposition of the binder may result in formation of active products (HCl) that could promote further degradation of the binder or corrosion of the recording head surface. During the investigations two tapes (RCA 617 and Memorex 79L), in which the presence of Cl was demonstrated, showed performance deficiencies. However, one tape (3M 871) that also gave a positive test for Cl, performed relatively well in loop and reel-to-reel tests. Thus, a simple relationship between halogen content in the binder and the behavior of tape in use could not be derived.

Table III

PRESENCE OF CHLORINE-CONTAINING COMPOUNDS IN TAPE COATINGS

Tape Identification	Test Result
3M 888	Negative
3M 777	Negative
3M 351	Negative
3M 871	Positive
3M 551	Positive
Memorex 63L	Negative
Memorex 79L	Positive
Crolyn	Negative
RCA 617	Positive
RCA 6050	Positive
Memorex Quantum	Negative

2. Characterization of Binder

The suspected dependence of head/tape adhesion problems on the nature of the binder system suggested a close examination of the organic compounds recovered after Soxhlet extraction and LCC separation in accordance with the methods described in Section II.C.

Infrared spectrograms obtained on extracts of several tape coatings from one manufacturing source (3M Company) suggested the polyurethane nature of the binder systems of these tapes. This is indicated in Fig. 9 (3M 888) where the peak at 3300 cm^{-1} corresponds to an NH stretching frequency. The absorption at 1535 cm^{-1} is due to a combination of NH bending and CH stretching modes, and the band about 1700 cm^{-1} represents a C=O amide stretching vibration. The ester carbonyl absorption frequency at 1730 cm^{-1}, accompanied by stretching bands at 1230 cm^{-1} (C-O) and 1420 cm^{-1} (CH_2-C=O), suggest the ester modification of the urethane polymer. An absorption band at 1600 cm^{-1} is indicative of the presence of aromatic compounds in the binder. The aromaticity of the binder is also supported by an absorption peak at 825 cm^{-1} (in-phase, out-of-plane wagging vibration of adjacent hydrogen atoms or an aromatic ring) which suggests the presence of para-substituted compounds, such as might result from reactions of p-phenylene diisocyanate, according to investigations conducted elsewhere.[32]

The relative similarity of the instrumentation tape (3M 888) binder and that of a computer tape (3M 777) was noted in subsequent studies (Fig. 10a). However, closer analysis of the band spectrum in the far infrared range suggested a lesser preponderance of para-substituted compounds in the binder of the computer tape, as suggested from the relative intensities of the absorption peaks at 820 and 760 cm^{-1}. Also, the extract of the computer tape contains more heavily substituted aromatic compounds, as indicated by the presence of an absorption band at 850 cm^{-1} for the 95/5 $CHCl_3$/MeOH LLC fraction (Fig. 10b). These compounds could possibly be responsible for the more aromatic character

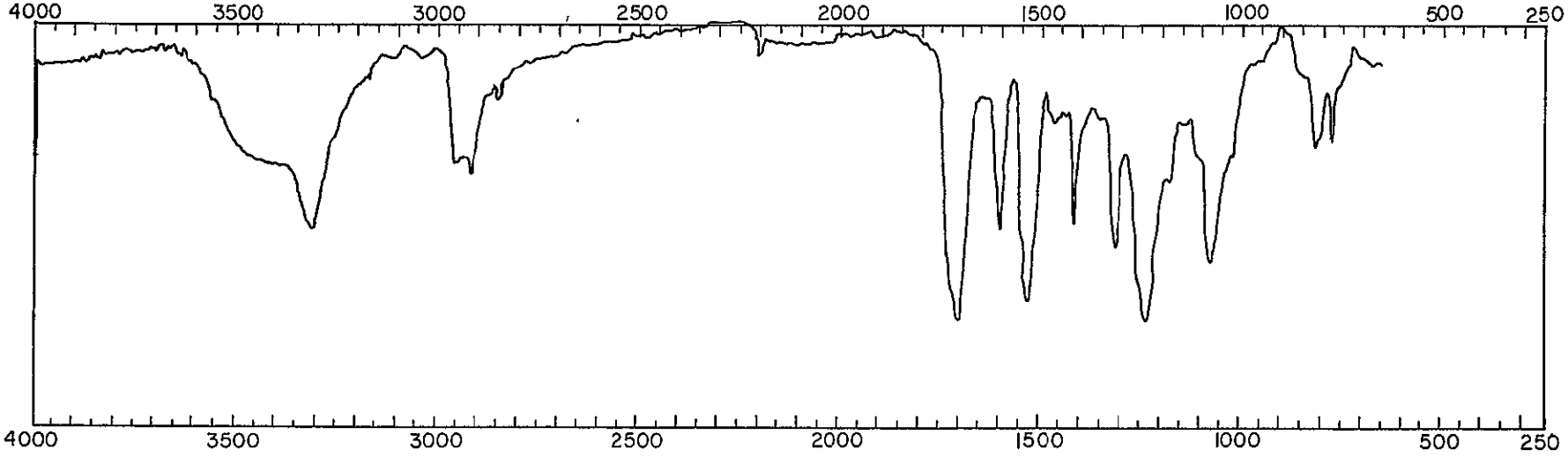


Fig. 9 INFRARED SPECTROGRAM OF 3M 888 INSTRUMENTATION TAPE COATING

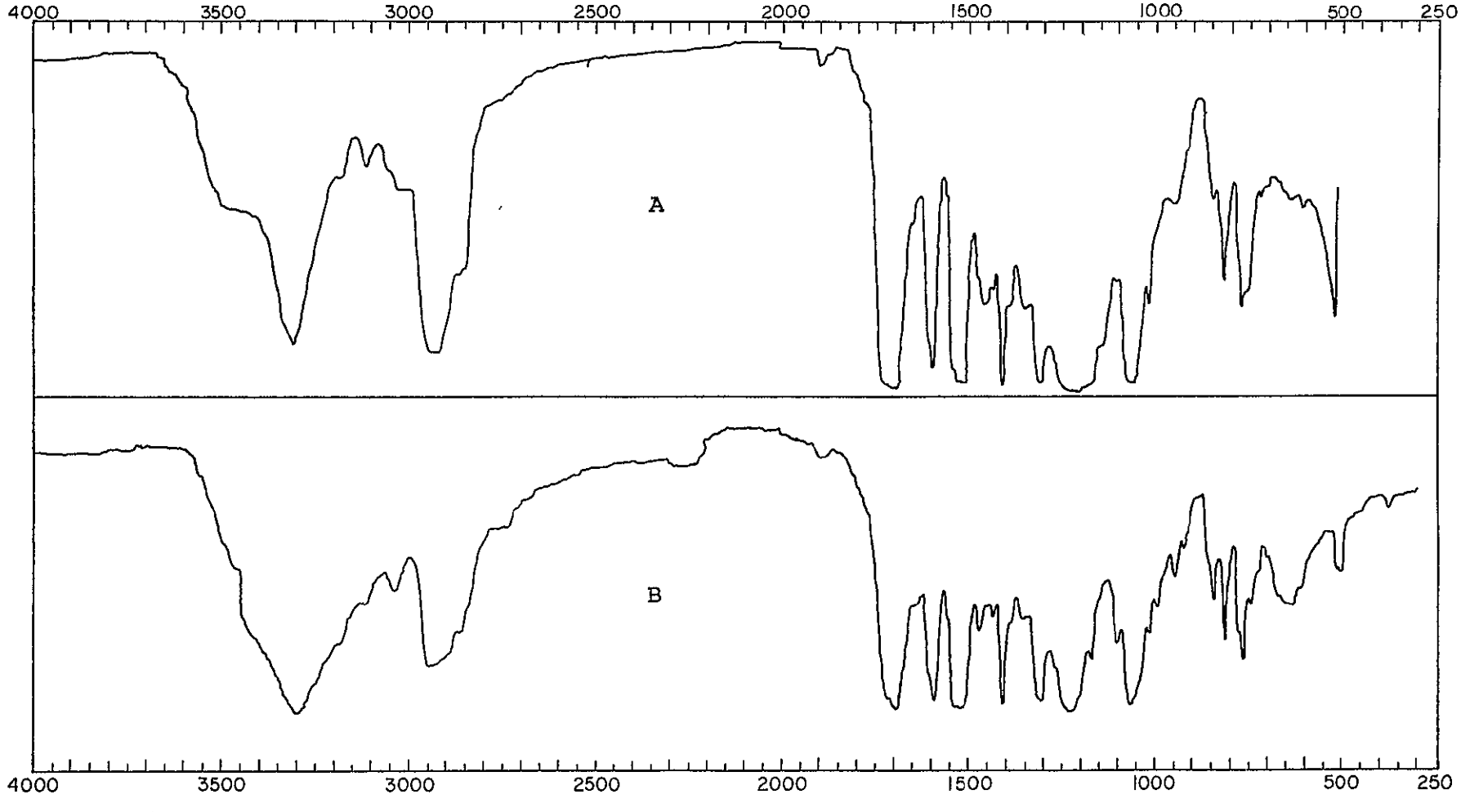


Fig. 10 INFRARED SPECTROGRAMS OF 3M 777 COMPUTER TAPE COATINGS

of the computer tape as suggested by the more pronounced peaks at 3120, 3040, and 530 cm^{-1}.

The polyurethane nature of the binder was also disclosed in investigations of a video tape (3M 351) obtained from the same manufacturing source (3M Company) as the previously considered tapes. The pronounced presence of oxygen-containing binder constituents in the 1000 to 1100 cm^{-1} absorption range (Fig. 11a) made the LCC fractionation of this binder extract particularly desirable. As suggested by Si-O-Si vibrational frequencies at 1020 and 1085 cm^{-1} and $Si-CH_3$ absorption bands at 800 cm^{-1} and 1270 (Fig. 11b), the compound recovered in the first fraction represents a silicone. This silicone, although present in more substantial amounts in the video tape, was also isolated in LCC fractions of other tape binders of the same commercial origin (3M Company), including the instrumentation (3M 888) and computer (3M 777) tapes. The advantage gained from investigating the binder after fractionation becomes apparent when the later fractions of the video tape extract are inspected. Here, in the 90/10 $CHCl_3$/MeOH elution system, the urethane nature of the binder is clearly indicated (Fig. 11c) after the silicone constituent was removed. The video tape (3M 351) is also characterized by its pronounced aromatic nature as indicated by absorption bands in the 3000 to 3200 cm^{-1} range.

Examination of an instrumentation tape binder of different commercial origin (RCA 617) indicated its basic urethane nature (Fig. 12a). The binder contained a significant proportion of silicone lubricant, which was largely recovered in the first LCC fraction as well as in an extraction with n-hexane (Fig. 12b). The similarity of the lubricant with a commercially available silicone oil (DC-200, Dow Corning Company) is indicated in the spectrum of the commercial compound (Fig. 12c). In addition to silicone, this instrumentation binder exhibited broad hydrogen-bonded OH-functionalities (3400 cm^{-1}), as well as absorption

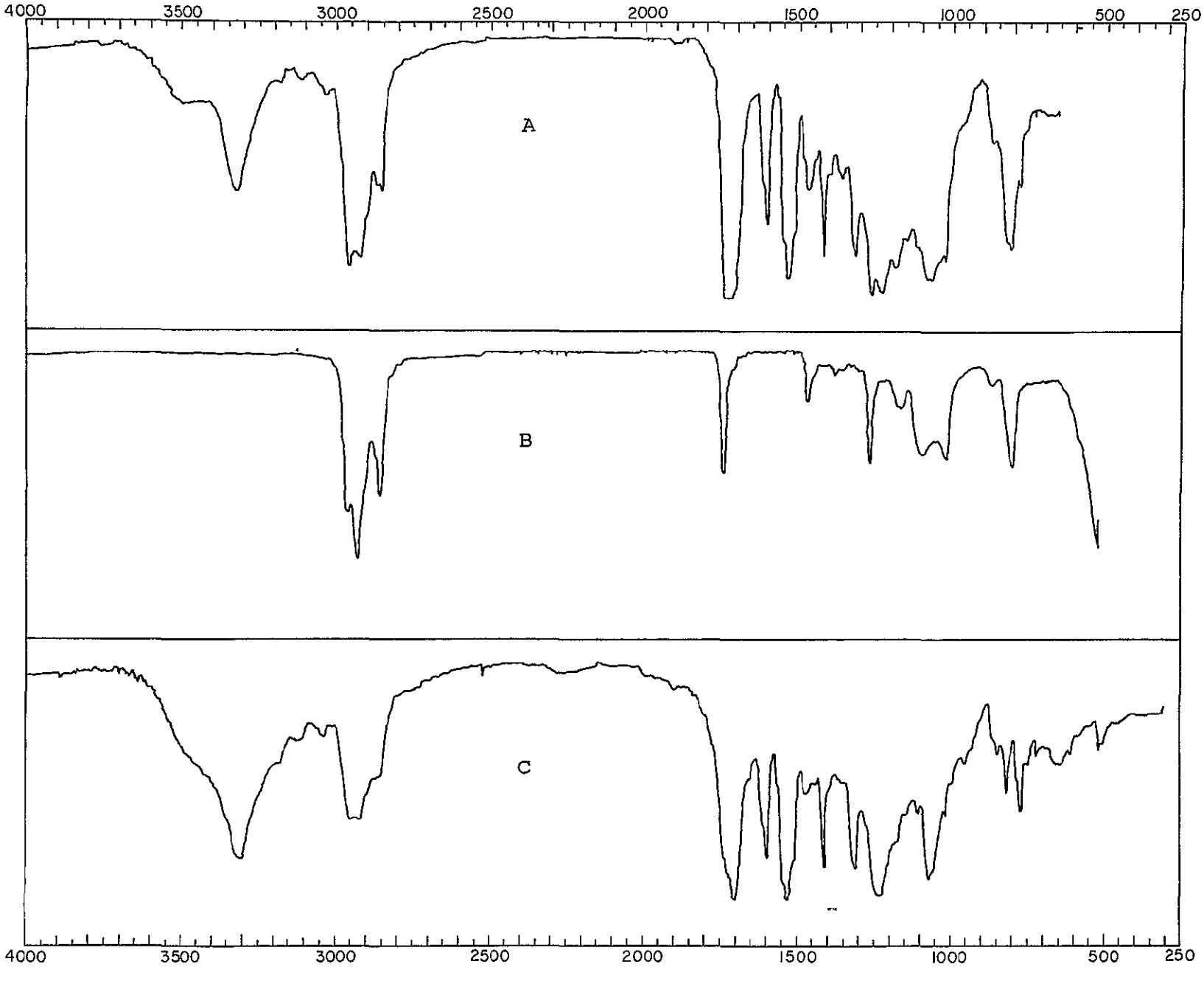


Fig. 11 INFRARED SPECTROGRAMS OF 3M 351 VIDEO TAPE COATING

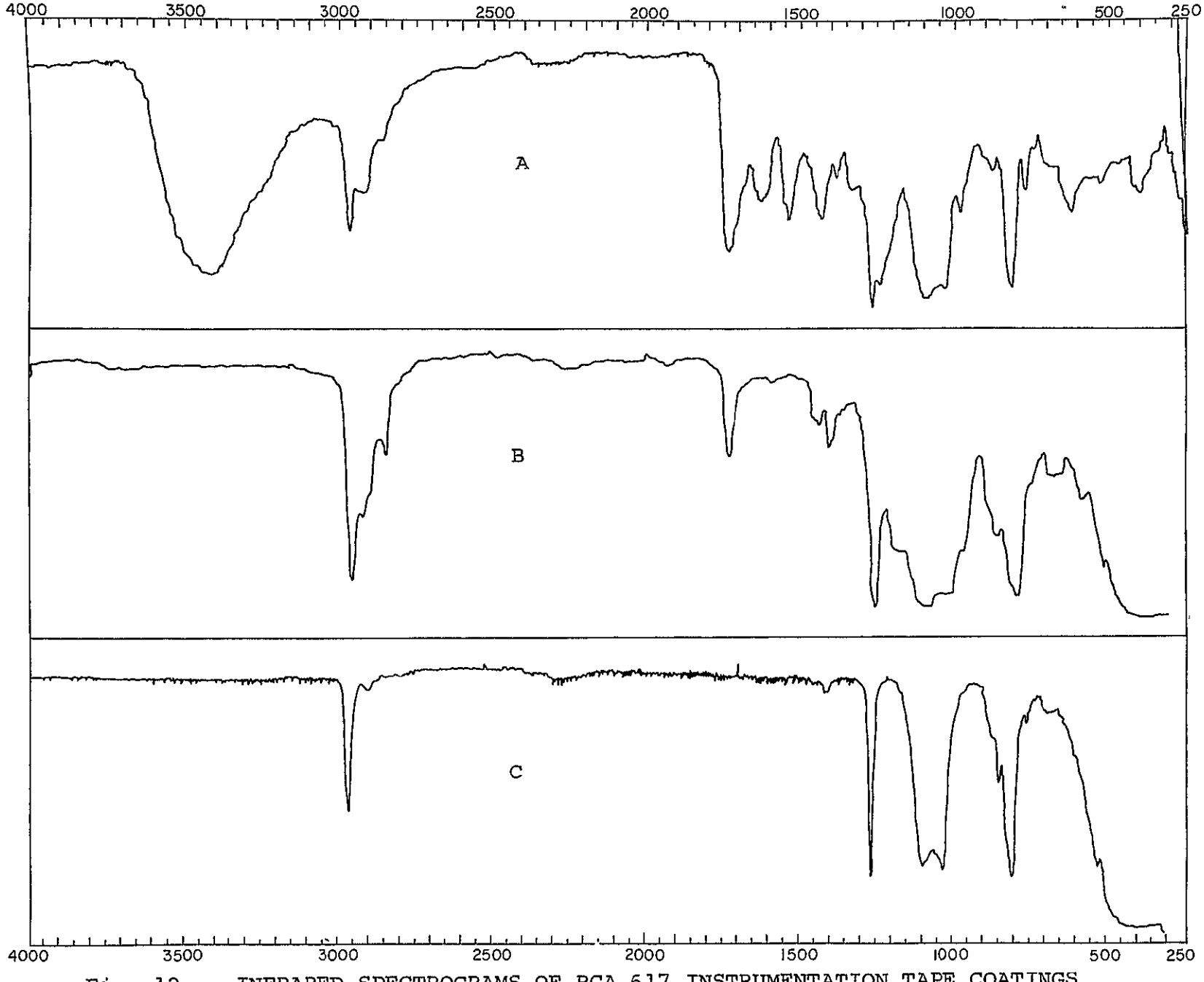


Fig. 12 INFRARED SPECTROGRAMS OF RCA 617 INSTRUMENTATION TAPE COATINGS

bands indicative of NH_2 groups (1620 and 870 cm^{-1}) and trans C=C unsaturation (960 cm^{-1}). An absorption peak at 610 cm^{-1} could possibly be ascribed to a chlorofunctional compound, in view of the previous evidence of the presence of Cl-containing constituents in the binder. However, the noted absorption band could also have arisen from an out-of-plane deformation of an urethane-associated NH group. In general, the investigated instrumentation tape (RCA 617) as well as its very close analog (RCA 675) exhibited a more diverse binder than the tapes of other commercial origin. The presence of OH-, NH_2- and C=C groups could pose potential adhesion problems for this tape. Apparently, inclusion of a substantial amount of silicone in the binder was intended to reduce head/tape adhesion through boundary lubrication. The quantitative aspects of lubricant and its possible implications on tape performance are discussed later.

An instrumentation tape (DuPont's Crolyn) representing a recording medium with a magnetic pigment (CrO_2) differing from that of conventional tapes has been subjected to infrared analysis. The organic material obtained after 24-hour Soxhlet extraction was fractionated by the previously described LCC method. The first fraction (Fig. 13a) suggests the presence of a branched hydrocarbon, as indicated by the isopropyl group associated bending frequencies at 1375 and 1385 cm^{-1} and at 1150 and 1170 cm^{-1}. The pronounced peak at 1450 cm^{-1} and absorption bands at 2870 and 2960 cm^{-1} are characteristic of the CH_3-group asymmetric bending, and symmetric and asymmetric stretching modes, respectively. The peak at 720 cm^{-1} (CH_2) indicates a greater chain length for the hydrocarbon, thus suggesting its possible use as a friction-reducing (lubricating) agent. The infrared spectrograms of a later fraction ($CHCl_3$/MeOH = 99/1) reveals the presence of a long-chain polymer of the polyamide type (Fig. 13b). The characteristic absorption frequencies noted are those of the Amide I (1640 cm^{-1}) and

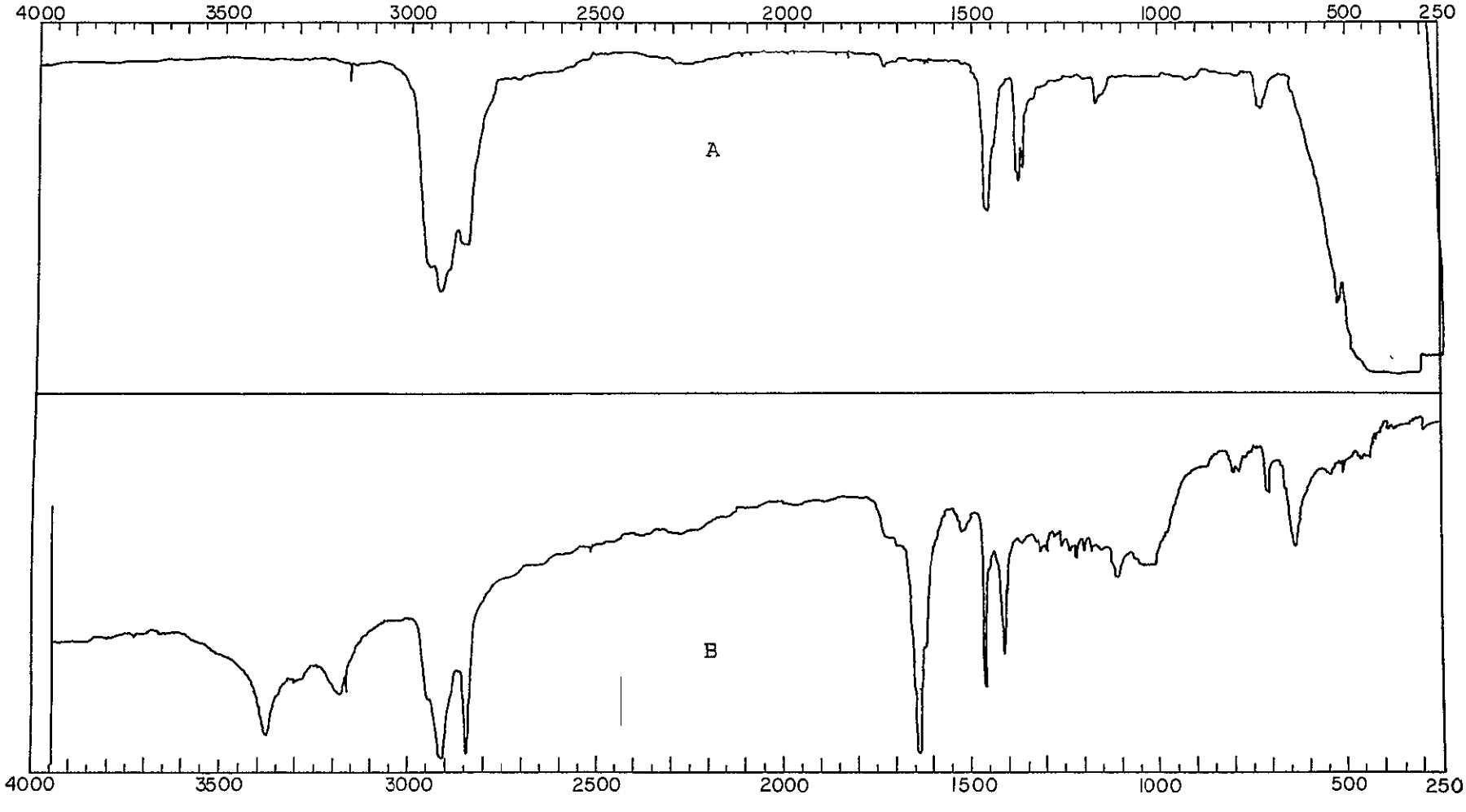
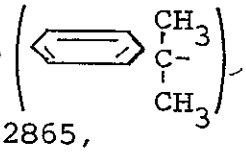


Fig. 13 INFRARED SPECTROGRAMS OF DU PONT CROLYN INSTRUMENTATION TAPE COATING

Amide II (1540 cm^{-1}) bands with the corresponding overtones at 3280 (2 x I), 3080 (2 x II), and combination bands at 3180 cm^{-1} (I + II). The absorption peak at 630 cm^{-1} suggests a combination band of the C=O and O=N bending modes of substituted amide frequencies.[33] The multiple peak structure in the 1200 to 1400 cm^{-1} region, indicative of CH_2-wag, suggests a methylene group arrangement in the polymer backbone as that of some ethylene-dicarboxylic acid-amides.

Since the fine structure of the CH_2 absorption peaks (1474, 1466, 1416 cm^{-1}) is missing, the polymer appears more amorphous. The hydrocarbon chain segments in the polyamide lattice would have a more open structure than pure hydrocarbons[34] since the interaction of polyamide groups should interfere with the alignment of hydrocarbon segments in the polymer,[35] unless such alignment was intentionally favored by treatment of the polymer during solidification. Considering the relatively small proportion of organic constituents in the chromium dioxide-pigmented magnetic coating, the choice of a more amorphous polymer may have been intentional on the part of the manufacturer to increase the compatibility between pigment and binder.

An epoxide-based binder was found to be represented in an extract of an instrumentation tape of another manufacturer (Memorex 63L). The broad band at 3400 cm^{-1} (Fig. 14a) indicates the presence of hydrogen-bonded OH groups, while peaks at 1365 cm^{-1} (OH bend) 1100 cm^{-1} (C-OH stretching mode) and 940 cm^{-1} (OH-wag) lend further support for the occurrence of this functionality. Absorption frequencies at 1380 cm^{-1} which appear to be associated with a dimethyl-phenyl group  , and the pronounced methyl absorption peaks at 2960, 2865, and 1455 cm^{-1} could likely represent a bisphenol A-type epoxide derivative. The ether linkages of the polymer are indicated by the absorption frequencies at 1290 cm^{-1} (symmetric C-O-C) and

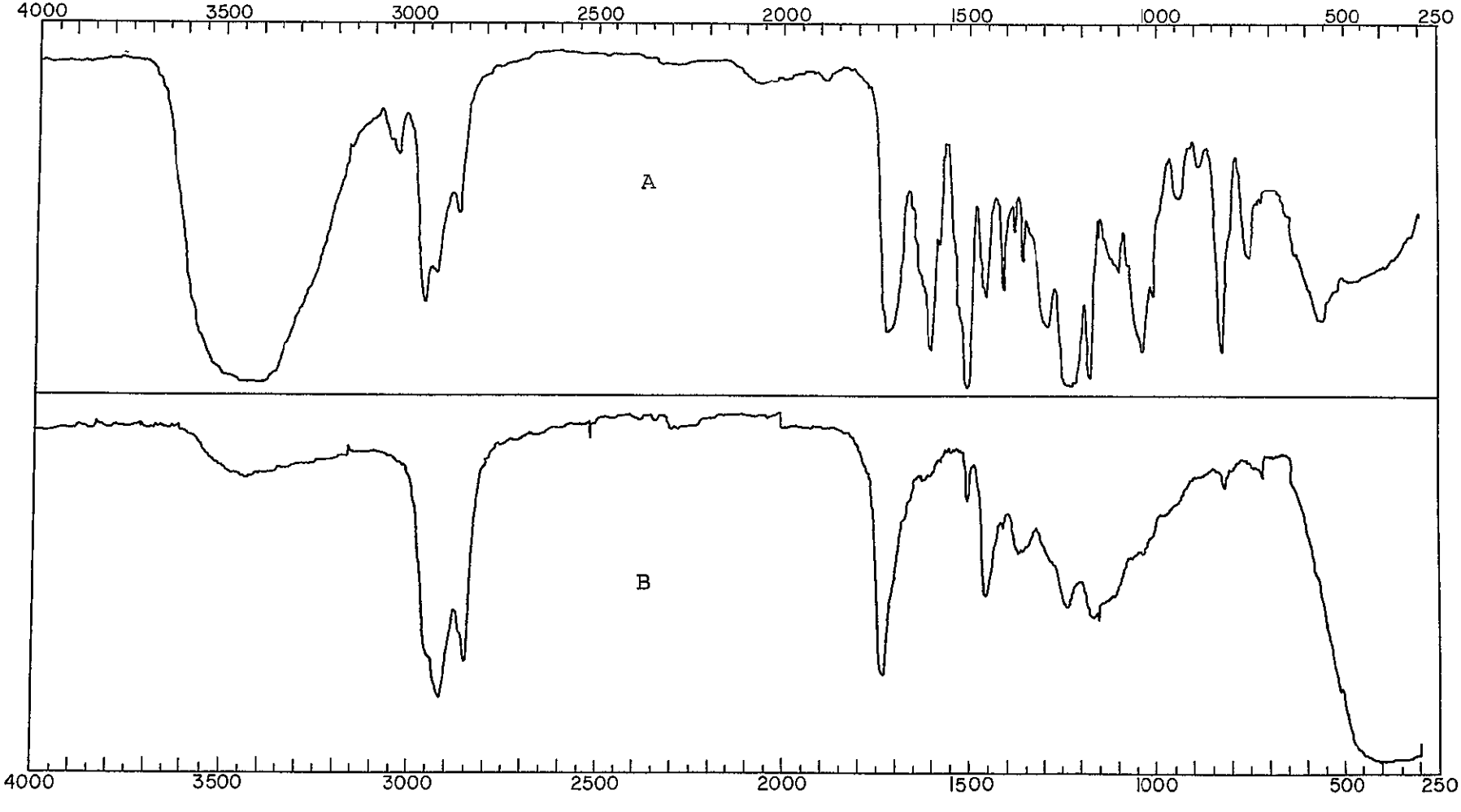


Fig. 14 INFRARED SPECTROGRAMS OF MEMOREX 63L INSTRUMENTATION TAPE COATING

1040 cm^{-1}, while its aromaticity is suggested by the peaks at 3040, 3065, and 1600 cm^{-1}. The carbonyl band at 1730 cm^{-1} has most likely its origin in an ester additive. This ester was partly isolated from the main binder system by extraction of the magnetic coating with n-hexane. The IR spectrum of this extract (Fig. 14b) indicates the suppression of epoxide-related peaks, while the ester absorbencies at 1730 cm^{-1} (C=O stretch), 1240 and 1275 cm^{-1} asymmetric C=O stretch) and 1110 cm^{-1} (symmetric C-O stretch) are much more apparent. In view of the absence of a silicone constituent in the binder, the ester additive appears to function in the coating as a lubricant.

Similar chemical relationships, as reflected in IR spectra, appeared between a computer tape (Memorex Quantum) and the instrumentation tape of the same manufacturer (Memorex 63L) as shown in Figs. 14a and 15a. The epoxide nature of the computer tape binder is again indicated. Also, the n-heptane extract of the coating (Fig. 15b) showed good correlation with a similar extract of the instrumentation tape (14b), thus suggesting the presence of an ester-type lubricant in the binder.

More substantial differences were noted between the instrumentation tape binder and a video tape binder (Memorex 79L) of the same manufacturer. Here (Fig. 16a), the terminal hydroxyl groups resulting from the epoxide reaction are not discernible (absence of broad OH band at 3400 cm^{-1} and peaks at 1100 and 940 cm^{-1}). This, in conjunction with the relatively sharp NH absorption frequency at 3320 cm^{-1} and amide overtone peaks suggest the polyurethane nature of the binder. The aromatic character of the binder is indicated by absorptions at 3030 and 3120 cm^{-1} as well as by peaks associated with out-of-plane hydrogen-bending frequencies at 810 and 890 cm^{-1}. The 890 cm^{-1} absorption band suggests greater substitution of the aromatic ring in comparison with the less substituted aromatic constituents of the instrumentation and computer tape binders of the same commercial origin.

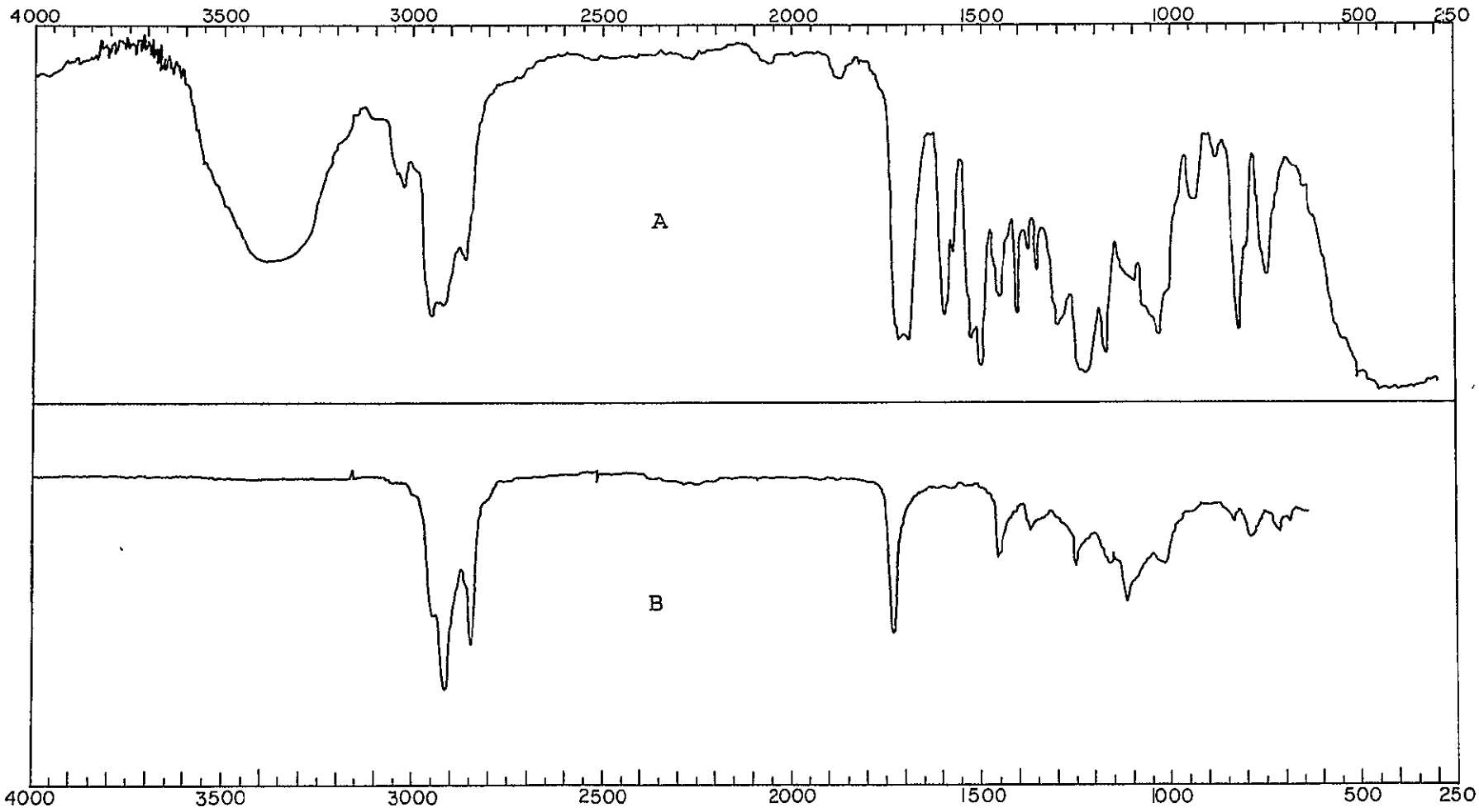


Fig. 15 INFRARED SPECTROGRAMS OF MEMOREX QUANTUM INSTRUMENTATION TAPE COATING

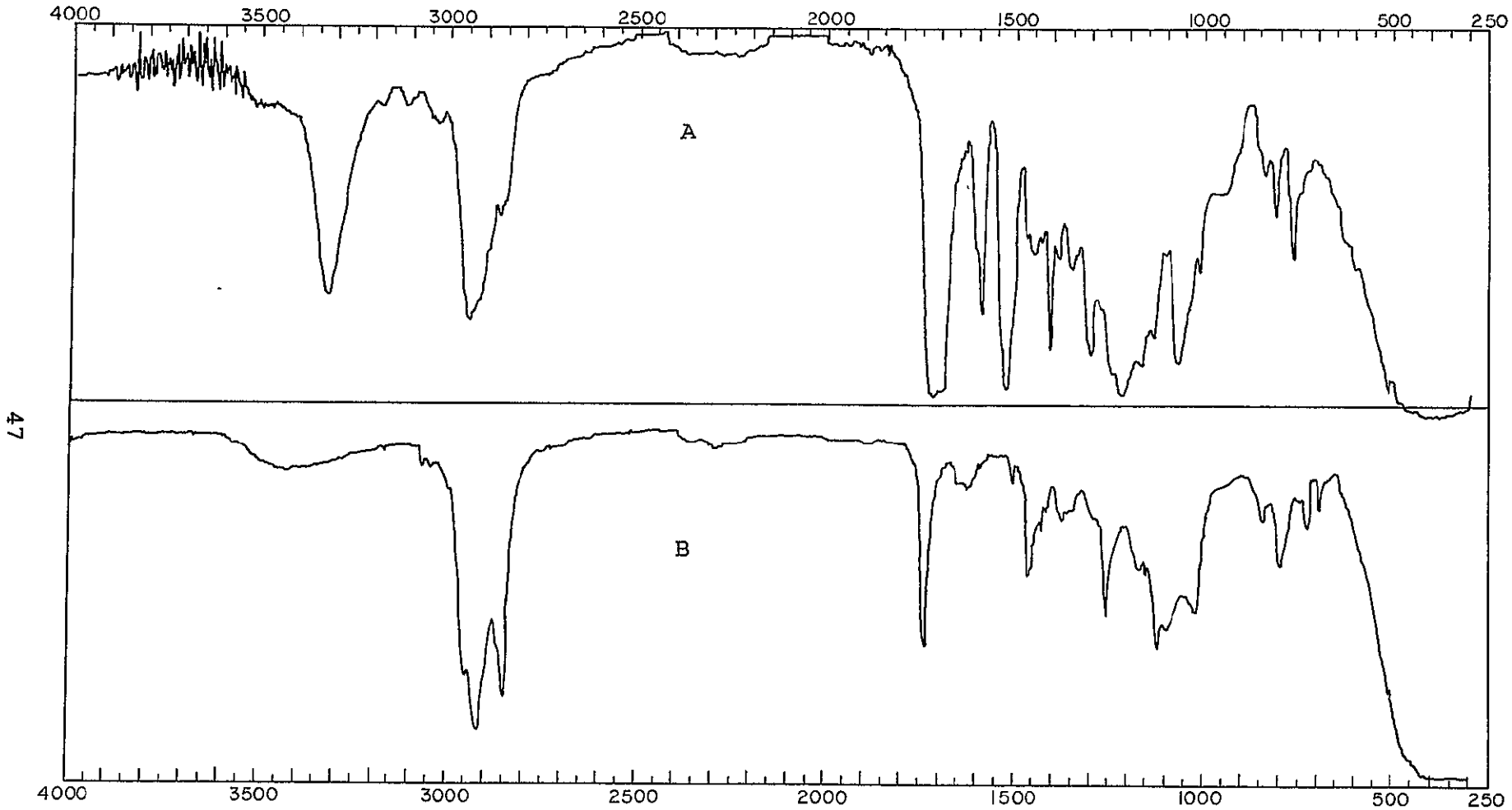


Fig. 16 INFRARED SPECTROGRAMS OF MEMOREX 79L VIDEO TAPE COATINGS

This is also supported by the greater ratio of absorption peak lengths of the video tape binder extract at 760 cm^{-1} (alkaline chain rocking modes) and 810 cm^{-1} (bending modes of adjoining hydrogens on the aromatic ring). The n-hexane extract of the tape coating (Fig. 16b) indicates Si-O-Si absorption peaks at 1020 and 1085 cm^{-1} as well as $Si-CH_3$ bands at 1270 and 800 cm^{-1}, thus suggesting the presence of silicon lubricant in the binder. The need of silicon lubricant in the video tape binder may have been dictated by thermal and low friction requirements imposed on the tape because of its contact with a fast-scanning head. The chemical characteristics of most tapes investigated are summarized in Table IV.

Infrared spectroscopic measurements performed on the binders of 3M 888 and 3M 951 tapes and the data obtained on weight proportions of organic matter extracted from the coatings of these tapes suggested a very close similarity, if not identical, of the binder systems. Also, the coatings of RCA tapes 617 and 765 showed a close chemical resemblance as indicated by IR measurements. However, differences were observed in quantitative relationships of the amount of coating applied to the tape substrate (tape 617 contained more coating) and the quantities of n-hexane extractable matter. The latter appeared to be related to the greater amount of silicon lubricant in RCA 617 (Table V).

3. Binder-Dependent Characteristics of Tape Coatings

a. Polar Constituents. In an attempt to obtain information about the amounts of extractable polar constituents from tape coatings, the yield of fractions recovered from the LCC separation with $CHCl_3$/MeOH eluent was quantitatively determined (Table VI). In a further effort to assess the polar nature of the binder systems of several representative tapes, the material isolated from the coating after extraction and LCC fractionation was selectively combined, and the amounts obtained

Table IV

IR IDENTIFICATION OF BINDER CONSTITUENTS

Tape	Binder Charaterization (Absorption peaks)	Aromatic (aliphatic) Compt's (Absorption peaks)	Carbon - Oxygen (Absorption Frequencies)	Lubricant (Absorption Peaks)
3M 888	Polyurethane based (PUB) NH3300 cm^{-1} NH bend - CH stretch: 1535 cm^{-1} C=O amide stretch: 1700 cm^{-1}	Aromatic Components: 825 cm^{-1} out-of-plane wag, 2 adjacent H 850 cm^{-1} subst'd ring 3040, 3120 cm^{-1}: arom. abs	Ester carbonyl, C=O 1730 cm^{-1} C-O stretch 1230 CH_2-C=O . 1420 C-C-O: 1225	Organo-polysiloxane Si-O-Si: 1020 and 1085 cm Si-CH_3: 800 and 1270 cm^{-1}
3M 777	Polyurethane based NH 3300 cm^{-1} NH bend - CH stretch: 1535 cm^{-1}	Aromatic components: 825 cm^{-1} out-of-plane H-wag 850 cm^{-1}: subst'd ring 3120, 3040 arom abs.	Ester carbonyl: 1730 cm^{-1} C-O stretching frequencies as in 3M 888 tape	Organo-polysiloxane As above. (present in lesser amount than 3M 888)
3M 351	Polyurethane based Absorption peaks similar to those above	Aromatic components. Several bands in 3000 to 3200 cm^{-1} range.	Ester carbonyl, C=O 1730 cm^{-1} CH_2-C-C=O 1415 cm^{-1} C-C-O 1225 cm^{-1}	Organo-polysiloxane Pronounced Si-O-Si 10 20 & 1085 cm^{-1} Si-CH_3: 800 and 1270 (considerable amount)
Memorex 63L	Epoxide based Terminal OH OH bend 1365 cm^{-1} OH wag 940 cm^{-1} H...OH 3400 cm^{-1} Dimethyl-phenyl group 1380 cm^{-1}	Aromatic components: 3040, 3065 and 1600 cm^{-1}	Ester carbonyl 1730 cm^{-1} Ether linkage: sym C-O-C 1290 cm^{-1} C-OH stretch 1100 cm^{-1}	Lubricity presumably afforded by aliphatic ester, extractable with n-hexane. Asymmetric C-O stretch : 1240, 1275 cm^{-1}
Memorex Quantum	Epoxide based OH bend, wag 1365, 940 cm^{-1} Broad H. OH 3400 cm^{-1}	Aromaticity: Several bands in 3000 to 3200 cm^{-1} range	Ester and ether absorption bands	n-hexabe - extractable aliphatic ester, similar to Memorex 63L
Memorex 79L	Polyurethane based NH stretch 3320 cm^{-1} (absence of epoxide-related OH bands) Amide overtones	Aromaticity: 3030, 3120 cm^{-1} out-of-plane H-bending 810 cm^{-1}	Ester carbonyl 1730 cm^{-1} CH_2-C-C=O 1415 cm^{-1} C-C-O 1225 cm^{-1}	Organo-polysiloxane SiO-Si 1020 and 1085 cm^{-1} Si-CH_3 1270, 800 cm^{-1}
DU PONT CROLYN	Polyamide based Amide I 1640 cm^{-1} Amide II 1540 cm^{-1} Overtone and combination bands 3280, 3180 cm^{-1}	Predominantly aliphatic Pronounced CH_2-wag in 1200 to 1400 cm^{-1} range Branched hydrocarbon i-propyl: 1375, 1385 cm^{-1}	C-O 1720 cm^{-1} comb. band C-O O=CN 630 cm^{-1} CH_2-C=O 1420 cm^{-1}	Lubrication presumably provided by long-chain aliphatic hydrocarbon 720 cm^{-1} CH_2-rock
RCA 617	Polyurethane based NH 3300 cm^{-1} NH bend - CH stretch 1535 cm^{-1} NH_2 1620 cm^{-1}	Aromaticity 3020, 1600 cm^{-1} Also contains C-C trans 960 cm^{-1} C-Cl (?) 610 cm^{-1}	Ester carbonyl 1730 cm^{-1} CH_2-C=O 1420 cm^{-1} H...OH 3400 cm^{-1}	Organo-polysiloxane Si-O-Si 1020, 1080 cm^{-1} Si-CH_3 1270, 800 cm^{-1}

Table V

QUANTITATIVE RELATIONSHIPS IN RCA 617 AND 765

Property	Tape Identification					
	RCA 617			RCA 765		
Weight of tape (4 ft) before X'n (g)	.8541	.8387	.8347	.7610	.7600	.7602
Weight of tape (4 ft) after X'n (g)	.8463	.8314	.8268	.7557	.7549	.7550
Amount of matter extracted (mg)	7.8	7.3	7.9	5.3	5.1	5.2
Amount of matter X'd per total weight of tape, percent	0.913	0.870	0.946	0.696	0.671	0.684
Average, percent		0.910 + 0.04			0.684 + 0.01	
Amount of coating in 4 ft of tape (g)	0.3699	0.3545	.3505	.2768	.2758	.2760
Amount of matter extracted per weight of coating, percent	2.11	2.03	2.25	1.91	1.85	1.88
Average, percent		2.13			1.88	

Table VI

CHROMATOGRAPHIC SEPARATION OF BINDER CONSTITUENTS

Tape Identification / Solvent System	Amount Extracted with Chloroform-Methanol Solvent, % by Weight of Chloroform Extracted Material							
Chloroform, %	100	99	98	95	90	75	50	0
Methanol, %	0	1	2	5	10	25	50	100
3M 888	13.2	—	—	68.9	5.5	5.4	0.7	1.0
3M 351	22.4	4.4	46.3	15.3	4.0	1.3	1.2	1.6
3M 777	2.9	2.3	49.2	31.1	5.4	1.7	2.0	5.2
3M 551	13.8	3.4	34.5	37.3	8.3	1.3	1.2	1.9
Memorex 63L	24.2	3.3	29.3	15.9	3.0	0.6	0.7	0.8
Memorex 79L	13.8	4.3	33.2	14.7	3.7	1.9	1.0	1.8
Crolyn	19.0	6.3	30.2	31.3	9.7	1.0	0.6	0.6
RCA 617	63.0	—	15.2	8.6	5.9	2.0	0.8	0.6

were gravimetrically determined. This material contained a fraction recovered from the first elution ($CHCl_3$/MeOH, 100/0) of the extract, corrected for the amount of nonpolar constituents (extracted with n-hexane) and fractions of LCC separations with 75/25, 50/50, and 0/100 $CHCl_3$/MeOH eluents, as well as material initially recovered from the tape coating in the sequential extraction with MeOH (first extraction - $CHCl_3$; second extraction - MeOH). The data representing the total material per unit weight of coating are shown in the bar graph of Fig. 17. As indicated, the Memorex 63L and RCA 617 tapes contain the greatest amounts of extractable polar material in their binders. The implications of these findings in regard to potential "sticktion" problems were assessed on the basis of the experimental evidence obtained in loop tests for these tapes.

b. Nonpolar Constituents. In a parallel effort to isolate the nonpolar components from magnetic tapes, the detached coatings of these tapes were subjected to extraction with n-hexane. The amounts of extractable material are shown in the bar graph of Fig. 18. It should be emphasized that, since the bulk of silicone additives and hydrocarbon lubricants would be found in the extract, the data reflect in large measure the amounts of lubricant in the tapes. On that basis, the relatively high content of extractable material in lubricant-rich tapes of the RCA 617 and 3M 351 type can be better understood.

c. Moisure Sensitivity. The equilibrium moisture absorption levels for the coatings of several magnetic recording tapes were determined by the Karl Fischer potentiometric titration method. This method involves reduction of iodine by sulfur dioxide in the presence of water in pyridine solution according to the following reaction:

$$2\ H_2O + I_2 + SO_2 \cdot 2C_5H_5N + 2\ C_6H_6N \rightarrow (C_5H_5N)_2 \cdot H_2SO_4 + 2\ C_5H_5N.HI \quad (3)$$

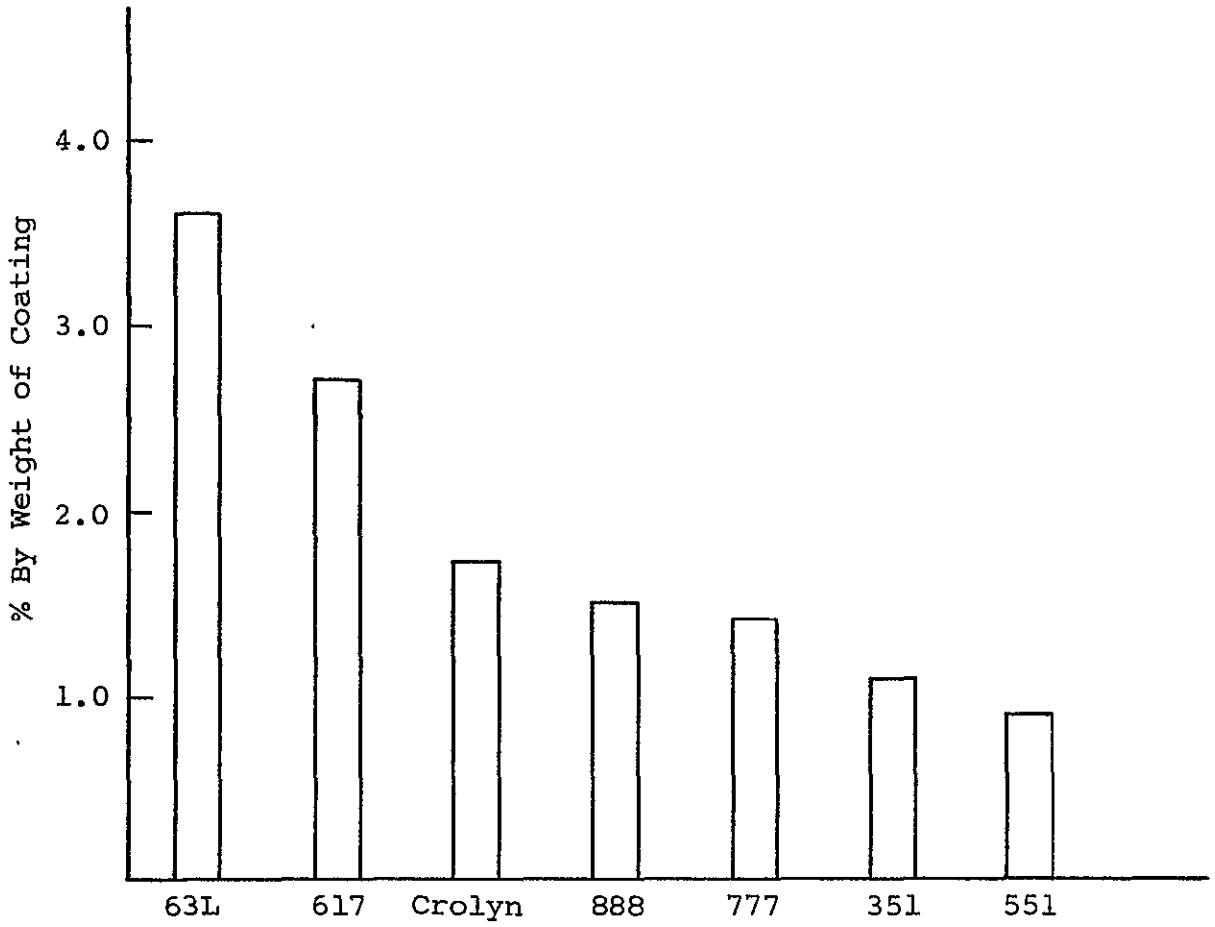


Fig. 17 EXTRACTABLE POLAR COMPONENTS IN TAPE COATINGS

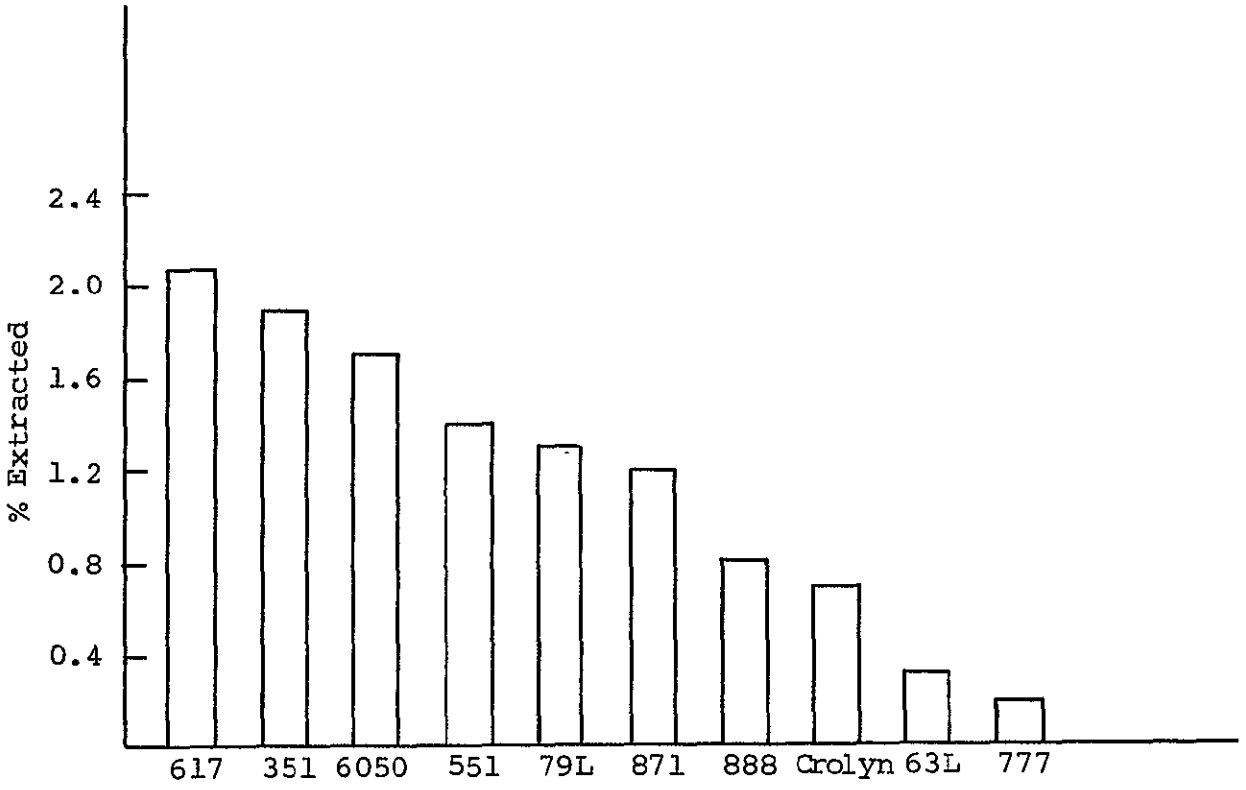


Fig. 18 HEXANE-EXTRACTABLE COMPONENTS FROM TAPE COATING

Thus, two moles of water reduce two gram-atoms of iodine. The end-point of the reaction is determined by a current-measuring device that indicates the depolarization of a pair of platinum electrodes.

In the experiment, 1 g-sample of coating material, detached from the Mylar tape substrate, as exposed to the environment provided in four large desiccators that maintained relative humidities of 7, 20, 43, and 75%, as obtained from saturated salt solutions of sodium hydroxide, potassium carbonate, potassium acetate, and sodium chloride, respectively. After conditioning for 96 hours, the moisture contents of the equilibrated samples were determined in triplicate measurements. The results obtained for several tape coatings and the polyester substrate, which was shredded and sieved through a No. 40 mesh screen, are shown in Fig. 19. As indicated, Memorex 63L shows a more pronounced moisture sensitivity than 3M 777 tapes. The equilibrium moisture absorption values range from 0.66 to 1.18% for Memorex 63L, and 0.26 to 0.52% for 3M 777, for the two extreme humidity conditions (7 and 75% RH), respectively.

These results appear to agree with the data obtained in the extraction of polar constituents from tape coatings (Fig. 17) and the spectroscopic evidence of the hydroxyl-functionality of Memorex 63L, as may be expected, a greater affinity for moisture from more polar compounds. However, on further analysis of the absorption relationships in magnetic tape coatings, the overriding importance of the continuity of the coating (as provided by the binder) around oxide pigment particles, in regard to absorption effects is realized. The extent to which the organic envelope is disrupted around the oxide pigment will determine, to a great extent, the susceptibility of the coating to water vapor absorption, since the relatively hydrophilic metal oxide represents the major constituent in the coating. Thus, the polyester substrate, when considered alone, (without pigment)

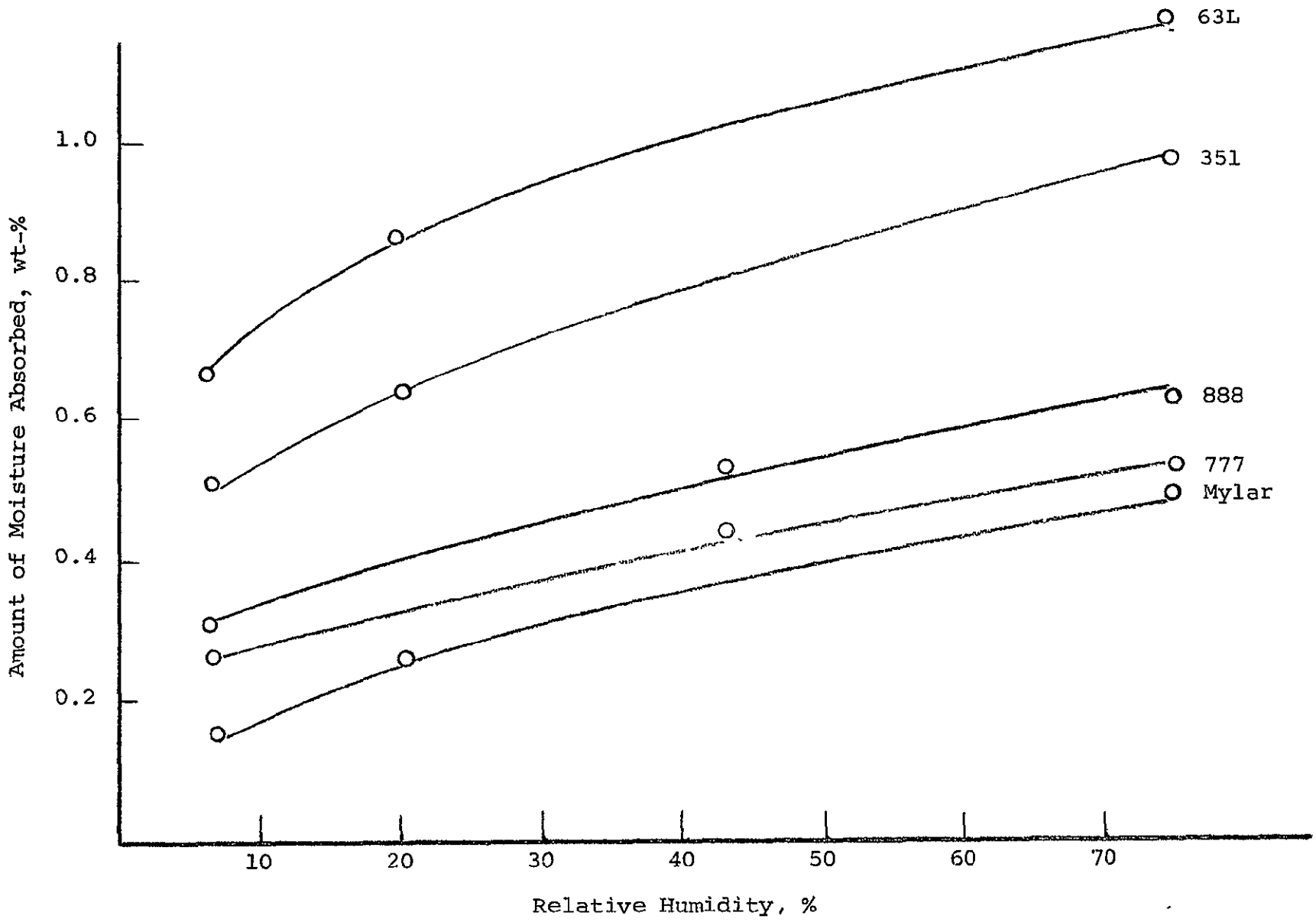


Fig. 19 EQUILIBRIUM MOISTURE CONTENT IN MAGNETIC TAPES

exhibits a lesser susceptibility to moisture than the pigment-containing coatings of magnetic tapes. In comparing the relatively high absorption value of 3M 351 with other tape coatings, where this result does not correlate with the relatively nonpolar nature of extractable binder constituents of this tape, the observation is related to the coating inefficiency of the binder. However, another explanation can also be found in the presence of substantial amounts of silicone lubricant in 3M 351, as the helical configuration of polysiloxanes affords little protection against water vapor permeation, although the coating provides a relatively good barrier for liquid water penetration.[36]

d. Corrosion Susceptibility of Copper Foil in Contact with Magnetic Tapes. In a limited effort to assess the corrosion-sensitizing properties of selected magnetic tapes for metals, 3M 888, 3M 777, 3M 951, Memorex 63L, and RCA 617 tapes were brought into close contact with copper surfaces at 65°C, in a nitrogen and air atmosphere maintained at 2 and 100% RH. In the experimental procedure, a three-foot long tape specimen was wrapped around a 0.005 x 0.75 x 5 in. copper foil strip (with the coating facing the metal surface). The strip was draped lengthwise over a 1 x 3 in. microscope glass slide that provided structural support for the foil and permitted tight winding of the tape in crosswise overlapping segments. To ensure the desired contact between metal and tape, the wound tape was overwrapped with 1/2 inch wide Dacron gauze that kept the sandwich construction in place without impairing the transfer of moisture to the tape/metal interface. After 96-hour exposure, the copper foil surfaces disclosed little change in appearance except for Memorex 63L and RCA 617 tapes. The metal foil in contact with Memorex 63L tape exhibited slight discoloration and the presence of a thin film that gave rise to interference colors when viewed from an oblique angle. A similar

but less pronounced effect was noticed for RCA 617. Moisture appeared to enhance the corrosion of foils in contact with Memorex 63L, while the presence of oxygen or nitrogen affected the appearance of the foil to an insignificant degree.

The observations made indicate, with the possible exclusion of one tape (Memorex 63L), little tendency of tape coatings to corrode copper surfaces. However, the relatively mild conditions under which the tests were conducted preclude more conclusive arguments. In actual use, where the tape remains in sliding contact with metal surfaces, the possible degradation of the binder could elicit more pronounced corrosion responses from the recording heads.

e. Volatilizable Components. In investigating the tendency of magnetic coatings to release volatilizable compounds during heating, as might be the case with organic substances that are heated in friction, gas chromatographic measurements were performed on 5 to 15 mg samples of the coatings that were obtained by mechanical removal (scraping) from the tape substrate. The specimens, placed in platinum boats, were introduced into an inductor (heating) unit of a Varian Associates Aerograph instrument, where they were maintained at 200°C; the volatile components were swept into a gas chromatographic column. The helium carrier gas flow through the SE-30 packed column whose temperature, programmed from ambient to 300°C at 8°C/min, was maintained at 30 ml/mil; the hydrogen and air flow rates to the ionization flame detector were 3 ml/minute.

Among the coatings investigated were those obtained from 3M 888, 3M 951, 3M 351, 3M 777, Memorex 63L, Memorex 79L, and RCA 617 tapes. The 3M 888 and 3M 951 coatings showed relatively similar gas chromatographic patterns with the major peaks located around 90°C (most tapes exhibited the first peak in this range), 180, 210, 235, and 245°C. The volatilizable components of

3M 351 coatings covered the range of 140 to 270°C, with a major constituent appearing at 235°C, and smaller peaks showing at 240, 245, and 265°C. Memorex 63L and particularly 3M 777 coatings exhibited a lesser number of volatile constituents, as the major peaks appeared at 275 and 292°C for Memorex 63L; and 3M 777 coating showed few peaks of small amplitude in the higher temperature range. By contrast, RCA 617 and, to a smaller extent, Memorex 79L showed a greater number of volatile constituents in the 170 to 270°C temperature range. The RCA 617 coating revealed major peaks at 170, 203, 249, 287, and 295°C, while peaks of greater amplitude appeared for Memorex 79L at 192, 260, and 298°C.

A comparison of gas chromatograms of RCA 617 coatings in their pristine state and after prolonged use of the tape on the loop tester disclosed the absence of a characteristic component (peak at 249°C) in the coating of the used tape. In an attempt to obtain information about the nature of the missing component, the compound associated with the 249°C was introduced from the chromatograph, at the time of its appearance, into a mass spectrometer. Among the fragments identified for this component were those with mass numbers 57-55, 43-41, and 29-27, representing butyl, propyl, and ethyl units, respectively. A base peak of 56 appeared to have originated from an isocyanate fragment ($-CH_2-NCO$), while another peak at 73 mass units suggested the presence of an amide fragment from the polyurethane resin. The molecular weight of the missing component used in RCA 617 tape was estimated at 284. This could well be a larger fragment of the polyurethane resin that was partially removed from the binder as a result of continuous friction between the coating and recording head in the loop test. The explanation must be qualified by the admission that an unambiguous identification of chromatographically isolated compounds by mass spectrometric methods would require comparison of the mass spectrograms obtained for the unknown specimen with spectrograms of those compounds whose identity with the substance in question is strongly suspected.

The chemical and analytical studies of magnetic coatings of a number of instrumentation, computer, and video tapes have some similarities as well as considerable differences between the materials investigated. A complete characterization of the tapes studied requires critical examination of the property-performance relationship for these tapes.

SECTION III

CHARACTERIZATION OF PRINCIPAL TAPES TESTED

A number of manufacturers are currently producing magnetic tape of various designs for the audio, instrumentation, digital, and video markets. Although it was known that differences existed in the formulation and coating procedures for these tapes, specific information concerning these distinctions was generally unavailable. Therefore, it became necessary to evaluate the properties of each tape used during the test program.

Most of the organizations visited during Phase I indicated preference for instrumentation tapes in satellite recording applications. This choice was apparently based upon familiarity, superior short-wavelength capability, and increased volume packing density, due to the 1-mil rather than 1.5-mil tape thickness dimension. However, since one of the objectives of this program was to isolate the effects of certain tape properties, it was felt that the variation in these properties would be enlarged by examining digital and video tapes as well as the instrumentation types commonly used. Therefore, the choice of the nine principal tapes to be tested was based upon both historical satellite usage and parameter variation. The tapes chosen and the batch numbers of the individual reels procured are shown in Table VII. (Audio tapes were neglected.) Additional tapes utilized near the conclusion of the program are listed in Appendix A.

The initial characterization of the tapes chosen was based upon information derived from the specification sheets provided by the manufacturer. These specifications contained basic data on physical properties, such as dimensions and tensile strength, intrinsic magnetic properties, and relative wavelength response measurements. However, general format, quantity of information, and procedures used in obtaining the data varied considerably between manufacturers. Therefore, only limited data were actually

Table VII

PRINCIPAL TAPE TYPES EVALUATED

Manufacturer	Tape Designation	Batch Number	Reason For Selection
3M	351	64748043046 64748043029	Video tape designed for high head-to-tape speed
3M	777	63904-390-1036 63904-390-1011	Computer tape with thicker substrate and oxide coating
3M	871	69333-3-10-16 69333-6-20-19 74768-5-11-9	Reportedly made for low speed operation
3M	888	55512-46-10-17 67760-40-11-12 (1 reel unknown)	Commonly used instrumentation tape
Memorex	63L	15092-A2-6A 15092-A1-17A	Commonly used instrumentation tape
Memorex	79L	E59159-C2-5D ME56857-A1-5B	Video tape designed for high head-to-tape speed
Memorex	Quantum	27E-HJ-3712C-A1-M30160-B3 (2 Reels)	Computer tape with thicker substrate and oxide coating
DuPont	Crolyn	832211036119 832211066324	Unique oxide and binder system
RCA	617*	Unknown	Previous use in satellite applications

*RCA 617 is an experimental tape not commercially available.

derived from these specifications. The data are tabulated in Table VIII. In accordance with the procedures discussed below, the remaining physical and magnetic properties were measured using the particular reels of tape incorporated in the testing program.

A. Surface Finish

The surface finish for each of the tapes was measured using a Bendix Proficorder, Model RLC-2. The stylus radius used was 0.1 mils and the applied pressure was 0.3 grams. Extreme care was exercised in maintaining cleanliness of the tape and optical flatness upon which the tape was placed. Measurements were repeated over approximately two-inch lengths in several areas of the tape. Two measurements were made: surface waviness and roughness. Basically, these measurements differ in the cutoff wavelength, with waviness being only the long-wavelength effect. Little or no difference was observed between the waviness of most tapes. The values of roughness, however, varied considerably in both peak-to-valley amplitude and number of discontinuities per linear distance. Figure (a) through (h) show the strip chart results for roughness.

B. Flexibility

The flexibility of test procedures incorporated in the Guidelines, Vol. I, was identical to that used during the test program. A three-inch, free length of tape was suspended over a sharp corner with the oxide side pointing down. By observing the x and y coordinates at the end of the tape, the angle of flexibility was computed as the tan x/y. The environmental conditions were laboratory ambient, i.e., 71°F and 27% RH. Results for the tapes measured are shown in Table IX. One tape, an experimental high temperature tape, was not considered measurable because of an S-shaped reverse-bend formed by the free length.

Table VIII

SUMMARY OF PROPERTIES SPECIFIED BY TAPE MANUFACTURERS

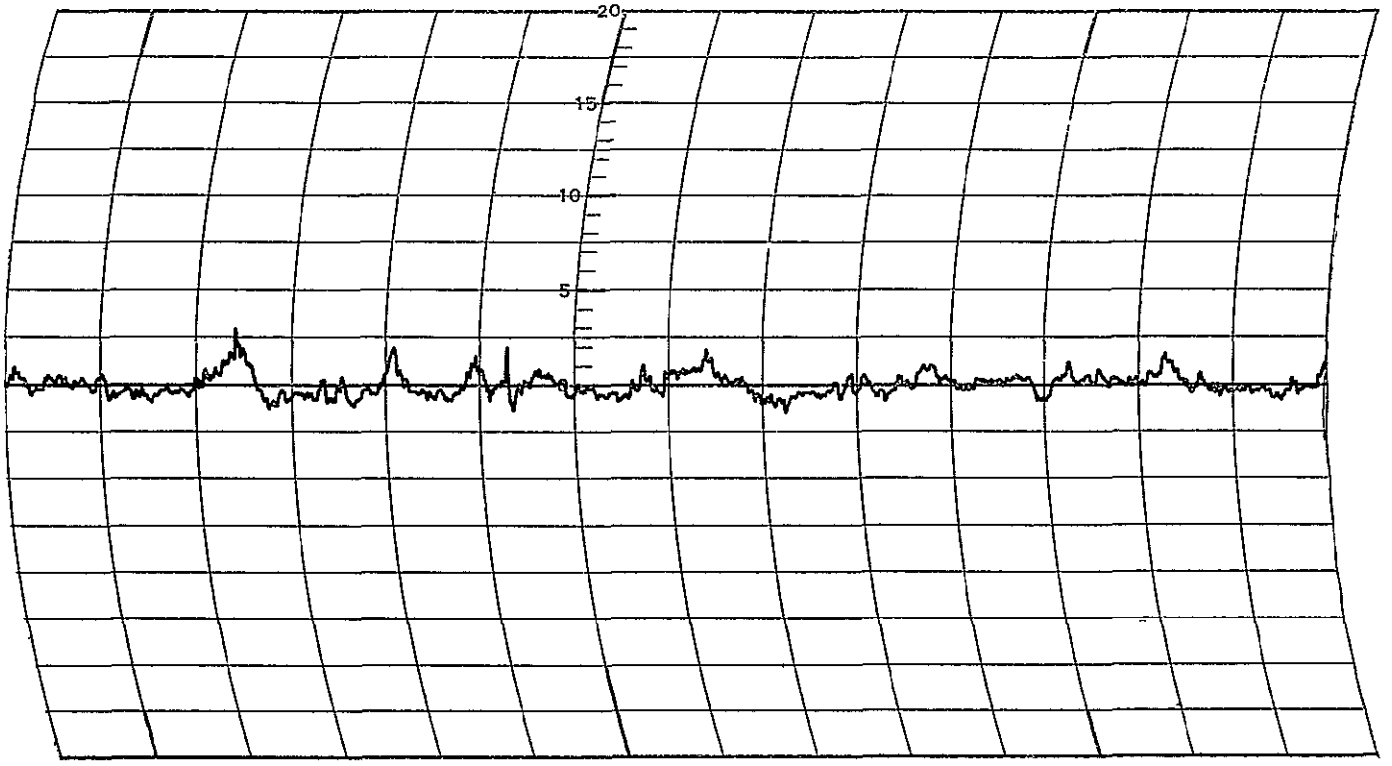
Tape Type	General Use	Thickness Backing (mils)	Thickness Coating (mils)	Yield Strength (lbs)	Breaking Strength (lbs)	Coating Resistance ($x10^6\Omega$/sq)
3M 351	Video	0.92	0.21	16 (1" width)	24 (1" width)	<200
3M 777	Computer	1.42	0.55	11	NS	0.5-100
3M 871	Instru-mentation	0.92	0.32	4.0 (¼" width)	6.8 (¼" width)	<100
3M 888	Instru-mentation	0.92	0.21	4.0 (¼" width)	6.8 (¼" width)	<50
Memorex 63L	Instru-mentation	0.92	0.17	7	12	NS
Memorex 79L	Video	0.92	0.17	14,000 psi	24,000 psi	<50
DuPont Crolyn	Computer	1.42	0.20	11	NS	<10

NS = Not Specified

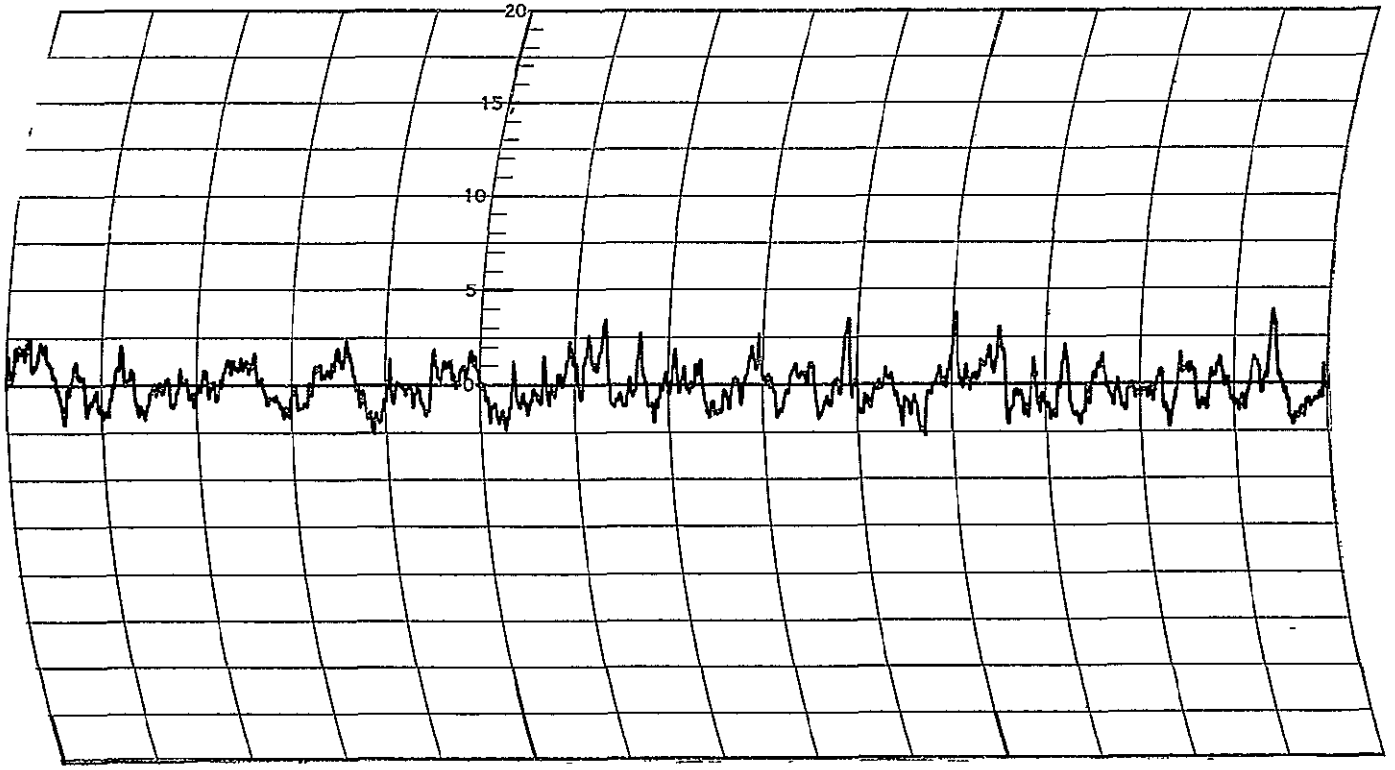
Table VIII (Continued)

Tape Type	Frictional Drag (grams)	Coercivity (Oersteds)	Retentivity (Gauss)	Remanence (Maxwells)	Uniformity	Coefficient of Expansion Thermal $x10^{-5}$/°C	Coefficient of Expansion Hygroscopic $x10^{-5}$/% RH
3M 351	NS	300	900	NS	± 1db (λ = 15mil)	NS	NS
3M 777	130	275		1.45	± 10% (800 bpi)	NS	NS
3M 871	NS	285	985	0.5 (lines/¼")	± 10% (λ = 10mil)	NS	NS
3M 888	NS	290	960	0.32 (lines/¼")	± 5% (λ = 10mil)	NS	NS
Memorex 63L	NS	290	900	0.56	± 1db (λ = 10mil)	2.7	1.1
Memorex 79L	NS	270	1100	NS	± 1db	1.5	1.1
Dupont Crolyn	130	400		1.0	± 10% (800 bpi)	NS	NS

NS = Not Specified



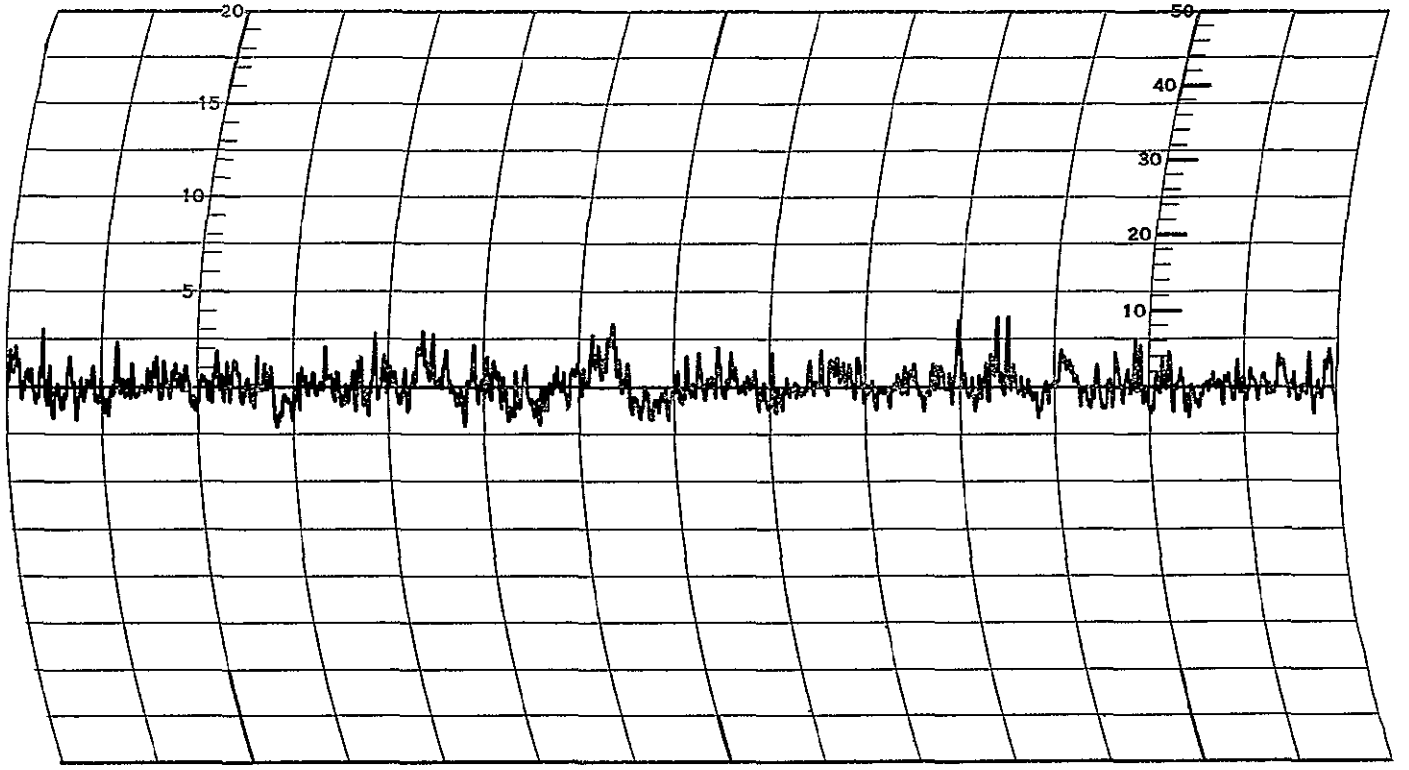
(a) 3M 871



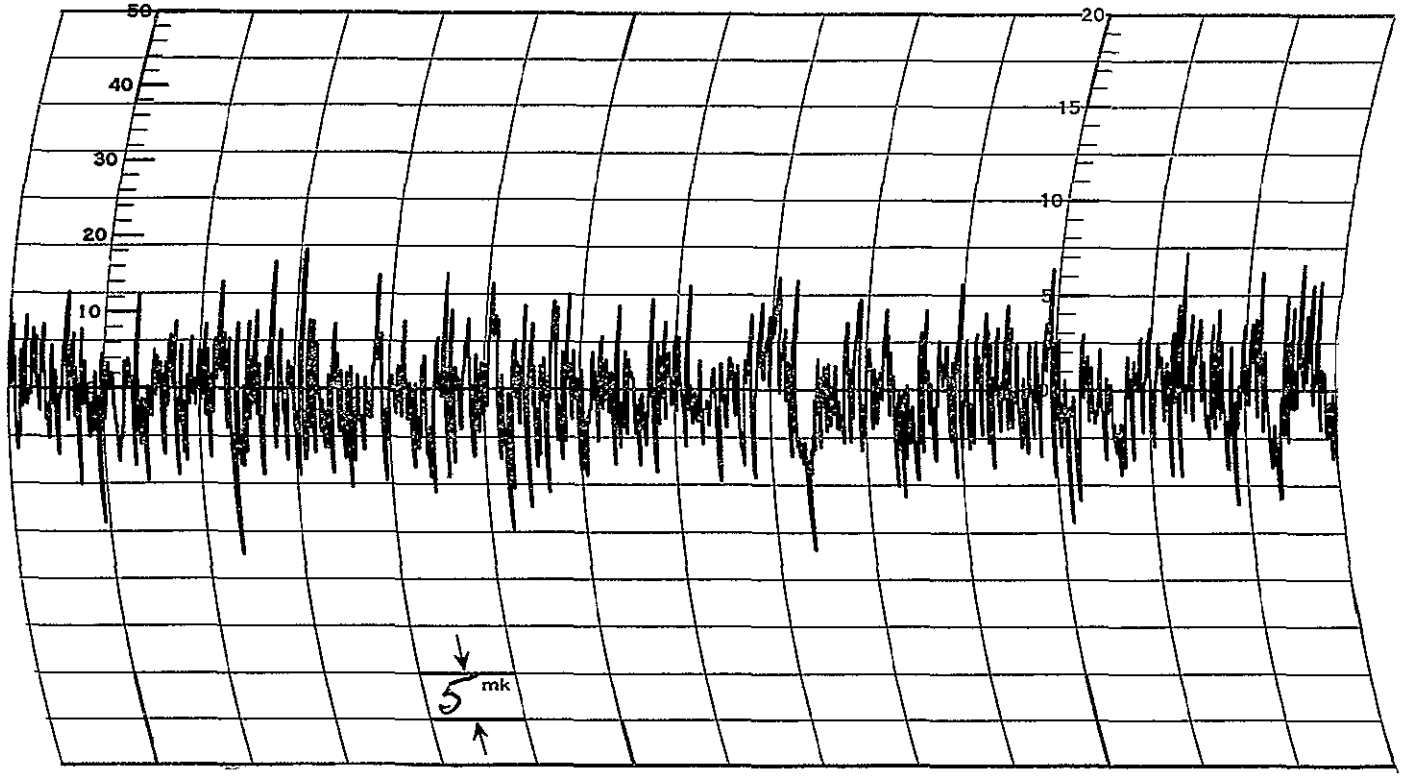
(b) 3M 551

Vertical 5 μin/Div Horizontal 20 mil/Div

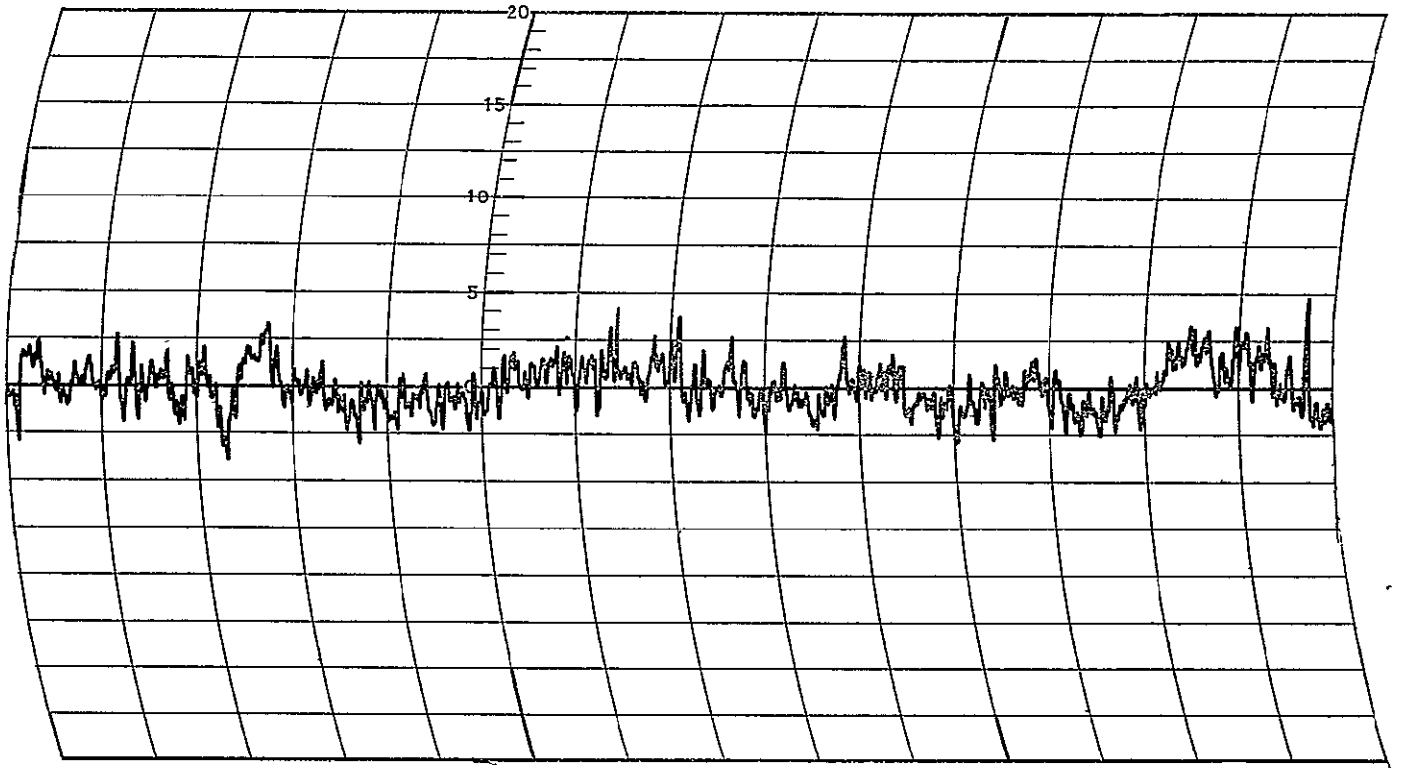
Fig. 20 TAPE SURFACE FINISH



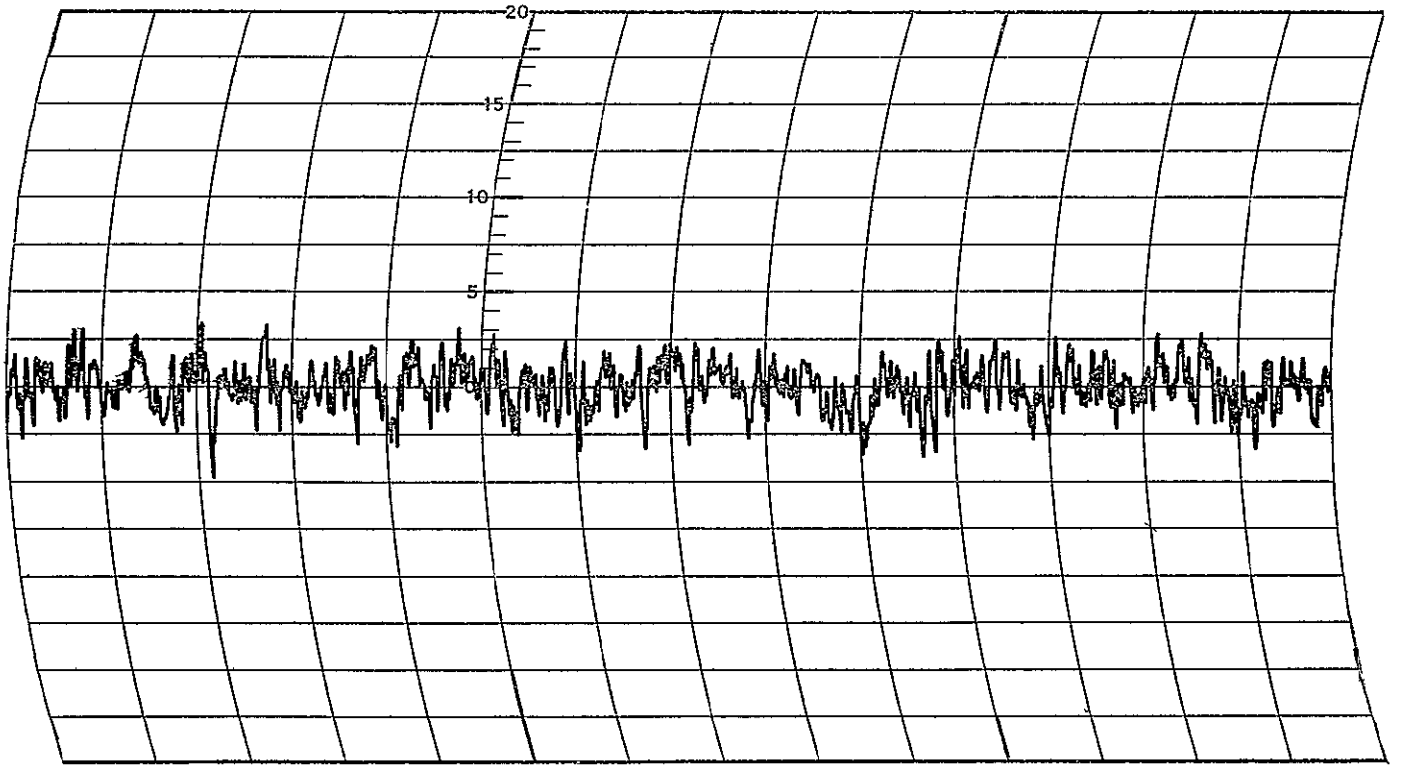
(c) 3M 888



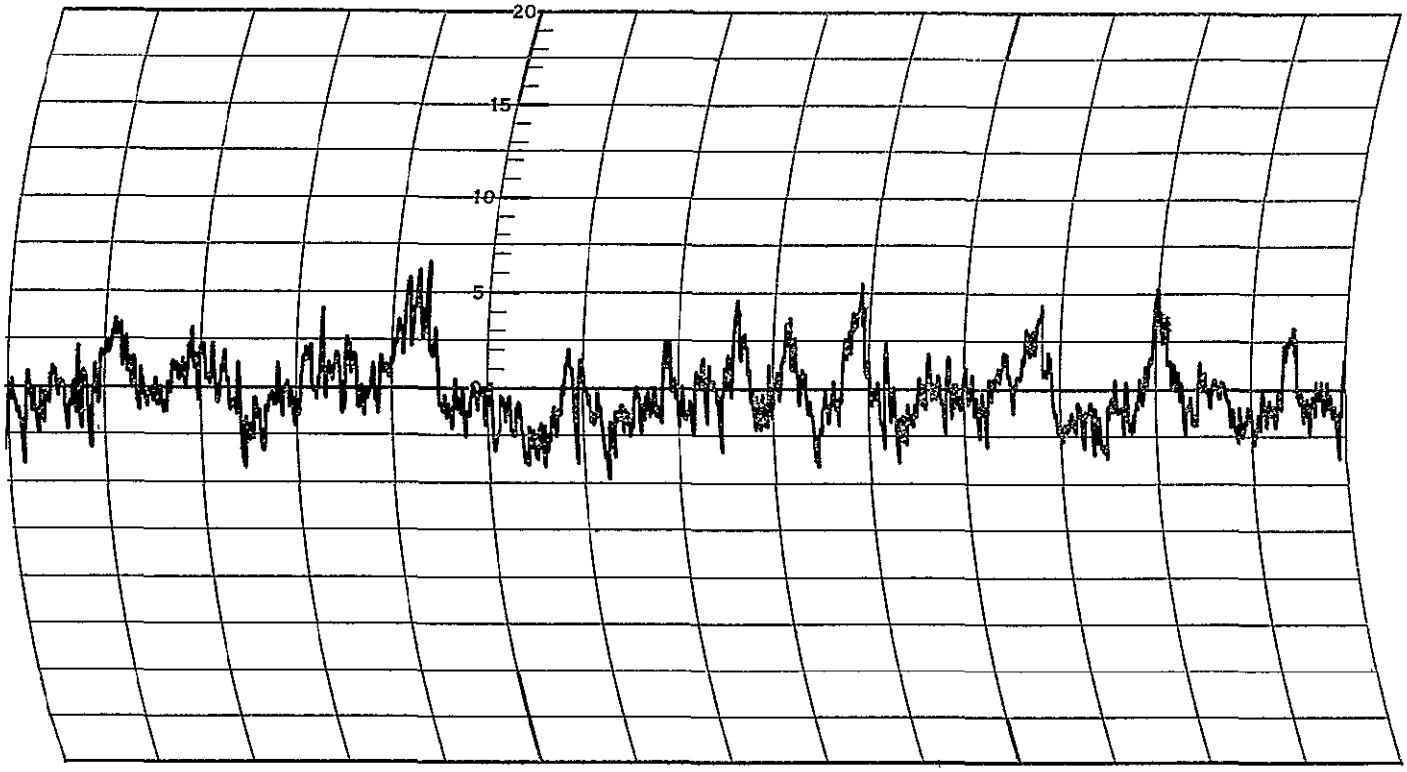
(d) 3M 851



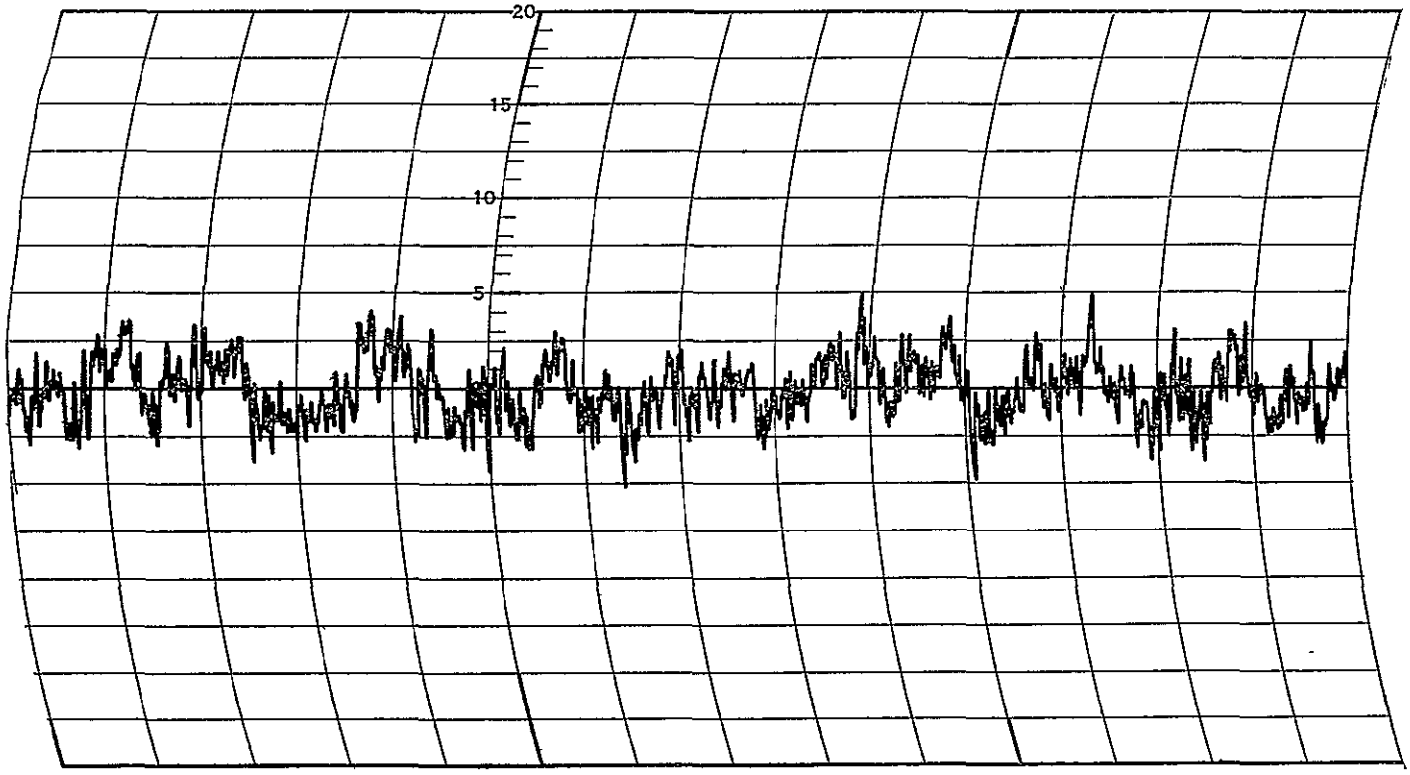
(e) DuPont Crolyn



(f) Memorex Quantum



(g) Memorex 63L



(h) Memorex 79L

Table IX

ANGLE OF FLEXIBILITY MEASUREMENTS

Tape Type		Coordinates Y	Coordinates X	Deflection
3M	551	2.4	1.6	33.7°
	888	2.3	1.72	36.8
	851	2.4	1.55	32.8
	871	2.4	1.55	32.6
	351	2.55	1.20	25.2
	777	1.80	2.35	52.6
Memorex	Quantum	2.27	1.70	36.8
	63L	2.65	1.10	22.5
	79L	2.38	1.65	34.8
RCA	617	2.45	1.46	30.8
	6050	2.43	1.80	31.7
DuPont	Crolyn	2.0	2.1	46.4

C. Surface Resistance

During the test program, the surface resistance of all the tape types was measured at room temperature and at two relative humidities, 0% and 84%. The environments were established by placing a dissicant, $CaSO_4$, or an aqueous solution of KBr in a sealed plastic container. The tapes were placed on a plexiglas table and tensioned with eight-ounce weights on each end, as described in the Guideline Tests, Vol. I. The normal force on the two-inch long probe was eight ounces. Probe resistance was $1.2 \times 10^{14}\Omega$ at 0% RH, and $7 \times 10^{13}\Omega$ at 84% RH. After allowing the tapes to stabilize for 24 hours at each humidity, the results shown in Table X were obtained. The measuring instrument was a Hewlett-Packard High Resistance Meter, Model 4329A, and the relative humidity was maintained using a Hygrodymanics, Inc. Humidity Indicator type 15-3001.

D. Modulus of Elasticity

The modulus of elasticity and yield point for each of the tapes was determined using an Instron Tensile Tester. Operating speed was 0.005 inch/minute. Both complete tapes and the mylar along were measured several times and averaged to reduce experimental error. The modulus of elasticity was determined from the slope of the stress strain curve in the linear region between 0 and 3,000 psi. The yield point was designated as a 0.2% offset from linearity. Test results are shown in Table XI.

E. DC Noise Spectrum

The tape noise of each tape was evaluated by magnetizing the tapes to saturation with a permanent magnet, and then monitoring the resultant output of a standard IRIG head while running the tape at 30 ips. The arrangement of test equipment is shown in Fig. 21. The initial measurements made using this setup were of wideband noise using the VTVM indications. System noise was measured by running the tape out of contact with the head. The

Table X

SURFACE RESISTANCE MEASUREMENTS

Tape Type	Tape Voltage	Tape Resistance 0% Humidity	Tape Resistance 84% Humidity
3M 551	25V	$38 \times 10^7 \Omega$	$46 \times 10^7 \Omega$
Reel #2	50V	37.5×10^7	46×10^7
B & H W-4	10V	1.8×10^6	1.5×10^6
Reel #1	25V	$.18 \times 10^7$	$.15 \times 10^7$
Memorex	25V	325×10^7	382×10^7
79L Reel #1	50V	330×10^7	381×10^7
3M 871	25V	1.85×10^7	2.38×10^7
	50V	1.8×10^7	2.3×10^7
Memorex	25V	3.3×10^7	4.2×10^7
Quantum	50V	3.2×10^7	4.2×10^7
3M 888	25V	1.05×10^7	1.32×10^7
	50V	1.05×10^7	1.35×10^7
	10V	1.05×10^7	1.33×10^7
RCA 788F	25V	36×10^7	41×10^7
	50V	36×10^7	41×10^7
RCA 617	25V	$.22 \times 10^7$	$.25 \times 10^7$
	50V	$.22 \times 10^7$	$.25 \times 10^7$
Epoch IV	25V	2.5×10^7	2.88×10^7
Graham	50V	2.5×10^7	2.82×10^7
3M 351	25V	32.0×10^7	30.5×10^7
	50V	32.1×10^7	30.5×10^7
Memorex 63L	25V	24×10^7	31.8×10^7
	50V	24×10^7	31.6×10^7
3M 777	25V	1.31×10^7	1.85×10^7
	50V	1.3×10^7	1.85×10^7

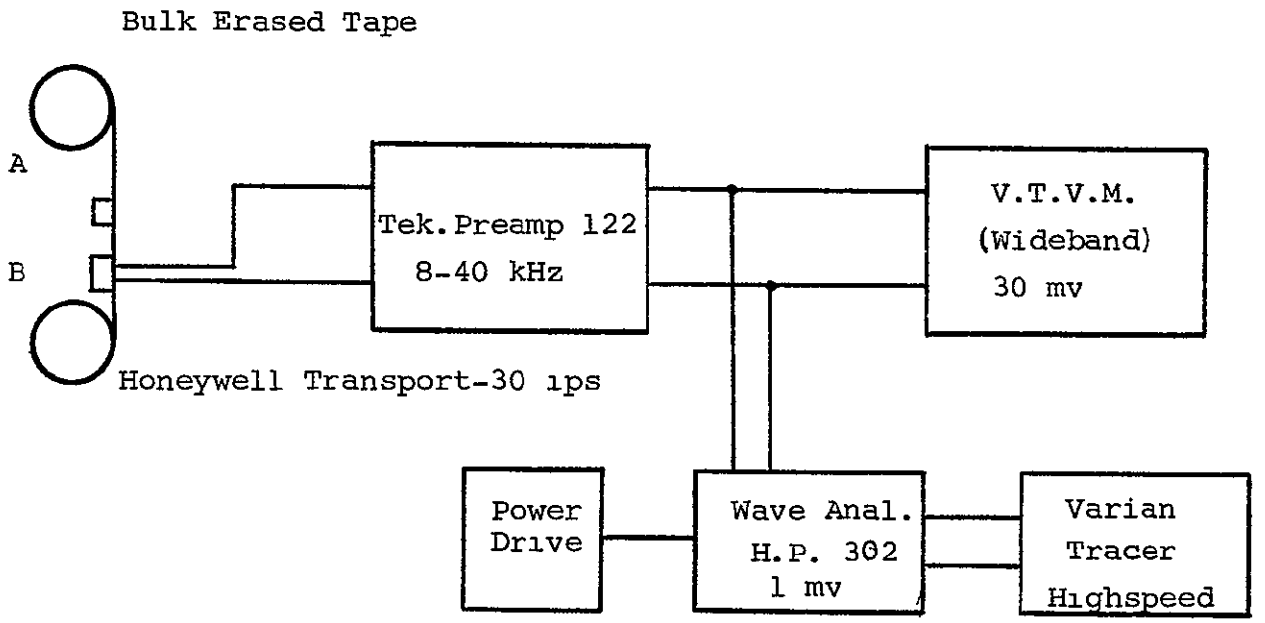
Table XI

INSTRON TESTER RESULTS

Tape Type	Thickness		Modulus of Elasticity	0.2% Offset Yield Point Stress
	Backing (mils)	Coating (mils)	(psi)	(psi)
3M 871	0.92	0.32	510,000	6,600
	0.92	0.32	520,000	6,600
	0.92	0.32	520,000	6,700
	0.92	0.32	520,000	6,300
	0.92	0.32	510,000	6,800
	0.92	0	680,000	8,000
	0.92	0	650,000	9,400
	0.92	0	660,000	9,500
	0.92	0	670,000	9,200
	0.92	0	700,000	8,600
3M 888	0.92	0.21	615,000	7,300
	0.92	0.21	615,000	7,300
	0.92	0.21	565,000	7,900
	0.92	0.21	590,000	7,300
	0.92	0.21	600,000	7,300
3M 351	0.92	0.21	465,000	7,500
	0.92	0.21	520,000	6,700
	0.92	0.21	535,000	6,400
	0.92	0.21	515,000	6,600
	0.92	0.21	505,000	6,800
3M 777	1.42	0.55	515,000	3,700
	1.42	0.55	520,000	3,600
	1.42	0.55	495,000	3,800
	1.42	0.55	455,000	5,400
	1.42	0.55	495,000	4,300
2 Mil Mylar Tape	2.0	0	690,000	11,300
	2.0	0	705,000	10,600
	2.0	0	695,000	11,300
	2.0	0	740,000	6,100
	2.0	0	755,000	5,400
Memorex 63L	0.93	0.17	665,000	6,900
	0.93	0.17	610,000	7,400
	0.93	0.17	630,000	7,100
	0.93	0.17	660,000	6,500
	0.93	0.17	660,000	6,600

Table XI (cont)

Tape Type	Thickness		Modulus of Elasticity	0.2% Offset Yield Point Stress
	Backing (mils)	Coating (mils)	(psi)	(psi)
Memorex 79L	0.93	0.17	570,000	7,200
	0.93	0.17	570,000	7,300
	0.93	0.17	570,000	7,000
	0.93	0.17	575,000	7,100
	0.93	0.17	595,000	7,000
Memorex Quantum	1.42	0.55	525,000	5,400
	1.42	0.55	525,000	5,400
	1.42	0.55	595,000	4,400
	1.42	0.55	585,000	4,500
	1.42	0.55	575,000	4,700
	1.42	0	700,000	6,800
	1.42	0	720,000	6,300
	1.42	0	650,000	8,500
	1.42	0	730,000	6,100
	1.42	0	725,000	6,200
RCA 617	0.91	0.41	410,000	6,200
	0.91	0.41	445,000	5,600
	0.91	0.41	435,000	5,900
	0.91	0.41	445,000	5,700
	0.91	0.41	450,000	5,600
	0.91	0	655,000	7,900
	0.91	0	625,000	9,300
	0.91	0	645,000	9,200
	0.91	0	655,000	9,300
	0.91	0	640,000	9,400
DuPont Crolyn	1.42	0.20	520,000	6,900
	1.42	0.20	515,000	6,700
	1.42	0.20	560,000	5,700
	1.42	0.20	545,000	5,700
	1.42	0.20	535,000	5,800
	1.42	0	650,000	5,800
	1.42	0	660,000	6,100
	1.42	0	630,000	6,600
	1.42	0	625,000	6,900
	1.42	0	625,000	6,700



A: DC magnet

B: Replay head (Honeywell)
Track No. 2

Fig. 21 TEST SET-UP, DC NOISE TEST

results are shown in Table XII. The largest deviation observed was between the 3M 888 and Memorex 79L tapes. Using these two tapes, a number of other frequencies and bandwidths were evaluated in order to maximize the measurable differences. The results of these tests are shown in Figs. 22 and 23. The final bandwidth selected was 10-20 kHz at 30 ips.

F. Oxide Orientation

The magnetic characteristics of each tape were measured using a 60-Hz curve tracer. After both the longitudinal and tranverse directions were measured, the tapes were reversed and remeasured. Thus, a total of four figures for the coercivity, remanence, and saturation magnetization were obtained for each tape. It was hope that comparison of the squareness ratios for each of the directions would indicate a degree of oxide orientation. However, the results, as shown in Table XIII, indicated a similar degree of orientations among all tapes.

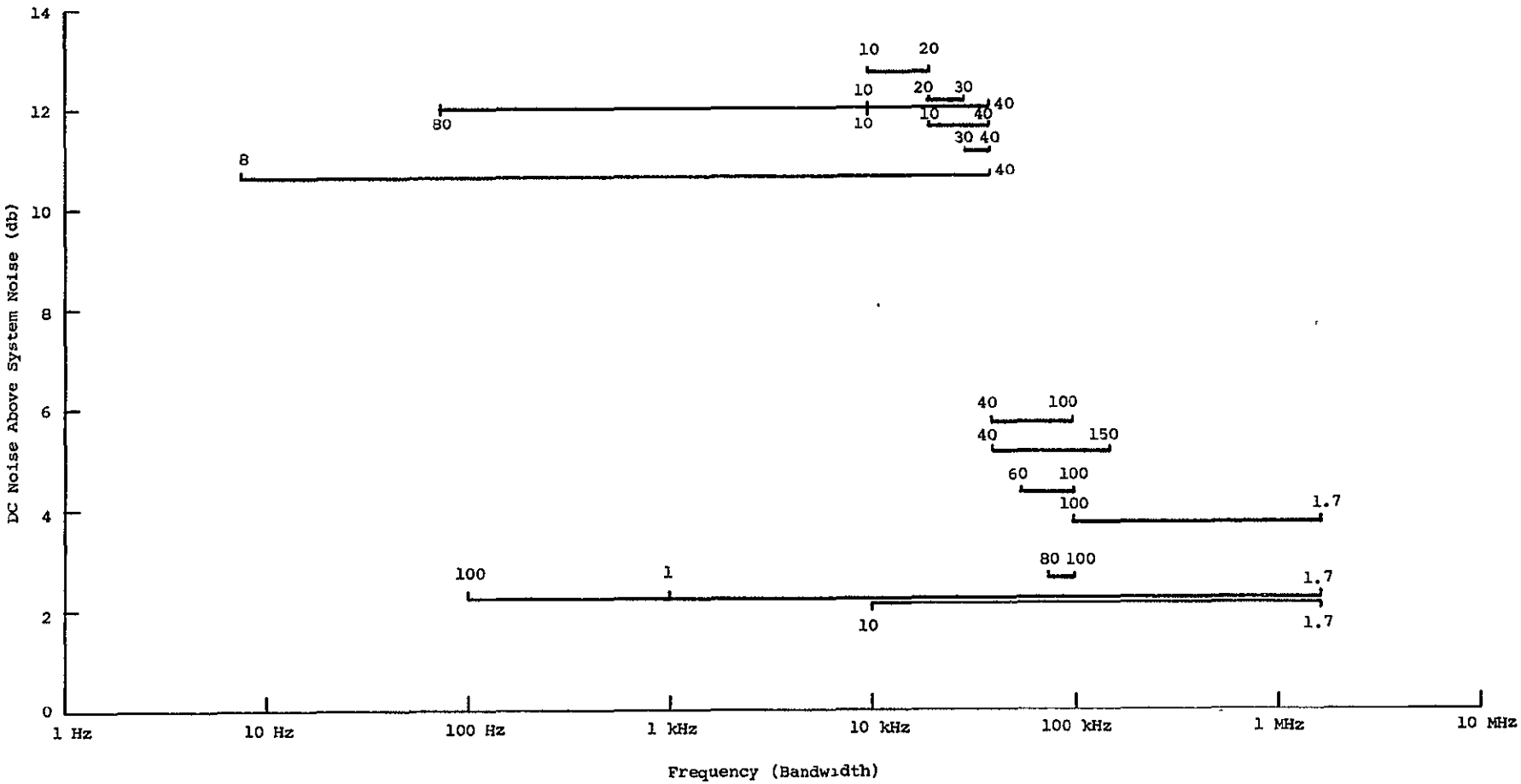


Fig. 22 EVALUATION OF DC NOISE SPECTRUM, TAPE TYPE MEMOREX 79L

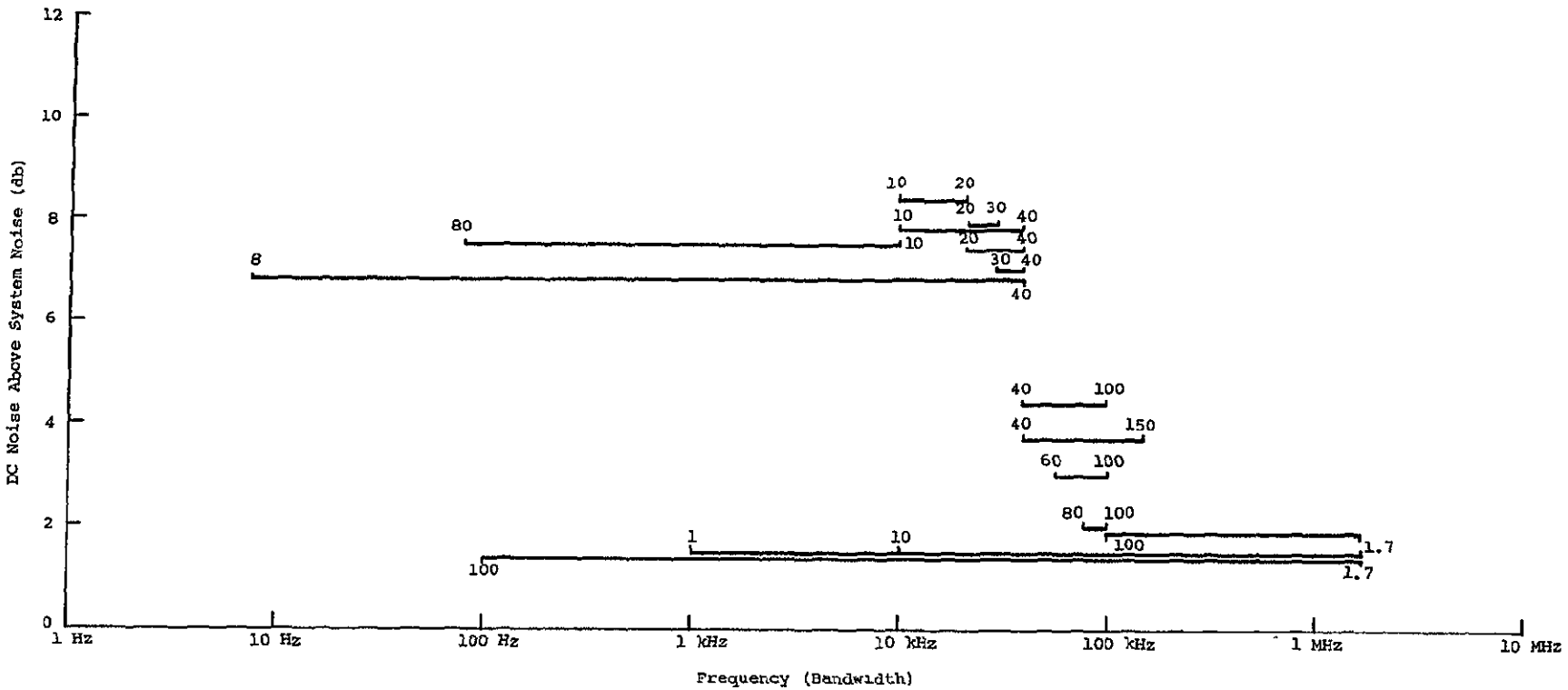


Fig. 23 EVALUATION OF DC NOISE SPECTRUM, TAPE TYPE 3M 888

Table XII

DC NOISE TEST

Tape Type		Wideband Noise	Instrument Noise	Noise Ratio
DuPont Crolyn		-36.9 db	-45.2 db	8.3
3M	551	-38.0	-45.2	7.2
	888	-37.9	-45.0	7.1
	851	-33.6	-45.2	11.6
	871	-36.0	-45.2	9.2
	351	-35.6	-45.2	9.6
	777	-36.6	-45.0	9.4
Mem. Quantum		-35.3	-45.2	9.9
	79L	-34.2	-45.0	10.8
	63L	-34.9	-45.2	10.3
RCA	617	-34.8	-45.3	10.5
	6050	-35.7	-45.2	9.5

Table XIII

MAGNETIC CHARACTERISTICS

Tape	Type	Direction	H_C	B_R	B_M
3M	351	x	300	760	1000
		$\bar{x}$	300	760	1000
		y	230	470	1000
		$\bar{y}$	230	470	1000
3M	551	x	290	790	1000
		$\bar{x}$	290	790	1000
		y	230	480	1000
		$\bar{y}$	230	480	1000
3M	777	x	280	760	1000
		$\bar{x}$	280	760	1000
		y	220	420	1000
		$\bar{y}$	220	420	1000
3M	851	x	270	780	1000
		$\bar{x}$	270	780	1000
		y	220	430	1000
		$\bar{y}$	220	430	1000
3M	871	x	280	790	1000
		$\bar{x}$	280	790	1000
		y	200	410	1000
		$\bar{y}$	200	410	1000
3M	888	x	300	760	1000
		$\bar{x}$	300	760	1000
		y	220	460	1000
		$\bar{y}$	220	460	1000

Table XIII (cont)

Tape Type	Direction	H_C	B_R	B_M
Memorex 63L	x	300	720	1000
	$\bar{x}$	300	720	1000
	y	240	500	1000
	$\bar{y}$	240	500	1000
Memorex 79L	x	270	780	1000
	$\bar{x}$	270	780	1000
	y	210	430	1000
	$\bar{y}$	210	430	1000
Memorex Quantum	x	270	780	1000
	$\bar{x}$	270	780	1000
	y	210	420	1000
	$\bar{y}$	210	420	1000
DuPont Crolyn	x	400	880	1000
	$\bar{x}$	400	880	1000
	y	390	460	1000
	$\bar{y}$	390	460	1000

NOTE: $\bar{x}$ and $\bar{y}$ measurements obtained by physically reversing the sample in the B-H Loop Tester.

SECTION IV

TEST EQUIPMENT

Throughout the test program, two basic types of transports -- the reel-to-reel and the endless loop -- were used as basic test equipment. Three reel-to-reel tape testers, Model 9209, were purchased from the Video Research Corporation, Rockville, Md., while four endless loop transports were fabricated at IITRI. The purpose in utilizing both transport configurations was to first accelerate tape wear and establish trends using the endless loop transports, and then to confirm these trends using longer lengths of tape in a situation more nearly simulating satellite recorders. This approach was found to be very successful throughout the program. The following paragraphs describe the general specifications and calibration procedures for these units.

A. Reel-to-Reel Transports

The reel-to-reel transports used during the program were specifically designed for the testing of magnetic tape. As such, the operation was very versatile in that tape tension, speed, and head position were all variable. The transport was arranged so that only the head contacted the oxide side of the tape. Crowned guidance was used, and the diameters of all the tape path elements were two inches or greater.

The factor measured on this transport was frictional drag. To accomplish this, the head was mounted on a 20-mil thick cantilevered beam equipped with strain gages. Arranged in a bridge configuration, the strain gages allowed measurements of drag as low as 0.05 ounces. This head mounting system, in turn, was mounted on a precision micrometer adjustment to allow changes in head height and penetration. The detailed specifications of the overall tape tester are listed below.

1. Tape Transport

a. Tape Quantity:

1000 ft. of either 1/4 in. or 1/2 in. wide tape, nominally 1 mil thick. Reels shall be 7 in. precision with NAB hubs. Two reels shall be supplied.

b. Tape Speed:

Continuously variable from 0.4 to 60 ips in four ranges.

c. Tape Speed Accuracy:

0.5% maximum deviation from initial setting after 1 hour of operation.

d. Flutter:

0.4 ips	2% P/P	(0.2 to 36 Hz)
7.5 ips	2% P/P	(0.2 to 300 Hz)
30 ips	1% P/P	(0.2 to 5 kHz)

e. Bidirectional Operation:

Adjustable end of reel sensors shall be provided to allow automatic bidirectional operation. Transport shall meet specifications in both directions. No pinch rollers shall be used.

f. Tape Guides:

Crowned guidance shall be utilized.

g. Head Mount:

Universal clamp shall accept heads up to 1 in. wide x 2 in. deep with adjustable positioning in 3 planes. Wrap angle shall have micrometer adjustment from 0° to 30° normal to the tape plane.

h. Dynamic Drag Transducer:

A precision cantilever and strain gage arrangement shall be provided on the head mount to permit head-to-tape friction measurement over the full range of speeds and tensions. Sensitivity on the most sensitive range shall be nominally 10 microamps per ounce of drag.

i. Tape Break Sensor:

A lamp and photocell combination shall be incorporated to sense tape break or physical end of tape. These conditions shall cause the transport to be switched to the STOP mode with power removed from the reel motors.

j. Enclosure:

The recorder shall be mounted in a gasketed case with maximum dimensions of 23 in. x 18 in. x 21 in. The case shall be capable of maintaining a positive pressure of 2 psig. Two valves, located on opposite ends of the 23 in. dimension, shall be supplied.

k. Environmental:

The transport shall operate over a temperature range of -10°C to +65°C with relative humidity from 0 to 99% (no condensation).

2. Control Unit

a. General Description:

A control unit shall be supplied which shall contain the necessary electronics to allow operation of the transport in accordance with the above specifications.

b. Transport Operating Modes:

Backlighted, pushbutton switches shall select Power On-Off, Stop, Forward, and Reverse.

c. Tape Tension Control:

Tape tension shall be variable from 2 ounces to 20 ounces (1/2 in. tape) within 1 ounce of the nominal setting.

d. Tape Speed Control:

Capstan servo control of the tape speed shall be provided to allow variable speed from 0.4 ips to 60 ips. A four-position speed range switch and a vernier dial shall be provided for speed selection.

e. Drag Measurement:

A meter shall be provided which shall indicate drag forces experienced by the cantilevered head mount. Meter range shall be 0-50 microamps. In conjunction, a zero adjust potentiometer and a four-position switch shall be provided. The switch shall select "off" or one of three drag force ranges.

f. Tape Tension Switch:

A toggle switch shall be provided to allow the tape tension to be turned on or off while the transport is in the Stop mode. A reset button shall also be provided to restore tape tension following activation of the tape break switch and subsequent rethreading of the transport.

g. Front Panel Connectors:

BNC connectors shall be provided to allow monitoring of the capstan servo tachometer signal (after squaring) and the drag transducer. A BNC connector shall also be provided from which a positive pulse can be obtained at the time of tape reversal to facilitate counting the number of passes.

h. Enclosure:

The control unit shall be housed in a 19-in. rack enclosure having nominal dimensions of 32 in. x 23 in. x 17 in.

1. Environment:

   Standard laboratory conditions.

3. Interconnecting Cables

   Interconnecting cables between the transport and control unit shall be supplied. The length shall be nominally 15 feet. Connectors shall be potted.

Calibration of the drag sensors for the three units was accomplished by suspending fixed standard weights from the head mount in a manner that applied force in the direction of tape motion. The conversion from the amplifier output in microamps to ounces of drag was:

Unit 1:	10.15 ua = 1 oz
Unit 2:	10.3 ua = 1 oz
Unit 3:	11.35 ua = 1 oz

Some hystersis was seen in the drag sensors between a given tape motion and standby. Further, small (<5%) differences in tape tension occurred between forward and reverse operation due to differences in the peripheral speed of the two capstans. Therefore, all drag measurements were the average between the forward and reverse readings.

The environment of the containers for these transports was controlled throughout the tests. Temperature was maintained using two-500 watt heating rods controlled by a Versa-Therm Model 2157-1, Electronic Indicating Temperature Controller. The sensor was a Versa-Therm Model 2157-6 Platinum Temperature Sensor. Relative humidity was controlled by use of a dessicant, or for the higher humidity tests, physically adding moisture. Humidity was monitored with the Hygrodynamics Humidity Sensor previously mentioned.

B. Endless Loop Tape Testers

The endless loop tape transports were designed to have an essentially square tape path with the oxide on the outside. A single capstan drive was utilized. This capstan was belt coupled to a DC servo-controlled capstan motor. One of the idlers was mounted on a counter balanced arm appropriately weighted to provide tape tension. Crowned tape guidance was used, and roller diameters were 2 inches or greater to minimize tape stresses.

The DC motor, generator, and speed control was Motomatic Model E-550, built by Electrocraft Corp., Hopkins, Minn. The motor generator has two windings on the same armature, one for driving the motor and the other for generating a voltage proportional to speed. This voltage is then compared to a reference set by the speed control adjustment, thereby maintaining constant speed.

Two measurements -- starting and running torque -- were made on the loop transports. These measurements were obtained from the DC capstan motor current. In monitoring starting torque, the speed adjustment was first set for the desired speed (usually 1 ips), with the torque (motor current) limiter turned to zero. The torque limiter was then slowly increased until tape motion began.

The calibration of the torque readings was accomplished by running tape on the transport out of contact with the head. In this manner, losses due to the motor brushes and bearing friction could be subtracted. The torque was expressed in terms of capstan torque, i.e., motor torque times a 5.89 ratio of speed reduction. A typical set of values for motor starting torque without tape drag was:

8.8	8.9	
9.0	9.5	
9.3	9.2	Average 9.28 oz-in at the capstan
9.8	9.3	
9.1	10.0	
9.4	8.9	

Figure 24 shows the variation in running torque with temperature.

Two of the endless loop transports were run in an environmental chamber (Tenney Model TH-10) capable of controlling both temperature and relative humidity. Maximum operating temperature was 65°C. Other operating specifications were:

Tape Speed:	0.37 - 90 ips
Tape Direction:	Bidirectional
Flutter at 30 ips:	0.25% RMS
Tape Tension:	2-20 ounces for 1/2 inch tape
Tape Width:	1/4 or 1/2 inch

A thermo-fusion tape splicer was used to band the free ends of tape forming loops for evaluation on the endless loop transports. This splicer, Model TF4550, Mark II, was built by Prestoseal Manufacturing Corp., Corona, N. Y. Use of this equipment minimized the splice effects possible when using endless loops of tape for evaluation.

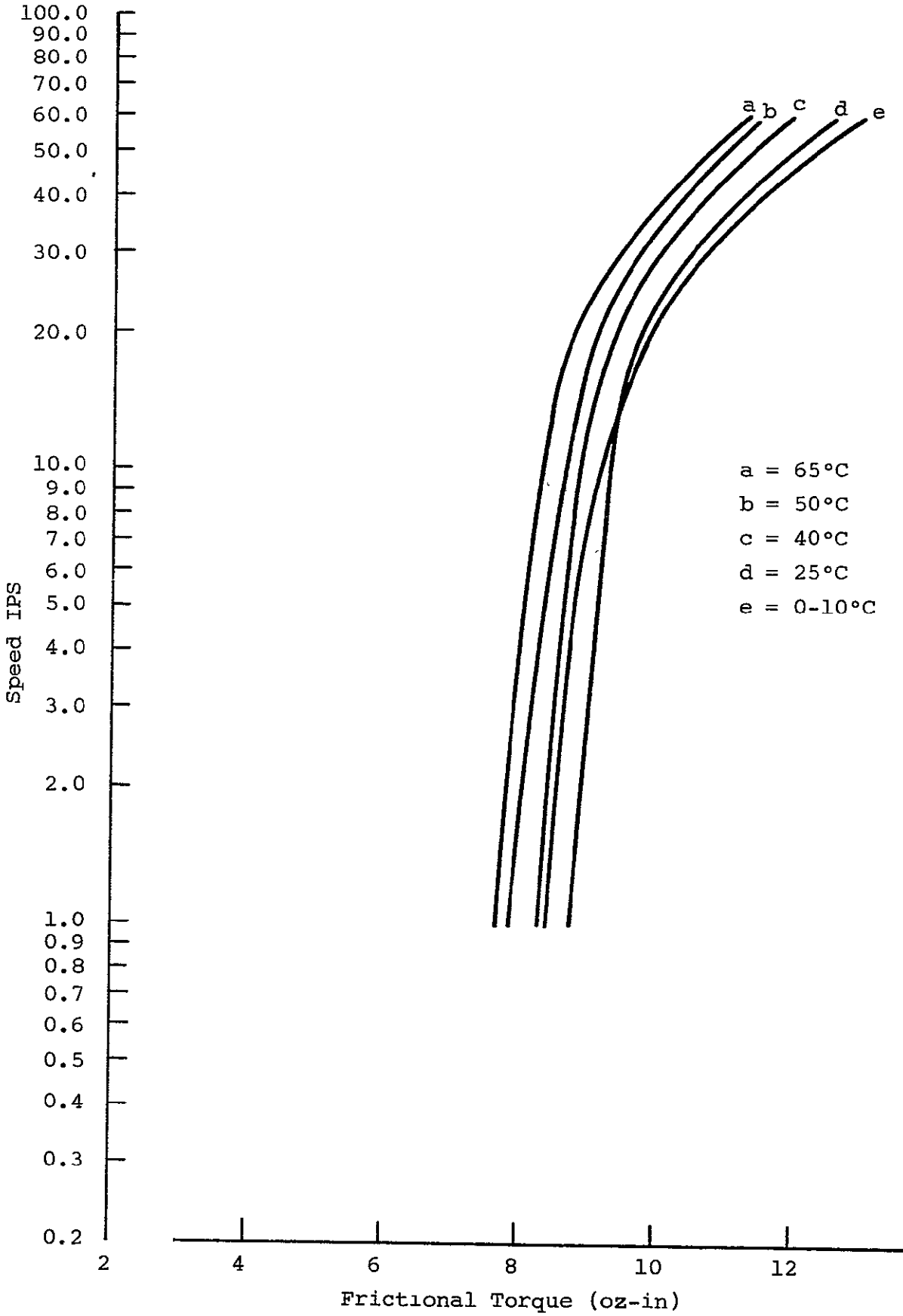


Fig. 24 VARIATIONS IN MOTOR AND BEARING LOSSES WITH TEMPERATURE

SECTION V

TEST PROCEDURES AND SAMPLE RESULTS

The overall results of the Phase II test program are reported in Volume II of this report. Those results, in many cases, represented the interpretation of several tests performed to isolate individual parameters. The procedures used in conducting these tests are described in this section. In addition, sample results of the data obtained are presented.

The majority of tests conducted were intended to study factors relating to head/tape adhesion and oxide/binder debris. Therefore, this section is divided accordingly, even though both drag and debris measurements were obtained during certain of the tests.

A. Head-to-Tape Adhesion Testing

1. Temperature and Relative Humidity

A large matrix of tests was run during the adhesion portion of the test program to determine the relationship between friction and environment. These tests were conducted on the endless loop transports in the Tenney environmental chamber at temperatures of 65, 45, and 25°C. Relative humidities of 45, 30, and 15% were run at the two upper temperatures. However, the 25°C, 15% RH tests were not run because of chamber instability and the lack of significant effects observed at 25°C.

The basic test conditions, initially defined during the failure mode analysis work described in Volume II, remained constant for each tape type tested at each of the environments. The standard test conditions were:

Tape Tension: 12 ounces
Tape Speed: 1 ips and 30 ips
Tape Direction: Bidirectional

Tape Length: 52 inches

Head Material: Brass

Head Radius: 0.25 inch

Wrap Angle: 12°

Definition of Failure: An increase in starting or running torque of 10 oz-in. at the capstan. Capstan radius was 1 in.

Definition of No Failure: 10,000 passes of tape over the head without a 10 oz-in torque increase

The procedure used in running these tests consisted of alternately running the tape in forward at 1 ips and reverse at 30 ips. Starting torque was measured at the beginning of each interval; running torque was monitored at the beginning and end. The number of passes at the two speeds was defined as follows:

No. of Passes	Tape Speed ips	Tape Direction
0 - 1040	30	CCW
1040 - 2110	1	CW
2110 - 3670	30	CCW
3670 - 3720	1	CW
3720 - 5800	30	CCW
5800 - 5870	1	CW
5870 - 7950	30	CCW
7950 - 8020	1	CW
820 - 10,100	30	CCW
10,100 Retest	1	CW

It was found that the conditions and procedures imposed, though more severe than would be normally used in a flight recorder, provided a valuable range of performance for the various tape types. Therefore, particularly the results at the upper temperatures and humidities were also used to establish initially the tape related trends discussed in Volume II.

Tables XIV(a) through (q) are sample results of the torque measurements obtained at various environments. It can be seen that, under the test conditions used, certain tapes were operated successfully for 10,000 passes, while others exhibited high frictional drag after considerably fewer passes.

2. Single Direction Tests

Tests were also conducted on the endless loop transports with the tape running in only one direction. These tests were initially intended to isolate the directionality factor. However, it was found that calibration results were extremely repeatable, and therefore, the head material tests were also run unidirectionally. The procedure used for these tests involved running the tape at 30 ips to accumulate passes, but periodically monitoring the starting and running torque both at 30 ips and 1 ips. The test intervals were every 200 passes up to 1000, followed by every 1000 passes up to 10,000. Sample test results are shown in Table XV(a) through (d).

3. Coating Sensitivity

In addition to the large subsets of data obtained with the loop transports, several small tests were run to isolate individual parameters. One such test was the evaluation of a possible preferred direction due to the coating process. Two loops of each tape type were fabricated and designated A or B, depending upon the coating direction. Both tapes were run 100 passes in the same transport direction; therefore, loop A was run in the direction of tape coming off the reel, and loop B the opposite. After 100 passes, the tapes were physically reversed on the transports, and the new starting and running torques measured. These tests were run both at 25°C and 65°C, but as can be seen from the 65°C data, little correlation was observed between coating direction and performance. Table XVI(a) through (e) shows the test results.

Table XIV

RESULTS OF TORQUE MEASUREMENTS
OBTAINED AT VARIOUS ENVIRONMENTS

(a) Tape 888
65°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	4.8 oz in	0.5 oz in
1040	0.8 oz in	30**	1*	3.8	2.0
5440	1.5	1	30	2.2	0.7
7520	0.3	30	1	7.2	4.7
7590	1.7	1	30	3.9	0.4
9670	0.5	30	1	1.8	4.8

(b) Tape 871
65°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	0.9 oz in	0.8 oz in
1040	0.8 oz in	30	1	4.0	2.1
2140	1.1	1	30	1.6	1.2
3700	0.9	30	1	3.0	2.0
3750	1.3	1	30	1.6	1.1
5830	0.9	30	1	5.2	2.1
5900	1.5	1	30	2.7	1.0
7980	0.8	30	1	5.5	2.1
8050	1.5	1	30	4.0	1.3
9090	0.8	30	1	7.0	2.1
9190	1.4	1	30	3.2	1.0
10230	0.8	30	1	6.6	2.1

*1 ips run clockwise for measurements in Table XIV
**30 ips run counter-clockwise

Table XIV (Continued)

(c) Tape 617K
65°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	2.2 oz in	0.8 oz in
1040	1.9 oz in	30	1	11.3	6.8
2140	5.7	1	30	7.5	1.8
3700	2.3	30	1	26	14

(d) Tape Crolyn
65°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	0.9 oz in	0.4 oz in
1040	0.9 oz in	30	1	3.1	1.3
2140	2.7	1	30	3.4	1.3
3700	1.4	30	1	4.6	2.5
3750	2.9	1	30	3.6	1.5
5830	1.3	30	1	4.9	2.8
5900	2.9	1	30	3.5	1.4
7980	1.3	30	1	4.9	2.6
8080	2.9	1	30	3.4	1.3
10120	1.6	30	1	5.2	3.0

Table XIV (Continued)

(e) Tape 63L
65°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ıps	0.5oz ın	0.8oz ın
1040	0.7 oz ın	30	1	4.1	2.8
2110	1.6	1	30	4.4	1.3
3670	0.6	30	1	8.5	2.7
3720	2.0	1	30	4.6	0.6
5800	0.6	30	1	11.5	2.8
5870	2.1	1	30	6.6	0.8
7950	0.6	30	1	>22	---

(f) Tape 3M 888
65°C, 15% RH

Passes	Final	Old	New	Start	Run
0	---	0	30 ıps	4.0oz ın	1.2oz ın
1040	1.2 oz ın	30	1	2.6	1.6
2280	1.0	1	30	2.1	1.0
3810	1.3	30	1	4.3	1.8
3880	1.3	1	30	6.3	1.1
6480	1.8	30	1	2.1	2.1
6570	1.3	1	30	1.9	1.2
9170	1.9	30	1	5.5	2.3
9220	1.5	1	30	1.0	1.3
10260	1.8	30	1	5.4	2.2

Table XIV (Continued)

(g) Tape 3M 871
65°C, 15% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	0.9oz in	1.0oz in
1040	2.4 oz in	30	1	2.1	1.5
2180	1.2	1	30	4.5	1.6
4260	2.4	30	1	4.9	1.8
4520	1.0	1	30	6.3	1.6
6600	2.9	30	1	7.5	1.7
11170	1.8	1	30	2.9	1.9
12210	2.6	30	1	3.9	2.0

(h) Tape MEM 79L
65°C, 15% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	3.4oz in	0.4oz in
1040	1.3 oz in	30	1	4.6	1.8
1110	0.9	1	30	1.3	0.7
2150	1.3	30	1	7.1	2.0
3310	1.3	1	30	5.9	0.9
4870	1.7	30	1	7.1	1.9
4960	1.3	1	30	5.4	0.9
7040	2.5	30	1	17.0*	---

*Restart after 2 hrs., 28.0 WET STICK

Table XIV (Continued)

(i) Tape MEM 63L
65°C, 15% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	3.5 oz in	0.8 oz in
1040	1.7 oz in	30	1	3.5	2.5
2180	1.7	1	30	1.5	1.4
3740	1.6	30	1	7.5	3.5
3790	1.8	1	30	1.4	1.5
5350	1.5	30	1	14.6	3.8
5420	1.8	1	30	5.9	1.3
6450	1.9	30	1	28	3.8

(j) Tape RCA 617
65°C, 15% RH

Passes	Final	Old	New	Start	Run
0	---	---	30 ips	3.7 oz in	1.6 oz in
1040	2.4 oz in	30	1	7.1	4.1
2140	2.6	1	30	6.0	2.6
3700	4.3	30	1	21.0	13
3770	4.6	1	30	5.7	3.0
6370	5.0	30	1	19	12

Table XIV (Continued)

(k) Tape Crolyn
65°C, 15% RH

Passes	Final	Old	New	Start	Run
0	---	---	30	1.5 oz in	1.2 oz in
1040	1.8	30	1	4.1	2.1
5420	1.5	1	30	6.0	1.6
6980	2.0	30	1	3.5	2.8
7050	1.7	1	30	6.3	1.6
9650	2.5	30	1	7.0	2.7
9740	1.7	1	30	4.8	1.6
10780	2.3	30	1	6.6	2.7

(l) Tape 888
45°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30	0.7 oz in	0.4 oz in
1040	0.5	30	1	2.5	1.6
2140	1.2	1	30	1.2	0.0
3700	0.3	30	1	2.1	1.3
3750	1.1	1	30	2.5	0.2
5830	0.0	30	1	1.9	1.4
5900	1.2	1	30	1.0	0.0
7980	0.1	30	1	1.6	1.6
8050	1.2	1	30	1.0	0.0
10120	0.2	30	1	0.7	1.3

Table XIV (Continued)

(m) Tape 871
45°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30	0.9 oz in	0.7 oz in
1040	0.9	30	1	3.9	1.7
2140	1.9	1	30	2.8	1.1
3700	0.9	30	1	5.0	2.2
3750	1.6	1	30	1.9	0.9
5830	0.9	30	1	4.6	2.7
5900	1.8	1	30	2.3	0.8
7980	1.0	30	1	6.4	3.2
8050	1.9	1	30	2.6	0.9
10120	1.1	30	1	4.9	2.9

(n) Tape 79L
45°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30	3.0 oz in	0.7 oz in
1040	0.5	30	1	3.3	1.3
2110	2.8	1	30	5.2	0.3
3670	0.5	30	1	8.6	3.0
3720	2.5	1	30	6.0	0.5
5800	0.3	30	1	10.9	3.7
5870	2.3	1	30	7.2	0.3
7950	0.5	30	1	14.4	5.0

Table XIV (Continued)

(o) Tape 63L
45°C, 30% RH

Passes	Final	Old	New	Start	Run
0	---	---	30	0.9 oz in	0.6 oz in
1040	0.9	30	1	0.9	1.4
2110	2.7	1	30	6.0	1.2
3670	0.8	30	1	14.9	3.4
3720	2.3	1	30	6.9	1.0
5800	1.2	30	1	14.9	3.3
5870	2.2	1	30	4.2	1.1
7950	1.1	30	1	15.0	3.2

(p) Tape 777
25°C, 45% RH

Passes	Final	Old	New	Start	Run
0	---	---	30	0.5 oz in	1.2 oz in
1040	1.4	30	1	1.7	0.8
2130	1.6	1	30	0.9	1.6
3690	0.9	30	1	0.6	0.8
3740	0.9	1	30	0.5	1.2
5820	1.0	30	1	0.3	0.7
5890	0.8	1	30	0.3	1.1
7970	0.9	30	1	0.4	0.5
8040	0.8	1	30	0.1	1.0
10120	0.8	30	1	0.2	0.5

Table XIV (Continued)

(q) Tape Quantum
25°C, 45% RH

Passes	Final	Old	New	Start	Run
0	---	---	30	3.2 oz in	1.2 oz in
1040	1.3	30	1	0.9	1.2
2130	1.5	1	30	1.7	1.0
3690	1.0	30	1	1.0	1.2
3740	1.3	1	30	0.9	0.8
5820	0.7	30	1	1.3	1.1
5890	1.2	1	30	0.9	0.6
7970	0.8	30	1	0.8	0.8
8040	0.9	1	30	0.9	0.4
10120	0.8	30	1	0.3	0.6

Table XV

ENDLESS LOOP TRANSPORT TESTS

(a) 63L Unidirectional Test
Brass Head 65°C, 45% RH
Unit 2 MEM 63L-1

Passes	Start	Run 1 (ips)	Run 30 (ips)
0	2.7 oz-in	2.4 oz-in	1.0 oz-in
200	2.7	1.9	0.9
400	2.8	1.7	1.0
600	3.0	2.0	0.9
800	3.1	1.9	0.7
1000	4.4	2.2	0.7
2000	5.0	2.5	0.6
3000	7.6	2.9	0.6
4000	5.6	2.7	0.7
5000	6.5	3.2	0.1
6000	5.6	2.8	0.7
7000	7.4	3.2	0.7
8000	6.9	2.9	0.6
9000	9.1	3.2	0.7
10,000	7.2	2.9	0.7

Calibration

Beginning start = 6.4, run 1 = 5.8, run 30 = 6.5

End start = 6.4, run 1 = 5.8, run 30 = 6.5

Table XV (Continued)

(b) 3M 888 Unidirectional(CCW)
Aluminum Head 65°C, 45% RH
Correlation Type Test on
Unit 1

Passes	Start	Run 1 (ips)	Run 30 (ips)
0	0.7 oz-in	0.6 oz-in	1.2 oz-in
200	0.8	0.8	1.4
400	1.9	1.1	1.4
600	1.9	1.4	1.5
800	1.5	1.5	1.5
1000	1.4	1.7	1.7
2000	2.9	2.4	2.2
3000	3.5	2.8	2.9
4000	4.1	2.8	2.9
5000	4.7	2.7	3.1
6000	4.3	2.4	2.7
7000	4.5	2.9	3.2
8000	4.3	2.6	2.6
9000	3.6	2.4	2.4
10,000	3.5	2.6	2.4

Table XV (Continued)

(c) MEM 64L-1 (200 pass lap)
Bidirectional Correlation
Type Test 65°C, 45% RH
Brass Head, Unit 2

Passes	Tape Direction	Start	Run 1 (ips)	Run 30 (ips)
0	CW	1.1 oz-in	1.4 oz-in	1.2 oz-in
20	CCW	1.8	1.9	1.3
200	CW	4.5	2.5	1.3
220	CCW	6.7	3.6	1.5
400	CW	5.0	2.9	1.4
420	CCW	6.2	4.2	1.5
600	CW	5.2	3.3	1.4
620	CCW	8.8	5.6	1.9
800	CW	5.6	3.4	1.4
820	CCW	9.0	5.6	1.8
1000	CW	6.0	3.5	1.4
1020	CCW	---*	---	---

*Tape Broke

Motor Torque

0	CW	6.7	6.6	7.2
20	CCW	7.1	6.6	7.2
200	CW	6.9	6.5	7.1
220	CCW	7.4	6.5	7.0
400	CW	7.3	6.5	7.0
420	CCW	7.3	6.5	7.1
600	CW	7.3	6.6	7.1
620	CCW	7.4	6.6	7.1
800	CW	7.4	6.8	7.2
820	CCW	7.5	6.6	7.1
1000	CW	7.4	6.7	7.2
1020	CCW	7.4	6.6	7.2

Table XV (Continued)

(d) MEM 63L-1 Unidirectional Correlation Type Test (CCW only) Brass Head; 65°C < 5% RH - Unit 2

Passes	Start	Run 1 (ips)	Run 30 (ips)
0	1.4 oz-in	1.6 oz-in	1.3 oz-in
200	0.7	1.1	1.0
400	0.8	1.1	1.0
600	0.8	1.1	1.1
800	0.8	1.1	1.0
1000	0.8	1.1	1.0
2000	0.9	1.2	1.1
3000	0.9	1.2	1.1
4000	0.8	1.2	1.1
5000	0.9	1.1	1.0
6000	1.0	1.1	1.0
7000	0.9	1.2	1.1
8000	1.0	1.1	1.0
9000	1.0	1.2	1.1
10,000	1.0	1.1	1.2

Motor Torque

Beginning start = 6.3, run 1 = 5.9, run 30 = 6.7

Final start = 6.3, run 1 = 5.9, run 30 = 6.7

Table XVI

COATING SENSITIVITY TESTS

(a) Crolyn

		A	B
0 Pass	Start	1.2	1.2
	Run	1.0	1.0
100 Pass	Start	0.7	1.5
	Run	0.9	0.8
Rev.	Start	1.1	0.7
	Run	0.9	0.8

(b) 3M 888

		A	B
0 Pass	Start	2.0	1.5
	Run	0.7	1.1
100 Pass	Start	1.6	1.2
	Run	1.3	1.1
Rev.	Start	1.4	1.5
	Run	1.3	1.1

(c) 3M 351
Head No. 15

		A	B
0 Pass	Start	1.4	1.3
	Run	1.2	1.1
100 Pass	Start	1.4	1.6
	Run	1.2	1.2
Rev.	Start	1.1	2.0
	Run	1.0	1.3

Direction "A" was the direction of tape coming off the reel.
Direction "B" was the opposite.

Table XVI (Continued)

(d) MEM 79L

		A	B
0 Pass	Start	1.6	1.5
	Run	1.5	1.3
100 Pass	Start	1.6	1.5
	Run	1.3	1.2
Rev.	Start	1.4	1.1
	Run	1.4	1.2

(e) MEM 63L

		A	B
0 Pass	Start	2.2	1.8
	Run	1.8	1.6
100 Pass	Start	1.2	1.4
	Run	1.0	1.1
Rev.	Start	1.6	1.6
	Run	1.5	1.4

Interpretation of RESULTS

$$C = \frac{A_2B_3 - A_3B_2}{\Sigma\ 2/4}$$

$$\Sigma = A_2 + A_3 + B_2 + B_3$$

		25°C, 15% RH		65°C, 15% RH	
		Start	Run	Start	Run
Crolyn	+ 0.25	0		-0.29	0
3M 888	+ 0.050	0		+0.088	0
3M 351	- 0.0070	-0.019		+0.11	+0.065
MEM 63L	- 0.125	+0.024		-0.039	-0.040
MEM 79L	+ 0.14	+0.18		-0.093	-0.018

4. Reel-to-Reel Tests

In conjunction with the endless loop tests, further evaluations were conducted on the VRC reel-to-reel transports described in Section IV. These transports permitted the use of longer lengths of tape, and were used primarily to verify the results obtained on the loop transports. The tape length was adjusted to allow one minute per pass at 30 ips, or 150 feet. The transports were enclosed in sealed containers with a dessicant (C_aSO_4) to maintain 0% relative humidity. Temperature was controlled, and on one of the units, the atmosphere could be purged and replaced with nitrogen.

The flexibility of the reel-to-reel transports aided in the measurement of effects due to changes in such parameters as tape speed, tape tension, and wrap angle. Therefore, most of the tests for these factors were performed on the VRC transports. The variable measured was frictional drag, as monitored by a cantilevered beam instrumented with strain gages. Strip chart recordings were used to measure starting force. Table XVII (a)-(e) contains sample results of data taken on these transports.

5. Static Tests

The preliminary evaluation of the effects of head material in the adhesion interaction was accomplished in a series of 24-hour static tests. Round bars (one-inch diameter) of the various materials being considered were lapped to a finish consistent with the front face of a magnetic head. Samples of each tape type were then hung over the bars with eight-ounce weights on each end. After 24 hours at the predetermined temperature, the tapes were carefully pulled with a spring scale until motion was observed. Breakaway forces for the tapes at several temperatures are shown in Table XVIII(a) and (b).

Table XVII

REEL-TO-REEL TEST RESULTS

(a) Tension, Wrap Angle, and Head Radius

3M 871 (Reel No.2) on VRC No.2, 45°C at ~ 0% RH

Tension - 4 oz Penetration - 0.574 in
Wrap - 5° Radius - 0.125 in

Number of Passes	Tape Speed (ips) 0.5	1.0	2.0	10	30	Comment
0	4.0	4.5	4.5	5	5	
200	4.0	4.5	4.5	5	5	No debris
1,200	3.5	4	4	4	4.5	
1,600	4	4	4	4	4.5	
2,050	4	3.5	4	4	4.5	
6,050	3.5	4	4	4.5	5	
8,000	4	4	4.5	5	5.5	
9,000	4.5	4	4	4.5	5.0	
10,500	4.0	4	4.5	5	5	

Conversion: 20.6 = 1 oz Drag

Debris Weight

Insert after test - 1.511225
Debris removed - 1.511141

84 μ grams

Head Wear

Center Average = 0.125 mil
Center Range = 0.068 - 0.185 mil
Edge No. 1 = 0.220 mil
Edge No. 2 = 0.360 mil
Edge Average = 0.290 mil

Table XVII (Continued)

(b) Tension, Wrap Angle, and Head Radius

3M 871 (Reel No.2) on VRC No.2, 45°C at ~ 0% RH

Tension - 8 oz Penetration - 0.574 in
Wrap - 5° Radius - 0.250 in

Number of Passes	Tape Speed (ips) 0.5	1.0	2.0	10	30	Comment
0 0	11	11	11.5	12	12	
200	9.5	10	10	10.5	11	No Debris
1,200	10	10	11	11.5	12	
2,480	10	11.5	12.5	13	14	
3,730	11	11	12.5	14	14.5	
4,930	12.5	12.5	13	14.5	15	
5,340	12.5	12.5	13	14	15	
7,700	12.0	14.0	14.5	15	15	
8,950	13.0	14	14.5	15.5	15.5	
10,200	13.5	13.5	14.5	15.5	15.5	

Conversion: 20.6 = 1 oz Drag

Debris Weight (Al No. 19)

Insert after test - 8.351632
Debris Removed - 8.351516

116 μgrams

Wear

Center - 200 μin
Edge 1 - 2.150 mil
Edge 2 - 1.02 mil
Edge Average - 1.575 mil

Table XVII (Continued)

(c) Tension, Wrap Angle, and Head Radius

3M 871 Tape on VRC No. 2 at 45°C, ~ 0° RH

Tension - 4 oz Penetration - 0.92 in
Wrap - 8° Radius - 0.250 in

Number of Passes	Tape Speed (ips) 0.5	1.0	2.0	10	30	Comment
0	7.5	7.5	8	8	7	
200	8.5	9	9	9	9	No Debris
1,260	7	8	8	8.5	8.5	
1,680	7.5	8	8	8.5	9	
2,690	7.5	8	8	8.5	9	
4,050	7.5	9	8.5	9	9	
4,450	7.5	8	8.5	9	9.5	
5,770	8	8	9	10	10	
6,765	8.5	9	9.5	10	10.5	
8,100	8.5	9	10	10.5	11	
9,330	9	9.5	10	11	12	
9,770	9	10	10.5	11.5	12	
10,000	9	10	10	11	12	

Conversion: 20.6 = 1 oz Drag

Debris Weight (AL No. 19)

Insert after test - 8.369613
Debris Removed - 8.369505
108 μgrams

Head Wear

Center - 0.150 mil
Edge 1 - 1.280 mil
Edge 2 - 1.100 mil
Average Edge - 1.190 mil

Table XVII (Continued)

(d) Guideline Verification, B & H W-4

B & H W-4 Tape, Reel No. 1, 45°C - Brass Head

Tension - 8 oz Wrap Angle - 12°

Number of Passes	Tape Speed (ips) 0.25	1	2	10	30	Comment
0	32	33	39	32	6	
200	45	44	43	40	5	
Debris on both sides						Debris not removable, dry Q-tip
1,200	43	41	42	25	5	Heavy debris both sides - evenly distributed
2,340	38	45	47	32	3	Very heavy debris on both sides

Tape would not track on crowned roller - rode up take-up reels. Heavy accumulation of debris on capstans, rollers, and tachometer.

Debris on head appeared to have molten look under magnification, not unlike droplets of solder.

Table XVII (Continued)

(e) B & H W-4 Tape, Reel No. 1, 35°C - Brass Head
Tension - 8 oz Wrap Angle - 12°

Number of Passes	Tape Speed (ips) 0.15	0.25	1	2	10	30	Comment
0	32	29	32	29	20	3	
1,340	34	29	36	36	19	1	Some debris and varnish on both sides
3,100	24	23	30	31	15	1	Heavy debris at bottom of tape
	Tape rode up on take-up reels about 1/4 inch. A solid al roller was substituted for the grooved rubber roller on the tachometer. Head was cleaned and test started again.						
4,400	20	21	22	22	9	2	Heavy patches of debris - tape will not track - rides up to flange on crowned roller

Table XVIII

STATIC TESTS - HEAD MATERIAL

(a) 27°C

Tape Type	Brass	Aluminum	Permalloy	Stainless	Chrome	Teflon
Crolyn	6.8 oz	10.0 oz	4.6 oz	6.6 oz	9.6 oz	7.8 oz
Quantum	7.2	5.6	6.4	6.2	9.6	11.8
3M 777	9.6	2.0	3.4	5.2	7.6	10.8
MEM 63L	9.0	6.2	6.0	8.8	8.4	8.6
Mylar	5.4	~0	~0	4.4	2.0	5.4
MEM 79L	7.8	6.4	7.6	7.6	7.8	10.6
3M 871	11.2	2.8	3.8	7.8	5.0	5.6
3M 888	6.8	1.8	8.2	4.4	5.0	8.8
RCA 617	13.2	6.8	5.6	10.8	9.4	14.0
3M 351	7.4	3.2	3.0	7.8	5.4	8.4
			(b) 65°C			
Crolyn	4.4	7.8	6.8	13.2	13.6	8.2
Quantum	>64	10.2	10.8	2.2	5.8	8.6
3M 777	10.4	5.8	3.8	14.2	16.4	11.8
MEM 63L	14.4	7.8	7.6	16.4	8.6	6.8
Mylar	1.0	4.2	4.2	2.4	6.2	3.4
MEM 79L	>64	8.6	22.0	41	7.2	10.4
3M 871	12.0	17.0	8.6	15.4	5.2	2.8
3M 888	15.2	8.0	5.6	7.8	5.2	7.6
RCA 617	6.6	7.4	9.2	7.6	8.2	11.8
3M 351	9.6	4.2	3.0	3.8	6.2	7.0

B. Oxide/Binder Debris

A quantitative evaluation of oxide/binder debris was more difficult to obtain than in the case of adhesion where torque and drag were readily measured. Three types of measurements were used: weighing the accumulation of debris on the heads, measurement of short wavelength reproduce signal loss, and dropout evaluation of the tapes after use. The most predominant method applied was weighing the debris. Qualitative descriptions of the heads and microscope photographs were also used throughout the tests.

The procedures for weighing the accumulated debris involved weighing the used head and debris, carefully cleaning the front surface of the head, and reweighing the head. Aluminum dummy heads were used for most of the tests to minimize head weight. A Mettler Instrument Corp. precision balance was used. Readings were estimatible to 1 μg. The following weighing procedure was used:

1. Stabilize the balance
2. Cool the head (minimum of 3 hr temperature stabilization)
3. Obtain a minimum of 7 readings
4. Average 5 median readings
5. Remove and clean the head with methyl ethyl ketone, followed by isoproply alcohol
6. Reweigh the cleaned head

Using this procedure, accurate and repeatable measurements of debris accumulation were obtained.

1. Endless Loop Debris Tests

A series of tests was performed on the endless loop transports to determine the debris generation properties of the various tapes at several environments. Aluminum dummy heads were used. The tape loops, 52 inches in length, were carefully

spliced, cleaned and placed in the chamber for stabilization. Tape tension was 8 ounces, and the wrap angle was 15 degrees. After stabilization, the tapes were run for 24 hours at 30 ips (over 49,000 passes) in one direction only. The accumulation of debris was then measured and the heads were described. Sample results are shown in Table XIX(a) and (b)

2. Measurement of Signal Loss

A number of tests were also run on the endless loop transports using actual reproduce heads rather than dummy heads so that playback signals could be monitored. These tests were run in room ambient environments up to 10,000 passes. The reproduce output of a 150-microinch-wavelength signal was monitored every 200 passes for the first 1000 passes, and then every 1000 passes. Typical results are shown in Table XX(a) and (b). The relative output levels refer to the signal-to-noise ratios in decibels.

3. Tape Dropout Evaluation

Following a few of the oxide/binder tests conducted, the 52-inch loops were evaluated on special test equipment capable of monitoring dropouts. Figures 25 and 26 show flutter and dropout counts for two loops of 3M type 351 tape previously run for 10,000 passes. The results shown in Fig. 25 were from a tape run over a quarter-inch diameter roller inserted in the standard endless loop transport tape path. The results in Fig. 26 were obtained from a tape not contacting this roller. The bit packing density for this evaluation was 3300 bits per inch; the tape speed was 0.3 ips and the dropouts measured were 6 db or greater.

Table XIX

OXIDE BINDER DEBRIS

Endless Loop Tests

(a) 65°C, 45% RH

Tape	Head Condition	Debris
63L	Two deep scratches; little varnish (blush coloration) moderate-heavy debris evenly distributed (light brown)	8.474705 8.474611 94μg
79L	Heavy scratches, moderate brown varnish; moderate-heavy debris (white and brown)	8.432167 8.432074 93μg
Crolyn	Heavy scratching, little varnish, minor amount of debris (brownish)	8.465330 8.465272 58μg
Quantum	One heavy scratch, no varnish, moderate brown debris	8.462310 8.462235 85μg
351	Heavy scratching; heavy brown varnish; heavy brown debris	8.464382 8.464176 206μg
888	No scratches except for two very deep ones; one heavy varnish band, light-moderate debris but heavy at varnish band	8.406280 8.406215 65μg
	(b) 65°C, 5% RH	
777	Light "melting" of head and minor scratches; heavy white varnish in places; a small amount of white debris	8.455691 8.455639 52μg
871	"Melting" but little other scratching; spotty light-brown varnish with a moderate brown debris	8.460662 8.460605 57μg
6176	Heavy scratching; heavy brown varnish; debris so heavy much lost on rollers and flaked off	Not Weighed Est. 2000μg

Table XIX (Continued)

(c) 45°C, <5% RH

Tape	Head Condition	Debris
Quantum	Two deep scratches; light gray varnish near scratches; light brown debris	8.371015 8.370983 32μg
777	Very deep wide scratches, very little varnish, moderate-light brown debris	8.447264 8.447255 9μg
79L	Moderate scratching gray-brown varnish, moderate dark brown debris	8.444243 8.444199 44μg
351	Moderate (fine) scratching, heavy brown varnish, very heavy debris on all four sides of contact	8.433858 8.433407 451μg
63L	Moderate scratching and one deep scratch near edge, moderate gray varnish, light debris	8.439677 8.439662 15μg
888	Moderate scratching but heavy in spots, moderate brown varnish in bands, light gray debris	8.357965 8.357948 17μg
871	Heavy scratching, moderate varnish, moderate debris	8.442549 8.442502 47μg
6176	Heavy scratching but not deep, very heavy brown varnish, very heavy debris, some loss as before	8.425719 8.424261 1457μg

Table XX

OXIDE/BINDER DEBRIS

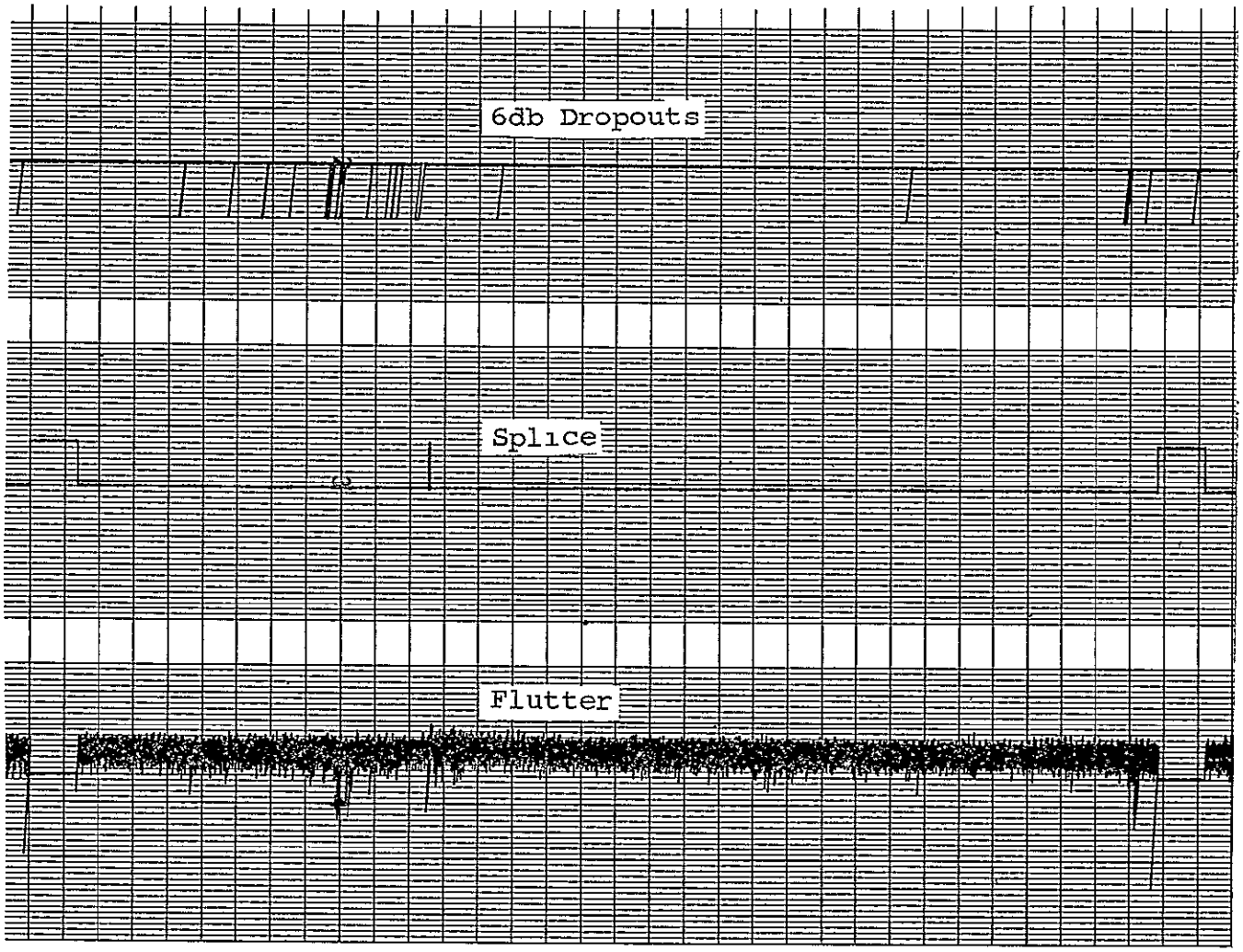
(a) 3M 777 8-oz Tension, 12° Wrap

No. of Passes	Relative 200 kHz-Output	
	Single Direction	Bidirectional
0	+29.5 db	+30.0 db
200	30.0	28.5
400	30.0	28.5
600	30.0	28.5
800	30.0	28.0
1,000	30.0	27.5
2,000	30.0	28.0
3,000	30.0	28.0
4,000	30.0	28.0
5,000	30.0	27.5
6,000	30.0	27.5
7,000	30.0	27.5
8,000	29.5	27.5
9,000	29.5	28.0
10,000	29.5	27.5

Table XX (Continued)

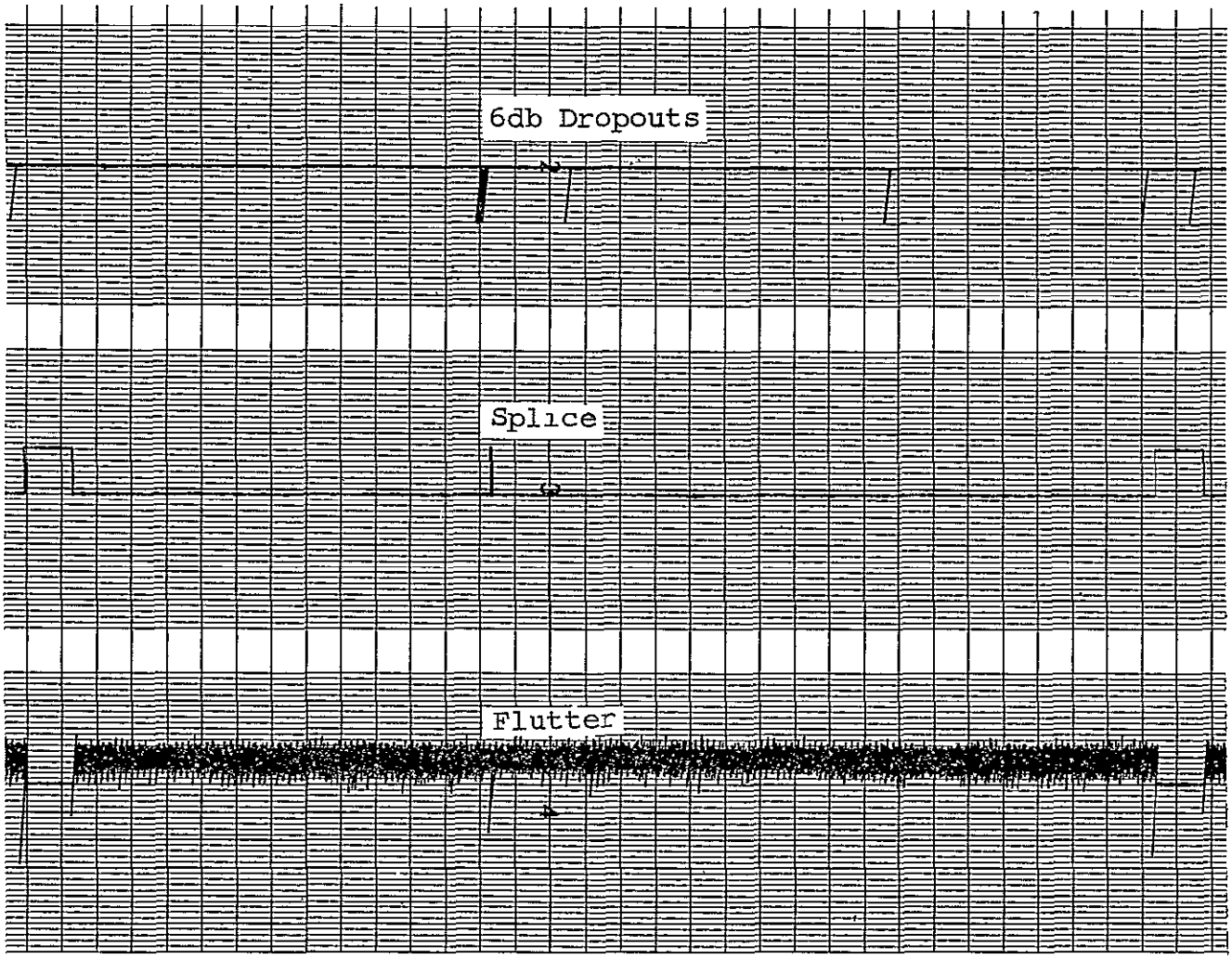
(b) 3M 888 8-oz Tension, 12° Wrap

No. O	Relative 200 kHz-Output	
No. of Passes	Single Direction	Bidirectional
0	+29.0 db	+32.5 db
200	29.0	31.5
400	29.0	30.0
600	29.0	28.5
800	29.0	28.0
1,000	29.0	27.5
2,000	29.0	27.0
3,000	29.0	27.5
4,000	29.0	27.0
5,000	29.0	27.5
6,000	29.0	27.5
7,000	29.0	27.0
8,000	29.0	27.0
9,000	29.0	27.0
10,000	29.0	26.5



Direction of Tape Motion

Fig. 25 DROPOUT EVALUATION AFTER 10,000 PASSES
3M 351 TAPE WITH SMALL ROLLER IN TAPE PATH



Direction of Tape Motion

Fig. 26 DROPOUT EVALUATION AFTER 10,000 PASSES
3M 351 TAPE WITHOUT SMALL ROLLER IN TAPE PATH

C. Logbooks

Test results and other data pertinent to the program have been recorded in the following IIT Research Institute logbooks:

C19382
C19391
C19397
C19645
C19646
C19647
C19648
C19649
C19692
C19809
C19814
C19893
C19979
C19984
C20210

D. Magnetic Tape Evaluated

Throughout the program, a number of tape types produced by several manufacturers were evaluated. These tapes, listed below, were purchased and tested between 1 July 1969, and 31 December 1970. Lot or batch identification numbers are included, as variations in certain parameters may occur between reels or batches of the same tape type designation.

Manufacturer	Type Designation	Lot Number
Ampex	772	29G7306-4 (1 Reel Unknown)
Bell and Howell	W4	10027834
DuPont	Crolyn	8322711036102 8322711036119 (1 Reel Unknown)
Graham Magnetics	Epoch IV	8B20558B-15
IBM	500	0 6 072271
Memorex	63L	15092-A2-6A 15092-A1-17A
Memorex	79L	E59159-C2-5D ME56857-A1-5B
Memorex	Quantum	27E-HJ-3712C-A1- M30160-B3 (2 Reels)
3M	351	64748043029 64748043046
3M	551	67618-71-11-41 67618-71-12-41
3M	700	72729-99-000-37
3M	777	63904-390-1011 63904-370-1036
3M	851	65849-6-10-7
3M	871	69333-3-10-16 69333-6-20-19 74921-10-10-12 74768-5-11-9
3M	888	55512-46-10-17 67760-40-11-12 (1 Reel Unknown)
3M	8119	72347-01-10-41

Manufacturer	Type Designation	Lot Number
3M	MTA 20294	70773-03-10-19
3M	MTA 20302	64883-1-2-2
3M	MTA 20478	75450-06-000-38
RCA	617	No Designation - Not A Commercial Tape

REFERENCES

1. Archard, J.F., J. Appl. Phys., 32, 1420, 1961.

2. Berlin, A.A., Bulacheva, S.F., and Morozov, Yu. L., Plasticheskie Massy No. 10, p. 3, 1962.

3. Schnurrman, R., J. Appl. Phys. 11, 624, 1940.

4. Schnurrman, R. and Warlow Davies, E., Proc. Phys. Soc. (London), 54, 18, 1942.

5. Hardy, W.B., Collected Works, Cambridge University Press, 1936.

6. Bowden, F.P. and Tabor, D., The Friction and Lubrication of Solids, Part II, Oxford at the Clarendon Press, 1964.

7. Shooter, K.V. and Tabor, D., Proc. Phys. Soc. B65, 661, 1952.

8. King, R.F. and Tabor, D., Proc. Phys. Soc. B66, 728, 1953.

9. Taylor, D. and Rutzler, J.E., Ind. Eng. Chem. 50, 928, 1958.

10. Deryaguin, B.V., Vestnik Akad. Nauk SSSR, 7, 10, 1954.

11. Frank, C., Kraus, G., and Haeffner, A., Ind. Eng. Chem. 44, 1600, 1952.

12. Houwink, R., Kolloid Z., 151, 143, 1957.

13. Skinner, S.M., Savage, R.L., and Rutzler, J.E., J. Appl. Phys. 24, 438, 1953.

14. Krotova, N.A. and Mrozova, L.P., "Research on Surface Forces, p.64, Consultants Bureau, Publishers, 1967.

15. Gutfreund, K., Brown, C., and Weber, H.S., Proc. Conf. SPI Reinf. Plastics Div., 1962, Section 10-A.

16. Scott, I.R., J. Paint Technol. 9, 218, 1944.

17. Aitken, A. and Barrer, R.M., Trans. Faraday Soc., 51, 116, 1955.

18. Voyutskii, S.S. and Shtarkh, B.V., Kolloidn, Zhur., 16, 3, 1955.

19. Forbes, W.G. and McLeod, L.A., IRI Trans. 34, 154, 1958.

20. Bayer, O., Angew. Chem. 59, 257, 1947.

21. Memorex Corp., Neth. Appl., 6,607,214, November 28, 1966.

22. Werst, G., et al., Ger. 1,241,490, June 1, 1967.

23. Jacobson, M.J. (to Ampex Corp.), Fr. 1,478,199, April 21, 1967.

24. Bishops, J.H., et al, Belg. 703,148, February 28, 1968.

25. Schell, G., et al, Ger. 1,260,132, February 1, 1968.

26. Rosenberg, M., U.S. 3,205,092, February 20, 1962.

27. Swann, M.H., Z. Anal. Chem., 164, 147, 1958.

28. Braun, D. and Mai, E., Kunststoffe, 58, 637, 1968.

29. Kubitz, K.A., Anal. Che., 29, 814, 1957.

30. Talley, J.P. (to Ampex Corp.), U.S. 3,144,352, August 11, 1964.

31. Cooper, W., Pearson, R.W., and Darks, S., Ind. Chemist, 36, 121, 1960.

32. Corish, P.J., Anal. Chem., 31, 1298, 1959.

33. Cannon, C.G., Spectrochim. Acta, 16, 302, 1960.

34. Muller, A., Proc. Roy. Soc., A 120, 437, 1928.

35. Baker, W.O. and Fuller, C.S., J. Amer Chem. Soc., 65, 1120, 1943.

36. Laird, J.A. and Nelson, F.W., Proc. 19th Conf. SPI Reinforced Plastics Division, 1964, Section 11-C.