



INTERNAL CORRESPONDENCE

CHEMICALS AND PLASTICS

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DATE April 25, 1977

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COPY TO

SUBJECT Test Program To Reduce RVC
Levels In Suspension Resin Slurry

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Suspension Vinyl Staff

As outlined in my letter of February 23, 1977, additional tests have been conducted to study the effect of stripping conditions on residual vinyl chloride monomer (RVC) of resin slurry leaving the blowdown tanks (BDT's). These tests were conducted during a three week period beginning February 21, 1977. Results of these tests indicate that Suspension resin slurry can be stripped below the 400 ppm level established by EPA. However, these results also indicate that higher stripping temperatures and pumping out under vacuum are necessary to prevent significant production losses which would result from previously employed stripping conditions.

The stripping procedure used during these tests is outlined below:

1. The slurry is moved from the autoclave to the BDT which is on the 15# vent header by autoclave pressure.
2. When the BDT pressure is below 20 psig, the BDT is switched to the 3# vent header. While on the 3# vent header the temperature is raised to 80°C.
3. After the BDT pressure is less than 8 psig and the temperature is at 80°C, the BDT is switched to the vacuum header. As this switch is made the steam (200 psig) is fully opened into the BDT for 30 minutes.

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4. After this 30 minute period the steam is reduced to half flow or stopped completely and the transfer from BDT to surge tank is begun with the BDT still under vacuum.

This procedure differs from the normal SOP in four respects in that the normal SOP requires that:

1. The temperature be raised to 85°C while on the 3# vent header.
2. Only enough steam be added on the vacuum header to hold the temperature at 85°C.
3. The BDT be held for a 30 minute period under vacuum conditions.
4. The BDT be switched back to the 15# vent header before beginning the transfer from BDT to surge tank.

With this "revised SOP" in effect, the stripping tests were begun on QSAH resin being produced on the East line. RVCN data are presented in Figure I with other data presented in Table 1. As you can see from the Table the stripping cycle increased about 25% with this revised SOP. However, this increased stripping cycle is somewhat inflated due to the increased time on the 15# vent which should not have changed with the "revised SOP". The other major contributor to the increased cycle is the pumpout time which resulted from pumping out under vacuum.

During this test a 22% drop in production rate was noted on the East line. Again, it should be pointed out that not all of this drop can be attributed to the "revised SOP" since other variables also affected production rate during this test. It is felt that with other variables constant a drop of about 15% in production rate would have been noted.

In order to compare resin quality before and after the test, it was decided that bin analysis would have to be used instead of BDT analysis. This is necessary because of the large variability in BDT analysis with time. Unless BDT samples are taken at the same time within the stripping process they cannot be easily compared. From Table 1 we see that bin analysis averaged 214 and 301 ppm RVCN for the week before and week after the test respectively. During the test the bin RVCN averaged only 155 which indicates a relatively small reduction in RVCN level in the dry resin. The BDT data presented in Figure I shows that the RVCN level of the slurry leaving the BDT can be reduced to 400 ppm in about 60 minutes; with levels of 40 to 70 ppm being reached in 90 minutes.

The bin analysis also indicates no significant rise in color index of the resin with 6.5 and 8.7 being averaged the weeks before and after the test and 7.6 during the test.

Using the same "revised SOP" stripping procedure as outlined earlier, tests were conducted on the South line while producing QSAL resin. Data from these tests are presented in Figures II and III and Table II. This data follows the trend of the earlier RVCM data on QSAH but with some exceptions in stripping cycle. For example, the total stripping cycle actually dropped 4% during the test under the "revised SOP" as compared with the normal stripping procedure. Examination of the data indicates this is mainly due to an improved time on the vacuum header which was a direct result of the procedure change. Pumpout time for the BDT's increased 12% while pumping under vacuum but was not a significant factor in the stripping cycle.

The RVCM data presented in Figures II and III indicates that QSAL resin would average about 400 ppm within 45 to 60 minutes after switching to the vacuum header. It is believed that sampling procedures on the final samples of batches 351, 353, 365 were the cause of the higher than expected RVCM levels. Tight control on batch 366 did result in a significantly lower RVCM level.

Again, from Table II we see that this "revised SOP" for stripping did result in lower RVCM level in the bin analysis, 31 ppm versus 40 ppm for the week prior to the test. It is felt that with stricter adherence to this "revised SOP" this RVCM could be lowered further. However, it should be noted that the color index of the bins did rise during this test from 4.5 to 5.8. While there is no color specification for homopolymers a color index above 6.0 is not considered desirable. This indicates that color may be a problem in resin slurry stripping on homopolymers but not quite the problem of copolymers.

Stripping tests on VSKK being produced on the North line were a little more complicated than those previously performed. The line was actually divided into two separate lines which used different stripping procedures. The West side of the line followed the same procedure that had been used on the other two lines and outlined earlier with the addition of five gallons of Bisphenol-A into the resin slurry prior to stripping. The East side of the line followed the same procedure except the stripping temperature was raised to 70°C on the 3-pound vent header and 10-pounds of sodium nitrite was added to the slurry prior to stripping. These materials were added to the slurry in order to try and determine their effects on resin color stability. Also, the temperature was raised on the slurry containing sodium nitrite because earlier tests had indicated it would be effective at the higher temperature and the higher temperature would be helpful in lowering RVCM.

Data for RVCM levels are presented in Figures IV and V for the East and West sides of the line respectively. Figure IV also presents data from batch 181 which contained no additives along with those batches containing sodium nitrite. The maximum temperature reached during stripping is noted as is the beginning and ending color index for each batch. All three batches which contained sodium nitrite and were stripped above 90°C reached the 400 ppm RVCM level in less than 30 minutes. While the color index was high at the beginning of the stripping operation an average increase in color index of only 1.33 is noted for those batches containing sodium nitrite compared to 8.6 for the batch without additives. Those batches containing Bisphenol-A rose an average of 1.40 in color index but required more than 45 minutes to reach the 400 ppm RVCM level at the lower stripping temperature.

The data presented in Table III is a bit surprising since the pumpout time under vacuum is actually less than under the 15-pound vent header. This, however, does not relate to production rates since production was down 7% during the tests. Please note that this Table combines all data for the North line stripping cycle and does not distinguish between East and West sides as does the RVCM data. We believe this drop in pumpout time resulted from a noted reduction in plugging problems with the slurry transfer line during the tests. This does point up the fact that many variables control production rates and not just equipment capacities.

Also presented in Table III are the average RVCM levels and color indexes for bins. While the RVCM level dropped significantly during the test from the previous week, 1298 versus 1662, the week following the test showed no significant rise, 1368. This suggests that the test did help reduce RVCM but not significantly. The average color index of the bins rose from 10.1 both the week before and after the test to 10.5 during the stripping tests. This color index increase is small but since the RVCM level is well above 400 ppm average a much larger increase would be expected if the 400 ppm level were reached. One reason for the high average RVCM level in the bins at the same time BDT samples are much lower could be the close adherence to procedure while BDT samples are taken on day shift and only casual observance of procedure at other times.

An overall look at the data presented in this report leads me to two major observations. The first is that boiling of the slurry in the BDT is extremely beneficial in rapidly reducing RVCM.

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The higher temperature employed during stripping allowed the slurry to boil sooner as the pressure was dropped via the vacuum header. It is felt that boiling and not just the temperature alone was the major factor in rapidly reducing RVCM levels. This suggests that if vacuum conditions could be reached more quickly in the BDT the temperature could be held at a lower level. This would require additional vacuum pumps and compressors. The approved project to install the second Nash Hytor compressor will help us move in this direction. The second observation is that with our system pumping under vacuum is essential if we are to reach the 400 ppm goal. While the present pumps will do the job, they will in the long run present more problems and add to the cycle time of the BDT's than if new pumps are installed. The present pumps are simply not designed for this type service and if required to operate in this mode we should install the proper equipment for the job.

No further testing is planned at this time but will be undertaken as deemed necessary. For example, as additional equipment is installed or resins are produced on which little or no data is available.

Your comments on this report are welcomed and should be directed to me at Building 114, Extension 672.

Many thanks go to Steve Dickerson and his Technicians who did the collecting and analyzing of the samples.

Sincerely,



K. E. Bowen

KEB/st
Attachments

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FIGURE I

QSAH
RVCM VS TIME
(Revised SOP)

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K-E SEMI-LOGARITHMIC 0.3 CYCLES X 10 DIVISIONS
KEUFFEL & ESSER CO. MADE IN U.S.A.

RVCM (ppm)

10⁴
10³
10²
10¹
10⁰
10⁻¹
10⁻²
10⁻³
10⁻⁴
10⁻⁵
10⁻⁶
10⁻⁷
10⁻⁸
10⁻⁹
10⁻¹⁰
10⁻¹¹
10⁻¹²
10⁻¹³
10⁻¹⁴
10⁻¹⁵
10⁻¹⁶
10⁻¹⁷
10⁻¹⁸
10⁻¹⁹
10⁻²⁰
10⁻²¹
10⁻²²
10⁻²³
10⁻²⁴
10⁻²⁵
10⁻²⁶
10⁻²⁷
10⁻²⁸
10⁻²⁹
10⁻³⁰
10⁻³¹
10⁻³²
10⁻³³
10⁻³⁴
10⁻³⁵
10⁻³⁶
10⁻³⁷
10⁻³⁸
10⁻³⁹
10⁻⁴⁰
10⁻⁴¹
10⁻⁴²
10⁻⁴³
10⁻⁴⁴
10⁻⁴⁵
10⁻⁴⁶
10⁻⁴⁷
10⁻⁴⁸
10⁻⁴⁹
10⁻⁵⁰
10⁻⁵¹
10⁻⁵²
10⁻⁵³
10⁻⁵⁴
10⁻⁵⁵
10⁻⁵⁶
10⁻⁵⁷
10⁻⁵⁸
10⁻⁵⁹
10⁻⁶⁰
10⁻⁶¹
10⁻⁶²
10⁻⁶³
10⁻⁶⁴
10⁻⁶⁵
10⁻⁶⁶
10⁻⁶⁷
10⁻⁶⁸
10⁻⁶⁹
10⁻⁷⁰
10⁻⁷¹
10⁻⁷²
10⁻⁷³
10⁻⁷⁴
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10⁻⁷⁶
10⁻⁷⁷
10⁻⁷⁸
10⁻⁷⁹
10⁻⁸⁰
10⁻⁸¹
10⁻⁸²
10⁻⁸³
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10⁻⁸⁶
10⁻⁸⁷
10⁻⁸⁸
10⁻⁸⁹
10⁻⁹⁰
10⁻⁹¹
10⁻⁹²
10⁻⁹³
10⁻⁹⁴
10⁻⁹⁵
10⁻⁹⁶
10⁻⁹⁷
10⁻⁹⁸
10⁻⁹⁹
10⁻¹⁰⁰

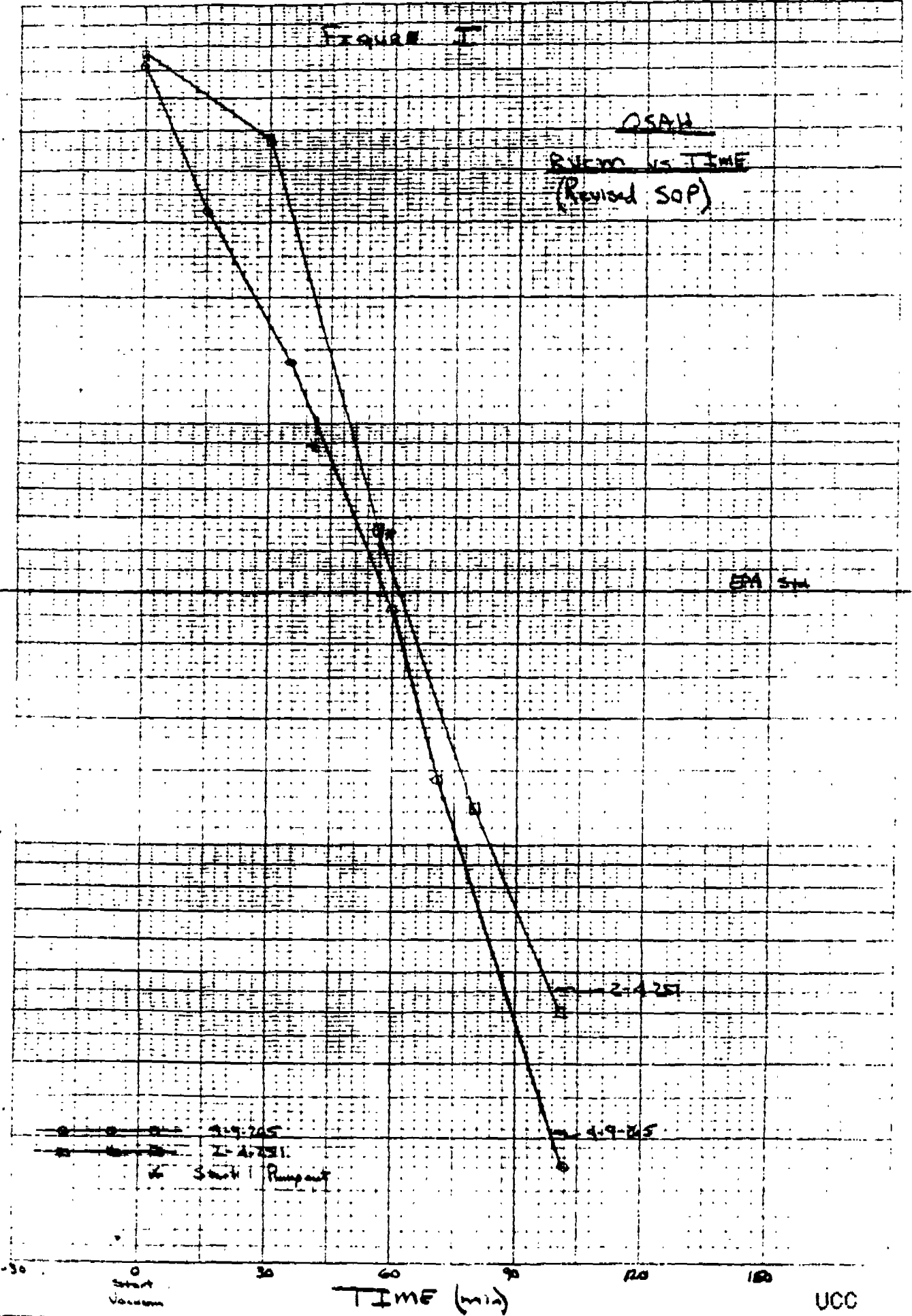


TABLE I
EAST LINE - QSAH

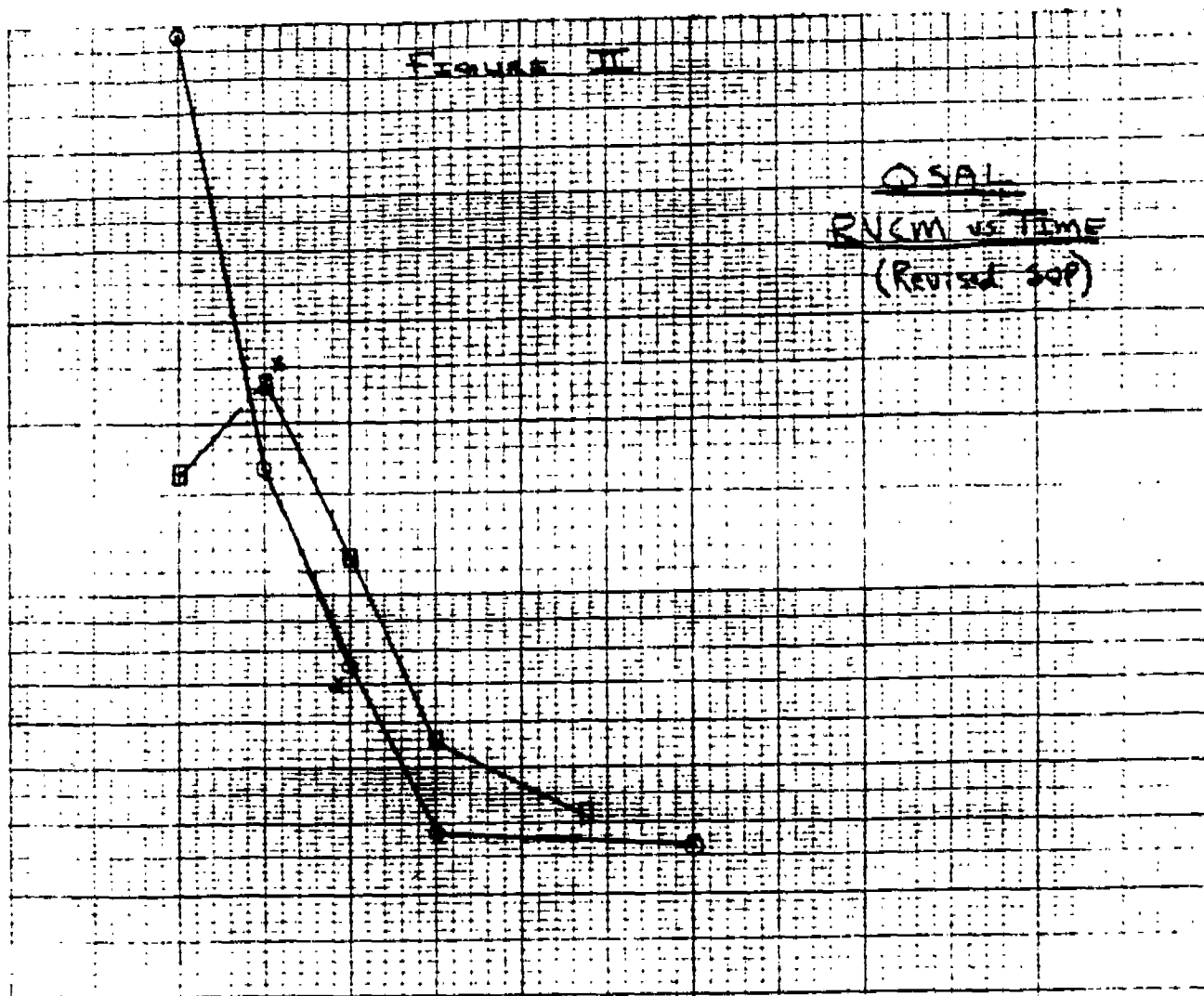
	<u>Std. SOP</u>	<u>Revised SOP</u>
Autoclave Cycle		
Charge & Heatup	2.17	2.07
Reaction & Coasting	9.75	12.87
Pullout	<u>0.57</u>	<u>0.92</u>
Total	12.49	15.86
BDT Cycle		
15-pound Vent	0.57	0.92
3-pound Vent	0.52	0.72
Vacuum	0.87	0.68
Pumpout	<u>0.75</u>	<u>1.06</u>
Total	2.71	3.38
Average Production (M lbs/day)	102.1	79.2
	week before test	week after test
Average Bin RVCM (ppm)	214	301
		155
Average Color Index	6.5	8.7
		7.6

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K-E SEMI-LOGARITHMIC - 3 CYCLES x 10 DIVISIONS
KEUFFEL & ESSER CO. NEW YORK

RUCM (ppm)

10⁹
9
8
7
6
5
4
3
2
1
10⁸
9
8
7
6
5
4
3
2
1
10⁷
9
8
7
6
5
4
3
2
1
10⁶
9
8
7
6
5
4
3
2
1
10⁵
9
8
7
6
5
4
3
2
1
10⁴
9
8
7
6
5
4
3
2
1
10³
9
8
7
6
5
4
3
2
1
10²
9
8
7
6
5
4
3
2
1
10¹
9
8
7
6
5
4
3
2
1
10⁰



20-12-381 (70% max) 3.5-3.6
18-3-352 (70% max) 4.3-4.0
Start Pumpout

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10⁰
9
8
7
6
5
4
3
2
1
10¹
9
8
7
6
5
4
3
2
1
10²
9
8
7
6
5
4
3
2
1
10³
9
8
7
6
5
4
3
2
1
10⁴
9
8
7
6
5
4
3
2
1
10⁵
9
8
7
6
5
4
3
2
1
10⁶
9
8
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6
5
4
3
2
1
10⁷
9
8
7
6
5
4
3
2
1
10⁸
9
8
7
6
5
4
3
2
1
10⁹

Start Vacuum

TIME (min)

30 60 90 120 150

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K-E SEMI-LOGARITHMIC PLOTTER
 REUFFEL & SMITH CO. NEW YORK, N.Y.

RUCM (ppm)

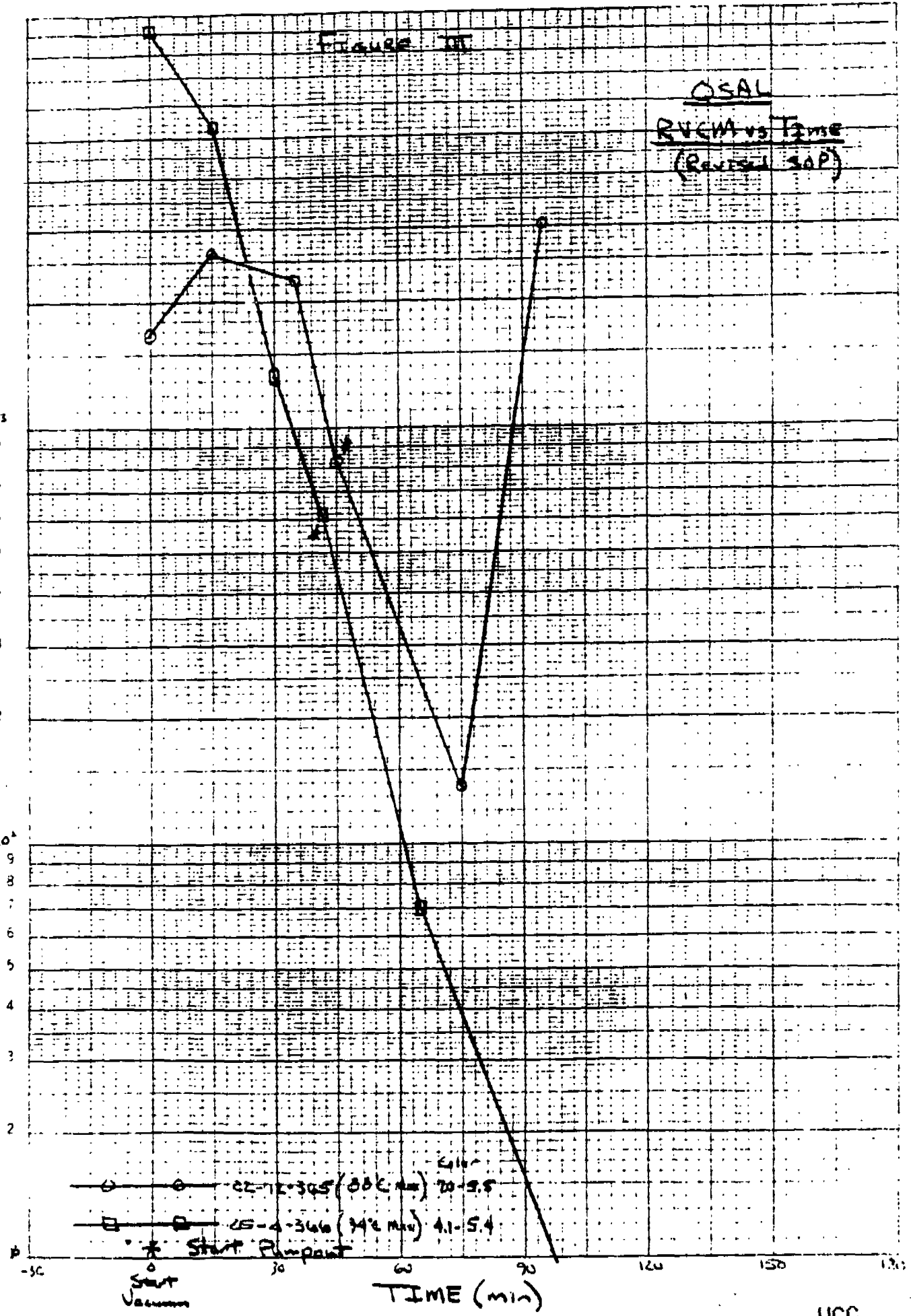


TABLE II
SOUTH LINE - QSAL

	<u>Std. SOP</u>	<u>Revised SOP</u>
Autoclave Cycle		
Charge & Heatup	1.77	1.80
Reaction & Coasting	9.33	8.90
Pullout	<u>0.50</u>	<u>0.84</u>
Total	11.60	11.54
BDT Cycle		
15-pound Vent	0.50	0.84
3-pound Vent	0.47	0.41
Vacuum	1.07	0.58
Pumpout	<u>1.00</u>	<u>1.12</u>
Total	3.06	2.95
Average Production (M lbs/day)		
	187.1	167.2
Average Bin RVCM (ppm)		
	40	31
Average Bin Color Index		
	4.5	5.8

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K-E SEMI-LOGARITHMIC PLOT OF RUCM VS. TIME
NEUPHEL & ESSER CO. 1000-1000

FIGURE IV

USKK
RUCM VS TIME

(Revised SOP with NaNO_2)

10³

9

8

7

6

5

4

3

2

1

0

10²

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10¹

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10⁰

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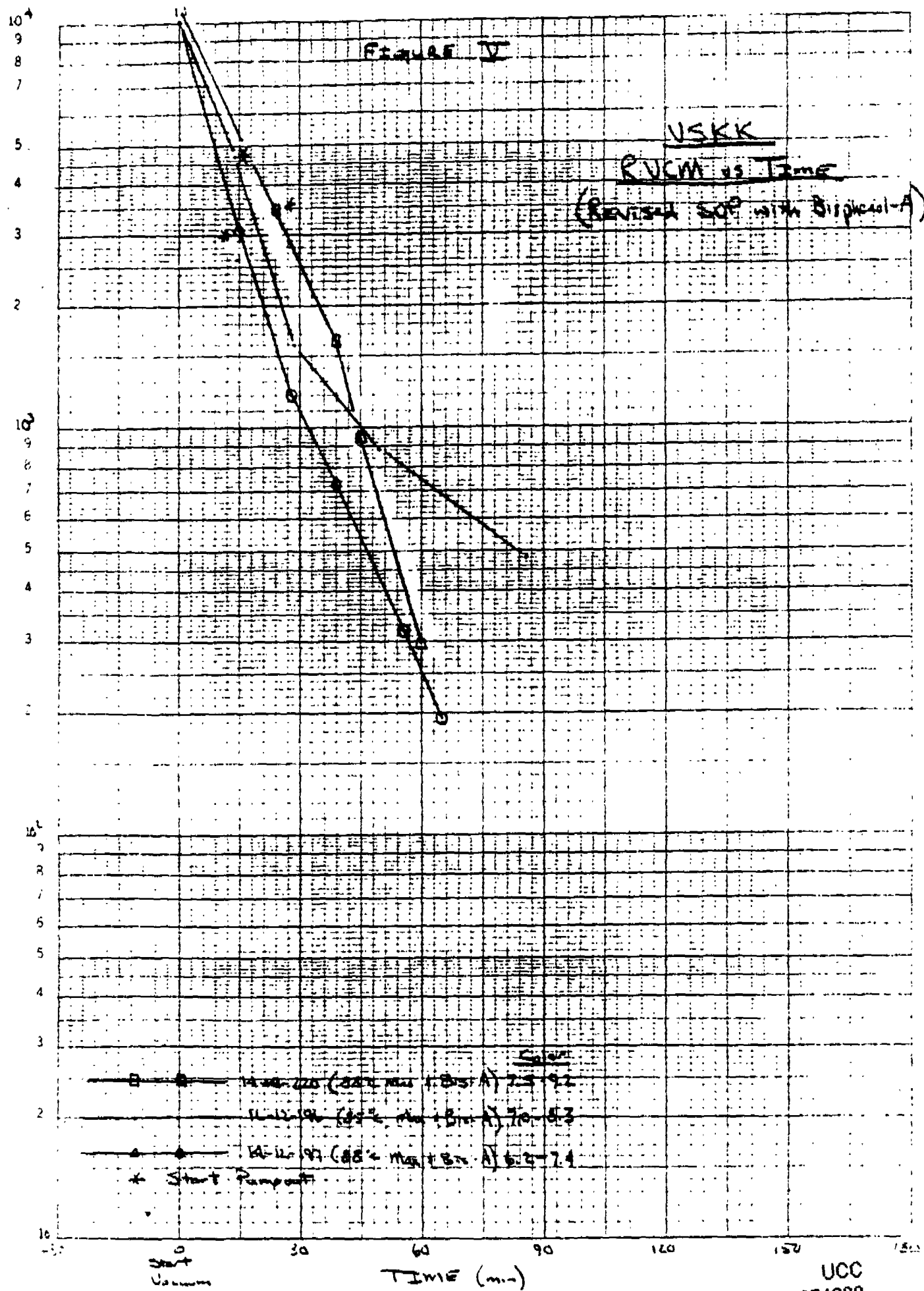
K-E SEMI-LOGARITHMIC CYCLES & DIVISIONS
NEUPHEL & ESSER CO. NEW YORK

TABLE III
NORTH LINE - VSKK

	<u>Std. SOP</u>	<u>Revised SOP</u>
Autoclave Cycle		
Charge & Heatup	2.24	2.24
Reaction & Coasting	10.04	10.26
Pullout	<u>0.59</u>	<u>0.72</u>
Total	12.87	13.18
BDT Cycle		
15-pound Vent	0.57	0.72
3-pound Vent	0.59	0.48
Vacuum	0.73	0.52
Pumpout	<u>0.93</u>	<u>0.86</u>
Total	2.84	2.58
Average Production (M lbs/day)	140.9	131.4
	week before test	week after test
Average Bin RVCM (ppm)	1662	1368 1298
Average Bin Color Index	10.1	10.1 10.5