J. I. Dye	-	F&F, Exp. Sta.
J. C. Fang	-	F&F, Exp. Sta.
E. B. Fitz Gerald	-	F&F, Exp. Sta.
W. R. Hendrix	-	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.
M. J. Roedel	-	F&F, Exp. Sta.
J. A. Simms	-	F&F, Exp. Sta.
D. J. Troy	-	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.
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.
J. R. 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.
R. L. Selby	-	Marshall Lab.
F. J. Shannahan	-	Marshall Lab.
C. B. Sheridan	-	Marshall Lab.
C. D. Smith	-	Marshall Lab.
W. B. Van der Linde	-	Marshall Lab.
E. M. Vary	-	Marshall Lab.
J. A. Vasta	-	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
August 28, 1975

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

CED:eak

GAS CHROMATOGRAPHY

PETER DEMCHUR

POLYESTER (VM-228) CONTAMINATION WITH DOWTHERM A

Several samples of VM-228 were submitted by Parlin to determine if they had been contaminated with Dowtherm A. (75% Diphenyl ether/25% diphenyl) A gas chromatographic procedure was developed using the internal standard technique with a 6' 5% Dexsil col. at 155° C column temperature using the flame ionization detector. The samples were cut into small pieces and dissolved in methylene chloride overnight. All samples tested were free of diphenyl ether except for B# 460136 which was found to contain 102 ppm of diphenyl ether. The lower limits of detectability are about 50 ppm.

PD:mb
8/27/75

LEAD AND CHROMIUM ANALYSES IN 825-LINE PRODUCTS

J. A. DEVLIN, JR.

Significantly high chromium values obtained by an outside laboratory on 825-Line products manufactured at Chicago for use as hopper car linings were confirmed by even higher results at Marshall Laboratory. Contrary to suspicions, we have established that the chromium source was not lead chromate but a natural contamination in the W-135 Low Micron Talc. The use of W-109 as a possible substitute in these products is suggested as it was found to contain only 25 ppm. Cr vs. 750 ppm for the W-135.

Since the reported lead values of ca. 10 ppm were confirmed here also, the low lead/high chromium ratio was erroneously considered to be part of the lead/chromium analysis problem referred to in a previous (July) report. However, our worst lead results with C. P. lead chromate showed 1/3 the true value, whereas those in the 825-Line samples gave only 1/100 the chromium equivalent.

X-ray fluorescence analysis performed by Plastics Dept. gave no lead signal above the noise level and they would have seen amounts of lead equivalent to the chromium. The reality of our values was confirmed when Plastics obtained, by wet ashing and AA, 144 ppm Cr (vs. our 194 ppm) and 5 ppm Cr (vs. our 9 ppm). In the light of these data we concluded that the source of chromium was not lead chromate contamination.

Subsequent analysis of samples of 825-Line raw materials for Pb and Cr showed very high Cr values (750 ppm) for W-135. This was confirmed by similarly high Cr found in a sample of W-135 from the Marshall Lab. weigh & load room. A sample of W-109 was found to contain only 25 ppm Cr.

Chromium values of 825-8600 and 825-8601 ran 125-200 ppm, whereas in 825-8500 they ran 330-400 ppm. Significantly, the W-135 content of the latter is twice that of the 8600 and 8601.

JAD, Jr./meg

DETERMINATION OF LOW LEVEL ISOCYANATES

G. L. EVANS/J. H. HARTSHORN

Analytical determination of hexamethylenediisocyanate (HDI) present at levels of 0-10 ppb by weight in aqueous/organic extracts is a difficult problem currently posed by work underway on urethane food-contact products. An existing colorimetric procedure in use for several years for determination of HDI in air samples (OSHA), is not sensitive enough for the extract analyses, and is also extremely time-consuming (hence expensive) in application to OSHA work.

A potential solution to the extracts analysis problem, as well as a much faster (less expensive) and more sensitive method for OSHA analyses, may be provided by a fluorometric procedure involving a reagent known as "fluorescamine". Under appropriate conditions the latter reacts rapidly with primary amines (hexamethylenediamine in the present case) to form a highly fluorescent species, the emitted energy intensity of which is proportional to amine concentration.

Preliminary evaluation of this approach was recently carried out (by J. H. Hartshorn) at Central Research, and results were sufficiently promising to justify undertaking a short-term methods development program on fluorometric determination of HDI in a variety of substrates for application to both aqueous/organic extracts and OSHA samples at Marshall Laboratory. The work will be carried out by Central Research, where required facilities are available.

GLE:JHH/mg

OSHA AND RELATED GOVERNMENTAL REGULATIONS AND ACTIVITIES

R. M. KIRKPATRICK

Comments on Proposed Ketone Standards:

Twenty-five companies and/or Trade Associations have been granted time to voice their approval or disapproval (in Washington on September 3) of the six ketone standards which will set the pattern for an additional 394 standards on various chemicals to be promulgated by OSHA in the next two years. Of the total of 18 hours of testimony expected OSHA has been granted 4 hours, Du Pont 1 hour and the National Paint & Coatings Association (NPCA), representing 1000 member companies, 1/2 hour.

The NPCA will contend, in their testimony, that the ketones in question (2-BUTANONE, 2-PENTANONE, CYCLOHEXANONE, HEXONE, METHYL-N-AMYL KETONE, and ETHYL BUTYL KETONE) present a low health hazard in the industrial environment although many of the standard's requirements are as stringent as those for more hazardous materials such as carcinogens. While NPCA supports the concept of "action levels" for monitoring it believes that, in light of the low hazard presented by these ketones, the action level of one-half the TLV (Threshold Limit Value) is arbitrary and unnecessary. Du Pont's comments will be handled on a corporate level and will undoubtedly support the NPCA comments in addition to offering background information on the history of the ketones in occupational areas.

NIOSH Trade Name Ingredient Survey

Phase II of the National Occupational Hazard Survey (NOHS) is now in progress. Under this program the Du Pont Company has received 1100 registered requests to supply compositional information on trade name products for the National Institute of Occupational Safety and Health. Of the 1100, F. & F. products account for 740. Through obsoletion and products already reported in Phase I we have cut our list to about 450 requests to be processed here. The survey requires us to identify every ingredient over 1% by weight.

Consumer Product Safety Commission Survey

We have recently completed 150 forms requesting product ingredient information to the one-tenth of one percent (0.1%) level for the CPSC. The CPSC claims that information to this level is needed to develop regulations and standards for consumer products. It is also needed, they claim, to assess whether consumer products may be toxic, corrosive, strong sensitizers, flammable, combustible, may generate pressure through decomposition, heat or other means, and/or may present a hazard because of synergistic effects of their components or otherwise present a hazard to the consumer.

RMK/meg
8/26/75

ANALYSIS OF COMPETITIVE PRODUCTS

JOHN W. LOCKHART

Of the competitive products analyzed during August, 1975, the following have been selected as being of possible general interest. Except where otherwise indicated, results are necessarily somewhat tentative, being based on an arbitrarily limited high spot survey analysis aimed at establishing the general nature of the sample and the classes of major ingredients present. Since detailed analysis of complex commercial products is usually difficult and/or time-consuming, it is undertaken only in special cases where the time and expense can be justified.

	Company	Product	End Use	Components Detected
1.	ICI	ICI P-101	Cookware Primer	Resin: Tetrafluoroethylene Pigment: Calcium sulfate dihydrate
2.	Reichhold Chemical	93-890-EPOTUF-PX-1254	Intermediate	Resin: Styrene modified epoxy ester
3.	Roman Adhesive Co.	Roman 91-61	Laminating Adhesive	Resin: Ethylene-vinyl Acetate copolymer Pigment: Calcium Carbonate
4.	Armco Steel	Zincrometal	Zinc-rich metal coating	Resin: Bisphenol A type epoxy Pigment: Metalic Zinc

JWL/meg
8/22/75

GENERAL SPECTROSCOPY

JOHN P. WINEBURG

Quantitative Infrared Analysis of % UF in Epoxy/UF Resins

A quantitative infrared method has been developed for the determination of % UF in epoxy/UF resins.

An IR spectrum of an epoxy/UF resin contains independent absorption bands for epoxy (827 cm^{-1}) and UF (1645 cm^{-1}). A calibration curve was constructed using samples containing known weight percentages of epoxy (RC 5952) and UF (RC 718) resins. The ratio Absorbance epoxy (827 cm^{-1})/Absorbance UF (1645 cm^{-1}) was plotted vs. weight % UF. J. M. Pecillo statistically analyzed our data and found the method to be reproducible to ± 1.09% and accurate to ± 1.35-1.56% within the range 2.5-25% UF. Although this method was developed for can coatings, it appears to be generally applicable.

Quantitative Infrared Analysis of Epon 828 in Epon 828/Polysiloxane Mixtures

A quantitative infrared technique has been developed which will determine weight % Epon 828 in the Epon 828 (G-1109)/Polysiloxane (KG 7523) mixtures used in our 955-802 Teflon® release coating.

An IR spectrum of an Epon 828/Polysiloxane mixture contains independent absorption for Epon 828 (1510 cm^{-1}) and Polysiloxane (1435 cm^{-1}). A calibration curve was constructed using samples containing known weight percentages of Epon 828 and Polysiloxane; the ratio Absorbance Epon (1510 cm^{-1})/Absorbance Polysiloxane (1435 cm^{-1}) was plotted vs. weight % Epon 828. Unknowns can be analyzed using the curve with an accuracy of ± 1%.

THERMAL ANALYSIS

Quantitative Thermogravimetric Analysis (TGA) of % Ethylene in Ethylene/Vinyl Acetate Copolymers

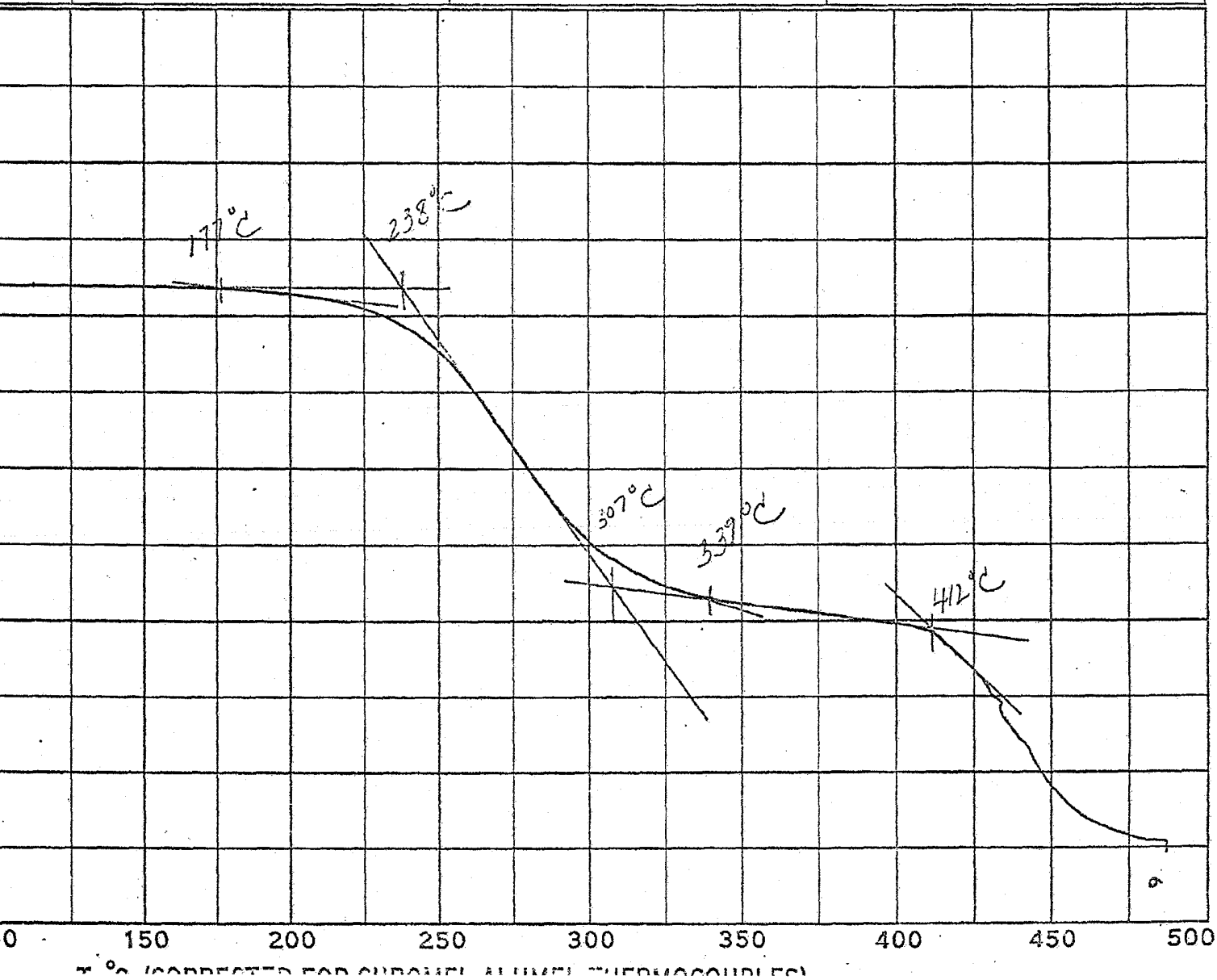
TGA has been used to measure weight percent ethylene in ethylene/vinyl acetate (E/VA) copolymers. E/VA copolymers thermally decompose in 2 steps between 0 and 500° C (see thermogram). The first step corresponds to loss of acetic acid. This weight loss is related to the weight % vinyl acetate present in the polymer; % ethylene is determined by difference.

The method suffers from the need to assume that only ethylene and vinyl acetate are present in the copolymer. The presence of an additional monomer or modifier will affect the accuracy of the results.

JPW/meg
Attachment

DUPONT INSTRUMENTS

X-AXIS	Y-AXIS	
TEMP. SCALE 50 °C / inch	SCALE 1 mg. / inch	RUN NO. 1 DATE
SHIFT 0 inch	(SCALE SETTING X2)	OPERATOR Simpkins
	SUPPRESSION 480 mg.	HEATING RATE 10 °C / min.
		ATM. AIR
		TIME CONSTANT 1 sec.



T °C (CORRECTED FOR CHROMEL-ALUMEL THERMOCOUPLES)