

# **LABORATORY NOTEBOOK**



## Outline of Uranine Tests

### I. Temperature

- A. Set three temperatures to evaluate relative peak locations and absorbance values (i.e. 75°F, 95 °F, 120°F) at 16 ppm
  1. Solutions prepared using LTM001, volumetric glassware.
    - a. Time of boiling 0.2% solution to be recorded for each sample.
  2. Temperature of each 16ppm solution will be controlled using a hotplate and thermometer to obtain necessary temperature.
    - a. Temperature of 16 ppm solution to be recorded.

- B. Repeat for two other lots

### II. pH

- A. Evaluate 16 ppm solutions at pH 5, pH 7 (neutral, D.I. water), and pH 9 for relative peak locations and absorbance values.
  1. pH 5 buffer prepared using 0.5L stock solutions of
    - a. 0.1M NaOH (2g:500mL H<sub>2</sub>O)
    - b. 0.1M Potassium Hydrogen Phthalate (10.2g:500mL H<sub>2</sub>O)
    - c. Use 172.18mL Potassium Hydrogen Phthalate: 77.82mL NaOH, 0.1M solutions.
  2. pH 9 buffer prepared using 0.5L stock solutions of
    - a. 50 mL of 0.025 M Borax (4.76 g/500 mL H<sub>2</sub>O) + 4.6 mL 0.1 M HCl (1.53 mL/ 500mL H<sub>2</sub>O using 22° Baume HCl)
    - b. This can be scaled up to use 228.94mL Borax (0.025M) + 21.06 mL HCl (0.1M)

- B. Repeat for two other lots

### III. Salt

- A. Add 0.5%, 5%, and 10% salt to 16 ppm solutions to determine effect on peak location and absorbance values.
- B. Repeat for two other lots

Test

Uranine pH

Obj. note:

Uniform Test  
method topology  
v. concentration

- Observe Luminescence
- Observe pH v. Lumin.
- Observe NaCl v. Lumin
- Observe NaCl v. Shade
- Observe Synthesis  
method (C.S.)

## OUTLINE OF URANINE TEST

### I. TEMPERATURE

A. SET 3 TEMPS, i.e. 120°F, 75°F, 95°F

1. EVALUATE SAMPLE VS. ITSELF AT DIFFERENT  
Temps for ABS. & PEAK LOCATION

AT 16 ppm

2. REPEAT FOR 2 OTHER LOTS

### II. pH

A. CHOOSE 3 BUFFERS: pH 5,  $\frac{\text{H}_2\text{O}}{\text{pH} 7}$ , pH 10

#### a) TEST pH

1. EVALUATE SAMPLE VS. ITSELF AT  
These pH VALUES AT 16 ppm.

2. REPEAT FOR 2 OTHER LOTS

3. TEST ON DILUTIONS of URANINE + SALT

### III. SALT

A. ADD 0.5% SALT, 5%, 10% TO  
DILUTION 16 ppm

1. IF changes, Run Conc. in Between.

2. Repeat for 2 other lots

OBJECTIVE, DETERMINE EFFECTS OF pH, TEMPERATURE  
AND SALT ON SHADE % of URANINE.  
ALSO, DETERMINE OPTIMAL pH TO CONTROL  
SHADE MEASUREMENTS.

**NOTEBOOK NO.** \_\_\_\_\_  
**ISSUED TO** \_\_\_\_\_  
**ON** \_\_\_\_\_ **20** \_\_\_\_\_  
**DEPARTMENT** \_\_\_\_\_  
**RETURNED** \_\_\_\_\_ **20** \_\_\_\_\_

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2. • When starting a page, enter the title, project number, and book number.
  - Use ink for permanence -- avoid pencil.
  - Record your work as you progress, including any spur-of-the-moment ideas which may be developed later.
  - Avoid making notes on loose paper to be recopied.
  - Record your work in such a manner that a co-worker can continue from where you stop. You might be ill and to protect your priority it could be urgent that the work continue while you are absent.
3. • Give a complete account of your experiments and the results, both positive and negative, including your observations.
  - Record all diagrams, layouts, plans, procedures, new ideas, or anything pertinent to your work including the details of any discussions with suppliers, or other people outside the Company.
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4. • After entering your data, sign and date the entries.
  - Explain your work to at least two wit-

nesses who are not co-inventors, and have them sign and date the pages in the place provided.

- Record the names of operators and witnesses present during any demonstration and have at least two witnesses sign the page. If no witnesses are present during an experiment of importance, repeat it in the presence of two witnesses.
- 5. Since computer programs can be patented these instructions apply to the development of computer software. In this case a description of the structure and operation of the program should be recorded in the notebook, together with a basic flow diagram which illustrates the essential features of the program. In the course of developing the code, the number of lines of code written each day should be recorded in the notebook, together with a statement of the portion of the flow diagram to which the section of code is directed.
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**If loss occurs, notify your supervisor immediately, and make a written report describing the circumstances of the loss.**

2/9/07  
1

TITLE BIC Red Ink #1

Project No. \_\_\_\_\_

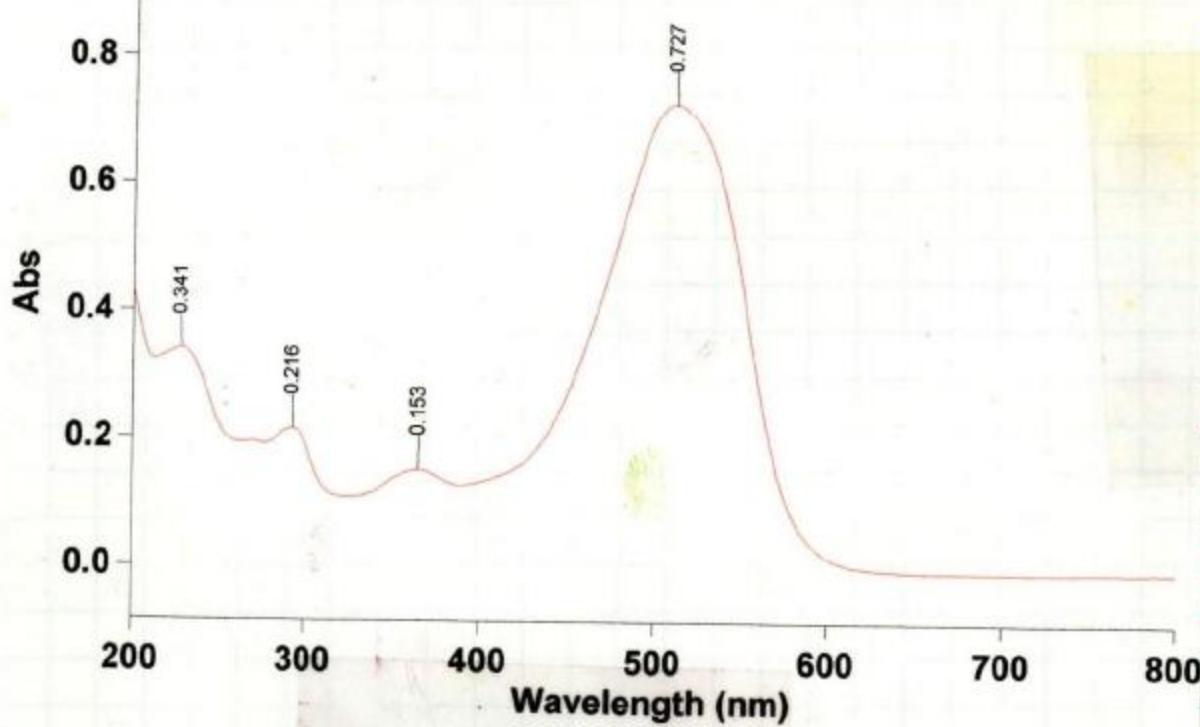
Book No. \_\_\_\_\_

From Page No. \_\_\_\_\_

- Scanned Bic Ink at 40 ppm
- unsuccessful getting an exact match
- relative products - ARY73, AR14, + BR 14
- Did Dips of all including BIC dye
- D.D Acid/Base Test using Sulfuric Acid + Caustic
- D.D TLC Test on all, none separated in different colors
- Lastly I did ■ A Fabric Dyeing test, results showed Acid dye + A touch of Basic dye.

2/13/2007 2:50:37 PM

Page 1 of 3



Witnessed &amp; Understood by me,

Date \_\_\_\_\_

Invented by: \_\_\_\_\_

To Page No. \_\_\_\_\_

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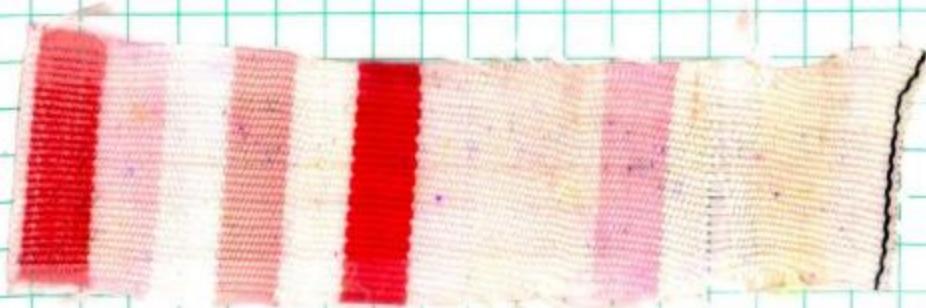
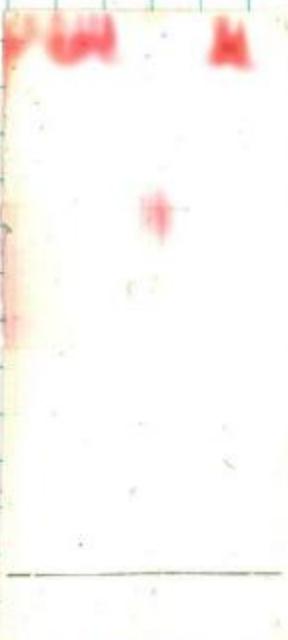
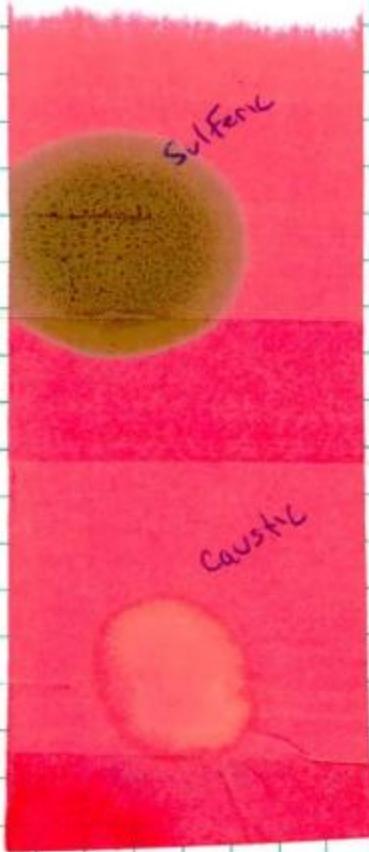
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Project No. \_\_\_\_\_

Book No. \_\_\_\_\_

TITLE \_\_\_\_\_

From Page No. \_\_\_\_\_

Acid  
Red  
73BIC Ink  
Red #1Acid Red  
14

To Page No. \_\_\_\_\_

Witnessed &amp; Understood by me,

Date

Invented by:

Date

Recorded by:

TITLE Blue /green /Pink Candle wax Match

Project No. \_\_\_\_\_  
Book No. \_\_\_\_\_

2/13/07  
3

From Page No. \_\_\_\_\_

I started with green and pink and searched for materials. Each either had a component that was inactive, or the dye itself was inactive. I search TB 43, Oil Dyes + TB 6, candle wax dyes. I then moved on to the blue. I tried a variety of Solvent Blues which failed, along with several Sol Red + Solvent Violets, none seemed to match. All samples sent, including the wax itself, contains optical brightener and fluoresces. I determined that the wax is of yellow color which is throwing off my results. I am uncertain if the sample beads were made with the same yellow wax I am using to match. My next step(s) is to find out if they were indeed used with this same kind of wax? Is there Titanium in any of the samples + wax, and will a white need to be added to my formula to match correctly?

To Page No. \_\_\_\_\_

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Project No. \_\_\_\_\_  
Book No. \_\_\_\_\_

1/17/07

TITLE Anti-freeze PAG Solution

From Page No. \_\_\_\_\_

306 - 036 - 51 - .01g - 100ml PAG

try - 53 Yellow - Zg - 100ml PAG

5g Try - 53 + 1.5g 306 - 036 - 51 = Solution

To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by:	Date
		Recorded by:	

TITLE Mahogany + Chocolate Samples

Project No. \_\_\_\_\_  
Book No. \_\_\_\_\_

From Page No. \_\_\_\_\_

Mahogany

13.5%	806-056-50	8.1g	203T399
34%	806-014-50	20.43g	205B235
20%	606-024-50	12.0g	206A304
3%	106-007-71	1.4g	206B286
15%	206-036-51	0.9g	205A400
28%	999-401-13	16.8g	(Stearic Acid)
100%		60.03g	

Chocolate

10%	106-007-71	206B286
12%	606-026-50	206B382
3%	706-013-50	206C360
51%	806-003-50	205D456
24%	999-401-13	(Stearic Acid)

- Procedure - gathered + weighed materials  
 - divided up 4, 8oz samples (2oz each)  
 - Blended in paint mixer plus 4 ball bearings  
 - Turned powder samples to liquid form using acetone

Results - Mahogany Plyam STD vs Mahogany - P

Strength - 93.42  
DE - 5.95

Chocolate Plyam STD vs Chocolate - P

Strength - 97.27  
DE - 3.75

To Page No. \_\_\_\_\_

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Project No. \_\_\_\_\_  
Book No. \_\_\_\_\_

TITLE Liquid / powder 501-610-50

From Page No. \_\_\_\_\_

Purpose - To compare strength ratio's between components that make up  
501-610-50

concentrations (Liquid)

11.2% 801-073-42  
 38% 801-007-40 }  
 13% 801-023-33 } 30g 1-500 solutions

concentrations (powder)

10% 801-073-52  
 16% 801-007-50 }  
 20% 801-023-71 } 30g 1-500 solutions

501-610-50

1.12g 801-073-42  
 3.80g 801-007-40 } Unknown concentration  
 1.30g 801-023-33 } Brought up to 100mls pr. glycol

To Page No. \_\_\_\_\_

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TITLE green fiber Liquid match

Project No. \_\_\_\_\_

Book No. \_\_\_\_\_

From Page No. \_\_\_\_\_

1g - 500 ml 201-009-00 2061384

1 - 500 ml 801-073-22 20519292

5g - 250ml of each solution

85% 801-073-22 40 ppm Solution

15% 201-009-00 40 ppm Solution

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Project No. \_\_\_\_\_  
Book No. \_\_\_\_\_

TITLE Keyacid Blue AFB + Keyamine Yellow Myp

From Page No. \_\_\_\_\_

Blue AFB

25'l. 201-009-50

35'l. 201-017-60

40'l. 999-400-98 natural Salt

Yellow MYP

65'l. 805-011-62

3'l. 605-081-60

33'l. 999-400-98 natural Salt ) 250mL

To Page No. \_\_\_\_\_

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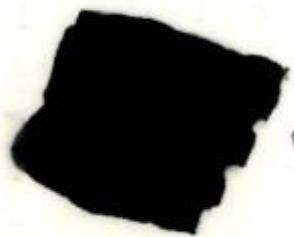
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3



1



2



A



3



A



2-10



B

TITLE Caustic Turquoise (NaOH vs Acetic Acid) Project No. \_\_\_\_\_  
 From Page No. \_\_\_\_\_ Book No. \_\_\_\_\_

- Start - NaOH 15%. (37.5g - 250ml) + to 10% Acetic Acid, then diluted to 1%.  
 - This raised the pH higher than the caustic solution itself (used a total of 100ml)
- NaOH 15% to 10% Acetic Acid, not diluted any further  
 - This slightly will and did raise the pH of the 1% a bit higher to about 13.10 (used a total of 100ml)
- NaOH 15% to Strait glacial Acetic Acid, added 30 ml which slightly brought down the pH level to 12.85 I then added an additional 20-25ml of the glacial which then drastically dropped the pH level to a neutralizing level of 7.10. (total 50-55ml)

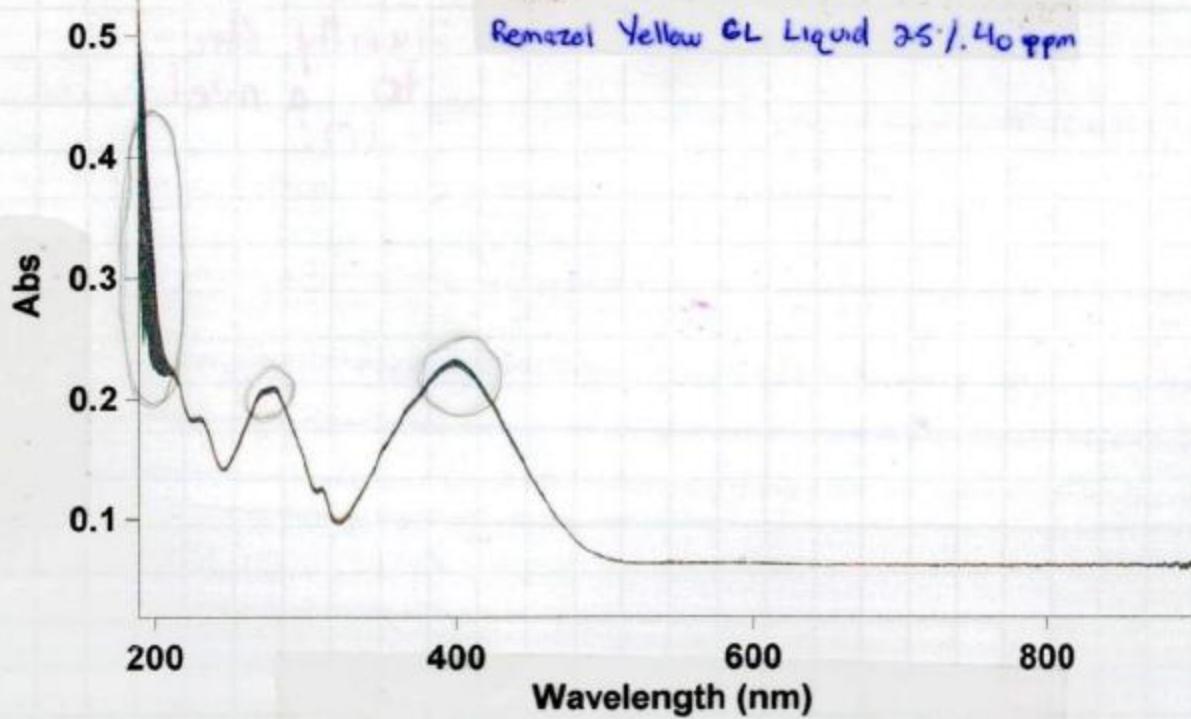
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To Page No. _____			
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From Page No. 12-

Procedure - Scan each of the reactive dyes at 40 ppm on the regular scanning software, label peaks and Save data

- make a caustic solution, 45.5g straight remazol dye to 7.5g NaOH, and mix on blade mixer for 15 minutes
- once all reaction dye has finished mixing, I rapidly make up 40 ppm out of it and Scan ■ using the kinetics software
- each scan was set for 180 cycle (3 hours) to see the change in peaks as the solution continued to react
- save data after 3 hours and Scan another 40 ppm Solution back in the regular Scan program and compare peaks to the non-caustic Scan originally taken
- bottle + Save Solutions

Results - very dissolved solution + mixed well



Witness

Recorded by:

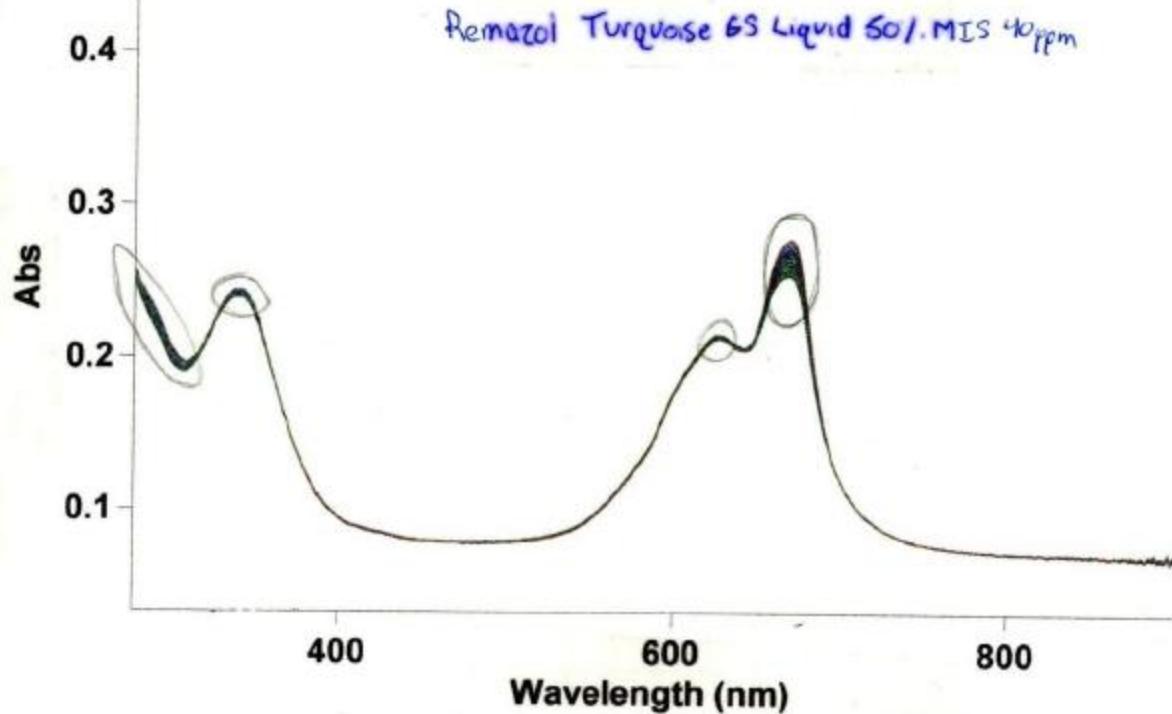
TITLE \_\_\_\_\_

Project No. \_\_\_\_\_

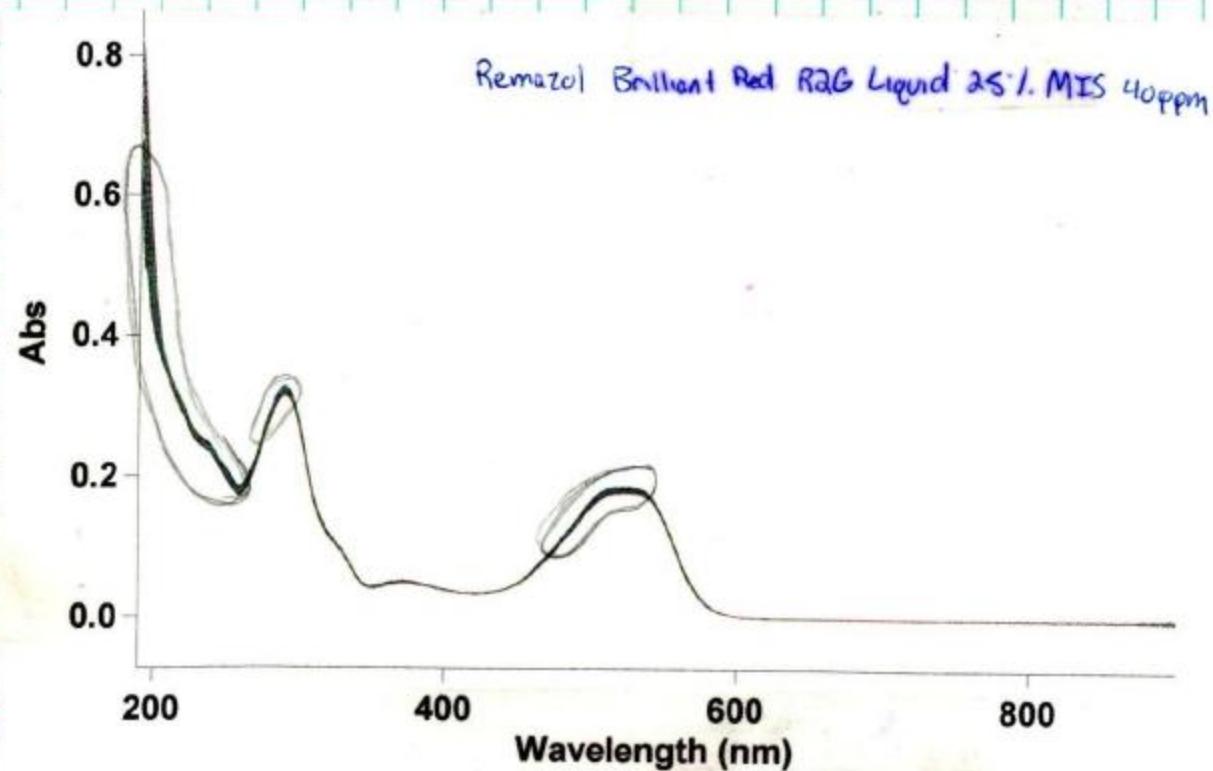
Book No. \_\_\_\_\_

From Page No. \_\_\_\_\_

- very dissolved solution + mixed well



- Dissolved + mixed well, thicker solution compared to others. but still smooth.



No. \_\_\_\_\_

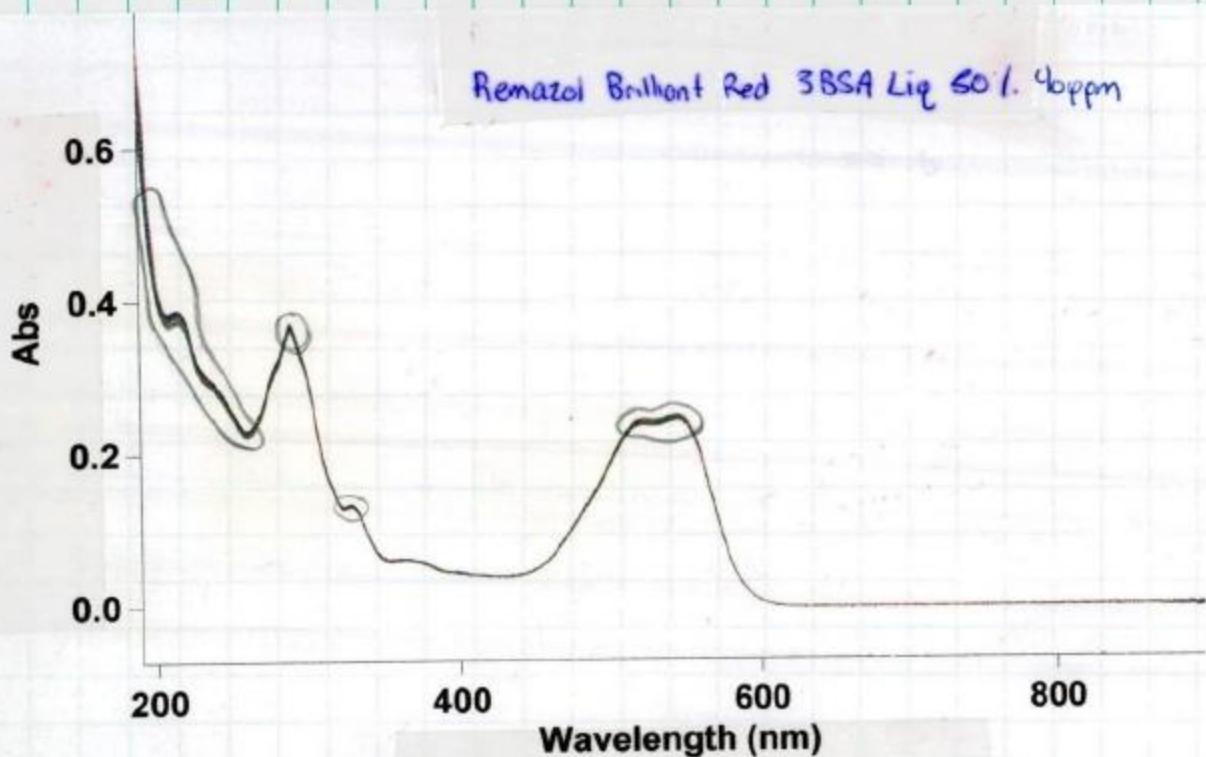
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Book No. \_\_\_\_\_

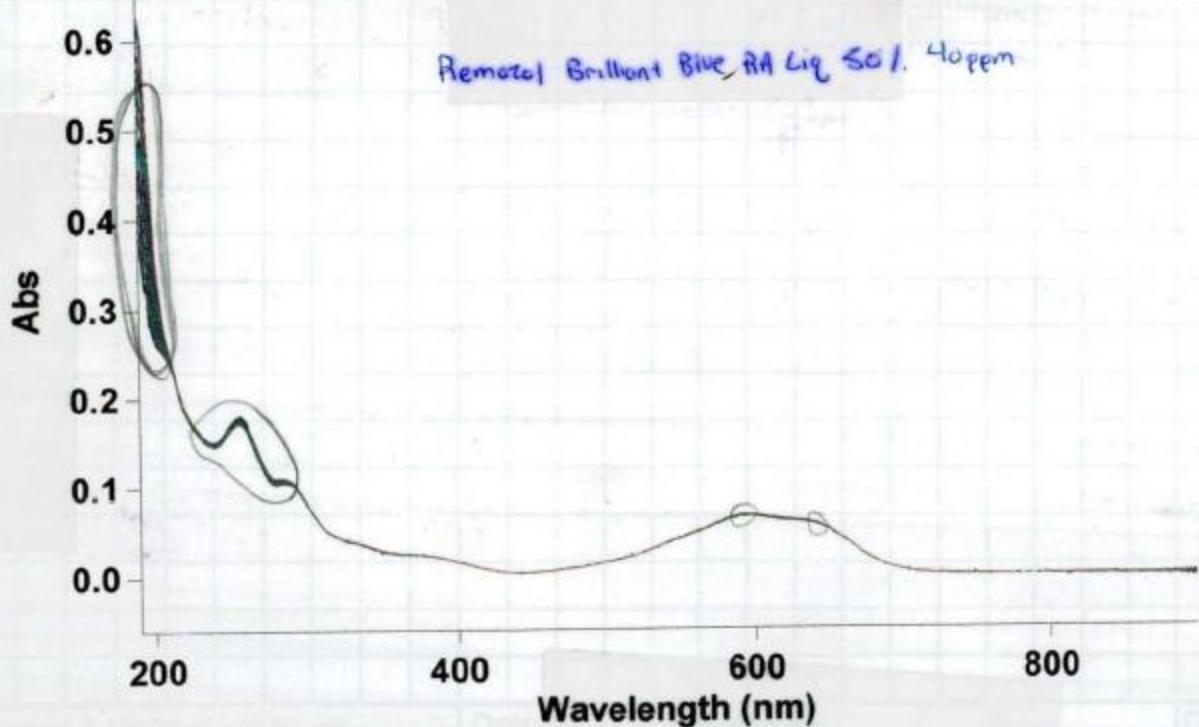
TITLE \_\_\_\_\_

From Page No. \_\_\_\_\_

- DID NOT mix well, very Pastey + Dry, Barely got 1g from it.



- Dissolved + Mixed Well



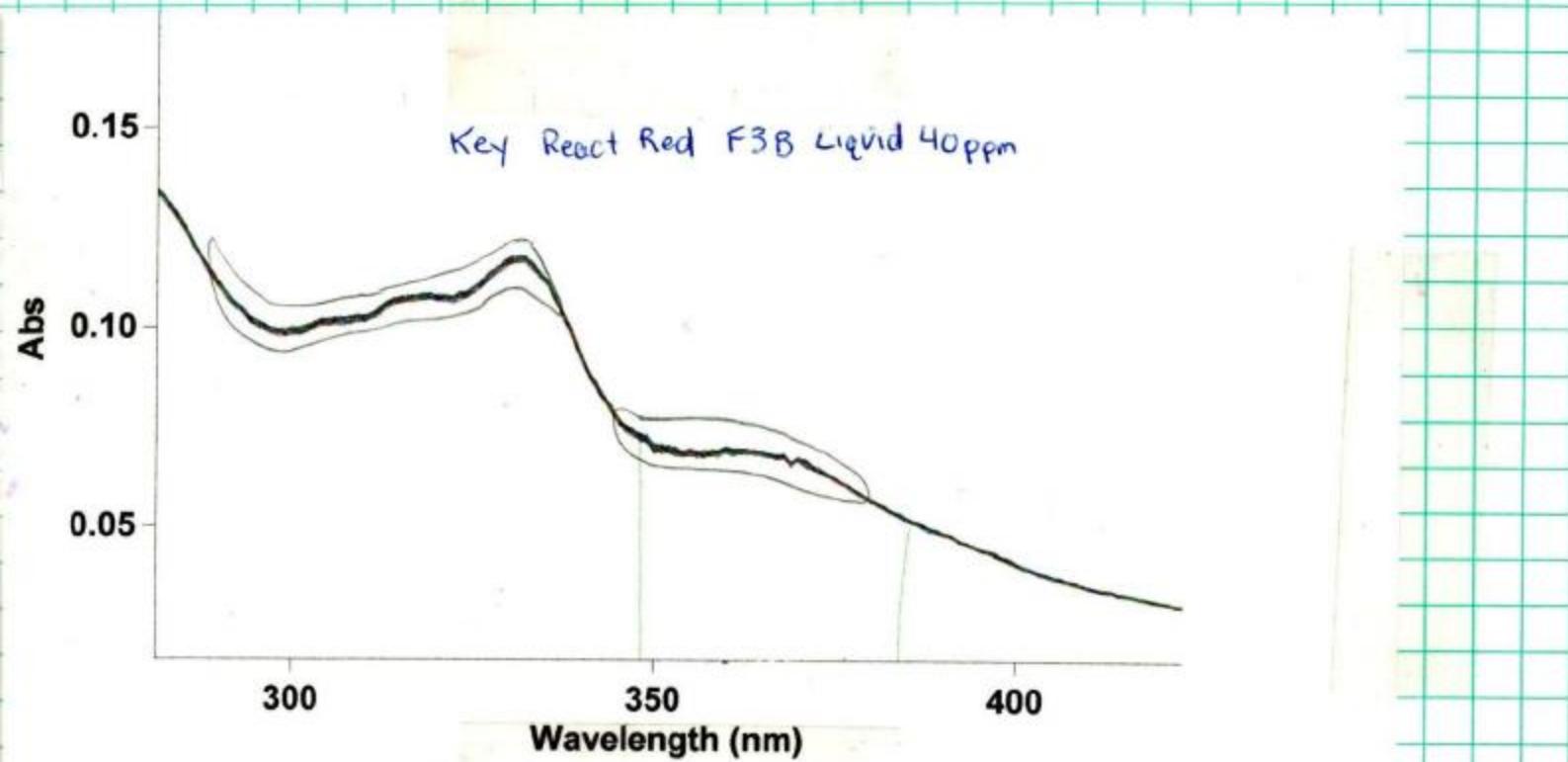
Project No. \_\_\_\_\_

Book No. \_\_\_\_\_

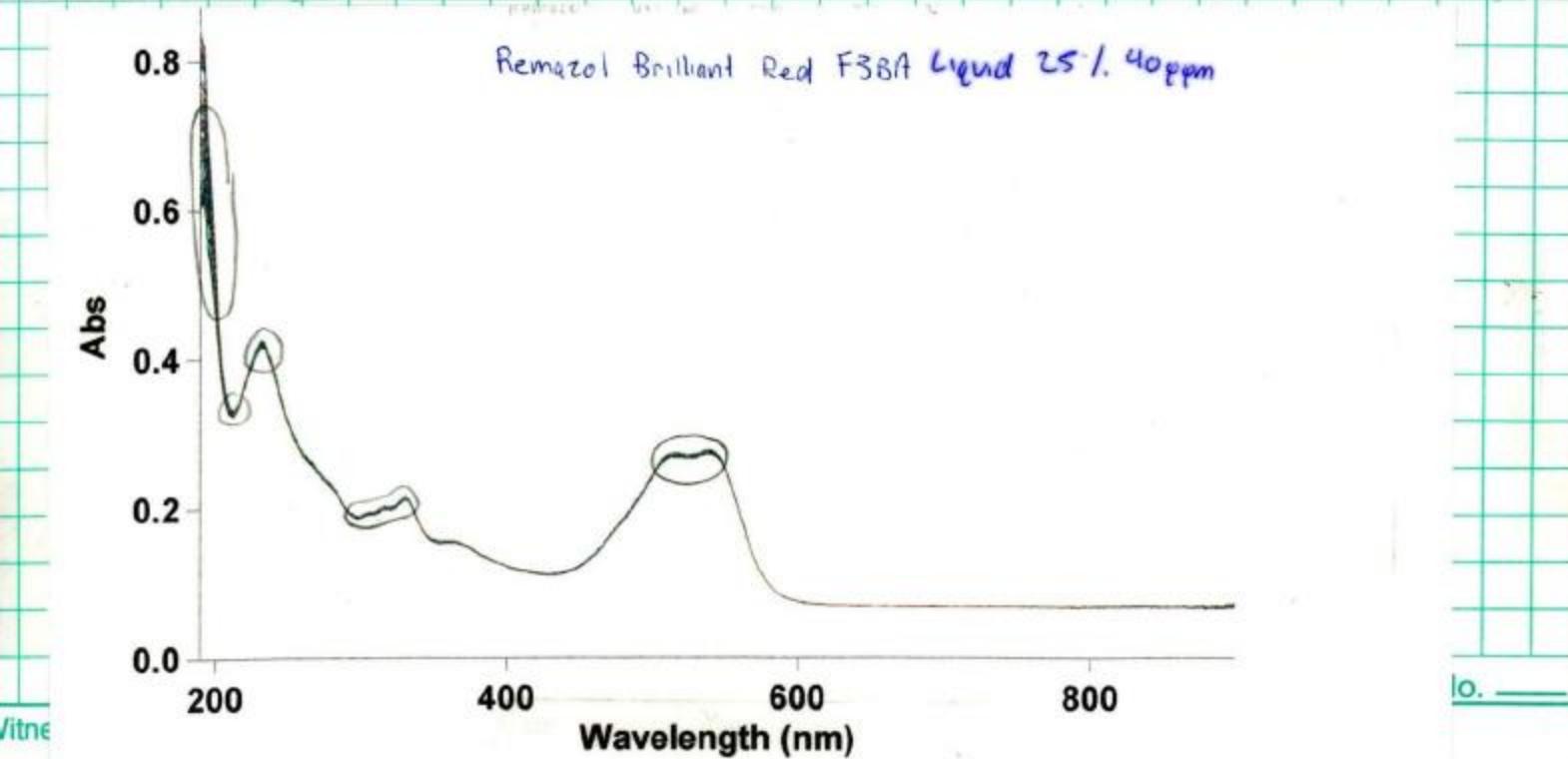
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From Page No. \_\_\_\_\_

- Dissolved + mixed well



- Dissolved + mixed well



Vitne

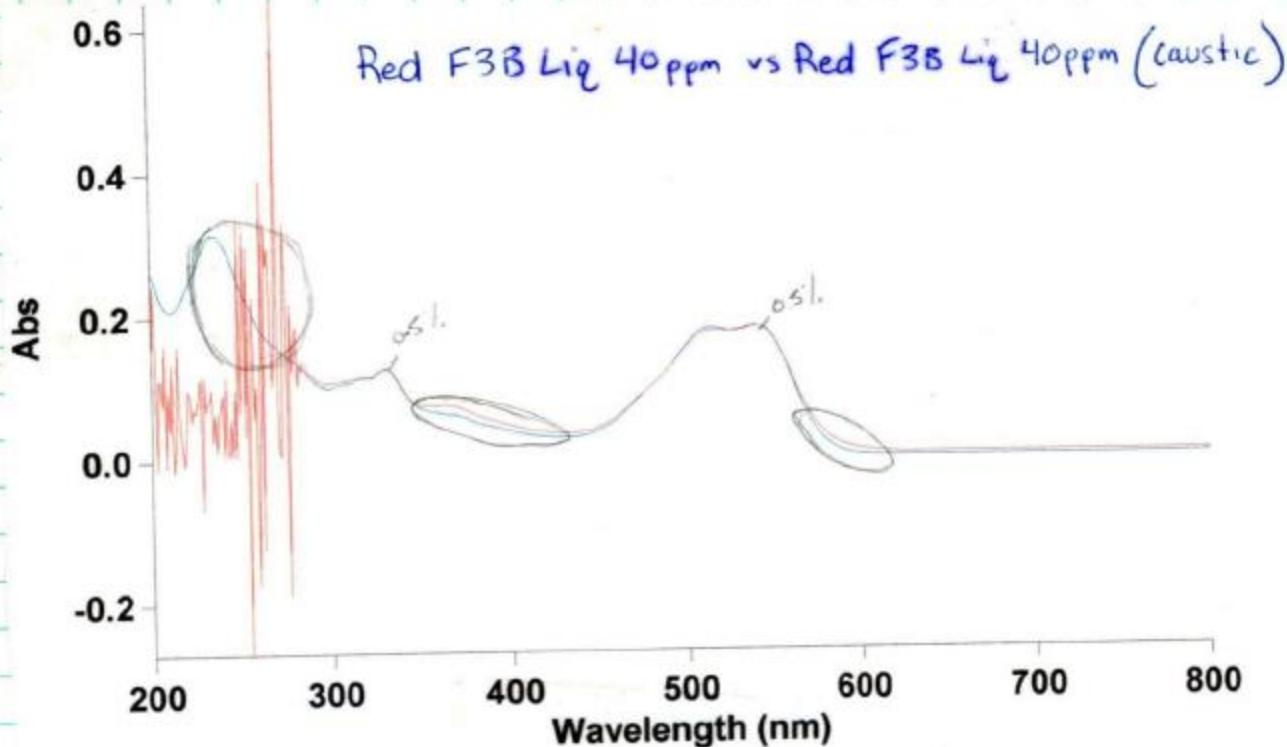
From Page No. \_\_\_\_\_

color matches + non working dyes

- 401-601-47 @ 40 ppm matched to 1.35g Remazol Yellow GL Liquid 25% +  
~~0.35g~~  
 0.35g Remazol Turquoise Liquid 50% brought to 40 ppm

Remazol Brilliant Orange R2G Liquid 25% MIS would not go into any type of solution because it would not even come close to mix. It was very pastey where I could not even draw liquid through a pipet. I assume that the caustic level was to high for this type of dye. I tried mixing it a second time with a higher speed blade mixer level with the same amount of caustic and again it failed and became a thick paste in the matter of a few minutes.

Red F3B Liq 40 ppm (caustic) follows very similar curves and peaks reused in strength 0.5%.



Witnessed &amp; Understood by me,

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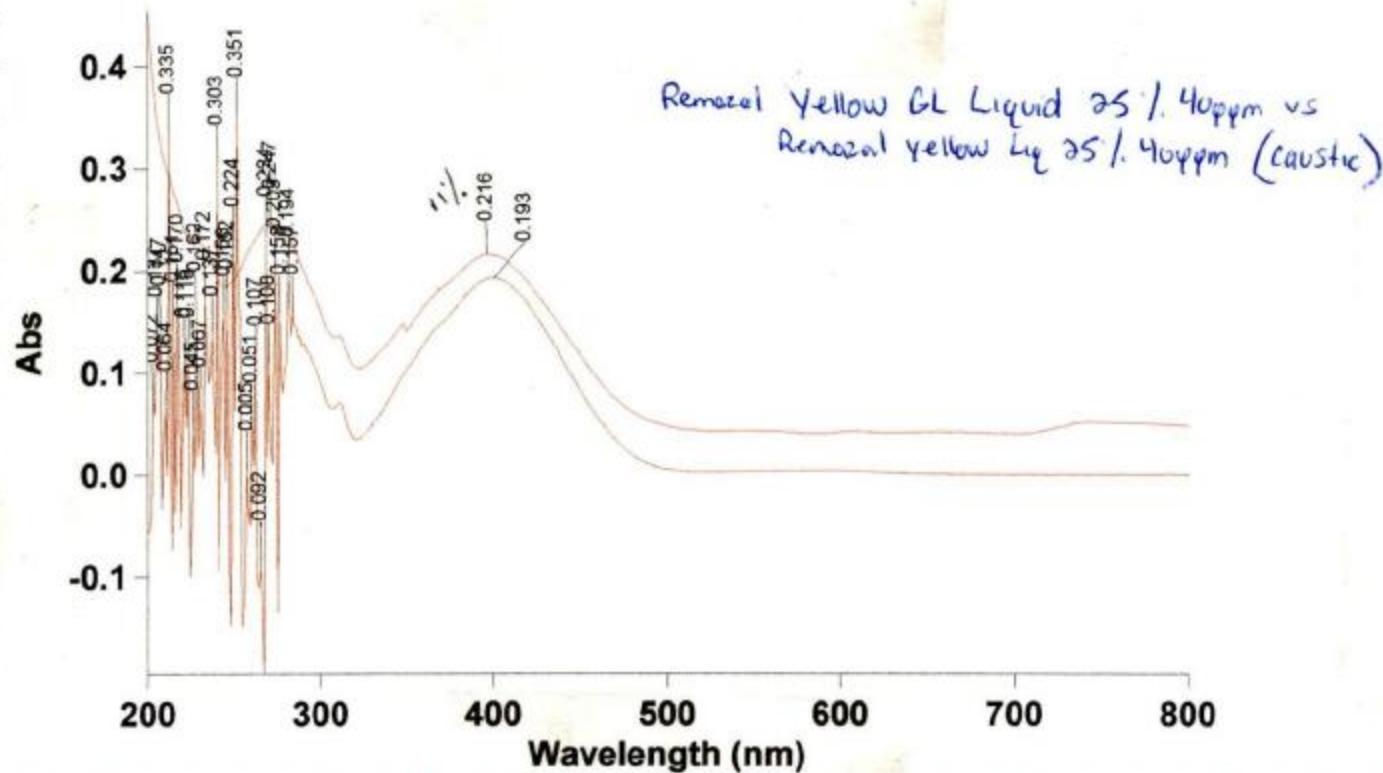
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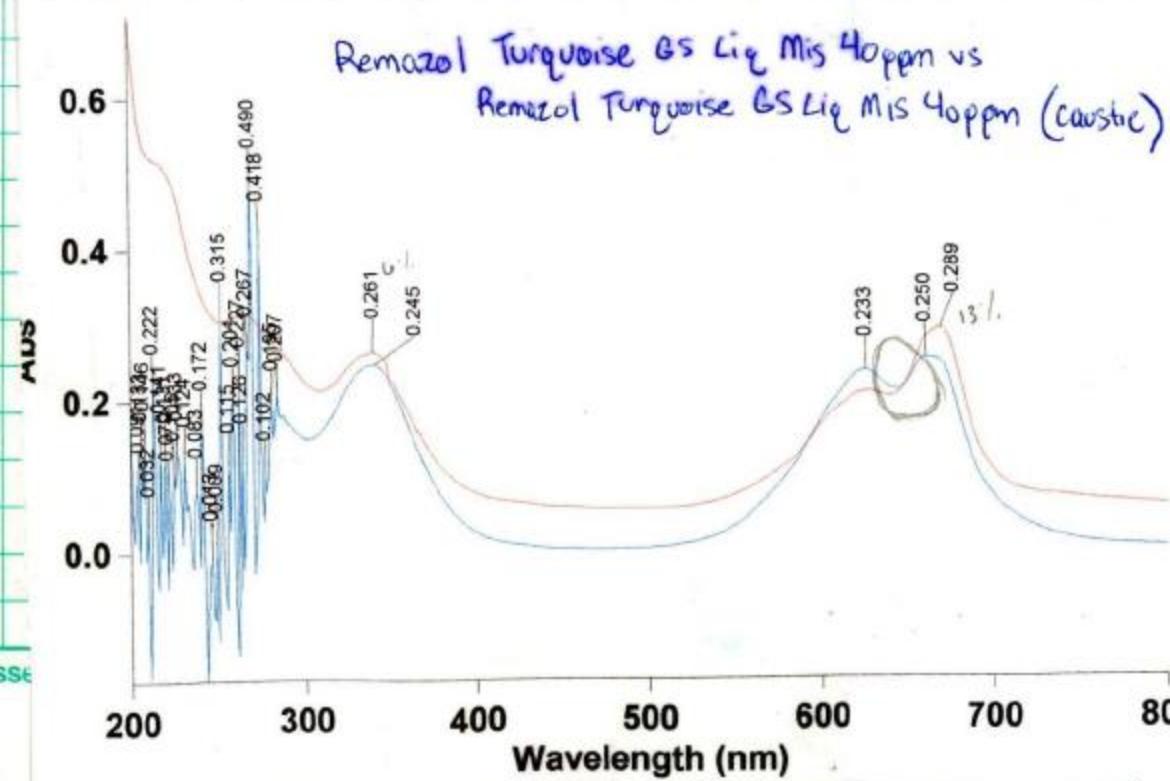
Book No. \_\_\_\_\_

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yellow GL Liquid 25%. 40ppm Caustic followed same curves with a slight peak shift + strength increased 11%.



Turquoise GS Liquid Mis 40ppm Caustic shifted peaks and was 13% strong in one and 6% in the other.



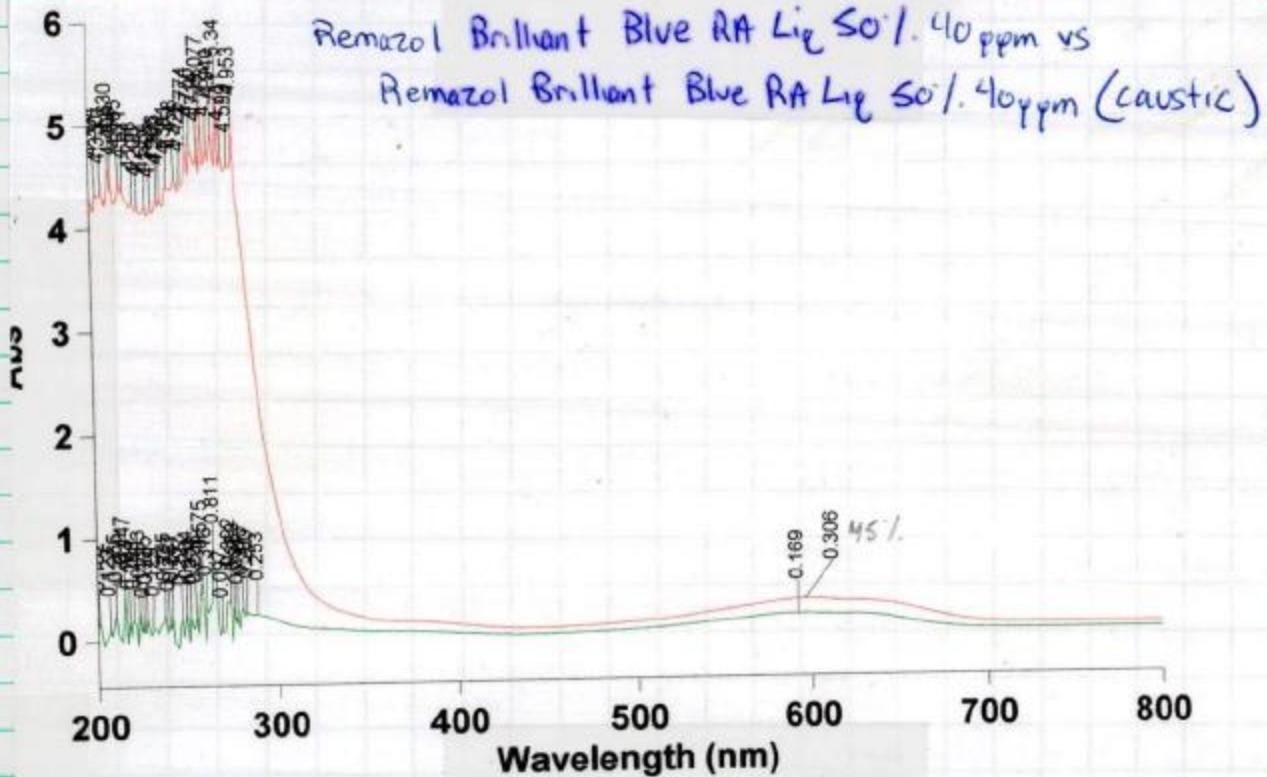
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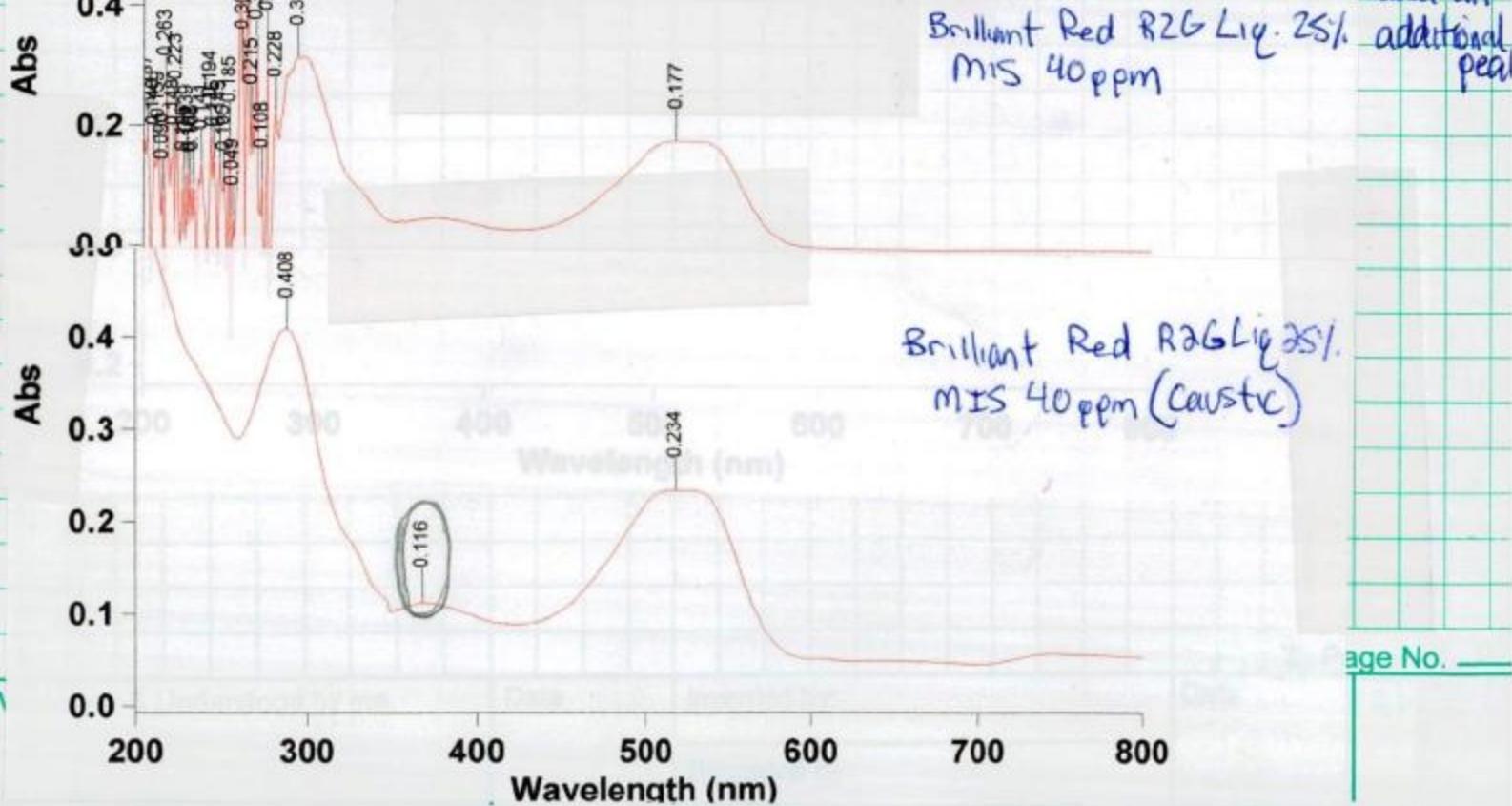
From Page No. \_\_\_\_\_

Brilliant Blue RA Lig 50%. Caustic was 45% stronger and peak shifted very slightly



Brilliant Red R2G is 24% stronger, peaks match with the exception that the Caustic added an

Brilliant Red R2G Lig. 25% additional peak MIS 40 ppm



### Sample Information

Customer code		Contact name	Doug Koerner
Company name	Keystone Aniline Corporation 2501 West Fulton Street	Telephone no	001 312 666 2015
City	Chicago	Email	
County/State		Category	Photochromic
Country	United States	End Customer (if known)	Duplicate samples sent to Del Sol
PostCode/ZipCode	IL 60612	Agent/ Distributor	Keystone
Request no	CGOD-75PBQH	AWB	
Date received	02/08/2007	Shipped by	
Received by	Claire Goddard	Assign sample to	Claire Goddard
Requested delivery date		cc:	Victoria Leyland
Overnight		Cost	
Request status	With Samples		
Sample closed by			
Dispatch date			

Product Name	Quantity	Batch
Encapsacol Green - 10 micron	100g	070705b
Encapsacol Purple - 10 micron	100g	070704b
Encapsacol Yellow - Slurry	20g	070705d

Additional Information

B5

M 1  
0.2 : 100 Ac

2

cut B<sub>3</sub>

B 3

2 : 100

B3

TITLE MARKER STRENGTH COMPARISON

Project No. \_\_\_\_\_

Book No. \_\_\_\_\_

From Page No. 27

Objective: To develop alternate marker ink for use in Sharpie. Solvent, Acid and Basic Dyes will be considered for substitute of existing black solvent dye. Violet, Red, Blue, and Black dyes will be considered as components of final product.

Dyes + Solvent:

- Solvent used is as follows (Solvent 1)

60% Carbitol

10% Glycol Ether DB

5% Benzyl Alcohol

7% Ethanol (Tech. Grade)

\* adjusted to 1.

- Dyes considered are as follows:

• 206-044-50 (F-6270)

• 606-135-50 (B-0497)

• 106-007-71 (Vendor Std)

• 606-052-51 (G-9368)

• 703-003-50 (203C065)

• 806-093-50 (AGD-L001)

• 806-072-51 (207J343)

• 806-114-50 (208G050)

Additives were as follows:

hydroxybenzoic acid

toluic acid

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Project No. \_\_\_\_\_

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TITLE MARKER STRENGTH

From Page No. 27

Dyes made as 3% solution, using Solvent 1

- 3.00g dye in beaker, add 75mL Solvent 1. Stir 5-10 min.  
check for undissolved dye in beaker.

- make marker using parts given.
- mark on paper for comparison.

MARKERS MADE

1. 106-044-50 (3g:80mL)

2. 106-007-70 (3g:80mL)

### Results:

- 106-044-50 runs from marker. Creep in markings.  
also need to alter solvation method (e.g., add dye to solvent)
- Solvent system not compatible with 106-007-70

Renamed 106-007-70 as 3.00g dye: 50mL solvent 1; 0.05g toluic acid  
• Stir 5-10 min.

- marker showed less creep.
- add 2.0 g dye to 6% solution. Added with stirring.  
dye still coagulated. Stir, 5-10 min.

### Mixtures of Dye Components

Solutions Made for second round of marker testing:

Dye Material	Amount			
106-007-71	5.0g	2.5g	2.0g	2.0g
806-093-50	0.5g	0.5g	0.4g	0.4g
806-072-51	-	-	-	-
806-114-50	-	-	-	-
806-053-50	-	-	-	-
Solvent 1	50.0mL	50.0mL	20.0mL	
o-toluiic acid	0.05g	0.05g	-	-
marker #	A	B	-	-
hydroxy benzoic acid	-	-	0.05	-

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Date

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unk 2 unk 2 unk 3  
unk 1

206-044 (206-044)

106-007-71 (106-007) (3%)

106-007-71 (6%)

106-007-71 (10%)

Marker B

(5% SB, 1% y)

Marker A (10% SB, 1% y)

10% in Xylene

10 ————— X

Standard Name

372 of 372

Today's Date: 13-Jan-09

test marker b

Batch Name

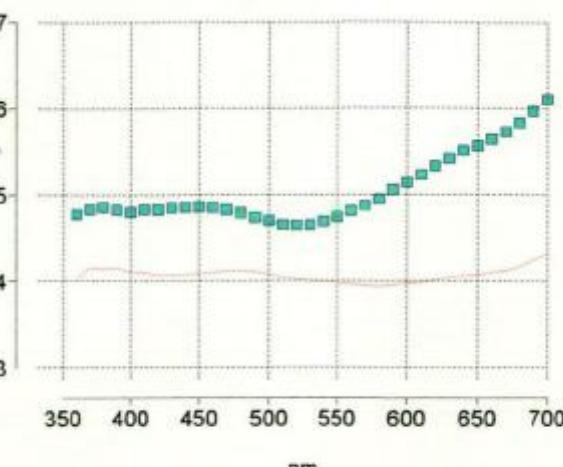
2 of 2

unk 3

Date: 13-Jan-09

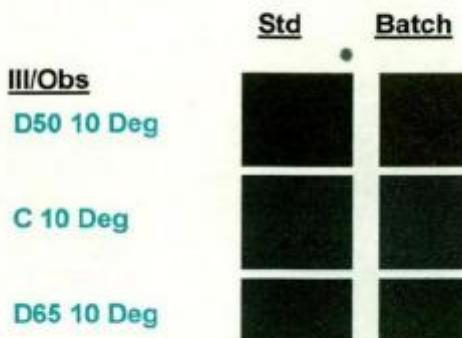
Time: 11:40:38

% Reflectance

**P/F : Fail**

Batch is :  
 lighter  
 more saturated  
 more red

<u>III/Obs</u>	<u>P/F</u>	<u>Decision</u>	<u>DE*</u>	<u>DL*</u>	<u>Da*</u>	<u>Db*</u>	<u>DC*</u>	<u>DH*</u>
D50 10 Deg		Fail	3.54	2.73	2.11	0.78	1.63	1.55
C 10 Deg		Fail	3.37	2.68	1.92	0.68	1.48	1.40
D65 10 Deg		Fail	3.39	2.67	1.99	0.66	1.53	1.43



78.85%

CMC DE = 3.91

test marker b

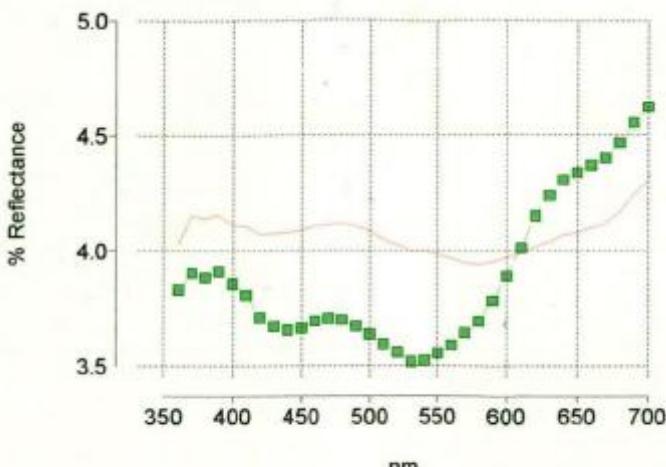
Batch Name

1 of 1

unk 1

Date: 13-Jan-09

Time: 11:39:27

**P/F : Fail**

Batch is :  
darker  
more saturated  
more red

Std      BatchIII/Obs

D50 10 Deg



C 10 Deg



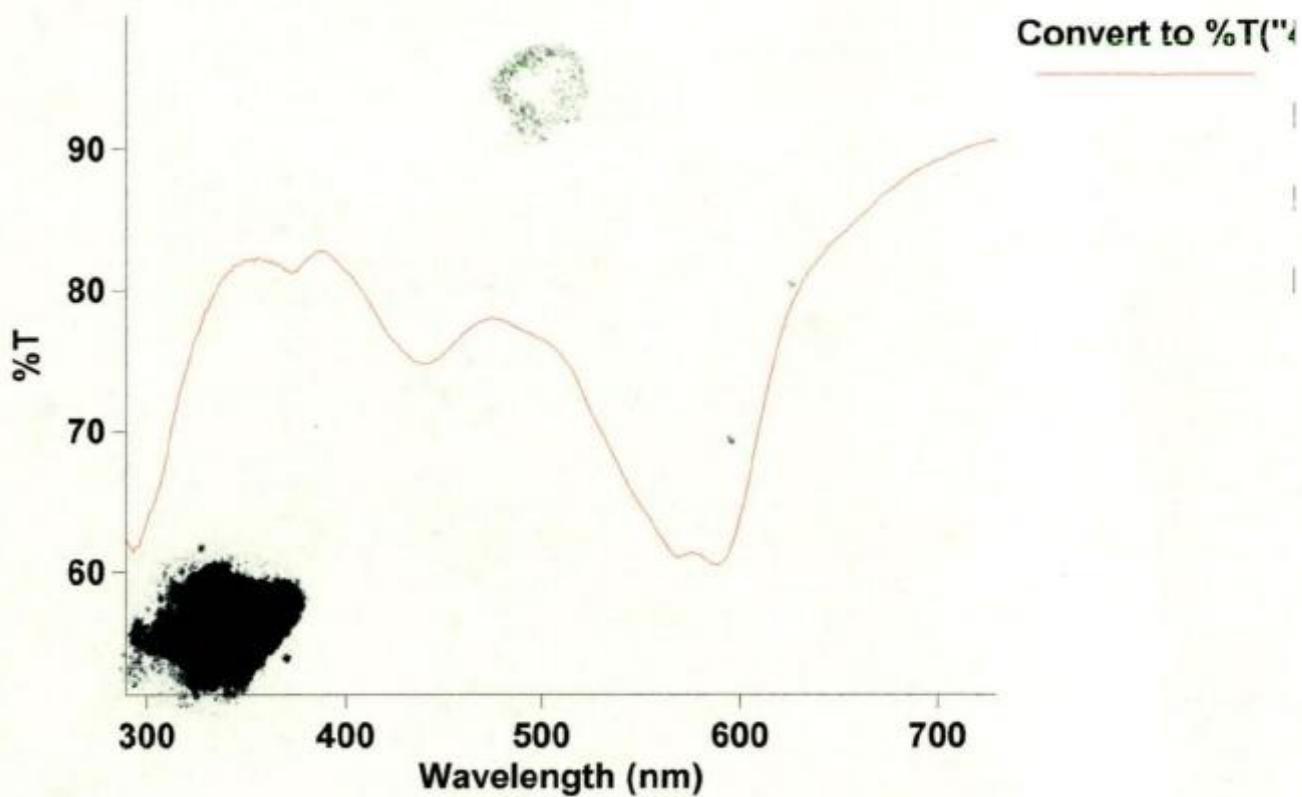
D65 10 Deg



P/F		<u>DE*</u>	<u>DL*</u>	<u>Da*</u>	<u>Db*</u>	<u>DC*</u>	<u>DH*</u>
<u>III/Obs</u>	<u>Decision</u>	2.38	-1.01	2.06	0.66	1.56	1.49
D50 10 Deg	Fail	2.18	-1.05	1.83	0.58	1.38	1.33
C 10 Deg	Fail	2.24	-1.06	1.89	0.55	1.43	1.36

105.64%

CMC DE = 3.31



## Scan Analysis Report

Report Time : Mon 12 Jan 09:30:45 AM 2009  
 Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\10603450 208C228 200ppm EtOH with 5 percent T.DSW  
 Software version: 3.00(182)  
 Operator:

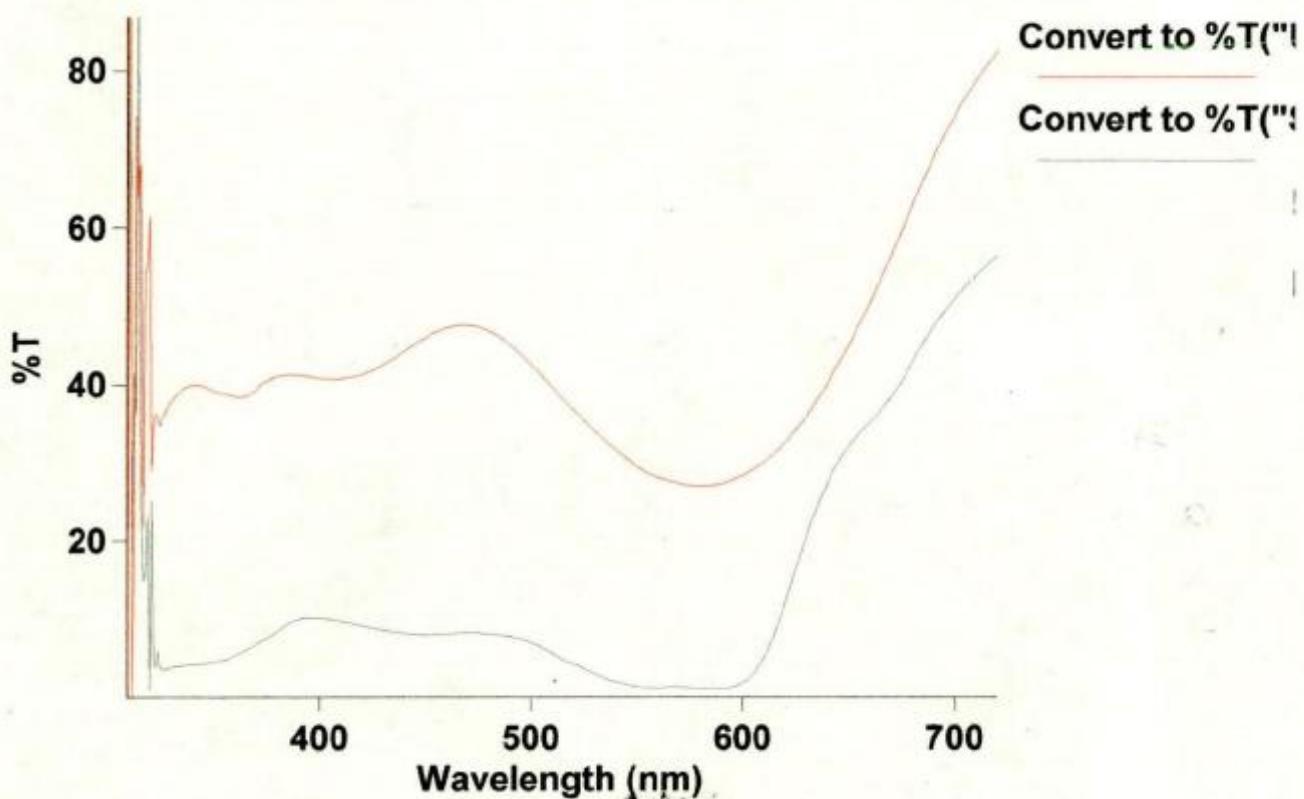
### Sample Name: 10603450 208C228 200ppm EtOH with 5 percent T

Collection Time 1/12/2009 9:30:45 AM

Peak Table	Peaks
Peak Style	
Peak Threshold	
Range	

Wavelength (nm) Abs

595.00	0.079
280.00	0.146
277.00	0.247
274.00	0.090
270.00	0.131
268.00	0.178
264.00	0.180
259.00	0.078
256.00	0.143
253.00	0.345



## Scan Analysis Report

Report Time : Mon 12 Jan 09:30:45 AM 2009  
 Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\10603450 208C228 200ppm EtOH with 5 percent T.DSW  
 Software version: 3.00(182)  
 Operator:

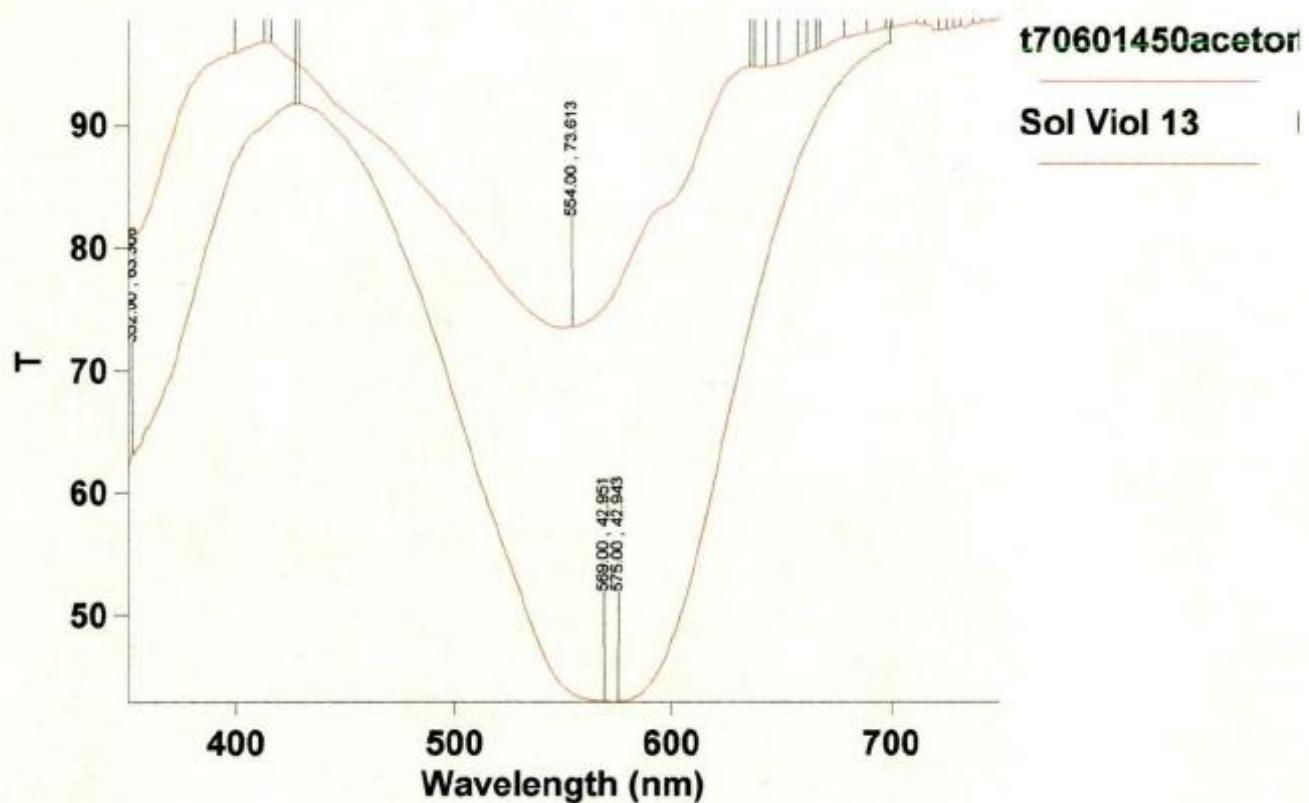
### Sample Name: 10603450 208C228 200ppm EtOH with 5 percent T

Collection Time 1/12/2009 9:30:45 AM

Peak Table	Peaks
Peak Style	
Peak Threshold	
Range	

Wavelength (nm) Abs

595.00	0.079
280.00	0.146
277.00	0.247
274.00	0.090
270.00	0.131
268.00	0.178
264.00	0.180
259.00	0.078
256.00	0.143
253.00	0.345



## Scan Analysis Report

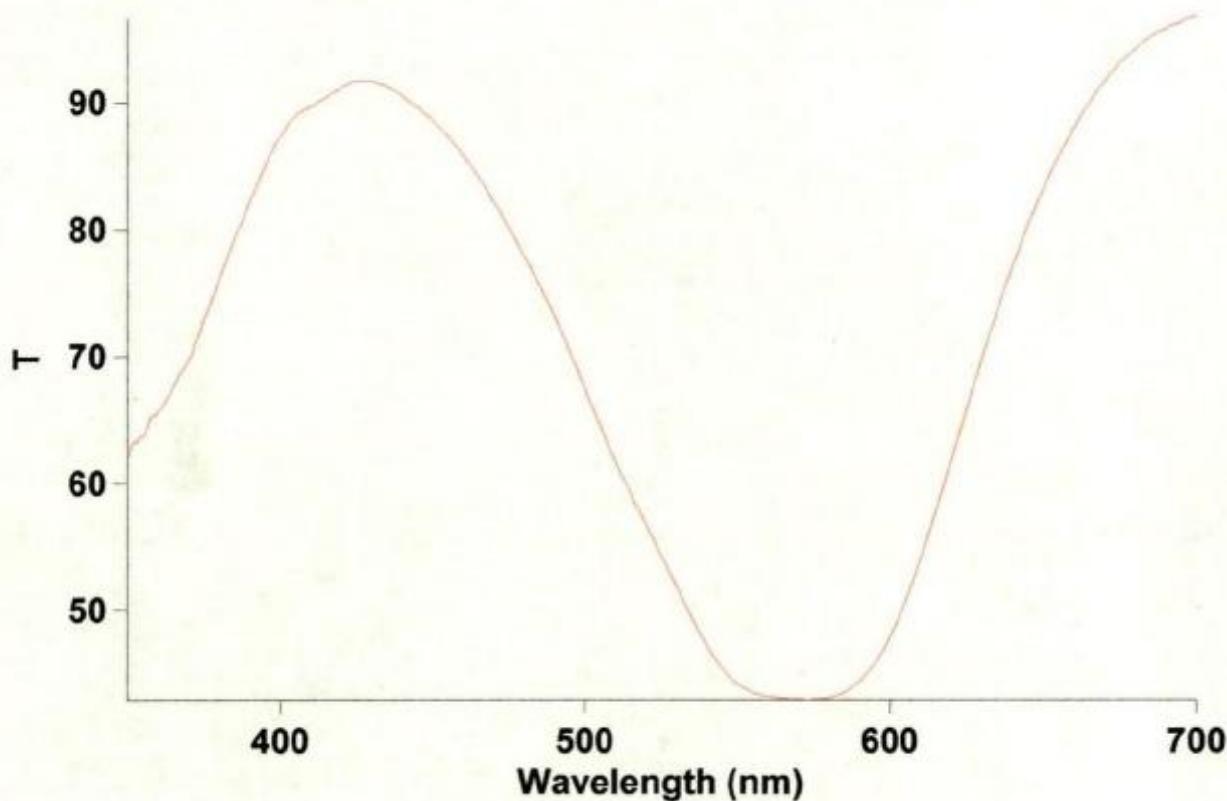
Report Time : Fri 09 Jan 02:38:40 PM 2009  
 Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\50643850 A208J070 200ppm EtOH with 5 percent H.DSW  
 Software version: 3.00(182)  
 Operator:

### Sample Name: 50643850 A208J070 200ppm EtOH with 5 percent H

Collection Time 1/9/2009 2:38:42 PM

Peak Table	
Peak Style	Peaks
Peak Threshold	0.0100
Range	800.00nm to 200.00nm

Wavelength (nm)	Abs
481.00	1.051
400.00	0.791
329.00	0.836
282.00	0.721
280.00	0.711
277.00	0.541
275.00	1.423
273.00	0.501
271.00	0.819
268.00	0.815



## Scan Analysis Report

Report Time : Fri 09 Jan 02:38:40 PM 2009  
Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\50643850 A208J070 200ppm EtOH with 5 percent H.DSW  
Software version: 3.00(182)  
Operator:

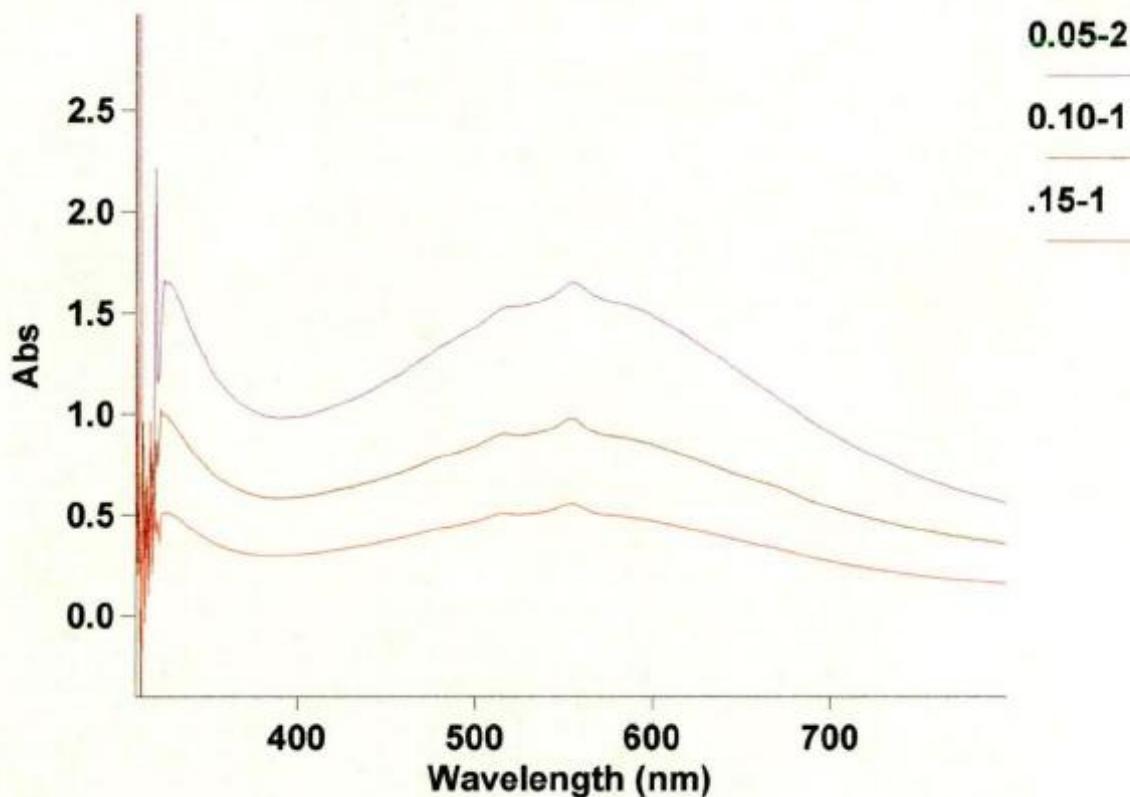
### Sample Name: 50643850 A208J070 200ppm EtOH with 5 percent H

Collection Time 1/9/2009 2:38:42 PM

Peak Table  
Peak Style  
Peak Threshold  
Range

Wavelength (nm)	Abs
481.00	1.051
400.00	0.791
329.00	0.836
282.00	0.721
280.00	0.711
277.00	0.541
275.00	1.423
273.00	0.501
271.00	0.819
268.00	0.815

Wavelength (nm)	Abs
481.00	1.051
400.00	0.791
329.00	0.836
282.00	0.721
280.00	0.711
277.00	0.541
275.00	1.423
273.00	0.501
271.00	0.819
268.00	0.815



## Scan Analysis Report

Report Time : Fri 09 Jan 01:51:54 PM 2009  
Batch: C:\Varian\Cary Winuv\Reference Scans\ETH SOL. RUN  
Software version: 3.00(182)  
Operator:

### Sample Name: .05

Collection Time 1/9/2009 1:51:59 PM

Peak Table  
Peak Style Peaks  
Peak Threshold 0.0100  
Range 800.00nm to 200.00nm

Wavelength (nm)	Abs
755.00	3.431
743.00	3.637
737.00	3.773
733.00	3.831
731.00	3.894
718.00	4.143
712.00	4.252
709.00	4.313
706.00	4.400
702.00	4.569
700.00	4.528

## unknown marker 2 red shade

Batch Name	3	of	3	Std Max Wt:	Sum of K/S	
				Bat Max Wt:	Status:	
sb 27				<b>Strength: 19.57 %</b>		
Date:	06-Jan-09	Time:	15:11:34	Batch is :	lighter less saturated more blue	
III/Obs	DL*	Da*	Db*	DC*	DH*	DE*
D50 10 Deg	22.74	-13.17	3.52	-11.47	-7.37	26.51
C 10 Deg	22.85	-11.82	3.48	-10.08	-7.08	25.96
D65 10 Deg	22.95	-12.01	3.70	-10.28	-7.22	26.17

Standard Name

332 of 332

Today's Date: 06-Jan-09

## unknown marker 2 red shade

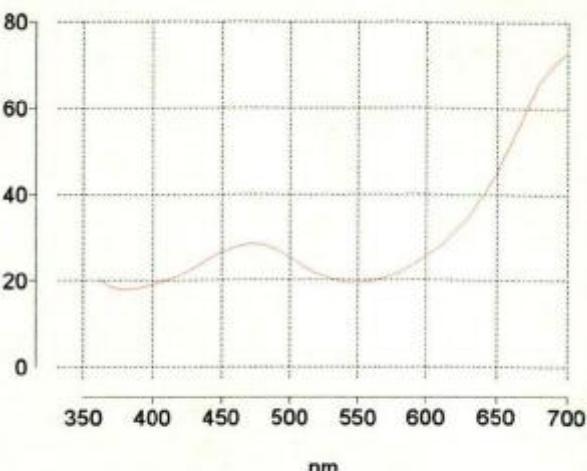
Batch Name

0 of 0

Date:

Time:

% Reflectance



P/F :

Batch is :

P/F

<u>III/Obs</u>	<u>Decision</u>	<u>DE*</u>	<u>DL*</u>	<u>Da*</u>	<u>Db*</u>	<u>DC*</u>	<u>DH*</u>
D50 10 Deg							
C 10 Deg							
D65 10 Deg							

III/Obs

D50 10 Deg

Std

Batch



C 10 Deg



D65 10 Deg



SB 45

SB 27

Standard Name

330 of 336

Today's Date: 06-Jan-09

**unknown marker 3 blk**

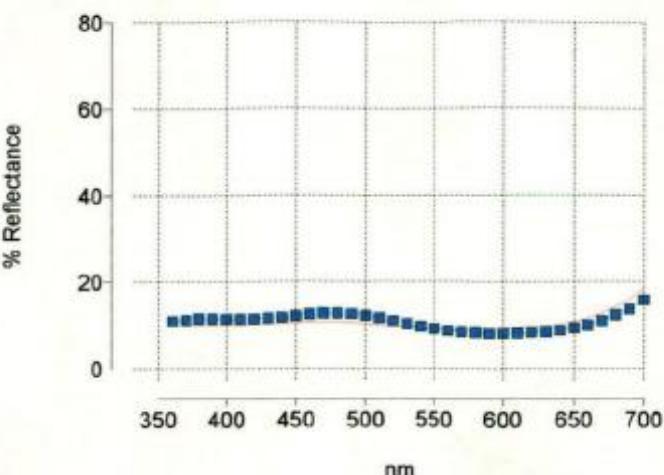
Batch Name

4 of 4

**sb3+sb27 strng1**

Date: 06-Jan-09

Time: 15:34:38

**P/F : Fail**

Batch is :  
**lighter**  
**more saturated**  
**more green**

P/F

<u>III/Obs</u>	<u>Decision</u>	<u>DE*</u>	<u>DL*</u>	<u>Da*</u>	<u>Db*</u>	<u>DC*</u>	<u>DH*</u>
D50 10 Deg	Fail	6.19	0.17	-4.43	-4.33	5.15	-3.43
C 10 Deg	Fail	5.48	0.33	-3.74	-3.99	4.51	-3.09
D65 10 Deg	Fail	5.63	0.36	-3.98	-3.96	4.64	-3.17

III/Obs

D50 10 Deg

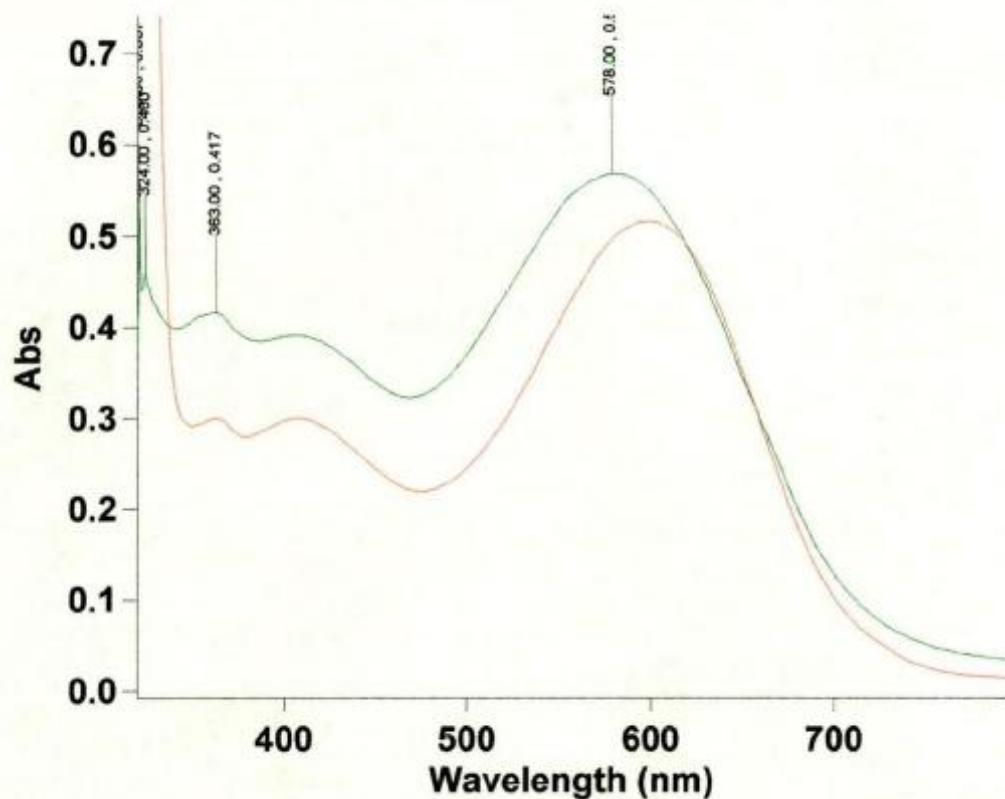


C 10 Deg



D65 10 Deg





## Scan Analysis Report

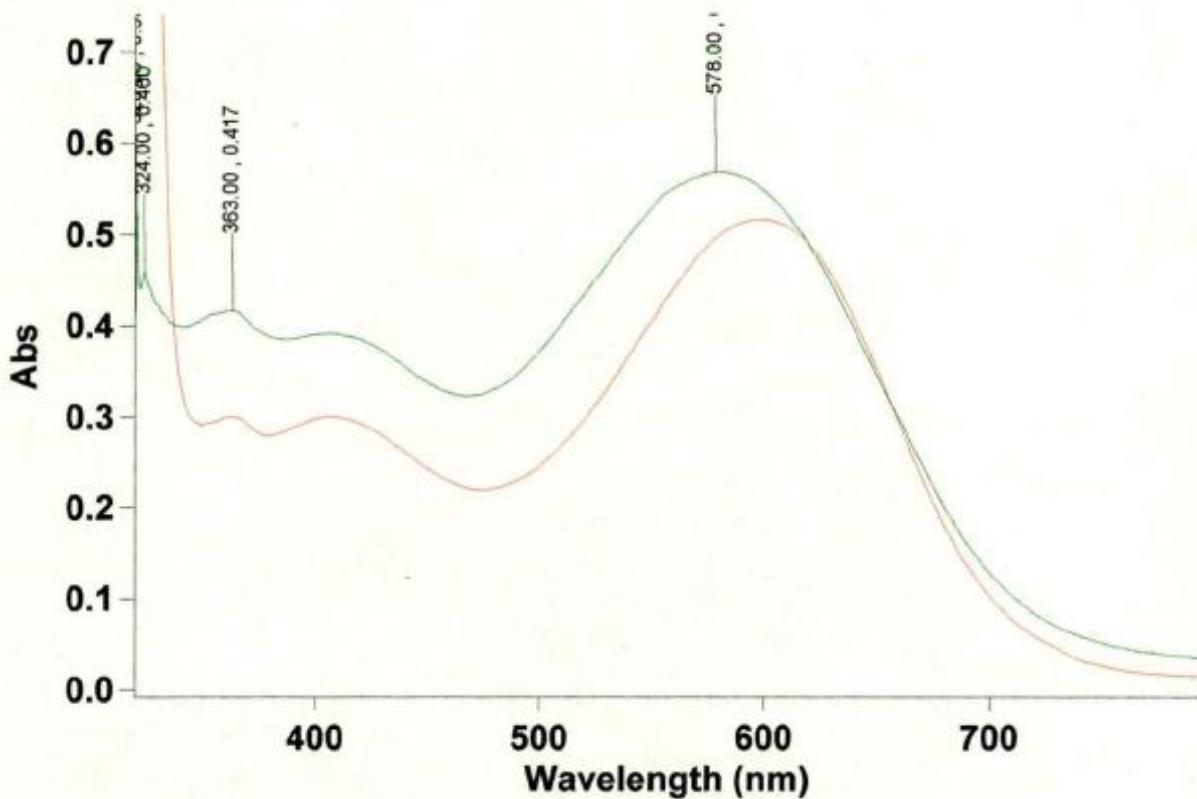
Report Time : Tue 06 Jan 03:32:58 PM 2009  
 Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\60600850 A208A266 200ppm MEK saturated solution.DSW  
 Software version: 3.00(182)  
 Operator:

### Sample Name: 60600850 A208A266 200ppm MEK saturated solution

Collection Time 1/6/2009 3:32:59 PM

Peak Table	Peaks
Peak Style	
Peak Threshold	0.0100
Range	800.00nm to 200.00nm

Wavelength (nm)	Abs
518.00	0.814
371.00	0.480
285.00	0.391
283.00	0.411
280.00	0.469
275.00	0.573
272.00	0.760
270.00	0.328
268.00	0.923
265.00	0.876



## Scan Analysis Report

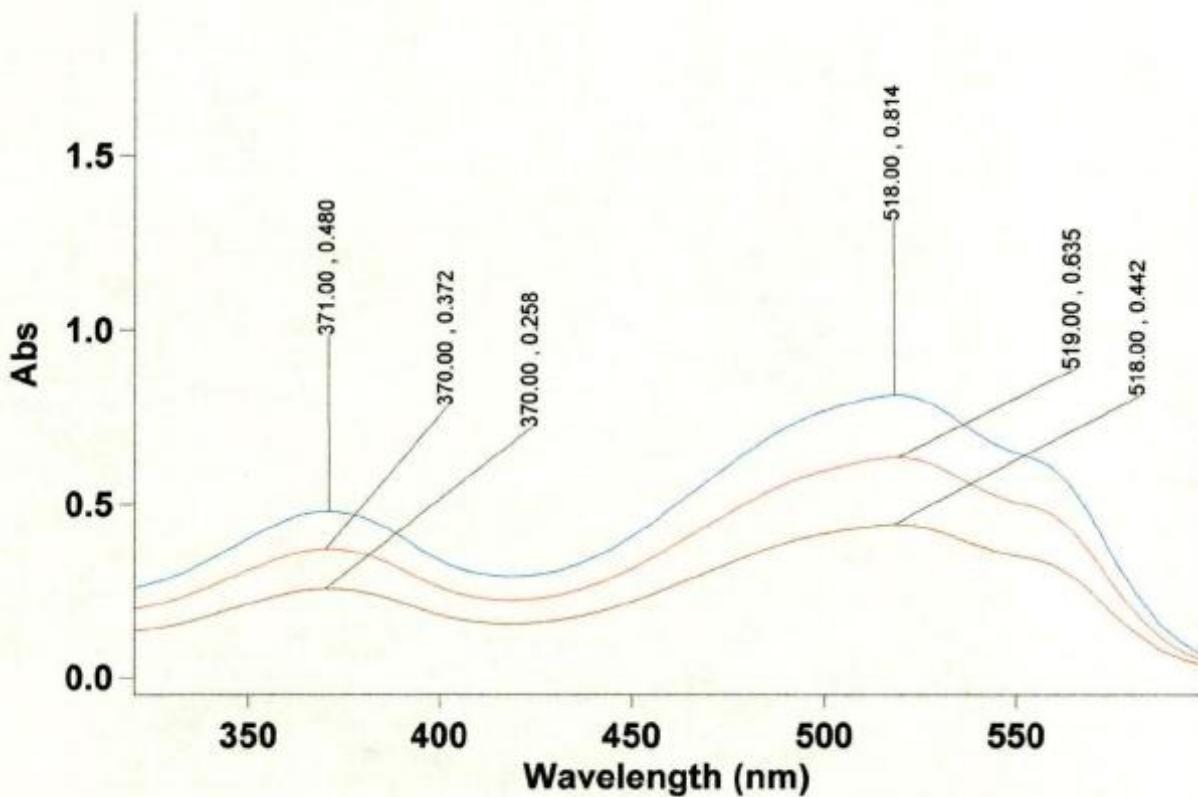
Report Time : Tue 06 Jan 03:32:58 PM 2009  
 Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\60600850 A208A266 200ppm MEK saturated solution.DSW  
 Software version: 3.00(182)  
 Operator:

### Sample Name: 60600850 A208A266 200ppm MEK saturated solution

Collection Time 1/6/2009 3:32:59 PM

Peak Table	
Peak Style	Peaks
Peak Threshold	0.0100
Range	800.00nm to 200.00nm

Wavelength (nm)	Abs
518.00	0.814
371.00	0.480
285.00	0.391
283.00	0.411
280.00	0.469
275.00	0.573
272.00	0.760
270.00	0.328
268.00	0.923
265.00	0.876



## Scan Analysis Report

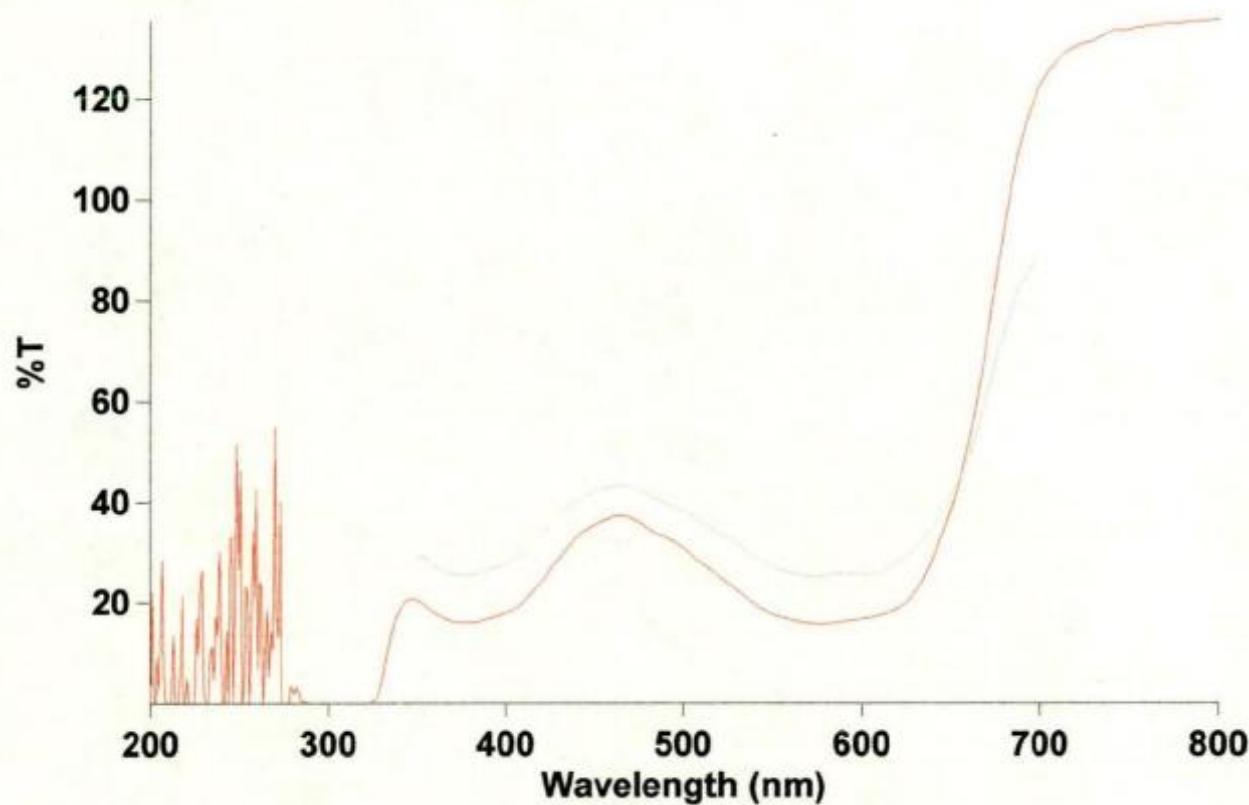
Report Time : Tue 06 Jan 03:32:58 PM 2009  
 Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\60600850 A208A266 200ppm MEK saturated solution.DSW  
 Software version: 3.00(182)  
 Operator:

### Sample Name: 60600850 A208A266 200ppm MEK saturated solution

Collection Time 1/6/2009 3:32:59 PM

Peak Table	
Peak Style	Peaks
Peak Threshold	0.0100
Range	800.00nm to 200.00nm

Wavelength (nm)	Abs
518.00	0.814
371.00	0.480
285.00	0.391
283.00	0.411
280.00	0.469
275.00	0.573
272.00	0.760
270.00	0.328
268.00	0.923
265.00	0.876



## Scan Analysis Report

Report Time : Tue 06 Jan 01:58:44 PM 2009  
Batch: C:\Varian\Cary Winuv\SpectralData\Gina\MEK and Ethanol Solubility\70600855 A204L553 10ppm EtOH.DSW  
Software version: 3.00(182)  
Operator:

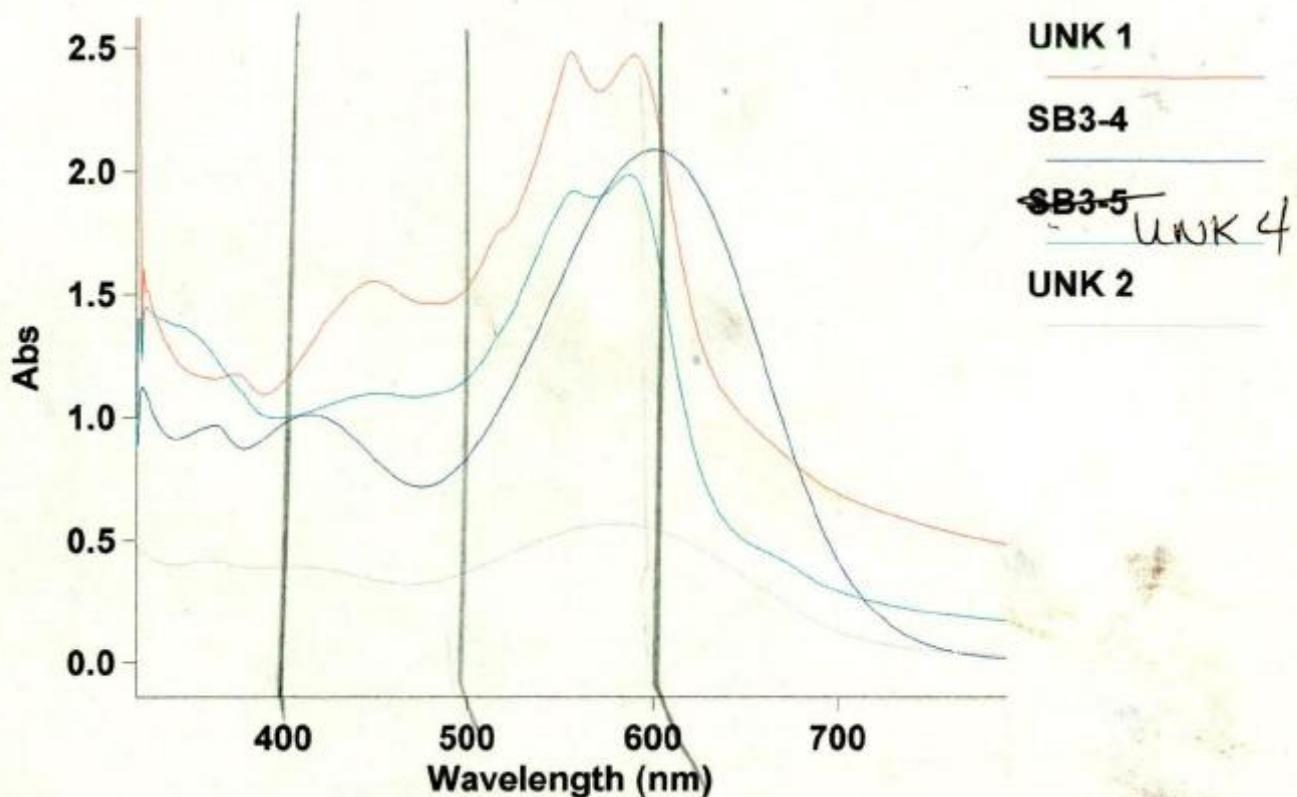
### Sample Name: 70600855 A204L553 10ppm EtOH

Collection Time 1/6/2009 1:58:45 PM

Peak Table  
Peak Style  
Peak Threshold  
Range

Peaks  
0.0100  
800.00nm to 200.00nm

Wavelength (nm)	Abs
579.00	1.690
302.00	0.430
282.00	0.224
278.00	0.229
274.00	0.238
272.00	0.260
269.00	0.300
267.00	0.266
264.00	0.484
261.00	0.328
258.00	0.220



## Scan Analysis Report

Report Time : Tue 06 Jan 01:19:44 PM 2009  
 Batch: C:\Varian\Cary Winuv\SpectralData\Gina\johns work\JIJ UNK 1.DSW  
 Software version: 3.00(182)  
 Operator:

### Sample Name: UNK 1

Collection Time 1/6/2009 1:19:53 PM

Peak Table	
Peak Style	
Peak Threshold	Peaks
Range	0.0100
	800.00nm to 200.00nm

Wavelength (nm) Abs

589.00	2.470
555.00	2.480
450.00	1.554
373.00	1.175
324.00	1.605
321.00	10.000
317.00	10.000
312.00	10.000
309.00	0.172
305.00	10.000
301.00	10.000

Standard Name

328 of 328

Today's Date: 05-Jan-09

## unknown marker 1 green

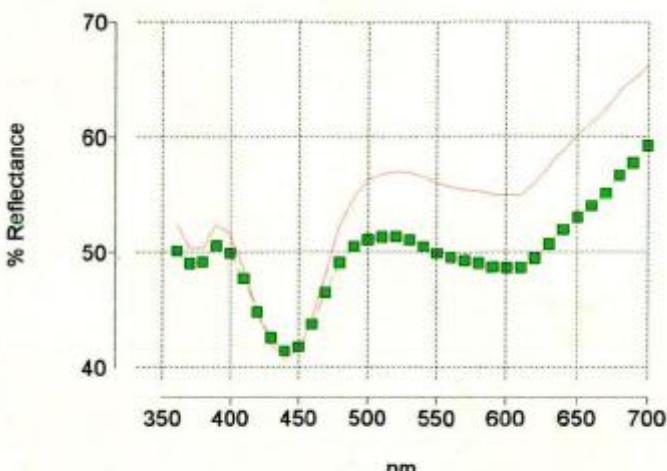
Batch Name

1 of 1

## marker 5 green1

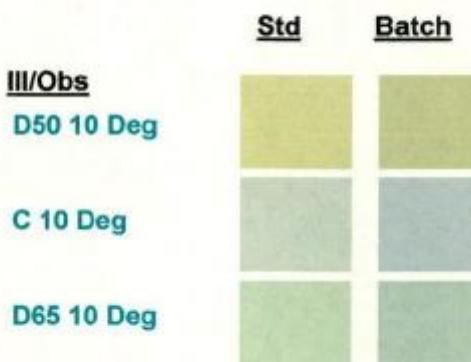
Date: 05-Jan-09

Time: 11:30:54

**P/F : Fail**

Batch is :  
darker  
less saturated  
more blue

P/F		<u>III/Obs</u>	<u>Decision</u>	<u>DE*</u>	<u>DL*</u>	<u>Da*</u>	<u>Db*</u>	<u>DC*</u>	<u>DH*</u>
D50 10 Deg	Fail	6.08	-3.42	0.49	-5.01	-4.91	1.07		
C 10 Deg	Fail	6.18	-3.35	1.15	-5.06	-5.08	1.07		
D65 10 Deg	Fail	6.13	-3.36	1.02	-5.02	-5.00	1.12		



CK

sy 37

Standard Name

327 of 327

Today's Date: 05-Jan-09

## unknown marker 1 red shade

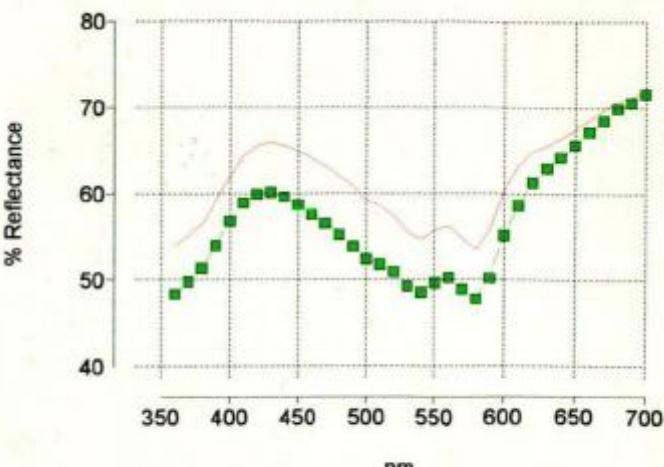
Batch Name

1 of 1

marker 5

Date: 05-Jan-09

Time: 11:25:37

**P/F : Fail**

Batch is :  
**darker**  
**more saturated**  
**more red**

P/F

<u>III/Obs</u>	<u>Decision</u>	<u>DE*</u>	<u>DL*</u>	<u>Da*</u>	<u>Db*</u>	<u>DC*</u>	<u>DH*</u>
D50 10 Deg	Fail	3.93	-3.28	2.15	0.17	1.48	1.57
C 10 Deg	Fail	3.89	-3.33	2.02	0.04	1.46	1.39
D65 10 Deg	Fail	3.91	-3.34	2.04	0.04	1.48	1.41



ck

—sv 38

—60627770

TITLE \_\_\_\_\_

From Page No. 27

Project No. \_\_\_\_\_  
Book No. \_\_\_\_\_Solubility Study

Objective: To survey additives that may increase the amount of Solvent Black 7 into solution.

~~Solvents & Dyes~~ Chemicals used

- Solvent 1

- Palmitic Acid (crystals)
- Oleic Acid (liquid)
- Stearic Acid
- Solvent Dye 7
- 4-hydroxybenzoic Acid
- o-toluic Acid

Technique: Dye + Additive added slowly to Solvent 1, then stirred 5-10 min.

The following chart indicates the dye mixtures

	1	2	3	4	5	6	7	8	9	10
Solvent Black 7	2.0	0.2	1.0	2.0	1.0	2.0	2.0	1.0	1.0	1.0
Palmitic Acid	0.1	0.1	0.05	0.05	0.05	1.0	0.5	0.2	—	0.1
Oleic Acid	—	—	—	—	—	—	—	—	—	—
<del>Boric Acid</del>	—	—	—	—	—	—	—	—	—	—
<del>Stearic Acid</del>	—	—	—	—	—	—	—	—	0.1	0.2
Solvent 1	20.0	20.0	20.0	20.0	20.0	20.0	20.0	20	20	20
	✓	✓	✓	✓	✓	✓	✓	✓	✓	✓

less clumping  
little sticking  
some clumping  
significant clumping  
significant sticking

Marker Trials

Objective: To attempt multi-component systems that match Solvent base markers.

Chemicals used:

Solvent Red 24

Solvent Yellow 42

Solvent Blue 36

Solvent 1 (from previous)

Solvent Orange 438

Solvent Red 207

Solvent Yellow 098

Solvent Blue 104

Solvent Violet 038

Witnessed &amp; Understood by me,

Date

Invented by:

Date

Recorded by:

To Page No. \_\_\_\_\_

Project No. \_\_\_\_\_

Book No. \_\_\_\_\_

TITLE \_\_\_\_\_

From Page No. 27

	1	2	3	4	5	6	7	8	9	10	C
Solvent Red 24										1.0	1.0
<del>Solvent Red 4</del>										—	—
Solvent Yellow 42								1.0	0.5	0.5	1.0
Solvent Blue 36								9.0	9.0	9.0	8.0
Solvent Orange 438								0	0	0	2.0
Solvent Red 207	1.0	1.0	0.5	0.8	0.5	0.5	0.5	—	—	—	—
Solvent Yellow 098	1.0	0.6	0.6	1.0	0	0	0	0	0	0	0
Solvent Blue 104	1.0	1.0	1.0	0	0.2	0.2	0	—	—	—	—
Solvent Violet 38	6.0	6.0	7.0	7.0	7.0	7.0	7.0	—	—	—	—
Solvent 1	20mL	20mL	20mL	10mL	10mL	20mL	20mL	20mL	20mL	20mL	50.

Results : See Data Color Scans

To Page No. \_\_\_\_\_

Witnessed & Understood by me,	Date	Invented by:	Date
		Recorded by:	

01/21/09

MIXTURES OF COMPONENTS FOR BLACK

8 ① ISR 24

1 sy 42

9 SB 36

20 Solvent 1

9 ② ADD

2 sy 42

10 ③ ADD

1 SR 24

11 ④ 8 SB 36

2 SO 438

50 solv. 1

12 ⑤ 8 SB 36

2 SO 438

0.5 SR 24

(1.2  
(.3 )

(0.075

.23

-

~~1.0~~ 542 solv. 1

1.6

.52

0.120/09

Starting base for making black from mixture

#

①

1 SR 207

1 sy 098

~~1.2~~ SB 104

6 SV 038

20 mL Solvent 1

③

.5 ~~SR~~ 207

.6 ~~sy~~ 098

1 SB 104

7 SV 038

20 mL Solvent 1

②

add 1 sy

④

.8 SR 207

1.0 sy 098

6 SB 104

7 SV 038

10 mL Solvent 1

⑤

0.5 SR 207

1.0 sy 042

~~0.2~~ SB 104

7.0 SV 038

10 mL Solvent 1

⑥

0.5 SR 207

0.5 sy 042

~~0.2~~ SB 104

7.0 SV 038

20 mL Solvent 1

⑦

0.5 SR 207

0.5 sy 042

0.0 SB 104

7.0 SV 038

20 mL Solvent 1

notes:

Change red

change violet

SV(-)

SR(-)

MoR Fast Black 108 (106-414-50)  
Solvent Black 3, 7, 5

MARKER Beaker 109.39 g  
#4

chk  
SB 46

\* Ink Dissolved in Xylene

\* Marker 3+4 possibly SB3 + SB27

MARKER BEAKER 49.98 g  
#1 INK (WHOLE) 0.20 g

Surplus

106-13-88  
106-14-50  
106-036-51

- 1) TLC → 1, 2, 3, 5, BS, B3 in Xylene
- 2) TLC → B3, 1, 2, B3 in Acetone

Bk 5 poor solubility  
Bk 3 close to b/k in marker 3



# KEYSTONE

TO: \_\_\_\_\_

FROM: \_\_\_\_\_ DATE: \_\_\_\_\_

60% Carbitol

2-2 Methoxypropanol

10% Glycol Ether DD (99-602-50)

5% Benzyl Alcohol (999-400-13)

7% Ethanol

82%

Xylene

1360

+

1 gall

~~73%~~  
~~12%~~  
~~6%~~  
~~8.5%~~

73.2%  
12.2%  
6.1%  
8.5%



## KEYSTONE ANILINE CORPORATION

2501 W. Fulton Street, Chicago, IL 60612 • (312) 666-2015 • (800) 522-4DYE • FAX (312) 666-8530

Phoenix, AZ: (623) 931-6442 • Inman, SC: (864) 473-1601

Huddersfield, U.K.: 01484 341466

[www.dyes.com](http://www.dyes.com)

**300**

1000

✓ 606 052 50

✓ 606 135 - 50

✓ 703 - 003 - 80 (41)

, 706-009

✓ 206-45

⇒ 44

806-093-50  
806-072-51  
806-053-50  
806-114-50

Try as 1% with

.05g/50mL + 5% or 10% SB

AB:

- USE 101-002-44 IN SB7 Blend
- ADD PALMITIC ACID

4

+

0.55

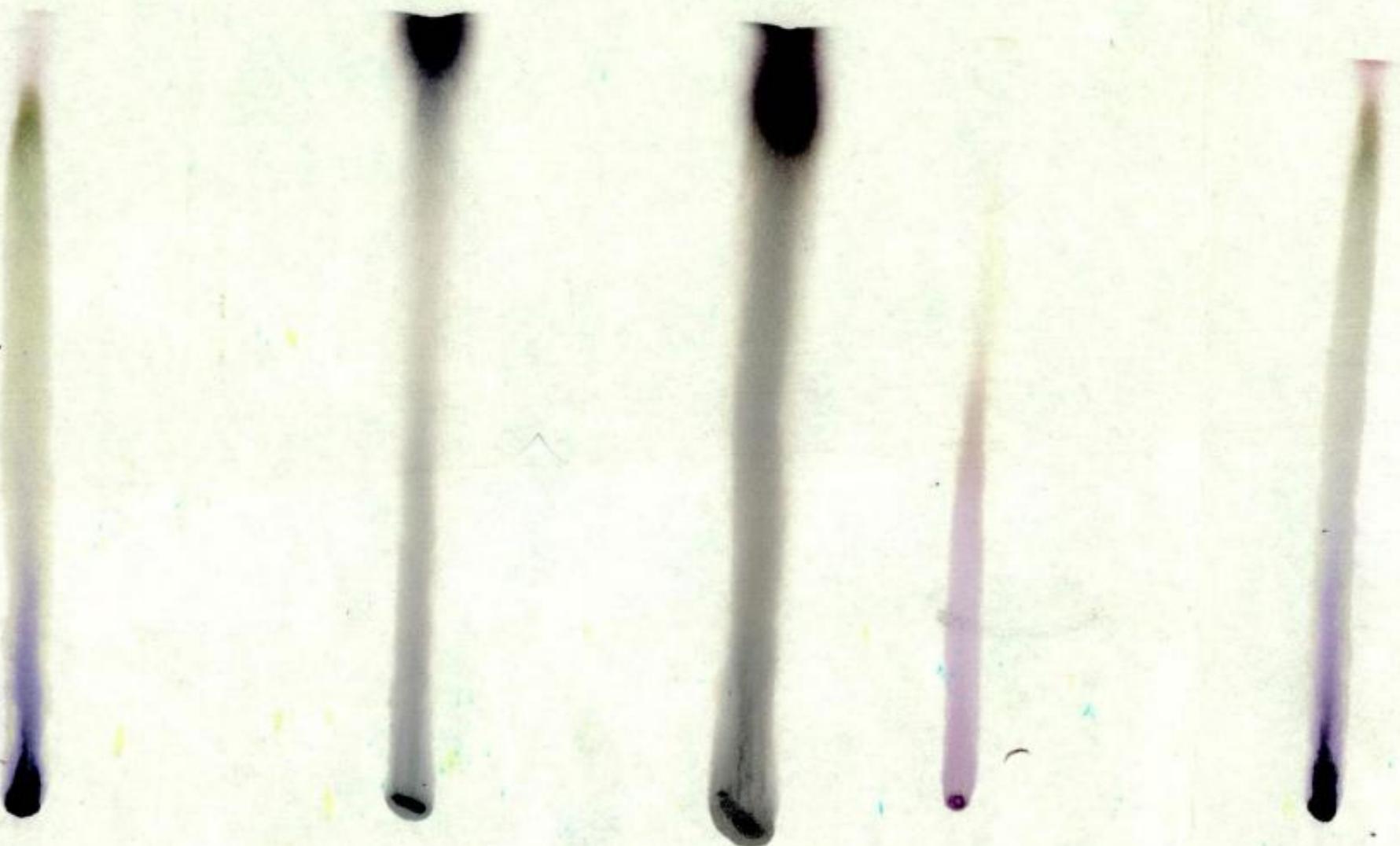
0.55

+

0.45

0.45

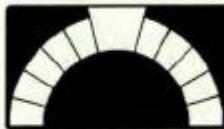
F



F







# KEYSTONE ANILINE CORPORATION

## Laboratory Report

### Reporting and Customer Information

KAC	Solubility of Solvent Black 5 in the provided Sanford ink system.			
Technician:	Chris Waring			
Project:	CMW000217		Status:	Done
Date Issued:	06/05/02	Date Completed:	06/05/02	Days Open: 1
Customer Name:	Sanford		Requested By:	Greg Godfryt
Customer Number:	723712		Phone:	
Contact:	Pat Nabor		Fax:	
Address:	2711 Washington Boulevard, Bellwood, IL, 60104			

#### Report:

Test samples of the Sanford standard plus Spirit Nigrsoine SSB and SJ - Solvent Black 5 (50 and 57 codes) in the provided Sandford ink system.

#### Procedure:

The above colourants were tested in the following ink system for their solubility and colouristic properties:

60.0% Cellosolve EE  
10.0% Cellosolve EB  
3.0% Benzyl Alcohol  
7.0% Ethanol  
10.0% Colour

The missing 10% was a resin of some nature that Sanford also added. For the purpose of the solubility test this was discounted from the overall formula.

#### Results:

The above ink mixtures were drawn down on to white Lenetta card. These were also reduced to a 5.0% ink and then drawn down again as the 10% formula showed little visual colour difference. (See attached draw downs)

#### DISCLAIMER

This data was obtained by testing according to our methods, with visual test deviations. Other test methods may give different results. Incoming evaluations, by the customer is therefore unconditionally recommended.

Out of the two Keystone samples, the 106-005-50 Spirit Nigrosine SSB material exhibited the worst solubility properties. This was closely followed by the Sanford material with the 106-005-57 Spirit Nigrosine SJ being the best of the three. Residual particles were present in both the Spirit Nigrosine SSB and Sandford material (DS-3091). Both of the Keystone samples were bluer in shade than the standard.

A TLC was run on the Sanford material to check for any added extras, but none were found. The Sanford material was closely matched by using the Spirit Nigrosine SJ shaded with 3.0% 306-012-50 (Bismark Brown Base RX) This mix appeared to be visually 10% stronger than the standard.

**Conclusion:**

The Spirit Nigrosine SJ was chosen to be submitted to Sanford because of its superior solubility properties.

A mix will be submitted to Sandford for their own testing and approaval, as the Bismark Brown may not possess the necessary lightfastness requirements.

Signed: \_\_\_\_\_  
Date: \_\_\_\_\_

**DISCLAIMER**

This data was obtained by testing according to our methods, with visual test deviations. Other test methods may give different results. Incoming evaluations, by the customer is therefore unconditionally recommended.