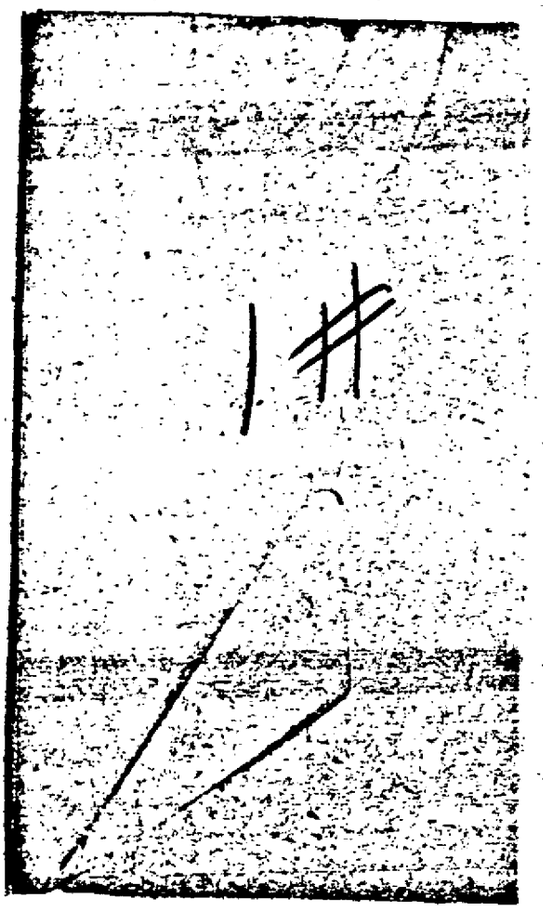


Neutral Red Prep

6/6/50
JRD

Material Ordered

1.00 gm KOH

A—0.1N added
350 cc
65 cc
415 cc
90 cc
325 cc A—0.1N + 7.6 gm added KOH
(= 265 gm)
= 341 g. Total

A—0.1N Distilled
114 cc Total (A—0.1N + 1.0N)
24 cc 1.0N
90 cc A—0.1N

341
71
270

So) 270/341 × .76 = 6.0 gm added KOH

Methyl added
350
70
420 cc × 0.95 = 360 gm Methyl
270 gm A—1N-KOH
630 gm Total added

360
165
195 gm Methyl
= 229 cc

653
1283
1118
165 Methyl Red

Need 60/50 × 220 = 236 cc

65

H_2O

$HOH \rightleftharpoons H^+ + OH^-$

$\frac{22}{65} = 33.8\%$

$300\,(0.869) = 265\,g\ KOH$

$\frac{265}{56}\ 18 = 85\,g$ water

$\frac{85}{15}$

$\frac{1}{88.3} = \frac{22.8}{17.7}\%$ reacted.

$\frac{43}{50}$ (19)

(0.50#) (56) $\frac{230}{50}$

$\frac{54.2}{1000}$

49.65
20.95
29.70
21.24
49.94
4.3
54.2

49.17
27.93
21.24

56 65
19
46

47 19

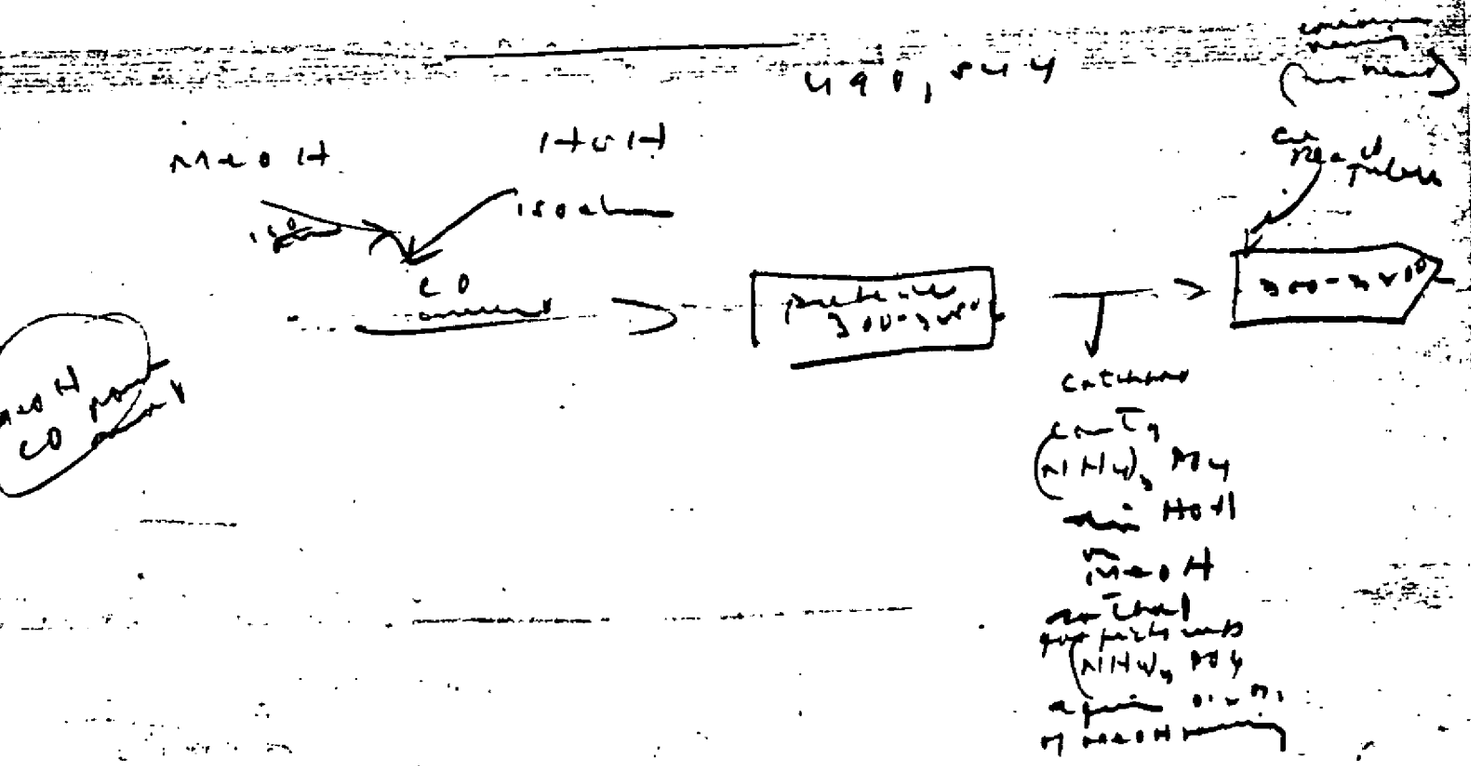
64
17
53

262,364

Dec 13, 1976

$3CO + 3Hv \rightarrow CO_2 + CH_3CO$

490,944



cool & Lower → Acetic

Pump Water Now → Full acid

200 am. KOH
480 am. AmOH
840 am. methanol
1520 am. Total

15.470
30.870
45.870

6/6/30 yld

56 100

15.4

56

1541 370

7700

8.11

70

7.41 730

370

3659

1541

490

1541

188

981

730

1711

1979

KOH 20 39 59

19.59 KOH

39

420g

420 9

10

56 x 2

18

18 171 = 5.89 KOH

+8.8

0

1153.7
1150.6
2.1

6.0
2.1
8.1

6/6/50
JM

Am. OH $156(0.814) = 122$ g

$\frac{122}{88}(56) = 77.5$ g. dry KOH

$\frac{7.50}{5.0} \times \quad \times 0.504 \times 0.0256 =$

6/6/50

Ex I

Ex V

Harry:

6/6/50 pm

1) The KOH (pelleted) is 86.3% KOH.

2) Abe would like a call before any further runs are made.

3) I may be somewhat late Thursday as I owe a visit to the County Clerk.

Bob

6/6/50

Memorandum Report ---- -G -90

Preliminary Operating Manual -Manfacture of Adipic Acid

Introdmntion:

This manuel covers the operating details for the manfacture of pure adipic acid from Hexalin. The procedure is covered in five sections as follows:

1. Vapor phase OXIDATION of Hexalin.

22. Refining of cyclo hexanone from vapor phase Oxida--tion.

3. Liquid phase Oxidation of Cyclohexanone to Adipic Acid.

44. Purification of Adipic acid--from liquid phase Oxidation.

5. Recovery of Aceticacid and Adipic acid and from purge liquor.

6/6/50

Summary of Process Materials

Materials	Per. Day	Quanities per month	PerYr.
Adipic acid	775 #	21,000 #	250,000#
Acetic acid	145 #	4,000 #	48 ,000 #
Cyclohexanone	91 gals.	2,500 gals.	30,000 gals.
Hexalin	116 gals.	3, 200 gals.	38,000 "
Manganese Acetate	1.7#	47 #	560 #
Darco	17 #	470#	5,600 #

Acetic acid make -up will be required at this rate, once the system is completely filled. Approximately 5,000 lbs. of Acetic acid will be required when first starting the process.

At least four feed mixes will be made before any recovered Acetic acid is available.

6/6/50

Part 1. Vapor Phase Oxidation of Hexalin to Cyclohexanone.

Discussion of Safety Precautions:

1. Hexalin and Cyclohexanone are inflammable materials. Explosion proof motors and lamps are used, There should not be smoking or open flames in this area. Also avoid bringing these materials in contact with the skin.

Outline of Process:

Vapor phase oxidation is accomplished by passing Hexalin vapor and air over silver gauze Catalyst, which operates at about 550 C. Although the reaction is slightly exothermic the converter is not self sustaining. External heat must be applied. The off vapors are condensed and decanted into a water saturated Cyclohexanone layer and a Cyclohexanone saturated water layer.The off gases are cooled and vented toatmosphere

A flow diagram of the process is attached (Figure 1).

Detailed Operating Instructions :

(A) Starting the Process

1. Since the freezing poi nt of Hexalin is approx-imately 25 C. the hexalin storage and all lines through which Hexalin passes must be steam heated and lagged. The temperature of Hexalin feed tank No. 3-A must be held between 30 and 35 C. Turn steam on feed tank No. 3-A and all steam traced lines.

2. Fill Hexalin feed No.3-A with refined Hexalin lines from the tank farm.

3. Turn water on converter NO. 4-A.

4. Check decanter No.5 and see that valves are set to put oil layer to No.7 storage and water layer to No. 6 storage

5. Turn full steam on vaporizer No. 3-B.

cont. from page 3

6/6/50 JB

6. Start air compressor.

7. Start Hexalin feed pump No. 3-D.

8. Turn heat on converter No. 3 -C and heat to 500 C.

9. Start flow of Hexalin of 3 to 4 G.P.H. to vaporizer.

10. Start very small flow of air to vaporizer .(Not more then 2 to 3 cu. ft. per. min.)

11 . As reaction "lights Off" gradually increase air flow to hold converter between 500 and 550 C.

12. Gradually raise Hexalin feed to 5 or 6 G.P. H.and increase air flow to correspond.

13. Check condenser No.4-A to be sure it is operating.

14. If at any time the Hexalin flow is stopped, turn off all flow immediately.

Shutting Down the Process.

1. Stop air flow to converter.

2. S tpp Hexalin flow to vaporizer.

3, Hexalin feed pump NO. 3- A.

4. Turn heat off condenser.

5. Turn steam off vaporizer.

6. Turn cooling water off cnndenser.

No. 4-A (Caution) In freezing weather leave small flow of water on condenser, if shut down is to be for some time, condenser and water lines should be drained).

C. Control of Process

No control is required for the vapor phase oxidation beyond maintenance of uniform flows of air and Hexalin being fed. Operating temperature of the converter

cont. from page 4.

must be between 500 and 550 C. It is necessary to supply some external heat to the converter.

As a general check on conversion and performance of the unit the laboratory should analyze thec crude product by precession distillation. Occasional analyses should be made of the off gas, to check the loss of material as CO and CO_2.

Part 12. Refining of Cyclohexanone

Outline of Process

The crude Cyclohexanone produced in the vapor phase oxidation is refined in a 200 gallon vacuum still. The process consists of drying the charge by refluxing through adecanter, producing a refined ketone fraction, aAsemi-refined ketone fraction, and a hexalin residue which is returned to hexalin storage.

Distillation Procedure:

See Fig. "2 for flow diagram.

A. Production of Refined Cyclohexanone from vapor phase Oxidation.

1. Charge still NO. 10-A.to approximately one inch from top of sight glass with crude Cyclohexanone from No. 7 tank.

2. Turn water on ketone still condenser No .10-C.

3. Be sure that still is vented through valve 1. at base of 10-H.

4. Turn full steam on coils to No.10 -A kettle and heat to about 80 C. Reduce steam flow to about 100 lbs. per. hour and bring still to reflux.

5. Set valve so that make passed to decanter No1 10-D water layer to water storage aNo. 6 and oil layer returned to

still kettle.

6/6/50 ZD

6. Decant water continuously at approximately 2:1 reflux ratio. The proper draw off is that which gives the maximum water removal.

7. When head temperature reaches 100 C. and no more water will separate turn off steam to kettle.

8. Close valve to decanter No 10-D and prepare still for vacuum operation.

9. Close valve on oil return to kettle.

10. Start vacuum pump as instruced below and gradu--ally reduce pressure on system .(At such a rate the normal boil up is maintained to 2.0 inches H. g.

11. The following cuts are made:

A. From water cut to R.I - 1. 4520 at 15.6 C. at a 4:1 reflux ratio - to crude ketone storage No. 7.

B. From R. I. equals 1.452 to 1.453 at 15.6 C. at 2:1 reflux ratio to refined ketone storage No.12

C. From R.I equals 14530 to 1.4540 at 15660 at 4:1 reflux ratio to refined ketone storage No. 12.

D. From R.I. equals 1.4540 to a head temper--ature of 88 to 90 C. (at 2inch Hg.) at 4:1 reflux ratio to crude ketone storage No.1.

E. The still residue is pumped (No. 9 pump) to crude hexalin storage at the hydrogenation plant.

12. The actual distillation procedure made , vary som--ewhat from the above depending upon the quality of the crude cyclohexanone charge. For low grades of crude ketone it will be necessary to take a semi - refined ketone out to be fed in a semi- refined storage No. 11 for redistillation

Figure 3 gives the boiling point pressure curves for cyclohexanone and hexalin.

Fig. 4 gives a pivot distillation of crude cyclohexanone as produced by vapor phase oxidation.

Recovery of oil from Cyclohexanone water layer.

1. Charge still No. 10-A approximately 1½ inch below the top of sight glass with water layer No. 6 tank.

2. Turn water on ketone still condenser No. 10 -C

3. Turn steam on kettle and establish reflux.

4. Pass draw off through decanter returning water layer to still kettle and oil layer to crude ketone storage No.7. The rate of draw off is set to give the maximum removal of oil.

5. When all oil has been removed shut down and discharge water im kettle to sewer.

Vacuum Pump Operation.

1. See that the oil is at the proper level in the oil separator tank. The proper oil is Opalube S.A.E 20 for winter operation and Opalube S.A.E.30 for summer operation.

2. To start Vacuum Pump.

A. Start small flow water to water jacket.

B. See that all valves in oil seal line are closed and remain closed untill pump is running.

C. Turn on power and start pump running.

D. Open wide plug cock in oil seal line.

E. Open one and one-half turns the valves allowing oil to flow into bearings, at each end of crank shaft.

F. Suction lines should be absolutely free of all foreign materials and should be perfectly tight.

6/4/5-D 21

Shutting down Pump.

A. Close plug, cock in oil seal line.

B. Allow pump to run thirty seconds to free itself of oil.

C. Shut off power.

D. Stop flow of cooling water to jacket.

Part 3. Liquor Phase Oxidation of Cyclohexanone to Adipic Acid.

Outline of Process:

Adipic acid is made from cyclohexanone by liquid phase oxidation. Air and cyclohexanone in an acetic acid medium are upassaddco-current through unpacked towers in series.

The towers are maintained at 80 C. by water circulation.

The adipic acid solution formed is collected in a hold up tank for subsequent purification. The spent air is first water cooled and then passed through a refrigerated condenser to remove acetic acid and cyclohexanone. The conde--nsate is returned to the oxidizers with the feed.

Air flow diagram is attached . Fig.5.

6/6/50 JDD

Starting the Process.

1. Aceticcacid Storage: The 200 gallon aluminum tank No.13 is provided for acetic acid storage. Since its only source of acetic acid is from the acid recovery still, this tank will be empty, when first starting the process.

2. Acid mother Liquor Storage; This storage No. 34 tank will also be empty when first starting the prpcess. Its source of acid liquor is from the centrifuge.

3. Refined Ketone Storage: The refined ketone storage No.12 should contain at least 50 gallons of refined ketone as produ--ced in Section 2. above.

4. Content of Oxidizers: If oxiddzers are empty add to No.20-A oxidizer, about 65 gallons of pure acetic acid from carboys.

This is done as follows:

A. Pump 75 gallons acetic acid from carboys through pump No. 18-B to feed tank No.20-C.

B. Pump acid from feed tank through pump No.20-B to oxidizers. If oxidizers are full from some previous shut-down they can usually be started without any charge.

5. Preparation of the Fill:

The feed is mixed in weigh tank No.18 and is suff- icient to last for twelve hours. The normal feed mix consists of the following:

A. Pure acetic acid from No.13 tank	equals	335#
B. Refined ketone from No.12 tank	"	360#
C. Acid mother liquor from No.34 tank	"	800#
		1495#

(Approx.174gals)

Catalyst equals 8lbs. manganese acetate dissolved in Acetic acid, per batch.

6/6/50
gl

Since it is necessary to add approximately 85 lbs of make-up acetic acid every 12 hours, the catalyst is dissolved in the quanity of acid in a carboy ahd pumped to the weigh tank. The remaining acetic acid required, (335)lbs. is run in from the acetic acid storage.

When first starting the process there will be no acetic acid in No.13 tank and no acid mother liquor in No.34 tank. The composition of these mixes will be as follows:

Pure Acetic acid from carboys equal 1135#

Refined Cyclohexanone from No.12 tank equals 360#

Total 1495#

Catalyst equals 8# maganese acetate dissolved in 85 lbs. of acetic acid. all of the acid and catalyst from the first mixes is pumped directly from carboys to the weigh tank. Rubber gloves and goggles should be used during this operation.

6. when the feed mix has been completed, circulate mix th-rough pump No.18-B to top of No.18 tank for two hours.

P 7. Pump feed to feed tanks No.20-C.

8. Water Circulating system.
Fill water hold up tank No.20-E with filtered water and circulate water through oxidizers.

9. Turn water on cooler No.20-F.

10. Turn ammonia on refrigerated column No.20-H.

11. Start feed pump No.20-D.

12. Start air compressor and set flow at 27 cu. ft.per. min. to oxidizers.

13. Turn small steam flow on, feed preheater No.20-N.

14. Start feed of about 5 G.P.H to oxidizers.

15. Adjust water circulating system to hold oxidizers at 80 C.

16. When feed has settled out at G.P.H. gradually raise to 14 G.P.H.

17. Check fl[illegible] of recycle condensate fr[illegible] separator No.20-M and 20-G. Leave rotameters wide open so that full flow returns to oxidizers. 6/6/50 JJ

18. Be sure that steam is on coil of hold up tank No.20-J. and all steam traced lines in this system. These lines should always be heated unless system is flushed and completely drained.

19. Control of process consists of maintaining constant flows of liquid feed and air and holding the oxidizer at 80 C.

Shutting down the Process.

1. Cut off feed to oxidizers.

2. Stop air flow to oxidizers, and shut down air compressor if not needed for vapor phase oxidation or isobutyl propionate

3. Shut down feed pump No.20-D.

4. Cut steam off preheater No. 20-N.

5. Stop cooling water to cover No.20-L and start a small steam flow to the water circulating system, or completely drained to hold up tank No. 20-J. If the shut down is to be for some time the oxidizers should be drained.

6. Turn water off spent air cooler No. 20-F. (Caution: Leave small flow of water on this condenser in freezing weather or drain completely).

7. Turn ammonia off refrigerated spent air cooler.

YOU WILL FIND ANALYTICAL METHODS ON SEPARATE SHEETS.

Part 4. Purification of adipic acid from liquid phase oxidation.

Outline of Process:

The effluent from the mother phase oxidizers is cooled in batch crystallizer and the adipic acid which crystallizes is centrifuged from the solvent medium and washed.

6/6/50
20

The crude w(:hed crystals are dissolve(:)n distilled water and recycled mother liquor with a proportionate amount of second crude crystals and treated with Darco. The hot liquor is filtered free of Darco and slowly cooled in a second batch crystallizer. The adipic acid crystals obtained are centrifu--ged from the mother liquor washed and dried. The Mother liquor from this step is divided into two portions. One portion is held in the water solution tank for dissolving additional adi--ic acid. The other portion purged to a recovery evaporator, where water and acetic acid are removed and adipic acid is recovered from the residue as second crude crystals.

A flow diagram is attached Fig. 6.

Summary of Operating Instructions.

A. Separation of first grade crude crystals from acid mother liquor.

1. Be sure that all lines from hold up tank No.20-J to crystallizer No.30 are heated.

2. Fill water jacket of No.30 Crystallizer and heat to 80 C. with steam.

3. Check distilled water supply,at least 50 gallons should be on hand.

4. Pump acid solution from No.20-J to No.30 crystallizer. Charge should be 250 to 300 gallons.

5. Start slow speed stirrer in No.30

6. Start flows of cooling water to No.30 jacket. Adjust cooling water rate to give cooling of approximately 10 C per hour.

7. When the acid liquor in No.30 has crystallized and colled to 20 C., Check centrifuge No.31 to be sure it will operate properly.

Place crude adipic acid crystal reciver under centrifuge discharge.

81 The centrifuging is done on the following cycle:

A. Set liquor valves to acid mother liquor storage N032.

B. Start centrifuge on slow speed and charge from No.30 crystallized. When cake begins to build up near the top of the basket, stop flow from No.30

C. Put centrifuge on high speed for one minute.

D. Put centrifuge on slow speed.

E. Set liquor valves to water mother liquor storage No.54.

F. Wash crystals with three gallons of distilled water.

G. Put centrifuge on high speed for one minute.

H. Put centrifuge on low speed and discharge basket to crude crystal receiver.

Repeat the above cycle as many times as it is necessary to empty No.30 crystallizer. A 300 gallon charge to No.30 should be handeled in about eight loads to the centrifuge.

9. When charge has been centrifuged, wash centrifuge with five gallons of distilled water. Run wash water to No.54 tank.

B. Purification and Drying of first grade crude crystals.

1. Besure steam is on heating element in water solution tank No.41 and all steam traced lines.

2. Check distilled water supply, at least 150 gallons should be on hand.

3. To No.41 tank add 45 gallons of distilled water and 50 gallons of water mother liquor from No.54 tank. Start agitator and heat to 80 C.

4. Add 395 lbs. of first grade crude crystals and 30 lbs. of residue crystals and dissolved,

6/6/50

5. Add 8(lbs of Darco and mixe well.

6. In case there are no residue crystals the batch is com-
-pleted with first grade crystals.

Distilled water is used when there is a storage of water mother liquor.

7. Check filter press No 46 be sure that it is in operating condition. Steam should be on all trace lines and heating elements in press.

8 . Start circulating pump No.46 A. and circulate water solution through filter press No.46 to top of No.41 tank.

Continue circulation untillall Darco has been removed from the solution.

9. Fill jacket to crysstallizer No.50 with water and heat to 80 C.

10. Turn steam on tracer system from filter press No. 50 crystallizer, these lines remain heated unless flushed and drained .

1 1. When water solution is free of Darco pump to cryst-
-allizer No.50.

12. Stop agitatur on No.41 and No.46-A. pump.

Caution: Do not turn steam off of heating system unless flushed and drained.

13. Start slow speed stirrer in crystalizer No.50 and starts cooling waterto jacket. Cool charge at the of 10 C per. hr. cool to 20 C. Care must be taken to avoid sudden cooling since rate of cooling governs crystals size. It may be necessary to "seed" the batch to start crystallization. This is done by introducing several crystals of pure adipic acid to the crystalizer

14. when the solution in crystallizer No.50 has been evolved to 20 C. and crystallized it is ready for centrifuging.

15. Place refined adipic acid crystals reciever under centrifuge discharge.

6/6/5 jpw

16. Centrifuging is done on the following cycle:

(A) Set liquor valves to water mother liquor storage No.54.

(B) Start centrifuge on slow speed and charge from No. 50 crystallizer. when cake begins to build up near the top of the basket stop flow from No.50 crystallizer.

(C) Put centrifuge on high speed for one minute.

(D) Put centrifuge on slow speed and wash cake with three gallons of water-distilled.

(E) Put centrifuge on high speed for one minute.

(F) Put centrifuge on slow speed and discharge crystals to refined adipic acid crystals receiver.

The above cycle is followed untill crystallizer No.50 is empty. A full charge of approximately 300 gallons in No.50 should be handeled in eight loads in the centrifuge.

17. Drying of adipic acid.

The drier No.56 provides for this operation is a counter-current rotary drier. Air heated to 140 C. by passing over steam coils, is drained through the drier in one direction and adipic acid is fed in at the other end.

Dry refined Adipic acid crystals are discharged at the other end.

The drier is of sufficient capacity to handle a twenty-four hour make of refined wet crystals in six hours. The actual performance of drier and rate of throughput must be adjusted by analysis.

C. Recovery Evapoator:

The portion of water mother liquor in No.54 tank which is not needed for dissolving crude adipic acid is pumped to recovery evaporator No.60.

6/6/50 JN

The water and acetic acid are evaporated to the atmosphere through the vent. The extent to which the evaporation is carried should be determined by cooling a sample of hot liquor and noting the crystallization occurs at 20 to 25 C. , the evaporation is stopped and the hot liquor discharged through a heated line to crystallizer No. 30. The crystallization and centrifuging of recovered adipic adid is the same as in Part B. above .

Analytical Procedure:

The pure adipic acid can be analyzed by dissolving a 1000 gram sample in methanol and tetrating with 0.5N caustic in methanol solution to the phenol-phthalein end-point.

$$\% \text{ Adipic acid equals } \frac{\text{CC N base} \times .073 \times 100}{\text{Wt. sample}}$$

A tytration of the base against 0.5 NHCl should be run for each determination.

Part 5 . Recovery of Acetic acid and Adipic acid from the Purge Liquor.

Outline of Process:

Lactone acids are formed as by-products in the liquid phase oxidation, these are soluble in the solvent medium and are purged from the system to prevent building up. The part of acid mother-liquor which is not recycled is charged to the acid recovery still. Acetic acid and ketone are recovered at the head of the still. Lactone acids are removed when adipic acid is centrifuged from the residue liquor.

A flow diagram is attached, figure 8.

6/6/50 JM

Operating Procedure:

A. Acid Recovery Still.

1. Charge acid recovery still kettle No.33-A to approximately one inch from the top of the sight glass with acid mother liquor from No.32 tank.

2. Turn water on condenser No.33-C.

3. Turn steam on coils to No.33A. The full steam flow may be applied until the base of the column begins to warm up. Then reduce steam flow to about 100 pounds per. hour and establish reflux.

4. Turn steam on tracer to discharge line No.33A to hold-up tank No.34 and heating coil in No.34. Leave heat on this system at all times unless completely drained.

5. The following cuts are made:

A. Ketone, water cut at 5:1 reflux ratio to ketone water layer storage No.6 (approx. 4 gals.)

6. B. Weak acid cut at 10:1 reflux ratio. This is drained from the receiver to a carboy to be discarded. (approx.5 gal)

C, Strong acid cut at 1:1 reflux ratio to receivers 33-E and 33-F and then dropped to pure acetic acid storage No.13 (approx.156 gals.)

D. The residue is discharged to No.34 hold-up tank.(approx. 34 gals)

6. When the required strong acid has been removed cut off steam and discharge hot residue to No.34 tank.

7. Turn water off condenser No.33 C.(Caution: Leave small flow of water on this condenser in freezing weather or drained completely). Since acetic acid freezes at 16C. care must be taken to avoid plugging the condenseer and draw-off lines.

Steam addition to the cooling water should be used when the raw water temperature is below 16C. The draw-off and reflux lines should be completely drained during shut-downs when the atmospheric temperatures is below 16 C. The quality of acetic acid produced is cchecked by the operator.

A 20 cc. sample of the acid is titrated with 1.0N. NaOH to the phenolphthalein end-point.

Calculation:
cc of 1.0N. NaOH 2.85 equals % acetic acid.

B. Adipic Acid Recovery from the acid still residue:

1. The acid still residue in No.34 tank is pumped to crystallizer No.30 and crystallized as instructed above for acid mother liquor.

2. The adipic acid crystallized from the residue is centrifuged and washed. The mother liquor is discharged to a drum for burning.

3. The crystals are washed with 3to4 gallons of water. The water is discharged to the sewer.

4. Discharge crystals from centrifuge to residue crystals storage. These crystals are reworked with first grade crude crystals to produce pure adipic acid.

FIG III
BOILING POINT
PRESSURE
CURVE

FILE DESCRIPTION

PHILADELPHIA FILE

SUBJECT HARRY GOLD

FILE NO. 65-4307

VOLUME NO. 1B12

SERIALS (2)

thru

(4)

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

VOLUME Bulky

PHILADELPHIA FILES

(1)

INVENTORIED BY RLH

REVIEWED BY RLH

Date: 6/78 (month/year)

File No: 65-4307

Re: HARRY GOLD

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)
1B-12(2) #1	—	COPY OF ENVELOPE	1	1	CONTENTS SENT TO NEW YORK (7/5/50)
1B-12(2) #2	—	DITTO	1	1	DITTO
1B-12(2) #3	—	DITTO	1	1	DITTO
1B-12(3) #1	—	DITTO	1	1	
	7/10/50	SA to SAC MEMO	1	1	
	VARIOUS	HANDWRITTEN NOTES OF CHEMICAL FORMULAS AND MATHEMATICAL EQUATIONS	21	21	
	—	SCALE DRAWINGS	1	1	
	—	HANDWRITTEN NOTES ON A. BROTHMAN & ASSOC. LETTERHEAD	16	16	
	—	HANDWRITTEN NOTES ON CHEMICAL PROCEDURE	3	3	
	—	HANDWRITTEN NOTES ON CHEMICAL FORMULAS	20	20	
	11/14/47	LETTER TO GOLD FROM KEUNEN MANUFACTURING CO. INC.	2	2	

FBI/DOJ

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

VOLUME BULKY

PHILADELPHIA FILES

(2)

INVENTORIED BY DL

REVIEWED BY DL

Date 6/78 (month/year)

File No: 65-4307

Re: HARRY GOLD

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)
18-12 CONT.	11/24/47	LETTER TO GOLD FROM SOCONY-VACUUM O.L CO.	1	1	
	12/8/47	LETTER FROM UNION BAY STATE CHEMICAL CO.	2	2	
	8/22/47	HANDWRITTEN NOTES ON A. BROTHMAN & ASSOC. LETTERHEAD	1	1	
	12/4/47	LETTER FROM DISTRIBUTING AND TRADING CO. INC	1	1	
	7/10/47	LETTER TO GOLD FROM ATLAS POWDER COMPANY	1	1	
	8/24/47	HANDWRITTEN NOTES ON A. BROTHMAN & ASSOC. LETTERHEAD	2	2	
	8/13/35	DRAWINGS OF CHEMICAL CLORINATION	1	1	
	—	GRAPH	1	1	
	9/6/47	UNION BAY STATE COMP. CHEMICAL ANALYSIS	1	1	
	11/17/47	HANDWRITTEN NOTES ON LETTERHEAD OF A. BROTHMAN & ASSOC.	7	7	
	—	"XR-3180 and XR-4357"	5	5	
	—	HANDWRITTEN NOTES OF CHEMICAL ANALYSIS	2	2	

FBI/DOJ

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

VOLUME Bulky

PHILADELPHIA FILES

(3)

INVENTORIED BY RL

REVIEWED BY RLS

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)
	8/21/46	THE PREP. OF UREA FORMALDEHYDE COLD-SETTING GLUE	1	1	
	—	HANDWRITTEN NOTES ON THE ABOVE GLUE	10	10	
1B-12(3) #2	6/6/50	COPY OF ENVELOPE	1	1	SENT TO NEW YORK (7/5/50)
1B-12(4) #1	6/6/50	DITTO	1	1	DITTO
1B-12(4) #3	6/6/50	DITTO	1	1	
	7/10/50	SA TO SAC PHILA MEMO	1	1	
	—	"WASHING DATA"	1	1	
1B-12(4) #4	6/6/50	COPY OF ENVELOPE	1	1	
	7/7/50	SA TO SAC PHILA MEMO	1	1	
	—	HANDWRITTEN NOTES "LIBRARY WORK"	2	2	
1B-12(4) #6	6/6/50	COPY OF ENVELOPE	1	1	SENT TO NEW YORK 7/5/50
#7	6/6/50	DITTO	1	1	DITTO

PBI/OOJ

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

VOLUME Bulky

PHILADELPHIA FILES

(4)

INVENTORIED BY RL

REVIEWED BY RL

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)
18-12(4) #8	6/6/50	COVER OF ENVELOPE	1	1	SENT TO NEW YORK (7/5/50)
#9	6/6/50	DITTO	1	1	DITTO
	7/7/50	SA TO SAC PHILA MEMO	1	1	
	—	"BLANK SHEETS"	1	1	
	4/22/45	RECORD OF EXPERIMENTS	1	1	
	6/6/50	COPY OF ENVELOPE COVER	1	1	
	7/7/50	SA TO SAC PHILA MEMO	1	1	
	6/6/50	Riboflavin Assays Snell - Strong	1	1	
	12/2/40	Handwritten notes Re: Clay Adam's Co's Centrifuge	2	2	
	1/26/40	Handwritten columns of numbers and notes	9	9	
#11	6/6/50	COVER OF ENVELOPE	1	1	SENT TO NEW YORK (7/5/50)
#13	6/6/50	DITTO	1	1	

FBI/DOJ

Inventory Worksheet
FD-5003 (2-16-77)

VOLUME Bulky

PHILADELPHIA FILES

(5)

INVENTORIED BY RL

REVIEWED BY RL

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)
(CONT.) #13	7/7/50	SA TO SAC PHILA MEMO	1	1	
	6/6/50	" MY IDEAS "	1	1	
		" CITRIC ACID SODIUM TARTARIC ACID CITRATE CREAM OF TARTAR "	1	1	
	4/22/42	LETTER FROM DOC TO HADOC	1	1	
	4/22/42	HANDWRITTEN CONTENTS OF LETTER ABOVE	3	3	
	12/5/40	A RECOMMENDATION FOR A RESEARCH PROJECT	2	2	
		HANDWRITTEN NOTES ASSAY WORK	4	4	
18-12(4) #14	6/6/50	COVER OF ENVELOPE	1	1	
	7/7/50	SA TO SAC PHILA MEMO	1	1	
	10/16/40	HANDWRITTEN NOTES ON READINGS IN CHEM LIT.	4	4	
	—	GRAPHS	2	2	
	—	STAND. CURVE FOR NICOTINIC ACID	3	3	

FBI/DOJ

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

VOLUME Bulky

PHILADELPHIA FILES

(6)

INVENTORIED BY RL

REVIEWED BY RLK

Date: 6/78 (month/year)

File No: 65-4307

Re: HARRY GOLD

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)
(CONT) #14	—	GRAPHS	2	2	
	10/14/41	STAND. CURVE DATA RIBOFLAVIN	9	9	
	—	GRAPHS	2	2	
	—	HANDWRITTEN NOTES ON RIBOFLAVIN	2	2	
	—	GRAPHS	4	4	
	—	STANDARD CURVE FOR RIBOFLAVIN	3	3	
	—	GRAPHS	2	2	
	—	CURVE ANALYSIS [redacted] FOR ABOVE	4	4	
	—	GRAPHS	2	2	
	—	CURVE ANALYSIS [redacted] FOR ABOVE	4	4	
	—	GRAPHS	2	2	
		CURVE ANALYSIS [redacted] FOR ABOVE	6	6	

FBI/DOJ

Inventory Worksheet
FD-503 (2-16-77)

VOLUME Bulky

PHILADELPHIA FILES

(7)

INVENTORIED BY RL

REVIEWED BY RLS

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)
(CONT) #14	—	GRAPHS	2	2	
	—	STANDARD CURVE DATA AND GRAPHS (CONT) RIBOFLAVIN	41	41	
	—	STANDARD CURVE DATA ~~THIAMINE~~ (CONT) THIAMINE	1	1	
	—	STANDARD CURVE AND GRAPH DATA (CONT) PANTOTHEMIC ACID	42	42	
	—	HAND WRITTEN NOTES ON RIBOFLAVIN STANDARD CURVE	1	1	
18-12(4) #15	6/6/50	COPY OF ENVELOPE COVER	1	1	
	7/7/50	SA TO SAC MEMO	1	1	
	—	"BACK ASSAY RESULTS	1	1	
	—	STANDARD CURVE DATA AND GRAPHS ON RIBOFLAVIN	8	8	
	—	NOTES ON ABOVE	1	1	
	—	STANDARD CURVE DATA AND GRAPHS ON PANTOTHEMIC ACID	8	8	
		NOTES ON ABOVE	1	1	

FBI/DOJ

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

VOLUME Bulky

PHILADELPHIA FILES

(8)

INVENTORIED BY RL

REVIEWED BY RLS

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)
(CONT) #15	—	STANDARD CURVE DATA AND GRAPHS ON RIBOFLAVIN	6	6	
	—	STANDARD CURVE DATA AND GRAPHS ON PANTOTHENIC ACID	17	17	
	—	STANDARD CURVE DATA AND GRAPHS ON RIBOFLAVIN	7	7	
	—	GRAPHS ON PANTOTHENIC ACID	2	2	
	—	HANDWRITTEN NUMERICAL CALCULATIONS	2	2	
	—	GRAPHS ON RIBOFLAVIN	2	2	
	—	HANDWRITTEN CALCULATIONS OF NUMBERS	1	1	
	—	GRAPHS ON PANTOTHENIC ACID	2	2	
	—	HANDWRITTEN NUMERICAL CALCULATIONS	1	1	BOTH SIDES OF PAGE
	—	GRAPHS ON RIBOFLAVIN	2	2	

FBI/DOJ

(Name of Contributor)

Received [illegible]

By [illegible]
(Name of Contributor)

To Be Returned to [illegible]
Yes (x)
No ( )

Description of Material Supplied: [illegible]

File No. [illegible]

65-4307-1B-12(2) #1

#8

#7

(Name of Contributor)

By _____ (Contributor)

Address _____

Date of Birth _____

To Be Returned: Yes ( ) No ( )

Description: _____

File No. _____

#2

65-4307-1B-12(2)

(2) #21

Received 6/15/65

(Name of Contributor)

By [illegible]

To Be Issued to [illegible]

Yes ( ) No (X)

Description Phenibut & number of 3 [illegible]

Franklin Hose Co. Equipment Committee [illegible]

File No. 65-4307-1B-12(2)

Date 6/15/65

65-4307-1B-12(2) #3

Date Received 6/6/50

From
(Name of Contributor)

(Address and Occupation of Donor)

By Fred C Bailey
(Donor or Agent)

To Be Returned Yes
No (X)

Description: Loose pages & miscellaneous papers found on shelf of wooden cabinet in basement of Guild Lane

File No. 65-4307-1 B-12(3)

65-4307-13-2(3) #1

SAC, Philadelphia 7/10/50

SA T. SCOTT MILLER, JR.

HARRY GOLD, was.
ESPIONAGE - R

EXHIBIT 65-4307-1B-12 (3)
LOOSE PAGES OF MISCELLANEOUS PAPERS FOUND ON
BOTTOM SHELF OF WOODEN CABINET IN BASEMENT OF GOLD'S HOME

On 6/22/50, GOLD went through these papers and stated that all of them concern work being carried on by A. BROTHMAN AND ASSOCIATES. He said that these papers consist of miscellaneous laboratory reports, work papers, correspondence, and are in the handwriting of GOLD, BOB GERSON, and PHILLIP LEVINE.

GOLD stated that these papers were another group of the numerous papers he took home just before he left the employ of BROTHMAN. At about the time BROTHMAN was having his showdown with the other members of the firm, GOLD told BROTHMAN that he wanted about two weeks to go over all of this material and assemble and annotate it. Subsequently, BROTHMAN told GOLD that the laboratory was locked to GOLD, so GOLD just kept the material.

TSM:HKF
65-4307

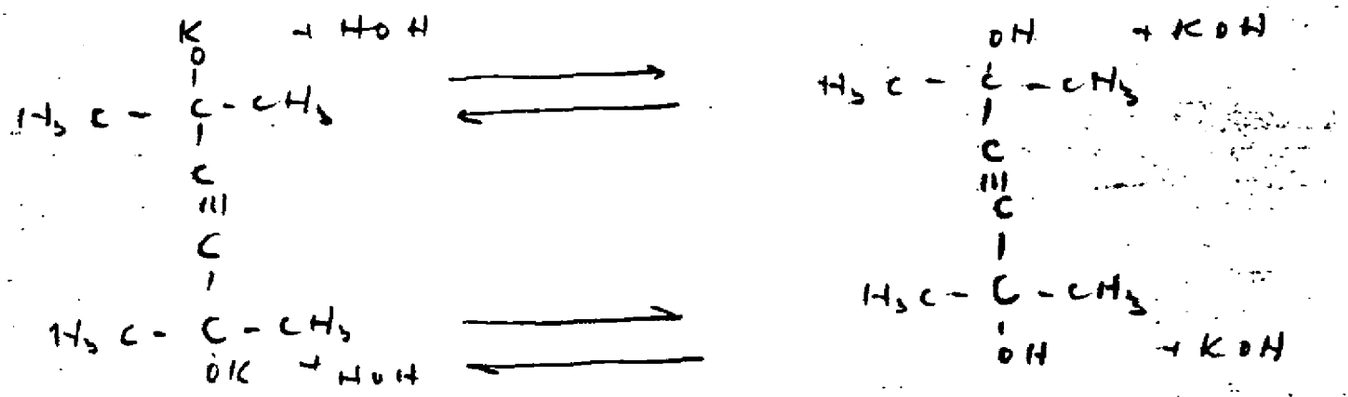
3-28-41

1.) must test ($CaCO_3$ should work for [illegible] - which we would like it if it is true as pertains

2. Recovery of KOH for re-use

110 gms KOH
250 gms H_2O

6/6/50
JDP



a - Determ. of optimum quantity of water addn.

b - Recovery of KOH

① Comp Scheme to Dispose

② m.e. because of KOH's affinity for H_2O

② Then volatile distn

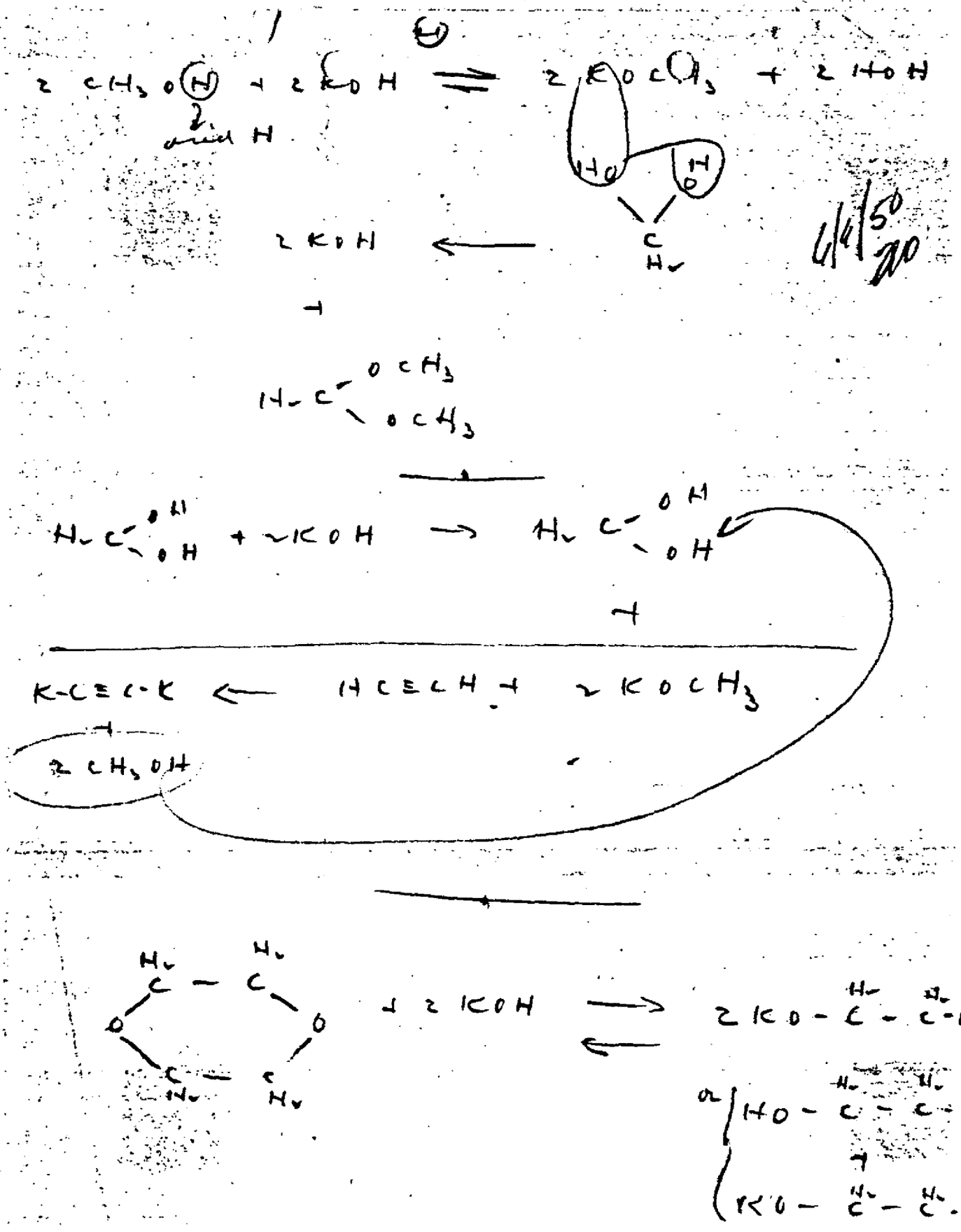
② m.e. $KOH \cdot xH_2O \rightleftharpoons KOH_{dry} + xH_2O$

↓

m.p. decreases as conc. decreases

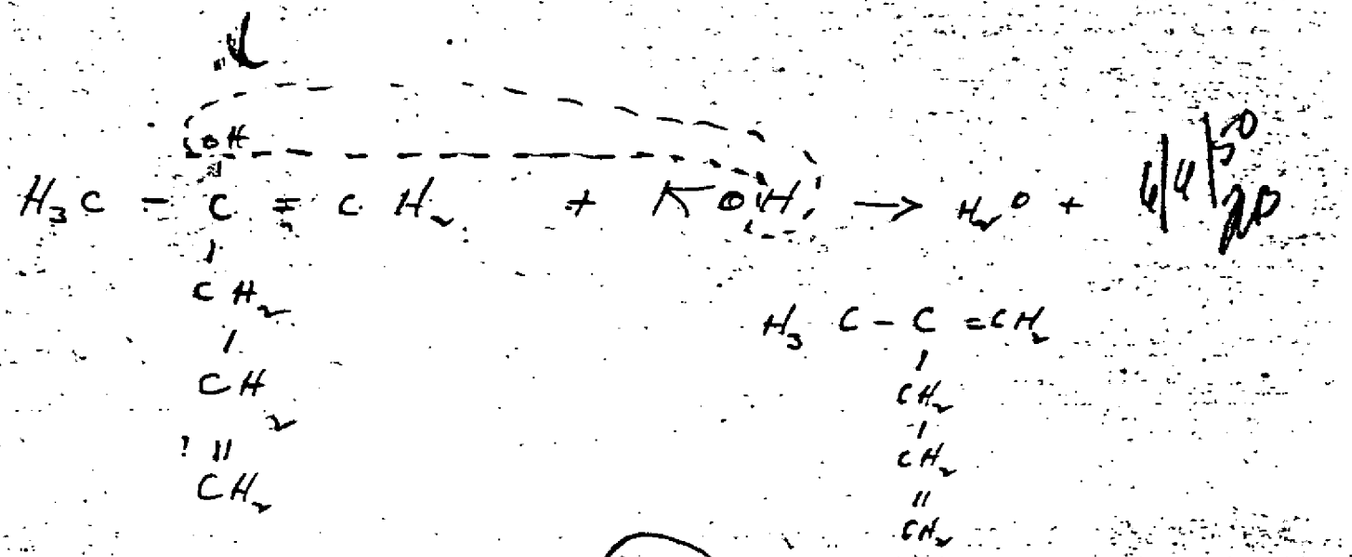
3. methylal as a catalyst

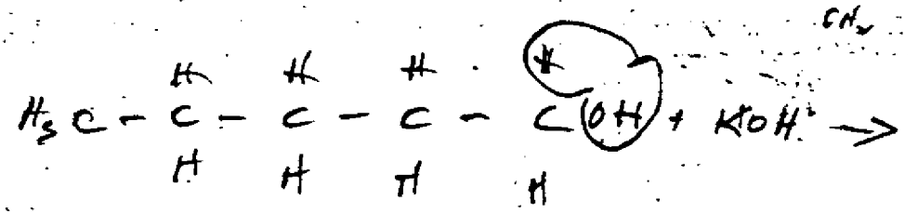
$H_2C{=}O + 2\,HOCH_3 \underset{\text{acid}}{\overset{KOH}{\rightleftharpoons}} H_2C(OCH_3)_2 + H_2O$

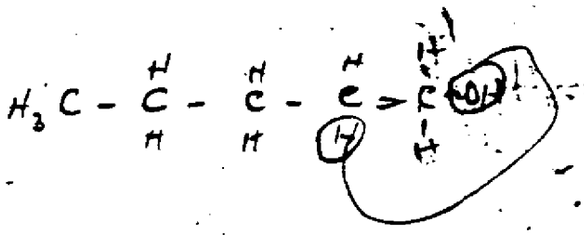


So ... for a similar reaction and ...

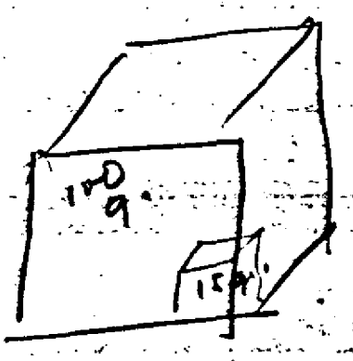
$CH_3\,CH_2\,CH(CH_2\,CH_2\,OH)$





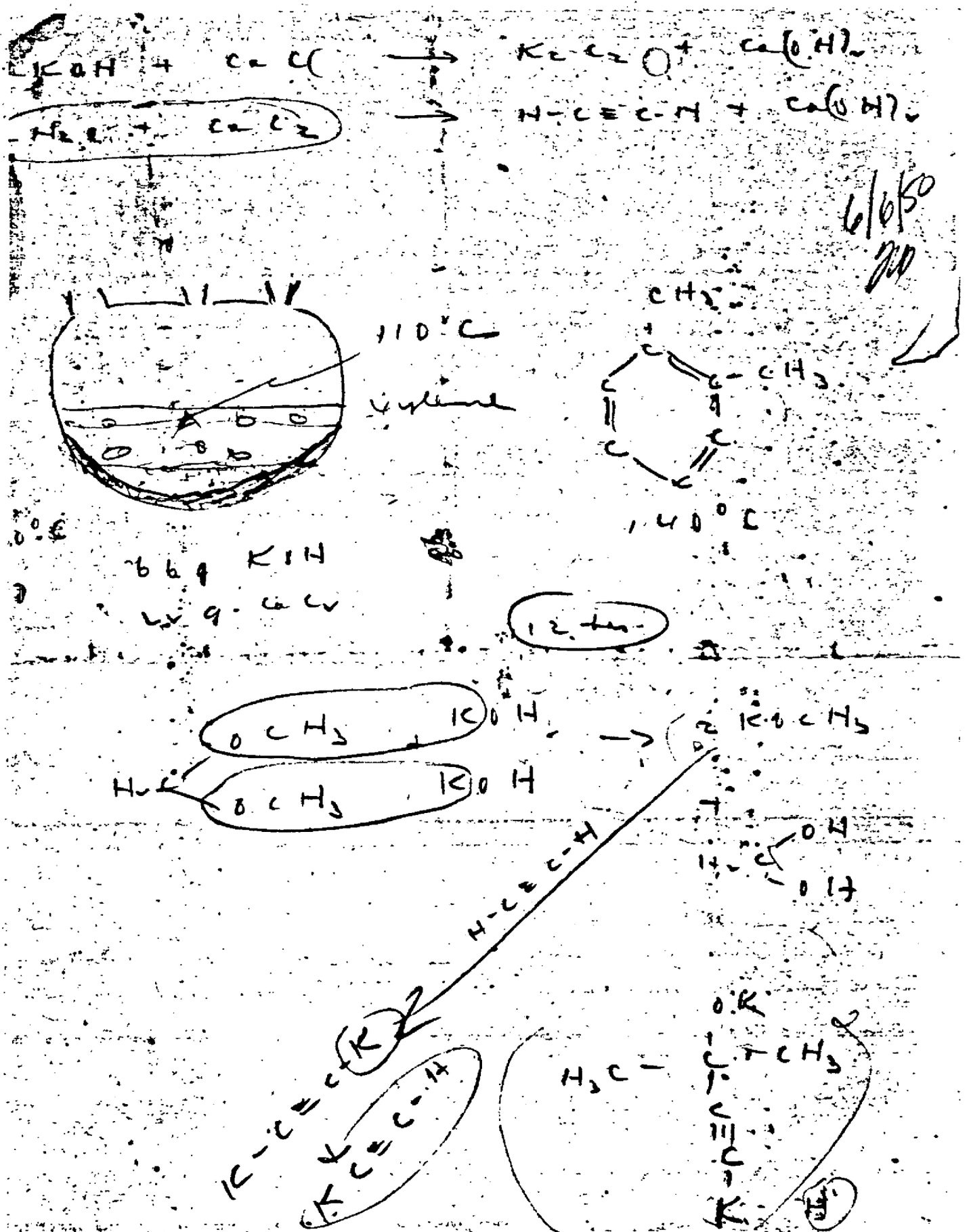


H_2C



8 grams

9 0 70


6/6/50
110°C
140°C
12 hrs.

6/6/50

1. dissolve 2 g KOH
2. add xylene (xylol)
3. wash through Et$_2$O Prom.
4. add NaNO$_2$ solution (1 g to ~10 °C) – watch out for heat rise
5. acidify for 1 hr at 13-15 °C
6. pass in HCl gas at 0 °C for 2 hrs
7. allow to rise to 13-15 °C
8. add acetone, cooling (at the same time cooling bath to 0 °C)
9. after 5 min solid at beginning
10. leave for 2½ hrs (13-15 °C)
11. decompose addition of ~ 4 H$_2$O at 0 °C.

Et 4 96 g

450 cc. methylal

132
132
264
450
716

$\frac{1}{8}$" sheet ($\frac{1}{4}$" sheet ○ $\frac{1}{2}$" sheet

80 cm. Me Mc
20 — gm. T.D.P. ✓
(80-20) $\frac{3.33}{6.66}$ = 99.5 — gm. T.B.P. ✓

125 cm. Me Mc
8.8 — gm. T.D.P. ✓
(125-7) $\frac{3.33}{6}$ = 133.8 — gm. T.B.P. ✓

220 cm. Me Mc
4.4 — gm. T.D.P. ✓
(220+4) $\frac{3.33}{6}$ = 240 — gm. T.D.P.

6/4/50
JP

4. T.D — 80-85°C (81-82)
T.D — 60-65°C (61°C)

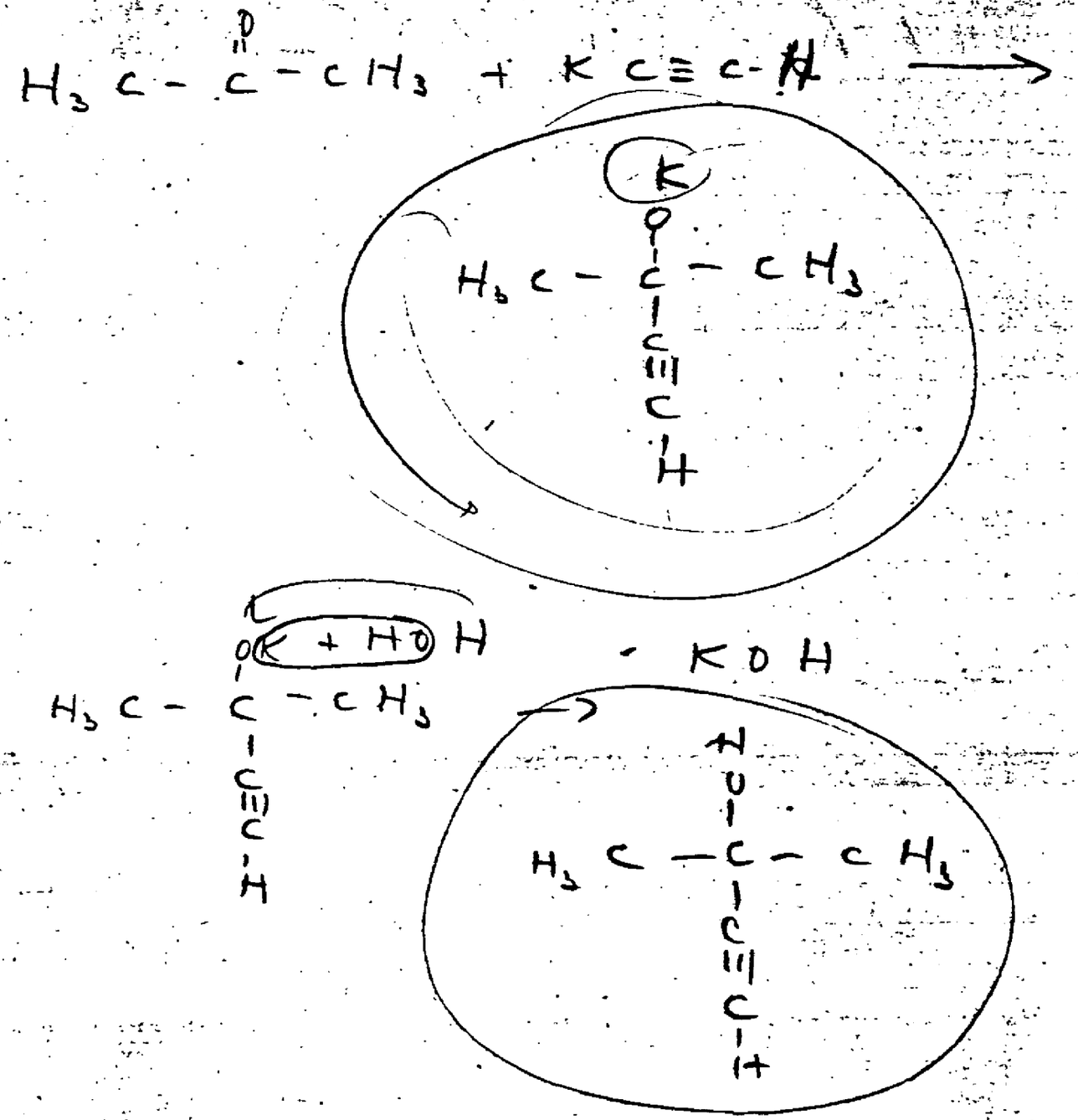
Hydrogenation	1:10	1:10	1:10
	1:45 ← mixed →	1:50 ← mixed →	1:50 mixed
	2:22 ←	2:30 ←	→ 2:50
0 min —	2:25	2:33	2:53
0. one —	2:55 (mixed)	3:56	4:43
0. two —	3:00	3:58	4:43
0 one —	9:15		

Bo 8-9692

ona 9.3795

0 min —
0. one —
mixed —
emulsion —

6/6/50


H3C - C - CH3 + K C≡C-H
K
H3C - C - CH3
C≡C
H
O
K + HOH
- KOH
H3C - C - CH3
C≡C
H
H3C - C - CH3
C≡C
H

$ClCH_2COOH$ $S=C(NH_2)_2 = 76$

35.5
14
45

$CH_2COOH = 92$
SH

94.5

4/6/50 gm

68% H_2SO_4
76% NaOH

36.75 moles $ClCH_2COOH$
32.5 " thiourea
75 moles NaOH
53.3 " H_2SO_4
2.3 " Zn

25.6 moles = 2360 lbs

$HSCH_2COOH$ $.62 - .675/lb.$
+ butyl ether + NH_3

$556 / ton $ $ClCH_2COOH$
742-866 $S=C(NH_2)_2$
60 NaOH
77 H_2SO_4
30 Zn

$1465
124
1589

Ex. 15

$CuSO_4 + HOH \rightleftharpoons Cu(OH)_2\downarrow + 2H^+$

$CuSO_4 + HOH \rightleftharpoons H_2SO_4 + CuO$

$Cu^{+2} + SO_4^{-2}$

$NaCl \rightarrow Na^+ + Cl^-$

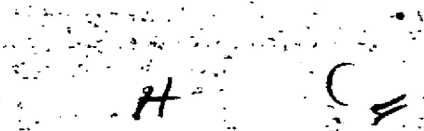
$NaC_2H_3O_2 + HOH \rightleftharpoons NaOH + HC_2H_3O_2$

Na_2O

$H_2S^{-2} + 8HN^{+5}O_3 \rightarrow H_2S^{+6}O_4 + 8H_2O + 8N^{+4}O_2$

$N^{+5} \rightarrow N^{+4} + 1e$ | x 8

$S^{-2} \rightarrow S + 8e$



6/6/50

33
29
3
32

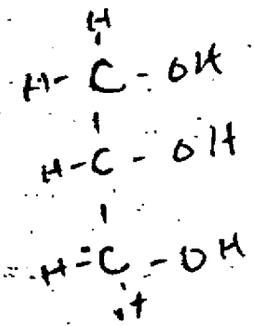
92

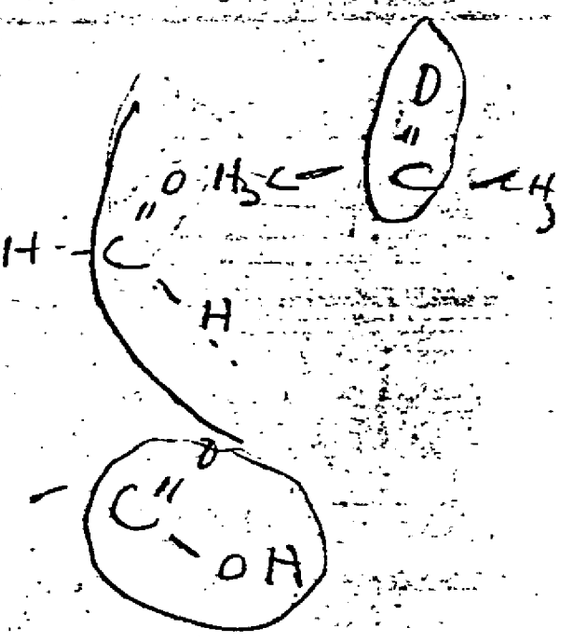
$\frac{33}{92} = 0.36$ # SH / # thioglycolic

$\frac{33}{76} = 0.434$ # SH / # thiourea

$\frac{0.36}{0.434} = 0.83$

$0.83(0.36) =$





1.6111
1.5934
0.0177

6/6/50
800

$\frac{66}{177} \times 1.5 = 0.57$

685

6870

7660 × 0.687 = 5270 #

$\frac{5270}{0.95}$ = 5547 #

7660
5250
5710

760 H_2O

300 $ClCH_2COOH$ (3.18 moles)

241 theorca (3.18)

168 g Na_2CO_3

or 268 $NaHCO_3$

660 g $NH_2 \cdot HCl$ 68.5% H_2SO_4 1.601

$C_6H_5CH_2COONa$

NH

45.6

96

1216

380 H_2O — 100 + 280

150 $ClCH_2COOH$ warmed & room T. & dissolve

120.5 theorca

84 g Na_2CO_3 added solid temp went to 40°. Added H_2O & make 350 cc.

330 g 68.5% H_2SO_4

64 g NaOH } Ran in at 25°C Temp
108 g H_2O } dropped from 80°C → 50°

Heated till temp reached 100°, Sol'n became clear at 3:15

Sample at 3:30

25.02 9.22

11.87 6.00

15.29 3.22

43.22

6.51

760

4:00 PM

22.00

5.27

800 cc,

4:45 To ½ of'n (330 cc.) added 32 g in 55 cc. H_2O

5 v.o.

5-3

3.18

Na_2CO_3 10.6 g/100 cc.

98%

1.59 × 40

63.6

107 g.

3 × 3.5

120

240

107

350

457

9/17/47

95g $ClCH_2COOH$ dissolved in 218cc. H_2O. Added 0.6 g. $CaCO_3$ then 76.1g. thiourea. Heated to 40°. Removed heat. Temp rose to 75° in ca. 4 min. & after a few min. began to fall. Maintained at 75° 15 min. Raised to reflux → clear very light yellow sol'n. Began to add 42g. NaOH in 60cc. H_2O at 12:45. After about ½ added in 5 min, fine needle cryst'l in such vol. that stir. in had to be stopped till stirr broke up mass. Finished add'n NaOH at 1 PM. Considerable heat evolved by add'n NaOH. Heated under reflux, pale yellow suspension. Cryst. mat. did not disappear. Titration of approx 1 c.c. req'd 4.5 cc. Added 2nd mole alkali at 4 PM, & titrated mins. Req'd 1 cc/cc. At 4:25 req'd 14.25; 5:00 – 15.4; 5:30 15.85; 6:00 – 16.2 cc. 0:30 – 16.5 cc.

Cooled to 40° & acidified with 70cc, 6 N H_2SO_4. Vigorous gas evol'n. Added 4g. Zn + 10 cc, add'l acid. Filtered → 520 cc. 5cc. sample required 73.5 cc. 0.1N sol'n. = 71g = 77%

$$\frac{536c}{025} \times hSH \times 0.077 \times 25 \times 14$$

2 mole NaOH = 80g.
1 mole = 40g.

$$14.7 \frac{73.5}{5} \times 0.077 \times 454 \times \frac{520}{3985}$$

~~75 H₂O~~
~~25 ClCH₂COOH~~

94.5 $ClCH_2COOH$
76.12 thiourea
53¼ = 13g Na_2CO_3
ca. 2½ H_2O used 85 cc.
~~½~~ 40 ml NaOH in 80 cc. H_2O

Added thiourea at 50. Temp dropped to 45°, then rose very slowly. Heated to 90° + removed flame. Temp rose with increasing velocity attaining b.p. (107°) in ca 2 min. Boiled 10 min. Cooled, filtered. Added 4½g NaOH in 60 cc. H_2O resulting in ppte. Added 20 cc. H_2O + NaOH + 60 cc. to reaction mixt clear on heating with

$$[\text{COOH}]^+ Cl^- \rightleftharpoons$$

$$+ \text{NH}=\text{C}(\text{NH}_2)\text{SCH}_2\text{COO}^- + NaCl$$

Sol'n did not become the alkali at 95° although partial decomp'n complex appear. Took place. Necessary to add 5g. NaOH to obtain clear sol'n.

550 cc

$$\frac{0.8}{45} = \frac{1}{x} \qquad x = \frac{45}{0.8} \quad 107$$

$$\frac{52}{5} \times 0.077 \times 454 \times \frac{538}{380} \quad 92g.$$
10.4 × .977

52.5 g = 57%

212.

$$CH_3\text{-}\overset{CH_3}{\overset{|}{\overset{+}{C}}}\text{-}\overset{O}{\overset{\|}{C}}\text{-}OH$$

dichloro Pentanes (mixed) ($C_5H_{10}Cl_2$)

6/6/50
JB

UP

1. mixed pentanes, C_5H_{12} + Cl_2

→ dichloropentanes + 2 HCl

from Natural Gas
(both n-pentane
& iso-pentane)

2. The process is a vapor phase one

M.P.

All isomers $C_5H_{11}Cl$

0.20 lb. wax / 400 ft²

$$\frac{0.20 \times 4}{400} = 0.002 \text{ gm/ft}^2$$

$$6.2 \times 6 \quad \frac{36}{144} = 0.25$$

$$0.227 \times 1.25 = 0.057 \text{ gm. wax}$$

$$\frac{0.057 \text{ gm. wax}}{36 \text{ in}^2}$$

$$\frac{0.057}{0.01}$$

$$0.75 \text{ pound} / 400 \text{ ft}^2$$

$$= 0.123 \text{ gm. pound} / 36 \text{ in}^2$$

$$\frac{37 \text{ lb.}}{5} = 7.4 \text{ lb/gal}$$

3.7 lb./gal

$$= 0.164 \text{ gm. sizing (air)}$$

81.291
78.691 Tue
2.600

81.377
81.291
0.086

81.517
81.377
0.140

0.226

81.073
78.691 Tue
2.382

81.158
81.073
0.085

81.269
81.158
0.111

0.196

80.937
78.691
2.246

81.016
80.937

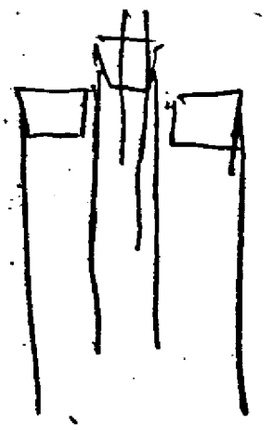
81.099
81.016

81.117
71.047

0.070 g

71.213
71.117

0.096



9.2 cc, original aq. soln

4.05 after extraction

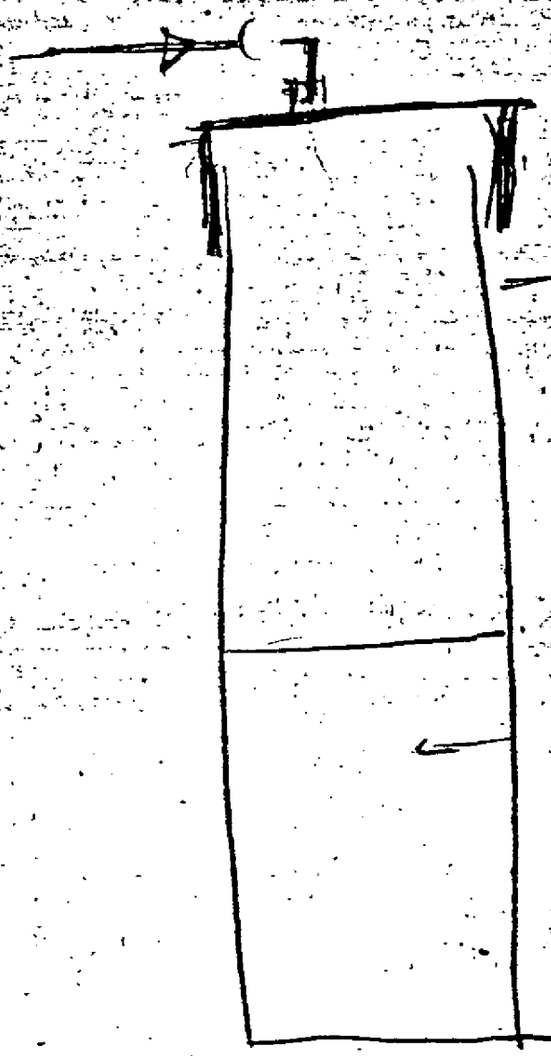
7.15 in ether — 5.23 in alcohol

$$K = \frac{4.05}{7.15} = 0.567$$

0.5

$$\frac{200}{150} \cdot 66 = 88 \text{ g}$$

05 | 01 | 9



Bender Bracket

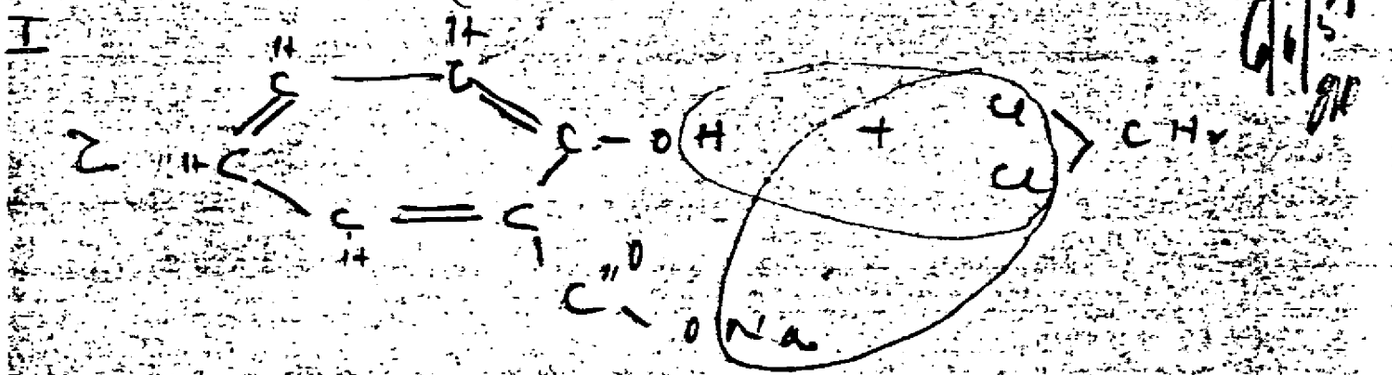
Given

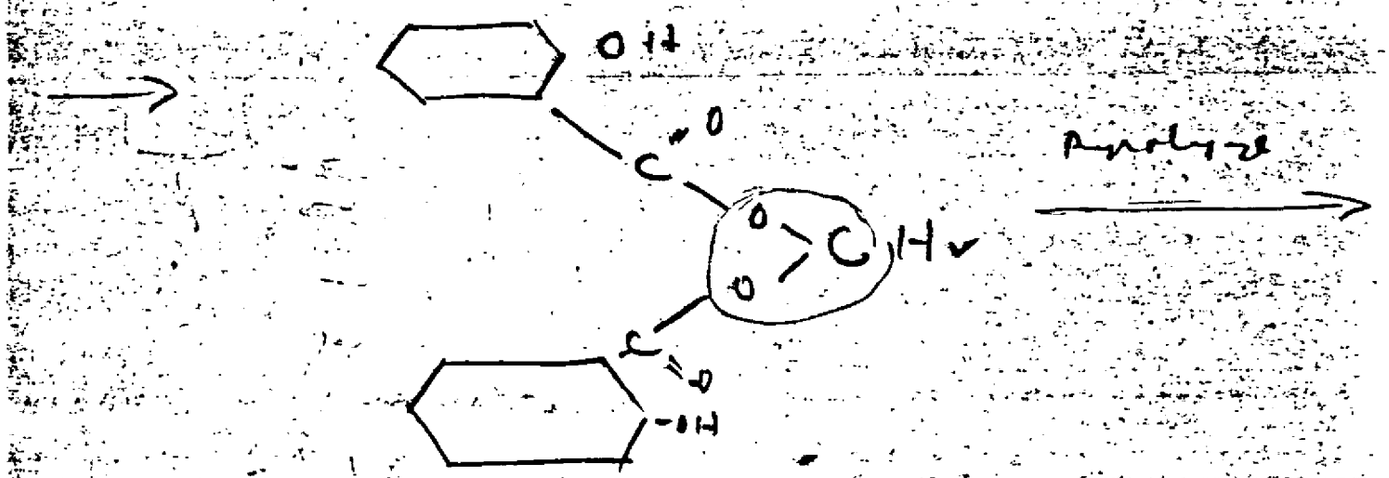
1: chord with C_{lv}

@ -11°+30°C

~ add

Project – Pascal

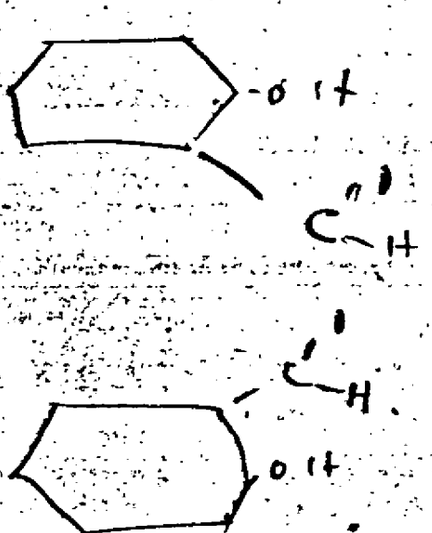




Analyse

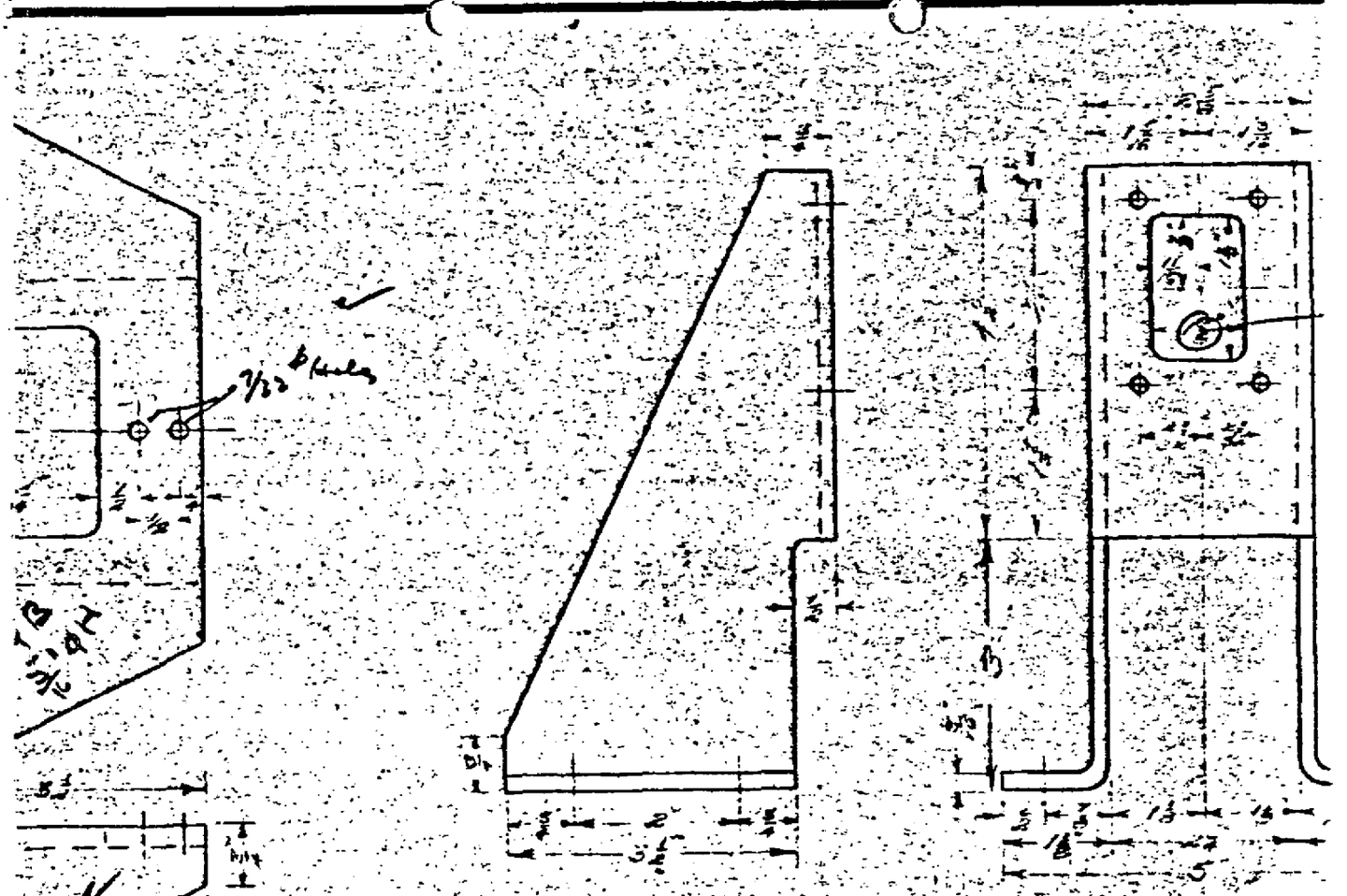
chemicals

CH_2Cl_2

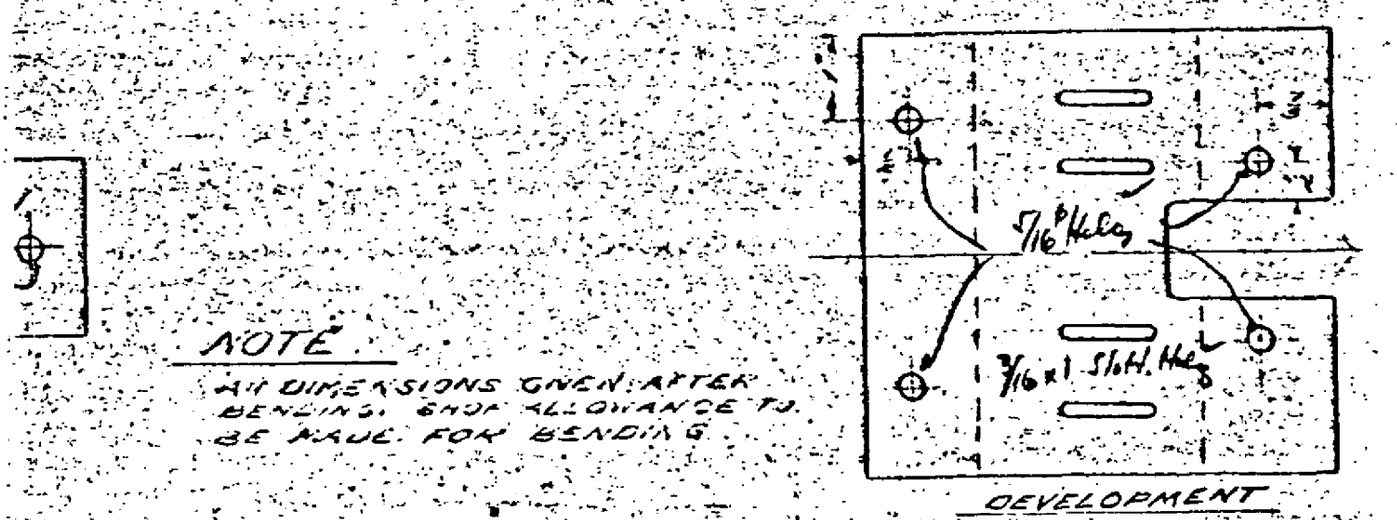


apparatus

3 neck flask
"Dreo" Heater
Distilling Column
Condenser
Receiver



E SUPPORT



NOTE.

ALL DIMENSIONS GIVEN AFTER BENDING. END ALLOWANCE TO BE MADE FOR BENDING.

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

6/6/50

No. of

Date:

By:

9/16

82.332 82.499

82.257 82.382

0.117

9/16

82.490 82.610

82.330 82.498

0.160 0.120

9/16

82.614 82.740

82.505 82.619

0.109 0.121

A. BROCHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By:

9/17 (1)

82.496
82.396
0.100

82.599
82.486
0.112

6/4/50

9/17 (2)

82.760
82.573
0.187

82.959
82.760
0.199

9/17 (6)

81.912
81.815
0.097

82.208
81.912
0.296

9/13 (3)

82.057
81.962
0.095

82.251
82.057
0.194

82.092
81.983
0.109

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

Date:

By:

A. BROCHMAN & ASSOCIATES

No. 2 of

Date: 11/v/47

By: H.G.

JOB: Thioglycollic acid

SUBJECT: A D A Process

material	Lbs.	mols	Price/unit	Total Cost
monochloracetic acid				
Thiourea	1850	24.35		665.00
NaOH		48.7		89.50
H2SO4		28.97		
Zn Dust				
anhyd NH3				
Buffed Cells				

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By:

90% 95%

18.5 moles acid (Theoretical) = 24.35 moles theoretical acid

6.8 × 1.95

Wet

24.35 = 100 lbs Theoretical

= 0.36/lb = 66

24.35 × 94.5 = 43 lbs ClAcOH

0.97

ClAcOH = 44.2 ClAcOH at 97 lbs

Have NaOH in excess to

ClAcH as 5 moles

2-NH2 = 266 lbs NaOH

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By:

32.8 ml NaOH

32.6 ml HCl

39.8 ml NaOH (76%)

97 counts NaOH

53.9 ml H2SO4 69% → 1076 ml

ml 2 Dec

101.6 ml H2O

6.8 ml H2O evap

1700 ml final titrate

=

18.5 mg

$\frac{18.5}{32.6}$ = 56.7% Cl

10 T-ln

$\frac{10 \times 12}{13}$ = 70%

$\frac{.031}{1700}$ = 1.8 / ml

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT

No.

Date: 6/4/50

By:

1.227
− 0.896
0.332 lb. saved

per
8 hrs/cycle or 96 hrs/week

1. Could find two times as much
2. Use in emergency or, multiply/mo
3 Sr. − 2, 000 × 1.33 = 7260
Saving/mo

A. BROCHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By:

$\frac{1750}{97.1} = 18.0$ units finished area

$\frac{18.0}{32.6} = 56.75\%$ build

$\frac{56.75 \times 16}{16} = 71\%$

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By:

material	mole wt	mix	total cost
H2O	3475	18.0	
H2	2475	36.9	
NaOH	3950 (anhyd)	97.0	38.7 lbs NaOH	41.5	
	7695 NaOH		
SO4	7656	53.8	1.9 gal/lb	
Na	150	46.1	
H2	550	32.4	13.6

NaOH + H2SO4 → Na2SO4 + 2H2O

$$\frac{2951 \times 0.76 \times 80}{62}$$

$$\frac{7656 \times 0.69}{98} = 1.4$$

6 mols

A. ROTHMAN & ASSOCIATES

4/4/50

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By:

A. BROTHMAN & ASSOCIATES

JOB: 0

SUBJECT: AMA

No. of

Date:

By:

$\frac{8.97 \times 144.7}{3.8} = 76.37$ C

$CaCO_3$ ... wt basis to

$CaSO_4 = 117$ lbs

@ end lb.

$= .34$

$\frac{1 \times 4 \text{ for } 1700 \text{ lbs.}}{}$

$\frac{1 \times 4}{17 \text{ lbs}} = 0.8 \text{ 96 / lb}$

A. BROTHMAN & ASSOCIATES

JOB: Thioglycollic acid

SUBJECT: Stanton Process

No. 1 of 2

Date: 11/2/64

By: H.G.

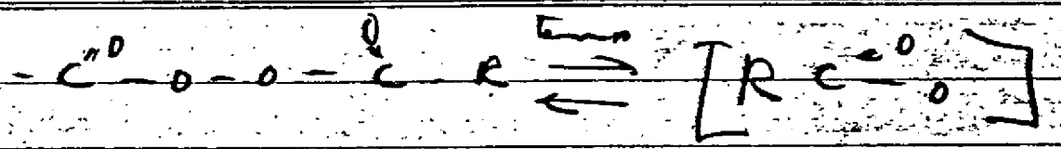
material	Lbs.	mols.	Price/unit	Total cost
Methyl acetate	3478	36.8	0.18	
Thiourea	2477	32.6	0.36	
NaOH ①	3950	97.0	0.045	
H_2SO_4 ②	7650	53.8	0.00185	
Zn Dust	150	2.31		
anhyd NH_3	550	32.4		
Butyl Ether				136

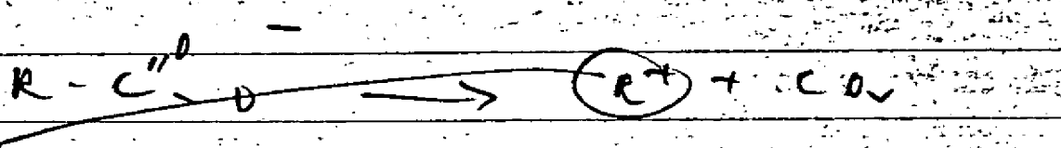
① $NaOH + HOH \longrightarrow 2NaOH$

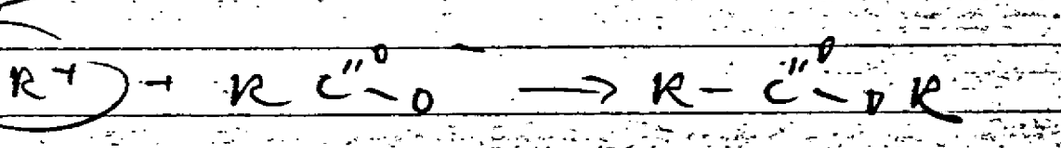
$$\frac{3950 \times 0.76 \times 80}{60} = 3870 \text{ lbs}$$

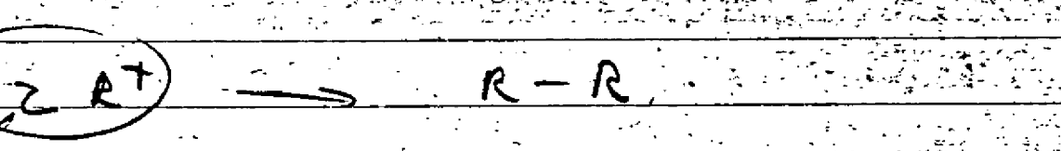
② H_2SO_4

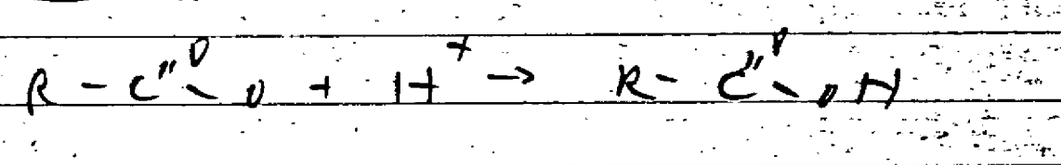
$$\frac{7656 \times 0.69}{98} = 538 \text{ mols } H_2SO_4$$



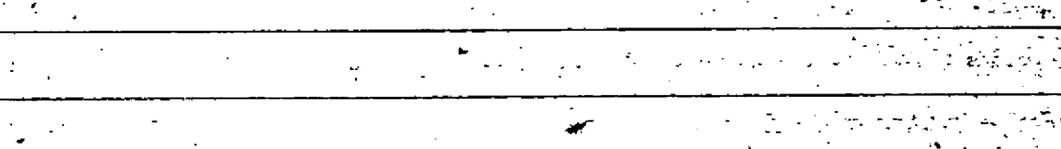








+ monomer → initiated molecules



So called energy of activation is really a measure between two forms.

1. Uniform rate of heating.

A. BROTHMAN & ASSOCIATES

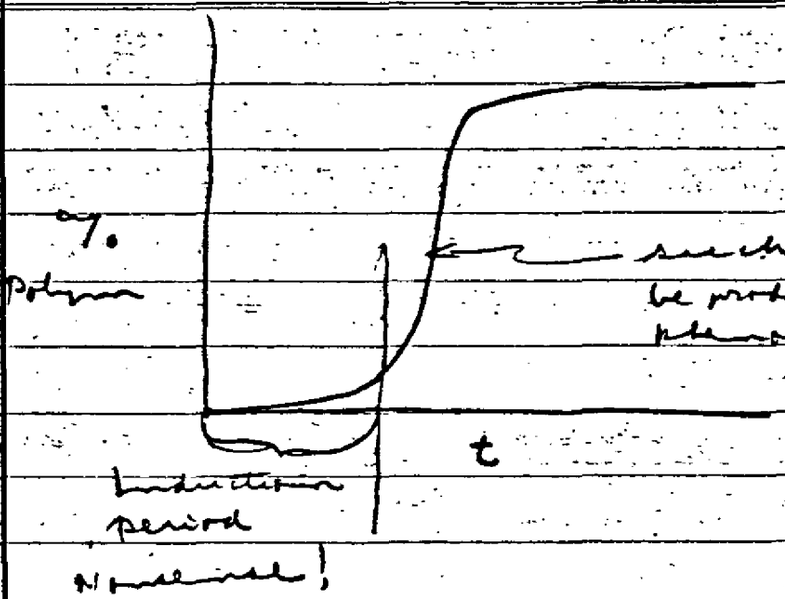
JOB:

SUBJECT:

No. of

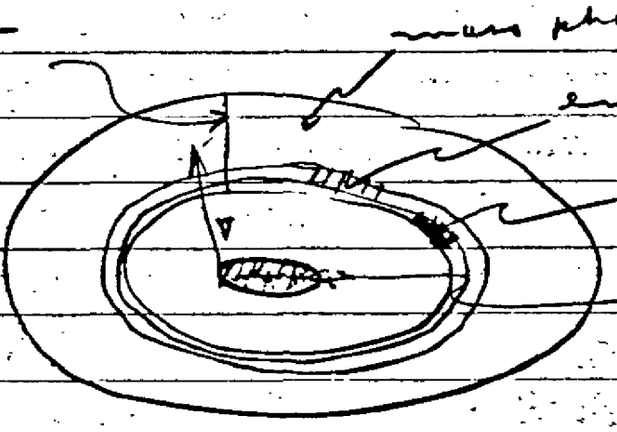
Date: 6/6/50

By: BG



2: must keep ↑, ↓ activated monomer (initiator)

conc. constant, i.e. do not deplete



also to react at t° at 80-88°C in order to maintain t under 80-88°, i.e. all migration of monomer into micelle

A. BROTHMAN & ASSOCIATES

JOB: 1/4

SUBJECT:

No. of

Date:

By:

1. Can these problems mill easy with particle size.

Pearl Polymer

only one problem - different - particle size

Emulsification agent → monomer into film ; ; ; reduction in emulsification agent in mix

Some floating microscopic $Al(OH)_3$ particles So need adequate suspension agent $Al(OH)_3$

Normally made from $Al_2(SO_4)_3$ & NaOH

must reproduce same $Al(OH)_3$

1. order of addn
2. rate of addn
3. Temp.
4. conditioning agent.

1. add 6 L am. ammonia + KOH to 15 L of m-amyl alc.
2. distill off the A—OH – H2O ternary azeotrope (b.p. 96° C) until the theoretical amt of H2O is removed & the still pot temp gradually rises and at the end of the distillation attains 128° C.
3. pour the still fluid while hot into a heavy and quickly placed in a desiccator (with vacuum).
4. place 220 L of methyl alc in a 1 liter flask and chill to 0° C. then pass in C2H2 to saturation (8 liters of C2H2 will be required). then add 50% excess C2H2-saturated methyl alc
5. add the A—OH-KOH solid (in as small particles as possible and with a little exposure to air as possible) and keep the temp at 0° C. when all A—OH-KOH has been added stir for ½ hr at 0° C.
6. remove the ppt of C2H2 and all the temp to rise to 13°-15° C.
7. add 218 g of dry acetone in small portions as possible keeping the

Dipropylene glycol

Methacrylic acid

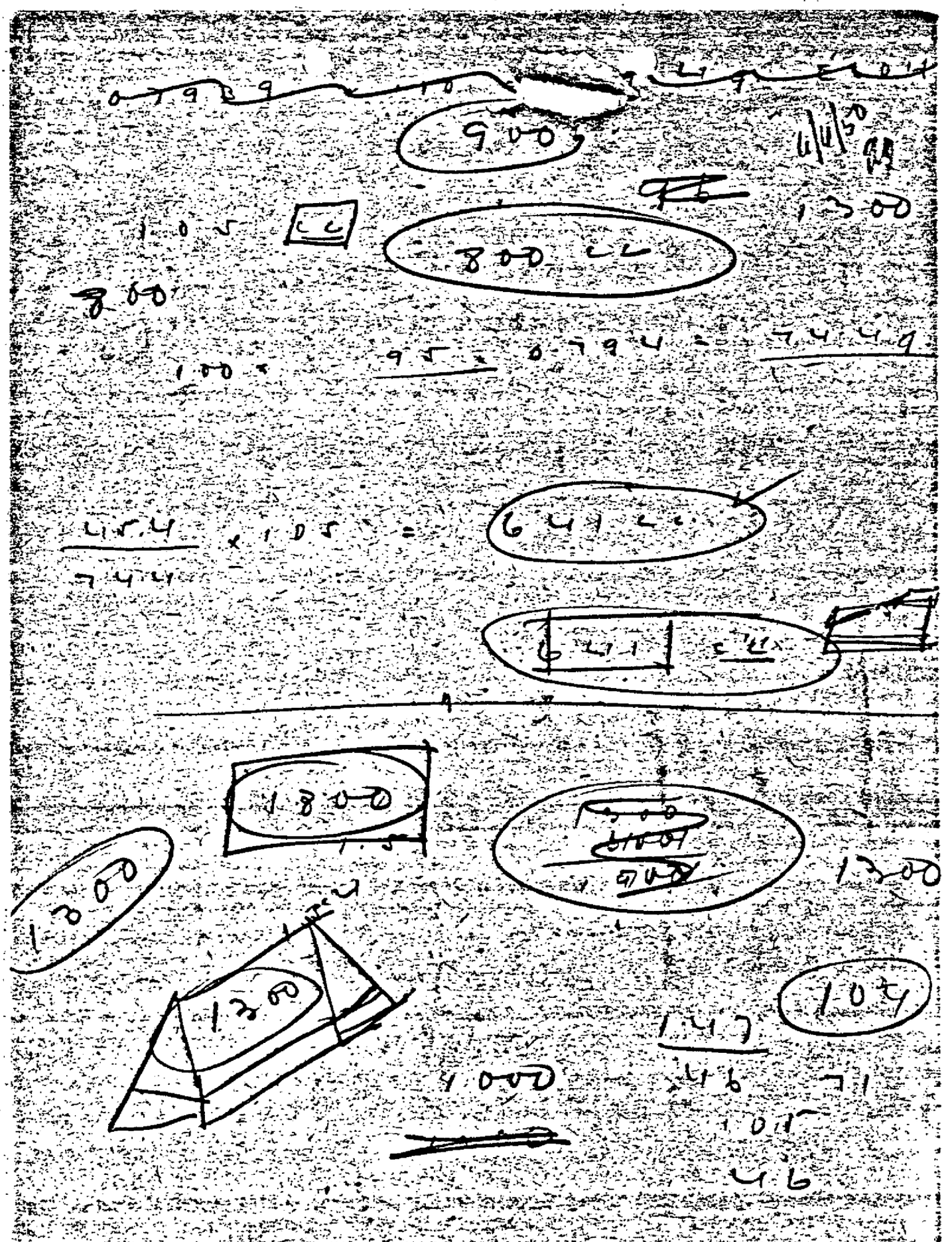
Triethylene glycol diacetate

Glycol Diacetate

Polyethylene glycol

Diethylene glycol

Butyl carbitol acetate



800 cc

7

800

800

100 / 105

0.79

147

605

13

33 × 36 = 1188

4400

1600

3400 cm

9

	CaCO3 added	Temp	NaOH	
	gm			
4 PM	15	14		Start
20	10	15		Foaming
25	15	15		Foaming subsiding with CaCO3
30	5	14		
35	~	14		Start for assay

$\frac{200}{64} \times 5.3 \times 0.1 \text{ N} \times 0.0357$

$= 9.8$ c(a)

Wet cake 28 gm = 14 gm Dry

3 25 AM	20	14	Start
30	10	15	
35	5	15	no cooling

$\frac{200}{1} \times 6.3 \times 0.1045 \times 0.0357$

$= 5.1$ gm c(a) $\times \frac{1}{2}$

Wet cake = 18 gm
(10% Moisture)
= 24 gm Dry

3 55	5	16	Start again
4 25		17	

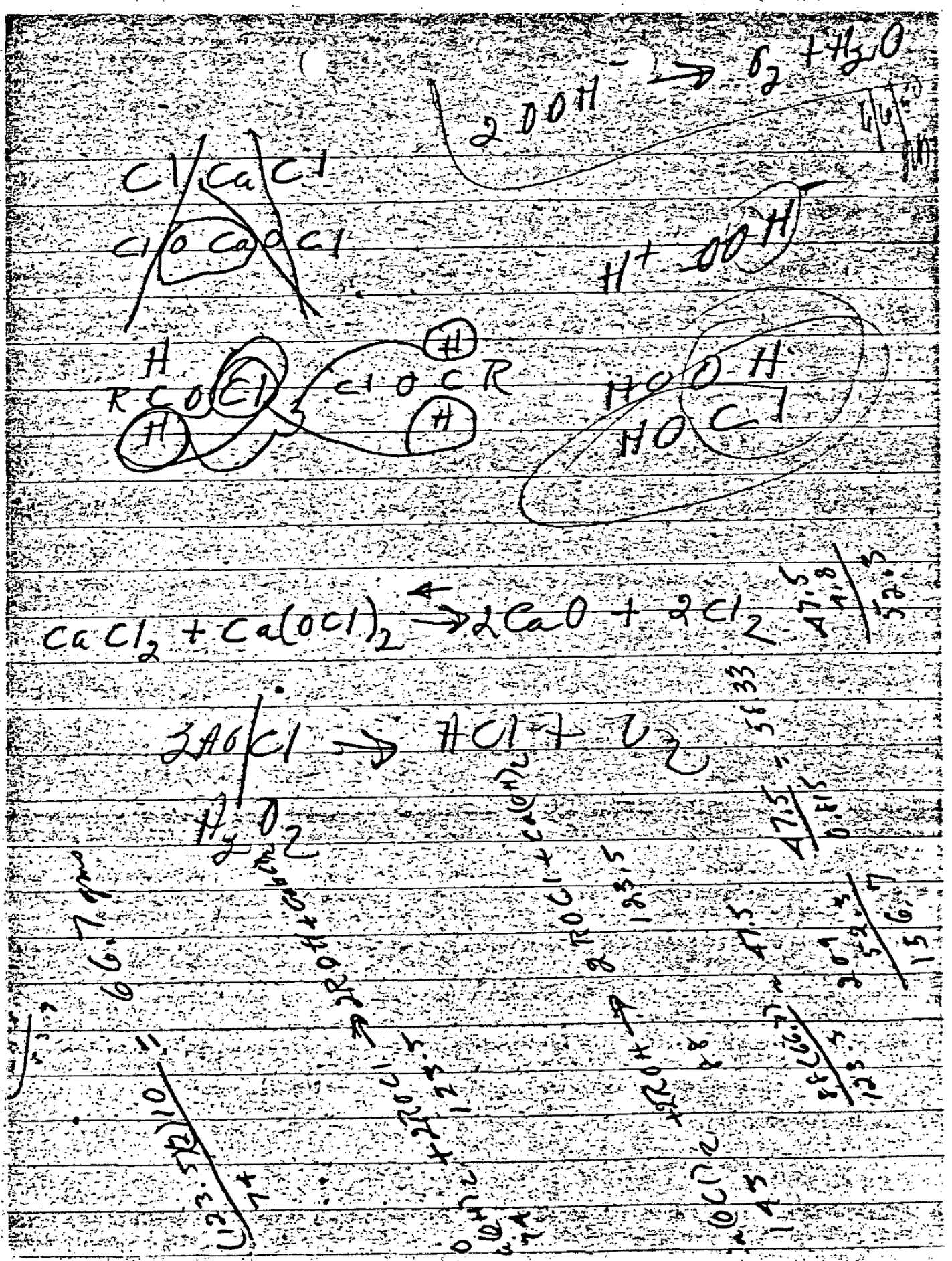
$\frac{200}{1} \times 9.6 \times 1 \text{ N} \times 0.0357$

$= 7.2$

Wet cake = 44 gm
(6.7% Moisture) = 26 gm Dry

4 30			
4 35		17	Filter 1/2 / 1/2

$$\frac{20.1 \times 0.10 \times 0.0533}{0.2040} = 5.730$$



Crude (Sodium salt) 1.63 1.25

Final Soln.

Buffer 0 7.65

74 × 37 = 1916

1443

9.19

559

19

419

alkyl oil

Petn No.	Titer HOH	Titer
1	5.0	3.6

2 alkyl oil used

1	10.6	8.4
2	10.2	15.2
3	7.6	17.6
4	12.5	23.8

$\frac{75}{1000} \times 23.8 \times 0.1019 \times \frac{0.1235}{2} = 22.4$ gms

ROC

$Ca(OH)_2$

$\frac{74}{2 \times 1.35} \times 22.4 = 6.7$ gms. Titer

or 3.5 gms. used

also $\frac{5.4}{1.42} \times 1.5 \times 3.5 = 13.8$ c HOH

will also 0.35 g. Triethylamine used

actually used

3.2 gms. $Ca(OH)_2$

$HOH = \frac{3.2}{3.5} \times 3.8 \times \frac{1.25}{1.5} = 4.0$

Reduction (Calculated in Titer) $\frac{23.8}{18.1}$ actual

$\frac{3.2}{5.7} \times 29.9 = 14.4$

NaOH
ROH + OCl⁻ + Ca⁺⁺
RCl
Ca(OH)₂
HOCl + NaOH
NH₂
NH₃⁺ + OH⁻

adding of 1/2 cup of rice 140

50 Tuesday 16.8 Red 23.8
16.8
7.0

12 Tuesday 16.0

23.8
16.0
7.7

17.1
16.8
10.4

10.4 11.4 .090

11.4 10.2 10.4

23.8
21.7

Titration No.	Titre	Cu+
1	11.8	15.0
2	12.2	27.1

$$\frac{27.1 + 30.1}{2} = 0.1 = \frac{57.2}{2} = 30.1 \text{ cm}^3 \text{ Rochelle}$$

$$\frac{74}{2 \times 12.5} \times 30.1 = 90.0 \text{ cm}^3 \text{ Tartaric Acid}$$

$$\frac{74}{74} \times 4.5 \times 1.1 = 4.9 \text{ cm}^3 \text{ H}_2\text{O}$$

2% TEA = 0.09 cm³ = 2 drops

check

24.9 Tartaric 150 cc.

3.8 cm³ Ca(OH)₂

after reaction with Ca(OH)₂ Titre = 23.3

Titration No. Titre Col.

1 6.7 7.7

2. 9.4 19.0

3 — 24.0

$\frac{175 \times 24 \times 0.1 \times 0.1 \times 3}{2} = 25.0$ gm. $\frac{12}{2}$ Rock

$\frac{74}{2+123.5} \times 26.1 = 7.3$ gm

$\therefore$ 26.1 − 4.3 gm. $Ca(OH)_2$

$\frac{4.3}{7.3} \times 233 = 137.5$ gm Mixed

$\frac{233}{111.0}$ $\frac{110}{127} = 86\%$

$50 \times 31.7 \times 0.1 \times \frac{0.1 \times 3}{4} = 5.7$ gm.

$\frac{143}{74} \times 4.3 = 8.3$

$\frac{5.7}{8.3} = 70\%$

KOSTER KEUNEN
MANUFACTURING CO., INC.

SAYVILLE · NEW YORK

CABLE ADDRESS:
KOSTER KEUNEN, SAYVILLE, N.Y.
A.B.C. CODE 5th EDITION

TELEPHONE
SAYVILLE 400-401

November 14, 1947

6/16/50
DLB

A. Brothman and Associates
2928 41st Avenue
L.I.C. 1, New York

Attention: Mr. Harry Gold, Chief Chemist

Dear Mr. Gold:

Thank you for your letter and inquiry of Nov. 10, 1947.

Under separate cover, we are sending you samples of our different MICRO-CRYSTALLINE WAXES which our chemists feel will fulfill your specifications. On these waxes, we quote you as follows:

HIGH MELTING POINT MICRO-CRYSTALLINE WAX 180/85 m.p.
PENETRATION 10-13
COLOR N.P.A. 3-4

@ 15½ ¢ per lb.
(for quantities not less than 500 lbs

HIGH MELTING POINT MICRO-CRYSTALLINE WAX 190/95 m.p.
PENETRATION 8
COLOR: 3-4

@ 25¢ per lb.
(for quantities not less than 500 lbs)

HIGH MELTING POINT MICRO-CRYSTALLINE WAX 190/95 m.p.
PENETRATION 8
COLOR: WHITE

@ 40¢ per lb.
(for quantities not less than 500 lbs)

6/6/50 JJB

MICRO-CRYSTALLINE WAX 900 YELLOW
MELTING POINT 165/70
PENETRATION 35-40

@ 14¢ per lb.
(for quantites not less than 500 lb

Packing of the above mentioned WAXES is in Slabs or Cartons, F.O.B. Sayville.

Delivery on all of these WAXES is Prompt.

If you have any further questions in regard to our waxes, please do not hesitate to contact us immediately.

Hoping to be of further service to you in the near future, we remain,

Very truly yours,

KOSTER KEUNEN MFG. CO., INC.

F. J. Koster

FJK:ps

cc: GNH

SOCONY-VACUUM OIL COMPANY

INCORPORATED

230 Park Avenue, New York 17, N. Y.

IN REPLY PLEASE REFER TO

A. Brothman & Associates
2928 - 41 Avenue
Long Island City 1, NY

November 24, 1947

Attention: Mr. H. Gold

Dear Mr. Gold:

Your letter of November 10th addressed to our 26 Broadway headquarters has been referred to this office for reply.

We would prefer that you be more specific as to the particular type of Micro-Crystalline or Paraffine wax that you desire. To aid you in this selection we have enclosed several technical publications regarding the application of waxes in the paper industry.

We feel sure that these bulletins will be of interest to you and we look forward to hearing from you further.

As you probably know, the supply situation in regard to Micro-Crystalline and Paraffine waxes is extremely tight and should your interest be in any one of our products, we cannot assure you that we would be able to make deliveries.

If we can be of any further technical service, please do not hesitate to contact this office.

Very truly yours,

SOCONY-VACUUM OIL COMPANY

Robert S. Sheie

Robert S. Sheie
Process Products Engineer

RSS/mf

UNION BAY STATE Chemical Company

SERVING INDUSTRY WITH CREATIVE CHEMISTRY

MANUFACTURERS OF INDUSTRIAL ADHESIVES AND SHOE CEMENTS
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TANK LINING COMPOUNDS - ORGANIC PEROXIDES - RESIN DISPERSIONS

90 HARVARD STREET
CAMBRIDGE 42, MASS.
TROWBRIDGE 6-8076

6/6/50 pp

December 8, 1947

A. Brothman & Associates
2928 - 41 Avenue
Long Island City 1, New York

Attention: Dr. Philip Levine, Ass't. Chief Chemist

Gentlemen:

Thank you very much for your interest in our N-525 Neoprene Paint. In compliance with your recent request we are forwarding to you, under separate cover, a laboratory sample of this material for your evaluation.

We are enclosing, herewith, a technical data sheet outlining the properties of this newly developed compound which is presently priced at $3.00 per gallon when purchased in drum quantities, $3.25 per gallon in 5's and $3.50 in single gallon containers. All shipments are made F. O. B., Cambridge, Massachusetts at our established terms of 1% ten days.

We would very much appreciate hearing from you after you have had the opportunity of evaluating this sample.

Very truly yours

UNION BAY STATE CHEMICAL CO., INC.

B. H. Arthur, Sales Dep't.

BHA:pm
encl.
cc: H. I. Barbey

N-525 NEOPRENE PAINT

6/6/50
ZP

DESCRIPTION: A solution of neoprene in an aromatic solvent with the addition of other materials to yield a chemically resistant paint.

GENERAL USE: As a coating for the protection of surfaces which are subjected to exposure to fats, oils, and greases or corrosive chemical liquids, solids, or fumes.

STANDARDS:

Solids -	36%
Specific Gravity -	0.949
Wgt/Gallon -	7.9#
Color (film) -	Translucent, amber color

MISCIBLE SOLVENTS:

Thinners - Aromatics, Ketones, Esters, Chlorinated Hydrocarbons

Diluents - Aliphatic Hydrocarbons, Mineral Spirits, Turpentine

SPECIAL FEATURES:

Shelf Storage - 2 months

Drying Speed - film loses tack in 20-30 minutes at room temperature (65-75°F) and dries completely in several hours, requiring no oxidation period.

Odor (film) - None

Chemical Resistance (film) - to acids and alkalies, oils, fats, waxes and greases - excellent.

Durability - Excellent resistance to abrasive action; rubber-like film will not chip or crack.

Adhesion - Excellent to wood, metal and other smooth surfaces.

APPLICATION: No special treatment of surface to be painted is necessary. However, the surface should be free from dirt, grease, rust, or other foreign materials prior to coating.

The paint may be flowed on from a full brush, avoiding as much as possible the re-crossing of partially dried painted areas so that a smooth continuous film is obtained. A surface free from gaps, ridges, and pinholes will prevent chemicals from penetrating beneath the paint and causing localized attacks which may spread and lift the film. If large areas are to be coated, adequate ventilation during drying should be provided. Drying at elevated temperatures should be avoided to eliminate air holes in the film.

The paint may be thinned with toluol or other thinners if a spray method of application is desired.

11/21/47

Address all inquiries to the Union Bay State Chemical Company,
50 Harvard Street, Cambridge 42, Massachusetts.

UBS

22 Aug 47

A. BROTHMAN & ASSOCIATES
Chemical and Mechanical Engineers
114 EAST 32nd STREET
NEW YORK 16, N. Y.

6/U/50 GB

Flow point of ground R & H compression molding powder.

13 x 160 t.t.

D. 145" plunger, 373 g.

Time	Temp	Remarks
1120 PM	125°C	
:21	132	Max temp after initial rapid heat-up.
:24	128	
:25	126	
:29	126	
:33	128	
:35	126	Plunger inserted - 3/16" "fall" from position w/o wts. to position w/ wts. Then set to 0 fall & begin measurement of 5/8".
:40	130	
:43	134	1/8" fall
:47	138	1/4" fall
:50	140	
:53	144	3/8" fall
2:00	150	
2:02	152	1/2" fall
09	158	5/8" fall

TELEPHONES:
ELDORADO 5-0775-7

CABLE ADDRESS: PROTRADE
CODE USED: A B C 5TH ED. MEP
BENTLEY'S SECOND PHRASE
BENTLEY'S COMPLETE

DISTRIBUTING AND TRADING COMPANY, INC.
444 MADISON AVENUE
NEW YORK 22, N.Y.

December 4, 1947

6/6/50 JB

A. Brothman Associates
85-03 57th Ave.
Elmhurst, L. I.

Attention: Mr. Harry Gold

Gentlemen:

With reference to our letter of November 14 and our sample shipment consisting of the following material

"DEETEE" American Ozokerite White 150/155

"DEETEE" American Ozokerite White 185/190

may we inquire whether these samples have reached you? We would appreciate hearing from you as to whether these products have your approval.

If any of our other waxes listed in our booklet are of interest to you, please do not hesitate to request samples of such types.

Assuring you of our best service, we are

Yours very truly,

DISTRIBUTING AND TRADING CO., INC.

W. Gantenbein

W.Gantenbein

WG/mls

ATLAS POWDER COMPANY

WILMINGTON 99, DELAWARE

July 10, 1947

6/16/50 AB

Mr. H. Gold
A. Brothman & Associates
85-03 - 57th Avenue
Elmhurst, Long Island

Dear Sir:

Mr. G. J. King of our New York Office has requested us to send you samples of Span 40, Span 60, and Span 65 (Sorbitan Tri-Stearate) and Spans and Tweens booklet.

We are enclosing the Spans and Tweens booklet and will send you promptly under separate cover by Parcel Post 4 ounce samples of Spans 40, 60, and 65, on a sample no charge basis.

We appreciate your interest and trust that if we may be of additional service you will not hesitate to call upon us.

Very truly yours,

ATLAS POWDER COMPANY

W. B. Comegys

Wm. B. Comegys

WBC:mm

Aug 24, 1947

A. BROTHMAN & ASSOCIATES
Chemical and Mechanical Engineers
114 EAST 32nd STREET
NEW YORK 16, N.Y.

6/6/50
90

Flow point of ground Lots 17 & 19:
(N.B. materials were rather brittle on grinding)

13 x 100 t.t., 1½" power height
0.145" plunger, 373 g. weight

Time	Temp	Remarks
11:05 AM	125°C	Begin 15 min const. temp.
11:20	125	Insert plunger & wts. 3/16" fall
:27	129	⅛" fall
:30	135	¼" fall
:37	143	⅜" fall
:44	149	½" fall
:51	157	⅝" fall

A. BROTHMAN & ASSOCIATES

JOB:

SUBJECT:

No. of

Date:

By: 6/6/50 MD

9 19/47 55 g ClCH$_2$COOH in 220 cc. H$_2$O + 0.6 g. CaCO$_3$ + 76.1 g. thiourea, Warmed to 35°, and heat left [illegible] on. Rose to 80° in 4-5 min. Then clear & pale yel. soln. Stayed there 5 min & began to drop. Heated to maintain at 80° 15 min. Added 80 g. NaOH in 120 cc. H$_2$O bringing to 95° without heat. Added at a rate to maintain at 95° without heating. After ca. ½ added no more heat evolved. Alkali ppted cryst mat. Applied heat to maintain at 90-95°. Took ca. ½ hr to add alkali. ½ hr. later 1 cc. titrated 13 cc. 0.1 N sol'n. 1 hr — 13.85 1½ hr. 14.4 cc. 2 hrs. 14.65 Added 2 g. NaOH 2½ hrs. 14.8. 3 hrs. 15.1 Cooled to 40° & added 75 cc. 68.5% H$_2$SO$_4$ by Fair amt. gas evol'n. Temp with cooling rose to 50°. Added 4 g Zn. No obvious fumes. Filtered ppt. H$_2$O used ppte. Then salts ppted. Filtered the salts 580 cc. obtained 5 cc = 54 cc. 0.1 N sol'n

$\frac{54}{5} = 10.8 \times 0.077 \times 454 \times \frac{580}{200} = 58\text{ g} = 62\%$ yield

Aug. 13, 1935. H. BENDER 2,010,841

CHLORINATION

Filed July 21, 1933

FIG. 1.

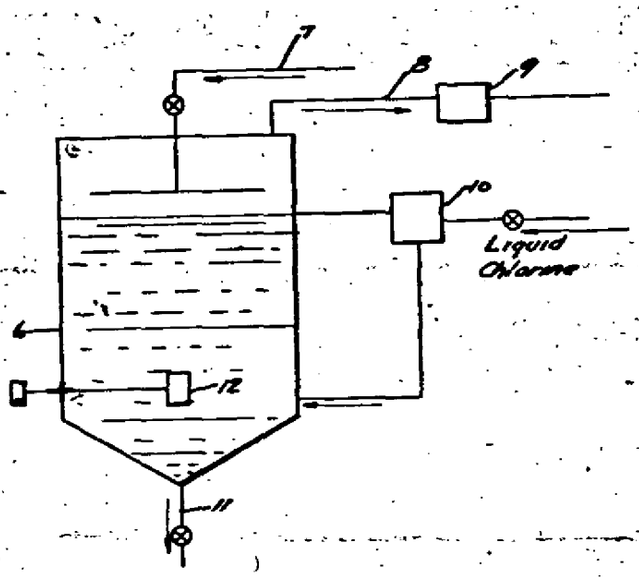
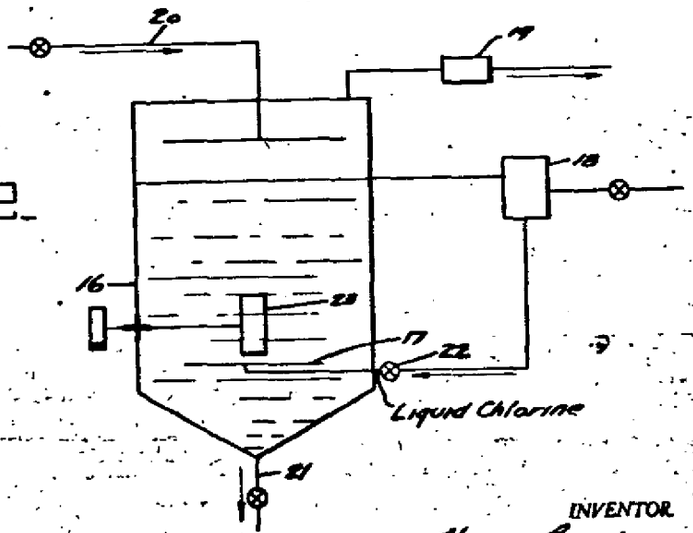
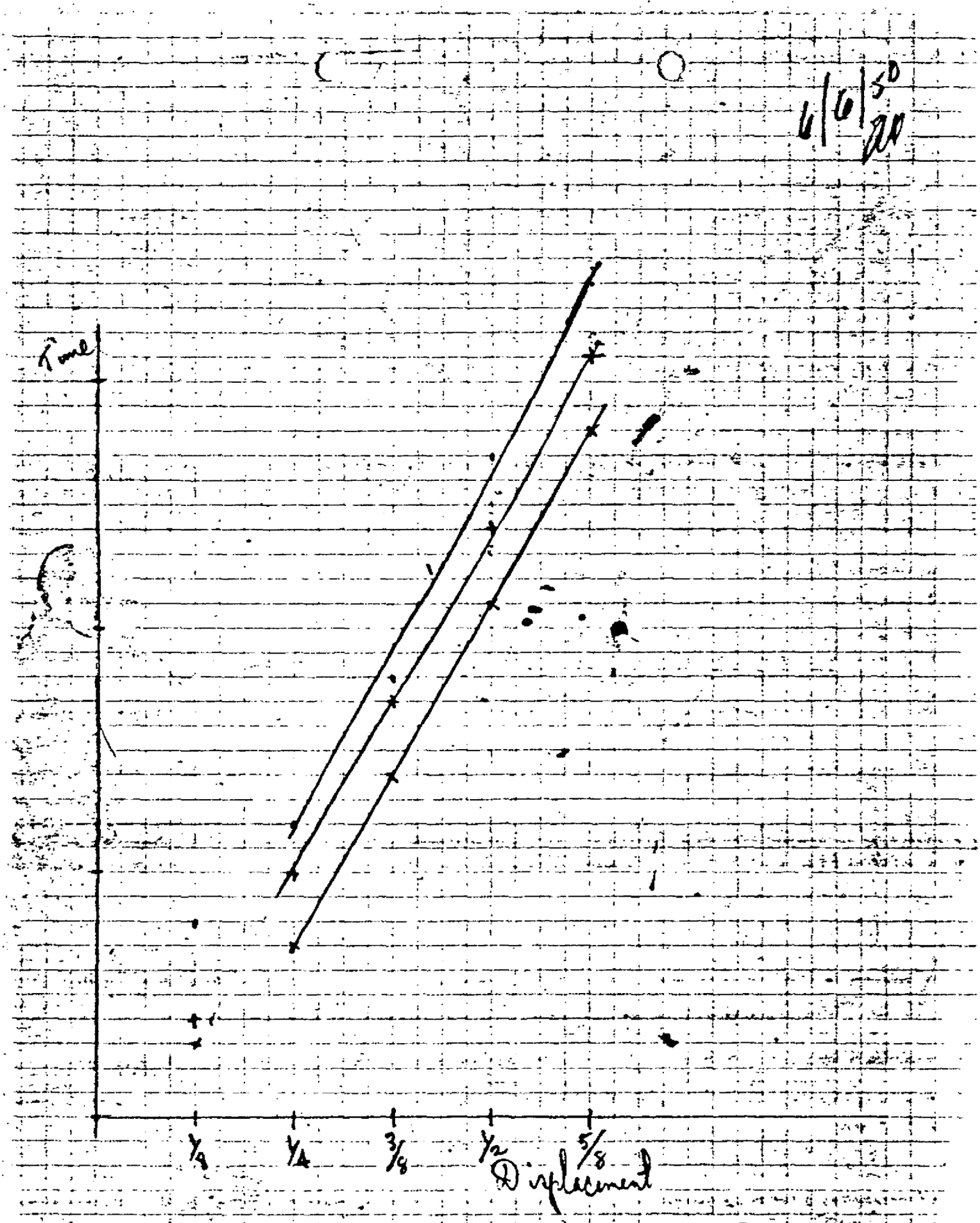


FIG. 2.



INVENTOR
Harry Bender
BY Robert H. Eckhoff
ATTORNEY.

6/6/50



(VION BAY STATE COMPANY 2/6/)

6/4/50 JD

PROPERTIES OF 60% TERTIARY BUTYL HYDROPEROXIDE

Molecular Weight	90
Specific Gravity @ 25°C.	0.859
Boiling Point	82-83°C.
Freezing Point	-30
Flash Point	18.3°C.
Refractive Index @ 25°C.	1.3960
pH in 10% water solution	4
O_2 available	10.6%
Color	Water-White

Stability:

a. Up to 76.6° C — indefinite
b. Above 76.6° C — decomposes at a rate proportional to the temperature.

Activators:

Hydroquinone and other like organic reducing agents have proved to be efficient activators when used in quantities up to 0.1 of TBO_2.

Solubility:

In Water	11%
Water in	6%
Short chain aliphatics	Excellent
Aromatics	Excellent

Price set-up:

$3.00 per lb. up to 279 lbs.
1.50 per lb. 279 lbs. or more

BURBAU OF EXPLOSIVES REPORT FROM CHEMICAL LABORATORY

"60% Tertiary Butyl Hydroperoxide"

The material is a water white liquid with a Specific Gravity of 0.860 at 15° C. It has a sharp penetrating odor that would serve as a warning in case of leaking packages.

The liquid is stable through prolonged heating at 75° C and did not decompose violently when heated up to 300°C. No noticeable pressure developed in a tightly closed bottle after standing five days at laboratory temperature.

The material is readily inflammable when ignited. The combustion of fine organic material saturated with the liquid is accelerated somewhat but not dangerously so. The material fails to explode when detonated by a blasting cap.

The flash point was determined as 62° F. This material is classed as an Inflammable Liquid and is considered sufficiently safe for transportation.

A. BROTHMAN & ASSOCIATES

JOB: matter

SUBJECT: H.T.H.

No. 7 of

Date: 11-17-47

By: H.H.

removed by washing with $NaHCO_3$

i.e. The ester decomposes on standing to give best yields of EtOAc

$$EtOCl \longrightarrow MeCHO + HCl$$

$$EtOCl + MeCHO \longrightarrow MeCO_2Et + HCl.$$

orig. article J.A.C.S. 47, p. 395-403

M. Taylor, R. MacMullen & C. Hammond

1. T, MacM & H are from Mathieson alkali
2. Part of research program to produce pure $Ca(OCl)_2$
3. Found that while EtOCl is too unstable for use in a technical process when it is pure, a soln. of it in CCl_4 (or any other inert solvent which is immiscible with H_2O) is stable for several hrs at 25° C.
4. Simple method - as above, but can mix 2% or more EtOH soln in CCl_4 and a soln loses only 1.2% of its available Cl in 2 days at 20° C in the diffuse light of the lab.
5. as above. EtOCl → EtOAc.

6. Ethyl hypochlorite reacts with alkali to

chlorides. Practically pure $Ca(OCl)_2$ can be prepared if such a water soln. be evaporated to dryness under a vacuum.

7. Another way - U.S. Patents { 1,491,039 / 1,491,041

a. add dry lime mixed with CCl_4 containing an excess of EtOH

b. a quantity of H_2O sufficient to form the trihydrate of $Ca(OCl)_2$ is introduced slowly with violent agitation. This water amount necessary to get good conversion, although too much water wets the lime particles and hinders conversion.

c. The alcohol regenerated remains in the CCl_4 which soln. is used over again to extract more HOCl from chlorinated carbonate soln.

a. process produces $Ca(OCl)_2$ containing 75% to 90% available Cl.

8. The suggested use of tert-amyl alcohol if a hypochlorite of greater stability is desired.

A. BROCHMAN & ASSOCIATES

JOB: water

SUBJECT: 1+T+H

No. 9 of

Date: 11-17-17

By: A.J.M.

9. Tate showed EtOH to be a true alcohol (by molecular data → that equal to EtOH not EtOH · HCl)

8. Also formed following ether hydrochlorides

n-propyl

isopropyl

n-butyl — all yellow unstable oils

sec-butyl

tert-butyl

iso amyl

sec amyl

tert amyl

9. Distribution coefficients - ether for alcohol

temp 25°C
total Cl = 0.3M

alc	water	ether
MeOH	3.1	17.7
EtOH	5.2	24.1
PrOH	6.1	21.7
iso-PrOH	4.5	23.5
iso amyl	7.3	20.7
sec butyl	1.5	22.1
tert butyl	1.6	22.5
sec amyl	3.7	21.4
tert amyl	3.4	22.9

A. BROTHMAN & ASSOCIATES 3/6/50 CD

No. 1 of
Date: 11-17-47
By: H.H.

JOB: Meetings

SUBJECT: H T H

10. HOCl is available in CCl_4.

11. T. etc studied hydrolysis of EtOCl.

a. For conc. of OCl < 0.03 mole/liter, hydrolysis is over 90% complete

b. Even a saturated soln of EtOCl in water containing 0.464 mole of OCl per liter is 69% hydrolyzed.

c. Effect of temp. on hydrolysis is not pronounced.

---

note 1 - In $CaCO_3 - Cl_2$ technique, the available Cl is present almost entirely as HOCl.

note 2 - Research program was to

a. Find a compound of HOCl whose physical properties would allow it to be separated from the HCl soln of the chloride formed by hydrolysis of the Cl_2, and

b. Whose chemical properties would permit reconversion to HOCl after separation.

A. BROTHMAN & ASSOCIATES

JOB: Matter

SUBJECT: C H T H

No. 6 of

Date: 11-17-47

By: H.A.

Lipid Information

EtOCl

Ref Biol. I, 304 (nothing)

Interchloridine Fauré = HOCl

Biol E I, 164 (nothing)

Biol E II, 305

1. To all pure EtOCl

a. Pass Cl₂ into wtd CaCl₂

b. shake with EtOH in CCl₄

orig Ref: J.A.C.S. 47, 395

Taylor, MacMullen, Summerl

2. EtOCl is stable in CCl₄ Rec 58, 572

3. T., MacM & Sa also find distribution coefficient bet HOH and CCl₄.

Ref Thorpe Vol 4 p 363-364. Dimethyl-Nitro-

1. Ber Bull. Soc. chim Belg IV 37, 717.

for improved apparatus

2. For T, MacM and H's method

a. Suspend 25 gms. CaCO₃ in 1 liter HOH

b. Pass in Cl₂ till 2.5 gms. are absorbed

c. Filter excess CaCl₂.

d. shake filtrate with 2% soln of EtOH in CCl₄. The EtOCl

A. BROCHMAN & ASSOCIATES

JOB: methods

SUBJECT: H.T.H.

No. 1 of

Date: 11-22-43

By: H.A.B.

orig. article U.S. Patent 1,493,039 May 6, 1924

T., Mac & Applicants
(methanol alkali)

1) eliminate suspension of $CaCO_3$ in HOH
so that the resulting solution contains
10-20 gms available chlorine per liter

2) Treat with equal vol of $Ca(OH)_2$ containing 2%
EtOH — use 0-5°C

3) Treated with $Ca(OH)_2$ containing ~1-3%
free HOH

Use ½ the amt of $HOCl$ required to
react completely with amt of lime used
add HOH amounting to 1½ - 2 times
the theo. amt of HOH → $Ca(OCl)_2 \cdot 3HOH$

→ 47% available Cl

{ 0.2% $Ca(OH)_2$
~0.7% HOH
~0.7% $Ca(OCl)_2$

4) Treat with HOH to dissolve only $Ca(OCl)_2$
& can recover by these following → 90-95% $Ca(OCl)_2$
4 gms; 18 ml; 0.2 gm

Transfer from settled ppt as
quickly as possible.

A. BROTHMAN & ASSOCIATES 6/6/50 PM

No. 13 of

Date:

By:

JOB: water

SUBJECT:

range tested being latitudes in terms of Thorne effecting products of production

---

C & D.

part work Hypothesis

71°/75 Variance.

$d \frac{v s^2}{40} = 0.3547$

Rule with dissenting.

---

part Run 0 cl

very stable Results of 79.6°/75°.

$d \frac{n!}{40}$

Use ps d test run conditions than

— cycles (ans).

(dyeing, Chemisizing, Performance

A. BROTHMAN & ASSOCIATES 4/6/50 RD

No. 1 of

Date:

By:

JOB: methods

SUBJECT: H+H:

orig patent 1948, 1449 Jan 5, 1949

1) eliminate most CaCO3 ⇒ 2-3 g available chlorine / liter

2) subject to conc to remove free Cl2 & most of CO2 (this reduces CaCl2 + CaCO3 content of finish product).

3) agitate slurry of this conc with limit of CO2 entry (3) & H+H.

⟶ leave a retard entry 5 grs available chlorine / liter

4) treat with hydrated lime entry 1-1½ lbs H+H - add an excess of 3+4 lbs - available chlorine such that required to react theoretically with all the lime

5) add H+H in small amts (very slowly & with thorough agit) so that total free HOH is 1½ - 2 times the theoretical HOH req to form Ca(OCl)2·2H2O ⟶ powdery product.

6) Filter ⟶ 50-70 Ca(OCl)2
42-30 HOH
1-2% CaCO3
1-2% CaCl2
balance Ca(OH)2

6/6/50
JHP

XR-3180 and XR-4357

The unmodified resins, XR-3180 and XR-4357, are tough, pale colored and have exceptional resistance to moisture, alkalis, acids, oils and greases. They impart high gloss, easy polishing characteristics, durability and freedom from water spotting to nitro-cellulose and ethyl cellulose lacquers. The flexibility and resistance properties of the XR-4357 have made it excellent for use in adhesives and plasticizers.

XR-3180

A permanently fusible resin having high alkali, acid, water and grease resistance. It gives high gloss, remarkable depth of luster and fullness, fair color, and extreme resistance to perspiration. It has a very low acid value. It requires combination with nitro-cellulose or/and ethyl cellulose for solution stability. It will not cook with oil.

It is used in both interior and exterior lacquers, lacquer coatings for paper and cloth, hardware lacquers for perspiration resistance, and auto-refinishing enamels and clears. Color retention is fair.

Typical uses are for product finishing lacquers and for exterior lacquers, clear and pigmented. Advantages:—high gloss, gloss retention on exposure (less chalking), good adhesion and non-tarnishing in clears over metal, resistance to moisture and water spotting in auto enamels, high durability, resistance to oils and butter fat in refrigerator lacquer tests, non-yellowing. An important property is its ease of polishing to a high gloss.

Clear lacquers, based on the following proportions, have given good durability in exposures.

NITROCELLULOSE	RESIN	PLASTICIZER (dibutyl phthalate)
1	1	0.5
1	1.7	0.3
1	2	0.1

The XR-3180 is in itself fairly flexible and requires less plasticizer as larger proportions of resin to cotton are used. The lacquers become slower drying and softer as the proportion of resin is increased, 1 1/2 parts of resin to 1 of nitrocellulose approaches the limit for general hardness.

Properties:

Color:		
Resin	—	2-4L(3:1 xylol)
Film	—	Very good to fair.
Specific Gravity	—	1.30
Melting Point (Ball & Ring)	—	125-150°F.
% Non-volatile	—	100%
Acid Number	—	1-3

6/4/50

XR-4357

XR-4357 is slightly softer and darker in color than XR-3180. It has excellent adhesive qualities and is good for plasticizing other solutions. In addition, it is used as an ingredient in adhesives and as a plasticizer for certain of the dispersion resin coatings.

Properties:

Acid No.	—	Not over 1.5
Color	—	3-9
Oil solubility	—	Poor
Melting Point	—	Approximately 117-121°F.
Specific Gravity	—	1.24
Weight per Gallon	—	10.3 lbs.

Stability of Solution

We do not offer a solution of XR-3180 for sale.

Cold cut solutions are less stable than those made by heating. Cold cut solution in cotton solutions or solvents that are cloudy when made, are certain to be unsatisfactory. In any precipitated solutions, the original condition of the solution made by heating is restored unchanged by warming up the separated mix and stirring at 140° F. to bring about re-solution.

Settling of an XR-3180 solution should be viewed as crystallization and may be prevented by the use of more powerful resin solvents, by proper dilution in these solvents or into a finished lacquer promptly after making up the solution, or by the use of temperatures high enough to prevent its starting. With slight alterations in final properties, XR-4357 designed for stability of solution, may be used.

Regarding the order in which solvents rank as to power and keeping of solutions, there is no numerical rating possible. However, the following list of solvents and other lacquer materials are given in the order of decreasing solvent power of toleration.

Solvents	Non-Volatile
Acetone	Tricresyl Phosphate
Ethyl Acetate	Dibutyl Phthalate
Toluol	Nitrocellulose
Butyl Acetate	Phenolic Resins (Bakelite)
Xylol	Natural & Modified Resins
Cellosolve	Oils
Alcohol	
Petroleum Thinners	

The most unexpected situation in this listing is perhaps that alcohols are such poor solvents for XR-3180. Petroleum thinners very markedly, some, such as the Solvessos, having about as good toleration as the alcohols. The usual mineral spirits, however, are completely immiscible.

Lacquers or resin solutions in which the combination contains the tolerable limit of oils, alcohols, petroleums, etc., are the most troublesome for precipitation. We have had no instances in which lacquers, made up to 25-35% solids with acetate and benzine series of thinners, have separated on two to three years storage, even when the solids were largely XR-3180. Equal parts of 3180 and toluol will precipitate in 10 to 30 days. A solution of two parts of toluol to one part of resin will normally last from several weeks to several months, and one of equal parts of toluol, ethyl acetate and resin will last somewhat longer.

Note: 50% solutions of XR-3180 in the stronger solvents must contain a little nitrocellulose, ethyl cellulose, alkyd, vinyl or other resin to stay in solution for definite periods of time. Solutions that are capped and not continually being disturbed seem to be most stable.

Discoloration of XR-3180 in Lacquer

While XR-3180 resin alone has little or no tendency to discolor on exposure to light, when used with nitrocellulose, the combination yellows more than either one alone. This discoloration depends on the intensity of the light; is greatest under the U.V.Arc, less on direct exposure to sunlight and still less in diffused light.

Very satisfactory white lacquers have been produced for indoor use as regards color or discoloration.

Any judgment of color change should be made on the basis of equal gloss or gloss retention. XR-3180 will permit higher pigmentation for equal gloss as compared with Damar. The gloss retention of XR-3180 is also excellent. While XR-4357 holds gloss still better than 3180, the XR-3180 will collect less dirt on outdoor exposure.

Plastic Checking

XR-3180 has a low melting point and when used excessively in a lacquer, will lead to plastic checking on exposure. This tendency may be reduced by using a moderate amount of XR-3180 (50-65% of solids on the average), and plasticizing to suit the conditions. Any modification with hardening or stiffening agents, such as hard or rubbery resins, pigments, especially of the fibrous type, or higher proportions of cellulose ester, should be effective in reducing plastic checking.

General

From a durability angle, XR-3180 does not seem to bolster up ester gum. Small percentages of XR-3180 give very little or no increase in life of the film. This may be due to poor compatibility and would apply equally to Damar.

The common lacquer resins are barely tolerated by XR-3180. The alkyds, damar, ester gum or rosin containing resins generally give hazy solutions at best with XR-3180. XR-3180 is probably the most compatible with cellulose of the above resins, and in some ways, almost resembles a solvent in its action.

Resins that readily combine with XR-3180 are less compatible with nitrocellulose (vinyls, styrols, etc.). Excellent compatibility may not be necessary for mixing to give the desired results.

Also, plasticizers which are not compatible with XR-3180 may not rule out their use (castor oil being an exception). Tricresyl phosphate is a solvent for 3180 and dibutyl phthalate makes an excellent plasticizer.

The following formula suggestions may be of interest to you:

LR-8285
LR-9859
LR-9470
XE-7472

BR-302:

This is viscous oil-modified resin which has a fairly deep yellow color and a phenolic odor. It gives low viscosity in lacquers and may, therefore, be used with larger proportions of higher viscosity nitrocellulose than usual. Gives clear and pigmented lacquers of unusual durability, body and water resistance. Due to color used only where yellowish Clear is acceptable or in dark and solid colors. Despite color and odor its high integrity in lacquers still continues its usefulness in specialties.

Properties

Color	—	4L-6 (3:1 xylol)
Specific Gravity	—	1.02
Viscosity	—	200-360 cp (3:1 xylol)
Solids	—	100%
Keeping Time	—	1 year
Compatible with	—	Nitrocellulose and bitumens

BJ-16580:

This is a non-oxidizing, heat-reactive C-9 ("Carbic" anhydride) type resin which will cold blend with nitrocellulose, ethyl cellulose, chlorinated

6/4/50
200

rubber and urea resins to give non-yellowing films having a high order of flexibility, adhesion and solvent resistance. BJ-16500 acts as a resin plasticizer for both lacquer and urea yielding a tough film with excellent adhesion along with alcohol resistance and durability. Excellent for paper and cloth coatings of all types.

Properties:

Acid No.	—	21-34
Baking time (alone)	—	30 mins. @ 250°F.
Color	—	Not darker than Z
Solid content	—	80%
Specific Gravity	—	1.05 (as is)
Viscosity	—	200-450 cp. (3:1 xylol)
Keeping Time	—	6 months or longer
Solvents	—	xylol
Thinners	—	xylol-Solvesso, ethyl acetate
Wt. per Gallon	—	8.3 lbs.

The synthesis of 2,5-dimethyl-4,5-dihydro-2-furoic

62 gms of 90% KOH are slurried in
150 gms of amyl alcohol. 6/6/50 gm

) 14.3 gms of $CaCl_2$ are added and the
mixture is distilled at 95°C under
constant agitation.

) The distillate in chilled to 0°C to
give a virtually quantitative separation
of the amyl alcohol as the upper layer
by means of a continuous chilling
and decanting device the amyl
alcohol is continuously returned to
the still pot.

) The distillation is continued till no
more water comes over with the amyl
alcohol.

5) The system is then set up for reflux
and the reaction continued in this
manner at 132°C for 1 hour.

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

amyl alcohol — the distillate contains 82.8 mole % of water. The distillate is chilled to 40°C in the decanting device and the upper layer of amyl alcohol containing no water is returned to the still pot. The rate 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 corresponding to a 9.0% KOH in the still pot

THE PREPARATION OF UREA FORMALDEHYDE COLD-SETTING GLUE

8/21/45

6/6/50 MP

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

To a 1 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 622 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 Hg. 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 2 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.

Fish glue

Source -

1) The waste products of the fish industry and fish glue which is the most important liquid glue.

2) This raw material are the skins, (especially that of cod and plaice) the bladders of various fish, and, chiefly, all varieties of fish offal.

3) The quality of the glue varies from ground fish such as cod, haddock, hake, etc. is better and the yield is greater than in the case of glue made from the other fish, e.g., herring.

Ref. Thorpe, Vol. VI, p. 24

Source

A. The bulk of the fish glue manufactured today is made from the waste products of the cod, hake, and haddock industries.

fish are also called round fish which are caught on the banks, usually together in the sand nets, and are classed on the round when consequently most of the fish that stink come to the glue factory are mixed; that is to say, the waste fish the various species of fish that have been dumped into the nets containing

5. some other species of fish other than that mentioned above are used in the manufacture of glue - indeed, any fish might be used for the making of glue - but for various practical and economic reasons only small quantities of glue are manufactured from other fish. The quality of the glue produced from the round fish is higher and the yield is greater than in the case of glue made from most other fish. Many species of fish, e.g., mackerel, which are the

...ut ... economically practicable t...
...ue ... for the manufacture of...
glue ... fish such as the hake...
and mackerel contain such large...
quantities of fat that special pro...
cedures must be followed to re...
move the fat from the fish in the...
glue making process.

C. Many fish which would otherwise be used are not caught locally in any one locality and consequently the supply of fish waste at any particular point is not large enough to justify the establishment of a glue factory. Other fish are caught only for short seasons which would cause the glue factories to be idle most of the year.

D. The ground fish waste is ordinarily divided into three classes:

(1) Fish heads

(2) Waste (viz.) and fish trimmings from fillets and boneless

(3) Skins from dressed salted fish

The fish and pieces are
hauled from the vessels into the
ground fish are cleaned with
the exception of the throats and sounds
fish, most of the dried salt fish
is skinned before it is packed for
shipping. The cod and cusk
skins are not mixed with the
skins of the haddock, hake and
pollock. The cod and cusk
skins which have a small
amount of salt fish adhering
to them constitute the skin-
glue stock. Most of the sound
fish and
cut into strips, trimmed of
the outer yellow portion and
pulled from bones. The trimmings
the bones, and the haddock,
hake and pollock skins constitute the salt-fish waste glue-stock
and is termed waste.

E. The fish skin glue and fish

(4)

Ref. The Chemistry and Technology of Adhesives and Glues by R. H. Bogue

P. 251-266.

The difference between a number of glutens derived from fish and from mammals are very slight.

Gelatin as such does not occur in the heads, bones, tails and other waste material from fish - but is produced as a result of the hydrolysis of certain proteins principally collagen, present in this waste.

Other proteins (then collagen), fats and pigments are the principal detriments to the production of a high quality glue from fish waste.

The most suitable raw material is the shins of fish obtained from filleting and curing factories.

Mixed offal contains all kinds of materials (such as fat, muscular tissue, etc.) and there is a much greater need for the introduction of impurities into the glue than in the case of shins, which are far more homogeneous in character

(2)

2. containing the following kinds of bacteria
K & S which they are known to obtain
a firm jelly from which they form
heads
of pot meat extracts
a- Heads and tails of cod, ling,
haddock etc.
d- Shoots of pot

3. K & S use in their nurses
a- a 24 wash (changing with water
3 times) with 0.2% NaOH
b- Followed by a 24 wash with
0.2% H_2SO_4 (changing wash
with 3 times)
c- Followed by a 24 wash with
H_2O.

Using this method they claim that a very good odourless glue can be obtained

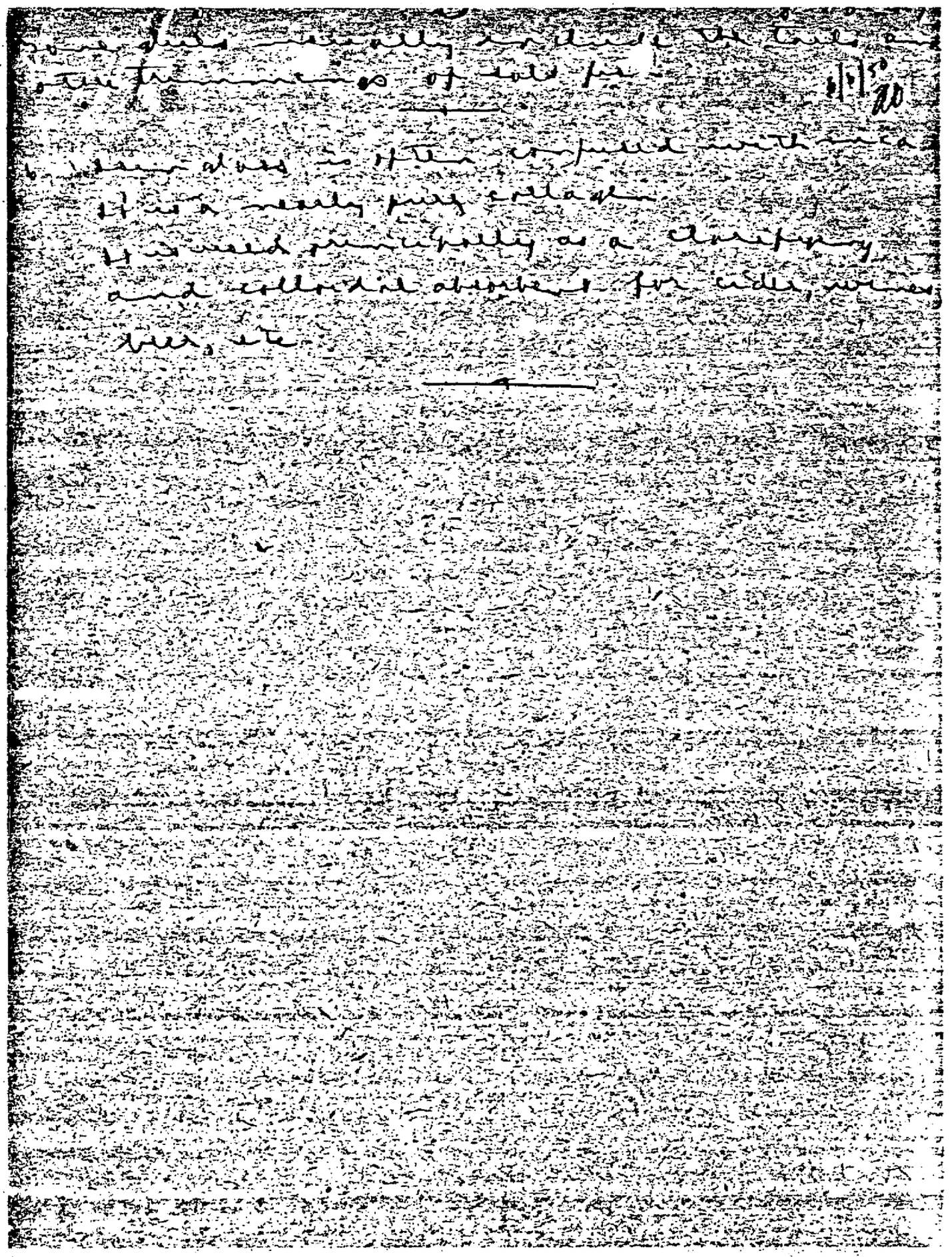
Ref Food Investigation Board, Report of Research Committee 1922-1923 Department, (Second Report 1924 p. 23-33)

material is gelatin contains the water-
soluble decomposition products (proteins)
such as albumoses, peptones and amino
acids. The more the albumoses the better
glue it yields; it is that a good glue
should be as free as possible from
other proteins from hydrolytic
splitting products and from non-
protein matter such as fats (i.e. heads,
skins, bones and meat scraps) which
contain little or no gelatin and should not
be used to make glue. But the presence of
some considerable quantity of fat
is fatal to the production of a
good glue.

Ref: White Dept. of Commerce
Bur. of Fisheries
Document No. 1052

also)

11. 1 ton of Hake → 50 lbs. of Granules
→ Liquors. (400 lbs.)



#3

6/6/52

Date Filed (Name of Contributor)

By Phillips Petroleum Company
(Name of Owner or Agent)

To Be Filed With Yes ( ) No (✓)

Description Some pages of miscellaneous papers found in
Woodlawn Bros. Inc. Account of Mobil Oil Co. Lease

File No. 65-4301-16-12(3)

Dupd 8 MM

65-4301-16-12(3)

#2

Date Received 6/4/50

From [illegible]
(Name of Individual or Organization)

By [illegible]

To [illegible]

Description [illegible]

Yes (1)
No (2)

File No. GS-4307-I-B-12(4)

Subject: Drilling #1 [illegible] natural [illegible]
[illegible] Kaibab [illegible]

6/15/50

GS-4307-I-B-12(4) #1

Received 11/6/80

(Name of Contributor)

To Be Returned

Description of Item(s) Submitted

File No. 65-4307-1B-1240

#3

65-4307-1B-1240

SAC, PHILADELPHIA

July 7, 1950

T. SCOTT MILLER, SA

HARRY GOLD
ESP - R

65-4307-1B 12 (4) Folder No. 3

On June 24, 1950 GOLD advised that the one sheet of paper in this folder was a sample of a proposed data sheet which GOLD prepared in connection with his work at Pennsylvania Sugar. GOLD stated that this sheet was to be printed up and used in Laboratory work at the Pennsylvania Sugar Laboratory.

TSM:EMC
65-4307

Fermenter no. Date (when?)

Aerating Data

Time			Temperatures, °C				Air			Analytical Data	
Start	Finish	Hrs.	Flame	Wall	Jacket	Exhaust	Pressure	Flow	Hrs.	A.V.	pH

Fermentation Data

Time			Analytical Data (Beginning of Ferm.)						
Start	Finish	Hrs.	P.V.	pH	% Solids	% Ash	% Total Nitrogen	% Total Starch	Beginning Temp.

Fermenter Emptying

Total Hrs. Ferm. + Aeration	Time Start 190 °C	Date Finish from Start	Lbs. Carbon Dioxide recovered from Fermenter-Boiler Complex	Emptying		
				Date	Time Finish	T

Date Received 1/16/50

By (Name of Individual, Firm or Corporation)

To Be Published (Yes or No)

Description

File No. 65-4307-18-12(4)

65-4307-18-12(4) #4

SAC, PHILADELPHIA

July 7, 1950

T, SCOTT MILLER, SA

HARRY GOLD
ESP - R

65-4307-1B 12 (4) Folder 4

On June 24, 1950 GOLD advised that the handwriting on the two sheets of paper in this folder was his own and that it was concerned with library work on January 30, 1943 which he did in connection with vitamin work for the Pennsylvania Sugar Company.

TSM:ERC
65-4307

Library Work

J. B.C. 147, 1 (Jan. 1943)

ad p.2 resp. of d-pantothenic acid

ad p.3 dl. aspartic acid (much)

in fact wholesale of pure amino acids for research.

ad p.4 gas driven stirrer 9224

9224-A

J. B.C. 147, 1. p. 185 - 187

a growth stimulant for Lactobacillus casei

by Maxwell a Pollack & Manfred Lindner (U. of Texas)

is present in natural extracts of a substance of unknown nature which stimulates the early growth of L. casei is demonstrated.

The properties of the growth factor are given.

Williams Turbidimeter was used for the tests

J. Biol. Chem. 83, 515 (1929)

Archives of Biochemistry 1 (Dec. 1942)

ad for book

Chemistry & Methods
of
Enzymes
by
James B. Sumner
&
G. Fred Somers

320 pp.
Ready in March '43.

) Looks like a very good (& the first) elementary book for ~~me~~ E & N HG.

---

chem & met. Jan. '43

11.13.7 chem. Eng'g bookshelf.

a Without Fame by Otto Loewi.

2. Textbook of Biochemistry
by
Roger J. Williams

733 pp.

D. Van Nostrand Co.

Fine text for a reader not too familiar with biochemistry

also a glossary of physiological & medical terms.

Fisher's "The Laboratory"
Vol 13, #5 Ind article on Chemistry in War.

Robert E.

Dept. of Justice
New York 7, N.Y.

7A

Date Received 6/16/65

From U.S. Courthouse

To Be Returned

(Name of Bldg.)

(Address)

(City)

Description

Re: Theodore Shapiro

File No. 65-4302 SAB-12(4) Subject: Theodore Shapiro

65-4302-1-B-12(4)#6

#13

Received [illegible]

(Name of Contractor)

By [illegible]

To Be Filled In By Contractor

Date Work Completed 2/15/52

Permit No. [illegible]

Under Authority of Contract Dated [illegible]

For Improvement of [illegible]

File No. ES-4207-17-8.12(4)

#7 (NJC-81-1014) 65-4307-1B-1514

Sent to NY
7/15/50

#10

File No. 65-4307-1B-12(4)

Date Received

(Name of Contributor)

(Name of Special Agent)

Description

65-4307-1B-12(4)

Name of Contributor

Address

By

To Be Filled In By Treasurer's Office

Date Received

Receipt No.

File No.

65-4307-1-B-12(4)

SAC, PHILADELPHIA

July 7, 1950

T. SCOTT MILLER, SA

HARRY GOLD
ESP - R

65-4307-1B 12 (4) Folder No. 9

On June 24, 1950 GOLD advised that the material in this folder was in his handwriting and consisted of laboratory notes on his work on CO 2 recovery at the Pennsylvania Sugar Company.

65-4307-1B 12 (4) Folder No. 10

On the same date GOLD identified the above folder as containing material on vitamin assays in connection with his work at Pennsylvania Sugar. GOLD said that this material is in the handwriting of himself and MORRELL E. DOUGHERTY.

65-4307
TSM:EMC

Folder #9 (WH)
6/6/50 AP

BLANK Sheets

RUN #1

Data Point No.	Time	Gas Rate, c.f.m.	Speed, r.p.m.	Gas Analysis, % CO_2			Temp, °C	Titration, ml. of N H_2SO_4			
				Inlet	Exit (Orsat)	Corrected Exit		Solution		Total	
								o.c.	M.O.	o.c.	M.O.
1	10:10	1.20 1.50 1.50	2380	9.8 10.4	6.9 7.9		47	25.5	51.0		
2	11:00	1.20 1.40 1.40	2410	10.4 10.2 10.2	7.8 8.0 7.9		48	24.1	51.9		
3	11:50	0.70 0.70 0.80	2320	10.4 10.0 9.4	4.6 4.0 2.60		49	23.2	51.0		
4	12:40	"	"	"	"	"	"	21.2	51.6	"	"
									/		

Notes

1 - E - Paint badly eaten, giving oily film

2 - Read from on top

Lewis INL 1111

Received 6/15/58

Name of Contributor

Address of Contributor

To Be Returned [illegible]

Description [illegible]

File No. [illegible]

SAC, PHILADELPHIA July 7, 1950

T. SCOTT MILLER, SA

HARRY GOLD
ESP - R

65-4307-1B 12 (4) Folder No. 9

On June 24, 1950 GOLD advised that the material in this folder was in his handwriting and consisted of laboratory notes on his work on CO 2 recovery at the Pennsylvania Sugar Company.

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65-4307
TSM:EMC

Riboflavin Assays

1. Snell-Strong

Presented 10:45-am
12-1-40

Re: Clay-Adams Co's Centrifuge.

After studying Catalogue, we recommend getting

Model CT-1010 - Page 5. (checked)

Reasons:

(1) This machine, as a unit, is designed for Bacteriological work, our primary use of it.

(2) The head covers, including tubes are inter-changeable, so that if need arises this machine could be changed into a general Laboratory unit at a very low cost - ($16.50) so that for a total expenditure of $62.50 we have two complete machines.

III Test tubes on this machine can be handled direct from incubation and no transferring of cultures is necessary; centrifuging done with cotton in tubes.

— The only point which is in doubt is the speed of the machine (CT 1010); the catalogue does not give it and we need at least 3000 R P M. Could you telegraph them? also, how quickly can delivery be made?

Dougherty
Sold

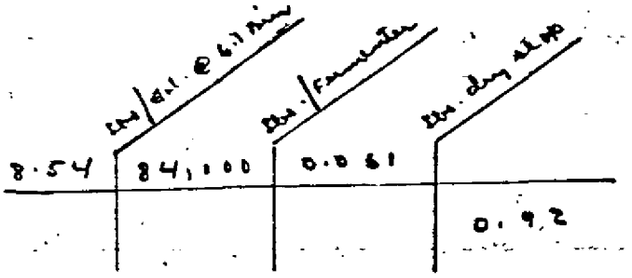
1	0.00	0.80	- 0.2	0.60
2	0.80	1.61	- 0.1	0.71
3	1.61	2.42	- 0.2	1.71
4	2.42	3.41	- 0.05	3.84
5	3.41	14.01		10.60
6	14.01	18.99	- 0.05	4.93
7	18.99	20.52		1.53
8	20.52	29.18		5.33
9	29.18	36.61		6.73
10	36.61	43.00		6.79
11	0.00	7.74		7.65
12	7.74	15.53		7.29
13	15.53	20.54		7.81
14	20.54	30.57		10.03

A-1	5.10	7.77	2.67
A-2	7.77	10.71	2.94
A-3	10.71	15.58	4.87
A-4	15.58	16.53	3.81
A-5	16.53	19.95	3.42
A-6	19.95	22.83	
A-7	22.83	25.70	
A-8	25.70	28.80	
A-9	28.80	30.57	1.77

2/6/41

Quantitative Distribution of Ribo-flavin between yeast and beer

Lbs. slop (8% H_2O) / Fermenter



= 47,600 Lbs. slop (8% H_2O)

Lbs. yeast (8% H_2O) / Fermenter

= 2000 {estimated}

and, at End of Fermentation

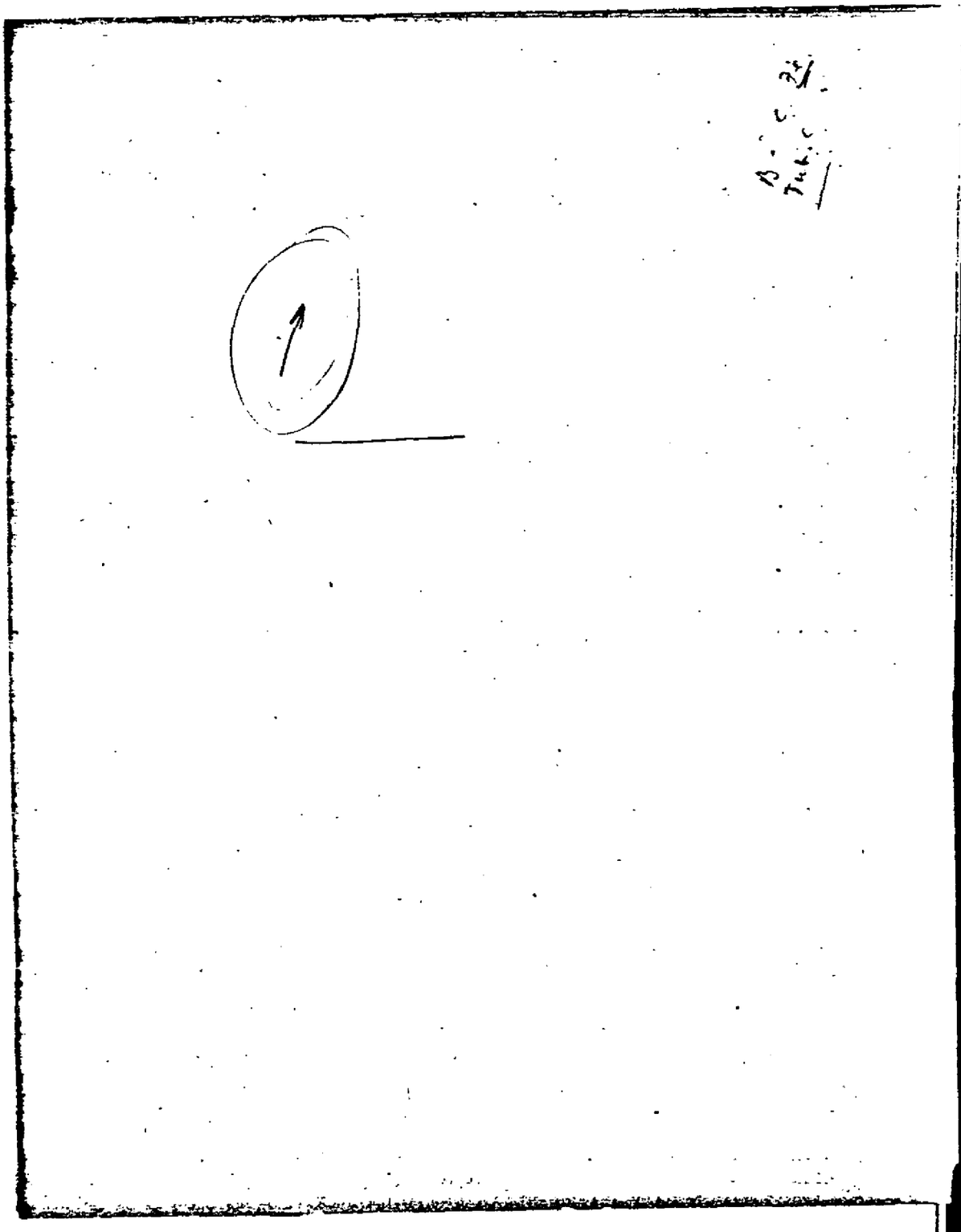
Riboflavin Value, γ / gm.

yeast	beer	Total.	% in beer
22.1	11.5	33.6	34

Ratio of Lbs. beer : Lbs. yeast = 23 : 1

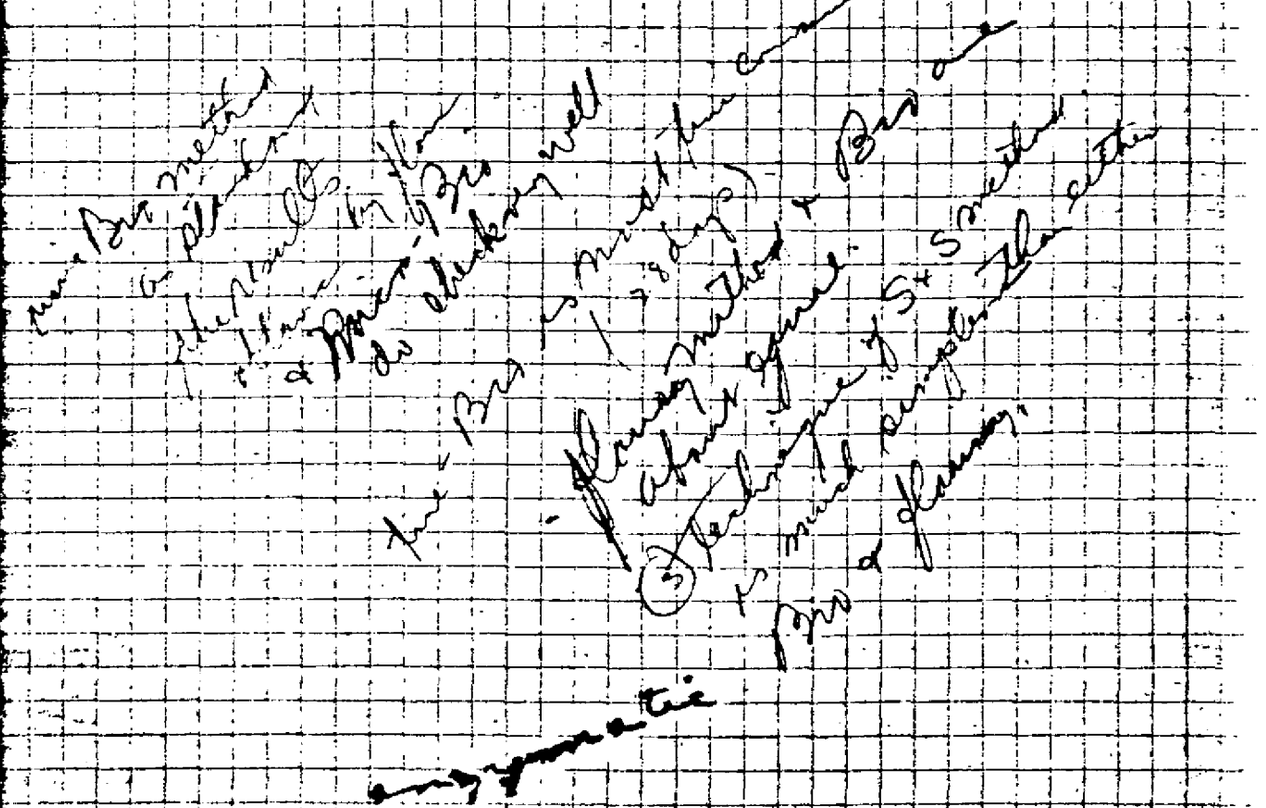
Ratio of Ribo in yeast : Ribo in beer 2 : 1

i.e., there is 11 Times as much (quantitatively) Riboflavin in the beer as in the yeast



(4) Possible flow in one area, but did not completely saturate the Vata Rabo by angle into well 1-0

The method used by the authors is Cryptomia Angeo — we intend to run Cryptomia comparisons on this Gravimetric irrigation with H'd & H'oa



Dr. Reich: Re: Assays ~~Vitamin~~

Assays — Vitamins? —

Question

(1) - Considerable discussion as to (a) accuracy, time, cost, [redacted] of various methods of doing.

Ans.

(1) (a) As to the accuracy of the Snell-Strong method -

An article in the [redacted] Journal of Industrial & Engineering Chemistry answers this -

Five men of the Parke-Davis Lab conducted research through their own laboratories and their findings are published [illegible] -

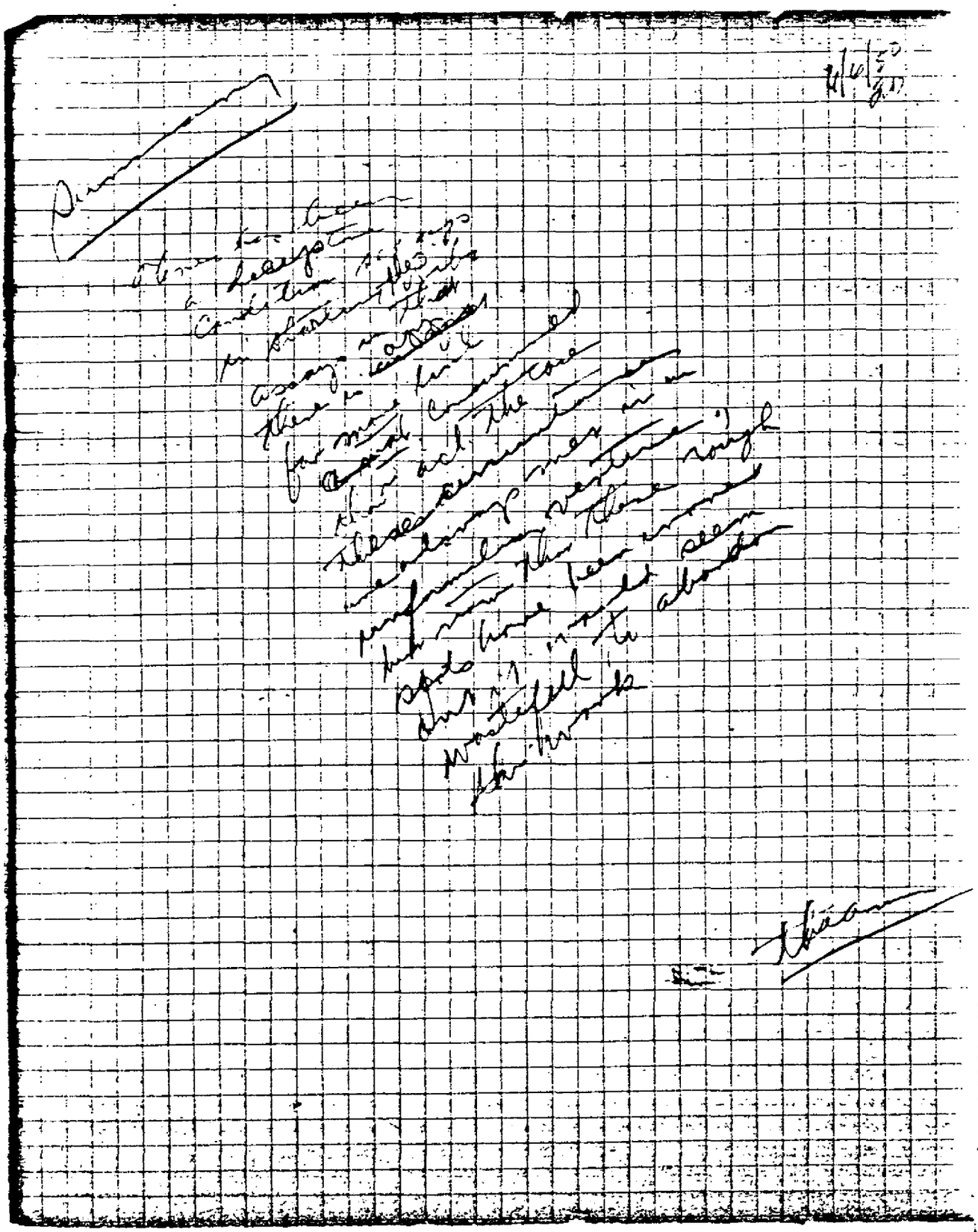
(b) - Time - 3 or 5 weeks in an organization such as ours - Each of said 3 or 5 weeks would require [illegible] We can give an over-all estimate in 3 days - (with)

(1) Reading chick [illegible] and more accurate, when results are [illegible] not known what dosage to administer [illegible] dosage of samples taken in question -

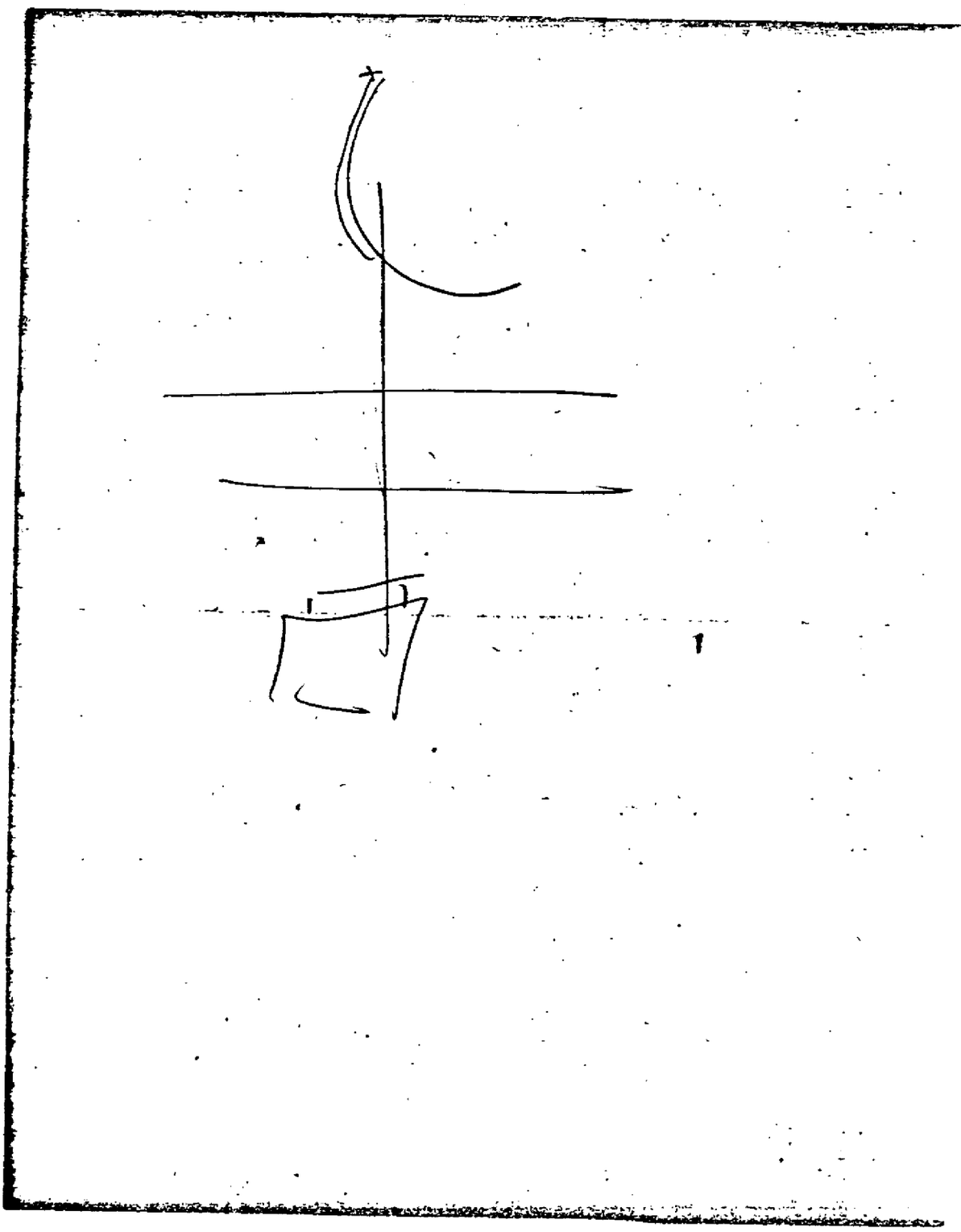
(c) Cost - (1) we have over-all labor well placed now on the way to being able to handle any [illegible] assays.

(2) If we do not have to have an [illegible] we should [illegible] much more work before the expense is taken

and of purchase - Where & when are we to be made of it - after all we do have a colorimeter machine and could we try to develop a method with



In making Riboflavin assays on a malted fermentation, we have encountered certain difficulties. Foremost, however, an evaluation of the stage of the mash was taken a sample of the mash was taken during fermentation and centrifuged to separate the wort and the beer. The moisture content of the samples was also determined so that the assay values could be calculated back to a dry basis. The data for the Riboflavin assays of the mash which has been separated into wort & beer are given below. The data were obtained graphically. The activities of the wort were plotted on them.



65-H307-1-B-12(4) #11

Received from [redacted]
Name of Contributor

[redacted]
(Address of Contributor)

[redacted]
(Name, Title, Organization)

Description: Relief #2 Supplemental Appropriation Act, 1973

Amount: $ [redacted]

Date: [redacted]

File No. CS-4309-IB-72(4)

65-4309-IB-72(4) #13

SAC, PHILADELPHIA

July 7, 1950

T. SCOTT MILLER, SA

HARRY GOLD
ESP - R

65-4307-1B 12 (4) Folder No. 12

On June 24, 1950 GOLD advised that some of the material in this folder is concerned with vitamins in connection with GOLD's work at Pennsylvania Sugar Company. The report dated 12/5/40 was material on production of lactic acid and a page entitled "Personal Affairs" with the following written thereafter in GOLD's handwriting:

a. another job
b. more dough here
c. own laboratory
d. debts

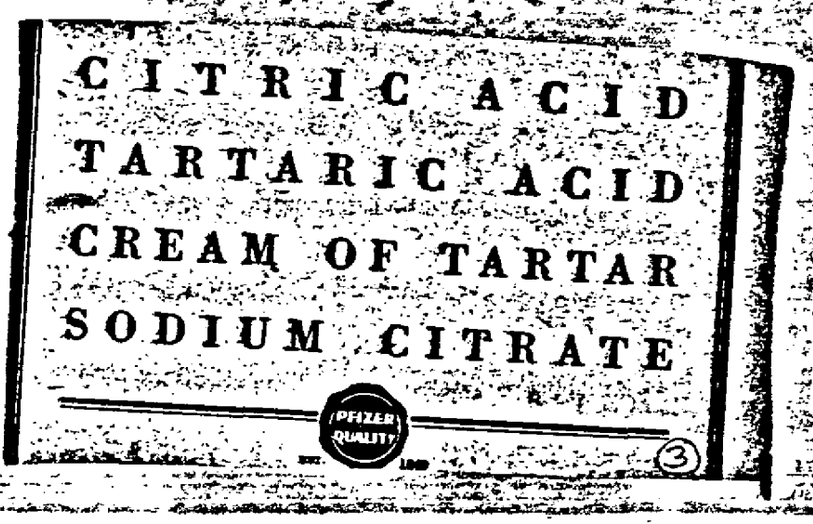
Attention is called to C. above which indicated that GOLD was considering a laboratory of his own as well as D. above which indicated that GOLD had debts. GOLD has advised that he was continually in debt because of his expenses in Soviet espionage.

GOLD said that the letter dated 4/22/42 was in the handwriting of MORRELL E. DOUGHERTY and was a work program for Dr. REICH.

65-4307
TSM:EMC

Date 11-15-11

My Ideas


CITRIC ACID
TARTARIC ACID
CREAM OF TARTAR
SODIUM CITRATE
PFIZER QUALITY
3

Thiamin Destruction In Baking

● A study of the fate of vitamin B_1 in baked goods by Food Research Laboratories shows that the average thiamin destruction in toasting bread is about 15%. This is the first time we have seen a figure for this operation. Also new is the fact that the thiamin loss in baking cake is only a little higher than that in baking bread, despite the higher pH. In angel food cake (pH 5.9) the thiamin loss is only 8%; in plain pound cake (pH 6.4) the thiamin loss is 20%; in sponge cake (pH 7.9) the thiamin loss is 24%. The complete report appeared in the *Northwestern Miller* for October 29.

● This new information is typical of evidences all along the line that the public never actually gets the vitamins it is supposed to from given portions of any food. There is many a slip between the source and the stomach. Many writers on nutrition will have to eat what they are saying about vitamin pills not being needed.

4-22-42

Doc:-

We are really serious about the program we mentioned to you on Saturday last., attached is a rough outline of the work set ahead for us. What do you think of it?

Hadoc.

4/6/50

4-22-42

Assay work

I
- a- Biotin ✓
- b- B_6 ✓
- c- Solutions
- d- Cultures ✓ ✓
- e- Possible trouble
- f- Keeping BY Extraction ✓
- g- Pantothen digestion time ✓
- h- Thiamin ✓
- i- Clarase amount ✓
- j- Other enzymes - "Multiphase" (Wallerstein) - "Pangestin" (Merck & Johnson)

II Work Program

(a) - Survey -

I - No. of samples
II - Place taken
III - method of sampling
IV - when start

6/6/42

(b) - Fortifying -

I - Check on amounts added.
(Theoretical & actual)
II - Method of adding
III - Check on BY.
1- exp. Pa - Niacin
IV Check more often on unfortified yeast and change fortification figures to suit.

C 9

c - Get Methods

1 - University of Texas Publication 4137.
2 - Dr. Lardy - P.M.A. Corp.
3 - Franklin Institute.

4/4/5 28

d - Check on time, amount, classes using same sample of yeast.

I - assay at 48 hrs - 2 gm classes
II - assay at 48 hrs - 4 gm classes
III - assay at 72 hrs - 2 gm classes.

} to be set Saturday April 25th

E - Write to:

Wallerstein
Mead-Johnson

F - Culture -

- Get new cultures at least every month.
- Check future results against different cultures.
- Set, on Saturday, extra tubes, with 5 cc BM 0.1% ribo and 2 c.c. of sample aliquot.

G - Protein -

I - Attempt our own assay method using l-arabinose

II - Try U. of Wisconsin method - l. casei

C q

h- Extraction of BY. 6/6/50 AP

1- H_2O - steaming ✓
2- H_2O - Room temp. + agitation and gradual withdrawal, wash 2 = washing.
3- MeOH + dilute HCl
4- Repeat #2 - with dilute AcOH.

i- B1 - Thiamine

1- Get Motor and drive.
2- Investigate method in U. of Texas Publication # 4137.
3 - Have some of our results on B1 checked outside (for free).

j- we would like to talk over with you, at your convenience, all the possible uses we could make of a Photometer

December 8, 1940

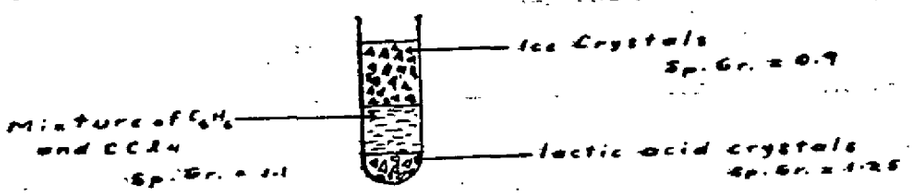
Harry Gald.

6/6/50

A Recommendation for a research project.

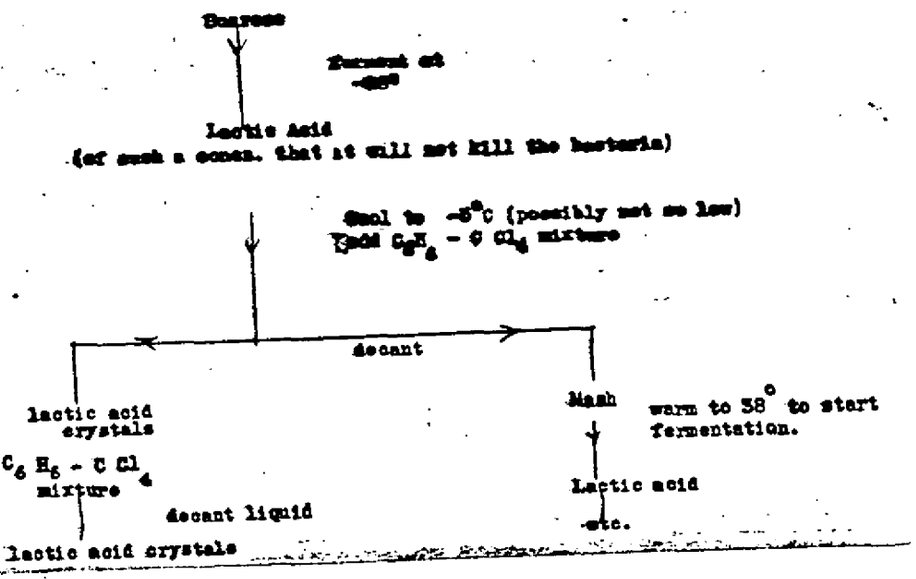
The continuous production of a high concentration lactic acid.

This process is an extension of the idea I have already mentioned for the production of lactic acid by the fermentation of sucrose. In the original method the thought was to utilize the freezing of a dilute solution of lactic acid to yield a mixture of acid and ice crystals; the components were then to be separated thus:



This did not consider the fact that before a water solution containing only lactic acid is obtained, the acid formed must first be continually neutralized with CaO or $CaCO_3$ to prevent the bacteria from being killed - and it is the Ca lactate which gives the trouble.

In order to avoid this step (the formation of the Ca salt) the following modification is proposed; when the point is reached where CaO would ordinarily be added, let the solution be cooled to - 5°C to separate out the crystals of lactic acid in the manner shown on page 1, then decant the mash and warm it up to 38° to start the fermentation again. The flow sheet would appear thus:



Harry Gold

December 5, 1940.

For this process to work, however, the following would have to be feasible.

1. The cost of the refrigeration for the freezing out of the crystals must not be too great.

2. A sufficiently high concentration of acid must be obtained before the point is reached where the bacteria are in danger.

3. The freezing must not kill the bacteria - otherwise the mash will have to be seeded again.

Harry Gold

I Assay work

a - Biotin

b - B6

c - Sol.

d - Cultures

e - Possible Troubles

f - Extra H B1

g - P.A. Digestion Time

h - Ref

i - clean

j - Temperature Control (incubator)

k - Incubation (water bath)

6/9/50

II Work program

a - Enzyme

(1) no of samples to be taken

(2) How to take Samples

(3) When Check

b - Fortifying

(1) Check on amts added to actual

(2) Check on method of adding

(3) Check on B1 itself - particularly P.A. & niacin

c - Set methods for B6 & B1

(1) Time of test meal 4 hrs - (1 hr -)

(2) Determined hourly of P.M. A-1

(3)

(h) Neutral (2) + dil AcOH.

(i) B. (Thiamine)

(1) Net water + dried

(2) Investigate. Unit of Texas method in Anal. 41137

(3) Have some of our results on B. checked outside (for Fell)

(j) We would like to talk over with you — at your convenience — all of the possible uses we could make of a photometer.

4/4/50

(9

II. Personal affairs

a. another job

b. more $ on hand

c. own lab

d. Debts

6/6/50
MP

65-4307-1-B-12(4) #14

SAC, PHILADELPHIA 7/7/50

T. SCOTT MILLER, SA

HARRY GOLD, was.,
ESP - R

65-4307-1B 12 (4) Folder No. 14

On June 24, 1950 GOLD identified this material as work he did at Pennsylvania Sugar in connection with a book which GOLD was going to draw up for Dr. REICH and which was concerned with Pennsylvania Sugar Company methods.

GOLD also stated that this folder contained vitamin assay material in connection with GOLD's work at Pennsylvania Sugar.

65-4307-1B 12 (4) Folder No. 15

On the same date GOLD advised that all of the material in this folder was concerned with vitamin assay work he did at Pennsylvania Sugar.

TSM:EMC
65-4307

rec'd 10/16/40. (U) Harry Gold

Report on Readings in the 6/6/50 gcb
Chemical Literature.

[C.A. 6489 (Oct. 10, 1940)] —— The construction and operation of a simple automatic multiple buret. J. B. Tapp. Can. J. Research 18, B, 217-22 (1940). — Unfortunately, just the value and varied uses of this apparatus (for delivering small measured quantities of liquid at regular intervals of time) are described — no drawing is given, therefore the original article will have to be consulted.

Value to us — small-scale labor in titration operations.

C.A. 6491C. (Oct. 10, 1940) — Exact sampling of a gas stream of varying composition and velocity. W. Krah. Chem. Fabrik 13, 126 (1940). By means of this apparatus a time-average gas sample can be obtained, even though the pressure of the gas may vary considerably.

Values to 24 — Furnace Gases; CO_2 Recovery.

Final Contribution — Conclusion and Abstract

3. Question — Could the pounding of the yeast in the pulverizer possibly affect the vitamin B_1 content? It has already been established that a too powerful juicer will lower the vitamin C content of orange juice

1/12/17 JB

Suggested outline Of work for:

"new" Primary Data and In-

formation

1. Standards

2. Useful apparatus and Devices

   a. Laboratory —

   b. Pilot plant —

   c. Large scale —

   d. miscellaneous —

3. Engineering Data (nomographs, etc.)

   a. Flow of Fluids

   b. Heat Transfer

   c. Evaporation and Distillation calculations.

   d. Tank capacities, etc.

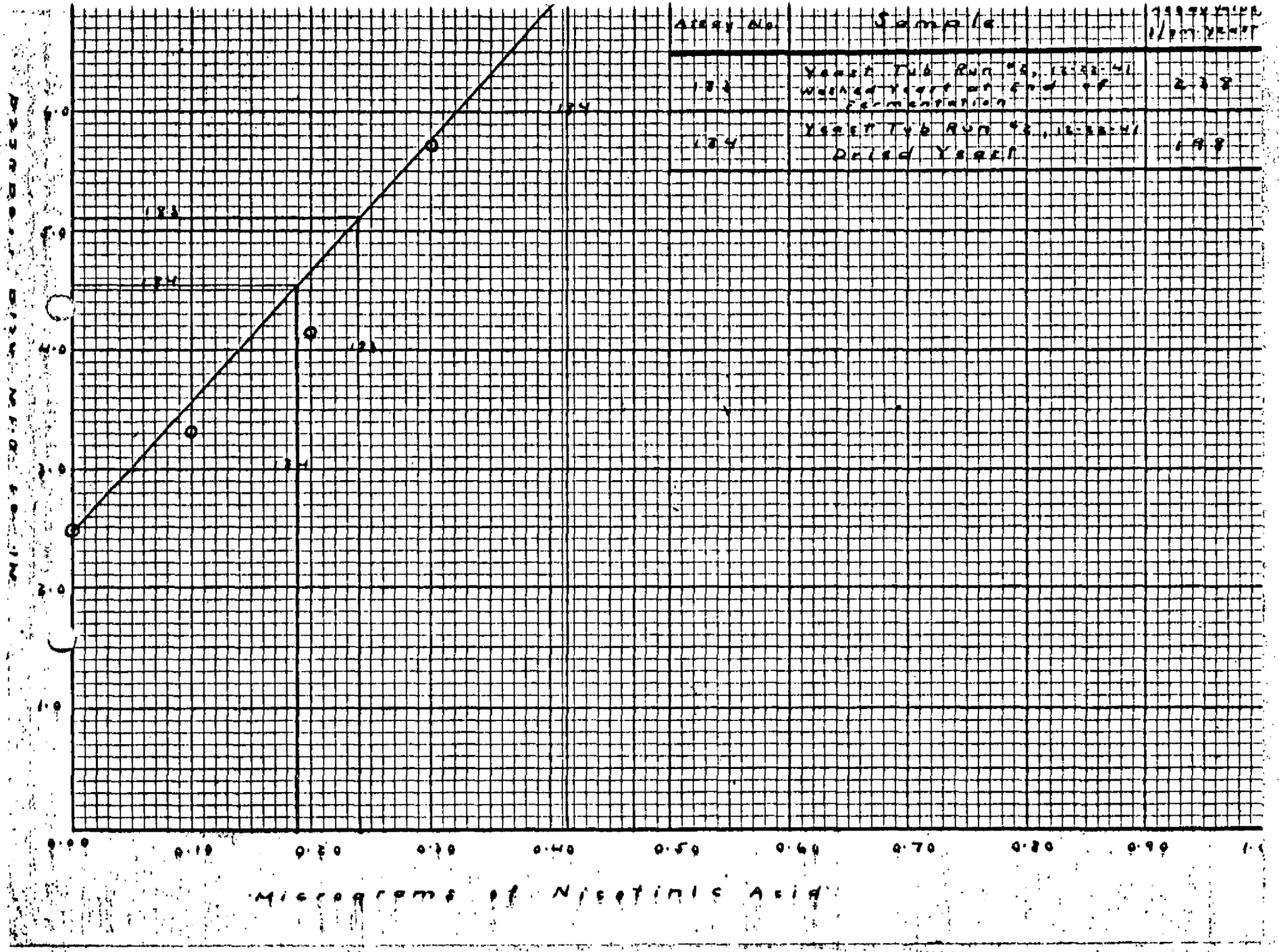
4. analytical chemistry

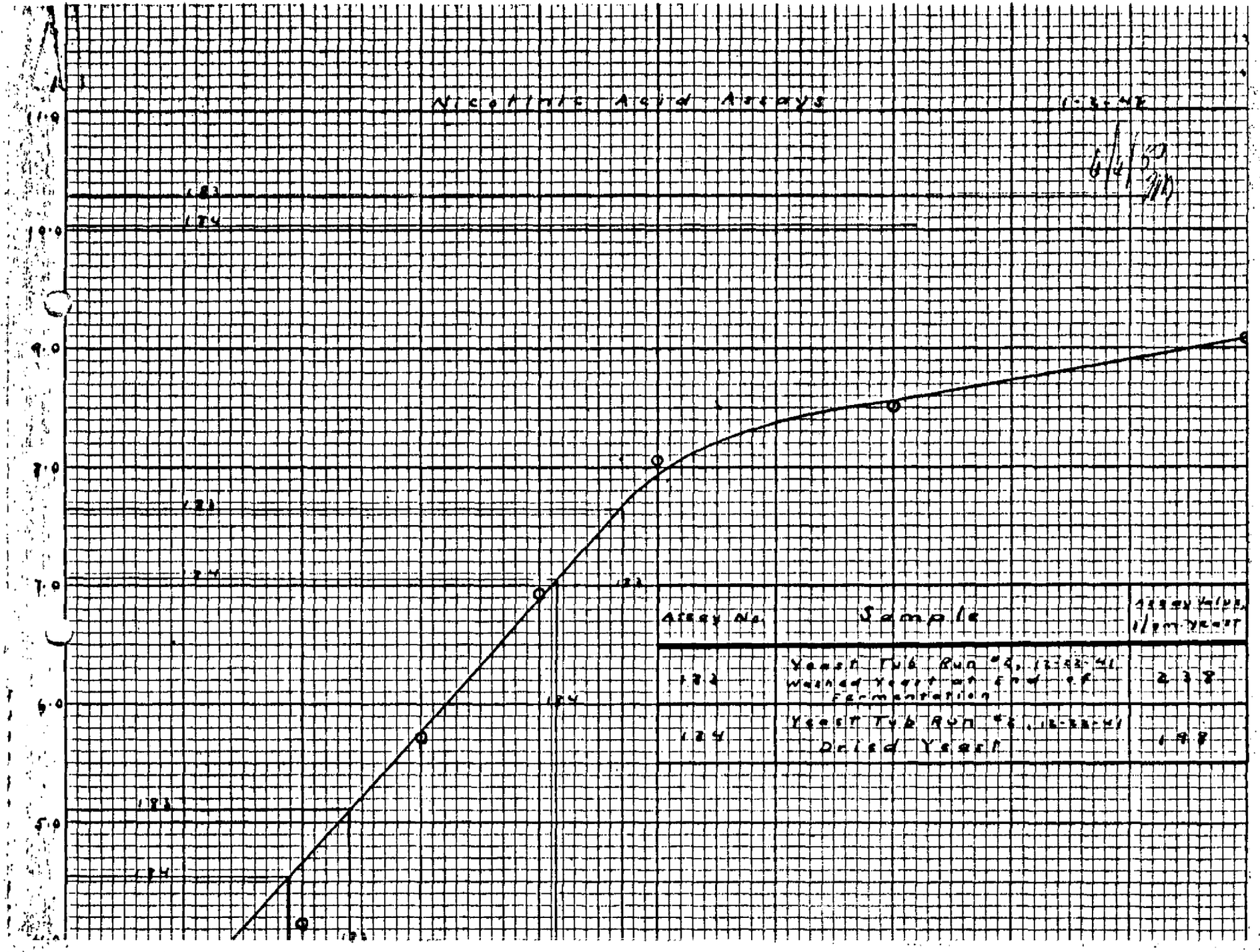
   a. rapid methods.

   b. [illegible] methods

5. short lists of all reports 6/6/50 MS

6. anything new developed in this laboratory.


Assay No.
Sample
Density
134
Yeast Tub Run "B", 12:22:41
Washed Yeast = 1 End of Fermentation
2.38
134
Yeast Tub Run "B", 12:22:41
Dried Yeast
1.98
Density
Micrograms of Nicotinic Acid
0.00
0.10
0.20
0.30
0.40
0.50
0.60
0.70
0.80
0.90
1.0
1.9
2.0
2.9
3.0
3.9
4.0
134
133
134
132
134


Nicotinic Acid Assays
10.0
9.0
8.0
7.0
6.0
5.0
Assay No.
Sample
Assay Units
1/2 mg-ml
124
Yeast Tub Run #4, 12-22-41
Washed Yeast from end of Fermentation
2.38
124
Yeast Tub Run #5, 12-28-41
Dried Yeast
6.98

Standard Curve Data for Nicotinic Acid

Assays Nos. 183 and 184

6/6/50 gm

Tube No.	Synthetic Nicotinic Acid		Buret Readings, ml.		ml. of 0.1 N NaOH	
	mcgms.	ml.	Initial	Final	Individual	Average
1	0.00	0.00	0.57	3.10	2.53	
2	0.00	0.00	3.10	5.51	2.41	2.47
3	0.05	0.50	5.51	8.81	3.30	
4	0.05	0.50	8.81	12.13	3.32	3.31
5	0.10	1.00	12.13	16.22	4.09	
6	0.10	1.00	16.22	20.38	4.16	4.13
7	0.20	2.00	20.38	26.08	5.70	
8	0.20	2.00	26.08	31.82	5.74	5.72
9	0.30	3.00	31.82	38.78	6.96	
10	0.30	3.00	38.78	45.66	6.88	6.92
11	0.50	5.00	0.02	8.33	8.31	
12	0.50	5.00	8.33	16.14	7.81	8.06
13	0.70	3.50	16.14	24.65	8.51	8.51
14	1.00	5.00	24.65	33.82	9.17	9.17

10 mcgms./ml. → 1000 mcgms./ml.

1 mcgm./ml. → 100 mcgms./ml.

10 mcgms./ml. → 1 mcgm./ml.

1000 mcgms./ml. → 50 mcgms./ml.

6/6/50
220

Nicotinic Acid Assay No. 183

Gms. Sample

Dilution 1 gm. ⟶ 250 ml. ⟶ 25 ml. ⟶ 100 ml.

Tube No.	Extract		Buret Readings, ml.		ml. of 0.1 N NaOH		Found		
	mgms.	ml.	Initial	Final	Individual	Average	mcgms. chart	mcgms. per gm.	Avg. mcgms/gm.
1	1.00	1.00	33.82	39.06	5.18	5.10	0.240	240	238
2	1.00	1.00	39.00	44.02	5.02				
3	2.00	2.00	0.00	7.75	7.75	7.65	0.470	235	
4	2.00	2.00	7.75	15.30	7.55				
5	5.00	5.00	15.30	25.60	10.30	10.27	—	—	
6	5.00	5.00	25.60	35.83	10.23				

Comment:

6/4/50
JKP.

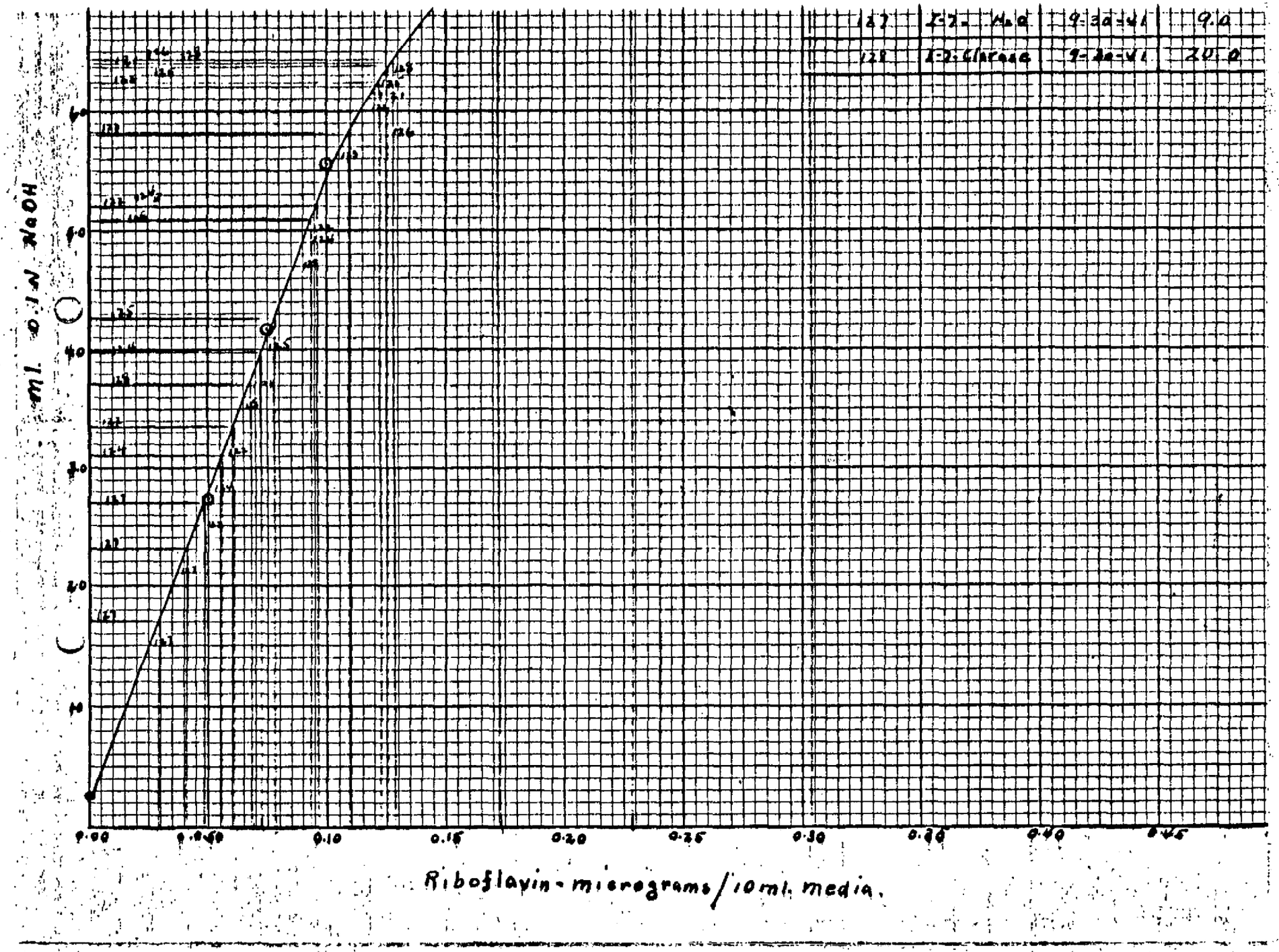
Nicotinic Acid Assay No. 174

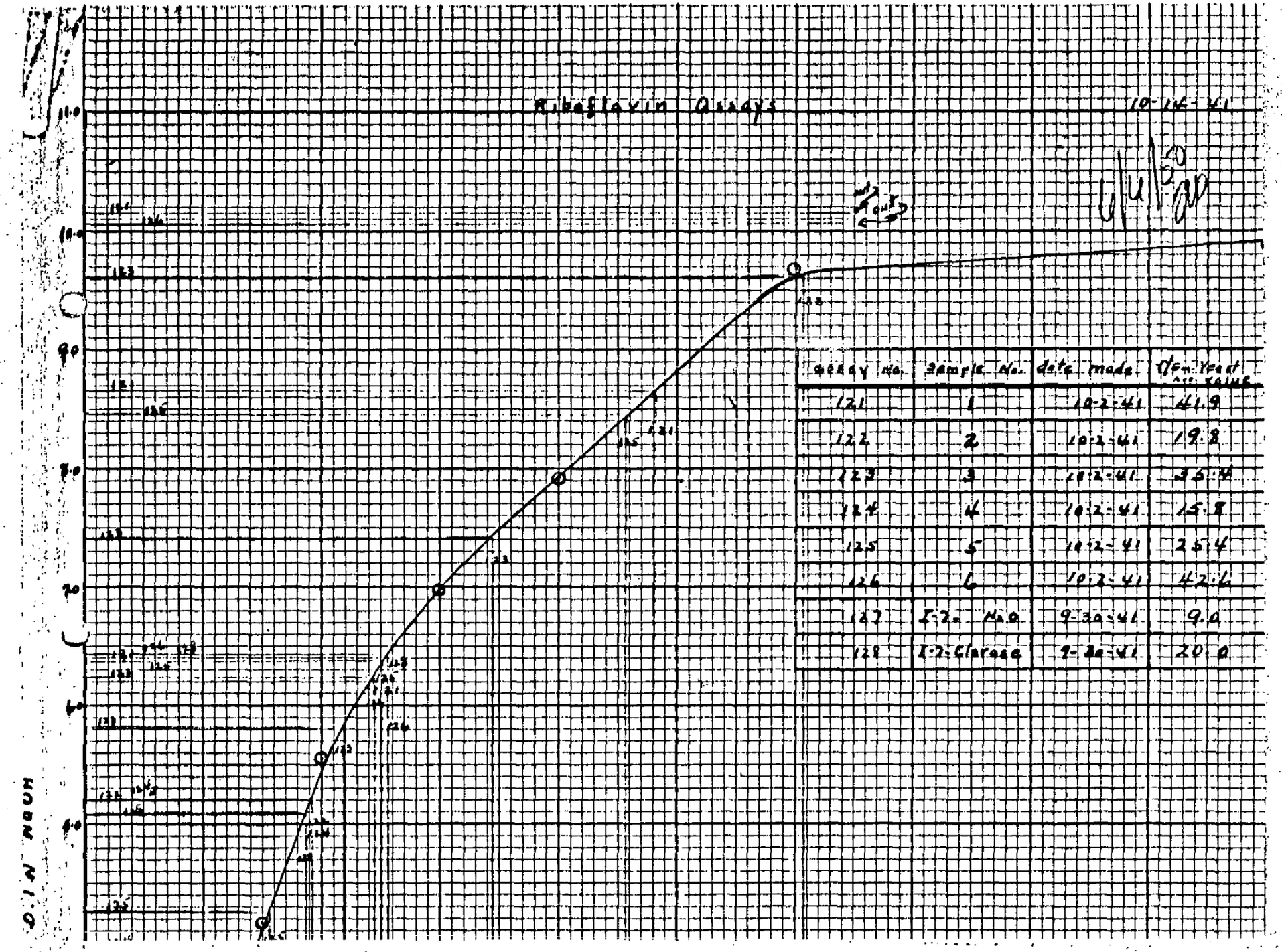
Gms. Sample

Dilution

Tube No.	Extract		Buret Readings, ml.		ml. of 0.1 N NaOH		Found		
	mgms.	ml.	Initial	Final	Individual	Average	mcgms. chart	mcgms. per gm.	Avg. mcgms/gm.
1		1.00	35.83	40.35	4.52	4.54	0.190	190	(198)
2		1.00	40.35	44.90	4.55				
3		2.00	0.00	7.13	7.13	7.06	0.412	206	
4		2.00	7.13	14.11	6.98				
5		5.00	14.11	24.26	10.15	10.04	—	—	
6		5.00	24.26	34.18	9.92				

Comment:


ml. 0.1 N NaOH
Riboflavin - micrograms / 10 ml. media.



assay no.	sample no.	date made	% from yeast
121	1	10-2-41	41.9
122	2	10-2-41	19.8
123	3	10-2-41	35.4
124	4	10-2-41	15.8
125	5	10-2-41	26.4
126	6	10-2-41	42.6
127	1-2-Na.A	9-30-41	9.0
128	1-2-Glucose	9-30-41	20.0

Hattie

10-14-41

Standard Curve Data for Riboflavin

Assays Nos. 121, 122, 123, 124, 125, 126, 127, 128.

4/4/50 gm

Tube No.	Synthetic		Buret Readings, ml.		ml. of 0.1 N NaOH	
	mcgms.	ml.	Initial	Final	Individual	Average
1	0.00	0.00	0.01	0.23	0.22	
2	0.00	0.00	0.23	0.50	0.27	0.25
3	0.05	0.50	0.50	3.20	2.70	
4	0.05	0.50	3.20	5.97	2.77	2.74
5	0.075	0.75	22.99	26.99	4.00	
6	0.075	0.75	5.97	10.31	4.34	4.17
7	0.10	1.00	26.99	32.55	5.56	
8	0.10	1.00	10.31	15.83	5.52	5.54
9	0.15	1.50	15.83	22.99	7.16	
10	0.15	1.50	32.60	39.47	6.81	6.99
11	0.20	2.00	39.67	47.60	7.93	
12	0.20	2.00	0.00	7.83	7.83	7.91
13	0.30	3.00	7.83	17.51	9.68	9.68
14	0.50	5.00	17.51	27.39	9.88	9.88