INTER-OFFICE MEMO TENNECO CHEMICALS, INC. MAR 2 1973

To Distribution List* At Date March 1, 1973

From Dr. R. T. Gottesman At Piscataway Copy to

Subject MONTHLY SUMMARY - FEBRUARY 1973

Attached are copies of the Monthly Summary reports for February 1973 covering the activities of the Piscataway laboratories and associated pilot plants. The following reports are involved:

Chemical Research Section	Dr. A. J. Deinet
Chemical Development Section	Mr. I. Huppert
Polymerization Section	Mr. R. S. Miller
Applications Development Section	Dr. F. Buono
Analytical Services Section	Mr. H. R. Johnson

R. T. Gottesman

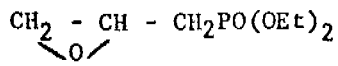
RTG/ec

Attachments

Distribution List*

Mr. M. Freifeld
Dr. D. Keyworth
Dr. P. A. Lobo
Dr. T. E. Maggio
Dr. M. Rosen w/o att.
Dr. L. A. Wigdor

Attempts to prepare an epoxyalkylene phosphonate



from epichlorohydrin via various schemes of halide replacement have been unsuccessful due to competing reactions and will not be pursued further.

The use of metallic salts as smoke suppressants in polyurethane foams has been investigated further. The zinc, tin, zirconium, lanthanum, cobalt and iron salts of dodecenyl succinic acid were prepared as well as the tin and iron salts of bis(2,3-dibromopropyl)phosphoric acid. All yielded high smoke density ratings and are thus considered ineffective.

III. COATINGS INDUSTRY

1. Can Preservative (H. Sidi, E. DiBella, J. Stanaback, O. Fuchs, G. Kagan, P. Ketterer)

Recent work aimed at a commercial can preservative process for our hydroxymethyl dichloroindazole has centered primarily on simplifying the final hydroxymethylation step to make it more adaptable to plant scale handling. Our goal based on the use of a crude dichloroindazole is to achieve: 1. an acceptably low color, 2. a low free dichloroindazole content and 3. a low non-volatiles (GLC) content. The first two objectives have now been achieved and work is well along on the third.

A novel decolorization system using zinc metal and ammonium chloride in trace amounts on the hydroxymethylation solution has achieved an eight fold reduction of the previously used large carbon requirements. This technique consistently achieves colors below APHA 200 (APHA 400 is tentative spec.). In addition sodium bisulfite has been found to be as effective in aiding color reduction as the previously utilized sodium formaldehyde sulfoxylate. Variable studies on the formaldehyde usage have fixed the minimum requirement to achieve a level of less than 5% of free dichloroindazole (unhydroxymethylated) in our product. Results showed this to be at 5.10 moles formaldehyde per mole dichloroindazole as follows:

Moles HCHO per Mole Dichloroindazole	% Free Dichloroindazole in Product
3.38	31.1
4.31	15.0
4.48	8.9
5.10	4.8

Since methanol is utilized during hydroxymethylation various ratios of methanol/HCHO are being screened to determine which achieves lowest non-volatile content to finalize our process. Pilot Plant work to verify the entire can preservative process will be undertaken in March.

A liquid version of our can preservative (45% solution in methylpyrrolidone) has shown acceptable physical characteristics similar to that of the powder in PVA and acrylic paints. These include odor, viscosity, color, appearance and pH stability after storage for one month at 120°F. Biological efficacy determinations are in progress.

IV. DYES AND INTERMEDIATES

A. Intermediates

1. Leucotetra (M. Feldman)

Our laboratory effort is aimed at developing a product that is acceptable to the trade. Beaufort plant material although used at Paterson has not been found acceptable when offered to potential outside customers. A TLC technique has shown that an impurity present in our plant material, but not present in acceptable Japanese material can be removed by proper sodium hydrosulfite reduction in the final reaction stage. Such a sample was submitted to Toms River Chemical Corporation last month, and on the basis of initial tests additional material was requested by them. Further material has been prepared and submitted. We are proceeding on the assumption that this material will be approved and are developing a procedure to incorporate the improvement into the current plant process.

2. Raw Material Evaluation (M. Feldman, L. Schinasi)

Samples of 1-aminoanthraquinone and aminoazobenzene hydrochloride were evaluated as alternate raw material sources. Biddle-Sawyer's 1-aminoanthraquinone was converted to 1-amino-2,4-dibromoanthraquinone and thence to ABH and found to be acceptable in all respects, while material from American Aniline Products was poor in quality and yielded inferior ABH.

Samples of aminoazobenzene hydrochloride obtained from American Cyanamid and American Aniline Products were converted to Yellow 4RL and found acceptable in dye tests at Paterson. The yields obtained with the American Aniline Products material were, however, too low to be acceptable on an economical basis. Costs were as follows:

Supplier	$ Aminoazobenzene/Lb. 4RL Type
Allied (Current)	0.475
American Cyanamid	0.460
American Aniline Products	0.705

3. Product Data Sheets (A. J. Deinet)

In cooperation with Beaufort and Reading new product data sheets were prepared for our dyestuff intermediates. These included DNS, NAS, PNTS, Leucotetr ABH, Amino J-Acid, J-Acid and J-Acid Urea and reflected the improved quality now obtained consistently with some of those materials.

B. Dyes

1. Direct Bond Yellow G Liquid (L. Port, M. Feldman)

A high priority need has existed to add a Bond Yellow Liquid to our paper dye line. Development of such a product has now been completed. The laboratory has developed a procedure for isolating Direct Yellow 84 as an ammonium salt presscake and formulating this as a liquid with polyethylene glycol and water. The product is an odorless, stable, freeflowing liquid of improved stability over the duPont "Pontamine" Yellow G product. Paper dyeing characteristics were acceptable in all respects. A Laboratory Process Proposal will be issued shortly.

2. Red NAS Liquid (M. Feldman, L. Port, G. McClain)

The addition of a Red NAS Liquid to our line of paper dyes has also been of high priority. The development of such a product is nearly completed. A revised laboratory procedure for isolation of the ammonium salt of Scarlet MG has been successfully verified in the Reading Pilot Plant. Two stable liquid formulations based on this presscake have been made using either urea and 2-amino methyl propanol or diglycolamine, both of which have given acceptable dyeings with improved strength over duPont's "Pontamine" Fast Scarlet 4B. Additional one quart samples have been submitted to Reading for final extensive evaluation. Since these products exhibit an ammoniacal odor the use of various odor masks is also being evaluated.

3. Brilliant Paper Yellow Liquid (Yellow 4) (L. Port)

This has been one of the A priority items on our liquid paper dyes program. A liquid formulation has been achieved from plant presscake using urea/triethylene glycol and this gave acceptable dyeings at Reading. Slight precipitation was noted on storage and is felt to be due to excessive strength (43.9%) as formulated. A lower strength formulation will be prepared and if found to be stable this material can be added to our product line.

5. Direct Blue 3RLP Liquid (L. Port, M. Feldman)

Our liquid formulations developed to date, although showing acceptable application properties on paper dyeings, have not shown acceptable stability properties. Recent formulations which incorporated carboxymethyl cellulose and Cellosize (both high and low viscosity) also showed thickening and some settling on storage. We will continue the screening of other dispersing agents (e.g. Nycol) on a high priority basis.

A. J. Deinet

AJD/srf

1-184 (50M)

INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

TO Dr. R. T. Gottesman AT Piscataway DATE March 1, 1973

FROM Mr. I. Huppert AT Piscataway COPY TO Dr. M. Rosen

SUBJECT MONTHLY SUMMARY - CHEMICAL DEVELOPMENT
FEBRUARY 1973

I. CHEMICALS/SPECIALTIES

A. Chlorination

1. p-Chlorobenzaldehyde (J. DeGaetano)

American Cyanamid evaluated a sample of p-chlorobenzaldehyde produced in the laboratory by sulfuric acid hydrolysis as a purification of standard Fords material. They found the material to be of excellent quality and placed an initial order for 5,000 lbs. Total annual potential is near 300,000 pounds. R & D personnel will work closely with Fords Plant to guarantee that the same high quality product is produced.

B. Phosphates

1. T-2,3-P (O. Blum, J. Sandstedt)

Pilot Plant work was completed which showed the effect of elevated temperature and elongated time cycle on emulsion formation on the $AlCl_3$ catalyzed reaction of DBP and $POCl_3$. Results are:

Run No.	Hold Time Hours	Acid Addition Time °C	Results
D-004:188	10	82	Emulsion
Fords 72-56	10	82	Emulsion
Fords 72-55	1	50	Low yield
D-004:199	1	50	Low yield
D-034:13	1	82	Off color product, low yield

The yield problem is directly related to dibromopropanol quality. The color is related to HCl addition temperature. The emulsion formation is due to a combination of elevated acid addition temperature and long hold time at the elevated temperature.

2. Dibromopropanol (S. Cinco, H. Gould, R. Caruso)

Laboratory work on heel recycle at various reaction temperatures generated the following data:

Reaction Temperature °C	DBP Yield %	T-2,3-P Yield %,$POCl_3$	T-2,3-P Yield %,DBP
10 ± 3	82.5 - 84	92.5-95	90.5-93
20 ± 2	82.5 - 84	93-94	91-92
35 ± 3	81 - 83.5	92-93	90-91
50 ± 2	82 - 83	87-90	85-88

Work at 42°C is in progress to determine the initiation point of the apparent sharp quality difference between 35 and 50°C.

Pilot Plant work verified the laboratory results:

Reaction Temperature °C	DBP Yield %	Laboratory T-2,3-P Yield %,$POCl_3$	Laboratory T-2,3-P Yield %,DBP
20	82.7	92.5	90.4
35	81.7	91.7	89.6
50	81.5	82.0	80.1

Conversion of DBP produced at 35°C to T-2,3-P in the Pilot Plant gave yields of 88.1% on $POCl_3$, and 85.6% on DBP apparently due to degradation during steam stripping. Resolution of this problem is in progress.

A continuous run was made at 50°C resulting in an 83% yield of DBP which converted to T-2,3-P in 88.7% DBP yield. This shows no apparent yield or quality difference between "shotwise" or continuous reaction on a laboratory scale.

Ferric chloride and stannous octoate were unsuccessful as catalysts for the preparation of T-2,3-P. Corrosion testing on T-2,3-P for twenty-six days at 60°C (140°F) showed insignificant changes in the T-2,3-P used and in test couponds of brass, copper, carbon steel, 304 stainless steel, 316 stainless steel and Lithcote LC-20X. The T-2,3-P used was Piscataway Pilot Plant batch 72-46. This corrosion test will be continued for a total of two months. Corrosion tests on Great Lakes and "Tenneco" DBP at 60°C(140°F) for two weeks using Lithcote LC-20X and LC-19 coatings showed the test coupons to be unaffected; however all DBP samples, including controls were badly discolored after 6-8 days.

C. Fuel Oil Additives

1. Chromium 8X (M. Ceprini, A. Fischer)

A revised procedure utilizing the double decomposition reaction of ammonium soaps (hexanoic and ethylhexanoic acids) and chromic chloride has been developed. This process eliminates washing to remove sodium (specification 0.02%, maximum) and is run at 8% chromium concentration. Twelve liter runs are scheduled for the first week in March prior to Pilot Plant verification. Attempts at a fusion process using ammonium dichromate were abandoned due to low chromium content (6.5%) and high viscosity (Z-2+) product.

II. PLASTICS INDUSTRY

A. Additives

1. Tin Stabilizers (M. Ceprini, S. Hoch)

Isooctylthioglycolate (IOTG) from UCB has been shown to be acceptable for the production of BD-600, BD-601, and V-1562 (modified by the incorporation of 5% tris compound). UCB appears to be our only available source. The modification of the V-1562 formulation is required to produce barely functionally acceptable product.

Work on determining factors influencing IOTG acceptability for V-1562 is continuing. Isomer distribution in isooctyl alcohol is apparently the cause of differences in IOTG performance. GLC data to date shows:

IOTG	Major Peak Number	% Area Composition
"Good"	(6	22.0-24.0
	(8	68.0-73.0
"Bad"	(6	29.0-35.0
	(8	54.0-60.0

A program for self-manufacture of IOTG has been initiated.

2. Low Toxicity Stabilizers (S. Hoch)

Two zinc-alkanolamine complexes were prepared for evaluation at Flemington versus standard powdered zinc PVC stabilizers. A laboratory prepared strontium nonylphenate was found to be better in early color in preliminary screening at Piscataway (Plastisol formulation) versus commercial Lubrizol strontium phenate (16.7% Sr). An experimental calcium octoate-isooctylthioglycolate also showed good early color functionality in this test and suggests an area for future consideration. Calcium benzoate,

which may be important as an FDA stabilizer intermediate, was prepared by dry fusion. Similarly prepared was zinc p-t-butylbenzoate. Analysis and evaluation will be undertaken.

B. Components

1. Polymeric Plasticizers (R. Caruso, I. Cooper, M. Koral, J. Sandstedt)

Pilot Plant Process Proposals for NUOPLAZES 6186 and 6187 were issued. NUOPLAZ 6186 is a revised process to reduce plant time cycle. NUOPLAZ 6187 is a new process for our medium molecular weight alcohol capped product.

Pilot Plant equipment has been modified to include a partial condenser to effect glycol economics and reduce time cycles. Initial runs on NUOPLAZ 6189 demonstrated a 25% reduction in polyester formation cycle and reduction of glycol in the water of reaction from 45 to 18%.

The Polymer Laboratory has achieved a one step process for the preparation of NUOPLAZ 6187 which affords single temperature operation, minimizes excess glycol and alcohol charges and significantly reduces polymerization time cycles. The process employs a 1.0/1.0/0.147 mole ratio of 1,3-butylene glycol/adipic acid/isodecyl alcohol and polymerization is completed in 10-1/2 hours (compared to best Pilot Plant times of 15 hours from start of reaction to final viscosity) at 220°C, 20 mm Hg, using the partial condensation technique, and TBT catalysis. Runs made with higher glycol excesses showed no advantages and led to high hydroxyl number product. A confirmatory run will be carried out prior to writing a Laboratory Process Proposal.

2. DHP (O. Fuchs)

A repeat preparation of dihexyl phthalate without catalyst was found to be functionally unacceptable by the Flemington Applications Laboratory. The previous preparation prepared without catalyst was acceptable. No further work is planned on this project.

3. AG-398B (I. Cooper)

The Pilot Plant prepared one drum of AG-398B (450 lbs. net) to fill a customer order. Upon approval of a pre-shipment sample this material will be released to GAF. Intermediates I-230 and I-231 were made via a revised process.

Additional effort will be required to fully develop the I-230 procedure (process control points) should AG-398B become a viable product.

III. COATINGS INDUSTRY

A. Driers

1. Calcium 5% in Toluene (A. Fischer)

A revised laboratory process for R-372, 5% calcium naphthenate in toluene, was developed incorporating recommendations by Manufacturing for production at the Long Beach, California Plant. The revised process includes use of A-47, in place of A-94, and all isolation operations in mineral spirits followed by dilution with toluene to minimize handling of this flammable solvent.

2. Water Dispersible Driers (J. DeGaetano, G. Kagan)

In an attempt to increase our product line standard Cyclodex Manganese 6%, Lead 24%, Cobalt 6% driers were prepared from naphthenates using prior formulations. However, it was found that calcium no longer will work in the Cyclodex formulation. A 4% water dispersible calcium was prepared from Calcium Naphthenate 5% which contains methylisobutyl carbinol acid phosphate (I-46). A 6% calcium was also prepared from NuXtra Calcium using Plurofac RA-40 as a surfactant. A 12% zirconium water dispersible drier was prepared from NuXtra Zirconium 18% using the Plurofac RA-40. Attempts to prepare 12% Cobalt and 9% Manganese were unsuccessful. Evaluation of additional surfactants is in progress. The Elizabeth Plant has prepared Cyclodex Manganese 6%, Lead 24% and Cobalt 6% for customer sampling and will prepare 4 and 6% calcium and 12% zirconium early in March when they receive Laboratory Process Proposals.

Cyclodex Cobalt and Lead were compared to the Advacar (Cincinnati Milacron) and Aquadry (Borcher's) cobalt and lead in a black enamel. The data shown below indicates that the Aquadry cobalt and lead imparted a much higher gloss after aging (desirable) than either the Cyclodex or Advacar systems. However, they also provided the soft test film (undesirable). The Cyclodex and Advacar systems were comparable in gloss with the Cyclodex showing a harder film. All systems showed poor flexibility.

	Advacar Co & Pb	Cyclodex Co & Pb	Aquadry Co & Pb
Gloss 60°			
Initial	91	91	90
2 Weeks*	83	79	92
Hardness (Sward)			
Initial	3	4	2
2 Weeks*	5	8	4
Flexibility			
Initial	Good	Good	Good
2 Weeks*	Good	Good	Good

*Paints aged for 2 weeks @ 120°F

IV. LUBRICANTS (O. Fuchs, I. Cooper)

The preparation of I-1121, a Tenneco proprietary lubricant additive, using Alfol 1216 (C_{12}, C_{14}, C_{16} alcohol mixture from Conoco), or CO-1214N (Proctor and Gamble's analogue) in place of the no longer available Lorol 5 from DuPont has been completed. Results of the Falex EP test show Alfol 1216 is the preferred substitute alcohol. It will be used in future Pilot Plant production.

V. COLORS

A. Intermediates

1. p-Aminoacetanilide (E. Reinhold, G. F. McClain)

Work in the Reading Pilot Plant lab has resulted in a satisfactory solution of two of the major problems in the Pilot Plant verification of the laboratory process for this product, namely the nitration and the reduction steps. The use of a programmed acetanilide and mixed acid addition has eliminated crystallization on the vessel walls which detrimentally effected heat transfer during the nitration reaction. The use of 80 mesh in place of 60 mesh iron and 1.5 times the laboratory reaction solvent volume has increased reactivity and eliminated filtration problems in the reduction step. The remaining problem involves removal of acid from the nitro(PNAA)presscake prior to reduction. Two solutions are currently under investigation: 1) decantation of the aqueous-acid layer from the nitro slurry prior to filtration, 2) neutralization of the acid in the nitro slurry with liquid caustic prior to filtration. Work in the Pilot Plant proper will be reinstituted upon solution of the above problem.

B. Liquid Dyes

1. Blue 3RLP (G. F. McClain, E. Reinhold)

Differences in plant versus Pilot Plant production of Blue D (the base for Blue 3RLP liquid) have been traced to filtration temperature. The plant has routinely filtered at 80 to 85°C, while the Pilot Plant filters at 65 to 70°C. Pilot Plant runs at 80 to 85°C resulted in a hard, lumpy material on drying which required milling through an 0.027 inch herringbone screen prior to liquid formulation. Runs at 65 to 70°C gave soft, friable lumps on drying which readily formulated to Blue 3RLP. Yield of the three most recent pilot batches averaged 125% of the present plant standard and when final tabulation and testing is complete a Process Change Recommendation will be written for plant use and a demonstration batch will be scheduled in the Reading Plant. This should result in a plant Blue D satisfactory for producing Blue 3RLP.

2. Duronyl Fast Black KPD Liquid (E. Reinhold, G. F. McClain)

The two batches produced in January totalled 9,080 pounds and three additional batches produced in February bring the year to date total to 22,190 pounds.

C. Engineering

1. Thickener (G. F. McClain)

The objective of the thickener program is to determine its feasibility for use on various dyes with subsequent drum drying to eliminate manual operations associated with filter presses and tray driers. The Reading Plant uses a large centrifugal pump to feed the thickener, which has been shown to reduce crystals from 40 to 5-10 microns. The Pilot Plant unit was set up using a Moyno pump to prevent crystal attrition. It was determined that materials having crystal sizes below 15 microns will not satisfactory thicken. Eight products have been evaluated in the Pilot Plant. Only two, Blacks OB and GX, are routinely thickened in the plant. Agglomerate breakdown from 100 micron to less than 5 microns was observed in the Pilot Plant unit using Red C. Current work is determining whether agglomerate breakdown is due to the Moyno pump or the thickener itself. Other pumps, particularly the Sandpiper double diaphragm unit, will be evaluated.

I. Huppert

IH/sf

INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

To Dr. R. T. Gottesman At Piscataway Date February 27, 1973

From Dr. F. Buono At Piscataway Copy to Dr. M. Rosen

Subject

MONTHLY SUMMARY - FEBRUARY 1973
APPLICATIONS DEVELOPMENT SECTION

I. COATINGS INDUSTRY

A. Microbiocides (P. Minieri, H. Sidi, E. DiBella, G. Trautenberg, G. Kagan, M. Landau)

The overall objective of this program is to develop new and novel non-mercurial biocides. The current synthesis and screening emphasis has been directed toward the development of a non-mercurial fungicide for latex paints. The efforts in the can preservative program are concerned with screening potential materials that might show performance superior to our current candidate, N-hydroxymethyl dichloroindazole.

1. Screening (Biocide and Compatibility)

(a) TID Candidate Compounds

Two of the three TID compounds reported last month to possess significant antimicrobial activity (1-hydroxymethyl-2-methylimidazole and hydrazinium p-toluate) have shown unacceptable paint compatibility at the biologically effective levels. The remaining compound (hydrazinium isonicotinate) is being evaluated as a potential can preservative.

Paint compatibility evaluations on two potential fungicides, N-hydroxymethyl benzimidazole and dichloroindazole Zn composition, have shown acceptable performance with the benzimidazole (@1.5%) in PVA paint and with the dichloroindazole (@0.25%) in both PVA and acrylic paint. Both materials have been test fence exposed.

Two TID compounds (methyl-3,5-dichloro-4-hydroxybenzoate and $\alpha,\alpha,2',4'$-tetrachloroacetophenone) that are showing promise as potential fungicides (based on test fence exposure) have been evaluated for paint compatibility on the basis that an acceptable candidate in a PVA (methyl-3,5-dichloro-4-hydroxybenzoate) and an acceptable material in acrylic paint ($\alpha,\alpha,2',4'$-tetrachloroacetophenone), as a mixed blend, would give a system that might be adequate for both latex paints.

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February 27, 1973

(a) TID Candidate Compounds (continued)

The blends, however, were unacceptable because of pH instability, film discoloration, and yellowness (the blends consisted of varying fungicidal ratios of the two materials).

The evaluation of six TID compounds as potential can preservatives has been completed. One compound, hydrazine salt of p-tertbutylbenzoic acid, showed efficacy equal to our current candidate, but also showed unacceptable paint compatibility.

Twenty-five TID candidate compounds were primary (biocide) screened (2%) this month. Of these, six, shown below, showed antimicrobial activity but some paint incompatibility. These are being screened at lower levels in an effort to eliminate the paint incompatibility without loss in activity.

1. Benzoylhydrazine 98%
2. 5-Chlorobenzotriazole 99%
3. Isonicotinic acid azide
4. 1-Hydroxymethyl-2-methyl-5-chlorobenzimidazole
5. 1-Methyl-3-hydroxymethyl phthalazone
6. 1,2-Dihydroxymethylbenzimidazole.

Twenty-six TID candidates have been submitted this month for primary screening.

(b) Tenneco Pasadena Laboratory Candidate Compounds

Four submissions underwent primary screening. Three compounds, 4-phenyl-3-butyn-2-one, 6-phenyl-5-hexyn-4-one and 1-phenyl-2,4-hexadiyne-1-one showed good to excellent fungicidal activity in the latex paints. However, all three showed color and/or imparted an unacceptable odor to the paint. The Pasadena Laboratories anticipate further work with these materials.

(c) Maybridge Chemical Candidate Compounds

None of the twenty compounds evaluated showed antimicrobial activity. A three month summary report on all of the Maybridge compounds screened thus far (50) will be issued next month.

(d) Chevron Candidate Compounds

Two potential vinyl biocides from Chevron, NU74-2 and NU4-5, have been further evaluated to determine their lowest level of efficacy. Based on surface fungal resistance and zone of inhibition formation, 0.5% on total formula weight for both materials was the lowest efficacy concentration. The zone formation of these materials, at equal biocide concentrations, was superior to both Fungitrol 11 and Fungitrol 22, although the surface fungal resistance was comparable. Studies on weatherability will be undertaken. NU74-2, which also shows excellent fungicidal activity in latex paints, is undergoing paint compatibility evaluations.

2. ASTM - Can Stability

Paint spoilage cultures obtained via the ASTM "round-robin" testing program are being readied for can stability evaluation. This program is to eventually lead to the elimination or modification of the present inadequate ASTM method, ASTM D2574-67T.

3. Competitive Products

(a) Fungicides - Nopcocide N-96

The previously reported biocidal efficacy of the above product from the Nopco Division of Diamond Shamrock showed that, at equal biocide levels (1.5, 1.0 and 0.5% on weight of paint), Super Ad-It would out-perform Nopcocide N-96 in latex paints while in oil paint, Nopcocide N-96 and Fungitrol 11 were comparable. The paint compatibility evaluation of the Nopco product has now been completed. The material was only found acceptable at the 0.5% test level in oil and PVA paints. Higher levels showed photosensitivity, discoloration, viscosity and pH instability and/or poor brightness. It was not acceptable at any test level in the acrylic paint. Panels containing Nopcocide N-96 are being exposed in Florida and Piscataway to generate performance data.

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B. Driers (G. Kagan, M. Landau, A. Fischer)

1. NuXtra Zirconium 24%

A NuXtra Zirconium 24% was developed to match a competitive Ferro product and to round out the Tenneco line of zirconium driers. The two products are chemically equivalent, as determined by infra-red analysis. The NuXtra drier is more readily soluble than the Ferro product in raw linseed oil (a common test vehicle) even though the viscosity of the latter is lower than that for the NuXtra drier. The functional evaluation (thru-dry) shown below indicates that the NuXtra product is comparable to the competitive product. Data is also given for the regular production NuXtra Zirconium 18% vs. Ferro's Zirconium 18% for comparison.

	Thru-Dry Times Hours:Minutes			
	24% Zirconium		18% Zirconium	
	NuXtra	Ferro	NuXtra	Ferro
Initial	2:50	2:50	2:35	2:30
Aged for:				
1 Month @120°F	2:25	2:40	2:30	2:30

2. Pb Driers

Four-ounce samples of lead 2-ethylhexoate (40.7% Pb) and lead naphthenate (35.2% Pb) were prepared in the absence of solvent for toxicity studies, initiated and conducted by New York University.

3. Loss-of-Dry Inhibitor

The primary objective of this program is to develop a proprietary non-staining loss-of-dry inhibitor.

Blends consisting of cobalt/calcium/zinc and cobalt/calcium/zirconium are continuing to show promise as loss-of-dry inhibitor/composite drier systems after two months' aging. This is shown in the table below using a black enamel that is quite prone to loss-of-dry. Similar data were obtained in a red enamel. The response of cobalt/calcium/zinc blend at 5% by weight on vehicle solids is comparable to that shown by Cobalt 254.

3. Loss-of-Dry Inhibitor (continued)

	Thru-Dry Times Hours:Minutes			
	Control	Control (0.7% Co 254)	5% Co/Ca/Zr	5% Co/Ca/Zn
Initial	4:05	4:10	3:35	4:00
Aged for:				
1 Month @120°F	14:00	6:05	6:15	6:25
2 Months @120°F	17:15	6:15	9:20	6:00

These blends are now undergoing evaluation in several oil-based house paints and other paint systems which are prone to loss-of-dry on aging. The staining potential in a white paint is currently under investigation.

The basic soluble calcium in conjunction with soluble cobalt which has shown promise as a loss-of-dry inhibitor showed unacceptable staining in a white paint. No further work on this system is planned.

C. Thickeners (G. Kagan)

The objective of this program is to develop a thickener for latex paints showing performance characteristics equal to or superior than the cellulosics (i.e., hydroxyethyl cellulose, Natrosol 250HR). The program has almost exclusively involved evaluation of experimental guar-type thickeners from Stein-Hall.

A sample of hydroxypropyl guar (HPG) has been evaluated in both PVA and acrylic systems. This product gives considerably better grinds in both paints when compared to Nuvis 44WA (hydroxyethyl guar, HEG). In two PVA paints, grinds approaching those of the Natrosol 250 HR comparator have been obtained. However, in two acrylic test paints, grinds with HPG have been unacceptable (i.e., HPG shows grinds 2 Hegman units lower than the 250 HR). Nevertheless, these grinds are a significant improvement over those shown by Nuvis 44WA. HPG has shown good viscosity stability in the two acrylic paints but has shown poor gloss in an acrylic semi-gloss paint.

In general, HPG exhibits better overall performance when compared to HEG but is still not acceptable as a broad spectrum thickener. Stein-Hall will be provided with these results so they may direct their program toward improved versions of HPG and/or other guar modifications for our evaluation.

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February 27, 1973

II. LUBRICANTS (M. Knapp, R. Stanabach)

This month's summary of the R & D effort in lubricants can be found in the monthly summary prepared by Mr. I. Huppert.

F. Buono

F. Buono

FB/amp

I-184 (5M4)

INTER-OFFICE MEMO **TENNECO CHEMICALS, INC.**

TO	Dr. R. T. Gottesman	AT	Piscataway	DATE	February 27, 1973
FROM	Mr. H. R. Johnson	AT	Piscataway	COPY TO	Dr. M. Rosen
SUBJECT	MONTHLY SUMMARY - FEBRUARY 1973 ANALYTICAL SERVICES				

METHODS DEVELOPMENT

1. Microbiocides (I. Wakeman, D. Tylicke)

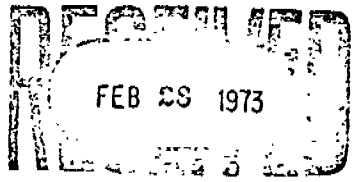
   A procedure based on flame ionization gas chromatography has been developed for the determination of our can preservative candidate in PVA and acrylic paints. Removal of the dichloroindazole via an ether extraction technique is quantitative, and determination of levels down to at least 0.005% are feasible.

2. NUOPLAZ 6187 (I. Wakeman)

   The compositional analysis of NUOPLAZ 6187 by gas chromatography of the saponified sample has been issued as Procedure No. GC-72.0.

H. R. Johnson

HRJ/sf

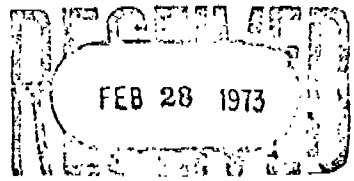


R. T. GOTTESMAN

INTER-OFFICE MEMO - TENNECO CHEMICALS, INC.

TO	Dr. R. T. Gottesman	AT	Piscataway	DATE	February 26, 1973
FROM	Mr. R. S. Miller	AT	Piscataway	COPY TO	Dr. M. Rosen

SUBJECT MONTHLY REPORT
POLYMERIZATION R&D SECTION, FEBRUARY 1973



PLASTICS INDUSTRY

Additives

1. Flow Modifier (D. Goodman, L. Gross)

Previous work had established that substitution of Alcolac, liquid, high purity sodium lauryl sulfates for Duponol WAQE reduced haze in Supercryl/PVC compounds to levels of commercial competitors. Alcolac's recommended statistically optimized candidate (D021-30, 32, 34) was obtained and compared to the best previous Alcolac liquid (D021-31), and two Onyx Chemical solid candidates in Supercryl 100 polymerizations with the following results:

TABLE I

Flow Modifier Evaluation

Sample	Surfactant	Supplier	Light Trans. (%)	Haze (%)
Supercryl 100	Duponol	DuPont	89.5	8.0
Ionac PA-910	-	-	93.4	4.9
Acryloid K-120 ND	-	-	93.1	4.7
D021-30	DV-813-J	Alcolac	90.0	5.9
D021-31	DV-813-D	Alcolac	92.3	3.9
D021-32	DV-813-J	Alcolac	92.9	5.2
D021-34	DV-813-J	Alcolac	92.6	5.9
D021-33	Maprofix LK	Onyx	91.2	5.4
D021-35	Maprofix 563	Onyx	94.3	4.6

Sample D021-31 rather than the Alcolac recommendation again appears to provide the most haze-free product of the liquids and will be obtained for plant scale trials. Maprofix 563, which appears to offer improved light transmission, will not be pursued at this time since dissolving would require additional process steps in the plant.

2. Impact Modifier (D. Goodman, L. Gross)

Previous laboratory impact modifiers based on Firestone SR-6133 latex (1100-1300Å) had good impact strength, clarity and crease whitening resistance but poor mill stick properties. These deficiencies have been overcome in samples based on Firestone SR-6189 and SR-6196 latex (600-800Å) which were prepared by the two stage graft method and isolated by salt coagulation and oven drying. Several materials were comparable to Kureha BTA-III in properties examined.

TABLE II

Impact Modifier Evaluation

Sample	Izod Impact Strength (ft lbs)	Mill Stick (min)	Early Color	Latex
BTA-III	8.0	17.5	Good	-
D021-26	6.7	35 +	Comparable	SR-6196
D021-7	9.5	35 +	Fair	SR-6189
D021-11	8.6	35 +	Comparable	SR-6189

More extensive functional evaluation is underway on 46/37/17 butadiene/styrene/methyl methacrylate compositions from SR-6189 and SR-6196 to determine impact efficiencies and compounding sensitivity. A progress report on SBR based laboratory impact modifiers and a patent analysis are being completed to obtain a legal opinion on this very complicated patent situation before expanding this program into Pilot Plant studies.

PVC Resins

1. Chemical Foam Blending Resin (J. C. Fisher, M. K. Rosen)

Efforts were continued to confirm the non-wicking characteristics of gelatin BR-501 resin in the Armstrong test. Although the standard charge procedure gelatin batches gave good foam, the resin is too coarse. Homogenization of the initial charge is now under study as a means of reducing the particle size. Initial evaluation of this technique produced resin with poorer wicking characteristics, however batches prepared with an improved balance of gelatin levels and homogenization conditions are required for a definitive answer.

Further work with Acrysol G-110 (ammonium polyacrylate) has verified its poor wicking characteristics when used as the sole suspending agent or in combination with gelatin. Some improvement was noted with a TAIC (triallyl isocyanurate) modified version of the standard Methocel BR-501 formulation which will be followed up.

2. Tri-Catalyst System (J. C. Fisher, M. K. Rosen)

A Pilot Plant study of appropriate tri-catalyst ratios and levels for use in 250 resin has been completed. The results confirm earlier work with 225 resin and verify more even cooling water utilization at current plant cycle time and avoidance of heat kicks and full cooling water utilization at reduced reaction times as shown below:

TABLE III

Tri-Catalyst System, 250 Resin

Catalyst			Cooling Water Temp. (°F)						
199 P (phm)	223 M (phm)	L.P. (phm)	1 hr	2 hrs	4 hrs	6 hrs	8 hrs	10 hrs	Complete hrs
	0.0291	0.0136	127	124	120	118	115	113	11.0
0.0138	0.0169	0.007	122	121	121	121	121	120	11.3
0.0151	0.0186	0.0077	116	116	117	116	116	-	9.7

A 1-2 hour reaction time reduction is projected for the plant with this system

3. PPG Industries, EHP (D. Goodman, M. Koral)

Simultaneous evaluation in bottles of di-2-ethylhexyl peroxydicarbonate (EHP) catalysts supplied by Lucidol and PPG Industries produced equivalent results in mono, dual and tri catalyst systems when charged on an equal activity basis. PPG has failed twice to provide EHP to an equivalent dilution to Lupersol 223 and the low assay prevents using the two catalysts on an equal weight solution basis which is common plant practice. Laboratory efforts continue to get PPG to supply EHP at equivalent assay so they can be approved as a supplier. Kinetic results are tabulated below for suspension PVC bottle polymerization.

TABLE IV

PPG Versus Lucidol EHP Kinetics @ 132°F

Catalyst System	Comparison	Conversion (%@) 3 hrs	5 hrs	7 h
Lucidol, EHP (.03)	Equal Activity	25.6	42.6	58.
PPG, EHP (.03)	Equal Activity	25.6	42.6	58.
Lucidol, EHP/LP (.028/.009)	Equal Activity	26.9	46.3	60.
PPG, EHP/LP (.028/.009)	Equal Activity	27.5	46.9	60.
Lucidol, EHP/LP/199P (.0143/.0107/.0159)	Equal Activity	33.1	55.6	76.
PPG, EHP/LP/199P (.0143/.0107/.0159)	Equal Activity	33.1	55.6	76.
Lucidol, EHP (.03)	Equal Solution Weight	27.5	43.7	63.
PPG, EHP (.03)	Equal Solution Weight	23.5	38.1	55.

4. Terpene Modified PVC (D. Goodman, M. Koral)

Additional PVC/β-pinene copolymer was prepared at optimized incorporation and catalyst levels to test the improved heat stability of these copolymers reported in U.S. 3,666,734 for a possible use of Newport Division products.

5. Dispersion Latex Defoamers (J. C. Fisher, M. K. Rosen)

Burlington 1757 latex was treated with Colloids 987 defoamer in the Pilot Plant and processed along with a tributyl phosphate control. No differences in filtration rate, cake moisture or standard Q.C. test results were observed. for this lower cost defoamer. Applications tests are pending on these samples.

R. S. Miller

RSM/srf