

(27)

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Now set free gum by 5.0g Bu₂N₂ from alc. sol. cinchonin salts tot 4.68g cinchonin
and 3.9g gum [α]_D²⁰ = +14.34 (p = 4.0); this was first treated with 8.5g Bu₂N₂ ext C₁₂H₃ etc
tot by xx from sec H₂O & 20cc alcohol very slowly a large curl of xx needles: decided at
this point to set free gum again tot back 3.5g weigh with 3.4cc ph. hyd & 4cc alcohol gave
12.78 ph. hyd vapor with 170°-180° with [α]_D²⁰ = +5.57 See No. 71 for continuation.

The orig. alc. filtrates from previous salts I + II, 19.6g + 5.9g, still contained about
18g Bu₂N₂ salts which would not xx: set free by 8g Bu₂N₂ tot 8.9g Bu₂N₂ and
4.6g brown gum with [α]_D²⁰ (p = 4.0) = +23°. Treated this gum with 8.4g Chlorine (to alk sol)
F₂ on distilling off H₂O etc 13.4g salts which dissolved in 40cc abs^l alcohol and scratch
gave 1.0g xx mpt 170°; is sticky: dissolv^d xx in H₂O, filter rmp + tot 0.57g of a chlorine salt
CH₃OH-CH₃OH-CH₃OH-CH₃OH from Osm A.
The main lot of alc. sol. chlorine salts was now set free by 5.0g Bu₂N₂ tot 5.88g Chlorine
and 2.0g gum sol in 300cc acetic ether (cold) with [α]_D²⁰ (p = 4.0) = +24; the latter gum with 2cc ph. hyd & 4cc alc
mpt 170° which is diff. soluble in H₂O as well as in alcohol; is it a keto acid
washed with cold alcohol.

and 2.0g " " spirit with [α]_D²⁰ (p = 4.0) = +17.3 (p = 4.0)
The (2.0g gum (+17.3) ext 5x with 100cc ether R.F.
left 0.8g insol gum (by diff) & the 0.8g insol gum gave with 1cc ph. hyd & 500cc cold ether
0.198 ph. hyd mpt 190-145°. An acc. det. of the part soluble in 500cc cold ether
gave 1.3g [α]_D²⁰ = +14°; this digested with Chlorine, ext 3x other etc gum a very sol in alcohol
Chlorine salt: dis cand. 40 days (Jan 14 Monday) 5.0g d. glucose, wog H₂O, 8g Bu₂N₂ & H₂O
[α]_D²⁰ = -12.45 rmt 69-71% sugar
H₂O mkt 23.5g neutral B₂O₃ + 2.3 Keto sugars
5.0g d. glucose, wog H₂O, 8g Bu₂N₂ & H₂O
[α]_D²⁰ = +9.17:

(73) 4. Glucose + 1/6 eq. Bu₂N₂
and let stand 0.7 corked for 40 days: add 6 57cc n H₂O, etc tot making filtr up to 1 L in 1 de lube = +9.17:
[α]_D²⁰ = +3.4. Sugar to H₂O (ineq 0.4g) gum pack 47% neutral sugars + 0.8% Bu₂N₂ ext 3x other etc gum a very sol in alcohol
Calc₃ left 135g und Calc₃ + ag. filtr gum finally 47% neutral sugars + 0.8% Bu₂N₂ ext 3x other etc gum a very sol in alcohol
cont 8.16% Ca rmt of Calc₃ gum by oxalic K₂ etc 1.75g and gums (alc ext) [α]_D²⁰ = +6.0 (p = 4.0). Discard
The 47g neutral gums + 200cc H₂O + 25.5g Br₂ 0.7 days, 44g phos₃P₂Ag₂CO₃ + H₂O etc gave finally
making up to 1 L in 1 de lube = -0.41: [α]_D²⁰ = -4.1. Total ag. sol. with 15g Calc₃ 10 hrs at 60° formed 10.15g
und Calc₃ tot on distilling off H₂O etc de a lube (w 2 cc ph. hyd) of 2.27g (see ext) and gum with [α]_D²⁰ = +25.3
+8.44 and cont 8.23% Ca; ph. hyd by oxalic K₂ etc 19.6g Cinchonin gave 20.48g xx d. glucose cinchonin gum (discarb).
(c = 3.47) + on digesting it work up 10.8g Cinchonin & 8.35g d. glucose cinchonin gum (discarb).
from this got by 9.0g Bu₂N₂ etc 10.8g Cinchonin & 8.35g d. glucose cinchonin gum (discarb).
The rest of the non-xx gummy cinchonin salts in filtrate gum by 11.5g Bu₂N₂ etc 7.15g cinchonin
and 8.45g gum [α]_D²⁰ = +19.53 (p = 3.38); treated & then treated with 16.5g Bu₂N₂ tot by xx 2.51g
d. Mannoic Bu₂N₂ mpt 170° (dis cand); the ag. alc. filtrate gum back 2.34g salts which
are being xx from 3cc H₂O & 50cc alcohol & an ext with xx lube (Oct 1 1911) → discarded Oct 13-11
(74) 4. Glucose + 1/6 eq. Bu₂N₂
[α]_D²⁰ = +11.4: mpt 170°; 4.45g phos₃P₂Ag₂CO₃; ag. filtr made up to 1 L gum d in 1 de lube = +0.84: [α]_D²⁰ = +8.4
4.45g phos₃P₂Ag₂CO₃; ag. filtr made up to 1 L gum d in 1 de lube = +0.84: [α]_D²⁰ = +8.4
18 days gum by CO₂ etc 3.43 phos₃P₂Ag₂CO₃ and

aq. filtr needs to 1d gun & in 1dc tube = +0.66 $\therefore$ [27]² = +6.0% (still not quite compl. equilib.).
 Now distill off H₂O, & pptd Ca salts g.m. p as usual (not weighed & solis caused). The aq. alc. filtrate cont.
 $\therefore$ the neutral sugars + osms A, B, C was then treated in the solution (500cc H₂O) with 50g Brn etc. for 5 days
 got by av, PbO₂, Ag₂SO₄, H₂SO₄ etc making up to 1d & in 1dc tube = -0.27 $\therefore$ [27]² = -11.27.
 The aq. sol. digested at 60° for 10 hrs took up 9.6g CaCO₃ & then got by H₂O alc etc a total of
 53.65g pptd Ca salts (in 3 crops, dried in vacuo) with 8.03% Ca and [27]² = +5.08 & rest of Ca salts
 by oxalic etc gun 42.0g acid gun [27]² = +29.03 (c = 4.2). These then took up 51.75g Anichlorine
 & gun by xx, in 2 crops, a total of 62.32g d. gluconic Anichlorine (c = 4.82) = +33.4. This gun with
 got 36.07g Anichlorine & 24.1g crude d. gluconic Anichlorine & $\therefore$ was for a long time
 Burein etc a bent. xx salt which looked different from d. gluconic Burein (see No 72) containing an. an. of
 considered as a new hexonic (d. gluconic) acid: it is known d. gluconic acid (see No 72) containing an. an. of
 d. Mannonic acid. on oxid. 7.6g of the gun with 31g HNO₃ 1.21 at 60° for 48 hrs & distilled off in vacuo (85° at 1mm) got
 5.6g xx gunny oxide which (c = 2.24) gun in 1dc tube = +1.042 $\therefore$ [27]² = +63.4. Titration of 0.224g took 22.4cc
 $\frac{1}{10}$ NaOH sol & 29.9cc hot stock of 500cc (10% NaOH sol): the aq. filtr. distilled off in vacuo & gun large amt of yellowish xx needles of a diff. sol
 got back 1.7g excess: the aq. filtr. distilled off in vacuo & gun large amt of yellowish xx needles of a diff. sol
 Ca salt; 4.12g dried in vacuo with 15.6% Ca theory for Ca H₂O & Ca = 16.13 $\therefore$ c.K. Rest of Ca salt + oxalic etc
 and keeping temp. of aq. sol. at 50° only making up c = 4.0 gun & in 1dc tube = +1.02 $\therefore$ [27]² = +29.65.
 the distilling off H₂O in vacuo & dissolving in cold acetone & heating nail. gun in vacuo to 90° the gun, 2.6g,
 gun [27]² = +86.5 $\therefore$ [27]² = +4.0. The gun now gun with Chlorine in 3 crops, 8.48g of xx diff.
 have been d. Saccharine. The gun now gun by Brn etc 1.3g free acid gun [27]² = +80.1 $\therefore$ [27]² = +4.9
 sol in H₂O. These took 400cc hot H₂O to dissolve gun by Brn etc 1.3g free acid gun [27]² = +80.1 $\therefore$ [27]² = +4.9
 easily sol in cold acetone or got them finally from acetone sol 1.05g gun 90° in vacuo to 90° the gun, 2.6g,
 on evap. aq. sol. of this gun in vacuo it then gun [27]² = +43.5 $\therefore$ obviously gun a mono- and
 a di-lactone (cf. lit.) dis. carb. Took a portion of the above 24.1g d. gluconic gun i.e. 14.4g & got
 ready aq. alc. filtr. from d. gluconic Anichlorine 6.23g crops I & II in S.T. with 72cc H₂O + 14g pyridine
 3 1/4 hrs at 150-160° etc 15g Brn etc 12.5g gun [27]² = +46.8. Treated it with 99.7g Burein got by
 xx a total of 14.6g d. Mannonic Burein, npt. 212; on xx this from 50cc H₂O + 100cc aq. alc. hot more
 got 12.02g xx needles npt 212 $\therefore$ [27]² = -24.03 (acc det). Now set free lactone from 14.5g salt original
 got 9.5g Burein + 4.2g xx lactone: this gun with Anichlorine etc a salt gunny easily sol. in cold
 about a total of 11.7g xx lactone: set free gun again got by alc etc 1.22g xx npt 135-150° &
 and 3.4g gun + xx the or on warming with 100cc aq. alc. hot more
 then by 3" treatment of alc filtr. by aq. alc. etc got 0.53g xx the crop III npt 135-140°: the
 alc filtr. of crop III distilled off & aq. alc. & scratch also got mostly solid. Residue of I, II & III
 now gun + 93.09 $\therefore$ +75.4 and +75.5B respectively $\therefore$ obviously had mostly the B or less
 stable form of d. Mannonic lactone in this case.
 Crop I, 12.2g [27]² = +93.9 now gun with 2.22g Chlorine a salt easily sol in cold H₂O & xx from 400cc aq. alc.
 dried very slowly got 2.13g gun needles npt 163-5 with [27]² = +63.75 (c = 4.0) dis. carb.
 then gun with 15g Brn etc 1.32g Chlorine & 0.65g gun (alc ext) [27]² = +63.75 (c = 4.0) dis. carb.
 crop II 11.7g [27]² = +75.4, heated with 24.5g Burein etc gun a Burein salt which xx out from
 5 pts water in cooling in porous deshe volum. xx had the exactly like d. Mannonic Burein: set
 free lactone again from all salt by 2.0g Brn etc got 2.45g Burein & 1.05g gun (alc ext) with
 [27]² = +57 (c = 4.0) dis. carb. The alc. filtrates from the 14.6g d. Mannonic Burein gun on further xx then
 11.8g xx the npt 153°: crude d. gluconic Burein (cont. traces d. Mannonic acid) & 8.4g gunny salts
 which on further xx the xx in gun mostly xx salts dis. carb. at this point

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hydroxymethyl
carb. sugar

Got brines the 53.65 g pples Ca salts (p 28) also 48.65 neutral 2- and 3- ketose sugars ~~from~~
which gave $[\alpha]_D^{20} = -17.3$ and carb. by titr betw. 65-67.5% Sugar on hand in alcohol Oct 1 1911).
The ^{orig} aq. alc. filtrate from the 62.32 g d. glucosic cinchonin (p 28) giving us now xx salts gave
with 22.0 g BuM₂ etc 14.9 g Cinchonin + 15.1 g gum, $[\alpha]_D^{20} = +26.5$; treated it then treated with
with 32.0 g BuM₂ etc 14.9 g ^{crop I} 6.32 g d. Mannosic BuM₂ npt 205; then the gum by 3.05 BuM₂ etc
41.9 g BuM₂ and 18.8 g gum or by scratching etc with Spnt got 0.85 g xx salt npt 149-151° $[\alpha]_D^{20} = +53.6$ (discard).
The orig. aq. alc. filtrate gave by further xxllim of 43.85 g salts left from ~~see~~ 100 cc alcohol
13.54 g ^{crop II} from xxllim npt 120-130° air dry; these lost 0.82 g xx this in vacuo the npt 140-160.
got 5.3 g ^{crop III} similar salt npt 130°-150° when 2.9 g ^{crop IV} similar salt or total of 2.1 g xx BuM₂ in
salts leaving 18.1 g gum (p 11) 32 days
(75) d. Mannose + 1/2 eq CaO₂ (p 16 March 4). 50 g d. Mannose, 100 cetho + 1.9 g CaO₂ corked & t.
for 8 days: whereas in case of d. glucose & d. fructose (74 & 75) got at first a clear solution was approx pple
on standing - in this case got on 2nd day a sep. of large amt of wood Ca salts which could not be got
& dissolve entirely on shaking during first of period of standing but some wood. salts subseq dissolved.
The comp. xpts with CaO₂ + galactose (72 and 76) gave insol. Ca salts at first on adding the CaO₂ +
shaking but these dissolved within 24 hrs on subseq ppn of Ca salts was observed in xpts with
d. glucose & galactose + d. fructose on standing. In case of d. Mannose xpt got after 8 days of standing
etc 2.16 g ^{pple} CaO₂ (theory 2.57 g) tag. filtr. used up to 1 L gum $[\alpha]_D^{20} = +3.0$ incomplete action.
Now deshell off H₂O + treat again with wood Ca salts for 6 days o.t. corked & got by Ca etc
2.1 g ^{pple} CaO₂ + found aq. filtr. needed to 1 L was ± 1 ; incomplete action. Total again with 100 cetho
+ 1.9 g CaO₂ for 18 days o.t. corked & got by ^{pple} CaO₂ 1.9 g ^{pple} CaO₂ tag. filtr. used up to 500 cc
gave $[\alpha]_D^{20} = +0.5$. Reaction still incomplete (not weighed) as observed; treat neutral sugars + ^{pple} CaO₂
Now deshell off in vacuo sppt + discard Ca salts. The aq. sol degraded 10 hrs at 60° took up 5.18 g CaO₂ got
of 2. from aq. alc. filtr. with 250 cetho + 28 g BuM₂ o.t. for 5 days got air pple, Ag₂ etc
anaq. filtr. (C=10) with $[\alpha]_D^{20} = -2.7$. The aq. sol degraded 10 hrs at 60° took up 5.18 g CaO₂ got
finally (in 3 crops) a total of 26.97 g pples Ca salts (theor in vacuo) with $[\alpha]_D^{20} = \pm 0.0$ + 7.93 g Ca: Pot
gum by xaline etc 20.25 gum (ale xtr) $[\alpha]_D^{20} = +40.46$: on digesting these with cinchonin 22.95 g
alkaloid dissolved then got by xx 22.25 g d. glucosic gum (discard after getting 2.09 g xx of
by 10 g BuM₂ etc 12.42 g Cinchonin + 9.3 g d. glucosic gum (discard after getting 2.09 g xx of
a polylactone (keep) - The orig. aq. alc. filtrate then pair with 13 g BuM₂ etc 9.8 g cinchonin
and 10.3 g gum (ale xtr) $[\alpha]_D^{20} = +44.2$ (this after titr treatment of rest with 20.4 g BuM₂ etc 9.8 g cinchonin
15.8 g d. Mannosic BuM₂ npt 145 g ep. salt npt 2120 with $[\alpha]_D^{20} = -25.71$ (p = 4.0 acc del)
from 300 cetho + 80 cc alcohol gave 14.5 g ep. salt npt 2120 with $[\alpha]_D^{20} = -25.71$ (p = 4.0 acc del)
this then gum with 5.0 g BuM₂ etc 10.18 g BuM₂ + 4.55 g d. Mannosic gum (theor in vacuo + powder) 4.25 g xaline
H₂O + xaline on BuM₂ + rubbing alcohol got 3.3 g wood xx powder npt 135-137°
npt 120-135°. On digesting this with 300 cc alcohol got 3.3 g wood xx powder npt 135-137°
Now deshell 3.7 g of the crude lactone in 120 cc alcohol R. 7. got rapidly on standing a large
amt of clear gum. Some grouped fine needles 2.12 g npt 149-153° with $[\alpha]_D^{20} = +53.4$; the
The orig aq. alcohol filtrate from the 15.8 g d. Mannosic BuM₂ etc 9.8 g cinchonin
gave back 15.3 g BuM₂ etc 9.8 g cinchonin salts which xx from 300 cetho + 80 cc alcohol
gave 5.05 g from d. xxllim npt 120-125°

which lost 0.25 g xx H₂O over H₂O in various other matters from 140°-160°; (broken Oct 17 1911).
aq. alcoholic filtrate and ∴ 10g salts is on hand Oct 1-1911.

Besides the acid given got also from the aq. alc. filtrates of the pples Ca salts.
a total of 25.1g neutral 2-3-Ketohexoses ~~hydro~~ much made up to 500cc given α in 1 de tube = -0.74
∴ [α]_D²⁰ = -14.74 and found by titr that sugars present were below 68.5 + 70% (used 0.2g)
∴ have 24.9g given on hand Oct 1-1911

(76) 1. Galactose 6g CaCl₂ (Jan 14) of No 45. Took 100g Galactose, 200cc H₂O
and 3.8g CaCl₂ of course 6 days: CO₂ de given 5.13g theory 3.38g pples CaCl₂; aq. filtrate made up to 1L given α
in 1 de tube = +3.46 ∴ [α]_D²⁰ = +34.06; sugar titration given between 76-80% sugar only ∴ much
change. Only not got rid of small amt Ca salts present by pptn with alcohol ∴ treated all with
500g H₂O and 60g Br₂ of 7 days, then air 107g PbCO₃ ag. sol. H₂O de got breaking filtr up to 1L
α in 2 de tube = -2.92 or [α]_D²⁰ = -14.06. Neaq. sol. digested to hrs at 60° took up 11.8g CaCl₂ +
finally given in 3 crops, a total of 64.5g pptd Ca salts made in vacuo which gave 7.91% Ca and
[α]_D²⁰ = +4.0 = +6.22. Rest of salts with 16.0g oxalic made given 5.38g given (alc. ex tr) [α]_D²⁰ = +2.67 and sugar
-30.03 (c = 5.38). got also 41.2g neutral 2-3-Ketohexoses ~~hydro~~ with [α]_D²⁰ = +2.67 and sugar
percent between 57-58%. the letting these stand dissolved in vac. and H₂O adding alcohol + scratch

soon got a separation of xx lls of l. Sorbose and d. Tagatose in large amts: after 1 mo. follow
off 40g xx lls, [α]_D²⁰ = +22.4 supt 150° [Keep] → continued See No 101.
The aq. alc. filtrate distilled off and again dissolved in vac. with H₂O and alcohol slowly giving more
xx lls June 10th - Oct 1 1911 (under hood) got only 98.5g of 150° crop II. the alc filtrate given much
must investigate 1st carefully properties of d. Galactose, l. Idonic and

l. gulonic acids in order to separate these acids quantitatively.
(77) 1. Arabinose + 6g BaCl₂ of 45 days (Jan 13 March 1). 50g l. Arabinose supt 150-160°
100g H₂O + 8g BaCl₂ of 45 days: add 52cc n H₂O etc. got working up filtr to 1L α in
1 de tube = +0.19 ∴ [α]_D²⁰ = +3.08. Sugar titration (0.3g used) found a shade below 65%.
The aq. sol. digested for 6 hrs. at 60° with CaCl₂ took up 1.77g only but could not separate the Ca salts of
org. acids from the neutral given ∴ treated all of it with 250cc H₂O + 25g Br₂ for 7 days; air +
then 42g PbCO₃; ag. sol. H₂O de finally by pptn, in 3 crops, a total of
The Ag. sol. now degraded 10 hrs at 60° took up 3.3g CaCl₂ + given finally by pptn, in 3 crops, a total of
18.53g Ca salts (in vacuo) containing sugar 14.3g, these gave 7.54% Ca and [α]_D²⁰ = +4.04 (see dech)
got also 25.0g neutral given I and very little Ca with [α]_D²⁰ = +0.6 and % sugar between
45-46% (used 0.3g) ∴ 24.7g left: took 1/10th of this ∴ 2.47g and made in cold with sol. of sample hydr
in 5cc 50% acetic acid. 0.50g from the box place only after 17 hrs standing paper 2 weeks of 7 got only
0.63g oxym well washed with cold H₂O + alcohol + supt 150-180° very diff. sol in boiling H₂O.
Keep out of neutral given I. got also 9.4g neutral given II containing nl. non Ca
3.6% CaO by quant def. [0.47g supt] I. with [α]_D²⁰ (c = 4.7) = +3.19. Keep solution of given II

(78) 1. Xylose + 6g BaCl₂ (Jan 13 March 1). 50g l. Xylose supt 150; 100cc H₂O + 8g BaCl₂ of 45 days: add 52cc n H₂O etc. got working up filtr to 1L [α]_D²⁰ = +2.6.
let stand of 45 days: add 52cc n H₂O etc. got working up filtr to 1L [α]_D²⁰ = +2.6.
Sugar titration (used 0.4g) was a shade under 65%. Could not separate the Ca salts from oxams + sugars
present ∴ decided to treat all with 250g H₂O + 25g Br₂
of 7 days. with air stream, 42g PbCO₃; ag. sol.

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Hrs etc gave making up to 1 L α in 10 tubes ($c = 5.0$) = $+0.19$ $\therefore [\alpha]_D^{25} = +3.8$.
 Now deposit ag. sol for 10 hrs at 60° and found 3.22g CaO₂ had dissolved finally got from
 the filtrate a total of 20.43g of Ca salts (in vacuo), containing sugar + osms, which gave
 79.2% Ca and $[\alpha]_D^{25} = +6.94$ (acc. det.) $\therefore$ treated these with oxalic acid and got 15.6g pure
 $[\alpha]_D^{25} = +9.94$ ($c = 3.12$) (Keep) $\therefore$ a mixture of all 8 portions $\therefore$ with d. series predominating
 got also 23.4g neutral sugars + osms, cont little Ca, giving $[\alpha]_D^{25} + 2.178$ ($c = 468$) + 46%
 sugar by titr with Fehd solution; $\frac{1}{10}$ this $\therefore$ 2.31g, then gave when mixed with fresh solution of
 5cc ph. hydr in 5cc 50% acetic at O.T. only after 17 hrs standing a sep. of osm after 2 weeks
 got 0.53g osm well washed with cold H₂O + spirit. mpt $130^\circ - 140^\circ$ + very diff. sol. in
 boiling water.
 Got also 7.65g neutral gums II, cont more Ca than I, which gave $[\alpha]_D^{25} = +4.07$ ($c = 3.825$)
 and Keep rest of gum. $\rightarrow$ Cont. No. 105

(79) Crystallization of d. Mannose. Took 10g Carl's d. Mannose mpt $135^\circ - 140^\circ$ + dissolved it in
 5cc hot water + added 25cc spirit + stood O.T. 2 days but got no xx even on seeding $\therefore$ dissolved it in
 residual gum 10.75g in 10cc about alcohol: on standing and seeding got after 4 days O.T. 115g xx (mpt $135^\circ - 140^\circ$)
 + filtrate on standing gave 1.4g more forms xx mpt $135^\circ - 137^\circ$ (not clear) with $[\alpha]_D^{25} + 13.81$ (i.e. with 0.964g
 xx + 23.1215g H₂O + found α in 10 tubes 10' after mixing was -0.09 : after 20' was -0.08 : after 48 hrs was
 $+0.56$. The alcohol filtrate distilled off gave a gum which on long standing got entirely solid &
 on adding alcohol + rubbing got 6.5g xx mpt $125^\circ - 138^\circ$ (not clear) $\therefore$ d. Mannose is very
 hard to get crystalline and in xx condition is probably entirely free and dry. (Keep)

(80) Crystallization of d. Fructose. Took 10g d. Fructose (fr. Dublin mpt $100^\circ - 113^\circ$) + dissolved it
 in 5cc hot water and added 25cc spirit + got nothing. To separate out in 2 days standing (+ seeding) $\therefore$
 distilled off water + found gum left 10.95g dissolved easily in 50cc about alcohol and on cooling
 and seeding (warming gently + first to dissolve gum which came out) got large amt xx needles after 4 days O.T.
 got 9.02g mpt $105^\circ - 112^\circ$ with $[\alpha]_D^{25} = -94.05$ i.e. took 1.1065g xx + 26.5015g H₂O gave α in
 10 tubes = -3.81 (13' after mixing). (Keep)

(81) Cane sugar + 1/10 eq NH₄Cl. Took 5g Cane sugar + 0.15g NH₄Cl ap. + 30cc H₂O and heated Buns
 in small flask c. cap. tube shrs. Solution was colored + faintly acid and $[\alpha]_D^{25}$ ($c = 5.0$) = $+21.08$; since $[\alpha]_D^{25}$ of
 cane sugar used was found to be $+66.9$ ($c = 5.0$) no mutarotation it is obvious that one hydrolysis to
 glucose + dextrose took place + that laevulose had been converted slightly into H₂O + laevulose.
 Malt sugar used had mpt $205^\circ - 206^\circ$ + showed mutarotation

(82) Malt sugar + 1/10 eq NH₄Cl. Malt sugar used had mpt $205^\circ - 206^\circ$ + showed mutarotation
 15. took 1.0302g xx + 19.6048g warm water + revolved gave α in 2 tubes + 6.02 at first after 17 hrs
 was $+5.35$: after 24 hrs was $+5.33$ $\therefore [\alpha]_D^{25} = +52.7$ ($c = \text{about } 5.0$)
 Took 5.0g Malt sugar + 0.15g NH₄Cl + 30cc H₂O shrs Buns as in 10.81 + got a colorless solution with
 $[\alpha]_D^{25} = +52.6$ ($c = 5.0$) α in 10 tubes = $+2.63$ $\therefore$ absolutely no change!
 (83) Malt sugar + 1/10 eq NH₄Cl. Malt sugar used had mpt $115^\circ - 125^\circ$ and showed mutarotation
 giving finally $+131.1$ for $[\alpha]_D^{25}$ i.e. took 1.2072g xx + 22.9440g H₂O gave α in 10 tubes 15' after mixing
 $= +6.08$: 17 hrs after = $+6.63$: 24 hrs after = $+6.63$.

TOOK 5g Maltogen + 0.15g NH_4Cl + 30 cc H₂O plus B.W.B (as in 81) T found solution was faintly colored but got $[\alpha]_D^{25} (c=5.0) = +129.4$ i.e. practically no change.

(84) Equisanin laevulose + dextrose + $\frac{1}{10}$ eq NH_4Cl . Took 2.5g each of reagents named + 0.15g NH_4Cl + 30 cc H₂O sealed B.W.B (as in 81) for 10 hrs; solution was colored faintly acid and gave

$[\alpha]_D^{25} = -18.7 (c=5.0)$ i.e. no action practically except slight decomposition of laevulose.

(85) d. glucose + $\frac{1}{10}$ eq NH_4Cl . 5.0g d. glucose + 0.15g NH_4Cl + 30 cc H₂O sealed (as in 81) B.W.B 10 hrs was faintly colored and gave $[\alpha]_D^{25} (c=5.0) = +53$ - therefore no action.

(86) d. Fructose + $\frac{1}{10}$ eq NH_4Cl . 5.0g laevulose + 0.15g NH_4Cl + 30 cc H₂O sealed B.W.B (as in 81) for 10 hrs was strongly colored but gave $[\alpha]_D^{25} = -89.6 (c=5.0)$ i.e. very little change.

(87) Crystallization of d. glucose. Took 10g Cat K's d. glucose m.p. $144-6^\circ$ dissolved it B.W.B in 5 cc water and add 25 cc spirit over it. Seeding after 5 days standing 6.08g d. glucose x x lls

upt 154°: $[\alpha]_D^{25} 10' = +99.04$ i.e. took 0.965g x x lls + 23.0753g H₂O and found d in 2 d cube

10' after mixing = $+8.07$; 22' after was $+7.40$ i.e. rapid change to limit (in lit) $+52.5$ (Keep)

(88) Crystallization of d. galactose. Took 10g Cat K's galactose m.p. $159-162^\circ$ dissolved it B.W.B in 5 cc H₂O sealed 25 cc spirit. On cooling comes out quickly as a gum i.e. warm again seal and

let cool warm gently etc so that x x not oil drops separate out. After 2 days of. got 7.1g x x lls

upt 162°-5°; then gave $[\alpha]_D^{25} = +134.3$ 10' after mixing i.e. 10.338g x x lls + 24.8342g H₂O

gave d in 2 d cube 10' after mixing = $+10.85$ 15' after was $+10.07$ i.e. goes down rapidly to limit

about $+8.0$ [Keep]. Please give 91.6% Sugars compared with d. glucose. 10.75 cc 0.5% sol. necessary to decolorize 10 cc alk. sol. (98 g/cc)

(89) Conversion of dl lactic acid into dl lactyl lactic acid (cf Nos 11 & 12). Took 25.0g Cat K's dl

lactic acid 1.21 spgr = 86.7% placed in d.f. with slow capillary stream over B.W.B for 17½ hrs sent

19.45g very stiff but slightly mobile in cold green. On dissolving this in 400 cc H₂O ext to 3X by other sol.

other sol. with Cat K's ext for 12.8g gum (100% view). On ext to eq. sol. 5X more with other got out

2.7g more gum. The under gum 15.5g on shaking with 100 cc cold H₂O dissolved quickly in great

part leaving only 1.5 cc insol thick Poly lactyl lactic acid. This forms can't convert lactic

gum at 100° into monolactyl lactic acid but possibly it can be done at 90°-95° with heating.

Also some lactic acid must have remained in the aqueous solution.

(90) Laevulose and HNC. Took 50g d. Fructose and dissolved it in 98g 10% ANC

(Heavy 75g) and let stand of. for 24 days. Now smells of NH_3 but not of HNC solution was deep

brown. Add 100g B.W.B. 8 hrs + 500 cc H₂O sealed 3 hrs oil bath 120° until no more smell of

NH_3 in eq. distillate. Add 31.1g H₂SO₄ etc over K₂CO₃ etc 49.95g gum (ale ext) with

$[\alpha]_D^{25} = +40 (c=4.995)$. On heating with Linchurin (1/2) found got w x x salt from alcohol i.e. turned over

all material to Javitsky for thesis. The powder both 20x7 Cat K's d. Mannuric & d. gluconic acid are formed.

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(91) The 2 lactones of d. Mannonic lactone (cf. Nos 67 & 68)

Took 50 g d. Mannose + 95 cc H₂O + 57.75 g Br₂ for 7 days, then air at 67° when 85 g /
 PbO₂, Ag₂SO₄, H₂SO₄ etc. The aq. solution gives decided test for sugar i.e. reaction incomplete.
 Now treat aq. sol. at 60° for 8 hrs with 100 g pure CaCO₃ found 13.7 g mol CaCO₃; further 8 hrs
 heating at 60° left 9.1 g mol CaCO₃ finally in heating 8 hrs left 8.17 g CaCO₃ i.e. 11.83 g dissolved
 instead of 13.9 g theoretical if all CaCO₃ had been oxigenized. Small amt of H₂O in mass other dissolved in mass
 but H₂O applied to alcohol 2x getting 50.8 g; Shaky Ca salts I and 5.8 g Ca salts II besides
 8.0 g given left in alcohol giving $[\alpha]_D^{20} = +8.75$ (p = 4.0) containing much sugar (by Fehling test).
 On xx the 5.6 g Ca salts from 70 cc hot water got rapidly transparent flat needles of Ca₂ pure
 Ca d. mannate 2H₂O (precipitated in mass) 45.5 g with $[\alpha]_D^{20} = -7.52$ i.e. took 0.9458 g salt + 22.7297 g
 H₂O acid found 2 in 2 dc tube = -0.56. (= 0.8726 g anhydrous + 22.8029 g H₂O)
 The aq. filtrate gave 1.6 g more xx Ca salt which melted with 45.3 g hot water in H₂O solution
 with 127.4 g oxalic acid 100 cc about. alcohol rubbing till all thickiness was gone got by
 the digesting these with 100 cc about. alcohol rubbing till all thickiness was gone got by
 filling 28.55 g crop I crude d. Mannonic lactone up to 130° - 153° $[\alpha]_D^{20} = +8.5$
 i.e. 0.77 g xx salt + 23.3369 g H₂O gave α in 2 dc tube = $+2.03 + 6.088$; after 44 hrs at 67°
 reading in 2 dc tube had gone down to $+3.12$ i.e. $[\alpha]_D^{20} = \text{now} + 38.53$; on crop this aq. solution
 over H₂O in mass got back the lactone in xx salt up to 125° - 140° after pouring drying in mass.
 These were given $[\alpha]_D^{20} = +79.3$ i.e. 10334 g xx salt + 24.9350 g H₂O gave α in 2 dc tube = $+6.38$
 but after 40 hrs. reading was $+3.10$ i.e. $[\alpha]_D^{20} = \text{now} = +38.5$ solution is strongly acid
 Now swap aq. solution on Br₂. adding at first 0.11 g HCl (as noted) got all as a granular fine
 powder with up to 150° - 153° and $[\alpha]_D^{20} = +51.097$ from Fischer's lactone. Took 0.9432 g
 salt + 22.6583 g H₂O got α in 2 dc tube = $+4.20$ after 18 hrs at 67° reading still was $+4.20$
 i.e. no change in this case. Now take this substance sheet in a flask with H₂O for 5 hrs on Br₂
 when evap solution in mass and got xx salt up to 120° - 145° $[\alpha]_D^{20} = +57.2$ or after standing
 for 64 hrs $[\alpha]_D^{20}$ went down to $+47.2$ (18 hrs. was $+49.06$) i.e. $\beta + \gamma$ lactone present
 Lactone is changed on Br₂ into a mix. lact.
 Got also from alcoholic mother liquor 2.15 g crop II xx lactone up to 130° - 140° $[\alpha]_D^{20} =$
 $+50.3$ i.e. took 0.97015 g xx salt + 23.3369 g H₂O gave α in 2 dc tube = $+2.03$ or after 18 hrs
 reading was $+1.063$ a mix. lact. of β & γ lactones present
 On treating the 2.15 g crop II fraction in H₂O solution with 0.21 g HCl swap Br₂ got all transformed
 into Fischer's lactone up to 149° - 155°; and on xx this from 60 cc abs. alcohol got rapidly
 1.75 g ap. xx needles up to 151° (in 2 crops) D.E.D.
 Took 3.0 g of crop I $[\alpha]_D^{20} = +8.5$ and xx this from 75 cc hot abs. alcohol got 2.28 g up to 138° - 145° which
 xx again from 100 cc abs. alcohol & sent to give 1.45 bulky needles up to 138° - 140° $[\alpha]_D^{20} = +71.53$ i.e.
 and after 44 hrs α was $+1.077$ after 66 hrs was $+2.02$ ($[\alpha]_D^{20} = +50$)
 The orig. lact. filtrate from which 2.28 g xx had separated out
 gave on evap to a small volume

tubular xxlle and right needle npt 135-150°, 0.42g with $\text{[} \Delta T_{\text{D}}^{\text{D}} = +64.9$ i.e. $0.4208 \times x + 10.145 \text{ g}$ H₂O
 gave α in 1dc tube = $+2.62$: reading after 18 hrs was $+1.098$ and after 42 + 66 hrs was $+1.85$ $\text{[} \Delta T_{\text{D}}^{\text{D}} = +46.0$
 The alc. filtrate obt from xx 2.28g xxlle (resp out 1.45g) gave on conc to a small volume 0.47g
 bulky dark xx needles npt 140-2° $\text{[} \Delta T_{\text{D}}^{\text{D}} = +35.6$ + small ~~amount of solid + yellow line~~ i.e. took 0.4668g xx lle
 and 11.2437g H₂O got α in 1dc tube = $+1.043$ reading same after 18 hrs: wrap eg. sol in vacuum powder
 and got back 0.4g npt 138-141° with $\text{[} \Delta T_{\text{D}}^{\text{D}} = +36.0$ (see det) :: wrap again in vacuum & keep
 B. lactone: $\text{[} \Delta T_{\text{D}}^{\text{D}} = +1.97$ npt 157° as follows: $\text{[} \Delta T_{\text{D}}^{\text{D}} = +15.1$ cool and

and got back 0.49 ml 138-141.
 prep. as pure ~~B~~ lactone.
 Now took the pure Fischer lactone $[\alpha]_D^{20} = +51.97$ nept 157° as follows:
 0.9939 g lactone + 6.78 cc n NaOH + H₂O up to total weight = 20.28g ^{after} shaking Bwt 15': cool and
 add 6.78 cc n HCl (= 6.78g) + read 4' after adding HCl + got 2 in 10c tube = -0.064; 6' after = -0.01;
 20' after = +0.05; 1 1/2 hrs after = +0.35; 17 hrs after = +1.04 (wt NaOH = 0.3966g) ∴ $[\alpha]_D^{20}$ now (17 hrs) was +27.6.
 after 21 hrs was +1.13 after 41 hrs was +1.15 (= $[\alpha]_D^{20} = +30.5$) practically constant but was noticeably
 slowly going up (to be noticed only on long standing). Now a vap. eq. sol in mens. arr. H₂O 9. m.p. &
 some which was got out only x x blew rubbing spinning. $[\alpha]_D^{20}$ now found to be +92.4.
 1.3113 g (instead of 1.3905g then) slight loss in handling.

$\therefore$ at first a jump which over-
wrote total weight of xable cont Nacl now found to be 1.3113g lactone.
 $\therefore$ total weight of xable cont Nacl now found to be 0.0262g Nacl + 0.053g lactone.
 $\therefore$ total wt = 0.0792g distributed as follows 0.0262g Nacl + 20.1278 Hrs given & in 1 Octole = +4° 17' after 18 hrs o.t. reading
 $\therefore$ 0.9409g lactone + 0.3704g Nacl + 20.1278 Hrs given & in 1 Octole = +4° 17' after 18 hrs o.t. reading
was +1° 57' after 42 hrs = +1° 48' $\therefore [x]_D^{20}$ = now +32°, after 66 hrs was 1° 52' slowly going up.
1st lecture is main product & that in the solution w.r. this is converted to
the 2nd lecture which finally slowly gives us big slanting
the 3rd lecture.

was +1.054
This shows that ~~the~~ lactone is
excess of 70-85% into free 2. Mannonic acid (which means = the ~~lactone~~ lactone.
the ~~Y~~ ~~acid~~ ~~lactone~~ (not affected by cold H₂O) but normally only ~~or scheduled lake~~ ~~measurable~~ ~~x~~ the
Now got pure ~~lactone~~ ~~lactone~~ $(\alpha)_{D}^{20} = +111^{\circ}$ up to 154-6° in ~~Temp~~ heavy ~~measurable~~ ~~x~~ the
as follows (see below). Further experiments with crops I & II etc.
Titration of xllle up to 130-153° $(\alpha)_{D}^{20} = +85^{\circ}$ crop I. Took 0.6289g in cold water: it latches from beginning
as a lactone in adding $\frac{21}{10}$ NNaOH but ~~ring~~ ring opens rapidly at first up to 10cc: then more
slowly from 10cc to 20cc: still more slowly from 20-30cc but regularly opens. After 30cc to 35cc
ring opened so slowly could not notice any decolorization in cold: and 40cc $\frac{21}{10}$ NNaOH + heat BBrO₃
as usual & titrate back got finally 34.67cc $\frac{21}{10}$ NNaOH required instead of 35.32cc = theory for $\frac{1}{2}$ lactone.
Titration of Fialar ~~Y~~ ~~lactone~~ $(+51.97)$ took 0.2917g spread whole at once like a lactone but
14cc $\frac{21}{10}$ NNaOH then finally BBrO₃ etc.
11.11cc theory for $\frac{1}{2}$ H₁₀O₆.
when $(\alpha)_{D}^{20}$ must be down to

Titration of Fiala's Yucca
 for more slowly than under (+85°) from: look up slowly in
 a total of 16.7 cc $\frac{N}{10}$ NaOH instead of 16.4 cc theory for 2 H₂O.
Titration of crude Lactone (+85°) after standing o.t. in H₂O for 49 hrs when it must be down to
 1.3. 0.5768 g lactone [d₄²⁰ = +85° crop I was dissolved in much cold H₂O and let
 (+38° 53'). Titrates now with $\frac{N}{10}$ NaOH like a free acid up to
 +15.8 cc to 22 cc then slowly from 22-28 cc
 (- theory),

$+38.5$ (cf. above p. 33) : $+38.5$ would now be $+38.5$ instead of $+32.40$ (= theory),
 stand v.t. for 49 hrs : $+32.18$ cc instead of $+32.40$ cc (= theory),
 15.5 cc then like a lactone rings opens rapidly from $+15.80$ cc to $+15.5$ cc.
 Final result B.B. etc was 32.18 cc instead of 32.40 cc (= theory).
 from 28-30 cc. Final result B.B. etc was 32.18 cc instead of 32.40 cc (= theory).
 The composition of various lactone mixtures obtained in above experiments except c.p. Fischer lactone
 on B.B. for 6 hrs then finally powder only on theory in new gear 3.0 g lactone up to $125-150^\circ$
 which gave $+57.1$ and this finally went down after 41 hrs to $+47.5$: mainly X & Y lactones formed
 on B.B. just pure Y as when Hal is used. i.e. 0.6468 g X + 15.8012 g the four d in 10 cc
 tube = $+2.27$ after 17 hrs was $+2.02$: after 41 hrs was $+1.89$. Evap. ag. sol. in new v.t. gave pure
 xx all of the 3.0 g + lactone recovered from 140 cc absol. alcohol leaving with pure Y lactone and got
 not $152-40$ (d) 7% = $+51.6$

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1.2. 0.5424g xx + 13.1165g H₂O gave α in 10c tube = +2.07 and reading (3x) was same even after
 42 hrs standing at r.t. $\therefore$ sp. y lactone.
 The alc. filtrate from 1.8g pure y lactone (and 1.25g xx lactone) was evap. to a small volume and
 got xx needles octahedra side by side 0.62g up to 140-50 $[\alpha]_D^{25} = +60.1$ or after 46 hrs at r.t. $[\alpha]_D^{25} = +48.0$
 $\therefore$ ~~xx needles octahedra side by side 0.62g up to 140-50 $[\alpha]_D^{25} = +60.1$ or after 46 hrs at r.t. $[\alpha]_D^{25} = +48.0$~~
 The pure ~~xx~~ lactone is unquestionably present mainly y lactone and very little if any ~~xx~~ from D.E.D.
 The pure ~~xx~~ lactone from crop I and 2 $[\alpha]_D^{25} = +85$; Took 3.0 xx lte up to 130-153 $[\alpha]_D^{25} = +85$
 and kept at first with 100cc absd. alcohol till only heavy xx octahedra remained and is solved
 + decant + wash these in all with 50cc more hot alcohol. Got 0.98g heavy transparent
 octahedra of ~~xx~~ lactone up to 153-150 $[\alpha]_D^{25} = +111.1$ i.e. 0.6015g xx + 14.3735g H₂O gave α in
 10c tube 12' after solution = +4.51; reading after 3 hrs was +1.23 $\therefore$ constant $[\alpha]_D^{25} = +30.3$
 after 45 hrs was +1.14; after 69 and 93 hrs was +1.02 $\therefore$ constant $[\alpha]_D^{25} = +30.3$
 Now evap. of sol. in vacuo H₂O gave 9.4g p. powder + dry well and found nearest crystals now gave
 $[\alpha]_D^{25} = +90.5$ only $\therefore$ ~~xx~~ y lactone ~~are~~ slowly being formed D.E.D.
 Now took rest of crop I (+ 85 $[\alpha]_D^{25}$) lactone mix lte 20.33g + xx this from 900cc absd. alcohol
 following seeding with pure ~~xx~~ lactone and got 7.0g magnificent octahedra like xx lte, very strongly
 refractive + transparent up to 150cc (11.23g) The alc. filtrate (including also that obtained from
 digesting the 1st 3.0g lot above with alcohol in all) gave on conc B.P.B. to a small volume 11.77g crop II
 up to 135-142 $[\alpha]_D^{25} = +67.0$ sol in xx from 330cc absd. alcohol seeding with pure y lactone
 gave 6.58g xx needles up to 149-157 $[\alpha]_D^{25} = +57.02$ i.e. O.K. sp. Fischer y lactone as the
 ap. sol after 18 hrs at r.t. still gave same reading for $[\alpha]$. The alcoholic filtrate from the y lactone
 (and i.e. 4.63g xx lte) gave on conc B.P.B. at first octahedra xx of ~~xx~~ lactone + then needles of ~~xx~~ lactone
 y lactone + then on top of needles of ~~xx~~ lactone $\therefore$ warm till last named xx lte $[\alpha]_D^{25} = +57$ and
 dissolved and decant still cool when got 2.6g bulky needles up to 137-8 $[\alpha]_D^{25} = +57$ and
 after 64 hrs at r.t. $[\alpha]_D^{25} = +34.33$ i.e. a mixture of ~~xx~~ and ~~xx~~ lactones + ester)
 The orig. alcoholic filtrate from 11.77g crop II above containing only about 3.0g xx was
 distilled off in vacuo ~~xx~~ taken up R.T. in 400cc acetic ether seeding with ~~xx~~ lactone + got ~~xx~~ α β
 1.24g volum. needles up to 137 $[\alpha]_D^{25} = +57.2$ or after 65 hrs at r.t. $[\alpha]_D^{25} = +35.0$ i.e. mixture of ~~xx~~ α β
 lactones. Note the 1.24g lot and the 2.6g lot in order to get more of pure ~~xx~~ lactone
 from these (if necessary), including also 0.7g xx lte up to 138-140 $[\alpha]_D^{25} = +52.8$
 subsequently obtained on concentrating the alcoholic filtrate of 2.6g lot
 i.e. have on hand 4.42g crude ~~xx~~ lactone and from 20-25% ~~xx~~ $[\alpha]_D^{25} = +35.6$
 0.47g pure ~~xx~~ lactone $[\alpha]_D^{25} = +35.6$
~~xx~~ (92) The 2 lactones and the ester of d. gluconic acid (of No 69)
 The 10.25g ~~xx~~ lactone, up to 145-150, gave $[\alpha]_D^{25} = +60.6$ 10' after solution i.e. 0.5845g xx +
 13.9477g H₂O gave α in 10c tube 10' after solution = +2.46; 15' after reading was +2.36; 25' after = +2.19
 80' after = +1.26; 160' after = +0.53; 250' after = +0.37; 310' after = +0.33 $\therefore$ $[\alpha]_D^{25} = +8.11$
 24 hrs after was +0.39 $\therefore$ slowly going up again!!
 Now evap. of sol. in vacuo gave 9.4g p. powder + got back the xx lactone

NB Should have tried solution + m.p. then mainly β lactone was present — This not

but it is now sticky & could not be got entirely free of stickiness over H₂O in vacuum by drying & rubbing; until it with concp. aq. solution of ap. lactone (see below)

Na ap. β lactone: Now took 2.0 g of the crude β lactone (orig 10.25 g) and found it took 120 cc hot abs^l alcohol to dissolve it and on standing got quickly transparent conc. grouped slender needles

1.1 g npt 152-40 $[\alpha]_D^{20}$ (12') = +61.7; 15.0.8019 xx + 19.3220g H₂O gave α in 10c tube 12' after solution = +2.48; 13' after +2.46; 14' after +2.43; 21' after +2.28; 22' after +2.24; 23' after +2.23; 24' after +2.22; 1 1/2 hrs after = +0.83; 2 hrs 35' after = +0.40; 4 hrs 25' after = +0.31; 20 hrs 41' after = +0.25; minimum $[\alpha]_D^{20}$ = +6.24; 46 hrs after = +0.32; 70 hrs after = +0.39; 104 hrs after = +0.40; slowly giving up like β lactone had been reached (this' from acetone after quills between free acid & β lactone)

2. Mannonic lactone after quills between free acid & β lactone gave 0.24g non xx like npt 145-150°

3. γ and β lactone The alc. filtr on cone. on B.W.B. gave 0.24g non xx like npt 145-150°

4. β lactone which can obviously be xx from abs^l alcohol without much change. Now concp. aq. sol. of 0.8019g non β lactone (mixing with aq. sol. of 0.8845g and β lactone see p 35) heating on B.W.B. for a long time others trying to get Fischer's γ lactone + did not succeed.

Then from now decided to take all gums = 3.15g + heat B.W.B. with 35.0 g B.W.B. & then add H₂O & got back at 40° in vacuum starting up in absolute alcohol + on distilling off this alc solution in vacuum 35° soon got a solid mass of bulky xx needles while much alcohol was still present; since the xx were so sticky on attempting to remove rest of alcohol over H₂O in vacuum, decided to dissolve these in about 250 cc hot alcohol and on standing got a large amount of xx needles 17.92g npt 50-60° of d. gluconic ethyl ester

5. β lactone on distilling off the acetone on standing got a large amount of xx needles 13.33g of xx npt 45°-54° (air dry). These last much xx alcohol $[\alpha]_D^{20}$ = about +0.00; the xx dissolved easily in warm acetone + on distilling off the acetone on B.W.B. and adding alcohol got by xx 11.38g and npt was 60°-65° and $[\alpha]_D^{20}$ = -0.997

6. β lactone + weighed went down in 5 days to 11.38g and npt was 60°-65° and $[\alpha]_D^{20}$ = -0.997

7. β lactone over H₂O + weighed went down in 5 days to 11.38g and npt was 60°-65° and $[\alpha]_D^{20}$ = -0.997

8. β lactone i.e. 0.5000g xx + 12.0950g H₂O gave α in 10c tube = -0.04; few other $[\alpha]_D^{20}$ was +2.0; slow change this' hydrolysis etc. Titration 0.4262g in water titrated with a lactone and took 10.09 cc of 1.0N H₂SO₄ (the 19.02 cc for the ester) Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.) Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

9. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

10. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

11. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

12. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

13. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

14. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

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56. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

57. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

58. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

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80. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

81. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

82. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

83. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

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89. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

90. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

91. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

92. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

93. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

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98. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

99. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

100. β lactone Now took rest of the ester 11.88g (and some other, obtained & heated by hydrolysis etc.)

free d. galactonic acid has been obtained in
[d] as -11.7 by Fischer Ruff

xx from whale by Kiliani Ber 18, 1887 sets
Ber 33 2146 (2-yr old gum)
xx from whale by Kiliani Ber 18, 1887 sets
Ber 33 2146 (2-yr old gum)
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xx from whale by Kiliani Ber 18, 1887 sets
Ber 33 2146 (2-yr old gum)

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(93) The lactones of d. galactonic acid (cf No 70)

The 4.24 g vials dried Ca d. galactonate, H₂O gave [d]_D²⁰ = +0.37. i.e. look 1.1782g salt
and 28.2690g H₂O found α in 2dc tube = +0.03.
Now had Ca salt + 3.9g crop II Ca salts with 12.52g oxide H₂O got 35.35 g crude d. galactonic lactones
(alc xtr). The alcoholic solution on long standing (3 weeks) got entirely solid with xxlll; got 8.13g, crop I,
mp 95-105 (after washing with mixture of alcohol + acetic ether); these gave [d]_D²⁰ = -36.73
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Took 1.0743g of these xxll + 6.78g N₂O₄ + H₂O 13' BWS & weight on cooling = 20.0g; now add 6.78g
(∴ 0.3966g H₂O) N₂O₄ found α in 2dc tube 6' after adding H₂O was -0.96 [d]_D²⁰ = -11.65
1/2 hr after α was -0.68; 15 1/2 hrs after = -0.91; 25 hrs after = -1.12; 40 hrs after = -1.52 ∴ goes up
? this experiment is somewhat doubtful as to data obtained because on cooling of ag. sol in
vials got small of acetic acid
The alc solution from the 8.13g crop I gave on evap on BWS & stirring 27.34g xxll.
Now observed that the crop I xxll (6.5g left) on dissolving in 30 cc alcohol left some und
gummy stuff + capillaries ∴ can't run cooling got 2.65g gelatinous xxll mp 95-105
(∴ d. galactonic ethyl ester) very difficultly soluble in hot acetic
the crystallizing the 27.34g xxll from 30 cc alcohol got solid with xxll ∴ add more
alcohol & filtered off 6.6g xxll mp 60-70 containing a shiny gelatinous body ∴ dissolved
it in 10 cc hot acetic ether from some insol. gum adding also acetic ether & steam
from 2.65g lost sign on distilling off in BWS. 6.65g xx mp 66; these xx from alcohol gave
3.0 xx needles mp 110-115 [d]_D²⁰ = -77.61 (very little change on standing) Blue line
Finally decided that only one lactone with hydrite present which can't be easily
separated by xx from alcohol, acetic ether or acetic ether ∴ re-treated all gums
& start afresh. Had 17.7g which heated BWS 15' with 21g BWS etc & take up in absol.
alcohol: on conc. alc. sol. in vials in d.f. it all xx rather volume was small ∴ pour out into
dish & evap out of alc in vials there: got 16g crude xx mp 70-95 [d]_D²⁰ = -63.06 no
change in [d]_D²⁰ after 48 hrs ∴ lactones must be present Blue line its hydrite for this acid present

Now xx 15g of this crude mixture from alcohol got in all 5 crops as follows:
Crop I 4.45g mp 108-115 [d]_D²⁰ = -77.0 ∴ crude Blue line [keep]
0.8342g crop I + 6.2g H₂O + 6.78g N₂O₄ BWS 13' cool & add H₂O total weight = 14.18g & then add 6.78g
6.78g N₂O₄ (∴ 0.397g H₂O) found α in 1dc tube 5' after adding H₂O was -0.60 [d]_D²⁰ = -14.63
α after 8 hrs of was -0.54; after 22 1/2 hrs was -0.78; after going up slowly, according to lactone
but ? does it first go down towards 0.0 due to Blue line from alcohol which should be +.
Crop II 2.40g mp 90-110 [d]_D²⁰ = -50.5 p = 4.0 ∴ crude Blue line [keep]
1.33g mp 100-115 [d]_D²⁰ = -76.3 p = 4.0
Crop III 2.14g mp 90-105 [d]_D²⁰ = -62.4 dis. can't
Crop IV 2.95g mp 70-95 [d]_D²⁰ = -50.3 dis. can't
Crop V 2.95g mp 70-95 [d]_D²⁰ = -50.3 dis. can't
Keep crude Blue line for seeding time in new sight
with large amount of material (Webb).

TOK 0.9450g crude leucine + 7.74 cc n NaOH + H₂O = -0.66 ? going down . 44 hrs after = -1.08
in 2d tube 4' after = -0.82; 15' after = -0.79; 60' after = -0.66 New wrap, eq. sol. in vacuo thru 9 mg
Total weight of gum + NaCl (0.453g) + H₂O = 2.55g ∴ H₂O + salt = 2.317g spread & in
to 7th = -1.04. Then again make up to total weight = 2.3770g ∴ H₂O + salt = 2.317g spread & in
~~the same way~~
1d tube 20' after solution = -1.04; 3 hrs after = -1.01; 18 hrs after = -1.03
or to 7th now = -32° (going up).
leucine + 7.74 cc n NaOH + H₂O BNB 13' cool (wt = 16.64g) total
-0.46; 7' after = 0.48; 16' after = -0.44

duplicate: 0.9879 g and 2 y leadline + 774 cc N₂ Wash + H₂O BUBB 13' cool ($w_t = 16.64g$) total
or total now = -32° (giving up).
1 October 20' after solution = -1.04; 3 hrs after = -0.46; 7' after = 0.48; 16' after = -0.44.
1 hr after = -0.40; 2 hrs after = -0.39; 3 hrs after = -0.40; 5 1/4 hrs = -0.41; 8 hrs after = -0.44.
22 hrs after = -0.58 31 hrs after = -0.68 total weight = 24.38g (Vacc = 0.453g).
unstable + rotating & lac tone but data resub closely
in presence of an stable + rotating & lac tone but data resub closely
now dissolved in 500cc hot acetic

1 hr after = 1
22 hrs after = -0.58 stable + rotating
No indication of presence of an unstable + rotating
those of d. galactonic acid (9.6%).
The main lot of l. ascorbic lactone (36.93g) was now dissolved in 500 cc hot acetone
to a small volume of l. ascorbic lactone (31.6g)
the main lot of l. ascorbic lactone (36.93g) was now dissolved in 500 cc hot acetone
to a small volume of l. ascorbic lactone (31.6g)

The main lot of ...
 & decanted from our and used for other cones.
 of the pure Y lactone w/o 98-107° [d]_D²⁰ = -72.2 [2.0, 4.96] xx + 1.1%
 tube = -2.92 (same after 46 hrs O.T.).
 Now took 5.0g of the pure Y lactone sheeted in H₂O solution with 1 1/4 cc. Br₂. B.W.B 15' t.p.h
 by H₂O etc distilling off 45° at 20 mm slaking up in alcohol and getting rid of alcohol war
 H₂O in vacuo (flash) mainly a x x lactone which gave [d]_D²⁰ = -53° which was not with y form)
 changed in H₂O 24 hrs O.T.: substance must be mainly a new β lactone (the heating this in
 H₂O solution [d]_D²⁰ goes up this formation of γ lactone but did not work up further,
 should repeat distilling off H₂O at low temp when use no alcohol trap off H₂O in vacuo
 at low temp to see if really a new β lactone is formed.
 on hand a total of 32.27 g ap. γ-lactone.
 bumped by tx from acetone.
 505 l. Xylene + 500 g H₂O + 60 g Br₂ O.T. 7 days
 then took up 18 hrs at 60°

should repeat distillation of a new batch
 at int temp to see if really a new product
 Now have on hand a total of 32.27 g of Y. L. Carabine
 from Oct 29 '06 which was purified by tx from acetone.
 50g l. Xylase + 500g H₂O + 60 g Br₂ at 7 days
 lactones of l. Xylonic acid. 50g l. Xylase + 500g H₂O + 60 g Br₂ at 7 days
 still induced. Soln. took up 18 hrs at 60°
 Ca salt from H₂O sol by alcohol. 50g
 Ca salt was +11.05 ie. 57712

Now leave on hand
from Oct 29 '06 which was pumped
(95) The 2 lactones of l. Xylonic acid. 50g l. Xylonic
acid + then 100g CaCO_3 , Ag_2CO_3 , H_2SO_4 : aq. filtrate still reduced field. solution took up :
149.5g CaCO_3 (just of 16.66g (theoretical) : 2nd still off H₂O in vacuum filtered Ca salt from H₂O sol by alcohol took
57.95g (in vacuo) with 9.6% Ca (theory 10.81% : xx H₂O present) : 2nd of Ca salt was +11.5% i.e.
took 1.8450g salt + 44.2785g H₂O and found 2 in side tube = +0.93. Total out of Ca salt 57.71g
with 17.2g of xylonic acid + 2 (like extra) 39.53g and a l. Xylonic lactone found which got partly
crystalline : took up in 500 cc acetone, decant from some insol tar + Ca salt took on acentr
clear solution to a small volume 20.3g pure y l. Xylonic lactone m.p. 99-103
I₂W₂ = +89.56 i.e. took 0.5312g xx + 12.719g H₂O and

Cinchonin l. xylmate: Now took eq. sol. of leachings (4.75 g) shealed BWSB with excess cinchonine
to 100 cc & found α in 1 October = +1.096 $^{\circ}$. after 18 hrs was 2.00
 γ & β leachings.
taken up 9 lbs BWSB tag. filtrate concentrated at room temp to 60 g gave on cooling
4 pts hot water
leaves from absolute alcohol.
to blades from absolute alcohol. (Keep pump).
= +5.09.

[2]₂ = +126.2
Now leave left 14.18 ap. y. last time
(96) L. gulonic and L. Idonic lectures.
24 days no action had taken
cool slab stand at 150g
Added 500g M
NH₃ noticed after a time that solution had got quite warm; cool slab stand at 150g
NH₃ no longer noticeable in distillate.
400c now give solution was deep brown colored. Heat pink smell of NH₃ no longer noticeable in distillate.
oil bath for 3 hrs till smell (said to be L. gulonic base br salt) x
base br salt (said to be L. gulonic base br salt) x
which got entirely x x
pouring out latter into

The dish trap + snow
nupte 182°-50 ∴ all filter contained
The 21.35g lot on taking out + rubbing with H₂O Kue trap BurB. 103g none c.p. leachd or
quite from but on breaking this with H₂O Kue later got 1.03g none c.p. leachd = 2.931 (19) after
in snow while hanf. plates nupte 182°-50 slater got H₂O gave 2 in 10 clute = 2.931 (19) after
∴ snow [d] no = -57.1 i.e. 1037.2 g xx + 24.8768g H₂O gave 2 in 10 clute = 2.931 (19) after
21 km = -2.23 ∴ after 46 km = -2.23 ∴ pract no change in H₂O at 0.7. (used for Bismar salt)
1.78°C n NaOH BurB 13' cool + add quickly 6.78cc n HCl total weight = 14.913g
at the adding HCl was -0.46; 94' after = -0.40; 234' after
∴ at first down then up 11 solid

[illegible]

Took 5g cp. l. galonic lactone with 1 1/2 mols BaCl₂ 8H₂O + H₂O in BWRB 15' and so₂ etc + distilled off H₂O quickly at 18 mm and at lowest possible temperature then take up gum in absol. alcohol + wrap over H₂O₂ in vacuo gum p + for mainly 4.0 Yl. galonic lactone kept 1850 but also a small amt 0.9g gum still containing the Y lactone which gives $\alpha D_{20}^{25} = -17.9$ on wrap this gum on BWRB it gave 0.5g xx Y lactone up to 150-50 showing that the gum is slowly converted into the Y lactone. The new form (?) of l. galonic lactone may even be + rotating.

l. galonic Buein: took the 1.0372g lactone (used for opt. det. p 39) + 1.0g more cp Y lactone + heated BWRB. with 4.4g Buein + then distilled off 8.25g xx salt which dissolved in 3cc H₂O + 30cc alcohol soon gave large amt of glistening heavy eye group of flat needles 5.21g (in vacuo) up to 112-40 $\alpha D_{20}^{25} = -18.967$ i.e. 0.9775g xx + 23.627g H₂O gave α in 10c tube = -0.75 (Keep prep).
Now have on hand 19.45g cp. Y lactone + 3.7g crude Y lactone

l. Ideric gum. The total amt of crude l. Ideric gum obt. from de. filtrate of l. galonic lactone was 21.5g; it was treated with 40g Buein + gave 718 xx salts which xx from 20cc H₂O + 30cc alcohol gave 2 in 10c tube = -0.98. Keep 3.8g as prep use

1.2. took 1.020g xx + 2.44737g H₂O gave 2 in 10c tube = -0.98. Keep 3.8g as prep use
not 40g for making for l. Ideric gum as the de. filter from crop I l. Ideric brown on long standing (19.3g) in 3cc H₂O + 11g BaCl₂ 8H₂O gave 25.6g Buein (in vacuo) and the 40g l. Ideric Buein + 600cc H₂O + 11g BaCl₂ 8H₂O gave 25.6g Buein (in vacuo) and 10.3g gum (acetic ext.) $\alpha D_{20}^{25} = -54.06$ (C = 4.12) one charge in 175 after 17 hrs. it.

The 10.3g gum was digested BWRB 20' with H₂O + 12g BaCl₂ 8H₂O and H₂O₂ etc + distilled off with can quickly at lowest possible temp + then take up in absol. alcohol; redistilling off alcohol in vacuo to small volume got a separation of 11g xx plates of Yl. galonic lactone + 1850 and 10.3g problem on converting 1.092g by 6.13cc H₂O Na = 1.337g) found 2 in 10c tube = +0.89 $\alpha D_{20}^{25} = +11.57$ Q. End.
The abs. filtrate was gum back 8.25g crude l. Ideric lactone gum (on hand).

(97) Behavior of d. Fructose towards H₂O and dilute acids. ? try with pyridine + water !

- (a) Took 5g cp. d. fructose (see below) + 10cc H₂O in ST. 9 hrs 130-135° then 8 hrs 140-150° (mostly 140-5) + got a deep brown colored solution which made up to 50cc gave 2 in 10c tube = -0.475 $\alpha D_{20}^{25} = -75$ not very accurate but d. fructose has been very little affected.
- (b) 5g d. fructose + 20cc 7.5% by vol H₂O₂ heated BWRB cap tube in a small flask for 3 hrs gave 0.34g insol humic acid + then by 5x ext. with other dry cells got (100% vacuo) 1.0g gum (20x7 CH₃ propand + laevulinic acid see below). The H₂SO₄ solution still reduces Fehd sol. strongly and gave (C = 2.5) $\alpha D_{20}^{25} = -33.3$ i.e. found 2 in 10c tube = -0.983.
- (c) 5g d. fructose + 15cc 10% H₂SO₄ heated BWRB cap tube for 1 1/2 hrs and got 0.23g humic acid (insol tar) and 0.8g gum ext. by other etc. H₂SO₄ solution (C = 2.5) gave $\alpha D_{20}^{25} = -41.02$ i.e. found 2 in 10c tube = -1.903
- (d) 5g d. fructose + 30cc 5% H₂SO₄ heated BWRB 9 hrs cap tube gave 0.51g humic acid and 1.15g gum ext. by other etc. the H₂SO₄ solution (C = 2.5) made up to 200cc) gave 2 in 10c tube = -0.46 $\alpha D_{20}^{25} = -18.4$
- 20% left (80% gone)

Dissolved the 195g ^{ppled} Casalts with tar & sugar in 100cc hot water and add shaking 500cc Spirit and got 95g ^{ppled} Casalts II and 92g Sugars ~~117g~~ (note see below)
Redissolved the 95g ^{ppled} Casalts II in 50cc hot water and ^{incl 92g previously lost} ~~ppled~~ by 750cc Spirit shaking & got a resolution into 71g ^{ppled} Casalts III and into 117g Sugars ~~117g~~ II
Dissolved the 71g Casalts III in H₂O strat K₂C then decant off & decolor in 80cc H₂O then add 350cc Spirit test by pptn & scratching 51.1g Casalts IV (in vacuo) containing only 6.21% Ca! The aq. alc. filtrate now gave united a total of 130g Sugars + ~~6.21% Ca~~ II
On digesting ^{130g} Sugars + ~~6.21% Ca~~ with 1L absol. alcohol got a total of 56g ^{incl 56g} a" & subsequent 3x extraction with 1L absol. alcohol gave a total of 101g Sugars ~~101g~~ a" leaving :: by diff 29g insol Casalts containing sugar.
The 51.1g Casalts IV cont 6.21% Ca was now treated with 9.97g oxalic acid etc: the residual gum on extraction R.7. dissolved to extent of 27.58g in absol. alcohol and 16.2g dark brown gum, mainly tar, containing sugar remained undissolved; made up the latter to 100cc and took 5cc :: 0.81g and got by ignition 0.007g P₂O₅ + a sugar determination by Fehd solution showed only 38% sugar present & titration of 0.2025g took only 3.9cc $\frac{N}{10}$ NaOH soln :: very little acids present. Now dissolve the rest of the 16.2g gum in 8cc H₂O then add 70cc Spirit and got 6.55g ^{ppled} gums which gave (c = 3.275) $\alpha_D^{20} = +10.4$ and by titr with Fehd solution about 38% sugar and had 1.72% ash? CaO (or K₂SiO₃ Na₂SiO₃) :: now discarded this; the aq. alcohol filtrate from 6.55g ^{ppled} gum about 8.9g gums was now added to the

27.58g lot in manner to be stated below.
The 27.58g gum (alcohol soluble) obtained from 51.1g ^{ppled} Casalts etc. was made up to 500cc spirit & in 100cc tube = +0.27 :: $\alpha_D^{20} = +10.0$ about. Titration taking 0.2755g (i.e. 5cc) took 12.34cc $\frac{N}{10}$ NaOH, then for C₁₂saccharin = 17cc :: sugar present
Titration for sugar showed between 15 and 20% !! (used 0.5g) - Now digested rest of aq. sol. of 27.58g gum with 6.17g ^{ppled} CaO₃ at 60° for 18 hrs then left 1.5g insol
CaO₃ when add to solution the 8.9g gum above & digested aq. sol. for 18 hrs longer at 60° when only 0.6g insol CaO₃ left :: 5.57g ^{back} Casalts containing sugar; dissolved. On distilling off aq. filtrate got 3.7g
The ^{ppled} Ca salts (cont 8.44% Ca and only traces of sugar) which gave with 3.85g oxalic acid & more were resolved by many digestions R.7. with 1L absol alcohol each time, etc. (a/b/H/c)
14.48g ^{ppled} Ca salts (see ext) α_D^{20} (c = 3.9) = +10.0; the Na salt of this gum (0.9g) was found to be optically (used $\frac{30}{100}$ lbs). Rest of saccharin gum 8.6g was treated with Styrchium but could not get any xx Styrchium B dextro metasaccharinate altho' got gummy Styrchium salts diff sol in alcohol :: set free gum & got back 7.9g (also ext) treated with Buecin but could not get Buecin B dextro metasaccharinate by setting :: set free gum again - as must obviously have a mixture of 6 Dextro saccharins present (see Galactose ext No 100) (See p. 124)
of 6 Dextro saccharins present (see ext) b" $\alpha_D^{20} = +7.95$ with 0.4% CaO; note see p 43 bottom
8.0g Sugars (alcohol. alc ext) c) into 13.0g Casalts containing sugar

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The (C) Fraction, 13g Ca salts containing sugar, was dissolved in 26 cc H₂O and lost 26.0 cc spirit when heat R.F. & shake; decant clear solution when cold and found 3.0g sugar in aq. alc filtrate and is by diff must have left 10g insol Ca salts (Ca salts IV) + unite see top p 44

Examination of Sugars ~~10g~~ ^{10g} ~~insol~~ ^{insol} ~~Ca salts~~ ^{Ca salts} ~~IV~~ ^{IV} + unite see top p 44
and 85g insol green coal Ca salts.

Green A, 103g; made up to 500cc + det ash in 5cc: 1.03g as 0.0012g CaO: only 0.116% CaO present
[α]_D²⁰ found ± 0.00; ^{made up to 500cc} % Sugar present = bts. 76-79%. Green dissolved in 50cc H₂O was not precipitated by 250cc
spirit on long standing O.T. Distill off H₂O syrt back 95g A

Green B, 85g; made up to 500cc + det ash in 5cc: 0.85g as 0.0012g CaO: 0.14%; [α]_D²⁰ (c=4.0) ± 5.25
and % Sugar present was between 77 and 79%. Green dissolved in 42cc H₂O and 210cc spirit
gave no ppt on long standing O.T. Distill off H₂O syrt back 81g B

Green C, 147g; made up to 500cc + det ash in 10cc: 0.94g as 0.0009g CaO: 0.096% + 70% Sugar was between
74 and 78% and [α]_D²⁰ (c=4.0) was +8.0: Green dissolved in 22cc H₂O and 220cc spirit nothing
came out O.T. on long standing. Distill off H₂O syrt back 42g C

Green D, 30g; made up to 250cc + det ash in 5cc: 0.6g as 0.0019g (0.167%) + [α]_D²⁰ (c=4.0) = +8.0.
D' 18g was subsequently added to D: total D = 48.0g; further extr. of the 75g moist gums
cont. Ca salts 7.7x with 1L alcohol raised finally Green D total up to 72g and the

moist gums cont Ca 51g by diff were now united with the corresp. 29g insol Ca salts
containing sugar obtained from the orig 130g ~~Ca salts~~ ^{Ca salts} + Sugars II (see p 42).

The 130g ~~Ca salts~~ ^{Ca salts} + Sugars II were redissolved by degumming with alcohol
into Green a', 56g, which was subsequently increased to 101g by further addition of
alcohol ex. hts. leaving 29g moist Ca salts containing sugar (unite see above)

Green a', 56g, made up to 500cc and det ash in 10cc: 1.12g as 0.0018g CaO: 0.16%; found [α]_D²⁰ (c=4.0)
= +4.075 and Sugar % was slightly below 75%.

The 51g + 29g Ca salts containing sugar from I + II respectively were now combined (i.e. about
80g) + added to less in 70cc H₂O by 400cc spirit ~~degummed~~ ^{degummed} then cool; got 48g green Ca salts and
a mass still containing over 20% Sugar as det by total solution and 28g Sugars cont Ca

in the aq. alc. filtrates. These were each now boiled out many x with much absolute
alcohol and got as final result (a) 13.20g Ca salts containing less than 2.5% Sugar and 8.00% Ca
with Shochin when with Bismar gum entirely similar results to corresp. expts with 9.8g Saccharins I + II

unite these and set for total gums by 200g BaH₂ + 252g BaH₂ + 252g BaH₂ (see p. 44) + 669g (see p. 44) + 4.7g (see p. 44)
got a total of 12g Sugars b' which was treated with
Br₂ and water [p 42]

(b) got also 31.0g Sugars E cont 0.87% Ca and giving [α]_D²⁰ = +6.45 (c=6.2) in all
(c) got 27g Ca salts containing Sugar which was dissolved in 54cc H₂O and added to in all
500cc spirit then heated R.F. shaking; then cool decant clear liquid - unite with
3.0g lot above & degum (after removing H₂O alcohol) with 250cc + 500cc abs alcohol and
got a total of 12g Sugars b' which was treated with
Br₂ and water [p 42]

(44)

The pptd Ca salts c including portions left und by abs. alcohol of June 6" 12g
containing 10g Ca salts & finally got

240g Ca salts (in vacuo) cont 10.45% CaO :: treated these with 5.3g oxalic + got Knc etc

127g Saccharin gum (alc extr) cont sugar) $[d]_D^{20} = +4.33$ (c = 5.08)

and 110g Ca salts IV in vacuo about 10.74% CaO $[d]_D^{20} = +8.46$ (c = 3.9) :: treated these with 2.53g oxalic & got Knc etc

17.8g Saccharin IV (alc extr) cont sugar, with $[d]_D^{20} = +8.46$ (c = 3.9) :: treated these with 2.53g oxalic & got Knc etc

Summary of Sugars
A = 95g; B = 81g; C = 42g; D = 72g finally; E = 31g; a" final = 95g; b" final 127g

Saccharin I = 9.8g; Saccharin II = 7.8g; Saccharin III = 12.7g; Saccharin IV = 7.8g
Total = 38.1g

Now resolve the Sugars further as follows: in 12 about alcohol shaking
Gums A, B, C, D+E, and a" were each dissolved g.m.p. in 12 about alcohol shaking

plain: in each case some gum partly xx. separated out: decant clear solution and
det. and of final gum in each case to be as follows:

A 12g $[d]_D^{20} = +9.0$ (c = 4.8); B 10g $[d]_D^{20} = +12.5$ (c = 4.0); C, 2g; D+E 43g $[d]_D^{20} = +8.4$ (c = 4.3)
a" 27g $[d]_D^{20} = +10.0$ (c = 5.4). Unite all these insol gums in one fraction total = 94g

this dissolved in 50cc H₂O and add 250cc spirit (gave a resolution into
27g pptd Sugars H $[d]_D^{20} = +7.04$ (c = 5.4) cont 1.2% CaO (0.54g gave 0.0065g CaO)

disolved off H₂O and got back 25g gum H which was oxidized by 25g Br₂ and 250cc H₂O
0.7. for 6 days then air pber₃ ag₂s etc got a gum which on trying to dissolve in 300cc absd

alcohol got solid with insol. phosphorus material 15.6g melt 80-120° which was at first
thought to be d. allornic lactone; alcohol solution gave 8.9g gum (partly solid) (see below).

The al. alcohol filtrate from gum H gave back 23g Sugars G (see below).
The cold clear absd. alcohol. solutions obtained from Sugars D+E and a" (147g by diff)

was now concentrated in vacuo until volume was about 1L then decant gums which
had separated out by heating gums cool & decant clear solution after 24 hrs 0.7.

got 43.0g insol Sugars $[d]_D^{20} = +9.07$ (c = 8.6); the absd. alcoholic solution cont: 7.8g
by diff was then distilled off in vacuo till volume about 1/2 L then warmed R.T. cool &

decant as before gave 31.0g Sugars D $[d]_D^{20} = +5.5$ (c = 5.5) or after getting rid of H₂O again got 49g Sugars D.
accounted to 55g Sugars D

The cold clear alcoholic solutions obtained from Sugars A, B, & C (by diff 194g)
were now distilled off in vacuo till vol. was about 1L then warm R.T. cool & decant gums
and got 80g insol Sugars $[d]_D^{20} = +7.0$ (c = 8.0) which was united with the 43.0g corresp

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Sugar 7 117g + G 73g were now further resolved as follows:
Sugars G 2x R.F. with 1 L absol. alcohol each time, cool & decant shed large amt of
unsol. xx hygroscopic sugar &
Gum 7 similarly digested 2x each with 1.5 L then with 1 L alcohol, cool & decant shed
large amt of unsol. xx hygroscopic sugars: the unsol portions of 2 lots were united
and got (acc. det) 27g gum H' cont 1.8% CaO; % Sugar (a shade below) and
[d] = +6.67 (c = 5.4).
The cold clear absol. alcohol solutions containing res of Gum 7 + G nsp. were allowed
to stand O.T. seeding with d. glucose x x lls but got nothing: distilled off sweeping over
with H₂O and got back 110g gum F and 50g gum G; mixture of 2 solns was
the crude d. alloric gum obtained by oxid of 25g gum H (see p 44) had been resolved by alcohol
into 2 fractions 8.9g alcohol soluble + 15.6g alcohol unsol partly solid gums. 0.85g oxalic acid
[d] of 15.6g lot was +1.093 and it contained 2.24% ash? CaO: heated it with
and got back 15.0g gum: made up to 200cc stock 5cc: 0.3g found it took 11.7cc $\frac{n}{N}$ NaOH
(instead of 16.8g theory for C₁₀H₁₀O₆) that was acc heated with 22.0g Buein + the dist off ag. sol
got much for Buein! $\therefore$ n.g. $\therefore$ extract excess of Buein by C₁₀H₁₀ but got no x x salts
 $\therefore$ n.g.
The 8.9g alc. sol. acid gum gum [d] = +2.07 (c = 4.45): heated it with 0.1g oxalic acid & H₂O again
back the gum & heated it & heated it with Buein on this basis & found too much Buein
as in other case got nothing x x after ext to excess Buein with C₁₀H₁₀ etc. $\therefore$ now
Set fire gum for both lots to 30.0g Buein. But the gum got back 14.0g gum
d. Alloric gum total which was be acetylated with 75cc acetic anhydride & 1.75g H₂O. The gum got back 29.35g acetylated
gum & melting pt 35 mm Hg. Galactose gum got back 11.75g I: 11.15g II & finally by 33cc 50% H₂O, $\therefore$ discarded
gum to be present in unfractionated milk sugar (see 14 11), Took 500g d. Galactose in 100cc H₂O
(100) d. Galactose 6g (see 14 11), Took 500g d. Galactose in 100cc H₂O
+ add 19g C₁₀H₁₀ in small lots in course of 1 hr that stand in corked flask O.T. for 52 days
Parse for this brown solution O.T. for 3 hrs & then heat to 60° C and got 8.0g ppt. CaCO₃ instead of
25.65g theory $\therefore$ CaCO₃ 17.65g (comp to about 60g sugar) left in solution.
Distill off H₂O in vacuo take up gum in 200cc hot water & add 2 L. of alcohol that stand O.T. 2 days
& then decant clear solution from ppted. Ca salts etc. Got finally 185g A soluble in absol alcohol
[d] (c = 3.7) = +18.75 and containing only 0.002% CaO (i.e. 0.92g gum by ignition 0.002g CaO); then got 154g B
(sol. in absol. alcohol) [d] = +17.4 (c = 3.85) and containing 0.17% CaO; and 33.5g gum B' [d] = +11.2
(c = 6.7) and containing 0.58% CaO $\therefore$ unite B + B' and heat ag. sol. with 1.1g oxalic acid and got
back 190g gum which was dissolved in 100cc H₂O and add 500cc alcohol & resolved by decantation
into 92g soluble sugars B and into 91g ppted sugars C. Gum A dissolved in 50cc H₂O
+ add 250cc spirit & seeding with xx d. Galactose (is giving on long standing a very large amt of
(?) d. tagatose l. soluble x x lls: got after 3 weeks 43.75g x x lls with 140+55 [d] = +80.5 (13.1)
could not sep by xx from 250cc H₂O + 100cc spirit: that all (43.75g) with 400cc H₂O + 12.85cc Br₂ (40g) O.T. for 6 days
Gum A left now = 136g [d] = +8.16 (c = 13.6)
Got also brines gums A, B, and C, 165g ppted. Ca salts containing tar and much
sugar which were dissolved in 170cc H₂O & ppted with

~~and add~~ 500 cc Alcohol & then heat R.F., shaking, then cool & decant & got
 48 g soluble gums and 96.0 g pptd Ca salts. On digesting the 48 g gum R.F. with
 a mixture of 75 cc H₂O + 42.5 cc Alcohol & then cool and decant got for clear filtrate
 (used 0.548) with $[a]_D^{20} = +9.0$ ($c = 5.4$); 2.2 g Ca salts of Saccharin present.
 Add 400 cc Alcohol & then heat

a mixture of 75cc H₂O (used 0.54g)
27.0g gum C with 1.17% CaO and with $[27]^{20} = +9.0$ ($c = 5.4$) $\therefore$ 2.2g Ca salts of Saccharin present.
 The 96.0g pptd Ca salts were now dissolved 2x (N.B.) in 100cc H₂O + add 400cc Alcohol + then heat
 R.F. many shakes cool + decant (2x) and got finally 70g insol. Ca salts. The aq. alc. filtrate
 was now mixed with the insol portion of 48g lot (from which 27g gum C had been removed)
 and total weight found to be 46.0g. There was now digested 3x each time with a mixture of
 + 425cc Alcohol (many shakes) + got finally 32.0g more soluble gum C' with
 + 2.5g CaO (used 0.64g) $\therefore$ 6.14g Ca salts of G. saccharin present
 + 11.0g more

75 cc H₂O + 425 cc alcohol (used 100% CaO (used 100%))
 [X]_D²⁰ = +7.0 (c = 6.4) and with 2.7% CaO (used 100%)
 The insoluble Ca salts dissolved in 15 g H₂O and 60 cc alcohol then poured
 50 cc H₂O + 200 cc alcohol + well stirred with the 70 g pptd Ca salts above; 11.0 g more
 pptd Ca salts which were united with the 70 g pptd Ca salts above; lighter than
 H₂O solution with K₂Cr₂O₇ (n.g.) then made up to 1 l + shake 5 cc :: 0.405 g + got by ignition
 17.35 oxalic acid
 9.43% CaO or 6.73% Ca in Ca salts :: total wt of salts with 15.7 g more
 Saccharin (sol in spirit) and 2.65 g oxalic acid and
 (2.65 g more)

0.0382g Cal: 4.43 gms given
and got Kue etc 4.14 (31.36g + 26.46g left) was
The gms C' and C 57.0 gms Ca free Sugars D.
back 57.0 gms ~~total of 23.3g~~ ~~Sugar separated out on cooling = 7.6g~~ E' with
soluble saccharis gave ~~total of 23.3g~~ ~~Sugar E + E' (and Tar).~~ Saccharins I
semi solid ~~total of 23.3g~~ back 33.3g Hro again digesting

The 41.4 g alc soluble
 added a large amt of insol with E and heat to sol
 $d)_{20} = +5.3$ (C = 3.8) i. unite with Saccharose and got back $d)_{20} = -3.0$ (C = 6.66) the remaining 120 g
 of alcohol from soluble Saccharose 4 with 3 L acetic ether (?)
 distill off alcohol slightly over $d)_{20} = -3.0$ including Saccharose I mix to with 4.0 more
 this solution slightly then uniting with Saccharose and then 4.0 more
 Acetic ether (C = 6.75) containing only traces of Sugar: and then 141.3 g total (oh hand)
 unite A + B Saccharose = 141.3 g total
 b.p. solution; solution:

[illegible][illegible]

13.09 m. 100 g
The ag. alcoholic solutions
which were now digested R.F. with 150 cc
of 10% sugar D¹ (white with D¹ + D²) got from alcoholic for
work. Sugar D¹ (white with other D sugars now are, del as 60.59 cent 0.55
10% sugar D¹ (white with other D sugars now are, del as 60.59 cent 0.55
between 45 and 50% sugar only. Found however on treating rest of ag. sol. of sugar D
that got no ppt. of Ca oxalate and finally got back 60. - grs Sugar. I and II
III. H. m.

The sugar was added by titr. between
and by 0.68g oxalic acid that got up
with 0.68g oxalic acid added more sugar solution
to which was subsequently added more sugar D^{III} and into 25.5g alc. sol. Saccharins C: on
The insol portions I + II (by diff = 33.5g) less now further more
+ cooling into 7.8g insol Sugar R.F. 3x with 1 L acetic ether got 4.95g more
deposited latter Saccharins C (lost fine fine sugar) [d] no = -2.4
mixture with B & above 14.3g

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makes a total of 19.25g Galacto saccharins (most free of sugar). The insoluble tarry
gum, 18.5g from 25.5g alc. sol. Saccharins III was now acylated with 55 cc anhydrid for
8 hrs after solution) on B.W.B. and got 32.5g acylated gums of which 20.4g dissolved in
hot ether and rest 14.5g was an insol tar (discarded latter).
Hydrolysis of the 20.4g ether soluble acylated Saccharins (containing ? sugar) gave
10 hrs c. 42° 14.6g 2nd hyd. gave 12.5g formed about 19 sugar present; 3rd hyd. analysis 9 hrs B.W.B. with 37.5cc 5%
Hydroxy gave 14.6g 2nd hyd. gave 12.5g formed about 19 sugar present; 3rd hyd. analysis 9 hrs B.W.B. with 37.5cc 5%
Underlying this 3x with 1L ethylacetate about 5g of London (discarded rest) & this with other Saccharins above gave a total of 23.1g products
The 7.8g Sugar D III see bottom p 46 was now found to contain only between 10 and 12% sugar
(1.2g used) ∴ distill off & got back 6.6g Sugar D III which acylated with 20 cc anhydrid
B.W.B. 7 hrs after solution gave a total of 12.5g gum acylated of which only 4.9g (on hand)
dissolved in hot ether & gum on by hydrolysis
and 7.9g tar remained undissolved (discarded latter).
The Sugar D 60.5g (p 46 bottom) which had been treated with a small amt of oxalic acid but gave
no ppt of CaC_2O_4 was now freed from water by dist in vacuo so that total weight was 85g (∴ 25g H₂O)
& then added (hot) 250 cc alcohol & got 44.0g of Sugar D and 17.7g alc soluble gums
which dissolved except 13.0g in 700 cc abs. alcohol; on cooling this separated out 13.0g
gum cont between 19 and 20% sugar (to test full solution) (0.5g used) ∴ now acylate the
13.0g lot with 40 cc anhydrid in B.W.B. for 8 hrs after solution and got 22g acylated gums of
which 12.0g dissolved in hot ether waste with 4.9g acylated gum above ∴ 16.9g acylated
gum
The total amt of insol ppted sugar D (including subsequent additions) now found to be 47.0g
& these acylated for 10 hrs B.W.B. (after clear solution) with 181 cc anhydrid gum 86g acylated
gums of which 69.3g dissolved in hot ether (white)
The total acylated (white) gums 69.3g + 4.9g + 12g ∴ 86.2g gum on 10 hrs by hydrolysis (few sol) with 600 cc
H₂O - 63.7g sugars; 2nd hyd. gave 50.7g 3rd hyd. gave 48.9g Sugar D (p 72)
using 300 cc 5% H₂O by full sol = betw 15-20% Malum 0.31g work 2.9 cc 2% NaOH in cold & total of 9.73 cc 2% NaOH
∴ + 6.45 Now distill off the 3rd gum (31g) which was much lactic acid & then by Ba⁺⁺ 31.0g Sugar D (p 72)
6.5g black acylated gum (80° 20 min) which was much lactic acid & then by Ba⁺⁺ 31.0g Sugar D (p 72)
got 21.5g other sol. acylated gums (of all out) which was much lactic acid & then by Ba⁺⁺ 31.0g Sugar D (p 72)
(101) d. Galactose + 1.9g CaO₁₂ (cont of No. 76) The 53.8g acid gums (p 72) = -30° 3 made up to
1L starting 10 cc (∴ 0.538g) took a total of 26.33 cc 2% NaOH ∴ treated Rest of aq. sol on B.W.B. 9 hrs with 25g powder
CdCO₃ & found 8.7g insol CdCO₃ + aq. sol. on cooling some gum & crops total of 37.7g Cd (p 41.07) 2, H₂O
corresp. to 25.8g d. galactonic lactone; the aq. filtrate freed Cd by H₂O etc gave 16.35g gum (alc extr)
[α]_D²⁰ = +2.5 (c = 3.27) gum gave decided test for sugar; the portion left behind in alcohol require
brakes with H₂O etc gum back 6.6g gum and hence Ba⁺⁺ and giving [α]_D²⁰ = +22° (c = 3.3) also gave a
very strong test for sugar: titration taking 0.33g, work 7.62 cc 2% NaOH for 0.33g of lactone = 18 cc + ∴ much
sugar but tar present ∴ discard. (∴ much d. galactonic acid)
Titration of the 16.35g gum [α]_D²⁰ = +2.5, gum taking 0.327g a total of 14.91 cc 2% NaOH
whereas they call for 18.34 cc (∴ for 0.1106).
Rotation of the NaOH of 16.35g lot: now work

The 41.2 g neutral sugars ~~gave~~ $[\alpha]_D^{20} = +2.67$ and about 58% sugar given in xx from H₂O and alcohol. 7.0 g l. sorbose d. gave xx the up 150 $[\alpha]_D^{20} = +22.4$; and the eq. alcoholic solution which had stood all summer gave only 0.83 g non crop II sorbose tetraose xx l. l. now degraded R.T. with 200 cc absd. alcohol. (shakes etc) and into 15.9 g insoluble

The 41.2g gum was dissolved in 70% alcohol & filtered. The solution which had stood all summer gave only 0.83g non-crystalline residue. Now distilled off the alcohol in vacuum and got back 32.15g gum ($d_{20}^{20} = -2.0$) ($c = 6.23$). This gum (free from water in vacuo by dist.) was now digested R.T. with 200 cc absd. alcohol. (shakes etc) when cool decanted and added into 18.15g alc soluble sugar + 20.9g ash free. The 18.15g lot moved up to 500 cc gum & in 10 c tube = ± 0.00 and gave by titr with Feh. Sol between 49 and 50% Sugar (on hand)? ash free) + 3.9g subsequently added = 22.05g. Decant & add alcoholic

The 15.90 g lot made up to 250 cc ($\therefore c = 6.36$) quite a bit of sugar
it contained between 40 and 45% sugar (near 40%) and $\therefore$ obviously has sugar
+ 200 cc absol. alcohol; cool + decant + add a little
tar which contained only 0.65% CaO
6/15/58 lot (see above)
6/15/58 lot (see above)

Now digested gum (free of H₂O) with 200 cc of
solvent cont. 3.9g gum to 18.15g br (see above).
got in portion insol. in alcohol, 9.6g Sugars + tar which contained
1.8g of 20.5g anhydrous gum (see above) 7/100
H₂O 5% 15-20%
H₂O 5% 15-20%

solution in alcohol
 got in portion insol. in alcohol
 on analysis (keep)
 Took $\frac{1}{5}$ of this it melted it after getting rid of H₂O (see No. 43)
 after solution with 14 cc anhydrous ether sol acetylated gums (discolor)
 used in ether soln 5.9g ether sol acetylated gums 21 hrs with 25 cc H₂O & 20 cc sugar later 15-20%
 Took another $\frac{1}{5}$ " 4.1g gums sheeted B.N. 73, cap tube 2.25 g gum (2) $\frac{2}{3}$ = +4.04 + no sugar later 15-20%
 tar gumme acid + aq. filtrate with Ba²⁺ etc get lithium of 0.33 g gum (2) $\frac{2}{3}$ = 7.4 cc $\frac{2}{3}$ NaOH in cold solution of 14.2 cc $\frac{2}{3}$ NaOH (left)
 " now leave only $\frac{2}{5}$ of 20.95g or acc. dec. 12.3 g of ketose sugars
 Now digest gum (11.37g) with H₂O & 22.74g Chlorine
 (cont. see No. 43) 40.53g salts 50° 20 min) which digested with 100 cc
 Chlorine up to 200° - 204° (corresp to 20g lactone); the
 100 cc alcohol gum 20 x x the same
 153g

(102) Mallory + Currier (cont., see No. 43) Now digest gum (11.37g) with H₂O & 22.74g Chitin
for after filter extra the sol 3X with other 40.53g salts (50°-25 mm) which digested with 60 cc
alcohol alcohol gum back 6.92g & dissolved Chitin upr 200-204 (corresp to 20g lactone); the
ale filtrate gum back 27.08 salts which dissolved in 60 cc alcohol gum 20 xx the corn
on seeding with 2 dextro sacch Chitin Xlle ∴ set free gum by 11.78 Buell 20g 155g
Chitin and 8.95g gum ale extr [in hand Oct '12] Took the 11g gum steated BuB with 200 g
Glycerine + Currier (cont., see No. 41). The ale. filtr. gum back giving salts only & then
set ag. sol 3X other ale 30.25g salts ward xx from 60 cc alcohol gum
195-200 ∴ set free gum by 14g Buell 2 gpt
Oct 1912.

Chlorine
 [103] Glycerose 24.0g ext 3X
 Chlorine ext by filter ext ag. ext 195-200; the res. set free from 1912.
 11.7g & boresch. Chlorine ext 195-200; the res. set free from 1912.
 dissolved in 60 cc absol. alcohol extract gave 0 in hand oct 1912.
 Chlorine & 9.3g sucrose in gum (alc ext)
 16.73g Chlorine & 9.3g sucrose in gum (alc ext)
 [104] l. Anabin se & 6.0g Bartha (out from No 77). took the 14.3g acid gum -3.85 & ext
with 2 l alcohol which took not only 2.5g gum id w = -10.8 add to remain for analysis
new gum (14g) with 45cc essigs. analyzed for 6 hrs at 100° after dear solution sgt 2 4.5g acid gum
which 18.3 g dissolved in last ether 1 1/2 hyd wt with 3pts the gum back 11.1g gum 12 with hyd gum
10.2g gum & 3 hyd wt with 31cc 50% H₂SO₄ (13 1/2 hrs no) gum some tar sgt Bar alc 8.15g
gum (alc ext) id w = -9.8. The gum contains very little sugar some ext with
acetone sgt 7.4g gum probably free of sugar (discard).

117g x 100cc abso. alcohol
 dissolved in 60 cc abso. alcohol
 16.73g chlorine & 9.3g saccharine gum (see ex)
 (104) l. natrii & 1/6 cc BaMe (cont from No 77). Took the 14.3g acid gum - 0.1
 R.F. with 2 d. alcohol which took not only 2.5g gum (d) w = -10.8; add to main lot saccharate
 new gum (14g) with 45 cc. essigs. anhydrid for 6 hrs at 100° after clear solution to get 4.5g saccharate gum
 which 18.3 g dissolved in hot ether: 1 1/2 hyd. vol. with 3 pts. the gum back 11.1g gum 12 w hyd. vol. gum
 10.2g gum (ple ext.) (d) w = -9.8. The gum contains very little sugar, some ext. with
 acetone got 2.4g gums probably free of sugar (discard).

∴ heated mixture from No 78, with 2 L ethylacetate & this took out
the rest of the oil

(105) L. Xyloze $\frac{1}{6}$ 29 10 11 12
 3 acid gums (+9.94) next R.F. with
 14.0 acid gums which are
 14.0 acid gums which are

using 33 which still contains
+71 37 neutral genes II (+2, 78) + 18.76 a few genes (discard),
Cas 1, treated with 0.64% xal

The neutral green (descart)
test back 7.0f green
Ce⁺⁺Mn⁺⁺, correct from No 99.
by titr between 75-80% mag.
a shade below 7500 u.

Succinyl

" $\frac{49}{8}$ " " " "

takes $\frac{1}{10}$ th of this D with 2.5 gms. of water
about 90% sugar

- 70% = +5.0 found by titr betw. 15 & 20 gms.
- 60% = +5.0 found " " about 90% sugar

- exalic etc + ych 12g sugar

Had 1568 Σ H^+ cont 0.833% $SD = \frac{6.0}{\sqrt{1568}}$
 Had 278 " I^- cont between $\frac{III}{IV}$ this carded 4
 Had 125 " Σ H^+ by tel + rest 13 cap tube for 18 hrs with 96 cc 5
 2 tube = +0.47; got by Bafate
 acylated gums of

TURK 1st th of 7 & g
acid res. filter w/acetate x 6%
the in other: the 2.2.2. made up to 100 cc
xx the benzal with 110 cc 1% H₂O₄ cap + then for

$\frac{1}{2}$ Dec tube = +0.84.
Other made up to vol by tech
comb (23cc) K in a total of 5.4 cc N₂/H₂.
2000 5% H₂O; got by filter

10.3 g acetylated gums or after extr with
 10.3 g Na acetate + 2.3 cc anhydrous
 8 hrs 100° with 1g Na acetate
 ppt by Ba⁺⁺ + a solid etc. + got vac. 10.3 g
 0.65g lumps
 E: 6.69 g
 10.3 g

with other parcel left 1.9g incl. NaOH + Tar) got 7.5g acetylated from 5 giving large amt of XX of p. Butacetyl. d. given XX etc. $\therefore$ needs up to 100 cc & found α in $\frac{1}{2}$ d tube = $+0.60$ - Hydrol. of this by 40 cc 10% H₂SO₄ then Bomb cap tube gave back Ba⁴ etc. 4.2g given came a shade below 55% sugar (by titer) + giving Vol = 100 cc α in $\frac{1}{2}$ d tube = $+0.44$: by titration of 0.42g using 24 cc $\frac{N}{10}$ alkali formed 5.94g $\frac{N}{10}$ NaOH used up 1 hr OT.

tube = +0.44: by ...
 up 1 hr OT.
Run D - look to the of 49g, 4.9g, treated 18 hrs BMB cap tube with 25 cc 5% H₂O₂ + 50
 got 0.7g luminic acid sap. filter made up to 100cc gave α = +0.29 in $\frac{1}{2}$ de tube. Ppt by Ba"
 in 1 cc acetic acid + 1 cc water 3.3 g gum which with 0.7g Nacacetate + 17cc anhydrid BMB 8 hrs
 gave 1.3 g wool Nac + 1 cc water 4.5g anhydrid gum giving us β Pentaacetyl d. glucose in
 Seeding + giving in making up to 100cc with other α = +0.17 in $\frac{1}{2}$ de tube.
 Got back by Ba" ate Now hydrol by 25 cc 10% H₂O₂ 8 hrs BMB cap tube got back 2.1 g
 gum cont below 50-53% sugar + in taking 0.525 g gum + 30cc $\frac{2}{10}$ alkali found
 + 20cc $\frac{2}{10}$ NaOH used up OT. 1 hr.
 + 7.9g + treated 18 hrs BMB cap tube with 45 cc 5% H₂O₂ + 50
 gave α in $\frac{1}{2}$ de tube = +0.24 $\therefore$ ppt by Ba" in
 + 12 Nac + 23 cc anhydrid + 1 cc

Seedling & growing. Now up to 100% sugar & on taking 0.520 g green
 got back by Ba⁺⁺ rate. Green cont below 50-55% sugar & on taking 0.520 g green
 green cont below 50-55% sugar & on taking 0.520 g green
 11.59 cc $\frac{2}{10}$ NaOH used up at 1 hr.
 Green B 799 took $\frac{1}{10}$ th 7.98 g heated 18 hrs BMB cap tube with 45 cc 50% H₂SO₄
 got 0.93 g tannic acid & eq. filler up to 100% green & in $\frac{1}{2}$ detube = +0.24 i.e. ppt by Ba⁺⁺ in
 presence of tannic acid & eq. filler up to 100% green & in $\frac{1}{2}$ detube = +0.24 i.e. ppt by Ba⁺⁺ in
 green 1.75 g used NaOH + tar & 7.0 g ether sol. acetylated sugar giving brown of pentacetyl sugar
 xx for making up to 100 cc (ether) poured & in $\frac{1}{2}$ detube = +0.50.
 Now hydrolyze green by 35 cc 10% H₂SO₄ BMB 8 hrs cap tube & got back 3.5 g green
 cont below 60-65% sugar & take of 0.42 g using 24 cc alkali took at 1 hr 9.6 cc
 12 g oxalic & thus got Ca free & formed (27) = +7.22 (C=6.4)
 60-65%. Now take $\frac{3}{10}$ th i.e. 8.1 g & then
 with made up

gum 1.150
 xx son making up
 Now hydrolyze gum by 35cc 10% H₂SO₄
 cont b/w 60-65% sugar & titr of 0.42 g using 24cc
 N. N. A. A. discarded rest just 4%
 Gum H¹ 27%, was treated with 1.2g oxalic & thus got Ca free & found (d) = +7° 22' (c = 6.4)
 and found 70% sugar present was between 60-65%. Now take $\frac{3}{10}$ th i.e. 8.1g ~~the~~
 heated B₁B₃. 18 hrs with 45cc 5% H₂SO₄: got 0.83g humic acid & sug. filtr made up
 to wce gum & in $\frac{1}{2}$ de tube = +0° 48'. Ppt by Ba⁺⁺ in presence of acetic & got back
 with heated B₁B₃ 18 hrs with 1g Val + 29cc anhydrous gum 1.8g wool Nal
 in $\frac{1}{2}$ de tube = +0° 76'. Hydrolyze by 50cc 1% H₂SO₄
 in $\frac{1}{2}$ de tube = +0° 41' cont between 60-55% sugar.
 2.075g wool humic

to wcc gun & 10g other sol. acetylated gums (20 x x)
 5.8g gum which made up to wcc gun & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 + 10g other sol. acetylated gums (20 x x) & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 which made up to wcc gun & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 8g gum 4.6g gum made up to wcc gun & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 Gum I, 12g Ca for, 10g gum made up to wcc gun & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 and + 2g. filter wcc gun & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 which with 2g NaOH + 4.8cc anhydrous 8g gum 4.0g gum made up to wcc gun & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 15.5g ether sol. acetylated gums (20 x x) & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 back 7.38g gum cont below 30-40mg sugar + made up to wcc gun & in $\frac{1}{2}$ de tube = +0.7841 & 10g gum
 = +0.7841 & 10g gum

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June B 9th :: 71g was treated with 700cc H₂O & 70g Br₂ 0.7. 7 days etc & got on making
up to 500cc & in 1dc tube = -1.27 ($\therefore [\alpha]_D^{20} = -9.9$), got 64.7g gums (60° 20 mm) which
dissolved in 200cc hot abs. alcohol & set stand 0.7. 4 days gave a small amt of
wood gum which was mixed with a comp. lot from June D (see below) & total wt
of June call B'D' was 6.4g; this gave $[\alpha]_D^{20} = -0.46$ (C = 6.4) & balance of 0.32g +
18cc $\frac{N}{10}$ alkali used up 0.7. 1hr 4.68cc $\frac{N}{10}$ NaOH; sugar balance was only a shade below 25%,
 $\therefore$ much neutral tar present. Now mix B'D' gum with the 52.7g comp.
gums E' 7' 6' obtained from E, 7 & 6 gums see below & det total amt of
E' 7' 6' B'D' gums to be 57.7g; there was now resolved by diss. in 250cc H₂O
(a) $[\alpha]_D^{20} = +3.00$; sugar tetr a shade
below 25% & and ltr of 0.366g + 20cc $\frac{N}{10}$ alkali used up 1hr 0.7. 4.45cc $\frac{N}{10}$ NaOH
and into 42g alc. sol. gums (b), $[\alpha]_D^{20} = -2.5$; sugar tetr below 40-45%; ltr of 0.42g
+ 23.3cc $\frac{N}{10}$ alkali used up 1hr 0.7. 9.48cc $\frac{N}{10}$ NaOH $\therefore$ bal rest of ag. sol. of b with
26g powd CaO & got a total of 46g Ca salts + gums $[\alpha]_D^{20}$ (C = 4.6) = -7. There were
resolved by diss. in H₂O & pptn with alcohol into 17.37g Ca salts I (in mass)
and into 25g Ketose sugars and some Ca salts I (in mass) & 76g Ketose sugars
400cc abs. alcohol gave 14g Ketose sugars
Ca salts - 121 (Ked up as shown in p. 52)
The June D 9th :: 44g was treated with 400cc H₂O & 44g Br₂ 0.7. 7 days etc & got
ag. 3 H₂O etc making up to 1L & in 1dc tube = -0.22 ($\therefore [\alpha]_D^{20} = -5$ approx).
Dissolved off for back 39g gums 60° 20 mm, which dissolved easily in 150cc hot alc
& on standing gave small amt of wood gum (see above). The combined
alc. sol. acid gums B'D' now found to weigh 101g. Balance of 0.505g + 29.1cc
 $\frac{N}{10}$ alkali 0.7. 1hr used up 11.9cc $\frac{N}{10}$ NaOH [$\frac{12}{28}$ this of theory] and balance of sugar found
below 40-45% - therefore bal rest of ag. sol. with 6.6g powd but CaO form for
clear sol 0.7. with some coloration however - after soln of the partly acid sol. distilled
off & made up to 2L gave 2 in 1dc tube = -0.57 $\therefore [\alpha]_D^{20} = -10.6$.
Dissolved off at room temp 110g Ca salts + gums & there pptd 2x by H₂O alc
gum 38.4g Ca salts II, in mass; the 65g gums and some Ca salts found in
ag. alc. filtrates on boiling out R.F. with 200cc abs. alcohol gave 56g Ketose
sugars B'D', $[\alpha]_D^{20}$ (C = 11.2) = -18; cont 66% sugar as det by titr & 0.56g
+ 22cc $\frac{N}{10}$ alkali standing 0.7. 1hr used up only 3.68cc $\frac{N}{10}$ NaOH "dis cord all"
The portion left in abs. alc., 9g by diff, was worked
up for Ca salts etc as shown in p. 52.

Gum Σ 9 this $\therefore$ 59g treated with 540 g H₂O + 60g Br₂ 0.7 days etc then
press H₂O etc making up to 500 cc sugar & in 100 tube = -0.87 $\therefore$ [d] Σ = -80.
Now distill off sugar 56.1g gums which dissolved in 100 cc abso. alcohol & let stand
0.7. 4 days do do. with corresp. gum 7 G see below + got a total of
52.7g insol gums Σ 7 G see p 51 [d] Σ = ± 0.0 ; with sugar ttr bks 40-45%
ttr of 0.527g + 29.1 cc $\frac{1}{10}$ alkali after 1 hr 0.7. used up 11.05 cc $\frac{1}{10}$ NaOH. unite see
p 51.

The main Σ gum sol. in abso. alcohol was united with corresp gum from 7. G.
Gum 7. G $\frac{1}{10}$ this 140g, treated with 800 cc H₂O + 140g Br₂ 0.7 days then
press H₂O etc. making up to 1 L. gum & in 100 tube = -0.66 $\therefore$ [d] Σ = 14 $\frac{1}{10}$ alkali
Now distill off 20 mm + got 118g gums which digested with 400 cc abso. alcohol
0.7. 4 days gum incl. corresp. gum from E a total of 52.7g insol
gums Σ 7 G (see p 51). The alcohol soluble acid gums Σ 7 G combine
now found to weigh 125g; they cont 35-40% sugar; ttr. of 0.625g + 84 cc $\frac{1}{10}$ alkali
used up 1 hr 0.7. 16.95 cc $\frac{1}{10}$ NaOH $\therefore$ 50% of C acids present). Now treat rest of ag.
sol. with 9.4g powder lost Cas 0.7. till clear solution (with some coloration). The
mainly acid sol. now made up to 2 L gum & in 100 tube = -0.52 $\therefore$ [d] Σ = -7.7.
Now distill off sugar 150.5g Casalts + gums which ppld 2x by H₂O + alcohol gave
65.2g Casalts in vacuum desicc. The 66.5g gum cont Ca salts or from ag. etc

filtered, on digestion with 700 cc abso. alc R.7. took out in all incl. 14g from alc
sol Σ 7 G B'D gums (see p 51) 69g Ketose sugars Σ 7 G [d] Σ = -18.0 (C = 13.8)
and cont 60% sugars by ttr. volume of 0.69g + 26 cc $\frac{1}{10}$ alkali 0.7. 1 hr used up
5.19 cc $\frac{1}{10}$ NaOH [dis card] all Ketose sugars Σ 7 G (see p 51). Had left 11.5g alc insol
gums cont Ca salts, which was united with that up as shown on B'D gums 9g
and from Σ 7 G B'D gums 14.0g or in all by diff 31.5g; actual det. of
wt of those gums (gum 43g; this ppld 2x by H₂O + alcohol gave 18.3g
+ 11g Ketose sugars Σ 7 G dis carded) 65.2g + 38.4g + 17.37g + 11.45g
The total amt of ppld Ca salts I II III IV 11.45g Casalts IV
amounts to 132.42g! Now ppt 3rd time from H₂O + alc + got
[d] Σ (C = 40) = +2.76 g cont 7.86% Ca; treat all with 31.1g oxalic acid and got
92g acid gums. 35g insol gums + the latter dissolved in 27 cc H₂O + 300 cc
alcohol gave by decant 29.0g sol gums unite c 62.8g
+ 6.0g insol gums cont Ca + sugar
in last amt $\therefore$ discard

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The combined alc. sol. gives ~~91.8g~~ 62.8g gum $[d]_D^{20} (c = 6.28) = +25.8$ & the
 29.0g gum from $[d]_D^{20} (c = 5.8) = +30.1$ unite 91.8g (as they contain little sugar)
 & treat with Cinchonin & found 100.35g alkaloid dissolved & got in 2 crops
 108.7g d. glucose Cinchonin which with 40g BaCl₂ gave 64.3g crystalline
 + 38.8g d. glucose lactone which xx from 100cc alc. gave 7.6g xx crystals 60-120°;
 6.53g xx crystals 90-130°; 2.2g xx crystals 55-60°; 3.6g xx crystals 50-60°; 1.2g xx crystals 80-110°
 ∴ a total of 26.17g xx of d. glucose ethyl ester + y d. glucose lactone; alc. filtr was
 still giving xx crystals when discarded all.
 The alc. filtr from the 2 crops of d. glucose Cinchonin gave 86g gummy salts & there
 with BaCl₂ gave 34.6g cinchonin & 46.0g acid gums $[d]_D^{20} = +21.1 (c = 4.6)$. This
 gum with 95g BaCl₂ extr 3x C₆H₆ etc gave 20.75g d. Mannoic BaCl₂ salts 210°
 & the alc. filtr gave 131g xx salts which xx from H₂O gave 55.53g furrows xx crystals
 crop I $[d]_D^{20} = -23$ (acc det) & next crop 120-140°; crop I was resolved by further xx into
 into 13.15g xx crystals 130-150° & into 9.25g xx crystals 150-160° $[d]_D^{20} = -18.63$ & these gave
 BaCl₂ etc 3.65g gum $[d]_D^{20} = +17$ and 2.6g gum $[d]_D^{20} = +31.1$. The former gave
 with ph hydr etc 1.83g ph hydr. with 180-190° Fletcher's gum with ph hydr etc 2.57g ph hydr
 next 20° $[d]_D^{20} = +13.36$ ∴ probably d. glucose ph. hydr.
 crop I alc. filtrates & sent by diff 33.15g salts gum with BaCl₂ etc 9.5g gum
 $[d]_D^{20} = +10.5$ & this with ph hydr etc gave 3.8g ph hydr crystals 180-190°;
 The main alc. filtr from crop I salts 55.53g gum on xx 13.55g xx crystals crop II $[d]_D^{20}$
 -22.5 & this with BaCl₂ etc gave 3.7g gum $[d]_D^{20} = +17$ & this with ph hydr etc
 gave 1.94g xx ph hydr crystals 185-195°.
 Then got 9.65g crop III xx crystals $[d]_D^{20} = -22.6$ & this with BaCl₂ etc gave 2.55g gum (alc. extr)
 $[d]_D^{20} = +14.01$ which with ph hydr etc gave 10.2g xx ph hydr crystals 185-195°.
 residue filtr from crop III and 33.1g gummy salts which with BaCl₂ etc gave 9.7g gum (alc. extr)
 $[d]_D^{20} = +14$ & this with ph hydr etc gave 1.98g xx ph hydr crystals 185-195°.
 Now unite all xx hydrazides 2.57g + 1.83g + 3.8g + 1.94g + 10.2g + 1.98g ∴ total = 13.08g & got by
 BaCl₂ etc 6.4g gum (alc. extr) $[d]_D^{20} = +30.5$ which degraded with Cinchonin to crop 8.0g & gave by xx
 10.43g d. glucose cinchonin alc. filtr and only 3.9g gummy salts obviously d. Mannoic
 salt - as we found by cinchonin of gum $[d]_D^{20} = +9.5$ into BaCl₂ salt 10.2g crystals 180-190° off.
 Now extr. all alc. sol. gummy by hydrazides (above) in H₂O sol by other etc got by by BaCl₂ etc
 19g dark brown gum $[d]_D^{20} (c = 3.8) = +9.47$; mixed this with 42g BaCl₂ extr 3x C₆H₆ etc
 gave 14.35g crop I next crop 130-150° $[d]_D^{20} = -23.48$ & 4.0g
 3.9g gum $[d]_D^{20} = +1.2 (c = 3.9)$ which with ph hydr etc gave
 this gum by BaCl₂ etc no xx ph hydr ∴ set free gum again for back 2.8g
 gum $[d]_D^{20} = +2$ & this with Cinchonin etc was
 found to give a xx very sol. in alc. sol. salt
 (discarded).

(154)

the filtrate from crop I salts 53, pour on xx 8.45g xx the crop II impure
 $130^\circ [\alpha]_D^{25} = -22.04$ then 8.45g xx the crop III impure $125 - 140^\circ [\alpha]_D^{25} = -21.07$
discuss all material at this point $\rightarrow$

(107) Titration of Lactulose with $\frac{N}{10}$ NaOH. (a) Took 0.5g lactulose in 50cc H₂O tasted
15 cc $\frac{N}{10}$ NaOH heated B.W.B 10' stirred back finding 12.57 cc $\frac{N}{10}$ alkali had been used up;
now run in 39.2 cc more $\frac{N}{10}$ alkali into the neutral solution heated B.W.B 15' + shake
back and found total amt of $\frac{N}{10}$ Alkali used up was 30.5 cc [theory 28 cc about
for conv. of all Hexose into C₆ saccharin].

(b) Took 0.5g lactulose in 40 cc $\frac{N}{10}$ NaOH heated B.W.B 1 hr + still found much sugar
left in solution as shown by mt. of Fehd sol (qual.).
(c) took 0.5g lactulose in 28.32 cc $\frac{N}{10}$ NaOH 0.7. 24 hrs + shake back and found
only 3.22 cc $\frac{N}{10}$ NaOH had been used up $\therefore$ V.K. Q.E.D.

(108) Lactulose + acetic anhydride. (a) Took 5g lactulose (from Lulin) and 20 cc
cong. anhydrid heated B.W.B 9 $\frac{1}{2}$ hrs (it took 2 $\frac{1}{2}$ hrs for lactulose to dissolve completely)
tst back 10' run 8.5g acetylated gum $\therefore$ incomplete acylation; these dissolved
with 10 cc anhydrid 8 hrs B.W.B 10' 8.5g mobile but acetylated gums; these dissolved
easily in 20 cc warm ether + distilled off other tanning see alcohol to extract
got no xx the $\therefore$ read up to 20 cc with alcohol found α in 10 tube = -0.10 $\therefore$
 $[\alpha]_D^{25} = -1.12$ only. Now distill off alcohol + get back 8.5g gum which heated with
20 cc H₂O in B.W.B. dissolved in part only + next day mostly part incl. that which
sep. out on cooling had gotten entirely solid $\therefore$ can get xx pentaacetylated d. fructose.
Now heat B.W.B 10 hrs with 20 cc H₂O + get back 6.5g gum; 2nd hydr. 8 hrs etc
gave 5.95g gum much sweeter $\therefore$ n.g. $\therefore$ must hydrolyze with 0.25% H₂SO₄ see
No (b) a second exp. with 30g lactulose 10 cc anhydrid + 15g Naacetate showed a
violent reaction with much sweating $\therefore$ n.g. $\therefore$ conversion into 20g material prepared took
place on heating mixture in a hot (100°) bath $\therefore$ n.g.

(c) Took 5g lactulose + 5 cc glacial acetic acid + 20 cc H₂O heated B.W.B cap tube 12 $\frac{1}{2}$ hrs
found $[\alpha]_D^{25} (c=5.0) = -9.3^\circ$ $\therefore$ no decomposition
(d) 5g lactulose + 15 cc H₂O + 5 cc conc H₂SO₄ mixed 0.7. 24 hrs found up
to 20 cc gum $[\alpha]_D^{25} = -97^\circ$ $\therefore$ no action; now distill off H₂O in vacuo 45° + 20 mm +
until total weight in d.f was only 13.8g; solution was now slightly yellow + got more color
on standing 2 hrs. 0.7. found $[\alpha]_D^{25} (c=5.0) = -74^\circ$. Now distill off again to total weight 17.3g
(at 20 mm bath 50°) + then keep bath at 50° for 4 hrs longer + then let stand 0.7. 10 hrs longer
got (c=5.0) $[\alpha]_D^{25} = -64.4$ found % sugar present was between 45-50%.
(e) Took 10g lactulose + 15g Naacetate + 60 cc cong. anhydrid in bath at 80° + got clear sol in 1 hr
heated then 8 hrs longer in 80° bath. Distill off in vacuo bath 60°-80° + shake up residue in
ether: got 19.85g 90° 15 mm gum which could not be got to xx by adding

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alcohol to scratch. Found in heating gun with hot water much dissolved but could get no xxlle.

(f) On heating 50 g lauricose + 5 g Naacetate + 50 cc anhydrid in bath at 80° got clear solution in 3/4 hr. on heating on BMB for 5 1/4 hrs longer gets deep brown in color. Add H₂O, etc with other wash with aq. soda sol got 14.5 g very dark gum which was strongly dextr. $[\alpha]_D^{20} = +15^\circ$ over could not read sharply as was too strongly colored. Now hydrolyzed this gum 2 1/4 hrs BMB cap tube with 150 cc 0.25% H₂SO₄; made up to 250 cc and got d in 1/4 dc tube = -0.53.

(g) Took 5 g lauricose 80 cc H₂O + 10 g best Cals₃ heated BMB cap tube at 60° for 17 1/2 hours: solution did not color + got back 0.9 g Cals₃ + aq. filter made up to 100 cc gum d in 1/4 dc tube = -2.29 ∴ no change

(h) Took 5 g lauricose + 5.0 g H₂SO₄ + 5 cc H₂O + 10 g best Cals₃ heated BMB cap tube at 60° for 66 hrs got dark brown solution. $[\alpha]_D^{20} = -91.6$ is found d in 1/4 dc tube = -2.29 ∴ no change. Add H₂O to make 100 cc + got d in 1/4 dc tube = -3.00 ∴ $[\alpha]_D^{20} = -60^\circ$. Sugar tetralum was between 30-35%? Is condensed sugar. Lactobion found (cf Wold); now make up to 200 cc and heat BMB for 1 hr made up to 250 cc found $[\alpha]_D^{20} = -75^\circ$ (-1.35 in 1/4 tube)

(i) Took 5.0 g best lauricose (r. lundin) + 5 g H₂SO₄ sp. conc + 10 cc H₂O got yellowish colored solution at 66 hrs: made up to 200 cc found $[\alpha]_D^{20} = -78^\circ$ (α in 1/4 tube = -3.9) + found 70 sugar was between 65-70%! Now make up to 200 cc heated BMB 1 hr then make up to 250 cc found $[\alpha]_D^{20} = -80^\circ$.

109 5 g d. glucose + 20 cc Essigsaure anhydrid were heated BMB found it took over 25 hours for sugar to dissolve clear ∴ heated 10 hrs longer after this got 100° warm

10.9 g acylated gums easily sol in 200 cc warm ether. On distilling off starting wce absol. alcohol got 6.35 g xxlle up to 80-110° $[\alpha]_D^{20}$ (c = 6.35) in C₁₁H₆ = +35.6 ∴ a mixt. of 2/3 β Pentacetate + 1/3 α Pentacetate d. glucose (+101°). The alc. filter gum back 3.55 g gum which dissolved in 4 cc absol. alcohol + seeded gum no further xxlle and got 9.9 g sheet BMB 10 hrs

gum (c = 3.55 in alc) $[\alpha]_D^{20} = +35.5$. Now unite gum + xxlle 9.9 g sheet BMB 10 hrs for 7.3 g (after filling a solution) with much H₂O got back 8.4 g gum 2nd hydr 10 hrs for 7.3 g gum ∴ obviously impossible to hydrolyze with H₂O. Q.E.D.

(h) 10 g dextrose + 50 cc anhydrid + 18 g pure Naacetate gum clear solution in 1/2 hr on BMB + heated in all 9 hrs: add H₂O, ether + wash with dil NaOH till alkaline react stops form then dry Coll₂ etc got 15.33 g xxlle up to 110-120° by xx from 50 cc absol. alcohol + alc. filter gum 4.4 g gum (100° warm) from which by xx got 2.48 g more xxlle up to 80-105°. Main product in this case is the β pentacetate up to 130 which is of NaA, 0.5 g finally in above reaction got α pentacetate as product

dodo. if heat β pentacetate at 100° with 4pts. anhydrid + 0.05 parts Zulu fused.
 (c) Took 10g crnd β Pentacetate mpt $100-125 + 50$ cc 1% H₂SO₄ that BrB cap tube
 + got a clear sol in 40' : heated BrB 5hrs in all; made up to 100cc (c = 4.62, α_D^{20}
 2 in 10 tube = $+2.49$: $\alpha_D^{20} = +53.9$: complete by analysis D.E.D. Now ppt 50%
 by Ba" + got 4.6 g d. glucose from which dissolved in 2cc H₂O + add 10cc alcohol from give
 seeding large amt xxlle of d. glucose for 2.87g mpt 156° -
 seeding large amt xxlle of d. glucose for 2.87g mpt 156° -
d. Galactose + Essigs. anhydrid. (a) 5.0g d. galactose (or : impure) + 20cc anhydrid

110 Took BrB 10hrs to give clear solution then on heat 10hrs longer + got (100° 20 min)
 10.7g acetylated sugar easily sol. in 170cc warm ether; on dist. of adding 10cc alcohol
 alcohol got no xxlle in scratching; made up to 100cc with alcohol + got 2 in 10 tube
 = $+3.06$: $\alpha_D^{20} = +28.6$. Found could not hydrolyze this from with H₂O in BrB (c = 13.8-14.2
 No 109 108). β pentacetate mpt $138-142$
 (b) Took 10g orl. galactose + 40cc anhydrid + 1g fused Naacetate + got BrB a clear sol in 15'
 + got 8.10g d. galactose + 40cc anhydrid + 1g fused Naacetate + got BrB a clear sol in 15'
 + got 8.10g d. galactose + 40cc anhydrid + 1g fused Naacetate + got BrB a clear sol in 15'

Tris browning : least 4hrs longer + add H₂O other did NaOH + got by xx of neutral
 from alcohol 8.10g β pentacetate mpt 142° in colorless plates
 besides 8.7g sweet colorless acetylated sugars very sol. in cold alcohol. alcohol
 + giving 2 in 10 tube = $+3.68$ in alc. 100cc, 8.7g. In a previous expt when
 30g galactose 15g NaOH + 100cc anhydrid was used a violent reaction took place with
 much browning in BrB + got 40.7g brown neutral from which got 9.1g ap.
 β pentacetate in plates mpt 142° of Kemp's Ber 22 2207

(c) Took 10g of the pure β Pentacetate mpt $142^\circ + 50$ cc 1% H₂SO₄ BrB cap tube &
 from for clear sol. in 45' : heated 5hrs in all then made up to 200cc (c 4.62) + got 2 in
 10 tube = $+3.73$: $\alpha_D^{20} = +80.7$: complete by analysis. Now ppt 50% by Ba" + got
 4.6g from which dissolved in 2cc H₂O + add 10cc alcohol from seeding 3.6g xx
 d. galactose mpt $163-50$ D.E.D. with lactose!
d. Galactose + 50% H₂SO₄. Took 5g orl. d. galactose + 30cc 5% H₂SO₄ that BrB cap

111 tube for 11½ hrs + got no lactic acid + yellow colored sol. made up to 100cc gave 2 in 10
 tube = $+3.68$: $\alpha_D^{20} = +73.6$: very little change &
 l. Arabinose + 5% H₂SO₄ : an entirely analogous expt with 5.0g l. Arabinose heating
 8 hrs BrB gave some browning + on making up to 100cc gave 2 in 10 tube = $+5.0$: $\alpha_D^{20} = 100^\circ$
 : no change.

112 l. Xylose + 5% H₂SO₄ ; an entirely analogous expt. with 5g l. Xylose 8 hrs BrB gave
 some browning + on making up to 100cc gave 2 in 10 tube = $+4.05$: $\alpha_D^{20} = +21.8$: no change.
113 Trehalose of yd. Maumic lactose ; took ap. Fischer's mpt $149-152^\circ$: $\alpha_D^{20} = +51.02$ &
 found 0.41035 xx in H₂O + add 29.3cc $\frac{1}{2}$ NaOH + 23.55cc $\frac{1}{2}$ NaOH required (V₀ = 2.304cc)
 to decolorize alk. sol. colored by alk. : 23.55cc $\frac{1}{2}$ NaOH required 28.45cc $\frac{1}{2}$ NaOH
 a duplicate expt dodo. he let stand 6.7 1½ hrs required 28.45cc $\frac{1}{2}$ NaOH
 : can take all lactones of 1hr - D.E.D.