2nd Section

| | Math | Verbal | Music |
|---|---|---|---|
| 12 | 45 | 29 | 550 |
| 2 | 60 | 45 | 700 |
| 9 | 70 | 47 | 675 |
| 20 | | | |

Dec. 2, 1946

Distillation of Ether and Acetone from

Started distillation at 1:30 p.m. under 200 mm pressure. Raised the bath temperature slowly to 75°C at 3 o'clock. Temp. at top of column remained at 21°C throughout.

| Time | | Bath Temp | Vapor |
|---|---|---|---|
| 4:00 | | 78 | 24 |
| 05 | | 82 | 26 — changed receivers |
| 20 | Pressure 100 mm | 80 | 29 |
| 5:00 | Changed receivers for smaller ones, there was same product in the previous receiver. Changed the distilling flask for a smaller one. (1 L). | | |
| 5:30 | (Pressure 100) | 85 | 29 |
| 36 | 4 | 90 | 39 |
| 6:00 | | 90 | 40 |
| 6:30 | | 110 | 38 |
| 7:00 | | 130 | 43 |

Butane yield 10.9 g

11.1.10

B.P. of $H_3C-\overset{\overset{OH}{|}}{\underset{\underset{C\equiv C-H}{|}}{C}}-CH_3$

$\frac{84 \times H}{348} = 11.7$

$H = 96.2$ cals/g.

$2.303 \log \frac{760}{760} = \frac{96.2 \times 84}{1.986}\left[\frac{1}{348} - \frac{1}{T_1}\right]$

$-1.33\ T_1 = (3.98 \times 10^3)(1.17 \times 10^{-3}) - \frac{3.98 \times 10^3}{T_1}$

$-1.33 T_1 = 11.43 T_1 - 3.98 \times 10^3$

$12.76 T_1 = 3.98 \times 10^3$

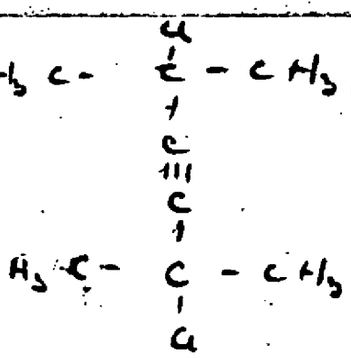
$T_1 = 312°K$

$t_1 = 39°C$

---

$H_3C-\overset{\overset{OH}{|}}{\underset{\underset{C\equiv C-H}{|}}{C}}-CH_3$

B.P. $^{\circ}C$



$$\frac{179 \times H}{423} = 22.7$$

$$H = 53.6 \text{ cals./g.}$$

$$\log \frac{P}{760} = \frac{53.6 \times 179}{1.986}\left[\frac{1}{423} - \frac{1}{T_1}\right]$$

$$-1.33\,T_1 = (4.83 \times 10^3)(1.37 \times 10^{-3}) - \frac{4.83 \times 10^3}{T_1}$$

$$1.33\,T_1 = 11.42\,T_1 - 4.83 \times 10^3$$

$$12.75\,T_1 = 4.83 \times 10^3$$

$$T_1 = 379^{\circ}K$$

$$t_1 = 106^{\circ}C$$

$$\frac{0.95}{0.5} = (5.39 \times 10^{3})(2.11 \times 10^{-3}) - \frac{5.39 \times 10^{3}}{T_1}$$

$$11.42 = 5.39 \times 10^{3} - \frac{201 \times 66.5}{T_1}$$

$$\frac{201 \times 66.5}{T_1} = 5.39 \times 10^{3} - 11.42$$

$$T_1 = \frac{201 \times 66.5}{5.39 \times 10^{3} - 11.42}$$

$$T_1 = 2.48 \text{ K}$$

$$T_1 = 2.5 \text{ K}$$

6/15/19

(B.P. of $CH_4$)

$$\frac{119 \times H}{334} = 44.7$$

$$H = 90.8 \text{ cals/g.}$$

$$2.303 \log \frac{440}{760} = \frac{\cancel{20.8} \; 5.9 \times 119}{1.986} \left[ \frac{1}{334} - \frac{1}{T_1} \right]$$

$$2.303 \log 0.463 = 4.25 \times 10^3 \left[ 3.0 \times 10^{-3} \right] - \frac{4.85 \times 10^3}{T_1}$$

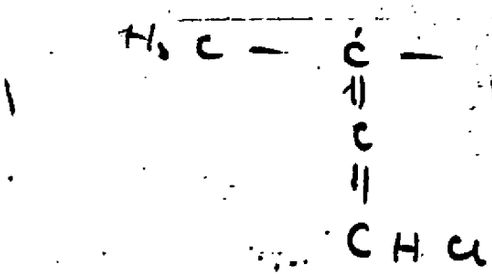
$$2.303 (-0.334) = 14.57 - \frac{4.85 \times 10^3}{T_1}$$

$$-1.33 \; T_1 = 14.57 T_1 - 4.85 \times 10^3$$

$$15.90 \; T_1 = 4850$$

$$T_1 = 305 \; ^\circ K$$

$$t_1 = 32 \; ^\circ C \qquad \text{Ans} \rightarrow 45.2$$



$$\Delta H = 94.2 \text{ cals./g.}$$

$$-1.33 T_1 = \left(3.98 \times 10^3\right)\left(-1.65 \times 10^{-3}\right) - \frac{3.98 \times 10^3}{T_1}$$

$$-1.33 T_1 = 10.56\ T_1 - 3.98 \times 10^3$$

$$11.89\ T_1 = 5.98 \times 10^3$$

$$T_1 = 335^\circ K$$

$$t_1 = 62^\circ C$$

$$2.303 \log \frac{0.00658}{60} = (5.39 \times 10^3)(1.1 \times 10^{-4}) - \frac{5.39 \times 10^3}{T_1}$$

$$-2.303 + 0.992 = 11.42\, T_1 - 5.39 \times 10^3$$

$$\frac{-0.97\, T_1}{11.42\, T_1} = 5.39 \times 10^3$$

$$T_1 = 394^0\ K$$

$$t_1 = 121^0\ C$$

## Distillation

| | | Pressure | Vapor Temp | Bath Temp | Remarks |
|---|---|---|---|---|---|
| started | 1.30 | 350 mm | — | — | |
| | 1.45 | 250 | 21 | 30 | |
| | 2.00 | 250 | 22+ | 44 — | No separation on adding equal vol of water |
| | :10 | 250 | 24 | 46 | |
| | 20 | 250 | 25½ | 47 | |
| | 30 | 250 | 26¼ | 48½ — | " " |
| | 40 | 250 | 26¾ | 46 | |
| | 45 | " | " | 47½ | |
| | 3:00 | " | 27½ | 49 — | " " |
| | 30 | " | 28 | 51 — | " " |
| | 40 | " | 28½ | 52 | |
| | 4:00 | " | 29 | 56 | No sep. on adding equal vol. $H_2O$ |
| | 15 | " | 29 | 57 | Yellow oil on adding 2x $H_2O$ but no sep. |
| | 30 | " | 30½ | 57½ | " |
| | 5:00 | " | 33½ | 65½ — | Separ. |
| | 30 | " | 40 | 72 — | Separation but less than before |

M.A.D

| | | |
|---|---|---|
| 100 cc | [illegible] | [illegible] |
| [illegible] | [illegible] | [illegible] |
| 500 | [illegible] | [illegible] |
| 1000 | [illegible] | [illegible] |

[illegible]

[illegible] 1st [illegible] 81°C

[illegible] [illegible] [illegible]

$$P_2O_5 + 3H_2O \longrightarrow 2H_3PO_4$$

C 35 - 22 gm, 19 cc.

C 36 - 34 cc

C 38 - 38 gm. 49 cc.

C 39 - 42 cc

C 33 butine 60 cc

C 34 butine 10 cc

6/6/15-0

[redacted]

[redacted]

[redacted]

| | | | |
|---|---|---|---|
| 3:50 | 2° | 7° | |
| 4:00 | 2 | 6 | |
| 4:05 | 5 | 7 | |
| 4:10 | 1 | 7 | |
| 4:15 | | | |
| 4:28 | -3° | 5° | reddish brown film on flask noted |

4:34 - all added

| | | | |
|---|---|---|---|
| 4:49 | start adding $H_2O$ | | |
| 4:50 | -3 | 18° | |
| | -10 | 40-50 | |
| 5:06 | -3 | 16.5 | |
| 5:12 | - | 11 | |
| 5:15 | -14 | 7° | |
| 5:30 | -15 | | |
| 5:45 | -15 | | |

6/6/50

| | |
|---|---|
| | 35 |
| | 40 |
| | 350 |
| | 400 |

L-12 NOV 29 1946

acetone

NOV 29 1946

tare - 489.3
1000
1487.3

NOV 29 1946

tare - 487.7
800.
1287.7

KOH
= 296

$CaC_2$

259 - tare
360
619

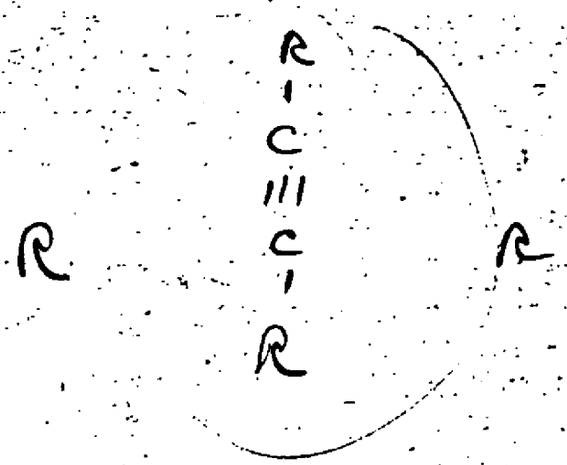
KOH

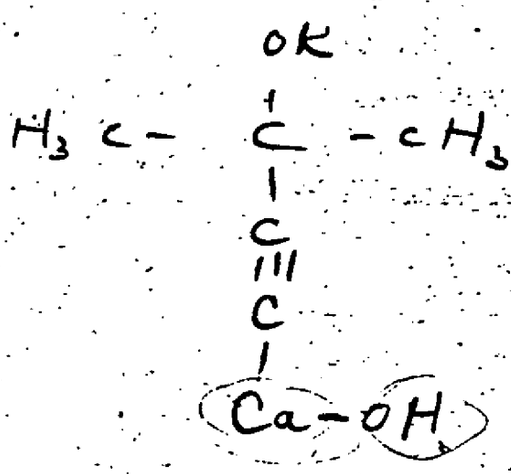
tare - 620.2
296.
916.2

$C_{10}$ start 3 37

| time | bath | reaction |
|---|---|---|
| 3 37 | 0° | 3° |
| 3 39 | 2° | 6° |
| 3 41 | 2° | 10° |
| 3 43 | 1.50 | 12° |
| 3 44 | 0° | 10° |
| 3 46 | −2° | 7° |

61.4 D  R = $Me_2CO$
38.8 H





519

397

18.5 Decimal

40

8.4

$\frac{64}{84} \times$

$\frac{84}{64} \times$

$\frac{18.5}{14}$

142

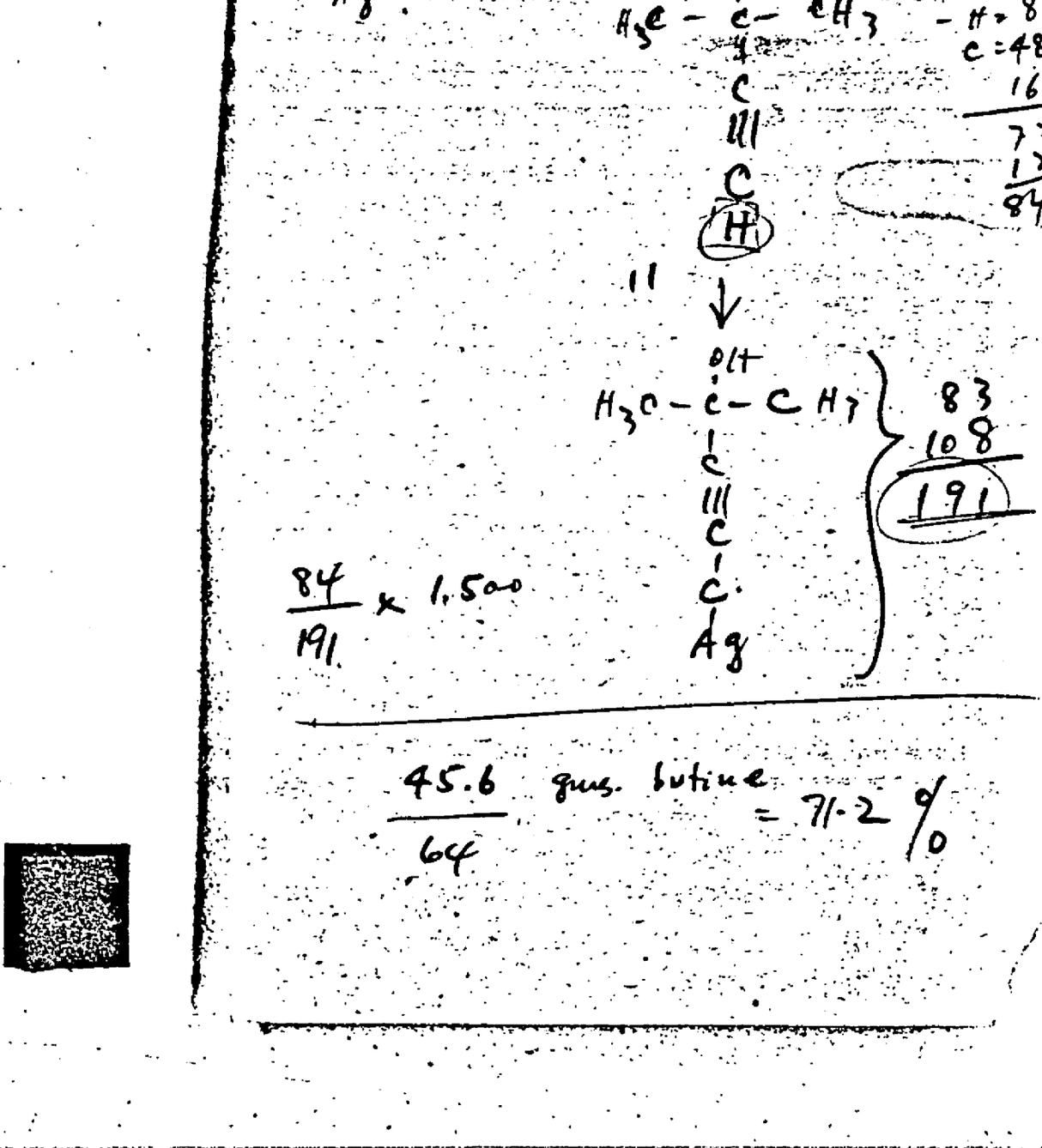
6.4

=

18.5 · $\frac{8.1}{1}$ $\frac{5.1}{8.4}$

1)

Ag = 108




$\frac{84}{191} \times 1.500$

$\frac{45.6}{64}$ gms. butine = 71.2 %

Ketone

49

4.7
0.5
5.7 g

$\frac{98}{84} \times 4$

$\frac{5.2}{0.90} = 5.8\ g.$

$\frac{5.7}{0.75} = 7.0\ g.$

$\frac{5.7}{0.90} = 10.4\ g$

$\frac{7.7}{0.75} = 10.3\ g.$

$\frac{5.7}{0.10} = 57.0\ g.$

12-2-46

C-10 ① lower layer after sludge removed by filtration, had a small amount of original upper layer and this was added to main upper layer.

② remainder of lower layer — about 400 mls. neutralized with 149 mls con. HCl

③ extract about 300 mls. of neutral solution with equal amount of ether; run off $H_2O$ - added balance of 300 mls. $H_2O$ solution to cancel ether + separated water.

the water solution was then re-extracted in the same manner

5:30 – start distilling.

| | B.P. | distillate | bath | Mm |
|---|---|---|---|---|
| 6:00 – | 45° | 29° | 52° | 700 |
| 6:30 | 43 | 45 | 60 | 680 |
| 7:00 | 41 | 47 | 70 | 700 |
| 7:20 | 55 | 48 | 75 | 675 |

12/3/46

| | distillate | vessel | bath |
|---|---|---|---|
| 11:25 – 80 mm. | 23.5° | 23.5 | 42° |
| traps – –30° to –40° | | | |
| 11:33 – 60 mm. | 28° | 27° | 45° |
| traps — 28° | | | |

11:45 — cut at 60 mm., 28°

2:00 – 150 mm. – take off bumps after, need to make residue from 5 liter to 1 liter flask

| | | dist. flask | b.t. | |
|---|---|---|---|---|
| 2[13] – 160 mm | | 26° | 34° | 41° |

mostly ether – acetone –
apparently close to cutting ft., since

2[17] – cut –

| | | | | |
|---|---|---|---|---|
| 2[21] | | 31 | 35 | 53 | 30 |
| 3[10] | 30 mm | 35 | 46° | 52 | 30 |
| 3[23] | | 38 | 48° | 65° | 30 |

close to end of lunimal

| | | | | |
|---|---|---|---|---|
| 3[37] | | 36 | 43 | 58 | 30 |
| 3[47] | | 39 | 48 | 70 | 28 |

bumping, close to end of fraction

| | | | | |
|---|---|---|---|---|
| 4[02] | | 42 | 48 | 72 | 28 |
| 4[12] | | 32 | about 55 | 80 | 25 |

↓ back of vapor → thermometer out of liquid

from about 3rd on, most

NOTE (I) of the material coming over was water, big allowance of condensate - apparently column does perform some separation

4th - stop - flask has about 5 mls. of material left

NOTE (II) 2 layers not visible. Some lactone in 5 cc layer since the residue was soluble in hot water

NOTE (III) yield - 518.6
tare - 208.7
309.9
2
312

51
4.5
255
20
2 29.5

29.5
2
59.0
279.0

C = 0.1 g Cumar / 100 c.c. THF

11-6-46

Poured in Me Hi (27° BP) at 4:00

5:20 — no powder

5:45 — Stop reaction —

some agglomeration — not hard

6/15/47

Mixed 300 mg Urinase in 300 ml 1% KOH, heated and filtered. Poured ~~off~~ 200 cc in 1 l flask with Hirschberg stirrer and reflux condenser.

Produced too much acid so added 100 cc water to ½ the volume

(50 mg Urinase/100 g KOH)

3.5 -

added 2 g Monomer Me Me
150 mg Benzoyl Peroxide
193.1 mg U-Butyl H.P.

results - Agglomeration

B - Same as A - Poured in Me Me at 1:40

results: Same as A

ARMA
ARMOURS

Ether –

tare flask + ring – 480.5
ring – 80.5

flask – 400.0

480.5
300 – ether
780.5

KOH

780.5
132
912.5

$CaCl_2$

913.6
160.
1073.6

acetone

tare – 239.6
116.
355.6

12-6-46

butine by Kazarian's method

Quantities

KOH - $\frac{56}{0.85} \times 2 = 132$ gms.

Ether - $150 \times 2 = 300$ gms.
(about 423 mls.)

$CaCl_2$ - $\frac{64}{0.8} \times 2 = 160$ gms.
(162.7)

acetone - $58 \times 2 = 116$ gms.
(about 145 mls)

ice-water - $150 \times 2 = 300$ mls.

423
145
200
300
1068

[redacted]

11/16

| Time | Min | Flask | Bath | Notes |
|---|---|---|---|---|
| 6:00 | 0 | 5° | 0° | start |
| 6:20 | 20 | 7° | 3 | - reddish brown film thinning up to top of flask |
| 6:25 | 25 | 6 | 4 | |
| 6:30 | 30 | 7 | 4 | |
| 6:35 | 35 | 7 | 5 | |
| 6:40 | 40 | 6 | 5 | |
| 6:45 | 45 | 8 | 5 | |
| 6:50 | 50 | 7 | 4 | |
| 6:55 | 55 | 7 | 4 | |
| 7:00 | 60 | 6 | 4 | |
| 7:05 | 65 | 7 | 3 | |
| 7:10 | 70 | 7 | 4 | |
| 7:15 | 75 | 8 | 6 | |
| 7:20 | 80 | 7 | 5 | |
| 7:25 | 85 | 7 | 4 | all |
| 7:30 | 90 | | | |

[redacted]

12/6/46

Neutron Data (cont'd)

| Time | mins | Fresh | Bath Water |
|---|---|---|---|
| 8[illegible] | 120 | 7 | 6 |
| 8[illegible] | 155 | | |
| 9[illegible] | 140 | | |
| 9[illegible] | 165 | | |
| 9[illegible] | 130 | | |

FILE DESCRIPTION

PHILADELPHIA FILE

SUBJECT HARRY GOLD

FILE NO. 65-4307

VOLUME NO. 1-B-13

SERIALS PART

B

# NOTICE

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Inventory Worksheet
FD-503 (2-18-77)

VOLUME 1-B-13 Part B

PHILADELPHIA FILES

INVENTORIED BY

REVIEWED BY

File No: 65-4307

Re: Harry Gold

Date: 6/78 (month/year)

| Serial | Date | Description (Type of communication, to, from) | No. of Pages Actual | No. of Pages Released | Exemptions used or, to whom referred (Identify statute if (b)(3) cited) |
|---|---|---|---|---|---|
| 13 | 6/6/50 | Bulky Exhibit Form | 1 | 1 | |
| 12K | — | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Folder containing notes of Harry Gold | 8 | 8 | some pgs both sides |
| 12L | | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Folder containing notes of Harry Gold | 23 | 23 | some pgs both sides |
| 12M | | Cover Sheet | 1 | 1 | |
| | | Folder containing notes of Harry Gold | 4 | 4 | |
| 12N | | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Folder containing research notes of Harry Gold | 20 | 20 | |
| 12O | — | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 5 | 5 | |
| | 6/6/50 | Folder containing notes of Harry Gold | 5 | 5 | |

FBI/DOJ

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FD-503 (2-18-77)

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|---|---|---|---|---|---|
| 12P | | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Folder containing notes of Harry Gold | 6 | 6 | |
| 12Q | | Cover Sheet | 1 | 1 | Enclosures sent to N.Y. |
| 12R | | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Folder containing notes of Harry Gold | 7 | 7 | |
| 12S | | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Folder containing "Notes from DoE" | 7 | 7 | |
| 12T | | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Notes of Harry Gold on $CO_2$ Recovery | 90 | 90 | |
| 12U | | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Folder containing notes of Harry Gold | 20 | 20 | |
| 13 | | Cover Sheet | 1 | 1 | |

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|---|---|---|---|---|---|
| | | Box containing 33 packets of notes of Harry Gold | | | |
| Packet #1 | — | Cover Sheet w/ notes | 24 | 24 | |
| #2 | — | | 5 | 5 | |
| #3 | — | | 3 | 3 | |
| #4 | — | | 3 | 3 | |
| #5 | — | | 3 | 3 | |
| #6 | — | | 19 | 19 | |
| #7 | — | Cover Sheet w/ let. from Bradin to A. Brotherman | 3 | 3 | |
| #8 | — | Cover Sheet w/ notes | 9 | 9 | |
| #9 | — | | 2 | 2 | |
| #10 | — | | 4 | 4 | |
| #11 | — | | 8 | 8 | |

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FD-503 (2-18-77)

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|---|---|---|---|---|---|
| pg #12 | — | cover sheet w/ notes | 6 | 6 | |
| #13 | — | | 3 | 3 | |
| #14 | — | | 2 | 2 | |
| #15 | — | | 2 | 2 | |
| #16 | — | | 4 | 4 | |
| #17 | — | | 4 | 4 | |
| #18 | — | | 2 | 2 | |
| #19 | — | | 2 | 2 | |
| #20 | — | | 2 | 2 | |
| #21 | — | | 6 | 6 | |
| #22 | — | | 12 | 12 | |
| #23 | — | | 4 | 4 | |

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PHILADELPHIA FILES

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| Serial | Date | Description (Type of communication, to, from) | No. of Pages Actual | No. of Pages Released | Exemptions used or, to whom referred (Identify statute if (b)(3) cited) |
|---|---|---|---|---|---|
| #24 | — | cover sheet w/ notes | 2 | 2 | |
| #25 | — | | 2 | 2 | |
| #26 | — | | 2 | 2 | |
| #27 | — | | 2 | 2 | |
| #28 | — | | 2 | 2 | |
| #29 | — | | 2 | 2 | |
| #30 | — | | 2 | 2 | |
| #31 | — | | 3 | 3 | |
| #32 | — | | 9 | 9 | |
| #33 | — | | 27 | 27 | |
| Encl. 14 | | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 1 | 1 | |

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FD-503 (2-16-77)

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|---|---|---|---|---|---|
| | postmark 6/2/48 | Letter from Dr to H. Gold w/ envelope | 3 | 3 | |
| encl 15 | | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 1 | 1 | |
| | — | misc. notes; booklet containing notes of H. Gold | 55 | 55 | |
| encl 16 | | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH. | 1 | 1 | |
| | 2/18/36 | U.S Patent Office Reprint | 2 | 2 | |
| encl 17 | | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH. | 1 | 1 | |
| | 9/26/44 | U.S Patent Office Reprint | 2 | 2 | |
| encl 18 | | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 1 | 1 | |

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|---|---|---|---|---|---|
| encl. 19 | — | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA Memo to SAC, PH | 1 | 1 | |
| | POSTMARK 11/25/47 | Envelope w/ "memo for Prep. of Thioglycolic Acid..." | 2 | 2 | |
| encl. 20 | — | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 1 | 1 | |
| | POSTMARK 12/3/47 | Envelope containing notes / documents of Harry Gold | 34 | 34 | |
| encl. 21 | — | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 1 | 1 | |
| | 6/6/50 | notes of Harry Gold | 8 | 8 | |
| encl. 22 | — | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH. | 1 | 1 | |
| | 3/3/32 | notes of Harry Gold | 86 | 86 | |

FBI/DOJ

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|---|---|---|---|---|---|
| encl. 23 | — | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 1 | 1 | |
| | 9/5/40 | 3X5 cards RE: METHYL METHACRYLATE | 32 | 32 | |
| encl 24 | | Cover Sheet | 1 | 1 | |
| | 7/10/50 | SA MEMO TO SAC, PH | 1 | 1 | |
| | — | 3X5 card w/name, add, phone # | 1 | 1 | |
| 14 | 6-6-50 | Cover Sheet | 1 | 1 | |
| | 6-6-50 | Investigative notes of SA. Burgess re: Harry Gold. | 47 | 47 | |

FBI/DOJ

Entire exhibit (about 1" thick) submitted by Special Agent [illegible] (continued on 2-1-51)

FD-161
(7-1-48)

BULKY EXHIBIT

Date received 6-6-50

HARRY GOLD

ESP R

(Title of case)

Retyped

Returned 7-11-52 per [illegible]

Submitted by Special Agent Fred C. Birkby

Source from which obtained Search of Subject's residence

Address 6823 Kindred St., Phila.

Purpose for which acquired Aid in investigation--evidence

Location of bulky exhibit Exhibit Room

CS-4 J67-1-B-13

Estimated date of disposition 12-1-50

Ultimate disposition to be made of exhibit to be determined

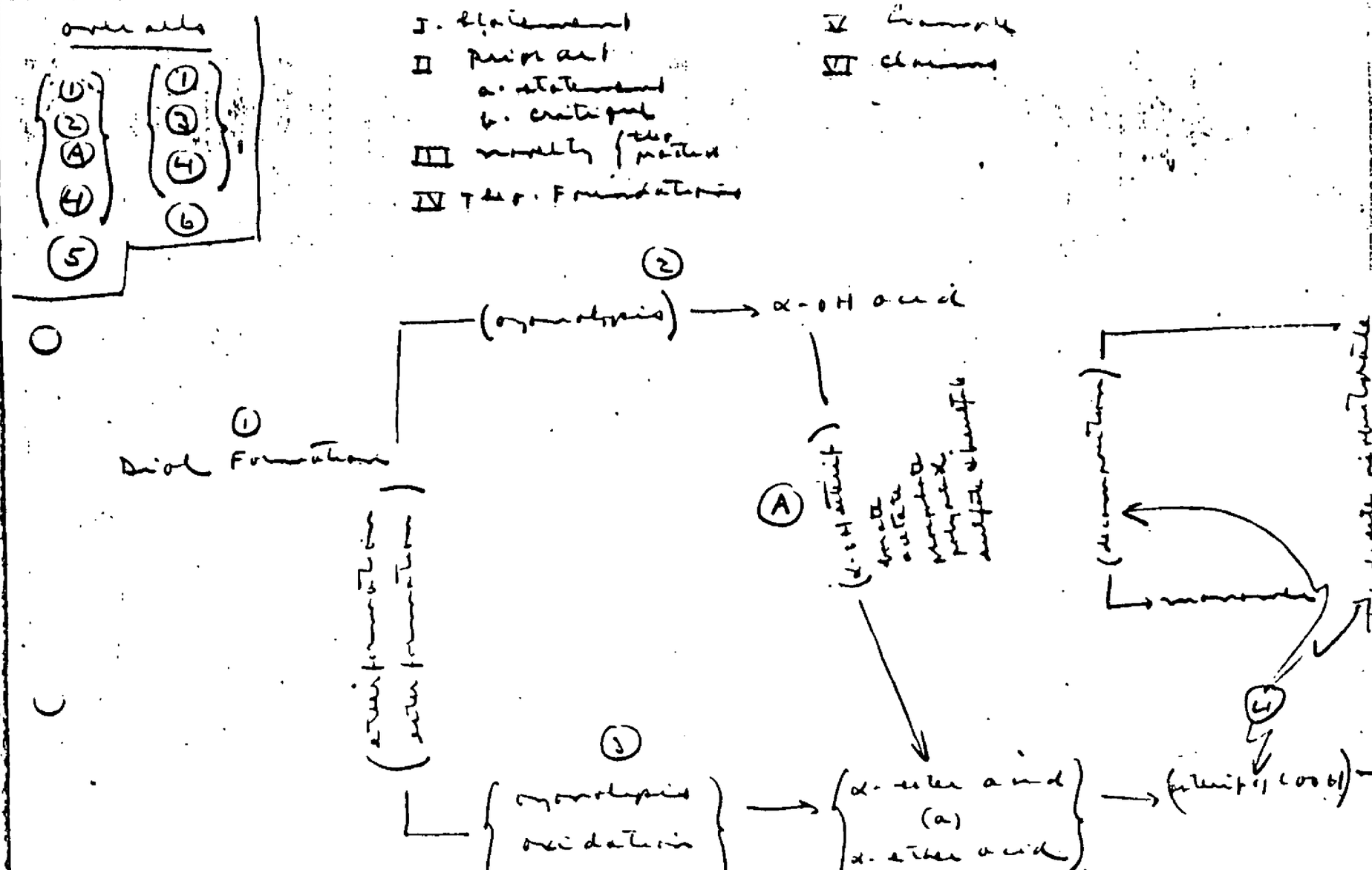
| | |
|---|---|
| SEARCHED | INDEXED |
| SERIALIZED | FILED |
| | NOV 21 1951 |
| | FBI - PHILA. |
| | 12B |

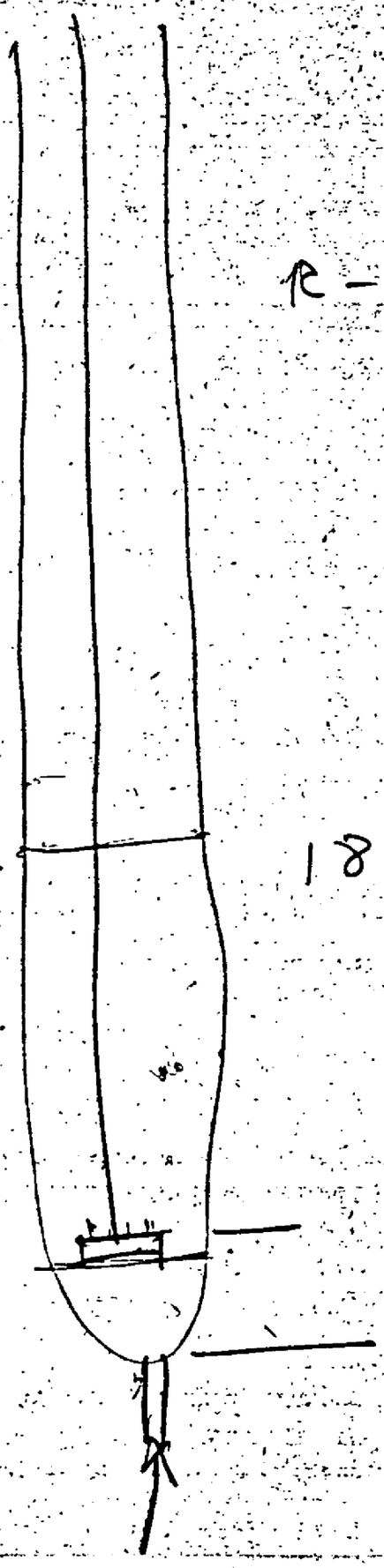
List of contents:

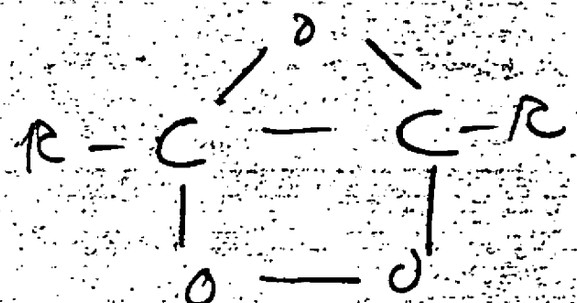
1. 21 manila folders numbered 12A thru 12U inclusive containing misc papers found on fourth shelf of wooden cabinet in basement.
2. 140 misc sheets of paper handwritten from fourth shelf of wooden cabinet in basement.
3. One envelope addressed to subject postmarked June 2, 48 at Phila from Morrell E. Dougherty 5617 Litchfield St. Phila 43, Pa., containing one sheet of paper signed Doc.
4. Bodge Composition Book containing chemical formulae.
5. Photostat of US Patent Office document #2,030,901.
6. Photostat of US Patent Office document #2,359,212.
7. Federal Standard Stock Catalogue L-P-406a Jan 24, 1944.
8. One 21 page "Memorandum for the preparation of Thioglycolic Acid Patent Papers" enclosed in brown envelope.
9. One brown envelope bearing return address "The Girdler Corp., Louisville, Ky. containing 15 misc papers.
10. Manila folder containing six misc sheets of paper.
11. 47 sheets of loose leaf paper containing seven punch holes and bearing penciled notations. Found on first shelf of wooden cabinet.
12. 20 3X5 index cards. Found on second shelf of Wood cabinet.
13. Slip of paper containing name of James A. Devlin. Found on 2nd shelf of wooden cabinet in basement at 6823 Kindred St.
14. Investigative notes of SA Burgess re search of Gold's home on above date.

Part B = 12K thru 14

Case
2/6/50



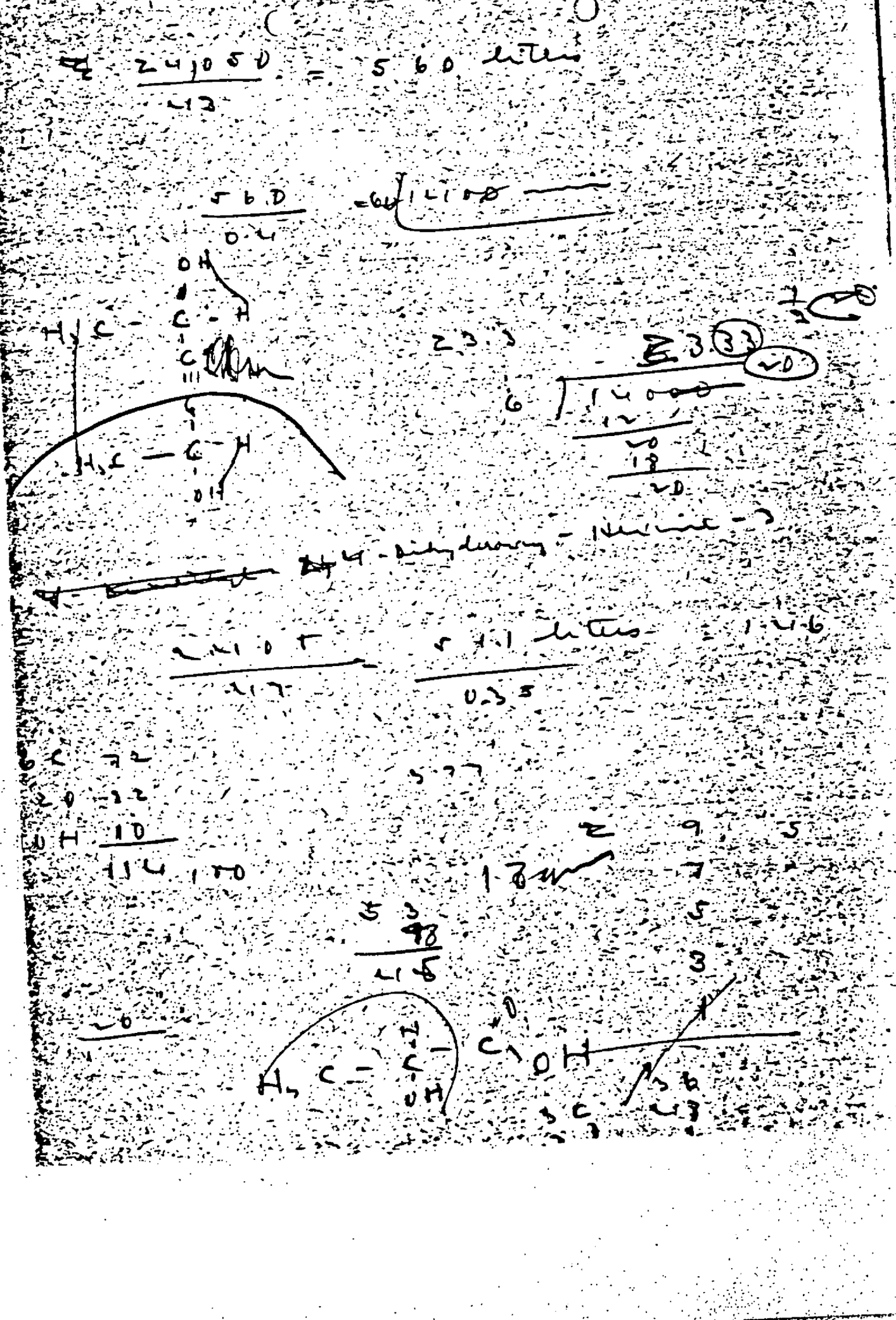


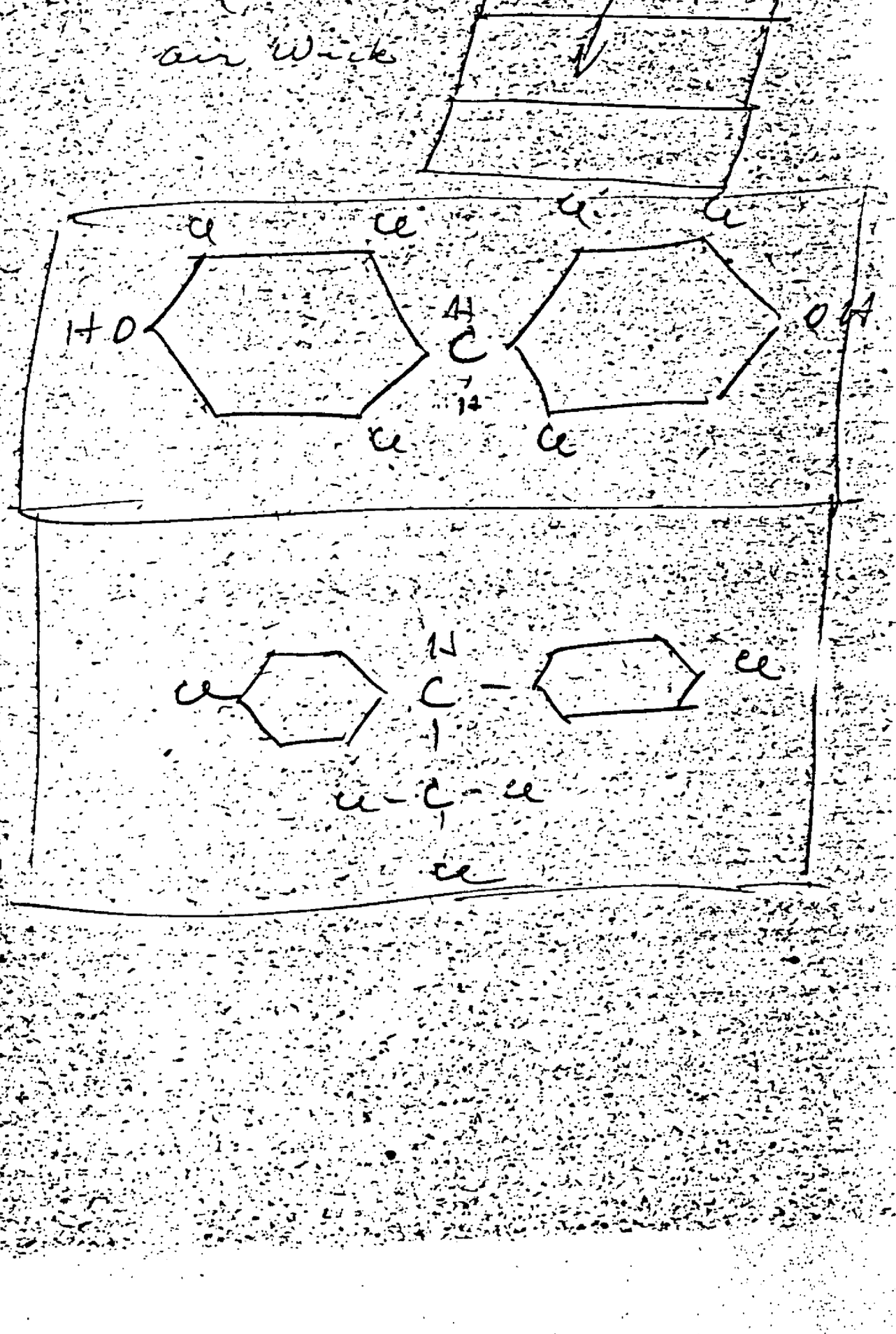


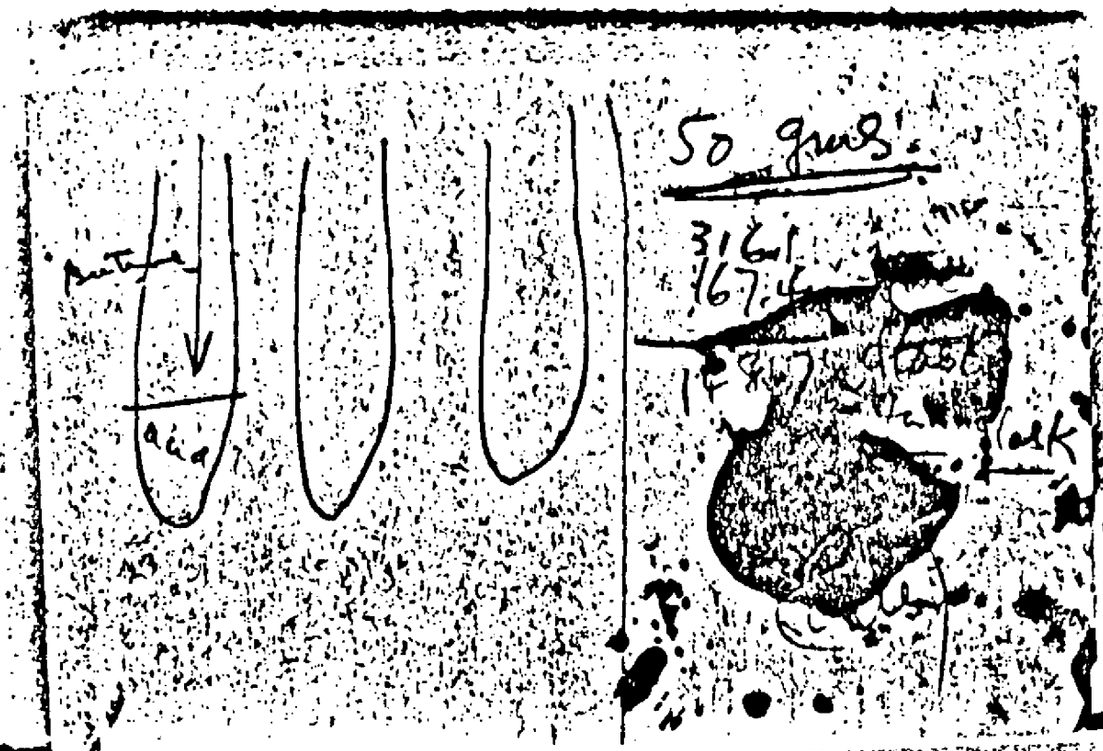
18 cm

1. Titration in alkaline
titrate with KI
for organics in
$O_3$ or $H_2O_2$

3. Extract with
ether for acid






50 grms

start 4:30

| time | dist. | flask | bath |
|---|---|---|---|
| | | 78° | 106° |

column temp. - 35°

4:41 - 60° - 99.5 133

first drops

4:51 - 100° 104° 148

6/6/50

$2Y_2 = 4Y_2 = 8$
$4K_4 = 16K_4 = 64$

$2FeS + H_2SO_4 \rightarrow H_2S + 2FeSO_4$

16 min/cuft

16 min

1 cuft = 7.5 gal / .475 gpm

$\frac{91(98)}{37(0.69)} = 370$ #/hr

$\frac{370}{60(8.35)(1.53)} = 0.475$ gpm

150 ea
100 ea
250
350

18 | 18 ⇒

370 gpg

1200

6000 ea
1240
124
480 ea Valves
1928
700 ea
2629
6336 ea

$60

$862 ea

1088
108.80
1197.8
165

Size #4, 1½" Fig 735-02 Flowdon.
end-bell type "F" pneumatic controller
all welded parts constructed of Hast "C"
complete with 1½" Hast "C" D.M.V assy.
for a press. drop of ____ PSI gr. @ max exp.
To control a normal flow rate
of 0.475 GPM of 69% $H_2SO_4$ @ 70°F.

Hart → $863 ea

Lead → $636 ea

65-1347-1-B-13(1)

C-5-4307

7212

Note:

(1) Stated reflux at 2$^{12}$ — No steady reflux, but irregular bumping.

(2) Vapor temp. under approximately equilibrium conditions was 64-66°

(3) Flask temp. thermometer remained close to 77° — at least from time thermometer was inserted at about 4$^{00}$ PM.

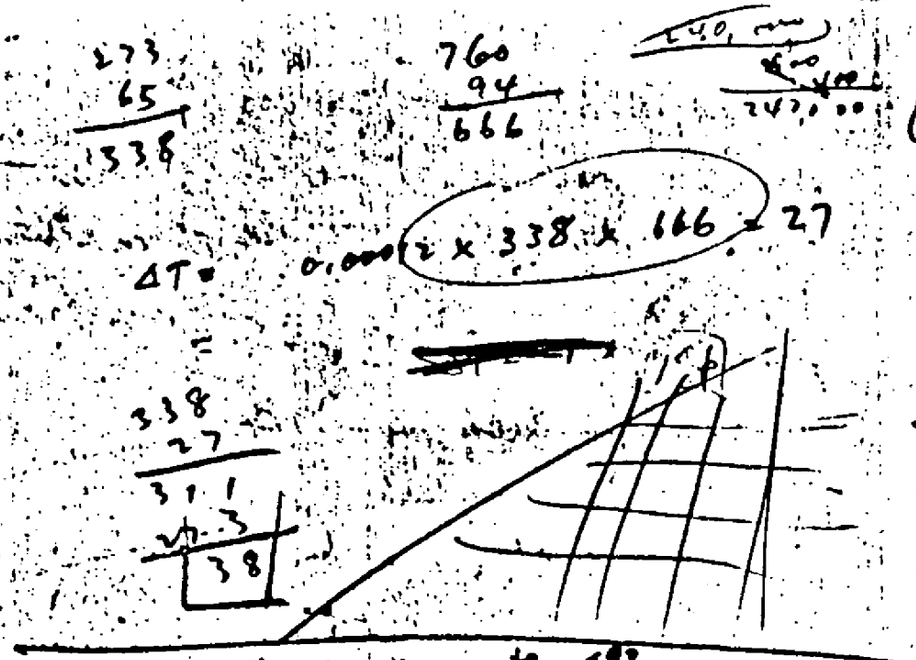
Submitted:

Turned off flame at 7:15

6-6-50
ae

Buffalo Locate Tom
Rolling Mill
Am. B+E Ltr
50 @ 2300
cl 3-4600

7098-E 2 2



I $CH_3-\underset{CH_3}{\overset{OH}{\underset{|}{\overset{|}{C}}}}-COOH + HOCH_3 \xrightarrow[\text{dry}]{HCl} \xrightarrow{\text{silica gel}}$ to 79°

II $CH_3-\underset{CH_3}{\overset{OH}{\underset{|}{\overset{|}{C}}}}-COOCH_3 \rightarrow$ distil under vacuum

III reflux ↓ silica gel → distil under vacuum
residue - HCl odor
distillate - methanol odor

IV or only methanol, OH acid to 79°, + water

Possibilities of

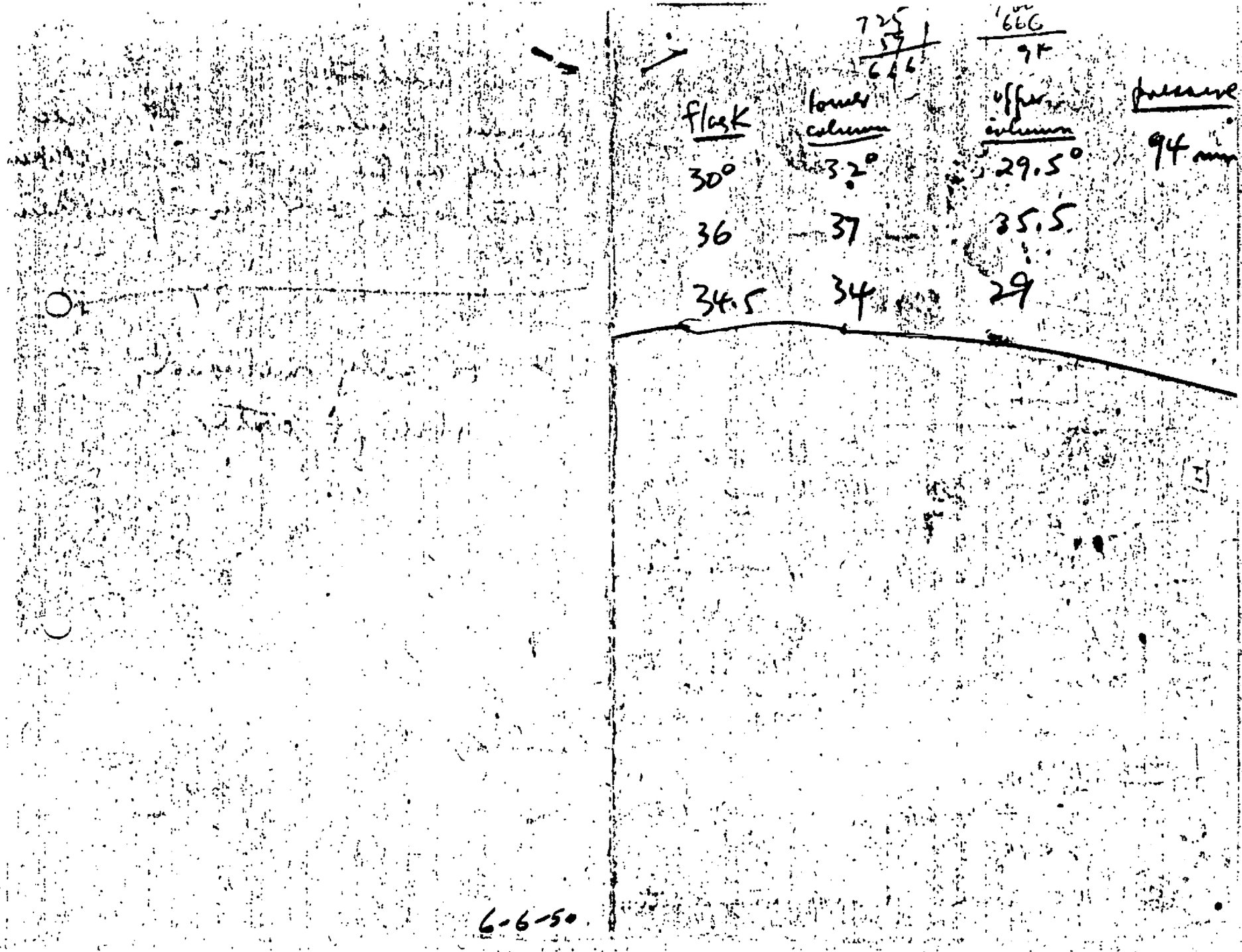
① $CH_3-\underset{CH_3}{\overset{Cl}{\underset{|}{\overset{|}{C}}}}-COOCH_3$ formed during esterification

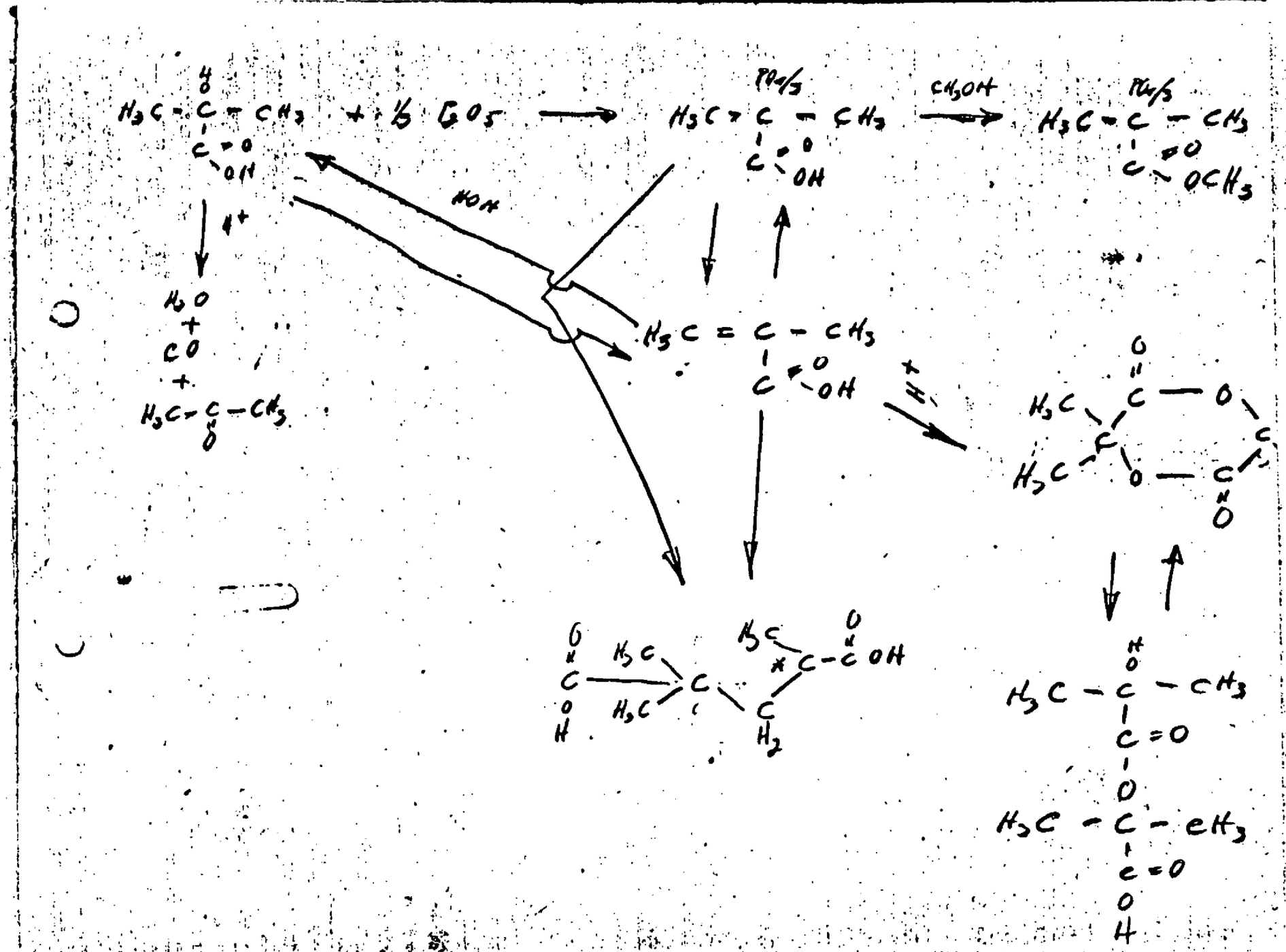
② HCl formed during esterification

α-OH substituent to acid + methanol

④ on distillation, HCl azeotrope remains behind as residue, while possibly another azeotrope of methanol + HCl comes over, giving methanol odor

yesterday's reflux exp.


725
666
flask
lower column
upper column
pressure
30°
32°
29.5°
94 mm
36
37
35.5
34.5
34
29
6-6-50.

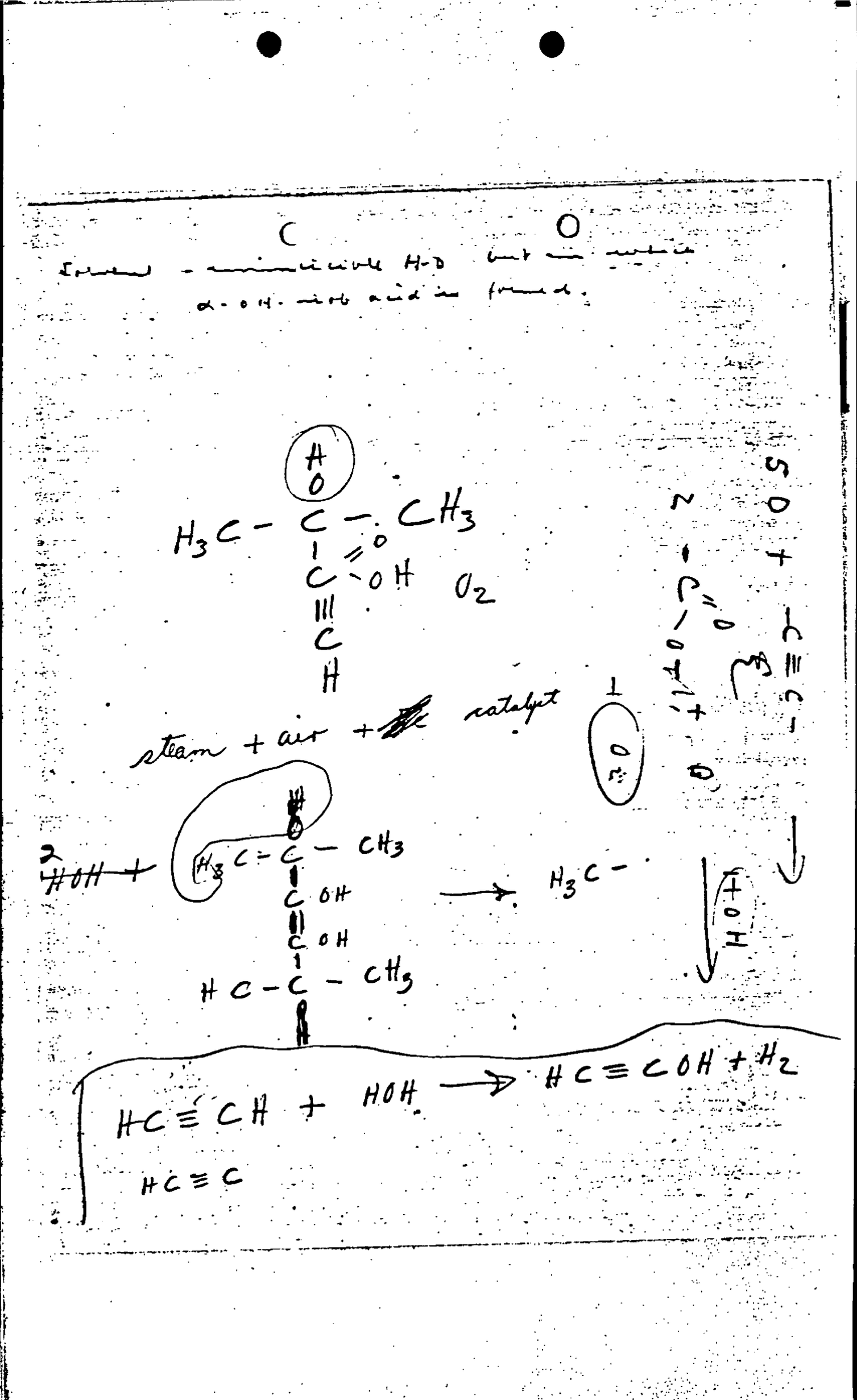

H3C - C - CH3
C = O
OH
+ ½ CO5
PO4/3
CH3OH
OCH3
H+
H2O
+
CO
+
H3C - C - CH3
O
HOH
+H+
H3C
C
O
OH
H
H2
H3C - C - CH3
C = O
O
H3C - C - CH3
C = O
O
H

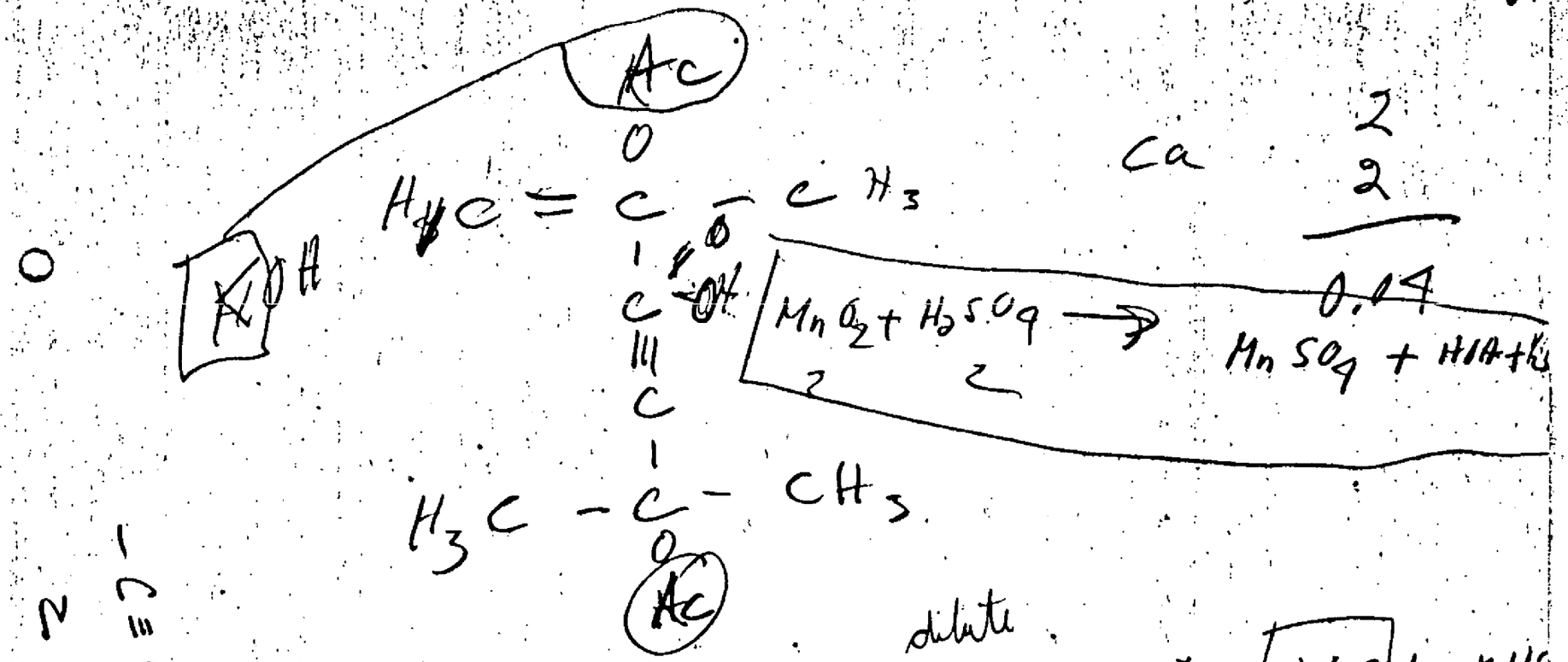
C O

1. Pyrolysis with {air / oxidizing agent} of acetylene cpds.

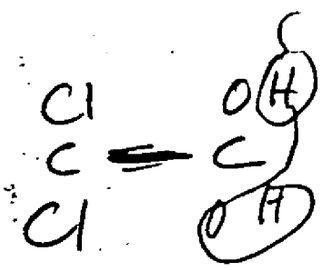
Vapor phase esterification

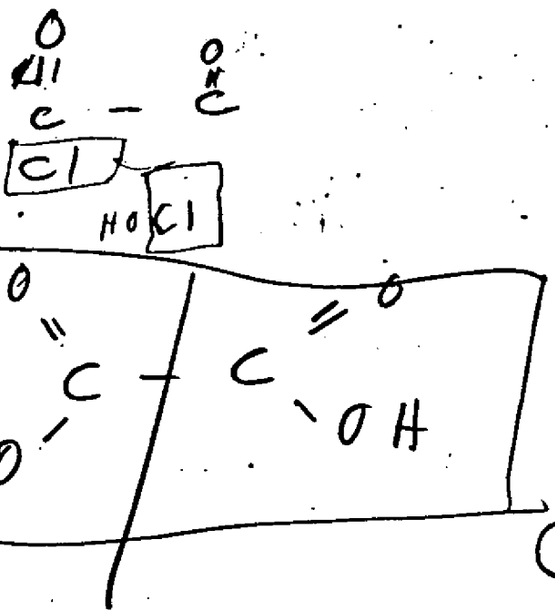
Hood article

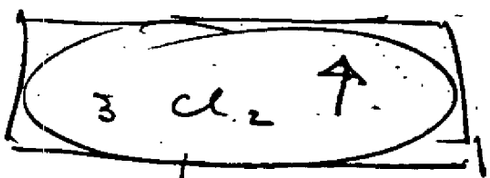

steam + air + catalyst
2 HOH +
$H_3C-C\equiv C-CH_3$
COH
COH
$HC-C-CH_3$
$H_3C-$
$HC\equiv CH + HOH \rightarrow HC\equiv COH + H_2$
$HC\equiv C$

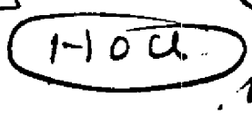

Ac
OH
$H_2C = C - CH_3$
O
C
C
$H_3C - C - CH_3$
O
Ac
$MnO_2 + H_2SO_4 \rightarrow MnSO_4 + H_2O$
0.04
Ca
2
2
0.04
dilute
$KAC + HHSO_4 \rightarrow HAC + KHS$
1 mol
1 mol

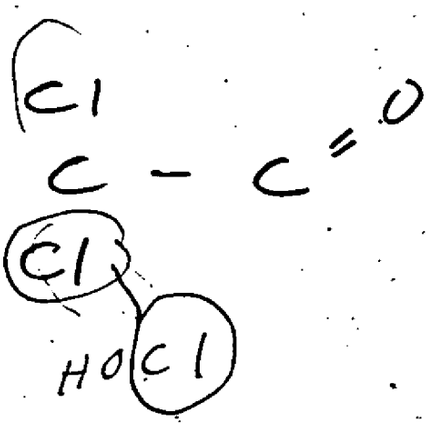
1.

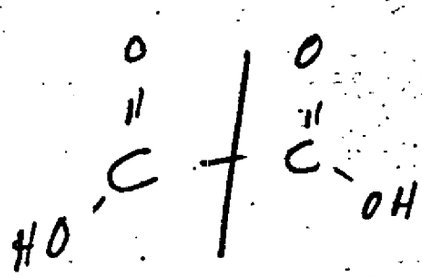


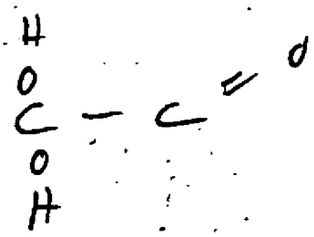












~~[illegible]~~ with $Et_2O$

~~[illegible]~~

cool to 0°C & add 300 cc of methylal

let temp rise to 13–15°C & agitate

for 1 hr at this temp.

Pass in $CH_2O$ for ~1 hr at 0°C

let rise to 13–15°C.

add 38 gm. acetic slowly ~~[illegible]~~

& keep temp at 13–15°C

~~[illegible]~~ warm to room temp & let stand for

31–35 hrs. Keep at 13–15°C.

Keep up reaction for 2 hrs after this.

CA. [illegible],3926,

CA. 33,1259

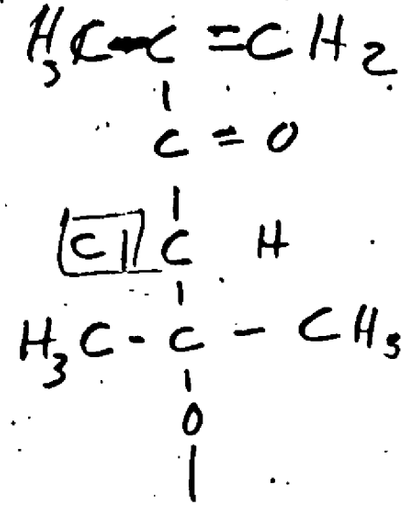
CA. 29,2169

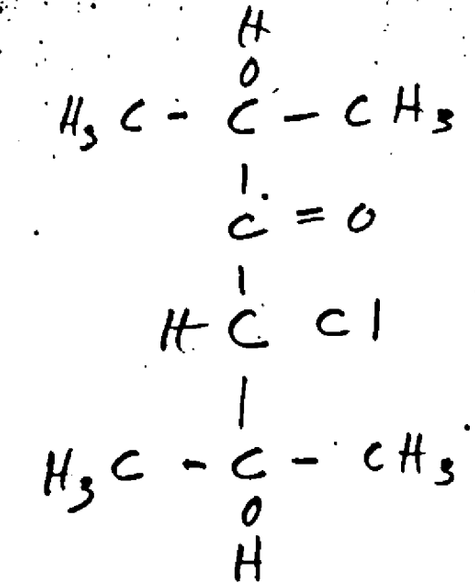
U.S.P. 1,396,161

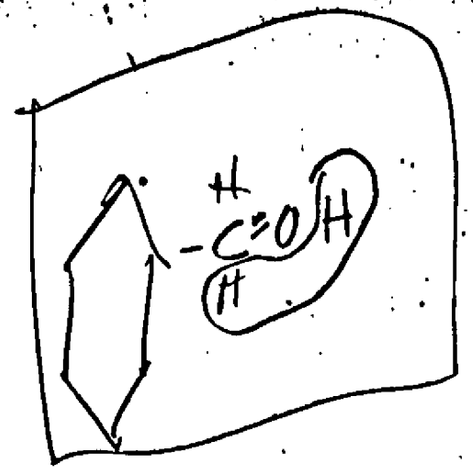
CA. 21,6502

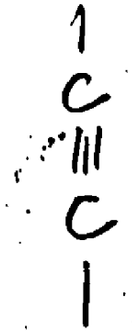
Sintet Kautschuk 1934 (9) 12

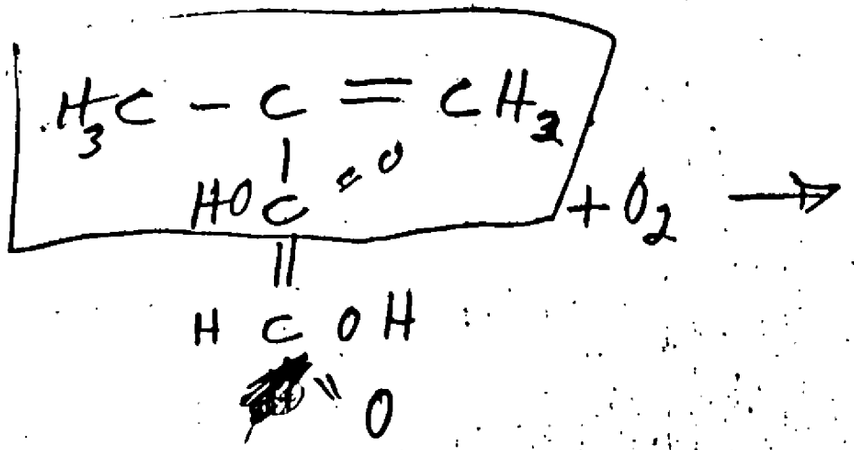
Sintet Kautschuk 1934 R7



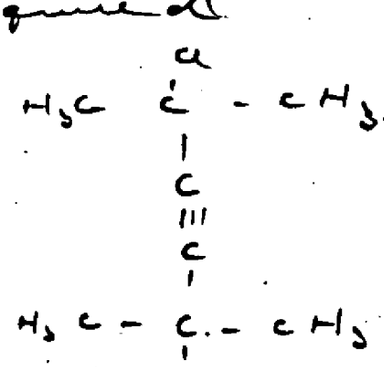


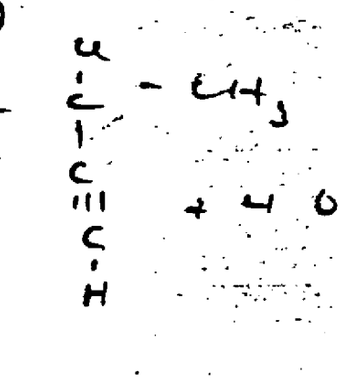






$O_2$ required





| | |
|---|---|
| 5 C | 60 |
| 7 H | 7 |
| 1 Cl | 35 |
| | 102 |

102 g req 64 g. O

$\frac{5}{102} \times 64 = 10\sqrt{\frac{30}{306}}$ 3.1 g. O

3 Vol % $O_2$ = 1.009 Ltr.

50 × 0.02 = 1.0163 =

$\frac{1.5}{1.46}$

$\frac{90}{60}$

$\frac{15}{1.190}$ 1.19 g.

$\frac{3.1}{2.5} \times 50 =$

$\frac{31}{50}$

$\frac{1510}{}$

70 cc

$10\sqrt{1510}$

124

$\frac{70\ cc}{50} \times 0.\frac{5}{} = \frac{35}{0.175}$ g. $FeSO_4$

3 moles α-C dissolved in MeOH
1 mol 4 mol
Silica gel HCl dry for 2 hrs
Cool to RT and pass HCl

Stop agitation
Filtered
Wash silica 2X 1½ c.c. gCH₃OH
Distill
α-hy acid 71°C
α-Chlor acid
Methyl α-hydroxy isobutyrate 126
Methyl α-Chlor isobutyrate 135

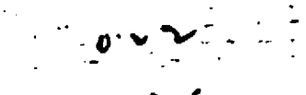
Cuts up 75°
75-100
100-120
120-130
130-145

Reflux

1-6-50

Ternary solubility of $NaOH$, $C_6H_6$ & $H_2O$
at 20°C

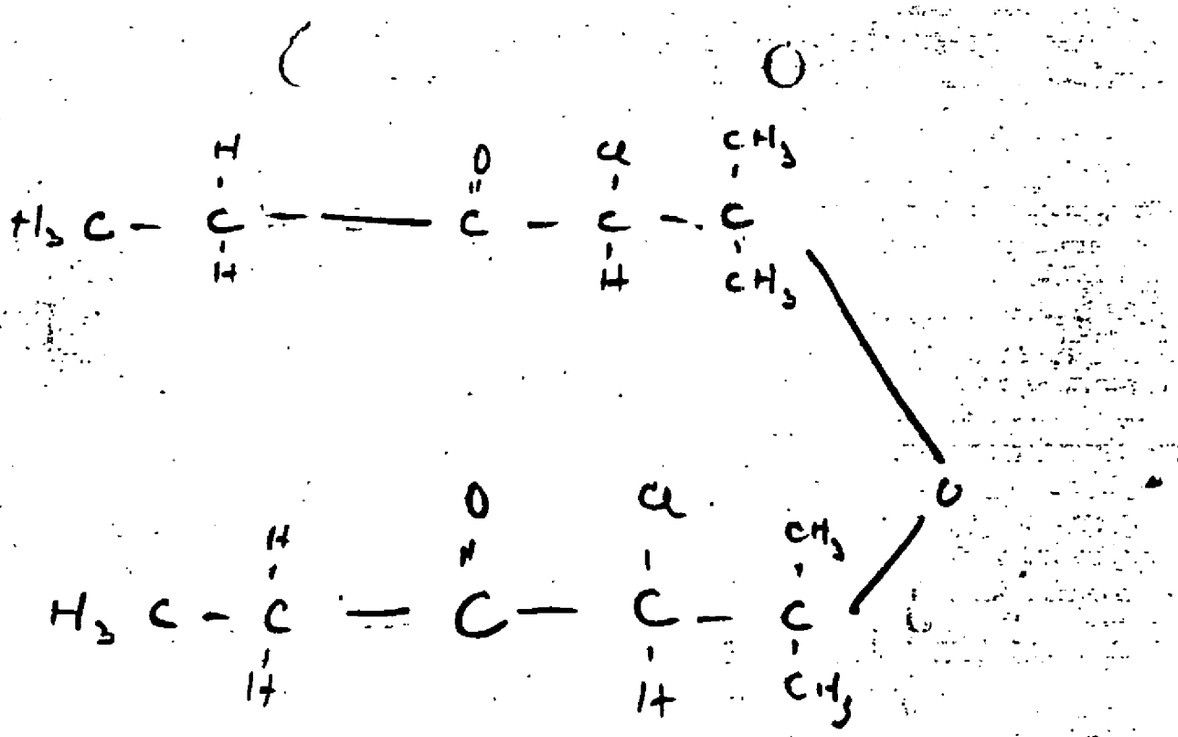
| wt % NaOH in NaOH-$C_6H_6$ mixture | wt % NaOH in the powdered clear soln. | wt % NaOH in the moist solid |
|---|---|---|
| 0.0 | 0.95 | |
| 1.0 | 1.66 | |
| 2.0 | 2.57 | |
| 3.0 | 3.57 | |
| 4.0 | 4.13 | |
| 5.0 | 5.1 | |
| 6.0 | 6.1 | |

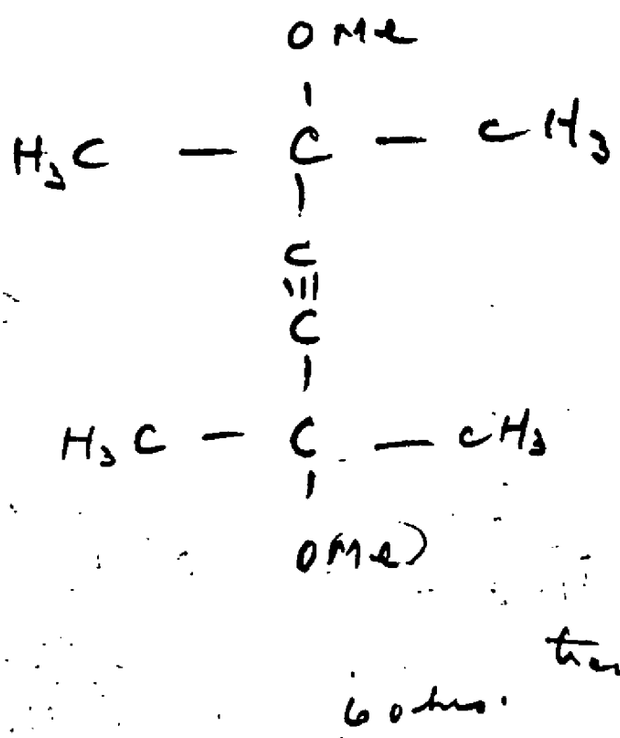


Moist solid

per 100 cc $C_2H_5OH$

| cc $H_2O$ | cc $C_6H_6$ |
|---|---|
| 5.0 | 0.15 |
| 3.0 | 0.25 |
| 2.0 | 0.49 |
| 1.0 | 1.00 |
| 1.4 | 1.9 |
| 1.0 | 9.0 |
| 0.50 | |

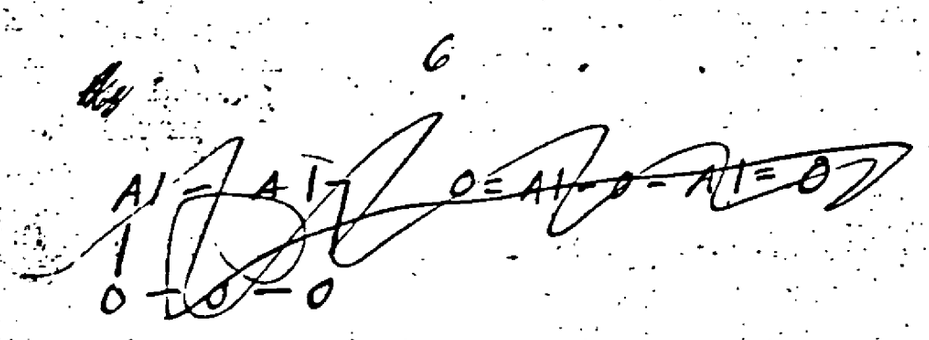




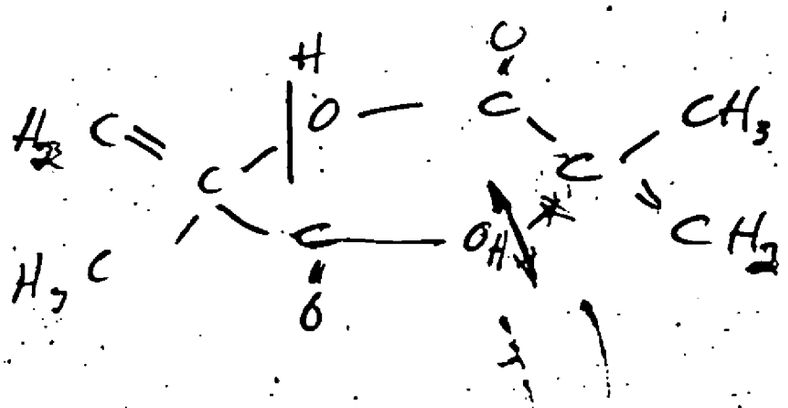
$CH_3OH$

tracce I

$Al_2O_3 + 3HOH \longrightarrow 2Al(OH)_3$



$Al_2O_3 + 3HOH \rightleftharpoons H_3Al_2O_3 \rightleftharpoons H^+ + H_2Al_2O_3^-$



SOLUBILITY - GEW PER 1000 g. H2O
10
20
30
40
50
0
20
40
60
80
100
120
140
160
TEMPERATURE - DEGREES C
SOLUBILITY OF KOH IN WATER AS A FUNCTION OF TEMPERATURE
CURVE CALCULATED FROM MIN

6-9-47

Vanishing Cream Formulae

No. 1 - T.E.A

| | |
|---|---|
| Stearic acid | 72 |
| A { T.E.A | 3 |
| A { Glyc | 24 |
| B { H O H | 201 |

No. 2 - $K_2CO_3$

| | |
|---|---|
| Stearic acid | 72 |
| $K_2CO_3$ | 0.9 |
| Glyc | 24 |
| H O H | 1000 |

L° = 82

No. 3 - Lanolin

| | |
|---|---|
| A { Stearic acid | 80 |
| A { Lanolin | 4 |
| B { H O H | 66 |
| B { Glyc | 8 |
| B { $K_2CO_3$ | 0.5 |

A. BROTHMAN & ASSOCIATES

No. of
Date:
By:

JOB:

SUBJECT:

Scientific Glass

J-1214 Pyrex glass helices $5.00
1/4 lb 1/8" coil diam

J-1057 Distilling column, Intejoint, pyrex
1- Column length 30cm. I.D. 12mm $4.00
1- " " 60 cm. " " 12mm 4.50
1- Air jacket #1
1- " " #3

J-1706- Flasks, 3-neck, vertical side joint
2- 29/42 joints on all 3 necks

~~J-1697~~
J-1691- Flask r.b. short neck. 24/40 ♀ joint
1- 100 cc. flask
2- 250 cc. "
1- 500
1- 1000
1- 2000



100 ± 15 605 Jun 2 rounds Cover cap

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By:

3 only # S-752 Channel Controlling and Ch.

3 only # S-754 " " " "

3 only # S-743 " " " "

1 only # 14-900 Control Room Console

1 only 14-930 Middle

1 only # 14-570 Furniture

## The Manufacture of Thioglycolic Acid
(8/20/47)

A tank of 8½ I.D. is used. Other measurements not taken. The following is the sequence of operations:

1. Fill tank with water to height of 26 inches.
2. Heat to 40° C.
3. Add 3,475 lbs. monochloracetic acid. ) No additional heat during this period.
4. Add 2,475 thiourea. ) Agitation is maintained throughout the addition.
5. Heat is then applied through the coils (steam) to the solution, bringing it up to 80° C. Additional heat is then put into the system for a period of one hour to achieve a temperature of 95° C., at which it is maintained for two hours, making a total of three hours of sustained heat.
6. The system is then cooled to 70° within one hour.
7. Add 3,950 lbs. caustic soda with an equal weight of water and mix. This mixture is added over a period of 1½ hours; faster addition will make for unbearable ammonia fumes.
8. After all of the soda is mixed with the water, bring the temperature up to 95° C. for three hours.
9. Cool to 50° within one hour.
10. Add 44 carboys (net weight each carboy, 174 lbs.) of sulfuric acid, of a sp.g. of 1.600. After the addition of 15 to 17 carboys, excessive foaming takes place, so at about that time the addition of the acid to the system should take place slowly. Once the foaming period is over, the addition can be more rapid.
11. Add 150 lbs. zinc dust. (This material causes terrific odors, sneezing, etc.)
12. Cool to 25° C.

---

### Raw Materials Required

1. Water
2. 3,475 lbs. monochloracetic acid.
3. 2,475 thiourea.
4. 3,950 lbs. NaOH
5. 7,656 lbs. $H_2SO_4$
6. 150 lbs. zinc dust

V.C. Formulations

135 gm Citric acid
- 25 c.c. H2O
- 1 gm. NaOH
- 100 gm. Citric acid

Heat to 90°C + add Citric acid

Add 2 gms. Talcum Powder and

Let cool.

# THE PREPARATION OF UREA FORMALDEHYDE COLD-SETTING GLUE

8/21/46

The following is the procedure for the preparation of the urea-formaldehyde cold-setting glue:

To a 5 liter, three-necked flask immersed to batch-content-level in a bath capable of maintaining the batch at a temperature between 20° and 25° C., add 2622 gms. of 37% by weight formaldehyde-in-water solution. 1080 gms. of urea should be added to the formaldehyde, under agitation. The solution should then be corrected to a pH of between 7.3 and 7.5 by the addition of approximately 13 mls. 1N NaOH, the amount depending upon the initial pH of the formaldehyde solution. The reaction mixture should be maintained within the specified pH and temperature levels for a period of 24 hours. At the end of this time the conversion of formaldehyde and urea to methylol urea and dimethylol urea should be virtually quantitative.

The solution should then be adjusted to a pH of 5.0 by the addition of approximately 26 cc. of 1N concentration acetic acid. The temperature of the mass should be raised to reflux temperature in a period of not more than 30 minutes. The solution should then be adjusted to a pH of approximately 7 to 7.5 by the addition of approximately 40 cc. of 1N NaOH. The solution should then be concentrated to 70% resins-in-solution concentration under a vacuum of 200 to 400 mm. of $H_2O$. At this point the formation of the resin glue solution has been accomplished.

The preparation of the glue mixture involves the following procedure:

To 100 gms. of resin glue solution add 7 gms. of walnut shell flour. The walnut shell flour should be added progressively and dispersed as well as possible in the glue solution. This can be accomplished by hand-stirring, employing a glass rod in a beaker, when walnut shell flour of +200 mesh to -300 mesh is employed. The proper dispersion of the walnut shell flour depends on the addition of the flour at a rate under continuous agitation such that at no time is there a significant amount of walnut shell flour present in an undispersed form.

To the thus prepared flour-and-glue-solution dispersion, there should be added 1 cc. of a water solution of 11.5 gms. of ammonium chloride in 250 cc. of water. This catalyst mixture should be well dispersed in the flour-glue-solution dispersion and the resulting mixture should then be allowed to rest for one and one-half hours. This mixture should demonstrate, at the end of the mentioned one-and one-half hours, a pH of about 4.5.

The final glue mixture should then be spread between the yellow birch veneer panels comprising the ultimate plywood composite board, so that 20 to 25 gms. of glue mixture are spread for each square foot of glue line. The plys should then be placed in a press at 100 psi pressure for a period of 24 hours and maintained at a temperature of 83 F. for the entire interval. At the end of the 24 hours the resulting plywood should be permitted to cure at a temperature of not less than 75 F. for a period of six days. At the end of the specified interval, specimens may be cut and prepared for testing.

A. BROTHMAN & ASSOCIATES

No. ____ of ____

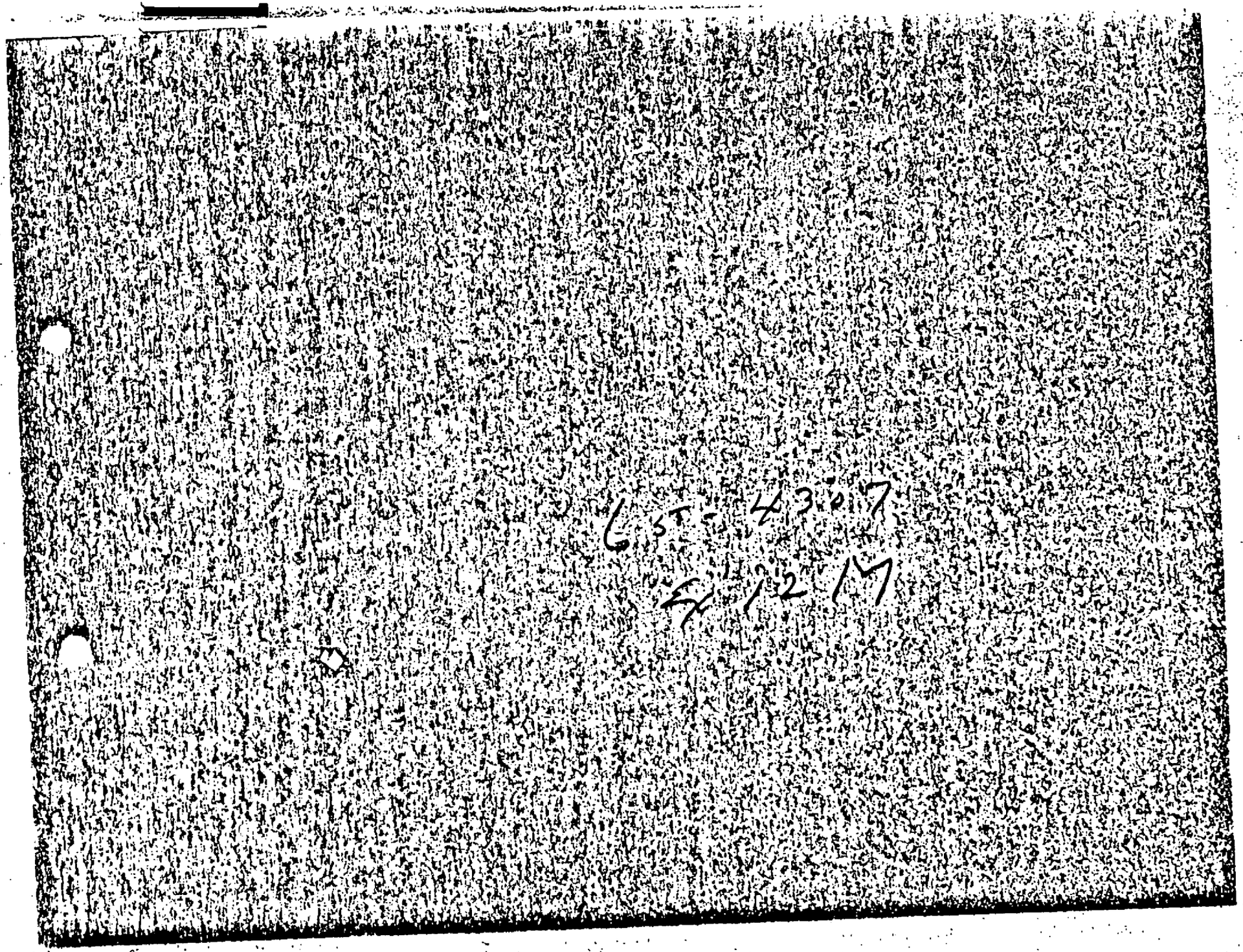
Date: ____

By: ____

JOB: ____

SUBJECT: ____

1. Satisfactory Monomeric Mixture
   a. complete formation of methylol & dimethylol urea
2. Satisfactory Polycondensation Procedure
   a. constant pH control — pH
   b. end-point test — true solution
      a. viscosity
      b. "extension principle"
3. Hardening Operation
   a. filler addition
      1. grinding?
      2. quantity
   b. anti-crazing reagent
   c. pH vs time curve


GST 43.17
EX 12.17

12 M
2nd
6-6-50

3-10-48

Synthesis of Dimethyl Nitric Acid

Solvent AcOH (glacial) 140 cc

Charge 258.7
7.1 gms. Acid (20 mols)
265.8

Time 2.4 gms.

% Conv in bulk 0.703 gms./Pt

Time of synthesis 40 min.

Vol of Gas 3.7 ft³ (Time)

Gas passed Thru 2.52 gms.

Gas absorbed 0.89 gms.

3-30-68

Synthesis of Dimethyl Nerolic Diol

Solvent H O H 200 cc.

Isobutyraldehyde 14.2 (0.1 mole)
3.8
18.0

Diethyl zinc 21.8 grams.

70% hexane in bottle 0.705 gm/ml Rate 0.496 liters/min @ 9:30
Mass concentration.

Tank Final 9:05

Tank at start 2:45

Vol. Rec. 13.117
6.148
6.969

Integrated Rec. 6.8276

Rate 0.62 L/min @ 9:52

Final Vol of Solution 217 cc

Ideal Rise from 10°C → 22°C C

Titration of Final Solution (calculated) 4.870

4.31 = 0.970 × 0.104 × $\frac{217}{10}$ = 9.414 mm.
α-OH J.B. and

Titration of Ideated (30 minutes) Solution

100 cc → 9.8 cc NaOH 52.9

Isobutyraldehyde b.p. 155°C (m. 156°C Tag.)

3-10-48

Synthesis of methyl Butanoid

Solvent H O H 8 1 cc

Charge 3.6 gms.

Time of run

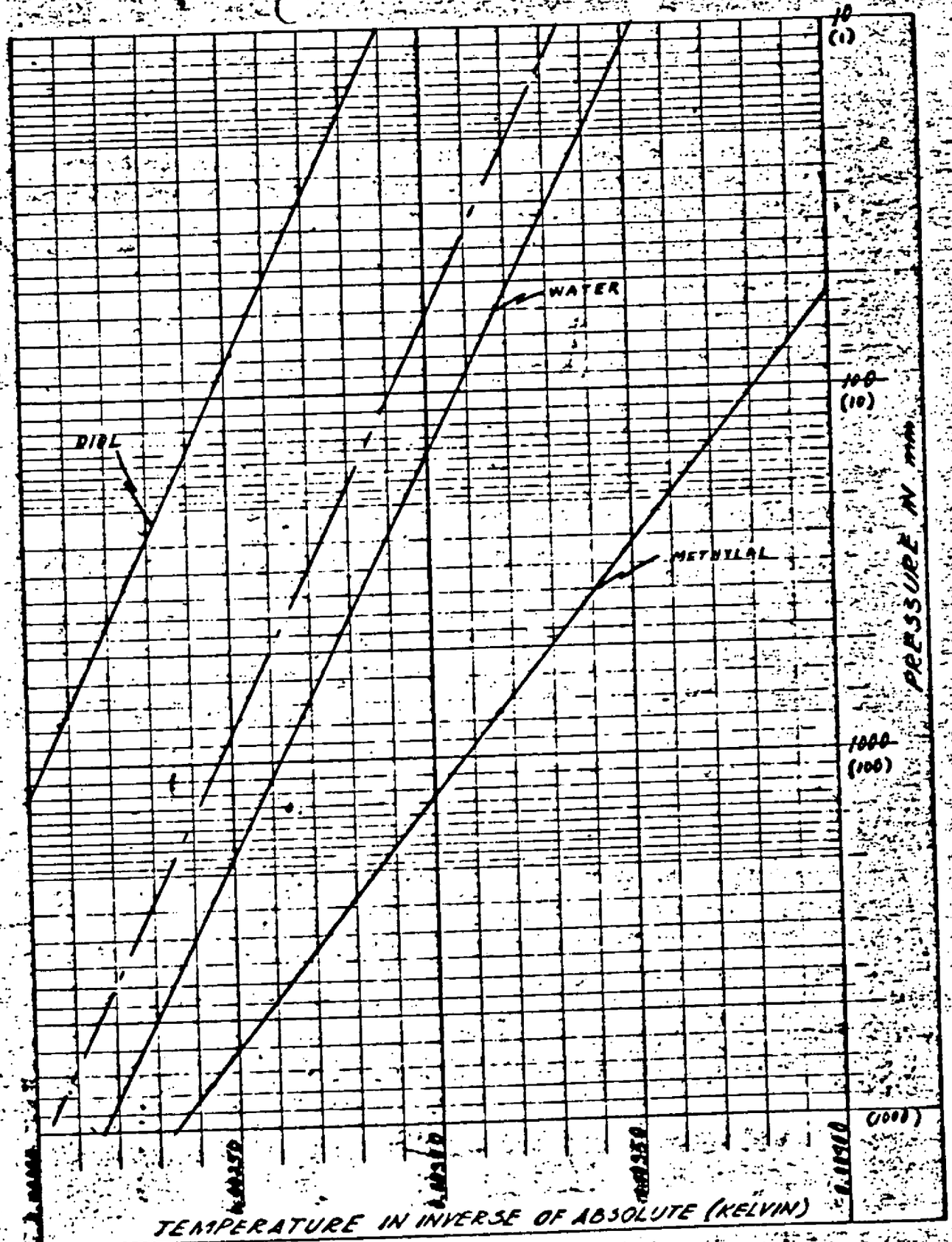
% of ester in total run

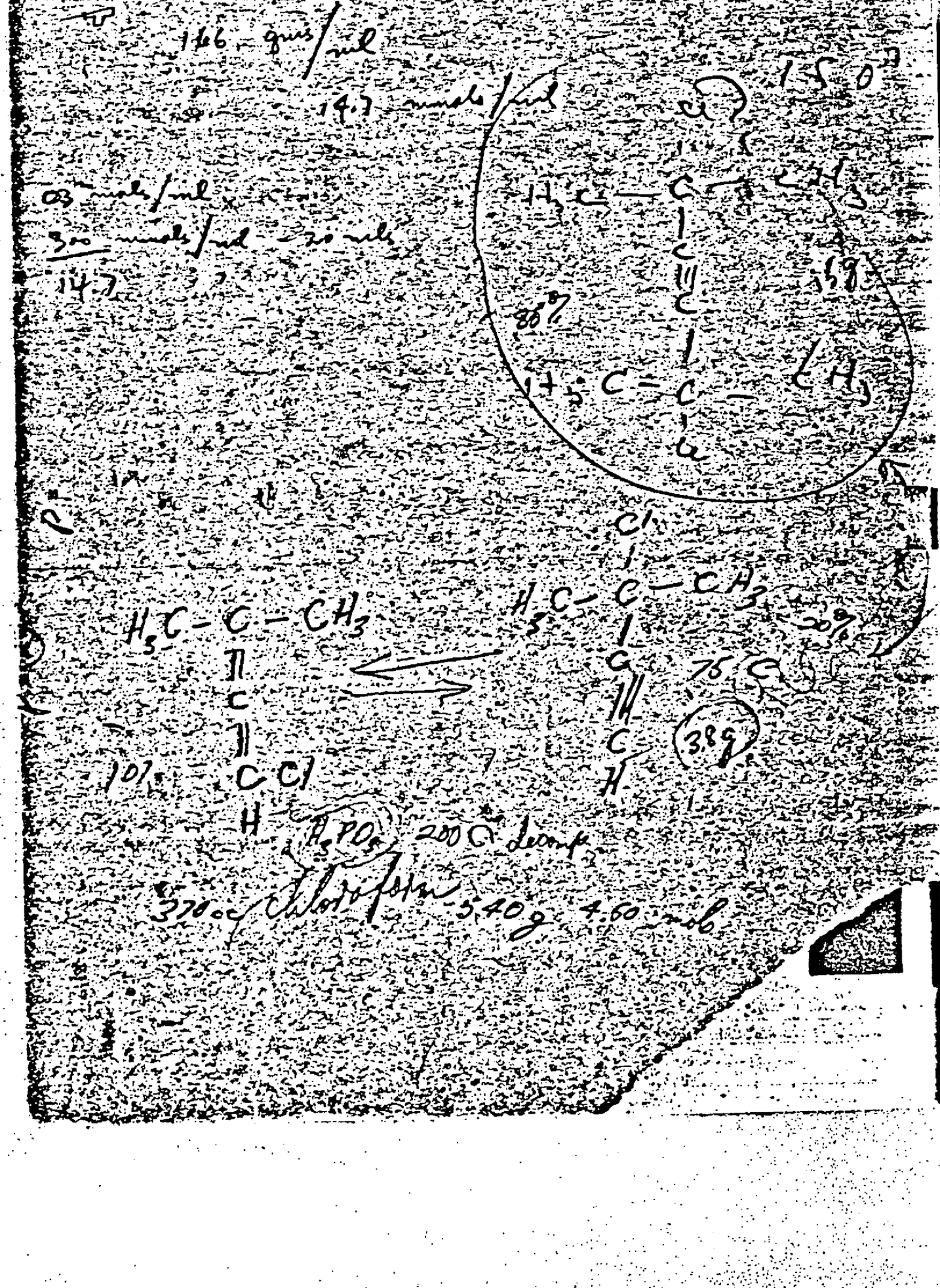
Time of synthesis

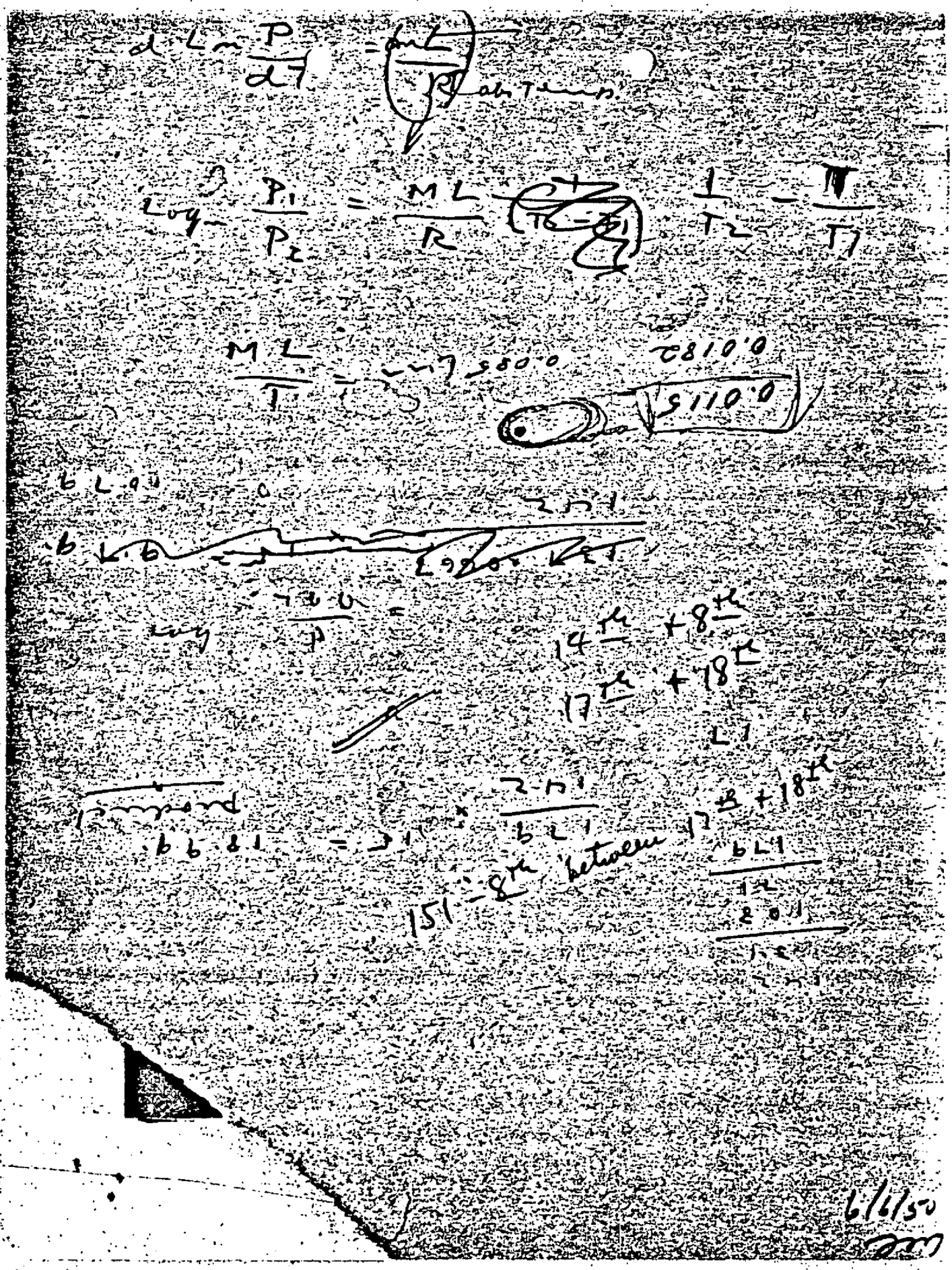
O )

PATENT LITERATURE
SEARCH ON METHYL METHACRYLATE

700
6/615


10
(1)
WATER
100
(10)
DIBL
METHYLAL
PRESSURE IN mm
1000
(100)
(1000)
TEMPERATURE IN INVERSE OF ABSOLUTE (KELVIN)


166 gms/mol
14.7 mmols/ml
30 mmols/ml x 20 ml
14.7
10%
370 g
Chloroform
5.40 g
4.60 mol
200 C decomp
H3PO4
75 C
3.89
20%
86%
1.50



6/6/50

| Time | Gross Weight | Diff |
|---|---|---|
| 5:00 | 727.5 | 4.5 |
| 5:30 | 725.3 | 2.2 |
| 6:00 | 721.7 | 3.6 |
| 6:30 | 719.2 | 2.5 |
| 7:00 | 715.9 | 3.3 |
| 4-11-86 9:30 AM | 688.0 | |
| 10:30 | 687.3 | |
| 11:00 | 687.2 | |
| 12:00 | 687.1 | |
| 1:30 | 686.9 | |
| 3:30 | 686.7 | |
| 4-12-86 9:30 | 686.7 | |

Dry weight of PETN = 269.3 g

Water = 100.0

Drying surface = 475 cm²

Yield = $\frac{269.7}{316} \times 100 = 91.7\%$

MP 136°C

To recrystallize use 2.6 g of Acetone @ 45°.
For each gm of PETN. Add ~~Carbon~~ $(NH_4)_2CO_3$
first to dissolve $CO_2$. Then add 8 g $H_2O$ for
each gm of PETN. Melting point of recrystallized
material = 137.5°C

6/6/15

4-9-46

Fuming nitric acid (6pb 50) 850 g
PE 136 g
4:20 started
Acid cooled to 5°C by ice-water bath.
PE added slowly. Color changed to brownish-
yellow after about 20 g of PE was added.
Temp. rose to 71°C in about 10 minutes.
all PE added in 40 min. temp maintained thruout
at 10-14°C. Temp regulated by the rate
of addition of PE.

Washed with cold water. Used about 2 l.
Then with 1 l of 2% $(NH_4)HCO_3$ solution. Next
washed with about 2 l of hot water (75°C).
Finally washed with cold water.

(4-10-46) Drying ~~in~~ glass dish. Diameter 7"
Material 1" deep. 50°C oven.

| | |
|---|---|
| Gross weight | 785.7 g. |
| Tare | 396.0 |
| | 389.7 |

In oven at 10:30 (4-10-46)

| Time | Gross weight | Diff. | |
|---|---|---|---|
| 10:30 | 785.7 | | |
| 11:00 | 780.3 | 5.4 | |
| 11:30 | 776.3 | 4.0 | 9.4 |
| 12:00 | 771.1 | 5.2 | 9.2 |
| 12:30 | 767.6 | 3.5 | 8.7 |
| 1:00 | 761.0 | 6.6 | 10.1 |
| 1:30 | 758.6 | 2.4 | 9.0 |
| 2:00 | 753.8 | 4.8 | 7.2 |
| 2:30 | 750.0 | 3.8 | 8.6 |
| 3:00 | 745.0 | 5.0 | 8.8 |
| 3:30 | 740.4 | 4.6 | 9.6 |
| 4:00 | 736.3 | 4.1 | 8.7 |
| 4:30 | 732.0 | 4.3 | |

4-16-46

## Preparation of PETN

Same quantities used as before. Temp kept between 3 and 5°C. PE added to $HNO_3$ during a period of 2 hrs. 25 min. Longer period of addition was required in order to keep the temperature below 5°C.

## Drying at 70°C

Wt. of round glass dish 394.2 g

| Time | Gross Wt. | Diff. |
|---|---|---|
| 4-16-46 10:30 am | 955.5 g | |
| 11:00 | 953.0 | |
| 11:30 | 949.5 | 6.0 |
| 12:30 | 940.4 | 9.1 |
| 1:30 pm | 930.8 | 9.6 |
| 2:30 | 920.6 | 10.2 |
| 3:30 | 910.9 | 9.7 |
| 4:30 | 899.5 | 11.4 |
| 5:30 | 891.1 | 8.4 |
| 6:48 | 876.5 | 14.6 |
| 7:30 | 871.4 | 7.1 |
| 4-17-46 10 am | 753.1 | 118.3 |
| 11 | 744.0 | 9.1 |
| 12 | 735.6 | 8.4 |
| 1 pm | 727.5 | 8.1 |
| 2 | 720.0 | 7.5 |
| 3 | 712.9 | 7.1 |
| 4 | 704.7 | 8.2 |
| 5 | 698.8 | 5.9 |
| 6 | 694.3 | 4.5 |
| 7 | 691.4 | 2.9 |
| 4-18-46 1:30 | 684.3 | 7.1 |

Weight of dry material = 684.5 − 394.2 = 289.9 g

Water driven off = 955.5 − 684.5 = 271.4 g

Yield = 289.9 / 316 = 91.7%

MP 136°C

4-22-46

## Preparation of PETN

PE 129.4 g (recrystallized from water)
$HNO_3$ (93%) 810 g

Started adding PE at 11:00 am. Finished 2:18. Acid and PE added in portions, as in previous experiment. Worked as in previous experiment.

MP 136°

$$\text{Yield} = \frac{286.7}{300} \times 100 = 95.4\%$$

---

4-23-46

PE 131 g; $HNO_3$ 850 g

After filtering from $HNO_3$, PETN was put back into fresh $HNO_3$ and agitated for 1½ hr. Filtered, washed as in previous experiment.

MP 137° C

6/6/56

11-16-46

# Preparation of PETN

Total weight of acid 850 g (570 ml). PE 136 g.

Put in nitrator 140 ml of acid and then slowly added 1/6 of total PE. Then 80 ml of acid added and addition of PE continued. This procedure was followed till the end of nitration (from 11:45 to 3:20 pm). Washing started at 4:00 pm. Washed with cold water, cold $NH_4HCO_3$ soln, hot water and finally with cold water. Another part washed with $H_2SO_4$.

Wt of round glass dish = 394.8 g

| | | |
|---|---|---|
| 4-18-46 | | |
| 11:10 | 641.1 | 70.8 g washed with $H_2SO_4$ |
| 4:00 pm | 570.0 | and then washed with $H_2O$, |
| 4-19-46 10 am | 617.1 | $NH_4HCO_3$ etc. |

MP 136°C MP 136°C

$$\text{Yield} = \frac{293.1}{316} \times 100 = 92.8\%$$

5-3-46

# PETN

PE 136 g  850 g $HNO_3$

PE added within 1½ hrs. Acid decanted and new acid added. This was agitated for over ½ hr. Filtered and washed with cold water.

(1) One portion washed with ammon. carbonate and then with cold water.

(2) Another portion washed with ammon. carbonate. Then with hot 1% $HNO_3$, then with cold water.

(1) Gross wt. 664.9
Glass dried 524.6 g  MP 136-136.5
Wt. of PETN 140.3 g

(2) Gross wt. 538.8
Dish 394.2
Wt. of PETN 140.6

Total PETN = 140.3 + 140.6 = 280.9 g

After drying under vacuum for several days the portion washed with dil $HNO_3$ had a strong odor of $HNO_3$.

Yield = 280.9 / 300 = 93.6%

4-24-46

PE = 136 g, $HNO_3$ (93%) - 850 g

Add acid gradually in flask, and PE added during a period of 45 min.

① One portion put back into fresh portion of 93% $HNO_3$ and agitated for 1½ hr. Filtered, washed with about 5 liters of cold water, then with Amm carbonate. Followed by cold water to pH of wash water 8.4. Dried gradually. In the morning, color slightly green.

② Another portion was not washed with $HNO_3$. Otherwise treated same as above. White crystals. Color did not change because this portion was contained much less water than the first portion.

Dried about 1 g of the latter portion at 50°C overnight. In the morning green color

| | | | |
|---|---|---|---|
| M.P. of portion not washed with $HNO_3$ | 5-3-46 | 136.0 | MP 134-135° |
| | | 394.2 | |
| | | 194.4 g | |

M.P. of portion washed with 93% $HNO_3$ 5-3-46 MP 135-136°C

## Recrystallization of PETN from Acetone

300 g PETN dissolved in 800 g Acetone at 50°C. Added about 1 g of $NH_4HCO_3$ and filtered through filter paper.

Precipitated PETN with 1400 ml of cold water. Decanted, washed 3 times with 1000 ml of water each time. After final decantation the material was filtered and washed with cold water.

Gross wt. 768.0
Wt. of dish 394.5 g
Wt. of wet material = 373.5 g

Acetone distilled: 1047.8 g of acetone solution recovered which contained 60.4% acetone according to specific gravity, which was .894

Percent recovery of acetone = $\frac{632}{800}$ = 79%

5-22-46 PETN put in 50°C oven 5-20-46

Gross weight 669.8
Dish 394.5
275.3

5-29-46 Wt. of dry PETN = 668.7 − 394.5 = 274.2

Recovery = $\frac{274.2}{300}$ = 91.4%

# A. BROTHMAN & ASSOCIATES

JOB: Improved

SUBJECT: Idaho Bend

No. of

Date:

By:

| Lbs Initial | Units Initial | M.F. Initial | Lbs Final | Units Final | M.F. Final |
|---|---|---|---|---|---|
| 3790 | 62 | 0.0756 | 8790 | 62 | 0.151 |
| 47,500 | 62 | 0.763 | 43,900 | 236 | 0.576 |
| 2,330 | 132 | 0.161 | 2,160 | 132 | 0.273 |

7-25-46

Me Me Molding Powder

1 { 100 g Monomer
1 g Benzoyl peroxide
33.5 g Carbon tetrachloride

2 { 700 g Sat. Salt solution
70 ml 2% starch solution

BP. in $CCl_4$ dissolved in Monomer and poured in fine stream into the brine-starch solution over a period of 1 hr. Fairly good foaming occurred at the beginning of the experiment. Later it decreased. At the end of 1½ hr the Me Me coagulates into one mass. No evidence of powder formation.

The experiment repeated by pouring (1) into (2) all at once. Same result.

---

Carbon tetrachloride precipitates Polymer from acetone soln.

The following do not precipitate the polymer.

Amyl Acetate
Acetylene Tetrachloride
Trichloroethane
Tetrachloroethylene

7-26-46

100 g Mono.
1 g BP
700 g saturated salt soln.
70 cc 2% starch soln.

Catalyst dissolved in Mono & poured all in the 3 liter starch soln. Refluxed at 80-82°C for one hour before Mono coagulated into a mass.

7-26-46

* Experiment repeated using 70 cc of 5% starch soln, adding the mono to the salt soln dropwise during a period of 30 min. One hour after start of addition temp rose to 89°C at which time there is evidence of powder. Temp rose to 90°C 15 min later. Cooled to 70°C 1½ hr after start of addition of monomer.

The product varied from small perfectly spherical to about 3/8" egg shaped.

7-29-46

* 100 g Monomer
1 g BP
700 cc sat. salt soln.
70 cc of 10% starch solution

Monomer and catalyst dissolved & added to the salt-starch soln over 30 min. About 30% of the material formed spherical pearls, the size of which varied considerably.

8/1/46

100 g Monomer
1 g BP
700 g Sat. salt solution
3.5 g sod. stearate in 70 ml water
} Catalyst dissolved in monomer and poured in salt-stearate solution

| Started | 3:00 | min | Cart |
|---|---|---|---|
| | 3:15 | 78°C | 9x |
| | 3:20 | 81 | 9x |
| | 25 | 83 | 9x |
| | 30 | 84½ | 9x |
| | 40 | 85 | 9x |

Foaming decreased
Stirrer stopped for ½ min.

Some spherical particles but most of the material formed lumps. This suggest that with better agitation it might be possible to obtain a good product

7-30-46

100 g Monomer
1 g BP
} added dropwise

700 g Salt soln.
14 g Areme shampoo

Started adding Mon at 10:48
11:00 very little foam left
05 Mixture viscous; Mn Mc agglomerate

8.1.46

1 1/8-7-46

100 g Monomer (washed with NaOH soln + water)
1 g BP
700 g Saturated salt soln at 83°C
7 g starch in 70 ml sat. salt soln

Monomer ran in dropwise into salt + starch solution from 10:35 to 10:45

| | | |
|---|---|---|
| 10:45 | 84°C | |
| 50 | 86 | |
| 55 | 86½ | emulsified to decrease |
| 11:00 | | |
| 05 | 87 | |
| 07 | 87½ | |
| 08 | 88 | |
| 8½ | 90½ | Cooled to 7°C at 1120 |
| 09 | 91½ | and filtered |
| 10 | 91 | 616.5 |
| 15 | 90 | 800 |

The powder obtained is not perfectly white probably because rock salt was used.

41.8 g of powder obtained. This fairly uniform in size and most of it consist of spherical particles.

30.8 g of the material is imperfect consisting small lumps and shells of pearls. This portion can be reduced greatly by adding the monomer faster (or all at once) or by controlling the temperature around 85°C or lower, say 82-84°C.

1—Methyl-butanolone 100 parts

Chlorine (3 mols) 216 parts

Sodium hydroxide (4 mols) 160 parts

H Cl (1 mol) 36 parts

give (75% yield)

Hydroxy-isobutyric acid 78 parts

Chloroform 90 parts.

2—Vapor phase esterification.

Hydroxy-isobutyric acid 73 parts

Methanol 24 parts (used 100 parts)

give (95% yield)

Methyl hydroxy-isobutyrate 85 parts.

3—Methyl ester 85 parts

Chlorosulfonic acid 85 parts

give (95% yield)

Methyl methacrylate [illegible]

4—Chloroform 90 parts

Acetal 10 parts (used 200 parts)

Potassium hydroxide 6 parts (used 60 parts)

Acetone 54 parts

give

Chloretone 90 parts

(Note: It is possible to recover 6,5 parts acetone and 14 parts chloroform).

5—Methoxy-isobutyric acid (Other alkoxy-acids can be made analogously).

| | |
|---|---|
| Chloroform | 90 parts |
| Methanol | 30 parts (used 13⅓ parts) |
| Sodium hydroxide | 80 parts |
| HCl | 20 parts |
| | give |
| Methoxy-isobutyric acid | 42 parts |

6—Methyl methoxy-isobutyrate.

| | |
|---|---|
| Acid | 42 parts |
| Methanol | 18 parts (used 36 parts) |
| Sulfuric acid conc. | 8 parts |
| | give |
| Ester | 38 parts |

7—Catalytic de-alkoxylation. Ester 38 parts

give (90% yield)

Methyl methacrylate 34 parts

Methanol 8 parts

December 19th, 1946

5-2-47

Synthesis of 2,[ Dimethyl-2,5-Dihydroxy-1-Hexyl-3

62 gms of 90% KOH are slurried in 150 gms of amyl alcohol.

2) 14.3 gms of $CaC_2$ are added and the mixture is placed under a pressure of 75 - 100 lbs (abs.) of $C_2H_2$. The mixture is continually agitated at a temp. of °C. The conversion to $KHC_2$ and $KH_2C_2$ is quantitative and is complete in minutes.

3) The pressure is released and the acetylene is drawn off to storage.

4) The mass is cooled to 13-15°C and 18.3 gms of acetone are added slowly under agitation. The temperature is maintained at 13-15°C.

5) The reaction is continued for 2 hours (after 1 minute the mixture sets to a hard mass) at 13-15°C.

6) 75 cc. of ice are now added under agitation and the temperature of the system is kept at 2°C. Then 75 cc of water are slowly added with mixing.

of Hexitol (2) 5-2-47

1) Two layers form:

a- The lower (water) layer contains substantially all of the KOH plus a sludge of $Ca(OH)_2$ on the bottom.

b- The upper (amyl alcohol) layer contains the 2,5-Dimethyl-2,5-Dihydroxy-Hexane-3 plus some small amount of amyl-hydrolyzed potassium alcoholate of the diol.

a) The lower layer is treated thus:

a- The water soln of KOH is decanted.

b- The $Ca(OH)_2$ sludge is washed twice with 25 cc portions of water to remove the adhering KOH solution. These washes are added to the main batch of KOH soln.

The KOH soln. (approximately 20%) is then dewatered as follows to yield a 90% KOH:

c- To 150 gms. of amyl alcohol at 95°C in a still equipped with an column, condenser, and a chilled decanting device is slowly added the KOH aqueous solution. The water distills at 95°C

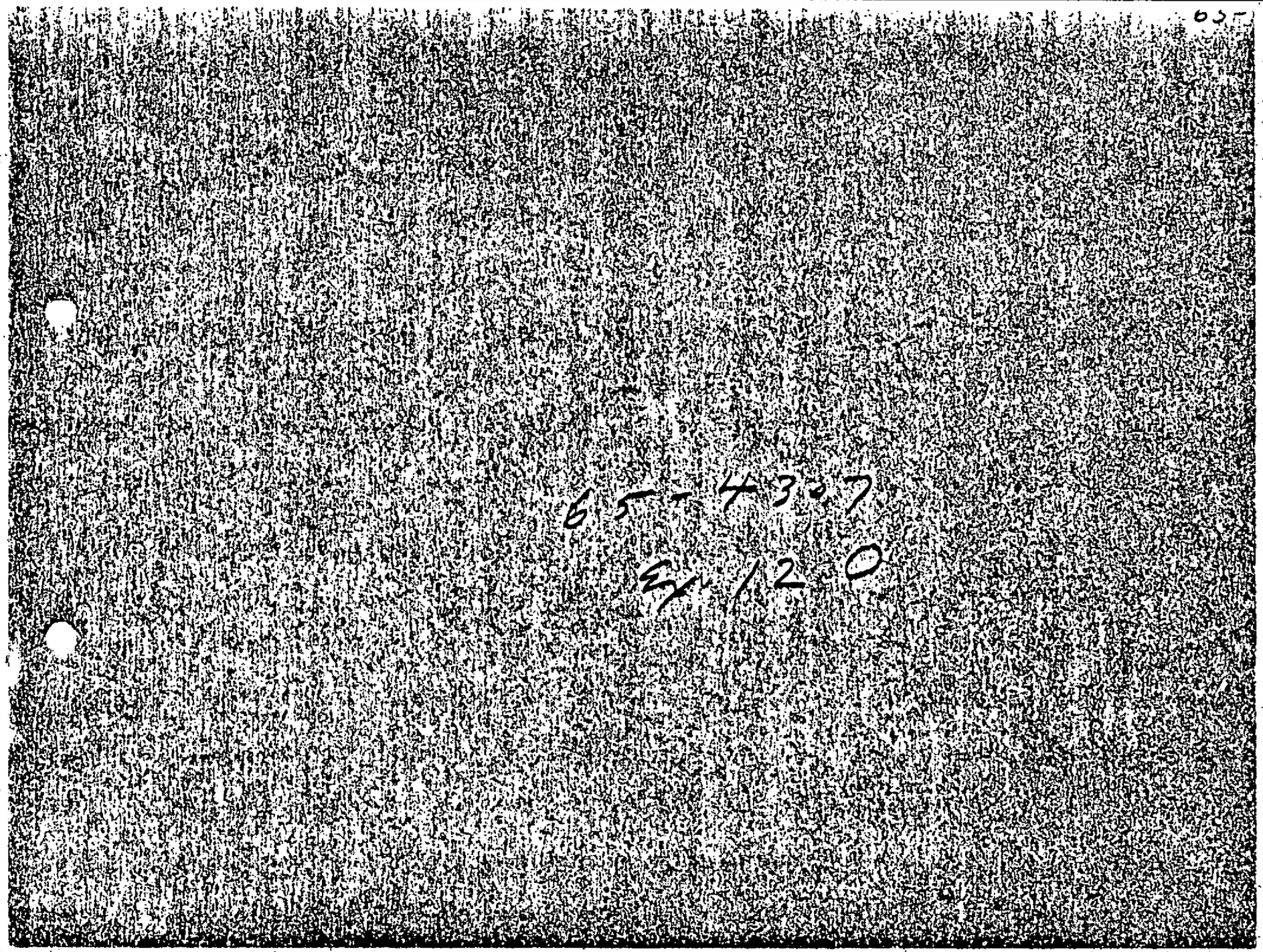
mixture of Neutral
amyl alcohol – the distillate contains
82.8 mole % of water. The distillate is
chilled to 0°C in the decanting device
and the upper layer of amyl alcohol
containing no water is returned to the
still pot. The rates of distillation and
feed of KOH solution are adjusted so
that a constant level of amyl alcohol
is maintained in the still pot. The
distillation is continued until an
amount of water is taken over which corresponds to a 90% KOH in the still
pot. This finely divided material is
identical with the 6.2 gms. of original
KOH at the start of the reaction.

7) The upper layer is distilled in the
following manner to obtain the 2,5-
dimethyl-2,5-dihydroxy-1-hexene-3:
a. A stream of $CO_2$ is passed into the
solution and it is distilled. The first
material coming over is the azeotrope
of amyl alcohol plus the small amount
of water present in the Neutral solution;
this distills at 95°C. The pH is con-
tinually checked to assure that it

below 7.0. The distillate is chilled to recover the amyl alcohol.

b. When all of the water has been taken over the system is put under a vacuum of 100 mm. of Hg and the remaining amyl alcohol is stripped off at a temperature not exceeding °C in the still pot.

c. The yield of 2,5-Dimethyl-2,5-Dihydroxy-Hexine-3 is 22.6 gms. corresponding to 97% of theoretical.


65-4307
EW-120

7/10/50

SAC, Philadelphia

SA T. SCOTT MILLER, JR.

HARRY GOLD, was.
ESPIONAGE - R

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12D)

On 6/25/50, GOLD examined the material contained in the above folder, at which time he stated that the written material was in his handwriting and consisted of notes on work in connection with the synthesis of dimethyl hexine diol, which work he did in May of 1947, according to the notes. The remainder of the material is technical equipment literature. In connection with the folder on HENRICK mixing equipment, GOLD stated that there were two possibilities as to why he had this in his possession: (1) The material was obtained from BROTHMAN's files and was accidentally mixed in with material which GOLD was interested in when he left BROTHMAN's employ. GOLD added that he would have very little use for something of this nature. (2) BROTHMAN had given him this folder for GOLD to submit to the Soviet Union, which GOLD never did. GOLD said the first possibility was the more likely.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12H)

On the same date GOLD examined the material in this folder and gave the following explanations:

The letter dated 2/21/47 to BROTHMAN on the stationery of PENNIE, EDMONDS, MORTON & BARROWS was in connection with patents which related to processes on which the BROTHMAN firm was working. GOLD stated that he recalled BROTHMAN dealt with ARNOLD R. WORKMAN, an attorney who is listed as an associate of the firm.

Relative to the letter from the AMERICAN CYANAMID COMPANY, dated 1/7/47, this was in connection with the manufacture of acetone.

The three pages dated 12/12/46 and entitled "Seminar," GOLD stated, were notes taken at a seminar held in BROTHMAN's office. BROTHMAN started having these seminars to acquaint members of the organization who were not familiar with chemistry, etc., with the general theory in those fields. GOLD said these seminars never got beyond the chemical field as BROTHMAN finally came to the conclusion they were too time consuming.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12I)

On the same date GOLD examined the material in the above folder, at which time he stated that the majority of the handwriting was his own and that the work

TSM:HKF
65-4307

MEMO, SAC 7/10/50

was done in connection with GOLD's employment by BROTHMAN. GOLD identified this material as notes and work on the methyl methacrylate molding powder; the snythesis of B-complex vitamins in connection with a report being prepared for AMTORG TRADING CORPORATION in 1946; and the small 8" x 5" pieces of paper indicate research done on the alternate synthesis of nylon, which was connected with BROTHMAN's office and was not part of the data on nylon obtained by SLACK from HOWARD GOCHENAUR of the DUPONT COMPANY in Belle, West Virginia. Also contained in this folder are schedules of work to be done at the BROTHMAN laboratory. GOLD recognized that some of the handwriting on the material in this folder is that of SOL FANSHEL.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12J)

On the same date GOLD examined the material in the above folder, at which time he said that all this material consisted of laboratory notes, calculations, laboratory reports, etc., in connection with work being done by the BROTHMAN firm in late 1946 and early 1947. GOLD said he recognized his own handwriting as well as that of BILL ROHOLL, SY SILVERSTEIN, and BROTHMAN.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12K)

On the same date, after examining the material in the above folder, GOLD stated that all of the material in this folder, with the exception of the last page, dealt with a scheme for writing a patent on the methyl methacrylate synthesis and an ozonalysis process in connection with the work on methyl methacrylate. GOLD stated that in connection with the ozonalysis notes, he thought that he made them while conducting experiments in the laboratories of the OZONE PROCESSES COMPANY in Philadelphia, at a time when BROTHMAN was considering the purchase of an ozoniser to be used in research being conducted by the BROTHMAN firm at that time. GOLD said he made several trips to Philadelphia for the purpose of conducting these experiments, but that an ozoniser was not purchased.

GOLD stated that the last page of notes, which is on ruled paper, consist of rough draft notes in connection with the building of the plant for the STANTON LABORATORIES.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12L)

On the same date GOLD examined the material in this folder and gave the following observations:

Most of this material, according to GOLD, consists of laboratory notes of BROTHMAN in connection with the methyl methacrylate synthesis, material GOLD was to look

MEMO, SAC: 7/10/50

up in the library, and GOLD's instructions to people in the laboratory relative to experiments. GOLD also stated that there are a list of material to be ordered in connection with the STANTON job, patent numbers, and notes on the diol synthesis.

One piece of paper, which is marked "#1" in red by the writer, has, among other notations, the following: "Rochester and Buffab - Lincoln Terr. - Kelly Mem. - Avenue S and E. 14 - SO 8-2300 - CL 3-4600 - GR 3-8604." GOLD stated that he thought these notations were in the handwriting of SY SILVERSTEIN, who was employed by BROTHMAN, and he thought that these notes identified firms or individuals from whom material and chemicals were ordered by the BROTHMAN firm.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12M)

On the same date GOLD identified the three pages of notes in this folder as being notes he took on 3/10/48 while he was conducting an experiment in the laboratory of the OZONE PROCESSES COMPANY in Philadelphia, on the dimethyl hexine diol process. GOLD said that this was in connection with the experiments to determine if the BROTHMAN firm had use for an ozonizer.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12M)

On the same date GOLD examined the material in the instant folder and stated that with the exception of the group dated 4/9/46, the material consisted of his own calculations and BILL ROHOLL's notes on processes being worked on by the BROTHMAN laboratory.

Relative to the material dated 4/9/46 and marked "#1" in red by the writer, GOLD said that this material was in the handwriting of BILL ROHOLL and concerned the work on a production process of pentaethyl tetranitrate. GOLD said that this was an explosive which BROTHMAN made in his laboratory and which BROTHMAN commenced working on prior to the time that GOLD entered the employ of BROTHMAN. GOLD said BROTHMAN was working on this production of "PETN." (pentaethyl tetranitrate) so that it could be made in a plant in Palestine with the same equipment used in DDT, on which BROTHMAN was also working. GOLD said that BROTHMAN told him that the idea was to build plants in Palestine which produce a peacetime product such as DDT, but which could also be used to immediately produce a wartime product such as "PETN."

GOLD said that BROTHMAN was involved in this work of designing plants which would have a dual purpose, as described above, with an individual by the name of ELIA SHALLIT and a man by the name of SLOVAN. GOLD said he thought that it was for this purpose that the PALESTINIAN POTASH COMPANY was formed.

MEMO, SAC 7/20/50

GOLD said that BROTHMAN had a quarrel with SLOVAN in about August of 1946 and told SLOVAN that he was too busy on other work to continue his work with SLOVAN and SHALLIT.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12O)

On the same date GOLD examined the material in this folder, at which time he stated that this appeared to be notes of ROLFE WOLLAN in connection with the work on the molding of methyl methacrylate, which work, as shown by these notes, was done prior to the time GOLD was employed by the BROTHMAN firm. GOLD said that some of the notes are in BROTHMAN's handwriting.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12P)

On the same date GOLD identified all of the material in the above folder as being his own notes in connection with work on chlorine products for the METTUR CHEMICAL COMPANY, and notes dated 4/3/47 in connection with the work on a new vanishing cream on which the BROTHMAN firm was working for JULIAN BRODIE.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12R)

On the same date GOLD identified all the notes in this folder as being in his handwriting and pertaining to work on solvents. GOLD stated there was a possibility that these might be notes of his when tutoring someone in chemistry at one of his places of employment, either PENNSYLVANIA SUGAR or ABRAHAM BROTHMAN.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12S)

On the same date this folder, which was entitled "Notes from DOC," in GOLD's handprinting, was shown to him, at which time GOLD stated that the material consisted of notes of Dr. GUSTAV REICH, who was GOLD's superior at the PENNSYLVANIA SUGAR COMPANY. GOLD said there were also some notes in the handwriting of himself and MORRELL E. DOUGHERTY on their work at the PENNSYLVANIA SUGAR COMPANY.

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12T)

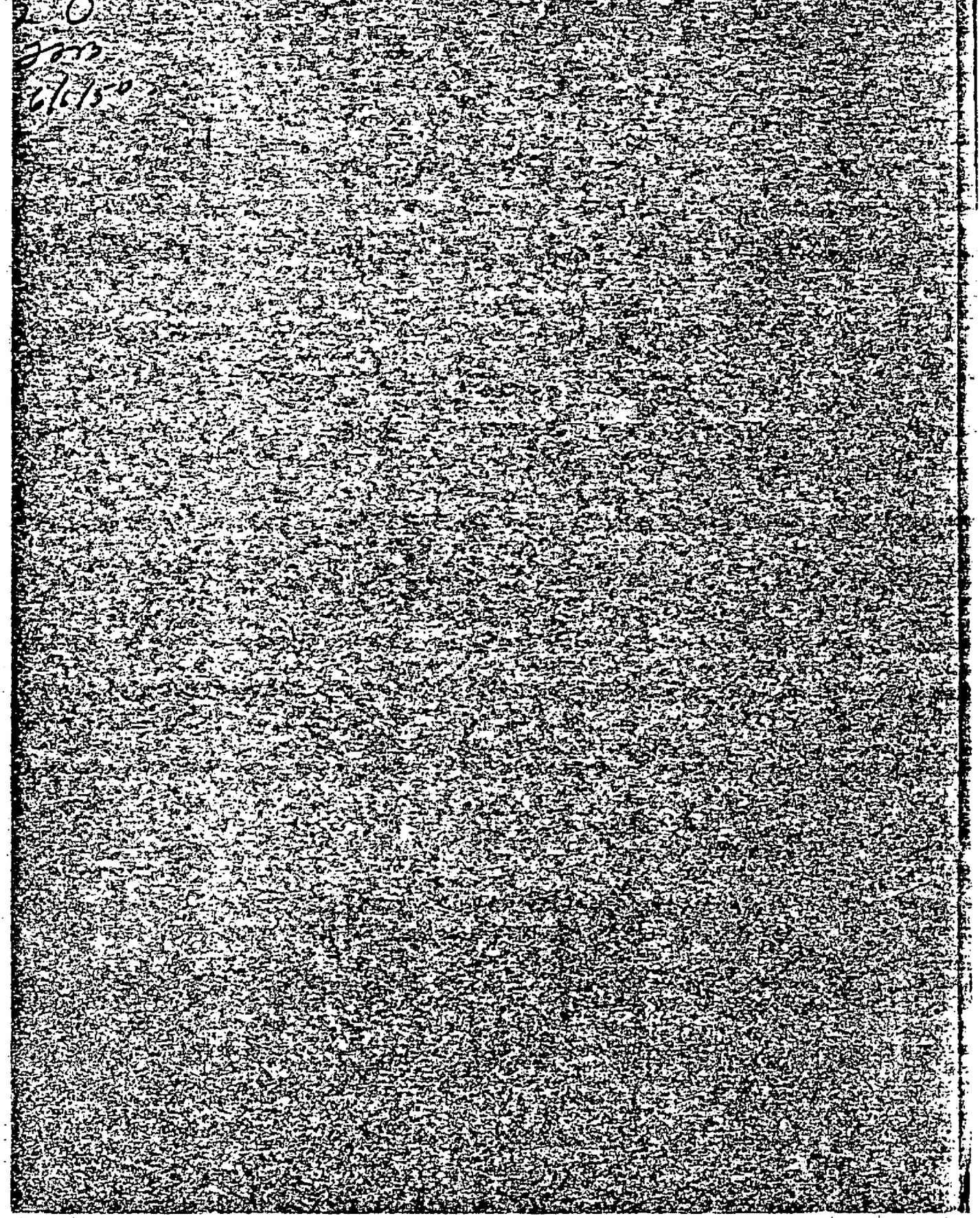
On the same date the above folder, labelled in GOLD's handprinting, "CO 2 Recovery," was shown to GOLD, at which time he stated that all of this material consisted of notes, etc. he had made in connection with work for Dr. REICH at PENNSYLVANIA SUGAR in 1941, on Dr. REICH's CO 2 recovery process. GOLD stated that he also saw some of Dr. REICH's handwriting in the material and, further, that some of the work was done in 1938.

7/10/50

MEMO, SAC

EXHIBIT 65-4307-1B-13 (1) (Exhibit 12U)

On the same date GOLD examined the material in the above folder, at which time he said that the white pieces of paper contain his handwriting and are concerned with his work for Dr. REICH on the CO 2 recovery process. GOLD stated that his handwritten notes on the yellow pieces of paper are his tutoring notes in organic chemistry.



| | | | |
|---|---|---|---|
| mall size | 116 sheets | | |
| 0.29. | 40 mg | 60-65° | Perfect sheet |
| 0.29. | 40 mg | 60-65° | No haze or bubbles but sheet broke |
| 0.29. | 40 mg | 68-73° | slight haze |
| 0.29. | 40 mg | 65-70° | haze |
| 0.29. | 50 mg | 60-65° | " |
| 0.29. | 50 mg | 70-75° | " |
| 0.29. | 50 mg | 60-65° | " |
| 0.29. | 30 mg | 60-65° | Perfect sheet |
| 0.29. | 40 mg | 60-65° | " " (gently slight haze) |

ig Sheet

| | | | |
|---|---|---|---|
| .29. | 40 mg | 60-65 | haze & brittle |
| .29. | 25 mg | 76-80° oven & chemical | " sheet broke |
| .29. | 30 | 60-65 | perfect but it broke |

601 & 116

Sheets

3/60

270 20 mg

5 3 sheets in dying mix - biggest one developed bubbles - all subtyped but spots. But 2 smaller ones came out OK.

Big Sheet

270 20 mg 59-64°

Bubbles along side at end of high oven. Dissolved in low oven. Flaws look white annealing no large sheet noticed by glass blowing. Otherwise perfect.

270 20 mg 60-65° Bubbles

270 20 mg 55-60° (?) One bubble - otherwise perfect.

219/498

1,30

| | | | | | |
|---|---|---|---|---|---|
| 9" | Mold | Ht. of Meniscus | In 80-85° oven | Low temp Oven | |
| 16 | Small | 250 g 25 mg BP | 1 hr 38 min | 55-60 overnight | Perfect |
| 9" | 10 x 10" | 250 g 25 mg BP | 1 hr 56 min | 55-60 Bubbles after one hr | Bubbles |
| 16" | Small | 100 g 40 BP | 1 hr 15 min | 60-65°C 1¼ hr | Perfect |
| 16" | 10 x 10" | 600 g 30 mg BP | 2 hrs. | 48-53°C overnight | Perfect |
| 3/16" | 10 x 10 | 400 g 80 mg BP | 1½ hrs. | 59-64°C 2 hr 55 min Glass broken after 1½ hr | Two bubbles otherwise good |
| 3/16" | 10 x 10 | 200 g 80 mg BP | 1¼ hrs | 60-65°C | haze |
| 3/16" | Small | 100 g 40 mg BP | 1 hr 40 min | 68-73°C | haze |
| 3/16" | Small | 100 g 40 mg BP | 1½ hr | 65-70°C sheet cracked | haze |
| 3/16" | " | 100 g 50 mg BP | 1 hr | 60-65 1 hr | haze |
| 1/16" | " | 100 g 50 mg BP | 1 hr 7 min | 70-74°C 55 min | haze pulling |
| 3/16" | " | 200 g 50 mg BP | | 76-80°C 1 hr 3¼ min | haze |
| 3/16" | Small | 100 g 30 mg BP | 1 hr | 60-65°C 3 1/4 hr | Perfect |
| 3/16" | " | 100 g (40) mg BP | 1 hr | 60-65 5 1/4 hr | slight Haze |
| 1/16" | 10 x 10 | 200 g 60 mg BP | 1 hr | 60-65°C overnight | Perfect crack |
| 1/8" | 10 x 10 | 450 g 90 g BP | 1 hr 12 min | 60-65°C bubbles after about 1½ hr | |
| 18" | 10 x 10 | 450 g 90 mg BP | | 55-60°C | |

# Castings

## 3/16" sheets

### Small size

| Catalyst | | Low temp oven | Results |
|---|---|---|---|
| BPE | BP per 100g Wt Mix | | |
| .2% | 5 mg. | 55-60° | Perfect sheet |
| .2% | 3 mg | 55-60° | " " |
| .3% | 5 mg. | 60-65° | Bubbles & haze |
| .3% | 3 mg | 60-65 | " " |
| .2% | 5 mg | 55-60° | Perfect sheet |
| .2% | 5 mg | 55-60° | " " |

### Big Sheets

| | | | |
|---|---|---|---|
| .2% | 5 mg | 55-60° | Bubbles - look glass |
| .2% | 5 mg | 48-53° | Perfect sheet |

PHYSICAL TEST SPECIMENS

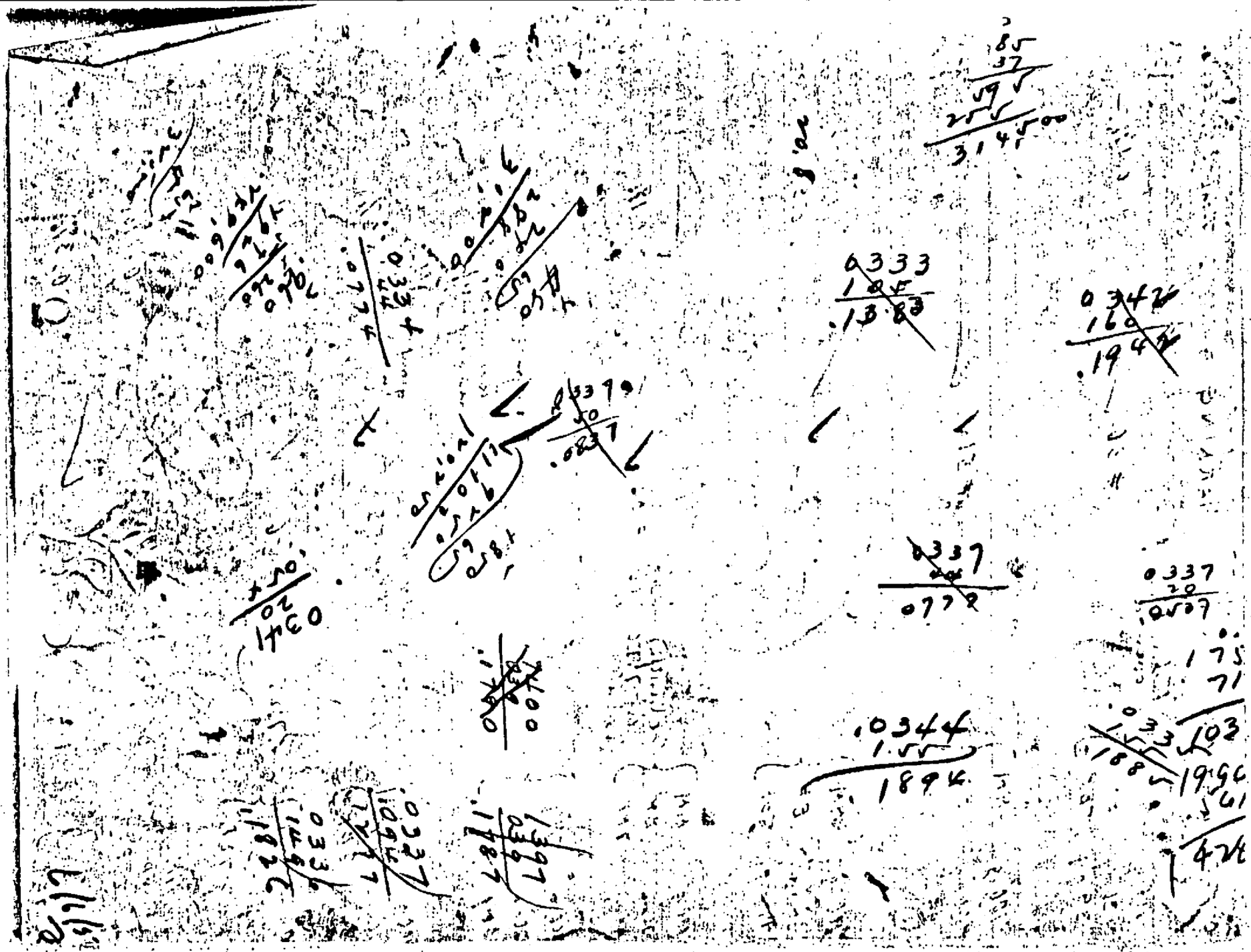
| FORMULATIONS | MARKING | | TOTAL B.P. EQUIV. |
|---|---|---|---|
| 100 g. MM<br>30 mg. BP<br>39 mg. TBP | back in high temp oven at | #50 | 0.1% |
| 100 g. MM<br>30 mg. BP<br>39 mg. TBP | | #51 ✓ | 0.2% |
| 100 g. MM<br>30 mg. BP<br>39 mg. TBP | clamps fell off at 7:30 but spot in center | #53 ✓ | 0.1% |
| 100 g. MM<br>20 mg. BP<br>0 mg. TBP | ditto | #54 ✓ | 0.2% |
| 100 g. MM<br>20 mg. BP<br>0 mg. TBP | clamps fell off at 7:50 | #55 ✓ | 0.3% |
| 0 g. MM<br>0 mg. BP<br>0 mg. TBP | 7:25 | #56 ✓ | 0.1% |
| 0 g. MM<br>0 mg. BP<br>0 mg. TBP | 8:00 | #57 ✓ | 0.2% |

+155
225
270
1987
1670
251
10
2240
+1000
-270
8972
500
39

2 vertical streaks of bubbles at end of high temp

group of bubbles in lower corner shortly after end of high temp

group of bubbles in lower corner shortly after end of high temp



GS-4307

4W12P

J. C. ① ○ 14-3-47

Chem. Form. I, 109

J. C. for Sum & Wind Burn

| | oz |
|---|---|
| Stearic acid triple pressed | 14 |
| apricot Kernel oil | 5 |
| Ethyl amino Benzoate | 0.5 |
| $K_2CO_3$ | 1 oz 175 gr. |
| Borax | 1 |
| Dist. $H_2O$ | 70 |
| Glycerine | 9 |

melt stearic acid & A.K.O. together and add E-A.B. stir until dissolved and strain through cloth. Dissolve $K_2CO_3$ & Borax in distilled water and filter then add glycerine. adjust t° of both the oil-stearic acid mixture and of the Borax-$K_2CO_3$ solution to 75°C, then add slowly while stirring the melted stearic acid and apricot Kernel oil to the aqueous solution. stir until completely emulsified and until temp. has dropped to about 40-45°C. Fill into jars or tubes.

2,5-Dichlorophenol

= 1,4-Dichlorophenol

| Probe | Quan | Value | Unit | Value |
|---|---|---|---|---|
| 220. | 151 | 188 | 1.25 | |

6-6-90

2,4 - Dichloro - Benzaldehyde
2, 4 - Dinitro - Chlorobenzene
1, 2, 4 - Trichlorobenzene
2, 5 - Dichloronitrobenzene

Critical Products

U.S. Tariff Commission
Washington D.C.
Publication
Synthetic Organic Chemicals
U.S. Production & Sales
Calendar 1944

1, 2, 4 Trichlorobenzene

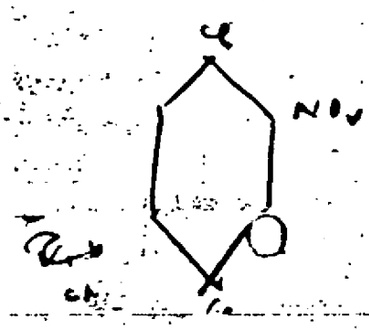
(1, 2, 3 - 1, 2, 4 mixture)

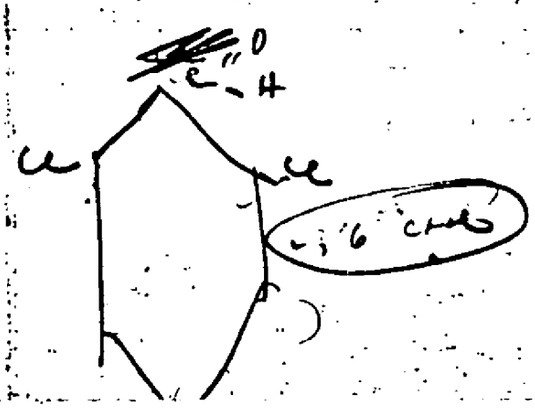
Folder

| Production | Quantity | Value (1000 $) | Unit Value per lb |
|---|---|---|---|
| 2,973 | 2,998 | 218 | 0.07 |

1-chloro-2, 4-dinitrobenzene

| | | | |
|---|---|---|---|
| 11,796 | 6,786 | 466 | 0.11 |



U.C. ⑥ 4-3-47

Chem. Form. I, 114

U.C.

| | lbs. |
|---|---|
| Stearic acid | 50 |
| Lanolin (anhydrous) | 9 |
| Triethanolamine | 2.5 |
| Carbitol | 18 |
| Water | 120 |

Preparation

In one container melt the stearic acid carefully and add the lanolin. Heat the T.E.A. and water separately to boiling and then add the melted fatty acid to it with constant stirring. When a smooth mixture is obtained, stir in the Carbitol to which has been added the perfume. Continue with even stirring while cooling until a heavy smooth cream is obtained, & then stir occasionally until cold. The cream will become thinner as it cools and the acid crystallizes.

Properties

A V.C. should be completely absorbed without leaving a greasy residue. It should not tend to flake or roll

(C.) (6) () 4-3-47

C. F. I, 113

continue stirring until mass is thoroughly cream. | allow to stand overnight. add no. 3. and mix for 20 mins. This cream is softer than the old-fashioned cream but typifies the highest grade modern vanishing cream. The perfection in this cream increases with age and is helped by stirring cold the next day.

a softer cream can be produced by increasing the amount of water.

a harder cream is made by pouring out or by increasing the amount of stearic acid; and also if stirring is very slow.

U. C. ② 4-3-47

( ) ( )

Chem. Form. I, 113

U. C. 9

U. C. 9 made with glycomine (a real forward step in cosmetics) should appeal to product perfect products, noteworthy becau

1. The use of caustic soda, potash and ammonia is eliminated.
2. No glycerin is necessary
3. A most beautiful pearly finish results.
4. Cloud fast will not shrink or dry.
5. It may be poured in jars when cold.
6. The batch is complete in 24 hrs.

Formula

| | | lbs |
|---|---|---|
| 1. | Stearic acid | 20 |
| 2. | Glycomine | 11 |
| | Water | 50 |
| 3. | Perfume | 12 oz |

Heat No. 2 to 200° F and add No. 1 (previously heated to 200° F) to it slowly with

$$\frac{\partial^2 \psi}{\partial x^2} + \frac{\partial^2 \psi}{\partial y^2} = -\omega$$

$$\frac{\partial \psi}{\partial y} = u$$

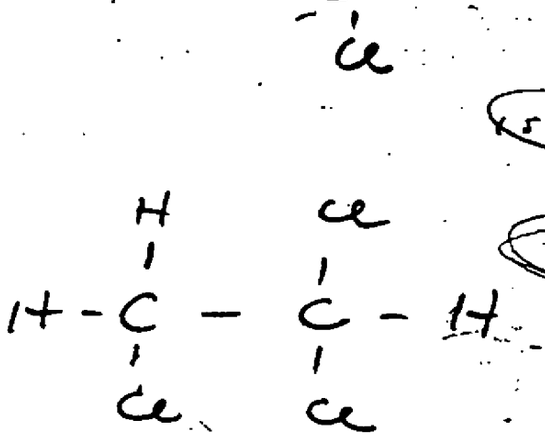
$$-\frac{\partial \psi}{\partial x} = v$$

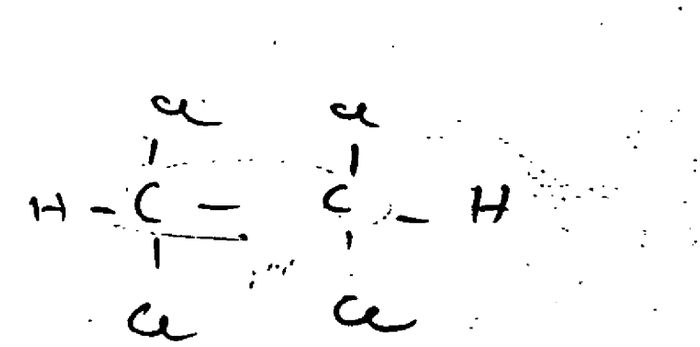
$$\sqrt{6}x^3 + 1 = 2x^2$$

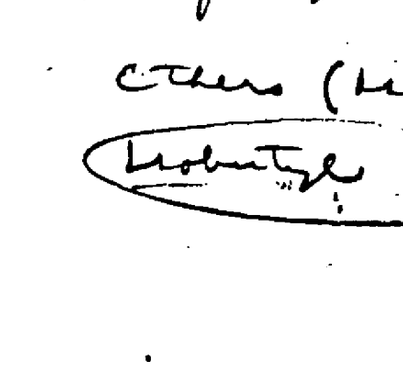
C

Q

Stoddard Solvent







Furfuryl alc

Ethers (Methyl)

Isobutyl alc

I $\frac{d}{dx}(c) = cx^{c-1}$

$y = x^3$

$\frac{dy}{dx} = 3x^2$

---

I(a) $\frac{d}{dx}x = 1$

---

II $\frac{d}{dx}(c) = 0$

$y = b \quad \frac{dy}{dx} = 0$

---

III $\frac{d}{dx}(u+v) = \frac{du}{dx} + \frac{dv}{dx}$

$y = 3x^2 - x^6$

$\frac{dy}{dx} = 6x - 6x^5$

---

IV $\frac{d}{dx}(uv) = u\frac{dv}{dx} + v\frac{du}{dx}$

$x^2 + y^2 = 20$



$y^2 = 20 - x^2$

$y = (20 - x^2)^{\frac{1}{2}}$

$\frac{dy}{dx} = \frac{1}{2}(20 - x^2)^{-\frac{1}{2}}(-2x)$

$\frac{dy}{dx} = \frac{-2x}{2(20-x^2)^{\frac{1}{2}}} = \frac{-x}{(20-x^2)^{\frac{1}{2}}}$

$1 = \frac{-x}{(20-x^2)^{\frac{1}{2}}}$

$\frac{x^2}{20 - x^2} = 0$

$x^2 = 0$

$x = 0$

$$y = (x)\sqrt{(2-3x^2)}$$

$$u = x \qquad v = (2-3x)^{\frac{1}{2}}$$

$$y = (4+x-3x^2)^{\frac{1}{3}}$$

$$\frac{dy}{dx} =$$

$$= \frac{1}{3}(4+x-3x^2)^{-\frac{2}{3}}(0+1-6x)$$

$$= \frac{1-6x}{3(4+x-3x^2)^{\frac{2}{3}}}$$

$x^2 + y^2 = 20$

$2x + 2y\frac{dy}{dx} = 0$

$2y\frac{dy}{dx} = -2x$

$\frac{dy}{dx} = -\frac{x}{y}$

$2x + y = 0$ slope $= -2$

$-\frac{x}{y} = -2$

$y = mx + b$

$y = (-2)x + (0)$

$x = x$
$y = y$

$-\frac{x}{y} = -2$

$x = 2y$

$x^2 + y^2 = 20$

$4y^2 + y^2 = 20$

$5y^2 = 20$

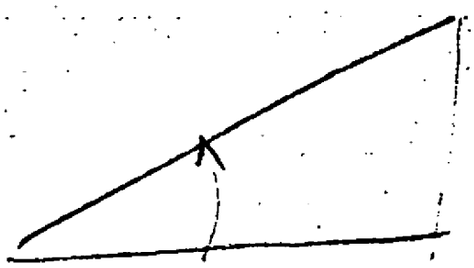
$y^2 = 4$

$y = \pm 2$

$y = x^3 - 2x^2 - x + 2$

$\frac{dy}{dx} = 3x^2 - 4x - 1 = \tan T$

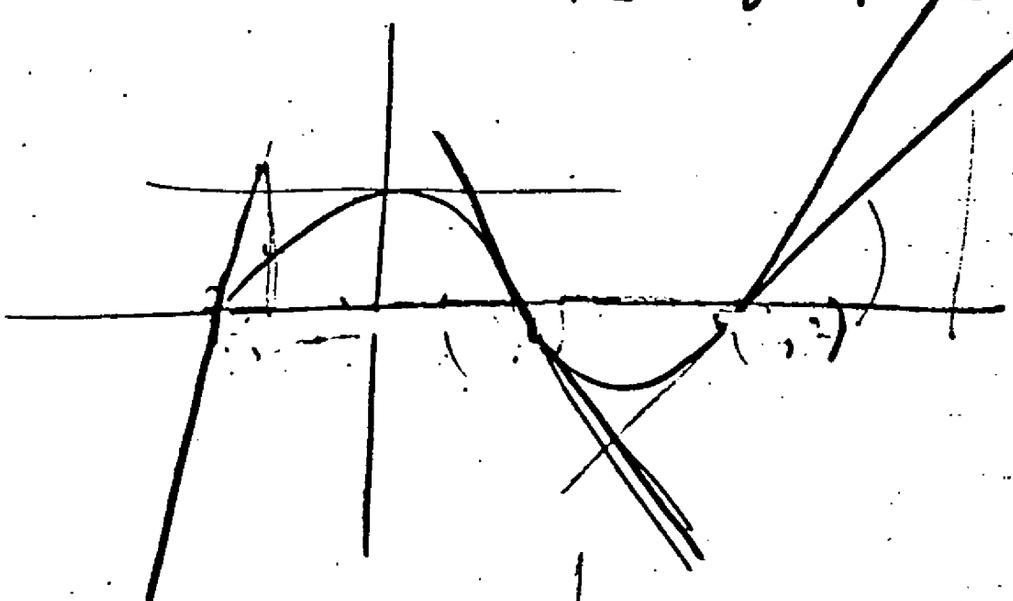
$(x-1)(x+\frac{1}{3})$ = slope

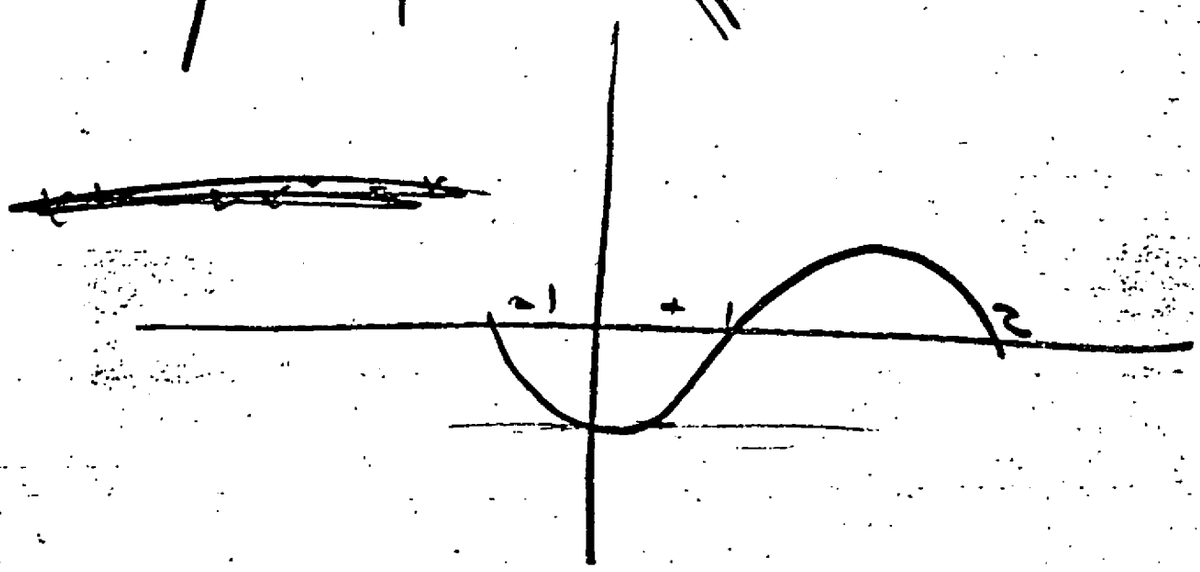


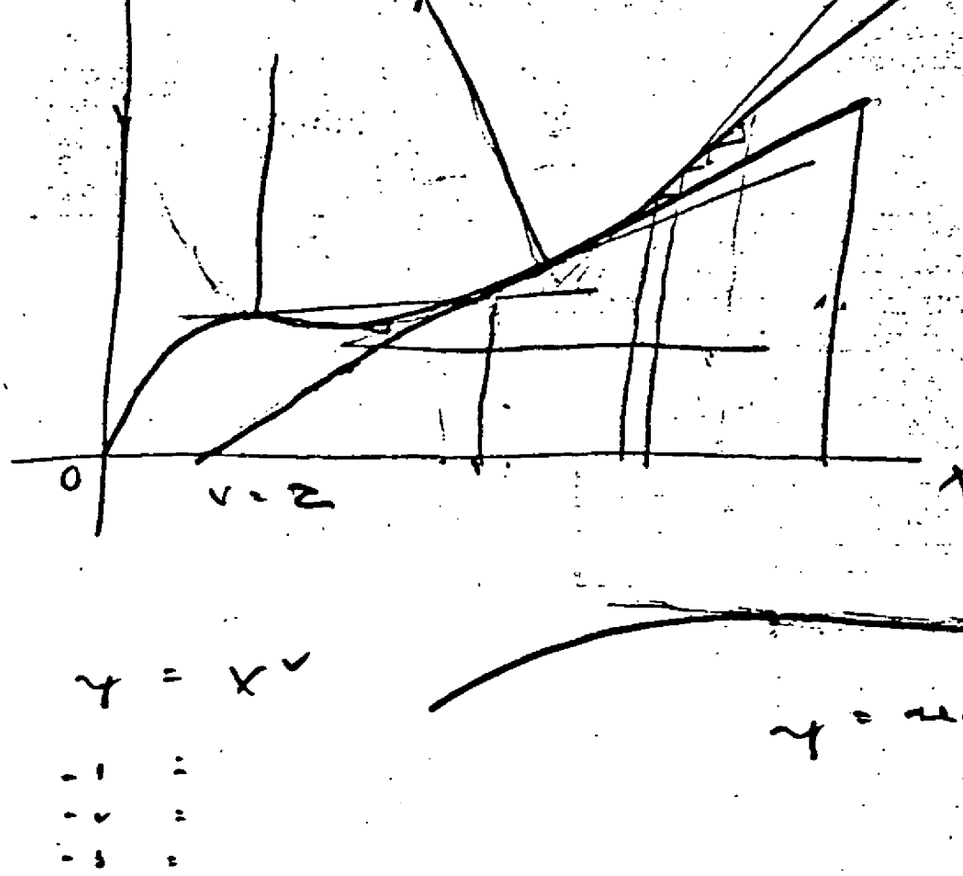
$3 - 4 - 1 = -2$

$3 + 4 - 1 = 6$

$12 - 8 - 1 = 3$

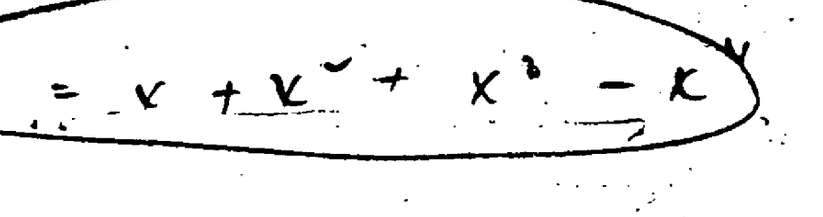







$y = x^n$

$n$ : $-1, -2, -3, \ldots, 1, 2, 3$




$y = x + x^2 + x^3 - x$

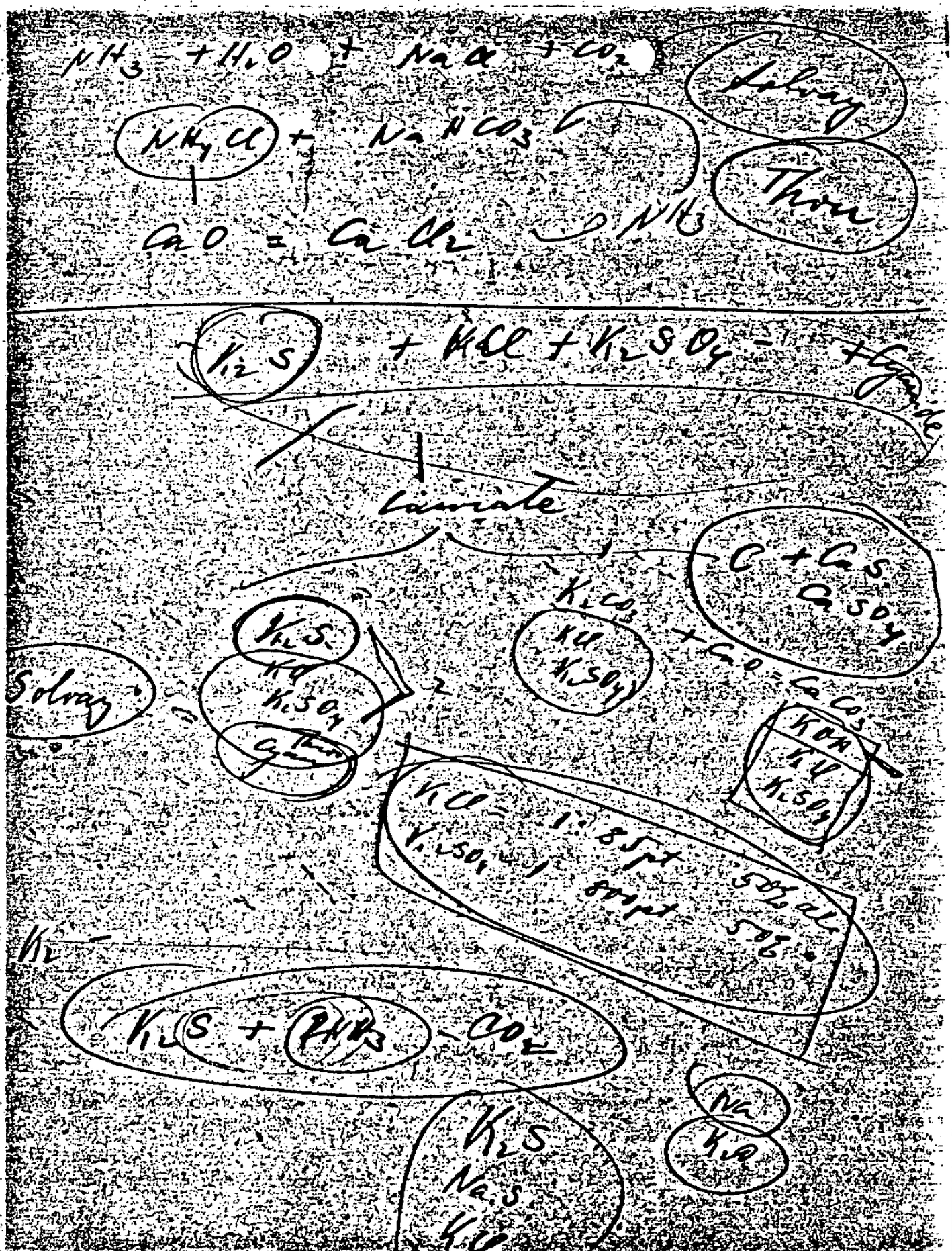
$$\frac{dy}{dx} = 1 + 2x + 3x^2 - 4x^3$$

$$= 1 + 4 + 12 - 32$$

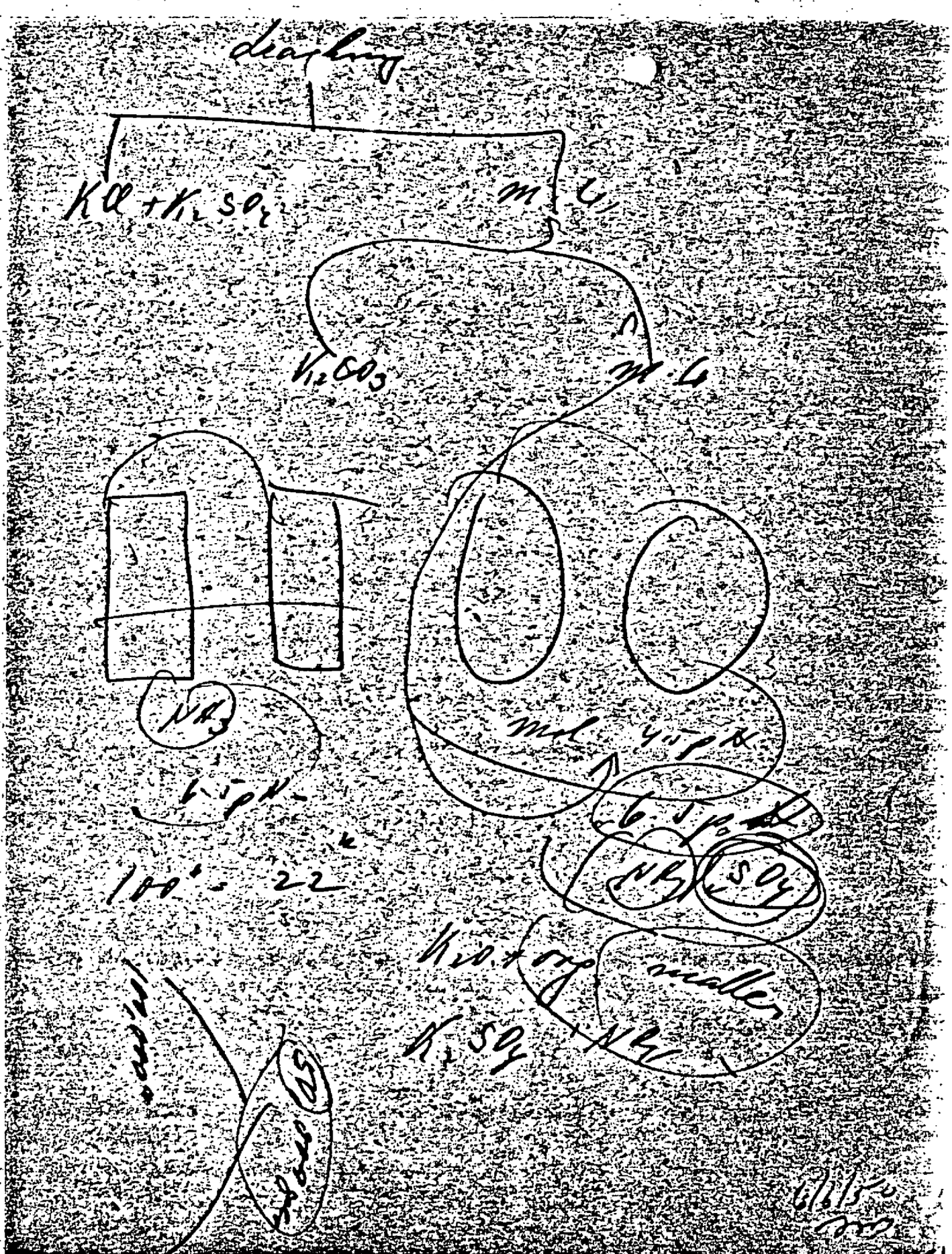
$$17 = -15$$

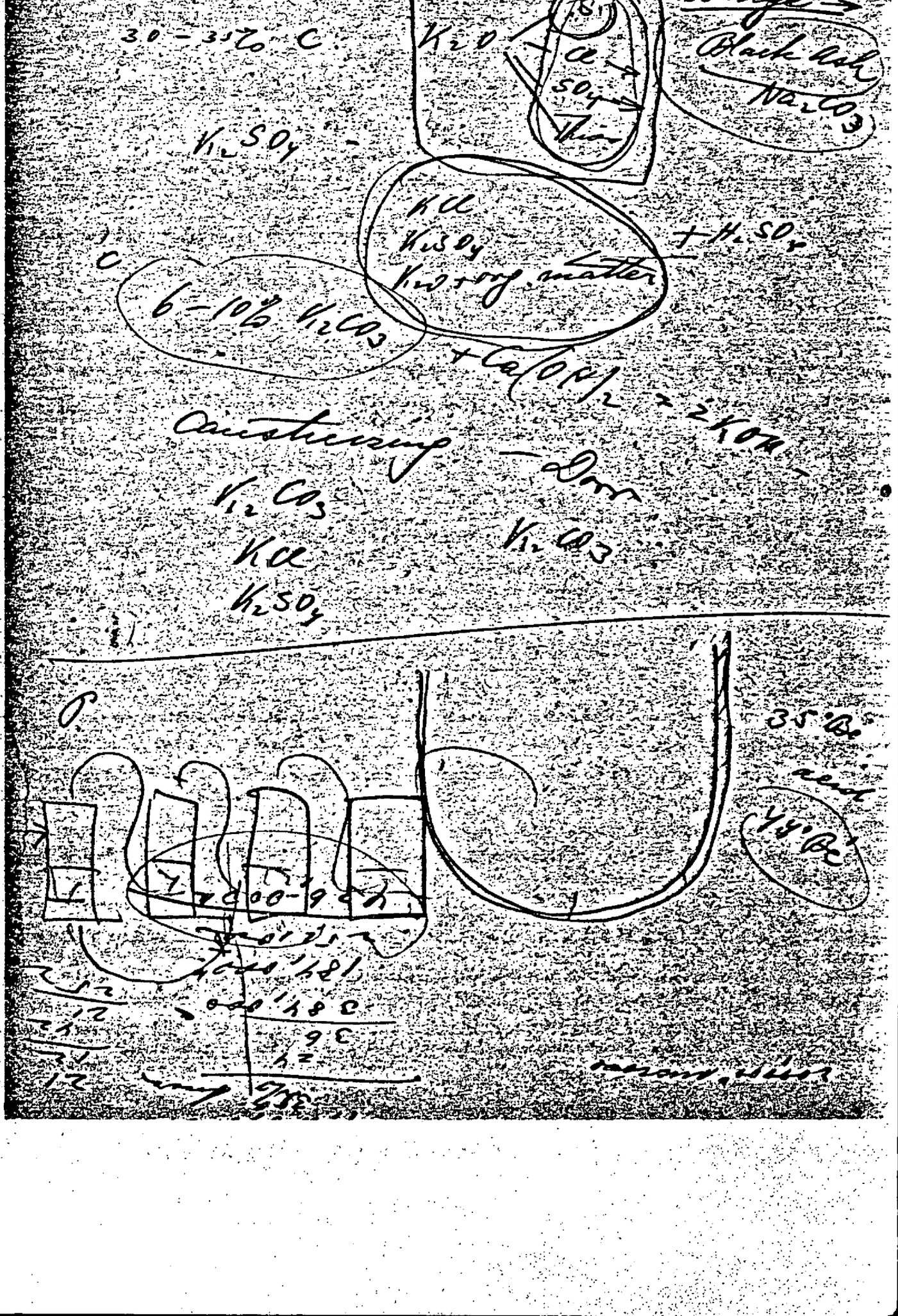
$$y = \frac{1}{2}\sin 2x$$

Notes from Doc

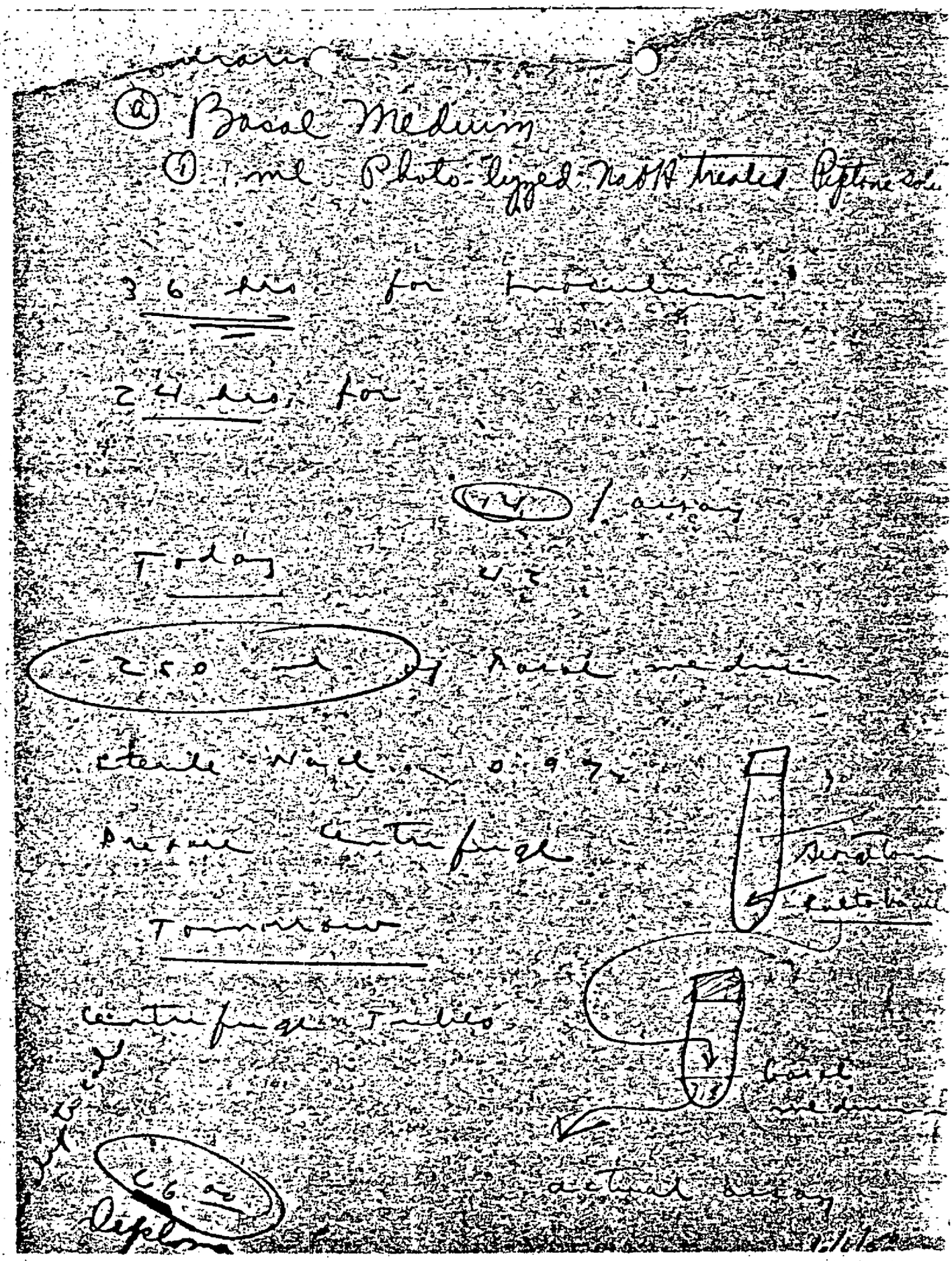

$NH_3 + H_2O + NaCl + CO_2$
$NH_4Cl + NaHCO_3$
$CaO = CaCl_2$
$NH_3$
$K_2S + KCl + K_2SO_4$
Limiate
$K_2CO_3$
$CaO = CaCO_3$
$K_2S$
$K_2SO_4$
$K_2S + PbS$
$CO_2$
$K_2S$
$Na_2S$
$K_2O$
Na
$H_2O$

CaO = 1.78
Yeast 1.25 - 6% acid
Ca SO4
Evap
Ca SO4
Ca SO4
Ca
Mg CO3
Ca O

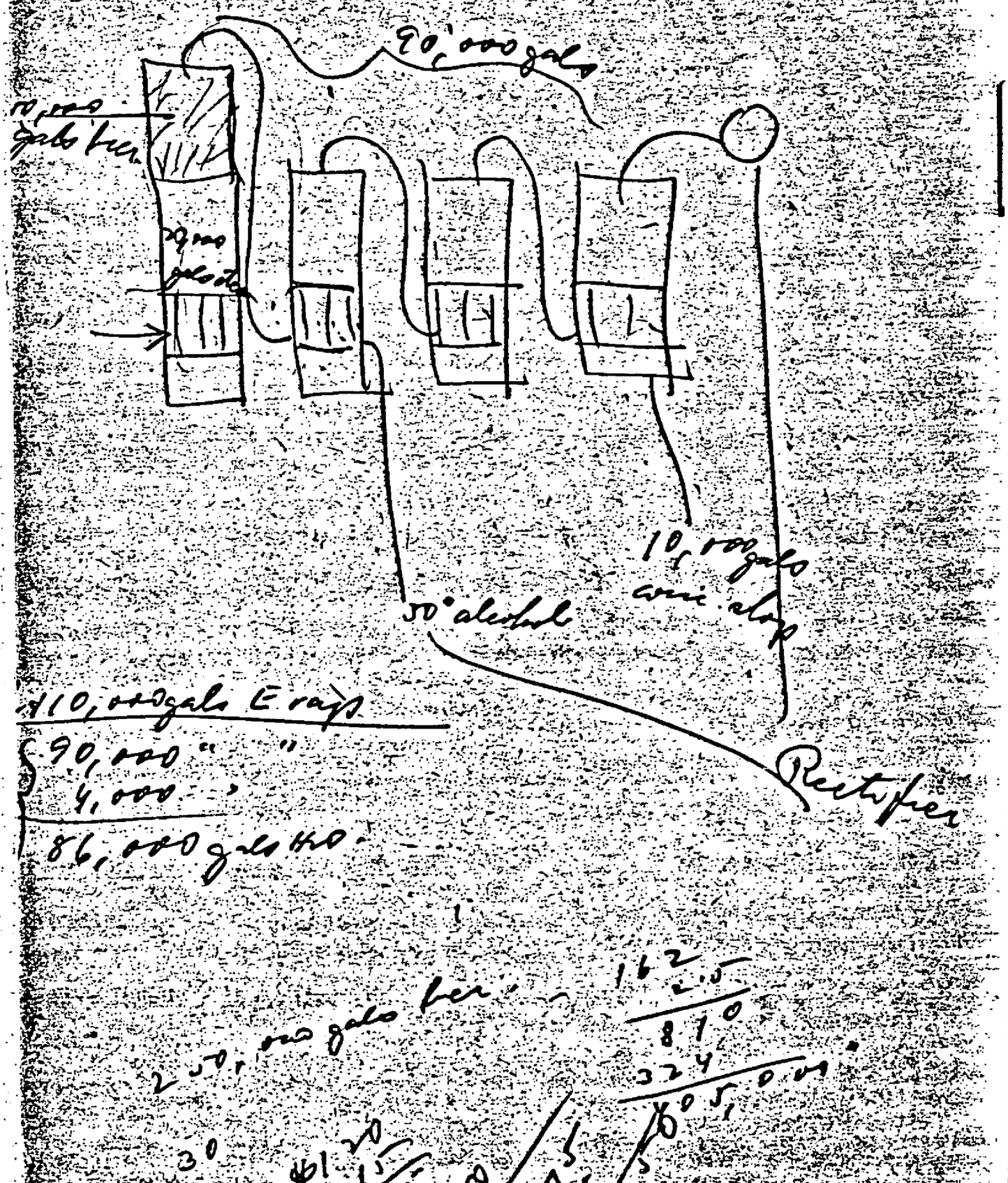



30 – 35 °C
$K_2O$
Si
Cl
$SO_4$
Mn
Dunge →
Black ash
$Na_2CO_3$
$K_2SO_4$
KCl
$K_2SO_4$
Impurity, water
+ $H_2SO_4$
1 C
6 – 10% $H_2CO_3$
+ $Ca(OH)_2$ + 2 $H_2O$ →
Centrifuge
$K_2CO_3$
KCl
$K_2SO_4$
– 0 °C
$K_2CO_3$
C
35 % $K_2CO_3$
rest
$K_2CO_3$

100,000
gals beer
9% alc
Beer Still
2# steam
140° alc
7000 gals alc.
3000 # H2O
10,000 gals
Rectifier
Rectifier
22# steam per 1gal 190°
7310
1470
1470
16,170 # steam from beer still
20,000 gals H2O
100,000 gals beer
+20,000 gals steam
110,000 gals
alcy.
E
Evaporator
100,000 gals
10,000
gals conc alcy
gals
Tubular
190°
100,000 gals beer
110,000 # dil. alcy
210,000 gals
7310 gals 190° alc.
10,000 gals conc alcy


a Basal Medium
1 ml Phyto-lyzed NaOH treated Peptone sol.
36 hrs for
24 hrs for
Today
250
Sterile water 0.9%
Sterile
Tomorrow
Sterile
Sterile
actual size
Light

17,000 gals
28½ N.S
168,000
46,000
214,800
3121,000
15
15-60
312
4,680 or
312,000
200,000
212,000
1:140
60,000
100,000 gals
200,000
70,000
alcohol
N.S
2:1
1:1
Liquefy N.S by removing the sugar
1,000 gals ale
360,000 gals
20 days
Mol + H2O
Ferment
Beer still
Centrifuge
Mol + H2O + H2SO4
Ferment
Evaporate
Mol
Saturate
Distillate
Mash
Grain
Evap


90,000 gals
10,000 gals beer
10,000 gals
50° alcohol
10,000 gals
corn slop
Rectifier
40,000 gals Erays
90,000 " "
4,000 "
86,000 gals 50°
2,500,000 gals beer
30
61.20
15
35.50
65
60
35
162
2.5
810
324
50,000

Citation of Vitamin of yeast
— Two others
J Biol Chem 1919, 40, p 383-4

Nearly pure growth promoting
fraction from dilute acetic acid
extract of yeast by fractional
precipitation with alcohol, no
definite substances were isolated.

$$\frac{63 \cdot 43^{\circ}}{W} \quad \frac{1}{1^{2}}$$

6/6/50

# $CO_2$ Recovery

Evaluation of the new unit for absorbing $CO_2$

Of the seven runs made, three were complete in that all the necessary data was obtained.

Summary:

The data obtained compared very favorably with that for the rectangular absorber and merits further investigation. Especially, is this true when it is considered that the mechanical difficulties encountered prevented the mixers from being given a complete and fair trial.

Sample Calculations:

1. Material Balance

a. CO2 by titration

Basis: 100 gms. H20

Total as Na2 CO3,

$$\text{cc} \quad \text{m.e.} \quad \text{ml. wt.} \quad \frac{100}{10} \text{ grms H20}$$

$$46.8 \times 0.053 \times \frac{100}{10} = 24.8 \text{ gms.}$$

Alkali as Na2 CO3 in Solution:

$$43.0 \times 0.053 \times \frac{100}{10} = \frac{22.8 \text{ gms.}}{2.0 \text{ gms.}} \quad \begin{matrix} \text{Na2 CO3} \\ \text{pptd. as NaHCO3} \end{matrix}$$

also in the solution

$$\text{cc} \quad \text{m.e} \quad 43.0$$

$$2 \times 16.4 = \underline{32.8}$$

$$\frac{10.2}{43.0} = 23.7\% \text{ NaHCO3}$$

and

22.8 x 0.237 = 5.4 gms. Na2 CO3 converted to Na HCO3

With the 2.0 gms. pptd. as Na HCO3, this gives a total of 7.4 gms. Na2 CO3 as NaHCO3 and,

7.4 x 0.415 = 3.07 gms. CO2

Volume of Solution

$$\frac{8.25^2}{144} \times 0.785 \text{ ft.}^2 \times 5.25 \text{ ft.}^3 \times 7.48 = 1.18 \text{ gals.}$$

$$= 4500 \text{ ml. (s}$$

$$= 4200 \text{ gms H2}$$

further

2. Results. - The figures are given together with those for Report #2 and for the rectangular absorber.

| Factor | Turbo-Mixer | | Rectangular Absorber |
|---|---|---|---|
| | Report #2 | Report #3 | |
| Average % of CO2 in exit gas | 4.4 | 2.1 | 3.0 |
| Apparent absorptive efficiency, % | 68 | 85 | 81 |
| Lbs. CO2 absorbed per gal. solution | 0.248 | 0.359 | 0.139 |
| Lbs. CO2 absorbed per gal. absorber sp. | 0.130 | 0.188 | 0.124 |

These results show a considerable improvement over those of the previous runs and in comparison with the rectangular absorber. The following conclusions may be drawn

a. The absorption is excellent; an average exit gas of 2.1% CO2 shows that the agitation is the principal factor in determining the efficiency of the absorption.

b. The CO2 absorbed per gallon of solution is 2 1/2 times as great as that for the rectangular unit. However,

c. Per gal. of absorber volume this figure is only 50% greater for the mixers.

d. The gas analyses show that each mixer takes out almost exactly the same amount of CO2 from the flue gas - 3.9% of CO2 is removed by each of the three.

Summary:

The absorption efficiency of 85% is better than any previously recorded;- the exit gas averaged 2.1% CO2. It was also determined that each of the mixers removes an equal amount of CO2 from the flue gas.

The causes of the mechanical difficulties were definitely located and can now be eliminated.

Witnesses:

Harry Gold

Outline of Rept.

Runs 1 & 2 - choke off to Exp. - no good tests. no data.

Run # 3 - good test - bubbles at breaker cups - poor absorption.
Poss. experimental error.

Runs # 4 & 5
615 r.p.m. vs. 1550 r.p.m. Chip Turbo data:
not ready yet

Run # 6 repeat each run of # 5 - slightly higher gas rate at end
(higher conc. alkali)

Run # 7 High gas rate (20% increase over #) → 8.0-9.0 Cir.
Average 5.4 Cir.

Tomorrow, Sun

1. Calculations { Cir & Circ Corr
Abs Corr, etc.
2. Tables
3. Report