A. G. Armour	-	F&F, Exp. Sta.
J. I. Dye	-	F&F, Exp. Sta.
J. C. Fang	-	F&F, Exp. Sta.
E. B. FitzGerald	-	F&F, Exp. Sta.
J. Hochberg	-	F&F, Exp. Sta.
B. G. Hunt	-	F&F, Exp. Sta
H. L. Jakubauskas	-	F&F, Exp. Sta.
D. M. Marsh	-	F&F, Exp. Sta.
P. H. Pettit	-	F&F, Exp. Sta.
W. Pregmon	-	F&F, Exp. Sta.
J. A. Simms	-	F&F, Exp. Sta.
J. J. Troy	-	F&F, Exp. Sta.
J. A. Vasta	-	F&F, Exp. Sta.
S. Wu,	-	F&F, Exp. Sta.
F. F. Huppe	-	Eng. Physics Lab. Exp. Sta.
F. A. Fluegge	-	Fairfield
C. W. Bullock	-	Troy Lab.
C. Ambrosio	-	Troy Lab.
T. P. Anderson	-	Troy Lab.
L. W. Crissey	-	Troy Lab.
F. M. Gavin	-	Troy Lab.
R. A. Hiss	-	Troy Lab.
R. O. Kenworthy	-	Troy Lab.
A. F. Nugent	-	Troy Lab.
T. A. Rettig	-	Troy Lab.
R. J. Sheppard	-	Troy Lab.
C. L. Steiner	-	Troy Lab.
R. D. Vest	-	Troy Lab.
J. L. Allen	-	Marshall Lab
V. Ananthakrishnan	-	Marshall Lab.
J. A. Antonelli	-	Marshall Lab.
W. S. Armstrong	-	Marshall Lab.
L. A. Becton	-	Marshall Lab.
G. C. Bell, Jr.	-	Marshall Lab.
R. D. Breazeale	-	Marshall Lab.
D. C. Carbaugh	-	Marshall Lab.
J. G. Carson	-	Marshall Lab.
M. M. Coburn	-	Marshall Lab.
B. V. Gregorovich	-	Marshall Lab.
P. Demchur	-	Marshall Lab.
J. A. Devlin	-	Marshall Lab.
A. V. Scancella	-	Marshall Lab.
C. E. DeBoer	-	Marshall Lab.
G. L. Evans	-	Marshall Lab.
J. W. Gkonos	-	Marshall Lab.
W. C. Golton	-	Marshall Lab.
D. M. Gowing	-	Marshall Lab
H. Halpin	-	Marshall Lab.
H. P. Hanisch	-	Marshall Lab.
J. H. Hartshorn	-	Marshall Lab.
G. W. Hiddemen	-	Marshall Lab.
S. Hochberg	-	Marshall Lab.
R. W. Laurrell	-	Marshall Lab.
Y. K. Lee	-	Marshall Lab.
G. E. Lewis	-	Marshall Lab.
R. G. Lindsey	-	Marshall Lab.
O. C. C. Lin	-	Marshall Lab.
G. W. Mansell	-	Marshall Lab.
R. L. Manley	-	Marshall Lab.
E. P. H. Meibohm	-	Marshall Lab.
M. P. Morse	-	Marshall Lab.
D. K. Ogg	-	Marshall Lab.
F. E. Schweitzer	-	Marshall Lab.
R. J. Shuba	-	Marshall Lab.
F. J. Shannahan	-	Marshall Lab.
C. B. Sheridan	-	Marshall Lab.
C. D. Smith	-	Marshall Lab.
C. M. Smyrk	-	Marshall Lab.
W. B. Van der Linde		Marshall Lab.
E. M. Vary	-	Marshall Lab
F. Welty	-	Marshall Lab.
J. P. Wineburg	-	Marshall Lab.
D. T. Wu	-	Marshall Lab.
E. L. Yuan	-	Marshall Lab.
W. S. Zimmt	-	Marshall Lab.
E. J. Zinser	-	Marshall Lab.
J. A. Haase	-	Chicago Plant
C. W. Johnson	-	Parlin Plant
R. L. McFarland	-	Toledo Plant
W. R. Williamson	-	Plastics, Chest. Run
R. A. Sandsted	-	S. S. Francisco

File: 1815

Marshall R&D Laboratory
November 25, 1975

FABRICS & FINISHES MARSHALL R&D LABORATORY
LABORATORY TECHNICAL SERVICES
MONTHLY REPORT
NOVEMBER, 1975

OSHA AND RELATED GOVERNMENTAL REGULATIONS

R. M. KIRKPATRICK

Occupational Cancer

To avert emergency situations such as those associated with vinyl chloride, methyl butyl ketone, and arsenic, the National Institute for Occupational Safety and Health (NIOSH) is beefing up its research programs on occupational cancer for the next three fiscal years. NIOSH hopes to help decrease the number of newly induced occupational cancers through a program of surveillance, industry-wide studies, and laboratory research.

The projected budget for the program is $6.8 million for 1976 and $11 million for 1977. NIOSH plans 15 industry-wide studies to be conducted when there is evidence of high rates of cancer among certain occupational groups or when animal studies indicate a particular substance used in the workplace causes cancer. NIOSH is also planning to develop a use-permit and registration system as part of its recommended criteria for standards on carcinogens.

Since October 1974 the National Cancer Institute has issued alerts on the following substances:

- Ethylene dibromide
- Chlordane
- Heptachlor
- Trichloroethylene

NIOSH has already taken action on some of these and is studying others:

- Chloroprene
- Asbestos
- Chromate pigments

RMK/meg
11/25/75

AIR TRANSPORTATION OF HAZARDOUS MATERIALS

R. M. KIRKPATRICK

On January 1, 1976 the U.S. Department of Transportation's new regulations on closed-cup flash point classification of materials becomes mandatory. F. & F. has been voluntarily shipping under these rules since July 1, 1975. However, we feel it is imperative to again emphasize that every F. & F. employee is responsible for complying with existing regulations. Heavy fines, and or imprisonment, can be levied on an individual employee for violation of the rules. At Marshall Laboratory we offer assistance by requiring all air shipments to be authorized by Bob Kirkpatrick or in his absence, Sal Mansi.

Hazardous materials shall not be carried as baggage. For your protection, special papers can be provided at the Philadelphia Works Office which will enable you to transport non-hazardous materials. In any event it is an F. & F. Departmental policy not to ship by air any corrosive, oxidizing, poisonous, or compressed gas materials. Flammable, combustible and unregulated materials may be shipped when properly classified and packaged. Under no circumstances are laboratory personnel to deliver shipments directly to the airport without proper classification, packaging and labeling papers.

RMK/meg

GAS CHROMATOGRAPHY/MASS SPECTROMETRY

R. J. SHUBA

Problems With Trace Level Organics:

Some recent analytical requests to identify low-level concentrations of organic materials required the use of gas chromatography/mass spectrometry equipment outside of our own laboratory. At best, only one or two of the components present in each sample could be identified with our present equipment.

Two different samples of Ft. Madison pond water were analyzed by GC/MS. The first sample was analyzed using a DuPont Model 491 GC/MS/Data System. (CR & D, Experimental Station). A second sample was analyzed using the Hewlett-Packard Model 5980 GC/MS/Data System, (Elastomers, Experimental Station). The organic materials for both samples were absorbed on activated charcoal and recovered in CS_2.

The following components were identified in the water sample (AR-75-10-3185) using the duPont 491.

diethyl ether	x-methyl dihydroindene
methyl ethyl ketone	cellosolve acetate
tetrachloroethylene	C_3 alkyl benzenes
toluene	C_4 alkyl benzenes
xylenes	naphthalene
1,1,2 trichloroethane	C_5, C_6, C_7 monobasic acids

Note: COS (carbonyl sulfide) and $CHCl_3$ (chloroform) were identified as impurities in CS_2.

The following components were identified in the water sample (AR 75-11-3360) using the Hewlett Packard 5980.

carbon tetrachloride*	toluene
chloroform*	xylene
methylene chloride	2 - methyl norbornene**
tetrachloroethylene	vinyl cyclohexene **

* probable impurities in CS_2

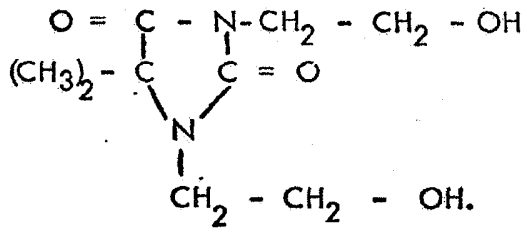
** tentative identifications (interference from CS_2)

RJS/bg
11/25/75

A NEW ENAMEL CROSSLINKER?

J. H. HARTSHORN

The computer infrared spectral search system along with GPC fractionation has frequently proven invaluable ~ for product analysis, but rarely as dramatically as shown by a recent high solids enamel study. Survey had found the enamel to be a blend of a styrene-acrylic copolymer and a cellulosic. This package is to be crosslinked with a hexamethylene diisocyanate adduct. The acrylic appeared to have been extended or reacted with some nitrogenous material which we suspected of being an isocyanate. Attempts to separate the resin with the usual solvents were complete failures. GPC however showed two distinct molecular weight peaks, which we believed would be the CAB and the modified acrylic. The higher molecular weight peak proved to be an unresolved mixture of the acrylic and the cellulosic; while the lower peak showed an unique double carbonyl compound, unlike anything in our files. A computer search of the full 150,000 spectra file provided a perfect spectral match for 1, 3-Bis (2-Hydroxy ethyl)-5,5-Dimethyl Hydantoin.



There is little doubt that this material is present, since other high solids samples from the same producer also showed the same compound. Without the aid of both GPC fractionation and computer IR file searching the identification or even detection of this compound would have been impossible.

JHH:mb
11/20/75

OSHA FILTERS

LEAD AND CHROMIUM DETERMINATIONS IN AIR PARTICULATE SAMPLES

J. A. DEVLIN, JR.

Methods have been established for the determination of lead and chromium in air particulate samples of lead chromate type pigment dusts, including Krolor®, but excepting basic lead silicochromate. A method has also been developed for these two elements in paints containing lead chromate type pigments, excepting Krolor® and basic lead silicochromate. Work is currently progressing on the Krolor®-pigmented paints and we are now recovering about 82-90% of both the lead and chromium contents. Because of the small incidence of use of basic lead silicochromate (1500 # of both W-666 and W-698 from January 1 to September 30, 1975 and probably less next year) we are not planning at present to expend any effort here on methods for either pigment dust or paint spray analysis.

Analysis of air particulate samples involving lead chromate type pigments presents a special problem. Ordinarily, different procedures in sample preparation are employed for each constituent sought, but the uniqueness of the particulate sample precludes such multi-faceted approach, and a method must be synthesized or invented which is specific for the sample type so that all the desired constituents may be determined from the one work-up. Other difficulties are sometimes introduced, too, by the OSHA-specified MCE filters, and methods of ashing, necessary when the sample is a paint spray, are much more critical in these filter analyses than in examination of a pigment, pigment fraction, or whole paint. All in all, it appears that individual methods, tailored to fit the specific sample type, will be necessary for each new type encountered.

JAD, Jr./mb
11/19/75

LIQUID CHROMATOGRAPHY

E. P. H. MEIBOHM

Work has now started to employ the DuPont 830 liquid chromgraph. The first runs made to become familiar with instrument operation included the separation of benzene, toluene, and xylene using a Permaphase ODS column and MeOH/H_2O eluents. In a second series of tests, a mixture of benzyl alcohol, methyl benzoate, benzyl acetate, methyl cinnamate, and dimethyl isophthalate was separated on a Permaphase ODS column using a linear gradient with H_2O as the A solvent and 25% MeOH/75% H_2O as the B solvent.

We are now trying to find a way to separate MDA from epoxies in Wellex. The molecular weight of the epoxies may be too high for a simple LC procedure and some sort of preliminary extraction may be needed to precede the LC work.

E. P. H. M.

EPHM:mb

GEL PERMEATION CHROMATOGRAPHY, LIQUID CHROMATOGRAPHY

E. P. H. MEIBOHM

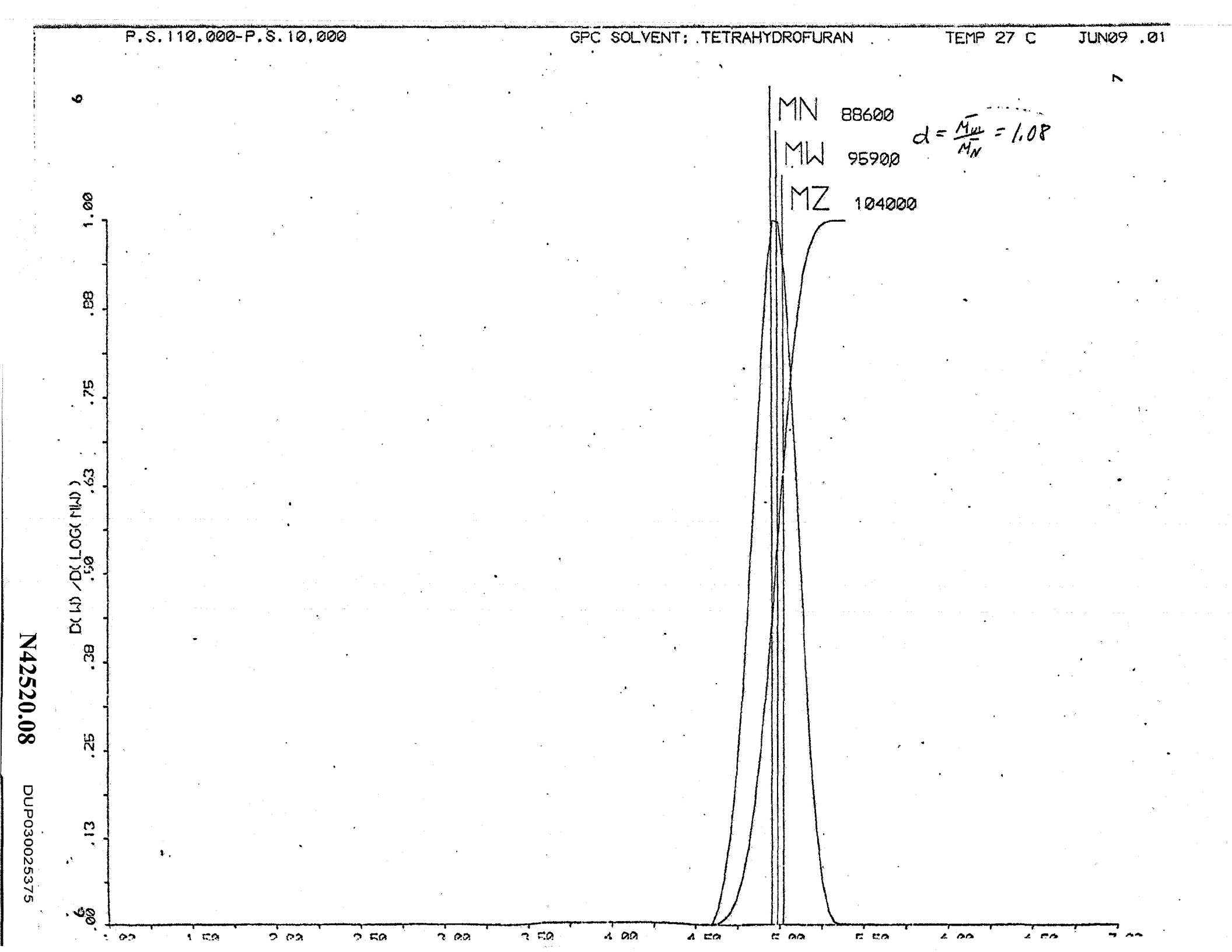
Gel Permeation Chromatography Values For Polydispersity:

The significance of the polydispersity factor, d, in GPC is not always understood. By definition $d = \bar{M}w/\bar{M}n$ and must always be 1 or greater. For most polymers d = 2 - 3. Several computer normalized GPC chromatograms are attached to illustrate typical distributions. In the polystyrene example, $d = \bar{M}w/\bar{M}n = 95900/88600 = 1.08$. According to the supplier d should be less than 1.06. Our slightly greater value may be due to the lack of a column broadening correction in the present computer program. Note that $\bar{M}w$ is slightly less than the peak molecular weight. $\bar{M}w$ is greater than the peak molecular weight for a symmetrical peak but not necessarily so for a skewed peak.

Newly Acquired High Pressure Liquid Chromatography:

In liquid chromatography the components of our system - a DuPont 830 liquid chromatograph, a DuPont 837 spectrophotometer, and an LDC two pen recorder have been set up. We are calibrating and otherwise getting acquainted with the operation of these instruments. An LDC refractive index detector will be worked in. Currently, we shall begin by setting up procedures for liquid chromatography and then add competence in high performance gel permeation chromatography (HPGPC).

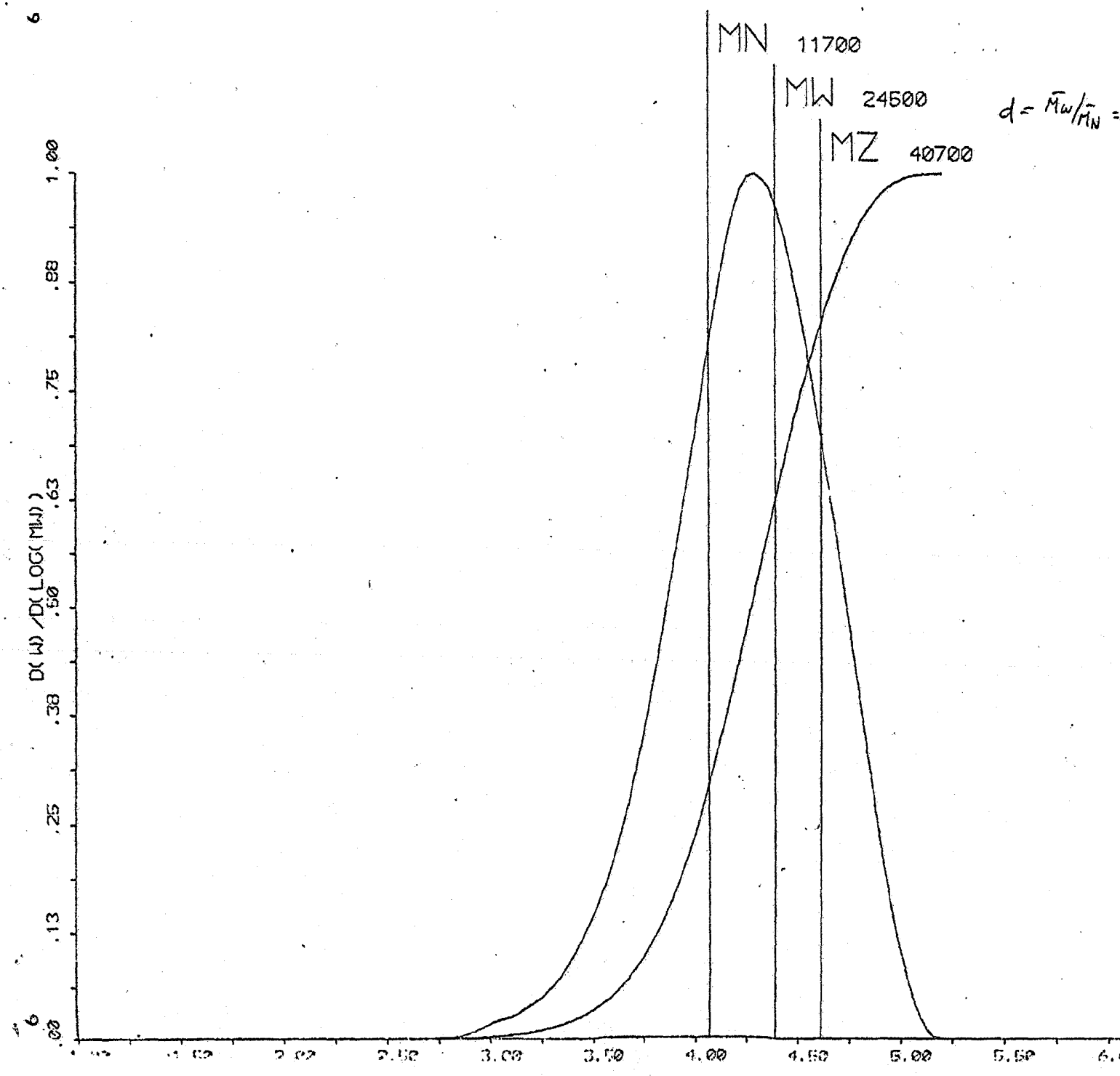
E. P. H. Meibohm

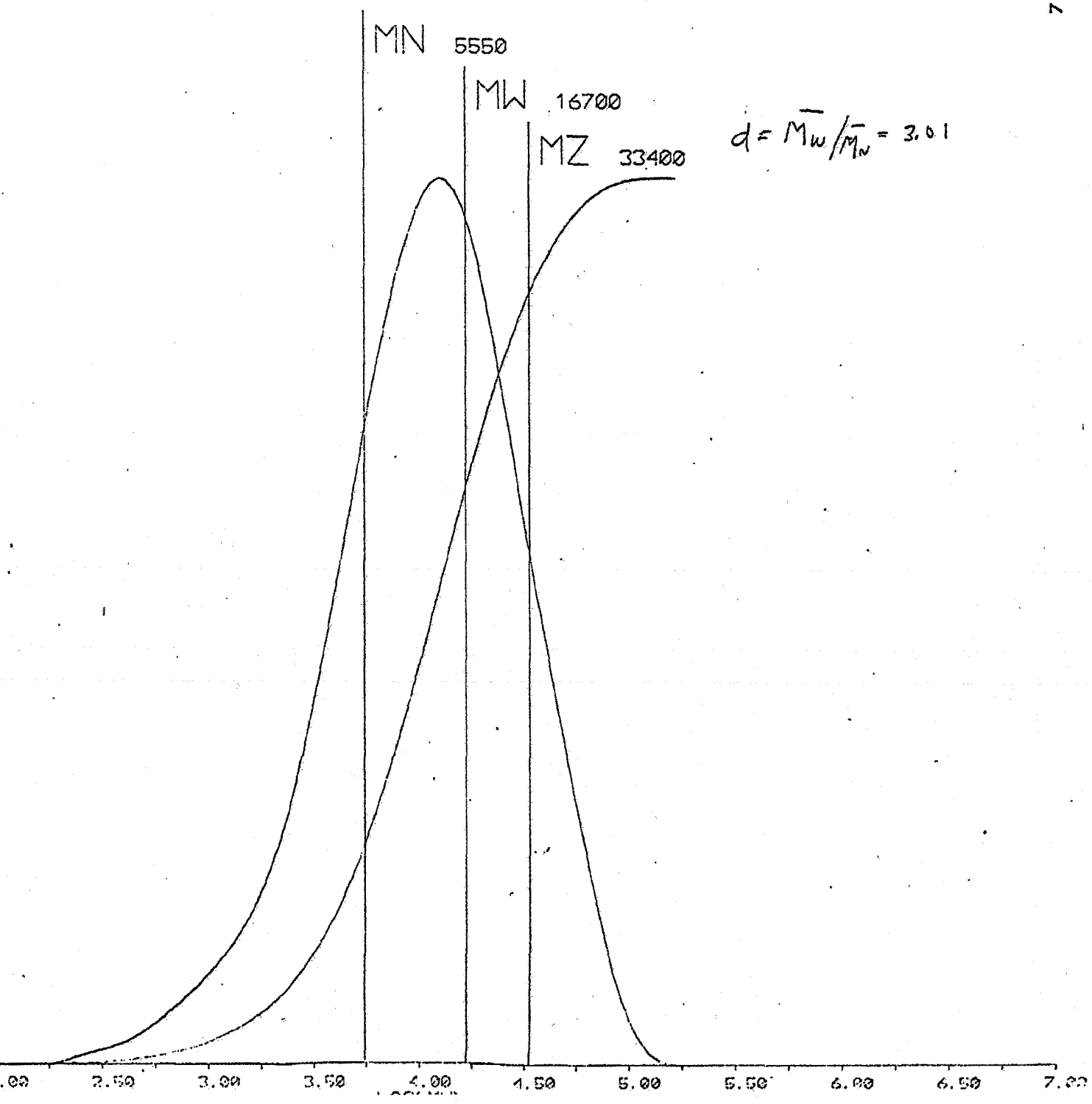


N42520.08

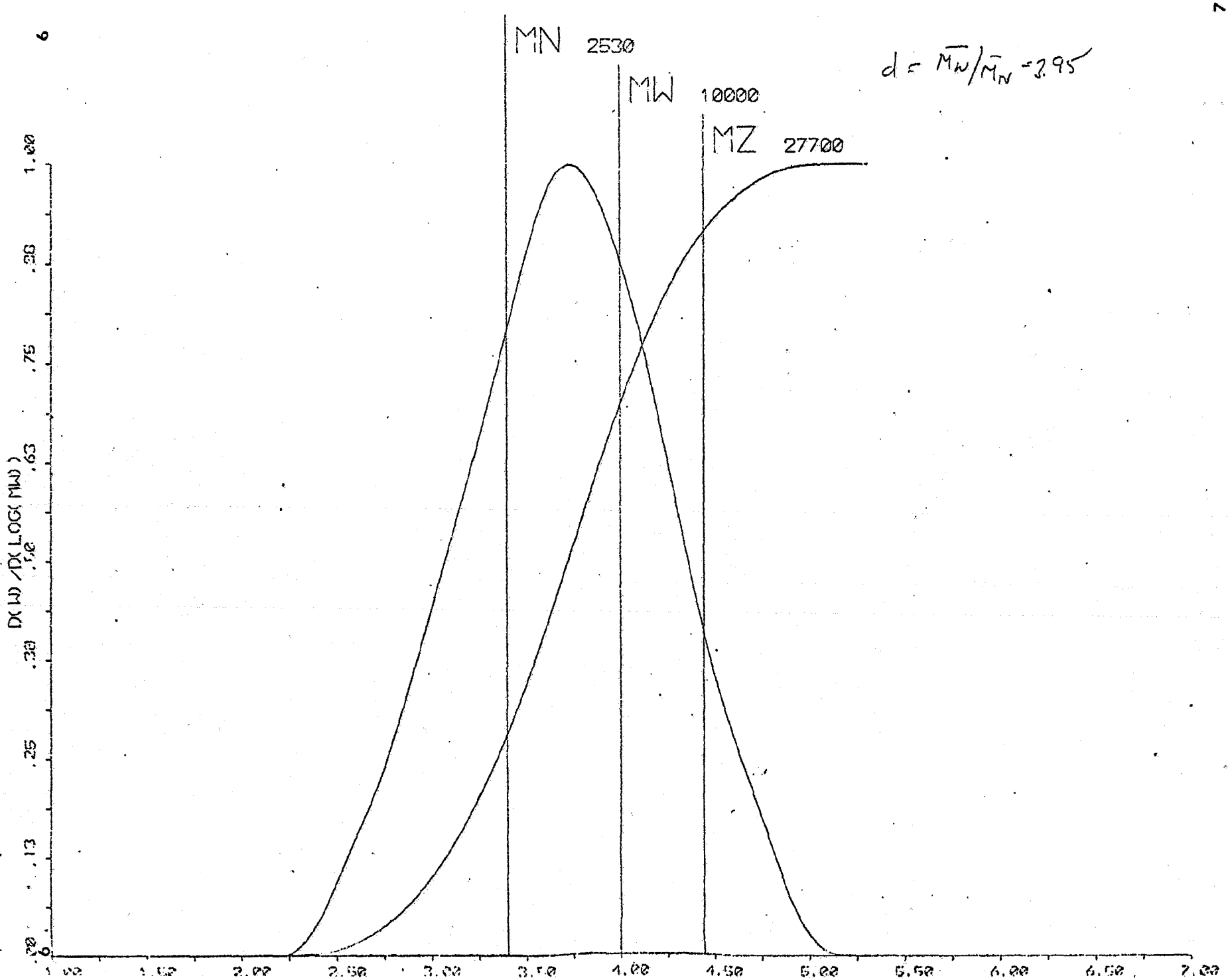
DUP030025375

TILLEY 75-2127 10546-83-A GPC SOLVENT: TETRAHYDROFURAN




MN 5550
MW 16700
MZ 33400
d = $\bar{M}_w / \bar{M}_N$ = 3.01
2.50
3.00
3.50
4.00
4.50
5.00
5.50
6.00
6.50
7.00

SCHMIDT 75-1284 210110-1 GPC SOLVENT: TETRAHYDROFURAN TEMP 27 C MAY12 .04



ANALYSIS OF PURPLE STAINS ON RED ENAMEL

J. P. WINEBURG

Investigations were recently conducted into the cause of purple stains appearing on equipment painted with red enamel at New Holland, Pa. Scanning electron microscopy (SEM), energy dispersive X-ray analysis (EDXA), and electron proble analysis (EPA) of stained and unstained surfaces showed that the stained areas contain comparatively larger amounts of sulfur. The valence state of the sulfur was found to be +6 by ESCA (electron spectroscopy for chemical analysis), indicating that the sulfur is present as sulfate. These results indicate that the stains are caused by atmospheric pollution interacting with dew to produce acidic droplets which attack the pigment in the enamel.

SEM, EDXA, and EPA were performed by W. E. Gresham of Micron, Inc., Wilmington, Delaware. ESCA work was done by R. S. Swingle, II of CR & D.

"KAPTON" ADHESION PROBLEM

ESCA (electron spectroscopy for chemical analysis) was recently used to investigate a problem involving "Kapton" polyimide film adhesively bound to specially treated copper plate. "Kapton" separation from the metal was occuring and appeared to be a result of either adhesive failure or "Kapton" film failure. However, ESCA analysis conclusively showed that "Kapton" separation was a result of cohesive failure of the Sn/Pb solder electroplated on the copper; neither the adhesive nor the "Kapton" was involved.

The copper plate had been electrocoated with 60/40 Sn/Pb solder, etched with ammonium persulfate, rinsed with water, and coated with a thin Sn immersion coating. The adhesive used to apply the "Kapton" was predominately acrylic (35% acrylonitrile/5% methacrylic acid/60% butyl acrylate).

Three samples were examined by ESCA:

(1) the metal surface after tinning but before the "Kapton" was applied, (2) the underside of a piece of "Kapton" peeled from the metal strip, and (3) the metal surface exposed by peeling off the "Kapton." The most important sample proved to be the underside of the peeled "Kapton." It showed the presence of substantial amounts of Sn, smaller amounts of Pb, but only trace amounts of nitrogen. Had either the adhesive or the "Kapton" failed, substantial amounts of nitrogen would have been detected.

ESCA analyses were performed by R. S. Swingle, II, of CR & D.

JPW:mb
11/19/75

MICROSCOPY

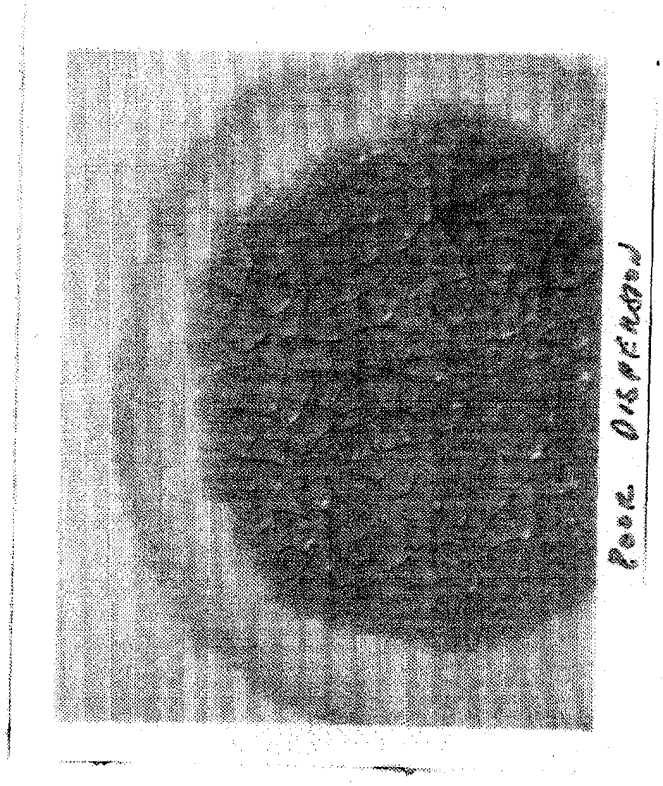
G. W. HIDDEMEN

DISPERSION QUALITY TEST:

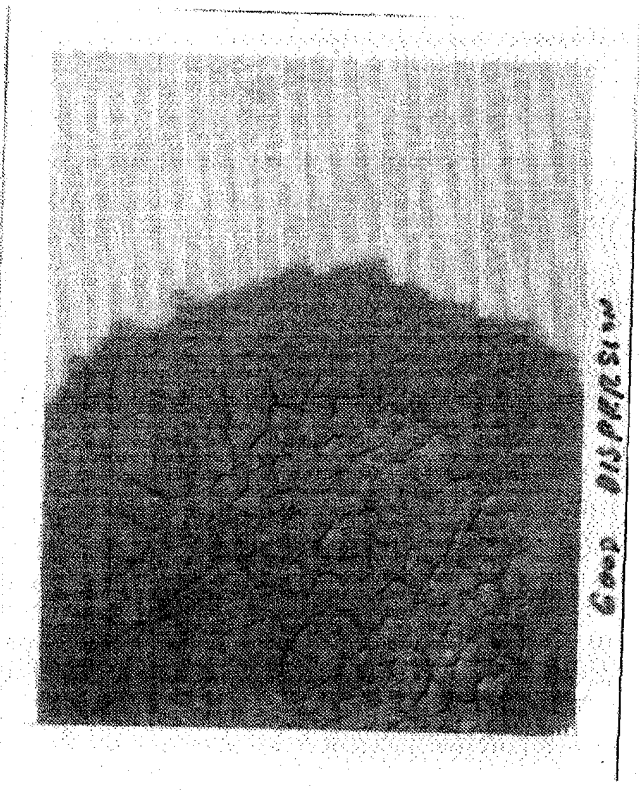
A series of color photomicrographs were taken for T. Nelson of the Manufacturing Support Group. These photographs will be used to illustrate the results of a new spot test that shows dispersion quality. The photographs below show a good and bad example of 547-5112 Dalamar Yellow Dispersion. In the bad dispersion you see a separation of the vehicle from pigment due to particle size.

The photographs were taken with the Bausch & Lamb dissecting microscope using a 10X power.

GWH:mb



Bad Dispersion



Good Dispersion