J. C. Fong	-	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.
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. San Francisco

File: 1815

Marshall R&D Laboratory
July 30, 1975

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

CED:eak

OSHA AND RELATED GOVERNMENTAL

REGULATIONS AND ACTIVITIES

JULY 1975

R. M. KIRKPATRICK

UPDATE ON NOISE STANDARD

Heated debates over OSHA's proposed noise standard have been in progress between OSHA and the Environmental Protection Agency (EPA) since January of this year. The controversy focuses on what the allowable noise level should be. OSHA has proposed an upper limit of 90 decibels; the EPA wants a level of 85 decibels. Labor is endorsing the EPA's 85 level while many businesses, already burdened with the high cost of meeting OSHA standards, favor the 90 decibel level. The EPA believes that the hearing loss risk percentages for 8 hours daily, 5 days per week, with exposures for 40 working years would result in a 21 to 29% hearing loss at the OSHA 90 decibel limit, while only a 10 to 15% hearing loss at the EPA 85 decibel level. This translates to 10 to 15 more persons per hundred could incur hearing handicaps at a lifetime working exposure at 90 versus 85 decibels.

On the matter of protective equipment to control noise level there is also a difference of opinion between labor and employers. The former want engineering controls while many of the latter group believe this is unrealistically expensive and would rely on ear plugs and ear muffs to deaden the noise level. Our policy at the Philadelphia F&F site is that ear muffs are used only as a temporary device to protect the worker while the noise level is being investigated. Either engineering controls, or redesign or repair of the equipment to reduce the noise level is the policy here.

Hearings on the noise level problem are expected to continue through August.

UPDATE ON SIX KETONE OSHA STANDARDS

OSHA has extended the comment period on the proposed standards reported in June. Hearings on the proposals have been rescheduled to begin on September 3, 1975. F&F currently uses three of these, i.e. methyl ethyl ketone, cyclohexanone, and methyl iso-butyl ketone in large quantities.

AIR SHIPMENTS

As of July 1st, 1975 F&F has adopted the closed-cup flash point system for classifying products as flammable or combustible for air shipments. In addition to local operating shipping papers - shipments for overseas require a certified letter and shipping papers from F&F's International Section (A. C. Coleman).

CARCINOGENS

The National Institute for Occupational Safety and Health has published a list of 2,000 suspected cancer-producing agents. This list can be found in the Federal Register of Monday June 23, 1975, Volume 40-Number 121, Part II. The list is published as a request for comment by the public to determine health research needs. It is interesting to note that one of the 2,000 suspects is ethyl alcohol.

RMK.

7/25/75
R.M.K.:hfm

Marshall R&D Laboratory

TABLE OF CONTENTS SERVICE

The Information Systems Department is now offering a journal current awareness program entitled Table of Contents - TA/T. TA/T presently includes 75 journals, covering chemistry, physics, engineering, and chemical engineering. TA/T subscribers receive a table of contents for each journal selected within 1-2 days of journal receipt from the publisher and can then request abstracts or full text copies of any articles of interest. These articles are usually sent within 1-3 days after TA/T receives the request. The cost of this service is $40.00/year for each title selected and approximately $2.00/copy for each full text or abstract requested.

The Library is considering this service as a possible replacement for some of the journals currently being received. We currently subscribe to approximately 180 titles and receive multiple copies of 28 of these titles. The TA/T subscription rate is less than the subscription rate of many of these journals. Replacement of full subscriptions by the TA/T service would be limited to those journals which are of interest and importance but which are less frequently used by lab personnel. Comments and questions on this service should be directed to N. L. Van Saun.

N. Van Saun
NV:hfm
7/24/75

ANALYSIS FOR LEAD AND CHROMIUM IN PAINTS AND PIGMENTS

J. A. DEVLIN, JR.

Possible anomalies in lead and chromium determinations in paints and pigments prompted us to evaluate our methods of analysis for these two in the presence of each other. The ashing procedure for both lead and chromium involves heating in air to 500° C. For lead the ash is then dissolved in nitric acid with heat, while for chromium it is treated in a Teflon®-lined bomb with sulfuric acid/permanganate. Both elements are then determined by atomic absorption. These procedures are similar to ASTM proposed methods - we collaborated in the chromium round-robin.

Our procedures were evaluated using C. P. lead chromate. Two general conclusions were:

1) Ashing is not necessary if pigment only is present.

2) From previous experience it is known that ashing is required if a vehicle is involved, i.e., a paint or coating.

We found further that if a sample of $PbCrO_4$ is not ashed

1) we get quantitative recovery of both lead and chromium if the sample is dissolved in nitric acid (bombing with sulfuric acid/permanganate is unnecessary).

2) Neither AN/PVCl copolymer nor cellulosics interfere in the quantitative recovery of both lead and chromium, at least as long as they are not present as binders.

3) The valence of the chromium in the final AA measurement is immaterial but the anion of the acid does affect both the lead and chromium results. If hydrochloric acid is used instead of nitric the yields of both metals will be low.

On the other hand, if the sample is dry-ashed (max. 500° C)

1) Both lead and chromium are quantitatively recovered from C. P. lead chromate alone even when nitric acid is used to dissolve the sample.

2) When organic matter is present, even if not as a binder, low yields are obtained for both lead and chromium if nitric acid is used as solvent for the ash, even in the Teflon® lined bomb.

3) Quantitative chromium yields are obtained if the ash is dissolved by treatment with sulfuric acid/permanganate in the bomb. This treatment precludes lead determination since insoluble lead sulfate is formed.

In summary, we can obtain quantitative recovery of chromium in the presence of lead regardless of the presence or absence of organic matter. But the latter causes low yields of lead in the presence of chromium if a sample requires ashing, as paints and coatings do.

JAD/meg
7/29/75 Jad Jr.

ANALYSIS OF COMPETITIVE PRODUCTS

JOHN W. LOCKHART

Of the competitive products analyzed during July, 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) Flecto Co.	Ebony Wiping Stain	Consumer Paint	Resin: Long oil o-phthalic alkyd Extender Pigment System: Calcium carbonate and China Clay
2) Pittsburgh Plate Glass	High Solids Enamel	Appliance Finish	Resins: Short Oil o-phthalic alkyd and hexamethoxymelamine
3) Inmont	E9IBDIII Topcoat	Automotive Finish	Resins: Styrenated acrylic and hexamethoxymelamine

JWL/meg

GEL PERMEATION CHROMATOGRAPHY

E. P. H. MEIBOHM

In the past period gel permeation chromatography has continued its use as a monitor of resin development and production technology. Systems studied have included several alkyd series, acrylic resins, and high solids oligomers. In addition we have been characterizing resins representing potential candidates for licensing and we are supplying data to the Pigments Department for studying the interaction of pigments and resins.

We have decided to purchase the Du Pont LC 830 liquid chromatograph with gradient elution control and flow control and a spectrophotometer to be used along with a differential refractometer as detectors for High Performance Liquid Chromatography (includes GPC and LC). Most of this equipment will be available in from 6-10 weeks. A period of development will be needed to become familiar with the new instrumentation before routine use can be established The Jackson Laboratory is continuing its work to develop equipment for long distance on-line transmission of data, a technique we will need for on-line real time computations, but their schedule is being lengthened by manpower problems.

EPHM/meg

INFRARED INTERPRETATION

JACK H. HARTSHORN

Several plant and laboratory locations have infrared equipment, but they have not been able to take full advantage of this valuable technique because no one there was qualified to interpret the spectra. The spectroscopists here at Marshall Lab can assist other division laboratories by interpreting any curves sent over the "Magnafax". Transmitted curves are of adequate quality and arrive much faster than a sample sent by mail to the Analytical Group. As a case in point, recently a batch gelled at Toledo plant. Troy Laboratory ran curves of the gel, the polymer, and a control polymer, and sent them to us by wire. It was obvious to those who are familiar with IR spectra that the gel contained predominantly acid monomer. Informed of this, Toledo found the acid monomer had begun to polymerize in the drums. The turn-around-time was one day, versus a week by mail and normal routine. Infrared curves run on site by the people most interested are, of course, the earliest which can be produced, and they are usually the easiest to interpret, since the information to answer a specific question is either there or it isn't. If the required information is not in the curve, we can advise the next step to provide it.

For the quickest possible IR service, make your own spectra and send them by "Magnafax" to Jack Lockhart, Jack Wineburg, or Jack Hartshorn for interpretation. In addition, you may get some added benefits - in the case above, we were able to spot an unsuspected instrumental problem.

URETHANE-FOOD CONTACT PRODUCTS

Toluene diamine - the hydrolysis product of toluene diisocyanate - has been declared an Experimental Carcinogen by Haskell Laboratory, i.e. it has been shown to produce cancer in test animals. According to the "Delaney Amendment", there can be none of this chemical in any food product. Thus, the use of urethanes and TDI containing formulas for food contact end-uses should be limited. Where such products are already in use, they should be tested under use-conditions for the presence of toluene diamine. We have a procedure which will detect as little as 10 parts per billion in aqueous extracts. If it cannot be found at this sensitivity, we believe the formula will be acceptable to Haskell and to the FDA. However, when that food contact product is to be reformulated, attempts should be made to repace the TDI with another material.

JHH/meg
7/24/75

SUPPORT OF HIGH SOLIDS FINISHES TASK FORCE

G. L. EVANS

In order to ensure compliance with regulatory requirements, a valid test procedure is being sought which will demonstrate 80% volume solids content (prior to cure) of our current enamel formulas. Present emphasis is being placed on a broadly-applicable procedure which involves volume solids determination on enamel residue remaining after several hours vacuum treatment at 45° C to remove solvents without curing the enamel. Using this approach, our first enamel submittal (705-H-41175) was found to contain 79.8% volume solids, versus a theoretical 80.2%. Agreement between theoretical (88.7%) and observed (87.2%) weightsolids was also reasonably good.

Efforts are also under way to define "active ingredient" weight solids of both trimethylpentanediol/mixed methyl esters and trimethylpentanediol/isophthalic acid oligomers, used as high solids enamel ingredients. Since such oligomers contain 10-20% free and relatively volatile trimethylpentanediol (one of the active ingredients), weight solids cannot be directly obtained by even mild heating under vacuum. A gas chromatographic solution to this problem is currently being evaluated.

7/28/75 GLE

ELECTRON MICROSCOPY

VITOLDS E. KAZMERS

In the second half of February, 1975, the transfer of the freeze-fracturing instrument from Marshall Laboratory and its installation at the Experimental Station was completed.

Together with existing equipment, the new freeze-fracturing instrument provides us with the unique capability of being able to examine samples electron-microscopically from the liquid state to rubbery or low Tg materials to solid samples.

Since almost all of the polymeric systems of interest to the F & F Department pass through the liquid-state stage, information relating to the microstructure in this state can be of vital importance. Due to the lack of literature covering freeze-fracturing studies of polymers and the complex nature of many polymer systems, interpretation of the micrographs is sometimes difficult. It may require the design of additional experiments and a careful freeze-fracturing study of the components involved.

During the past four months, the freeze-fracturing instrument was used to investigate the following polymer systems:

1) High solids automotive paint for:
   a) Pigment dispersion quality
   b) Aluminum flake dispersion quality
   c) The mixing of components
   d) Phase structure of the system
2) Organosols

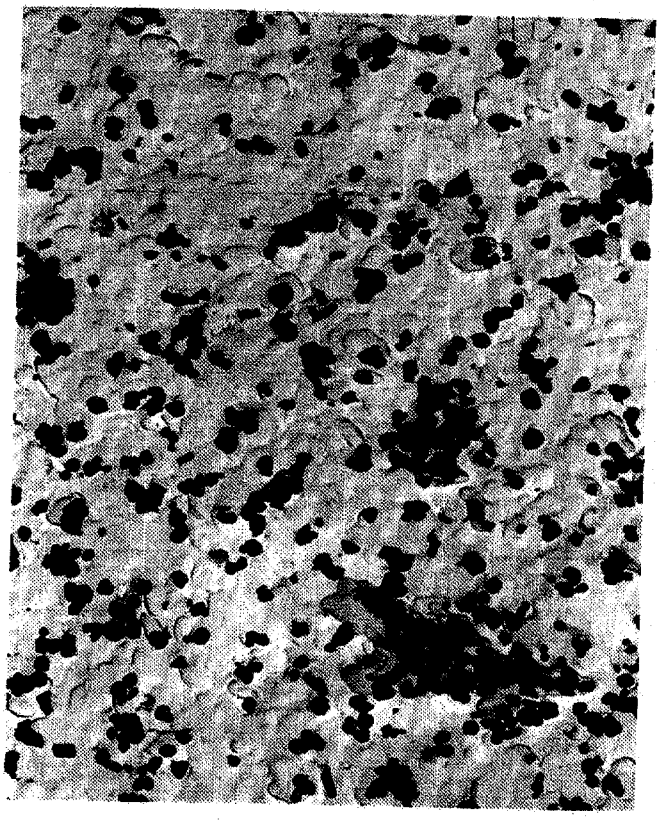
   The structures of various organosol-type lacquers and enamels were examined.
3) Flexible automotive primers for:
   a) Pigment dispersion quality
   b) Phase structures of the systems
4) Hand cleaner, "Good 'N Clean"

   "Good 'N Clean" was examined for batch-to-batch differences in liquid structure correlatable with variations in freeze/thaw stability.

Most of the investigations mentioned above are in the initial stages only and, in order to understand the systems more thoroughly, additional experiments were designed and studies are continuing. (REFJr)

VEK:ayk
6/16/75

FREEZE FRACTURING
(VITOLDS E. KAZMERS)



PIGMENTED HIGH SOLIDS PAINT SYSTEM
MAG. 19,000X

PIGMENTED FLEXIBLE AUTOMOTIVE PRIMER
MAG. 24,000X