

TO Distribution List AT DATE October 28, 1974
FROM Dr. R. T. Gottesman AT Piscataway COPY TO
SUBJECT MONTHLY SUMMARY - OCTOBER 1974

Attached are copies of the Monthly Summary reports for October 1974 covering the activities of the Piscataway Laboratories and associated pilot plants at Piscataway and Flemington.

R. T. Gottesman
R. T. Gottesman

RTG/ec

Attachments

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(b) Candidates from Jet Propulsion Laboratories (Caltech)

Three candidates from JPL underwent primary bioscreening this month. Two compounds (3,3-Ionene and 6,4-Ionene) showed significant broad spectrum activity and additional quantities have been requested for paint compatibility evaluations.

2. Fungitrol 11 (G. A. Trautenberg)

The complete Piscataway test fence competitive product exposure series involving Fungitrol 11 has been updated. The data (up to 48 months' exposure) dramatically demonstrates the superiority of Fungitrol 11 over Vancide PA, Dow S-13, Skane M-8 and Nopocide N-96. Charts and slides are being prepared for presentation at the Tenneco agents' breakfast at the Paint Show and for additional technical presentations and literature.

3. Fungitrol 11 Dispersion (G. M. Kagan)

In 1973 a Fungitrol 11 dispersion was developed as a potential liquid adjunct to the powdered version. This dispersion consisted of 50% Fungitrol 11, 39% Nuosperse 657, 10.8% mineral spirits, and 0.2% Exkin 2. At Marketing's request attempts have been made to increase the active level of Fungitrol 11 from 50% to 55%. Two dispersions were prepared successfully at the 55% Fungitrol 11 level; however, these exhibited poor viscosity stability (compared with the 50% version) after aging for two weeks at 120°F. Since viscosity stability is an important consideration in a product of this nature and the original formulation is acceptable, the dispersion will be finalized on this formulation.

* B. Wacker's Vinnapas Dispersions (G. M. Kagan)

A sample of a Union Carbide terpolymer (i.e., vinyl acetate/vinyl chloride/ethylene) latex designated Ucar 1000, which is being offered to the paint trade on a commercial development basis, has been obtained for evaluation because it represents potential competition for Wacker's terpolymer CEF 10/21. Scrub results in three test (PVA, acrylic, Air Products terpolymer) formulations show that Ucar 1000 imparts substantially poorer scrub resistance than CEF 10/21 and apparently exhibits no advantage in this respect over the conventional PVA and acrylic latexes previously evaluated. These results lead to two conclusions: (1) Ucar 1000 would not be a serious threat to CEF 10/21 for interior paints and (2) the mere presence of vinyl chloride in a polymer does not necessarily guarantee good scrub resistance.

C. Nuosperse 657 (G. M. Kagan)

In an effort to develop data to promote Nuosperse 657 as a substitute for soya lecithin which is in short supply, dispersion studies have been conducted with these dispersants in five pigmentation systems, i.e., titanium dioxide, red iron oxide, yellow iron oxide, violet toner, and phthalocyanine green. Substitutions made on a formula cost replacement basis have shown performance advantages for Nuosperse 657 over soya lecithin in titanium dioxide (faster grinds, better color response) and red iron oxide (faster grinds, better gloss and color development) dispersions. Studies are currently underway comparing these products on a weight replacement basis. Results obtained thus far indicate that Nuosperse 657 provides better color development of a permanent violet toner. These data will be used to prepare a technical bulletin showing the advantages of Nuosperse 657 over soya lecithin.

* D. Servo Additives

1. Ser-Ad MSB 203 (G. M. Kagan, M. Landau)

Ser-Ad MSB 203, an anti-settling agent, was evaluated in Servo's recommended wood stain formulation. At room temperature, the Servo product showed good performance (no settling) compared to a control with no additive (severe settling). However, samples aged for one week at 120°F showed slight settling in both cases. Ser-Ad MSB 203 provided lower Hegman grinds than the control and particularly marked grind differences were evidenced with the 120°F samples (control = 7 grind; MSB 203 = 1 grind). Based on the heat sensitivity and poor grind responses of Ser-Ad MSB 203, no further work will be done with this product.

2. Serdas MOS Defoamer (G. M. Kagan)

Serdas MOS was evaluated versus Foamacure 142, Nuodex AF-100 and Serdas GFN in accordance with Functional Procedure FP-13. It exhibited significantly poorer (unacceptable) performance than the three comparators in both PVA and acrylic latexes. No further work will be done with this product.

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II. LUBRICANTS

* A. Crankcase Lubricant (M. H. Knapp, R. S. Smith)

Analysis of the scuffed and worn rocker arm pivots from our latest IIC test indicated that failure was due to fretting corrosion. This is a type of wear caused by small reciprocating sliding motions of machine parts--in this case the steel rocker arms against the aluminum rocker arm pivots. It is called corrosion because of the characteristic presence of oxidized wear debris. A new candidate has been formulated by Chevron Research using additives which inhibit aluminum oxidation. Although Chevron has not revealed the chemical identity of these additives, it is likely they are using 1,2 or 1,3 alkanediols and/or aliphatic or alkaryl β -ketols which are known to be effective. Attempts to do our own evaluations of these additives were unsuccessful due to the limitations of our test equipment (Falex tester).

A 55 gallon drum of base fluid (DTDA) was sent to Chevron for their use in blending candidates. A candidate incorporating the fretting corrosion inhibitor will be sent out by Chevron to Southwest Research during the week of October 28. Results are expected by mid-November. If the candidate passes, the L-38, VC, and IIC tests will be run.

B. Compressor Lubricants (M. H. Knapp)

A sample of Westvaco's "Diamyl ester of Westvaco Diacid" CHR-74113 was received and evaluated as a potential synthetic lubricant base oil. In comparison to DTDA, it was found to have a higher acid number, lower decomposition temperature, and a tendency toward forming deposits at elevated temperatures. For these reasons it was dropped from consideration.

* C. Two-Cycle Lubricants (M. H. Knapp, R. G. Smith)

The intake air vents were modified as planned and this was found to reduce some of the noise generated by the test engine. It did not, however, totally eliminate noise transmission downstairs to the Order Department. The sound level there is still 3 dB(A) over the existing noise level (64 dB(A)). Additional soundproofing material has been ordered to reduce sound transmission through the concrete walls and floors.

F. J. Buono
F. J. Buono

FJB/amp

COLORITE 011452

TENNECO CHEMICALS, INC.

TO Dr. R. T. Gottesman AT Piscataway DATE October 28, 1974
 FROM Dr. A. J. Deinet AT Piscataway COPY TO
 SUBJECT MONTHLY SUMMARY - OCTOBER 1974

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I. CHEMICALS/SPECIALTIES

A. Formaldehyde Stabilizer (E. DiBella)

- * Laboratory work has been completed on a preparative procedure for our proprietary formaldehyde stabilizer, palmitic diisopropanolamide, from palmitoyl chloride and diisopropanolamine. This route has advantages over the previous one which utilized palmitic acid in that no formaldehyde insoluble by-products are formed, thus yielding a haze-free formaldehyde and eliminating a troublesome filtration. A pilot plant run will be undertaken to supply sufficient stabilizer for plant trials.

B. Monochlorotoluene (E. DiBella, H. Gould, H. Sidi)

- * Various parameters associated with the production of monochlorotoluene (MCT) were studied in the laboratory in order to facilitate planned plant revisions aimed at increased productivity. These indicated the following:
 1. Steam distillation of crude MCT from caustic soda as practiced in the plant does not eliminate side-chain chlorine. Plant data, however, has shown that in a proper chlorination the benzyl chloride content is <0.02% and that this is not a problem. Treatment with hot aqueous hexamethylenetetramine will, however, readily remove benzyl chloride should this become a necessity.
 2. A chlorination curve based on sulfur monochloride as a catalyst was developed for MCT and indicated maximum monochlorotoluene (98%) content at a specific gravity of 1.07. A curve for cascade overflow reactors is also being developed.
 3. Chlorinations at 35 and 50°C. indicate no differences in p/o isomer ratios or benzyl chloride content. The lower temperature is favored due to less volatilization losses.

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4. A ten-fold increase in sulfur monochloride catalyst over plant level offered no advantages in p/o ratio and tended to coagulate iron powder if present.
5. Use of toluene containing 1000 ppm of moisture is not detrimental to the chlorination and thus enables recycle of toluene without special drying.

Future work will concentrate on improving the p/o ratio and will include catalyst studies, isomerization techniques and alternate chlorination methods (e.g. with ferric chloride). The use of the latter has shown a p/o ratio of 9/1 in a trial run based on prior literature, but requires simplification to be economically feasible.

II. PLASTICS INDUSTRY

A. Additives

- * 1. Flame-Retardants (E. DiBella, H. Sidi, H. Gould, M. Reale)

A simplified procedure has been developed for our prime-candidate R-525, bis (1,3-dichloropropyl) tribromoneopentyl phosphate which eliminates two of the three vacuum strippings. A pilot plant run is scheduled via this procedure to provide material for an additional lab-line run and possible customer sampling.

Evaluation of our encapsulated HBB-2 at Scott Paper indicated unsatisfactory flame-retardancy in both polyether and polyester systems, but no objectional odor was noted. Further encapsulation studies will be held in abeyance pending the disposition of HBB-2 at Asco. Poor flame-retardancy and foam formation was obtained in our evaluation of encapsulated boric acid with FR-2 (30 PHP and 20 PHP respectively).

Pentabromophenol was an effective flame-retardant at 5 PHP, but foams were somewhat tight. Our formulation will be modified to compensate for this.

An R-525 analog, phenyl (1,3-dichloropropyl) tribromoneopentyl phosphate was an effective flame-retardant in polyurethane foams at 10 and 15 PHP, but showed no advantages over R-525. This material, however, will be included as a patent example in an ICDS for these types.

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2. Tin Stabilizers (S. Hoch, M. Spalding)

A sample of basic dibutyltin bis propyl maleate (25% Sn) was prepared and found to be a functional match for Ferro 837 in plastisol applications (static oven heat stability and physical and optical property comparisons). This material is of particular utility where long term color hold is of particular importance. A method for its preparation has been developed and a Laboratory Process Proposal is to be issued.

3. Powder Stabilizers (S. Hoch, J. Dunn)

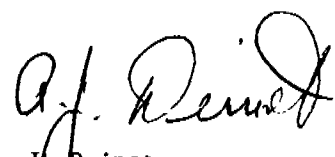
Samples of the zinc, calcium and barium salts of Westvaco's 1485 dimer acid previously submitted to the Flemington Plastics Applications Laboratory were found to be essentially equal to the stearate controls on an equal metal basis both as the individual metal salts and as stabilizer components (NUOSTABE V-1250 and V-1005) in PVC oven heat stability tests. These materials can thus be considered as possible stearate substitutes should stearic acid pricing and supply warrant this.

Samples of the cadmium salts of Emery's 657 (C₈), 658 (C₈ and C₁₀) and 658 (C₁₀) fatty acids as well as a basic cadmium laurate have been prepared for evaluation as possible substitutes for cadmium pelargonate (I-282). The latter acid is in short supply and required for powder stabilizer production at the Piscataway plant.

III. COATINGS INDUSTRY

A. Driers (A. Fischer)

Runs of Manganese driers based on the use of Diamond-Shamrock's new reactive manganese oxide have shown that fusion runs e.g. with 2-ethylhexanoic (A-97) and isononanoic (A-242) acids are feasible and these have shown reaction times as short as 2 hours. Procedures are being developed for our line of manganese products based on this raw material since productivity over our current double decomposition route would be increased and raw material cost lowered. In addition, there would be no problems in meeting low sodium specifications, which can be a problem with the current route.


A. J. Deinet

AJD/msk

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

TO Dr. R. T. Gottesman AT Piscataway DATE October 28, 1974
FROM Mr. I. Huppert AT Piscataway COPY TO
SUBJECT MONTHLY SUMMARY - CHEMICAL DEVELOPMENT
OCTOBER 1974

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I. ORGANICSA. p-Chlorobenzal Chloride (J. DeGaetano)

A sample of "P/C Benzal Chloride" from Hooker was evaluated analytically and synthetically. This material, containing 89.4% p/Cl benzal chloride and 8.67% combined p and o/Cl benzyl chloride, was converted to specification-acceptable p-chlorobenzaldehyde via the zinc phosphate catalyzed steam hydrolysis process. The yield based on p/Cl benzal chloride assay basis was 86.3% which represented 0.55 pounds of product from each pound of p/Cl benzal chloride on a weight basis. If economics are satisfactory, this material is recommended for use as a Fords Plant raw material.

B. LubricantsAnderol 415 (414) (O. Fuchs, M. Q. Ceprini)

Approximately 10-11 gallons of Anderol 415 Watch Oil, also known as Anderol 414, have been prepared from existing raw materials. Functional tests are in progress at the Chestertown Control Laboratory.

Anderol 416 (R. Caruso, M. Q. Ceprini, O. Fuchs)

A new supply of I-4902 was prepared in the Pilot Plant and was converted to two 12 gallon batches of Anderol 416 in the Process Development Laboratory. Functional evaluations are in progress in the Chestertown Control Laboratory, after which time these supplies will be incorporated into inventory for customer sales.

C. Foamacure 142 (L. Schinas)

Another plant batch (Lot #72999) of Foamacure 142 was made at the Elizabeth Plant on September 20, 1974 incorporating a process modification recommended in Research Report 3401-7, July 30, 1974. This modification entailed adding only the amount of Nuosperse HOH needed to fluidize the batch after cooling the batch to 100°F. Stability tests at ambient temperature and 120°F showed no separation after 4 weeks.

COLORITE 011456

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II. POLYMERS

- A. Plasticizer Expansion (M. Q. Ceprini, S. Cinco, J. DeGaetano, O. Fuchs, J. McBroom, L. Schinasi, F. Roesler, G. Trautenberg, G. Tirpak)

A literature search covering 1962-1972 revealed no fundamentally new concepts in plasticizer manufacture. A laboratory evaluation of the process currently in use at Chestertown was made and compared to the only large scale commercial process (Rhone-Progil) for which information was at least partially available. Results of this comparison, presented below, showed the Chestertown process to be equal to the competitive process with respect to yield. In addition, the TOP process is a proven process with no "unknown" values such as quality of recovered alcohol, the necessity of its subsequent rehydrogenation prior to recycle and the required decomposition of by-product dialkyl sulfates.

YIELD COMPARISON OF VARIOUS DOP PROCESSES

	<u>LABORATORY</u>	<u>CHESTERTOWN</u>	<u>RHONE-PROGIL</u> ⁽²⁾	
<u>% Yield, On Phthalic Anhydride</u>			<u>Before Recycle</u>	<u>After Recycle</u>
Unfiltered	99.3	98.5 ⁽¹⁾		
Filtered	98.2	98.0	99.0	99.3
<u>% Yield, On 2-Ethylhexanol</u>	99.3	99.3	98.1	99.2

(1) Estimated 0.5% hold-up in filter.

(2) These are projected yields based on recovery of phthalic anhydride and 2-ethylhexanol from procedures which are not yet commercially proven.

The slightly lower yield (on phthalic anhydride) in the TOP plant is due to the fact that the reaction is prematurely terminated at acid no. 1.0, which is equal to 0.8% DOP yield. This data further verifies the yield to be quantitative or potentially so by present TOP technology.

The possibility of developing "low" temperature processes was investigated using tetrabutyl titanate (TBT), sulfuric acid and p-toluene sulfonic acid (PTSA) as catalysts. Maximum reaction temperatures of 175°, 185°, 200°C were chosen since they correspond to operating temperatures from steam at 150, 225, 300 psig, respectively. The use of a "foreign" azeotroping agent (other than excess 2-ethylhexanol) or vacuum was also examined. Results of these trials are summarized in the following table.

DOP - "LOW" TEMPERATURE PREPARATIONS

- 3 -

Expt. No.	Maximum Reaction Temp., °C	Catalyst, %	Excess Alcohol %	Reaction Time, Hrs.	Final Acid No.	FINAL PRODUCT			Carrier	Remarks
						APHA	Acid No.	Yield, % ⁽¹⁾		
(1) D-071-9,10	245	0.03 TBT	22	2.8	0.8	5	0.1	98.2	Excess 2-FH	Control-Atm. Press.
(2) D-071-12	185	0.075 TBT	22	4.0	1.1	5	0.07	97.8	Vacuum	185°C-Vac. Strip (25 mm)
(3) D-071-13	200	0.075 TBT	22	3.0	0.4	5	0.01	98.0	Vacuum	195°C-Vac. Strip (25 mm)
(4) D-071-11	200	0.10 TBT	22	2.5	0.6	10	0.12	98.1	Vacuum	200°C-Vac. Strip (25 mm)
(5) D-071-16	175	0.25 TBT	22	3.0	0.9	5	0.02	97.8	Vacuum	175°C-Vac. Strip (25 mm)
(6) D-073-13	175	0.50H ₂ SO ₄	20	1.5	2.8	20	0.03	96.0 ⁽²⁾	Cyclohexane (5%)	Neutralized before Strip.-20°C-Vac.
(7) D-073-15	185	0.50H ₂ SO ₄	20	1.5	2.6	25	0.04	97.2	Vacuum	Neutralized before Strip.-200°C-Vac.
(8) D-073-11	175	1.24H ₂ SO ₄	25	1.0	6.8	-	-	-	Cyclohexane (5%)	200°C Strip-decomposed in pot.
(9) D-057-43	190	0.10 PTSA	10	2.5	6.1	> 50	0.03	94.6	Toluene (8%)	Pure PTSA-H ₂ O-240°C Strip
(10) D-057-45	180	0.25 PTSA	10	3.0	1.5	10	0.03	98.1	Toluene (8%)	Pract. PTSA-H ₂ O-185°C Vac.-Strip
(11) D-057-46	180	0.25 PTSA	22	2.5	1.6	> 50	0.03	97.8	Vacuum	Pract. PTSA-H ₂ O-175°C Vac.-Strip
(12) D-057-47	180	0.40 PTSA	10	2.5	1.6	5	0.03	98.3	Toluene (8%)	Pract. PTSA-H ₂ O-175°C Vac.-Strip
(13) D-057-44	175	0.50 PTSA	10	2.0	1.9	10	0.03	98.1	Toluene (8%)	Pure PTSA-H ₂ O-180°C Vac.-Strip

(1) Unless otherwise indicated, all yields are filtered yields. In the laboratory, losses due to drainage and hold-up in filter cake was approximately 1.1%.

(2) Yield before filtration; inadvertent losses.

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These data demonstrate "low" temperature processes are feasible with all three catalyst systems evaluated. Functional evaluation of selected DOP candidates from the preceding table, for electrical properties showed all processes (TBT, H_2SO_4 , PTSA) to produce acceptable material. The only marginal products were D-073-17A and D-073-17B from the H_2SO_4 catalyzed process which were neutralized with sodium carbonate instead of sodium hydroxide. D-073-17B was given an additional high temperature ($120-130^\circ C$ - 2 hours) treatment to destroy dialkyl sulfates if present. This carbonate treatment on a PTSA catalyzed run (D-057-49-1 and D-057-48-1) showed that special treatment to destroy di-organo sulfates was not necessary. (see table attached).

As a result of the favorable synthetic and functional results on the DOP procedures studied, factorial designs on the PTSA and H_2SO_4 catalyzed systems were set-up and implemented. The variables in these studies involved catalyst levels, maximum reaction temperature and whether an azeotroping agent (other than excess 2-ethylhexanol) was required. Results of these runs showed that in the H_2SO_4 catalyzed runs, optimum reaction time was obtained using 0.25% H_2SO_4 at $175^\circ C$ (D-050-181). The resulting APHA color of this product was 10. The best result from the PTSA factorial design (D-057-54) was obtained with 0.75% PTSA at $175^\circ C$ and the DOP had an APHA color of <10. Previous results show that satisfactory results can be obtained with 0.5% PTSA at $175^\circ C$. In both cases an azeotroping agent (other than 2-ethylhexanol) was used. When run in vacuum significant color developed. These runs are summarized in the following tables. Functional evaluations (electricals) are in progress for the best samples.

In addition, other catalyzed systems such as $KHSO_4$ and aqueous H_2SO_4 , as well as varying levels of TBT at $175^\circ C$ are being screened for DOP production. (see tables attached).

Based on this work confirming runs to verify the detrimental effect of vacuum were made. Work will now concentrate on determining the recyclability of recovered alcohol and corrosion effects on 316 stainless steel of the optimum sulfuric acid and para-toluene-sulfuric acid catalyzed systems at $175^\circ C$ with entraining agents and at catalyst levels of 0.25 and 0.50%, respectively.

Emulsion problems originally encountered in continuous sodium hydroxide wash experiments with DTDP (plant batch) were overcome by raising the settling tank temperature from $25^\circ C$ to between $50-90^\circ C$. Functional evaluations showed the DTDP so obtained to be of acceptable "electrical" quality. The feasibility of continuous neutralization was thereby established.

FUNCTIONAL EVALUATION - "ELECTRICALS" OF EXPERIMENTAL DOP'S

Sample No.	VRX10 ¹³ OHM-CMS, 23°C. (1)	VRX10 ¹³ OHM-CMS, 50°C.	Catalyst - Azeotrop. Agent	APHA Color	Remarks
(1) Plant Lot 27336	30.73 ⁽²⁾	0.55 ⁽²⁾	0.03% TBT - Excess 2-EH.	Spec. <20	Standard plant run . Atm. Press. - 255°C.
(2) D-071-10	19.81	0.20	0.03% TBT - Excess 2-EH.	5	Laboratory version of standard plant run.
(3) D-071-11	20.12	0.32	0.10% TBT - Vacuum	10	Yellow in sheet.
(4) D-073-13	21.66	0.32	H ₂ SO ₄ - Cyclohexane	20	Sodium hydroxide neutralizatio
(5) D-073-15	26.41	0.52	H ₂ SO ₄ - Vacuum	25	Sodium hydroxide neutralizatio
(6) D-073-17A	8.99	0.09	H ₂ SO ₄ - Vacuum	50	Prep. as for D-073-15 ex- cept Na ₂ CO ₃ neut.
(7) D-073-17B	11.55	0.12	H ₂ SO ₄ - Vacuum	50	As for D-073-17A except 2nd Na ₂ CO ₃ treat. - 130°C.
(8) D-057-47	16.32	0.17	PTSA Toluene	5	-
(9) D-057-49	17.57	0.16	PTSA Toluene	-	Prep. as for D-057-47.
(10) D-057-48	19.73	0.19	PTSA Toluene	-	As for D-057-49 except Na ₂ CO ₃ treat. - 130°C after NaOH neut.

(1) Using WE-644 - Specification calls for 5×10^{13} ohm-cms, minimum at 23°C.

(2) A previous plant batch (lot #27705) had values of : 26.10×10^{13} (23°C) and 0.4241013 (50°C).

COLORTITE 011460

FACTORIAL DESIGN - SULFURIC ACID CATALYZED PREPARATION OF DOP

Expt. No.	REACTION						FINAL PRODUCT				FUNCTIONAL EVAL.		
	Maximum Temp., °C	Catalyst Level, %	Carrier	Excess Alcohol, %	Reaction Time, Hrs.	Acid No.	Filtered ⁽¹⁾ Yield, %	Color APHA	Acid No.	Assay, %	VRX10 ¹³ OHM-CM, 23°C	VRX10 ¹³ OHM-CM, 50°C	
(1) D-073-35	175	0.10	6% Cyclohex.	10	3.0	1.4	97.3	10	0.08	99.9			
(2) D-050-181	175	0.25	6% Cyclohex.	10	1.50	2.6	99.3	10	<0.03	99.7			
(3) D-073-29	150	0.10	6% Cyclohex.	10	4.25	1.4	98.0	5	0.05	99.9			
(4) D-073-37	150	0.25	6% Cyclohex.	10	2.50	2.2	97.4	20	0.10	99.9			
(5) D-073-39	175	0.10	Vacuum	22	1.50	1.3	97.3	20	0.03	99.7			
(6) D-050-175	175	0.25	Vacuum	22	1.25 ⁽²⁾	2.3	98.3	50	0.01	99.6			
(7) D-050-179	150	0.10	Vacuum	22	2.5	2.5	97.8	250	<0.03	99.2			
(8) D-050-177	150	0.25	Vacuum	22	1.75	2.5	98.3	100	<0.03	99.5			

(1) Average yield loss due to filtration and drainage est. at 1.5%

(2) After one hour, acid no. was 2.7 indicating reaction essentially finished since acid no. for 0.25% H₂SO₄ = 2.86. Reaction allowed to continue for an additional 0.75 hour, where an acid no. of 2.3 was obtained.

COLORITE 011461

FACTORIAL DESIGN - PTSA CATALYZED PREPARATION OF DOP

Expt. No.	REACTION						FINAL PRODUCT				FUNCTIONAL EVAL.	
	Maximum Temp., °C	Catalyst Level, %	Carrier	Excess Alcohol, %	Reaction Time, Hrs.	Acid No.	Filtered ⁽¹⁾ Yield, % (on PAN)	Color APHA	Acid No.	Assay, %	VRX10 ¹³ OHM-CM, 23°C	VRX10 ¹³ OHM-CM, 50°C
(1) D-057-55	175	0.25	8% Toluene	10	2.75	1.70	97.9	<5	0.02	99.7		
(2) D-057-54	175	0.75	8% Toluene	10	1.75	2.41	98.7	<10	0.02	99.7		
(3) E-022-102	150	0.25	8% Toluene	10	6.17	1.75	97.0	20	0.03	99.5		
(4) E-022-100	150	0.75	8% Toluene	10	3.75	2.63	97.8	10	0.03	99.6		
(5) D-057-52	175	0.25	Vacuum	22	2.50	1.51	98.3	40	0.03	99.5		
(6) E-022-103	175	0.75	Vacuum	22	1.25	2.56	98.3	>500	0.08	99.7		
(7) D-057-53	150	0.25	Vacuum	22	5.00	1.51	98.4	150	0.02	99.8		
(8) E-022-104	150	0.75	Vacuum	22	2.25	2.68	97.9	300	0.20	99.5		

(1) Average yield loss due to filtration and drainage - 1.11%.

MONTHLY SUMMARY - OCTOBER 1974

A major effort is continuing to try to understand and improve the treatment of waste waters at the Chestertown plant to eliminate pollution. Results to date showed that caustic washes, from Nuoplaz 1538 production, after adjustment to pH 4 to 4.5 and filtration of the resulting stannic oxide and benzoic acid, reduced the total COD load on the Chestertown pollution control system to 40% of its original requirement. A technique to treat phthalate ester washes by adjusting the alkaline washes to pH 1.5 was also developed. The filtrate, after filtration of the phthalic acid showed a COD value with only 10% of the original loading. Because this latter approach would require special materials of construction and neutralization prior to adding to the equalization basin, a tentative program calling for an economic evaluation and implementation of benzoate treatment is being considered first. In an effort to improve efficiency, a study is also underway to investigate the microbiological profile of the waste treatment system at Chestertown. A weekly program of microbiological analyses of the equalization basin and the three aerated digestion ponds has been started. Initial studies have shown unusual variations in numbers and species of microorganisms between the three ponds receiving the same effluent and nutrient feed. To monitor "in-house" studies COD and BOD capabilities are being set up in the Biocontrol Laboratory. In addition, seasonal effects on microorganism population distribution are being followed.

I. Huppert (msk)

I. Huppert

IH/msk

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

TO Dr. R. T. Gottesman AT Piscataway DATE October 28, 1974
FROM Mr. H. R. Johnson AT Piscataway COPY TO
SUBJECT MONTHLY SUMMARY - OCTOBER 1974
ANALYTICAL SERVICES

Methods Development (I. Wakeman)

Chlorotoluenes: The gas chromatographic analysis of crude chlorotoluene mixtures has been revised (GLC 83.0) to permit the determination of the chloroform and carbon tetrachloride contaminants present in the recovered toluene.

Chloride: A colorimetric procedure based on mercuric thiocyanate has been set up to determine trace quantities of chloride ion in aqueous solution (M-000.98). The procedure can detect 0.2 microgram of chloride ion per milliliter and is suitable for air quality monitoring for hydrochloric acid.

H. R. Johnson
H. R. Johnson

HRJ/ec

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COLORITE 011464

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

TO Dr. R. T. Gottesman AT Piscataway DATE October 25, 1974
 FROM Mr. R. S. Miller AT Piscataway COPY TO
 SUBJECT MONTHLY REPORT - OCTOBER 1974

NOTICE

COMPLIANCE

These materials are
 used in the
 production of
 the product
 and for the
 purpose of

PLASTICS INDUSTRYPVC Resins

1. VCM Compliance - VCM Removal From Dispersion Resin (J. C. Fisher
 D. Goodman, M. Koral, P.J. Patel, M. K. Rosen)

* The use of an air or nitrogen sparge plus vacuum demonstrated very promising results in the laboratory with respect to VCM removal from dispersion resin. Testing a plant 1730 latex, a 140 mm Hg vacuum plus air sparge at 0.22 ft³/hr/(ca. 0.8 cfh air/gal. latex) eliminated over 90% of residual VCM in the first two hours and upwards of 98% of residual VCM in five hours at 45°C. Substitution of nitrogen for air as the sparge gas affords similar results. Utilization of higher temperatures such as 50°C further shortens the VCM removal time. Filter cake characteristics at 45° and 50°C are satisfactory. When a temperature of 55°C was employed, additional improvements in time occurred, but the filter cake behavior may be borderline. The results are outlined below:

<u>Time (Hrs)</u>	<u>VCM (ppm/Latex Solids)</u>		
	<u>45°C</u>	<u>50°C</u>	<u>55°C</u>
0	20,575	15,475	13,103
2	1,570	1,043	295
3	-----	-----	35
4	-----	153	N.D.
5	128	40	-----
21.5	N.D.	-----	-----

The use of a sintered glass disc (to give better gas dispersion than with an open end dip tube) for nitrogen sparging gave a slightly faster removal of VCM. Flushing only the head space above the latex with nitrogen during stripping gave much slower VCM removal rates. The indication is that bubbling the sparge gas through the latex gives a greater gas/liquid interface, which is a critical parameter to effect transver of MVC from the latex.

Pilot Plant stripping studies have verified the laboratory trends. Use of higher agitation rates (65, 120 and 220 rpm) to improve latex turnover in the reactor and thus increase latex/gas surface exposure gave improved MVC removal rates. The use of a nitrogen sparge showed a further significant improvement in MVC removal rates. Foam problems limited the use of the nitrogen sparge to .19 cfh nitrogen/gal. latex. The results of the Pilot Plant runs using 1740 latex from Burlington are:

<u>Time(hrs)</u>	<u>VCM (ppm/latex solids)</u>				
	<u>65 rpm</u> <u>45°C</u>	<u>120 rpm</u> <u>45°C</u>	<u>120 rpm</u> <u>50°C</u>	<u>220 rpm</u> <u>50°C</u>	<u>N₂ Sparge</u> <u>120² rpm/45°C</u>
5	5100	3000	2500	800	2200
10	4400	2000	1400	490	500
19	3700	1200	700	260	68
24	--	910	510	200	10
36	--	570	--	115	--
42	--	--	--	92	--

Considerable foaming problems were encountered during nitrogen sparging which limited the nitrogen rate. Further trials are planned with equipment changes to permit higher nitrogen rates with foam control.

Additional Pilot Plant studies are in progress using a latex circulation loop around the reactor with nitrogen gas dispersion in the loop. A study of latex turnover during stripping will also be conducted pending purchase of an improved agitator impeller design recommended by Mixco.

Since the increased latex/gas interface required to give improved MVC removal rates has also lead to significantly higher foam levels in the laboratory, pilot plant, and production plant, a program to evaluate improved antifoam agents has been initiated in the laboratory.

2. Carbon Adsorption (J.C.Fisher, P.J.Patel)

An extended run of coconut base adsorptive carbon (PCB, 4 x 10 mesh) from Calgon has shown significantly better performance than the bituminous coal base carbon (BPL, 4 x 10 mesh) previously evaluated. Both of these carbons will maintain a relatively high adsorptive capacity for a certain number of cycles, followed by a fall-off in capacity. The BPL evaluations were terminated shortly after the capacity fall-off. The PCB evaluation showed the bed capacity leveled-off at a lower plateau after the fall-off and was then continued for a total of 82 cycles without a further decrease in bed capacity.

<u>Carbon Type</u>	<u>Bed Capacity(% MVC)</u>		<u>Fall-off Point (cycles)</u>	<u>Total Cycles</u>
	<u>Initial Plateau</u>	<u>Second Plateau</u>		
BPL, run 1	21	---	20	27
BPL, run 2	19	---	18	22
PCB	22	12.5	34	82

A verification of the PCB performance is planned with a different lot of this carbon type. An evaluation of Witco 256 (4 x 10) (a petroleum base carbon) has been initiated.

Scanning electron microscope examination of virgin and exhausted BPL carbon showed an increase in pore size and a less complex surface structure on the exhausted carbon. Analysis of carbon by Iodine Number, as a measure of surface area, has shown some correlation with bed capacity (12.5% reduction in I_2 No. $\approx$ 50% reduction in bed capacity).

A run is also in progress to evaluate the effect of repeated steam and nitrogen regeneration cycles (no MVC adsorption) on the surface adsorption of the carbon as measured by I_2 No.

Material balance studies are in progress to establish the amount of bed(fines) carry-over and the efficiency of bed regeneration.

3. Copolymer Stripping -Solvent Assist (J.C.Fisher, M.K.Rosen)

Studies are in progress in the Pilot Plant to study the effect of various parameters which could influence the rate and efficiency of MVC removal from copolymer slurries. These studies employ a post-polymerization addition of vinyl acetate to function as a solvent to assist in the removal of MVC from the copolymer resin particles. The effect of agitation, temperature, nitrogen sparging and reflux are being evaluated. Problems have been encountered during recovery

in the Pilot Plant because of a gel-like build-up in the recovery system. These evaluations are still in progress.

4. TCE Removal (M.L.Feldman, D.Goodman)

Preliminary laboratory results indicate that the residual trichloroethylene in 10.5 R resin can be reduced by about one-half (3400 ppm to 1600 ppm residual TCE) by the vinyl acetate add-back treatment recommended for treatment of suspension copolymer slurry. Exhaustive methanol extraction of dry 10.5 R resin in the lab demonstrated a reduction to 590 ppm from a starting value of 1530 ppm residual TCE. A search for other FDA chain transfer agents is under way.

5. Small Sample Processing (J.C.Fisher, A.C.Shah)

There has been no acceptable method of processing small dispersion latex samples to permit functional testing of the effect of laboratory additions (stabilizers, antifoams, etc.) required for more rigorous stripping of dispersion latex. The most difficult problem has been to adequately grind small samples of lab dried resin to permit preparation of plastisol pastes suitable for rheological and film properties.

Various fluid-energy mills are now being evaluated with a 4-inch Micronizer (Strutevant Co.) showing excellent results, grinding as little as 600 grams of sample with product recoveries of better than 82%. Pastes prepared from these ground samples show reasonable paste viscosities and low grain films comparable to production or pilot plant Mikro-atomized resins. One additional grinding trial is scheduled prior to an equipment purchase recommendation.

6. Color Stabilization (M.L.Feldman, D.Goodman, R.J.Stanaback)

Thermal treatment of homopolymer to remove residual VCM has had the adverse side effect of causing the resins to darken in color. The color development occurs in slurries heated from 70 - 100°C, with the rate of color development increasing at the higher temperatures. Using the plant homopolymer stripping conditions, 95 - 100°C for 1/2 hour, the laboratory data has shown that promptly cooling and maintaining the temperature to 70°C provides minimal color changes in Tenneco 105 R resin. The relative viscosity of heat treated and dissolved resin was examined. There were no changes in molecular weight indicating that there is little or no crosslinking or chain scission accompanying the color formation.

There has been an unexpected result in the treatment of homopolymer slurry with oxidants. Use of hydrogen peroxide or oxygen sparged through the slurry significantly reduces color formation versus similar treatment in a nitrogen atmosphere. Attempts are being made to understand and exploit this effect. Combinations of hydrogen peroxide and epoxidized soybean oil have been tested. The best results

to date (minimum color development) have been with 0.25phr H_2O_2 and 1.0 phr Nuoplaz 849.

Bottle polymerization of VCM, using a plant 10.5R formula, was employed to evaluate the stabilizing effect of Epoxol 9-5. Epoxol 9-5 was studied at zero, normal, and twice normal levels (0, 0.45, and 0.90 respectively). Relative viscosity measurements of polymer made with zero and with 0.45 phm Epoxol 9-5 demonstrate no significant molecular weight differences. However, the result with twice the normal amount of Epoxol 9-5 showed a considerable reduction in molecular weight. This suggests that the epoxy compound is acting either as a polymerization inhibitor or as a chain transfer agent. If the latter proves to be the case, Epoxol 9-5 or other compounds containing the epoxide structure may be replacements for TCE as a chain transfer agent. These results are being repeated to verify the results.

7. OC Coating (M.L.Feldman, D.Goodman)

Several domestic materials were screened for solubility and wettability characteristics relative to Shin-Etsu coating material. A water soluble candidate did not coat steel and should not be considered. A program has been set up to test whether the Shin-Etsu. OC coating system is effective in preventing wall scale in the dispersion resin towers. Coated stainless steel coupons will be tested against uncoated controls.

Additives

1. Supercryl (D. Goodman, M. Koral)

In order to trim raw material costs, the previously approved Duponol WAQE, was substituted into the Supercryl formulas replacing the higher priced Avirol POL-1-N. A set of four plant runs using Maprofix LCP (a still lower cost sodium lauryl sulfate from Onyx at 21.8¢/lb.) was made in a program of total solids increases from 35 to 40%. The polymerization processes over this range of solids with the Maprofix LCP were satisfactory. Full functional testing of these batches is pending.

Supercryl 100H was characterized at higher total solids. The use of the rotating flash evaporator proved to be too foamy so that the latex was concentrated by simple water evaporation. As demonstrated in the outline below, there is little change in latex viscosity at 44% solids, but at 52% solids the viscosity may pose a spray drying problem.

Total Solids %	Brookfield Viscosity, LVT #2 Spindle, 30°C, 30RPM (cps)	Surface Tension 25°C, (dyne/cm)
38.5	13	45.8
44.0	75	47.5
52.0	1,250	52.4

R. S. Miller jcs

R. S. Miller