E. I. DU PONT DE NEMOURS & COMPANY

PIGMENTS DEPARTMENT
NEWPORT PLANT

SERIAL NO. KN-67-15

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NEWPORT PLANT

PIGMENT COLORS RESEARCH REPORT

SUBJECT: FLASH DISTILLATION - ENGINEERING DEVELOPMENT - BLUE B BREACHING STUDY - GREEN G BREACHING STUDY

PERIOD COVERED: JUNE 1966 - FEBRUARY 1967

KN-67-15

DATE: 8/24/67

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SUBMITTED BY: D. H. EASTHAM DATE SUBMITTED: 7/25/67

APPROVED BY: J. W. MINNICH DATE RELEASED: 8/24/67

ABSTRACT

The theory and development of a continuous flash steam distillation process is described. A brief study of pertinent variables was made to demonstrate feasibility of flash distillation process for ODCB removal from Green G and Blue B pigment slurries. Effects of temperature, hold time and ODCB/Pigment ratio on quality were also determined. Blue B products were also evaluated in soft vinyl.

TABLE OF CONTENTS

I. INTRODUCTION

A continuous steam distillation process has been designed and tested on semi-works scale to remove o-dichlorobenzene (ODCB) from pigment slurries. A description of the process, laboratory results and data on demonstrated performance on green and blue products are included.

A brief study was made on the effects of significant variables in the breaching of green and blue products.

II. CONCLUSIONS

1. The flash distillation process is an effective, reproducible method for removing ODCB from pigment slurries.

2. By controlling pigment-solvent contact time and temperature, a green or blue product of standard strength with a significantly darker, more transparent masstone can be produced.

3. The flash distillation process can be incorporated to remove other water immiscible solvents from a water-pigment dispersion.

4. Flash distillation will make possible a reduction in distillation cycle of about 50% as compared to batch distillation.

III. SUMMARY

A steam distillation process was developed to effectively remove ODCB from pigment slurries. The process is carried out continuously and under conditions that do not produce foam which is currently a frequent serious operating problem.

The equipment required is relatively simple; a steam jet eductor provides a "pump" as well as high efficiency contactor for steam and slurry coupled with a cyclone separator to separate the solvent-free slurry from the vapors. The contacting is done in the absence of air which prevents the formation of foam which occasionally causes serious operating problems in the plant. The process produced green and blue products of standard strength with a much darker, more transparent masstone. The rapid continuous removal of solvent makes relatively close control possible of solvent-pigment contact time. This led to a study of other pertinent variables affecting breaching, ODCB/Pigment ratio and temperature.

IV. DISCUSSION

A. Theory

Flash distillation incorporates the same steam distillation principle on a continuous basis that is currently used on batch basis. To determine operating parameters we have the following.

IV. DISCUSSION (Cont'd)

A. Theory (Cont'd)

$$\frac{W_A}{W_B} = \frac{M_A}{N_S M_B} \left(\frac{P}{P_B} - N_S \right)$$

W_A = Mass flow rate of carrier vapor (steam), lb./hr.
W_B = Mass flow rate of material in vapor (ODCB), lb./hr.
M_A = Molecular weight of carrier (steam), 18
M_B = Molecular weight of material (ODCB), 147
P = Total pressure, 760 mm
P_B = Partial pressure of material, assumed to be 0.9 x vap. press., 63 mm Hg
N_S = Vaporization efficiency, assume unity

$$\frac{W_A}{W_B} = \frac{18}{(1)\ (147)} \left[\frac{760}{0.9(63)} - 1 \right]$$

$$\frac{W_A}{W_B} = 1.52\ \frac{\text{lb. Steam}}{\text{lb. ODCB}}$$

B. Heating Requirement

Typical Breaching Slurry Composition

	Gallons	Lb.	Moles	Mole % (Approx.)
Water	1,082	9,050	503	98
"Staybelite"	12*	91		
ODCB	46.5	507	3.45	0.7
CPC	96	1,200		
NaOH	2.5(50%)	32(50%)	0.4	
	1,239	10,870		

*Solid - estimated.

1. Steam for ODCB Removal

1.52 (507) = 770 Lb.

Heating Requirement

Heat 10,870 lb. slurry from 60°F. to 210°F.
Assume C_p = 1 BTU/Lb. °F.

$Q = MC_p \Delta T$
$Q = (10{,}870)\ (1)\ (210-60)$
$Q = (10{,}870)\ (1)\ (150)$
$Q = 1{,}630{,}000$ BTU

IV. DISCUSSION (Cont'd)

B. Heating Requirement (Cont'd)

1. Steam for ODCB Removal (Cont'd)

$$Q_{lb.} = \frac{1{,}630{,}000 \text{ BTU}}{10{,}870 \text{ lb. Slurry}}$$

$$Q_{lb.} = 150 \frac{\text{BTU}}{\text{Lb. Slurry}}$$

$$(150 \text{ BTU/Lb. Slurry}) \left(\frac{\text{Lb. Steam}}{980 \text{ BTU}}\right) \left(\frac{10{,}870 \text{ Lbs. Slurry}}{1{,}239 \text{ Gal. Slurry}}\right)$$

$$1.345 \frac{\text{Lb. Steam}}{\text{Gallon Slurry}}$$

$$\text{Total Heating Steam} = (1{,}239 \text{ gal.}) \left(1{,}345 \frac{\text{Lb. Steam}}{\text{Gal. Slurry}}\right) =$$

1,661 Lb. Steam

2. ODCB Vaporization Steam Requirement

Latent Heat of Vaporization ODCB = 140 BTU/lb. - DG 14.1A

$$\frac{Q_{Vap.}}{\text{Lb.}} = \frac{140 \text{ BTU/Lb. ODCB}}{980 \text{ BTU/Lb. Steam}} = 0.143 \frac{\text{Lb. Steam}}{\text{Lb. ODCB}}$$

$$Q_{Tot.} = (507)(0.143) = 72.5 \text{ Lb. Steam}$$

3. Summary of Steam Requirement

	Lb. Steam
Heating	1,661
ODCB Vaporization	73
Distillation	770
Total	2,504 Lb.

Add 15% for heat loss

1.15 (2504) = 2,880 Lb. Steam

$$\frac{2{,}880 \text{ Lb. Steam}}{1{,}239 \text{ Gal. Slurry}} = \frac{2.33 \text{ Lb. Steam}}{\text{Gal. Slurry}}$$

C. Semi-Works Testing

A semi-works scale unit as shown in Figure 1 was assembled. Test results indicated a steam requirement of about twice theoretical is required to achieve satisfactory removal of ODCB. A steam to slurry ratio of 5 lb. steam per gallon of slurry is recommended for design purposes.

IV. DISCUSSION (Cont'd)

D. Cyclone

A cyclone was provided to separate the ODCB-free slurry from the vapor stream.

Design of semi-works still cyclone was not entirely in accordance with Design Standard DG2.2D because required pipe size was unavailable. Lab cyclone re-entrained slurry at exit vapor velocities (Section dimension e of DG2.2D) of 10 ft./sec. If cyclone had been fabricated as outlined in DG2.2D, exit velocity would have been 4 ft./sec. and no re-entrainment would have occurred. Slurry/steam ratio at cyclone corresponds to 5-6 gal./1,000 ft^3. A special spray separator with a conical bottom in accordance with DG2.2D is recommended.

E. ODCB Removal Efficiency - Blue B

Tests were conducted under various operating conditions to determine optimum for removal of ODCB. Steam to feed ratio was found to be the most effective variable. Below a ratio of 4 lb. steam/gal. slurry, the percentage removal of ODCB begins to fall off rapidly with slurry at room temperature. The effect of velocity has some influence, higher velocity-better removal, but the effect is much less significant than steam to feed ratio. At higher slurry temperatures, a lower steam ratio can be tolerated.

F. System Operability

In all cases, the semi-works still operated well. Most problems experienced were associated with the steam supply and not the still operation. The exclusion of air apparently prevents the formation of foam which was at no time evident in any of the tests.

G. System Pressure Drop

Estimating pressure drop in a system containing two phases is difficult and in most cases accurate only to within $\pm$ 30%. Provisions were made to measure pressure drop under design conditions in semi-works. Results are shown in Figure 2. The pressure drop in the steam-slurry transfer pipe is expected to be 8.8 lb./sq.in./100 ft. pipe at proposed design conditions of 50 ft./sec. velocity.

H. Analysis of ODCB Removal - Blue B

To determine the effectiveness of ODCB removal, the material processed was placed in a flask and vigorously distilled in the lab. The vapors were condensed and the volume of ODCB remaining measured. By material balance

IV. DISCUSSION (Cont'd)

H. Analysis of ODCB Removal - Blue B (Cont'd)

the percentage removal was calculated. This does not take into account the ODCB that cannot be removed by distillation.

I. Other Uses

Considerable effort has been put forth to improve quality of dispersion milled blue products. Some thought has been voiced that extended contact with perclene in the plant extraction tank was causing crystal growth. If this is the case, a flash still can also be used to heat the wetted blue mill powder (ground alum and CPC mixture) and remove the perclene rapidly. Several tests were conducted that indicated a standard product could be made in this fashion.

J. Quality - Blue B

The effect of breaching variables was studied using the still as a tool to rapidly remove the ODCB. Effects of temperature, breaching time and ODCB/Pigment ratio were studied and are shown in Figures 3-8 and Table 3. A significant improvement in masstone can be achieved by reducing the ODCB-Pigment contact time. The effect of these variables is also shown on 5-15 cut soft vinyl in Table 3.

K. Quality - Green G

The development of a flash distillation process made possible close control of solvent contact time by rapid removal of ODCB. A series of tests were conducted to determine the effect of ODCB/Pigment ratio, solvent contact time and temperature on tinctorial properties of Green G as the process would relate to plant scale flash distillation.

Two levels of solvent were used at various contact times and temperatures. The Solvent/Pigment ratios studied were 1.2 (current plant standard) and 0.5. No significant difference was noted in quality by rubout between the two levels. The results are shown in Figures 9-12. Figures 1 and 2 show the temperature as the most effective variable affecting strength and masstone over the levels of ODCB examined. The solvent concentration does serve to reduce the time and temperature somewhat but not to a major degree. Hence, the lower solvent concentration is more desirable since it can be removed at a higher rate in a continuous distillation and would be an advantage in cycle time.

IV. DISCUSSION (Cont'd)

K. Quality - Green G (Cont'd)

Figures 11 and 12 show the effect of contact time. Figure 11 shows a gradual decline in masstone darkness and transparency of about 10 points over a period of 3 hours while the tinting strength was not affected. Figure 4 shows the effect at a higher level of ODCB and again shows similar behavior. One additional test was conducted at the higher ODCB level which showed strength and desirable masstone properties can be attained by holding at room temperature for about 4 hours.

The flash still can be utilized to produce a product of standard strength with a significantly darker, more transparent masstone because of close control of solvent-pigment contact time.

/mc

EN-2216

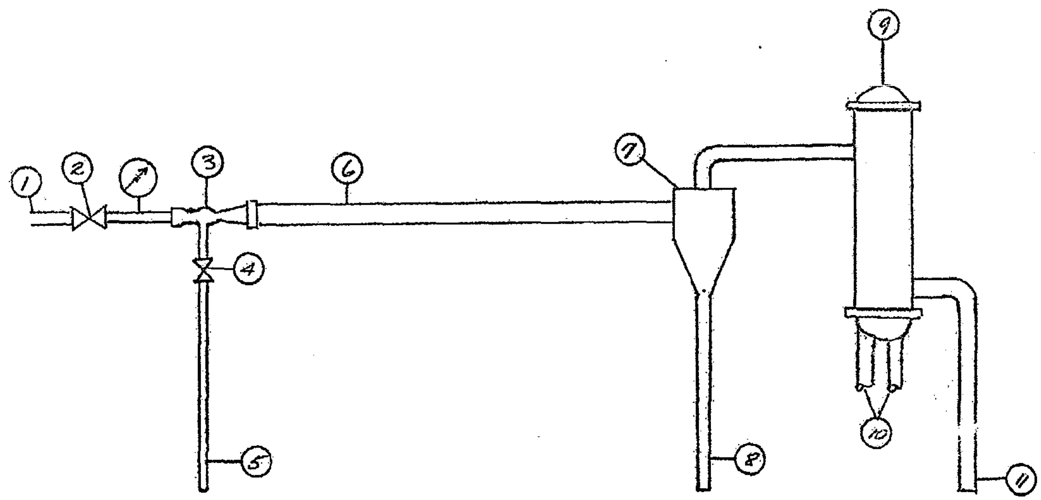
ENGINEERING DEPARTMENT SKETCH SHEET SHEET No. ___

PROJ. No. ___ PLANT NEWPORT TITLE LABORATORY FLASH STILL

BLDG. OR AREA ___ FUNCTION ___ SUBJECT FLASH STILL

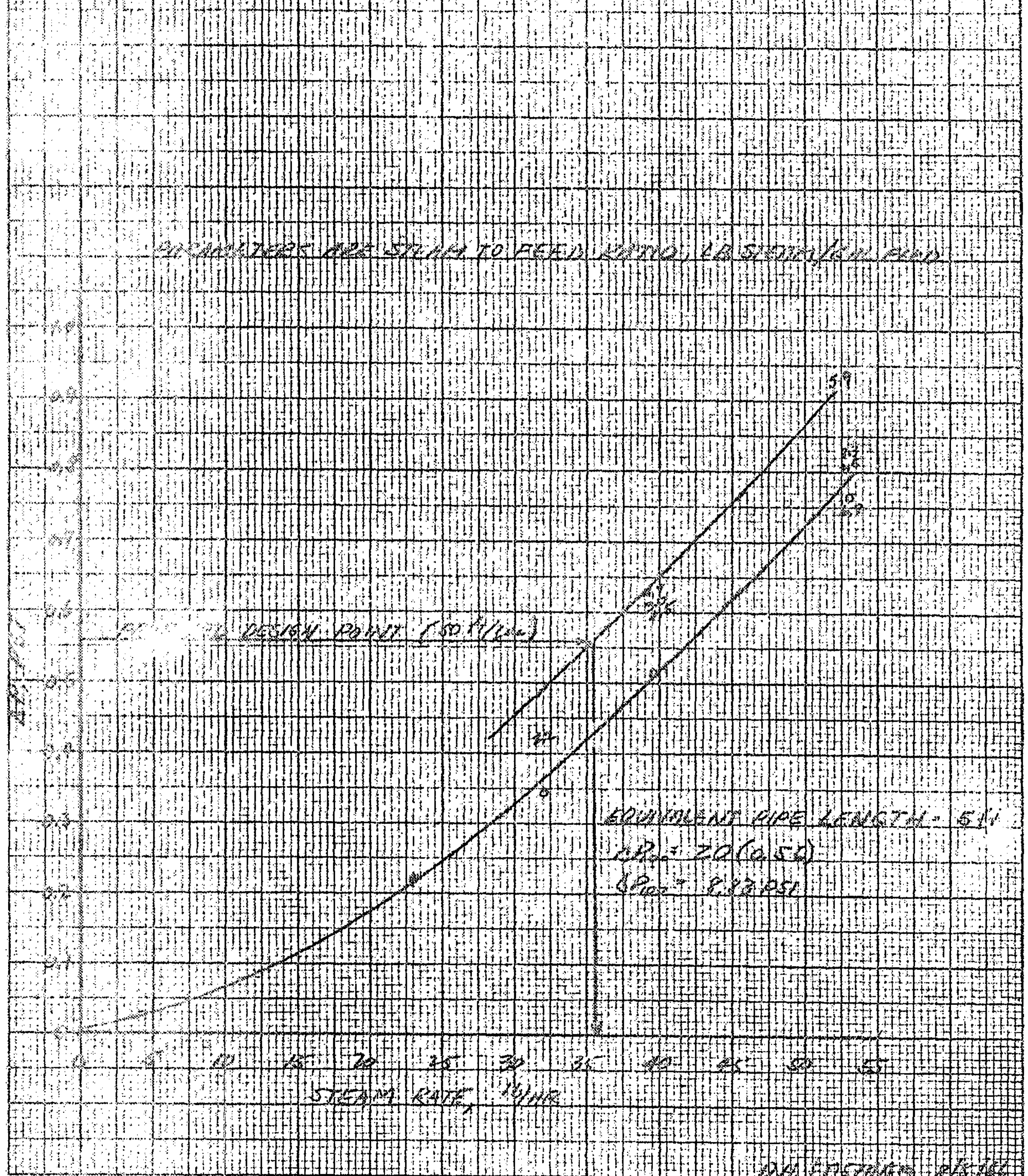
NAME D.H. EASTHAM DATE 7/14/66

FIGURE 1



1. 150 PSIG STEAM SUPPLY
2. STEAM CONTROL VALVE
3. SCHUTE - KOERTING FIG. 217 STEAM JET SYPHON
4. FEED CONTROL VALVE
5. FEED INLET
6. TRANSFER LINE, 1,2 & 3"
7. CYCLONE
8. SOLVENT FREE SLURRY DISCHARGE
9. CONDENSER - MODEL # 3587-N-15
10. COOLING WATER INLET & OUTLET
11. STEAM - ODCB CONDENSATE

DUP050027572



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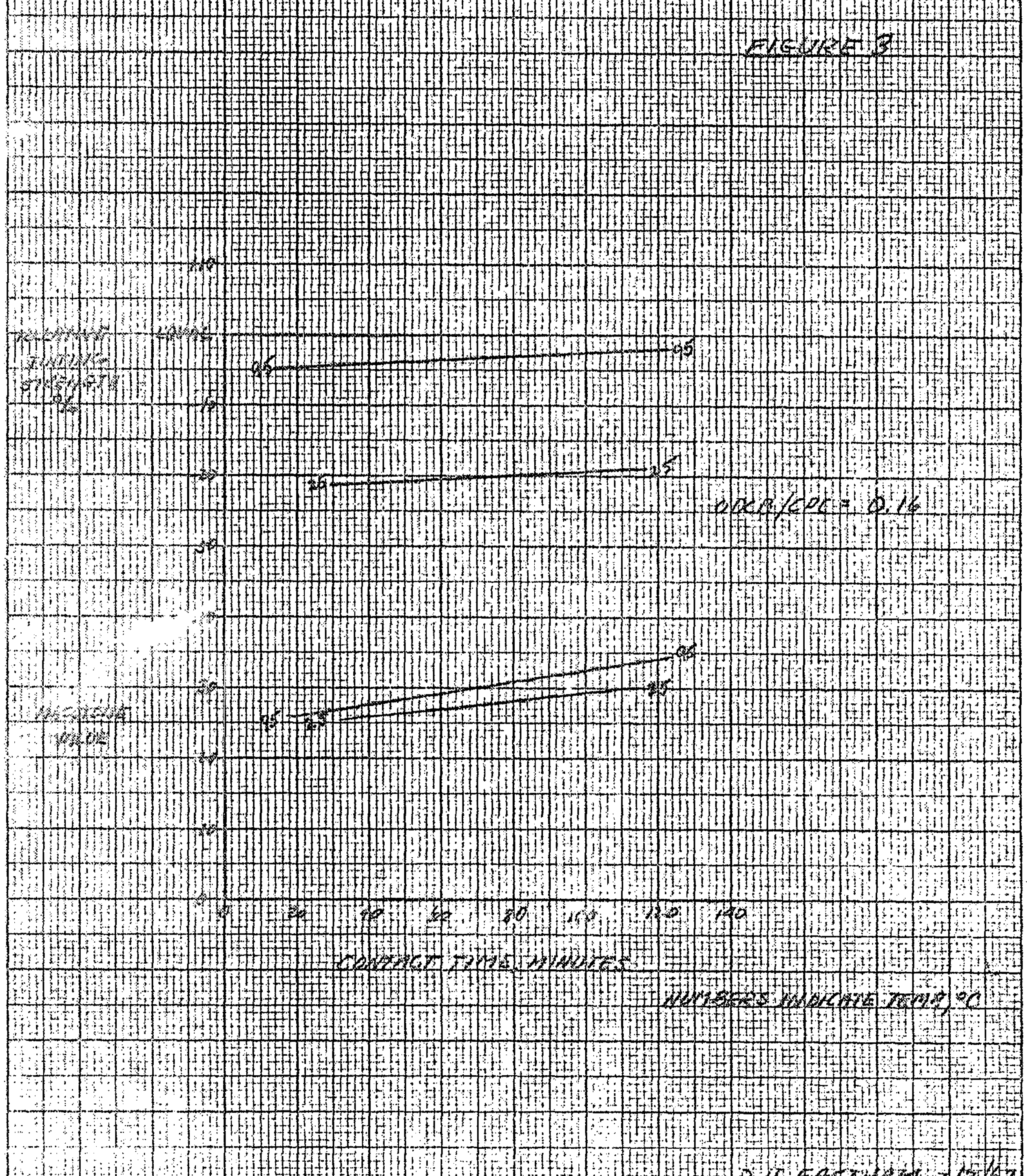
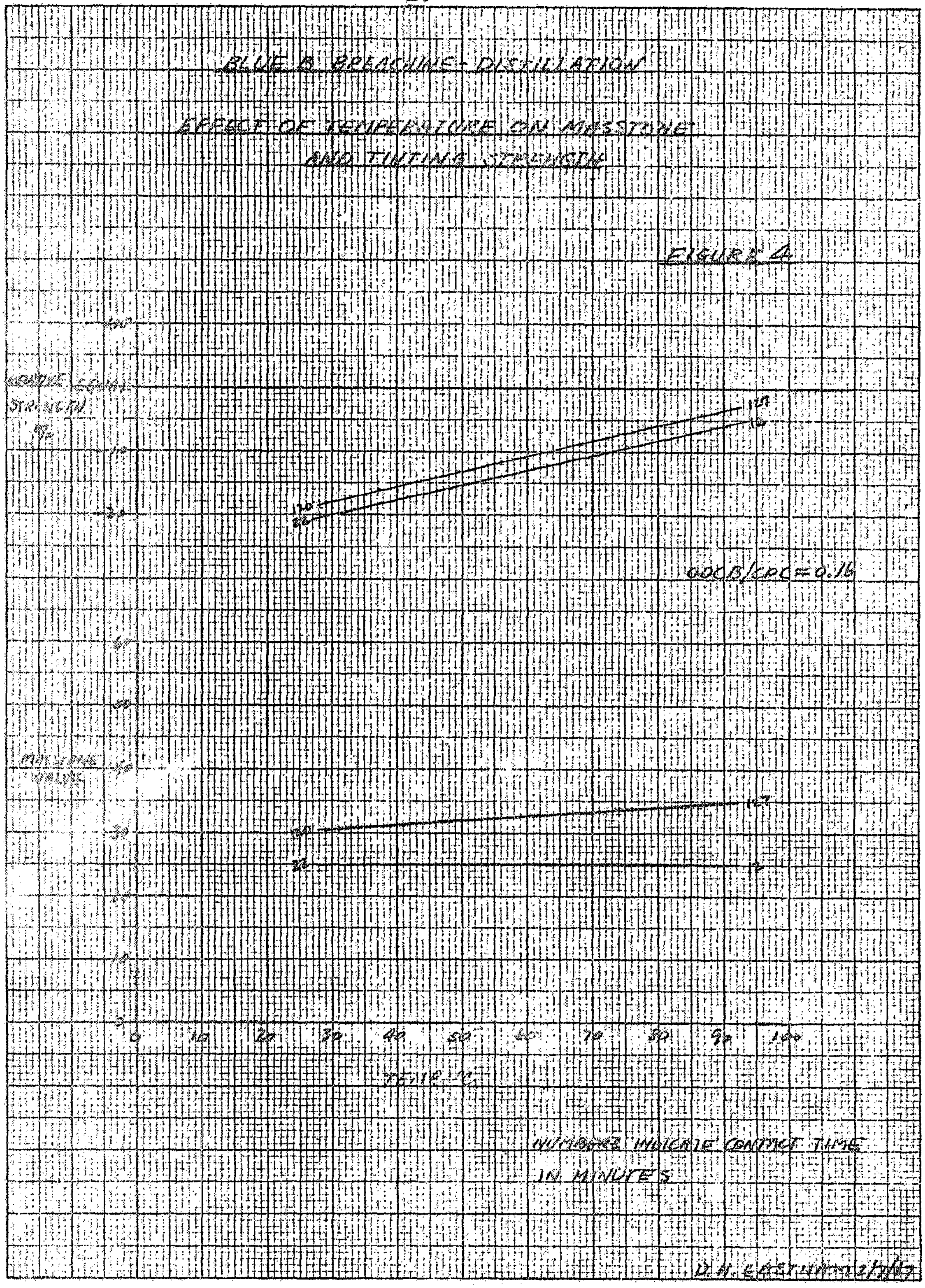
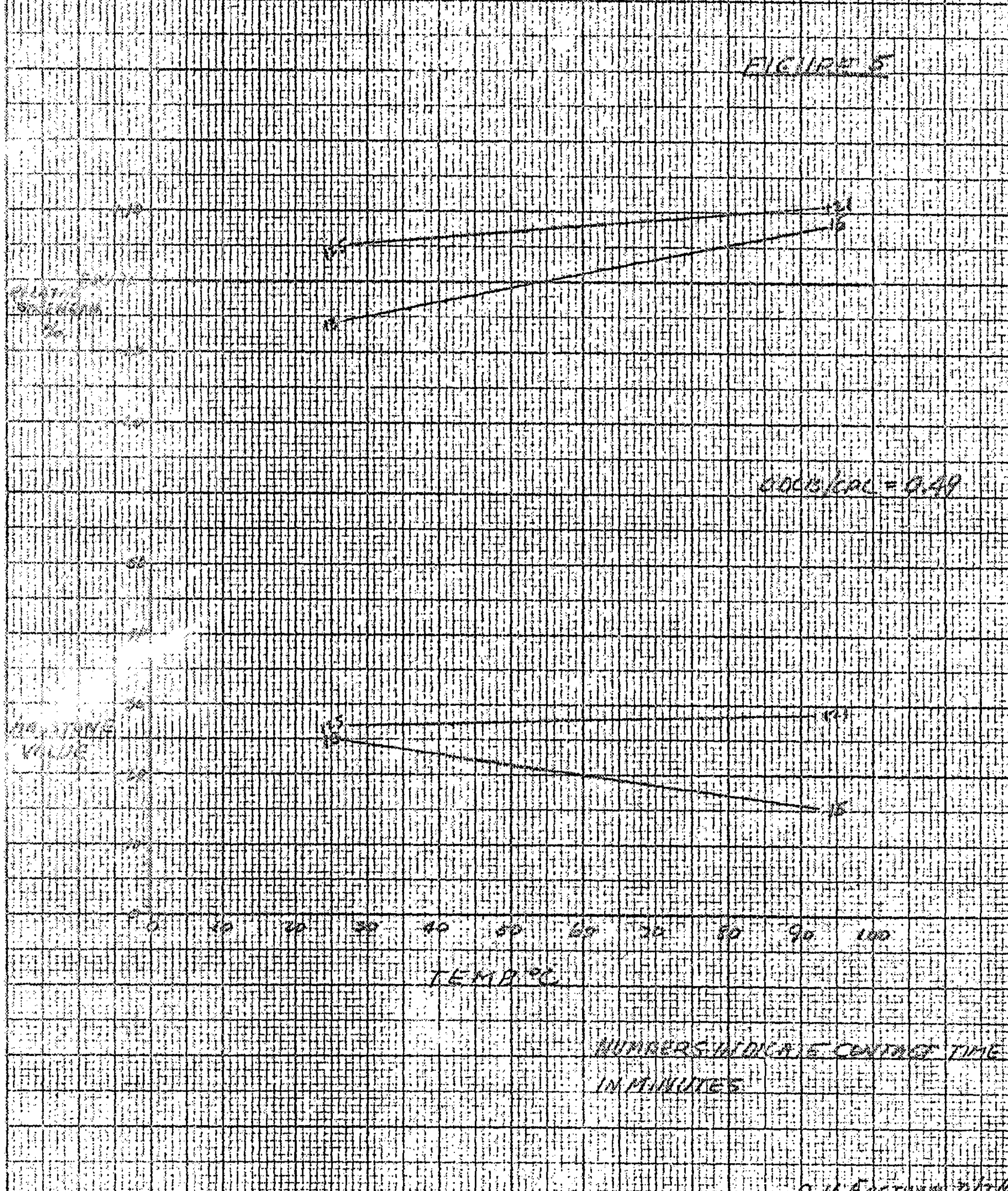


FIGURE 3
ODCB/CPC = 0.16
0
20
40
60
80
100
120
140
CONTACT TIME, MINUTES
NUMBERS INDICATE TEMP, °C
D.H. EASTHAM



BLUE B BLEACHING - DISTILLATION
EFFECT OF TEMPERATURE ON MASSTONE
AND TINTING STRENGTH
FIGURE 4
CDCB/CPC = 0.16
0
10
20
30
40
50
60
70
80
90
100
NUMBERS INDICATE CONTACT TIME
IN MINUTES



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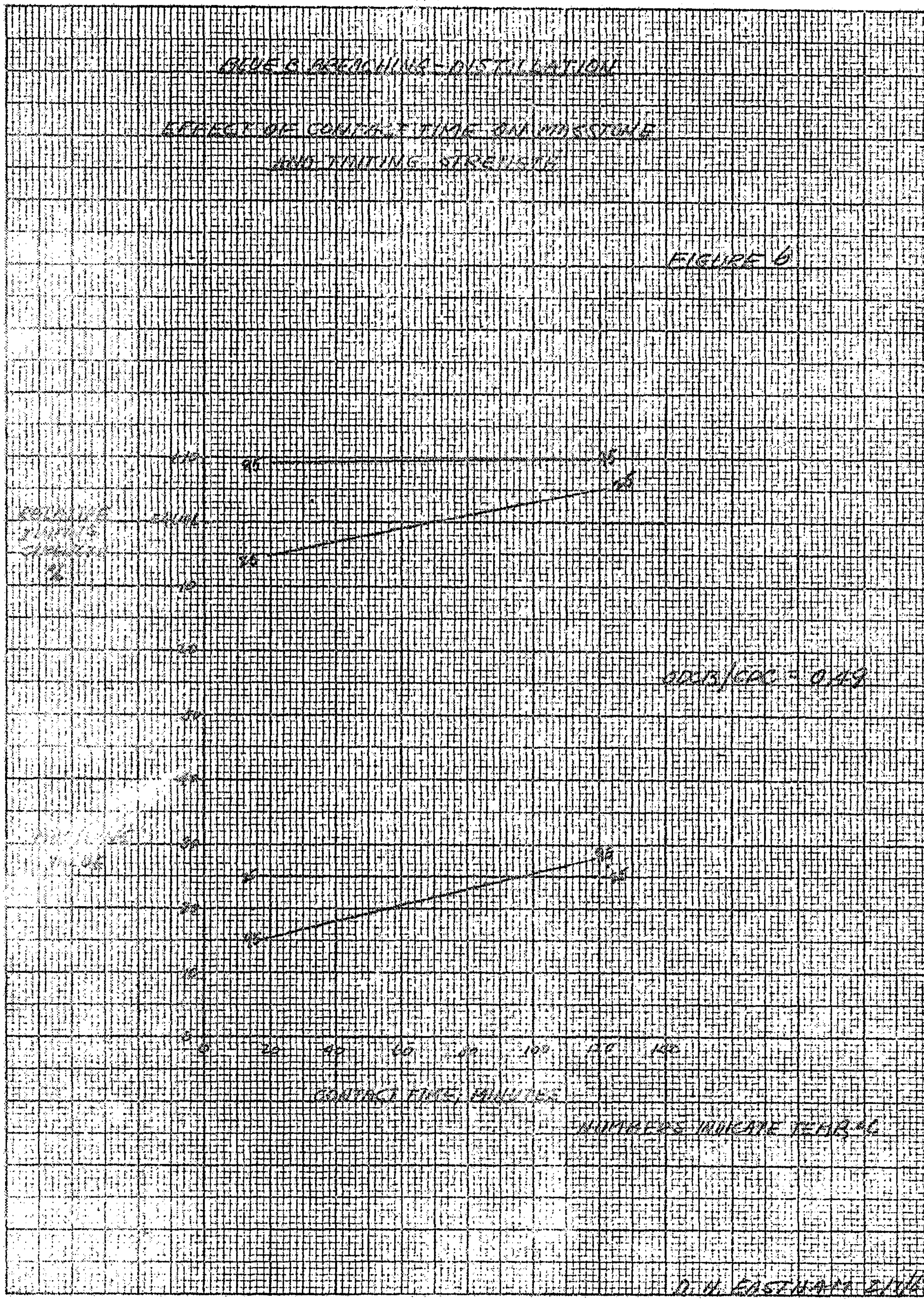
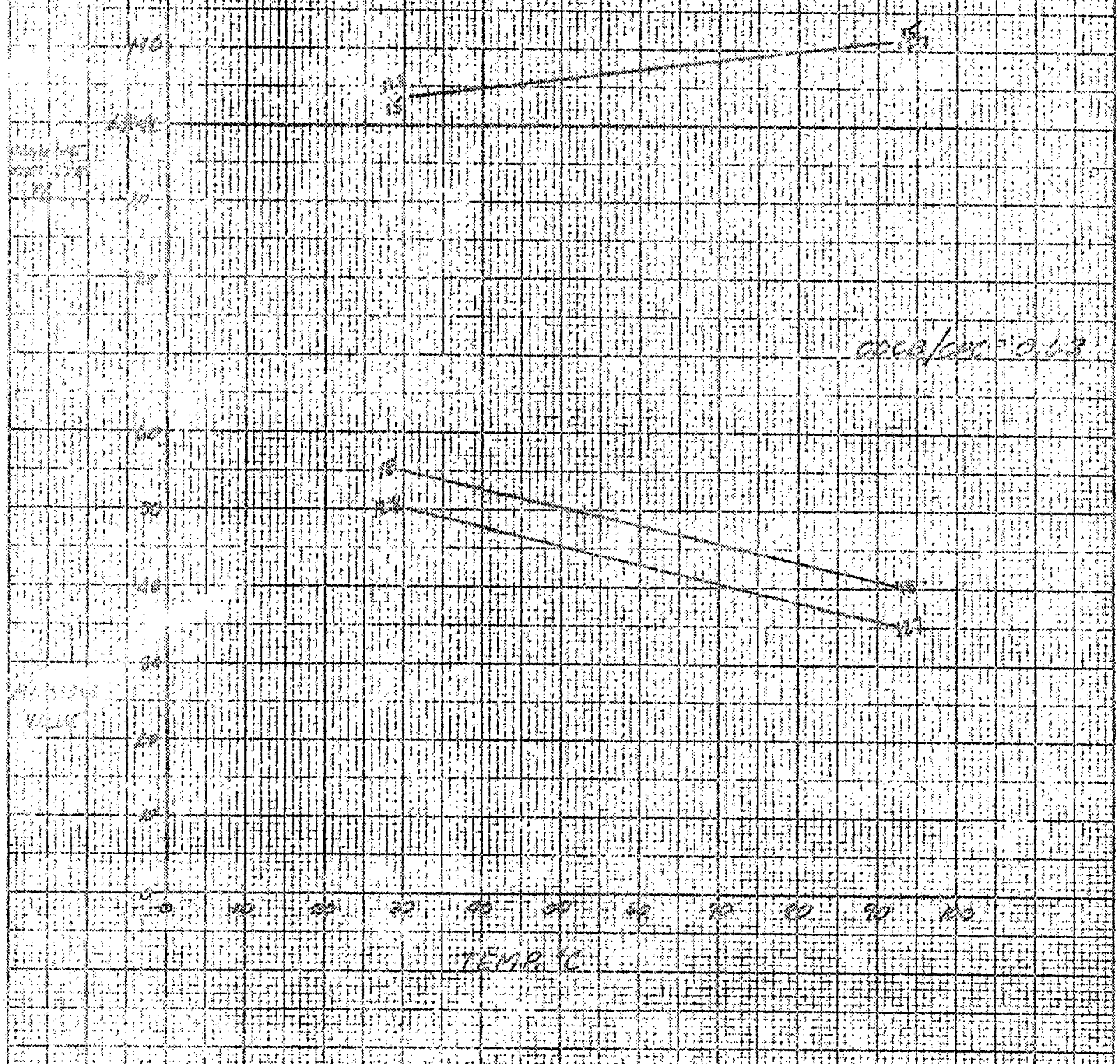

EFFECT OF CONTACT TIME ON MOISTURE
AND TINTING STRENGTH
FIGURE 6
0 20 40 60 80 100 120 140
CONTACT TIME, MINUTES
NUMBERS INDICATE TEMP. °C

BLUE B BREAKING-DISTILLATION

EFFECT OF TEMPERATURE ON MASSTONE AND TINTING STRENGTH

FIGURE 7



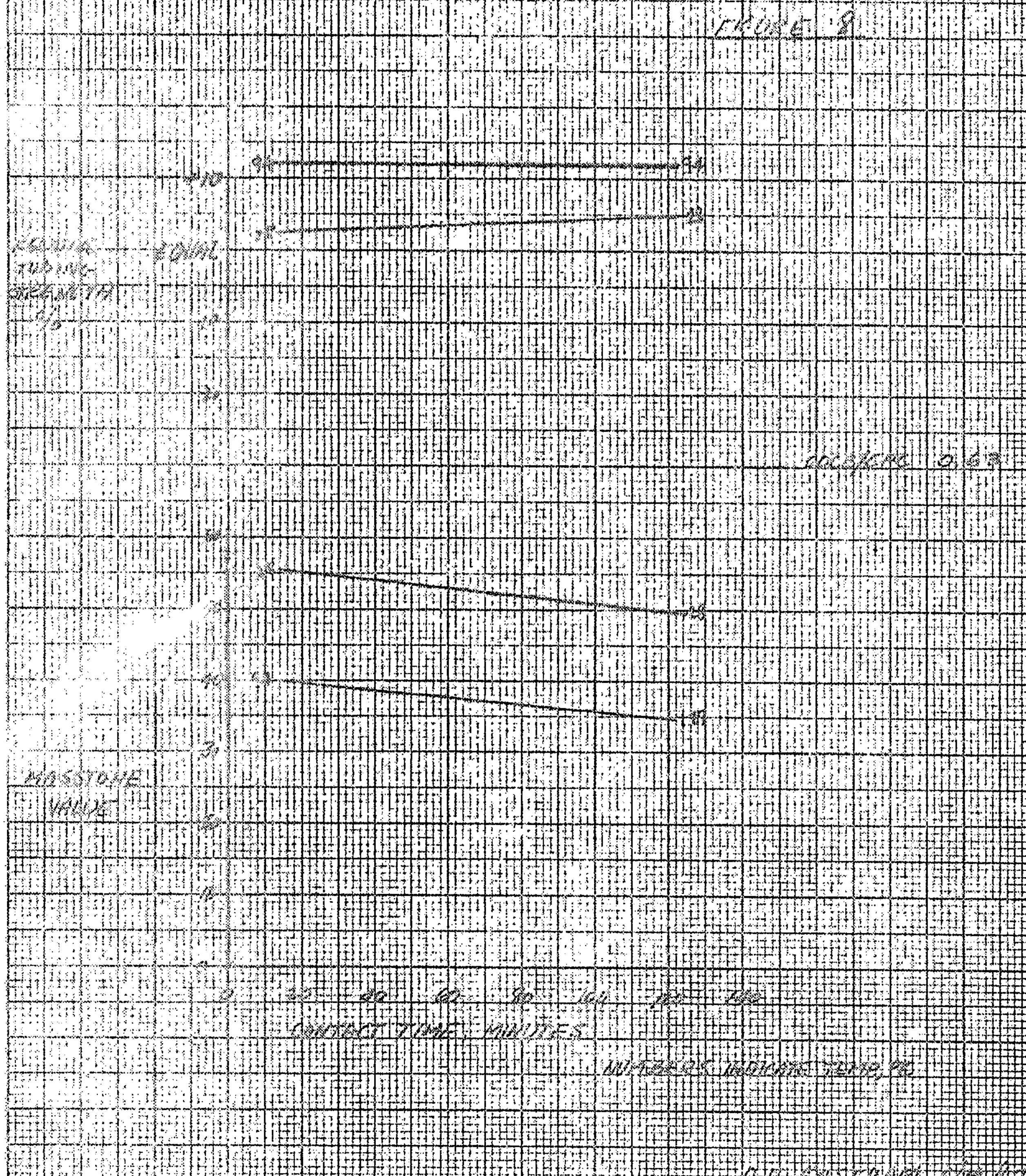
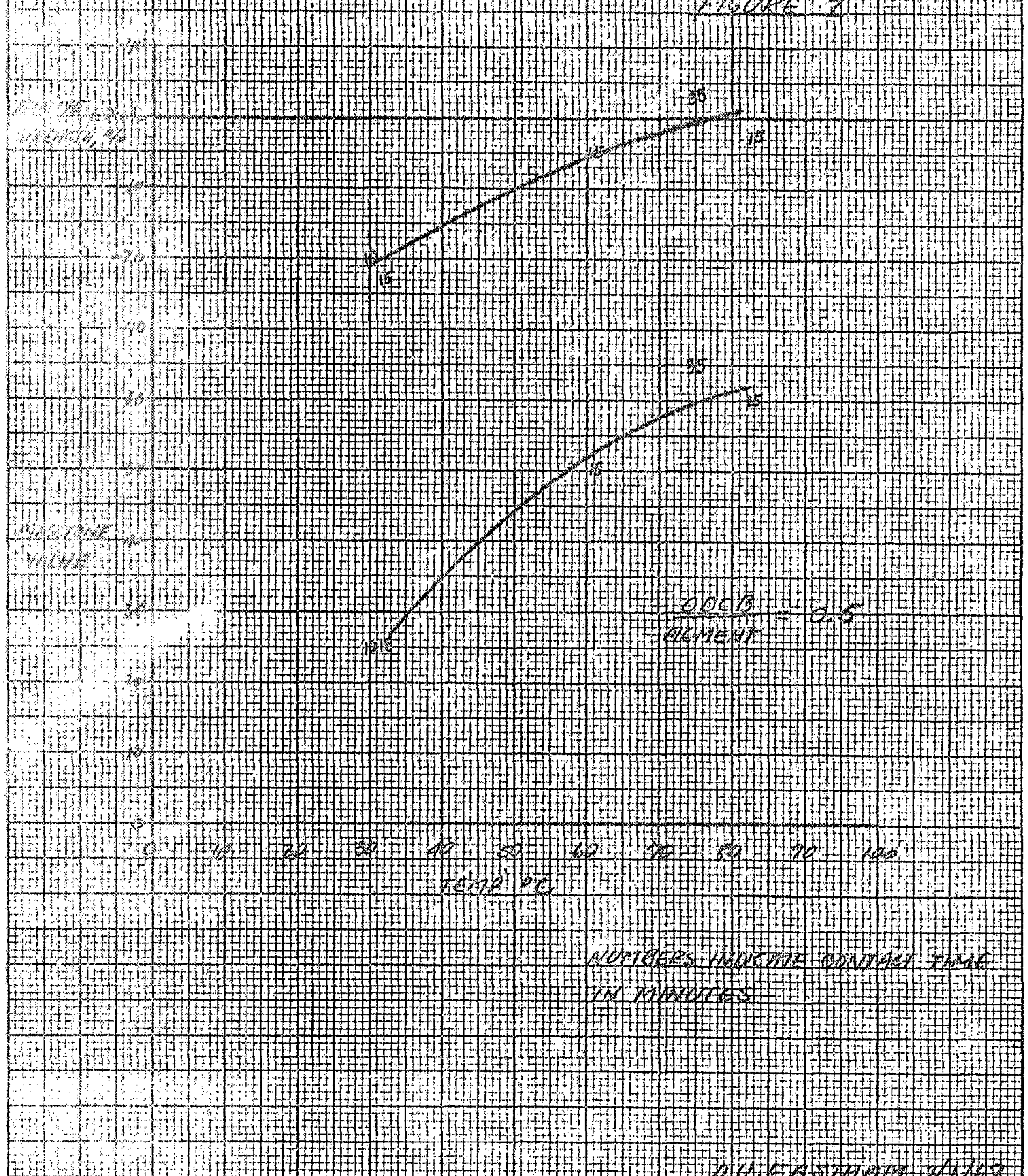


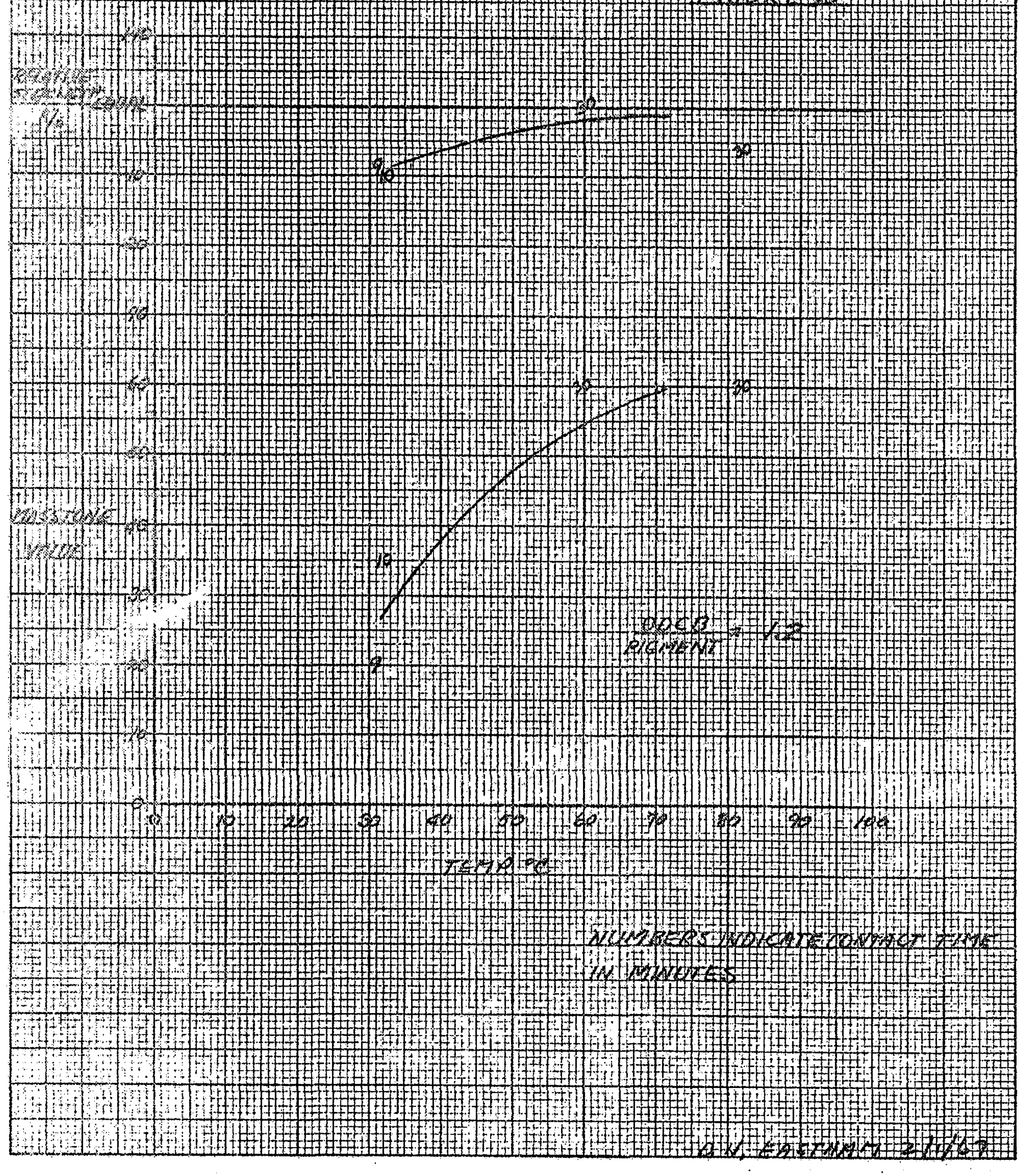
FIGURE 8
MASSTONE VALUE
CONTACT TIME - MINUTES

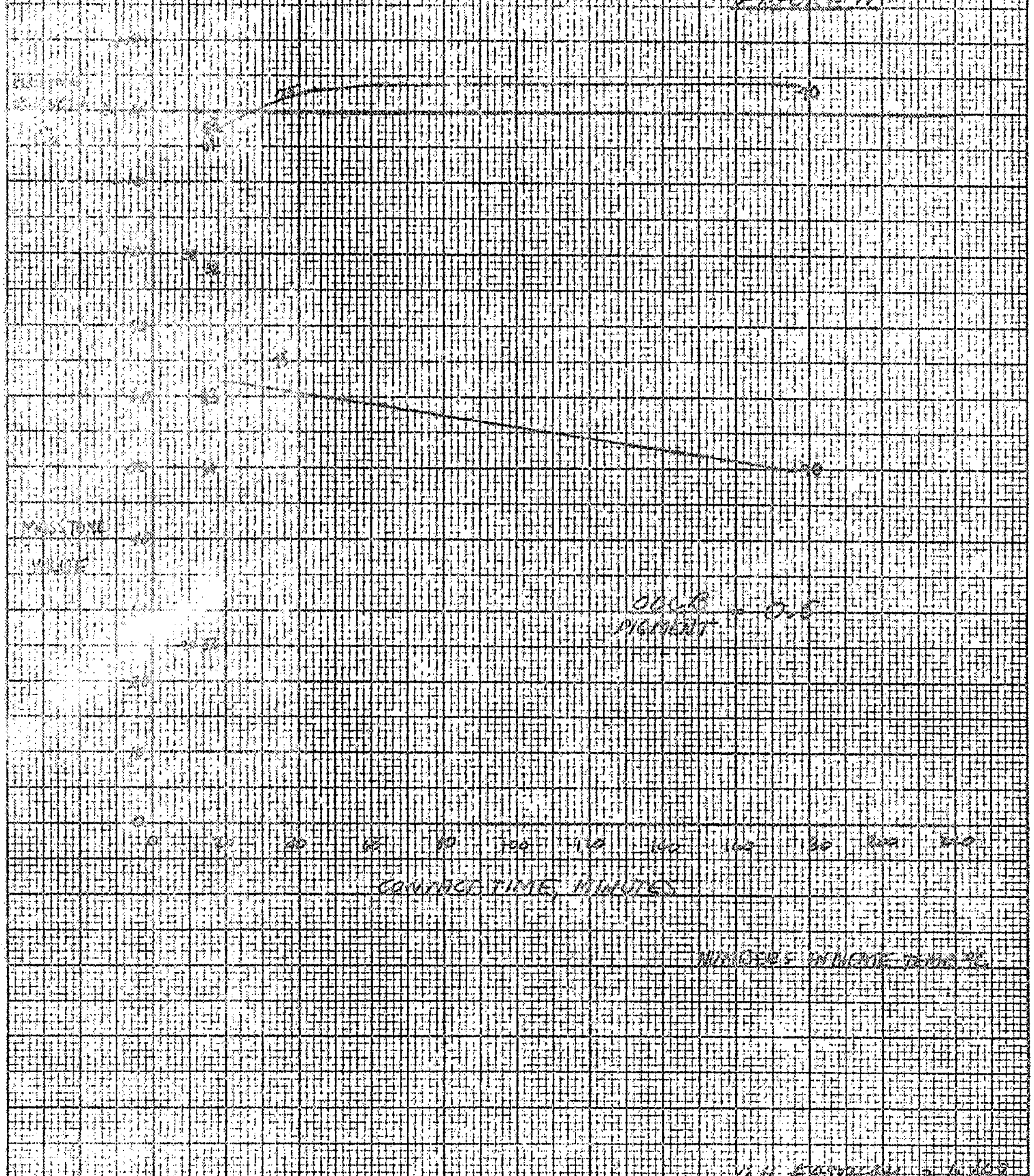
K+E 10 X 10 TO ½ INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.



DUP050027580

K+E 10 X 10 TO 1/2 INCH 46 1323
7 X 10 INCHES MADE IN U.S.A.
KEUFFEL & ESSER CO.





DUP050027582

EFFECT OF CONTACT TIME ON MASSTONE AND TINTING STRENGTH

FIGURE 12

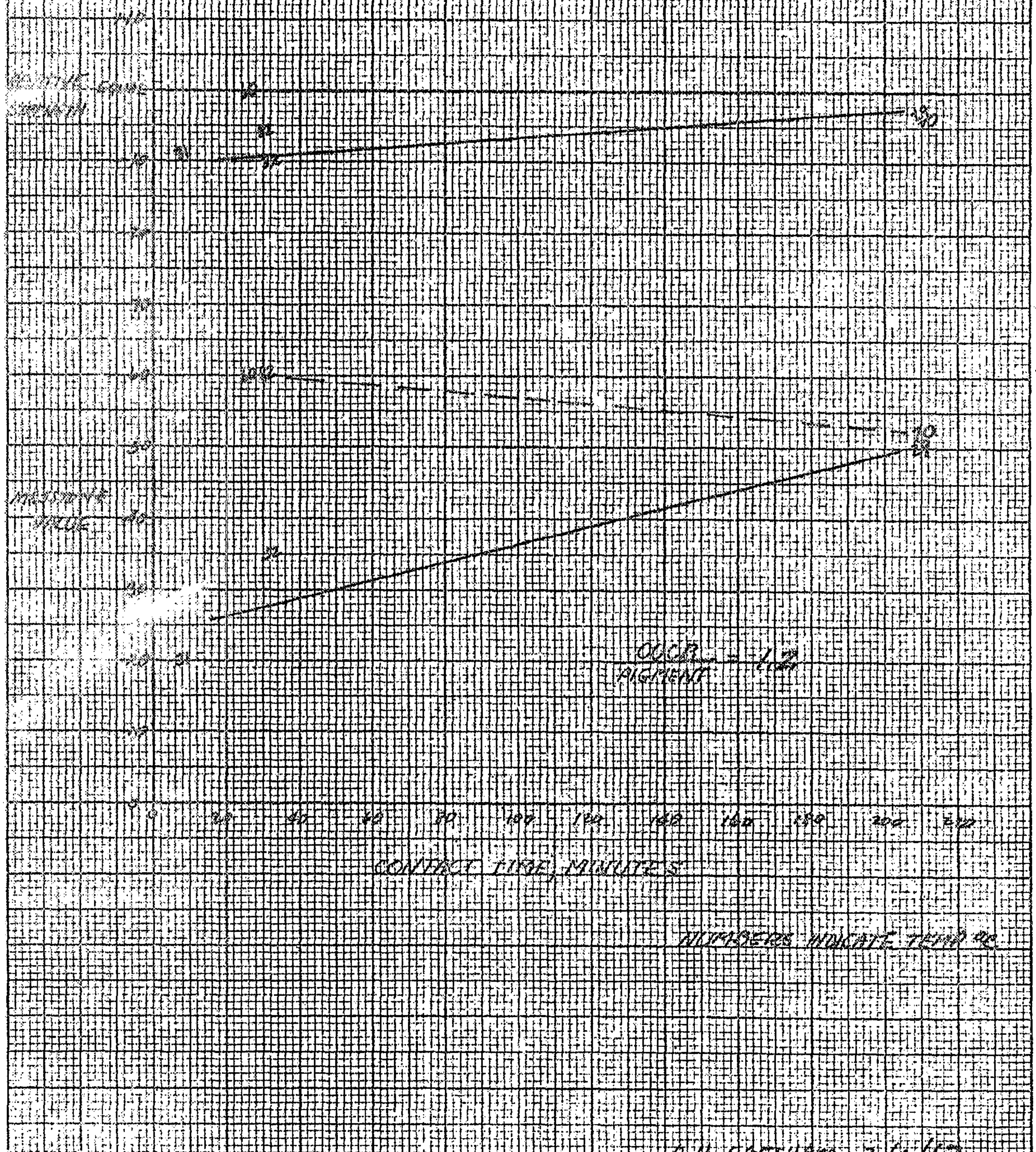


TABLE 1

FLASH STILL EXPERIMENTAL DATA

Run No.	Feed Rate ml/min.	Distillate Rate ml/min.	Steam Rate lb./hr.	Steam/Feed lb. steam/gal. slurry	Velocity ft./sec.	Residence Time sec.	% Removal ODCB
1	243	149	22	5.7	30.5	0.125	98
2	438	138	22	4.0	30.5	0.125	95
3	365	148	23	4.0	32.0	0.120	100*
4	267	112	19	4.5	26.0	0.147	96
5	-	134	27	-	37.5	0.102	95
6	161	169	25	9.8	34.5	0.111	99+
7	338	122	20	3.7	7.5	0.443	89
8	175	141	20	7.2	7.5	0.443	95
9	103	138	17	10.5	23.5	0.163	99+
10	202	142	21	6.4	28.3	0.135	97

* Run 2 material on second pass.

TABLE 2

Theoretical Steam Requirement for ODCB Steam Distillation

Vapor Pressures per DG15A

T°C	VP H_2O mm Hg	VP ODCB mm Hg	Total Press. mm Hg	VP H_2O / VP ODCB
50	94	6.2	100.2	15.15
60	150	11	161	13.62
70	230	19	249	12.10
75	300	22	322	13.65
80	360	28	388	12.85
85	420	36	456	11.68
90	520	43	563	12.10
95	625	50	675	12.50
100*	760	63	823	12.10
105	900	77	977	11.70
110	1,100	92	1,192	12.00

*Design Basis

TABLE 3

BLUE B BREACHING – FLASH STRIP DISTILLATION

EXPERIMENTAL DATA

RUN NO.	ODCB CFC	TEMP. °C	CONTACT TIME AT TEMP., MIN	FEED RATE GAL/HR	STEAM FEED LB/GAL	MASSTONE	STRENGTH	HUE	CHROMA	SOFT - VINYL* STRENGTH 5v5	STREAKING 5v5	SPECKING 5x5	STRENGTH 15v15	STREAKING 15v15	SPECKING 15v15	X RAY β RATIO
1	0.163	25	22	8.0	5.0	VD 25	121:100	—	—	127:100	B	B	124:100	E	E	—
2	0.489	25	13	8.0	5.0	25	106:100	—	—	118:100	B	B	114:100	B	B	—
3	0.163	25	120	8.0	5.0	30	119:100	—	—	128:100	B	B	120:100	E	E	—
4	0.489	25	125	8.0	5.0	25	96:100	R_{12}	SD_2	118:100	B	B	109:100	E	B	—
5	0.163	95	12	5.0	8.0	25	105:100	G_6	D_6	116:100	B	B	118:100	E	E	—
6	0.489	95	15	6.0	6.7	15	91:100	R_8	D_8	108:100	B	B	106:100	B	B	—
7	0.163	95	127	5.5	7.3	35	102:100	R_6	D_8	123:100	B	B	116:100	E	E	—
8	0.489	95	121	6.0	6.7	29	90:100	R_{10}	D_{10}	105:100	B	E	103:100	B	B	—
9*	0.52	100	—	—	—	15	90:100	R_{10}	D_{10}	—	—	—	—	—	—	—
10	0.63	28	15	7.0	5.1	55	98:100	R_{20}	D_{20}	118:100	B	B	110:100	B	B	trace
11	0.63	28	128	7.0	5.1	50	95:100	R_{10}	D_8	110:100	B	B	107:100	B	B	trace
12	0.63	94	15	7.0	5.1	40	88:100	R_{10}	D_{10}	97:100	E	E	99:100	E	E	0.05
13	0.63	94	127	7.0	5.1	35	88:100	R_{15}	D_{15}	94:100	E	E	101:100	B	B	0.04

* Control: Made by standard lab breaching. Soft vinyl results compared to control.
Intent strength compared to N930 Std (LPC 830)

TABLE 4.

GREEN G BLEACHING- DISTILLATION

EXPERIMENTAL DATA

RUN NO.	ODCB / PIGMENT	TEMP. °C	HEAT UP TIME, MIN	CONTACT TIME AT TEMP	FEED RATE GAL/HR	STEAM/FEED lb / GAL	MT	STR
1	0.51	29	—	10	3.2	7.1	VOL 25	120:100
2	0.51	61	10	15	3.9	6.0	50	105:100
3	0.53	32	—	15	4.0	5.8	25	123:100
4	0.53	93	25	5	4.7	4.9	60	103:100
5	0.53	75	—	35	4.2	5.5	65	97:100
6	0.53	70	—	180	4.4	5.2	50	97:100
7	1.20	31	—	9	4.1	6.6	70	104:100
8	1.20	29	—	210	4.0	5.8	50	103:100
9	1.20	60	210	30	4.0	5.3	60	100:100
10	1.20	32	—	10	8.8	3.8	35	110:100
11	1.20	82	—	20	8.7	3.8	60	106:100
12	1.20	70	—	210	9.9	3.8	50	103:100

	MT	STR
LAB CONTROL	VOL 35	98:100

STANDARD: N 764 IN PROCESS STANDARD – CPC 864.1.