Electronics Park
Syracuse, New York
February 18, 1955

Subject: Lacquer Studies

The following report is the result of preliminary laboratory work done in attempting to set up a method of evaluating the nitrocellulose lacquer in present use.

Certain chemical and physical tests were performed on each of the three components of the lacquer, to arrive at some limitations on their acceptability.

This report is meant to serve only as a guide. Additional tests and refinements of existing procedures will be reported as they develop.

Victor Betar

Victor G. Betar

Paul L. Dee

Paul L. Dee
Materials and Processes
CATHODE-RAY TUBE SUB-DEPARTMENT

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EVALUATION OF SOLVENTS

Distillation

A 15 inch distilling column packed with unglazed porcelain is used. A controlled heat source (electric mantle) is necessary. A water cooled condenser will be satisfactory to condense the vapors.

Place 100 cc of the solvent into a round bottom flask. Drop in a few boiling chips. Assemble the remaining apparatus and apply heat. The column may have to be insulated (aluminum foil) to enable the vapors to reach the take off arm. As vapors start to condense, adjust the heat to allow a steady take off rate of about 2 drops per second. Record the temperature of the first drop and at intervals of 10cc until no further distillate comes over. Collect each 10cc fraction in a separate bottle. Additional heat may be required as the distillation progresses.

By comparing the specific gravity* of the solvent and the boiling range and refractive indecies of each fraction with the data compiled on known mixtures, it is possible to approximate the composition of the solvent. To be usable, the ester content should be 90% or greater.

For example, in determining the composition of an unknown solvent after the following information is obtained:

Initial Specific gravity - 0.864 at 23°C

1st drop	128°C	N^{25}_{D}
10	132	1.3945
20	134	1.3952
30	135	1.3959
40	136	1.3965
50	137	1.3968
60	137	1.3972
70	138	1.3976
80	139	1.3980
90	140	1.3984
Pot.	-	1.3988

The following analysis can be used:

Comparing the results with those on chart number 1, we find that the specific gravity compares favorably with mixtures 4, 5, and 6. This tentatively eliminates mixtures 1, 2, and 3 even though their fractions have refractive indecies fairly close to these in the unknown. The boiling ranges of 1, 2, and 3 also would be such as to disqualify their consideration.

Closer examination of the boiling range data on mixtures 4, 5, and 6 tends to indicate that the unknown compares closest to mixture number 5.

Therefore, first by comparing the specific gravity and then by further comparing the boiling range and the refractive indecies, a fairly close approximation of the unknown can be made.

It is quite evident from the data compiled on the various mixtures that:

1. The specific gravity tends to decrease as the alcohol content increases.
2. The refractive indecies of the first fractions are lower when the mixture is richer in iso butyl acetate or butyl alcohol.
3. The boiling ranges of the 90% ester content mixtures are in the 130-140 °C. neighborhood where as those of less ester content are in the 120-135°C range.

As a final check on the ester content of the solvent, reference should be made to the section on neutralization.

Description of Apparatus

15" distillation column Will Cat. #11966
20" water cooled condenser Will Cat. #9636
100 cc round bottom flask
25-200°C thermometer
100 cc Heating Mantle Will Cat. #15981
Unglazed porcelain
Collecting bottles
Abble Refractometer Will Cat. #23001
Constant Temp. Bath. Will Cat. #28521

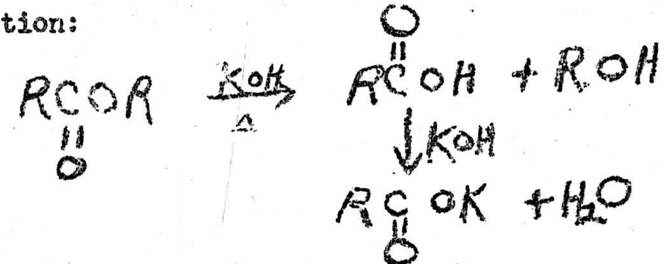
* The specific gravity was taken with an araometer at 23°C. Scale 0.8500-1.000

Ester Hydrolysis (Neutralization)

On an analytical balance, weigh out approximately 1 gram of solvent in a weighing bottle. Pour it into a 1000cc round bottom flask. Pipette 25cc of 1N KOH (alcoholic) into the weighing bottle and pour into the flask also. Rinse the bottle with 10cc of ethyl alcohol and add to the flask. Place the flask on a heating mantle and insert a vertical water cooled condenser into the mouth. Apply heat slowly and reflux for one hour taking care not to lose any of the vapors out the top of the condenser. Refluxing of about 1 drop per second is sufficient.

After refluxing for 1 hour cool to room temperature. Add a few drops of phenolphthlein to the flask and titrate with 1N HCL.

Reaction:



$$\underset{\overset{\|}{O}}{RC}OR \xrightarrow[\Delta]{KOH} R\overset{\overset{O}{\|}}{C}OH + ROH \xrightarrow{KOH} R\underset{\underset{O}{\|}}{C}OK + H_2O$$

Weight of ester = (mlKOH - mlHCl) Milli Equiv. Wt. Ester

$$\% \text{ Ester} = \frac{(\text{wt. of ester})}{(\text{wt. of solvent})} \; 100$$

Milli equiv. wt. of amyl acetate = .130

NOTE: Only valid when Normality of KOH = Normality of HCL.

Determination of Free Acid - (CH_3COOH)

Pour 200cc of solvent in a separatory funnel, add 200cc of deionized water. Shake vigorously for five minutes. Let stand to settle and collect the aqueous layer. Add a few drops of phenolphthalein and titrate with .05N NaOH.

Add a few drops of phenolphthalein to 200cc of deionized water and titrate with .05N NaOH as a blank.

$$\frac{\text{Grams of Acid}}{\text{liter of Solvent}} = (V_1 - V_2)\,N(NaOH)\,(MEq\ Wt)\ \frac{1000}{200}$$

V_1 = ml of NaOH to neutralize aqueous layer

V_2 = ml of NaOH to neutralize blank

N(NaOH) = Normality of NaOH

MEqWt - Milli equivalent weight of CH_3COOH

$$\frac{\text{Grams of Acid}}{\text{liter of Solvent}} = (V_1 - V_2)\ (.05)\ (.06)\ 5$$

The maximum acidity allowable is .01%

Moisture Content

An analysis of the percent moisture in the solvent is necessary to qualify its usefullness. A Carl Fischer titration is suitable for this determination. The Organic Section in the Electronics Laboratory is set up for such an analysis.

The maximum water content allowable is 0.3%.

Traces of Metal Impurities

Tests should be made to determine if metal impurities are present in the solvents. Special attention should be focused on Copper and Iron.

If this test shows more than a trace, an analytical analysis is necessary. The Inorganic Section in the Electronics Laboratory is qualified to do this.

EVALUATION OF LACQUER

% Solids Determination of Nitrocellulose Lacquers

On an analytical balance weigh a clean, dry, flat weighing dish with a ground glass cover (approximately 35ml capacity). Pour in approximately 10ml of lacquer and cover immediately. The lacquer depth should not be over 1/8 of an inch. Weigh on an analytical balance and record. Remove the cover and place the dish in a well ventillated oven set at 80°C for 48 hours. Remove and cool to room temperature. Weigh again and record.

Weight of dish = wt. 1

Weight of dish + lacquer = wt. 2

Weight of dish + lac. + 48 hrs. at 80°C = wt. 3

$$\% \text{ Solids} = \frac{(\text{wt } 3 - \text{wt } 1)}{(\text{wt } 2 - \text{wt } 1)} \; 100$$

Viscosity

A Brookfield viscosity determination on incoming lacquer should be taken. A #3 spindle at 30RPM is the proper range. Care should be taken to submerge the spindle to the proper depth and to keep the lacquer temperature at 25°C. The correct viscosity range is 1800-2200 centipoises.

EVALUATION OF NITROCELLULOSE

Because the lacquer is manufactured by an outside vendor, a sample of the alcohol wet nitrocellulose used in each lacquer batch should be obtained for the following evaluation:

1. **Falling Ball Viscosity Determination**

   The sample of nitrocellulose should be dried for this in a well ventillated oven set at 60°C and the viscosity determination can be made as per Hercules Nitrocellulose Handbook (ASTM D301-33). This gives the viscosity range of the nitrocellulose. The correct range should be 60-80 seconds.

2. **Infra Red Analysis**

   A sample of the alcohol wet nitrocellulose can be sent to Schenectady for infrared spectrographic analysis. This test will show any organic compounds in addition to the nitrocellulose. The sample would have to be dried of alcohol, then made up in a water free solvent to the proper solids concentration. A run on the sample in solution and the solvent alone would be required.

EVALUATION OF PLASTICIZER

A sample of the B-400 plasticizer, Carbon and Carbide, used in the lacquer, should be obtained from the vendor and should compare favorably with the following:

Specific gravity at 20°C	0.991 to 0.996
Refractive index at 20°C	1.4487
Flash point (open cup)	430°F
Maximum acidity	0.05 milliequiv. per gram
Maximum water content	0.25%

V Betar, Bldg. #6
WH Bertrand, Bldg. #6
J Gorman, Bldg. #6
IE Halt, BTP
ET Peterson, Cleveland

Electronics Park
Syracuse, New York
June 24, 1955

Mr. J. Dana
Chemical Products Plant
1099 Ivanhoe Rd.
Cleveland 10, Ohio

As you requested in your earlier letter submitted with the original lacquer sample, I am forwarding the viscosity measurements made in the factory.

The two liter sample you indicated had a viscosity of 45.3 seconds at 25°C with a no. 7 Parlin cup. With a no. 8 pipet we get a value of 25.9 seconds at 25°C. As you know, this turned out to be slightly below the range of 30-31 second lacquer that we currently are using in production.

To assist us in further viscosity standardization I would very much appreciate the following:

1. A 7% solids of the low viscosity (59-61 second R.S.) in H.B. amyl acetate.
2. A 7% solids of the medium viscosity (70 second R.S.) in H.B. amyl acetate.
3. A 7% solids of the high voscosity (80 second R.S.) in H.B. amyl acetate.

If you could take viscosity readings with the proper Parlin cup and then forward a two liter quantity of each of the three lacquers to us we will obtain our readings for correlation.

This will, I believe, allow us to more closely define our choice of nitrocellulose viscosity within the 60-80 second range.

Paul

P.L. Dee
Supervisor
Monochrome Chemical Engineering Unit
CATHODE RAY TUBE SUB-DEPARTMENT

PLD:jfe

W. H. Bertrand, Bldg. #6
V. Betar, Bldg. #6
J. M. Gorman, Bldg. #6
I. E. Halt, BTP
E. T. Peterson, Cleveland

Electronics Park
Syracuse, New York
June 15, 1955

Mr. J. Dana
General Electric Co.
Chemical Products Plant
1099 Ivanhoe Rd.
Cleveland 10, Ohio

As agreed in our telephone conversation this morning, the following plan of action on test lacquers will be followed.

A sample of 60-80 second nitrocellulose (7% solids) in high boiling amyl acetate will be the first objective. If filtering this 7% solution proves difficult then two different 4% solids solutions will be formulated, i.e., diluting a quantity of 7% in high boiling amyl acetate to approximately 4% with high boiling amyl acetate would be satisfactory for testing at the Syracuse Tube Plant. Similarly, dilution of a quantity of 7% in high boiling amyl acetate to approximately a 4% solids by using Pearce and Stevens lacquer thinner M-5925 would be necessary for evaluation at Buffalo.

It would be appreciated if we could receive small samples of both the HB and the M-5925 for our own distillation studies.

Paul L. Dee

Paul L. Dee
Supervisor
Monochrome Chemical Engineering Unit
CATHODE RAY TUBE SUB-DEPARTMENT

PLD:jfe

W Bertrand, #6
PL Dee, #6
W Highfield, #6
SS Sadowsky, #6
HN Sherwood, #6
LE Swedlund, #6
PN Taggett, #6

Electronics Park
Syracuse, New York
October 19, 1955

The following is a summary of the lacquer work done during the past four weeks. The effort has been concentrated on the improvement of the present lacquer (FAN Sy-1776), and also on a higher solids butyl acetate lacquer.

Shrinkage records of lacquers A through E are incorporated in daily factory records and the indication is that lacquer B has improved shrinkage somewhat, but it is still below the desired level.

The most promising of the Butyl Acetate lacquers is the 3.5% 18 sec in 44% butyl acetate and 56% amyl acetate with DBP in the ratio of 2.3 S to IP.

Further work is continuing presently on this lacquer.

Victor Betar

Victor Betar
Monochrome Chemical Engineering Unit
CATHODE RAY TUBE SUB-DEPARTMENT

VB:jfe

Formulas

Lac A.	1335 cc 6.2 % 18 sec Stock (AA) 465 cc Amyl acetate 720 cc Toluene 720 cc Butyl Acetate 720 cc Butyl Alcohol 32.4 cc DBP 21.6 cc OA
Lac B	Same as A less 465 cc Amyl Ac.
Lac C	1335 cc Stock 720 cc Toluene 720 cc Butyl Acetate 720 cc Butyl Alcohol 29.6 cc DBP 19.7 cc OA
Lac D	Same as C with 35.6 cc DBP and 19.7 cc OA
Lac E	40 cc Stock 20 cc Toluene 20 cc Butyl Acetate 20 cc Butyl Alcohol 1.86 cc DBP 1.11 cc OA

Lacquer Summary

Formula	TA @ 20 f.l.	% Solids	Plast	S/P DBP	S/P OA	Streak 451	Tear 452	Holes 453D	Bare Spots 453E	Blistering	Mottling	Sec. Grade Nitrocellulose
Lac A FAN Sy-1776	103	2.2	DBP OA	2.3/1	3.4/1						OK	18
Lac B	105	2.4	DBP OA	2.3/1	3.4/1						OK	18
Lac C	105	2.4	DBP OA	2.5/1	3.8/1						OK	18
Lac E (40-20-20-20)		2.5	DBP OA	2.4/1	4/1						OK	18
40-20-20-20 (1.24 DBP)	112	2.5	DBP	1.8/1	0	1/12	0/12	0/12	0/12		OK	18
40-20-20-20 (0.97 DBP)	100	2.5	DBP	2.3/1	0		yes	yes	yes		OK	18
100% Bu Ac	98	2.3	DBP	2.5/1	0	no	no	yes	no		OK	64
100% Bu Ac	89	2.3	TCP	2.5/1	0	no	no	yes	no		OK	64
100% Bu Ac	92	2.3	DBP TCP	2.5/1	0	no	no	yes	no		OK	64
80% Bu Ac 10% Am Ac		2.3	DBP TCP	2.3/1	0	no	no	yes	no		OK	64
80% Bu Ac 20% Bu OH		2.3	DBP TCP	2.3/1	0	no	no	mild	no		OK	64
63% Bu Ac 37% Am Ac		2.3	DBP TCP	2.3/1	0	no	no	yes	no		OK	64
44% Bu Ac 56% Am Ac	112	3.5	DBP	2.3/1	0	no	4/12	no	no	5/12	OK	18

V. Betar

BAKE OUT TESTS

The bake out tests were conducted inside a metal container on top of a hot plate controlled by a 16 amp. variac. The container was 4" by 6" by 3-1/2". There was no bottom or top to the container. The 4" by 4" plates tested were supported 1-1/2" from the hot plate by four bolts. The container was insulated with asbestos and aluminum foil. A thermometer was positioned 1-1/2" from the surface of the hot plate inside the container. A pyrex glass cover was used to view the bake out. One bolt was removed and an air hose attached to permit the air flush test.

As shown in Chart 2, the film seemed to start burning at approximately 185°C and usually had completed burning at approximately 205°C. The film usually would darken in one small circular area. This darkened area gradually increased in size until the entire plate was covered. The aluminized plates obscured this darkening, but the comparison of the gloss readings on Chart 1 between the control and the baked plates tends to indicate no hindering action due to the aluminum.

Microscopic examination was made after bake out in order to detect any signs of charring. Charring was noticed only on one plate that had a folded film. After baking out for 30 mins. at 205°C, a single charred streak was visible where the film was folded over. The unusual three-fold thickness of the film at this point prevented complete combusion, and the result was a charred streak. No other portion of the film charred.

The use of the Gardner gloss meter as a measure of film presence after bake out is based on the gloss difference between the screen and film (2 and 13).

No difference was noticed between the plate baked out with air flush and those baked without it.

Victor Betar

Victor Betar

VB:ch
5/12/55
cc: PL Dee
C Dichter
FN Taggett

CHART 1

	°C Temp	Time- Mins	Color	Gardner Gloss Meter
Screen only	--	--	Pale yellow	1.5 (Control)
Screen & film	--	--	Grayish yellow	13 (Control
	205	30	Pale yellow	2
(air flush)	205	30	Pale yellow	2
	180	30	1/2 Grayish yellow 1/2 Pale yellow	1/2 11 1/2 4
	230	30	Pale yellow	2
Screen and film & aluminized	--	--	Gray	11 (Control)
	205	30	Bluish gray	7
	300	30	" "	8
	200	30	" "	9
	230	30	" "	7

CHART 2

Screen and film	(Mins.) Time	°C Temp	
No Air Flush	5	63	
	10	69	
	20	78	
	30	85	
	35	154	
	37	179	
	38	185	- Film started to darken
	39	190	
	40	195	- Entire film darkened
	45	196	
Air Flush	5	60	
	10	150	
	15	180	- Film started to darken
	20	195	
	25	205	- Entire film darkened
Screened, filmed, and aluminized	5	50	
	10	150	
	15	175	- Could not see any darkening
	20	200	
	25	210	
	5	50	
	10	135	
	15	180	- Could not see any darkening
	20	220	
	25	270	
	30	300	

W Bertrand, Bldg. #6
PL Dee, Bldg. #6

Electronics Park
Syracuse, New York
August 31, 1955

Mr. J. Dana
Chemical Products Plant
1099 Ivanhoe Road
Cleveland 10, Ohio

Per cent solids determinations and viscosity measurements were made on your lacquers and I am sending you my findings. The per cent solids of the undiluted lacquers were determined by weighing analytically a small portion, and then by reweighing after oven drying for 48 hours @ 80°C. Since my values did not correspond to yours, a second check was made and dried for 96 hours @ 80°C with no change. The per cent solids of the diluted lacquers were calculated values after using the standard factory dilution ratio of 1650 cc stock to 2350 cc of amyl acetate.

As can be seen from the data, the regular factory lacquer (60-80) when diluted from 1650 cc of 7% stock with 2350 cc of amyl acetate is 2.88%. This lacquer gives a 60 cp. reading @ 25°C. The three diluted lacquers 3831-59, 69, and 78 all were near 90 cp. @ 25°C.

The present factory filming conditions have favored a fast dry lacquer and all attention has been focused on this. I have tested a series of butyl acetate lacquers and I am sending you the results also. If these lacquers continue to be as good under further testing we may ask your assistance in formulating them.

Victor Betar

Victor Betar
Monochrome Chemical Engineering Unit
CATHODE RAY TUBE SUB-DEPARTMENT

VB:jfe
attach.

VISCOSITY CHECKS ON CLEVLLAND LACS.

As Received	Clev. % Solids	Syr. % Solids	Cleve. Visc.	Syr. Visc.	Graphic % Solids
3831-59	7.02	7.60	29.4 Sec #20 @25°C	2184 cp.★ @ 25°C	—
3831-69	6.95	7.47	30 Sec #20 @25°C	2160 cp.★ @25°C	—
3831-78	6.87	7.20	42.5 Sec #20 @25°C	1768 cp.★ @25°C	—
Reg. Fac. Lac.	—	2.88	—	32.5 Sec #8 Pipette @25°C 60 cp.@25°C	—
Diluted					
3831-59	—	3.13	—	44.8 Sec #8 Pipette* 91.5 cp. @22°C ⊖	3.26 @22°C
3831-69	—	3.05	—	47.2 Sec #8* 94.5 cp @22°C ⊖	3.30 @22°C
3831-78	—	2.83	—	40.6 sec #8* 86.5 cp @22°C ⊖	3.18 @22°C

* @25°C

★ #3 spindle @30 RPM

⊖ #2 spindle @60 RPM

The lacquer as received from Cleveland was diluted in the usual factory dilution ratio of 1650cc stock lac to 2350 cc of solvent.

The graphic % solids determination was based on previous work where 75 sec nitrocellulose in amyl acetate was plotted showing % solids vs. viscosity (cp.)

BUTYL ACETATE LACQUERS

(ua) Brightness	Plasticizer	S to P	% Solids	B-flies	Holes	Swirls	cc of Lac.
—	B-400	1.4 to 1	2.5	No	Yes	No	1.0
—	"	"	"	No	Yes	No	1.0
—	B-400	2.5 to 1	2.3	Yes	No	"	1.2
—	"	"	"	Yes	"	"	"
—	"	"	"	Yes	"	"	"
110	B-400	1.3 to 1	"	No	"	"	"
107.5	"	"	"	"	"	"	"
—	DBP	2.5 to 1	"	"	Yes	"	"
102	"	"	"	"	No	"	"
94	"	"	"	"	No	"	"
89	TCP	"	"	No	No	"	"
—	"	"	"	Yes	"	"	"
95	TCP + DBP	"	"	No	"	"	1.3
90	" "	"	"	"	"	"	1.3
91	" "	"	"	"	Yes	"	1.3

Note: 64 sec. R.S. nitrocellulose was used in all lacquers. TCP + DBP was mixed 1 to 1

Terminology:

B-400 - Flexol B-400 plasticizer
DBP - Dibutyl phthalate
TCP - Tricresy phosphate
S to P - Solids to plasticizer ratio
B-flies - butterflies or bare spots (incomplete coverage)

4 - Development Studies of Flotation Filming Lacquers.

a) Tank Studies on Lacquers - P. L. Dee, V. Betar

A 24 x 24 inch stainless steel tank with a 15-1/2 x 20 inch elliptically shaped frame insert was constructed for the purpose of testing lacquers. The boundary of the insert was as similar as possible to that encountered by the lacquer during the regular film spreading. The depth of the tank was four inches, and the depth of the water cushion used was one inch. The tank was covered by a plexiglass lid with a center hole for lacquer dispensing.

In testing the various types of lacquers, one inch of cushion water (six liters) at 23°C was used. One cc of lacquer was hand dispensed with a pipette. The dry time noted is when a wet glass rod will depress the film 1/4 of an inch without puncturing it and without the film sticking to the rod.

Variations of the following lacquers were tested:

(1) 4.9% R.S. 18 sec. nitrocellulose in amyl acetate A.R.
(2) 5.4% R.S. 36 sec. nitrocellulose in amyl acetate A.R.
(3) 5.2% R.S. 75 sec. nitrocellulose in amyl acetate A.R.
(4) 4.9% R.S. 145 sec. nitrocellulose in amyl acetate A.R.

All of the above were plasticized with dibutyl phthalate in the ratio of 1.8 to 1, solids to plasticizer. In certain lacquers, the use of 0.5 cc of octyl acetate per 100 cc of working solution was added for comparison.

The evaporating rate recorded on the data is based on the rate of n-butyl acetate equal to 100.

The above stock solutions were prepared from dried nitrocellulose.

Conclusions:

From the preliminary testing of lacquers made from various grades of nitrocellulose, the following trends are noticed:

Lacquers of high solids content are faster drying than those of lower solids content regardless of nitrocellulose grade.

For each grade of nitrocellulose an optimum percent solids value exists where the spreading area will be the greatest. This is the point where the spreading pressure minus the retarding effect of edge drying is at its maximum.

Octyl acetate in general increases the spreading area of a lacquer and increases its dry time.

Of the lacquers tested, the following have been chosen for more detailed study:

1.	4%	18 sec.	3.	4.5%	75 sec.
2.	4.5%	36 sec	4.	3.5%	145 sec.

LACQUER					AREA (Sq.In.)		
R.S. Grade	% Solids	Visc (Cps.)	Evap. Rate	Dry Time (Mins.)	Width	Length	Area
18 Sec.	4.9	92.5	59	4	15-1/2	16	248
18 "	4.0	52.5	59	7	15-1/2	19	294
18 "	4.0	52.5	58	11	15-1/2	19	294 *
18 "	3.0	27.5	59	7	15-1/2	17	263
18 "	2.5	17	100	(Rauland Lac)	15-1/2	20	310
36 "	5.4	338	59	3	15-1/2	17	263
36 "	4.5	220	59	8	15-1/2	16	248
36 "	4.5	220	59	11	15-1/2	19	294 *
36 "	4.0	147	59	7	15-1/2	15	232
75 "	5.2	450	59	3	15	17	255
75 "	4.5	280	59	4	15	18	270
75 "	4.5	280	59	11	15	19	285 *
75 "	4.0	170	59	11 *	15	19	285
75 "	2.9	60	70	7	15	19	285 *
145 "	4.9	556	59	3	14	15	210
145 "	4.0	270	59	-	15-1/2	19	294
145 "	3.5	175	59	11	15-1/2	19	294
145 "	3.0	105	59	10	15-1/2	20	310
145 "	2.0	35	59	14	14	16	224

* Added 0.5cc of actyl acetate per 100cc of lacquer.

b) Aging Effect on Lacquer - P. L. Dee, V. Betar

The method used to evaluate the effective power of various ester solvents on nitrocellulose is the toluene tolerance test. Nitrocellulose lacquers from such solvents can tolerate additions of toluene (a non-solvent) up to a certain point, after which gelling occurs. Localized gelling takes place at the tip of the burette delivering toluene to the mixture, even though mechanical stirring is used. As an arbitrary end point, a one-minute time limit was set on the disappearance of the gel. That is, toluene was added until the first signs of gelling, then, one ml. additions were added every minute, with stirring, until the gel failed to disappear.

Toluene tolerance titrations were made on six types of nitrocellulose lacquers diluted to a 3% solids consistency with:

1. Pierce & Stevens lacquer thinner.
2. Commercial solvents Amyl Acetate (H.B.)
3. Malinkrodt Amyl Acetate (A.R.)

The tests attempted to find the effect of time upon the solvency power of each solvent used.

Each of the lacquers was made to a 7% and 4% solids content with 70 sec. nitrocellulose (R.S.) in each of the three solvents above. The 7%

solids lacquer was chosen to approximate the present incoming lacquer from Raffi and Suanson. The 4% solids lacquer was chosen for comparison tests.

At various intervals, these stock lacquers were diluted to a 3% solution with each solvent type. A 25cc amount of each of these 3% solutions was then titrated with toluene as described. The first titration (0 days) was made after the stock lacquers were mixed and had rolled for 72 hours. The time schedule of the other titrations was based on the first titration being zero days.

Brookfield viscosity readings were taken on the stock solutions at the time of each toluene titration.

Toluene titrations of 7% stock lacquers diluted to 3% with methyl ethyl ketone and methyl isobutyl ketone were also taken, and the results are listed in the data.

Conclusions:

No pronounced effect is noticed in toluene titrations on lacquer solutions for the duration of the test. The trend, however, is for the titrations to level off after the second week. The viscosities recorded seem to fall into this same trend except for the readings at 27 and 57 days. The unexpected increase in the viscosities of the 7% lacquer at 27 days was probably due to an increased submersion of the Brookfield spindle. The slight decrease in the 7% lacquer readings at 57 days was probably due to a 1/2 degree temperature reading error.

The leveling trend of the toluene titrations and viscosities after two weeks indicates that this period is the minimum time requirement for the complete solvency of the lacquer. A period of one month would provide a slight safety factor.

The 7% to 3% dilution seems to produce slightly higher toluene tolerances than the 4% to 3% dilution. However, the lack of erratic fluctuations in the 4% to 3% tolerance and viscosity readings would favor the use of a 4% stock lacquer as the incoming material.

Based on the viscosity tests, the Pierce and Stevens lacquer thinner, which is approximately two parts amyl acetate to one part butyl acetate, would appear to be the more active solvent of the investigation.

Based on the toluene tolerance tests, much better solvent action can be expected from faster evaporators such as methyl isobutyl ketone and methyl ethyl ketone, but their effects on a lacquer film would limit their usefulness. The analytical reagent amyl acetate would more often impart a slightly greater tolerance than either the Pierce and Stevens thinner or the commercial solvents amyl acetate. The differences become less and less after two weeks. The choice of solvent, assuming uniformity from barrel to barrel, could easily be determined by current price quotations.

	0 Days		5 Days		13 Days		27 Days		57 Days		120 Days	
	Visc. 20°C	Ml. Tol.	Visc. 20°C	Ml. Tol.	Visc. 21.5°C	Ml. Tol.	Visc. 22°C	Ml. Tol.	Visc. 23°C	Ml. Tol.	Visc. 22°C	Ml. Tol.
7% P&S	2600	*	2340		2280		2320		2120		2260	
3% with P&S		64		68		68		72		70		73
3% with CSAA		66		66		66		70		68		69
3% with AA		68		68		68		70		68		68
7% CSAA	2860		2640		2584		2640		2460		2540	
3% with P&S		64		68		68		69		68		69
3% with CSAA		65		66		66		67		67		69
3% with AA		70		70		70		70		69		69
7% AA	2772		2608		2496		2500		2320		2440	
3% with P&S		70		70		68		69		68		68
3% with CSAA		66		67		66		67		66		68
3% with AA		70		70		68		69		69		69

*Viscosity in centipoises

	0 Days		5 Days		13 Days		27 Days		57 Days		120 Days	
	Visc. 20°C	Tol.	Visc. 20°C	Tol.	Visc. 21.5°C	Tol.	Visc. 22°C	Tol.	Visc. 23°C	Tol	Visc. 22°C	Tol.
4% P&S	363		355		342		340		300		327	
3% with P&S		61		64		64		66		65		66
3% with CSAA		62		65		64		66		65		67
3% with AA		64		66		66		68		66		68
4% CSAA	430		412		408		396		360		387	
3% with P&S		63		64		64		65		64		66
3% with CSAA		63		63		63		64		63		68
3% with AA		62		63		63		64		62		66
4% AA	405		380		356		355		330		342	
3% with P&S		61		64		63		64		63		65
3% with CSAA		64		65		64		64		62		67
3% with AA		66		67		66		66		64		67

Viscosity Conversions to 20°C

	0 Days	5 Days	13 Days	27 Days	57 Days	120 Days
7% P&S	2600	2340	2395	2470	2355	2410
7% CSAA	2860	2640	2699	2790	2695	2690
7% AA	2772	2608	2611	2650	2555	2590
4% P&S	363	355	342*	340*	300*	327*
4% CSAA	430	412	408*	396*	360*	387*
4% AA	405	380	356*	355*	330*	342*

Note: Readings are in centipoises.

P&S = Pierce & Stevens lacquer thinner
CSAA= Commercial solvents amyl acetate (H.B.)
AA = Malinkrodt amyl acetate (A.R)

*These readings are at the temperatures listed on page 13.

Stock	Dilution	With	0 Days ml Toluene	6 Days ml Toluene
7% CSAA	3%	50-50 IPA + MEK	104	
7% AA	3%	"	105	
7% P&S	3%	"	108	
4% CSAA	3%	"	84	
4% AA	3%	"	85	
4% P&S	3%	"	86	
7% CSAA	3%	MEK	118	120
7% AA	3%	MEK	121	123
7% P&S	3%	MEK	120	122
7% CSAA	3%	MIBK	98	100
7% AA	3%	MIBK	103	104
7% P&S	3%	MIBK	102	104

Note: 25 ml samples were used in titrations.
IPA = Iso propyl alcohol
MEK = Methyl Ethyl Ketone
MIBK = Methyl Iso butyl Ketone

c) Combined Screening and Filming - F. L. Dee

Through a period of nearly four weeks of three-shift operation a filming defect, bare spots in the corners of the screen, has been the largest single cause of shrinkage.

From extensive observations of this defect at the pouroff platform, the film appears wet in the center and dry along the edges. In other words, when spreading the film on what is in effect an elliptical shaped plane, the lacquer adequately covers an area out to the end of the minor axis. At this point the lacquer reflects off the glass wall on each boundary of the short axis and partically folds or runs back on itself. This pattern which has been observed at lacquer dispensing leads to irregular areas on the pouring edge but more harmful is the extra thickness of film and its wetness. In contrast, the lacquer just spreads out to the extremities of the major axis and in so doing is thinned out sufficiently to be considerably drier than lacquer at the end of the minor axis.

The low viscosity and volatile solvents in this RAULAND lacquer actually accentuate a condition that is present with any lacquer cast on this type of plane. The center to edge wet and dry variation is so magnified, however, with this lacquer that normal tolerances are sharply reduced.

As a result, the water to pipette distance, the timing and quantity of lacquer, referencing of dispenser and other variables, such as temperature, require degrees of exactness that are becoming more extreme as additional process knowledge is obtained.

In attempts to keep within the present system, some eight minor changes have been made in the current lacquer. These involved small variations in plasticizer or ratios of stock solution to diluting solvent. It is significant that in all such lacquer modification tests the resultant product was poorer than the lacquer it was meant to correct.

It would appear then that a different type lacquer could be of value in allowing wider tolerances in actual processing. Development work for some has been directed toward this goal, and as promising lacquers arise, they will be tested under actual process conditions.

5 - Color and Brightness Control - F. D. Gordon

a) Phosphor Lot Approval.

Seven phosphor lots were tested for color and brightness during March for both the Syracuse and Buffalo Tube plants. As per the S.I., readings of four tubes per 2,000 lb. lot were made using the laboratory spectroradiometer. The results are listed below--all readings are average values of the CIE color coordinates and brightness.

PROGRESS REPORT NO. 6 (D.A. 30113) June 24, 1953

TITLE: Filming Investigations - Triphenyl Phosphate Plasticizer

PERSONNEL ASSIGNED: Paul L. Dee

PROGRESS FROM: January 15, 1953 to February 27, 1953.

FOREWARD:

This Progress Report is a compilation of past work using Triphenyl Phosphate Plasticizer. Although separate reports have been issued covering the majority of this work, the data is collected here for review and future planning.

The accompanying sheet shows the brightness and life test results on special aluminized 27EP4's that had been flotation filmed with lacquer containing 0.4 gms. of Tri-Phenyl Phosphate Plasticizer per 50 cc's of solution. The control tubes, also 27EP4's, had the regular B-400 Plasticizer in the lacquer (1.0 c.c. per 50) for their filming operation.

This work was prompted by the reduced brightness of factory 27EP4's, a condition that was first suspected by noting the excessive mottling in the film at light inspection. It was believed that the B-400 Plasticizer which takes up 3.3% water by weight was the principal cause for film blemishes and decreased brightness in a finished tube. Our present lacquer suppliers, Raffi and Swanson, had suggested Tri-Phenyl Phosphate among other plasticizers as a possible means of getting around this water absorption problem. The increased brightness with Tri-Phenyl Phosphate lacquer bears out the wisdom of their suggestion.

There are, however, certain processing problems that have arisen in using lacquer containing this new plasticizer. A slightly thinner film is cast when the same volume of lacquer that is used with B-400 solutions is dispensed, judging from the interference colors at pouroff. Increasing the amount of lacquer in part compensates for this, but the Tri-Phenyl Phosphate films set up much faster and are ready for pouroff of cushion water in approximately two-thirds of the time that B-400 films require. Literally presaturating the water 1-2 seconds before dispensing this new plasticized lacquer did not lengthen the setting time sufficiently. For this reason small amounts of octyl acetate were added to the lacquer to increase the set up time. While this proved successful, it resulted in a drop in brightness. Additional work with the octyl acetate (Specials 4 & 5) would indicate there is an optimum amount for lacquer containing the 0.4 gms. of Tri-Phenyl Phosphate that will yield consistently bright tubes.

This rather roundabout approach to convenient set up time has been necessary because of limitations at where lacquer can be dispensed on the conveyor.

Future work is planned for increasing the amount of Tri-Phenyl Phosphate and also continuing a study of octyl acetate. A larger volume of tubes will also bear out or refute an unanswered question on film flexibility with the Tri-Phenyl Phosphate.

Life test racks were very limited during the course of these tests (Jan. 20, 1953 to Feb. 27, 1953) and while the controls were not put on life, regular factory production life test results are listed here for comparison.

Paul L. Dee
Materials & Processes
CATHODE-RAY TUBES

PLD:f

Distribution: VCC
A.F.Carl - B.T.P.
GLC
CHD
HJElias - B.T.P.
IEHalt - B.T.P.
JW Wright - B.T.P.

SUMMARY OF PAST WORK USING TRI-PHENYL PHOSPHATE PLASTICIZER IN FILMING SOLUTION

27EP4

	I_b @ 20 ft.l.		500 Hrs.					
	0 Hrs.	500 Hrs.	Cut Off	I_s	50V I_s	% Orig. I_s	Gas Ratio	Screen
SPECIAL-1	106 ua	115 ua	46	1100	1254	95	.005	OK
" -2	120	130	50	1150	1150	88.5	.02	OK
3	104	110	36	760	1246	92.6	.02	OK
4	133	-			Not On Life			
5	120	-	54	1220	1086	86.1	.22	OK
CONTROL-1	135	-			Not On Life*			
2	126	-			" " "			
3	126	-			" " "			
4	145	-			" " "			
5	145.5	-			" " "			

*Life Test Racks Not Available.

All Specials had 0.4 gms. of Tri-Phenyl Phosphate per 50 c.c. of lacquer.
Special - 4 in addition had 2.0 c.c. of octyl acetate per 50 c.c. of solution.
Special - 5 in addition had 1.5 c.c. of octyl acetate per 50 c.c. of solution.

Regular Factory Production 27EP4's Showed the Following Life Test Results:

	Hours	50V I_s	% Orig. I_s	Gas Ratio	Screen
Jan. Production	788	1311	98.7	.01	OK
Jan. Production	1100	1280	93.7	.01	OK
Feb. Production	805	1295	98.8	.01	OK
Feb. Production	306	936	71.7	.01	OK