

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

TO Distribution List AT DATE August 31, 1973
FROM Dr. R. T. Gottesman AT Piscataway COPY TO
SUBJECT MONTHLY SUMMARY - AUGUST 1973

Attached are copies of the Monthly Summary reports for August 1973 covering the activities of the Piscataway laboratories and associated pilot plants at Piscataway, Flemington and Reading.

R. T. Gottesman
R. T. Gottesman

RTG/ec

Attachment

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MONTHLY SUMMARY - AUGUST 1973

August 31, 1973

* B. R-125 Pigment Dispersant (G. Kagan, M. Landau)

Three pilot plant lots of R-125 (035-131,136,141) have demonstrated acceptable functional performance vs. Tamol 731 in PVA and acrylic paints. A product data sheet is being prepared and material is available for customer sampling and introduction at the Paint Show in November. The pilot plant process proposal was issued and cooperative work with Process Engineering to initiate manufacture at Garfield is underway.

C. Thickeners (G. A. Trautenberg, G. Tirpak)

A series of new cellulosic thickeners (XD-7630) from Dow Chemical reputed to be enzyme resistant have been evaluated and found to be quite enzyme resistant. Compared to the standard cellulosic, Natrosol (HEC), the experimental products were about six to seven times more resistant to microbial degradation. Based on this data, these experimental thickeners would not need biocidal protection, although Dow is recommending the use of an antimicrobial.

II. LUBRICANTS

* A. Automotive Crankcase Lubricants (M. Knapp, R. Smith)

A comprehensive program for the development and testing of a synthetic multigrade (10W-40) crankcase oil has been developed through in-house experience and extensive contacts with the major automobile manufacturers. The initial efforts involve optimization of our current crankcase oil, Anderol 472.

* B. Rotary Combustion Engine (RCE) Lubricant (M. Knapp, R. Smith)

Walker Manufacturing tested Anderol 472 in an RCE-powered car for 20,000 miles and found that this synthetic lubricant produced slightly less wear and lower emissions, but also resulted in more severe engine deposits.

Walker has agreed, at recent meetings with Tenneco R&D personnel, to conduct additional tests in their dynamometer-mounted RCE starting the first week in September. Initial testing will be carried out on a recently-developed low ash shear stable version of Anderol 456, which was chosen as a starting point because it will meet the viscosity requirements for a Dexron II type fluid (which is the General Motors' requirement for this application).

F. Buono
F. Buono

FB/amp

COLORITE 011708

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

TO Dr. R. T. Gottesman AT Piscataway DATE August 31, 1973
FROM Dr. A. J. Deinet AT Piscataway COPY TO
SUBJECT MONTHLY SUMMARY - AUGUST 1973

I. CHEMICALS/SPECIALTIES* 1. p-Hydroxybenzoic Acid (M. Reale, R. J. Stanaback)

A laboratory fusion carried out in all-glass equipment in which dipotassium salicylate was isomerized to para acid utilizing time-temperature cycles (2 hrs. at 275°C.) as recently carried out at Bethlehem Corporation gave a 91% yield of quality product. Although some 4-hydroxyisophthalic acid is formed as by-product, final acidification to pH 3 avoids the coprecipitation of this impurity with the para acid. Since the feasibility of good quality and yield has now been demonstrated, confirming runs in the laboratory autoclave are underway to determine if the same can be achieved in steel equipment prior to a final trial at Bethlehem.

A work-up procedure to be compatible with the equipment capabilities at the Garfield salicylic acid plant is being developed. The use of hydrochloric acid for partial neutralization of the reactor liquor prior to decolorization with carbon in the steel column at Garfield is deemed unsuitable because of chloride ion corrosion. While the use of sulfuric acid was found to be unsuitable due to the poor solubility of potassium sulfate, carbon dioxide was shown to be entirely suitable for the partial neutralization and is already available in the plant. Bethlehem reactor liquor so treated, followed by decolorization and final precipitation with hydrochloric acid (rubber lined equipment available for this stage) gave an excellent quality product.

2. Dinitro-p-chlorobenzotrifluoride (E. DiBella, R. J. Stanaback)

A revised nitration technique has been developed which minimizes the extensive solvolysis encountered when p-chlorobenzotrifluoride is nitrated in the presence of fuming sulfuric acid. It was found that by initially carrying out a mononitration by use of strong nitric acid alone followed by the use of mixed acid to achieve the dinitro stage a 74% yield of product assaying 98% was achieved. This technique will be utilized to prepared a ten pound sample for BASF.

COLORITE 011709

II. PLASTICS INDUSTRY

1. Flame Retardants (E. DiBella, R. J. Stanaback)

Samples of our flame-retardant emulsifiable concentrate composition based on T-2,3-P/TOF with Tergitol P-28 and NP-27 emulsifiers have been forwarded to the Masonite Corporation and the J. F. Donoval Corporation for evaluation.

The evaluation of hexabromobutene-2 (HBB) is continuing on a high priority basis. An emulsion system in which HBB is dispersed in N-methylpyrrolidone/toluene/water with Tween-20 was developed and utilized in padding application to various fabric types. Preliminary results indicated flame-retardancy was imparted to acetate and wool. In the plastics area, HBB has been successfully incorporated into both high and low density polyethylene and polypropylene. Incorporation into ABS and polystyrene is in progress and then flammability tests on the entire set of plastics materials will be carried out for use in support of a patent application.

The use of diphenyl isodecyl phosphite in conjunction with T-2,3-P has been evaluated for anti-scorch characteristics in polyurethane foam. Although some reduction (similar to that found when using Weston's DHOP) was achieved the results were not comparable to those with Fyrol-2. The basis for this trial was a recent French Patent (#2,143,488) which claimed the use of such phosphites for imparting better stability to T-2,3-P.

Based upon an earlier observation that boric acid imparts some smoke suppression to PVC, a sample of dibutyl-2,6-di-t-butyl-4-methyl phenylborate was evaluated in PVC at 15 PHR as a partial replacement for DOP. Only slight flame-retardancy was imparted (LOI of 23.8 vs. 22.6 for control) with no apparent smoke suppression.

III. DYES AND INTERMEDIATES

A. Dyes

1. Liquid Paper Dyes (M. Feldman, H. Gosch, L. Port, N. Conzo)

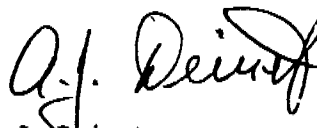
- * Based on analytical results on competitive liquid paper dyes which have shown them to be essentially aqueous formulations emphasis has been placed on developing this type of formulation. Aqueous solutions of 35% strength have been achieved for both Blue 3RLP and Red NAS by conversion to the triethanolamine salts. These sales have enhanced water solubility and were readily prepared by heating the corresponding ammonium salts with triethanolamine directly in a water system. Stability thus far (2 weeks) is excellent and the systems have a desirably low viscosity. Dyeing tests on paper are to be carried out and pilot plant verification of these processes will follow. Attempts to isolate these two products as the free acids prior to conversion to the triethanolamine salts were unsuccessful.

The triethanolamine salt of Bond Yellow G was also prepared, but was found to have insufficient water solubility to achieve 35% strength. Tenneco Bond Yellow G is a phosgenated product, whereas DuPont's "Pontamine" Bond Yellow G has now been identified by the Analytical Department as being a triethanolamine salt of 2-(p-aminophenyl)-6-methylbenzothiazole-7-sulfonic acid which has been diazotized and coupled with a pyrazolone. Synthesis has been initiated, therefore, on related structures based on p-aminophenyl benzothiazoles coupled with other economical components.

2. Yellow 3G (L. Schinasi)

Trial laboratory runs have shown the feasibility of a solventless process for the preparation of this material which will enable manufacture in non-explosion proof equipment. The procedure consists of a gradual addition of quinoline carboxylic acid (QCA) to molten phthalic anhydride with a short hold at 210°C. for reaction completion, drowning the melt in aqueous caustic soda, treating the solution with activated carbon and finally springing Yellow 3G with sulfuric acid. Using a QCA to phthalic anhydride ratio of 1.0:4.25, an 85% yield of product was obtained which showed acceptable dyeings and strength.

A tentative Laboratory Process Proposal will be issued shortly prior to pilot plant verification.


A. J. Deinet

AJD/srf

TO Dr. R. T. Gottesman AT Piscataway DATE August 31, 1973
FROM Mr. I. Huppert AT Piscataway COPY TO
SUBJECT MONTHLY SUMMARY - CHEMICAL DEVELOPMENT
AUGUST 1973

I. CHEMICALS/SPECIALTIESA. Phosphates1. Dibromopropanol (H. Gould, S. Cinco)

Additional work on the continuous "heterogeneous" process for preparing DBP dealt with the use of heptane and mineral spirits (S-434) as the initial reaction solvent in place of hexane. Yields for both runs were 86+% with G.C. purities of 99.9% for the batch distilled product. Further work on reaction systems is pending until Process Engineering can economically evaluate the continuous reaction system.

The soda ash treated crude DBP was continuously distilled through a wiped film evaporator under the conditions described below:

Run No.	Temp. °C	Pressure (mm Hg)	Feed Rate (ml/Hour)	Yield (%)	DBP Assay* (%)	Moisture (%)
1	185-210	30	100	85.6	97.9	0.49
2	170-200	15	110	87.7	97.6	0.37
3*	175-220	27-30	200	80.7	99.3	0.73

*Only one-half the volume of 8% soda ash solution was used in this case as compared to Runs No. 1 and 2 to remove acidity.

In all runs, the main cut of DBP did not meet the required maximum moisture specification of 0.05%. Emphasis on continuous DBP distillation will be maintained until a suitable system is found.

*

A sample of Dow Chemical DBP (1440495) was converted in the laboratory to T-2,3-P via the MgO catalyzed plant process in a yield of 85.7% (on POCl_3) and 82.9% (on DBP). This source of DBP is recommended for T-2,3-P manufacture provided Dow can continuously meet our moisture specification of 0.05%.

2. T-2,3-P (J. P. Sandstedt, S. Cinco)

A demonstration batch of T-2,3-P using predistilled DBP and the $AlCl_3$ catalyst was made for Fords Plant personnel in the Piscataway Pilot Plant. Yields were 84.0% on DBP and 86.4% on $POCl_3$. Quality was acceptable. Arrangements are being made to reintroduce this process at Fords sometime in September.

Pilot Plant HCl and water washed crude T-2,3-P was worked up in the laboratory to evaluate the effect of Glucoquest AC as a sequestering agent to remove traces of aluminum. Results indicate that a 5% concentration in the soda ash wash facilitates separation and reduces emulsification tendencies. The resulting product met all LV specifications. This system will be held in reserve in case it is required in plant operations.

Corrosion studies were conducted on Celanese Cellulose Acetate Dope containing Tenneco LV Grade T-2,3-P (Fords No. 73-42) in the presence of "Black" Iron. After 28 days at 58°C, the iron was insignificantly affected (corrosion rate = 2.7 mils/year). The Dope formulation was unchanged as determined by color and consistency. Similar studies to aid Marketing are in progress using galvanized iron.

B. Catalysts (A. Fischer)

A quart sample of R-465, a 6% iron (III) 2-ethylhexanoate was prepared and submitted to United Foam Corporation as a match for competitive Cincinnati Milacron Iron 6%. The properties of R-465 are compared below with those of the competitive product.

	<u>R-465</u>	<u>C.M. Iron 6%</u>
Total Iron, %	6.0	6.0
Iron (II), %	0.18	0.6
Non-Volatiles, %	49.8	42.5
Viscosity (Gardner-Holdt)	A-3	A-1
Specific Gravity (77°F)	0.903	0.897

C. Cal/Ink Vehicles (O. Fuchs)

Two processes for the preparation of two Cal/Ink vehicle compositions are presently being evaluated in the Process Development Laboratory. One, General Purpose Dispersion Liquid, has been prepared on the first attempt according to the plant procedure supplied by Cal/Ink and meets their specifications for acid number, color, viscosity and hydroxyl number. This material will be sent to Cal Ink for functional evaluation. The other, Uni-Cal Vehicle, was still being worked on due to processing difficulties (which may be related to scale-down to glassware) when using the Cal Ink procedure.

II. PLASTICS INDUSTRYA. Additives1. Stearates (S. Hoch)

Two pound samples of Experimental Water Dispersible DLG-20 (R-439) were prepared and sent to each of the following for evaluation:

Inmont Corp., Morgantown, N. C.
Mobil Chemical, High Point, N.C.
Lilly Co., High Point, N. C.
Reliance Universal, High Point, N. C.
Warlick Paint Co., Statesville, N. C.

Two pound samples of Experimental Water Dispersible Stearates: S-1058 (zinc stearate); S-1244 (calcium stearate); and USP magnesium stearate; each containing 5% Aerosol OT-B, have been prepared and are available for customer sampling. This initial approach is the first phase of our overall program to develop a line of water dispersible stearates.

2. Plasticizers (I. H. Cooper)

Evaluation of the Artisan falling film evaporator has been hampered by mechanical problems. The latest was determined to be the supply of an improperly designed unit for vacuum steam stripping by Artisan. Results to date show the unit to be overdesigned for continuous alcohol removal from DOP. Work is attempting to establish the maximum throughput through the thirteen stages that can be achieved. Best results to date are 125.0 pounds per hour of DOP and 3.5 pounds per hour of steam yielding DOP with less than 0.05% alcohol (specification value) compared to Artisan's stated maximum of 60 pounds per hour to achieve 0.01% for this unit. Operating conditions are 165°C and 5 mm Hg. Emphasis will be changed to evaluating simpler units to accomplish the same job.

IV. COLORSA. Intermediates1. Oxylyl Para-Phenylene Diamine (G. F. McClain)

An oxalylation batch was run in the Pilot Plant to provide presscake for pilot studies of the reduction step and recycling of the unreacted iron. The oxalylation batch ran without incident and a wet weight yield (1057 lbs.) was obtained which paralleled the lab process (906 lbs.). Three reduction batches have been run. The first had to be clarified by filtration because of problems encountered in trying to decant. Some product was lost and the yield was only 84%

of expected, however 20% of the initial iron charge was recovered and used in the next batch. The second batch returned 101% of expected yield and the procedure was improved. A recovery of 30% of the iron which was used in the third batch was made. The third batch has been run and again the decant was successful, however, yield data is not yet available. It is planned to run three more reduction batches which will consume all of the available OPNA and make it possible to calculate yield back to PNA. This should complete Pilot Plant work. Reading Plant personnel have been invited to watch the Pilot Plant reduction and decantation for their comments on the mechanics of the system.

2. p-Nitroaniline (E. Reinhold)

A portion of the PNAA (p-nitroacetanilide) presscake from a Pilot Plant PAA (p-aminoacetanilide) batch was hydrolyzed with liquid caustic by a procedure described in Fierz-David to PNA (p-nitroaniline). The hydrolysis was simple and the yield was 72% of theory. Fierz-David projects 72.4% of theory. The PNA derived was converted in the laboratory to Black 10BR by a standard Reading process and the resultant dyestuff was slightly red in shade (this is the shade normally produced by this process in the plant). The yield on a lab basis was greater than expected by 20%. After economic evaluation of this procedure, it will be verified in the Reading Pilot Plant.

B. Dyes

1. Direct Fast Blue 3RLP Paper Liquid (G. F. McClain)

Presscake from Pilot Plant ammonium chloride precipitated Plant Blue D Crude slurry was converted into liquid. The conversion was made in an enamel kettle with a horseshoe agitator which closely resembles the kettle in the Reading Plant planned for the manufacture of paper liquids. During the processing a considerable amount of ammonia vapor was evolved so that the plant design will require an adequate scrubber. No problems were encountered in the processing and the resultant liquid is very fluid at 40% strength versus dry powder Code 2323. Previous formulations at 35% of the same code were often quite viscous.

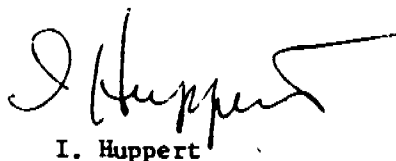
2. Yellow FWL (R. Reinhold)

Plant Batches 22, 23 and 24 consumed 2646 pounds of Yellow FWL base which at standard yield should have produced 7938 pounds of color. Actual color yield is 8512 pounds or 107.2% of standard. The shades were trace red, trace red and very slightly green respectively, all other tests were acceptable. This process is now ready to be run in the Reading Plant when installation of the new scrubbing equipment is complete. A Pilot Plant Process Proposal will be written.

C. Miscellaneous1. Dyeing Assistant (S. Cinco, R. Caruso)

Two thousand pounds of DUROCAR P-73 were prepared in the Piscataway Pilot Plant from Fords chlorinated o-chlorotoluene and are available for customer sampling. Corrosion studies to determine storage vessels and shipping containers gave the following results:

After 7 days at 60°C, carbon steel was corroded on the surface and edges and the DUROCAR 73 was slightly discolored. Heresite was unaffected and did not discolor the DUROCAR 73. At 40°C for 7 days polyethylene and phosphatized steel were unaffected as was the DUROCAR 73. The control as well as tested samples of DUROCAR 73 emulsion at both 40° and 60°C were unstable resulting in a two-phase system. Initial sampling will be in polyethylene gallon, 5 gallon and 15 gallon containers.



I. Huppert

IH/sgf

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

TO Dr. R. T. Gottesman AT Piscataway DATE August 31, 1973
FROM Mr. H. R. Johnson AT Piscataway COPY TO
SUBJECT MONTHLY SUMMARY - AUGUST 1973
ANALYTICAL SERVICES

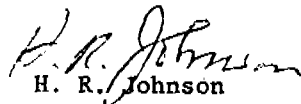
METHODS DEVELOPMENT1. Liquid Chromatography (P. Ketterer, N. Conzo)

The high speed liquid chromatograph equipment has been assembled and is now in operation. A program is underway to set up procedures for the analysis of raw materials and intermediates used in the development and manufacture of dyes. With this technique, for example, the low assay (60-75%) 1-aminoanthraquinone manufactured via the nitration route is readily distinguishable from the high assay (over 90%) product from the sulfonation process. Preliminary runs show that the 1,5- and 1,8-dihydroxyanthraquinones can be separated, and this will serve as the basis for a specific assay method for anthrarufin. Pure reference materials are being prepared using conventional column chromatography techniques.

As detailed analytical data become available from liquid chromatography, it should not be necessary to carry out laborious and time consuming dye syntheses to evaluate raw materials.

2. Nuclear Magnetic Resonance Spectroscopy (N. Conzo)

The Varian A-60 NMR spectrometer has been delivered. Installation and performance check-out is scheduled for completion by September 14.


H. R. Johnson

HRJ/ec

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

TO Dr. R. T. Gottesman AT Piscataway DATE August 31, 1973
 FROM Mr. R. S. Miller AT Piscataway COPY TO
 SUBJECT MONTHLY REPORT - AUGUST 1973

PLASTICS INDUSTRYAdditives* 1. PVC Processing Aids (D. Goodman, M. Koral, M. Spaulding, E. Szabo)

While clear Supercryl 100 has been repeatedly demonstrated in the laboratory and verified in the Polymer Pilot Plant by substituting low levels (0.75, 0.5, 0.375%) of Avirol POL-1-N for the 1% Duponol WAQE in the polymerization process, extended production plant trials during August on this system have failed to confirm the laboratory results.

A plant program was conducted to vary spray drying conditions on the standard Duponol system and the 0.75 and 0.5 phm Avirol systems to determine whether drying temperatures, an apparently significant factor in earlier work, could be adjusted to yield improved clarity product. In both the Fords plant dryer and a spray drying trial at Nichols Engineering there were no clear trends that would indicate that other drying conditions would lead to improved clarity performance. These results are summarized below:

Sample No.	Surfactant	Dried At	Sample Point	Dryer Temp, °F Inlet	Outlet	% Haze
K-120N	---	---	---	---	---	3.1
K-120ND	---	---	---	---	---	2.7
S-100 lot 20132	1% Duponol	Fords	Bagger	---	---	5.1
S-100 lot 20133	1% Duponol	Fords	Bagger	---	---	5.4
D-021-092-A	1% Duponol	Fords	Dryer	350	---	4.8
D-021-092-B	1% Duponol	Fords	Dryer	500	---	5.6
D-021-092-C	1% Duponol	Fords	Screenner	350	---	4.6
D-021-092-D	1% Duponol	Fords	Collector	350	---	3.4
D-021-092-E	1% Duponol	Fords	Collector	500	---	4.1
D-021-092-F	1% Duponol	Fords	Bagger	350	---	5.2
D-021-092-G	1% Duponol	Fords	Bagger	500	---	4.9
D-021-092-AA	1% Duponol	Nichols	Chamber	350	175	5.0
D-021-092-CC	1% Duponol	Nichols	Chamber	502	185	3.1
D-021-092-EE	1% Duponol	Nichols	Chamber	645	190	4.4
D-021-092-M	0.75% Avirol	Fords	Bagger	350	---	5.8
D-021-092-N	0.75% Avirol	Fords	Bagger	500	---	4.4
D-021-092-RR	0.75% Avirol	Nichols	Chamber	350	170	4.8
D-021-092-U	0.5% Avirol	Fords	Bagger	350	---	6.1
D-021-092-V	0.5% Avirol	Fords	Bagger	500	---	5.0
D-021-092-GG	0.5% Avirol	Nichols	Chamber	655	190	7.9
D-021-092-LL	0.5% Avirol	Nichols	Chamber	350	170	4.4

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Since the Avirol batches made during these trials were not acceptable, a second series of plant runs were made at lower Avirol levels and also with a redox catalyst system both of which had led to haze-free product in the laboratory. Prior to making these runs, the Fords raw materials were sampled and run in the laboratory and a lab batch was prepared with the Fords water supply. Both runs yielded clear products. The subsequent plant runs at Fords again did not show any improvement with these changes. During these runs samples of the latex from the reactor were taken and dried in the laboratory. These latexes did not exhibit the haze-free performance obtained from laboratory polymerization of the same raw materials.

The results indicate that a discrepancy exists between the lab and the plant in the polymerization process which may be related to possible contamination, deterioration or cleaning procedures. R&D and Process Engineering are investigating possible sources of contamination in the plant, the influence of latex heels in subsequent batches and improved reactor cleaning procedures.

2. Impact Modifier (J. C. Fisher, D. Goodman, M. Koral, M. K. Rosen)

The Polymer Pilot Plant has prepared impact modifier latex for a series of drying tests in late August, in conjunction with Process Engineering at Louisville Dryer and Nichols Engineering to define equipment requirements to successfully dry at commercial rates to 0.5% moisture content. This low a moisture content has yet to be realized in spray drying and flash drying trials to date. The Louisville Dryer trials will be on a rotary dryer and a flash/fluidized dryer which they have recommended, based on preliminary testing of impact modifier samples which were sent to Louisville. The Nichols test will be the final spray drying test in this series.

Coagulated impact modifier was tested at Sharples on their 14 inch and 30 inch basket centrifuges to determine dewatering characteristics of the cake and whether the plow discharge from this centrifuge will give free flowing wet product for dryer feed. These tests dewatered the cake to the 60% moisture level and the plow discharge was only partially successful in breaking the cake. This polymer is being sent to Louisville Dryer for their trials. Louisville indicates that they have a cake breaker that can handle this problem which will be investigated. Previous trials with pilot plant equipment and a Wyssmont Rotocage Lumpbreaker during August have not resolved this problem which greatly influences drying.

Only a limited amount of laboratory time was spent on this program during August to improve crease whitening properties due to the high priority given the Supercryl project. Rubber latex suppliers were contacted for additional SBR starting latex samples and some runs with compositional changes, increased mercaptan levels and procedural changes have been made but are not yet evaluated.

PVC Resins

1. Copolymer (J. C. Fisher, M. K. Rosen)

Samples of mixed A and B gelatin from Hormel were evaluated versus standard Atlantic gelatins in pilot plant polymerizations. Stable slurries were polymerized in the pilot plant with the Hormel gelatin mixture used for both T-315 and T-389 and samples are under evaluation at the Plastics Applications Laboratory.

R. S. Miller
R. S. Miller

RSM/srf