MILLIKEN RESEARCH CORPORATION

Spartanburg, South Carolina

LABORATORY NOTEBOOK

Name Chineta Camp Book № 10118

Used from June 4, 1996 To July 30, 1996

Laboratory Book Nº 10118

Date Received: June 4, 1996

Assigned To: Chinetta Camp

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The early date of record may mean the difference between getting an important patent for the Research Corporation in your name, or losing it to some other company which can actually keep our stockholder mills from using the idea.

If after further thought and discussion, or a few experiments, you decide that the idea may have value, you may draw up a formal description, either as a memo or on the invention record form provided, for submission to the Patent Division.

It is a function of the Patent Division to review the yellow sheets in this notebook as forwarded by you and to cull from them all ideas having patentable novelty, but the invention record form submitted in this way will serve to accent those ideas considered by you to be most important.

PROCEDURE FOR
KEEPING LABORATORY NOTEBOOKS

1. Make all entries in ink and as legible and complete as possible.

2. Every pair of sheets is consecutively numbered, and all entries should be made on the white sheets with a carbon paper between the white sheet and the next succeeding yellow sheet. The yellow sheets, which are perforated for easy removal, are torn out daily and forwarded to the administrative office of the Research Corporation. No white pages (containing the original of the entry) should be torn out under any circumstances.

3. All experimental data is to be entered in this book, and as far as possible, all calculations, graphs, drawings and notes of any character should be entered herein. When patents are referred to, include patent number. When literature is referred to, include complete journal references (Author, Volume, Page, Date).

4. Make all entries in this notebook **first**. It is extremely important that this notebook be what the law calls a "notebook of original entry." Make your first notes about anything—weight, temperature, time, ideas for future work, drawings, sketches—**in this notebook**. If you want to keep a separate tabulation in special cases, do your transcribing of data from the notebook into the tabulation, but not the other way.

5. When starting a problem use the following heading:

Subject:_____ Project No._____

Experiment No. _____ Test No._____

If work done on the problem for the day fills more than one page, continue on next page and start heading:

Project No._____ Continued

If work done on the problem for the day fills part of page, use the remainder of page for the next problem worked on that day and fill in the complete heading. Make all your entries in strict chronological order. If you are working on several experiments at once and making alternate observations, use the full heading at the start of each experiment each day, and for later entries on that day you need only note the experiment number beside the date.
Date and sign every page or after each problem on the same page.
The full date should be entered, e.g., July 1, 1946, not 7/1/46.

Do not leave blank spaces after experiments. If there is only a small amount of space, 1 to 2 inches, left at the end of the page when an experiment is finished, draw diagonal lines through this space.
Have an associate, if possible a superior, witness the record of the entire experiment and the signature of each experiment. He should be someone qualified to understand the record.

6. Erasures should never be made. Draw a line through the incorrect work or entry. Never make corrections on a page after signing and dating. If it is at any time observed that a cancellation or addition should have appeared in an earlier entry, a new entry should be made, stating what should be cancelled and what addition should have appeared in the earlier entry.

7. When an experiment must be started on one day and completed some days or a week later, space should not be left at the first entry for subsequent results, but when the results are finally received, the data from the original day's work may (but need not) be brought forward (with suitable reference to the page where they first occur) and the whole experiment summarized on the day on which the final data are obtained.

8. **Do your thinking in the notebook.** Whenever you have an idea of any kind on future experimental work, whether it bears on your own problems or those of other laboratory or research sections or of others with whom you may be engaged during the course of your work, jot it down in your notebook at once, with as much detail as possible. Do the same for proposed apparatus layouts and experimental procedures.
After any discussion, formal or informal, that bears on the work of the Research Corporation or other organizations engaging the services of Research Corporation, note what it was about and who was present. If you watch somone else carry out an experiment, make a note of what you saw and who was there.
In short, make this notebook a diary so that anyone can go back to it years later (five to ten years is not unusual in patent actions) and report without question what you did, what you saw, what you thought, who was present, whom you spoke to, what you discussed and **when all this occurred.**

9. Index experiments on index pages in front of book.

10. When describing experimental work in weekly or monthly reports, always include the notebook and page numbers on which the work can be found.

11. When a book is filled, it is checked and approved by your section or division head and returned to the Patent Divison for filing, unless it is to be retained for reference. However, only one finished book may be retained, and all other finished books must be returned to the Patent Division unless specific permission for their retention is obtained from the Director of Research. If other notebooks are later needed for reference, they can be charged out.

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All pipets are serialized and class A

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(DV Methods)

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Method List (CONTINUED)

(DV Methods)

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SF8320 Acid Number 10118-1

lot 10023-74

Purpose:

To perform acid number testing on the following given sample product.

Apparatus:

1 - 250ml ~~Erlenmeyer flask~~ CYC 6/19 beaker
2 - magnetic stirrer & stir bar
3 - 45 ml burette

Reagents:

1 - Reagent grade Methanol
2 - Phenolphthalein indicator
3 - 0.5 N KOH (aqueous)

Raw Material Charges:

Epal 1Ø 4EO/2PO (lot 10023-74) - 3.5g
→ 75% Acetic Anhydride Rxn.

Epal 1Ø 4EO/2PO (lot 10023-74) - 3.5g
→ 100% Acetic Anhydride Rxn.

Procedure:

1) add 3.5g of smpl. to 250ml beaker
2) add 100ml Methanol to beaker
3) add stir bar & stir for 5 min. (until smpl. dissolved)
4) add 6-8 drops of phenolphthalein indicator to beaker
5) titrate sol'n using 45ml burette of 0.5N KOH
6) Record Results & do calculation

Date June 19, 1996 Signature Chineta J. Camp
Date June 21, 1996 Witness [illegible]

→ cont. p. 2

SF8320 Acid #

→ continued from pg. 10118-1

Results: DV 12

75% Rxn.			Acid #
(4E/2P)	3.5165	(37.66)	→ 300.40

100% Rxn.			Acid #
(4E/2P)	3.5275	(37.34)	→ 296.92

SPECIFICATIONS: (Triton DF-20 Surfactant)

Acid Number (mg KOH/g)

low spec	>	290
high spec	>	335

Conclusion:

both samples had acid #'s within the given range

Date June 19, 1996 Signature Chineta Y. Camp

Date June 21, 1996 Witness [illegible]

SF8320 Viscosity 10118- 3

lot 10023-74

Purpose:

To predict the viscosity performance on the following given sample.

Apparatus:

1 - top load balance
2 - viscometer w/ cylinder & water bath

Reagents: NONE

Raw Material Charges:

Epal 1Ø 4EO/2PO (lot 10023-74) - 8g
→ 75% Acetic Anhydride Rxn.

Epal 1Ø 4EO/2PO (lot 10023-74) - 8g
→ 100% Acetic Anhydride Rxn.

Procedure:

1) set viscometer on the following settings: 25°C, 20 RPM, & CP display
2) allow smpl. to become room temp.
3) add 8g smpl. to viscometer cylinder
4) let viscometer read smpl. for 30 min.
5) once reading has stabilized, record results

→ cont. p. 4

Date June 19, 1996 Signature Chinetta Y. Camp

Date June 21, 1996 Witness [illegible]

→ continued from pg. 10118-3

Results: DV26

75% Rxn.	viscosity	
(4E/2P)	590.0 cP	@ 30 min.

100% Rxn.	viscosity	
(4E/2P)	557.5 cP	@ 30 min.

SPECIFICATIONS: (Triton DF-20 Surfactant)

Viscosity (cP)		
low spec	>	600
high spec	>	700

Conclusion:

the 75% Rxn. was below the given Range & the 100% Rxn. was also below the given Range for viscosity

Date June 19, 1996 Signature Chineta Y. Camp

Date June 21, 1996 Witness [illegible]

SF8320 Cloud Point 10118- 5

lot 10023-74

Procedure:

To determine the cloud point of the given sample. (5% in 6.8 N KOH)

Apparatus:

1 - 150ml beaker
2 - 1L Volumetric flask
3 - magnetic stirrer & stir bar
4 - hot plate
5 - °C thermometer

Reagents:

1 - 6.8N KOH solution (aqueous)

Raw Material Charges:

Epal 1Ø 4EO/2PO (lot 10023-74) - 5g
→ 75% Acetic Anhydride Rxn.

Epal 1Ø 4EO/2PO (lot 10023-74) - 5g
→ 100% Acetic Anhydride Rxn.

Potassium Hydroxide (lot J3599) - 381g
- Dewey

Procedure:

KOH soln Preparation:

1) in a 1L Volumetric flask, ~~mix~~ CYC 6/19 add 381g KOH flasks & add d.i. water to mark-off line

Date June 19, 1996 Signature Christa Y. Camp

Date June 21, 1996 Witness [illegible]

→ cont. p. 6

→ continued from pg. 10118-5

Procedure: (cont.)

KOH sol^N/ Prep: (cont.)

2) stir mixture for about 15 min. (or until KOH is completely dissolved)
3) place flask in bucket of ice for about 30 min. to allow mixture to cool
4) pour sol^N/ in amber bottle & set aside

Cloud Pt. Procd: ~~(C~~ CYC 6/20

1) in a 250 ml beaker, add 95g of ~~smpl.~~ CYC 6/20 given sol^N/ & 5g of smpl.
2) add a stir bar to sol^N/ & heat slowly until sol^N/ begins to cloud
3) Record temp. at which sol^N/ reached its cloud point

Results: DV 582 (5% in 6.8N KOH)

75% Rxn.		Cloud Pt.
(4E/2P)	@	70 °C

100% Rxn.		Cloud Pt.
(4E/2P)	@	69 °C

→ cont. p.7

Date June 20, 1996 Signature Chineta Y. Camp
Date June 21, 1996 Witness [illegible]

→ continued from pg. 10118-6

Conclusion:

the 75% Rxn. & 100% Rxn. were both ~~out of~~ above the given Range for cloud points

CYC 6/20

NOTE: while smpl. were dissolving in sol'n, the 100% Rxn. took about 5 min. longer than the 75% Rxn. to completely disperse before the cloud point was Reached

SPECIFICATIONS: (Triton DF-20 Surfactant)

Cloud Point (5% in 6.8N KOH)

low spec > 54°C

high spec > 64°C

~~110~~ CYC 6/20

~~69~~ CYC 6/20

Date June 20, 1996 Signature Chinota Y. Camp

Date June 21, 1996 Witness [illegible]

SF8320 Hydrolyzation of OSA lot 10023-74

Purpose:

To hydrolyze the OSA after it has already been mixed with the acetylated alcohol.

Apparatus:

1 – 3 neck/500ml rxn. flask
2 – stirring apparatus
3 – controlled thermometer
4 – heating mantle

Reagents: NONE

Raw Material Charges:

Epal 1Ø 4EO/2PO (lot 10023-74) → 75% Acetic Anhydride Rxn.	–	38 g
Epal 1Ø 4EO/2PO (lot 10023-74) → 100% Acetic Anhydride Rxn.	–	38 g
OSA-NJC (lot TRL-3/Dewey) → stock # 680085	–	57.1 g
Deionized Water	–	4.9 g

Procedure:

1) in 500ml rxn. flask, add 57.1g of 50% OSA & 38g of smpl.
2) then slowly add 4.9 g of d.i. water
3) add stirring apparatus & controlled thermometer to flask and place it in heating mantle

Date June 21, 1996 Signature Christopher J. Camp
Date June 21, 1996 Witness [illegible]

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SF8320 Hydrolyzation

→ continued from pg. 10118-8

Procedure: (cont.)

4) cook mixture at 90°C for about 3 hrs.
5) after cook time, allow mixture to cool ~~for~~ to 50°C & then pour into 8oz. smpl. jar
CYC 6/21 6) Run usual product analysis tests on smpl.

Results:

Rxn. completed & OSA is now totally hydrolyzed

Conclusion:

OSA completed & mixed thoroughly with the acetylated alcohol

Date June 21, 1996 Signature Chinetay Camp

Date June 21, 1996 Witness [illegible]

Cloud Point
lot 10118-8

Procedure:

To determine the cloud point of the given sample. (5% in 6.8N KOH)

Apparatus:

* same items listed on pg. 10118-5

Reagents:

* same products listed on pg. 10118-5

Raw materials Charges:

Epal 1Ø 4EO/2PO (lot 10118-8) – 5g
→ 75% Acetic Anhyd. Rxn.

Epal 1Ø 4EO/2PO (lot 10118-8) – 5g
→ 100% Acetic Anhyd. Rxn.

Potassium Hydroxide (lot J3599) – 381g
→ Dewey

Procedure:

KOH sol'n Prep: * same steps outlined on pp. 10118-5 & 10118-6

Cloud Pt. Procd: * same steps outlined on pg. 10118-6

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Date June 21, 1996 Signature Chinetta Y. Camp

Date June 21, 1996 Witness [illegible]

SF8320 Cloud Pt.

→ continued from pg. 10118-10

Results: DV582 (5% in 6.8N KOH)

75% Rxn.		Cloud Pt.
(4E/2P)	@	70°C

100% Rxn.		Cloud Pt.
(4E/2P)	@	69°C

Conclusion:

both Rxns. were above the given Range for cloud points

SPECS: low - 54°C / high - 64°C

Date June 21, 1996

Date June 21, 1996

Signature Chineta Y. Camp

Witness [illegible]

SF8320 Viscosity 10118-12

lot 10118-8

PROCEDURE:

To determine the viscosity performance on the given sample product.

Apparatus:

* same items listed on pg. 10118-3

Reagents: NONE

Raw Material Charges:

Epal 10 4EO/2PO (lot 10118-8) — 8g
→ 75% Acetic Anhyd. Rxn.

Epal 10 4EO/2PO (lot 10118-8) — 8g
→ 100% Acetic Anhyd. Rxn.

Procedure:

* same steps outlined on pg. 10118-3

Results: DV26

75% Rxn.	viscosity	
(4E/2P)	255.0 cP	@ 30 min.
100% Rxn.	viscosity	
(4E/2P)	515.0 cP	@ 30 min.

→ cont. p. 13

Date June 21, 1996 Signature Chineta Y. Camp

Date June 21, 1996 Witness [illegible]

SF8320 Viscosity 10118-13

→ continued from pg. 10118-12

Conclusion:

both Rxns. were below the given Range for viscosity

SPECS: low-600cP / high-700cP

Date June 21, 1996 Signature Christy Y. Camp

Date June 21, 1996 Witness Rick Rib

SF8320 Acid Number lot 10118-8 10118-14

Purpose:

To perform acid number testing on the given sample product.

Apparatus:

* same items listed on pg. 10118-1

Reagents:

* same products listed on pg. 10118-1

Raw Material Charges:

Epal 10 4EO/2PO (lot 10118-8) — 5.0g
→ 75% Acetic Anhyd. Rxn.

Epal 10 4EO/2PO (lot 10118-8) — 5.0g
→ 100% Acetic Anhyd. Rxn.

Procedure:

* same steps outlined on pg. 10118-1

Results: DV 12

75% Rxn. (4E/2P) 5.0193 (44.08) → Acid # 246.34

100% Rxn. (4E/2P) 5.0164 (51.42) → Acid # 287.52

Conclusion: SPECS: low-290/ high-335
both Rxns. were below given Range CHC 6/21 Acid #'s

Date June 21, 1996 Signature Christy Y. Camp
Date June 21, 1996 Witness [illegible]

SR604 MEHQ Analysis 10118-15

"NO" lot #

Purpose:

To check the parts per million of MEHQ in the given samples of SR604.

Apparatus:

1 - UV/Vis Spectrophotometer
2 - 100ml Volumetric flask w/ Stopper (4)
3 - 2ml pipette /TD (2)
4 - 10ml pipette /TD (2)
5 - 5ml pipette /TD (1)

Reagents:

1 - Sulfanilic Acid sol^N/
2 - Sodium Nitrite sol^N/
3 - 25% Sulfuric Acid (aqueous)
4 - 8% Sodium Hydroxide (aqueous)
5 - 50/50 Methanol - Water sol^N/

Raw Material Charges:

Sulfanilic Acid (lot SR3541/Aceto)	-	0.76g
Sodium Nitrite (lot C9058/Dewey)	-	0.34g
Caustic 50% (lot B4130/Dewey)	-	16g
Sulfuric Acid 98% (lot W2343/Dewey)	-	25g

Procedure:

Prep. of Sulfanilic Acid sol^N/:
→ dissolve 0.76g sulfanilic acid in 100ml d.i. water

Prep. of Sodium Nitrite sol^N/:
→ dissolve 0.34g sodium nitrite in 100ml d.i. water

Date June 24, 1996 Signature Chiquita Y. Camp
Date July 2, 1996 Witness Philip Smith

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→ continued from pg. 10118-15

calc. 50% caustic
⇓
50 NaOH/50 H_2O
(100g)(0.08) = 8g NaOH ⇒ 16g caustic

Procedure: (cont.)

Prep. of 8% Sodium Hydroxide sol^N/:
→ dissolve 16g caustic 50% in 84g d.i. water

Prep. of 50/50 Methanol-Water sol^N/:
→ mix 500ml methanol w/ 500ml d.i. water

Prep. of 25% Sulfuric Acid sol^N/:
→ dissolve 25g sulfuric acid 98% in 75g d.i. water
NOTE: Always add acid to water & NOT vice versa

Analysis:

1) in 4oz. clear vial, pipette 10ml sulfanilic acid sol^N/, 2ml H_2SO_4 (25%) sol^N/, & 10ml $NaNO_2$ sol^N/
2) add lid, shake well to mix, & allow to set for at least 5 min. before use
3) accurately weigh 1.0000g MEHQ samples into 100ml Vol. flasks & record weight
4) fill flask approx. 1/2 full w/ 50/50 MeOH-H_2O
5) pipette ~~8~~ 2ml NaOH sol^N/ to flask
6) pipette ~~2~~ 5ml of sol^N/ in step [1] to flask & mix thoroughly
7) dilute mixture to mark w/ 50/50 MeOH-H_2O
8) allow samples to set for 30 min.
9) obtain UV/vis of sample (using 50/50 MeOH-H_2O as the blank) on the spectrophotometer
10) calculate the absorbance concentration

→ cont. p. 17

Date June 24, 1996 Signature Chineta Y. Carey
Date July 2, 1996 Witness Philip Smith

SR604 MEHQ

→ continued from pg. 10118-16

Results: DV601 SW=1.0000g

* smpl. are ppm MEHQ in Diethylene Glycol

Smpl.	*	smpl. wt.	wavelength(nm)	absorbance	corrected concente
1) 916 ppm	(920)	1.0215	512.5	0.65506	900.6
2) 1008 ppm	(1010)	1.0042	510.5	0.71036	1005.8
3) 1111 ppm	(1115)	1.0294	512.5	0.78099	1083.2
4) 1213 ppm	(1220)	1.0238	512.5	0.90328	1191.6

* ck. plot graph to get # to divide by smpl. wt. to calc. the corrected concentration

Conclusion:

the MEHQ analysis of the given product is completed; all sample concentrations were within their given range

⟹ CONT. on pg. 10118-24

Date June 24, 1996 Signature Chinita U. Camp

Date July 2, 1996 Witness [illegible]

3F8320 Acetylation of Epal 10 4EO/3PO

10118-18

lot 10000-58

Purpose:

To acetylate a 1-liter batch of Epal 10 4EO/ PO with acetic anhydride while purging the mixture with nitrogen. When reaction is complete, vacuum off the excess acetic acid.

Raw material Charges:

Epal 10 4EO/3PO (lot 10000-58)	- 496g
Acetic Anhydride (lot B54760)	- 102g

CALCULATIONS:

given: $\frac{56,100}{OH\#} = \frac{56,100}{113.2}(1) = \boxed{496g}$ Epal 10 4EO/3PO

$100\% \rightarrow (1.00)(102) = \boxed{102.00g}$ Acetic Anhydride

Procedure:

1) add 496 g Epal 10 4EO/3PO to 1L Rxn. flask
2) slowly add 102g acetic anhydride to flask
3) attach heating mantel, stirring apparatus, & N_2 purge to flask
4) heat flask to 120°C for 2 hrs. (w/ N_2 purge)
5) after cook time, run GC inj. on Rxn. & record results
6) if results unsatisfactory, ~~until~~ cyc cont. cook time & GC inj. until desired results achieved

Date June 26, 1996 Signature Chaineta U. Camp

Date July 2, 1996 Witness Philip Smith

→ cont. p. 19

SR8320 Acetylation

→ continued from pg. 10118-18

Procedure: (cont.)

7) after Rxn. complete (shown by GC inj.), cont. to purge w/ N_2 while vacuuming off the excess acetic acid
8) after 1 hr., run acid # on mixture & Record Results
9) if results unsatisfactory, cont. to vacuum (w/ N_2 purge) until desired results reached

SPECIFICATIONS:

- GC inj. btwn. 24-30% (shown by 4th set peaks)
- acid no. less than 5 (DV 12)

Results:

GC inj. (%)	Acid #	
1- * no calculation needed	1- 1.66	@ 2:50 PM
2- - Rxn. complete per R. VanDahm 6/27/96	2- —	@ —

*chromatographs found in PC on Project: DF-20 Quick Set Samples (6/27/96)

Conclusion:

Rxn. complete & all specs. were met

Date June 27, 1996 Signature Chintalal Campo

Date July 2, 1996 Witness Philip Smith

SF8320 OH# Analysis 10118-20

Epal 1Ø 4EO/3PO lot 10000-58

Purpose:

To analyze the hydroxyl values of the given sample product.

Apparatus:

1 - 250ml Erlenmeyer flask
2 - water-cooled condensers
3 - hot plate
4 - boiling beads
5 - analytical balance
6 - magnetic stirrer & stir bar
7 - 45ml burette

Reagents:

1 - Reagent~~s~~ grade Methanol (CYC)
2 - Phenolphthalein indicator
3 - 0.5N KOH (aqueous)

Raw Material Charges:

Pyridine (lot BJ902)	-	83g
Acetic Anhydride (lot B54760)	-	25.4g
Epal 1Ø 4EO/3PO (lot 10000-58)	-	2g

Procedure:

To run OH#'s on smpl., 1st need to make OH sol^N of pyridine/acetic anhydride:
1) mix 83g pyridine & 25.4g acetic anhydride in amber jar & mix for 15 min. (keep in hood)
→ OH sol^N should titrate btwn. 44-46 mls. KOH

Date June 27, 1996 Signature Chineta Y. Camp
Date July 2, 1996 Witness Philip Smith

→ cont. p. 21

→ continued from pg. 10118-20

Procedure: (cont.)

Blank Prep:

~~1) pipette 5ml pyrd./act.anhyd. sol^N/~~ CYC

~~1) add 0.5~~ CYC

~~1) add g smpl. to flask~~ CYC

1) pipette 5ml pyrd./act.anhyd. sol^N/ into flask & record weight
2) add 15ml pyrd. & 10ml d.i. water to flask and swirl to mix (then cap & set aside)
3) add stir bar & phenolphthalein indicator (4-6 drops) to flask
4) wash sides of flask w/ methanol, then titrate & record results

Sample Prep:

~~1) add g smpl. to flask~~ CYC

~~2) pipette 5ml p~~ CYC

1) add 2 g smpl. to flask
2) pipette 5ml OH sol^N/ into flask & record weight
3) add 15 ml pyrd. & 6-8 boiling beads to flask
4) place flask under condensers & place on hot plate (on high setting)
5) when boiling starts, cook for 1 hr.
6) after cook time, add 10ml d.i. water to condensers & cook another 10 min.
7) cool both flasks to room temp. & rinse w/ methanol down condensers
8) add stir bar & phenolphthalein indicator (4-6 drops) to flasks
9) titrate (occasionally washing flasks w/ methanol) & record results

Date June 27, 1996 Signature Chinetal Y. Camp

Date July 2, 1996 Witness Philip Sinh

→ cont. p. 22

SF8320 OH# Analysis 10118-22

→ continued from pg. 10118-21

Results: Trial #1 DV169

$SW = \frac{224}{\text{theo.OH}} = \frac{224}{112} = 2g$

smpl.	smpl.wt.	soln.wt.	CB	mls.KOH	Results
B	—	5.0630	—	46.60	—
1	2.0167	5.1096	47.03	39.26	108.07
2	2.0047	5.0423	46.41	38.66	108.44
				avg.	108.26

→ base (turned pink)

base # = 0.56

Conclusion:

the OH# of the given product was too low & will run another hydroxyl later

Bill's OH = 113.2
theo. OH = 112.0
my OH = 108.26
corr. OH = 108.26 − 0.56
= 107.7

⇒ CONT. on pg. 10118-23

Date June 27, 1996 Signature Shineta Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 OH# Analysis

→ continued from pg. 10118-22

Results: DV169 SW=2g

Trial #2

smpl.	smpl. wt.	soln. wt.	CB	mls. KOH	Results
B	—	5.0630	—	46.60	—
1	2.0026	5.0222	46.22	38.36	110.09
2	2.0008	5.0682	46.65	38.80	110.05
				avg.	110.07

→ base (turned pink)

base # = 0.56

Conclusion:

the OH# of the given product was within range & corrected

Bill's OH = 113.2
theo. OH = 112.0
my OH = 110.07
corr. OH = 110.07 − 0.56
= 109.51

Date June 27, 1996 Signature Shineta Y. Camp

Date July 2, 1996 Witness Philip Smith

SR604 MEHQ Analysis 10118-24

→continued from pg. 10118-17

Results: DV601 SW=1.0000g

*smpl. are ppm MEHQ in Diethylene ~~Glycol~~ Glycol

Trial #2

smpl. (ppm)	smpl. wt.	wavelength	absorbance	corr. conc.
1) 916 (920)*	1.0029	512.0	0.72440	917.3
2) 1008 (1010)	1.0144	512.5	0.76328	995.7
3) 1111 (1115)	1.0174	512.5	0.83713	1095.9
4) 1213 (1220)	1.0089	513.0	0.90264	1209.2

*ck. plot on graph to get # to divide by smpl. wt. to calc. the corrected concentration

Trial #3

smpl. (ppm)	smpl. wt.	wavelength	absorbance	corr. conc.
1) 916 (920)*	1.0172	512.5	0.72129	904.4
2) 1008 (1010)	1.0146	513.0	0.78973	995.5
3) 1111 (1115)	1.0063	513.5	0.90050	1108.0
4) 1213 (1220)	1.0103	514.5	1.0088	1207.6

Conclusion:

the MEHQ analysis is completed; all smpl. concentrations were within the given range

Date June 28, 1996 Signature Chineta Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Acid # 10118-25

lot 10118-18

Purpose:

To perform acid number testing on the given sample product

Apparatus:

* same items listed on pg. 10118-1

Reagents:

* same products listed on pg. 10118-1

Raw Material Charges:

Epal 10 4EO/3PO (lot 10118-18) - 3.5g
-mixed w/ milliken OSA F/A-100% 62% (lot 10023-52)

Procedure:

* same steps outlined on pg. 10118-1

Results: DV 12 SW = 3.5g

(4E/3P) 3.5174g (37.68) → AN = 300.48

Conclusion: specs. → low-290 / high-335

CYC ~~Rxn. h~~ 6/28 smpl. had acid # within Range of given specs.

Date June 28, 1996 Signature Chineta Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Cloud Pt. 10118-26

lot 10118-18

Procedure:

To determine the cloud point of the given sample. (5% in 6.8 N KOH)

Apparatus:

* same items listed on pg. 10118-5

Reagents:

* same products listed on pg. 10118-5

Raw Material Charges:

Epal 1Ø 4EO/3PO (lot 10118-18) - 5g
- mixed w/ Milliken OSA F/A - 100% 62g (lot 10023-52)

Potassium Hydroxide (lot J3599-Dewey) - 381g

Procedure:

KOH solⁿ/Prep: * same steps outlined on pp. 10118-5 & 10118-6

Cloud Pt. Procd: * same steps outlined on pg. 10118-6

Results: DV582 (5% in 6.8 N KOH)

(4E/3P) @ 65°C

Conclusion: specs. → low/54°C - high/64°C
cloud pt. was within given Range

Date June 28, 1996 Signature Christa M. Camp

Date July 2, 1996 Witness [illegible]

SF8320 Viscosity 10118-27

lot 10118-18

Procedure:

To determine the viscosity performance on the given sample product.

Apparatus:

* same items listed on pg. 10118-3

Reagents: NONE

Raw Material Charges:

Epal 1Ø 4EO/3PO (lot 10118-18) — 8g
- mixed w/ Milliken OSA F/A- 100%
62% (lot 10023-52)

Procedure:

* same step outlined on pg. 10118-3

Results: DV26 setting: 25°C, 20RPM & cP display

(4E/3P) → 730.0cP @ 30 min.

Conclusion: specs. → low-600cP / high-700cP

viscosity for given sample was within range of the given specs.

Date June 28, 1996 Signature Chineta Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Acid # 10118-28

lot 10118-18

Perform acid # on given smpl. product.

→ used same apparatus items listed on pg. 10118-1

→ used Reagents listed on pg. 10118-1

Raw Material Charges:

Epal 1Ø 4EO/3PO (lot 10118-18) — 3.5g
— mixed w/ Milliken OSA F/A-100% 60% (lot 10023-52)

→ same procedure steps outlined on pg. 10118-1

Results: DV12

(4E/3P) 3.5178 (36.42) → AN=290.40

Conclusion: specs. → low 290/high 335

acid # for given sample was within the spec. Range

Date June 28, 1996 Signature Chinita Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Cloud Pt. 10118-29

lot 10118-18

Determine the cloud pt. of 5% of the given smpl. in 6.8 N KOH solⁿ.

→ same apparatus items listed on pg. 10118-5

→ same Reagents listed on pg. 10118-5

Raw Material Charges:

Epal 1Ø 4EO/3PO (lot 10118-18) — 5g
— mixed w/ Milliken OSA F/A-100% 60% (lot 10023-52)

Potassium Hydroxide (lot J3599) — 381g
— Dewey

→ same procedure steps outlined on pp. 10118-5 & 10118-6
* for KOH solⁿ preparation

on pg. 10118-6
* for cloud pt. procedure

Results: DV582 (5% in 6.8 N KOH)

(4E/3P) @ 58 °C

Conclusion: specs. → low 54°C / high 64°C

cloud pt. for given sample was within the spec. Range

Date June 28, 1996 Signature Chineta Y. Camp

Date July 2, 1996 Witness [illegible]

SF8320 Viscosity lot 10118-18 10118-30

Determine the viscosity performance of the given smpl.

→ same apparatus items listed on pg. 10118-3

→ NO reagents were used

Raw Material Charges:

Epal 1Ø 4EO/3PO (lot 10118-18) — 8g
- mixed w/ Milliken OSA F/A - 100% 60% (lot 10023-52)

→ same procedure steps outlined on pg. 10118-3

Results: DV26 settings: 20 RPM, 25°C & cP display

(4E/3P) → 660CP @ 30 min.

Conclusion: specs. → low 600cP/high 700cP viscosity for given smpl. was

CYC 6/28 ~~July~~ June

Date ~~July~~ 28, 1996 Signature Chineta Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Hydrolyzation of OSA 10118-31
(in Epal 10 4EO/3PO) lot 10118-18

Hydrolyze the OSA ~~free acid~~ cyc after it has already been mixed w/ the acetyl. alcohol.

→ same apparatus items listed on pg. 10118-8

→ NO reagents were used

Raw Material Charges:

Epal 10 4EO/3PO (lot 10118-18) – 38g

OSA-NJC (lot TRL-3/Dewey) – 57.1g
stock# 680085
& d.i. water – 4.9g

→ same procedure steps outlined on pp. 10118-8 & 10118-9

Results:

Rxn. is complete & OSA has been completely hydrolyzed

Conclusion:

OSA has been completely hydrolyzed & is now 100% free acid and mixed thoroughly w/ the acetylated alcohol

Date ~~June~~ July 1, 1996 Signature Christopher Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Acid # 10118-32

lot 10118-31

Perform acid # on given smpl. product.

→ same apparatus listed on pg. 10118-1

→ same Reagents listed on pg. 10118-1

Raw Material Charges: Epal 1Ø 4EO/3PO (lot 10118-31) w/ hydrolyzed OSA in alcohol – 3.5g

→ same procedure outlined on pg. 10118-1

Results: DY 12

(4E/3P) 3.5207 (36.98) → AN = 294.63

Conclusion: specs. → low 290 / high 335

the acid # for the given smpl. product was within Range

Date July 1, 1996 Signature Shinata Y. Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Cloud Pt. 10118-33

lot 10118-31

Determine the cloud point of 5% of the smpl. product in 6.8 N KOH solⁿ/.

→ same apparatus items listed on pg. 10118-5

→ same reagents listed on pg. 10118-5

Raw Material Charges:

Epal 10 4EO/3PO (lot 10118-31) w/ hydrolyzed OSA in alcohol — 5g

Potassium Hydroxide Dewey/ (lot J3599) — 381g

→ same procedure outlined on pp. 10118-5 & 10118-6 (KOH solⁿ/ prep.) and pg. 10118-6 (cloud pt. procd.)

Results: DV582 (5% in 6.8 N KOH)

(4E/3P) @ 57 °C

Conclusion: specs. → low 54°C / high 64°C

the cloud pt. for the given smpl. product was within range

Date July 1, 1996 Signature Christally Camp

Date July 2, 1996 Witness Philip Smith

SF8320 Viscosity 10118-34

lot 10118-31

Determine the viscosity performance of the given smpl.

→ apparatus items listed on pg. 10118-3

→ NO Reagents were used

Raw material Charges: Epal 10 4EO/3PO (lot 10118-31) w/hydrolyzed OSA in alcohol — 8g

→ procedure outlined on pg. 10118-3

Results: DV26 settings: 20 RPM, 25°C & cP display

(4E/3P) → 700.0 cP @ 30 min.

Conclusion: specs. → low 600/high 700

viscosity for given smpl. was within Range

Date July 1, 1996 Signature Chinata Y. Camp

Date July 2, 1996 Witness Philip Smith

HHPA Analysis

Neutralization

lot K504731-A-1

10118-35

CYC ~~Procedure:~~ Purpose:

To hydrolyze the given anhydride w/ the addition of an excess amnt. of base & then to back titrate w/ acid to determine the amnt. caustic required to neutralize the anhydride. (neutralization no.)

Apparatus:

1 - 250 ml Erlenmeyer flask w/ stopper
2 - 50 ml burette (self zeroing)
3 - magnetic stirrer & stir bar
4 - 50 ml graduated cylinder

Reagents:

1 - Reagent grade acetone
2 - Phenolphthalein indicator
3 - 0.5N KOH (aqueous)
4 - 0.5N HCl (aqueous)

Raw material Charges:

*NOTE: use 1g for of smpl. HHPA products

CYC 7/1

HHPA-BK (lot K504731-A-1) — ~~2g~~ 1g

Procedure: * RUN IN DUPLICATES

CYC 7/1

1) add ~~2g~~ 1g of smpl. to Erlenmeyer flask & record wt.
2) add 50 ml acetone & 50 ml KOH to flask & replace stopper
3) stir at room temp. for 15 min.
4) back titrate w/ HCl & phenolphthalein indicator, using 50 ml self-fill burette
5) record burette reading & calc. neut. no.

Date July 1, 1996 Signature Chiquita Y. Camp

Date July 2, 1996 Witness Philip Smith

→ cont. p. 36

HHPA Neutralization 10118-36
Analysis

→ continued from pg. 10118-35

CALCULATIONS: (given eq^N/)

$$\frac{(\text{ml KOH} \times N) - (\text{ml HCl} \times N) \times 56.1}{\text{sample weight}} = \text{neut. no.}$$

Results: DV-TI204

1st set

prod.	lot #	smpl. wt.	mls. HCl	neut. no.
HHPA-BK	K504731-A-1	(1) ~~2.0136~~ 1.0093	24.7	703.13
		(2) ~~2.0178~~ 1.0169	24.1	714.42

QC 7/2

(Range 720 ± 10)

Conclusion: specs. → low 710 / high 730

the neut. no. for the given smpl. was within the Range of specs. for trial #2, but was below the Range for trial #1

Results: DV-TI204

2nd set

prod.	lot #	smpl. wt.	mls. HCl	neut. no.
HHPA-BK	K504731-A-1	(1) 1.0216	23.9	716.63
		(2) 1.0239	24.0	712.28

Conclusion:

the neut. no. for smpl. was within Range on both trials

Date July 2, 1996 Signature Christaly Camp

Date July 2, 1996 Witness Philip Smith

HHPA Solidification 10118-37
Analysis lot K504731-A-1

Purpose:

To find the temp. at which the given smpl. completely solidifies.

Apparatus:

1 - med. test tube
2 - thermometer

Reagents: NONE!

Raw material Charges:

HHPA-BK (lot K504731-A-1) — amnt. may vary

Procedure:

1 - fill test tube about 1/2 full w/ smpl.
2 - stir smpl. in t.t. w/ thermometer until smpl. completely gels (this is known as solidification)
3 - Record the temp. at gel point

Results: specs. → 35°C (min.)

HHPA-BK solidification point is 36°C

Conclusion:

the solidification pt. is within the spec. Range

Date July 2, 1996 Signature Chineta Y. Camp
Date July 2, 1996 Witness Philip Smith

HHPA Analysis

10118-38

Product: HHPA-BK lot#: K504731-A-1

I) APHA Color (std. method on molten material) DV30

→ material was too hazy to Read

II) Clarity @ 60°C DV599

→ material should have no haze at a temp. of 60°C, but it did proving that the material contains "free acid"

Date July 2, 1996

Date July 2, 1996

Signature Chineta U. Camp

Witness Philip Smith

SF8320 Hydrolyzation of OSA 10118-39
(in Epal 1ø 4EO/3PO) lot 10118-18

VOID

Date July 8, 1996
Date July 8, 1996

Signature Chineta Y. Camp
Witness Rick Rodz

SF8320 Acetylation of Epal 1Ø 4EO/3PO lot 10000-58

I) Acetylate a batch of Epal 1Ø w/ acetic anhydride (while purging w/ N_2). Then vacuum off excess acetic acid. (3 x's)

II) Epal 1Ø 4EO/3PO (lot 10000-58) — 248 g
Acetic Anhydride (lot B547160) — 51 g
→ NOTE: 1/2 of original charges (found p.10118-18)

III) procedure same as on pg. 10118-18 & 10118-19
→ same specs. on pg. 10118-19

Results: *chromatographs found in PC on Project: DF-20 Quick Set Samples (7/8/96)

GC inj. (%)	RXN. 1	Acid # (DV12)
1 - no calculations;		1 - 3.08 @ 9:40A (7/9)
2 - Rxn. complete per R. VanDahm (7/8/96)		2 - ___ @ ___

* ——— *

GC inj. (%)	RXN. 2	Acid # (DV12)
1 - no calculations;		1 - 1.38 @ 2.35P (7/9)
2 - Rxn. complete per R. VanDahm (7/8/96)		2 - ___ @ ___

* ——— *

GC inj. (%)	RXN. 3	Acid # (DV12)
1 - no calculations;		1 - 6.55 @ 1:20P (7/9)
2 - Rxn. complete per R. VanDahm (7/8/96)		2 - 3.48 @ 2:25P (7/9)

Date July 8, 1996 Signature Chineta Y. Camp
Date July 18, 1996 Witness [illegible]

→ cont. p.41

SF8320 Acetylation

→ continued from pg. 10118-40

SPECIFICATIONS:

→ GC inj. btwn. 24-30% (shown by 4th set of peaks)
→ Acid no. less than 5 (DV12)

Conclusion:

Rxns. are complete & all specs. were met

Date July 8, 1996 Signature Chineta Y. Camp

Date July 18, 1996 Witness Rick Rehg

SF8320 Hydrolyzation of OSA 10118-42
(in Epal 10 4EO/3PO) lot 10118-40

I) Hydrolyzed the OSA after it has already been mixed w/ the acetylated alcohol. (3x's)

II) Epal 10 4EO/3PO (lot 10118-40) — 38 g

OSA-NJC / Dewey (lot TRL-3) stock # 680085 — 57.1 g

d.i. water — 4.9 g

III) apparatus same as on pg. 10118-8

IV) NO Reagents!

V) procedure same as on pg. 10118-8 & 10118-9

Results:

Rxn. is complete & OSA has been hydrolyzed

Conclusion:

OSA has been completely hydrolyzed & is now 100% free acid and mixed thoroughly w/ the acetylated alcohol

Date July 8, 1996 Signature Chinetaly Camp

Date July 18, 1996 Witness Rick Rib

Nonyl Phenol 2.5PO — Product Analysis 10118-43

lot 10000-61

I) Perform various testing on the given product.

~~II) Epal 10 4EO/3PO~~ CYC 7/8

II) Nonylphenol 2.5PO (lot 10000-61) — varied amnt.

Pyridine (lot 74506/1) — " "

Acetic Anhydride (lot B54760) — " "

III) PROCEDURE:

(A) Color → Gardner color (DV31)

(B) Base # → 1) add 10g of smpl. to 100ml Methanol
2) stir until dissolved completely
3) add phenolphthalein indicator
4) solⁿ should turn pink – Run base # (DV12)

CYC 7/8 ~~NOTE: if stays clear – Run acid #~~

(C) OH # → steps outlined on pg. 10118-20 & 10118-21 (DV169)

(D) % water → Aqua tester (DV13)

(E) % solids → 1) add 0.5g of smpl. to foil pan
2) Record wt. of smpl. & foil pan separately
3) heat in 113°C oven for 1/2 hr.
4) Record wt. of foil pan (w/ smpl.) & do calculations

*NOTE: eqⁿ ⟹ (gross pan wt. – empty pan wt.) / smpl. wt. × 100 (DV1)

Date July 8, 1996 — Signature Chineta Y. Camp

Date July 18, 1996 — Witness [illegible]

→ cont. p. 44

Nonyl Phenol Analysis 10118-44

2.5 PO → continued from pg. 10118-43

Results:

(A) DV31 → 2 Gardner color

(B) DV12 → 0.73 (pink, Run base#) $\left[\frac{(1.30)(0.1)(56.1)}{10.0085}\right]$

(C) DV169 → SW = 1.5g theo. OH = 154
Bill's OH = 153.8

Trial #1

Smpl.	smpl. wt.	soln. wt.	CB	mls. KOH	Results
B	—	5.0923	—	46.56	—
1	1.5018	5.0793	46.44	38.04	156.89
2	1.5131	5.0276	45.97	37.60	155.16

Trial #2

Smpl.	smpl. wt.	soln. wt.	CB	mls. KOH	Results
B	—	5.0923	—	46.56	—
3	1.5107	5.0855	46.50	38.06	156.71
4	1.5076	5.0449	46.13	37.74	156.10

⇒ CORR. OH = 156.22 − 0.73 = 155.49 (subt. b/c base) avg. of 1,2,3,4 > 156.22 my OH

(D) DV13 → 0.149 % Aquatest (for % water)

(E) DV1 → SW = 0.5g

Trial #1

	empty pan wt.	smpl. wt.	gross pan wt.	% solids
1	1.0858	0.5099	1.5916	99.20
2	1.0673	0.5019	1.5653	99.22

Trial #2

	empty pan wt.	smpl. wt.	gross pan wt.	% solids
3	1.0781	0.5068	1.5822	99.47
4	1.0777	0.5002	1.5748	99.38

avg. of 1,2,3,4 → 99.32

Date July 8, 1996 Signature Christaly Camp

Date July 18, 1996 Witness Rick Rich

→ cont. p. 45

NonylPhenol 2.5PO Analysis 10118-45

→ continued from pg. 10118-44

NO SPECIFICATIONS given!

CALCULATIONS: (for % solids)

eq^N → (gross − empty) % smpl. wt. ×100 = % solids

Conclusion:

all tests performed ~~were~~ CYC gave results that were normal for the given product

NOTE: a 1-qt. smpl. was sent to Sartomer Co., Oaklands Corporate Cntr., 502 Thomas Jones Way, Exton, PA 19341, attn: Tom Hazell

mailed sample on 7/16/96

was assigned my NB # (10118-44) but made from Bill's NB # (10000-61)

Date July 8, 1996 Signature Chineta Y. Camp

Date July 18, 1996 Witness Rick Rib

SF8320 Property Testing 10118-46
(Epal 10 4EO/3PO) RXN. ~~#~~ 1 lot 10118-42
cyc

I) Perform an acid #, determine the cloud pt. in 6.8N KOH sol^N/, & establish the viscosity of the given smpl.

II) Epal 10 4EO/3PO (lot 10118-42) w/ hydrolyzed OSA in alcohol — varied amounts

Potassium Hydroxide Dewey / (lot J3599) — 381g

III) Acid # (DV12) procedure outlined pg. 10118-1

Cloud Pt. (DV582) KOH sol^N/ prep. outlined pg. 10118-5 & 10118-6
cloud pt. procedure outlined pg. 10118-6

cyc ~~Viso~~ Viscosity (DV26) procedure outlined pg. 10118-3

Results: for RXN. 1

Acid # → 3.5032 (36.48) / AN = 292.09
specs. — low 290 / high 335
Cloud pt. → 5% in 6.8N KOH @ 58 °C
specs. — low 54°C / high 64°C
Viscosity → 20RPM, 25°C, cP, 30 min → 712.5 cP
specs. — low 600 / high 700

Conclusion:

the acid # & cloud pt. were within given Range, but viscosity was slightly higher than given range

Date July 10, 1996 Signature Christeta Y. Camp
Date July 18, 1996 Witness [illegible]

→ cont. p. 47

→ continued from pg. 10118-46

RXN. 2

Results:

Acid # → 3.5196 (36.20) / AN = 288.50
Cloud pt. → @ 54 °C
Viscosity → 345.0 cP

Conclusion:

the acid # & viscosity were lower than given range, but the cloud pt. was within the given range > cook another 3 hrs. @ 90°C & run tests again (cont. on pg. 48) →

RXN. 3

Results:

Acid # → ~~3~~ 4 5.0290 (49.38) / AN = 275.42
Cloud pt. → @ 59 °C
Viscosity → 545.0 cP

Conclusion:

the acid # & viscosity were lower than given range, but the cloud pt. was within the given range > cook another 3 hrs. @ 90°C & run tests again (cont. on pg. 48) →

Date July 10, 1996 Signature Chineta Y. Camp
Date July 15, 1996 Witness [illegible]

SF8320 Properties 10118-48

→ continued from pg. 10118-47

RXN. 2

Results:

Acid # → ~~3.5~~ 4.5230 (43.36) / AN = 268.90 (cyc)
Cloud pt. → @ 56 °C
Viscosity → 462.5 cP

Conclusion:

the acid # & viscosity were lower than given Range, but the cloud pt. was within the given Range

RXN. 3

Results:

Acid # → 4.5207 (44.52) / AN = 276.24
Cloud pt. → 58 °C
Viscosity → 665.0 cP

Conclusion:

~~the acid # &~~ (cyc)
the acid # was lower than the given Range, but the cloud pt. & viscosity was within given Range

Date July 11, 1996 Signature Chinota Y. Camp
Date July 18, 1996 Witness [illegible]

SF8320 Acetylation of Epal 10 4EO/3PO lot 10000-67

10118-49

I) Acetylate a batch of Epal 10 w/ acetic anhydride (while purging w/ N_2). Then vaculum off excess acetic acid.

II) Epal 10 4EO/3PO (lot 10000-67) — 514g
Acetic Anhydride (lot B547160) — 102g

calc. $\frac{56,100}{OH\#} = \frac{56,100}{109.2} = 514g$ Epal 10

100% → (1.00)(102) = 102g Act. Anhyd.

III) procedure outlined on pg. 10118-18 & 10118-19
→ same specs. as on pg. 10118-19

Results: *chromatographs found in PC: Project DF-20 Quick Set Samples (7/12/96)

GC inj. (%) | Acid #

1 – no calculations; Rxn. complete per RAV (7/12) | 1 – 1.39 @ 9:30A (7/14)

—*—

~~NOTE: Run acid #'s on Rxn. throughout cook time to determine completion~~

~~Acid #'s (before heating) w/ N_2 *while stirring~~

~~1~~
~~2~~

*all corrections by CYC (7/12)

Conclusion:
Rxn. is complete & all specs. were met

~~→ cont. p.50~~ NOTE: ~~save for~~ later use

Date July 12, 1996 Signature Chineta Y. Camp
Date July 18, 1996 Witness [illegible]

SF8320 Acetylation

→ continued from pg. 10118-49

Results: (cont.)

*while stirring

Acid#'s (while heating)* not on temp. (w/N2)

1-
2-

Acid#'s (while heating)* on temp. (w/N2)

1-
2-
3-
4-
5-
6-
7-
8-
9-
10-
11-
12-

VOIDED

Date July 12, 1996

Date July 18, 1996

Signature Chineta Y. Camp

Witness Rick Leb

SF8320 OH# Analysis 10118-51

lot 10000-67

I) Analyze hydroxyl values of given smpl.

II) apparatus on pg. 10118-20

III) Reagents on pg. 10118-20

Raw material Charges:

Pyridine (lot BJ902) — 83 g
Acetic Anhydride (lot B547160) — 25.4 g
Epal 1Ø 4EO/3PO (lot 10000-67) — 2 g

~~Procedure:~~ CYC

IV) procedure on pg. 10118-20 & 10118-21

Results: [DV169] $SW = \frac{224}{\text{theo. OH}} = \frac{224}{112} = 2g$

smpl.	smpl.wt.	soln.wt.	CB	mls. KOH	Results
B	—	5.0334	—	46.14	—
1	2.0085	5.0752	46.52	38.92	106.14
2	2.0159	5.0459	46.25	38.52	107.56
3	2.0162	5.0591	46.38	38.72	106.57
4	2.0042	5.0628	46.41	38.80	106.51

avg. [106.7]

base# [DV12] (turned pink)

$\frac{(0.25)(0.1)(56.1)}{5.0218} = 0.3$ } avg. OH − base# = CORR. OH
106.7 − 0.3 = [106.4]

Conclusion:

OH# was normal for given smpl.

theo. OH = 112
Bill's OH = 109.2
my OH = 106.7
CORR. OH = 106.4

Date July 16, 1996 Signature Chinatary Camp

Date 9/18/1996 Witness [illegible]

SF8320 Acetylation of Epal 10 4EO/3PO lot 10000-67

10118-52

I) Acetylate a batch of Epal 10 4EO/3PO w/ acetic anhydride (while purging w/ N_2). Run neutralization numbers during cook time to determine completion of the rxn. Then vacuum off the excess acetic acid.

II) Raw material charges on pg. 10118-49

III) procedure on pg. 10118-18 & 10118-19, except run neut. #'s at set time intervals until rxn. complete
→ same specs. on pg. 10118-19

Results: DV204

time (min.)	smpl. wt.	mls. HCl	neut. no.
10	2.0062	37.3	521.52
20	2.0018	37.5	525.46
40	2.0061	37.5	524.34
65	2.0308	37.2	513.82
95	2.0527	37.1	506.97
120	2.0195	37.0	513.91
135	2.0518	38.2	522.23
155	2.0110	38.6	538.40
175	2.0399	39.1	537.65
195	2.0639	39.7	539.55

neut. no. is stabilized; rxn. complete

Acid# (DV12)

$1 - 2.0256(0.12) = \boxed{1.66}$ @ 2:20P (7/16)

Conclusion:
Rxn. complete & all specs. were met

Date July 16, 1996 Signature Chanetta Y. Camp

Date July 18, 1996 Witness Rickles

SF8320 Hydrolyzation of OSA 10118-53
(in Epal 10 4EO/3PO) lot 10118-52

I) Hydrolyze the OSA after it has already been mixed w/ the acetylated alcohol.

II) Epal 10 4EO/3PO (lot 10118-52) — 38g

OSA-NJC/Dewey (lot TRL-3) stock# 680085 — 57.1g

Deionized water — 4.9g

III) apparatus on pg. 10118-8

IV) NO Reagents!

V) procedure on pg. 10118-8 & 10118-9

Results:

Rxn. is complete & OSA has been hydrolyzed

Conclusion:

OSA has been completely hydrolyzed & is now 100% free acid and mixed thoroughly w/ the acetylated alcohol

Date July 17, 1996 Signature Chineta Y. Camp

Date July 18, 1996 Witness [illegible]

SF8320 CYC ~~Perfo~~ Properties Testing 10118-54
(of Epal 10 4EO/3PO) lot 10118-53

I) Perform various properties tests to determine if given smpl. is within Specs. Range.

II) Epal 10 4EO/3PO (lot 10118-53) w/ hydrolyzed OSA — varied amounts

Potassium Hydroxide (lot J3599) / Dewey — 381g

III) Acid # (DV12) procedure on pg. 10118-1

Cloud pt. (DV582) @ KOH sol^N prep. on pg. 10118-5 & 10118-6
procedure on pg. 10118-6

Viscosity (DV26) procedure on pg. 10118-3

Results:

Acid # → 3.5058 (36.02) / AN = 288.20
Cloud pt. → (5% in 6.8N KOH) @ 57°C
Viscosity → 20 RPM, 25°C, cP, 30 min. / 860.0 cP

NOTES: specs. > acid # — low 290 / high 335
cloud pt. — low 54°C / high 64°C
viscosity — low 600cP / high 700cP

Conclusion:

acid # was slightly below spec. Range; cloud pt. was within spec. Range; & viscosity was greatly over spec. Range

Date July 18, 1996 Signature Chineta Y. Camp
Date July 18, 1996 Witness [illegible]

Palmer-based Inks [DV-VI506.R2] Washability testing

10118-55

*diff. lot#'s

Purpose: Fabric fugitivity (washability) of 100% Palmer-based inks (standards) on swatches (100% cotton & 50/50% poly-cotton)
*test 5 swatches (of ea. 100 & 50/50) for each color

I. Ink Preparation

Materials:
1 - Palmer colorant (std. smpl.) *no older than 6 mnths.
2 - 4 oz. jars (w/ lids)
3 - PVP K-15 (polyvinylpyrrolidone) lot 90408 (GAF)
4 - Poly (ethylene glycol) MW 200 lot 05911MX (Aldrich)
5 - Glycerin
6 - Deionized water
7 - Liquid 15 lot T1032 / Dewey
8 - Labels
9 - Diethylene glycol lot K9525 (QC) / Dewey
10 - Top loading balance

Procedure:
1) make up 50g of each color using the given amounts:

Color (lot#)	Colorant (g)	Glycerin (g)	DI water (g)
Yellow Q1061	10	7.5	32.5
Blue T1081	18.5	7.5	24
Orange S1151	15	7.5	27.5
Scarlett T1136	20	7.5	22.5
Magenta T1124 *	—	—	—
Black PP001	32.5	7.5	10
Yellow YB J1088	5	7.5	37.5
FL Red T1163 *	—	—	—

*magenta/colorant - 14.25
glycerin - 7.5
DI water - 23.25
liquid 15 - 5

*FL Red/colorant - 12
diethylene glycol - 2
poly(ethylene glycol) - 7
PVP K15 - 1
DI water - 28

Date July 17, 1996 Signature Chineta L. Camp

Date July 23, 1996 Witness Lm Chavis

→ Cont. p. 56

Palmer Inks Washability 10118-56

→ continued from pg. 10118-55

Procedure: (cont.)

2) Combine in 4oz. jars, Replace lids, & shake for 2 min.
NOTE: for FL Red, add PVP K-15 last & shake for 5 min.
3) label each jar by color name
~~4) R~~ cyc

II. Ink Application

Materials:

1 - 100% cotton swatches (fabric 419)
2 - 50/50% poly-cotton swatches (fabric 9406)
3 - permanent marking pen
4 - embroidery hoops
5 - 1ml syringes
6 - plastic transfer pipettes

Procedure:

1) label each swatch w/ marker (color, fabric, lot#)
NOTE: use 5 swatches of 100% & 5 swatches of 50/50% for each color
2) mount each swatch in an embroidery hoop
3) fill 1ml syringe w/ 0.6ml of ink (prepared in section I)
4) deposit ink in droplets in circular motion without placing them on top one another, at least 1 inch from edge of hoop (as quickly as possible)
5) use head of plastic pipette to spread ink evenly over swatch
6) allow swatch to air dry for 4 hrs.
7) after drying, wash swatch according to laundering procedure
8) Repeat steps 1-7 for each color

→ cont. p. 57

Date July 18, 1996 Signature Chineta Y. Camp
Date July 23, 1996 Witness Kim Chavis

Palmer Inks Washability 10118-57

→ continued from pg. 10118-56

III. Swatch Laundering

Materials:

1 – Swatches (prepared in section II)
2 – hand towels
3 – stapler
4 – Tide detergent
5 – 2L beaker

Procedure:

1) Using 2L beaker w/ water (at 105°F) continually flowing into it, places swatches between fingers & Rinse (one at a time) in 3 10-sec. intervals (emptying the beaker after each interval)
2) attach swatches to the towels using the stapler
   NOTE: put same color swatches (100 & 50/50) on 1 towel ea.
3) add 100g (1 scoop) of Tide to washing machine
   NOTE: use the following settings:

settings	KENMORE heavy duty (60 series)	KENMORE (70 series)
Wash	hot	hot
Rinse	cold	cold
water level	high	medium
wash cycle	cotton – medium soil	12 min.

   automatic temp. control (Model 242-A or 242-BM)
   MANUAL – off / WASH – 105°F / RINSE – cold
4) allow washer to agitate some & fill halfway before ~~add~~ putting in the towels w/ the swatches
5) after wash cycle, place load into dryer
   NOTE: use the following settings:
   FABRIC CARE – high (cotton/sturdy) / TIME – 30 min.
6) after drying, Remove swatches from towels & iron & trim them (separate by each color)
7) using piece of white paper as a background, compare these std. swatches w/ the std. swatches in Kim's book & Rate them
   NOTE: Rating scale 1-5 (5-worst & 1-best)

Date July 19, 1996 Signature Chinota Y. Camp

Date July 23, 1996 Witness Kim Charles

→ cont. p. 58

Palmer Inks Washability

→ continued from pg. 10118-57

IV. Results

SPECS. > Ratings
Ø (best) – 5 (worst)

COLOR	RATINGS (100%)	(compared w/ previous std. swatches) (50/50%)
Yellow	1	1
Blue	1	1
Orange	1	1
Scarlet	2	2
Magenta	2	1
Black	1	1
Yellow YB	Ø	Ø
FL Red	Ø	Ø

V. Conclusion

NOTE: Save results for later use per M. Valenti (7/19/96)

— D. Clearer wants us to hold onto these swatches until further notice from him on what to do with them (7/22/96)

Date July 19, 1996 Signature Chineta Y. Camp

Date July 23, 1996 Witness Lina Chavis

SF8320 OH# Analysis 10118-59

lot 10000-73

I. PURPOSE

Analyze the hydroxyl values of the given sample.

II. APPARATUS

found on pg. 10118-20

theo. OH = 112
Bill's OH = 117.1
my OH (avg.) = 111.9
corr. OH = 111.6

III. REAGENTS

found on pg. 10118-20

IV. RAW MATERIAL CHARGES

Epal 1Ø 4EO/3PO (lot 10000-73) — 2g
cyc ~~Pr~~ Pyridine (lot 02657)/Aldrich — 83g
Acetic Anhydride (lot B54760)/Acros — 25.4g

V. PROCEDURE

found on pp. 10118-20 & 10118-21

VI. RESULTS

DV169

smpl.	smpl. wt.	soln. wt.	CB	mls. KOH	Results
B	—	5.0524	—	46.26	—
1	2.0093	5.0910	46.61	38.54	112.66
2	2.0123	5.0757	46.47	38.48	111.37
3	2.0147	5.0616	46.34	38.36	111.10
4	2.0042	5.0362	46.11	38.08	112.38
					avg. 111.88

DV12

base # (turned pink) = $\frac{(0.25)(0.1)(56.1)}{5.0011} = 0.28$ } corr. OH = avg. OH − base # (subt. when base)

VII. CONCLUSION

OH# was normal for given sample

Date July 19, 1996 Signature Chineta Y. Campo

Date July 23, 1996 Witness Lisa Chavis

SF8320 Acetylation of Epal 10 4EO/3PO lot 10000-73

10118-60

I. PURPOSE

Acetylate a batch of Epal 10 w/ acetic anhydride (while purging w/ N_2). Run acid #'s during cook time to determine completion of Rxn. Then vacuum off the excess acetic acid.

II. APPARATUS

1 – 1 liter Rxn. flask
2 – heating mantle
3 – stirring apparatus
4 – thermometer
5 – hose (w/ N_2)

III. REAGENTS

1 – 50/50 Methanol/Water

IV. RAW MATERIAL CHARGES

Epal 10 4EO/3PO – 503g (lot 10000-73)
Acetic Anhydride – 102g (lot B54760)

calc.
$\frac{56,100}{111.6} = 503g$
$(1.00)(102) = 102g$

V. PROCEDURE

found on pg. 10118-18 & 10118-19, except Run acid #'s at set time intervals until Rxn. complete NOTE: same specs. on pg. 10118-19

EXCEPT Run acid #'s at different time intervals until Rxn. complete

VI. RESULTS

DV12

	time (min.)	smpl. wt.	mls. KOH	Acid #
before heating	15 min.	2.0158	7.74	107.70
after heating	15	2.0149	6.76	94.11
	30	2.0855	6.20	83.39
	60	2.0096	5.82	81.24
	90	2.0089	5.62	78.47
	130	2.0191	5.60	77.80
	180	2.0268	5.58	77.22

acid # is stabilized; Rxn. complete

SPECS: (during Rxn.) start – 185 / finish – 93 (after Rxn.) less than 5

Acid # (DV12)
1 – 2.0242(0.16) = 2.22 @ 10:45A (7/23)

VII. CONCLUSION

Rxn. complete & all specs met, though acid #'s were a little low

Date July 22, 1996 Signature Chineta Y. Camp
Date July 23, 1996 Witness Lisa Chavis

NOTE: save for later use!

SF8320 Acid Numbers 10118-61

(Epal 1Ø 4EO/3PO) lot 10000-73

I. PURPOSE Refine acid number method for acetylation reaction for given sample.

II. APPARATUS

1 – 250ml Erlenmeyer flask
2 – water-cooled condensers
3 – hot plate
4 – boiling beads
5 – 45ml burette
6 – magnetic stirrer & stirbar
7 – bucket of ice

III. REAGENTS

1 – Phenolphthalein indicator

IV. RAW MATERIAL CHARGES

1 – Epal 1Ø 4EO/3PO (lot 10000-73)* alcohol — 5g
2 – Acetic Anhydride (lot B54760) — 1g
3 – Deionized Water — 100ml

V. PROCEDURE

(A) 1 – dissolve 5g alcohol & 1g acetic anhydride in 100ml d.i. water
2 – wait 15 min. before titrating

(B) 1 – dissolve 5g alcohol & 1g acetic anhydride in 100ml d.i. water
2 – heat to reflux, remove & let cool, before titrating

(C) 1 – dissolve 5g alcohol & 1g acetic anhydride in 100ml d.i. water
2 – heat at reflux for 10 min., remove & let cool, before titrating

VI. RESULTS DV12 SPECS. > theo. acid # = 183

	alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
(A)	5.0171	1.0019	6.0190	38.28	178.39
(B)	5.0060	1.0418	6.0478	39.62	183.76
(C)	5.0093	1.0038	6.0131	38.34	178.85

VII. CONCLUSION Procedure (B) was the only one to produce acid # within range

Date July 23, 1996 Signature Chineta Y. Camp

Date July 24, 1996 Witness [illegible]

→ CONT. p. 62

SF8320 Acid # 10118-62

→ continued from pg. 10118-61

RESULTS: (DV12) SPECS.> theo. acid # = 183

Trial #1 > * heat to Reflux before titrating

alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
1- 5.0162	1.0421	6.0583	39.58	183.26
2- 5.0283	1.0825	6.1108	41.24	189.30
3- 5.0161	1.1164	6.1325	42.82	195.86
4- 5.0182	1.0129	6.0311	38.52	179.15

Conclusion: smpl. 1 was only one within Range

Trial #2 > * heat at Reflux 10 min. before titrating

alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
1- 5.0152	1.0084	6.0236	36.30	169.04
2- 5.0800	1.0026	6.0826	36.08	166.38
3- 5.0089	1.0197	6.0286	36.86	171.50
4- 5.0002	1.0020	6.0022	36.18	169.08

Conclusion: no smpl. within Range

Trial #3 > * heat at Reflux 5 min. before titrating

alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
1- 5.0142	1.0041	6.0183	38.16	177.86
2- 5.0216	1.0183	6.0399	38.70	179.73
3- 5.0197	1.0088	6.0285	38.50	179.14
4- 5.0007	1.0350	6.0357	39.80	184.96

Conclusion: smpl. 4 was only one closest to given Range

Date July 23, 1996 Signature Chineta Y. Camp

Date July 26, 1996 Witness Rick Lutz

Acid Dyes Comparisions (by Draw-downs) *diff. lot#'s

10118-63

I. PURPOSE

To determine if dyes from Dri-Mark are as strong (or stronger) than those from Crayola and Milliken.

II. APPARATUS

1 – 8½×11½ plain, white paper
2 – draw-down surface (plexi-glass)
3 – steel bar
4 – tape
5 – pipette
6 – spatula
7 – analytical balance
8 – 4oz. smpl. vials w/ lids

III. REAGENTS — NONE!

IV. RAW MATERIAL CHARGES

1 – Glycerin		— 1.5g
2 – Deionized water		— amnt. varies
3 – Dri-mark Blue (no lot#)		— " "
4 – Dri-mark Violet (no lot#)		— " "
5 – Crayola Acid Blue #9 (no lot#)		— " "
6 – Crayola Acid Violet #17 (no lot#)		— " "
7 – Liquitint WRG Blue (lot P1068)/st.#710295	MILLIKEN	— " "
8 – Access Violet 1967 (lot 10108-44)/KWC-NB	MILLIKEN	— " "

V. PROCEDURE

1) make up smpl. colors at the following percentages:

2% → 0.2 color	, 1.5 glycerin	, 8.3 d.i. water	
3% → 0.3 "	, 1.5 "	, 8.2 "	"
4% → 0.4 "	, 1.5 "	, 8.1 "	"
5% → 0.5 "	, 1.5 "	, 8.0 "	"
7% → 0.7 "	, 1.5 "	, 7.8 "	"

*weighed all on analytical balance & mixed in smpl. vials

2) ~~using~~ cyc pipette 5-6 drops onto paper and pull down steel bar

3) tape up color sheet until dry & then compare results

NOTE: compare all color sheets to 2% draw-downs of Dri-mark (blue & violet), to be used as std.

Date July 24, 1996 Signature Chinota Y. Camp

Date July 26, 1996 Witness Rick Ruby

→ CONT. p-64

Acid Dyes Comparisions

→ continued from pg. 10118-63

VI. RESULTS

(compared to 2% draw-downs on blue & violet by Dri-mark)

Blue

Crayola 2% – too weak
(B#9) 3% – matches closely

Milliken 2% – too weak
(WRG) 4% – cyc ~~too~~ weak
5% – matches closely

Violet

Crayola 2% – too weak
cyc ~~(B#9)~~ 3% – weak
(V#17) 4% – slightly weak
5% – matches closely

Milliken 2% – too weak
(Access 1967) 5% – weak
7% – matches closely

NOTE: all results were confirmed by M. Valenti (7/24/96)
—— * ——
K. Chavis has the actual draw-downs in her files

FINAL RESULTS: Pass >

Violet	Blue
2% Dri-mark	2% Dri-mark
5% Crayola	3% Crayola
7% Milliken	5% Milliken

VII. CONCLUSION

Dri-mark's acid dyes are stronger than those by Crayola & Milliken for colors blue and violet; based on draw-downs, Crayola & Milliken colors add to be increased to match the 2% Dri-mark draw-downs

Date July 24, 1996 Signature Chinota Y. Camp

Date July 24, 1996 Witness Rick Ritz

SF8320 Acid Numbers 10118-65

(Epal 1Ø 4EO/3PO) lot 10000-73

I. PURPOSE Refine acid # method for acetylation rxn. for given sample.

II. APPARATUS
on pg. 10118-61

III. REAGENTS
on pg. 10118-61

IV. RAW MATERIAL CHARGES
on pg. 10118-61

V. PROCEDURE
1) add given amnts. of alcohol & acetic anhydride to flask
2) dissolve contents in 100ml d.i. water
3) add boiling beads, set on hot plate (high), & connect to condensers
4) heat contents accordingly ~~#~~ CYC
5) turn off hot plate, leave condensers connected, & place flasks in ice bath for 10-15 min., then rinse condensers w/ d.i. water & remove
6) add 6-8 drop of phenolphthalein & stir bar to flasks, place on magnetic stirrer, & titrate
7) Record results & do calculations

VI. RESULTS DV12 SPECS. → theo. acid # = 183

A.) heat to reflux

	alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
1 -	5.0168	1.0167	6.0335	39.20	182.24
2 -	5.0047	1.3477	6.3524	52.10	230.06

B.) heat at reflux 5 min.

	alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
1 -	5.0182	1.0056	6.0238	38.86	180.95
2 -	5.2773	1.0131	6.2904	39.16	174.62

Date July 25, 1996 Signature Chineta Y. Camp

Date July 26, 1996 Witness Rick Rutz

→ Cont. p. 66

→ continued from pg. 10118-65

VI. RESULTS (cont.) DV12 SPECS. → theo. acid # = 183

C.) heat at Reflux 10 min.

	alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
1 –	5.0119	1.0337	6.0456	39.80	184.66
2 –	5.0136	1.0211	6.0347	39.52	183.69

D.) heat at Reflux 15 min.

1 –	5.0093	1.0202	6.0295	39.30	182.83
2 –	5.0029	1.0263	6.0292	39.70	184.70

E.) heat at Reflux 20 min.

1 –	5.0018	1.0186	6.0204	39.28	183.01
2 –	5.0359	1.0123	6.0482	39.12	181.43

VII. CONCLUSION REPEAT sets which are closest to theo. acid #

"REPEATS" SPECS. → theo. acid # Range = 178–188

* heat to Reflux

1 –	5.0098	1.0178	6.0276	38.78	180.47
2 –	5.0132	1.0373	6.0505	39.88	184.88
3 –	5.0193	1.0442	6.0635	40.12	185.60
4 –	5.0171	1.0029	6.0200	38.68	180.23

* heat at Reflux 10 min.

1 –	5.0156	1.0113	6.0269	38.68	180.02
2 –	5.0297	1.0070	6.0367	38.52	178.99
3 –	5.0186	1.0254	6.0440	39.32	182.48
4 –	5.0207	1.0265	6.0472	39.48	183.13

* heat at Reflux 15 min.

1 –	5.0485	1.0349	6.0834	39.38	181.58
2 –	5.0231	1.0106	6.0337	38.66	179.73
3 –	5.0272	1.0087	6.0359	38.82	180.40
4 –	5.0103	1.0381	6.0484	39.88	184.95

Date July 25, 1996 Signature Chineta Y. Camp

Date August 1, 1996 Witness Kyle Stephens

→ cont. p. 67

SF8320 Acid #'s 10118-67

→ continued from pg. 10118-66

Results: (cont.)

	alcohol	act. anhyd.	smpl. wt.	mls. KOH	Acid #
* heat at Reflux for 20 min.					
1 –	5.0072	1.0055	6.0127	38.54	179.79
2 –	5.0029	1.0032	6.0061	38.58	180.18
3 –	5.0214	1.0139	6.0353	39.14	181.91
4 –	5.0389	1.0178	6.0567	39.42	182.56

~~CONCLUSION~~ cyc

"REPEATS" *

~~*heat~~ cyc

CONCLUSION:

all results were within ~~range~~ bounds of the theo. acid # range cyc

Date July 29, 1996 Signature Chineta Y. Camp

Date August 1, 1996 Witness Kyle Stephens

SF8320 Physical Properties Testing 10118-68

lot 10000-73

PURPOSE Determine freeze point of product. Also find specific gravity (vs. temperature) of product (@ -1.1, 15.6, 32.2, & 48.9 °C)

REAGENTS A

VOID

Date July 26, 1996 Signature Chineta Y. Camp

Date August 1, 1996 Witness Kyle Stephenson

SF8320 Physical Properties Testing lot 10000-73 10118-69

PURPOSE Determine the specific gravity & viscosity (vs. temperature) of the given product at the following temp. intervals > -1.1, 15.6, 32.2, & 48.9 °C.

APPARATUS

1- temp. controlled water bath
2- 25ml Volumetric flask w/ stopper
3- viscometer w/ cylinder (Rotating)
4- top load balance

REAGENTS

NONE

RAW MATERIAL CHARGES

Epal 10 4EO/3PO — 8g (lot 10000-73)

PROCEDURE (Spec. grvty.)

1) place smpl. in flask up to mark & replace lid (* weigh flask w/o smpl. 1st & record wt.)
2) set water bath on 1st temp. & wait for it to get on temp.
3) place flask in container & sit in water bath
4) wait at least 10 min., remove from bath, weigh flask & record wt.
5) Repeat steps 1-4 for setting at each temp.

calc. → (wt. empty − wt. flask w/ smpl. after bath) = ?/25 = specific gravity

NOTE: plots spec. grvty. pts. to give linear graph

PROCEDURE (viscosity)

1) place 8g smpl. in cylinder & allow to become room temp.
2) set viscometer at the following setting: given temp. CYC ~~25°C~~, ~~20°~~ 10 RPM, cP display
3) follow directions of viscometer & place cylinder in slot
4) allow to read smpl. at least 30 min. to stabilize & record results
5) Repeat step 1-4 for setting at each temp.

NOTE: plot viscosity pts. to give linear graph

RESULTS (spec. grvty.) * initial flask wt. = 24.4974

°F	°C	final wt.	spec. gravity
30	-1.1	48.5075	0.9604
60	15.6	49.0683	0.9828
90	32.2	48.8401	0.9737
120	48.9	48.5468	0.9621

* NOTE: Chris K using his spec. grvty. cup (wt. cup & smpl.)

30	2.7311	2.7311
60	2.7291	2.7291
90	2.7276	2.7276
120	2.7259	2.7259

calc. (smpl. wt) x (0.01202)

RESULTS (viscosity)

°F	°C	viscosity (cP)
30	-1.1	160.0
60	15.6	75.0
90	32.2	50.0
120	48.9	35.0

CONCLUSION ~~all testing was normal~~ CYC

all testing ~~was~~ gave normal results for the product given

Date July 26, 1996 Signature Chinita Y. Camp

Date August 1, 1996 Witness Kyle Stephens

SPEC. GRVTY. #'s off; Ran again on pg. 10165-01

SF8320 Physical Properties Testing 10118-70

lot 10000-73

PURPOSE Determine the haze point & freezing point of the given product. (& long term hazing)

APPARATUS
1- large test tube
2- thermometer

REAGENTS
1- Acetone
2- crushed ice
3- dry ice

RAW MATERIAL CHARGES
Epal 1Ø 4EO/3PO (lot 10000-73) - varied amnt.

PROCEDURE (haze pt.)
1) place smpl. in test tube (until ½ full) & immerse thermometer in smpl.
2) place t.t. in container of crushed ice & acetone
3) stir smpl. continuously w/ thermometer
4) Record temp. at which a cloud appears in smpl. (haze pt.)

PROCEDURE (freezing pt.)
1) place smpl. in t.t. (½ full) & immerse thermometer
2) place t.t. in container of dry ice
3) stir smpl. continuously w/ thermometer
4) when smpl. has solidified, record temp. (freezing pt.)

RESULTS

Haze pt. → -24 °C
Freezing pt. → -36 °C
Long term hazing → slight haze
(5° above freezing pt. = @ -31°C)

CONCLUSION
all tests gave normal results for the product given

Date July 26, 1996 Signature Chineta Y. Camp
Date August 1, 1996 Witness Kyle Stephens

SF8320 Phys. Properties Testing 10118-71

lot 10000-73

PURPOSE Determine the freeze/thaw cycle & flash point of the given product.

APPARATUS

1 - lg. test tube
2 - thermometer ~~(°C & °F)~~ °C
3 - Ring stand
4 - gas source
5 - Cleveland open flash cup apparatus

REAGENTS

1 - Acetone
2 - crushed ice
3 - dry ice

RAW MATERIAL CHARGES

Epal 10 4EO/3PO (lot 10000-73) - varied amnt.

PROCEDURE (freeze/thaw cycle)

1 - place smpl. in t.t. (½ full) w/ thermometer immersed in smpl.
2 - place t.t. (1st) in container of dry ice
3 - when smpl. solidifies, record temp.
4 - place t.t. (2nd) in container of crushed ice
5 - when smpl. liquifies, record temp.
6 - Repeat steps 1-5 three times & record results

RESULTS

	(freeze)	(thaw)
cycle ~~1~~ 1 -	-37°C	-14°C
2 -	-34°C	-16°C
3 -	-36°C	-14°C

* 3 cycles

PROCEDURE (flash pt.) this training is now complete (RLR)

1 - pour smpl. in metal cup to mark & suspend thermometer (from ring stand) about 1/4 inch into cup
2 - heat smpl. rapidly until continous stream of smoke appears, then regulate heat to 5-6°C per min.
3 - cont. heating until a flash appears at any pt. on the surface of smpl. & record temp. (flash pt.)

RESULTS (flash pt.)

NOTE: ① avoid breathing over smpl. ② perform in non-ventilated hood

Flash pt. → ~~116°C~~ 175°C

CONCLUSION

all test gave normal results for the product given

Date July 29, 1996 Signature Chineta U. Camp

Date August 1, 1996 Witness Kyle Stephens

SF8320 Phys. Properties Testing 10118-72

lot 10000-73

PURPOSE Determine the oven aging, Rusty nail, & shelf stability factors of the given product.

APPARATUS
1- testing oven
2- lg. (9 in.) nails
3- 4 oz. vial w/ lid
4- 8 oz. jar w/ lid

REAGENTS
1- tap water

RAW MATERIAL CHARGES
1- Epal 10 4EO/3PO (lot 1000-73) - varied amnt.

PROCEDURE (oven aging)
1- place smpl. in 8 oz. jar & replace lid
2- set in 60°C oven for 2 weeks & ck. smpl. every 2 days
3- Record observations (i.e., color, viscosity, specs, separation)
NOTE: put in oven 7/29/96 @ 8:15 AM

RESULTS

day 2 → (7/31)	no changes	
4 → (8/2)	"	"
6 → (8/5)	"	"
8 → (8/7)	"	"
10 → (8/9)	"	"
12 → (8/12)	"	"
14 → (8/14)	"	"

CONCLUSION
no changes in appearance or structure of material after 2 weeks in oven

PROCEDURE (Rusty nail)
1- place smpl. (1/4 full) in one vial, add nail, & Replace lid
2- place water (1/4 full) in another vial, add nail, & Replace lid
3- let stand 24 hrs. & record observations (any Rust on nails)
NOTE: put in vials 7/29/96 @ 8:45 AM

RESULTS
7/30/96 vial 1 (smpl.) → no Rust at all on the nail
vial 2 (water) → nail is extremely Rusty in H_2O

CONCLUSION smpl. "PASSES" Rusty nail test

→ cont. p. 73

Date July 29, 1996 Signature Chineta Y. Camp
Date August 1, 1996 Witness Kyle Stephens

SF8320 Phys. Properties

→ continued from pg. 10118-72

PROCEDURE (shelf stability)

NOTE: info. taken from observations made of test samples (i.e., color, viscosity, specs., phys. form)

RESULTS

"PASS" — all samples had no adverse changes; they looked as close to the same as they did when I 1st placed them on the shelf

CONCLUSION

all tests gave normal ~~result~~ cyc results for the product given

Date July 29, 1996 Signature Chineta Y. Camp

Date August 1, 1996 Witness Kyle Stephens

SYNFAC PRODUCTS

Cloud Point

diff. lot #'s

10118-74

PROCEDURE Determine cloud points of each of the Synfac products.

APPARATUS
1- 125ml beaker
2- thermometer
3- magnetic stirrer
4- stir bar
5- hot plate 6- 1L Vol. flask

REAGENTS
1- Sodium Chloride sol'n
2- Deionized Water

RAW MAT. CHARGES
SF8120 (lot F1112) - 6g
SF8145 (lot PP103) - 6g
SF8145 (lot 9914-23) - 6g
Sodium Chloride J.T. Baker (lot 439356) - 100g

PROCEDURE

~~Prep. of 100g~~ c/c

Prep. of NaCl sol'n:

1) in 1L Volumetric flask, add 100g NaCl & 900 ml d.i. water (to dissolve)
2) shake vigorously & set aside

Prep. of Samples: (1g SF & 99g NaCl sol'n)

1) in 125ml beaker, add 99g NaCl sol'n & slowly add 1g of Synfac smpl.
2) add stir bar & heat slowly until combined sol'n begins to cloud (cloud pt.)
3) Record temp. of cloudiness in smpl.

Prep. of Samples: (5g SF & 95g NaCl sol'n)

1) in 125ml beaker, add ~~5g~~ c/c 95g NaCl sol'n & slowly add 5g of Synfac smpl.
2) Repeat steps 2-3 (from above) to complete procedure

→ cont. p. 75

Date July 29, 1996 Signature Chineta Y. Camp

Date August 1, 1996 Witness Kayle Stephen

SYNFAC products → Cloud Pt.

continued from pg. 10118-74

RESULTS

Cloud Points:

SF8120 (lot F1112)	1g –	97 °C
	5g –	95 °C
SF8145 (lot PP103)	1g –	86 °C
	5g –	84 °C
SF8145 (lot 9914-23)	1g –	82 °C
	5g –	80 °C

CONCLUSION specs. → cloud pt. = 80°C or greater

each amount of sample gave results that were within the given spec. range; but SF8120 had cloud pts. that were "slightly" higher than range

Date July 30, 1996

Date August 1, 1996

Signature Chineta Y. Camp

Witness Kyle Stephens