

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

TO H.B. Carr AT Burlington DATE February 15, 1972
FROM F.W. Kanzler AT Burlington COPY TO R.J. Eckman
SUBJECT Plate-Out in 3805-D-Black P. Gawason
Record Resin (All-Disc) ✓ File

As part of the effort to explain the plate-out observed in the subject compound, the following is a summary of the work done and the results of the tests performed in the Burlington Analytical Laboratory:

1. Use of Recovered Monomer

The type 384 lots listed in Table I show that lots 641062-63 had 1,000 pounds of recovered monomer charged with only one (1) out of fourteen (14) batches charged with virgin monomer. Lots 641068-69 recovered monomer level was at 2,000 pounds with seven (7) out of eighteen (18) batches containing virgin monomer only. The first four (4) lots manufactured in 1972 were charged with 1,000 and 2,000 pounds for twenty-three (23) batches while thirteen (13) batches had a charge of virgin monomer only. Lots 642005 through 642008 had a charge of 1,000 pounds for a total of twenty (20) batches and fourteen (14) batches with virgin monomer. Lot 642009 consisted of nine (9) batches with all the batches charged with 1,000 pounds of recovered monomer.

2. Use of Ninol

Ninol is added to the type 384 resin routinely at a level of 1:8000 parts of resin. To better interpret the effect of Ninol on the finished compound, Resin Plant Production suggested the use of compounds prepared with resins treated as listed in Table II. The pressouts were set up as follows:

- a. 3805-D-Black compound, pressed from dry blend.
- b. 3805-D-Black compound, pressed from roll milled sheet
- c. Type 384 resin with carbon black only, pressed from dry blend (same test as run in the Compound Plant).
- d. Type 384 resin with double the amount of carbon black added in c..

3. Microscopic Analysis

To best investigate the physical appearance of the type 384 resin treated as outlined in Table II, microscopic analysis was used. In addition, the pressed sheets were subjected to the same analysis in an attempt to characterize the imperfections and deposits appearing on the surface.

The above outlined investigations and analyses give rise to the following conclusions:

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To 1. - Use of Recovered Monomer

Table I (attached) and the summary in 1. demonstrate that no unusual amounts of recovered monomer were added. The change in the RVCM/RVAcM ratio is based on the gas chromatographic analysis results obtained from the master mix tank analysis. This analysis also reflects on the RTCE level changes. No formulation changes were made throughout the period of the lots listed. The virgin VCM origin for the last three (3) months (November and December, 1971 and January, 1972) was about thirty (30) tank cars of acetylene process monomer and approximately twenty-five (25) tank cars of oxychlorination process monomer per month.

In addition, physical testing data obtained showed no unusual variations for the lots listed with the exception of lot 642001 (higher screens thru 200) and 642005 (low on screens +80). A change in the resin based on monomer is therefore rather remote and can be ruled out.

To 2. - Use of Ninol

The pressouts of the various compounds prepared yielded the following results:

a. 3805-D-Black Dry Blend (Pressed from Dry Blend)

The compound of this pressout was prepared without prior heat history and was mixed in the Ronson Blender. The compounds of the four (4) samples (Table II) were pressed under the conditions normally used in the Compound Plant Quality Control Laboratory. The surface inspection showed no surface imperfections previously observed (blue areas). The area of the original location of the compound powder was easily distinguishable, however, by the non-migration of what is suspected to be carbon black. This is manifested by a brown tint in that area.

b. 3805-D-Black Dry Blend (Pressed from Roll Milled Sheet)

The compounds were prepared the same as in a. (above). After roll milling and pressout, the surface of the samples showed surface imperfections which were caused by the surface imperfections from the roll milled stock. The rectangular outline of the milled stock is very prominent. No other surface imperfections were noted in the areas of resin flow.

c. Type 384 Resin Mixed with Carbon Black Only

This test was developed by Compound Plant Quality Control as a quick check for possible plate-out caused by the resin. Sample A (Table II) showed none of the surface characteristics observed in the samples prepared and pressed in the Compound Plant. Flow lines are attributable to poor admixing of the resin with

the carbon black. Sample B (Table II) showed surface imperfections caused by carbon black. Sample C (Table II) showed some imperfections on the top of the pressout while the bottom was very difficult to release from the bottom plate. A very dull surface can be seen by the naked eye and is probably due to the large agglomerates in the resin. (See Microscopic Analysis below). Sample D (Table II) showed the best surface and it is suspected that the excess of Ninol acted as a lubricant.

d. Type 384 Resin Mixed with Double the Amount of Carbon Black Added in c.

Samples A through D were prepared with double the amount of carbon black. This was done because in pressout samples of plant compound submitted by the Compound Plant, microscopic examination showed deep black areas with a white crystal structure on the surface. To see if this may be caused by the carbon black, the amount of carbon black was doubled. Aside from carbon black flow lines, no unusual surface abnormalities were observed.

To 3. - Microscopic Analysis

With the exception of sample D (Table II), all other samples air-dried from slurry showed a relatively large amount of agglomerates when examined under the microscope. This was particularly pronounced in sample C. These agglomerates are suspected to be contributing to the dull surface described in To 2.,c. (above). Indeed the surface showed, when inspected under the microscope, poor admixed resin particles. All surface deposits observed in all the pressouts were treated with cold and hot water as well as cold methocel. In no case were these surface deposits removed by this treatment. It is safe to assume, therefore, that the plate-outs observed were not caused by the Ninol. The small amounts of deposits were not sufficient to be used for positive identification in this laboratory.

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Attachments (2).

FWK/jt

TABLE I

<u>Lot Number</u>	<u>Date</u>	<u>RVCM</u>	<u>RVAcM</u>	<u>RTCE</u>	<u>Number of Batches Run</u>	
		<u>Lbs.</u>	<u>Lbs.</u>	<u>Lbs.</u>	<u>With Recovery</u>	<u>Virgin Only</u>
641062	11/7/71	600	400	7	7	1
641063	11/7/71	600	400	7	6	-
641068	12/8/71	1600	400	16	10	2
641069	12/9/71	1600	400	16	1	5
642001	1/4/72	600	400	4	9	-
642002	1/6/72	600	400	4	4	5
642003	1/7/72	1100	900	16	5	4
642004	1/7/72	1100	900	16	5	4
		1500	500	24		
642005	1/15/72	600	400	9	9	-
642006	1/15/72	600	400	9	1	4
642007	1/16/72	550	450	8	3	8
642008	1/16/72	600	400	9	-	-
		550	450	8	7	2
642009	2/3/72	650	350	21	9	-

TABLE II

<u>Sample Designation</u>	<u>Additive</u>	<u>Level of Addition</u> <u>Additive:Resin</u>	<u>Origin</u>
A	Emcol CC-9	1:8000	Stripper
B	No Additives	None	Stripper
C	Ninol	1:8000	Blend Tank
D	Ninol	3:8000	Blend Tank

NOTE: The resins used were from
batches used in lot 642009.