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PARLIN PLANT LABORATORY REPORT NO. 28

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THE DETERMINATION OF GLOSS OF FLATTED "DULUX" AND "DUCO" FURNITURE FINISHES

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PREPARED BY: George P. McAndrews
GEORGE P. MC ANDREWS

APPROVED BY: R H Bell
Plant Manager

C B Sheridan

S. H. Reindl

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PARLIN PLANT LABORATORY REPORT NO. 28

THE DETERMINATION OF GLOSS OF FLATTED "DULUX" AND "DUCO" FURNITURE FINISHES

INTRODUCTION: (Condensed in part from Parlin Plant Laboratory Report No.14, February 1, 1956, by C. N. Swinehart)

Since 1949 the standard method for determining gloss of furniture finishes has been that outlined in TM-92-D. Briefly, this method consists of pouring the material on a black glass plate, drying and baking in a prescribed manner, and finally reading the gloss by use of a Universal Glossmeter.

In 1956, as a result of tests conducted by C. N. Swinehart, the basic TM-92-D gloss test was modified in an attempt to achieve better reproducibility. Basically this modification added a one-half hour flash in a constant temperature-humidity room to the test and eliminated a 15 minute open-oven drying step.

In spite of these modifications, experience at the Parlin Plant and Control Laboratories has shown that TM-92-D, as presently constituted does not satisfy requirements for a test both reproducible and accurately indicative of the gloss level experienced when the product is applied to furniture in the field.

Because of the apparent unreliability of the TM-92-D test procedure, much time is lost in production by hold-up of a plant batch to allow for more extensive testing and retesting. There are also cases of remixing completed batches because the last can sample or a sample during the take-out will not check for gloss. It is believed that much of this is due to an unreliable testing procedure in which certain critical variables are not controlled.

The Sales Department has complained that gloss level determinations arrived at by using TM-92-D are generally 10 to 15 points lower than the actual gloss level obtained on furniture in the field. This discrepancy is attributable to inherent characteristics of the present test method.

An additional factor influencing the search for a better test method is the fact that no constant temperature-humidity room is available in the Parlin Control Lab and thus the Control Lab technicians must use the facilities located in the Plant Lab for the test.

OBJECTIVES:

The objectives of this work were:

1. Modify the present test or devise a new test procedure that will permit accurate reproduction of gloss values within narrower limits than those presently obtainable using TM-92-D.

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OBJECTIVES (cont'd)

2. Modify the present test or devise a new test procedure that will permit achieving gloss values in the range presently obtained when the "Dulux" or "Duco" flat is applied to furniture in the field.

3. Subject to the above two objectives try to eliminate the need for a constant temperature-humidity room in the test procedure.

SUMMARY AND CONCLUSIONS:

1. Experimentation under actual Control Lab conditions shows that the present tolerance limits of ± 2 gloss units for the TM-92-D test are unrealistic.

2. A gloss test utilizing an automatic Bird Film Applicator does make possible a test with 95% experimental confidence limits of ± 1.964 at a gloss level close to that experienced by the product in the field. Quality control difficulties arising solely from the test procedure could still be expected from:

a.) Statistically 1 in 20 samples will fall outside these limits.

b.) Less than strict adherence to the test procedure would give faulty results.

3. It is not deemed advisable under present tolerance limitations to eliminate the use of a Constant Temperature-Humidity Room.

A. RECOMMENDATIONS:

1. It is recommended that the following test procedure for gloss be adopted: (See Appendix A)

a.) Panels should be prepared under constant temperature and relative humidity conditions.

b.) Samples should be prepared with attention to preventing the formation of air bubbles. (Spatula mixing of accelerator rather than agitation by shaking.)

c.) Automatic Bird Film Applicator with a .007" drawdown blade should be used.

d.) Panels should be flashed in a horizontal position in the Constant Temperature-Humidity Room for 30 minutes in an area free from air currents and accumulated foreign vapors.

e.) Panels should then be baked for one hour at 65°C. in a Precision Scientific Co. Forced Draft Oven. The panel should be placed horizontally on a solid sheet metal shelf and covered on four sides with a shield to minimize draft variations.

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A. RECOMMENDATIONS: (cont'd)

2. Gloss limits should be extended to ± 3 units. It is felt that product quality will not suffer if this step is taken since a practicable testing procedure for gloss of furniture flats is inherently variable as shown in this report. Costly testing and retesting would be eliminated with no sacrifice in the quality of the product.

3. If tolerances cannot be extended to ± 3 units it is recommended that the present limits of ± 2 units be retained and each time a gloss sample falls outside the ± 2 limits but within ± 3 units an additional test sample be made before making any gloss-changing additions to the batch.

B. DISCUSSION:

1. Statistical Evaluation of TM-92-D.

a.) Tests conducted over the period 6/15/60 to 8/30/60 using a test batch of RF-5283, serial J-921400, with a standard gloss of 25.84, have demonstrated that present tolerance limits of ± 2 gloss units for "Dulux" and "Duco" furniture flats are unrealistic. Only under carefully controlled experimental laboratory conditions is it possible to achieve 95% confidence limits within this tolerance. During this period optimum Control Lab techniques were utilized and with close adherence to the TM-92-D procedure, a standard deviation of 1.064 was the best that could be attained. This value alone would place the 95% confidence limits outside the present tolerances and, even if the limits were extended to accommodate these values, one sample out of 20 could be expected to give a faulty picture of the gloss level of a batch. According to statistical information gathered by C. N. Swinehart (Parlin Plant Laboratory Report No. 14), the standard deviation of TM-92-D increases with increasing gloss level. Since the test batch of RF-5283 used in this experiment has a gloss value (25.84) that falls close to the level for minimum standard deviation as determined by Swinehart, an increase in the confidence limits at higher gloss levels could be anticipated. This should convincingly demonstrate that desired reproducibility within the present tolerance values is impossible.

2. Variables influencing gloss determination.

Swinehart's work accurately and effectively demonstrated the dependence of gloss on the temperature and relative humidity of the testing area. In an effort to isolate other factors influencing the gloss level of a sample, extensive testing has shown that gloss is heavily dependent on:

a.) Uniformity of film build.
b.) Thickness of dry film build.
c.) Length of flash time.
d.) Type of oven used for bake and location of panel in oven.
e.) Existence of entrapped air in the test sample.
f.) Number of other test panels in the baking oven.
g.) Gloss of glass test panel prior to application of sample test film.

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B. DISCUSSION: (cont'd)

a. Uniformity of Film Build

No effective method of measuring film uniformity is available at the Parlin Plant Lab. Since gloss is inherently dependent on film uniformity, it is reasoned that the pour method of film application, relying on air flashing from a vertically positioned panel, is not the most suitable for quality control work. Other experimenters bear out this conclusion and, as a result, the automatic Bird Film Applicator has been widely adopted in industry to achieve uniform films. (ASTM Bulletin, March, 1948, pp. 95-96) No valid evaluation of film uniformity achieved by the Bird Film Applicator as compared with that achieved by the pour method has been formulated. However, work on uneven films achieved by dipping the test panel in the sample has shown the advantage of an automatic drawdown blade in achieving reproducible results.

b. Thickness of Dry Film Build

Tests have demonstrated that gloss determinations are very sensitive to variations in dry film thickness. (See Appendix C, pp. 10) In the lower film build range, gloss varies more than one gloss unit with each .1 mil increase in dry film. This rate of increase levels out at higher film builds. A 7 mil Bird Film Applicator was selected because it gives a dry film build of approximately 1.6 mils (dependent on % solids) which is near the plateau of gloss sensitivity, while not being so heavy a wet film as to allow excessive run-off of the wet sample on the glass plate. Tests using 1.5, 3.3, 4.2, 5, 6, 7, and 10 mil Bird applicators show that the 7 mil applicator provides the best reproducibility.

Sales states that the actual dry film build used in the trade is between 2 and 2.5 mils. Of the four methods tried: pouring, dipping, spraying, and casting; only spraying enables the securing of a uniform film build in this range. Only an automatic spray machine gives uniform films using this method, but tests show that even the degree of uniformity possible here is not satisfactory for gloss testing. The mere cleaning of the spray booth exhaust vents was sufficient to lower the final gloss of the test panels approximately 8 points. Due to an increase in air flow, flashing was more rapid with consequent decrease in gloss.

c. Length of Flash Time

In an effort to eliminate the one-half hour flash time in the constant temperature-humidity room, tests were run in which the panel was placed in a 65°C. draft oven immediately after application of the wet film. It was reasoned that since the relative humidity of air at this temperature is fairly constant, the need for the CTR would disappear. It is felt that basically this idea is sound but effects introduced by oven variables adversely affect test results. (See paragraph (d) below).

The physical separation of the two laboratories at the Parlin Plant and the necessity to make three trips for each test exactly at the proper time, introduces a potential variable that cannot be overlooked. An increase or decrease of 5 to 7 minutes in the Constant Temperature-Humidity Room flash time results in a gloss change of 3 to 5 units. Increasing the flash time lowers the gloss. (See pp. 7, Section II)

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Efforts to render the sample less sensitive to flash time requirements by the addition of a retarding agent, (H-224) were unsuccessful. Tests show that the use of this agent makes the test results even more unpredictable.

d. Type of Oven Used for Bake and Location of Panel in Oven

R. E. Thompson, (Parlin Plant Lab. Notebook No. 97) has conducted tests, confirmed by this writer, demonstrating the variables introduced into gloss determinations by oven conditions. It has been found that differences in gloss of up to 5 points may be attributable to different types of draft ovens. This difference was found in a comparison between test results in which the panel was placed immediately into the oven after drawdown. Both draft ovens were kept at 65°C. The Fisher "Isotemp" oven in the Control Lab gave consistently higher results than did the Precision Scientific Co. draft oven located in the Plant Lab.

Tests show that vapor accumulation in a static oven prevents accurate gloss determinations. Reproducibility is entirely unsatisfactory. Static oven tests also fail to achieve gloss levels in the ranges obtained in the trade. Gloss values, even at high film builds, are generally at the same level as those obtained using the present TM-92-D. (Parlin Plant Lab Report No. 14, pp. 6)

Tests also show that gloss differences of up to four points may be attributed to the location of the test panel within the oven. In tests conducted using the Precision Scientific Co. draft oven in the Plant Lab., a gloss average of 53 in the lower left hand corner of the oven compared with one of 56 in the bottom right and 55 in the upper center of the oven. (See pp. 7, Section III). It has been determined that a simple rectangular shield that protects the panel from direct draft and yet permits steady removal of solvent vapors levels out this variable. This shield has been incorporated in the proposed test procedure.

e. Existence of Entrapped Air in the Test Sample

TM-92-D calls for allowing the test sample to stand until bubble free before making the test pour. In the case of "Dulux" where 4502 Accelerator (Octyl Acid Phosphate) is added and mixed, this problem of air bubbles is acute. Observation of tests run in the Control Lab indicates that this problem may be a factor in poor gloss test results. Experiments show that those pours made immediately after mixing, even where moderate care is taken to limit air entrapment, result in gloss levels up to 4 points lower than those achieved after waiting for one-half hour prior to pouring and up to 5 points lower than those achieved after waiting for 1 and 1/2 hours. This effect is minimized by mixing the 4502 with the "Dulux" by means of a spatula rather than by violent agitation of the sample. Tests have also shown that the use of a Bird Film Applicator further reduces the effect of bubbles on the final gloss. This is another attribute of the proposed new test method.

f. Number of Other Test Panels in the Baking Oven

It has been noticed throughout the test period that gloss reproducibility is seriously impaired when the number of test panels in the draft oven increases to four or more. In the static oven, the

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effect is even more pronounced. This is probably attributable to an increase in solvent vapors in the oven since it is most noticeable when the panels are placed directly into the oven without prior flashing.

g. Gloss of Glass Test Panel Prior to Application of Sample Test Film

Tests in which the glass test panels were coated with opaque films prior to having the furniture flat applied have shown that the final gloss of the flat is dependent on the gloss of the substrate. Extending this principle, gloss readings were taken on the glass panels used in TM-92-D, and it was found that these glass panels themselves do not possess gloss reproducibility with 95% confidence limits of ± 1 units. If this gloss variability is transposed undiminished through the transparent test film, this would be another argument for enlarging the 95% confidence limits to ± 3 gloss units. (See pp. 8, Section V.)

EXPERIMENTAL DETAILS:

Section I. Statistical Comparison of Methods.

	Sample	Gloss	Std. Deviation
Present TM-92-D	RF-5283	25.84	1.064
No CTR Method*	RF-5283	53.27	1.016
Proposed Test Method	RF-5283	40.90	0.982

*The values here are for those tests tun in the Precision Scientific Co. Forced Draft Oven. The gloss average, using immediate baking at 65°C. with no flash time in the Constant Temperature-Humidity Room, went to 55.8 when the Fisher "Isotemp" Oven in the Control Lab was used.

Section II. Variation in Gloss Due to Change in Flash Time.

Sample	Method	Gloss (30 min. flash)	Gloss (37 min. flash)
RF-5283	Proposed	40.9	36.4

Sample	Method	Gloss (No flash)	Gloss (5 min. flash)
RF-5283	Auto.Spray (two passes)	30.2	27.5
RF-5283	Auto.Spray (five passes)	47.9	44.3

Section III. Gloss Variations Due to Location of Panel in Oven

Oven: Precision Scientific Co. Forced Draft Oven.
Test: No Flash - Immediate Bake @ 65°C.

Location of Panel	Gloss			Avg.
Top Left	54.3,	54.0,	54.1	54.33
Top Center	54.5,	55.0,	55.4	55.00
Top Right	54.3,	53.7,	54.5	54.17
Bottom Left	52.9,	52.8,	53.2	52.97
Bottom Center	53.8,	52.3,	54.7	53.60
Bottom Right	55.1,	56.0,	56.7	55.87

Section IV. Increase of Gloss Using Proposed New Test Method as Compared with that Achieved Using TM-92-D

Sample	Gloss (TM-92-D)	Gloss (Proposed Method)	Increase
RF-5283	25.84	40.90	+ 15.06
14281	30.70	43.70	+ 13.00
RF-5295	32.30	39.00	+ 6.7
RF-5285	60.00	68.00	+ 8.0

Section V. Variability of Glass Test Panels Prior to Application of Sample Test Film

Type	Gloss Average	Std. Deviation	95% C.L.
4" x 6" (Window)	91.3	.505	± 1.010
5" x 12" (Plate)	91.64	.4468	± .8936
5" x 7" (Plate)	93.58	.684	± 1.368

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APPENDIX A

TM-92-D (Current Method)

"A" "DULUX"

1. Material to be tested is let down with 2 parts 4502 (Octyl Acid Phosphate) to 98 parts of sample. This material is thoroughly mixed and allowed to stand until bubble-free.

2. The material is now poured on a black glass plate, starting the pour exactly one inch from the top of the panel.

3. This pour is allowed to air drain in an upright position under constant temperature and humidity conditions (78°F. and 50% R.H.) for one-half hour.

4. The panel is now baked for one hour at 65°C. in a forced draft oven.

5. After cooling the panel the 60° gloss is determined by using a Universal Glossmeter in the prescribed manner.

"B" "DUCO"

The procedure for "Duco" material is identical with the above except that the sample is not mixed with 4502 Accelerator.

- -

Proposed Method of Determining Gloss

"A" "DULUX"

1. Material to be tested is let down with 2 parts 4502 (Octyl Acid Phosphate) to 98 parts of sample. This material is thoroughly mixed with a spatula and allowed to stand until bubble-free.

2. The Automatic Bird Film Applicator fitted with a .007 inch Bird Doctor Blade is now used to make a drawdown of the sample on a 4" x 6" window glass panel.

3. The glass panel is allowed to air flash in a horizontal position in the constant temperature and humidity room (78°F. and 50% R.H.) for one-half hour.

4. The panel is now baked for one hour at 65°C. in a forced draft oven (Precision Scientific Co.). The panel is placed horizontally on a solid sheet metal shelf and is covered on four sides by a sheet metal shield.

5. After cooling the panel the 60° gloss is determined by using a Universal Glossmeter in the prescribed manner.

95% confidence limits = ± 1.964 gloss units
if test procedure is strictly followed.

"B" "Duco"

The procedure for "Duco" material is identical with the above except that the sample is not mixed with 4502 Accelerator.

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APPENDIX B

TEST EQUIPMENT

OVENS:

Supplier:	Precision Scientific Co.
Type:	Forced Draft
Serial:	821-501
Location:	Parlin Plant Lab.

Supplier:	Precision Scientific Co.
Type:	Forced Draft
Serial:	825-307
Location:	Parlin Plant Lab.

Supplier:	Fisher Scientific Co.
Type:	Fisher "Isotemp"
Location:	Parlin Control Lab.

AUTOMATIC FILM APPLICATOR:

Supplier:	Gardner Laboratories Inc.
Type:	Automatic Bird Film Applicator, Item 1410 with Bird Vacuum Plate Item 1420.
Location:	Parlin Plant Lab. (On Loan from Philadelphia Plant)

DOCTOR BLADES

Supplier:	Gardner Laboratories Inc.
Type:	Bird Applicators, Items 1390 thru 1405.

TEST PANELS:

4" x 6" Disposable Window Glass Panels

TEST SHIELD:

Supplier:	Parlin Millwright Shop
Specs.:	Rectangular aluminum shield 5" x 8-1/4" x 6-1/2"

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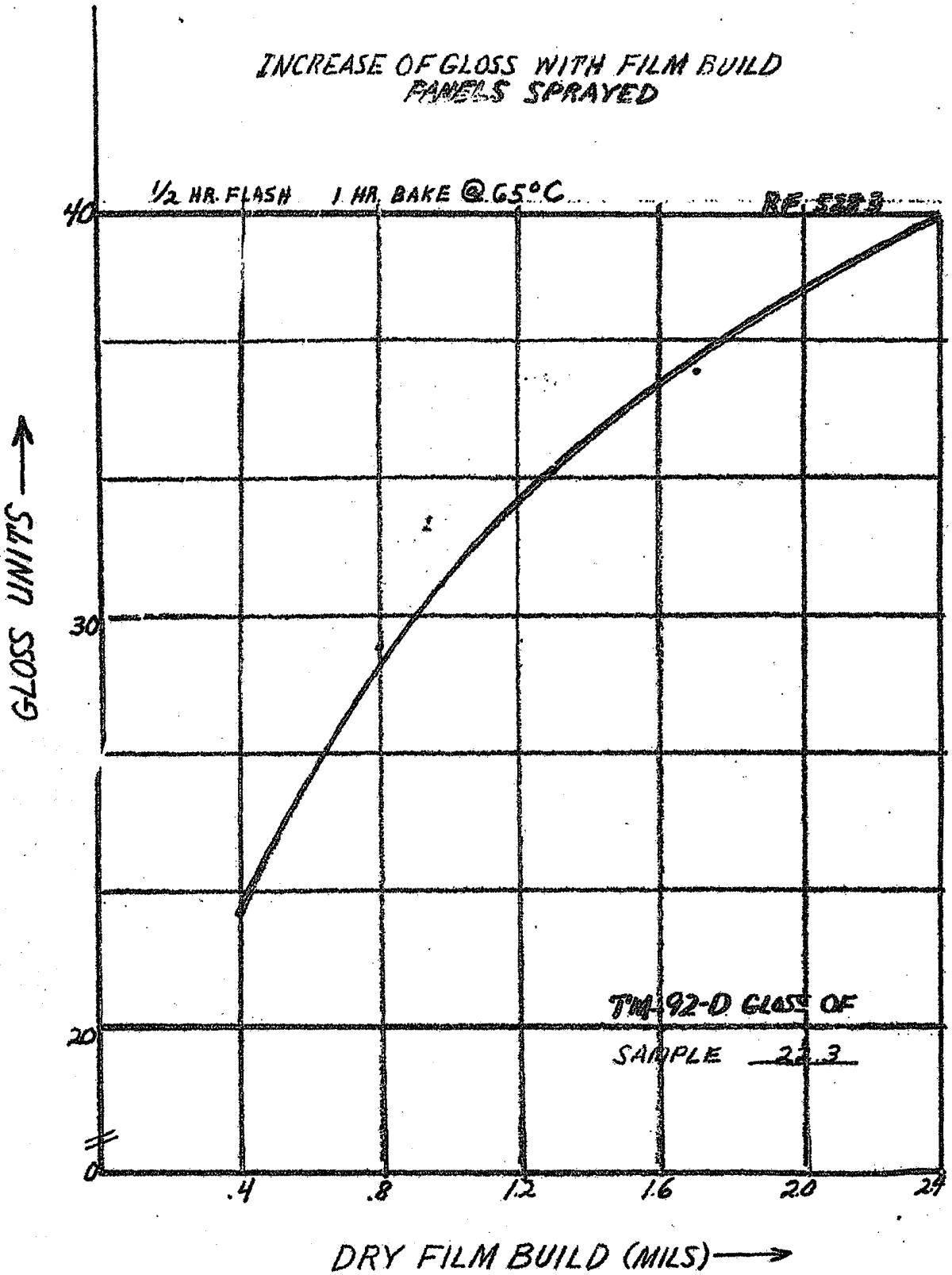
APPENDIX C



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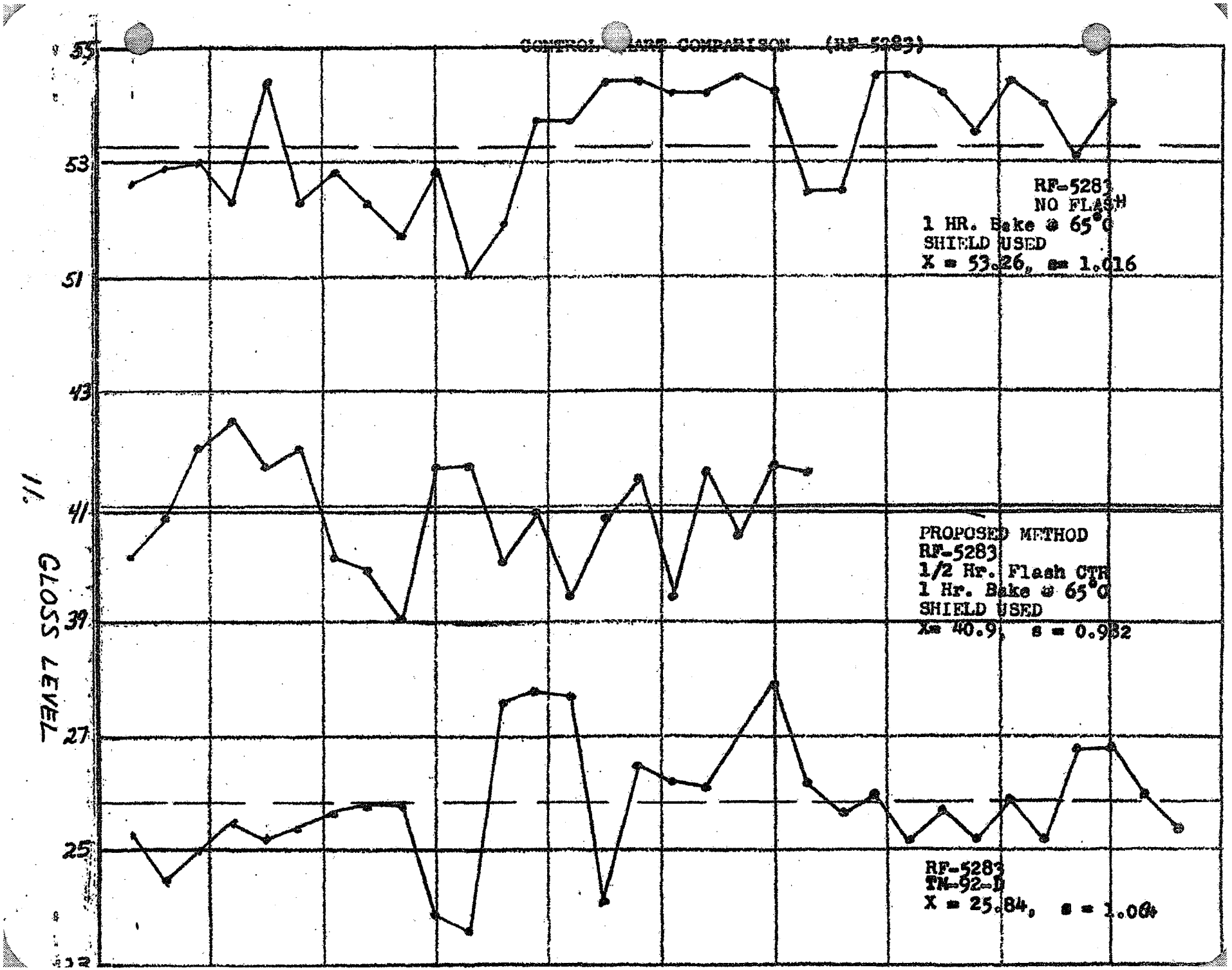

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