

84 Bromacetic, 12g, and Naglycerin (pure) by 10gr dried at 170° in H₂ atmosphere
120 cc absol ether mixed in cold: keep cool ice salt & seal up -
gits yellow sticky. after standing 24 hrs in cold heat B.W.B. ket. 8 hrs -
got on desk off ether etc 5 cc oil in sol in H₂ which smells like Bromacetic
∴ nothing definite

850 Dissol. of Naglycerin (pure) (final) of 54. Took 150g Naglycerin in a 1 L flask
and heat cond. adapter B.W. + B.W. with 100% NaOH & agit. that solder bath 240°-70°
solved at first (210°-220°) that about 10 cc ethylalcohol came off (∴ not dried)
long enough at 170°. After getting off 4d. of gas, let this escape & in this operation
accidentally got some NaOH into 18th B.W. ∴ cleanout temp. & begin again.
Time total 2 hrs & 40' heating; temp 240-270°. much frothing at first but at end
all subs. down to clean white salt residue in d.f. got 12 L of gas which with
previous 4d makes 16L in all. Had 13.3g dist in 18th B.W. which boiled
10-75 cc at 78°-98° + 3.25 cc 97°-103°. On drying 2x K₂CO₃ got 7.3 cc of 71°-80°
worth 71°-40°. Mixture of CH₃OH and C₂H₅OH. alcohol on distilling off alc.
Boiled out residue in d.f. 3x with 3 L of absol. alcohol on distilling off H₂O &
H₂O & still off B.W. in vacuo. got 76 gr gummy salt (soluble)
Dissolved this in 125 cc H₂O & added 40g of cone HCl when distilled off H₂O &
vol. fatty acids in vacuo (no oil in dist as not enough added). It took 33.9 cc *
of 100% NaOH to neutralize them. Took up residue left in d.f. again in 100 cc H₂O
and added 20 grs more cone HCl, filtered from small amt of gum & ex to 5x with ether
+ dry CaCl₂. One soap other glycidic acid. This was dissolved in H₂O & filter
8g yellow sheep smelling acid (some by spill). One of these was kneaded with
fine sw. and brand then divided into 2 equal pts. One of these was kneaded with
6g ZnCO₃ & H₂O & 4.9g zinc salt (some by spill) ∴ it xx from one solution in Zn
needles rising oil in H₂O. Analysis: Zn = 26.38 C = 30.37 H = 4.36 Zn = 26.89
Other pt was treated with 5g CaCO₃ & H₂O ∴ can't be glycidic acid as this would
+ is not changed by plus heating (on 100°-130° still remains unchanged). On soap got
Zn Ca glycidate ∴ heated 9 hrs d.f. 130°-135° still remains unchanged. They were dried at 105°
a gummy Ca salt which gum. Anal. Ca = 16.16 C = 40.32 H = 5.47 Zn = 26.89
110° and analyzed. Analysis = Ca = 16 C = 28.8 H = 4.0 Zn = 26.89
0.7541g Zn 0.1707g Ca 0.1328g H₂O } Zn = 26.89 C = 29.6 H = 4.1
0.2688g " 0.3974g Ca and 0.1328g H₂O } Zn = 26.89 C = 29.6 H = 4.1
The H₂O solution which had been ex to 5x with ether was distilled off in vacuo
+ still gave no oil in distillate & got also final no vol. fatty acids as it
look only 3 cc 100% NaOH to neutralize total ag. dist. The residue extracted
with ether gave about 10 cc mobile oily acid but residue still contains
only Na salt besides NaCl ∴ dist it again in 100 cc H₂O & add 10 grs cone of HCl
again distill off in vacuo etc. Found now 39.2 grs white resid. final from
NaCl. The ether solution from last soaking gave 29 grs oily acid heated in
vacuo to 70°: this was put with 10 cc obtained before and dissolved in 110 cc H₂O
+ ex to 5x with ether. 4 cc oily acid only went into the ether & this was
converted into zinc salt & analyzed after xx from H₂O together with other salt (4.7g)
previously obtained. them for C₆H₁₀O₆ = 181.35
Ca = 33.10
H = 4.6

(27)

muscle + or - Lactic

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The aq. filtrate gave on evap. in vacuo + 70° 27.5 grs of oily acid. This was distilled over pumice at 480°-500° time 1 1/2 hrs. got much gas, Cd. 1/2 Co₂ mostly from (H₂O + NaOH) also smell of eth. acid. Had 2 flasks, 2nd in 7.M. 1st had 12 cc dilute + 2nd had 4 cc. on 2x dist got 2.5 cc bpt 31°-70° cfly 31°-45° (eth. acid?) with odor of pure acetaldehyde and 1.2 cc bpt 80°-90° (formal?) and 3.8 cc = H₂O bpt 98°-100° + 1.8 cc bpt 105°-50° which is not pumice acid. Two fl. bps. lost. This expt was carried out to see if could get proof that π glycidic acids were present: these should give pumice acid or its decomp products by $\text{H}_2\text{O} + \text{Co}_2$. The orig. salt residue (after treating) which had been freed from salts sol in ether by 3 days of 2 lbs each weighed 37.7 grs after drying H₂SO₄ in vacuo. 2.999 grs gave 0.2788 Co₂ or 0.679 Na₂CO₃ salt contains 16.6 pct of Na₂CO₃. Took out of salt and bled out once more 2 lbs R.F. with 35 cc absol ale but 0 dissolved: dry again in vacuo. Had now 28 grs left (98 used in analyses) which reacted with H₂O and 28 g of conc HCl + drop off H₂O in vacuo (B.W.B. gave in H₂O dist up HCl (: not enough HCl used) but took 30 cc NaOH to neutralize: just with vol of salts sol in alcohol. An exothermic salt residue (7 x H₂O) with ether got a gummy acid with fatty acid smell when hot 6.7 grs: a very thick oil don't fit solid. This was converted into a Ca salt by using 5 g CaCO₃ + H₂O B.W.B. It gave no xx of Ca gly collate at all and forms a gummy Ca salt quite sol in cold H₂O; the gummy salt changes finally on drying B.W.B. to white granular powder quite sol in cold Water: glycidic acid. (probably a glycidic acid) investigated vol. with H₂O in vacuo + (acid) formed now in vacuo taking 33.9 cc 100% NaOH to neutralize etc) Got from salt sol in alcohol this treated with 10 cc H₂O + 6.0 g H₂SO₄ conc and exlv on evap. 8.2 gr Na salt. This treated with 10 cc H₂O + 6.0 g H₂SO₄ conc and exlv 5x with ether bpt 39°-45° at 8 mm or 100° O.P. cfly 134°-9° 5 grs (only 3-4 drops 118°-30°) 5.2 g acid bpt 39°-45° at 8 mm or 100° O.P. cfly 134°-9° 5 grs (only 3-4 drops 118°-30°) got from salt insol in alcohol (taking 30 cc NaOH to neutralize etc) 6.35 g Na salt this treated with 10 cc H₂O and 5.3 g H₂SO₄ conc and exlv 5x with ether dry CaCl₂ etc. 4 grs of fatty acid bpt 58-73° at 5 mm or bpt O.P. 115-140° cfly 130-40° (2.8 g) this was treated with 10 cc. conc H₂SO₄ in d.f. vapors up 0.5 cc conc off (250 cc) (3.8 g) $\therefore$ H₂CO₃ present. Still off fatty acid O.P. some 80% formed got 4.5 cc dist bpt 100-160°. The 5 gr acid obtained above was treated in same way gave some 20% less and 5.5 cc dist bpt 100°-160° (outside temp 290°). Under the 2 distillates [4.5 + 5.5 cc] exlv with ether + dry CaCl₂ etc. got 4.8 g bpt 36-80° at 1 mm or O.P. 134-138° cfly 120° 5 drops 120°-130° $\therefore$ dissolves in H₂O neutr with H₂ON and small amt H₂O. got 9.4 grs of propionate or O.P. 134-138° cfly 120° 5 drops 120°-130° to sol of 12 g H₂O in small amt H₂O. got 9.4 grs of propionate (they = 11 gr). This was also in 350 cc

boiling water and sub. to fast xx. I. 4.5 grs II 11 grs III 1.05 grs
IV 0.4 grs V 0.3 grs. all pts come out in needles sup blackening
on BWS & they look homogeneous.
Analyzed Fr III and V for ag.
III 0.2398 g salt gave 0.1428 g ag or 59.53 pct
V 0.1644 g " " " 0.0976 g ag or 59.37 pct
∴ practically pure propionic acid free from acetic acid is formed,
in expt.

86) 20g Bromoacetone (2 mol) and PbO 20g (1 mol = 16g) dried 120° heated in
s. 100° for 10 hrs no action ∴ heat 130°-135° for 10 hrs when all laborm. got
on working up except black tar

87) 20g Bromoacetone and add slowly 2.4g Na glycerin in small lots get hot &
vol of liquid increases at first then gets brown and thick. Heat finally to 100° for
20 minutes (R.F.) got a resinous product a thick gummiol in ether.
n.g. this is efly s. dibromoacetone

88) mesq. dibromoacetone (obs as side prod. 1082) was heated in absol. ether
solution with of benzate and of acetate in st. heating finally to 100°.

got in both cases much pressure in tube and found much free acetic
acid. Benzoic acid had been formed in expt.

20g Kaecate in 60g absol alcohol sheet finally 85°-90° for 1 1/2 hrs.
70°-91° at 7 mm, added to a boiling solution of
30g dibromoacetone bpt 73°-91° at 7 mm, added to a boiling solution of

30g Kaecate in 60g absol alcohol sheet finally 85°-90° for 1 1/2 hrs.
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30g Kaecate in 60g absol alcohol sheet finally 85°-90° for 1 1/2 hrs.
70°-91° at 7 mm, added to a boiling solution of
30g dibromoacetone bpt 73°-91° at 7 mm, added to a boiling solution of

X N.B. from now on 93° Oct 11902 all ~~the~~ an corrected for
(29) ~~maximeter~~ readings are O.K. i.e. correct

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(91) mpgr of Acetolacetate: 300g bromoaceton (38-46° at 7mm) and 225g lactale
(heavy 214.5) and 450g absd alcohol sheet mixture R.T. before adding
bromoaceton (sept funnel) 10-15 minutes & then heat 2 1/2 hrs R.T. 85-90° filter
spurt in vacuo. got 178g bpt 65-72° at 15 mm - a second expt gave
184 grs. This is impure material which decomposes on keeping and
∴ must be used up at once for expts (it contains bromoaceton)
after 10 days standing took a 50g lot and found it only partly soluble in
H₂O (leaving thick tar) ∴ labelled [net: 282 grs] using 88g 50%
I 19.2 g bpt 40-47° at 6 mm; II 6.7 g bpt 57-64 at 6 mm } or total 268.19
II 10.3 g " 47-57 at 6 mm; III 31.7 g bpt 64-76 at 6 mm } leaving 19 gr black
tar in d.f. Repeat net in same way. Found finally in I on dist. O.T.
bpt 72-100° that it contains ethylacetate and N₂O fumes and got a product
from II & III finally which boils 29.2° from 108-120° at O.T. & is Essenzig.
the acetolacetate on standing a short time darkens. This can all
be promoted see later.

(92) Took 30g acetolacetate bpt 55-72° at 10 mm (3 days old) and treat with
9g aceton fr. 150° in 8.1. 130-150° for 4 hrs found = 8.3 g and distill
in vacuo at 12 mm. got 1.8 g bpt 30-40°; 6.4 g bpt 40-60° and 11.3 g bpt 60-80°
leaving small amt of tar in d.f. Repeat at 7 mm and got oils.
3.7 g bpt 40-50° [acetol] 5.4 g bpt 50-60° and 6.8 gr bpt 64-66° last part
is soluble in H₂O and odorless -

(93) mpgr of Epiglyhydrin. N.B. 100g epiglyhydrin 277 grs (I prod) (1 1/2 mols) and
130 cc absd alcohol heated R.T. 80-100° for 17 hrs gave on pouring into H₂O
170g oil which ex. ether washed H₂O 1070. ex. ether wash etc gave
higher small amt. much alcohol in H₂O. Since abs. alcohol
a total of 80 grs Epiglyhydrin & 225 g KF 22 mols, 50 cc abs. alcohol
Repeated with 50g Epiglyhydrin & 225 g KF 22 mols, 50 cc abs. alcohol
16 hrs at 90-100° and got as fuel prod 38.5 g pure Epiglyhydrin
(77 g oil sep by H₂O at first) " no advantage over first method.
If use 50g Epiglyhydrin and 180g prod KI 2 mols in 8.1. 130-150° for 16 hrs
got 82 g oil by H₂O finally got 31.6 g oil bpt 53-65° at 17 mm with
same epiglyhydrin in formic acid. no advantage.
82 gr. of pure Epiglyhydrin bpt 54-62° at 18.5 mm is 1982 at 24°

(94) It is impossible to convert propylene bromide into diacetate by heating
R.T. 170-180° bath for 9 hrs with 1 mol fused Na⁺acetate 40.6 g + 50g glac. acetic
practically no action takes place: reversed 78g bromide.
Also got no action by using the " " with 37.8g Na⁺acetate (1 mol)
and 76g absd alcohol heating R.T. 110-120° for 8 hrs - reversed 70 gr.
on heating this (70 gr) with 58g Na⁺acetate (1 mol) in oil bath to 150-170°
with 100 cc Essenzig got an action but Result is to form much
diacetate leaving much bromide + little propylene
acetate prod ∴ impracticable.

95° The Reduction of Chlorhydrin by Na to propyl alcohol found n. g.
 used 20g and 100gr H₂O & 2 atoms Na as 80% Na¹⁰⁴⁵ cooling in ice water -
 + kept neutralizing alkali by conc. HCl and finally added 50g more Na¹⁰⁴⁵.
 a similar expt with 20g pure glycerol gave similar results

96° Epichlorhydrin and H₂O. must allow to react slowly otherwise get blowup!
 heat 1 day at 60° 1 day at 80° & finally 2 days at 100° B.P. in fall bot.

Took finally 6 lbs each containing 35.3g epichlorhydrin 127.4g dry ag₂O, 200cc alcohol
 ether. Filter & distill in vacuo. 34grs ep. diglycid ether

200g epichlorhydrin gave after 3 part distilling 34grs ep. diglycid ether
 bpt 103° at 17 mm spgr 1.119 at 23°. colorless oil, sol. in cold H₂O.
 analyzed Dec 18th '02. Found a considerable percentage 23.2% which is a
 mixture of Epichlorhydrin and Glycidol (!).

97° In an expt. using 200g epichlorhydrin 720g KI (2 mols) and 360cc abs. alcohol
 heating in 2 lots for 11 hrs at 80° - 85° got finally 205g pure epichlorhydrin.
 heating in 2 lots for 11 hrs at 80° - 85° got finally 205g pure epichlorhydrin.

98° Took 200g glycerol 100cc of water containing 1cc ep. H₂O₂ conc & heated in
 a d.f. 100°-120° 8 hrs (no distillate) and then for 9 hrs from 120-150° when got
 65cc distillate which was no test with Phillips sol or with Phenylyl bromine
 in 2 hrs. 80°. then heated 2 hrs longer bleached 46°-172° & got a black
 in 2 hrs. 80°. the residue in d.f. is now a black
 smell of acrolein (final temp 200°) it is definite this way

99° Epichlorhydrin alcohol and Acetate 22grs react on heated R.T.
 90°-105° pure ethylacetate and only emulsion products but no
 glycid acetate. heated Epichlorhydrin 50g in S.T. with 1 1/4 mols
 Acetate 66g powdered S.T. 110°-115° for 8 hrs. Take up in ether and
 put in vacuo. got 17g Epichlorhydrin and 4.8g oil bpt 70-100° at
 24 mm or 160°-170° at o.p. which is probably glycid acetate and

14.8grs oil bpt 160°-220° at 24 mm (o.p. temp 275°). Considerable
 light yellow sticky oil left in d.f. not distill. The fraction 14.8grs on
 ordish boils 162°-178° at 27 mm (1/3 left in d.f.) Found also some thick
 oil with KCl residue. From this it is clear that to make glycid acetate
 must use Epichlorhydrin + acetate. For getting dimolecular glycid acetate

use above method. cf. Breslau's J. pr. (2)

$$\begin{array}{c} \text{CH}_2 \\ | \\ \text{CH} - \text{O} - \text{C} - \text{CH}_2 \\ | \quad \quad | \\ \text{O} \quad \quad \text{O} \end{array} \quad \text{CH}_2 - \text{O} - \text{C} - \text{CH}_2$$

100° Distill. of Epichlorhydrin. took 50g bpt 114°-5° ca 7K. Temp 450°-460° pressure.
 time 2 1/4 hrs drop in by sep funnel. Had a d.f. and 1 BW in ice H₂O tagometer.
 got 3 1/2 grs inflammable + colored green (leaves CH₃CO). got in d.f. 43g oil which
 is free from HCl fumes but smells like acrolein. Distill at first o.p. got 4.3cc bpt
 80°-110° II 14.3g bpt 110°-140° III 14.8g bpt 140°-160° last portion colored :-
 put III in d.f. and distill at 17 mm. got 7.1g bpt 48°-70° and 11.9g bpt 70°-77°
 residue in d.f. now combined with 7.1g & fraction in vacuo continued

clavering? in lab

(311)

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got 3 cc bpt 45-65° at 33 mm [put this with fraction bpt 120-150° S.P.] and
also 4 g bpt 85° at 33 mm ∴ in all 11.9 g + 4 g = 15.9 g high boiling oil
The spgr of oil bpt 70-77° at 17 mm was 1.344 at 23° and this is i.
a dichlorohydrin as proved by converting it ~~back~~ to Epichlorohydrin by
pouring NaOH (10.5 g) : got 5.6 cc bpt 114-6° ∴ O.K.
the fraction bpt 80-120° (2 1/2 cc) was heated in a S.T. with 15 cc H₂O at 110° for 17 hrs
1.25 cc of black oil left. distill ag. filtrate in vacuo and got bnd so. And
2.6 g of bpt about 120-130° at 18 mm [(!) chlorohydrin]. The two solutions gave
a xx prep with Ph₂CO & acetic anhydride present.
Funct bpt 120-150° (6.75 cc) heated with 14 cc H₂O in S.T. 100° for 17 hrs gave
- 2.25 cc black oil & eq. filtrate distilled over, bnd so. And giving last for
+ 0.5 cc yellow oil (sharp) bpt 80-110°? (acetal) at 25 mm.
+ 0.7 cc bpt 110-180° + rest is tar. all oils except last fraction dichlorohydrin
have strong smell of acrolein. chloroacetone $CH_2=CH-C(=O)CH_3 = CH_3-CH=O + CH_3CO = C + HCl$
(?) $CH_2=CH-CH_2 \rightarrow$ gives $CH_2=CH-C(=O)CH_3 = CH_3-CH=O + CH_3CO = C + HCl$
 $CH_2=CH-CH_2 + HCl =$ a dichlorohydrin.

It is certain that no part of acrolein formation was obtained
Dissoc. of chlorohydrin bpt 128-130° at 22 mm. Ca 7.7 K used 50 grs.
Temp 440-600°. Time 4 1/2 hrs, dropped in over pressure: got 1300 cc gas inflam
got 35.4 g and f 2.5 g in Bnd. much HCl in app. - much clavering.
distilled oil (37.9 g) on Bnd. B. & got 2.75 cc oil with much HCl gas: of this
low layer = 0.5 cc and upper layer a fragrant sharp smelling oil = 2.25 cc is
insol in the gas. Distilled rest in vacuo got 11.3 g bpt 20-40° at 20 mm
and 2 g bpt 100-180° (outside temp 180°) leaving charred residue in d.f.
The fraction bpt 30-40° at 20 mm boils 108-109° at 0.1 P. is chiefly
aqueous HCl.

(102) chlorohydrin bpt 119-122° at 22 mm Ca 7.7 K, 20 gr with 1 mol of
Kacetate 2.2 g, in S.T. 110-115° for 9 hrs - no pressure. take up in ether
when much oil is left insoluble. distill off ether filtrate & part resid oil
in vacuo. got 2 drops of formic acid which redist boils 5 gr betw 145-150° at 19 mm
bpt 140-60° at 23 mm poor yield in any case.
? is it monoacetyl glycerol. Ca 7.7 K. 114-150 20 g, and 20 cc absol alcohol in S.T. 13 hrs
3.4 g bpt 40-85°

(103) Epichlorohydrin Ca 7.7 K. 114-150 20 g, and 20 cc absol alcohol in S.T. 13 hrs
150-160° gave on fract. 6.2 g oil bpt up to 40° at 30 mm is pure $CH_2=CH-CH_2-CH_2-CH_2-CH_2$
at 25 mm and rest = 15.75 g oil bpt 85-88° at 25 mm = pure $CH_2=CH-CH_2-CH_2-CH_2-CH_2$
no HCl in 1/2 fraction consists of alcohol & bases unchanged
Epichlorohydrin

104 Took the body bpt 85-88 at 25 mm 15.75g, and heated it in a cold
with 2 vol. 9 gr powder ap. NaOH adding latter in small amt. warm up i. Cool
tap. after 35 minutes add other filler shell. got 8 cc Glycid ethyl
ether bpt 125-135° aft 127-80°
Repeated expt 103° using 100g Epichlorhydrin + 100cc alcohol in 4 S.T.
11 hrs at 140-150° got 40cc alcohol bpt 75-100° 28.3 gr Epichlorhydrin bpt
114-120° + Ethyl chlorhydrin 52 gr bpt 80-85° at 17 mm
The ethyl chlorhydrin, 52g, gave with 30g Powd NaOH etc 30.4 grams
the ether bpt 122-130°

glycid ethyl ether bpt 122°-130°
 105° glycid 20g bpt 65°-80° at 21 mm and Essing 2 vols 32.5 grs
 mix in cold (no action) :: let stand 22 hrs + got on heat materials
 back again unchanged :: let stand again longer - ^{small amt} residue of Polyglycids
 remains in d.f. not rd at 75°-80° ^{with} and pressure 21 mm. but chiefly no action.
 Heated finally 4 hrs at 75°-80° ^{with} and then fract. got back 19.5g
 acetic acid (bpt up 65° at 22 mm) and 4.7g bpt 50-100° at 22 mm
 and 11.1 gr oil bpt 168°-240° at 27 mm (outside temp 280°)
 leaving thick almost colorless residue in d.f. about 5 gr
 as to nature (bis glycid) and Poly glycids (certainly monoacetyl glycid)
 20g ^{12.4g} is acid react with great violence on mixing in cold
 The oil is rheal uns 7 1/4

106° as to nature (bis glycid) and 12.48
 Glycid + formic acid react with great violence on mixing in cold
 so heat explosion took place & lost all material ∴ repeat using 7th
 and add 7.75g. Also sep funnel to glycid 10grs - at -10°. To rise of
 temp but when 7th was more temp rose fairly rapidly: did not allow it to
 get above 30°. At cooler heat for 14 hrs when no more heat cool
 destill at reduced pressure. got 3.5g bpt up to 100° at 27 mm & small
 of 7th. and 2.8grs bpt 160° - 210° at 27 mm 4th and left in d.f. with
 thick gummy colorless oil not distill outside temp 280° at 27 mm ∴ can't
 be more formic glycid. Took 37.1 grs, made 104, bpt 122-30° bpt 125-135°
 Glycid ethyl ether. Took 10.5 grs as in 100°. Got 2800 cc gas in 1st
 as in 100°. Got 2800 cc gas in 2 hrs.

distill at 2.8 grs bpt 180
of H₂O and not distill outside temp 180
thick gummy colorless oil
to monoforming glycerine. Took 37.1 grs, made 104, bpt 122-30° bpt 125-135°
Dissoc. of Glycid ethyl ether. ^{at 100°} as in 100°. Got 2800 cc gas in 1st
107 Dissoc. of Glycid ethyl ether. ^{at 100°} as in 100°. Got 2800 cc gas in 1st
and run over juice at 450°. Then 2400 cc more gas in 2 hrs:
2½ hrs & then stopped for 1½ hrs and then collected 2400 cc more gas in 2 hrs:
about 5.5 d gas in all. Gas is inflammable and has odor of ethyl ether as
was also H₂O in azotometer. The d.f. had 28 cc distillate which dist
O.P. gave 13.5 cc bpt 45-85° #57 sec bpt 85-120 #7.6 sec bpt 120-140°
and 4 b of bpt 140-160°. Residue in d.f. was distilled at 120-140° pressure
got 3.48 bpt 75-120° effy 80-85° at 18 mm. Oil is diff sol in cold H₂O & heavier than
H₂O & dissolves slightly in H₂O.
Refraction 1.0 f bpt 120-150° at 18 mm having little residue in d.f.
Got also 1.5 cc bpt 20-70° effy 30-50° [Acetaldehyde & crotonaldehyd]
all but 0.6 cc of this dissolves in 3.5 cc H₂O. [Acetaldehyd & crotonaldehyd]
Refraction 1.0 f bpt 70-80° dissolves except 0.2 cc in 3.5 cc H₂O [Acetaldehyd & crotonaldehyd]
Got also 1.5 cc bpt 85-120° which dissolves easily in eq. vol H₂O. [Acetaldehyd & crotonaldehyd]
has strong odor like acrolein. The H₂O product
got also 4.25 cc oil bpt 100° for 3½ hrs using 8.5 cc H₂O & then distilled in vacuo
got H₂O only and about 1 cc gummy residue. The H₂O product
Gave with K₂CrO₇ 0.6 cc oil. CH₃COCH₂CH₂CHO?

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108) Bromaceton and Ag₂O (cf No. 107) $\frac{1}{2}$ mol Ag₂O in 40 cc
in 15 cc absd ether to 25.4 gr Ag₂O (own make due 100-1150) suspended 30 g Ag₂O in 40 cc
an action that expt not continued. In next expt suspended 30 g Ag₂O in 40 cc
absd ether and then added the solution of Bromaceton (30 g) in ether (15 cc) slowly
thru a hyp. funnel to Ag₂O, cooling with 7 M. In morning from 7 M. acts slowly
slit temp. got as high as 130° still stand over night. Other heat R.F. 157 gr of
ether for 4 hrs. - filter & wash. got 5.1 g Oil bpt 48-70° at 22 mm 3 inch. Bromaceton
and 1.8 g bpt 70-120° (22 mm) and 3.1 g bpt 120-140° 22 mm (! Acetoether).
Had about 3 g residue in d.f. at a temp. 200° which would not distill over.
much Ag₂O in salt residue + little Ag. $\frac{1}{2}$ mol = 25.4). Suspended the Ag₂O in
Bromaceton less in 20 cc absd ether
take out of 7 M. in

(110) Bronze 80gr, Oil bpt 48-60° at 23 mm, and 729 fused Nacestate (1^{1/2} lbs)
heated R.F. 100°-120° for 6 hrs. and then add ether filter
which can't be sep by K2CO3 solution.

(110) Bromacetic, 80g, bp 100°-120 mm R.F. 29.4g Oil bpt 27 mm
and 72 g Essigsig got 71 g Essigsig and Essigsig which can't be sep fr by
distill in vacuo. acetolacetate and shake with conc aq. K₂CO₃ solution +
this is a mixture of acetolacetate and ether and shake with conc aq. K₂CO₃ solution +
fract. dist. ∴ take up in ether and shake with conc aq. K₂CO₃ solution +
dry ether callz schell. got 15.8 g v. cp. acetolacetate bp 73°-4
any other callz schell. keeps indefinitely. *but have used solid K₂CO₃ in ether. sol*
at 18 mm which keeps indefinitely.

(111P) an expt with 80g. Dibromoacetic 90°-110° at 27 mm, and 91g Vinylacetate
(3 mols) and 91g Essigsig R.F. 6 hrs 110°-120° gave o definite except
large amt of tar, sol in H₂O, 20g. ∴ n.g.

(112) Took 80g Bromaceton and 60g xx catK Naformiate ($1\frac{1}{2}$ mols) and 15g xx HCOOH in S.T. and heated for 8 hrs at 100° . Added ether & filtered & poured 7.3g oil bpt up to 60° at 22 mm and 32.3g bpt $60^\circ - 73^\circ$ at 22 mm. Refracted more this to be HCOOH and got 30.2g oil bpt $60^\circ - 75^\circ$ at 22 mm. Heated this in S.T. with 52g CH_3OH (5 mols) at 100° for 9 hrs & distilled off HCOOH on Bw B at 0° . & fraction residue in Vaevu. Got 9.5g Oil bpt $48^\circ - 53^\circ$ at 22 mm. This redistilled after 2 days had changed to fair amt of gum not volatile on Bw B at 21 mm. Got broad is 0.5g oil bpt $53^\circ - 60^\circ$ at 21 mm and 1.0g oil bpt $90^\circ - 120^\circ$ at 21 mm leaving ~~perhaps~~ a gum in d.f. which begins to come over when bath is 250° at 21 mm. and glycerin (12.4g mol) stood 40 days in cold bath.

113) Glycerol 10 gr and Acetol
absolutely in action. infusory Acetol
100 and 65 gr Na

absolutely no action. 114° 100g Bromacetic and 6.5g Na formate ca 7K [mol Navoet, H₂O = 62.7g] (X)
and 120g CH₃OH heated R.T. 85°-105° for 9 hrs. Distill off oil on BW B vent in
vacuum. bpt 52° for runner up to 52° at 22 mm and then 29g of Acetol
bpt 52-60° at 18 mm [It still contains Bromacetic and don't keep]
and 5gr Oil bpt 60°-90° at white temp = 203°. Refract 802 25g Acetol
bpt 47°-53° at 19 mm. and 5.1g Oil bpt 125-135° at 19 mm
after 9 days standing the Acetol was found to have much gum on
2nd distillation in fact it always changes and can't be kept. It also has
taint smell of Bromacetic. (converted this into Oil No 117°)
Took 100g Bromacetic bpt 48°-60° at 23 mm and
then heat

2nd trial: Took 100g Bromoacetone bpt 48-60 at 23 mm and
 faint smell of Bromoacetone. Took 100g Bromoacetone bpt 48-60 at 23 mm and
 (115°) Acetolacetate ref. gr. Took 100g Bromoacetone bpt 48-60 at 23 mm and
 75g Kacetate and 150g absol alcohol mix in cold. warm up, then heat
 2 hrs at 85° R.F. Filter & fract 2x. Got 65gr oil bpt 65-80 at
 18 mm cflg 72-4°. This don't keep but gets colored at once after distillation.
 Treated it in the solution with a conc aq. solution of 50g K_2CO_3 in 100 cc tho
 + dry $CaCl_2$ & got back 55gr Oil bpt 72-6° at 18-19 mm. Still changes
 after 24 hrs standing ∴ heat R.F. with 20g glacial acetic & 70g Kacetate
 for 9 hrs add ether wash with K_2CO_3 (over) in 200 cc tho soln in dry $CaCl_2$.
 Got 31.5g oil. Acetolacetate bpt 73-4° at 18 mm with absolutely
 no odor of Bromoacetone a sweet smelling Oil which keeps indefinitely
 at No 110. 2nd trial: took 1gr Acetol (fr 114) & treated with a sol of 2 gr K_2CO_3
 in 135 solid K_2CO_3 . Warm up

got 3.15 g
 no. 10 of Bromoaceton a white
 ct. No 110
 (116) Semicarbazone of Acetol: took 1 gr Acetol (fr 114) & treated with a sol of 2 gr HCl
 of semicarbazone (theory 1.5g) in 5 cc H₂O (which had been added 1.3g solid K₂CO₃). Warm up
 & got 5 cc at once. add 5 cc more of H₂O & filter. got 12.5 grs crude solid. Recryst
 from 20 cc hot H₂O on cooling got 0.95 grs white flake needles stark light broken
 with deph about 200° of snow melting point is 195°. The S. carbazone is most used in
 either C₆H₅ and CHCl₃.
 (117) Oxime of acetol: Took 3g of Acetol (from 114) and add to solution of 4g H₂NOH, HCl (theory 3g)
 in 10 cc H₂O to which had been added 4g K₂CO₃ solid. let stand over night - then add
 in 10 cc H₂O to which had been added 4g K₂CO₃ solid. let stand over night - then add
 + on distilling off ether on W.B. oil left soon
 got 3g crude

Took mass of material, 1.2 grs, obtained from No 130 (see) dis. expt. nearest found body can be xx from H₂O (12 cc). came down (35) slowly in clear group. Nadeln 0.95 grs up to 145°. xx not well defined but an flat needles. Note special Acetolacetate (16 grs) can't be separated to Acetol by means of ether & KOH. (mole 8 grs in cold get going out). The University of Chicago O.K. of Bloch

which melts at 68°-70° and boils at 123°-5° at 18 mm. Is an odorless xx solid

118° Oxim of Acetolacetate: Took 3g Acetolacetate (of 115°) and treated with 2.4g H₂NOH, H₂O 1.8 cc H₂O + 2.4g dry KO₂. Warm up: cool tap. let stand 20 hrs + xx to 4x other dry cells 73st 2. f. thick colorless oil bpt 118°-9° at 16 mm with faint sweet odor. not yet solid. is probably the oxim.

119° Semicarbazone of acetolacetate: Took acetolacetate 1g and add to solution of NaHSO₃ Na₂SO₃ H₂O 1.48, H₂O 1.8 cc H₂O + 2.4g dry KO₂ (0.95) and 5 cc H₂O. fit hot and solidifies at once. Stand 30 hrs. Filter off. got 0.9g crude salt. This is ~~very~~ soluble even in cold water. rexx 0.9g from 2 cc H₂O and got dark heavy needles up to 145° (try again). The body is slightly sol in cold ether and can be xx from hot C₆H₆. from which it comes down in needles -> analyzed for H₂O note top page

120° S. carbazon of Acetol obt from glycerin (see No. 65); Took fraction bpt 33°-40° at 10 mm (corr) and called Acetol and redistilled it [9 months old] again and got 1.5 gr bpt 45°-52° at 20 mm [or 38°-60° entirely] having yellow gum in d.f. on B.W.B. Treated this with 3g S. carbazon H₂O in H₂O cc H₂O + add 2g K₂CO₃: fits warm & soluble solid sep out. Let 8 (and 5 hrs for NO₂) analyzed for H₂O. got 1.25 grs bpt 195° (peculiar quick on stone) crude solid which rexx from H₂O gave 0.85 grs S. carbazon, deft 195° identical with S. carbazon from acetol (see 116°) as proved by mixing & determine deft. = 2000 at 42-70 (40 + 32 mm)

121° Took fraction bpt 50-70 at 10 mm (corr) see No 65 obt from deft of Glycerin: 9 months old and redistilled it: It boils 2/3 between 48°-60° at 18 mm [wt = 2.9 gr] and: probably mostly Acetol, leaving an oil which comes over in part between 60°-140° at 18 mm - Took the 2.9 gr obtained + treated with 3g H₂NOH, H₂O 3, K₂CO₃, and 10 cc H₂O + add oil. fits warm: cool tap slower 2 days. distill off H₂O in vacuo (60°) and ext to residue with ether (4x) (of No 117°). got a fair amt of oil, which boils between 100°-130° at 19 mm smells like Oxim of Acetol but could not get xx of oxim in impure.

122° The fraction bpt 80-90° at 10 mm (corr) = 12.9 gr from deft of glycerin 65 No: 9 months old was dissolved in 3 vols H₂O and ext 3x with ether and found that got about 2.3g oil to go from H₂O into ether as 2.3g oil bpt 100°-110° at 30 mm was obtained. The H₂O solution distilled in vacuo gave 7.5g oil bpt 94°-102° at 20 mm: proves that oil goes from H₂O into ether but only partially. Note also that the H₂O distillate contained about 2g oil carried over by H₂O: body is not entirely non volatile with H₂O. Took now of combined oil, 9.8 grs for following expts. a) the oil reduces Feh. sol:

(36)

b) see behavior towards Ph. hyd. and evening No 64
c) It does not react with S. Carbazone: took 1g bpt $94^{\circ}-102^{\circ}$ at 20 mm and
2g HCl S. carbonyl, 1.3g K₂CO₃ 6cc H₂O + let stand in cold 30 hrs [no precip] -
d) Took 7.6g of the oil and treated it with 7.6g H₂NOH, HCl, and 7.6g K₂CO₃ and
20g H₂O. no warming. let stand 2 days in cold and then distilled off H₂O in vacuo (60°)
and extract residue with ether (4x) and got fr. dist.
3.8g Oil bpt $83^{\circ}-110^{\circ}$ at 18 mm. unchanged and 14g Oil bpt $110^{\circ}-125^{\circ}$ at 18
which smells faintly of an oxime. no action with H₂NOH except with
traces of acetal present. 7.7g H₂O

123 Took fraction bpt $90-100^{\circ}$ at 10 mm (cor) [see descrip of gas. in No 65]
and redistilled it; got 7.7g bpt $93-98^{\circ}$ at 16 mm [with a thermometer looking
like No. 7]. Treated this with 7.7g H₂NOH, HCl, 7.7g K₂CO₃ 20g H₂O, no warming
let stand in cold 2 days. proceed. distilling off H₂O (60° at 25 mm) 1
and then extract 4x ether etc. got back 4.6g Oil bpt $89-110^{\circ}$ at
17 mm. mainly unchanged.

124 Took 3.8g oil bpt $83-110^{\circ}$ at 18 mm from 122 and 4.6g Oil bpt
 $89-110^{\circ}$ at 17 mm from 123 and distilled again. got 3.1g bpt $70-90^{\circ}$
and from $98-100^{\circ}$ at 17 mm [for 5 after - run] and 4.8g Oil bpt
 $90-98^{\circ}$ at 17 mm [bpt $90-93^{\circ}$] main oil still reduces Fehling's
solution very markedly. Assumed body was Propylene glycol and
worked on this basis and found this incorrect. body can't be C₅H₁₀O₃ as later

Took all of Oil [3.1g + 4.8g = 7.9g] and added it slowly thru sep. funnel
to 15cc conc. Cp. H₂SO₄ kept in 7.14. temp inside varied from -8°
to 0° and noticed heating on every drop oil added. Solution turns deep
dark brown. [Propylene glycol dissolves with faint yellow color only at 0°]. Let
stand at 0° for 3 hrs and then pour on ice and neutralize acid by adding
220cc 10% NaOH keeping faintly acid at end. then distilled off 100cc H₂O
on oil bath: noticed that a very volatile strong smelling body came over with
the water which can be separated off by adding solid K₂CO₃. is this
the chief reaction product but was not investigated at this point
and worked on basis body was propylene glycol. The 22% solution
was not made slightly alkaline by trace NaOH and rest of H₂SO₄ nothing
distilled off in vacuo. Salt residue extracted with ether gave nothing
so Propylene glycol can't be there.
It now follows that body off in descr. of glycerine can't be
lactic aldehyd or acetal ether or propylene glycol. It is probably an
acetal condensation product of this acetal hydrate (??) see later
C₅H₁₀O₃ but acetal of glycerine C₆H₁₂O₃ or 3H₂O

The CH_3OH solutions of Acetol, about 200 gr, were kept in cool room for about 20 days (over Xmas) and the crystals of bis alcoholate sep off by filtering & then examined filtrate to see how Acetol had kept. It should be mentioned that in 1st expt described above (627) total Brn acetol used a dimolecular Acetol of bpt 113-120 at 18 mm which on standing 2 days goes over into thick π Acetol gum. Don't keep - never got this again. Also noticed that upon Acetol in CH_3OH solution forms maddening 2 drops of ep come HCl a x precip very quickly of bis Acetol alcoholate? if want it again with pure material
 Dec 31st filtered off 32 g more bis alcoholate & let filtrate stand till Jan 27th when filtered off 19.6 g more. Acetol bpt 40-50 at 17 mm & no condensation distilled in vacuo & got 22 grs. Acetol bpt 40-50 at 17 mm & no condensation product formed - appears as though the bis alcoholate is constantly coming slowly forming - & hence found H₂O as well as CH_3OH or put out the Acetol. (if cool)
 very little bis alcoholate was left in the CH_3OH solution of Acetol.

126° Mfr of $\text{CH}_3\text{COOH}-\text{CH}_2\text{Cl}$ and of Propylene oxides of allylchloride from allyl alcohol
 allyl alcohol could not be converted into propylene glycol by treatment with conc H₂SO₄ at 0° but allylchloride gives almost quant. allylchloride as follows: Took 50 g allyl alcohol in 7.M. leaving R.F. long and made allylchloride as follows: Took 50 g allyl alcohol in 7.M. leaving R.F. long and added 50 g PCl_3 slowly, this sep. funnel shaking and let stand 24 hrs and then distill being great care to cool well & have pieces of ice in receiver to take up HCl (much comes off) then wash oil with 100 cc H_2O & dry CaCl_2 . Big yield goes as well using 223 g allyl alcohol in one lot.
 Took 234 g allylchloride and added it to 713 g cp. H₂SO₄ in 7.M. leaving R.F. & shaking violently from time to time. The oil floats on acid but quickly goes into solution as fast as all dissolved inside of 2 hrs with deep brown coloration. After 4 hrs standing in 7.M. poured on ice and distill at once used w/ sep off black oil which sep off (about 70 cc). The total solution amounted to about 3 Ltrs. Distill off on oil bath and redistill the fractions again & treat as distilled with K_2CO_3 etc. Got finally 213 grs of oil which dried slowly - treated the 204 g in 2 lots with 280 gr coarsely powdered KOH in a d.f. heating gently at first and 105 grs Propylene oxyd which on redist. putty in 6 cc off from portion expt gave 101 grs Propylene oxyd bpt 36-45 clearing a small amt of water & a thick oil (?) much lighter possibly traces propylene glycol.

127° 127° Propylene oxyd tube unchanged but finally stops. Heat 10 hrs on B.W.B. and for some time rolls up tube unchanged but finally stops. Heat 10 hrs on B.W.B. and

128° Acetol and K_2CO_3 . Took 11.9 g Acetol and 1.1 g K_2CO_3 in cold water night: gets yellow next day added 1/2 gr more K_2CO_3 and let stand 2 days. Then dist off H₂O directly in vacuo (bath 40°) and got 18.2 g H₂O 18.25 and the rest 5.5 g oil bpt 40-50 at 20 mm. unchanged Acetol. d.f. contains K_2CO_3 with only a slight amount of gum: no condensation to speak of. Took the H₂O 18.25 and the Acetol and added 2 g K_2CO_3 to 3 cc H₂O for mixing and heated S.T. 100° for 3 1/2 hrs. Found on distill. a thick brown gum (small amt & slowly) with very little acid. Did not look for acids formed.

The University of Chicago

129 Acetol about 3cc, and about 5cc conc sp. N₃ shaken about 2 days in cold & then boiled off in vacuum (17 mm). Got a thick brown oil bp about 100° at 17 mm. This better taste, is very thick, odorous & colorless in the pure state by adding to Et₂O but gives off N₃.

Acetolactate. Took 42g sp. N₃ 73-4 at 18 mm = 4 drops a minute & d.f. Worn & Bull then B.W. with

trans bitter taste, is very thick, or less viscous in the
 130 Extraction of Acetolacetate. Took 42g sp. Bpt 73-4 at 18 mm - 4 drops a minute
 over furnace at 430-450°. Time 5½ hours. Had d.f. Worm & BW + then BW with
 10% NaOH and pycnometer. No charring. St 1200 cc gas in 1st 2½ hrs - for burns
 like Co - after a time the NaOH showed signs of presence of acetaldehyde & smells
 like crotonaldehyde on disconnecting. Sppt 36 cc dist in d.f. only = brown liquid with
 strong odor like acrolein. Or better carefully sppt 53 cc yellow dist bpt 100-140°
 outside bpt 170°. On redistilling this boils from 30-70° 3 cc in dist 2 cc effy from
 85-100°. a mixture of acetaldehyde with acrolein crotonaldehyde? kept these oils
 in dense in dark for 20 days you need not get only 1.6 cc vol. on B.W.B. with strong odor -
 this added to 5 cc of alc. HBr gave in separation of mostly acetic acid bpt 71-80°
 not likely body was acrolein. gave 7.6 g bpt 50-71° at 22 mm and 21 g bpt 71-80°
 Residual oil distilled in vacuo gave 3.25 g crude carbonyl or xx H₂O
 leaving about 2.5 cc brown residue in d.f. (temp 160°). This oil 3 g treated
 with 10 cc 27% K₂Cr₂O₇ gave 3.75 cc + no gave 3.25 g crude carbonyl or xx H₂O
 unchanged Acetolacetate.
 1 - 1 with ether

Residual oil
at 21 mm leaving about 2.5cc
with 5.2g s. carbon
got 2.25g
took total oils
dry K_2CO_3 syst. finally
1310) Acrolein refer
best method finally was to use 250g em. glyc 625 fused $KHSO_4$ powder sheet in 4L
flask quickly & leave PBO in necks to clamp 502 at house & to cool well &
distill the dist on 12w. B not filtering. got 67g Acrolein bp 57-55 (or 40-60)
2g s. carbon. not evidently in 7. white but no precip of carbon
Acrolein 2g s. carbon. not evidently in 7. white but no precip of carbon
distilled by dist off the oil.

132 Akroling except 0.2 g white wool solid by dist off H₂O etc.
comes out (see no) have been isolated by dist off H₂O etc.
may be very sol. in H₂O & could have been isolated by dist off H₂O etc.
ethyl alcohol heated at 50°C for 5 days
11.5 cc + 3.5 cc ethyl alcohol heated at 50°C for 5 days
the sin 3 days cold acetone smell all gone.
ether ex, wash 5 x H₂O dry Cal 2

comes out
may be very sol. in H₂O than
133° Acrolein 11.5 cc + 3.5 cc ethyl alcohol used in
of new distillate came c.p. H₂O in 3 days in cold acetone solution
change. as it was washed with ether 5 x H₂O & dry each 2
Pon who H₂O cont'd NaOH & wash with ether 8x, wash 5 x H₂O & dry each 2
got 8.5 g CH₂-CH(OH)-CH₂-CO₂H bpt 80-83° at 23 mm with slight foaminess.
The alkaline ag. solutions for H₂O H₂O giving probably CH₂-CH(OH)-CH₂-CO₂H did not work up
with water containing free H₂O H₂O added to 15 cc H₂O (1.49) in F.M. gives solution and on standing
the alkaline ag. solutions for H₂O H₂O added to 15 cc H₂O (1.49) in F.M. gives solution and on standing
the alkaline ag. solutions for H₂O H₂O added to 15 cc H₂O (1.49) in F.M. gives solution and on standing

1340 Aerolein 5g added to 15cc HBr (1.49) in F.M. [the amt in 6 hrs].
 got semi solid green of sweet odor [the amt in 6 hrs].
 If use dry HBr to 8.5 cc aerolein. in F.M. till increase in wt was 11.3g got
 a thick oil which 5cc remains insoluble on adding 10cc H₂O + shake -
 water solution on test in vacuo showed a brown body present. To get the desired
 Brown proportional (dry) proceed but as shown late using alc HBr acc to
 woul. cf No 136

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(1350) Acroline + Phenylhydrazine see to Fischer. Cystine gave no def. results
i.e. could not Cystine's body. Fischer's body did not crystallize. As to
purity of acroline was but is evidently not accepted for identifi. of acroline in a
mixture.

136 Aerolin + alc HBr: found 160cc absol. alc about 200 dry HBr in 7.M. Aeroline had increased enormously. Added 45g ap. Aerolin (KHSO₄ melted) to flask containing 108cc of absol alc HBr in 7.M. and an oil sep out at once which contains for 1 lin. The upper layer on adding more alc. HBr gave much more oil again. ∴ probably advantageous to use 3x Volume of aerolin taken. Got in all about 40g acetal chloride (CH₃COCH₂Cl). This boils at about 80°-90° at 20 mm. The HBr is removed by adding NaHCO₃ at first when pour oil into conc. sol. of NaHCO₃ + ex. ether etc. To convert this into β-Bromopropion aldehyde but proceed as follows. Take oil and put with eq. wt of water & wash freely specially 5 minutes on 2 W.B. when get complete solution. At end action is violent & solution gets dark ∴ probably best not to heat. The solution now distilled in vacuum & neutralize HBr by NaHCO₃, then distill - resulting solution at 18 mm 40-45° slngpr. HBr by NaHCO₃, 200 H₂O spat at 10-15° H₂O 40-45° with

by using 80% CH_3OH (warms up) and a definite pinch NaOH was used
but on fresh in vacuo for a definite pinch NaOH was used
200 ml of Propylaldehyde: made in all 530 grs from n. Propylalcohol 2 kilos
It boils 48-50°

137° mfg of mfg
acc to M. ...
10 National day from 1961 to 1980
... 1980 ... 2.2 g ...

S. carbazyl of Purpuraceae group
rec 138° - at once giving an oil which even solid. (C₆H₅) comes out in need less
quite soluble in cold water; brist xx from cell No 149
melt 102° got 135 g. cp. w/it axes activity and add 2 drops conc H₂O₂
Gschl.: took 5 gpt 48-60% impure) and add 2 lbs act. and

(390) Parafronolalepis: 100% OK.
Worms up some when left O.T. keep 2 1/2 hrs in 4% NaOH. 28 grs for 66 at 17 mm: OK.
old H. 1.5 cc. 4.5 cc. 5.0 cc. 5.5 cc. 6.0 cc. 6.5 cc. 7.0 cc. 7.5 cc. 8.0 cc. 8.5 cc. 9.0 cc. 9.5 cc. 10.0 cc. 10.5 cc. 11.0 cc. 11.5 cc. 12.0 cc. 12.5 cc. 13.0 cc. 13.5 cc. 14.0 cc. 14.5 cc. 15.0 cc. 15.5 cc. 16.0 cc. 16.5 cc. 17.0 cc. 17.5 cc. 18.0 cc. 18.5 cc. 19.0 cc. 19.5 cc. 20.0 cc. 20.5 cc. 21.0 cc. 21.5 cc. 22.0 cc. 22.5 cc. 23.0 cc. 23.5 cc. 24.0 cc. 24.5 cc. 25.0 cc. 25.5 cc. 26.0 cc. 26.5 cc. 27.0 cc. 27.5 cc. 28.0 cc. 28.5 cc. 29.0 cc. 29.5 cc. 30.0 cc. 30.5 cc. 31.0 cc. 31.5 cc. 32.0 cc. 32.5 cc. 33.0 cc. 33.5 cc. 34.0 cc. 34.5 cc. 35.0 cc. 35.5 cc. 36.0 cc. 36.5 cc. 37.0 cc. 37.5 cc. 38.0 cc. 38.5 cc. 39.0 cc. 39.5 cc. 40.0 cc. 40.5 cc. 41.0 cc. 41.5 cc. 42.0 cc. 42.5 cc. 43.0 cc. 43.5 cc. 44.0 cc. 44.5 cc. 45.0 cc. 45.5 cc. 46.0 cc. 46.5 cc. 47.0 cc. 47.5 cc. 48.0 cc. 48.5 cc. 49.0 cc. 49.5 cc. 50.0 cc. 50.5 cc. 51.0 cc. 51.5 cc. 52.0 cc. 52.5 cc. 53.0 cc. 53.5 cc. 54.0 cc. 54.5 cc. 55.0 cc. 55.5 cc. 56.0 cc. 56.5 cc. 57.0 cc. 57.5 cc. 58.0 cc. 58.5 cc. 59.0 cc. 59.5 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Aldol of Propionaldehyde: Add H₂O: fits quite hot for a few seconds temp rose to 60 but mostly
in a flask cooled by ice H₂O: fits quite hot for a few seconds temp rose to 60 but mostly
+10 - fits very thick + viscous & slightly yellow. After 20 minutes add ether + then?
water die 18-20 3.4 g Aldol bpt 110°-120° at 20 mm -
Another expt with 5g crude aldol and aqueous K₂Cr₂O₇ shows that when used 5g K₂Cr₂O₇ and
5g H₂O or 10 grs H₂O than any slight action takes place in cold on standing 24 hrs or more
but if we use 15 grs H₂O to 5 grs K₂Cr₂O₇ quant. conv. to Aldol also takes place on shaking
used in 24 hrs. got finally 2.3 g pure aldol (some spelt) ∴ O.K.

141) mfg of α Bromo propional aldehyd: Took 20g crude aldehyd bpt $48^{\circ}-60^{\circ}$ and 20g Essacy and 80g water and hwr 17, then all 50g (1 mol) Br₂ thru sep. funnel keeping temp at 43° . The Br drops at once & solution ft and is colorless (20 minutes). Add 20 cc H₂O & dry Na₂CO₃ until almost neutral. no oil sep out ∴ distill at. on oil bath - the 1st distillates the same over, I = 19g less spec. grav. 1.524 at 25° } dried these with CaCl₂ until # 17g " " " 1.42 at 25° } no more H₂O sep off + got after 4 hrs drying 27g bpt $29^{\circ}-40^{\circ}$ at 35 mm mostly $29-31^{\circ}$ Oil with terrible action on eyes. It boiled at. as follows: I $90^{\circ}-103^{\circ}$ at 15 mm II and 18.25 grs $105^{\circ}-115^{\circ}$ & is unquestionably mainly α Bromo propional aldehyd.

142) mfg of Lactic aldehyd: prelim. expt. Took the 24.25 crude α Bromo prop. aldehyd (from 141) and hwr it with 16g Naformiate xx and 20g EtOH R.T. for 20 hrs & then work up as acetol. i.e. distill off on 200 B.P. & then take up in ether & filter NaBr. Distill off ether and got 10.6g Oil bpt $40-70^{\circ}$ (outside temp 160) leaving small amt gum in df. Oil smells of bromaldehyd ∴ lost gain with 4.3g EtOH and 3grs Naformiate 8 hrs $90^{\circ}-100^{\circ}$ and 7 hrs at $115^{\circ}-130^{\circ}$. Odor of Brom aldehyd now practically gone + on next got 5g Oil bpt $40^{\circ}-55^{\circ}$ at 15 mm or redist 4.5g bpt $45^{\circ}-60^{\circ}$ cfly $50-53^{\circ}$ at 13 mm. Still has very faint odor of Brom aldehyd - and seems much more stable than Acetol.

143) Dissoc of Chloroacetone. Took 60g capk bpt $117^{\circ}-119^{\circ}$ at rate of 4 drops a minute over furnace at $440^{\circ}-450^{\circ}$. Time $\frac{1}{2}$ 5 1/2 hrs. df, worm, BW, BW with NaOH & agt - got 21.00 cc gas in first 3 hrs if gas inflame with green tinged flame. Much HCl in apparatus. in 1st 2 hrs and warm then sucking back of NaOH. Much HCl in apparatus. got in d.f. 35.1g brown Oil. Bpt at. up to 110° = 74.5g and 27.2g bpt $110^{\circ}-120^{\circ}$ Redist. over 4g bpt $50^{\circ}-75^{\circ}$ The first $110^{\circ}-120^{\circ}$ color on standing but seems to be unchanged Chloroacetone. The lower boiling oil has acrolein smell but not certain it is this body: more Acetol. distillates. This is chief mixture. Also indication that Acetone is present (perhaps this is chief product). The oil 4g is quite soluble in water (gets warm) and on 200 B.P. Supr 2cc Oil bpt $53^{\circ}-70^{\circ}$ cfly $53^{\circ}-62^{\circ}$ = 1.5 cc. This dissolves in H₂O & prep by K₂CO₃

144) Took 50g Oil about out H₂O from CaO carefully hwr using & clean dropper Oil over at temp $430-450^{\circ}$. Time 3 hrs. Got 6 L. of inflame gas bpt $58-70^{\circ}$ + drying K₂CO₃ like H₂ in 1st 2 1/2 hrs - had 2 d.f. lost one in a 7. m. got 5.6g oil in 1st and 7.6g in 2nd d.f. which distilled over 10 cc bpt $58-60^{\circ}$ with smell of pure acetone. the CaO furnace was not examined but may have pyrolytic acid present (?).

2nd. Redace Feb. 80 on boiling (4/2)

It has a faint camphor like smell esp. in boiling. from other 6 H₂O Went

145° The bis acetol methylalcohol xx beautifully and Benzol (cf 125°). It dissolves but does not react with Phenylhydrazine on B.W.B. It does not react in cold with H₂NO₂ fze. Took 2g xx and 2 mol of H₂NO₂ H₂O + eq. wt K₂CO₃ + water 75cc in all to necessary to keep the bis alcohol in solution. not hot sharp? Calons heat xx exam.

146° bis acetol methylalcohol + 2% H₂O quant conversion into Acetol. Took 10g bis alcohol + added to a sol. of 50g H₂O + 3.1g of conc H₂SO₄ & heated in S.T. 15 minutes on B.W.B. Complete change solution is faintly yellow. and 2.4g K₂CO₃ added till just faintly alkaline & then distill off H₂O etc in vacuo. for 57.9 g of residue no part boiling above 32° at 25 mm: Acetol & H₂O can't be separated easily. The residue in d.f. is K₂CO₃ with faint tinct of gum only. H₂O solution must contain all the Acetol. Added it 33g solid K₂CO₃ & 5x with ether etc but got only 2cc oil left after distilling off & this done (0.8g only) between 50-60° at 25 mm = Acetol leaving about same amt solid gum in d.f. This residue with 1.6g S. carbonyl H₂O 8cc H₂O + 1g K₂CO₃ gave 0.4g crude S. carbonyl or xx H₂O got 0.33g of. flang Nadeln depth 200°. no question of conversion into Acetol but can't get Acetol sep from H₂O & xx it from H₂O by K₂CO₃ & ether. And quant. or ext. it from H₂O by K₂CO₃ & ether.

Proof that conversion is quant. & other. Took 10g bis alcohol, 50g H₂O + 3.1g of conc H₂SO₄ & heated on B.W.B. for 10 min (K.F.) & let cool. All changed: add 2g K₂CO₃ powder & heat & filter. Filtrate = 60cc (no H₂O added) Took of this 10cc and added to it 2g S. carbonyl H₂O (it dissolves on gentle warming) & then 1.3g K₂CO₃ solid: all gets solid. Filter got 2.1g crude S. carbonyl - which xxx from 25cc H₂O gave 1.8g of S. carbonyl depth 197° beaut. flang Nadeln: O.K. Tried to get out Acetol from remainder of solution (50cc) by adding 40g H₂O & got 33g dist mostly H₂O but some Acetol fused cells & distilling in vacuo. see of this gum on adding 1g S. carbonyl H₂O & then in it as found by following: 0.7g K₂CO₃ only 0.17g S. carbonyl precip. B.W.B. at 30 mm to draw the CaCO₃ residue in d.f. is yellow heated B.W.B. at 30 mm to draw off Acetol & probably containing much Acetol gum. ∴ N.G.

147° An expt. with 10g bis alcohol and 30cc conc H₂SO₄ at -10° gave deep brown solution but nothing definite on working up after 1 hour by pouring on ice adding NaOH solid etc. 2 expts. In 1st exp used a dropping funnel to let in oxyd

148° distill. of Propylbenzoyl 2 expts. In 1st exp used a dropping funnel to let in oxyd but broke to distill in the oxyd from a d.f. as can't regulate well otherwise. 1st exp. drop. funnel, pneumatic lamp, a d.f. (2nd exp in ice H₂O) & then a worm & B.W.B. then a safety bottle & got 35-37° for 1.2 L. of inflaming gas. Total dist = 16.3g used 20g Propylbenzoyl & 1.2 L. of inflaming gas. Temp 450°. from 37-45° a small amount smelling strongly of pure propylaldehyde. This boils from 37-45° (a small amount) from 45-57° and about 1.5cc not volatile on B.W.B. app. 1.4g oxyd & 5.2g pure fresh. 2nd exp same app. except distill in fused 14.8g from 1st exp + 5.2g pure fresh. Temp = 480-505: here 4hrs & got 5.4 L. of gas in 1 1/4 hrs - (not measured after words)

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about $\frac{1}{3}$ of dist is at an worm still $\frac{2}{3}$ in d.f. $\frac{1}{3}$ to bal amt = 8.5 grs it but
 8 grs from 42°-60° (fly 42°-50°) leaving small amt new in d.f. in B.W.B. (most
 heated the 8 grs oil with 20 cc H₂O in a S.T. for 13 hrs at 100° B.W.B. Kek (most
 of it dissolved in cold but some oil floats on top). Distill in B.W.B. 87.5 cc left
 much dried 2x each 2 grs H₂O then dist got 3 cc left 48°-60° B.W.B. 87.5 cc left
 in d.f. + not over by heating to 160° bath (?? purified)
 3 cc left 50°-56° leaving small of pure propyl ald chyd (or residue left in d.f.).
 got 2 grs propyl ald chyd on working up H₂O solutions & bpt 94°-8° at 15 mm -
 The 3 cc oil obtained was allowed to stand 14 hrs with 38 K₂CO₃ in 9 cc H₂O to get
 quant conversion into aldol but most small as the 2 cc oil sep off on top gave an
 dist unchanged aldol (3.4 cc) which may contain acetone but does not diss in aq. wt
 of H₂O. Therefore that main product is propyl ald chyd but not certain regarding
 absence of ketone.
 3rd expt using s. carbon method to sep acetone + aldol (see 149°)
 20 grs ap. oxyl of propyl ald used; lamp 48°-50° (fly 50°); time 2 hrs. got 7.3 L.
 of gas - 12 cc dist in worm in d.f. which mixed at once with 25 cc H₂O then
 heated in a S.T. for 7 hrs at 100° B.W.B. Kek. Open tube after reboils, yellow liquid in top
 + distill off in B.W.B. - got 7.5 cc distillate 1 cc = H₂O + 6.5 cc a layer of aldol
 containing H₂O. An redist this on
 acetal (?) + propyl ald hydrate (leaving residue in d.f.). This was in medium
 poured into a sol of 8 grs S. carbon H₂O in 19 cc H₂O + 5.2 grs K₂CO₃. An oil
 sep out at first which soon solidifies. After 40 hrs filter off precip which
 dried ep. in vacuum residue degraded with Benzol gave on soap of Benzol
 (slight smell) 1.55 grs xx scales mpt 85°-90° = Propyl ald S. carbon
 The precip 1.55 grs was only slightly sol in benzol: must be acetal S. carbon sly.
 boiled it out with 20 cc C₆H₆ + got 1.3 grs insol salt mpt 170°-180°: This diss
 in 6 cc H₂O gave fine xx needles 0.75 grs mpt 86° unscharf: O.K. The H₂O
 filtrate on standing over night gave more xx needles. Rexx the part sol in Benzol
 0.12 grs mpt 80°-110°: propyl ald S. carbon + 0.12 grs from about 10 cc boiling Benzol
 and obtained from H₂O solution 1.55 grs + 0.12 grs mostly about 92° = Propyl ald S. carbon
 for 1 grs quant. needles mpt 90°-102°
 149° Quant sep. of ketone + Propyl ald by S. carbon; is only reliable method
 as ap. K₂CO₃ not reliable see above. But find it diff to get sharp mpt on
 ald. S. carbon as it xx with C₆H₆ when dissolved in H₂O set at
 85°-120° very when the deposit xx only after
 When xx from Benzol it melts anywhere from 85°-120° when the deposit xx only after
 once small of propyl ald of the H₂O solutions even when the deposit xx only after
 some standing: the body is quite sol. in the steamy from excess sol. on
 crys from H₂O or on soap from H₂O sol. mpt goes up to 145°-153° should say body melts
 at 153° but uncertain. The H₂O sol.

44
coaf in vacuo from S. carbazon mpt 83° - 100° when rexx from C₆H₆ again (needed)
The sublt comes out from H₂O in grain. flat needles. 1.35 gms
(mpt 150°): Rediss in 10 cc guv o after standing edays but on standing swap
in dish for 0.4 gms mpt 135°-145° the body is easily sol in hot benzene
+ can easily be separated from S. carbazon of Acetone which is not sol
in cold benzene.

Acetone S. carbazon mpt 187° is obt thus: on adding 2 1/2 cc acetone to a sol of 5 gms. carbazon in
in 12 cc H₂O add 3.25 gms K₂CO₃ 3 gts S. gms crude precip was left with 5 cc H₂O. This xx
from 30 cc H₂O gave 2.0 gms ap. S. carbazon mpt 187°: it is sol in C₆H₆ M, slightly in boiling C₆H₆
most in ether 3 others 870-880° 2 gms.
quant sep of acetone & propylaldehyd. Took 10 gms S. carbazon Mcl 24 cc H₂O 16.5 gms

K₂CO₃ added = mixture of 2 1/2 cc each of acetone and of propylaldehyd. sol
5.35 gms crude precip, not washed except with mother liquor. This cleared in WKB
with 80 cc Benzol & filtered hot guv on cooling 0.3 gms more precip. Total wash
precip = 3.55 gms. Wash xx from 20 cc H₂O guv 2.0 gms ap. Acetone S. carbazon
∴ much remains in H₂O. The Benzol filtrate guv in vacuo gave 0.9 gms
propylaldehyd S. carbazon mpt 85-90°: OK. Much S. carbazon of propylaldehyd
does not come down + is to be found in 2g. mother liquor by dist off

150° Dissolve. of Propylaldehyd: Took 26 gms bpt 76° at 21 mm. 100° from oxide
+ H₂O; dropped in this sep. funnel. Temp 500°; rate 4 drops a minute. Purance
d.f. in ice H₂O, warm + BW, a black oil. Benzol + acetone. Got 3.5 L. gas, Time
2 3/4 hrs; no charring. Got 20.8 gms on BW. mixed with 30 cc H₂O oil but 2-3 cc
distill this got 13 cc bpt 50-55° on BW. Took 5 cc of this oil
dissolve. Redistill in BW & got 11.5 cc bpt 52-57°. Took 5 cc of this oil
(small old smell) and add to it to a sol of 80 gms S. carbazon Mcl in 24 cc H₂O &
6.5 gms K₂CO₃. Got 1 1/2 gms crude precip. (washed in lig) which mpt. 90-100°.

night, benzol, warm, with 30 cc Benzol + acetone. The Benzene filtrate on evap.
at 230° + is chiefly K₂CO₃ which mpt 85-90°
guv 1.2 gms xx of S. carbazon, which mpt 85-90°
∴ can't be anything except S. carbazon. The H₂O filtrate mpt 85-90°
in vacuo gave 3.2 gms more S. carbazon of propylaldehyd. This with
-92° or 3.55 gms including what was found in C₆H₆ mother liquor 40 Acetone
12 gms obtained from precip. water 4.75 gms from 5 cc oil taken. D.E.D.

150° Dissolve. of Glycid 150° (cf 62°) Temp 450°; glycid = 40 gms. bpt 74-5° at 30
this sep. funnel. much charcoal in tube. lead to stop before all glycid available
was left up (only 40 gms used). had d.f. in ice H₂O, worm + BW, viscosity not segot.
got 6.7 gms in 3 1/2 hours when let out escape. Time expt 5 hrs. Rate
4 drops a minute. Got 23 gms distillate nothing from C₆H₆ in worm tube
this distillate guv 9.5 cc bpt 29-45° on BW 13; distillate this with 18 cc H₂O guv
clear oil. next day distill guv got 6.75 cc bpt 24-28° which dried. Proved this
redistilled guv 5 cc bpt 22-30° (1/2 between 22-25°). Acetone sol. Proved this
by adding to this 1 drop conc H₂SO₄ got 2.7 cc para acetald bpt 115-21°: D.E.D.
vol. = 3.25 cc with CaCl₂. Got 1.1 gms bpt 85-90° with sharp small yellow oil (won from
on heating the oil left on BW. B. (after getting off 9.5 cc bpt 29-45°) to 115° in ball
got 1.1 gms bpt 85-90° (1/2 at 115°) in all 1.5 gms; dry CaCl₂ = H₂O) mpt
∴ undetermined but probably C₆H₅CHO

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The resid. oil heated in bath to 115° now distilled in vacuum gave
 2.7 grs bpt $40-55^\circ$ at 20 mm & 3.2 grs bpt $55-60^\circ$ at 20 mm. The
 residue in 1st now began to decompose (4.5 grs) & pressure goes up & it smells
 strongly of para formalin & the residue of Buick becomes covered with opaque
 fl formaldehyde. (bath 110°). Took 2 fractions (3.2 + 2.7 grs) & immediately got
 2 grs bpt $28-53^\circ$ at 22 mm & 2.4 grs bpt $53-70^\circ$ at 22 mm leaving a
 few drops of gum in 1st. on Buick at 17 mm; the lower fraction is chiefly H₂O with
 traces of acetone & gly. Took the 2.4 grs oil and added it to a solution of
 2 grs S. carbazil in 10 cc H₂O + 1.3 grs K₂CO₃. Got 1.1 grs crude S. carbazil which
 xx from 40 cc H₂O gave a small amount of flocc precip that fell off & then on
 further cooling got 0.55 grs of a small amount of flocc precip that fell off & then on
 noticed that the orig. mother liquors on standing gave much of same flocc precip
 on standing: this prob. S. carbazil and (prob) gly. (the precip is oil
 in traces in boiling H₂O). No acetone in: formed.

152) Dissoc. of Disposed ether (96). Took 32.1 grs freshly distilled bpt 105° at 17 mm -
 Temp 3 hrs. much charring.
 head a d.f. in ice then warm & Buick & 8 cc of Buick gave 3 cc oil bpt $45-100^\circ$
 got 19.7 grs dist in d.f. except (lines). This dist in bath to 150° gave 4.2 grs + per formal
 (some para formaldehyde) no trace in d.f. side (b.c.). The resid got 1.5 cc bpt $42-70^\circ$ + per formal
 old, too little to dist. This 1.5 cc oil has sharp odor & prob. is a mix of Acetone & acetone
 the part left in d.f. (bath 130°) now dist in vacuum. Got 12.6 grs oil bpt $80-115^\circ$
 at 95 mm leaving about 2 cc gum in d.f. Distillate has odor of acetone & then dist in
 vacuum the remaining yellow sol with sharp smell - got H₂O & then a slight
 bpt $170-260^\circ$ at 22 mm; main oil is a very thick gum bpt $260-261^\circ$
 at 22 mm = 8.4 grs: probably [CH₃OH - CH₃OH - CH₂CO] leaving only 2 grs gum in d.f.
 The H₂O solutions reduce Filings solution: add 2.5 grs S. carbazil in 6 cc H₂O
 & 1.7 grs K₂CO₃ slab stands. got no precip: conclude that little Acetol or
 acetol ether can be present. Dissoc. exp not very decisive.

153) Dissoc. of Propylaldehyde continued of 150. Reex the 4.75 grs total propylaldehyde S. carbazil
 from 30 cc benzene & got 3.5 grs needles which dist in vacuum well bpt $80-90^\circ$
 at 60° for 4 hrs in bath: still melts $83-95^\circ$: uncertain about C₁₁H₂₂XX (cf 149)
 (probably a trace of S. carbazil itself is present which melts at $80-85^\circ$)
 Took rest of oil obtained 6.5 cc bpt $62-75^\circ$ (may be due to by prod formal). Dist after distilling
 but was not successful (1/4 of 140) in exp. with K₂CO₃. Got after standing 30 min
 again: used 5 cc bpt $48-60^\circ$ of $45-52^\circ$ in exp. with K₂CO₃. Got after standing 30 min
 O.T. de 4 cc oil on Buick. only 1.5 cc bpt $80-140^\circ$ at 23 mm: not great conv. to acetol.
 - exp from only Propylaldehyde & acetone is formed

(for method: (46) details of exp. of 141°)
 154° 192g Na formate H₂O + 230g Box heating R.F. for 26 hrs. lumps up: filter & break
 up lumps & heat again 16 hrs R.F. rising with little CH₃OH, distill in vacuo after
 filtering fine precip & washing down. Got 92.2g oil bpt 40°-65° at 35-25 mm
 heavy white salt in d.f. on B.W.B. extract this with ether & redistill in open air
 B.W.B. after getting rid of ether - gave 8.7g more bpt 50-84° at 22 mm
 These oils all have faint smell of much Bromaldehyde & on redist next day found
 much less endense & after every distillation much less than this source. Did
 not succeed in fact out a pure product but exclude from subsequent expts that
 body obtained must have been Acetol. This' this was not pure Ac by comparison
 into a bis acetate or into a Castor. It is certain that this method is
 a poor one & don't give Lactic aldehyd but probably Acetol.

1550. Oxidation of Acetol by CuO & NaOH to Lactic acid.
 Took 20g bis acetol ex. distillate, 100cc H₂O & 6.2g ap. conc HCl heated R.F. for 10 min
 cool & add dil NaOH till neutral, then add H₂O to make up vol to 400cc; then add
 22.7g NaOH sp. in 68g H₂O [2½ mols on basis of acetol present] & immediately
 thereafter a sol of 56.6g CuSO₄ H₂O [1 mol] in 283g H₂O. Reduction to Cu₂O
 takes place instantly in cold. Decant at this point that a unitate has been made in
 clear filtrate from Cu. Decant and 2 mols Endry 5H₂O :: ½ ketol must still
 be left in solution. After 1 hour standing of hot filtrate :: cool and add a solution
 of 22.7g NaOH in 68g H₂O & sol of 56.6g CuSO₄ H₂O in 283g H₂O in cold
 precip at first greenish white but gets yellow quickly on warming on B.W.B. & shaking
 all Cu comes down from sol. in 1½ hrs heating (precip is broken). Filter hot & to
 dark yellow solution add in cold 35.7g ap. conc HCl (12.5g HCl calc as 35%)
 and this corresponds to 1½ x theory for all lactic acid that could be set free.
 Distill in vacuo vol of dist 110-130 cc & contains trace of HCl & by titrating for
 to Endry only 0.74g to make neutral. Extract residue in d.f. with cold ether
 & distill off ether heating mild oil in vacuo to 70° at 15 mm. Had 15.7g yellow
 oil left in d.f. only about ½ cc H₂O with traces HCl went off :: dissolve in new H₂O
 & heat with 12g zinc carbonate on B.W.B. An conc filtrate got 8.5g zinc leat.
 jump mother liquor 1.3g more pure zinc salt :: 13.8g zinc salt. Took
 xx from 170cc H₂O for 1.3g more pure zinc salt. 9.6g more pure salt :: 13.8g zinc salt.
 this all and rexx from about 200cc boiling H₂O got 9.5g air dried salt
 which analyzed (guar) after drying to constant wt 8½ hrs at 100-105°, weighed after standing
 9.3387g after drying to constant wt 8½ hrs at 100-105°, weighed on H₂O in vacuo
 7.6444g dry salt
 :: loss = 1.6943 = H₂O theory for 2 Zn (C₆H₁₀O₆) + 3 H₂O
 18.16

Found 18.14
 A zinc det. of a portion of dried salt gave 0.1598g Zn
 0.479g gave theory for Zn C₆H₁₀O₆ Found 26.81
 Zn = 26.89

156° Oxidation of Acetal to Lactic acid by aqueous $CuSO_4$, $5H_2O$.

Took 10 grs Acetal [bp 50°-60° at 18 mm] in $\frac{1}{2}$ L H_2O + added a solution of 3.38 grs $CuSO_4 \cdot 5H_2O$ (1 mmol) in $\frac{1}{2}$ L H_2O + heat R.T. for 10 hrs in oil bath (bath 130°). Cu separates out at once (no CO_2 or SO_2 formed). Solution is light brown + contains only traces of Cu. Neutralized with 16.3 grs $NaOH$ in H_2O , gas given showing traces of a heat B.W.B. of filter. Acet [35-40] = theory to get for all lactic acid + then distilled off H_2O in vacuo + ex. met salt residue with ether. After getting rid of ether + heating to 70° at 30 mm got 9.2 grs oily lactic acid which mixed with 9.3 grs pure carbonate $NaHCO_3$ (to get salt white) gave on concentration + treatment of some of filtrate with $CaCl_2$ to get salt white. The 10.4 grs $\pm$ of snow white Zn lactate + hydrolyzed as in 155 + then in a previous exp. used 20 grs in $CaCl_2$ + Zn lactate + hydrolyzed as in 155 + then made sol. up to 500 cc + worked up but got 6.1 grs + Zn lactate air dried. In this exp. more concaveing on working up but got 6.1 grs + Zn lactate air dried. A portion of this was 661 snow white for analysis (no Cu used). Results:

2.4038 grs salt air dried + 100°-105°	Theory for Zn ($C_6H_{10}O_6$) + $3H_2O$	Found
1.9675 grs at 100°-105°	18.15	
0.4363 grs H_2O	18.16	
	$H_2O = 0.1652$ grs Zn O	
	Found	
	Zn ($C_6H_{10}O_6$)	26.82
	Theory for Zn	26.89

0.4948 grs dry salt gave in ignition

notes made observation the follow. substances are not effective test. C_6H_5CHO , C_6H_5COOH , C_6H_5COCl , C_6H_5COBr , C_6H_5COI , C_6H_5COF , $C_6H_5COClO_2$, $C_6H_5COBrO_2$, $C_6H_5COIO_2$, $C_6H_5COF_2$, $C_6H_5COBr_2$, $C_6H_5COI_2$, $C_6H_5COF_3$, $C_6H_5COBr_3$, $C_6H_5COI_3$, $C_6H_5COF_4$, $C_6H_5COBr_4$, $C_6H_5COI_4$, $C_6H_5COF_5$, $C_6H_5COBr_5$, $C_6H_5COI_5$, $C_6H_5COF_6$, $C_6H_5COBr_6$, $C_6H_5COI_6$, $C_6H_5COF_7$, $C_6H_5COBr_7$, $C_6H_5COI_7$, $C_6H_5COF_8$, $C_6H_5COBr_8$, $C_6H_5COI_8$, $C_6H_5COF_9$, $C_6H_5COBr_9$, $C_6H_5COI_9$, $C_6H_5COF_{10}$, $C_6H_5COBr_{10}$, $C_6H_5COI_{10}$, $C_6H_5COF_{11}$, $C_6H_5COBr_{11}$, $C_6H_5COI_{11}$, $C_6H_5COF_{12}$, $C_6H_5COBr_{12}$, $C_6H_5COI_{12}$, $C_6H_5COF_{13}$, $C_6H_5COBr_{13}$, $C_6H_5COI_{13}$, $C_6H_5COF_{14}$, $C_6H_5COBr_{14}$, $C_6H_5COI_{14}$, $C_6H_5COF_{15}$, $C_6H_5COBr_{15}$, $C_6H_5COI_{15}$, $C_6H_5COF_{16}$, $C_6H_5COBr_{16}$, $C_6H_5COI_{16}$, $C_6H_5COF_{17}$, $C_6H_5COBr_{17}$, $C_6H_5COI_{17}$, $C_6H_5COF_{18}$, $C_6H_5COBr_{18}$, $C_6H_5COI_{18}$, $C_6H_5COF_{19}$, $C_6H_5COBr_{19}$, $C_6H_5COI_{19}$, $C_6H_5COF_{20}$, $C_6H_5COBr_{20}$, $C_6H_5COI_{20}$, $C_6H_5COF_{21}$, $C_6H_5COBr_{21}$, $C_6H_5COI_{21}$, $C_6H_5COF_{22}$, $C_6H_5COBr_{22}$, $C_6H_5COI_{22}$, $C_6H_5COF_{23}$, $C_6H_5COBr_{23}$, $C_6H_5COI_{23}$, $C_6H_5COF_{24}$, $C_6H_5COBr_{24}$, $C_6H_5COI_{24}$, $C_6H_5COF_{25}$, $C_6H_5COBr_{25}$, $C_6H_5COI_{25}$, $C_6H_5COF_{26}$, $C_6H_5COBr_{26}$, $C_6H_5COI_{26}$, $C_6H_5COF_{27}$, $C_6H_5COBr_{27}$, $C_6H_5COI_{27}$, $C_6H_5COF_{28}$, $C_6H_5COBr_{28}$, $C_6H_5COI_{28}$, $C_6H_5COF_{29}$, $C_6H_5COBr_{29}$, $C_6H_5COI_{29}$, $C_6H_5COF_{30}$, $C_6H_5COBr_{30}$, $C_6H_5COI_{30}$, $C_6H_5COF_{31}$, $C_6H_5COBr_{31}$, $C_6H_5COI_{31}$, $C_6H_5COF_{32}$, $C_6H_5COBr_{32}$, $C_6H_5COI_{32}$, $C_6H_5COF_{33}$, $C_6H_5COBr_{33}$, $C_6H_5COI_{33}$, $C_6H_5COF_{34}$, $C_6H_5COBr_{34}$, $C_6H_5COI_{34}$, $C_6H_5COF_{35}$, $C_6H_5COBr_{35}$, $C_6H_5COI_{35}$, $C_6H_5COF_{36}$, $C_6H_5COBr_{36}$, $C_6H_5COI_{36}$, $C_6H_5COF_{37}$, $C_6H_5COBr_{37}$, $C_6H_5COI_{37}$, $C_6H_5COF_{38}$, $C_6H_5COBr_{38}$, $C_6H_5COI_{38}$, $C_6H_5COF_{39}$, $C_6H_5COBr_{39}$, $C_6H_5COI_{39}$, $C_6H_5COF_{40}$, $C_6H_5COBr_{40}$, $C_6H_5COI_{40}$, $C_6H_5COF_{41}$, $C_6H_5COBr_{41}$, $C_6H_5COI_{41}$, $C_6H_5COF_{42}$, $C_6H_5COBr_{42}$, $C_6H_5COI_{42}$, $C_6H_5COF_{43}$, $C_6H_5COBr_{43}$, $C_6H_5COI_{43}$, $C_6H_5COF_{44}$, $C_6H_5COBr_{44}$, $C_6H_5COI_{44}$, $C_6H_5COF_{45}$, $C_6H_5COBr_{45}$, $C_6H_5COI_{45}$, $C_6H_5COF_{46}$, $C_6H_5COBr_{46}$, $C_6H_5COI_{46}$, $C_6H_5COF_{47}$, $C_6H_5COBr_{47}$, $C_6H_5COI_{47}$, $C_6H_5COF_{48}$, $C_6H_5COBr_{48}$, $C_6H_5COI_{48}$, $C_6H_5COF_{49}$, $C_6H_5COBr_{49}$, $C_6H_5COI_{49}$, $C_6H_5COF_{50}$, $C_6H_5COBr_{50}$, $C_6H_5COI_{50}$, $C_6H_5COF_{51}$, $C_6H_5COBr_{51}$, $C_6H_5COI_{51}$, $C_6H_5COF_{52}$, $C_6H_5COBr_{52}$, $C_6H_5COI_{52}$, $C_6H_5COF_{53}$, $C_6H_5COBr_{53}$, $C_6H_5COI_{53}$, $C_6H_5COF_{54}$, $C_6H_5COBr_{54}$, $C_6H_5COI_{54}$, $C_6H_5COF_{55}$, $C_6H_5COBr_{55}$, $C_6H_5COI_{55}$, $C_6H_5COF_{56}$, $C_6H_5COBr_{56}$, $C_6H_5COI_{56}$, $C_6H_5COF_{57}$, $C_6H_5COBr_{57}$, $C_6H_5COI_{57}$, $C_6H_5COF_{58}$, $C_6H_5COBr_{58}$, $C_6H_5COI_{58}$, $C_6H_5COF_{59}$, $C_6H_5COBr_{59}$, $C_6H_5COI_{59}$, $C_6H_5COF_{60}$, $C_6H_5COBr_{60}$, $C_6H_5COI_{60}$, $C_6H_5COF_{61}$, $C_6H_5COBr_{61}$, $C_6H_5COI_{61}$, $C_6H_5COF_{62}$, $C_6H_5COBr_{62}$, $C_6H_5COI_{62}$, $C_6H_5COF_{63}$, $C_6H_5COBr_{63}$, $C_6H_5COI_{63}$, $C_6H_5COF_{64}$, $C_6H_5COBr_{64}$, $C_6H_5COI_{64}$, $C_6H_5COF_{65}$, $C_6H_5COBr_{65}$, $C_6H_5COI_{65}$, $C_6H_5COF_{66}$, $C_6H_5COBr_{66}$, $C_6H_5COI_{66}$, $C_6H_5COF_{67}$, $C_6H_5COBr_{67}$, $C_6H_5COI_{67}$, $C_6H_5COF_{68}$, $C_6H_5COBr_{68}$, $C_6H_5COI_{68}$, $C_6H_5COF_{69}$, $C_6H_5COBr_{69}$, $C_6H_5COI_{69}$, $C_6H_5COF_{70}$, $C_6H_5COBr_{70}$, $C_6H_5COI_{70}$, $C_6H_5COF_{71}$, $C_6H_5COBr_{71}$, $C_6H_5COI_{71}$, $C_6H_5COF_{72}$, $C_6H_5COBr_{72}$, $C_6H_5COI_{72}$, $C_6H_5COF_{73}$, $C_6H_5COBr_{73}$, $C_6H_5COI_{73}$, $C_6H_5COF_{74}$, $C_6H_5COBr_{74}$, $C_6H_5COI_{74}$, $C_6H_5COF_{75}$, $C_6H_5COBr_{75}$, $C_6H_5COI_{75}$, $C_6H_5COF_{76}$, $C_6H_5COBr_{76}$, $C_6H_5COI_{76}$, $C_6H_5COF_{77}$, $C_6H_5COBr_{77}$, $C_6H_5COI_{77}$, $C_6H_5COF_{78}$, $C_6H_5COBr_{78}$, $C_6H_5COI_{78}$, $C_6H_5COF_{79}$, $C_6H_5COBr_{79}$, $C_6H_5COI_{79}$, $C_6H_5COF_{80}$, $C_6H_5COBr_{80}$, $C_6H_5COI_{80}$, $C_6H_5COF_{81}$, $C_6H_5COBr_{81}$, $C_6H_5COI_{81}$, $C_6H_5COF_{82}$, $C_6H_5COBr_{82}$, $C_6H_5COI_{82}$, $C_6H_5COF_{83}$, $C_6H_5COBr_{83}$, $C_6H_5COI_{83}$, $C_6H_5COF_{84}$, $C_6H_5COBr_{84}$, $C_6H_5COI_{84}$, $C_6H_5COF_{85}$, $C_6H_5COBr_{85}$, $C_6H_5COI_{85}$, $C_6H_5COF_{86}$, $C_6H_5COBr_{86}$, $C_6H_5COI_{86}$, $C_6H_5COF_{87}$, $C_6H_5COBr_{87}$, $C_6H_5COI_{87}$, $C_6H_5COF_{88}$, $C_6H_5COBr_{88}$, $C_6H_5COI_{88}$, $C_6H_5COF_{89}$, $C_6H_5COBr_{89}$, $C_6H_5COI_{89}$, $C_6H_5COF_{90}$, $C_6H_5COBr_{90}$, $C_6H_5COI_{90}$, $C_6H_5COF_{91}$, $C_6H_5COBr_{91}$, $C_6H_5COI_{91}$, $C_6H_5COF_{92}$, $C_6H_5COBr_{92}$, $C_6H_5COI_{92}$, $C_6H_5COF_{93}$, $C_6H_5COBr_{93}$, $C_6H_5COI_{93}$, $C_6H_5COF_{94}$, $C_6H_5COBr_{94}$, $C_6H_5COI_{94}$, $C_6H_5COF_{95}$, $C_6H_5COBr_{95}$, $C_6H_5COI_{95}$, $C_6H_5COF_{96}$, $C_6H_5COBr_{96}$, $C_6H_5COI_{96}$, $C_6H_5COF_{97}$, $C_6H_5COBr_{97}$, $C_6H_5COI_{97}$, $C_6H_5COF_{98}$, $C_6H_5COBr_{98}$, $C_6H_5COI_{98}$, $C_6H_5COF_{99}$, $C_6H_5COBr_{99}$, $C_6H_5COI_{99}$, $C_6H_5COF_{100}$, $C_6H_5COBr_{100}$, $C_6H_5COI_{100}$, $C_6H_5COF_{101}$, $C_6H_5COBr_{101}$, $C_6H_5COI_{101}$, $C_6H_5COF_{102}$, $C_6H_5COBr_{102}$, $C_6H_5COI_{102}$, $C_6H_5COF_{103}$, $C_6H_5COBr_{103}$, $C_6H_5COI_{103}$, $C_6H_5COF_{104}$, $C_6H_5COBr_{104}$, $C_6H_5COI_{104}$, $C_6H_5COF_{105}$, $C_6H_5COBr_{105}$, $C_6H_5COI_{105}$, $C_6H_5COF_{106}$, $C_6H_5COBr_{106}$, $C_6H_5COI_{106}$, $C_6H_5COF_{107}$, $C_6H_5COBr_{107}$, $C_6H_5COI_{107}$, $C_6H_5COF_{108}$, $C_6H_5COBr_{108}$, 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$C_6H_5COI_{142}$, $C_6H_5COF_{143}$, $C_6H_5COBr_{143}$, $C_6H_5COI_{143}$, $C_6H_5COF_{144}$, $C_6H_5COBr_{144}$, $C_6H_5COI_{144}$, $C_6H_5COF_{145}$, $C_6H_5COBr_{145}$, $C_6H_5COI_{145}$, $C_6H_5COF_{146}$, $C_6H_5COBr_{146}$, $C_6H_5COI_{146}$, $C_6H_5COF_{147}$, $C_6H_5COBr_{147}$, $C_6H_5COI_{147}$, $C_6H_5COF_{148}$, $C_6H_5COBr_{148}$, $C_6H_5COI_{148}$, $C_6H_5COF_{149}$, $C_6H_5COBr_{149}$, $C_6H_5COI_{149}$, $C_6H_5COF_{150}$, $C_6H_5COBr_{150}$, $C_6H_5COI_{150}$, $C_6H_5COF_{151}$, $C_6H_5COBr_{151}$, $C_6H_5COI_{151}$, $C_6H_5COF_{152}$, $C_6H_5COBr_{152}$, $C_6H_5COI_{152}$, $C_6H_5COF_{153}$, $C_6H_5COBr_{153}$, $C_6H_5COI_{153}$, $C_6H_5COF_{154}$, $C_6H_5COBr_{154}$, $C_6H_5COI_{154}$, $C_6H_5COF_{155}$, $C_6H_5COBr_{155}$, $C_6H_5COI_{155}$, $C_6H_5COF_{156}$, $C_6H_5COBr_{156}$, $C_6H_5COI_{156}$, $C_6H_5COF_{157}$, $C_6H_5COBr_{157}$, $C_6H_5COI_{157}$, $C_6H_5COF_{158}$, $C_6H_5COBr_{158}$, $C_6H_5COI_{158}$, $C_6H_5COF_{159}$, $C_6H_5COBr_{159}$, $C_6H_5COI_{159}$, $C_6H_5COF_{160}$, $C_6H_5COBr_{160}$, $C_6H_5COI_{160}$, $C_6H_5COF_{161}$, $C_6H_5COBr_{161}$, $C_6H_5COI_{161}$, $C_6H_5COF_{162}$, $C_6H_5COBr_{162}$, $C_6H_5COI_{162}$, $C_6H_5COF_{163}$, $C_6H_5COBr_{163}$, $C_6H_5COI_{163}$, $C_6H_5COF_{164}$, $C_6H_5COBr_{164}$, $C_6H_5COI_{164}$, $C_6H_5COF_{165}$, $C_6H_5COBr_{165}$, $C_6H_5COI_{165}$, $C_6H_5COF_{166}$, $C_6H_5COBr_{166}$, $C_6H_5COI_{166}$, $C_6H_5COF_{167}$, $C_6H_5COBr_{167}$, $C_6H_5COI_{167}$, $C_6H_5COF_{168}$, $C_6H_5COBr_{168}$, $C_6H_5COI_{168}$, $C_6H_5COF_{169}$, $C_6H_5COBr_{169}$, $C_6H_5COI_{169}$, $C_6H_5COF_{170}$, $C_6H_5COBr_{170}$, $C_6H_5COI_{170}$, $C_6H_5COF_{171}$, $C_6H_5COBr_{171}$, $C_6H_5COI_{171}$, $C_6H_5COF_{172}$, $C_6H_5COBr_{172}$, $C_6H_5COI_{172}$, $C_6H_5COF_{173}$, $C_6H_5COBr_{173}$, $C_6H_5COI_{173}$, $C_6H_5COF_{174}$, $C_6H_5COBr_{174}$, $C_6H_5COI_{174}$, $C_6H_5COF_{175}$, $C_6H_5COBr_{175}$, $C_6H_5COI_{175}$, $C_6H_5COF_{176}$, $C_6H_5COBr_{176}$, $C_6H_5COI_{176}$, $C_6H_5COF_{177}$, $C_6H_5COBr_{177}$, $C_6H_5COI_{177}$, $C_6H_5COF_{178}$, $C_6H_5COBr_{178}$, $C_6H_5COI_{178}$, $C_6H_5COF_{179}$, $C_6H_5COBr_{179}$, $C_6H_5COI_{179}$, $C_6H_5COF_{180}$, $C_6H_5COBr_{180}$, $C_6H_5COI_{180}$, $C_6H_5COF_{181}$, $C_6H_5COBr_{181}$, $C_6H_5COI_{181}$, $C_6H_5COF_{182}$, $C_6H_5COBr_{182}$, $C_6H_5COI_{182}$, $C_6H_5COF_{183}$, $C_6H_5COBr_{183}$, $C_6H_5COI_{183}$, $C_6H_5COF_{184}$, $C_6H_5COBr_{184}$, $C_6H_5COI_{184}$, $C_6H_5COF_{185}$, $C_6H_5COBr_{185}$, $C_6H_5COI_{185}$, $C_6H_5COF_{186}$, $C_6H_5COBr_{186}$, $C_6H_5COI_{186}$, $C_6H_5COF_{187}$, $C_6H_5COBr_{187}$, $C_6H_5COI_{187}$, $C_6H_5COF_{188}$, $C_6H_5COBr_{188}$, $C_6H_5COI_{188}$, $C_6H_5COF_{189}$, $C_6H_5COBr_{189}$, $C_6H_5COI_{189}$, $C_6H_5COF_{190}$, $C_6H_5COBr_{190}$, $C_6H_5COI_{190}$, $C_6H_5COF_{191}$, $C_6H_5COBr_{191}$, $C_6H_5COI_{191}$, $C_6H_5COF_{192}$, $C_6H_5COBr_{192}$, $C_6H_5COI_{192}$, $C_6H_5COF_{193}$, $C_6H_5COBr_{193}$, $C_6H_5COI_{193}$, $C_6H_5COF_{194}$, $C_6H_5COBr_{194}$, $C_6H_5COI_{194}$, $C_6H_5COF_{195}$, $C_6H_5COBr_{195}$, $C_6H_5COI_{195}$, $C_6H_5COF_{196}$, $C_6H_5COBr_{196}$, $C_6H_5COI_{196}$, $C_6H_5COF_{197}$, $C_6H_5COBr_{197}$, $C_6H_5COI_{197}$, $C_6H_5COF_{198}$, $C_6H_5COBr_{198}$, $C_6H_5COI_{198}$, $C_6H_5COF_{199}$, $C_6H_5COBr_{199}$, $C_6H_5COI_{199}$, $C_6H_5COF_{200}$, $C_6H_5COBr_{200}$, $C_6H_5COI_{200}$, $C_6H_5COF_{201}$, $C_6H_5COBr_{201}$, $C_6H_5COI_{201}$, $C_6H_5COF_{202}$, $C_6H_5COBr_{202}$, $C_6H_5COI_{202}$, $C_6H_5COF_{203}$, $C_6H_5COBr_{203}$, $C_6H_5COI_{203}$, $C_6H_5COF_{204}$, $C_6H_5COBr_{204}$, $C_6H_5COI_{204}$, $C_6H_5COF_{205}$, $C_6H_5COBr_{205}$, $C_6H_5COI_{205}$, $C_6H_5COF_{206}$, $C_6H_5COBr_{206}$, $C_6H_5COI_{206}$, $C_6H_5COF_{207}$, $C_6H_5COBr_{207}$, $C_6H_5COI_{207}$, $C_6H_5COF_{208}$, $C_6H_5COBr_{208}$, $C_6H_5COI_{208}$, $C_6H_5COF_{209}$, $C_6H_5COBr_{209}$, $C_6H_5COI_{209}$, $C_6H_5COF_{210}$, $C_6H_5COBr_{210}$, $C_6H_5COI_{210}$, $C_6H_5COF_{211}$, $C_6H_5COBr_{211}$, $C_6H_5COI_{211}$, $C_6H_5COF_{212}$, $C_6H_5COBr_{212}$, $C_6H_5COI_{212}$, $C_6H_5COF_{213}$, $C_6H_5COBr_{213}$, $C_6H_5COI_{213}$, $C_6H_5COF_{214}$, $C_6H_5COBr_{214}$, $C_6H_5COI_{214}$, $C_6H_5COF_{215}$, $C_6H_5COBr_{215}$, $C_6H_5COI_{215}$, $C_6H_5COF_{216}$, $C_6H_5COBr_{216}$, $C_6H_5COI_{216}$, $C_6H_5COF_{217}$, $C_6H_5COBr_{217}$, $C_6H_5COI_{217}$, $C_6H_5COF_{218}$, $C_6H_5COBr_{218}$, $C_6H_5COI_{218}$, $C_6H_5COF_{219}$, $C_6H_5COBr_{219}$, $C_6H_5COI_{219}$, $C_6H_5COF_{220}$, $C_6H_5COBr_{220}$, $C_6H_5COI_{220}$, $C_6H_5COF_{221}$, $C_6H_5COBr_{221}$, $C_6H_5COI_{221}$, $C_6H_5COF_{222}$, $C_6H_5COBr_{222}$, $C_6H_5COI_{222}$, $C_6H_5COF_{223}$, $C_6H_5COBr_{223}$, $C_6H_5COI_{223}$, $C_6H_5COF_{224}$, $C_6H_5COBr_{224}$, $C_6H_5COI_{224}$, $C_6H_5COF_{225}$, $C_6H_5COBr_{225}$, $C_6H_5COI_{225}$, $C_6H_5COF_{226}$, $C_6H_5COBr_{226}$, $C_6H_5COI_{226}$, $C_6H_5COF_{227}$, $C_6H_5COBr_{227}$, $C_6H_5COI_{227}$, $C_6H_5COF_{228}$, $C_6H_5COBr_{228}$, $C_6H_5COI_{228}$, C_6H

159 Propylene oxide 22.3 grs put in a flask cool in 7 M. + shake + pass over it dry HCl gas. at end 1 hr 17 grs increase in wt (Heavy = 14 grs) :: stopped + let stand in 7 M for 4 hrs: then add solid K_2CO_3 + 70 cc H_2O to fill rid of excess HCl + sep of 36.4 grs oil which dried K_2CO_3 + dist. oil. gave 19.2 grs bpt up to 129° = 32.1 grs 12.9 grs at $129-140^\circ$

leaving small amt residue in d.f. not vol. both 200% poly glycol. (Dried K_2CO_3)
 Repeat I + II separately but got no indication of presence of two isomers. Spt
 30.8 grs bpt $100^\circ-132^\circ$ effy $127^\circ-129^\circ$ of $CH_3CH_2CH_2CH_2CH_2CH_3$ and $CH_3CH_2CH_2CH_2CH_2CH_3$
 :: tried to oxid later see No. 159 by HNO_3 and by $H_2SO_4 + CrO_3$ see No.

160 Dissoc of Acetol and Water. Took 16.1 grs acetol bpt $50^\circ-60^\circ$ at 25-28 mm
 freshly distilled immediately dilute with 4 grs H_2O :: 20.1 grs mixture taken (eq. vol.)
 + dropped this over furnace sep funnel at rate 4-5 drops a minute.
 Time 2 hrs. Temp. $450^\circ-60^\circ$. had d.f. in ice H_2O , brown + B.W. safety B.W. + Agol.
 got 1100 cc gas last 300 cc inflame + like CO . No dist in worn.
 got 14.7 grs dist in d.f. slightly colored, app. 50% of aprot has smell of pure Acetaldehyde,
 with trace of crotonaldehyde. Heated dist on B.W. got 1 cc dist sol in eq vol
 of H_2O except trace yellow crotonaldehyde. Residue in d.f. dist in vacuo gave
 dist almost that of pure Acetaldehyde. Residue in d.f. dist in vacuo gave
 got 7.1 grs H_2O + Acetol bpt $25^\circ-28^\circ$ at 20 mm and 4.6 grs Acetol recovered bpt
 $50^\circ-60^\circ$ at 20 mm two cond. products analogous to glycol diss. expts.

→ Took these together and add 2 cc H_2O + 0.7 grs NaOH ap. + let stand. gets yellow
 after 20 hrs o.t. neutr. with dil HCl, make faintly alkaline by K_2CO_3 + let stand in vacuo
 (bath at end 140°). got all to boil over except 4 grs salt residue having some π Acetol gum.
 The distillate has mostly bpt of H_2O but a part 3.2 grs boils up to 50° at 20 mm -
 On redistillation of H_2O got run higher boiling oil :: put with 3.2 grs. str. fract.
 got 2.5 grs bpt $50^\circ-52^\circ$ at 24 mm or refract gave 2.3 grs bpt 49° at 20 mm
 this (2.3 grs) boiled with 4.6 grs S. carbonyl HCl, 3g K_2CO_3 + 12.5 cc H_2O - got
 3.4 grs crude S. carbonyl which xx from about 40 cc H_2O gave 3.06 grs ap. S. carbonyl 19%
 1.1 grs more S. carbonyl. Q.E.D.

The H_2O + formers gave with same amt S. carbonyl HCl + K_2CO_3 - 1.1 grs more S. carbonyl.
 :: It is certain that Acetol was unchanged in dissoc. exp. and subseq. treatment
 with NaOH did not change it to lactic acid. in cold but left

unchanged tho' a part went over to π Acetol-gum
 unchanged by H_2SO_4 and CrO_3 . Took 20 grs Acetol bpt $50^\circ-60^\circ$ at 18 mm

161 Oxid. of Acetol by H_2SO_4 and CrO_3 . Took 26.5 grs $K_2Cr_2O_7$ + 46.6 grs 95% H_2SO_4
 + dissolved in $\frac{1}{2}$ L H_2O + add this sep funnel a sol. of 26.5 grs $K_2Cr_2O_7$ + 5 H_2SO_4 i.e. 4 atoms of O for 1 mol Acetol
 in $\frac{1}{2}$ L H_2O calc on basis of $Cr_2O_3 + 3H_2O$ (294) + 5 H_2SO_4 (98) = 52 grs $K_2Cr_2O_7$ in 200 cc H_2O + filler from
 cool with tap for 1 hr + then let stand over night. Reacts slowly in cold + next day is
 all green. Heat B.W. + heat again adding a sol. of 52 grs $K_2Cr_2O_7$ in 200 cc H_2O + filler from Cr_2O_3
 again + then add 28.6 grs conc. (sp. HCl 35%) (= heavy for expts. 1 mol acetol used). distill off
 H_2O in vacuo + looked for acetic + 2 drops of tar. Ag. dist = 1000 cc contains only trace HCl + took
 3.88 grs NaOH to neutralize. Underp. neutr. sol bpt 8.2° Nasalts which with 10 cc ap. H_2SO_4 + 20 cc H_2O
 + treated with 800 cc H_2O and 13.5 grs Ag_2O (= 2 mols) gave on B.W. and filtering
 from AgCl. 5 grs of Agacetate (about eq. amt Ag).
 no trace of lactic!!

1650 Acetol + H₂O; Acetol made by hydrolysis of 18 gms bis-allyl alcoholate using 100 cc H₂O, 6.2 gms op H₂O, heating 10 minutes on B.W.B reactor, subseq. by dil NaOH and then add to freshly prep'd H₂O in about 2.5 L. H₂O. Made H₂O from 82.5 gms C₂H₅COOH = 1½ mols. H₂O. Saponified at 46 hours as it reacts at once. H₂O still left; heat B.W.B (much more reduction) can't filter: sat with HCl & soap filtrate in vacuo to small volume then treat with 15 gms fine carbonate & H₂O (250 cc); very little effervescence. Test filtrate with fine C & soap to dryness but got only very small amt. of very dark salt residue: no fine lactate & acetic + formic acid must be other products. ~~residue: no fine lactate & acetic + formic acid must be other products.~~

166° mfr of 2 Brown prop. 2.5 gr oil bpt 38°-50° at 27 mm
 Liebig 99.4 grs H₂O + 628 grs Br etc. 2.5 gr oil bpt 38°-50° at 27 mm
 Took this at once and treated in S.T. with 20 grs NaAmiate H₂O (Heavy 14.5 grs) + 5 grs X from
 S.T. 120°-140° for 8 hrs. Some charring. Took up c. ether, still gummy, sp. oil.
 Got 5.9 grs bpt 45°-60° at 27 mm 4.4 grs bpt 60°-100° at 27 mm 57 grs 120°-120° at 27
 with bath 100° at and leaving small amt (28) tar in d.f. Took all dist p/b (3 frs)
 & heat in S.T. 100° for 9 hrs with 15 grs CH₃OH. Bubble in vacuo 2 x got 2.35 grs
 oil with sweet smell. No gummy. Shews thin trace H₂O. bpt 50°-60° at 34 mm.
 Rest is C₁₃ or H₂O. There is left each time a gummy residue [1st time 1 cc 2nd time
 1/4 cc] looking like π Acetol gum. The oil was poured at once into a sol
 of 4 grs s. carb. in H₂O. 26 grs CO₂ and 10 cc H₂O. Reacts & gets solid at once. Got 2.7 grs
 crude salt which mxx from 37 cc H₂O from 2.1 grs brnt. xx needles easily, measure
 0.2986 for theory for C₄H₇O₂N₃ 36.64
 0.4026 gr CO₂ and 0.1863 gr H₂O. Found 36.77
 6.93
 gwm p. 16 paper
 all acetic gum standing in cold 0.75 gr
 10 cc acetol

The formers makes with Ph. hydr. dil acetic acid
 0.2956 gm theory 36.64
 C 6.87
 H 6.99
 The formers makes with Ph. hydr. dil acetic acid
 0.52 gm ref 146 of Q.E.D.
 1670 Bromacetophenone 24.4 gm bp 140° at 12 mm and 18 gm of acetate bcc abrd
 13.8 gm sp. Acetate bp 170°-20° at 29 mm. Ref. of
 other pure 8.5.100 for 9 hrs 13.8 gm sp. Acetate bp 170°-20° at 29 mm. Ref. of
 for 12 gm solid xx. This xx from ether (bucc) pure bcc abrd 10 gm and
 in every respect with a body obtained from benzyl carbinol 10 gm and
 acetic anhydride (10 gm) Ref. R.F. 9 hrs at 160° at 12 mm. Ref. of
 bp 144° at 12 mm. Ref. R.F. 9 hrs at 160° at 12 mm. Ref. of
 a specimen from 18% case ref remains 49°-50° ∴ unquest. identical &
 Benzyl carbinol must be $C_{13}H_{17}O$ and not $C_{13}H_{17}O$ - CH=O.
Bromacetophenone and Na formate alone + with ether present do not
form succars.

Bromacetoform
168° Sucrose
Gum and methyl butyrate gives Acetolactate and not the ar.

169° Acetol by direct acylation gives Acetolactate and not the ar.
Acetol 2.1 grs and acetic anhydride 3.2 grs let stand in cold 4 days and then
heat in St. 100° put back for 15 hrs give a dash in vacuo 1.3 grs left up to 40° at 13 mm
effly 30-35° = acetic acid and then give a few drops at 40-63°. Rest boils 2.3 grs between
63°-65° at 11 mm Benzyl gum 2.0 grs needles melt 138-40° An rest this from 12 grs 2x
precip - which mxx with 145° heat needles melt 138-40° An rest this from 12 grs 2x
(1/2 gr) left up to 145° heat needles melt 138-40° An rest this from 12 grs 2x
Succinyl benzyl gum Acetolactate from Acetone Benzyl gum it melt 138-40°
same B.E.D. analyzed for N see the analysis page

at 10 mm
of Benzoylcarbonyl mfr 600
171° mfrgr. of a Brm professional delyd: 50.8 grs mfrgr. in
139 grs Brz then sep funnel cooling with ice H₂O + oil and let warm up spm. in
30°-40° log. rid of last of Brz & then warm. Distill in vacuum bath using "fl.
a d.f. in ice water for receiver as dist often warms up & splashes in Brmhl.
I 35.5 cc oil = 57.9 grs (i.e. spgr. about 1.58) II 36 cc oil = 48.3 grs III 20.3 grs oil =
17.3 cc. Took I II III 91.8 cc + traces at once with fresh CaCl₂ shaking & cooling
under 2 layers appear sep of lower layer & dry it fresh CaCl₂ as well as upper
layer which usually gives little or no more heavy oil. Got 76 grs dry oil which with
20 grs oil from a previous expt gave 96 grs oil which dried & filtered gave a dist in
vacuum. I 48.6 grs bpt 42°-44° at 63 mm spgr. 1.523 at 21° [West phal]
II 14.9 grs 44°-51° " 60 mm } outside temp. at end bar. (Bar 73)
III 8.8 grs 51°-52° " 60 mm } leaving about 20 gr heavy in sol oil & expt
I 43.5 grs bpt 43°-5° at 60 mm i.e. poss. to I (not put in)
as shown by analysis but it is not poss. to heat without CaCl₂ to heat (both ways)

20 gr oil from 42 mm
I 48.6 gm
II 14.9 gm
III 8.8 gm

Refract II & III got 15.5 grs bpt 43-5 at 60 mm :: forced to I, (not put in)

The oils contain about 10% Essing as shown by analysis but it is not poss.
to remove them by redist. in much H₂O and extract (with or without CaCO₃ to react
acid) without much loss of Bromal. That hydrolysis can carefully treat both ways).

It was impossible to get acetic ald. acetate from this oil by treating in
about. ether sol with dry acetale S.T. But B 100° got only acetic acid (see March 11)
reaction products in these cases (2x very carefully tried with much material).
To accomplish this we had to make a 10% suspension of aldehydes (see below) either by
The above crude Bromal. can only be used to make Acetol either by
treatment with N-pinnate etc or by Kacetate + cis oil (see below) (178)

Analysis Fraction I above.

43.6 x 9 g (Carus)
16.62 g H₂O

Memo of 10% acetic
present =

The above
Treatment with K₂CO₃

Analysis of Ether (Caries)
O. 2962 grs
O. 1854 grs
d. 4304 grs

Found
C 26.28
H 3.65
Br 58.39

Nearly pure C₂H₅OBr
Heavy 16% Acetic
present =

Heavy 16% Acetic
present =

172 Naphthalene & Iodine
Apr 42-4 at 63 mm and found gets a sol. of 40grs KI (heavy 33grs) in 40cc H₂O & shock
in sep. funnel reacts once; bubble added 22grs more KI to H₂O solution after
2 hours sep. of heavy oil 14cc = 25grs diss in ether dry CaCl₂ & bottle in vacuo -
got 11.9 g oil bpt 40° at 12 mm brown color horrible in effects on eyes (leaving
much green in d.f.) Took oil and immediately sealed up in a tube with 20cc absolute
ether (heavy 11grs) after standing over night heat 30°C for 9 hrs. Some
and 15 grs dry kacetate (heavy 11grs) distill off ether & heavy oil in vacuo got 2.3 grs
press. in tube Take up in ether & Eiseberg got 2.6 grs oil bpt 50-53° at 18 mm (both 90°
on heating both to 120° main came over there is left in d.f. 2cc green
main fraction: 2.6 + .1 = 2.7g at 18 mm.

The oil of 1 has a sweet but faintly sharp odor: took 2 grs of it & poured into a sol of 4 grs S. carbonyl & 2.6 grs K_2CO_3 in 10 cc H₂O got large precip in short time on shaking. The max this (3.4 grs crude? wt) from 30 cc H₂O got 1.9 grs of light yellow needles. This was analyzed at 163°: H₂O sol. group spot gave 0.3 grs from 100 cc 160°-1°. This was analyzed and found to be lactic aldehyde acetate S. carbonyl.

Took rest of oil 1.7 grs & poured into H₂O: oil sinks & is only slightly soluble & has sweet fragrant odor. add 1 grs pure K_2CO_3 (H₂O = about 15 cc) & heat S.T. B.W.B. for 8 hrs. Filtrate 19 cc has sweet smell: add 2 grs S. carbonyl & 1.3 grs K_2CO_3 & heat light brk. got only 0.3 grs precip. which xx from 5 cc H₂O gave 0.23 grs shiny & sharp light brk. crystals. Had clear depts 195 (flow) & looking in every way like Acetol. S. carbonyl.

Mother liquor 19 cc (from 0.3 grs precip) was comp off in vacuo & residue ext. with Benzol. got nothing: has residue in 5 cc H₂O & 100 grm.

is brown and smells sweet (? Acetol grm).

173° infus of a brown & 1/3 acetol depts (Feb 24 03) O.K. took body & tried its action towards Nafumiate & little Heort. Na formiate & Acetolacetate in excess succeeded: N.G.

174° Tried to Hydrolyze Lactic al. acetate S. carbonyl & Acetolacetate in H₂O sol. S.T. 100° for 4 hrs: got precip, but not bodies desired.

175° Bromoacetone & Nafumiate [cf No 112] Took 70 grs Bromoacetone bpt 47°-50° S. carbonyl & Na formiate & 13 grs xx formiate & heated 9 hrs in a S.T. at 18 mm and 53 grs powder. Nafumiate H₂O and 13 grs xx formiate & heated it on B.W.B. by dist off 25.8 grs bpt 50°-65° at 20 mm.

B.W.B. Press. on perming. much charring: Wash ether & more it on B.W.B. by dist off Oil left from 1st run 2.9 grs bpt up to 50°: at 20 mm 15.4 grs bpt 65°-75° at 20 mm. Flowing 5 grs less in d.f. B.W.B. Shook I in ether: sol with K_2CO_3 aqueous (1/2) & got after nitroizing ether only 2-3 cc oil left leaving from full smell of Bromoacetone: discards.

Took fraction III & heated in S.T. with 30 grs CH_3OH B.W.B. for 9 hours. An dist. in vacuo (2X) got 1.9 grs bpt up to 40° at 15 mm & 3.4 grs oil bpt 40°-45° at 15 mm = Acetolacetate (got 0.9 grs in filling). Tried adding a drop of H₂O to this fraction which bis Acetolacetate (got 0.9 grs in filling). Tried adding a drop of H₂O to this fraction which fillrate (12 cc) but got no more bis ale. to sep out. (the drop: but ali as best is)

Not a reliable method for getting Acetol and it tends to go over to bis ale

176° Bromo propionaldehyde & Na formiate [cf No 166] 7.5 grs Bromo propionaldehyde bpt 42°-4° at 63 mm and 7 grs Nafumiate H₂O & 2.8 grs xx $COOCH_3$ heated in S.T. B.W.B. for 20 hrs. no precip. or charring and no smell of Bromoal. left. Took up in ether etc & mid oil dist in vacuo got 17 grs bpt up to 50° at 30 mm and 2.55 grs bpt 50°-75° at 30 mm B.W.B. leaving a small amt. of H₂O in d.f. Took both fractions and heated in a S.T. B.W.B. for 9 hrs with 8 grs CH_3OH . An dist. in vacuo got 1.25 grs oil bpt 40°-50° at 20 mm this found above into a sol of 2 grs S. carbonyl & 1.3 grs K_2CO_3 in 5 cc H₂O. Reacts at once & got 0.95 grs xx which xx from 15 cc H₂O gave 0.72 grs light needles/dept 195° ident.

177° Bromoacetone and CH_3OH Kacetate. Bromoacetone 33.5 grs less spgr. 1.634 at 23° West ph. Acetolacetate (from 26 grs Kacetate + 9 grs CH_3OH) got only the Acetolacetate obt (from acetic acid in it) which can be removed by K_2CO_3 & heat in S.T. 140° for 5-7 hrs got Acetol big yield. 29. took 8.2 grs bpt 60°-70° at 20 mm + 2.2 grs bpt 40°-60° at 20 mm = Acetolacetate chiefly and heated S.T. 5 hrs 140° with 15 grs CH_3OH