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LABORATORY NOTEBOOK

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Date Received 4-10-96

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NOTEBOOKS ARE NOT TO BE TAKEN OUTSIDE OF THE COMPANY OR CORPORATION PREMISES WITHOUT THE PERMISSION OF DIRECTOR OF RESEARCH.

The procedure outlined below is required for one purpose primarily---to make the notebook acceptable as indisputable legal proof in patent proceedings of what was done, and exactly when it was done. The exact date on which a given idea was first in mind has often been the basis for awarding valuable patents. Therefore, as soon as you have any idea, even though it may seem trivial or obvious to you, you will, under the procedure outlined, set it in your notebook with complete details. This will apply whether it is a small modification in experimental procedure or apparatus design which may give improved yields or operation, or what may be a major improvement or innovation in products or processes. **GET IT DOWN.**

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 data.

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 someone 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 Division 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 will be charged out.

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#10007

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Hydrolyze the Bulk of B.H. 9990-43, ref. 10043-47

Add ~600mL of 20% sulfuric acid to a 2L, 3 neck flask. Mount over Mag. stir plate. Add stir bar, heat mantle, thermometer, condenser, + drop funnel.

Beginning stirring + heat to ~50°C at ~9:30am. Reached temp. at 9:46am. Charge drop funnel with 279.56g of product B.H. 9990-43, + begin dripping into flask when temp. is 50°C. Finish dripping at ~ 10:30am. (or a little more)

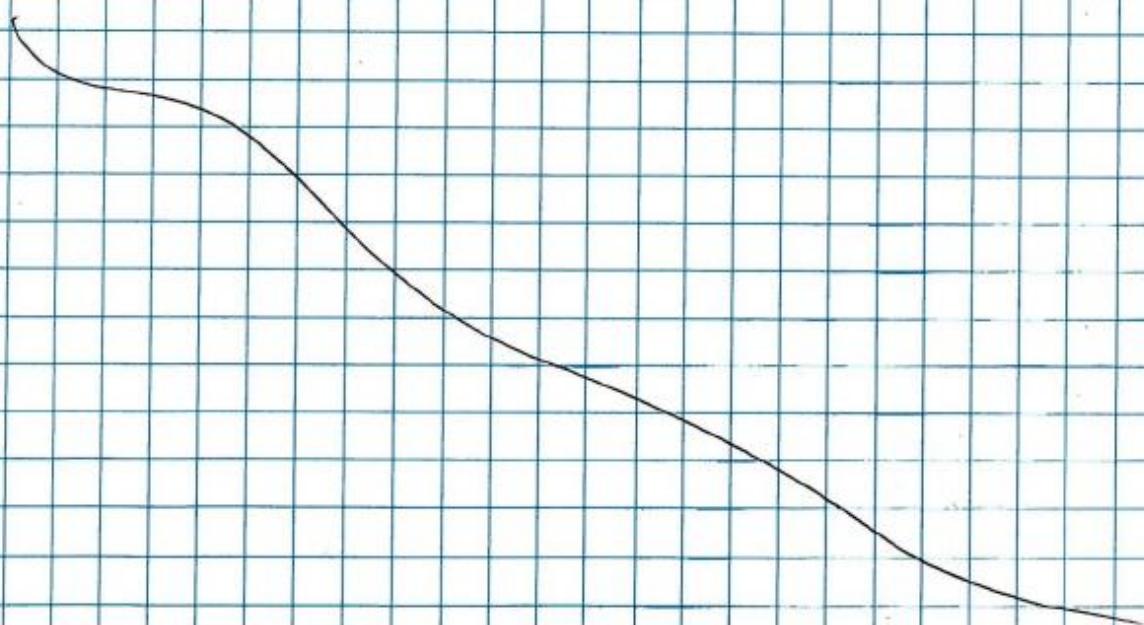
No fizzing noticed. Material is a deep brown color.

Raise temp. to ~100°C + let run. (10:30am)

At 10:50am, temp. ~82°C, some foaming or fizzing noted in center of surface. Definite fizzing + refluxing at ~84°C, 10:58am.

Reached 90°C @ ~12:15pm; still fizzing
Reached 93°C @ ~1:30 to 2pm; still fizzing

Stopped Rxn at 4:30pm.



Date April 11, 1996

Date April 26, 1996

Signature

M.R. Filling

Witness

Doris S. Fisk

Cont'd on 10007-3

Determine Hydroxyl # N.H. 9990-43

Use D.V. #9

Expected # is 94.9, so $\frac{420}{94.9} = 4.42$ g desired sample wt.

Sample "1" wt = 4.2314 g

Sample "2" wt = 4.2412 g

Add 10mL OH soln. to each ~~J~~ & to beaker for a blank

Added 3 glass beads to both samples, installed in condensers, heated to boiling; started timer for 5min.

Added 2.5mL H₂O to blank

After 5min, add 1mL DI H₂O to samples, wait 10sec. + add 4mL DI H₂O

After 1min. remove from heat; after 1min. rinse condensers in methanol; after 1min. remove flasks from condensers.

Add 50mL 0.5N KOH to all three

Fill buret to 0 mark with 0.5N KOH (for each)

Add 5 drops phenophthalein indicator to each & titrate till turns pink.

mLs. of .5N KOH added to each:

Blank = 38.26mLs

Sample 1 = 14.81mLs

Sample 2 = 15.05mLs

Formula: $\frac{(\text{Blank mLs} - \text{Sample mLs})(0.5\text{N})(56.1)}{\text{Sample wt}} = \text{Hydroxyl \#}$

Sample 1 Hydroxyl # = 155.45

Sample 2 Hydroxyl # = 153.50

Avg. Hydroxyl # = 154.48

Date April 11, 1996

Date April 12, 1996

Signature

M. R. Villaluz

Witness

Terese Foster

Cont'd from 10007-1

Hydrolysis of the Bulk - of D.T. H. 9990-43

Pour product into a beaker & place this in a bucket with water; begin mag. stirring

Rinse rxn. flask with D.I. H₂O & add this to beaker; also add more D.I. H₂O to bring total to \approx volume of product (~750 to 800 mL)

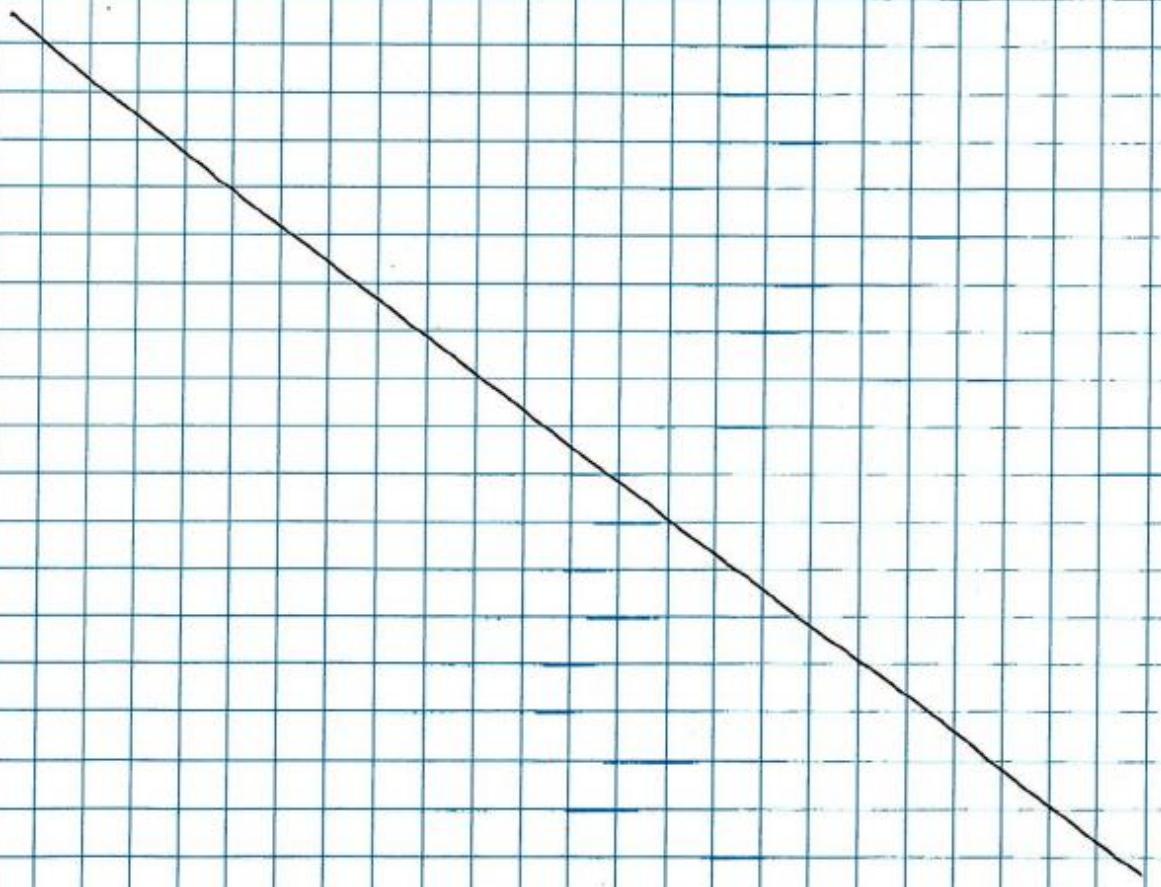
Initial pH = 0.95 ; Put a thermometer in beaker to monitor temp.

Drip 50% NaOH into beaker until pH \sim 13.

Add ice to bucket as needed to keep temp. from going too high

After adding 350.97 g NaOH, pH was 12.20. Continued adding NaOH, but pH began dropping! Added 258.00 g more NaOH & pH was 11.70.

Stopped; total NaOH added = 609.07 g with 11.70 pH.



Date April 12, 1996

Date April 17, 1996

Signature

Witness

M. R. Pithawalla
Dentise Fuchs

Cont'd on 10007-4

Cont'd from 1007-3; started on 1007-1

A thin dark layer is present on top of product. Pour into a sept. funnel & separate this from the rest; submit to analysis.

Heat the bulk of product (less the above mentioned layer) 'till it separates.

Turned cloudy at $\sim 60^\circ\text{C}$

At 85°C , some separation (forming a darker layer) noticed; put in sept. funnel, separation not well defined.

Returned $\sim 300\text{ml}$ to a heater & heat to near 100°C . This separated @ $\sim 95^\circ\text{C}$ & yielded 8.98g from the top layer.

M.W. of material is 593, so should be $\sim 279.0\text{g}$ of hydrolyzed product.

~~Heat~~ Heat ~~the~~ remainder of material likewise.

Heated to 97°C ; separation.

Place in Sept. funnel; drain off bottom layer. Total wt. of top layer = 66.11g (dark)
Evaporate top layer to dryness & submit sample for NMR, Mass spect & IR.

Take $\sim 100\text{ml}$ of bottom layer, & neutralize with dilute sulfuric acid, 20% methyl group may still be attached. Look for another separation.

Initial pH ~ 14.00 ; final pH ~ 7.0 . Total sulfuric used = 61.33g

(Increased volume by $\sim \frac{1}{3}$)

Heated to $\sim 90^\circ\text{C}$; thin layer separated.

Date April 15, 1991

Date April 26, 1991

Signature

M. R. Pellegrino

Witness

Deanne Foster

Cont'd on 10007-7

Combining Various Quats or Colants in Mobil 1 Oil

Materials:

10049-51, Fluorescein-Quat-Hydrocal

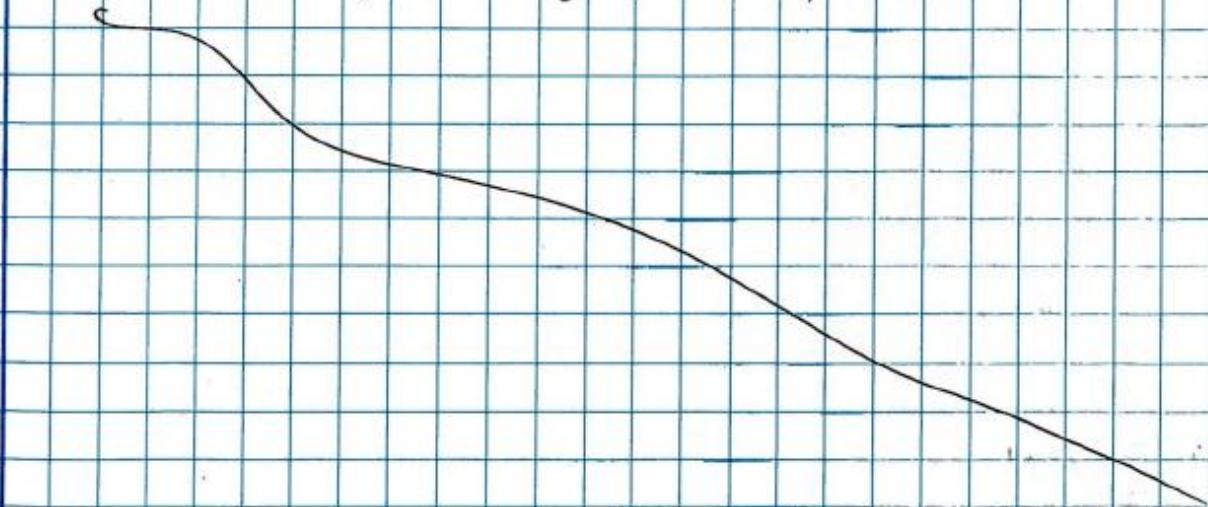
10049-52, Uranine / Quat / Hydrocal

10043-68, Chromatech Yellow 131SC - Automate Yellow HF

Add these to oil as follows & check fluorescence: (Total WT \approx 85.00g (n/100ml)

Sample ID	Sample WT.	Oil WT	Fluorescence
A 10049-51	0.32g	84.70g	barely
B 10049-51	0.64g	84.36g	barely
C 10049-51	1.28g	83.72g	slightly more
D 10049-52	0.32g	84.68g	barely
E 10049-52	0.64g	84.36g	barely
F 10049-52	1.28g	83.73g	slightly more
G 10043-68	0.32g	84.69g	This is the standard & it's extremely fluorescent
H 10007-G1	0.32g	84.69g	still very fluorescent

G1
make 12.9 gdm of
10043-68 in hydrocal
needs hydrocal.



Date April 15, 1996

Date April 26, 1996

Signature M.R. Poldberg

Witness Jettie S. Aarø

Conf'd from 10043-62

2 Week L.A.B. Readings of 10043-62 Detergent Samples Colored in Various Polymers

Deltas

Sample I.D.	L	A	B	E	L
10043-62A2, Red ST/H ₂ O	97.46	3.89	0.03	1.41	-0.60
10043-62B2, Red ST/PVA	98.83	1.28	1.97	0.67	-0.13
10043-62C2, Blue HP/H ₂ O	96.74	-1.49	-0.44	2.12	-0.99
10043-62D2, Blue HP/PVA	98.26	-0.63	1.10	0.99	-0.20
10043-62E2, Red ST/Propylene Glycol	96.54	4.30	0.83	2.28	-1.06
10043-62F2, Red ST/Propylene Carbonate	94.61	7.02	0.13	2.05	-0.99
10043-62G2, Blue HP/Propylene Glycol	96.47	-1.85	-1.01	4.80	-0.27
10043-62H2, Blue HP/Propylene Carbonate	94.99	-2.70	-2.50	1.68	-0.64
+ 10043-62I, Control White Tide	99.43	0.37	2.47	0.59	0.15

(Note)

* The filter paper left in each sample showed NO signs of visual discoloration or staining.

These samples were returned to the Tenney Humidity Chamber at 80°F & 80% r.h. & will be checked again on 4-29-96.

Date April 15, 1996
Date April 26, 1996

Signature M.R. Pilling
Witness Dentice Foster

cont'd on 10007-19

10007-9

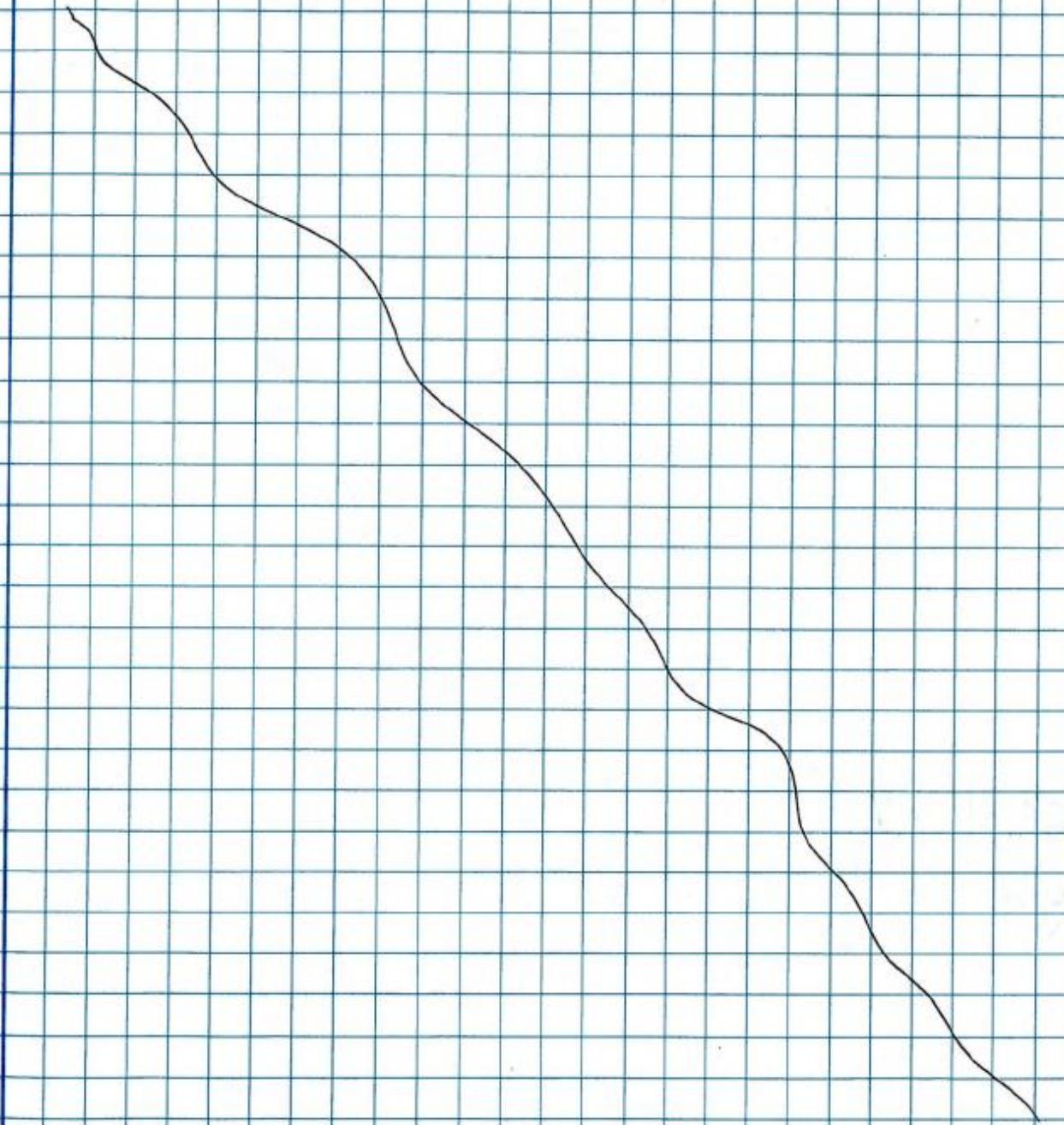
Cont'd from 10007-4; started on 10007-1

Hydrolyze P.H. 9990-43

Take the neutralized sample & extract with dichloromethane, in equal volume, twice.

Drain off bottom layer of resultant separation & evaporate this bottom layer to dryness.

Submit for NMR, Mass Spec, I.R.



Date April 16, 1996
Date April 26, 1996

Signature M. R. Kellberg
Witness Dentice Foster

Can't on 10007-9

Arm & Hammer®

Creating Colored Laundry Detergent in PVA, Using ALL® & F&F®; ref. 10043-59

Materials:

Airvol 523, P.V.A., 71% soln in DI H₂O lot 10043-59

Red ST AD101

Blue HP W1155

D.I. water

ALL® Ultra Laundry Detergent by Lever Brothers

~~F&F® Ultra - Color Plus~~

Arm & Hammer, Heavy Duty - Perfume & Dye Free by Church & Dwight

Mix Color solns. as follows:

Color & Absorb	Color Wt	P.V.A. Wt.	D.I. H ₂ O Out.
A Red ST, 10	0.75g	-	33.10g
B Red ST	0.75g	33.10g	-
C Blue HP, 15	0.50g	-	33.35g
D Blue HP	0.50g	33.35g	-

Spray these solns. on to uncolored detergents as follows, using Crown Spray-Tool & power sprt.
Pre-dry detergent in oven #298@ 77°C for ~2min, 155cc.
(need 50.0cc)

Type detergent	Wt.	Deterg. Dry Time	Soln. & Wt. Sprayed on	Comments
A1 ALL®	50.03g	2 min, 14 sec	"A" Red ST/H ₂ O - 0.86g	sprayed well, fairly even
B1 ALL®	50.02g	2 min, 16 sec	"B" " PVA - 0.84g	Pink deterg. with red clumps
C1 Arm & Hammer®	50.07g	2 min, 14 sec	"C" Blue HP/H ₂ O -	sprayed well, very even
D1 ALL®	50.05g	3 min, 14 sec	"D" " PVA - 0.95g	same as B1
E1 Arm & Hammer®	60.03g	2 min, 13 sec	"E" " PVA - 0.96g	barely sprayed, no to D1
F1 ALL	50.06g	2 min, 14 sec	"F" Blue HP/H ₂ O - 0.89g	same as C1
G1 A.H.	50.03g	2 min, 13 sec	"G" Red ST/H ₂ O - 0.89g	same as A1
H1 A.H.	50.03g	2 min, 13 sec	"H" Red ST/H ₂ O - 0.89g	initial L. abs.

Combine these colored bases with uncolored detergents as follows:

Colored Base I.D.	Base Wt.	uncolored deterg. & Wt.	L	A	B	
A2 A1	(3.62g)	ALL - 22.9g	96.45	-0.11	0.31	These samples were sealed in
B2 B1	3.62g	ALL - 23.0g	96.49	0.00	2.15	boxes & placed in Tegney
C2 C1	(5.02g)	AT-H-39945g	98.24	1.86	3.16	Humidity Chamber @ 80%RH &
D2 D1	8.61g	AT-H-23.0g	99.11	-0.35	4.66	80°F. They will be checked on
E2 E1	15.02g	AT-H 400.0g	101.50	-1.19	1.72	5-2-96
F2 F1	8.62g	AT-H 23.0g	101.13	0.04	-2.60	
G2 G1	15.01g	AT-H 400.0g	101.13	4.52	2.42	
H2 H1	14.96g	AT-H 400.0g	101.78	2.95	-1.98	
I None	-	ALL - 22.82g	101.18	0.26	3.48	
J None	-	AT-H 20.72g	102.44	0.44	-1.25	

Date April 18, 1996

Date April 25, 1996

Signature M. J. Kellaway

Witness Justice Foster

cont'd on 10007-25

Cont'd from 10007-7; started on 10007-1

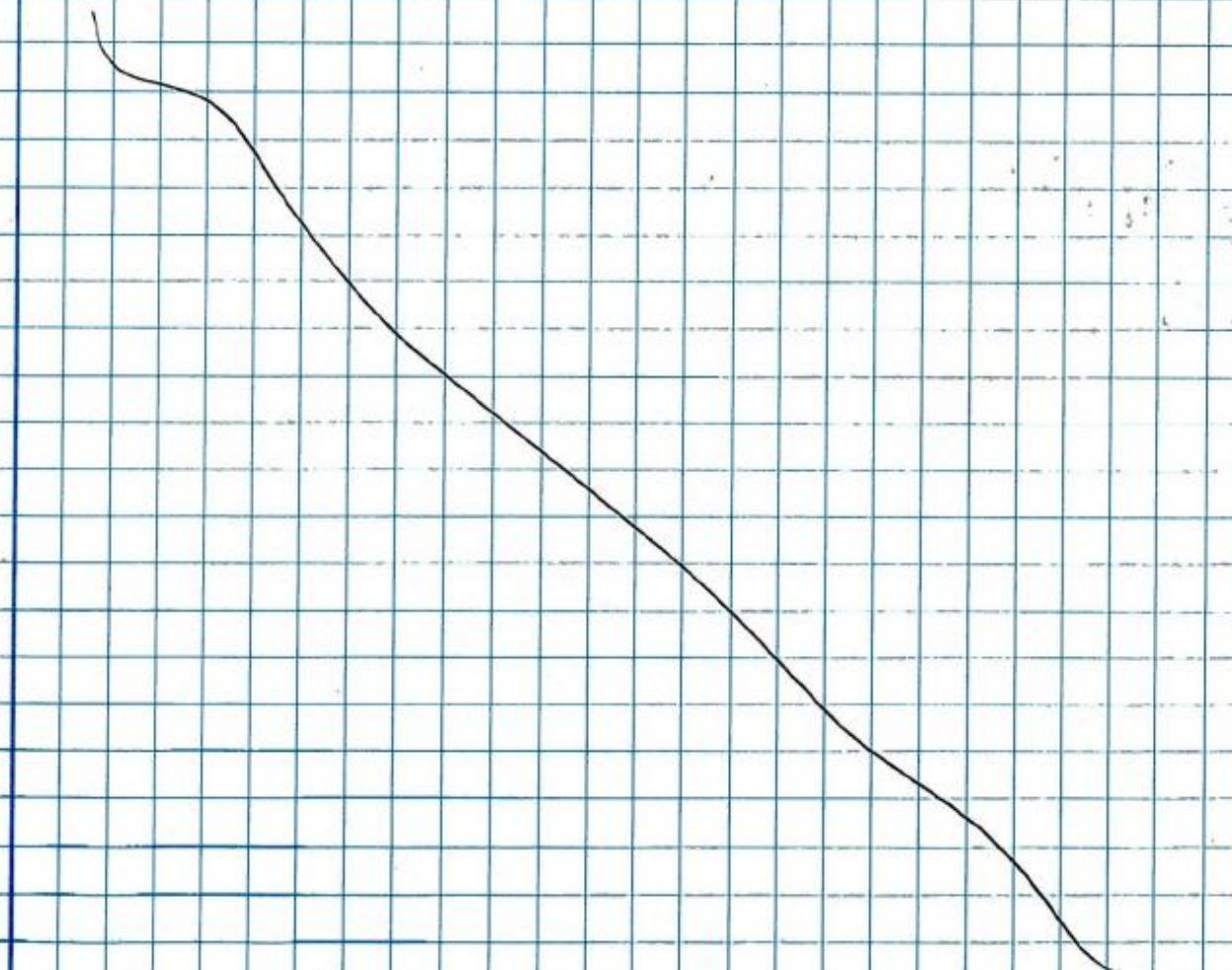
Neutralize & Extract Remainder of Bott. 9990-43

Use dichloromethane in ~ equal volume to extract. (do twice.)

Initial pH \approx 14. Neutralize with 20% sulfuric Acid.Added \sim 94.00 g of 20%, pH \sim 13.Added concentrated sulfuric 'till' pH \sim 7 to 8. (Volume now \sim 2800 mL)Heat to \sim 90°-95°C to see if it separates.Separation at \sim 80°CPlace in sept. funnel & drain off bottom layer; total volume was \sim 2700 mL

Wt. of top layer = 91.76 g; evaporate to remove water

Filter out salts from product.



Date April 18, 1996

Date April 26, 1996

Signature M. R. Ballou
Witness Dertise Porter

10007-10

Determine Amine Equivalent of 10007-1; Extracted Version & Top Layer of separation.

Use D.V. Method T311

Use Acidic Acid, Lot #2504KPMF & Perchloric Acid, lot #14804 KN O, 1016N

Extracted Version

Sample WT	1st Titration	2nd Titration	Top Layer of Sep.	1st Titration	2nd Titration
	0.7040g	0.7040g		0.6956g	0.6974g
mls. Perchloric	1.45 mls	1.40 mls		14.55 mls	14.78 mls

Amine			
Equivalent	4783.46	4949.38	470.55
			464.42

Milli-equivalents	0.21	0.20	2.13	2.15
-------------------	------	------	------	------

Av. Amine
Equivalent = 4866.42

Av. Milli-Equivalents = 0.205

Date April 22, 1996
Date April 26, 1996

Signature M.R. Nelling
Witness Justice Forfe

10007-11

Absorb of RLM 10045-41, -43, & -46; cut in Hydrocal & Checked in Toluene.

Sample I.D.	100mL flask [#]	10mL pipet [#]	wt.	conc.	wL	abs	Absorb
10045-41	923, 939		322	0.1204g	0.1204	420	0.7101 5.89
10045-43	887, 122		126	0.1137g	0.1137	422	0.5683 5.00
10045-46	880, 928		185	0.1139g	0.1139	422	0.5700 5.00

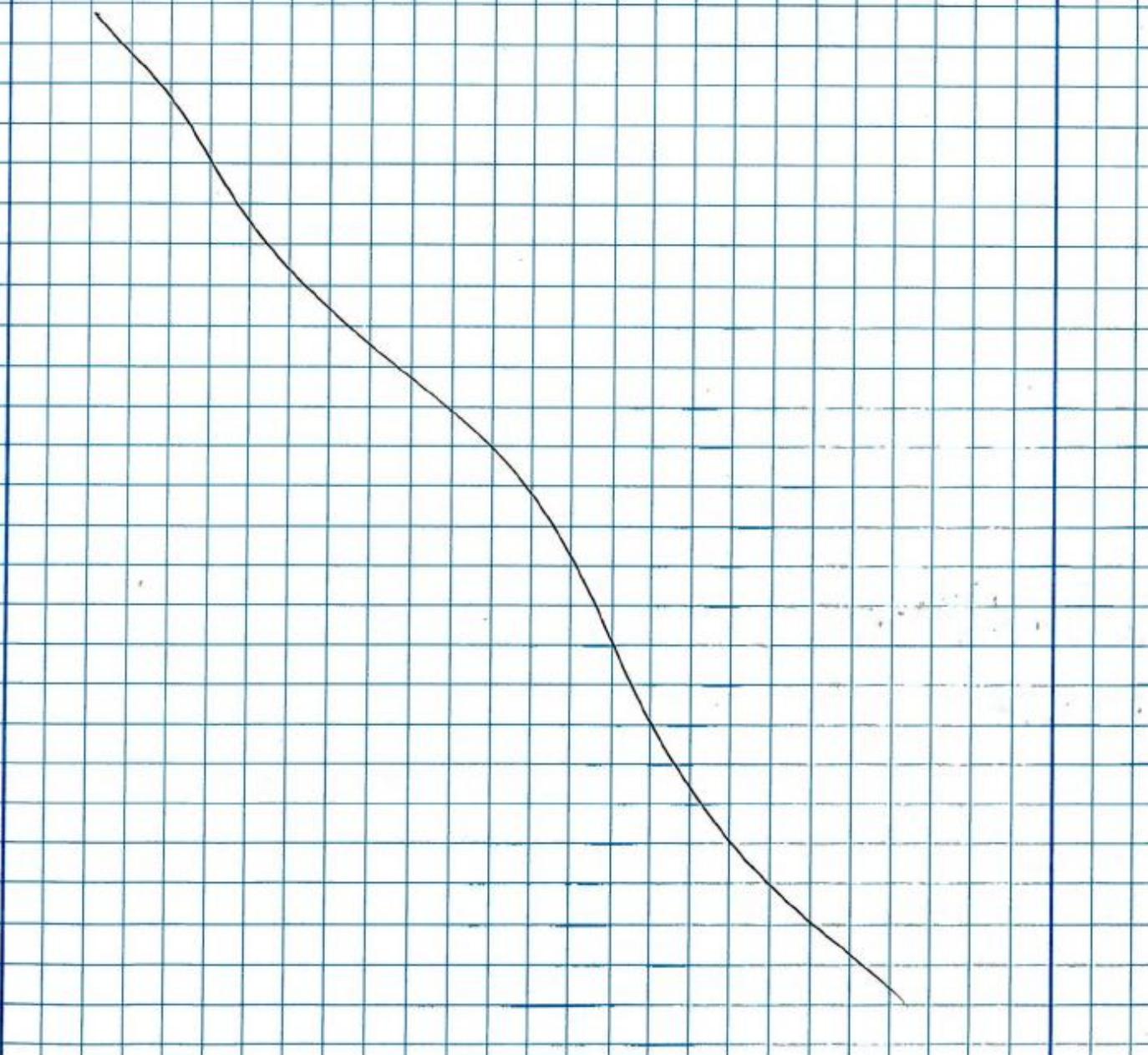
Date April 22, 1996
 Date April 26, 1996

Signature M. R. Kellberg _____
 Witness Donice Foster

10007-12

Check Absorb of MP 9974-32 + ALM 9815-33 in Methanol

Sample /D.	100mL flasks	# pippets	wt.	conc.	wL	abs	Absorb
9974-32	162, 887, 923	5mL #813 & 10mL #126	0.1632	0.00766	259 282 443	0.3650 0.3706 0.3309	47.65 48.38 43.20
9815-33	7529, 928	2mL #7516	0.1193	0.02394	253 415	0.5801 1.6643	24.23 69.52

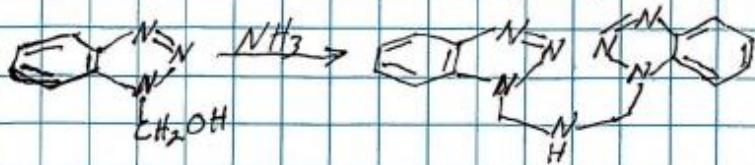


Date April 23, 1996

Date April 24, 1996

Signature M.R. Kelley
 Witness Denise Foster

Work-Up of RIM 10045-58 (1-hydroxy-methyl benzotriazole)



Make a 2% Ammonia Soln. using Ammonium hydroxide, lot 17628/HG (28-30% NH₃)

Ammonium Wt = 7.1g

D.I. H₂O Wt = 92.9g

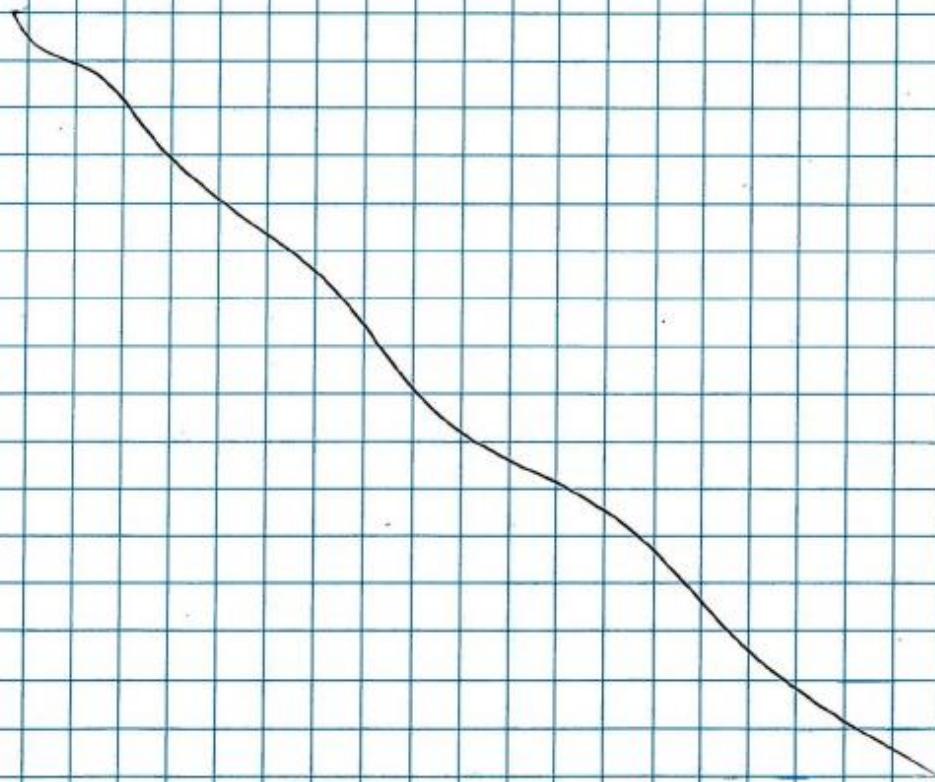
To 42.4g of 2% NH₃, add:

1 drop phenolphthalein, then.

Acetic Acid, glacial, 4ml Pink color disappears (2.24g)

Dissolve 11.9g of 10045-58 in 100ml methanol. Heated to 45°C to dissolve, then placed in ice bath to return to room temp., (~25°C)

Combine the two solns. & keep at ~25°C while stirring, 4L 5pm.
Put in refrigerator overnight. Turned pale pink/milky in less than 5 minutes



Date April 23, 1996

Date April 26, 1996

Signature

Witness

M. R. Bellamy
Denise Parker

cont'd on 10007-15

Testing for Amine in MP 10007-1 (Top Layer)

D) Dissolve a few crystals of Cuprous Chloride, lot 763155 (lab #1) in 10mL D.I. H₂O. ^{1 drop} Color went from a pale green to a pale greenish blue.

2) To a 140mL beaker, add: 0.48g of Quinizarin, Lot 40290; 0.25g of Leucoquinizarin, Lot R9293; 2.5mL D.I. H₂O; 2.74g of amine 10007-1

Hook-up a stream of nitrogen to blow gently into beaker,
Heat with stirring to near boiling.

Initial color was an orange-violet, which quickly turned violet.
After about 40min, water had evaporated & material began to "spic" out.
Added more water. Now has an orange cast to it.

3) Try ^{test} step (1) again, adding 1 drop Jeffamine M-715, lot N0708
Color turned a blue-green; slightly blue than previous.

4) In an erlinmeyer flask, add: 1.02 g of amine

~5mL Acidic Anhydride, lot 2420KJGP (Ac₂O)
a "Pinch" of 4-Dimethylaminopyridine, 91%,
lot 4023THKJ, Acetone, lab 32

Add 2 boiling stones & heat to ~100°C for 5 min.
Let cool

Add H₂O, D.I., to break down Ac₂O, ~25mL. Then add sodium carbonate, lot #06121CW until pH is ~8. Initial pH ~4. Final pH ~8 to 9 (some solids)

Drain off bottom layer, ~25mL, & evaporate to dryness.

After evaporation is complete, run an I.R., watching for carbonyl peaks at ~1630 - 1700cm⁻¹ & 1700 to 1730.

Dispose of top layer (water)

Date April 24, 1996

Date April 26, 1996

Signature J.M. R. Pilewicz

Witness D.J. T. Foxter

10007-15

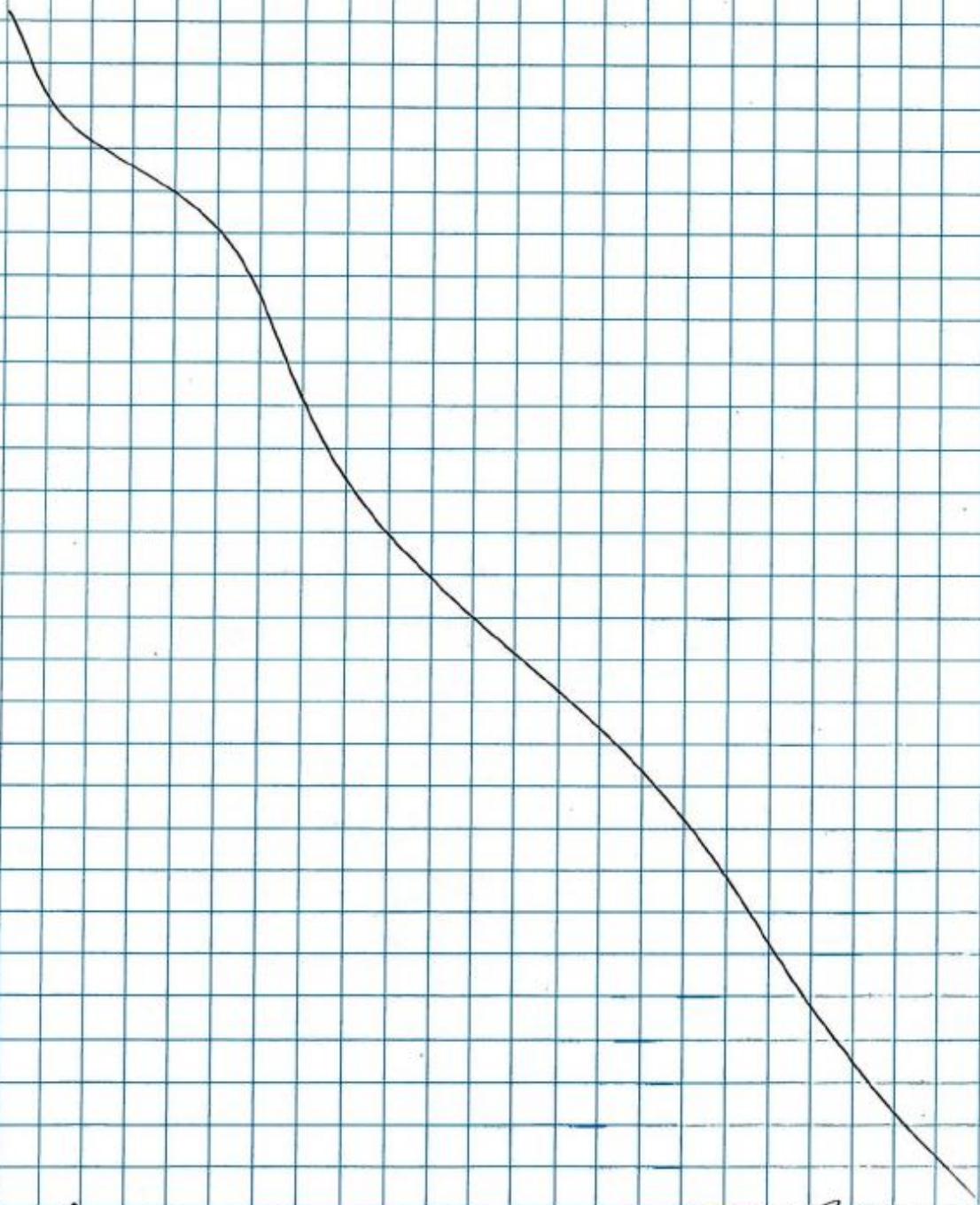
Cont'd from 10007-13

work-up RLM 10045-58

Remove from refrigerator.
Filter product

Let dry, then check melting pt.

M.P. \approx 177 - 180 °C



Date April 24, 1996
Date April 26, 1996

Signature M. R. Pilling
Witness Darlene Foster

Cont'd on 10007-16

10007-16

Can't from 10007-15; Started on 10007-13

work-up of ALM 10045-58

Work-up remained of product same as the small sample.

Take addition 2% Ammonia Soln.:

64.96g of Ammonia Hydroxide, lot 176 281HG
850.04g D.I. H₂O

57.60g remainder of previously mixed batch (10007-13A)

~~Take~~ Take 968.94g of 2% Ammonia

Add 23 drops of phenolphthalein soln. (turns soln. Pink)

Add 56.23g Acetic Acid Glacial (makes soln. clear)

Dissolve 272.40g of RIM 10045-58 in ~2.9L methanol.

Heated to ~45°C, then placed in ice bath to return to room temp. (~25°C)

Combined the two solns. & stir, keeping near room temp. for 5 hrs. Particulates began to form after ~2 min.

Place in refrigerator overnight.

Date April 25, 1996
Date April 26, 1996

Signature M-R. *Pallmyer*
Witness Justice Porter

Cont'd on 10007-18

10007-17

Cont'd from 10043-53

L.A.B. Readings on 10043-5/ Colored Detergent Samples (water passed in Tide Q)

Sample I.D.	2 Weeks			4 weeks			Deltas	
	L	A	B	L	A	B	E	L
10043-51A1, Blazon/H ₂ O	97.62	2.47	-0.21	97.55	2.35	-0.07	0.86	0.18
10043-51B1, VT Violet X80/H ₂ O	97.45	0.49	-0.74	96.05	0.83	-2.28	2.83	-1.89
10043-51C1, Int'l Blue 45X/H ₂ O	98.51	0.35	0.19	98.39	0.43	-0.33	1.97	-0.96
10043-51D1, VT Red II/H ₂ O	98.79	1.69	0.72	98.22	2.13	0.46	0.80	-0.45
10043-51E1, VT Blue II/H ₂ O	97.30	0.06	-0.81	96.99	0.03	-0.99	1.11	-0.70

Visually, all samples looked good.

Date April 25, 1986
 Date April 26, 1986

Signature M.R. Palermo
 Witness Justice Forte

10007-18

Cont'd from 10007-16; started on 10007-13

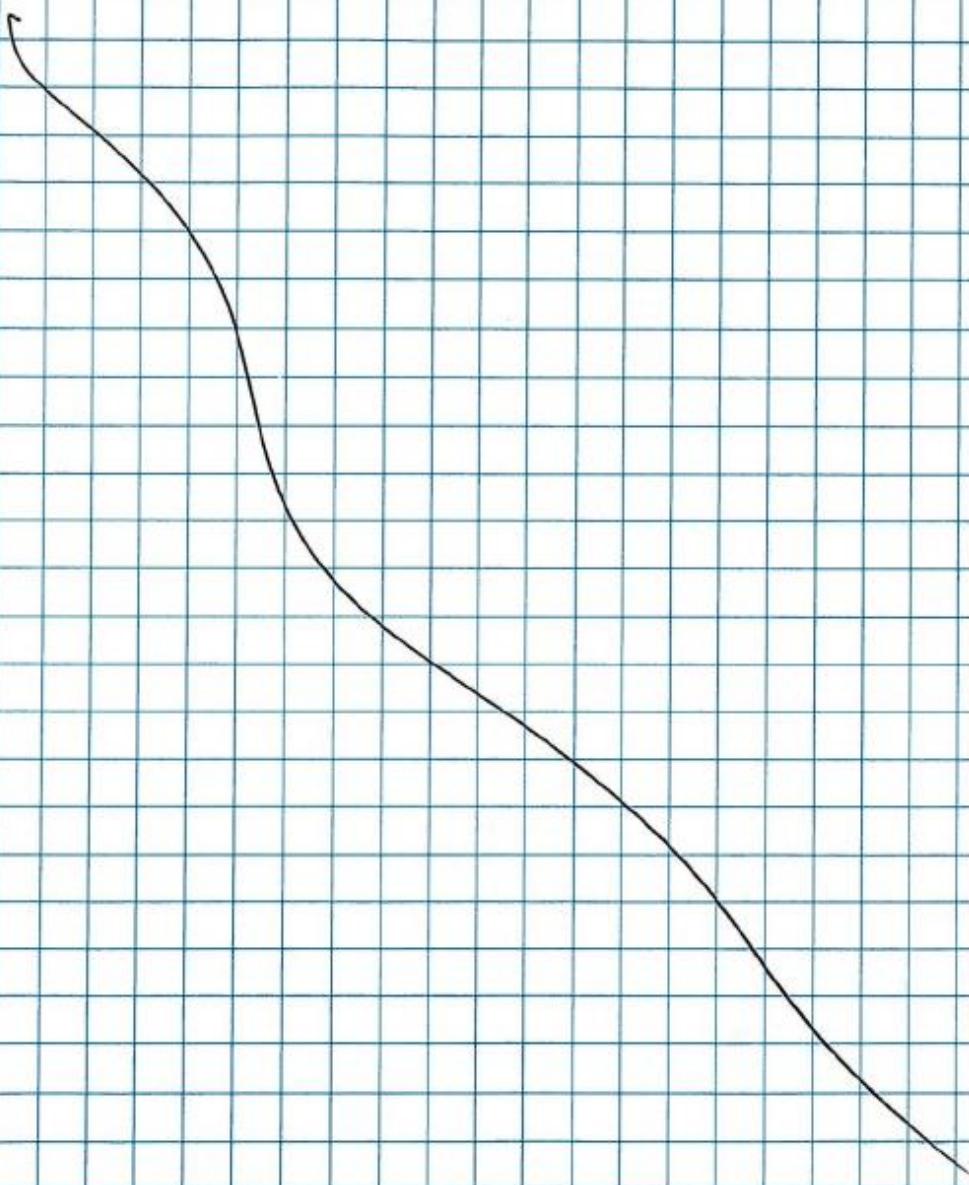
work-up of RLM 10043-58

Remove from refrigerator,

Filter product

Let dry.

M. P. \approx 177 to 179 °C



Date April 26, 1996
Date May 9, 1996

Signature

Witness

Mr. R. Pellegrino
Doris S. Foster

10007-19

Cont'd from 10007-6

4 week L.A.B. Readings of MP 10043-62 Detergent Samples Colored in Various Polymers

Deltas

Sample I.D.	L	A	B	E	L
10043-62A2, Red ST/H ₂ O	96.61	4.71	0.11	2.46	-1.45
10043-62B2, Red ST/PVA	98.72	1.24	2.15	0.55	-0.24
10043-62C2, Blue HP/H ₂ O	96.44	-1.75	-0.71	2.58	-1.29
10043-62D2, Blue HP/PVA	98.19	-0.73	1.53	0.70	-0.27
10043-62E2, Red ST/Propylene	96.16	4.47	1.07	2.51	-1.44
Glycol					
10043-62F2, Red ST/Propylene	94.77	6.75	0.26	1.71	-0.83
Carbonate					
10043-62G2, Blue HP/Propylene	96.08	-2.14	-1.05	5.11	-0.66
Glycol					
10043-62H2, Blue HP/Propylene	94.74	-2.87	-2.63	1.97	-0.89
Carbonate					
10043-62I, Control White	99.20	0.32	2.67	0.39	-0.08
Tide					

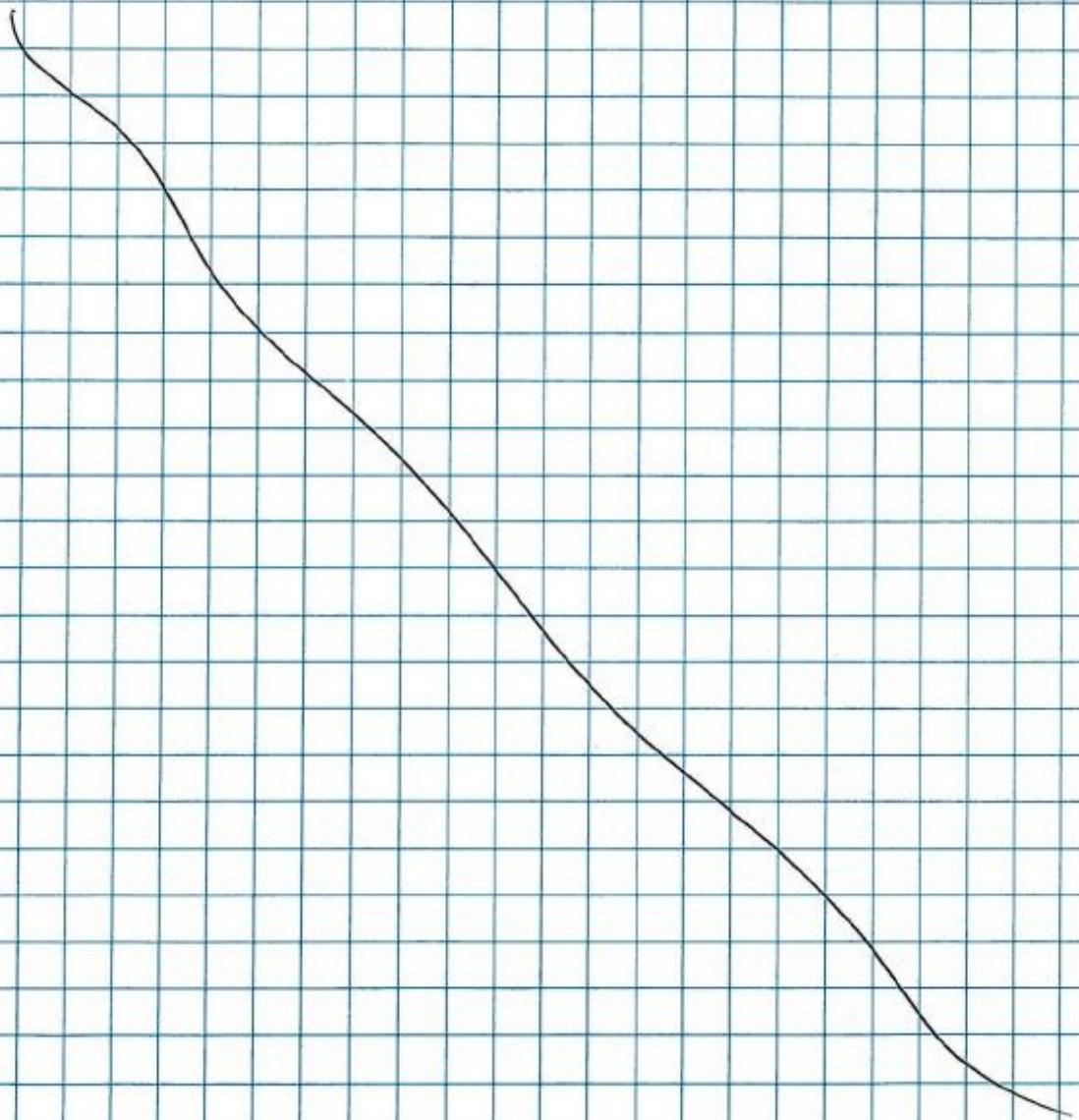
Date April 29, 1996
 Date May 9, 1996

Signature M.R. Pihlwan
 Witness J. T. T. S. Foster

10007-20

Monitoring Absorb of RL/M 10045-66

Time	Wt.	100mL flasks	Pipet #	Conc.	wL	ab.s	Absorb
11:30am	0.2693g	887,928	5mL, 127	0.13465	407	0.3818	2.84
4:00pm	0.2641g	same	same	0.13205	426	0.4338	3.29



Date April 29, 1996
 Date May 9, 1996

Signature M. R. McElroy
 Witness Denise S. Foster

10007-21

Determine Hydroxyl # of ALM10045-67, W-Chloro-mePEG

Use D.V. #9

Expected # is 0, so use 10.000g sample wt.

Sample "A"

wt. = 9.9322g

mLs of 0.5N KOH soln added:

38.40g

0.17

Hydroxyl #

Sample "B"

wt. = 9.9577g

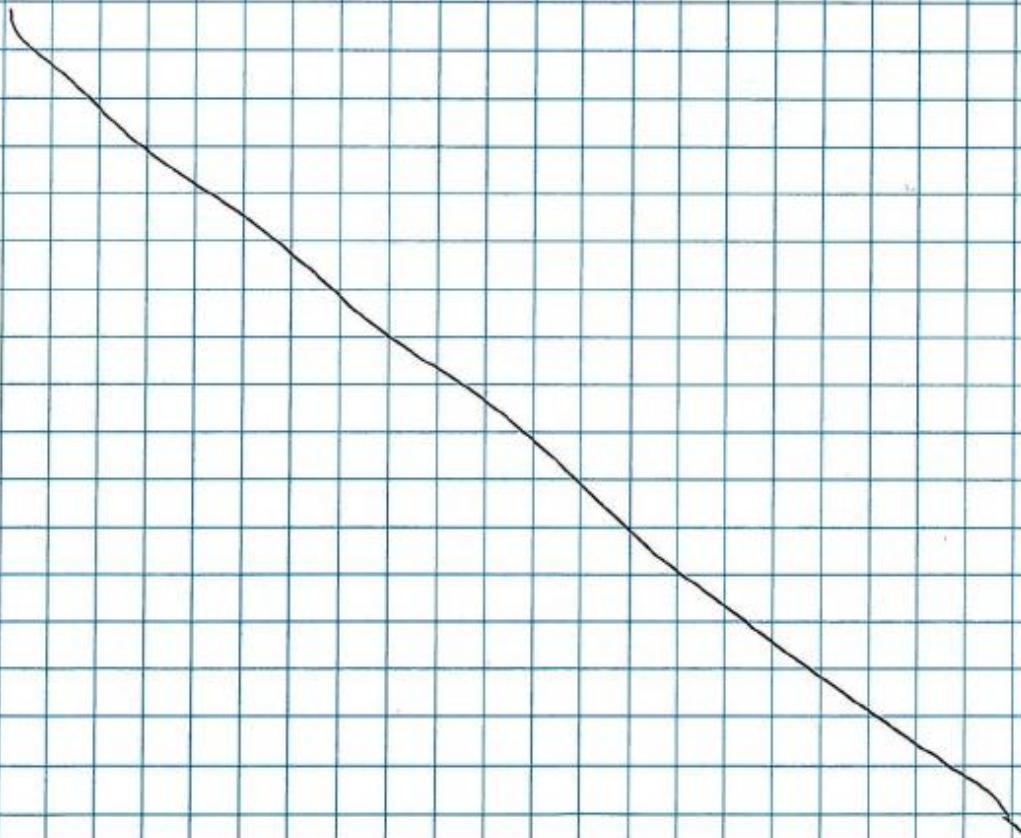
(BLANK)

38.30

38.46g

0.45

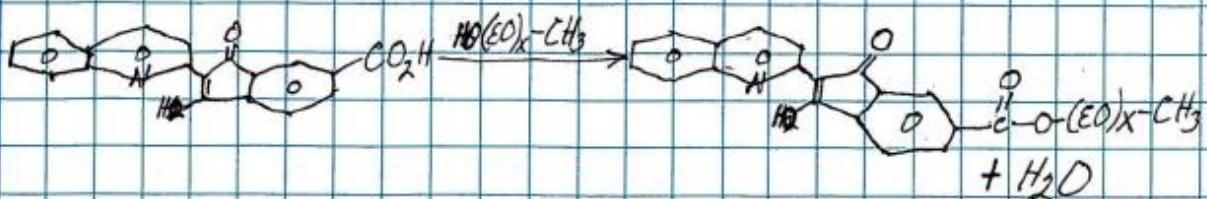
An Hydroxyl # = 0.31



Date: April 30, 1996
Date: May 9, 1996

Signature: M.R. Phillips
Witness: Jerry S. Foster

Rxn of PTSA · H₂O + Quinophthione Acid



Materials:

Materials:	Lot#	M.W.	Moles	wt. needed	Actual wt.
(PTA) ^{H2O} p-Toluenesulfonic Acid monohydrate	09208BF	190.22	0.006	0.3g	0.30g
Quinopthalone acid	041937-51	317	0.05	15.85g	15.85g
(Me ₂ PEG500) Poly(ethylene glycol)methyl Ether, Avg. M.W. 550 1992MX		550	0.05	27.50g	27.50g
Toluene	8608KPM		~200mL	~200mL	
(DMF) N,N-Dimethyl-formamide	4929KTK		~100mL	~100mL in 25mL intervals	

Combine all but DMF in a 1L 3-neck flask with overhead stirring, Dean Stark Trap, condenser, & drop funnel with heat mantle. Add static nitrogen line.

13'40

Heat to reflux; material not in soln.

Add, ~25 mL DMF thru drop funnel; not in soln.

Added 1.0g of $\text{PTSA} \cdot \text{H}_2\text{O}$ to 25ml DMF & added thru drop funnel; ~~beginning to go~~ ^{no in soln.} Small amount of H_2O came off.

Stop rxn at 5pm

Date: May 1, 1996
Date: May 9, 1996

Signature

Witness

M. P. Lublins
Dept. S. T. O.

WITNESS.....

10007-23

Cont'd from 10043-66; Started on 10043-59

2 wk & 4 week

L.A.R. Readings on Surplex "M" "Thm" "T" Detergent Samples in Various Polymers

Sample I.D.	2 weeks			4 weeks			Delta ^{visual} staining on filter paper	2 wks	4 wks	
	L	A	B	L	A	B				
10043-66M, Red ST/ Styrene-co-maleic Anhydride	98.01	2.75	0.90	98.58	2.86	0.59	1.98	-0.83	none	yes
10043-66N, Red ST/poly(NVEMA) Methyl Vinyl Ether-co- maleic Anhydride	97.64	2.64	0.74	97.97	2.90	0.65	1.45	-0.65	none	none
10043-66O, Red ST/Poly(SSS) sodium- <i>t</i> -Styrene sulfonate	98.39	2.22	0.74	97.88	2.87	0.47	1.63	-0.64	none	yes
10043-66P, Red ST/Bl./(VPAA) (<i>t</i> -vinylpyrrolidone-Acrylic acid)	97.90	2.90	0.43	97.70	3.33	0.33	2.44	-1.19	none	yes
10043-66Q, Blue HP/Poly(SMA)	98.68	-0.40	0.05	98.28	-0.53	-0.40	1.54	-0.55	none	none
10043-66R, Blue HP/Poly(NVEMA)	98.70	-0.38	0.10	97.97	-0.76	-0.64	1.72	-0.64	none	none
10043-66S, Blue HP/Bl./(SSS)	97.79	-0.81	-0.87	97.63	-0.95	-1.30	2.08	-0.76	none	yes
10043-66T, Blue HP/Poly(VPAA)	97.40	-0.94	-0.85	97.33	-1.09	-1.10	2.05	-1.12	none	none

Date: May 1, 1996
Date: May 9, 1996Signature: M. R. Pillbury
Witness: DENTISLE S. FOSTER

Cont'd from 10007-22

10007-24

Rxn of PTSA + Quinophthalone Acid

At 8am, much solid in flask.
With pipet, removed some liquid from top & put in H₂O.
This yielded some precipitate & some seemed to be soluble.

Added 2.00g of PTSA to rxn & restarted @ ~8:30am.

Stopped rxn @ 4:45pm

Still much solids presents & ~~little~~ if any additional
H₂O come off.

This rxn ^{product} will be wasted.

Date May 2, 1996
Date May 9, 1996

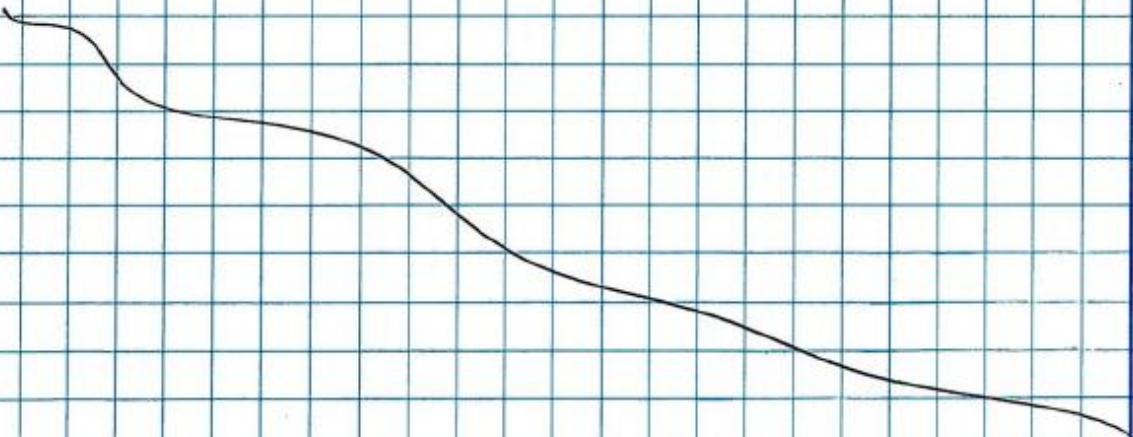
Signature M. R. Williams
Witness Denise S. Foster

Cont'd from 10007-8

2 Week L.A.B. Readings on Colored Detergents in P.V.A. (NL® and Arm+Hammer®)

Sample I.D.	A	A	B	E	L	Deltas on filter paper	visual staining on filter paper
10007-8A2, Red ST/H ₂ O Base in ALL®	93.20	8.56	0.49	5.86	-3.29	major	
10007-8B2, Red ST/ ^{PVA} H ₂ O Base in ALL®	97.39	2.84	2.60	1.41	-0.85	major	
10007-8C2, Blue HP/H ₂ O Base in Arm+Hammer®	97.77	-1.46	-6.82	2.77	-1.34	none	
10007-8D2, Blue HP/PVA Base in ALL®	95.71	-1.87	0.22	2.45	-1.79	moderate	
10007-8E2, Blue HP/PVA Base in Arm+Hammer®	99.76	-0.50	-4.38	2.32	-1.37	minor	
10007-8F2, Blue HP/H ₂ O Base in ALL®	93.55	-3.19	-2.10	4.08	-2.59	moderate	
10007-8G2, Red ST/H ₂ O Base in Arm+Hammer®	96.61	8.36	-3.70	4.78	-2.55	moderate	
10007-8H2, Red ST/PVA Base in Arm+Hammer®	98.36	5.99	-3.21	4.08	-2.42	minor	
10007-8I, ALL® detergent, no color	98.71	0.55	4.32	0.65	-0.47	none	
10007-8J, Arm+Hammer, no color	102.13	1.02	-1.32	0.33	-0.33	none	

These samples were returned to the Temney Humidity Chamber at 80°F & 80% R.H. for 2 more weeks. Will be checked on 5-16-96



Date May 7, 1996
 Date May 9, 1996

Signature M. R. Pillbury
 Witness JETCO S. Foster

Cont'd on 10007-34

Prep. of $\text{MeO}-(\text{CH}_2\text{CH}_2\text{O})_x\text{CH}_2\text{CH}_2\text{Cl}$; ref. 10045-67(8Lm)

Materials:

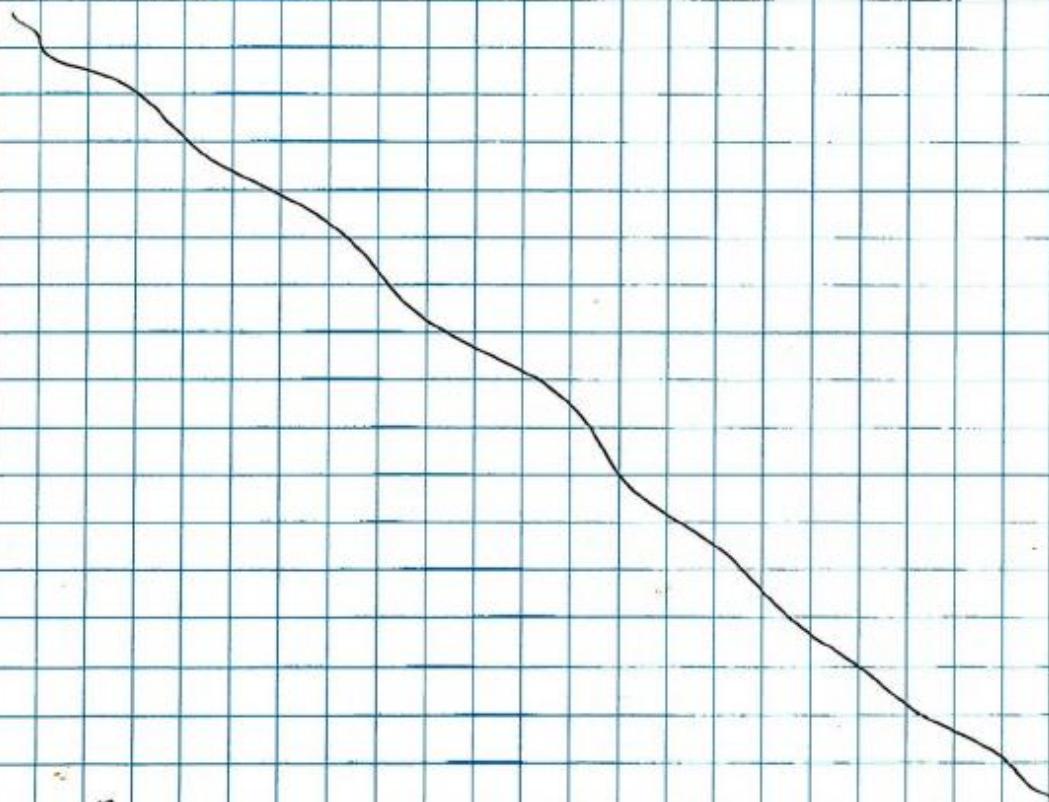
	Lot # of 1st Bkt 035204W	M.W. 107.22M	Moles 0.536	wt. needed 295.0g	Actual wt. 295.0g
(Me-PEG) Poly(ethylene glycol)methyl Ether	04712LG	119	1.13	133.95g	133.95g
(SO ₂ Cl ₂) Thionyl Chloride	02404W	-		~300mL	~300mL
(CH ₂ Cl ₂) Dicloro-methane					

Combine Me-PEG with CH₂Cl₂ in a 1L round-bottom flask, including condenser, Thermometer, Add-funnel & overhead stirring. Connect a gas trap to condenser.

Charge Add-funnel with SO₂Cl₂ & drip in over ~1hr. period, starting @ 2:40pm. Temp. never rose above 36°C during dripping.

Heat to Reflux starting at 3:50pm

Stopped rxn at 5pm



May 1, 1996
Date April 2, 1996
Date May 9, 1996

Signature: M. R. Pellegrino
Witness: Justice S. Foster

Cont'd on 10007-27

10007-27

Cont'd from 10007-26

Prep. of $\text{Me}-(\text{CH}_2\text{CH}_2\text{O})_x\text{CH}_2\text{CH}_2\text{Cl}$

Restart rxn @ 7:55 am.

Cut-off heat ~ 10:30 am

While rxn is cooling, set-up a vacuum distillation as follows:

Remove all glassware & adaptors from flask except stirrer set-up & thermometer.

Grease replacement glassware. Add a stop-cock adapter to release vacuum pres.; a Claz-on adapter with stopper on top - add to claz-on arm a vacuum distillation adapter with a 500ml round-bottom flask at bottom in an ice/acetone bath.

Connect vacuum hose to vacuum distillation adapter port.

With stirring on, start vacuum, & start heating slowly.
Lowest temp. was ~ 12°C. Heat to ~ 90°C, Slowly!.

Stopped distillation when temp reached 94°C; let cool to room temp.

Neutralize liquid vacuumed into 500mL flask & dispose of; use ^{sodium} bicarb.

Now, neutralize product.

Starting pH ≈ 0 ; temp is 27°C

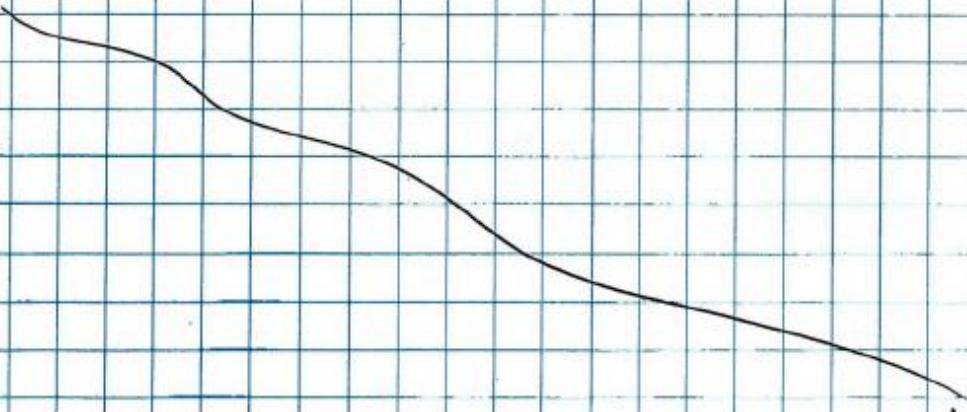
As pH begins to rise, product changed from a clear material with pale tan color to a milky tan (cloudy) ~~solid~~ liquid.

At pH 1 to 2, forming began to occur. Final pH ≈ 6 to 7

Evaporate on Roto-vap at 70° to 80°C for ~45 min.

Filter to remove solids.

Submit for I.R. & Mass spec



Date May 3, 1996
 Date May 9, 1996

Signature

Witness

M.P. Polkinghorne
 Jessie S. Foster

work on Tech Service # 9606168, Color Matching to Replace Colors
in Dawn Dishwashing Detergent

Check Absorbs of current Dawn Colored detergents;
use their uncolored base for blank

Color	Cone.	WL	abs
A) Dawn Yellow	straight	419	1.0466
	"	442	1.0297
	"	495	0.3177
B) Dawn Blue	"	411	0.0586
	"	633	0.9067
C) Dawn Green	"	418	0.5529
		442	0.4873
		499	0.1910
		634	0.6825

Make ~~1%~~ solns. of the following colorants:

color Colorant	wt.	D.I. Water WT	colorant Lot#	% Soln.
D Quinoline Yellow water soluble	1.00g	99.00g	08919JF - Aldrich	1%
E Pyranine 120	1.00g	99.00g	N11216, Miles	1%
F Uranine	1.00g	99.00g	47201-D, Hilton Davis (5154bgi)	1%
G Naphthalimide	10.00g	90.04g	RHM9657-72 (6.3 absorb)	10%

Check Absorbs on these solns. (Run in Hydron, dry Buffer, pH 7, tot 0.505PPF)

Colorant	100mL flasks*	Pipette#	Cone.	WL	abs	Absorb	wt.
D Quinoline Yel.	752.9	322	2.038	410	1.0256	0.5	0.2038g
E Pyranine	844.2	185	2.504	368	0.5455	0.22	0.2504g
F Holtranine	"	"	"	380	0.5340	0.21	"
G Naphthalimide	"	"	"	403	0.7486	0.30	"
	"	"	"	453	0.3664	0.15	"
F 1% Uranine	839.9	126	1.461	489	1.2519	0.86	0.1461g
G 1% Naphthalimide	167	5	1.109	451	0.7052	0.64	0.1109g

Date May 8, 1996
Date May 9, 1996

Signature M. R. Kellberg
Witness D. T. Foster

Cont'd on 10007-29

Cont'd from 10007-28

10007-29

Tech Service # 9606168

$$100 \text{ mL of Dawn Base Soap} = 106.61 \text{ g}, \text{ so, } \frac{106.61 \text{ g}}{100 \text{ mL}} = \underline{\underline{1.066 \text{ Density}}}$$

Make 1% solns. of Sunbeam Yellow & Yellow LP as follows:

	I. D.	Lot #	color wt.	D. I. H ₂ O wt.
A) 1% Sunbeam Yellow	C1150	1.00g	99.00g	absorb=10
B) Yellow LP	91061	1.00g	99.01g	absorb=19

Run Absorbs of these solns. in pH 7 Buffer

	100mL flask #	Pippet #	wt.	Conc.	wL	abs	Absorb
1% Sunbeam Yellow	122	—	0.2025g	2.025	400	0.2068	0.102
Yellow LP	928	—	0.2316g	2.316	429	0.4084	0.176

Calculate amount of Quinoline Yellow needed to match Dawn Yellow (in soap)

C) + make soln. ; get absorb.

$$1.05 \text{ Dawn Yellow abs} : 0.5 \text{ Quinolin Absorb} = 0.00219 \text{ mL} ; 0.00219 / 106.61 \text{ density} = 0.2239 \text{ g in } 106.3861 \text{ soap}$$
$$\text{abs} = 0.7688 \text{ at } 420 \text{ wL}$$

D) Try 0.21g of Quinolin soln. in 99.79g soap ; abs = 0.8501 @ 420mL

E) 0.2167g Quinolin soln. in 99.5524g Soap ; abs = 0.6375 @ 420mL ; why is it lower ? try again

F) Recalculate from sample "D" ; $\frac{1.0466 \text{ abs of Dawn Yellow}}{0.8501 \text{ abs of "D"} } \times 0.21 \text{ g } 1\% \text{ Quinolin soln.} = 0.2588 \text{ g}$ of
1% Quinoline needed
(need 0.2588g)

1% Quinoline soln. wt = 0.2645g

(need 99.79g)

abs = 1.2905 at 419 wL

Date 2 May 13, 1996 Signature M. R. Polkberg

Date 1 May 31, 1996 Witness Detective S. Foster

Cont'd on 10007-30

10007-30

Cont'd from 10007-29; started on 10007-28

Tech. Service #9606168

Match Down Yellow detergent as follows: (abs = 1.0466 or 1.0297 at 442)

(in 100g soap)

II, All answers divided by 1000

#2

Colorant	Calculation*	Color Lit. Soap wt.	WL	abs	Calculation	Color Lit. Soap wt.	WL	abs
A Quinoline Yellow	1.05 X 2.23399 = 0.22583	0.22583	99.78879	420	1.0743			
B Pyranine 120	1.05 X 106.61 = 0.37319	0.37319	99.64493	405	1.1820	1.0466 1.1820 X 0.37319 = 0.33029	99.66209	405 1.0380
C Uranine	1.05 X 106.61 = 0.17499	0.17499	99.83439	495	1.0139	0.33259 needed		455 0.4553
D Naphthalimide	1.05 X 106.61 = 0.17499	0.17499	99.82849	447	1.1586	1.0466 1.1586 X 0.17499 = 0.16019	0.16419	99.8335446 1.0107
E Sunbeam Yellow	1.05 X 106.61 = 1.04759	1.04759	98.90339	402	1.0425			
F Yellow LP	1.05 X 106.61 = 0.63609	0.63609	99.44019	429	1.1590	1.0466 1.1590 X 0.63609 = 0.57369	0.57369	99.42639 429 0.9640
F3 Yellow LP	1.0466 0.9640 X 0.57369 = .62279	.62279	99.37889	428	1.1020			

Date May 14, 1996

Date May 31, 1996

Signature M.R. Pilkington

Witness D. L. S. Rose

Cont'd on 10007-31

10007-31

Cont'd from 10007-30; started on 10007-28

Tech Service # 9606168

Matching Dawn Blue & Green detergents & Yellow

A Make a 1% soln. of Patent Blue, Lot J1182, as follows:
 color wt = 1.009 D.I. water wt = 99.009

B Take $\frac{25.009}{100.009}$ of this soln. & place in 75.01g D.I. water. (0.25% soln.)
 Check absorb:

wt = 0.1665g in 100ml flask #122; conc = 1.665 absorb = 0.087; 62.8wl @ 0.1442abs
 Check "A":

wt = 0.1441g in 100ml flask #928; 62.8wl @ 0.5052; Absorb = 0.35 conc = 1.441

Calculate wt needed to adjust abs to match Dawn Blue detergent.

$$\frac{0.9067 \text{ Dawn Blue}_2}{0.35 \text{ Patent absorb}} \times 0.1441g \text{ needed}$$

P. Blue 1% soln.

C1 wt = 0.1454g Dawn Base wt = 101.2516g WL = 634 abs = 0.4385

C2 wt = 0.2661g " " = 49.7278g " = 634 " = 0.8474; visually seems very close to 5th.

Need to match the 495wl, 0.3177abs peak in Dawn Yellow & blend with our yellows:
 #1

Colorant	Calculation	Color wt ^{500P} wt ₁	WL	abs	adjustments when blending with yellow
D 1% Pyranine 10007-28E	$\frac{0.3177}{0.5139} \times 0.5742 - 0.2372g$ needed	0.2333g	99.7670g	455	0.3174 ✓
E 1% Naphthalimide 10007-28E	$\frac{0.3177}{1.1586} \times 0.1773 - 0.0486g$	0.0480g	99.9659g	446	0.3067 ✓
F 1% Uranine 10007-28F	$\frac{0.3177}{1.0139} \times 0.1764 - 0.0572g$	0.0571g	99.9437g	495	$\frac{0.3177}{0.3353} \times 0.0571g = 0.0571g$ needed

Make solns. of 1% Sunbeam Yellow with Fluorescent mat'l's.

1%	Sunbeam lot#, FL Mat'L & wt.	Base wt,	WL	abs	WL	abs	WL	abs
G1	1.0923g NI, 0.3053g	98.331g	432	2.3507	439	2.3577	444	2.3247
H1	1.0935g Pyranine, 0.294g	98.722g	405	1.8088	455	shoulder 0.81593		

G+H need
work

Date May 15, 1996

Date May 31, 1996

Signature M. R. Pillai

Witness D. S. F.

Cont'd on 10007-32

10007-32

Cont'd from 10007-31 ; Started on 10007-28

Tech. Service # 9606168

Make Solns. of 1% Quinoline Yellow with Fluorescent mat'l's.

	1% Quinoline wt	Fl. Mat'l. + wt	Base wt	wL	abs	wL	abs	wL	abs
A1	0.2243g	NI, 0.3054g	99.4681g	427	2.4384	443	2.5544	444	2.5595
B1	0.2222g	1% Pyranine, 0.2330g	99.5399g	408	1.5573	443	1.3692		

Adjust color levels of the yellows with Fluorescent mat'l's. ($\frac{\text{desired abs}}{\text{current abs}} \times \text{last wt.}$)

	Colorant + wt. ^{need}	Fl. Mat'l. + wt. ^{need}	Base wt ^{need}	wL	abs	wL	abs	wL	abs
31G2	1% Sunbeam (0.4849g)	0.4843g	NI (0.1366g)	99.3739g	359	0.6976	437	1.1973	
31H2	1% Sunbeam (0.6327g)	0.6325g	Pyranine 0.0968g	99.4343g	405	0.9267	shoulder 455	0.40809	
32A2	1% Quinoline (0.0963g)	0.0944g	NI (0.0415g)	99.8750g	422	0.7234	443	0.7675	
32B2	1% Quinoline (0.1493g)	0.1505g	Pyranine 0.1887g	99.5126g	408	1.1711	443	0.9834	

The 1% Quinoline soln. is beginning to "fall out" Make a new batch & filter;
 C 100g Quinoline Yellow, Lot 08919 JF ; 99.00g D.I. H₂O.

Recalculate color levels as follows & mix :

	Colorant wt. ^{wt.}	Fl. Colorant ^{wt.}	Base ^{wt.}	wL	abs	wL	abs
31G3	1% Sunbeam, 0.5466g	0.5483g	10% NI, 0.1627g	0.1521g	99.3012g	99.3083g	437 1.2812
31H3	1% Sunbeam 0.6327g	0.6321g	1% Pyranine 0.0500g	0.0512g	99.3173g	99.3147g	405 0.7534
32A3	1% Quinoline 0.1441g	0.1442g	10% NI, 0.0612g	0.0622g	99.7447g	99.8947g	423 0.9221
	(45% increase over A2)						
32B3	1% Quinoline 0.1493g	0.1491g	1% Pyranine 0.0600g	0.0633g	99.7907g	99.8901g	418 0.7350
	(Go with 31H1 for stabilities) (Also use 10007 3EDR, naphthalimide in stabilities)						

Date May - 16, 1996

Date May 31, 1996

Signature M-R. Palermo

Witness J. T. S. Jr.

Cont'd on 10007-33

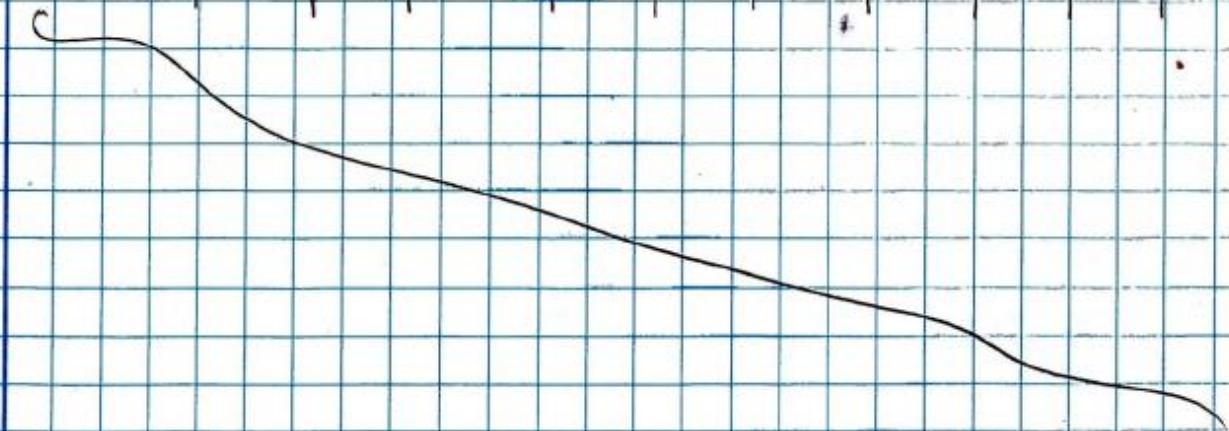
Cont'd from 10007-32; started on 10007-28

10007-33

Tech. Service # 9606168

Additional adjustments to yellow blends:

Colorant	Fl. Colorant		Base		wt.	wt.	abs	wt.	abs
	wt needed	wt	wt needed	wt					
3164 1% Sunbeam	0.5461g	0.5507g	10% N.I.	0.1217g	0.1206g	99.7322g	99.3655g	435	1.1453
32A4 1% Quinoline	0.1543g	0.1565g	10% N.I.	0.0603g	0.0591g	99.7854g	99.8949g	422	1.0479 443
32B4 1% Quinoline	0.1678g	0.1652g	1% Pyranine	0.0600g	0.0650g	99.7782g	99.7731g	418	0.9036 442
3165 1% Sunbeam	0.5507g	0.6511g	10% N.I.	0.0903g	0.0881g	98.5488g	98.5262g	432	0.9610
31H4 1% Sunbeam	1.0935g	1.0857g	1% Pyranine	0.400g	0.429g	98.7665g	98.7801g	404	1.4495
32A5 1% Quinoline	0.1700g	0.1733g	10% N.I.	0.0500g	0.0515g	99.7998g	99.8460g	421	1.0740 442
32B5 1% Quinoline	0.1899g	0.1866g	1% Pyranine	0.0600g	0.0656g	99.7558g	99.7505g	419	0.9777 442
31H5 1% Sunbeam	1.0935g	1.0876g	1% Pyranine	0.0715g	0.0707g	99.8419g	99.8664g	404	1.2473
✓ 32A6 1% Quinoline	0.208g	0.2074g	10% N.I.	0.0360g	0.0350g	99.7826g	99.7635g	421	1.1892 442
32B6 1% Quinoline	0.2345g	0.2395g	1% Pyranine	0.0360g	0.0366g	99.7271g	99.7156g	419	1.2220 442
33C1 1% Sunbeam	1.100g	1.0099g	1% Uranine	0.0451g	0.0445g	98.8549g	98.8444g	403	0.8154 491



Date May 17, 1995

Signature M. P. Pilling

Date May 31, 1995

Witness D. S. Foster

Cont'd on 10007-35

10007-34

Cont'd from 10007-25; started on 10007-8

4 Week L.A.B. Readings on Colored Detergents in PVA (All® & Arm & Hammer)

Sample I.D.	L	A	B	E	L	Delta's on filter Paper	Visual staining
10007-8A1, Red ST/H ₂ O base in All®	93.09	9.74	-0.63	7.23	-3.40	major	
10007-8B1, Red ST/PVA Base in All®	97.20	3.92	1.31	2.96	-1.04	major	
10007-8C2, Blue HP/H ₂ O Base in Arm + Hammer®	97.41	-1.64	-7.67	3.69	-1.70	very minor	
10007-8D2, Blue HP/PVA Base in All®	95.87	-1.93	-1.18	3.42	-1.63	moderate	
10007-8E2, Blue HP/PVA Base in Arm + Hammer®	99.55	-0.70	-5.20	3.14	-1.58	minor	
10007-8F2, Blue HP/H ₂ O Base in All®	93.11	-3.43	-3.17	5.19	-3.03	moderate	
10007-8G2, Red ST/H ₂ O base in Arm + Hammer®	96.65	8.47	-4.12	5.38	-2.51	moderate	
10007-8H2, Red ST/PVA base in Arm + Hammer®	98.54	6.32	-3.59	4.36	-2.14	minor	
10007-8I, All®, no color	99.13	0.67	3.02	1.05	-0.05	none	
10007-8J, Arm + Hammer® no color	102.43	1.01	-1.38	0.15	-0.15	none	

Date May 17, 1996
 Date May 31, 1996

Signature M. R. Elling
 Witness D. T. S. Fisher

10007-35

Cont'd from 10007-33; started on 10007-28

Tech. Service #9606168

Scale up Yellow detergent samples as follows:
Also scale Blue detergent sample

	1. D. Colorant + wt	Fluorescent Color & wt	Base wt	wL	abs	wL	abs
A	30D2 10% NI	10% NI, 0.44g	259.57g	445	1.0821		
B	31G5 1% Sunbeam, 1.49g	10% NI, 0.28g	268.28g	434	1.0583		
C	31H5 1% Sunbeam, 2.94g	1% Pyranine, 0.19g	266.87g	404	1.2636		
D	32A6 1% Quinoline, 0.56g	10% NI, 0.10g	269.35g	421	1.2180	442	1.2408
E	33C1 1% Sunbeam, 2.73g	1% Urarine, 0.13g	267.13g	403 394	1.0107 1.0103	442	0.3267
F	31C2 1% Patent Blue, 0.73g		269.28g	634	0.9889		

Samples of these were placed in oven #297, set at 50°C & oven #298, set at 38°C for stability testing, to be checked after 2 wks & 4 wks.

Included in these samples were "G", Dawn Yellow control,
& "H", Dawn Blue control.

Additional samples will be placed in the Xenon apparatus:

"G", Dawn Yellow	419	1.0466	442	1.0297	495	0.3177
"H", Dawn Blue	633	0.9067				

Date May 20, 1996
Date May 31, 1996

Signature M. R. P. Blang
Witness D. S. Guter

Cont'd on 10007-36

Cont'd from 10007-35; started on 10007-28

Tech. Service # 9606168

Results of Initial Xenon testing:

I.D.	3 hr results Visual	6 hr. results								
		wl	abs	wl	abs	Visual	wl	abs	wl	abs
35A, 10% NI	yellower than control "35G"	444	1.0498			more difference than at 3 hrs	445	1.0373		
35B, 1% Sunbeam + 10% NI	"35G" slightly redder	434	0.9450			"35G" is paler & redder	436	0.8728		
35C, 1% Sunbeam + 1% Pyranine	"35G" redder than this	404	1.1879			~ same as "35B"	405	1.0823		
35D, 1% Quinoline + 10% NI	~ same as "35C"	421	1.1888	442	1.2079	~ same as "35B"	422	1.1653	442	1.1859
35E, 1% Sunbeam + 1% Ullmannine	~ same as "35C", but seems paler	407	0.7944			~ same as "35B"	407	0.6332		
35F, 1% Patent Blue	~ same as "35H"	634	0.9203			~ same as "35H"	634	0.8887		
35G, Dawn Yellow	Control; seems slightly redder & less fluorescent than initial	419	0.906	442	0.8855	paler & less fluorescent than initial	419	0.9491	442	0.8822
35H, Dawn Blue	Control; ~ same as initial	633	0.7609			~ same as initial	633	0.6496		

9 hr & 12 hr results are on 10007-39

Date May 21, 1996
 Date May 31, 1996

Signature M.R. Johnson
 Witness D. S. Foster

cont'd on 10007-37

10007-37

Contd from 10007-36; started on 10007-28

Tech. Service # 9606168

Color match Dawn Green: Peaks at 418(5529), 442(4873), 479(4910),
634(6825)cal. amount of Patent Blue(1%) needed; $\frac{0.6875 \text{ abs}}{0.4469 \text{ abs}} \times \frac{0.14549}{0.14549} = 0.52679$ needed
 $0.52679 \text{ abs}(3101)$
 0.43805 cal. amount of 1% Sunbeam needed;
+ 10% N.I.
~~0.5524 abs~~ last trial; 0.56119 (3165)
0.09019

" " " 10% N.I.

" " 0.16419 (3002)

" " " 1% Sunbeam
1% Pyranine" " 1.08769 (3145)
0.07079" " " 1% Quinoline
10% N.I." " 0.20749 (3246)
0.10350" " " 1% Sunbeam
1% Uranine" " 1.00999 (33C1)
0.04459

Color	wt	wt	wt	Patent Blue Wt.	Base Wt.	WL	abs	WL	abs	WL	abs
A 1% Sunbeam	0.5420g	10% N.I.	0.0901g	0.2396g	99.1184g	425	0.9970	634	0.8928		
B1 — — 10% N.I.	0.1564g	422.84g	99.6109g	445	1.0490	634	0.8134				
C 1% Sunbeam	1.0491g	1% Pyranine	0.0700g	0.2293g	98.7252g	406	1.3082	634	0.8001		
D 1% Quinoline	0.1970g	10% N.I.	0.0375g	0.2345g	99.5408g	420	1.2192	442	1.1710	634	0.808
E 1% Sunbeam	0.0848g	1% Uranine	0.0488g	0.2267g	99.6669g	412	0.2015	495	0.2940	634	0.7942



Date May 21, 1996

Date May 31, 1996

Signature M. R. Pillbury

Witness D. T. S. Foster

cont'd on 10007-38

10007-39

Cont'd from 10007-37; Started on 10007-28

Tech. Service # 9606168

Continue color matching Dawn Green!

	Color	wt	P. Color	wt	MoPBlue/wt	Base wt	wL	abs	wL	abs	wL	abs	Comment
37A2	1% Sunbeam	0.3062g	10% NI	0.0487g	0.1965g	99.4586g	418	0.5543 0.5631	634	0.6853			very close match
37B2	—	—	10% NI	0.0834g	0.1975g	99.5480g	445	0.5606	634	0.6958			very close match
37C2	1% Sunbeam	0.4430g	1% Pyranine	0.0268g	0.2036g	99.3353g	406	0.5929	634	0.7198			too blue
37D2	1% Quindine	0.0860g	10% NI	0.0245g	0.1905g	99.6280g	420	0.6196	442	0.6016	634	0.6636	need more yellow
37E2	1% Sunbeam	0.2315g	1% Uranine	0.0349g	0.2004g	99.5215g	411	0.3277	495	0.2260	634	0.7100	need more yellow, less blue
37C3	1% Sunbeam	0.4537g	1% Pyranine	0.0318g	0.1953g	99.3210g	406	0.6117	423	0.4800	634	0.6888	add more yellow + less blue
37D3	1% Quindine	0.1486g	10% NI	0.0268g	0.1898g	99.6255g	420	0.9124	423	0.8996	442	0.8803	add slightly more yellow
37E3	1% Sunbeam	0.3893g	1% Uranine	0.0363g	0.1952g	99.4018g	410	0.4572 0.4117	494	0.2356	634	0.6758	good dye match
37F4	1% Sunbeam	0.4637g	1% Pyranine	0.0385g	0.1884g	99.7331g	406	0.6419	423	0.4951	634	0.6608	add more yellow
37D4	1% Sunbeam	0.1545g	10% NI	0.0302g	0.1864g	99.6345g	420	0.9787	423	0.9652	442	0.9467	add more yellow
37E5	1% Sunbeam	0.8929g	1% Pyranine	0.0456g	0.1961g	99.1561g	406	0.8936	423	0.7107	634	0.6935	too blue
37D5	1% Quindine	0.2047g	10% NI	0.0423g	0.1887g	99.5864g	421	1.2822	423	1.2604	442	1.2604	close but add more yellow
37C6	1% Sunbeam	1.3838g	1% Pyranine	0.0891g	0.1886g	98.3409g	406	0.8836 1.6653	423	1.3269	634	0.6575	too blue
37C7	1% Sunbeam	1.7307g	1% Pyranine	0.1208g	0.1430	98.2892g	405	2.0052	423	1.6061	633	0.4951	bring the blue up
37C8	1% Sunbeam	1.6840g	1% Pyranine	0.1130g	0.1623g	98.2020g	405	0.6288	423	1.3457	454	0.2543	
37C9	"	1.5732g	1% Pyranine	0.1197g	0.1832g	98.0002g	634	0.6357					needs more yellow

Date

May 22, 1996

Date

May 31, 1996

Signature

M. R. Pilkington

Witness

S. F. S.

Cont'd on 10007-39

Cont'd from 10007-38; started on 10007-28

Tech. Service #9606168

Further Xenon Testing of Samples from 10007-36:

ID	visual	9 hr. Results				12 hrs. Results				
		wL	abs	wL	abs	wL	abs	wL	abs	
35A, 10% NI	Samples "A" - "D"	423	0.8480	443	1.0248	A+B, ~;	423	0.8433	443	1.0152
35B, 18 Sunbeam + 10% NI	were all ~ & were yellower than the control	423	0.7744	437	0.8344	yellower than control "G" "G" also paler.	423	0.7240	438	0.7901
35C, 1% Sunbeam + 1% Uranine	"G"	406	0.9443	423	0.8475	"G" is redder	406	0.9245	423	0.7980
35D, 1% Quinoxoline + 10% N.I.		421	1.1533	442	1.1640	"G" is redder	422	1.1379	423	1.1308
442							442	1.1464		
35E, 1% Sunbeam + 1% Uranine pale	= to "G" control;	408	0.5792	423	0.5381	Paler than "G", but "G" is redder	409	0.5571	423	0.5206
35F, 1% Patent Blue	Looked bluer than control "H"	634	0.8538			More bluer than control "H"	634	0.8333		
35G, Dawn yellow	Paler than control after 6 hrs.	419	0.9352	423	0.9122	= redder than after 9 hrs	419	0.9194	423	0.8970
35H, Dawn blue	Paler than control after 6 hrs.	633	0.5316			Paler than at 9 hrs	633	0.4463		

Make a fresh lot of 71% P.V.A. as follows: ~~10007-38~~Air vol 523, lot. #D4248, wt #4402 - wt = 12.02g ; DI H₂O wt = 153.68g

Date May 23, 1996

Date May 31, 1996

Signature M. R. Kilbrey

Witness D. S. Foster

Cont'd on 10007-42

Creating Colored Dial Laundry Detergent in P.V.A. for Color Migration Tests; ref. 10007-8

Materials:

Lot#

Airval 523, P.V.A., 7.1% Soln. in DI, H ₂ O	10007-39
Red ST, ~10 absorb	R.D.101
Blue HP, ~15 absorb	R.1126
D.I. H ₂ O	

Dial® uncolored laundry detergent

Mix color solns. as follows:

Color	Color Wt.	P.V.A. wt	D. I. H ₂ O Wt.
A Red ST	0.75g need 0.75g	—	33.10g need 33.10g
B Red ST	0.75g	33.10g	—
C Blue HP	0.50g need 0.50g	—	33.10g
D Blue HP	0.50g	33.10g	—

Spray these solns. onto uncolored detergent samples as follows, using Crown Spray Tool & power pack.
 Pre-dry detergent in oven #298@ 77°C for ~ 2min, 15sec.

Detergent Wt. (g)	Deterg. Dry. time	Soln. + wt. sprayed on	Comments
A1 50.00g	2min, 16 sec.	"A", Red ST/H ₂ O, 0.86g	sprayed well; fairly even
B1 50.01g	2min, 14 sec.	"B", Red ST/PVA, 0.49g	Pink deterg.; red clumps; didn't spray well.
C1 50.03g	2min, 14 sec.	"C", Blue HP/H ₂ O, 0.98g	sprayed well, even
D1 50.02g	2min, 16 sec	"D", Blue HP/PVA	sprayed very badly
			* Try diluting both PVA solns with 31.00g H ₂ O(DI) to see if sprays better
E1 50.00g	2min, 14 sec	Red ST/PVA/H ₂ O, 1.40g	sprayed better; still a little clumpy
F1 50.05g		Red ST/PVA/H ₂ O	

Combine with uncolored detergent as follows:

Cont'd on 10007-41

Date May 24, 1996
 Date May 31, 1996

Signature M-R. Kellberg
 Witness Jennifer S. Parker

Cont'd from 10007-40

10007-41

Combine colored bases with white dial:
(need 5mg) (need 400.0mg) Initial

Colored-Base I.D.	Base wt.	Uncolored Dial wt.	L	A	B
40A2 40A1-Red ST/H ₂ O	400.05g	400.069	97.58	-1.60	5.41
40B2 40B1-Red ST/PVA	15.14g	400.069	99.12	-3.82	6.28
40C2 40C1-Blue HP/H ₂ O	15.11g	400.069	96.50	-5.82	3.15
40D2 40-D1-Blue HP/PVA	15.20g	400.15g	99.46	-4.29	6.42
40E2 40E1-Red ST/PVA/H ₂ O	15.01g	400.15g	99.02	-3.61	6.22
40F2 40F1-Blue HP/PVA/H ₂ O	15.15g	400.16g	99.01	-5.02	5.86

These samples were placed in Jenney
Humidity Chamber @ 80°F & 80% R.H.

Will be checked on

6-6-96

Date May 24, 1996

Date May 31, 1996

Signature

M. R. Pfeiffer

Witness

Jessie S. Foster

Cont'd from 10007-38; started on 10007-28

Tech. Service # 9606168

continue color matching Dawn Green

	1% Sunbeam wt.	1% Pyranine wt.	1% Patent Blue wt.	Base wt.	wL	abs	wL	abs	wL	abs
--	----------------	-----------------	--------------------	----------	----	-----	----	-----	----	-----

38C10	2.5601g	0.1925g	0.1937g	920892g	406	3.0378	423	2.4568	633	0.6493
38C11	1.7093g	0.1262g	0.1919g	979665g	407	2.0119	423	1.6082	634	0.6509

The amount of Blue was supposed to have been 0.1830g & the sample appears a little blue. We'll use this formulation with the other four "Greens" for stability testing.

Now, scale-up the "Green" samples for stability testing:

L.D.	Color & wt	Color & wt	Patent Blue wt.	Base wt.	wL	abs	wL	abs	wL	abs
A 37A2	1% Sunbeam, 0.83g N.I., 0.13g	1% Pyranine, 0.83g N.I., 0.13g	0.53g	268.54g	418	0.5646	423	0.5628	634	0.6495
B 37B2	—	10% NI, 0.23g	0.53g	268.78g	423	0.5089	445	0.5976	634	0.7063
C 37C11	1% Sunbeam, 4.62g 1% Pyranine, 0.34g	—	0.49g	264.51g	407	2.0911	423	1.6624	634	0.6388
D 37D5	1% Quinoline, 0.55g 10% N.I., 0.11g	1% Quinoline, 0.55g 10% N.I., 0.11g	0.52g	268.88g	420	1.2104	423	1.1962	634	0.6513
E 37E3	1% Sunbeam, 1.05g 1% Quinoline, 0.11g	1% Quinoline, 0.11g	0.52g	268.38g	409	0.4729	423	0.4328	634	0.6713
F Dawn Green	—	—	—	—	418	0.5529	442	0.4873	634	0.6825
					499	0.1910				

Samples of "A"- "E" were placed in ovens #297, set at 50°C & #298, set at 38°C & will be checked after 2 wks. & 4 wks.

Additional samples were placed in the Xenon chamber.

Date May 26, 1996

Date May 31, 1996

Signature Mike R. Pilling

Witness D. T. S. Foster

Continued on 10007-43

Cont'd from 10007-42; started on 10007-28

Tech. Service #9606168

Xenon Test Results on "Green" formulations

3 Hour Results

6 Hour Results

I.D.	Visual	wL	abs	wL	abs	Visual	wL	abs	wL	abs
42 A, Sunbeam/NI P. Blue	Greener than control; Bluer than "C" & "D"	421 418	0.4952 0.4933	423	0.4940	Greener than "F"; not quite as pale as "F" Slightly bluer though	421	0.4595	423	0.4586
42 B, N.I. / P. Blue	Much greener than "F"; slightly yellower than "A"	423 634	0.4996 0.6659	443	0.5596	Much greener than "F"; Bluer than "C"	423 634	0.4945 0.6462	443	0.5572
42 C, Sunbeam Pyranine, P. Blue	^{Yellowed} Much greener than "F"; Yellower than "A"	404 634	1.9802 0.6352	423	1.6007	"C" & "D" are almost alike - much greener/yellower	405 634	1.8694 0.6293	423	1.5291
42 D, Quinoline/ NI / P. Blue	^{Yellowed} Much Greener than "F"; Yellower than "A"; slightly bluer than "C"	420 422	1.2006 1.1747	423	1.1850	than "F" & yellower than all samples	421 442	1.1981 1.1607	423	1.1745
42 E, Sunbeam/ Uranine, P. Blue	Bluer than "F"; Pale than "A" & "D"; Much bluer than "A" - "D"	412 443	0.3391 0.1050	423	0.3035 0.6471	Aqua blue; bleakest sample	411 443	0.3117 634	423	0.2745 0.6471
42 F, Dawn Green	Paler than "A" - "D"; Paler than initial	418 442	0.4903 0.4276	423	0.4672	Not much paler than at 3 hrs.	418 442	0.4901 0.4276	423	0.4677 0.5289
				634	0.5659					

Date May 29, 1996

Date May 31, 1996

Signature

Witness

M.R. Pilbeam

Dentise S. Pilbeam

Cont'd on 10007-44

Cont'd from 10007-43; Started on 10007-28

10007-44

Tech. Service #9606168

More Xenon Test results on "Green" formulations

9 Hours Results

12 Hour Results

1. D	Visual	wl	abs	wl	abs	Visual	wl	abs	wl	abs
42A, Sunbeam/ NI / P.Blue	Slightly greener than "F"; Bluer than "B"	421 674	0.4217 0.6297	423 634	0.4209 0.6061	Slightly yellower than "F"; Much bluer than "D"	428 634	0.3910 0.6173	423 634	0.3974 0.5878
42B, NI / P.Blue	Much yellower than "F"; Bluer than "C"	423 634	0.4994 0.6061	440 634	0.5475 0.6170	Yellower than "F"; much Bluer than "E"	423 634	0.4950 0.5878	441 634	0.5385 0.6140
42C, Sunbeam/ Pyranine/P.Blue	Much yellower & deeper than "F"; Slightly yellower than "D"	406 634	1.7111 0.6170	423 634	1.4098 0.6170	Extremely yellower & deeper than "F"; Slightly yellower than "D"	405 634	1.5858 0.6140	423 634	1.3234 0.5944
42D, QuinoLine/ NI / P.Blue	Much yellower & deeper than "F"; slightly Bluer than "C"	420 442	1.1549 1.1202	423 634	1.1436 0.6050	Much yellower & deeper than "F"; a hair Bluer than "C"	421 442	1.1366 1.1004	423 634	1.1258 0.5944
42E, Sunbeam/ Uranine/P.Blue	Almost totally Blue; \oplus	412 634	0.2860 0.6077	423 634	0.2924 0.6077	\sim same as 9 hr. check	412 634	0.2747 0.5998	423 634	0.2437 0.4229
42F, Dawn Green	Very slightly paler than 6 hr. sample	418 442 633	0.4778 0.4182 0.4648	423 511	0.4578 0.0406	\sim same as 9 hr. check	418 442	0.4705 0.4108	423 633	0.4514 0.4229

Date May 30, 1996

Date May 31, 1996

Signature M.R. Pilbury

Witness J.L.S. Forte

Cont'd on 10007-46

10007-45

Calculate Absorbts of KLM 10091-12 & 10091-15

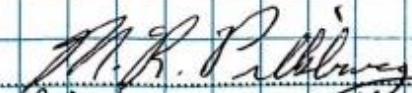
Run in Methanol, adding 4 drops of 10% NaOH to each dilution

L.D.	wt.	100ml flasks	2mL Pipet	Concn	WL	abs	Absorb
10091-12	0.1010g	167, 939	426	0.0202	496	1.2401	61.39
10091-15	0.1000g	880, 887	7516	0.0200	496	1.1507	57.54

Date May 31, 1996
Date June 14, 1996

Signature

Witness




Cont'd from 10007-44; started on 10007-28

10007-46

Tech. Service # 9606168

2 wk Absorbs checks on oven & shelf stabilities for yellows & blues

I.D.	wL	abs	wL	abs	I.D.	wL	abs	wL	abs	wL	abs
10007-35A	446	1.0880			10007-35E, shelf	402	1.0055	491	0.3224		
N.I., Shelf					Sunbeam, Iodamine						
10007-35A, 38°C	446	1.0934			10007-35E, 38°C	403	1.0285	491	0.3316		
N.I.					Sunbeam/Iodamine						
10007-35A, 50°C	445	1.1038			10007-35E, 50°C	407	1.0438	491	0.3309		
N.I.					Sunbeam/Iodamine						
10007-35B, Shelf	435	1.0581									
Sunbeam, NI											
10007-35B, 38°C	434	1.0683			10007-35F, shelf	634	0.9475				
Sunbeam, NI					Patent Blue						
10007-35B, 50°C	433	1.0824			10007-35F, 38°C	634	0.9424				
Sunbeam, NI					Patent Blue						
10007-35C, Shelf	406	1.2571			10007-35F, 50°C	634	0.9276				
Sunbeam, Pyranine					Patent Blue						
10007-35C, 38°C	405	1.2823			10007-35G, shelf	420		0.9892	442	0.9691	495
Sunbeam, Pyranine					Dawn Yellow	420		0.9892	442	0.9691	495
10007-35C, 50°C	405	1.3049			10007-35G, 38°C	420	0.9995	442	0.9748	495	0.2950
Sunbeam, Pyranine					Dawn Yellow	420	0.9995	442	0.9748	495	0.2990
10007-35D, Shelf	420	1.2254	442	1.2680	10007-35G, 50°C	419	1.0128	442	0.9748	495	0.2941
Quinoline/ Sunbeam Pyranine					Dawn Yellow	419	1.0128	442	0.9748	495	0.2941
10007-35D, 38°C	421	1.2244	442	1.2662	10007-35G, 50°C	419	1.0128	442	0.9748	495	0.2941
Sunbeam Pyranine					Dawn Yellow	419	1.0128	442	0.9748	495	0.2941
Quinoline, NI											
10007-35D, 50°C	421	1.2673	442	1.2807	10007-35H, shelf	633	0.8442				
Sunbeam Pyranine					Dawn Blue	633	0.8442				
Quinoline, N.I.					10007-35H, 38°C	633	0.8508				
					Dawn Blue	633	0.8508				
					10007-35H, 50°C	633	0.8802				
					Dawn Blue	633	0.8802				

Date: June 3, 1996

Date: June 14, 1996

Signature

M. R. Poblocki

Witness

Rich Dunstall

cont'd on 10007-47

see 10007-54 for wt. absorbs

Cont'd from 10007-46; started on 10007-28

10007-47

Tech. Service # 9606168

Perform stain testing on all ^{matched} formulations for Yellow, Blue, & Green ~~& control~~ samples. Use swatches of 13-fiber-strip & cotton Terry cloth for each.

Soaked ~~swatches~~ in samples for ~15 min. Remove swatches from samples & remove excess sample from fabric; let dry overnight. Wash swatches in cold H₂O with no agitation, changing water periodically 'til H₂O remains clear. Hang swatches to dry overnight then compare to untinted control swatches.

Stain Testing Results:

Exp. # & I.D. | Results:

10007-35A, N.I.	13-Fiber	NO Staining
	Terry	NO Staining
10007-35B, Sunbeam/N.I.	13-Fiber	" "
	Terry	" "
10007-35C, Sunbeam/Pyranine	13-Fiber	" "
	Terry	" "
10007-35D, Quinoline/N.I.	13-Fiber	" "
	Terry	" "
10007-35E, Sunbeam/Uranine	13-Fiber	" "
(5/18)	Terry	" "
10007-35F, Patent Blue	13-Fiber	" "
(20/25)	Terry	" "
10007-35G, Dawn Yellow	13-Fiber	" 4
	Terry	" "
10007-35H, Dawn Blue	13 Fiber	" "
	Terry	" "
10007-42A, S.b./N.I./P.Blue	13 Fiber	" "
	Terry	" "
10007-42B, N.I./P.Blue	13 Fiber	" "
	Terry	" "
10007-42C, Pyranine/P.Blue	13 Fiber	" "
	Terry	" "
10007-42D, Quinoline/N.I./P.Blue	13 Fiber	" "
	Terry	" "
10007-42E, Uranine/P.Blue	13 Fiber	" "
	Terry	" "

Date June 4, 1996

Date June 14, 1996

10007-42F
Dawn Green Terry

Signature M. R. Killeberg

Witness Rich Durstall

cont'd on 10007-48

10007-48

Cont'd from 10007-47; started on 10007-28

Tech. Service # 9606168, "g Color Retained from Xenon & Oven Stabilities"			
Expt # & ID.	Type	% retained	Xenon % retained
10007-35A, NI	shelf	445 1.0880	445 93.82
	28°C Oven	445 1.0934	
	50°C Oven	445 1.1038	
10007-35B, S.b./N.I	shelf	434 99.98	434 82.47(6 hrs)
	38°C	434 100.94	
	50°C	434 102.28	
10007-35C, S.b./Pyramine	shelf	404 98.49	404 73.16
	38°C	404 101.48	
	50°C	404 103.27	
10007-35D, Quinoline/NI	shelf	421 100.61	421 93.42
		442 100.58	442 92.39
	38°C	421 100.53	
		442 100.44	
	50°C	421 104.05	
		442 103.22	
10007-35E, S.b./Uranine	shelf	402 99.49	407 55.18
		421 98.68	
	38°C	402 101.76	
		491 101.50	
	50°C	402 103.27	
		491 101.29	
10007-35F, Patent Blue	shelf	634 100.06	634 88.00
	38°C	634 99.52	
	50°C	634 97.96	
10007-35G, Dawn Yellow	shelf	419 94.52	419 87.85
		442 94.11	442 82.89
		495 92.85	
	38°C	419 95.50	
		442 94.67	
		495 94.11	
	50°C	419 96.77	
		442 94.67	
		495 92.57	

Date June 4, 1996

Signature M. R. Pilliar

Date June 14, 1996

Witness Clinch Sunstall

Cont'd on 10007-49

Cont'd from 10007-48; started on 10007-28

10007-49

Tech. Service 9606168, "Cont'd Color retained from Xerox + Over Stabilities"

10007-50

Cont'd from 10007-49; started on 10007-28

Tech. Service #9606168; Cont'd 'odor retained' from Xenon & Oven Stabilities
Oven Stabilities

Exp # & ID 10007-42D, Quinidine/MT/PBlue	Type 38°C	% retained		Xenon	
		abs 2wks	4wks		
	50°C				
10007-42F, Sulphurine/PLBlue	Shelf	409	58.09	442	44.29
	38°C	423	56.31	495	32.89(3 hrs)
	50°C	499	27.70	634	89.35
10007-42F, Dawn Green	Shelf	418	85.10	442	46.61
	38°C	499	27.70(3 hrs)	634	61.96
	50°C				

Date June 4, 1996

Date June 14, 1996

Signature M. R. Pihlava

Witness

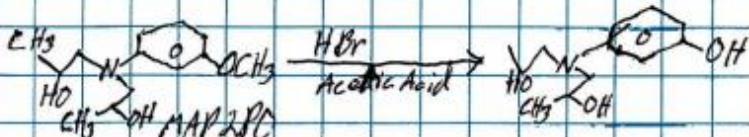
Oletha Junstall

Cont'd on 10007-54

Rxn. of Methamino phenol 2PO with Hydrobromic Acid + Acetic Acid
 starting (try to remove methal group)
 Materials:

<u>Lot</u>	<u>M.W.</u>	<u>mols</u>	<u>wt. needed</u>	<u>Actual wt.</u>
MAP 280	239	0.02	4.78g	4.83g
Hpt, 48%	10320EG	80.92	0.04	6.74g

Acetic Acid Glacial 2504 KPMF — — ~25mL ~25mL



Put charges in a 250mL oneneck flask & place on steam bath for 1hr, 20min

Remove from steam & neutralize pH with 10% NaOH.

At \sim pH 4 to 5 particulates began to form. 175.63g NaOH added at this pt.

At pH 6, larger globules of what should be product had formed.

At \sim pH 7, 281.30g 10% NaOH had been added. Large globules of crimson product are in a pale rose/cream colored fluid.

Pour into a 500mL sept. funnel & extract twice with ~~dimethyl~~ ^{dichloromethane} or

Wash in 5.75% Sodium bicarbonate soln. twice

Evaporate to remove dichloromethane.

Submit product & MAP2PO for Mass spec & NMR

Date Jane 5, 1996

Date June 14, 1996

Signature

Witnesses

M.R. Phillips

Rich Sunstall

Compare N.I. at Normal Level in Oil with 5 Times Normal

Use ALM10045-46; Di-PA-10-NI (in Hydrocal 45)
and Mobil 1 15W-50 oil

* The normal ratio is .32g NI/sdm. in 100g oil

A) Add 0.26 g 10045-46 to 80.04 g oil + stir

B) Add 1.28 g 10045-46 to 80.01 g oil + stir

The customer's current product is a much deeper than our's, though both are at same M.L. It was hoped that by increasing the amount of color used, the depth might match-up better. This did not happen.

Date June 5, 1996

Date June 14, 1996

Signature M.R. Robinson

Witness Quincy A. Howell

10007-53

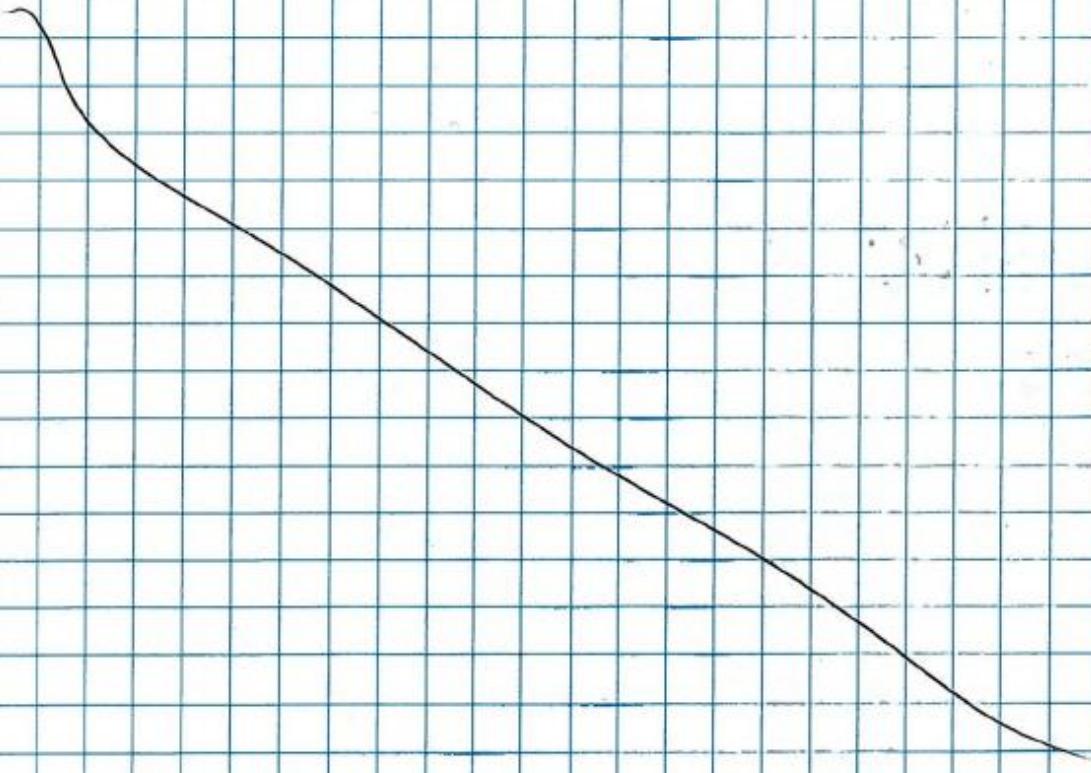
Cont'd from 10007-41; started on 10007-40

2 Week LAB Reading on Colored Dial ^(R) Deltag. in P.V.A.

Deltas

I.D.	L	A	B	E	L
10007-40C2, Red ST/H ₂ O/Dial	98.49	0.45	4.96	2.36	7.09
10007-40B2, Red ST/PVA/Dial	99.23	-3.49	6.43	0.41	0.11
10007-40C2, Blue H ₂ O/Dial	95.27	-6.02	1.73	1.89	-1.23
10007-40D2, Blue H ₂ PVA/Dial	98.48	-2.30	5.83	0.49	-0.29
10007-40E2, Red ST/ PVA/H ₂ O/Dial	98.48 98.48	-6.22 -2.30	98.48 5.83	1.47	-0.54
10007-40F2, Blue H ₂ O/ PVA/H ₂ O/Dial	98.41 98.90	-4.11	5.47	1.00	-0.11

Samples continue in Humidity chamber 'til 6:30-96



Date June 6, 1996
 Date June 14, 1996

Signature

M. R. Palmaro
 Linda Dunstall

Witness

Cont'd on 10007-66

10007-54

Cont'd from 10007-50; started on 10007-28

Tech. Service 9606168; ^B VK Absorb Checks on Over & Shelf Samples for Yellow fumes stability (Zwickson 10007-46)

I.D.	wL	abs	wL	abs	I.D.	wL	abs	wL	abs	wL	abs
10007-35A, NI, shelf	446	1.1523			10007-35E, shelf	403	1.0670	492	0.3413		
10007-35A, NI, 38°C	446	1.1665			S.B./Uranine						
10007-35A, NI, 50°C	445	1.1875			10007-35E, 38°C	402	1.1017	491	0.3526		
10007-35B, Shelf	434	1.0337			S.B./Uranine						
Sunbeam/N.I.					10007-35E, 50°C	399	1.1144	491	0.3505		
10007-35B, 38°C	434	1.1425			S.B./Uranine						
S.B./N.I.					10007-35F, shelf	634	1.0139				
10007-35B, 50°C	435	1.1674			Patent Blue						
S.B./N.I.					10007-35F, 38°C	634	1.0066				
10007-35C, shelf	406	1.3622			P.Blue						
S.B./Pyranine					10007-35F, 50°C	634	0.9924				
10007-35C, 38°C	404	1.3813			P.Blue						
S.B./Pyranine					10007-35G, shelf	420	1.0518	442	1.0300	495	0.3107
10007-35C, 50°C	405	1.3917			Dawn Yellow						
S.B./Pyranine					10007-35G, 38°C	420	1.0740	442	1.0467	495	0.3196
10007-35D, shelf	421	1.307	442	1.3337	10007-35H, shelf	633	0.9108				
Quinoline/N.I.					Dawn Blue						
10007-35D, 38°C	421	1.3294	442	1.3590	10007-35H, 38°C	633	0.9084				
Quinoline/N.I.					Dawn Blue						
10007-35D, 50°C	421	1.3638	442	1.3725	10007-35H, 50°C	633	0.8938				
Quinoline/N.I.					Dawn Blue						
10007-35E, shelf											

Date June 10, 1996

Date June 14, 1996

Signature M.R. Pillai

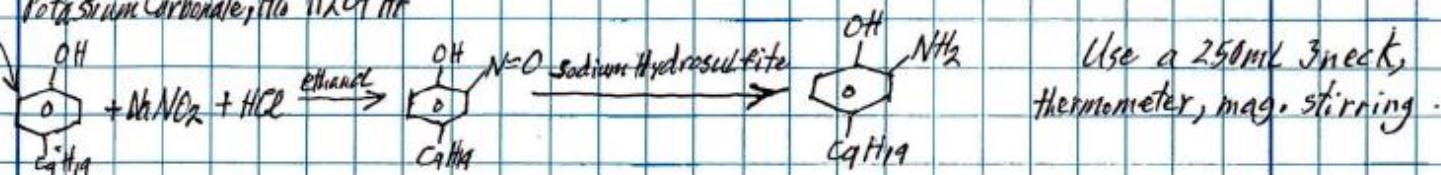
Witness Dr. J. Sunstall

cont'd on 10007-57

Rxn of Nonylphenol, Sodium Nitrite, & Sodium Hydro sulfite (ref. 10091-19, PLM)

Starting Materials:

	Lot #	M.W.	Moles	wt Needed	Actual wt.
Nonylphenol	JG13631EG	220	0.1041	22.91g	22.91g
Sodium Nitrite (NaNO ₂)	Y0320	69	0.1562	10.78g	
Ethanol, 200 proof	-	-	-	~ 52 mL	~ 52 mL
HCl (conc)	2612KHGV	36.96	-	~ 52 mL	~ 52 mL
Sodium Hydro sulfite	00711LT	174.11	0.1128	23.11g	23.11g
Potassium Carbonate, 99% 11204 HF					



10:20am The Phenol, Ethanol & HCl was combined & looked cloudy
 Cooled to < 10°C to 0°C (ice bath) Added Ethanol to clear soln.
 NaNO₂ added in portions

10:32am Clear soln.

10:35am Most of NaNO₂ added; pale yellow color.

10:39am All NaNO₂ has been added; slightly yellower color

10:46am Removed ice bath & Beginning to turn a brownish yellow

10:55am

Solu. brown with pale precipitate.

11:30 am Deep brown color

3:30pm Poured into DI H₂O; dark brown color; & extracted with dichloromethane (twice)
 Evaporated

Submitted for Mass Spec, I.R.

This 1st or intermediate product is "Nitroso"



Date: June 11, 1996

Date: June 14, 1996

Signature: M. R. Pillbury

Witness: Linda Sunstall

Cont'd on 10007-56

Cont'd from 10007-55

10007-56

9:00am The Nitroso 10007-55A was suspended in DI water (~175 ml) & 50% NaOH added to bring to pH 12. Solids present. Add Ethanol to increase solubility.

9:45am Added 23.11 g Sodiumhydrosulfite in portions. The dark brown color gave way to a deep brownish-orange. Stirred ~ 2 hrs.

11:45am Extracted in Dichloromethane; dried over Potassium Carbonate (use an ~~Erlenmeyer~~ erLinnmeyer flask, add product, add K_2CO_3 , swirl, let stand); ~~filter~~ filter; evaporate.

~~then~~ Submit for I.R. ; Greenish color

Submit for NMR

Date: June 12, 1996
Date: June 14, 1996

Signature: M.R. Pillai
Witness: Finch Junstall

10007-57

(see 10007-42)

Cont'd from 10007-54; started on 10007-28

Tech Service # 9606188; 2wk Absorb Checks on Open & Shelf Stabilities for Green's

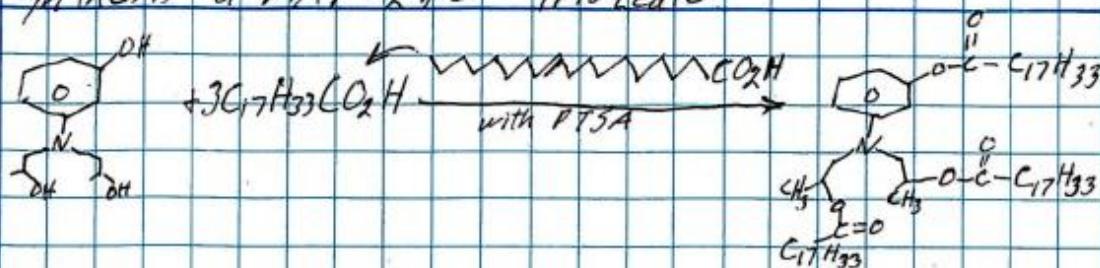
L.D.	wL	abs	wL	abs	wL	abs
10007-42A-Shelf	418	0.6021	423	0.6099	634	0.7632
NI/Patent Blue						
10007-42B-38°C	417	0.6239	423	0.6251	634	0.7593
SB/NI/P.Blue						
10007-42A-50°C	417	0.6567	423	0.6656	634	0.7847
10007-42B-Shelf	445	0.6310	634	0.7905		
NI/P.Blue						
10007-42B-38°C	445	0.6502	634	0.7548		
10007-42B-50°C	444	0.6370	634	0.7105		
10007-42C-Shelf	406	2.279	634	0.6909		
SD/Pyranine/P.Blue						
10007-42C-38°C	406	2.270	634	0.6845		
10007-42C-50°C	405	2.2845	634	0.6800		
10007-42D-Shelf	420	1.374	442	1.3008	634	0.774
Guanine/NI/P.Blue						
10007-42D-38°C	420	1.3364	442	1.3068	634	0.7113
10007-42D-50°C	420	1.3606	442	1.3252	634	0.7044
10007-42E-Shelf	410	0.5058	495	0.3334	634	0.7219
SD/Pyranine/P.Blue						
10007-42E-38°C	410	0.5064	495	0.3335	634	0.6920
10007-42E-50°C	407	0.5741	495	0.3384	634	0.7141
10007-42F-Shelf	418	0.5546	442	0.4893	499	0.1893
Dawn Green						
10007-42F-38°C	418	0.5792	442	0.5073	499	1.2016
						634
10007-42F-50°C	418	0.6217	442	0.5316	499	1.2152
						634
						0.6801

Date: June 12, 1996
 Date: June 14, 1996

Signature: M.R. Pillbury
 Witness: Lrich Junstall

Cont'd on 10007-62

Synthesis of MAP-2PO-Trioleate



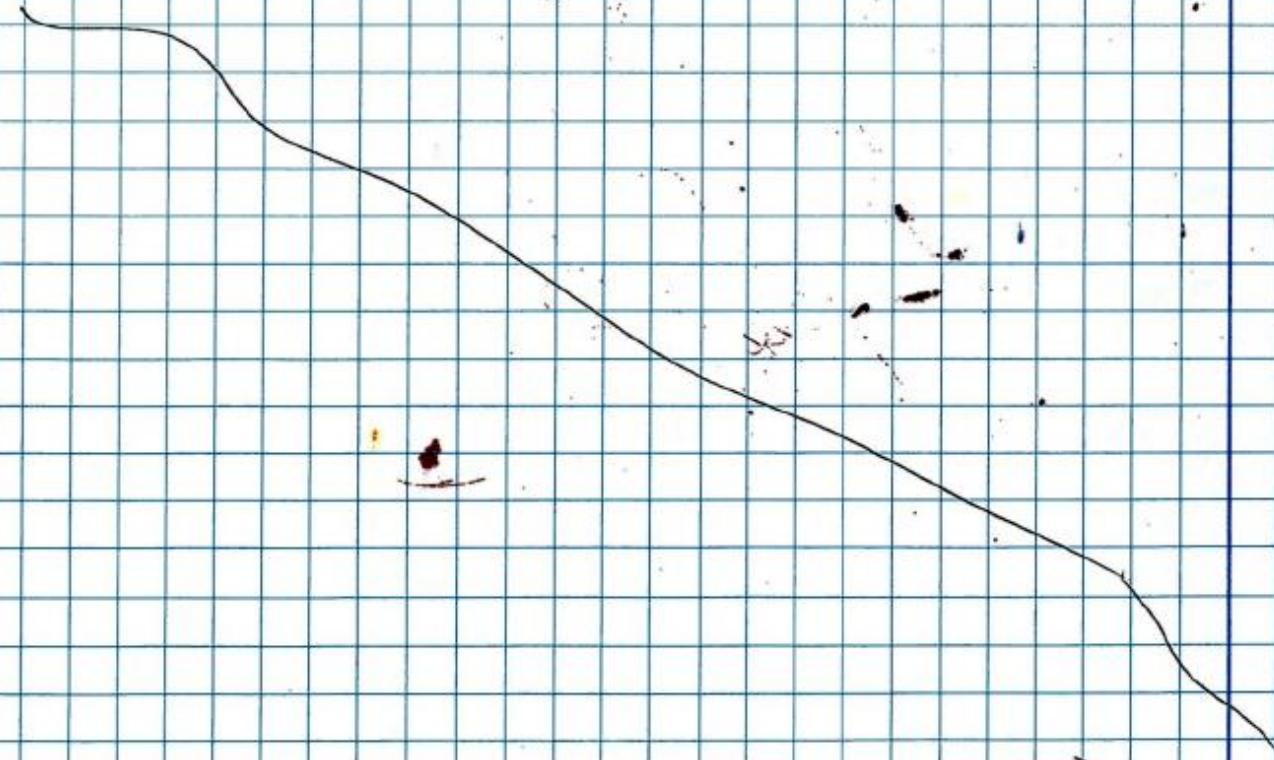
Starting Materials:

	<u>Lot #</u>	<u>M.W.</u>	<u>Moles</u>	<u>Wt. Needed</u>	<u>Actual Wt.</u>
Oleic Acid	G 9364	291	0.30	87.30g	87.30g
MAP-2PO (ink)	Batlaw	239	0.10	23.90g	23.90g
Toluene	-	-	-	~250mL	~250mL
PTSA (p-Toluenesulfonic Acid monohydrate)	02907HF	190.22	0.0105	2.0g	2.00g

Combine components in a 500mL 3-neck flask with boiling chips, heating mantle, condenser, & deanstark trap.

Heat to reflux & continue until ~0.30^{mol} H₂O comes off
(5.4g)

Let reflux overnight



Date: June 12, 1996
Date: June 14, 1996

Signature: M. R. Pihlay
Witness: Rich Windfall

cont'd on 10007-59

Can't'd from 10007-58

10007-59

Syn. of MAP-2PO - Trioleate

At 9:00am (after running overnight), only ~1mL of H₂O had come off

Changed set-up: Moved Dean-Stark trap/condenser to a side port;

Added thermometer & overhead stirrer. A temperature controller was connected to the heating mantle & set to maintain a constant 150°C.

The 1mL of H₂O was drained off.

Rxn restarted at ~10:30am.

Periodically drain toluene from trap 'til all is removed

Let rxn run.

At 11:45am; ^{most of the} Toluene is out

1:30pm; No additional water off yet. Add a Nitrogen line to sweep rxn.

1:45pm - A liquid coming off

2:05pm - The volume of liquid that has come off is ~7.5mL. This is more than the expected 5.4mL of H₂O; it's probably more toluene. Put some in beaker of H₂O-MsTol.

2:30pm - Raised temp. to 180°C; ~5mL more toluene came off

2:50pm - Raised temp. to 190°C; ~1.1mL more toluene came off

Stopped Rxn at ~4:30pm

Submitted for I.R. which indicated only a minor prospect of any ester product.

Date: June 13, 1996
Date: June 14, 1996

Signature: M. R. *M. R. McElroy*
Witness: *Rich Dunstall*

Purify RLM 10091-25

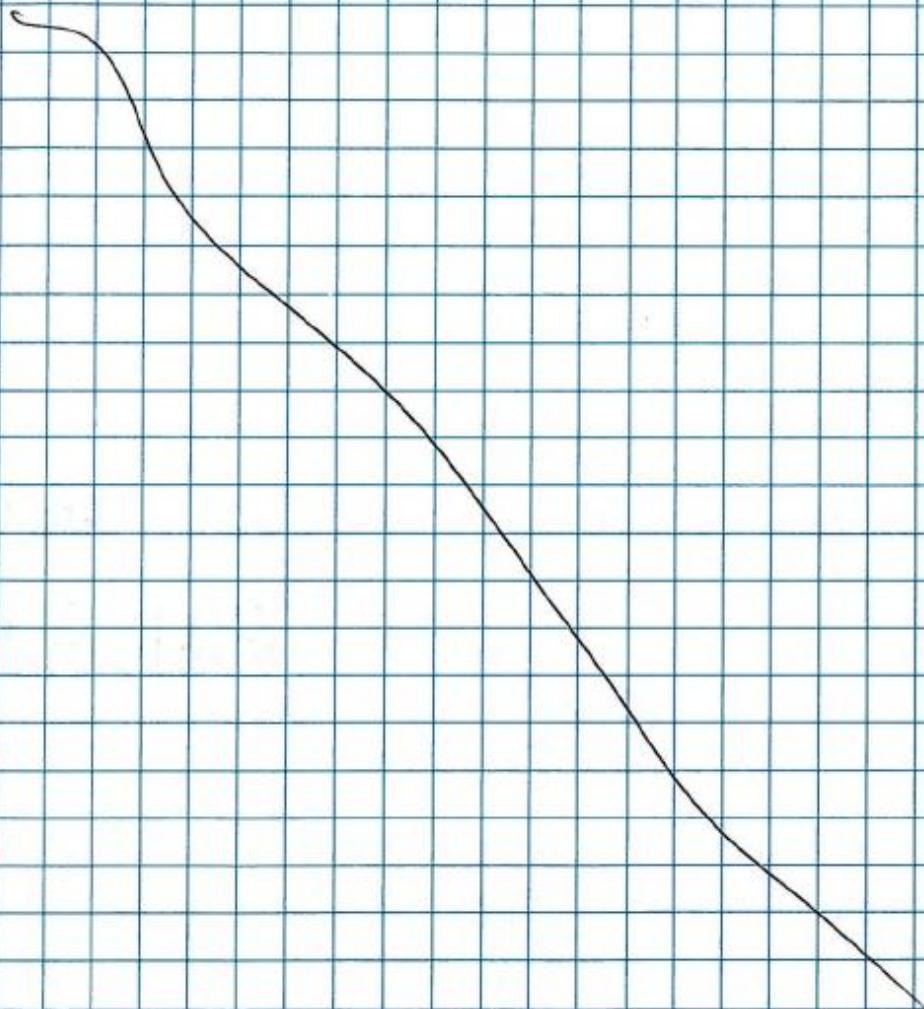
Dissolve product in Dichloromethane (CH_2Cl_2)

Wash 3 times with 50ml of 5% HCl in a sept. funnel

Evaporate the CH_2Cl_2

Run an I.R.

I.R. showed mostly starting material



Date June 13, 1996
Date June 14, 1996

Signature M.R. Palkovics
Witness Ruth Van Stall

Rxn of Dansyl Chloride, PA-12ETH, & Triethylamine

ref. 10070-33LT

Materials:

	Lot #	Moles:	M.W.	Wt. needed	Actual Wt.
Dansyl Chloride	05508KV	0.0185 0.0092	269.75	2.50g	2.51g
PA-12ETH (by Tomah)	50221B	0.0185 0.0092	200.00	1.85g	1.87g
Triethylamine, 99%	01828HN	0.0185	106.19	0.935	0.99g
THF	BJ022			35 mL to 70 mL	~ 50 mL

- 1) Charge Dansyl Chloride into a 1 neck 100mL flask with mag. stirrer.
 - 2) Add ~ 25mL ~~50mL~~ THF, stir & drop temp. to 0°C to 5°C in ice bath with IPA
 - 3) Pour in triethylamine
 - 4) Mix a soln of PA-12ETH & ~10 to 20mL THF & drip into flask
 - 5) Stir ~1 hr @ 0°-5°C
 - 6) Warm to room temp. & stir 1 hr
 - 7) Filter with aspirator
 - 8) Rot-a-vap.
- * with step "2" onward, use a thermometer adapter with side 24/40 outlet ~~outlet~~ into which a drying tube is placed.

Ratio is 1 to 1

In step "2", the Dansyl Chloride was not very soluble in THF;
 Temp. settled @ ~3°C (white looking)

In Step "3" temp. went to 4°C

In Step "4", began adding PA-12ETH & THF soln. at ~12:10pm

at 12:20pm, Dansyl Chloride appears to be starting to go into soln.
 (clearing up)

At 12:25pm, all PA-12ETH soln. is in; temp. 1°C

Remove from ice bath at ~1:25pm

At 2 pm, temp. was ~25°C; Step "6"; A clear, pale yellow soln.
 3pm; Filtered with aspirator; 3:15pm - Rota-vap till 3:45

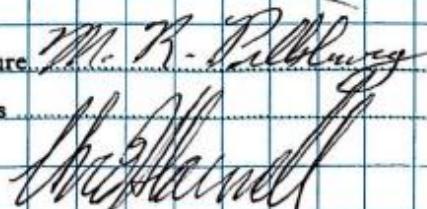
Product fluoresces brightly a blue shade.

Date: June 17, 1996

Date: June 28, 1996

Signature: M. K. Pilliar

Witness: J. P. McConnell



10007-62

(continued from 10007-57; started on 10007-28)

Tech Services 9606168; 4 WLR Absorb Checks on Oven & Shelf Stabilities for Yellow & Blue Samples in LDL

ID.	wl	abs	wl	abs	ID.	wl	abs	wl	abs	wl	abs
10007-35A, shelf	445	1.1441			10007-35E, Shelf	400	1.0075	491	0.3336		
NI					Sunbeam/Pyranine						
10007-35A, 38°C	445	1.1196			10007-35 E, 38°C	403	1.0517	491	0.3468		
NI					S.B./Pyranine						
10007-35A, 50°C	445	1.1435			10007-35 F, 50°C	397	1.0684	491	0.3457		
NI					S.B./Pyranine						
10007-35B, shelf	434	1.0676			10007-35 F, Shelf	634	0.9628				
Sunbeam/N.I.					P.Blue						
10007-35B, 38°C	433	1.0927			10007-35F, 38°C	634	0.9507				
S.B./NI					P.Blue						
10007-35B, 50°C	434	1.0941			10007-35F, 50°C	634	0.9496				
S.B./NI					P.Blue						
10007-35C, shelf	405	1.2867			10007-35G, Shelf	420	0.9976	492	0.9743	495	0.2988
S.B./Pyranine					Dawn Yellow						
10007-35C, 38°C	405	1.3114			10007-35G, 38°C	420	1.0189	492	0.9890	495	0.3109
S.B./Pyranine					10007-35G, 50°C	420	1.0651	492	1.0092	495	0.3084
10007-35C, 50°C	404	1.3433			Dawn Yellow						
S.B./Pyranine					10007-35H, Shelf	633	0.8651				
10007-35D, Shelf	422	1.2385	442	1.246	Dawn Blue						
Quinoline/NI					10007-35H, 38°C	633	0.8625				
10007-35D, 38°C	421	1.2776	442	1.2954	Dawn Blue						
Quinoline/NI					10007-35H, 50°C	633	0.8518				
10007-35D, 50°C	421	1.3064	442	1.3164	Quinoline/NI						
Quinoline/NI											

Date: June 17, 1996

Signature: M. R. Pillbury

Date: June 28, 1996

Witness: *[Signature]*Cont'd on 10007-64 *[Signature]*

Purification of 10091-29

Removed Rxn flask from heat & allowed to cool.

with temp. @ $\sim 32^\circ\text{C}$, poured into a 100ml beaker containing an approx. = volume of D.I H₂O at vol. of product ($\sim 250\text{mL}$)

Let stir $\sim 1\text{hr}$; Keep temp. $< 40^\circ\text{C}$; Temp. leveled out at $\sim 35^\circ\text{C}$

~~Temp.~~ Initial pH at 2pm ≈ 2 ; used 50% NaOH cat A 3932
2:30pm; pH ≈ 6 to 6.5 (CH₂Cl₂)

Extract in dimethyl dichloromethane twice; Remove bottom layer 1st, then extract. Remove bottom layer & extract top layer. - Use a 2L sept. funnel

Combine the bottom layers (product & CH₂Cl₂) in a 1L Sept. funnel.

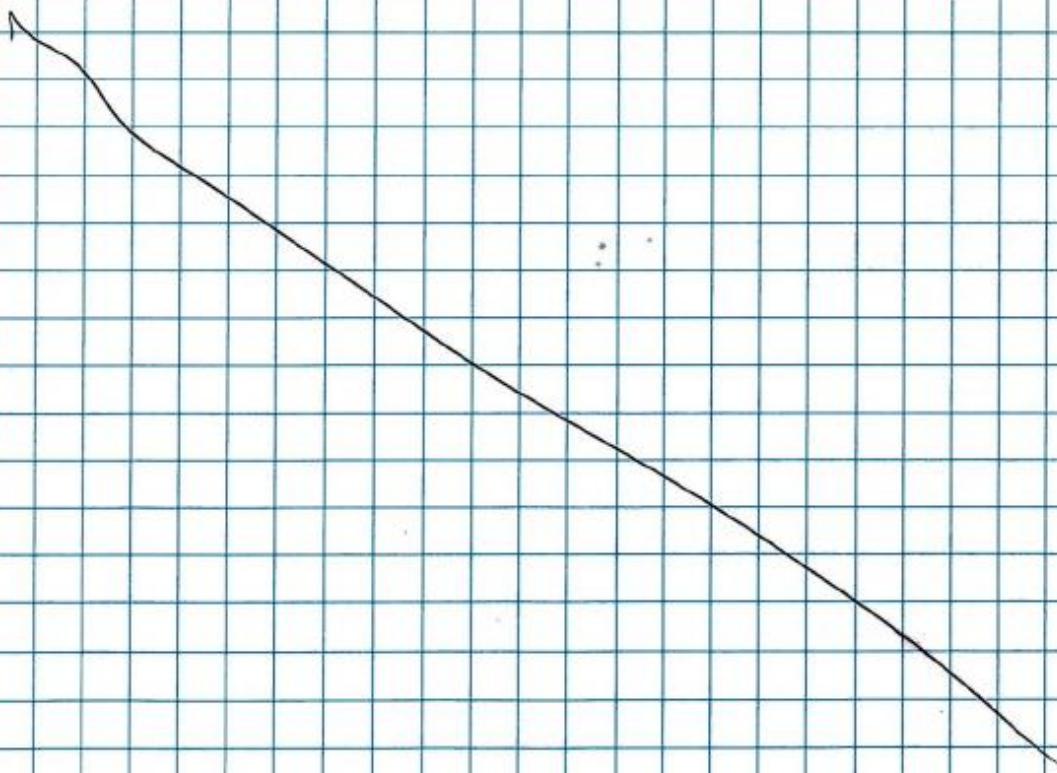
Total soln. vol. $\approx 450\text{mL}$. Add about 200mL of 5.75% Soln. of NaHCO₃ (bicarb.) & extract. * After shaking but before removing the bottom layer, check pH of top layer (NaHCO₃) to be sure it's basic. pH = 7

Repeat the NaHCO₃ extraction; pH of top layer \approx 8 to 9

Dry product layer in Potassium Carbonate.

Evaporate the CH₂Cl₂.

Submit for I.R.



Date: June 18, 1996

Date: July 2, 1996

Signature:

Witness:

M. R. P. Wilson
Dattle, Inc.

10007-64

Cont'd from 10007-62; Started on 10007-28

Tech. Service #9606168; Make larger batches of some colored LD Detergents to show Customer
 Make a fresh batch of 1% Quinoline Yellow, lot 088191JF: 0.50g Quinoline, 50.00g DT H₂O
 The customer-provided bottles will hold ~ 422.00g of LD, since density
 of Dawn® soap = 1.0661. Multiply all wts. previously used by 4.22
 Make samples as follows:

	Yellow (Colorant) 1% D.	Old wt. Wt. Needed	Actual wt.	Fl. Colorant 100% NI Lot 10007-286	Old wt. Wt. Needed	Actual wt.	Patent Blue, 1% 0.6475g 0.6475g 0.1646g	Old wt. Wt. Needed	Actual wt.	Base Wt Actual Wt. 422.00g	Actual Wt. 422.00g
A)	10007-35A Yellow	—	—	NI 100% 0.6475g 0.6475g 0.1646g	10007-286 0.889g 0.0350g	0.69g 0.15g	—	—	—	49.8319g 420.9516g	421.99g 421.05g
B)	10007-35D Quinoline Yellow	0.2074g	0.8752g	10% NI 0.0834g 0.0423g	0.86g 0.18g	0.15g 0.18g	—	—	—	49.7535g 420.2394g	421.05g 420.26g
C)	10007-35F Blue	—	—	—	—	—	0.2661g 0.1229g 0.12g	1.12g	1.12g	99.778g 420.85g	420.83g
D)	10007-42B Green	—	—	10% NI 0.0834g 0.0423g	0.35g 0.175g 0.185g	0.35g 0.83g 0.7963g	0.83g 0.83g 0.83g	99.5490g 420.0168g	420.10g	420.10g	
E)	10007-42D Quinoline Green	0.2047g	0.8638g	10% NI 0.0834g 0.0423g	0.86g 0.18g	0.18g 0.18g	—	—	—	99.5864g 420.2394g	420.26g

Used Top Loading Balance #310

New Dawn samples were purchased at the store for comparison.
 It seems the old yellow & blue Dawn stds have changed over time;
 the yellow looks redder & the blue has faded.

As compared to the ~~new~~ stds, both of our yellow samples
 seem paler in the customer bottles - "A", the NI sample is worst.
 The Blue & Green samples look close.

Will run absorbance tomorrow.

Date: June 19, 1996

Date: July 2, 1996

Signature: M. P. Pillbury
Witness: J. A. Johnson

Cont'd on 10007-65

10007-65

Cont'd from 10007-64 ; started on 10007-28

Tech. Service # 9606168 ; Check Absorbts of Samples from 10007-64

L.D.	wL	abs	wL	abs	wL	abs
10007-64A, Yellow 10% NI	446	1.0876				
10007-64B, Yellow 1% Quinoline/10% NI	445	1.5206				
Old Dawn Yellow	420	0.9866	442	0.9666	495	0.2991
New Dawn Yellow	420	1.8796	443	1.8717	498	0.6757
10007-64C, Blue 1% Patent Blue	634	0.9359				
Old Dawn Blue	633	0.8601				
New Dawn Blue	634	1.1176				
10007-64D, Green NI/P.Blue	445	0.5740	634	0.6991		
10007-64E, Green 1% Quinoline/NI/P.Blue	421	1.3013	442	1.2744	634	0.6774
Old Dawn Green	418	0.5321	442	0.4652	500	0.1929
	634	0.6363				
New Dawn Green	418	0.5991	442	0.5465	498	0.1965
	634	0.6063				

Date: June 20, 1996

Date: July 2, 1996

Signature: M.R. Pellew

Witness: Denise Foster

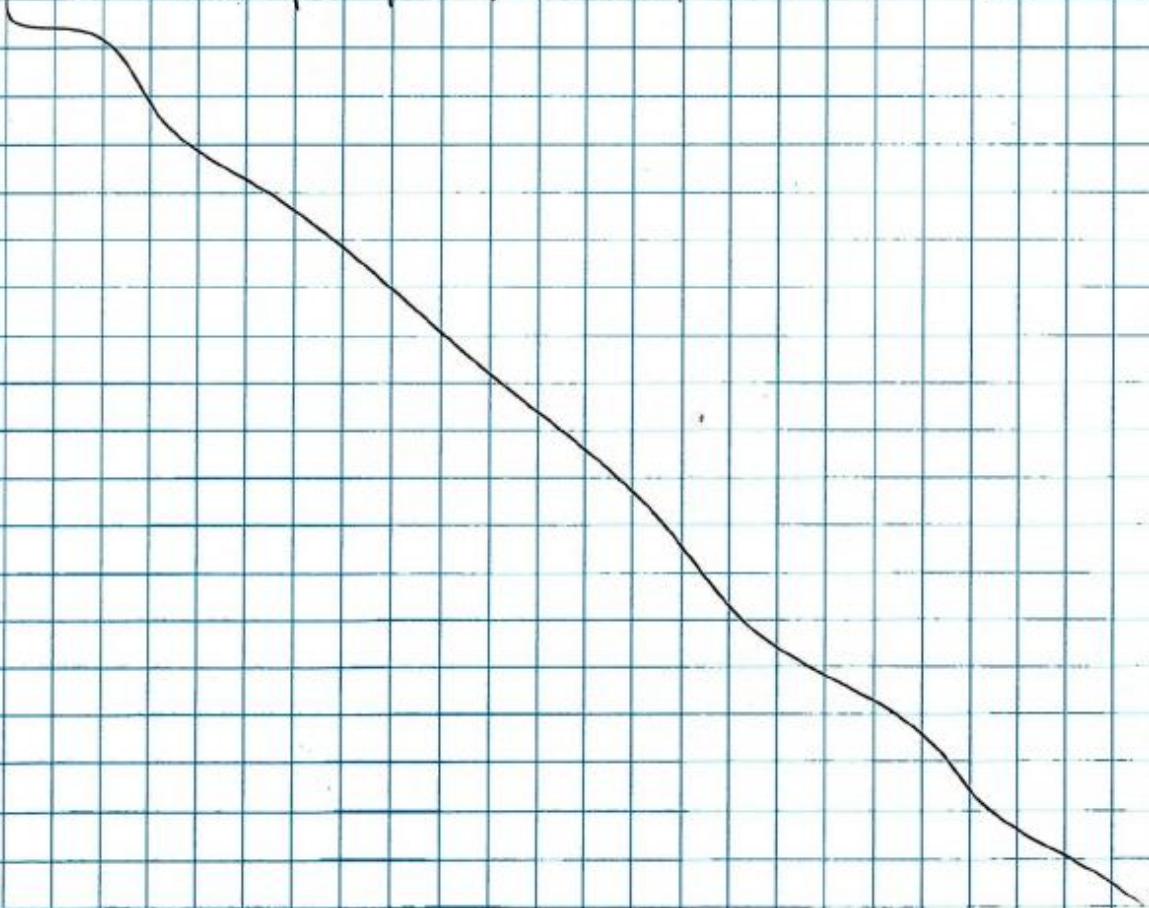
Cont'd on 10007-68

10007-66

Cont'd from 10007-53; started on 10007-40

4 Week L.A.B. Readings on Colored Dinh[®] Detergent in P.V.A.

I.D.	L	A	B	E	L	Deltas
10007-40A2, Red ST/H ₂ O/Dial	96.12	1.54	4.32	3.63	-1.46	
10007-40B2, Red ST/PVA/Dial	98.38	-1.90	5.63	2.18	-0.74	
10007-40C2, Blue HP/H ₂ O/Dial	94.46	-6.21	0.06	3.72	-2.04	
10007-40D2, Blue HP/PVA/Dial	98.53	-3.89	6.39	1.01	-0.93	
10007-40E2, Red ST/ PVA/H ₂ O/Dial	97.97	-1.35	5.44	2.61	-1.05	
10007-40F2, Blue HP/ PVA/H ₂ O/Dial	98.32	-4.13	4.82	1.53	-0.69	



Date: June 20, 1996

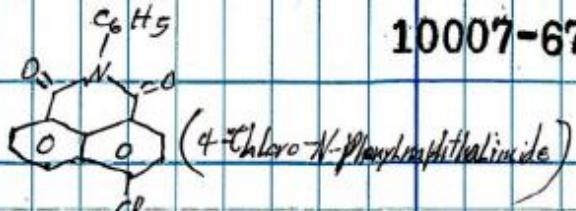
Date: July 7, 1996

Signature:

Witness:

M. R. Pillay
Dated 5/2001

Work-up RLM 10091-30 Product



Wash product in ethanol. Use a 2L filter flask & butner ~~filter~~
filter funnel, + #1 Filter paper

Some solids crystallized in flask.
Re-wash this, using #4 filter paper

Melting Point \approx 184°-187°C

Submit for I.R., Mass Spec, & NMR

I.R. indicated some anhydride may be present, but some product was there; peaks at 1736^{anhydride} & 1663^{product}

Added to ~200mL 5% NaOH & stirred ~40min. Filter & wash in DI H₂O;
pH of wash water (approx. 750mL) was ~13; need to be near 7 pH.
Will work on this more ~~Monday~~, let's do now.
Washed in ~300mL more H₂O; pH = ~7.

Date: June 21, 1996

Date: July 2, 1996

Signature: M. P. Polkberg
Witness: Dentise Foster

Cont'd from 10007-65; started on 10007-28

Tech. Service #9606168; Add RedST to Yellow LDL formulation

The customer "yellow" sample seems redder than ours. Salesman asked us to redden one of our samples.

Make a 3% soln. of RedST, lot # Rd 101: 1.50g RedST in 48.50g H₂O

Use the Quinoline, N.I., ^{mp} ~~Patent Blue~~ formulation, adding \approx amount of A 3% RedST as N.I. to start; decision will be based on visual appearance in customer oppaic bottles.

	Quinoline wt	10% NI wt	3% RedST wt	Base wt.	appearance
B 1	0.28g	0.05g	0.05g	134.63g	75 times too red

~~Double the H₂O in the RedST Soln & try again (added 48.6g)~~

	1.5% RedST				
B 2	0.28g	0.06g	0.01g	134.66g	not red enough
B 3	0.28g	0.07g	0.02g	134.66g	too red
B 4	0.28g	0.05g	0.02g	134.69g	too red
B 5	0.28g	0.05g	0.01g	134.74g	

These samples all seem to have a reduced brightness of yellow. Stop for now.

Date: June 24, 1996

Date: July 2, 1996

Signature:

M. R. Pillai

Witness:

Dentle S. Arora

Cont'd on 10007-69

10007-69

(Ref. 10007-57 & 42)

Cont'd from 10007-68; started on 10007-28

Tech. Service # 9606168; 4wk Absorb Checks on Open Stabilities for "Green" formulation

1.D.	wL	abs	wL	abs	wL	abs	wL	abs
10007-42A, Shelf, SB/NI/P.Blue	418	0.5751	634	0.5751				
	:			0.7191				
10007-42A, 38°C	418	0.5873	634	0.7094				
10007-42A, 50°C	416	0.6163	634	0.7093				
10007-42B, Shelf, NI/P.Blue	444	0.5893	634	0.7027				
10007-42B, 38°C	444	0.6083	634	0.7025				
10007-42B, 50°C	444	0.6262	634	0.6903				
10007-42C, shelf, SB/Ketamine/P.Blue	403	2.1091	634	0.6437				
10007-42C, 38°C	405	2.1231	634	0.5010				
10007-42C, 50°C	405	2.1418	634	0.5500				
10007-42D, Shelf, Quinoline/NI/P.Blue	419	1.2328	442	1.2070	634	0.6662		
10007-42D, 38°C	420	1.2445	442	1.2163	634	0.6587		
10007-42D, 50°C	420	1.2735	442	1.2339	634	0.6484		
10007-42E, shelf, SB/Ketamine/P.Blue	410	0.4829	495	0.3223	634	0.7060		
10007-42E, 38°C	410	0.5073	495	0.3216	634	0.7053		
10007-42E, 50°C	407	0.5334	495	0.3333	634	0.6951		
10007-42F, shelf, Draw Green	418	0.5301	442	0.4664	499	0.1678	634	0.6650
10007-42F, 38°C	418	0.5709	442	0.4667	499	0.1899	634	0.6693
10007-42F, 50°C	417	0.5897	442	0.5080	499	0.1993	634	0.6266

Date: June 25, 1996

Signature: M. R. Pillaireo

Date: July 2, 1996

Witness: Jennifer Foster

Make-up Various Fluorescent Oil Samples for Customer Meeting.

A Make a 15% soln. of Chromatech FL Yellow 1315C (yellow) in Hydrocal 45; 7.50g FL Yellow + 1.50g Hydrocal

Get density of motor oils to be used: density = $\frac{\text{mass}}{\text{volume}}$

$$\text{Haveline } \cancel{30HD} \rightarrow 30HD$$

$$\text{Density} = \frac{8.580}{10mL}$$

$$= 0.8580$$

$$\text{Mobil 10W-30}$$

$$\text{Density} = \frac{8.6379}{10 \text{ ml}} = 0.8633$$

$$\text{Density} = \frac{8,5889}{10 \text{ m}^3} = 0,8589$$

$$\text{density} = \frac{8.58009}{1000} = 0.8580$$

Get density of Colorant solns

"A," Chromatech Fl. Yellow in Hydrocal, density = $\frac{8.41889}{1000} = 0.8419$

$$RLM10046-46, D_i - PA-10-NT; \text{ density} = \frac{19.034 \text{ g}}{10 \text{ cm}^3} = 0.9034$$

To solve for Wt% of each component use the formula; $\text{Vol} \rightarrow \frac{(C_{100} + M_0)}{M_1} \frac{Wt}{d}$

Make samples as follows:

Havoline 30HD oil wt				Mobil 10W-30			
colorant	colorant	colorant	colorant	oil	WT	1000 FT. T.O.A.	oil
D-1	10007-70A WT	AKM 10045-46	ID	10045-46	ID	WT	WT
Weed	Actual	Need	Actual	need	Actual	Need	Actual
sec			sec		sec		sec
5% B 1	0.4520g	0.4523g	85.3710g	85.3740g	C-1	0.4710g	85.370g
25% B 2	0.5262g	0.5268g	85.2639g	85.2649g	C-2	0.564g	85.2652g
75% B 3	0.3153g	0.3150g	85.4783g	85.4740g	C-3	0.3394g	85.4779g
75% B 4	0.6314g	0.6324g	85.1565g	85.1520g	C-4	0.671g	85.1644g
2.5% B 5	0.2105g	0.2108g	85.5855g	85.5889g	C-5	0.2210g	85.5820g
					D1	0.4518g	85.981g
					D2	0.5253g	85.794g
					D3	0.3158g	86.005g
					D4	0.6323g	85.682g
					D5	0.2106g	86.1145g
					E1	0.4208g	85.898g
					E2	0.5890g	85.7925g
					E3	0.3383g	86.0076g
					E4	0.6787g	85.6858g
					E5	0.2255g	86.1163g

Mobil 1, 10W-30		Mobil 1, 15W-50	
(see D9)	0.2L WT. need Actual 1.0007-70A WT see "C"	(see F)	0.2L WT need Actual 1.0007-70A WT see "C"
5% F1	0.4528g	85.4711g	85.4700g G1
15% F2	0.5245g	85.3591g	85.3507g G2
75% F3	0.3152g	85.5671g	85.5688g G3
75% F4	0.6304g	85.2458g	85.2480g G4
25% F5	0.2110g	85.6753g	85.6760g G5
		0.4222g	0.4630g H1
		85.3544g	85.3714g H2
		0.5650g	0.5258g H3
		0.3386g	0.3159g H4
		0.6763g	0.6310g H5
		0.2267g	0.2102g H6
			85.4714g I1
			85.2678g I2
			85.4783g I3
			85.5659g I4
			85.5855g I5
			85.5854g I6
			0.4714g J1
			0.5648g J2
			0.3386g J3
			0.6777g J4
			0.2262g J5
			85.5867g J6

Stir each sample ~1 hr.

Date: June 27, 1996

Date: July 2, 1994

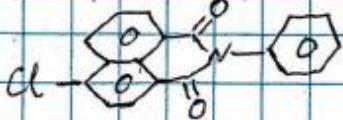
Sigantum

M.R. Poblocki

Witnessed

gentle sand

Prepare 4-Chloro-N-Phenylnaphthalimide (ref. KLM10091-30 & MP10007-87)



Starting Materials:

Lot. M.W. Moles. wt. needed. Actual wt.

4-Chloro-1,8-naphthalic anhydride 05130TY 232.63 0.2 46.53g 46.53g

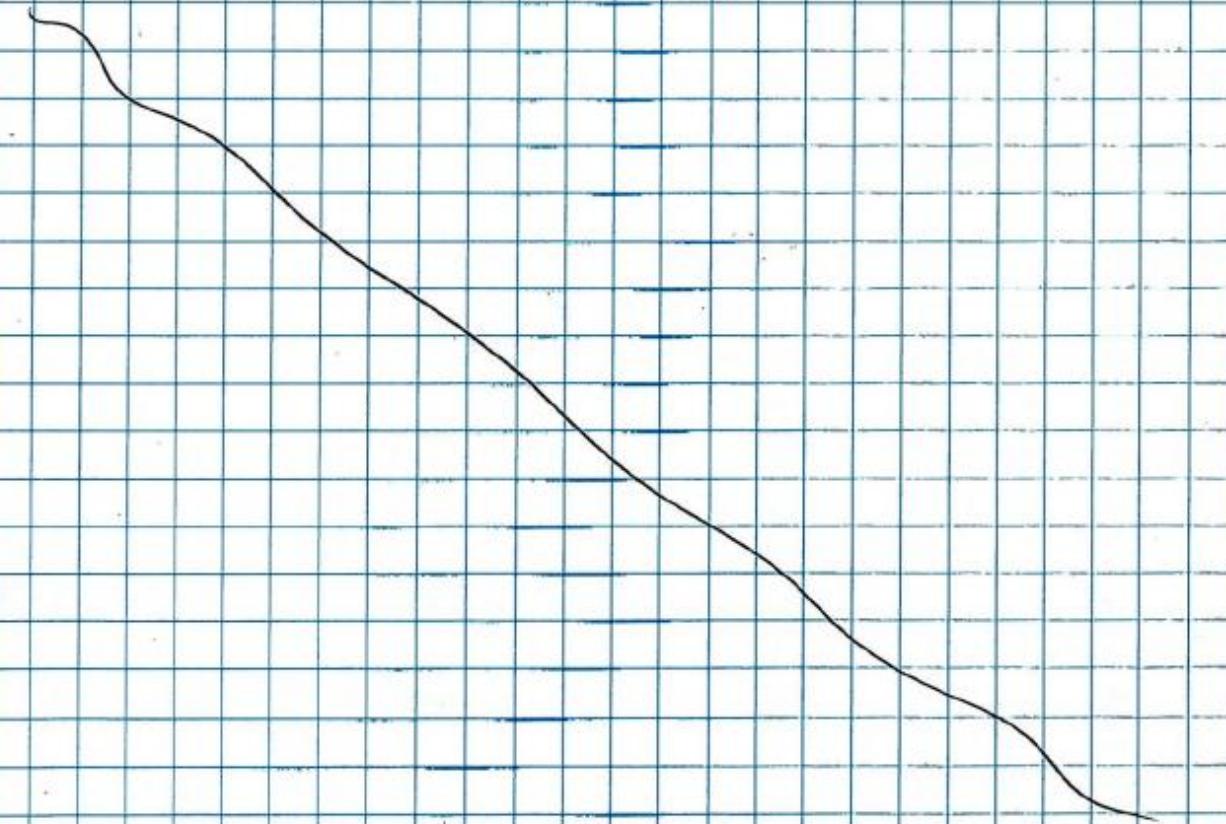
Aniline (Aldrich) 9720LG 97 0.3 29.0g 29.10g

Ethanol, 200 proof ~250mL

Heated to reflux. in a 500 mL 3 neck flask with condenser,
Refluxed ~ 1 hr, cut off for weekend.
↑

A solid ring of starting mat'l. was in bottom of flask after ~15 min.,
broke this up & added magnetic stirring.

t =



Date: June 28, 1996

Signature: *M. R. Pilliar*

Date: July 2, 1996

Witness: *Dentice Foster*

Cont'd on 10007-72

Cont'd from 10007-71

10007-72

7:40 am material is a solid; tan in appearance.
Restarted rxn.

8:15 am - Material has begun liquifying; stirred with spatula to break up, material is mostly a mushy substance & turned a more yellow shade with stirring. Restarted mag. stirrer. Total reflux time ~ 6^{3/4} hrs

1:30 pm - Cooled & filtered with aspirator. Washed in EtOH (~200ml). Solid is a tan color, filtrate a deep yellow. Rewash solid in EtOH, using #4 filter paper, but no aspirator as above.

Melting Point = 207°-210°C

Ran IR; The Anhydride peak at 1736 was gone & the product peak at 1663 was larger than on the IR of the earlier batch, 10071-30.

Added ^{product} V to ~600mL 5% NaOH & a beater & stirred ~40 min. Filtered & washed in DI H₂O & continued washing 'till pH wash ~7 (was 13 pH)

Place in drying dish over night
Total product wt \approx 62.95g

Date July 1, 1996

Date July 2, 1996

Signature

Witness

M. R. Pilkington
J. S. T. J.

Rxn. of 10007-71 (4-Chloro-N-Phenylnaphthalimide), AminePA-10, & Triethanolamine
r(CNPN)

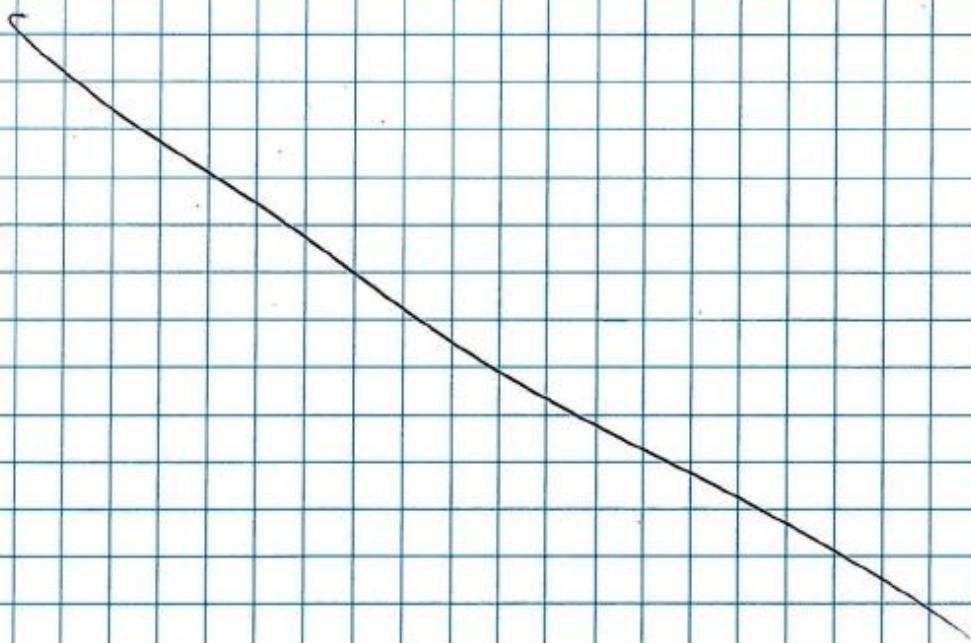
Materials:	Lot ⁴	M.W.	roles	wt.Needed	Actual wt.
CNPN	MP10007-71	309.5	0.1	30.95g ^{30.7} 31.00g	30.97g
AminePA-10 ^(comol)	S1020-205	165	0.11	18.15g ^{18.15g} 18.17g	18.17g
Triethanolamine ^{97%}	109197B (12)	149	0.11	16.90g	16.42g

Combine into a 500 ml 4-neck flask with overhead stirrer, thermometer with controller, & static nitrogen. Heat to 163°C for 10 hours

Began at ~10:30 am -

11:30 am - temp. range from 160° to 167°

Stop rxn at ~5 pm



Date July 2, 1996

Date July 2, 1996

Signature

Witness

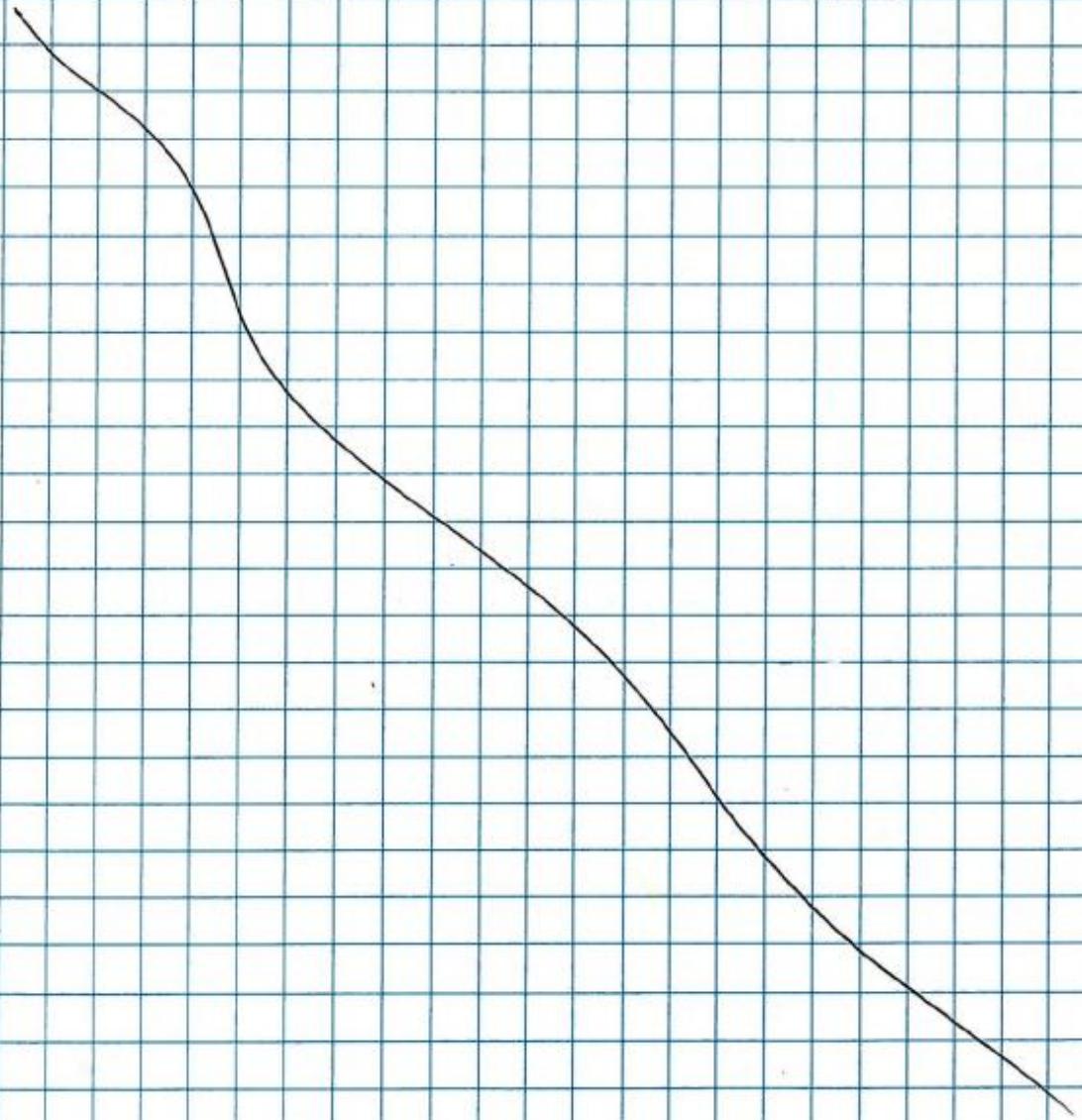
J. R. Toliver

Detise 705

Cont'd on 10007-75

Making Uranine Oil ~~saturated~~^{soluble} with Syn Fac DG

- A) Make a 1% Soln. of Uranine, Lot #47201-P, in Syn Fac DG (ultra lab); 0.3g Uranine in 29.7mg Syn Fac DG
Stir.
- B) Add 0.50g of the 1% Soln. to ~~85.56g~~^{85.56g} Mobil 1, 10w-30 oil (allow for oil density of 0.8589).
Uranine may be soluble ; Not - fluorescent.
- C) Add 2.67g Uranine to Soln. "A" to make a 10% ~~soln~~ soln.
- D) Add 0.50g of 10% Soln. "C" to 85.57g Mobil 1 oil; Looks cloudy ; still not very fluorescent



Date July 2, 1996

Date July 2, 1996

Signature

Witness

M. R. Ballou
Dentise Fox

Cont'd from 10007-73

Rxn of 10007-71, Amine PA-10, & Triethanolamine

Re-started rxn. at 8am. material was solid, deep yellow color.
Began melting @ in 100° to 110°C

9:15 am j nitrogen tank empty. Hooked up a portable tank & flushed out flask, then let run to completion.

12:30pm - Reduce heat ~~til~~ product is flowable (~120°C)
check absorb. Run in Toluene (not soluble in methanol)

100mL flask #3817608300, sample wt = 0.105g, 2mL pipett #7569, WL = 423, abs = 0.6009, Absorb = 25.35

Turn off heat & cut with Hydrocal 45 'til absorb ~5

amount of Hydrocal	sample wt	100mL flasks*	Pipet	Conc	WL	abs	Absorb	
200.00g	0.4220g	8176, 8942	10mL #5	0.427	421	1.007	2.37	sonicated

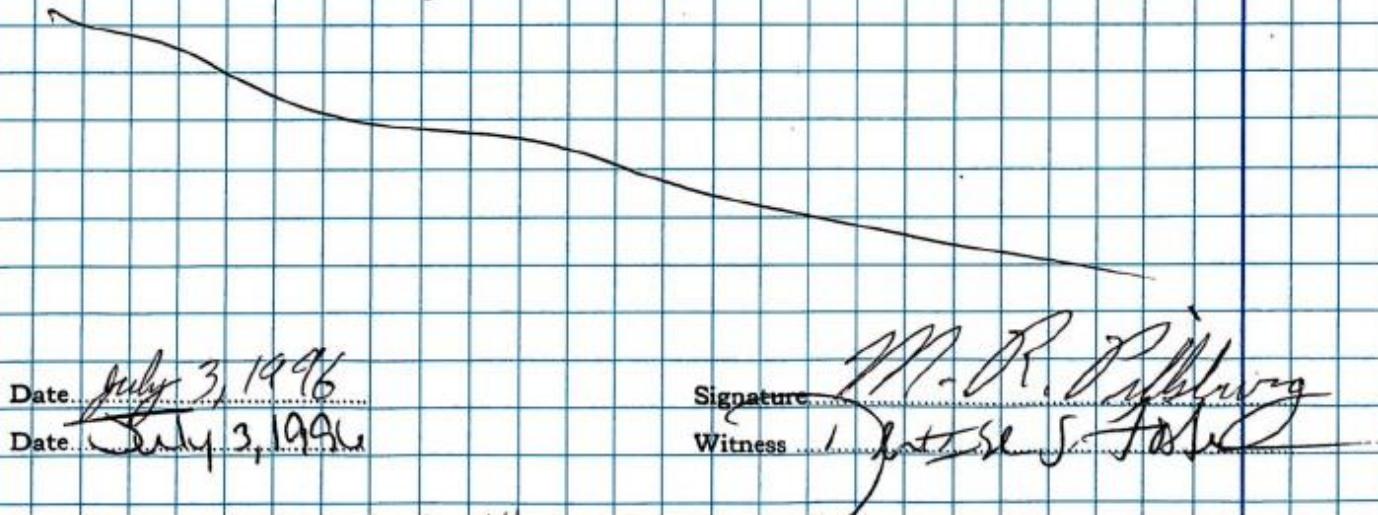
Filter product with aspirator, bottom funnel with #1 paper & just enough Hydrocal to wet paper.

Solids in filtrate; will hold for Dr Makoffey to determine what to do with it. It's a filmy substance; yellow with some brown material.

Run absorb on solids in funnel: (deep yellow; ^{looks} brown looks brown)

wt = 0.1004g, 100mL flasks - 8460+7513, 10mL pipett #938) conc = 0.1004, WL = 423, abs = 1.115, Absorb = 11.07

sample had ~~a~~ slight amount of salts? in initial flask.



Cont'd on 10143-1