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Spartanburg, South Carolina

LABORATORY NOTEBOOK

Name..... *Bruce Suddeth* Book No. 10038

Used from..... *January 26, 1996* To.....

Laboratory Book **Nº 10038**
Date Received: *January 26, 1996*
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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 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 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 can be charged out.

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Instrument ID Reference

UV/VIS	00292
Analytical Balance	00293, 00294
Top Load Balance	00208
pH Meter	00207, 00209
Microwave	00222
Ovens	00295, 00296, 00297, 00298

These pieces of equipment will be used unless otherwise specified.

11.21 11.2

, 051

14v

Lt. Violet w/ Sulfonic Acid

Purpose: React the Aniline 10 with Sulfonic Acid and
Continue w/ H₂SO₄ and Hydrogen Peroxide.

R/M	Lot#	mw	wt	Actual	Moles
Aniline 10	A1020	531	156.65	156.65	0.2950
Sulfonic Acid	Dewey	97.09	57.28	57.28	0.5900 2x

8:30 - Charge Aniline 10 to a 1L 3-neck. Charge Sulfonic Acid. Agitate. Add condenser.

8:35 - 28°C Begin heating Rx to 140°C.

9:25 - 80°C Aniline 10 is getting darker as it reacts.

9:40 - 125°C

9:45 - 147°C Cut temp back.

10:25 - 130°C

1:30 - 140°C Cut heat back.

Burn

R/M	Lot#	mw
-----	------	----

Aniline 10 + Sulfonic Acid 631 213.93 0.3096

Urea W2816 60 4.65 0.0775

PDMAIB Y3374 149.2 23.10 0.1548

H₂SO₄ (38%) 3553K3JG 98 15.17 0.1548

H₂O#1 DI 18 15.0

H₂O#2 DI 18 28.0

AMV Bahn 116.98 0.0017

H₂O₂ (35%) Y3295 34 10.52 30.08 0.3096

1:45 - Cut heat and allow to cool to <60°C before adding R/M.

H₂O (Peroxide) DI 18 30.08

2:10 - 60°C Charge H₂O#1 + H₂O#2, charge Urea, PDMAIB, AMV, and H₂SO₄.

2:15 - 65°C Begin reheating Rx to 95°C.

2:30 - 85°C

3:00 - 96°C Hold for 3 hours at 95-100°C.

3:45 - 100°C

4:30 - 98°C Cut heat & allow to cool.

Continue on page 3.

Date: January 29, 1996

Signature: Bruce Muddith

Date: January 31, 1996

Witness: Linda Marshall

Bermuda Grass Seed Patent w/ Colorants Only.

Continued from 9961-71.

10:30 - Using 1 gram of colored grass seed in a 100 ml beaker wash ~~sets~~ with DI water (90 grams). Agitate w/ dropper and let sit ~15 minutes.

Run an absorb curve of the washed seed.

Bermuda Green Wash

Peaked at 628 nm Abs of 1.1986
 " " 412 nm Abs of 0.3392

Polychem Wash

Peaked at 631 nm Abs of 2.8597 (2.39 x as much Bermuda)
 " " 408 nm Abs of 0.5384 Cut wash in half and run again. = 629 nm Abs of 1.7771

To look at the seeds you wouldn't think that the Polychem was darker in shade.

The Polychem seeds look lighter than the Bermuda Green.

Calculate Seed Coverage.

Absorb of Bermuda Green is 15

Absorb of Polychem Green is 15.48

Calculate Bermuda Green:

$$(0.108) \times (15) = 1.50 \\ (\text{on seed}) \quad (\text{Absorb})$$

Calculate Polychem Green:

$$(0.210) \times (15.48) = 3.10 \quad \text{or} \quad (2.07 \times \text{as much Bermuda}) \\ (\text{on seed}) \quad (\text{Absorb})$$

Continued on page 11.

Date: January 29, 1996

Date: January 29, 1996

Signature: Bruce A. Shabot

Witness: Linda S. McCall

bt. Violet w/ Sulfamic Acid.
Continued from page 1.

1/30/96

Take a small sample of condensate and dissolve in water. Check pH = 2.46. Made ~ a 10% solution. Heat Rx up to 95°C and hold.

10:25 - 97°C Hold for 1 hour.

11:30 - 97°C Prepare H₂O/H₂O₂ solution in add funnel.

11:35 - 97°C Start addition dropwise.

11:36 - 104°C Cut off heat. Add H₂O/H₂O₂ at a rate that holds temp at 105-108°C.

11:40 - 107°C

11:46 - 110°C Cut back add rate.

11:58 - 108°C Complete add. Turn on rheostat and hold at 95°C for 30 minutes.

12:10 - 100°C

12:30 - 100°C Cut heat and allow to cool.

2:00 - 40°C Measure DV-383 + DV-300

$$DV-300 = 55.67 \quad DV-383 \text{ in meth} = \frac{0.1779}{0.0726} \times 2 = 4.90 @ 591\lambda$$

$$CV = 8.80$$

Date: January 30, 1996

Date: January 31, 1996

Signature:

Bruce Andelth

Witness:

Ruth Dunstall

Bermuda Grass Seed Patent w/ Colorants Only
Continued from page 2.

Using the colored seed prepared on 8961-71 observe
the seeds and takes pictures with the micrograph.

The seeds with Polyphen Green do not look like they
are coated very evenly. They looked like the
dye was blotchy in color. They had dark spots of blue
dye on the seed.

The seeds with Bermuda Green appear to be
more evenly coated in color. The seeds appear
to have swollen more than the polyphen seeds.

Tim Monahan needs to look at these photos and
see if he can use them in the Patent as
evidence.

Date: January 30, 1996
Date: January 31, 1996

Signature: Bruce Addith
Witness: Linda Dunstall

Lt. Violet w/ Sulfamic Acid and ρ -Toluenesulfonic Acid.

Purpose: React the Amiline 10 w/ Sulfamic acid and continue w/ H_2SO_4 and H_2O_2 .
use ρ -Toluenesulfonic acid as catalyst.

R/M	Lot #	mn	wt	Act wt	Moles
Amiline 10	A1020	531	156.65	156.67	0.2950
Sulfamic Acid	Dewey	37.09	57.28	57.31	0.5300 2x
ρ -Toluenesulfonic acid	02807HF	190.2	11.21	11.3	0.05900

10:45 - Charge A10 + Sulfamic to a 1L 4 neck flask. Agitate.
 10:50 - Charge ρ -Toluenesulfonic Acid and begin heating to $\sim 140^\circ C$. Add condenser w/ stopcock and bubbler.
 11:15 - $80^\circ C$ Heat up to $\sim 100^\circ C$ and hold.
 11:35 - $110^\circ C$ Cut heat back.
 12:00 - $102^\circ C$ Cut heat. The Rx looks olive green in shade. Something is different than 10038-1.
 Possibly the PTSA made the difference. Allow to cool and run the Violets LS.

Calculate R/M Charges.

			23.95	Based on 691 mn of A10+Sulfamic
PDMA B	Y3344	149.2	32.07	0.577 + 0.1605
Urea	W2816	60	3.61	0.0602
Muriatic (31%)	X3201	36.46	7.02	0.1925
AMV	Bahr	116.98	0.13	0.13
$H_2O^{\#1}$	DI	18		11.53
$H_2O^{\#2}$ (Peroxide)	DI	18		34.45
H_2O_2 (35%)	Y3295	34	12.06	34.45
				0.3547

1:20 - $60^\circ C$ Charge PDMA B, Urea, AMV, and $H_2O^{\#1}$. Agitate and begin heating to $95^\circ C$.

1:25 - $65^\circ C$ Charge Muriatic & continue heating.

1:40 - $75^\circ C$

1:45 - $83^\circ C$

Continue on page 6.

Date: January 31, 1996

Signature:

Bruce Heldeth

Date: Jan 31, 1996

Witness:

Rich. Durrall

Lt. Violet w/ Sulfamic Acid and p-Toluenesulfonic Acid.
Continued from page 5.

1:55 - 90°C

2:05 - 95°C Hold at 95-100°C for 3 hours.

2:10 - 98°C

2:15 - 102°C Cut heat back to hold at 95-100°C.

2:30 - 100°C

2:50 - 97°C

4:00 - 96°C

4:30 - 97°C Cut heat and allow to cool.

2/1/96 8:00 - Begin to reheat Rx to 95°C. Very thick, will not stir.

8:30 - 80°C Agitating

8:55 - 91°C Condensate is a violet color.

9:30 - 102°C Cut heat back.

10:00 - 100°C Prepare H₂O/H₂O₂ solution in add funnel.

10:05 - 98°C Begin oxidation. Had a nice exotherm.

10:06 - 102°C Cut off heat and add at a rate that
hold temp at 105-108°C.

10:08 - 109°C

10:28 - 108°C Complete oxidation. Turn on rheostat
and hold at >95°C for 30 minutes.

10:40 - 99°C

11:00 - 98°C Cut heat and allow to cool.

3:00 - 40°C Run absorb + solids.

DV-300 = 58.982. Solid. DV-383 in MeOH

$\frac{0.9490}{.1000} \times 2 = 18.98$ CV = 32.18

Date: January 31, 1996

Date: January 31, 1996

Signature: Bruce Suddeth

Witness: Linda Dunstall

TS # 9602045

Continued from 9961-68

Check the stabilities by turning the vials upside down.

Allow to sit several hours and observe.

2/1/96 Blends #1 + #2 at RT look the same. Do not see any sedimentation in the bottom of vials.

Blends #1 and #2 at 140°F appear to have some differences. Blend #1 w Acid Blue 9 has sediment and a hazy film on the bottom.

Blends #3 and #4 w/o water, both have colored sediment on the bottom of the vial.

2/15/96 Check stabilities by turning vials upside down. Allow to sit several hours and observe.

Blends #1 and #2 at RT do not have sediment. There is a slight yellow cast on the bottom of vial containing Acid Blue 9.

Blends #1 and #2 at 140°F have the same differences as vials at RT.

Blends #3 and #4 w/o water, both have colored sediment on the bottom of the vial.

Date February 1, 1996

Date February 6, 1996

Signature Bruce Suddith

Witness Linda Juristall

Grass Seed Bulk Density.

Purpose: Needed to measure the density of the seeds for patent application purposes.

Use "Raw" bermuda seed from Arizona as the uncoated control. These seeds being tested are white color only.

Measure weight of seeds in a 10ml graduate.

$$\text{Raw Seed} = \frac{4.71}{10} = 47.1\%$$

$$\text{Polyphen Green} = \frac{4.80}{10} = 48.0\%$$

$$\text{Bermuda Green} = \frac{4.30}{10} = 43.0\%$$

The Bermuda Green sample is more expanded than the others, so therefore it has a lower bulk density.

Date February 2, 1996
Date February 6, 1996

Signature Bruce Suddeth
Witness Rich Sunstall

Highlight Colorants Match for Stewart Hall.

Purpose: Make an ink formulation with highlighters for Stewart-Hall.

Blnd #1 - Lt. Blue HP Lot # R1126	60%
Blazon Red Lot # Q1025	30%
Syntint Yellow Lot # B1097	10%

Weigh 3.0g BHP, 1.5g Red BL, and 0.50g Syntint Yellow in a vial. Shake well.

Add 1g of blend to 19g ink formula. Shake well.

Blend #2 - Lt. Blue HP (Above)	55%
Blazon Red (Above)	30 40 %
Syntint Yellow (Above)	15 5 %

Weigh 2.75g BHP, 1.5g Red BL, and 0.75g Syntint Yellow in a vial. Shake well.

Add 1g of blend to 19g ink formula. Shake well.

Add 4g of blend to 16g ink formula. Shake well.

Color appears to be too light. Color needs to be more yellow. Talked w/ Ken Starks about the color load. Ask if color load of 20% can be cost effective. He said he would talk to Paul Halphen and get more cost info.

Date: February 5, 1996

Date: February 6, 1996

Signature: Bruce Addeth

Witness: Rich Durstall

Sample of lignin-like Nature from Colgate Europe

Purpose: Lee Closs and Jim Spy visited Colgate in Europe. They gave them a sample of Nature. Lot # is not legible. They say it has sediment or sludge in the sample.

Received a lot # A1145 of Sunbeam Yellow with sediment in the bottom.

Nature uses Sunbeam Yellow so that might be where the sediment is coming from.

Drain off the good color Sunbeam Yellow and drain sediment into a beaker.

Lignin-like Nature sample - The 4oz bottle of Nature had a tremendous amount of salt in the bottom. Estimate about 20 grams of salt. This salt is a crystalline salt. The salt in Sunbeam Yellow is fluid not crystalline.

Spoon out ~5 grams of salt from Nature and wash with Methylene Chloride. Washed with about 100 ml of solvent. Crystals became mostly clear. Submit to analytical to determine what kind of salts they are.

See if lignin Teal has salts in it.
 Retained sample from T5 lab lot # V1012. Sample was grinded in 11/95. Poured out top product. Saw salt on bottom of quartz jar. Scrapped out ~2 grams w/ spatula. Will wash w/ Methylene chloride on 2/5/96.

9:30 - washed the salts with ~100 ml of Methylene Chloride.

Continued on page 11.

Date: February 5, 1996

Signature: Bruce Juddith

Date: February 5, 1996

Witness: Ruth Dunstall

Sample of Lignite Nature from Edgell Europe.

Continue from page 10.

Use a pipet and squirt ~ 2ml into funnel with Whatman #4 filter paper. Let Methane chloride rinse salts w/ each squirt.

10:00 - Have rinsed most of color from the salts. Allow to dry.

The salts do not look like salts in Nature. They are smaller and finer.

The salts in Nature are larger and more crystalline.

Submit to analytical for analysis to determine what type.

2/16/96 Received results from Bill Sims.

Micro-infrared analysis of the two salt samples showed them to be identical. The salt is totally inorganic. IR analysis showed the salt to be either a sulfate or phosphate type. XRF found the salt contains a large amount of phosphorous, confirming it to be a phosphate type. The cation associated with the salt is most likely sodium since the salt is water soluble (and XRF did not detect potassium).

Date February 6, 1996

Date February 6, 1996

Signature Bruce Anddeth

Witness Linda Sunstall

Technical Service #9603066

Originator: Spn

Customer: Colgate Palmolive
Belgium

Date In: 2/6/96

Objective: Fabuloso Fabric Softener.

Service: Apply Crimson at 0.025%. Try Red ST, Red HP, Red BL, and 70/30 Red ST/Sunbeam Yellow blend at appropriate levels. Try to get a more pink shade. Conduct thermal stability and staining. Adjust the pH to a 2 and a 3.5 to check for a shade shift. (down w/ HCl and up w/ NaOH).

Test: Using Palmer Scarlet lot # K1119 (Crimson), add 0.025g. to 39.975g. of Fabuloso base sent by Steven on 2/6/96. Added 0.025% in 39.97g. base. Agitate and pour into 4oz jar. Appears to be too orange.

Add Red ST lot # RD101 at 0.01%. Weight of 0.0100g., and 99.99g. of base. Stir on stir plate. Color appears to be too Red. Needs to be more pink. The amount of color is too much also.

Add Red HP lot # S1004 at 0.01%. Weight of 0.0113g. and 99.99g. of Fabuloso base. Stir on stir plate. Color appears to be a lavender shade. The red looks to have dried out making the Red HP dark. This color will not work by itself.

Add Red BL lot # G1025 at 0.017%. Weight of 0.0171g. and 99.98g. Fabuloso base. Stir on stir plate. The color is a purple/pink shade. Needs to be more peach. Needs some yellow.

Continued on page 13.

Date February 6 1996

Date February 6 1996

Signatures

Bruce Anddett

Witness

Sandy Quinball

Technical Service #9603066
Continued from page 12.

Prepare a blend of 70% Red ST Lot# RD101 and 30% Sunbeam Yellow Lot# V1129. 7.0g Red ST / 3.04g SBY.

Add 0.0075g of 70/30 RedST/SBY to 99.99g Fabuloso base.
Actual = 0.0087g of 70/30 RedST/SBY to 99.98g Fabuloso base.
Allow to stir into solution. Color appeared to not go into solution readily. Went in after ~30 minutes of stir time.

Color was too pinkish/lavender. Need to back off on the Red ST some.

Talked w/ Jim Spay and he looked at all the previous samples. He agreed that Lt. Crimson by itself is the best so far. He says the other reds are so dull they will never match the blends made of Acid Red 52. He suggested trying Lt. Pink by itself and see the shade.

Use Palmer Magenta Lot# H1105 at 0.011% in Fabuloso base. Use 99.99g of base. Weight = 0.0099g. Use 99.99g Fabuloso base. Agitate on stir plate. Color is real pink.

Blend Magenta Lot# H1105 (85%) with Sunbeam Yellow (15%).
Use 0.017g in Fabuloso. Weight = 0.0108g.
Use 99.99g base. Agitate on stir plate.
Color is too pink still. Try another blend w/ more Sunbeam Yellow.

Blend Magenta Lot# H1105 (50%) with Sunbeam Yellow Lot# V1129 (50%). Mix well. Use 0.01% in Fabuloso. Use 99.99g base.
Agitate on stir plate. Color looks really dull.
Bad shade match.

Continue on page 14.

Date February 7, 1996

Date February 6, 1996

Signature Bruce Adderly
Witness Finch Smith

Technical Service #9603066
Continued from page 13.

Jim looked at samples and wanted to try a blend
of 60% Crimson and 40% Pink.

Blend	Scarlett	Lot # K1119	60%	6 ⁰³
	Magenta	Lot # H1105	40%	4.02 grams

Agitate well.

Add 0.02% of blend to Fabrlos base. !

Weigh 0.01g of blend and 99.98g Fabrlos base. Agitate
well. Color is too pink.

Prepare another blend of 80% Scarlett and
20% Magenta.

8g Scarlett and 2g Magenta. Agitate well.

Weigh 0.02g of blend. Add 99.98g Fabrlos base.
Agitate well. Color is still too pink.

Prepare another blend of 90% Scarlett and 10% Magenta.

Add 0.02g blend to 99.98g base. Stir well.

Color is close. Needs to be a little darker.

Talked of Jim Song, he said to do stability & staining
on Fabrlos Reference, Crimson @ 0.025%, and
95/5 Scarlett/Magenta @ 0.03%.

Blend 0.0515g of Scarlett lot #K1119. Add 99.95g of
Fabrlos base. Agitate well. Allow to stir.

Blend 0.0617g of 95/5 Scarlett/Magenta. Add 99.94g of
Fabrlos base. Agitate well. Allow to stir on stir plate.

Use the Fabrlos Pink Reference and the 2 blends
above. Place nonfibrous test strips and terry
cotton swatches in each sample. Place in
respective solutions at 9:15. Allow to soak 15 minutes.
Remove from solutions and squeeze excess off.
Allow to dry over weekend.

Continue on page 15.

Date: February 8, 1996

Signature: Bruce Siddle

Date: February 13, 1996

Witness: Vicki Gundal

Technical Service #96D3066
continued from page 14.

Adjusts pH of blends to a pH of 2 w/HCl and 3.5 w/NaOH.

Fabuloso Pink Reference: Initial pH=3.3 Peach shade.

Prepare 50% solution of Caustic lot# V2568.

1/2g Caustic / 10g H₂O. Stir. + cool.

Prepare 50% solution of Muriatic lot# X3201.

1/2g Muriatic / 10g H₂O. Stir + cool.

Added 12.07g Caustic solution to base. pH=3.5
Pour sample into vial.

Began adjusting to pH 2 w/Muriatic solution,
while adding HCl the color began getting pinker.
pH=2.0 Pour sample into vial.

Int. Crimson @ 0.025%: Initial pH=2.5 Peach shade.

Adjusted down to pH 2 w/Muriatic solution.

Added 0.18g of Muriatic solution. pH=2.0 Pour up
sample into vial. No color change.

Adjusted pH up to 3.5 w/Caustic solution.

Added 0.56g Caustic. pH=3.45. Color is still peach shade.
Pour sample into vial.

95/5 Scarlett/Magenta @ 0.03%: Initial pH=2.5 Peach shade.

Adjusted pH down to 2 w/Muriatic solution.

Added 0.70g of Muriatic solution. pH=2.0 Pour up
sample into vial. No color change.

Adjusted pH up to 3.5 w/Caustic solution. Added 0.50g
Caustic solution. pH=3.5 Pour up sample into
vial. No color change.

Continued on page 16.

Date Feb. 9, 1996

Date February 13, 1996

Signature Bruce Addith

Witness Fred Jostal

Technical Service #8603066
Continued from 10038-15.

Thermal Stabilities:

Place in oven on 2/9/96 2:00 PM. 40°C

Samples: Pink Reference

Crimson @ 0.025%

95/5 Scarlet/Magenta

Use as is samples w/o pH adjustment.

Allow to stand in oven for 2 weeks @ 40°C. Visually observe samples, no colorimeter readings.

2/12/96 Dried fabric samples w/ softener.

8:00 - Rinse samples in cold water. Change out water frequently until water is clear. Use spatula to agitate fabric samples lightly.

11:00 - Squeeze out water and hang to dry.

3:30 - Prepare display with one Untinted Control. The overall staining of the Pink Reference is worse than Crimson on Exp 10038-14 (which is 95% Crimson / 5% Lt. Pink). The Pink Reference left a dirty appearance.

All of the terry cloth samples were stained badly.

	Pink Reference	Crimson 00.03%	Exp 10038-14
Acetate	Light Stain	No Stain	No Stain
SFT	Light Stain	No Stain	No Stain
Armed	Light Stain	No Stain	No Stain
Cotton	Stain	Heavy Stain	Heavy Stain
Creslan 61	Light Stain	No Stain	No Stain
Dacron 54	Light Stain	No Stain	No Stain
Dacron 64	Light Stain	No Stain	No Stain
Nylon 6.6	Light Stain	No Stain	No Stain
Orlon 75	Light Stain	No Stain	No Stain
Silk	Light Stain	No Stain	No Stain
Polypropylene	No Stain	No Stain	No Stain
Viscose	Light Stain	Light Stain	Light Stain
Date	February 9, 1996	Signature	Brace Suddeth
Date	Debra 13, 1996	Witness	Liz Juddall
Wool	Light Stain	No Stain	No Stain

Continue on page 25.

Synthesis of M-Toluidine 10ED and 2-Chlorobenzaldehyde

Purpose: Make a green color similar to 9881-49.
Use M-Tol 10 instead of Aniline 10.

R/M	Lab #	MW	WT	Actual	Moles
M-Tol 10	01061	547	131.88	131.80	0.2411
2-Chlorobenzaldehyde	07902CX	140.57		16.71	0.1189
Urea	W2816	60		4.2	0.0700
Miniratic Acid (312) X3201		36.46	2.46	7.93	0.0674
BQ	W2794	108.1		3.21	0.0297

8:20 - Charge M-Tol 10, 2-Chlorobenzaldehyde, and urea to a 1L flask. Agitate.

9:40 - 25°C Add Miniratic and begin heating to 95°C w/ condenser.

10:00 - 85°C Amber shade and green color yet.

10:30 - 90°C Light green shade.

11:00 - 95°C Hold at 95-100°C for 3 hours. light green

11:20 - 100°C light green.

1:20 - 100°C

1:45 - 100°C Cut heat and allow to cool to <80°C.

2:20 - 60°C Change BQ and reheat to 95°C.

2:45 - 83°C

2:55 - 92°C

4:30 - 90°C Cut heat and allow to cool to RT.

2/13/96 8:00 - Turn on heat and allow to heat to ~40-50°C. Agitate.

12:30 - 60°C

3:00 - 55°C Sample for DV-300 + DV-383

Solids = 94.6% Absorb in MeOH $\frac{0.0868}{0.828 \times 10} = 0.105 @ 6671$

Trash Rx.

Date: February 12, 1996

Date: February 13, 1996

Signature:

Ronald Juddith

Witness:

Liz Junstall

Acid Red 52 Absorb. Acid Red EXB 400

Purpose: Run absorb in water and check duplicability.

Weigh sample by pipet into a 100ml flask.

$\frac{0.5145}{0.0324} \times 10 = 158.80$ @ 584λ in H₂O
Pipet 10ml into 1000ml flask.

Repeat same sample as above.

$\frac{0.6632}{0.0420} \times 10 = 157.90$ @ 584λ in H₂O

Recheck Sample of Acid Red EXB 400 cut to a
5.56 absorb Notebook # 9967-59.

Weigh directly into a 1L flask. Water as diluent

$\frac{0.18445}{0.1520} = 8.56$ @ 584λ

Date February 12, 1996

Date February 15, 1996

Signature

Bonnie Suddith

Witness

Dwight Marshall

Sunbeam Yellow Absorb Pecheck.

Purpose: Denver had a bad batch of Sunbeam Yellow with low absorb & poor conversion.

Lot # A1145 There was a precipitate in the bottom of a quart jar.

$$\text{Top Layer} \quad \frac{0.4034}{.0859} \times 2 = 9.39 @ 403 \lambda$$

$$450\lambda = 0.4459 \quad 530\lambda = 0.0021$$

$$\text{Bottom Precipitate} \quad \frac{0.8748}{.1464} \times 2 = 11.95 @ 403 \lambda$$

$$450\lambda = 0.4459 \quad 530\lambda = 0.0040$$

The products should have a λ of 395. This lot # A1145 has a λ of 403. Not good!

The λ should be 395 for Sunbeam Yellow.

Date February 13, 1996
Date February 15, 1996

Signature Bruce Muddeth
Witness Ditch Smith

PG Blue Analysis

Purpose: The plant has changed the procedure of how to make PG Blue. Originally made with Aniline 55 and Propylene Glycol, we now make it with Aniline 10, (Patent Blue) Strip the water and cut w/ propylene glycol.

Need to run viscosity of batch prior to changes vs batches after changes.

Lot #		cps @ 25°C
21004	Prior	72.5
PP01	Change	1,788
PP02	Change	435
C1007	Change	405
C1008	Change	380

Lot # PP01 was too hot while stripping and was burned and lost color value. That is why the viscosity is so high.

Lot # PP02, C1007, and C1008 had viscosities in the 400 range, whereas lot 21004 had a viscosity of 72.5.

Date: February 13, 1996
Date: February 14, 1996

Signature: Bruce Suddeth
Witness: John Junstall

Sunbeam Yellow - Control Batch w/ ^{+W/O} Stainless Agitator

Purpose: The plant is having a problem making Sunbeam Yellow. The last 3 or 4 batches have gotten really low CV. Need to run 2 batches. Run 1 with a stainless agitator and 1 without.

<u>Rxn</u>	<u>Lot#</u>	<u>MW</u>	<u>wt</u>	<u>Moles</u>
Geffamine 715	N0708	715	313.25	156.63
Water #1	DI	18	73.5	36.75
Na Acetate	G6851	82	50.5	25.25
PABSC	Q1617	233.67	92.75	46.38
Muriatic #1 (312) x 320g		36.46	93.5	46.75
Muriatic #1 (313) x 320g		36.46	93.5	46.75
H ₂ O #2	DI	18	73.5	36.75
H ₂ O #3	DI	18	118	59
Na ND ₂		69	28	14
				0.2029

Cut above charges in half. Rx has teflon stir blade.

9:05 - Charge Geffamine + H₂O #1 to a 1L flask. Agitate.

9:30 - Charge 50ml Na Acetate.

9:45 - 35°C Start PABSC add. Keep temp. < 50°C.

10:20 - 43°C Adding PABSC.

10:45 - 48°C Complete PABSC add and begin heating Rx to 95°C.

11:30 - 92°C

12:00 - 95°C Hold at 95°C for 3 hours.

12:45 - 97°C

1:30 - 95°C

3:00 - 95°C Cut heat and cool to < 60°C.

2/15/96 Agitate and charge Muriatic #1 and heat to 95°C.

8:45 - 90°C

9:00 - 95°C Hold for 3 hours at 95°C,

10:05 - 96°C

11:15 - 95°C

12:00 - 95°C Cut heat & allow to cool.

Continued on page 23.

Date February 14, 1996

Signature Bruce Suddeth

Date February 15, 1996

Witness Linda Gunstall

10038-22

Smilean Yellow w/ Stainless Agitator

Purpose: Make a SBY with a stainless agitator during diazotization.

<u>R/M</u>	<u>CD#</u>	<u>MW</u>	<u>WT</u>	<u>Moles</u>
Jeffamine 715	N0708	715	156.63	0.2191
Water #1	DI	18	36.75	
Na Acetate	G6851	82	25.25	0.3079
PABSC	Q1617	233.67	46.38	0.1985
Muriatic #1(312)	X3201	36.46	46.75	0.3975
Muriatic #2(312)	X3201	36.46	46.75	0.3975
H ₂ O #2	DI	18	36.75	
H ₂ O #3	DI	18	59	
Na NO ₂		69	14	0.2029

9:30 - Charge Jeffamine, Water #1, and Na Acetate to a 1L flask. Agitate. Stainless agitator.

9:55 - 40°C
Begin adding PABSC slowly. Keep temp < 50°C.
Adding PABSC.

10:20 - 45°C
10:45 - 45°C
Rx to 95°C. Complete PABSC add. Begin heating

11:30 - 95°C Hold Rx at 95°C for 3 hours.

12:00 - 97°C

12:45 - 93°C

1:30 - 96°C

3:00 - 95°C Cut heat & cool to < 60°C.

3/15/96 25°C Agitate and charge Muriatic #1. Heat to 95°C.

8:45 - 90°C

9:00 - 95°C Hold at 95°C for 3 hours.

10:00 - 95°C

11:15 - 97°C

12:00 - 95°C Cut heat. Allow to cool.

Reaction is redder than 10038-21. Due to stainless agitator paddle.

Continue on page 24.

Date February 14, 1996

Date February 15, 1996

Signature Bruce Andrich

Witness Linda Simstall

Sundan Yellow - Control Batch w/Teflon Agitator
 Continued from page 21.

2/16/96 Begin cooling Rx down to $< 5^{\circ}\text{C}$.
 7:50 - 15°C Charge Mannite #2 and $\text{H}_2\text{O}^{\#2}$
 8:20 - 10°C Prepare Sodium Nitrite + $\text{H}_2\text{O}^{\#3}$ solution.
 8:30 - 3°C Begin nitrite solution add. Keep temp
 $< 5^{\circ}\text{C}$. Color of diazo at this point is a
 light yellow/tan color.
 9:45 - 0°C Complete add. Hold at $0-5^{\circ}\text{C}$ for
 3 hours.
 11:00 - 2°C
 1:00 - 2°C

Coupling Minic plant. Start coupling at pH of 8.
 Add diazo till pH drops to < 4 . Add Versene.
 Adjust to the 4-5 range. Adjust pH to 8 to
 phase.

Rxn	Lot #	wt	Actual
Pyrogallol	A4076	46.88	1451
H_2O	DI	94.05	
Versene 100XL	V2577	34.58	
Diazo		280.95g	291.5
Cannistic.	V2568	15.9	

11:35 - Charge Pyrogallol + H_2O to a 1L beaker. Agitate.
 Add Versene. Adjust pH to 8.0. Initial pH = 3.65
 Added 15.90g Cannistic pH = 8.0. Begin cooling
 to $< 10^{\circ}\text{C}$.

Coupler Temp $< 0^{\circ}\text{C}$. 1:00 PM.

1:05 - 3°C Begin coupling.
 1:20 - 8°C complete couple pH = 2.7 Add Versene
 to a pH of 4-4.5. Added 83.12g Versene. Hold
 for 3 hours at $< 10^{\circ}\text{C}$ pH = 4.1

2:40 - 5°C

3:30 - 6°C Allow Rx to sit over weekend.

Continued on page 26.

Date February 16, 1996
 Date February 22, 1996

Signature Bruce Suddeth
 Witness Linda Sunstall

Sunbeam Yellow w/ Stainless Agitator.
Continued from page 22.

2/16/96 Begin cooling Rx to $\leq 5^{\circ}\text{C}$
 7:55 - 10°C Charge Muriatic #2 and $\text{H}_2\text{O}^{\#2}$.
 8:15 - 7°C Prepare $\text{H}_2\text{O}^{\#3}$ and Sodium Nitrite solution.
 8:30 - 4°C Begin nitrite solution add.
 Keep temp $\leq 5^{\circ}\text{C}$. Drags began to foam a little while adding nitrite. Drags is a real red color. Does not look good.
 8:45 - 0°C Complete add. Hold at $0-5^{\circ}\text{C}$ for 3 hours.
 11:00 - 1°C
 1:00 - 3°C

Coupling Same as 10038-23.

Part	Lot #	wt	Actual
Polypropylene	A4076	46.88	
H_2O	DI	34.05	
Versene 100XL	V2577	34.58	
Drags		280.95	284.70
Caustic.		17.11	

11:35 - Charge Polypropylene and H_2O to a 1/2 beaker. Agitate. Add Versene. Initial pH = 3.65 Adjust to 8.0 w/ Caustic. Added 17.11g Caustic. pH = 8.10
 12:00 - Begin cooling to $\leq 10^{\circ}\text{C}$.
 1:00 - Coupler Temp = 0°C
 1:05 - 0°C Begin coupling.
 1:20 - 80°C Complete couple. pH = 2.6 Add Versene to a pH of 4-4.5. Added 86.36g Versene - pH = 4.1 Hold for 3 hours at $< 10^{\circ}\text{C}$.
 2:40 - 5°C
 3:30 - 7°C Allow Rx to stir over weekend.

Continued on page 27.

Date February 16, 1996
 Date February 22, 1996

Signature Brian Smith
 Witness Dick Junstaf

TS# 9603066

Continued from page 16.

1 week stabilities @ 40°C:

Pink Reference: Stable after 1 week, no color change.

Crimson @ 0.025%: Stable after 1 week.

75/5 Lt.Crimson/Lt.Pink @ 0.06%: Stable after 1 week.

All colors above are stable at room temp. 1 week.

2 week stabilities: 2 weeks

All samples are stable after 2 weeks.

Date February 16, 1956
February 22, 1956 P.P.

Signature

Bruce Ballou
Rich Swastik

Witness

10038-26

Simbea... Yellow - Control Batch w/ Teflon. Agitation.

Continued from page 23.

2/16/96 Batch has stirred over weekend.

10:30A 2/20/96 adjusted pH to 8.35 w/ 50g Caustic
w/w 37.8 grams

10:45A Place in Sep funnel and let sit in
60°C oven and let settle for 1.5 hrs.

Separate batch into Beaker. Product is on bottom
Product wt: 7.22
Salt by wt: 232.75

left in beaker overnight

2/21/96 Agitate product layer and measure absorbance solid.

DV-300 = 67.90% DV-383: MeOH $\frac{D_{7323}}{1348} \times 2 = 10.86 @ 4031$

$$CV = 15.99$$

$$SR = \frac{D_{3819}}{2911} = 1.31$$

Abs @ 450 1st Dil
Abs @ 530 2nd Dil.

Date February 16, 1996
Date February 20, 1996

Signature: Brian Sanderson
Witness: Dennis J. Martell

10038-27

Sundbeam (yellow w/ Stirring) Agitation.
Container & lid from page 24.

2/16/96 Allowed Rx to stir over weekend.

2/20/96 adjusted pH to 8.3 w/ 50g. Caustic
used 38.3 grams.

Place in Sep funnel and let settle in 60°C
oven for 1.5 hrs.

Separate Product layer into beakers / product is on bottom
product wt = 343
salt layer wt = 391.44

let stand in beakers overnight

2/21/96 Agitate product layer measure absorb + solids.
DV-300 = 67.28% DV-383 = 0.5616 x 2 = 9.69 @ 401λ
0.1159

$$EV = 14.40$$

$$SR = \frac{0.2847}{0.601} = 4.74$$

Date: February 16, 1996
Date: February 21, 1996

Signature: Bruce Andleeth
Witness: Linda Sunstall

Technical Service Request # 9603068

Originator: Syng

Date In: 2/16/96

Customer: Colgate Palmolive

Location: Belgium

Application: Fabric Softener Color

Technical Support

Contact: Viviane Tech

Fabricoso, Base

Objective: Fabuloso, Violets FV, TSR# 9602038

Service Requested: Match Violet FV. Try for brightness. Colormatch was achieved w/ 88.5/11.5 Pink/Violet shade. Please do stain-testing and thermal stability. Use Violet as a comparison in testing. Adjust the pH to a 2 and a 3.5 to check for a shade shift (down w/ HCl and up w/ NaOH).

Do 13 fiber staining and cotton terry staining.
Thermal Stability, 2 and 4 week @ 100°F.

Try:	A) 88.5 / 11.5 B) Violet C) 76 / 24 D) 82.5 / 17.5	Pink / Violets Red ST / Violets Pink / Violets	O. O16% O. O17%
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Received Reference Standard on 2/15/96.

Date: February 16, 1996
Date: February 23, 1996

Signature: Bonnie Leedleth
Witness: Rich Dunstall

Peach 96

Using the lower and upper Spec. limits of Acid Red 52 at 5.5 Absorb, and Sunbeam Yellow @ 10 Absorb calculate changes.

Acid Red E-XB 4010 @ 5.56 Absorb 9967-59

Sunbeam Yellow @ 9.95 Absorb lot V1129

Spec Range = 2.28 - 2.78 @ 395-405, 3.70 - 4.46 @ 553 - 569

Cow / Low

23.87_g S.B. Yellow / 70.91_g AR52 + 5.22 g H₂O

Actual 5.77 / 14.18 + 1.04

$$\text{DV-18 in H}_2\text{O} = \frac{0.77}{.2495} = 2.57 \text{ e4002A}, \frac{1.04}{.2495} = 3.95 \text{ e5641}$$

Low / Hi

23.87_g SB Yellow / 77.73_g AR52 - 1.6_g H₂O
4.53 / 15.54 - .32

Weight of vial = 37.07_g Dry down to 36.75_g

$$\text{DV-18 in water} = \text{SW} = \frac{0.2721}{.6958} = \frac{1.1718 @ 569}{399} = 4.31$$

Hi / Low

26.38_g SB Yellow / 70.91_g AR52 + 2.71_g H₂O

5.28_g / 14.18_g + .54

$$\text{DV-18 in H}_2\text{O} = \frac{0.7387}{.2610} = 2.83 \text{ e4002A}, \frac{1.0454}{.2610} = 4.01 \text{ e5641}$$

Hi / Hi

26.38_g SB Yellow / 77.73_g AR52 - 4.11_g H₂O
5.28_g / 15.54_g - 0.82_g

Weight of vial = 37.5_g Dry down to 36.68_g Dry to 36.74_g
Place low / Hi & Hi / Hi in 60°C oven at 11:30 AM.

12:00 - Take samples out of oven.

2/22/96 10:15 Place samples in 60°C oven, 11:30 - Take out of oven, allow to cool and reweigh.

11:45 - Whipped samples and did not dry enough.

Place back in 60°C oven,

Continue on page 30.

Date February 21, 1996

Date February 22, 1996

Signature Bruce Maddith

Witness First Dunstall

Peach 96

Continued from page 29.

While the Low/Hi and Hi/Hi samples are drying down. Prepare other 2 samples in fabric softener. Make in 1% solution in water. Add 3% of that into Fabuloso base. Agitate.

This color is supposed to go in New Variant base but we do not have any!

Next day - reweighed sample that had been in oven overnight.

500 = Dried down - vial, capt color 35.83 gms.

DV = 18 in water.

$$\text{SW} = \frac{0.2431}{0.7113} = 0.0754 @ 564 = 4.50$$

Prepare Low/Hi and Hi/Hi blends in H_2O , 1% solution of H_2O . Add 3% of each blend into Fabuloso base. Agitate.

Tabulate all #'s.

<u>Low/Low</u>	<u>Low/Hi</u>	<u>Hi/Low</u>	<u>Hi/Hi</u>	
2.57	2.56	2.83	2.92	Sunbeam
3.95	4.31	4.01	4.50	Acid Red 52

Concentrate at the 4 above blends in fabric softener.

Based on results the visuas correlate. The Low/Hi sample is the most red.

Steven wants to cut the blends to the specs already set. Start w/ Low/Low (B) sample.

$2.57/2.3 = 1.12$ or $3.95/3.7 = 1.07$ Charge 5 grams of blend. Add 0.60g H_2O and agitate.

Absorb = 2.19 @ 401A, 3.39 @ 564A

Hi/Hi (B)

$2.92/2.78 = 1.05$ or $4.50/4.50 = 1.0$ Charge 5 grams of blend to vial. Add 0.25g H_2O and agitate.

Absorb = 2.74 @ 401A, 4.23 @ 564A

Continue on page 32.

Date: February 22, 1996

Date: February 22, 1996

Signature: Bruce Hollie

Witness: Vicki Sunbeam

Pilot Plants package for 95/5 Lt. Crimson / Lt. Pink

Measure absorb of Lt. Crimson + Lt. Pink.

Crimson Lot# S1135 DV-478 in MeOH

$$\frac{0.3508}{.1167} = 3.01 @ 503\lambda \quad \text{Spec} = 2.80 - 3.20$$

Lt. Pink Lot# H1105 DV-478 in MeOH

$$\frac{0.1544}{.1151} \times 5 = 6.71 @ 556\lambda \quad \text{Spec} = 6.5 - 7.5$$

Prepared a sample of 95/5 Crimson/Pink. Added 0.03% to Fabuloso base. This is same as 10038-14.

Place samples in oven and dry down to top of absorb Spec.

Need to calculate the charges for top + bottom of R/M spec. ranges.

<u>Crimson</u>	
Low	88.67
Hi	100.99
Low	88.67
Hi	100.99

<u>Pink</u>		<u>Water</u>
Low	4.84	6.49
Low	4.84	
Hi	5.57	5.74
Hi	5.57	

Do not use these ratios but rather cut the R/M to top + bottom of spec. Blend all at 95/5 ratio.

Cut Crimson Lot# S1135 to a 2.80 Absorb in MeOH.
 $50g \times \frac{3.01}{2.80} - 50 = 3.75 \text{ H}_2\text{O}$ Stir well,
 $\frac{0.3583}{2.82} @ 503\lambda$

$$DV-478 = .1270$$

Cut Magenta Lot# H1105 to a 6.5 Absorb in MeOH.
 $16.65 \times \frac{6.71}{6.5} - 16.65 = 0.518 \text{ H}_2\text{O}$, Stir well,
 $\frac{0.2634}{6.26} \times 2 = 0.26 @ 557\lambda$

$$DV-478 = .0842$$

Continued on page 34.

Date February 22, 1996

Signature Bruce Suddeth

Date February 29, 1996

Witness Linda Junstall

Dench 96

Continued from page 30

Prepare 1% solution of Low/Hi (B) and Hi/Hi (B) in H₂O. Add 3% to Fabuloso base. Agitate well. Pour each into vial for comparison.

Low/Hi (B)

$2.56/2.3 = 1.11$, and $4.31/4.46 = 0.97$ This cannot be done w/o adding AR52 to sample.

Charge 5g of blend to vial. Add 0.50g H₂O.

Add 0.50g AR52 and agitate.

$$\frac{0.5076}{.2279} = \frac{2.23 @ 401\lambda + 0.9125}{0.2279} = 4.00 @ 564\lambda$$

Hi/Hi (B)

$2.83/2.80 = 1.01$, and $4.01/3.7 = 1.08$ Will have to backadd Sunbeam Yellow.

Charge 5g of blend to vial. Add 0.40g H₂O.

Add 0.20g Sunbeam Yellow.

$$\frac{0.6847}{.2317} = \frac{2.96 @ 402\lambda + 0.8148}{.2317} = 3.52 @ 564\lambda$$

The red peak was too low in both samples above.

Add AR52 to bring up absorb.

Low/Hi - Add 0.25g AR52 @ 5.5 Absorb to blend above. $\frac{0.5146}{.2406} = 2.14 @ 401\lambda, \frac{0.9752}{.2406} = 4.05 @ 564\lambda$

Hi/Hi - Add 0.75g of AR52 @ 5.5 Absorb to blend above. $\frac{0.7353}{.2464} = 2.98 @ 389\lambda, \frac{0.8731}{.2464} = 3.54 @ 564\lambda$

Use this Hi/Hi (B) sample to add to softener base.

Continue to work on the Hi/Hi (B) sample.

Continued on page 33.

Date February 26, 1996

Signature Bruce Middlecamp

Date February 28, 1996

Witness Linda Denotall

Peach 96

Continued from page 32.

Low/Hg B - Add 0.50g AR52 @ 5.5 Absorb to blend.

Also add 0.10g Sunbeam Yellow.

$$\frac{0.5242}{.2435} = 2.15 @ 401\lambda, \frac{.9968}{.2435} = 4.09 @ 564\lambda$$

Add 0.50g AR52 to Blend. Measure absorb.

$$\frac{0.4478}{.2296} = 1.95 @ 401\lambda, \frac{.9759}{.2296} = 4.25 @ 564\lambda$$

Values are still too low.

Add 0.2g Sunbeam Yellow and 0.75g AR52.
Stir well.

$$\frac{0.3915}{.1967} = 1.99 @ 401\lambda, \frac{0.8423}{.1967} = 4.28 @ 564\lambda.$$

Cannot get to the absorb I want with this sample.

Use a Low/Hg sample blend that Linda has.
Measure absorb.

$$\frac{0.5919}{.2284} = 2.57, \frac{0.9781}{.2284} = 4.28$$

Add 0.50g of AR52 to the vial sample. Agitate
Measure Absorb

$$\frac{0.5239}{.2146} = 2.44, \frac{0.9322}{.2146} = 4.34$$

Add 1.0g of AR52 to the vial above. Agitate.

$$\frac{0.4664}{.2103} = 2.22, \frac{0.9412}{.2103} = 4.48. \text{ Use this sample.}$$

Make a 1% solution in H₂O. Add 3% to Formless base.
Agitate.

Jim Sny has looked at the samples and
recognized the shade differences.

February 26, 1996
February 28, 1996

Bruce Suddett
Linda Sunbeam

• 95/5 Lt. Crimson / Lt. Pink
Continued from page 31.

The absorb of Crimson sample is fine for blending.
The Magenta sample needs color (higher absorb)
added to it.

16.98g in beaker. Add 0.50g of Palmer Magenta lot # C1131.
Agitate. $\frac{0.2151}{.12331} = \textcircled{6.50} @ 556\lambda$.

Measure absorb on samples of Crimson and Pink that
have been evaginated.

Crimson DV-478 in MeOH Spec = 2.80 - 3.20
 $\frac{0.3761}{.1130} \times 2 = 6.65$

$23.98 \times \frac{6.65}{3.20} - 23.98 = 25.85 \text{g H}_2\text{O}$ Agitate.

DV-478 = $\frac{.3888}{.1196} = \textcircled{3.25} @ 502\lambda$.

Pink DV-478 in MeOH Spec = 6.5 - 7.5
 $\frac{0.7632}{.1327} \times 2 = 11.50$

$21.40 \times \frac{11.50}{7.5} - 21.40 = 11.41 \text{g H}_2\text{O}$ Agitate.

DV-478 = $\frac{0.8472}{.1096} = 7.73 @ 556\lambda$. Cut Rx to 7.5

$32.22 \times \frac{7.73}{7.5} - 32.22 = 0.88 \text{g H}_2\text{O}$ Agitate.

$\frac{0.7201}{.0956} = \textcircled{7.53} @ 556\lambda$

Continue on page 35.

Date February 27, 1996
Date February 28, 1996

Signature Bruce Suddeth
Witness Vicki Sundall

95/5 Lt Crimson / Lt. Pink. None is Lt. Pink 10038
 Continued from page 34.
 Using the RYM adjusted to the high and low
 end of spec. prepare a 95/5 blend.

A) Low/Low	Crimson/Pink	9.5g / 0.5g
B) Low/Hi	"	"
C) Hi/Low	"	"
D) Hi/Hi	"	"

Add 0.03% to the Fabuloso base. 99.97% Fabuloso base.
 Stir each sample well. Pour into vials and
 observe color difference. Samples look real good,
 there is hardly a color difference.

Measure the absorbances of the above blends to
 hopefully establish specs. Use the Balmer procedure
 for measuring absorb. 0.50g in 100, pipet 2ml to
 100, use MeOH as solvent.

Low/Low DV-478

$$\frac{0.4892}{.3347} \times 2 = 2.92 @ 504.5\lambda$$

Low/Hi

$$\frac{0.5003}{.3375} \times 2 = 2.96 @ 505\lambda$$

Hi/Low

$$\frac{0.5863}{.3509} \times 2 = 3.34 @ 504.5\lambda$$

Hi/Hi

$$\frac{0.5640}{.3361} \times 2 = 3.41 @ 504.5\lambda$$

Continue on page 36.

Date February 27, 1996

Date February 28, 1996

Signature

Bruce Buddell
John Twissell

Witness

95/5 Crimson / Lt. Pink Name is Pink 10038.
Continued from page 35.

The Pinky is in such a low concentration it is being hidden by the Crimson.

Ask John Brinkhake about how to set specs. Need to measure Rm at 505λ and 557λ to see what each contributes to the spec.

Palmer Scarlett (Lt. Crimson) Lot # B1147
Max absorb = $\frac{0.5152}{.3454} \times 2 = 2.98 @ 502\lambda$

$$Abs @ 505 = \frac{0.5735}{.3454} \times 2 = 2.97 @ 505\lambda$$

$$Abs @ 557 = \frac{0.1999}{.3454} \times 2 = 1.16 @ 557\lambda$$

Palmer Magenta (Lt. Pink) lot # C1131
Max Absorb = $\frac{1.0932}{.3379} \times 2 = 6.47 @ 556\lambda$

$$Abs @ 505 = \frac{0.7535}{.3379} \times 2 = 4.46 @ 505\lambda$$

$$Abs @ 557 = \frac{1.0893}{.3379} \times 2 = 6.45 @ 557\lambda$$

Use the above numbers to calculate spec. ranges for the blend.

Continue on page 37.

Date February 28, 1996
Date February 28, 1996

Signature Bruce Suddeth
Witness Rich Denstell

95% Lt. Crimson / Lt. Pink Name is Pinta 10038

Continued from page 36.

Shade ratio at 557 / 505

$$= \frac{(\text{Crimson change})(\text{Crimson Abs @ 557}) + (\text{Pink change})(\text{Pink Abs @ 557})}{(\text{Crimson change})(\text{Crimson Abs @ 505}) + (\text{Pink change})(\text{Pink Abs @ 505})}$$

$$\text{Mav} = \frac{(\text{Crimson change})(\text{Crimson Abs @ 505}) + (\text{Pink change})(\text{Pink Abs @ 505})}{\text{Batch Weight}}$$

Pinto Scarlet Crimson

Mav Abs @ 502 = 2.98

@ 557 = 1.16

Abs @ 557 = 0.393 Mav

Pinta

Mav Abs @ 505 = 4.46

@ 558 = 6.47

Abs @ 505 = 0.683 Mav

Standard Changes = 95% Crimson 5% Pink

$$\text{Mav} = \frac{(5)(0.683)(6.5 - 7.5) + (85)(2.8 - 3.2)}{100}$$

$$= \frac{(22.3925 - 25.8375) + (85)(2.8 - 3.2)(266 - 304)}{100}$$

$$\text{Mav Spec Range} = 2.08 - 3.30$$

use $(2.8 - 3.5)$ as true range

$$\text{Shade Ratio} = \frac{(5)(6.5 - 7.5) + (85)(0.393)(2.8 - 3.2)}{(5)(0.683)(6.5 - 7.5) + (85)(2.8 - 3.2)}$$

$$= \frac{(32.5 - 37.5) + (104.538 - 119.472)}{(22.3925 - 25.8375) + (266 - 304)}$$

$$= \frac{(137.038 - 156.372)}{(288.3925 - 329.8375)} = 0.475, 0.415, 0.544, 0.476$$

$$[\text{SR Range} = 0.415 - 0.544]$$

Continue on page 38.

Date February 28, 1996

Date February 28, 1996

Signature Bruce Middleth

Witness Lincoln Denotali

95/5 Crimson / Pink None in Pink 10038
 Continued from page 37.
 Lab Batch

Palmer Scarlett (Crimson) lot # B1147
 Charge 380 grams to a 600 ml beaker.

Palmer Magenta (Pink) lot # C1131
 Charge 20 grams to above 600 ml beaker.
 Agitate well. Label as Lt. Pink 10038 lot # 10038-38A
 Measure Solids DV-300 = 32.49%
 pH DV-5 = 6.6
 Specific Gravity DV-33 = $10.80/10 = 1.080$

Prepare F1 form for an MSDS to be generated.

Measure Absorb of blend alone.

Max Absorb DV-478 = $\frac{0.7308}{.4745} \times 2 = 3.08 @ 505\text{nm}$ Spec = 2.8-3.5

Shade Ratio $\frac{552}{505} = \frac{0.7308}{.7308} \cdot \frac{.342}{.342} = 0.478$ Spec = 0.415-0.544

Blend Passes.

Prepare a second blend, same as above.

Palmer Scarlett (Crimson) lot # B1147

Charge 180 grams to a 400 ml beaker.

Palmer Magenta (Pink) lot # C1131
 Charge 10 grams to beaker alone. Stir well.

Label as Lt. Pink 10038 - lot # 10038-38 B

Measure Absorb of blend.

Max Absorb DV-478 $\frac{0.7324}{.4758} \times 2 = 3.08 @ 505\text{nm}$

Shade Ratio $\frac{552}{505} = \frac{0.3492}{.7324} = 0.477$ SR

pH DV-5 = 6.6 Solids DV-300 = 32.46%

Date February 28, 1996

Date February 28, 1996

Signature Bruce Scarlett

Witness Linda Crystal

Lignite-6 Points 10038

Using Lot #10038-38A run a viscosity and specific Gravity vs Temperature.

DV-28 and DV-33. Check each at 4 points

0°C 15°C 30°C 50°C

10:15 - Start at 0°C. Hooke #00249

Specific Gravity Flash + Top Weight = 24.7199
Use a 25ml Volumetric.

$$0^\circ\text{C} = 52.3086 - 24.7199 = 27.5887/25 = 1.1035$$

$$15^\circ\text{C} = 52.1633 - 24.7199 = 27.4434/25 = 1.0977$$

$$30^\circ\text{C} = 51.9611 - 24.7199 = 27.2412/25 = 1.0896$$

$$50^\circ\text{C} = 51.7054 - 24.7199 = 26.9861/25 = 1.0794$$

Flash Points - Same as Lt. Crimson and Pink

Viscosity 150 ml beaker #1 spindle @ 60 rpm.
Viscometer #00248

$$0^\circ\text{C} = 32 \text{ cps}$$

$$15^\circ\text{C} = 19.5 \text{ cps}$$

$$30^\circ\text{C} = 13.5 \text{ cps}$$

$$50^\circ\text{C} = 10 \text{ cps}$$

Freeze Point DV-156 = -10°C

Centrifuge Test DV-15 = No Sediment after Freeze/Thaw.



Centrifuge Test DV-15A Check for sediment after 30 minutes at 2800 rpm. Pass, No sediment.

Recommended Container - Plastic, Poly drum or glass

Date March 5, 1996

Date March 5, 1996

Signature Bruce Sundell
Witness Rich Sundell

Liquitint Pink 10038'

Freeze / Thaw Stability - Place lot #10038-38A in
freezer on 3/5/96. Run 3 cycles.
Initial - Max Absorb = 3.08 Shade Ratio = 0.478
F/T after 3 cycles = $\frac{0.7592}{.5033} \times 2 = 3.02 @ 505\text{nm}$
 $SR = \frac{0.3498}{.7592} = 0.461$

Oven Aging - 140°F for 2 weeks. Place in oven on
3/5/96.
Initial - Max Absorb = 3.08 Shade Ratio = 0.478

Shear Stability - Stable @ 5 minutes, foam slightly.

Decolorization Procedure - Charge 5.7 grams color
to beaker. Add 2.004 g H₂O. Agitate. Add
2.0 grams ~~sodium hydroxide~~ and warm to 110-120°F.
After holding for 15 minutes solution is colorless.
Allow to cool and observe if color returns.
Cooled to 30°C and no color evident.
For every gram of colorant add 0.35 grams
of NaOH T.

Date March 5, 1996
Date March 7, 1996

Signature Bruce Saldeth
Witness Richie Williams

Liquitint Red ST

Flash Point DV-86
Lot # RDDI

>500°F or >250°C

Freeze / Thaw Stability Start on 3/6/96 3 cycles

Initial = 10.15 After 3 cycles F/T = $\frac{0.5330}{.1042} \times 2 = 10.23$

Oven Aging - 140°F Start on 3/6/96 2 weeks
~~10.145~~ 3/11/96 $\frac{0.9231}{.1800} \times 2 = 10.26$ @ 521 hr in Month.

3/18/96 $\frac{1.0482}{.2060} \times 2 = 10.18$ @ 521 hr

Centrifuge Test - Pass after F/T + oven age.

Date March 6, 1996
Date March 7, 1996

Signature Bruce Holdeth
Witness Lynn Tunstall

Liquid to Pint. 10038 NIPQC

use lot # 10038-38A

<u>Analysis #1:</u>	<u>Analysis</u>	<u>Specification</u>	<u>Result</u>
2/28/96	Solids	60 Max	32.45%
	pH	5.5 - 7.0	6.6
	Max Absorb	2.8 - 3.5	3.08
	Shade Ratio	0.415 - 0.544	0.478

<u>Analysis #2:</u>	Solids	32.46%
2/28/96	pH	6.6
	Max Absorb	3.08
	Shade Ratio	0.477

<u>Analysis #3:</u>	Solids	32.58%
3/6/96	pH	6.6
	Max Absorb	3.14
	Shade Ratio	0.469

<u>Analysis #4:</u>	Solids	32.52%
3/7/96	pH	6.6
	Max Absorb	3.16
	Shade Ratio	0.471

<u>Analysis #5:</u>	Solids	32.54%
3/7/96	pH	6.6
	Max Absorb	3.16
	Shade Ratio	0.468

Recheck Analysis of Max Absorb.

<u>Analysis #6:</u>	Max. Absorb.	3.15
3/7/96	Shade Ratio	0.471

<u>Analysis #7:</u>	Max. Absorb	3.16
3/8/96	Shade Ratio	0.469

Continue on page 43.

Date March 7, 1996

Date March 11, 1996

Signature Bruce Muddith

Witness Ruth Dunstall

Lignite P.L. No. 10038 $\sqrt{D}P\ AC$
 Contained from page 42.

Calculate Solids Process Tolerance -
 $\bar{x} = 32.52 \quad \sigma_{x_{n-1}} = 0.0460$
 $\frac{0.0460 \times 6 \text{ sigma}}{60} \times 100 = 0.46\% \text{ PPT}$

Calculate pH Process Tolerance -
 $\bar{x} = 6.6 \quad \sigma_{x_{n-1}} = 0.00$
 $\frac{0.00 \times 6 \text{ sigma}}{1.5} \times 100 = 0.8\% \text{ PPT}$

Calculate Max. Absorb Process Tolerance -
 $\bar{x} = 3.154 \quad \sigma_{x_{n-1}} = 0.0089$
 $\frac{0.0089 \times 6 \text{ sigma}}{0.70} \times 100 = 7.67\% \text{ PPT}$

Calculate Sludge Ratio Process Tolerance -
 $\bar{x} = 0.4696 \quad \sigma_{x_{n-1}} = 0.00134$
 $\frac{0.00134 \times 6 \text{ sigma}}{0.129} \times 100 = 6.23\% \text{ PPT}$

Date March 7, 1996
 Date March 7, 1994

Signature Bruce Sandelth
 Witness Ruth O'Connell

Sunbeam Yellow w/ Experimental 9824-35 Amino Replacement

Purpose: Synthesize Sunbeam w/ John Brinkley
Amino (MEA + Benzaldehyde + HED).

RM	Lot #	mw	wt	Act wt	Moles
Exp 9824-35	9994-26	237	25.96	25.96	0.1096
Water #1	DI	18		18.38	
Na Acetate	668512388	82	12.62	12.62	0.1540
PABSC	Q1617	233.67	23.19	23.19	0.0993
Muriatic #1(31%)	X3201	36.46	7.25	23.38	0.1988
Muriatic #2(31%)	X3201	36.46	7.25	23.38	0.1988
H ₂ O #2	DI	18	18.38	18.37	
H ₂ O #3	DI	18		28.5	
Na NO ₂	A5586	69	7.00	7.00	0.1015

8:20 - Charge Exp 9824-35 to a 1L flask. Add H₂O #1.

Begin agitation.

8:30 - Charge Sodium Acetate.

8:40 - 28°C Begin adding PABSC. Keep temp < 50°C during add.

9:25 - 38°C Complete add. Check pH = 6.5 @ 38°C.

Begin heating Rx to 95°C.

9:40 - 70°C

10:08 - 85°C

10:15 - 90°C

10:20 - 94°C

Hold for 3 hours at ~95°C.

11:30 - 95°C

12:20 - 95°C

1:00 - 95°C

1:20 - 95°C

Cut heat & allow to cool to ~60°C.

1:50 - 45°C

Charge Muriatic #1 and reheat Rx to 95°C.

2:40 - 94°C

Hold for 3 hours.

3:00 - 95°C

Cut heat & allow to cool.

3/12/96 - 25°C

Begin cooling to ~5°C. Charge Muriatic #2

and H₂O #2.

Continue on page 45.

Date: March 11, 1996

Date: March 11, 1996

Signature: Bruce Mifflin

Witness: Richard Dainton

Sunbeam Yellow w/ Experimental 7824-35 Amine Replacement

Continued from page 44.

7:30 - Prepare $\text{NaNO}_2 + \text{H}_2\text{O}^{+3}$ solution. Mix well.

7:45 - 4°C Begin NaNO_2 solution addition slowly. Keep temp $< 5^\circ\text{C}$.

10:15Q 0°C Complete NaNO_2 add. Hold at $< 5^\circ\text{C}$

for 3 hours. Check excess nitrite w/ KI paper. Re does have excess nitrite.

Check nitrite every hour during diazotization.

10:52 - 0°C

12:00 - -2°C Check excess nitrite. Positive.

2:25 - 0°C - Slight excess nitrite. Dye is a light brown color.

Coupling - Prepare Coupling Solution.

BPM	100	wt
Polyazobenzene	R1786	46.88 23.44
H_2O	DI	94.05 47.03
Versene 100XL	V2577	17.29
Dye		140.5
2:30 -		

Add Polyazobenzene & H_2O to a 1L beaker. Stir.

Initial pH = 1.75. Add Versene & begin cooling to 5°C . pH after Versene is 3.75. Adjust pH to 8 w/ Caustic. Add V2568. Added 8.7g Caustic. pH 8.25. Continue to cool to 5°C .

3:15 - 4°C Begin to couple slowly. Keep temp $< 10^\circ\text{C}$.

3:20 - 8°C Add ~20 drops. Re thickened like normal. Keep pH > 5 while coupling initially.

3:45 - 9°C Allow to cool down.

3:55 - 5°C Continue coupling. Adding Versene slowly.

4:15 - 0°C Complete coupling. Added 73.41g Versene pH = 4.25. Continue to cool and let temp. rise overnight.

Continued on page 46.

Date March 12, 1996

Date March 19, 1996

Signature Bruce Sandell

Witness Ruth Ann Sunstall

Sunbeam Yellow w/ Experimented 9824-35 Amino Replacement

Continued from page 45.

4:30 - 5°C Allow to stir overnight. Cover w/ foil. pH = 4.20
3/13/96 - 20°C Rx appeared to have some foam on top.

Still a yellow color. pH = 3.95

Adjust pH to ~8.5 w/ 50% Canstic.

8:15 - 30°C Charged 25.97g Canstic lot # V2568. pH = 8.5

Pour Rx into separation funnel and place in 60°C oven for 1.5 hours. Hopefully Rx will phase separate in the oven.

10:30 - Remove separation funnel from oven.
Had a good phase. Product on bottom layer.

Retained 70.60g product. Agitate well.

Retained 258.95g of Salt/H₂O layer.

Measure Solids DV-300 and Absorb DV-383.

DV-300 = 59.35%. DV-383 in MeOH = 1.0613 $\times 2 = 13.50 @ 393\lambda$
1572

Color Value = 22.75 Theoretical Yield = 95.31 grams.

Product Layer = 66.27g Cnt to a 10 Absorb.

$66.27 \times \frac{13.50}{10} - 66.27 = 23.18 \text{ g H}_2\text{O}$ Agitate.

DV-383 in MeOH = 0.8721 $\times 2 = 10.28 @ 395\lambda$
.1698

DV-300 = 45.15 pH = 8.4 CV = 22.77

Place sample in 60°C oven for 2 weeks. In on 3/13/96

Date March 12 1996

Date March 19 1996

Signature Bruce Suddeth
Witness Rich Sunstall

Hightint Sunbeam Yellow Solids NIPQC.

Purpose: Checked Spec. from 65-70 (Monitor)
to 70 max.

Use Lot # C1150 from plant. This was a very
good batch w/ CV of 16.7.

4 minutes @ 50% power.

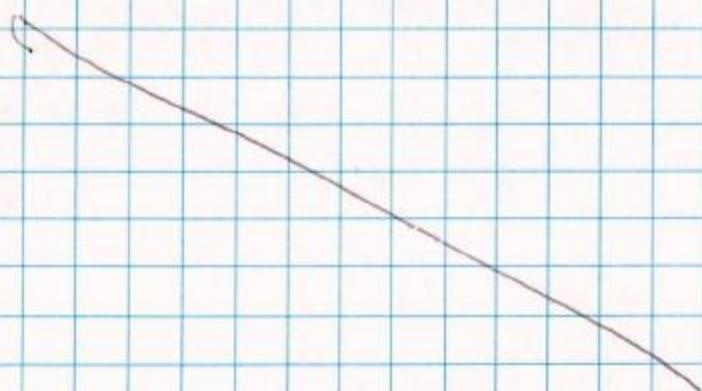
#1 @ 10:00AM	59.40
#2 @ 11:00AM	59.26
#3 @ 11:40AM	59.36
#4 @ 11:55AM	59.36
#5 @ 2:15PM	59.34

Spec = 70 Max.

Average = 59.34

Std. Deviation = 0.0518

Process Tolerance = 0.44%



Date: March 18, 1996
Date: March 19, 1996

Signature: Bruce Sundell
Witness: Vicki Sundell

Replacement for Lt. Pink 10038

Purpose: Colgate - Palmolive called on 3/18/86 and said pink 10038 stained badly after multiple washings.

Blend A

Try Lt. Peach 96 and see how far off the shade is. Add 0.03g Peach 96 (NIP&C sample) to 99.95g Fablosa base. Color is hard to stir in. Allow to fully disperse. While stirring, prepare a blend of AR52 + Yellow LP.

Blend A is too Peachy (too yellow). The color also would not go into solution well.

Blend B - Acid Red 52 @ 5.56 Alsoft lot #9967-59 and Yellow LP lot #M-1057.

1) AR52 @ 85% / Yellow LP @ 15% (4.25g / 1.75g)

Add 0.08g to 100g base. Color went in readily. Shade is too yellow. Need to go up on the red.

Blend C - Acid Red 52 @ 5.56 Alsoft lot #9967-59 and Yellow LP lot #M-1057.

1) AR52 @ 95% / Yellow LP @ 5% (4.75g / 0.25g)

Agitate 100g softene. base. Agitate. Add color slowly with micro dropper. Color appears to be slow to go into solution.

Blend D - Acid Red 52 @ 5.56 Alsoft lot #9967-59 and Yellow LP lot #M-1057.

1) AR52 @ 90% / Yellow LP @ 10% (4.5g / 0.5g)

Add 0.04g of blend to 100g base. Agitate well. Continue on page 49.

Date: March 19, 1986

Date: March 19, 1986

Signature: Bruce Suddith

Witness: Vicki Swanson

Replacement for Lt. Pink 10038.
Continued from page 48.

Also make a 1% solution of this blend in H₂O.
Add to 100g stirring softener. Color is too red.

Using Blend B make a 1% solution in H₂O.
Weigh 100g softener and agitate. Added 3.87g
of color solution to base. Color is too yellow.
Same for testing.

Using Blend D make a 1% solution in H₂O.
Weigh 100g softener and agitate. Add 3.18g
of color solution to base. Color is too pink.
Color need to be between Blend B + D.

Blend E - Blend 87% Acid Red 52 @ 5.56 Alcoh and
13% Yellow LP. Prepare a 1% solution of the 4.35g / 0.65g
blend in H₂O. Weigh 100g softener and agitate.
Added 4.84g of 1% solution. Color is still to yellow
compared to control sample. We will use this for
stain tests.

Staining Test.

- Reference from Colgate,
- Pink 10038 Cat # 10038-38A @ 0.03%
- Blend E at .04% (87% Acid Red 52 @ 5.56 Alcoh / 13% Yellow LP.)

4:00 - Soak Terry Cloth & Mittfitter in each base.
Allow to soak 15 minutes.

4:20 - Remove excess and hang on clothesline.
Allow to dry overnight.

3/20/96 Wash fabric samples in beaker of cold water,
Rinse several times to remove softener.
Continue on page 50.

Date March 19, 1996

Date March 18, 1996

Signature Bruce Sundell

Witness Karen Sunstall

Replacement for Lt. Pink 10038.

Continued from page 79,

3:00 - Have recorded 4 types of fresh water.

4:00 - Hang samples on clothesline and dry overnight.

3/21/96 - Apply double stick tape to samples and cut out.
Showed display of Untinted Fabrics, Pink Reference from
Colgate, Lt. Pink 10038, and Blend E of Acid Red 52.

The Pink Reference had the least amount of staining,
followed by Pink 10038. Blend E of Acid Red 52
had the very worst staining of all samples.

Dated March 20, 1996
Date April 4, 1996

Signature

Witness

Bruce Suddeth
Rich Sunstall

Lt. Patent Blue DOE Absorptivity

Objective: Reduce common cause variation with spectrophotometer. Talked w/ Hal Rice and he designed an absorb experiment with a TPA. Will use Patent Blue.

<u>Factors</u>	<u>1 (+)</u>	<u>2 (-)</u>
A) Absorbance	1.5	0.80
B) Cell Type	Quartz Clear	Quartz Black
C) Cap on Cell	Yes	No
D) Scan Speed	1200	600
E) Avg. # of scans	3	1

of factors in design = 5

of observations in design = 16

Assume a point other than λ for shade ratio.

Response Variable = Shade Ratio + Absorptivity.

Patent Blue

Lot # A1451

Continues on page 52.

Date March 27, 1996

Date April 4, 1996

Signature Bruce Suddeth

Witness Dutch Sundall

It, Patent Blue DOE Absorbance
Continued from page 51.

Layout of Design

	A	B	C	D	E	Absorb Resp. 1	Shade Ratio Resp. 2
1)	1	1	1	1	1	35.84	0.1216
2)	2	2	2	2	1	36.92	0.1204
3)	2	1	1	1	2	36.82	0.1207
4)	1	1	1	2	2	36.13	0.1230
5)	2	2	1	1	1	36.98	0.1216
6)	1	1	2	2	1	35.93	0.1212
7)	2	1	2	2	2	37.22	0.1196
8)	1	2	1	1	2	36.27	0.1202
9)	1	1	2	1	2	36.14	0.1207
10)	1	2	2	2	2	36.07	0.1200
11)	1	2	1	2	1	35.77	0.1209
12)	2	1	1	2	1	37.22	0.1196
13)	1	2	2	1	1	35.80	0.1206
14)	2	2	1	2	2	37.11	0.1208
15)	2	1	2	1	1	36.97	0.1204
16)	2	2	2	1	2	37.033	0.1194

Continue on page 53.

Date March 27, 1996

Date April 4, 1996

Signature

Bruce Sneed

Witness

Ditch Sunstall

Lt. Patent Blue DDE Absorptivity
Continued from page 52.

	A	B	C	D	E	Resp. 1(Absorb)	Resp. 2(Shade Ratio)
1)	1	1	1	1	1	36.49	0.1216
2)	2	2	2	2	1	36.31	0.1224
3)	2	1	1	1	2	36.52	0.1194
4)	1	1	1	2	2	36.80	0.1213
5)	2	2	1	1	1	36.17	0.1203
6)	1	1	2	2	1	36.57	0.1220
7)	2	1	2	2	2	36.48	0.1200
8)	1	2	1	1	2	36.72	0.1213
9)	1	1	2	1	2	36.83	0.1211
10)	1	2	2	2	2	36.75	0.1210
11)	1	2	1	2	1	36.56	0.1221
12)	2	1	1	2	1	36.28	0.1200
13)	1	2	2	1	1	36.55	0.1215
14)	2	2	1	2	2	36.10	0.1163
15)	2	1	2	1	1	36.28	0.1208
16)	2	2	2	1	2	36.36	0.1148

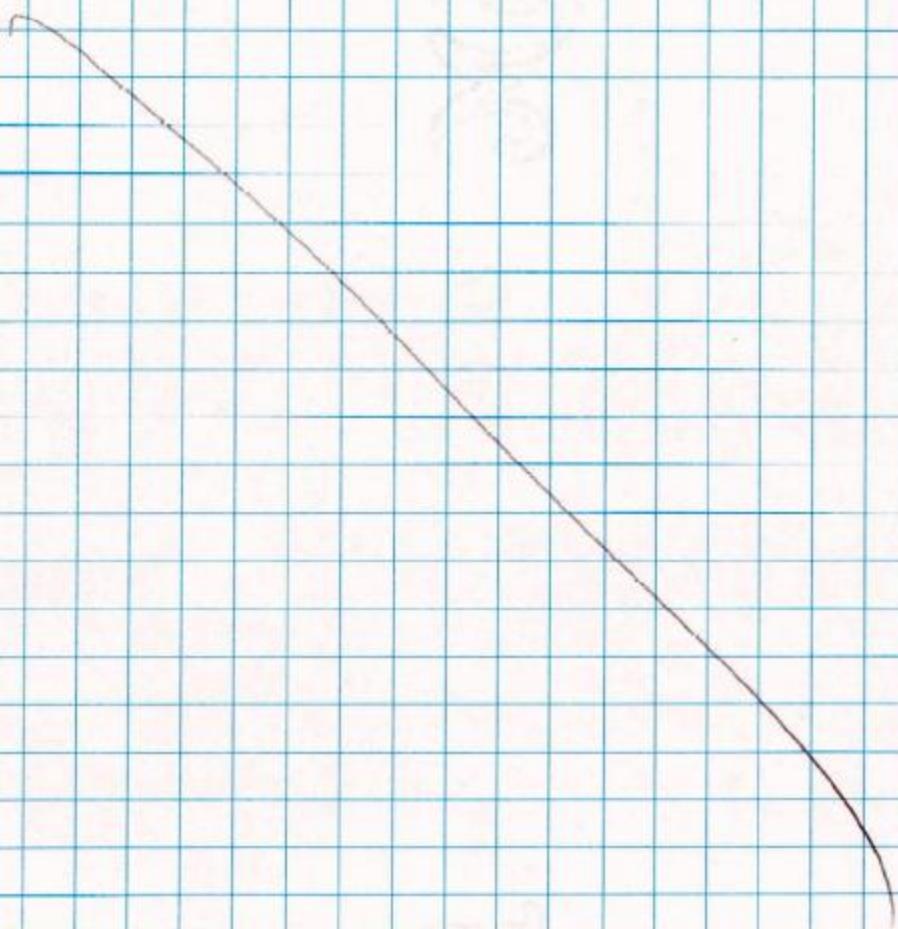
Continued on page 54.

Date: March 27, 1996
Date: April 4, 1996

Signature: Bruce Andleth
Witness: Rich Sunstall

Ht. Patent Blue DOE Absorptivity

Spoke w/ Hal Rice and will send data to him
to analyze. Appears so far that the
sample weight has a lot to do with the
absorb strength.
Send data on 3/28/96.



Date March 28/96

Date April 4, 1996

Signature Bruce Suddeth
Witness Rich Sandall

GPC Analysis of Texatint Colorants.

Purpose: A competitor has 3 colors similar to Versatints. Topaz, Garnet, and Sapphire.

Instrument Set-up. 0.5 ml/min THF, 15 ml/min sparge, RI Detector Sampling Rate of 1, Filter Time of 1, Sensitivity of 4 Auto-zero at Run start, Positive Blank, use an Styragel HR-3 column, use Poly(ethylene Oxide) standards. Weigh 0.50g each in THF. Use 300, 900, 1450, 3350, 4500, 8000, + 21000 mw standards.

Weigh 0.50g sample based on 100% activity. Inject 100ul of each sample. Run time of 40 minutes.

All runs had a negative peak at ~ 21.7 minutes. The low mw (300) samples injected had the tail of the peak incorporated into the negative peak.

Topaz -	RT = 13.887	MW = 8036
Garnet -	RT = 12.500	MW = 18712
Sapphire -	RT = 14.667	MW = 5262
	RT = 15.533	MW = 3498

1st Peak
2nd Peak.

300 mw	RT = 19.917
900 mw	RT = 18.150
1450 mw	RT = 17.300
3350 mw	RT = 15.683
8000 mw	RT = 13.850
21000 mw	RT = 12.483

Allow computer to process & report data of GPC-Polymer method.

Date April 1, 1996
Date April 14, 1996

Signature Bruce Siddle
Witness Finch Sunstall

Synthesis of Brighter Version of Vt. Yellow II.

Purpose: Make a yellow with higher absorb
for Michael Hunt. $\text{HgC} \left(\text{C}_6\text{H}_5\text{CO} \right)_2 \text{N}(\text{CH}_2\text{CH}_2\text{O})_5\text{NH}_2$

Item	Lot #	mw	wt	Actual wt	Moles
Water #1	DI	18	150	150	
p-Aminobenzoic Acid D2218	137	9.59	9.6	0.070	
Miniatia Acid (B12) K3201	36.46	165.03	16.55	0.1407	
Water #2	DI	18	15.21	15.2	
NaNO ₂	A5586	69	5.07	5.1	0.0735
Aniline 10	A1020	533	37.31		0.070
Sulfamic Acid Plant 89.5% 97	47	as needed	1.0g		
Constar, 50%		40	as needed		

10:15 - Charge water #1, PABA, to a 400ml beaker.
Agitate. Off white opaque color.

10:25 - 25°C Charge Miniatia slowly. Color went to tan clear. Look like it dissolved.

pH = 0.60

10:30 - Begin cooling to 0°C.

10:45 - 20°C Begin making NaNO₂ + H₂O^{1/2} solution.

10:50 - 6°C

11:00 - 2°C

11:02 - 0°C Start NaNO₂ solution add slowly.
Keep temp at 0°C.

11:35 1°C Complete NaNO₂ add. Check nitrite w/ KI paper. Positive nitrite. pH = 1.50
Begin to hold 1 hour at 0°C.

12:00 - 0°C Excess nitrite.

12:45 - 1°C Added 1g Sulfamic. Allow to stir ~10 minutes and recheck excess.

12:55 - 3°C No excess nitrite. Amps has a bright yellow shade. Had some foaming during killing of excess nitrite.

Continue on page 58.

Date April 1, 1996

Signature Bruce Fredrik

Date April 4, 1996

Witness Rich Judd

Synthesis of Lt. Version of Sintra Red.

Purpose: Make a Sinra Red with higher absorb for Michael Hunt.

R/M	Lot #	MW	WT	Actwt	Moles
2.5 DMA 1050	9106-47	593	118.6	118.6	0.200
Water	DI	18	79	79	
4,4'-Diaminodiphenylsulfone	23490	248	24.8	24.8	0.100
Muriatic(31%)	A3853	36.46	To Alkox	25.8	
NaNO ₂ (25%)	A5582	69	13.8	13.8 + 41.4 H ₂ O	0.200
Muriatic(31%)	A3853	36.46	To Maintain 18.0	18.0	H ₂ O

12:30 - To a 400mL beaker, charge 2.5 DMA 10, H₂O, and 4,4'-Diaminodiphenylsulfone. Agitate until in solution. Prepare NaNO₂ solution - 13.8g NaNO₂ + 41.4g H₂O.

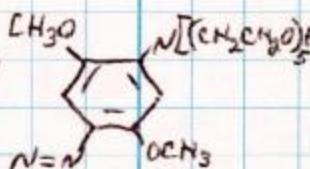
12:45 - Adjust pH to 0.8-1.3 w/ Muriatic. Initial pH = 5.7. Added 25.8g Muriatic. pH = 1.0

12:55 - 38°C Allow to cool down to RT before adding nitrite solution.

1:10 - 35°C Begin adding NaNO₂ solution. Keep pH in 0.8-1.3 range w/ Ammonic Acid.

Add NaNO₂ solution slowly. Keep temp < 40°C.

1:18 - 40°C

1:45 - 35°C Continue adding nitrite sln.  Keep pH at 0.8-1.3.

2:50 - 38°C Complete add. Continue to stir overnight to complete coupling.

4/2/96 10:30 - Measure Solids DV-300 and

Alcohol DV-383. DV-300 = 48.48

DV-383 in H₂O = $\frac{2.0873}{.1516} = 13.77$ @ 503λ
CV = 28.40

4/2/96 Give sample to Michael Hunt for analysis.

Date April 1, 1996

Date April 18, 1996

Signature Bruce Suddett

Witness Ruth Dunstall

Synthesis of ht. Version of Vt. Yellow II
 Continued from page 56.

22:00 - Started coupling Antine 10 into diazo.
 Turned Red immediately. Temp. @ 0°C.

1-DB - 0°C complete add. Allow to just stir
 as ice bath melts.

4:50 - 10°C Allow Rx to stir overnight

4/2/96 - 10:00 Check Solids DV-300 and Absorb DV-18.
 $DV-300 = 22.60\%$

$$DV-383 = \frac{1.7325}{1.1984} = 8.73 @ 464\lambda \quad CV = 38.64$$

4/2/96 Give sample to Michael Hunt for analysis.

Date April 1, 1996
 Date April 4, 1996

Signature Ronald Judd III
 Witness Robert W. Hall

Paired Ion Chromatography for Reactive Colors.

Purpose: Run PICA reagent for John Brinkley
to determine whether the azo molecule is
attached to the polymer.

Prepare the mobile phase - Mix Acetonitrile (HPLC
solvent) and Water (HPLC Grade) 50/50. Stir
in 1L graduate. Add 1 ampule of PICA Reagent (0.005M
(tetra butyl ammonium hydrogen sulfate). Allow
to stir ~10 minutes. Filter through a 0.45μm
filter (Nylon). Add to a 1L solvent reservoir.
Sparge w/ 50 ml/min helium.

Attach the C₁₈ column labeled for PICA Reagent
only.

Start w/ flow rate at 0.50 ml/min. Use the
PDA detector at 200 - 700 nm. Sparge at 10ml/min.
Use 600nm as where data is taken.

Tony Smith weighed w/ 3 samples: A) Reactive Blue 19,
B) Hydrolyzed Reactive Blue 19, C) Blue 19 Amine Reaction.
All samples were made up in the mobile phase
in 100ml volumetric.

Run samples for 1 hour.

Reactive Blue 19	1) Peak @ 13.138	2.0 AU
	2) Peak @ 19.872	0.5 AU

Reactive Blue 19 Hydrolyzed	1) Peak @ 14.382	0.005 AU
	2) Peak @ 20.232	0.060 AU

Blue 19 Amine Rev	1) Peak @ 9.735	0.40 AU
	2) Peak @ 10.302	0.40 AU
	3) Peak @ 14.302	0.05 AU
	4) Peak @ 20.302	0.05 AU
	5) Peak @ 21.952	0.05 AU

Continued on page 60.

Date April 2, 1996

Date April 7, 1996

Signature

Bruce Suddeth

Witness

Dick Dillotall

Paired-Tom Chromatography for Friday Colors.
 Continued from page 59.

4/4/96 Need to rerun the samples with multiple injections. Also run a sample of the Amine HED made by John Brumke. Turn pump weighed sample to mm. Cut run times back to 30 minutes. Shoot 50ul of each product, and have 2 injections each.

These injections are very similar to the 3 previous injections. We need to use a blend of Hydrolyzed Blue 19 and Blue 19 Amine Rx.

After looking at the chromatograms it appears that the product peak of John's colorant has two peaks. The product peak is split. Suspect having 2 Amines.

Used a blend of PGA + Reactive Blue 19. This chromatogram had only one peak for the product.

Need to investigate further. Talked w/ John about using a Rx made w/ Jeffamine to see if it has 1 or 2 product peaks. Shot a sample and the Jeffamine stayed on the column longer than the 30 minute run time. Stop set and reset run time to 60 minutes.

The Chromatograms only had 1 Amine peak indicate the Jeffamine replacement.

Date April 4, 1996
 Date April 4, 1996

Signature Bruce Bellith
 Witness Ditch Dumont

10038-61

Sample Request to Loveland Industries

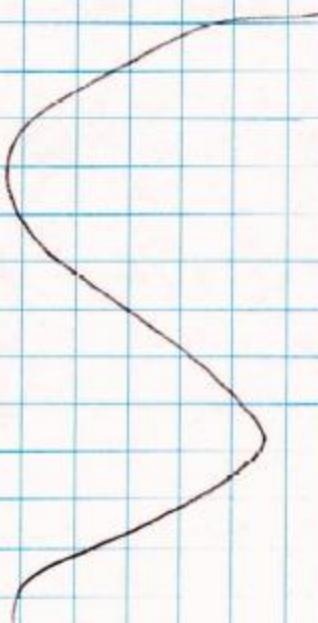
Purpose: Miller McClellan would like to
ship 5 gallons of Bullseye.

Customer: Loveland Industries
14520 WRC #64
Greeley, CO 80634
Attn: M. Atkinson.

Label as: Experimental Blue 9805-22
Lot # 10038-61

Sample came in from Pifco Plant labeled as:
Bullseye SAI
Lot # D1690

Ship sample on 4/4/96 by UPS.



Date April 4, 1996
Date April 4, 1996

Signature Bruce Laddith
Witness Girth J. Novotall

Ht. Patent Blue Absorptivity DOE

Repeat of 10038-51.
use Lot # A1451

$$\text{Sample weight } \begin{cases} 1 \\ 2 \end{cases} = \begin{cases} 0.3929 \\ 0.2221 \end{cases}$$

From Dr-516 with Methanol

Run the same parameters as the first series.

Calculate a shade ratio by dividing absorbance at 409 nm by absorbance at 628 nm.

Run the first series before lunch.

Using the same dilutions repeat the analysis after lunch.

#	<u>Alkohol #1A</u>	<u>Resp #2A</u>	<u>Alkohol #1B</u>	<u>Resp #2B</u>
1	35.61	0.0934	35.46	0.0955
2	35.38	0.0762	35.47	0.0793
3	35.71	0.0730	35.77	0.0788
4	35.83	0.0853	35.83	0.0880
5	35.15	0.0796	35.51	0.0814
6	35.65	0.0960	35.58	0.0979
7	35.84	0.0774	35.77	0.0759
8	35.97	0.0955	35.86	0.0954
*9	35.92	0.0920	34.34	0.0991
10	36.05	0.0957	35.83	0.0950
11	35.62	0.0862	35.48	0.0880
12	35.51	0.0771	35.51	0.0780
13	35.57	0.0967	35.44	0.0970
14	35.65	0.0714	35.40	0.0763
15	35.64	0.0803	35.26	0.0807
16	35.80	0.0762	35.60	0.0735

Date: April 8, 1996
Date: April 16, 1996

Signature: Bonje H. Pendlett
Witness: Robert M. Taff

Color Match for Pink Fabuloso

Talked w/ Jim Spry about the Pink 10038 in Fabuloso base. Colgate says the color stain too bad. Need to try to match w/ Supra Red if possible.

Prepare a 1% solution of Supra Red lot # X1087.

Prepare a 1% solution of Sunbeam Yellow lot # C1150.

(Blend A)

Charge 100g of Fabuloso base to a 250ml beaker.

Added 14.7g of Supra Red 1% and SBY 0.43g of SBY 1%. Color looked too dark.

Added 22.3g of Fabuloso base. Color is closer, but a little too Orange.

Total of 122.3g base. (0.147g Supra Red / 0.0043g SBY @ 100%)

Blend B

Charge 100g of Fabuloso base to a 250ml beaker.

use Supra Red at a straight concentration.

Blend 9.8g of Supra Red and 0.2g SBY. Shake well.

Use a micro pipet to add color to stirring base. Add slowly to insure color is fully dissolved.

Added 0.11g of color blend to base.

Shade is too pink. Need to increase the yellow.

Blend C

Charge 100g of Fabuloso base to a 250ml beaker.

Blend 9.7g of Supra Red and 0.3g SBY. Shake well.

Begin to stir base in stir plate. Add color dropwise and slowly. Added 0.14g of color to base.

Color is close but a little too dark. This blend is close enough to run a stain test.

Continue on page 64.

Date April 16, 1996

Date May 15, 1996

Signature Bruce Suddith

Witness Linda Marshall

Color Match for Pink Fabrioso.
Continued from page 63.

Blend D

For this blend I will use a Red similar to Supra Red except use 2.5 DMA 10FD. Sample of red labeled as E70 10038-57. Absorb = 13.8 @ 503λ. Also use Sunbeam Yellow lot # X1087. Use the same ratio of Red to Yellow as Blend C except correct for absorb.
 $\text{Red} = \frac{0.97 \times 0.60}{13.8} = 0.0422 \text{ g/100g}$ ~~4.22%~~ 4.22g

$$\text{SBY} = \frac{0.03 \times 10}{10} = 0.03$$

$$\underline{\underline{3.08}}$$

$$\underline{\underline{7.22}}$$

$$\text{Red} = 4.22 \div 7.22 \times 100 = 58.45\%$$

$$\text{SBY} = 3.0 \div 7.22 \times 100 = 41.55\%$$

This is the blend if colorants are straight.

Diluted w/ H₂O to 10 to achieve same strength as blend C.

Red	4.22g
SBY	3.08g
Water	2.78g

Stir well.

Charge 100g of base to a 250 ml beaker. Agitate. Add blend dropwise and slowly. Added 0.05g of blend. Shade is way too orange. The calculated dilution did not achieve the right shade.

Need to reblend the Red and Yellow with different ratios.

Continued on page 65.

Date April 16, 1996

Signature

Bruce Suddeth

Date April 19, 1996

Witness

Linda Jurstall

Color Match for Pink Fabuloso
Continued from page 64.

Blend E

Prepare a blend with the same R/M as blend D.
Use different ratios of red.

Red	6.0 g
SBY	3.0 g
Water	1.0 g

Stir well.

Charge 100g of base to a 250 ml beaker. Agitate.

Add blend E dropwise and slowly.

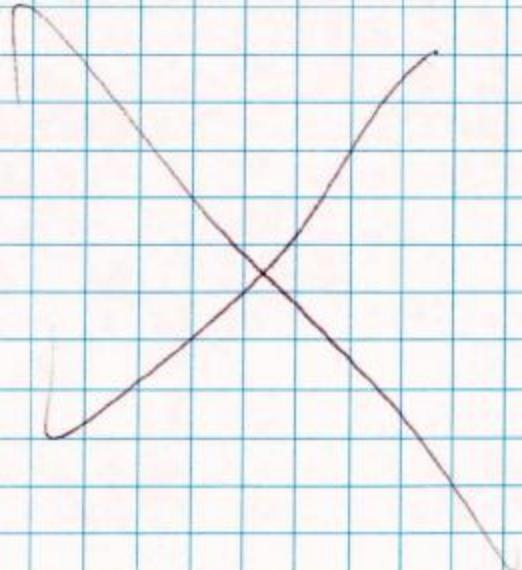
Added 0.03 g of Blend E to base. Color appears
to be too yellow, but will continue with this
sample for stain testing.

Blend F

Need to also use Orange X96 to the Fabuloso base.

Use lot # 9968-53 Orange X96 Uncut.

Added 0.01 g to 100g Fabuloso base. Use this
sample for stain testing.



Date April 16, 1996

Date April 19, 1996

Signature

Bruce Laddeth

Witness

Dickie Sundall

Stain Testing for Fabuloso Pink.

Purpose: Compare staining on Terry cloth and Multifiber with following colors.

Cut out swatches and soak in softener w/color for 15 minutes. Hang on line and allow to dry overnight.

- 1) Pink Reference
- 2) Lt. Pink 10038
- 3) Blend C of Supra Red / SBY #10038-63
- 4) Blend E of Supra Red 2.5 DMAID / SBY #10038-65
- 5) Blend F of Orange X96 #10038-65.

4/18/96 Angel Lee removed samples from drying rack. Washed w/ water and allowed to dry.

Get out samples and place on display.

- 1) Pink Reference - Appears all color washed out. The fibers have yellowed though.
- 2) Pink 10038 - Stained worse than reference. The cotton is worse.
- 3) Supra Red / SBY - Cotton looked very good. All other fiber samples were good also.
- 4) Supra Red w/ 2.5 DMAID / SBY - Surprisingly the cotton stained worse than the regular Supra Red.
- 5) Orange X96 - Cotton stained very badly.

Will proceed by testing pH stability of blend of Supra Red / SBY.

4/23/96 To a 400ml beaker, charge 200g of fabuloso base. Agitate. Using Blend C of Supra Red / SBY from page 10038-63 add 0.25g.

Continue on page 70.

Date April 17, 1996

Date April 19, 1996

Signature Bruce Indleth

Witness Linda Unstall

10038-67

Sample of Cotton Red (Exp Red 10038-67) to University of CA.

Ross Davis came by lab and wanted some
Cotton (into colors). The only one I have made
is Red.

This Red was made in Notebook # 9183-73.
End to a 0.60 Absorb.

Label sample as Exp. Red 10038-67 lot# 10038-67.
Ship 4oz of material.

Address: Lawrence Livermore National Laboratory
Physical Chemistry, Electrochemical Engineering
Department of Chemistry and Materials Science
PO Box 808, L-3690
University of California
Livermore, CA 94551
Attn.: Dr. John F. Cooper

Send R+D letter w/ sample.

Lee said it was OK to send Revision 6.0
of NIP.

Date April 19, 1996
Date April 19, 1996

Signature Bruce Bellotti
Witness Linda Dunstall

Sample Request #96-72

Customer: Pioneer Plastics Corp.

One Piomonte Road.

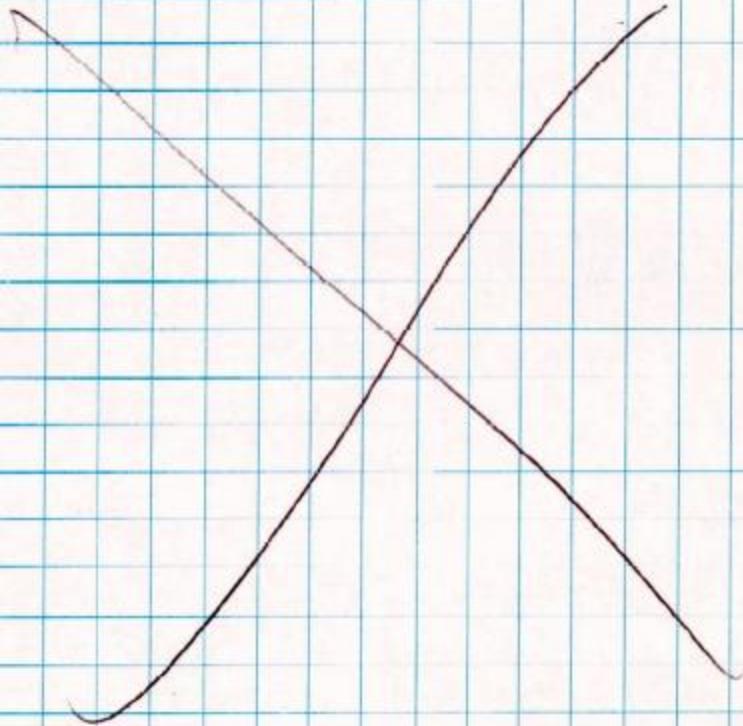
Watson, ME 04211

Attn: Paul Marshall

Phone: (207) 784-9111

Send Reactint Black X77 as Experimental 10038-68
Request 10 gallons to be sent from Cypress.

John Brumke is generating draft MSDS's.



Date April 22, 1996
Date April 23, 1996

Signature

Bruce Suddeth

Witness

Linda Sunstall

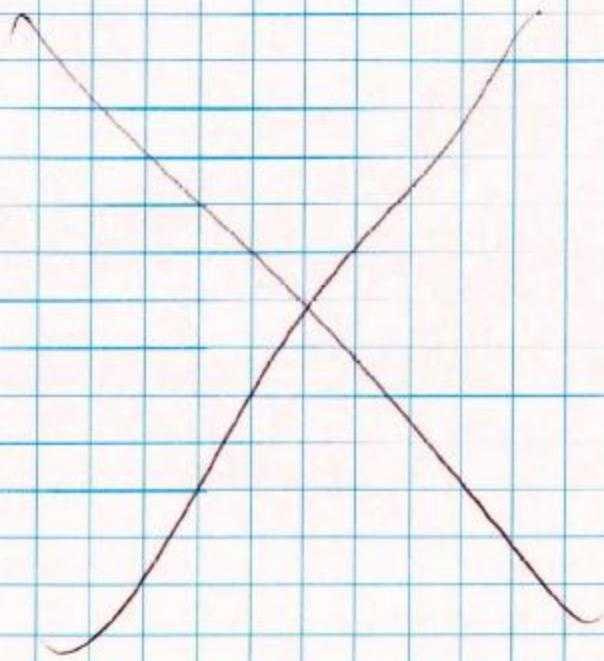
Sample Request # 96-72

Customer: Pioneer Plastics Corp
One Priorite Road
Auburn, ME 04211
Attn: Paul Marshall

Phone: (207) 784-9111

Send Reactant Block X95 as Experimental 10038-69.
Request 10 gal to be sent from Cypress.

John Brinkley is generating draft MSDS's.



Date April 22, 1996
Date April 23, 1996

Signature Bruce Andlett
Witness Linda Marshall

pH Stability of Supra Red / Sunbeam Yellow in Fabriles.
Continued from page 66.

Using the softener blend made on page 66,
prepare to check color change w/ pH adjustment.

Prepare 50% solution of Caustic (50%) Lot # A3932

10g Caustic / 10g H₂O Stir + cool.

Prepare 50% solution of Muriatic Lot # A3853

10g Muriatic / 10g H₂O Stir + cool.

Add 100g of base softener blend to 250 ml beaker,
agitate.

Initial pH = 2.4

Add 50% Muriatic to sample. Added 0.30g, pH=2.0
Pour into vial.

Add 100g of softener blend to 250ml beaker. Agitate.

Initial pH = 2.4

Add 50% of 50% Caustic to sample. Added 0.2g Caustic.
pH = 3.4

The color changed and went pale ~pH of 3.

Color change is noticeable but I do not know
if it is significant. I'll have Jim try look
at sample and see what he thinks.

Add 0.07% of Supra Red to Fabriles base. Stir well.

Place vials containing Pink Reference, Pink 10038, Supra Red/
Sunbeam Yellow, and Supra Red in a 40°C oven
on 5/14/96. Allow to stand 1-2 weeks. Observe for
shade change compared to RT.

5/14/96 - The Pink Reference has turned slightly orange. The other
3 look to have no change.

5/20/96 - Pink Reference not changed from 5/14. Pink 10038 is stable,
Supra Red faded orange & 2 weeks. Supra Red/Sunbeam faded
real orange. Color is not heat stable.

Date April 23, 1996

Date April 23, 1996

Signature

Bruce Sandford

Witness

Litch Sunstall

Synthesizing Rxn of Aniline PEG 30 and Aniline 10.

Purpose: Need to synthesize a Sunbeam Yellow replacement by diazotizing Aniline PEG 30 and coupling to Aniline 10.

R/M	Lot#	MW	wt	Act. wt	Moles	Concn%
A PEG 30	S1065	1429	141.90	175.90	0.0993	0.1225
NaNO ₂	C1405	69	9.40	4.72	0.1045	0.1362
Muriatic (HCl)	A3853	36.46	28.77	14.15	0.1986	0.2406
H ₂ O (NaNO ₂)	DI	18	18.75	35.00		2.083

8:35 - Charge A PEG 30 to a 1L 3 neck. Agitate. Begin cooling to 0-5°C. While cooling add Muriatic.

8:45 - 20°C pH = 0.90 Continue to cool. Prepare NaNO₂ / H₂O solution.

9:00 - 0°C Start NaNO₂ / H₂O solution dropwise. Keep temp < 5°C.

9:30 - 1°C Complete add. Rx does have excess nitrite. Hold 2 hrs at 0-5°C.

10:35 - 0°C Has excess nitrite.

11:30 - 0°C Has excess. Add 1/3 of Sulfonic Acid. Diazo began to form a little.

11:55 - 2°C No excess nitrite. Some foam still.

Coupling

R/M	Lot#	MW	wt	Act. wt	Moles
Aniline 10	A1020	531	32.58	65.15	0.1211
H ₂ O		18	32.58	65.15	

11:25 - Charge Aniline 10 + H₂O to a 400ml beaker. Agitate. Begin cooling to 0-5°C.

11:55 - 0°C pH = 6.25 Begin coupling slowly. Keep temp < 10°C.

12:20 - 2°C Complete coupling. Keep cold and allow to stir.

3:00 - 5% solids = 44.58 DV-300.

Absolute DV-18 = $\frac{0.0881}{.5721} = 0.1540 \text{ @ } 45.2 \text{ L}$.

Date April 24, 1996

Date April 26, 1996

Signature Bruce Juddith

Witness Ruth Dunstall

Synthesis of Yellow by Palmer Orange process.

Purpose: Prepare a yellow by the Palmer Orange process. This is to replace Dumbourn Yellow.

R/M	Lot #	mw	wt	Act wt	Moles
H ₂ O #1	DI	18		63.4	
Diethandamine	02114MZ	105.14		54.5	0.5184
Na Acetate	23789	82.03		21.6	0.2633
PABSCL	Q1617	233.67		60.8	0.2602
H ₂ O #2	DI	18		3.3	
Muriatic #1(31%)	A3853	36.46	18.45	53.5	0.5059
H ₂ O #3	DI	18		75.1	
Muriatic #2(31%)	A3853	36.46	14.07	45.4	0.3860
H ₂ O #4	DI	18		56.7	
NaNO ₂	A5586	69		18.0	0.2609
H ₂ O #5	DI	18		44.2	

4:00 - Charge H₂O #1, Diethandamine. Begin agitation.

4:20 - Charge Na Acetate.

4/25/96 8:00-25°C Charge PABSCL slowly. Keep temp <50°C.

8:10 - 50°C Complete add. Charge H₂O #2.

Begin heating Rx to 70°C.

9:00 - 50°C Rxn set-up. Continue to heat and melt.

9:30 - 70°C Rxn beginning to melt well and stir.

9:50 - 82°C

10:00 - 90°C Hold for 2 hours.

11:00 - 95°C

12:00 - 93°C Prepare Muriatic #1 and H₂O #3 solution.

12:00 - 92°C Charged Acid/H₂O solution.

12:10 - 70°C Release Rx to 90-95°C

1:00 - 90°C Hold for 2.5 hrs @ 90-95°C.

2:40 - 94°C Cut heat and allow to cool.

4/26/96 8:00 - Begin cooling to 0-5°C. While

cooling add Muriatic #2. Continue cooling

Container on page 73.

Date April 24, 1996

Date April 20, 1996

Signature Bruce Suddeth

Witness Rich O'Neill

Synthesis of Yellow by Palmer Orange Process.
Continued from page 72.

Propane $\text{NaNO}_2 / \text{H}_2\text{O}^{10}$ solution. Dissolve fully.
8:30 - 2°C Begin $\text{NaNO}_2 / \text{H}_2\text{O}^{10}$ addition slowly.
Keep Temp at 0-5°C.

9:30 - 0°C Complete add. Hold for 2 hours at 0-5°C.
Check Rx for excess nitrite.
Positive nitrite.

11:05 - 0°C

12:00 - 0°C Excess. Add 1g Sulfanilic. Agitate. Excess
12:08, 0°C Add 1.5g Sulfanilic. Agitate.
2:15 - 4°C Slight excess. Continue w/ coupling.

Coupling

RIN#	Lot #	ml	wt	Actual	Moles
Pipadone	R1786	323		61.44	0.1902
NaO	DI	18		122.88	
Versene 100XR	V2577	380		35.0,	
Caustic (50%)	A3932			25.5	

Charge Pipadone, H_2O to 1L beaker. Agitate. Add Versene.
 $\text{pH} = 3.7$ Adjust pH to 8.5 w/ Caustic. $\text{pH} = 8.5$

12:00 - 32°C Begin to cool to 0-5°C. Went to lunch.

2:15 - 10°C Cool up dry ice.

2:40 - 4°C Begin coupling slowly. Keep temp ~10°C
and $\text{pH} > 4.5$ w/ Versene.

3:00 - 8°C Have charged ~ 3/4 of diags. Added Versene
while coupling. $\text{pH} = 4.9$ Continue to couple.

3:45 - 10°C Complete coupling. Added 223g Versene.
 $\text{pH} = 4.7$ Keep cool and stir overnight.

Next day

checked pH 4.5 - looks like mustard color.

Adjusted pH w/ 50% caustic

At pH 7.0 - started turning red

Adjusted pH to 8.10 w/ 50.36gms. 50% caustic

Date April 26, 1996

Signature Bruce Madellett

Date April 29, 1996

Witness Ruth J. Mota

Synthesis of Yellow by Peltier. Orange Products. cont. from p. 73

Phased in oven 60°C for 2 hr.

Drained off layers - product = layer on bottom
water - ~~bottom~~ layer 230.699g
225.359g

Measure abs. and % sol-

$$\text{SW} = 1.244 \quad 1.7611 - 1.7709 \text{ at } 397.0$$

$$\text{Abs} = 14.16$$

$$\% \text{ sol} = 55.87$$

$$\text{P.V} = 25.34$$

Cut batch to a theoretical absorb of 10.

$$227 \times \frac{14.16}{10} - 227 = 94.43 \text{ g H}_2\text{O}. \text{ Allow to stir well.}$$

Stain test of Acid Yellow #17 (Abs = $\frac{1.5406}{.247}$) = 6.17 @ 400 nm DV-181
Sunbeam Yellow Lot# C1150

Yellow 10038-72 @ 10 Abs. Test a 1% solution based
on absorb with mulfiber and terr cloth.

Allow for fiber samples to soak for 15 minutes.

Squeeze out color and hang to dry. Allowed to
dry 2 hours. Wash w/ cold water @ 3:00 PM. until
there is no evidence of color being extracted.

5:00 - Rinse w/ cold water. Allow to dry on paper
towels overnight.

Fiber	Acid yellow 17	SBY	Exp 10038-72	File	Acid yellow 17	SBY	Exp 10038-72
Acetate	Slight	No	Slight	Otan 75	No	No	No
SEF	No	No	No	Silk	Heavy	Heavy	Heavy
Armed	No	No	No	Polyprop.	No	No	No
Cotton	No	No	No	Viscose	No	Slight	No
Greslan 61	No	No	No	Wool	Heavy	Slight	Slight
Damon 54	No	No	No				
Dacron 64	No	No	No	Terry Cloth	Slight	No	No
Nylon 66	Heavy	Slight	Slight to Heavy				

Date April 30, 1996

Signature

Linda Gunstall

Date May 8, 1996

Witness

Bruce Lindell

10038-75

Experimental Pink 10038-75

This is a blend of 97% Supra Red and 3% Sunbeam Yellow.

Jim is to turn in a Sample Request once the thermal stabilities are completed.

Date May 6, 1996
Date May 8, 1996

Signature Bruce Juddith
Witness Linda Juddith II