

Index - Sugar Expts 1907-1910

The University of Chicago

d. Galactose $\frac{11}{10}$ BaO $\frac{1}{2}$ (4)
 7.5% NaOH (4)
 $\frac{5}{4}$ N BaO $\frac{1}{2}$ (21)(9)(13)
 CaO $\frac{1}{2}$ (16) (27)(37)
 33% NaOH (17)(19)(51)(57)
 $\frac{3}{80}$ N NaOH (28)
 $\frac{11}{28}$ BaO $\frac{1}{2}$ (29)

d. Glucose 5% NaOH (5)
 $\frac{5}{4}$ N BaO $\frac{1}{2}$ (11)
 33% NaOH (18)(23)(52)
 CaO $\frac{1}{2}$ (40)

d. Fructose $\frac{5}{4}$ BaO $\frac{1}{2}$ (11)
 CaO $\frac{1}{2}$ (41)

Maltose $\frac{5}{4}$ N BaO $\frac{1}{2}$ (3)
 CaO $\frac{1}{2}$ (38)

Lactose CaO $\frac{1}{2}$ (39)

Dulcose 33% NaOH (30)

L. Xylose 33% NaOH (35)(64)(70)
 CaO $\frac{1}{2}$ (43)

L. Arabinose 33% NaOH (34)(68)
 CaO $\frac{1}{2}$ (42)

d.l. lactic acid $\left\{ \begin{array}{l} \text{oxid by Chlorine (33)} \\ \text{Baum salt (12)} \\ \text{ph. hydrazine salt (10)} \\ \text{acetic anhydride (15)} \\ \text{ext. by ether from aro sol (36)} \end{array} \right.$

uridine acid $\left\{ \begin{array}{l} \text{acetyl deriv} \\ \text{ph. hydrazine} (71) \\ \text{solubility} \\ \text{Baum salt} (81) \end{array} \right.$

d.l. 1-Oxy butyrolactone $\left\{ \begin{array}{l} \text{oxid to ampoules (32)} \\ \text{oxid to d. malic acid (45)} \\ \text{oxid of d. form to d. malic (63)} \\ \text{phenylhydrazide (11)(17)(20)} \\ \text{Styphurine salt (17)} \\ \text{Baum salt (4)(17)} \\ \text{Chinin salt (17)} \\ \text{Zn SO₄ salt (17)} \\ \text{ext. p. aro sol. by ether (36)} \end{array} \right.$

d.l. 2-Oxy butyrolactone (46) $\left\{ \begin{array}{l} \text{Baum salt} \\ \text{ph. hydrazide} \\ \text{oxid to d. malic acid} \end{array} \right.$

d. Galacto metasaccharine
 mp 144°

β d. Galacto metasaccharine
 Chlorine P₂S₅ saccharine

d. dextro metasaccharine

β -d. dextro metasaccharine

$\left\{ \begin{array}{l} \text{Ca salt} \\ \text{Baum salt} \\ \text{Styphurine salt} \\ \text{Chinin salt} \\ \text{ph. hydrazide} \\ \text{Ba salt} \\ \text{shifting to p (20)(6)(58)} \\ \text{reprec. anhydride (25)} \\ \text{oxid by HNO₃ (55)} \\ \text{Baum salt (51)(11)} \\ \text{Styphurine salt (17)(54)} \\ \text{Chinin salt} \\ \text{lactone analysis (57)} \\ \text{Ba salt} \\ \text{ph. hydrazide} \\ \text{oxid by HNO₃ (51)} \end{array} \right.$

$\left\{ \begin{array}{l} \text{shifting d to 3-60} \\ \text{Baum salt (51)} \\ \text{Ca salt 18(23)} \\ \text{ph. hydrazide (18)(23)} \\ \text{Styphurine salt (23)} \\ \text{Chinin salt (23)} \\ \text{lactone (23)} \\ \text{oxid by HNO₃ (54)} \\ \text{ext. from aro sol by ether (48)} \end{array} \right.$

$\left\{ \begin{array}{l} \text{lactone (23)} \\ \text{Baum salt (23)} \\ \text{ph. hydrazide} \\ \text{Chinin salt (23)} \\ \text{oxid by HNO₃ (53)} \end{array} \right.$

Shifting xpt 1060

call β in paper

call α in paper

α d. Dextro Saccharin
(α CH₃ d. arabinic lactone)

Brucein salt (21)
lactone (21) (41) (α 7)
Shydrine salt (21)
Chinine salt (21)
ph. hydrazide (21)
gastic acid (26)
salt from H₂O by ether (49)

2-oxyruenic lactone

from α galactometasaccharin acid
" α dextro metasaccharin
" β " " "

α Isosaccharin
α oxy methyl l. thio Sae

Ca salt
Chinine salt (24)
ph. hydrazide (24)
Shydrine salt (22)
lactone (22)
oxid by HNO₃ (64)
Soyuz salt (24)
salt from H₂O by ether (47)

dl 5 anthro Brucein (31)

d. thro C₅ Saccharin

oxid to 1,3 dioxo glutaric acid (61)
Chinine salt
ph. hydrazide
Brucein salt
lactone
Na salt
salt from H₂O by ether

Formose 33% NaOH (71)
CaH₂ (44) (73)

l. anthro C₅ saccharin

Brucein salt
ph. hydrazide
oxid by HNO₃ to meso dioxo glutaric (65)

l. Malic acid (62)
groups of
acid
Na salt
Brucein salt
Chinine salt

l. thro C₅ Saccharin

Brucein salt
ph. hydrazide

d. anthro C₅ thro gum

Ag salt (64)
Chinine salt (69) mixed with d. thro salt (72)
oxid by HNO₃ (67)

d. anthro C₅ Saccharin

oxid to meso 1,3 dioxo glutaric acid (66)
Brucein salt
ph. hydrazide
lactone

Sugars ex/ps-II

The University of Chicago

19 Lauridin $\frac{5}{2}$ N/3 at $\frac{1}{2}$ H₂O. $\frac{1}{2}$ 50g a. fine etc. (leaving trace Ba²⁺) transp
 heated 100° (20 min) 10 hrs and then add 90.5 cc conc H₂SO₄ etc. (100 min) [lactic + dihydroxy butyric]. Heated this gum
 aq. filtrate in open dish sept only 29.15 gum [lbs of lactic + dihydroxy butyric]. Heated this gum
 with 87.3 cc acetic anhydride (7 hrs 80-90°) sept 45.25 acetylated gums (100° vac) + this with
 ether (2x 300 cc) left 95 insol tar.
2nd Expt using 50g lauridin, 145.5g Ba(OH)₂ 8H₂O and 750 cc H₂O (16 hrs, 100°) stirred with H₂SO₄
 etc. sept, evap. open dish, 27.8 gum [C₃ & gums volatile?]. By acetylation got 40.58 gum which
 2x ether left 11.35 insol. Tar. 11.35 better use more alkali as in 1st expt. The combined
 these digested 2x R.T. with 500 cc ether took out 19.85 (acc. 16) C₃ & gums; the
 remainder, 17.55, dissolved easily except 15 tar in digestion R.T. with 500 cc
 acetic ether [C₃, C₆ gums]. A 2nd acetylation of the 19.85 gum gave 29.1g acetylated
 gums where on shaking 10' with 100 cc cold H₂O (in sep. funnel) ~~the~~ gave 16.5g
 gum, sol. in H₂O, or after 2x hydrolysis got 11.7g mobile C₃ & gums where 9.6g
 acetylated gum remained insoluble which on 2x hydrolysis gave 6.7g C₄ gum or
 the 11.7g C₃ & C₄ gum with Zulez etc a total of 9.65g Zulez etc 3 H₂O
 comp. to 6.0g dl. lactic acid. discarded rest of material. (mainly C₃, C₄, C₅ gums)
 2 expts, as in 1; except use 50g d. galactose in each case.
 which digested with ether, left only 5g insol tar.

29) Salutator $\frac{5 \text{ in Baht} + 2}{4}$

2) Solvent SS March 12
I, quat 30g gums (open dish) or 44.2g acylated gums, which " "
II " 27.5 gum (" ") or 42.1g " " , which " "
" I bust. lot of combined acylated gums sol in ether was 70.4g where ex hydrolisis gave 44 g gums
which ext^x R.F. so decather took up ether ext^x acc det [23.3 g]; unol. portion in ether
1x with sove acyl ether left only 1g insol tar and found acc. det of acetic ether portion
wt was 17.2g. Acylation of 23.3g br gum 34.6g gums or 17.2g br gum x hydrolisis of
the combined acylated gums, 57g were shaken with 5 pts cold H₂O & filter off; lydsolysis of
portion soluble in H₂O gum 16.6g gums + of acylated gums, unol in H₂O, gum 17g gums;
the latter gum was xx of Met a saccharin. [dis carded].
2 x pts, as in 1^o, except use 50g Maltose each time.
the other left 9.8g insol tar.

3^o) Malton & Bant, Eⁿ

I gum 31.4g gums + by acetylation got 40.8g " " " " " "
II " 35.6g gum + " " " " " " " " " " " "

The combined acetylated gums in ether = 89.9g there 2x hydrolyzed gave 52.8g acetic ether
Use combined acetylated gums in 1X digestion with 500 cc acetic ether or acc del had 52.8g acetic ether
except 3g tar, in 1X digestion with 500 cc ether found 57.2g C₃C₄ gums dissolved
in digesting this 3x with 500 cc ether found 57.2g C₃C₄ gums dissolved
(acc. del)

recrystallized each portion again & took each recrystallized gum with 5 pts cold H₂O & got
from combined aqueous filtrates by 2x hydrolysis 26.4g gum & from most recrystallized gums
in hydrolysis got 19.3g gums. [discovered] Oct 10 - Dec 9-07
[4] Galacton 7.5% NaOH. Took 180g d. galacton, 270g NaOH ^(100%) 3.6L H₂O
Heat Bubb 14 hrs (war) sudd 1,125g 21.7% NaOH etc. got (acc. det) 471.8g gum
which extr 3x with 500cc acetic ether took up [acc. det] 100g gums leaving 71.8g
insol gums + NaOH. The acetic ether solutions on standing gave 5.6g Metasaccharin
upt 144°. The filtrates gave 93.0g gums which extr 2x with 1L ether from
took up 58.9g C₃ C₄ gums (very mobile), leaving 34.1g insol gums, out 2x of Metasaccharin
The 71.8g gums + NaOH ^(if enough) 46.5g dissolved these in 2x hydrolysis gave 27.1g gum
on digestion, 3x, 500cc ether, 46.5g dissolved these in 2x hydrolysis gave 27.1g gum
containing 2x of Metasaccharin: combine with 34.1g C₃ & filtered off, after adding
sufficient of acetic ether, 6.05g Metasaccharin [4.6g from 27.1g C₃ & 1.45g from 34.1g C₃]
The total amt of gum found in filtrates now det acc as 50g which on recrystallization
gave 84g gums, which on steaming with 5 pts cold H₂O & insol in ether extract was lower
was reduced into recrystallized gums sol in H₂O & insol in ether extract was lower
lots from 58.9g C₃ C₄ gums (above) this 58.9g gum
first treated as follows: dissolved in H₂O, pass steam thru. sol & poured some acids
an volatile but only in small amounts by steam as shown by following:
1st - 990 cc aq. det: took 25 cc of this and found 9.17cc (0.999) ¹⁰/₁₀ K₂H₂O necessary
2nd - 825 cc " " 25 cc of this and found 3.17cc (0.999) ¹⁰/₁₀ K₂H₂O " "
3rd - 575 cc " " 25 cc of this and found 1.87cc (0.999) ¹⁰/₁₀ K₂H₂O " "
4th - 555 cc " " 25 cc of this and found 1.88cc (0.999) ¹⁰/₁₀ K₂H₂O " "
Now took all aq. distillates left & treated these up to 18.76g of Brucine etc & found
the Brucine salts obtained dissolved easily in 30 cc hot alcohol & making amount of
ether got 2x needles. Poured subsequently that both lactone & 13 dirty butyric acids are
carried over by this operation: a mixture of these acids from others: now take orig aq. sol
used is obviously n.g. for sep. of these acids from others: now take orig aq. sol
thru' which steam had been passed & got back (value 100) 52.3g C₃ C₄ gums; thus on
recrystallization gave 81.1g gum & from insol oils got 48.6g gums.
The 36.2g gum treated with ZnCO₃ etc now gave in all only 73.2g galacton 6 3tho XX: n.g.
and in getting rid of Zn from aq. filtrate gummy salts got back 3.15g gums which on steaming
will in cold with 150cc ether left 17.8g insol gum which in turn easily dissolved
(except 3g tar) in one digestion with 300cc acetic ether. The 48.6g gum digested 6x R7
with 500cc ether dissolved 23.2g; the other acid solutions on standing gave 0.5g Metasaccharin
XX and now got back from after making up to 1L & taking 20 cc: 0.44g gum full results.

The University of Chicago

40.77 cc $\frac{n}{10}$ KOH ^(100° men) necessary: [theny for 0.445 C₅S each = 43.8 cc] $\therefore$ nearly C₅ gums.
 Now distill off H₂O from resin aq. sol ~~of~~ & shuck gum left (22.08 - 0.445)
 very thoroughly in cold with 150 cc ether and found, by difference, that 7.2 g dissolved;
 the remainder of insol oil now found must all dissolved with ease in 500 cc rectified ether.
 These solutions were now united with concp. lts from 31.5 g gum (see above) ether
 by H₂O etc. $\therefore$ Had 23.9 g cold ether extracts and 25.9 g rectified ether extracts, not
 including portion left undissolved from 48.6 g after 5 x extr with ether etc, which
 took out 23.25 gum. This portion ~~48.6 - 23.25 = 25.35~~ was now dissolved 2 x with 500 cc rectified ether
 and all but 2.4 g tar (discard) dissolved; in cooling got 2.2 g of solid (see above, 100° men).
 100° men gum back 20.49 C₅ & gums: titration making up to 500 cc taking 10 cc $\therefore$
 0.408 g took 28.644 $\frac{n}{10}$ KOH [theny for 0.408 g C₅S each = 30.90 cc]
 now take rest of aq. solution ^{strong with glycolic} 55.4 g Bumein etc got 788 salts which xx from
 78 cc aq. gum 21.63 g crop I of crude β Metasaccharinic Bumein wpt 120° - 137°; and
 7.05 g, crop II of same salt $\therefore$ total of 28.68 g salts. These salts heated with BaCl₂ etc
 gum 18.1 g Bumein & 5.35 g crude β Metasaccharin. Took 1 g of this & heated it in cold
 with 1 cc aq. 57 cc ph. hydrazine (8 hrs). On scratching this got entirely solid & on adding ether
 & trying to filter got sticky $\therefore$ much loss [cf. Expt. 90, 1904-7]: it is likely that working
 with cold alcohol or rectified ether would work better $\therefore$ probably β Metasaccharin
 - identical with Kiliani's Parasaccharin. The alc. filtrates from crops I & II
 β Metasaccharinate of Bumein gum 40.45 g gummy [C₅ Bumein salts.
 cold ether extract, 23.9 g see above was now 1st heated with Zuev's etc & got only
 3.425 g xx zinc acetate 3 H₂O $\therefore$ n.g. $\therefore$ got out 16.65 g gum (100° men).
 titration making up to 250 cc take 5 cc $\therefore$ 0.333 g gum 33.53 cc $\frac{n}{10}$ KOH necessary
 [theny C₅S each = 30.43 cc] $\therefore$ had rest of aq. sol with 64.78 Bumein got 87.5 g salts
 by xx from alc got 13.2 g wpt 192-4° and 18 g wpt 185-190° of pure gum 1,3 dixy
 butyric Bumein other crop II 21.6 g wpt 208-210° of pure gum dl. lactic Bumein
 and then crop IV 5.45 g of pure 1,3 dixy butyric Bumein.
 Now prove the absence of glycollic Bumein in alc. filtrates with precision as follows:
 had only 17.2 g gummy Bumein salts left which were united with the 54 g gummy
 Bumein salts obtained from rectified ether extracts, 25.9 g gum (see below). These
 gum with BaCl₂ etc 46.78 Bumein and 125 gum [1 x 300 cc rectified ether]; the 125 gum
 digested 1 x R.F. with 300 cc rectified ether left 4.3 g insol C₅ gums & the ether sol.
 gums $\therefore$ 7.78 gum up in drying with excess of puror. Styracine (3 hrs BaCl₂)
 a total of 22.75 g much degraded except 5.3 g dense. Styracine wpt 288°
 wpt. H₂O easily in hot alcohol but pure

no xx of gly collie styphium on standing: set fire acids again by Ba₂H₂ & got
back 6.9g mobile gum; make up to 750cc & take 10cc: 0.276g & found 26.94cc $\frac{n}{10}$ KOH

necessary [they for C₄ saccharin = 27.24cc] gly collie carboxylat.
Rest of aq solution now loaded with 95.5g Buein etc gum 12.55g and 1.3
Dioxy butyric Buein whereas rest of salts are gummy ~~in low melting gums.~~
Acetic ether extract, 25.9g gum; made up to 500cc & take 5cc (0.259g)
took 24.38cc $\frac{n}{10}$ KOH [they C₄ sacch = 25.39cc] $\therefore$ treat rest of aq. sol. with 95.5g

Buein etc & got 143g Br salts; these gave a total of 61.64g xx Buein salts, esp
13 Dioxy butyric Buein ~~upt~~ in 3 fractions, I 13.3g, II 32.34g, III 16.0g
upt 140-180° - stable. filtrates gave 54.8g gummy salts which were
united with 17.2g gummy salts ~~or. from other expts~~ [see above].

1,3 Dioxy butyric latten; properties: took 17.53g of purest 1,3 Dioxy butyric Buein

[obtained from ether sacche ether extracts above] upt 194-6° & got with 8.04g Ba₂H₂ etc
13.02g Buein (as dry) & 2.85g latten as yellow mobile oil; this mixed with 3cc
phi. hydrazin & 3cc ac. sol. & then o.t. so has got solid with xx needles. Added much ether
& filtered off Hydrazin, which was sticky, put in sp. funnel with sun. amt. acetic ether
till suns white (much loss, water in acetic ether). got 2.5g crude material & this in xx
from 100cc acetic ether gave 1.5g suns while very bulky needles of dl 13.
Dioxy butyric phenyl hydrazin upt 130-132° which keep indefinitely [analyses, see
no 7]. Took the rest of the crude 1,3 Dioxy butyric Buein 6.67g
upt 140-190° & got with 8g NaOH etc 50.5g Buein (as dry) & 6.75g gum [ether expts, 1X only].

This was acylated (6 1/2 hrs 80-100°) & got 9.6g acylated gum which shatters in
etheral solution with ~~acetic~~ aq. sol of 5g Na₂CO₃ (100°) in 100cc H₂O. got 3.95g washed
acylated gum which on dist at reduced pressure decomposed in great part: n.g.

50% Dextrose and 50% NaOH (Oct 12, Dec 16, 07). 125g Dextrose 187.5g NaOH (100%) &
882g 19.4% H₂O etc; 2 expts of 125g each
3.75L H₂O heated Boil 14 hrs (no air) and 134.5g gums [in Acetic ether ac. det].
got about 169.9g gum (1X/1200 cc alcohol); in extracting this 3X with 1L acetic ether
had left 28.9g insol. tar & found had 134.5g gums [in Acetic ether ac. det]. This gum
is probably much lactic & dioxy butyric carried off this volatilization. This gum
dissolved 3X with 1L ether dissolved 58.1g (ac. det) leaving 76.4g insol. gum
acylation of 58.1g got gum 81.6g gum + of 76.4g got gum 111.5g gum of which 18g was
insoluble in hot ether: discarded. These acylated gums were washed with 5pts
cold H₂O & filter off insol oil etc. got by hydrolysis of acylated gums sol in H₂O 31.8g
C₃ & gums and gum insol oils got 76.7g gums. On shaking the 31.8g with 20cc
ether in cold found 14.95g (ac. det) & had dissolved and the soluble oil when
treated with 20cc 3% NaOH (by H₂O etc) gave only 5.7g xx Zela taste 3H₂O
which treated with S.T. etc

The University of Chicago

The portion of the orig. oil, 31.8g, left insoluble in cold ether: 16.85g, was discarded.
 The 76.7g gum ~~left insoluble in ether~~ obt. from by sol. insol acylated gums
 gum by 5x extr with 50cc ether 35.5g (acc. det) soluble gums, which must be
 mainly C₄ saccharins, for it gum in one run by dist. with 50cc H₂O 34.7g
 gum; this made up to 50cc state 5cc (: 0.347g) required 33.23cc $\frac{n}{10}$ KOH
 [theory for 0.347g C₄ saccharin = 34.0cc]. Now distill off H₂O from main portion &
 extract gum left in cold with 50cc ether and found 22.9g (acc. det) had dissolved
 leaving the for 10.3g very thick insoluble gum. The portion sol. in ether was a very
 mobile oil, 22.9g; made up to 50cc state 5cc (: 0.219g) and found 23.4cc $\frac{n}{10}$ KOH necessary
 (theory C₄ sacch = 22.4cc) : take rest of aq. sol strat with 87.5g Buem 116g Buem
 salts; then xx from a/c and got a total of 34.9g d.l. lactic Buem (in 2 crops), in Buem
 from which by Buem etc got 54.5g gum str. 7.03g xx galactate 3tho
 [N.B. notice that this gum may come from acylated gums used in H₂O]. The alc. filtrates
 from dl lactic Buem gum 75.9g gummy & very soluble Buem salts from which
 got by Buem etc 13.35g very mobile gum (probably lactic + 13 Dixy lactic in main).
 Titration of the 10.3g insol thick gum made up to 50cc state 10cc (: 0.206g)
 gum 17.7cc $\frac{n}{10}$ KOH [theory for 0.206 C₄ sacch. = 20.2cc] : C₄ C₆ gums; take rest of aq.
 with 49.5g Buem solution strat with 34.8g Buem 43.0g salts which gave us
 xx but gummy only : convert back to 6.3g acid gums (discarded)
 C₆ gums; the orig 76.7g gum, ext 5x by ether, should contain 41.2g C₄ C₆ gums : extract
 these 3x R.F. by 50cc acetic ether and found all but 2g tar (discarded) dissolved; on distilling
 off solvent we then got 38.6g gum (which gave no xx of Saccharin) : titration making up to
 10cc state 10cc, : 0.386g, took 27.36cc $\frac{n}{10}$ KOH [theory C₄ sacch = 29.24cc
 23.8cc]
 : treated rest of aq. sol with 10.67g Buem 75.6g salts which xx fine alcohol
 gum in all two crops of xx needles, 40.7g up to 10-140 from this got back 70.35g
 gum (acetic extr) : the rest of salts were gummy only : add Buem etc got back
 21.7g gum [action of acetic ether extr]. On dissolving the 10.35g gum from xx for salts
 in 400cc acetic ether it stood with xx of Saccharin, but no trace of gum xx etc : stable
 off solvent again got gum, 10.35g, R.F. this with 100cc acetic ether cool & decant
 and found only 3.6g gum had dissolved; the rest sol. & insol parts with comp. bts
 from 21.7g gum (discarded). Now extract the 21.7g lot 8x R.F. with 200cc ether
 and found 7.55g dissolved mainly C₄ gums; this made up to 250cc state 10cc (: 0.302g)
 + found 25.2cc $\frac{n}{10}$ KOH necessary [theory C₄ sacch = 29.6cc] discarded
 The insoluble portion, 14.15g, was now digested with 4x R.F.
 with 200cc acetic ether and found 2.3g insoluble gum.

Now distilled off solvent and now resolved gum left, about 11.8g, by digestion R.F. with (acetic ether small amt) into an easily soluble gum + a difficultly soluble (in acetic ether) gum uniting with curcup. lots from 10.35g by sec p. 5) ^{discovered}
 ∴ the org 10.35g + 21.7g now split into 3 components (10) Ether ext to 7.55g
 (20) 10.2g easily sol in acetic ether (3) 13.05g diff. sol. in acetic ether.
 took 1/3rd of (3) and treat with 1cc ph. hyd. + 1cc ac. ether stand 24 hrs. o.t. to get a xx ph. hyd
 but n.g. ∴ took gum (3) left ac. det = 12.15g and made up to 250cc + take 10cc
 in 0.486g, and found 30.1cc $\frac{n}{v}$ K_{OH} necessary (= chem & sacch) ∴ treat rest of
 aq. solution with 28.5g Buein and got 4.2g Buein salts; on xx these from 100cc H₂O
 got a rapid separation of 20.17g fibrous wooly needles mpt 165°; on xx these from 100cc H₂O
 and 100cc alcohol got 15.75g very porous fine needles mpt 175° ∴ treated this
 salt in aq. sol. with NaOH + got 10.8g Buein (anhyd) salt mpt 175° ∴ treated this
 this is ~~β-Dextro~~ ^{α-Dextro} ~~metasaccharine~~ ^{metasaccharine} [absolutely free from Saccharin]. It don't get xx ∴ treat on
 ac. sol (5cc) with 4.2cc ph. hydrogen on 24 hrs. o.t. got entirely solid in scratching; got
 4.8g crude ph. hydrogen salt washed with cold acetic ether. On xx this from 30cc alcohol +
 100cc acetic ether got 3.5g very heavy xx needles mpt. 124° - 6°. Finely xx this from 15cc
 hot absol. alcohol for analysis + ph. in water got very quickly fine. needles, dextro, schwan
 xxle 3.3g with mpt 124-6° which lose on drying H₂O in vacuo.
 0.2446g gum 0.478g CO₂ and 0.147g H₂O
 then for C₁₂ H₁₈ N₂O₅ Found
 C 53.33 53.30
 H 6.67 6.68

0.9954g subst + 19.7861g H₂O ∴ p = 4.79; d = 1.013; found α in 2d tube - 3, 14
 ∴ [α]_D²⁰ = -32° 37'. N.B. The ~~α-Dextro~~ ^{α-Dextro} ~~metasaccharine~~ ^{metasaccharine} ph. hydrogen salt, ^{repeated later see No 24}
 26, 1905 (9.11) gave [α]_D²⁰ as +19.6° ~~not accurate~~ ^{α-Dextro metasaccharine} Buein still contained 18.9g
 The alcoholic filtrate from crop I ^{α-Dextro metasaccharine} 11.46g crop II wooly needles containing
 Buein salts which xx from 1cc H₂O + 19cc alcohol got 120°-145° + lose 0.23g H₂O
^{α-Dextro metasaccharine} Buein - these have mpt 120°-145° + lose 0.23g H₂O
^{α-Dextro metasaccharine} Buein - these have mpt 120°-145° + lose 0.23g H₂O
 during in vacuo. On xx this crop II from 5cc H₂O + 75cc alcohol this separates out again
 very slowly ∴ can't be entirely free from ^{α-Dextro metasaccharine} Buein salts ∴ set
 free by NaOH got 5.35g gum which no saccharin could be detected; in treatment of
 the gum with CaCl₂ etc got a gummy Ca salt only (! ∴ must contain 3 body).
 (2) Had 10.2g gums easily soluble in acetic ether; made up to 250cc + taking 10cc for titration
 (∴ 0.408g) and found 27.5cc $\frac{n}{v}$ K_{OH} necessary. (chem for C₆ sacch = 25.2cc) ∴ treated
 rest of aq. solution with 26g Buein got 38.5g Buein salt, xx these from 38.5cc
 alcohol got 12.68g heavy xx needles, grouped & clear, with mpt 130° - 150°; on rexx these
 from 6cc H₂O + 75cc alcohol got quickly a large amt, ^{α-Dextro metasaccharine} Buein with mpt 135-140°; this salt gum with NaOH
 of ~~β-Dextro~~ ^{α-Dextro} ~~metasaccharine~~ ^{metasaccharine} the 6.07g Buein and 1.85g and a ~~β-Dextro~~ ^{α-Dextro} ~~metasaccharine~~ ^{metasaccharine} (which
 end not be fit solid ∴ treat it with

from all 22g Bismuth left got back 3.85g gum which with $CaCO_3$ etc
gave no xx of α -D-glucosaccharinate of Ca; it may contain saccharin
(6) α -Metasaccharin, and Hydroxyphenylhydrazine; 10g Metasaccharin, m.p. 144°, 50 cc H₂O
10 cc pyridine heated in S.T. 3 hrs 150°-160° & then pour into a solution of 10g BaH_2 8H₂O
in 50 cc H₂O heat 110°-120° (boiling off pyridine etc) & then add 3.11g H₂SO₄ etc & got
back finally by xx from alcohol 7.93g unchanged Metasaccharin and found
only 0.5g gum residue this $\therefore$ practically shifting of α OH group to give
Kilian's Pansaccharin = β Metasaccharin. (No 20)

(7) Lactulonic acid, its acetyl derivative acc. to Brist Ann 236 p.
To make sure no lactulonic acid is formed in expts with sugars & caustic alkalis
made some of the acetyl derivative acc. to Brist. got 4.33g b.p. 137° at
17 mm; it xx from 50 cc hot water fusing, on using 3.65g subst, 2.4g xx needle
m.p. 75-76° on cooling $\therefore$ body little soluble in cold water.

(8) Lactulonic acid, extr. by other means (see Fieser Ann)
H₂O was extr. 10x by ether & dry callz; got 4.3g acid b.p. 150°-160° at 20 mm; $\therefore$
incomplete extraction. (b) Bismuth salt of Lactulonic acid xx in needles and
is very soluble in alcohol & can be obtained in xx only when a very small amt of
absol. alcohol is used (c) phenylhydrazine [see Fieser Ann]
1g Lactulonic acid in 75 cc H₂O and add 1 cc phenylhydrazine in 1 cc 50% acetic
acid etc & got in 15 minutes standing S.T. 1.35g of the xx phenylhydrazine m.p.
105°-108° $\therefore$ but method of identifying Lactulonic acid in bases

(9) Galactose + $\frac{5}{4}$ BaH_2 (Oct 25 & Nov 07). 100g d. Galactose, 582g xx BaH_2 8H₂O
& 3 L H₂O leaves BMD at 12 hrs (in air) and 181g ap. H₂SO₄ die by H₂O etc through
ppt BMD will be decant. Total aq. dist = 5.2 L; on adding alkali to this noticed a
yellow color [due to oxy methyl furfural] and also noticed a peculiar odor on opening
my flask before adding H₂SO₄ as well as in aq. distillates. Titration with $\frac{n}{10}$ NaOH to
K Miso 4 shows that some other substances are present besides HCOOH [see Ann 335 p. 328]
Misen Linner and also unquestionably lactic + 13 dioxybutyric acid. Should also
prove with certainty whether CO_2 & H_2O or C_2H_5OH are formed [see Ann 335 p. 328]
200 cc of aq. dist took 28.24 $\frac{n}{10}$ NaOH (0.9889) for neutralization; from this can calculate
total amt. of HCOOH present as 3.34g. $\frac{1}{2}$ lost at 60°
[B]. 100 cc of aq. dist and Na₂CO₃ + 50 cc $\frac{n}{10}$ K Miso 4 (0.9962) and then add dil H₂SO₄
and 6.2 cc $\frac{n}{5}$ oxalic acid when titrate back with 1.71 cc $\frac{n}{10}$ K Miso 4
100 cc $\frac{n}{5}$ oxalic = 20.08 cc $\frac{n}{10}$ K Miso 4; total amt. of HCOOH
present is 4.68g (too high see No 11).

The University of Chicago

As there was still a fair test for Ba in non vol. gums left in d.f. residues there in
H₂O ppt Ba " so as only to leave slightest trace when distill off H₂O again in vacuo.
Total dist = 1465 cc; 150 cc of this took 17.45 cc $\frac{N}{10}$ KOH (0.999) $\therefore$ total H₂O = 0.78g.
wt of non vol. gums left in d.f. was now 76.5g; and in distilling then 1x R.F. with
12 acetic ether all except 3.26g tar dissolved on distilling off solvent finally
in vacuo got 75g gums. On extracting this 1x with 12 ether only 25g gum
remained insoluble (by diff). Other extract (acc dist) = 46.7g; made up to 500cc &
take 10cc ($\therefore$ 0.934g gum) and found 74.34 cc $\frac{N}{10}$ KOH in cold & finally 300cc at total
of 96 cc $\frac{N}{10}$ KOH necessary [theory for 0.934g & saccharins = 91.6cc]. Titration of the
25g gum made in ether (by diff) made up to 500cc & taking 10cc ($\therefore$ 0.5g) took in
cold 14 cc or in all 37.24 cc $\frac{N}{10}$ KOH [theory for 0.5g & saccharins = 30.88 cc].
Now distill off H₂O 100cc from each main eq. solution & got 43.8g & 23.7g gum resp.
Acetylation of the very mobile 43.8g gum gave 62.8g acetylated gums which shaking 10' with 300cc
cold H₂O left only 13.2g insoluble gum & this on hydrolysis gave 9.05g gum, which by drying
of portions soluble gave 32.7g very mobile gum; make up 32.7g to 500cc shake 5cc & found
34.5 cc $\frac{N}{10}$ KOH necessary [theory for 0.327g & saccharins = 32.06cc]. Now take rest of eq. solution
495cc divide into 2 lots 245 cc & 250cc respectively. (a) was treated with 67g Brucine
& gave 87g Brucine salts, (b) gum with Zues 3 etc. in all 13.65g Zues lactate 3 H₂O corresp
to 8.2g dl. lactic acid; the eq. solutions after ppt Zn etc gave 4.8g crop I, xx needed
20.24g Brucine de gum 24.8g Brucine salts & these xx from alcohol gave 4.8g crop I, xx needed
upt 205° a mixture of 2 CH₃ glycerinic and 1,3 Duxy lactic Brucine; 25g needed up 210-215°
(a) due to the 87g Brucine salts from 100cc alcohol gave 28g crop II, by emersion of a portion into
the filtrate gave 55.1g salts, which was also ppt 100° (see No 10); (b) by xx from Brucine salt
Zues lactate 55.1g salts, which was also ppt 100° (see No 10); (b) by xx from Brucine salt
the distilling off ale from filtrate from crop I got 55.1g Brucine salts which xx from 50cc alcohol
gave 6.55g crop II, up 110°-190° & on rexx this got 2.55g crude 2 CH₃ glycerinic acid above.
Now powder these very finely when brl at R.F. with 30cc of alcohol and found on filtering
her had left 2.5g insol xx, up 212°-218° = crude 2 CH₃ glycerinic Brucine; this gives out
very slowly & incompletely in dissolving in 3cc H₂O & 35cc absol. alcohol but gives xx needed as with
upt 210-230°. (It is all C₂ rather Brucine)
The ale. filtrate from crop II should contain about 47g Brucine salts; then gave with Brucine etc
back 9g gums, which with Zues 3 etc gave in all 6.0g Zues lactate 3 H₂O
& product was mainly d.l. lactic acid.
The 23.7g gum insol. in ether gum in acetylation 35.5g acetylated gums which,
il. shaking with 150cc cold H₂O, dissolved only 6.1g
oil, which was also on shaking with 18cc fresh H₂O of
gummy oil of H₂O & mainly insoluble; & brassy

C₆ gums nearly insoluble.

(10) Lactic acid phenylhydrazine, Took 1.1g CaK lactic acid 1.21 + 1cc phenylhydrazine + 1cc alcohol and mixture got solid with xx needles; add ether 1.3g xx needles up to 105°; on xx these from 5 pts lost acetic ether or a mixture of acetic ether slightly the salt comes out in silty needles or plates, up to 108-110° it is easily soluble in alcohol. 1g of anhydrous lactic acid from obs. from d. galactose [5 pts No 9] gave with ph. hydrazine etc in same way 1.25g lactic phenylhydrazine up to 105° although this salt comes out. This salt is not well adapted for identification & quant separation in presence of other acids.

(11) Dextrose + 5N BaCl₂. 100g Dextrose, 3L H₂O, + 582.0g BaCl₂ 8H₂O leave to Boil 12 hrs (no air) then add 181g H₂SO₄ etc. filter & decant. Ag. dest total = 5.2 L.

(a) 200 cc of this took 28.55 cc $\frac{N}{10}$ NaOH: total acid H₂SO₄ = 3.42g
(b) 100 cc of this with soda & 50 cc $\frac{N}{10}$ KOH: total acid H₂SO₄ + 14.11 cc $\frac{N}{10}$ oxal. c + 11.8 cc $\frac{N}{10}$ KOH: total acid H₂SO₄ = 4.33g (too high)
8.2 cc $\frac{N}{10}$ KOH: 10 cc $\frac{N}{10}$ oxal. c = 20.08 cc $\frac{N}{10}$ KOH: total acid H₂SO₄ = 0.49g

Now ppt. rest of Ba⁺⁺ except traces by 80% spirit 2nd ag. dest Vol = 1200 cc.
100 cc of this took 8.9 cc $\frac{N}{10}$ KOH or total acid H₂SO₄ + 1.8 cc H₂SO₄ + 1.8 cc H₂SO₄
Now neutralize all ag. dest together with NaOH group BaCl₂ + 1.8 cc H₂SO₄
15 cc H₂O + 5 cc conc H₂SO₄ + 5 cc ether, dry CaCl₂, distill slowly, got 1.8 cc H₂SO₄
ppt 90°-115° leaving fair amt of non-volatile matter + 13.5g butyric gum in def.
the d. galactose ag. dest elates (see 9°) give entirely analogous results: 1.8 cc H₂SO₄
a small amt, about 2g (404-7) with CaCl₂ acc. to Kiliari.

cf. expts No
Total non vol. gums left in d.f. = 75.7g which ext 1X R. 7. with 1L acetic ether
left only 4.3g insol tar + be salts. And gum left in solvent now acc. det as 74g
in extracting this R. 7. 1X with 1L ether had only 24.5g insol gum left
ether ext acc det = 47.6g very mobile oil; made up to 500 cc + took 5 cc spirit
a total of 49 cc $\frac{N}{10}$ KOH necessary [theory for 0.476g C₄saccharin = 46.7 cc].
Now took another 10 cc of ag. sol. (0.952g) notice of H₂O in conc. strong gum left in conc. with
1 cc acetic + 1 cc phenylhydrazine but got no ppt except small amt of tar separation: 1
no trace of barbituric acid can be present in gum (see No 8). Now distill off
H₂O from main ag. sol. of gum got back 44.3g mobile gum which by acylation gave 63.7g
gum & this by making with 300 cc cold H₂O left only 13.4g insol acylated gums.
Got from H₂O solution by 2X hydrolysis 32.95g mobile gum which with 300 cc H₂O gave a
total of 22.35g 3rd lactate 3 H₂O conc to 13.4g d. l. lactic acid: ag. filter ppted by
H₂O gave back 16.6g gum still containing much lactic acid; made it up to 200 cc & take
5 cc (0.415g) & found 38.1 cc $\frac{N}{10}$ KOH necessary (theory C₄saccharin = 40.7 cc)

Now had rest of ag. sol. with 57.8g BaCl₂ 8H₂O + 50.5g BaCl₂ 8H₂O which xx from alcohol
gum 16.6g crop I xx needles up to 205°; these dissolved R. 7. in 200 cc abs alcohol
in ending gum 11.55g xx needles up to 205-215°

The University of Chicago

got also crop II 10.75g with 190° xx needles which dissolved in 100 cc hot alcohol & on cooling gave 5.4g xx needles m.p. 205-8°. Now under these high melting salts 11.55g + 5.4g = 16.95g stirred with Bu₂H₂ etc got 12.73g dry Bu₂ + 3.7g gum; on shaking this gum [mainly 13 dixybutyric + 2 eth glycerinic acid] with 50 cc eth ether not much dissolved but on adding 100 cc more ether and on warming & shaking found 2.6g had dissolved + 0.9g remained insoluble; the latter was a very viscous oil got 0 on boiling stand 0.74 hrs with 100 cc eth ph. by dragin i. body was probably eth glycerinic. The other extract, 2.6g, was a very mobile pale yellow oil not easily soluble in ether. divide into 2 equal parts and work up (a) with Bu₂ + (b) with phenyl dragin. (a) zinc salt is giving very sol. in H₂O sis prop. by the same gets xx solid on stirring: got 1.52g which lost 0.19g H₂O on drying in vacuo (roughly done). Now weigh analytical balance and heat to 100° to constant weight then got a further loss of 0.1454g H₂O (∴ total H₂O = 0.3354g) 0.2881g anhyd salt gave on ignition 0.0978g Zn ∴ 21.69% Zn; theory for Zn (C₁₄H₂₀O₄)₂ = 21.55% ∴ O.K.

(b) 2nd lot with 135 cc ph. by dragin H₂O and alcohol gave 24 hrs in cold, ether etc 1.02g crude hydroxide which xx from 30 cc acetic ether gave 0.46g ap. 1,3 dixybutyric ph. by m.p. 130-20 in volume. xx needles m.p. 16.65g ∴ O.K. Bu₂ salts (see above) gave 49.1g salts from which by xx from alcohol got a quantity of all lactic Bu₂ salts (see above) gave 40.1g gummy Bu₂ salts which by ~~NaOH~~ etc gave back 9.85g very mobile gum (ak ext) this gum on ext 2x R.F. 300 cc ether took up 5.1g 1st time + then 0.3g 2nd time. & the insol portion on ext with 300 cc acetic ether took out 2g more from leaving 2.45g brown tar - ∴ probably had been a mixture of lactic & eth gums -

Orig gum, 24.5g, left insol in ether: was now made up to 500 cc & take 10 cc (∴ 0.49g) & from 34.3 cc to K₂ necessary (then C₆ succh = 30.24 cc). Now distill off H₂O from back 20.7g gum got by acylation 32.0g of which only a very small amt was soluble in shaking with 150 cc H₂O ∴ gums are [discarded].

(12) d.l. Lactic Bu₂: Took 1.65g C₁₇K's Lactic 1.21 stirred with H₂O + 6.3g Bu₂ very heavy cubes or prisms m.p. 212° = 5.15g (no loss in vacuo); these can be xx only from alcohol (5-7 hrs hot) & come out very slowly but almost completely on standing 2-3 days. If H₂O is present in alcohol nothing comes out ∴ can separate 1,3 dixybutyric Bu₂ + lactic Bu₂ by xx from alcohol as shown by later expts. O.K.

Mon 4-08, April 1-08

(13) Galactose + $\frac{5}{4}$ n Ba $\frac{1}{2}$ (of Nos 2 + 9), June 13 - Feb 3-08 2 lots of 100g Galactose
 npt. 150°-161° used taking 3 L. H₂O & 582g Ba $\frac{1}{2}$ H₂O for each lot 12hr 100°; add 186g
 conc H₂SO₄ each lot, leaving in a trace of Ba $\frac{1}{2}$ & then unite 2 lots & add 20cc 19.56% HCl & distill
 off H₂O. Take up gums, in 2 portions, 2x R.F. with 1 L ether and then 1x with 1 L acetic ether
 and let ether extracts combined stand in cold in about 2 L ether decant & let wt. of
 ether extract (free. 100° men) as 63.6g; rest of gums combined i.e. acetic ether extr
 + gum. amt. gum sep off ether combined from 61.5g gum (100° men). ~~Had also 34.5g~~
 gum + tar insol. in acetic ether, which was acetylated (120cc Essig anhyd) shes 80-90%
 got 42.1g acetylated material of which 23.7g dissolved in hot ether, leaving:
 19.5g insol tar [discard, still combined some gum]. ^{Hydrolysis of the 23.7g sol acetylated gum}
 gave 15.1g gum, ^{137.5g} total sacch gums obt from 20g galactose = 140.2g the 15.1g gum
 on extr 3x with 350cc acetic ether all dissolved except 2.7g tar (discarded)
 the 63.5g gum [acetic ether extr] was now extr 5x R.F. with 750cc ether which took out
 25.5g (by diff) & then gum left insol dissolved R.F. in 750cc acetic ether
 leaving only 0.5g insol tar (discard) & then solution standing gum was xx of Metasaccharin
 and of acetic ether extracts as 37.4g and 12.45g resp.
 Titration 37.4g lot made up to 500cc & take 5cc (i.e. 0.374g) and find 28.86cc $\frac{n}{10}$ KOH needed;
 therefor C₅saccharins = 28.33cc. Distill off H₂O & got back 37.1g gum which gum 55.1g
 acetylated gums of which 7g remained insol (= tar) on digestion with 15 L Ether. The
 ethereal solution containing 48.1g acetylated gums was now shaken with a sol of 30g anhyd
 Na₂CO₃ in 600cc H₂O: got 22.45g neutral acetylated gums unite (see below) of pure soda solutions
 after boiling 3hrs 100° & 107g 19.56% HCl etc gave 16.2g hydrolyzed gum which in extracting 4x
 with 0.5 L ether dissolved only 7.8g gum; rest of gum (except 0.7g tar) ^{partly dissolved in 500cc acetic ether}
 ether extract 83.9g gum was now acetylated & gave 117.7g gum which dissolved in 12 L cold ether
 & shook well with a cold solution of 40g anhyd Na₂CO₃ in 800cc H₂O; ag. sol. (see alk) was drawn
 off & shaken then BaO₃ when got acid: add 142.0g 19.56% HCl etc & got back 51.7g gum, which in
 1x hydrolysis gave 43.4g gum, easily soluble in 200cc cold ether.
 Got also 51.95g neutral acetylated gums and by 2x hydrolysis gave 38.4g gum dissolved in ether on
 after 7x extr 400cc ether all dissolved, 36g acc. det. (3 crops) leaving 2.4g insol tar (discard).
 On treatment of the 43.4g lot with 32g Zn + 3 H₂SO₄ etc got in all 35.66g Zn lactate 3 H₂O, corresp
 to 24.6g lactic acid: ag filter ppt Zn by H₂SO₄ etc got back 17.5g gum or after extr ether etc got 16.6g gum.
 The 36g lot on shaking in cold 1x with 1 L ether took up 26.5g gum & shaking 2x more with
 0.5 L. ether each time 4.5g + 1.0g resp. ^{neutral} acetylated gums got after 2x hydrolysis 18.7g gum
 4x Co gums, which were united with the 22.45g, ^{neutral} acetylated gums & got after 2x hydrolysis 18.7g gum
 which on digesting 1x with 350cc acetic ether all dissolved, except 2.2g tar (discard); in getting
 rid of some got back 16.5g gum which on extr 5x with 0.5 L ether dissolved 11.0g, leaving (i.e. by diff)
 5.5g insol gum which easily dissolved (except
 0.35g tar) in 200cc acetic ether.

Has selected gum 2.3g sp. salt up to 181° :: combine with 3.85g obt. from III and with 2.9g obt from II (see below). As alc. filtrate from crop I gum gummy Borneo salts only combine these with those obtained in III, :: in all about 125g Borneo salts said NaOH etc got back 24.5g gum (acetic ether extr) which was quite thick. This gum was shaken in cold degraded R.F. with 12 ether 2x; 1st time 8.5g dissolved + 2nd 5.0g (by diff) or by acc. dec. total ether extr = ~~13.9~~ 13.9; on shaking this in cold with 500 cc ether found 7.0g dissolved 1st time & with 2nd extraction in cold found 2.5g went in in cold and = 9.5g. Now add 7.2g gum, from V & VI, to insol portion, (also special gum from I & II & V. Borneo) dissolve further by digestion with hot ether subsequently with cold ether into 2 fractions. A, cold ether extract 16.2g rather thick gum - mainly C₄, C₆ gums (see p 15) B, acetic ether extract 14.55g r thick gum - " C₆ gums. " p 15

Titration of I = 8.9g make up to 250cc take 5cc (: 0.178g) found 17.4cc $\frac{N}{10}$ KOH used (they for C₄ sacch = 17.45cc) :: treat rest of ag. sol with 32.2g Borneo and got by xx 11.0g crop I gum which needles up to 196-212 $^{\circ}$ of 13 Duxy butyric + 2 C₆ glyceric Borneo; this crop requires 200 cc absol alcohol to dissolve by R.F. on cooling got 5.46g heavy fine needles up to 210-224 $^{\circ}$ on cone ag. filtrate for 2.4g xx needles depts 195-80. The alc. filtr from crop I gave 28g Borneo salts which by xx gum from 300cc alcohol gave 5.17g crop II with 76.8g gummy Borneo salts obt. from II (see below). Borneo salts only which was combined with 20.0g mobile gum (acetic ether extr); on shaking this 2x said NaOH etc got back 20.0g mobile gum dissolved + a 3rd digestion with 500cc hot ether in cold with 500cc ether found 12.5g hot dissolved + a 3rd digestion with 500cc hot ether dissolved 15g more leaving :: 6.0g insol gum combine with III & V (see above). The ether extract 14g by diff, now acc. dec as 13.25g ~~13.25g~~ mobile gum; on shaking it in cold with 70 cc ether all but 3g (discond) dissolved rest of sol. gum acc dec = 10.25g; this degraded B₂ with ZnCO₃ 8g etc gave only 1.8g gum lactate 3 H₂O :: hoty was mainly 13 Duxy butyric: now ppt Zn by H₂SO₄ etc got back (ether extr) 6.4g gum made up to 250cc take 10cc (: 0.256g) took 26.0cc $\frac{N}{10}$ KOH (they C₄ sacch = 25.1cc) :: treat rest of ag. sol. with 24.6g Borneo & got 3.14g salts which xx from 400cc alc gave 5.23g crop I xx needles up to 190-50 (= 13 Duxy butyric Borneo). alc. filtr gave 23.85g salts of lact. + Duxy butyric gum. Titration of II = 27.35g gum, made up to 500cc & take 5cc (: 0.2735g) found 25.27cc needed (they C₄ sacch = 26.8cc) :: treat rest of ag. sol. with 98.6g Borneo alc & got xx alc, crop I 26.57g derba Nadder up to 180-194 $^{\circ}$; this is a mixture for on digesting salt R.F. with 300cc of a 10% Isosaccharin solution (see p 13). The alc. filtr on cooling quickly gave 7.46g more insol and filtering hot got 2.9g insol. porous woody fibres up to 181 $^{\circ}$ of Isosaccharin unite with excess B₂ from III & IV (see p 13). The alc. filtr on cooling quickly gave 7.46g more porous XXX - a mixture of woody fibre salt & of 13 Duxy butyric & 2 C₆ glyceric Borneo. Mother liquors from crop I gave 16.32g crop II up to 170-185 $^{\circ}$ of same salts of finally got 76.8g gummy Borneo salts which were united with corresponding salts from I (see above) & worked up as stated there.

(see p. 14)

The University of Chicago

A, cold ether extract 16.2 g made up to 500 cc + take 10 cc ($\therefore 0.324g$) and find 28.4 cc

$\approx$ is not necessary [theory for C_5 sacch = 31.77 and for C_5 sacch = 24.55 cc] $\therefore$ that rest of ag. sol with 54.83 g Bueim got 72.0 g Bueim salt which $\times \times$ so as to give 21.25 g crop I, up to 140-180° obviously (1) 13 dry butyric Bueim (lost all filter by acc ident), and found to (x) contain much free Bueim and also woolly fibre salt; $\therefore$ that this crop I in H₂O solution, with ether 3 got 12 g Bueim + 4.85 g woolly fibre salt; $\therefore$ that this crop I, with 50 cc ether (alcohol) sol. dist off gum 9.2 g salt which digests with 50 cc ether (alcohol) dept. 180-181° ($\therefore$ combine with previous lots).

B, Acetic ether extr = 14.55 g was first heated R.F. with 500 cc acetic ether shaking well & then let cool, decant and found 7.65 g gum had dissolved (by diff) acc. det; insol gum acc. det = 7.0 g Titration of 7.0 g lot made up to 250 cc + taking 10 cc ($\therefore 0.28g$) needed 21.2 cc $\frac{N}{10}$ KOH (theory for C_5 sacch = 21.21 cc) $\therefore$ that rest of ag. sol. with 21.04 g Bueim got 27.0 g Br salt which $\times \times$ from 30 cc alcohol gave 0 definite but leave salt which are diff sol in alcohol $\times \times$ from rec H₂O & 30 cc alcohol gave 0 definite C_5 gums present.

alcohol some not as gums in evap. $\therefore$ obviously C_5 gums present. Titration of 7.65 g lot made up to 250 cc + take 10 cc ($\therefore 0.306g$) + find 26.4 cc $\frac{N}{10}$ KOH necessary (theory for C_5 saccharins = 23.18 cc) $\therefore$ that rest of ag. sol with 24 g Bueim got 1x ether 1x ether 9.3 g Bueim + 23 g salt which $\times \times$ from alcohol etc gave 3.17 g, crop I, Bueim salts up to 160° + then 0 more. Woolly fibre salt $\times \times$ from alcohol etc gave 3.17 g, crop I, Bueim salts up to 181° combined all salts obtained i.e. 4.85 g + 2.9 g + 3.85 g + 2.3 g = 13.9 g + that with H₂O & NaOH etc & got 9.35 g Bueim (insol) + 3.3 g gum extr 0.55 g insoluble; acetate ether extr = 2.8 g; only 0.5 g insoluble gum; that 2.8 g gum with 3 cc alcohol and 3 cc ph. hydrazine and let stand o.t. for 24 hrs got 0; used part heated in alcohol manner gum 0 $\therefore$ split off ph. hydrazine by 6.18 NaOH 98.3% 80 cc H₂O 9 hrs 100° etc got back 3.18 g gum or by extr 3x R.F. 300 cc ether for 2.55 g gum (discard wool portion); gum divided into 2 equal parts & heat (a) with ZnO 3 (b) with CaO 3 and found Zn salt gummy but pptd by finally at 90-110° (some decant) $\therefore$ all 0.2774 g H₂O (= 24.07%) faintly mobile gum; faintly mobile salt left = 0.8669 g

0.3581 g gum by gum (2x) $\therefore$ all 0.0914 g ZnO 14 g $\therefore$ Zn = 20.5% then for C_5 sacch salt = 18.0% Ca salt, same oppy by etc etc - found the salt can be $\times \times$ from small amt of H₂O when Ca salt not in very bulky needles which are too soluble in H₂O to handle easily $\therefore$ pptd with etc etc got 0.9374 g anhyd salt which lost 0.0488 g over H₂O in vacuo + undry 100° total loss H₂O = 0.0795 g $\therefore$ 0.8579 g anhydrous salt left. $\therefore$ Ca salt is $\pm$ (may contain traces of 13 dry butyric).

0.2774 g anhyd. salt gum theory for $(C_5H_9O_5)_2Ca = 11.83\%$ $\therefore$ Ca salt is $\pm$ (may contain traces of 13 dry butyric).

Now look rest of anhyd salt 0.4633 g + 17.6472 g H₂O $\therefore$ p = 11.83% $\therefore$ Ca salt is $\pm$ (may contain traces of 13 dry butyric).

Ca salt is $\pm$ (may contain traces of 13 dry butyric).

The high melting α CH₃ glycerinic Buerin: had 546g mpt ~~546~~ (see p 14); this with H₂O, NaOH etc gave 1.15g gum (acetic ether sol) and on treating it with 1.5cc alcohol + 1.4cc ph. hyd. reagent. got 2 after 24 hrs and then pptd a gum which don't get solid & much is easily soluble in acetic ether: now split off ph. hyd. with 7% NaOH etc. got back 1.1g gum. This with CaCl₂ etc gave a very sol Ca salt which is pptd by alc as gum but soon gets solid on scratching. got a heavy xx gran. powder. got 0.9388g salt an dry which lost drying 80-110° 0.0847g H₂O (9.02% H₂O).
 0.3325g anhydrous salt gave 2x ignition 0.0662g CaO: 14.7% and by H₂SO₄ etc
 for 0.1572g CaSO₄ Ca % = 13.92% then for Cs suach. Ca salt = 11.83% and for (C₄H₇O₄)₂Ca = 14.39% body is obviously α CH₃ glycerinic acid.
 Now took rest of anhydrous salt 0.5088g and 9.2091g H₂O & found [x] in 10 cubes = ±!

[14] Galactose and 35% Boric acid (or about 11/10) Took 10g galactose, mpt. 155-161° & 59g But 7/2 H₂O & 3.75g H₂O sheet But 12 hrs; no trace of sugar left as tested by Fehd sol. i.e. add 19g H₂O etc so leave twice Ba" other 2cc 19.56% 10cc sol. all off mpt. got 8-8.5g gum which ext 2x by 750cc acetic ether left 1.5g wool tar + br salts etc. Acetic ether extract = 6.8g very thick gum which extracted 4x by 500cc ether R.F. left 1.7g (by diff) wool gum. other extract acc. det = 54.5% is very thick gum: still ok. obviously mainly of saccharins [discont]. {can use 1/3 amt of Boric acid in one op. i.e. use 30gd. galactose in one op.}

[15] Lactic acid + acetic anhydride; Cs lactic acid had been found in neutral acylated gums since found in cold H₂O [see Vol 13] found why as follows: took Ca7K's acid heated 1/2 hr 90° at 25 mm + det wt of gum as 10.45g which gave with 33cc essy anhydrous 9 hrs 80-85° etc (no sweating or decamp) 14.7g acylated gums which dissolved easily in 200cc ether; on shaking this with 10g anhydrous NaClO₃ in 200cc H₂O got 1.7g neutral colorless mobile oil [1: acetyl deriv of lactic or of lactic anhydride].

[16] Galactose + CaH₂. Jan. 16-08, March 3-08. 100g galactose, 3L H₂O + 105g powder. CaO - that But 3, shaking often at first, for 12 hrs 1st day + for 8 hrs longer 2nd day + then add 200cc glacial acetic acid and then a sol of 220g acetic 2H₂O in H₂O sep. filter (66% Ca test) and then treated with 10cc 19.56% H₂O etc got 84.5g anhydrous gum which was ext 2x with 1.8L Acetic ether, and found in all only 27.4g gum had dissolved; the wool part 57.1g was acylated with 190cc Essy anhydrous got 87.5g acylated gums which ext 2x with 2d ether left 29.45g wool acylated tar (und in H₂O, diff sol in alcohol & ext. on and of ash only) This tar was now ext 1x more with 1L ether & then 2x hydrolyzed gave 33.9g gums, which got back 57.5g acylated gum from (ether) & there 2x hydrolyzed gave 31.5g gums ext 4x with 400cc Acetic ether left 5g wool tar (discont) & found 31.5g gums in Acetic ether (acc det).
 i.e. had 27.4g 1st Acetic ether ext
 " 31.9g 2nd " " "
 " " " " "
 i.e. total = 59.3g gums.

The University of Chicago

Use 27.4g lot was now ^{digested} shaken 7x R7 with 0.5L ether & then let 60cc ether ext stand in cold (Vol = 1.7 L) solvent & det not soluble gums as 14.6g acc. det.

Had left acc. det 11.8g insol. gums

Use 31.5g lot now ext 3x R7 with 1L ether and found 11.3g acc det head

dissolved making the form in combining with 14.6g 1st ether ext at 60cc of

25.9g ether extract. The portion used in ether was now combined with other

lot & acc. det go 28.85g this ext R7 with 50cc Acetic ether when all, but

2.5g brown ^{discuss} ~~had dissolved~~; the solutions gave no x x l of Metasaccharin as

standing over night with xx of it: distill off solvent so that only about 100 cc

of acetic ether remained. Let cool & decant from much insol gum. Found in

solvent 12.2g gum which on 7x digestion with 0.5L ether took up 9.7g gum (by diff)

& this was united with 25.9g ether extract above & total amt of ether ext

now found = 36.75g. The 2.5g of gum left in sol in the ether from 72.2g lot, sum

for xx of Metasaccharin in scratching suddenly sum. amt of alcohol W. 5.7g Temp 140-4;

Now found by combining all gums left not sol in ether head 13.05g (d).

The 36.75g gum was now dissolved by 4x digestion with 1L ether into 25.15g

soluble gum and into 11.6g insol gum; in shaking the 25.15g lot in cold with 1L

ether found 10.19g acc det = 16.9g dissolved; this shaken in cold with

50cc ether brushed with 50cc cold ether and found 10.19g dissolved (a); insol portion

is 6.8g now united with portion left from 25.15g lot and combined gums det acc as

14.5g (b). Gums now dissolved into 4 fractions (a) (b) (c) Hd 1.

(a) 10.19g most soluble gum, was made up to 250cc stock 5cc (0.202g) and found 21.0cc $\frac{2}{10}$ KOH

needed (they for C₄ sacch = 19.8cc) i.e. that amt of aq. sol with 40.5g Buem salt 52.1g Buem salts

which xx from 60cc alcohol gave 9.75g waxy fibre C₆ ~~Metasaccharin~~ ^{Metasaccharin} up to 170-50; ale. filter

gave 38.6g salts from which got indications of presence of dexty but, re as well as of

α CH₃ glycerine gum. after filling out these bodies as much as possible, found presence

of lactic in most soluble portions by setting free acid from (4.8g) shaking with Jones 3 etc

got 1.0g xx Zylactate 3 H₂O i.e. lactic is unquestionably present.

(b) 14.5g gum, sol in cold ether, made up to 250cc stock 5cc (0.29g) and found 24.5cc $\frac{2}{10}$ KOH needed.

[they for C₄ sacch = 28.4cc & for C₆ sacch = 21.9 (cc) i.e. that amt of aq. sol. with 47.3g Buem!

got 62.1g Buem salts which xx from 70cc alcohol sum got solid with xx of waxy fibre salt

crop 2 = 16.28g i.e. amt of salts were gummy.

(c) 11.6g gum, made up to 250cc stock 5cc (0.232g) and found 19.8cc $\frac{2}{10}$ KOH needed

(they for C₄ sacch = 17.6cc) i.e. that amt of aq. sol. with 38.2g

Buem salt 52.7g salts, which

on crystallization gave little definite but some very fine salt ^{was} ~~in~~ ⁱⁿ ~~the~~ ^{the} ~~most~~ ^{most} ~~present~~ ^{present}.
(d) 13.05 g lot, was dissolved in H₂O, filter got back 12.35 g gum which on digestion with
50 cc acetic ether R.F. & shaking well ~~dissolved only~~ ^{dissolved} ~~only~~ ^{only} ~~chyl~~ ^{chyl} ~~& decant~~ ^{& decant} and found 8.3 g gum
acc. det remained insoluble $\therefore$ 4.05 g dissolved (acc. det 1). Titration of 8.3 g lot made
up to 250 cc & take 10 cc ($\therefore$ 0.3325) and found 22.6 cc $\frac{n}{10}$ KOH needed (they for 8.3 g each
 $= 25.15$ cc for 8.3 g each = 20.5 cc). Conversion into Bueim salts are sure & definite.
The 4.05 g lot made up to 250 cc & took 10 cc ($\therefore$ 0.162 g) took 12.35 cc $\frac{n}{10}$ KOH (they for 8.3 g each =
12.27 cc). and conversion into Bueim salts are sure & definite.

(17) Galeatae + 33% NaOH, Jan 25 + Feb 19-08 March 19 08 [cf. March 8th]

1906 ~~glycerine~~ ^{glycerine} ~~paper~~ ^{paper} ~~expts~~ ^{expts} No 99. Added 100 g of oleic acid in 100 cc H₂O (hot sol) to a lot (60%)
Sol of 250 g 97.1% NaOH in 413 cc H₂O (time $\frac{1}{2}$ hr). Temp rose to 85 + then continue heat 73°
96° made in B.W.B. for 4 $\frac{1}{2}$ hrs longer. Add 1143 g 19.5% H₂O (they 1131 g) then add
off over (60%). Extract residue 2x with 2 L + 1.5 L ether resp and found 47.6 g acc det
dissolved. Now extract salt residue with 1 L alcohol got 42.2 g gum which exto 1x R.F.
with 1 L acetic ether dissolved 23.65 g gum (acc det); the insol gum 18.55 g gave by acylation
32.5 g gum which exto R.F. 1 L ether gave 20.9 g sol gum ~~10.0 g insol tar + NaCl~~ ^{10.0 g insol tar + NaCl} &
the former gave by 2x hydrol exto with acetic ether 10.4 g gum which gave no Metasaccharin

Now digested the 23.65 g lot 1x with 0.5 L ether & noticed much Metasaccharin xx in
xx: white with other acetic ether exto (see below).
used portion $\therefore$ decant and unite ether extract with 47.6 g lot & det gee as 54.25 g gum
which acylated gave 76.6 g gum & this worked in alcohol solution with a cold sol. of 40 g only
NaOH 3 in 800 cc H₂O gave 19 g neutral acylated gums or after 2x hydrol got 13.0 g gum (see middle p 19)
the soda solution leaked bks 100 cc gave after 1x hydrol. 37.6 g mobile gum which B.W.B. H₂O sol took
up 22.3 g Zucos & gave a total of 16.75 g Zuc lactate 3 H₂O (comp to 10.13 g dl lactic acid).

by H₂O the got back from gumming Zuc salts 24.8 g mobile oil which shook in cold with 100 cc ether
dissolved all except 4.55 g gum (ambr see below). The 20.25 g gum which dissolved in 100 cc
cold ether gave on 2nd treatment with Zucos 3 etc 8.55 g Zuc lactate 3 H₂O which shook in sep. funnel with
and on adding Zuc gum from filtrate got back 12.8 g gum which shook in sep. funnel with
70 cc cold ether gave 7.75 g sol. gum and insol portion united 2th other lot
gave together (acc. det) 9.1 g gum. The 7.75 g sol. gum made up to 200 cc & take 5 cc ($\therefore$ 0.19375 g)
and found 20.16 cc $\frac{n}{10}$ KOH needed $\therefore$ took rest of eq. sol. and treated with 31.13 g Bueim & got by xx
a total of 26.91 g 13 dioxyl butyric Bueim (see below) (the rest about 9.9 g salts ambr).

mainly of lactic Bueim (got 2.2 g ap. salt by xx).
The 9.1 g lot made up to 100 cc H₂O & took 5 cc ($\therefore$ 0.2275 g) & found 23.32 cc $\frac{n}{10}$ KOH needed $\therefore$
but rest of eq. sol. with 35.83 g Bueim etc got 31.88 g (remp) of prod ap. 13 dioxyl butyric
Bueim with 190-20. Rest of salts, about 12 g, mainly lactic & got 4.5 g ap. salt.
This proves much 13 dioxyl butyric gum when acylated goes into NaOH 3 free
version some lactic remains in neutral acylated gums, $\therefore$ n.g., also if use H₂O alone.
The 23.65 g gum above which had been exto 1x by 0.5 L ether now contained about 178 gum &
was ~~exto~~ ^{exto} dissolved R.F. in 50 cc acetic ether & this sol gave on standing
3.86 g Metasaccharin xx up to 140°-2°; now unite

acetic ether filtrate (cont about 10.4g gums) with about 10.4g least sol gums. solt
 wt acc as 22.8g this acetylated gum 35g gum & this dissolved in 100 cc cold ether
 trashed with cold sol. of 20g NaOH (dried 100°) in 400 cc H₂O got 11.4g neutral acetylated
 gums or by 2x by dr. gum 6.2g gums fairly soluble in 400 cc acetic ether stirring in
 standing 10.64g of Metasaccharin. The soda solution heated 5 hrs 100° & H₂O by distilled
 gum 13.55g gums, which extr 4x B.F. with 500 cc ether took out 4.5g gum. Now had insol
 part (full of xxlls) with small amt of steel ether and got 2.3g Metasaccharin (mpt 130-140°)
 now combine the filtrate with ether & filtrate from 6.2g gums got 11.9g gums which
 extr 4x with 0.5L ether took out 5.5g gum unite with 4.5g lot and sol acc. as
10.8g (is very mobile gum) ; the insoluble gums now weigh 5.3g & give in 300 cc acetic
 ether on long standing no more xxlls of Metasaccharin. but is mainly β Meta-
 saccharin (Kiliani's Perm Sol) ; made up to 250 cc & take 10 cc ($\therefore$ 0.212g) and found
15.51cc $\frac{2}{10}$ KOH needed (theory $\frac{1}{10}$ = 16.06) ; treated rest of aq. sol. with 14.67g
 Buene etc got by xx from H₂O sol 4.7g β Metasaccharin Buene in mpt 130-70° in
 dist. Naphtha ; rest of solids an mainly gum sol in all sol. $\therefore$ 6g gums.
 The 10.8g gum made up to 250 cc & take 5 cc ($\therefore$ 0.216g) and found 17.87cc $\frac{2}{10}$ KOH
 needed (theory for $\frac{1}{10}$ = 16.36cc for $\frac{1}{10}$ = 21.8cc) ; but rest of aq. sol. with 34.5g Buene
 etc got 45.2g Buene salts got a total of 30.34g and a 1,3 Dioxyl butyric Buene xxlls.
 The 13g gum from neutral acetylated gums (see middle p 18) made up to 250 cc & take 5 cc
 ($\therefore$ 0.26g) & found 25.77cc $\frac{2}{10}$ KOH needed ; but rest of aq. sol. with 49.75g Buene etc. got
 by xx a total of 28.66g + 5.95g = 34.61g and 1,3 Dioxyl butyric Buene ; the gum
 Buene salts left unite with those from 10.8g lots & got 44.2g salts which by NaOH etc
 got 8.85g fairly thick gum, which on shaking with 70 cc cold ether dissolved only
 about 2.0g ;
 Cude 1,3 Dioxyl butyric gum ; now took the various crops of crude Buene salts 20.1g + 20.8g +
3.55g + 4.35g + 5.95g + 1.92g + 14.43g + 17.82g + 17.45g + 9.09g - total = 115.4g & sol with NaOH etc got
16.3g and 1,3 Dioxyl butyric gum (1 L-ether extr) and found 6.8g gum (acetic ether extr)
 The 6.8g gum heated with 53g 98.3% NaOH and 600 cc H₂O in BFB 1 hr etc gum back (?)
 6.3g mobile gum of which on digesting 2x R.F. 200 cc ether found 5.2g dissolved $\therefore$ de H₂O.
 Took $\frac{1}{2}$ of 5.2g gum $\therefore$ 2.6g & heated it with 2.6 cc acetic ether & 2.6 cc ph. by digesting 36 hrs O.T. &
 smelt solid with xxlls - got solid cetyl hydrate. Now took 1.9g gum, from 16.3g lot, heated in similar way
 with 2.5 cc ph. hydrate & 2.8 cc acetic ether got 0.95g xx needles mpt 130-20. unite $\therefore$ 2.39g & rexx from
 100 cc acetic ether and got 1.66g c.p. buty mpt 130-10 in purity xx needles. 2x
 Now unite all of the crude oil left = 17.5g & heat R.F. with 250 cc
 ether and found 10.5g dissolved 1st time.
 3.25g u 2nd u 1

[20]

[20]

The gums were recombined again but not as 17.2 g; treat with 17 cc alc & 20 cc ph. hyd
 and (45 hrs. o.f.) got in all 7.95 g of 1,3 Diox-4 butyric ph. hyd resin (130°-2°). Hydrolyzed
 this by 6.1 g 98.3% NaOH & 80 cc H₂O (8 $\frac{1}{2}$ hrs 100°) got 4.4 g gum (acetic ether extr) which
 dissolved easily $\Rightarrow$ (3.9 g) in 100 cc cold ether + m. shaking with 100 cc more ether and
 only 0.3 g insol gum $\therefore$ 4.1 g gum; This now divided into $\frac{1}{8}$ & $\frac{7}{8}$ th lots.
 0.7 cc ph. hyd & 0.7 cc alcohol 20 hrs o.f. 0.68 g comb or

dissolved easily (0.1 g) only 0.3 g insol gum $\therefore$ 4.1 g gum; This now dissolved in 10 cc alcohol 20 hrs 0.7, 0.68 g each or $\frac{1}{8}$ of 4.1 g $\therefore$ 0.5 g gum gave with 0.7 g ph. lyd & 0.7 cc ph. lyd mixed m.p. $130^{\circ}-2^{\circ}$.
The other two bottles ph. lyd mixed m.p. 130° etc. of 1.2 g each (a, bc).
Of which dec.

$\frac{1}{8}$ of 4.1g $\therefore$ 0.5g gum juice with 0.7 g ph. ly^d
0.53g sp. 13 waxy bityie ph. ly^d mixed w/str 130-20.
 $\frac{7}{8}$ of 4.1g gum $\therefore$ 3.6g gum now divided into 3 eq. pts of 1.2g each (a, b, c).
(a) Bucium salt (used 4.25g Bucium & ext^d eq. solⁿ with CHCl₃) + got 4.7g salt - which dissolved
in 60cc abs alcohol + making got = large amt of transparent, very heavy cone grouped,
+ sides, prisms with pointed & square ends respectively, 3.52g with d_{cpt} 188°-190 or 185-90.
acc. as lost slowly or quickly). ~~These~~ Turk 2.47g of sp. Salt + vxx from 40cc abs alcoh
and got very slowly 2.0g of salt depts 186°. This gave [α]_D²⁰ as -27° 07' (no loss in volume during)
1.0004g undyd. salt + 24.002g H₂O $\therefore$ p = 4.0; d_x = 1.011; found d in d_c tubes = -2° 19'
Bz salts: 1.2g gum + excess Shychmin took up 4 hrs Bz B 2.75g alkaline
soln. and dissolve. Shych.) in 13 cc liq.

(acc. as last slow) 2.0g of salt up
 and got very slowly 1.0004g anhyd. salt + 24.002g H₂O ∴ p = 4.0; $\Delta T = 1.011$
 1.6) Strychnine, K, & Zn salts: 1.2g gum + excess Strychnine took up 4 hrs B₂B 2.75g alcohol
 + 4.05g gummy Strychn. salt easily sol (except sun. and disolv. Strych.) in 13 cc hot
 alcohol but $\frac{1}{2}$ xx not ∴ distill off alcohol, take up in H₂O and found 112.7 cc $\frac{N}{10}$ KOH
 necessary to get neutral: filter off Strychnine + distill off H₂O in vacuo for 2.0g K salt.
 which is gummy + slightly sol in hot about alcohol seems not as a gum ∴ distill
 off vacuo + add 3.0g 19.82% HCl etc got back 1.15g gum (acetic ether ex tr) which
 with Zn + 3 etc gave a gummy Zn salt very sol in H₂O but ppted + got 5xx with add.
 of acetic acid ∴ got 1.0451g (anhyd salt 18hrs) a very heavy powder; this lost on drying 14 hrs
 80-110° 0.1596g H₂O (∴ 15.3% of anhyd salt) + 0.0918g Zn (salt was probably slightly
 0.3312g anhyd salt gave 2x upon drying for Zn (ex H₂O 4) 2 22.26 (decompose)
 Zn = 21.55
 H₂O + 3.5g Chlorine then B₂B 2 hrs, cool 3x to 2x ether
 + alcohol + 2x

0.3312 g anhydrous
 21.55
 Zn =
 (c) Ca salt, Guinine salt; ^{high melting} ~~TOX~~ 1.29 g ^{Alum} H₂O, +3.5 g ^{Alum} in Heat Buns, cool 3x to 2x other
 4.5 g ^{Alum} salt which dissolved easily in 12 cc hot alcohol + 2 cc
 4.5 g ^{Alum} salt which dissolved easily in 12 cc hot alcohol + 2 cc
 out on standing; i. distill off alc add H₂O and 0.65 g powder ^{Alum} in Buns, cool 3x to 3x other,
 pass for this sol. etc. found Ca salt very sol gummy; i. filtered by alcohol tube got a heavy gum.
 powder: 1.6083 g (anhyd). This lost 0.1148 g H₂O when heated in vac & finally anhydrous
 90-110° had 1.3043 g anhyd. salt left (i. 18.9% H₂O loss).
 0.2726 g salt (heated 110°) pure 2x ignition 0.0604 g CaO (16%) or 0.1412 g CaSO₄ (2x) = 15.24% Ca
 as shown. ^{Alum} 1.0198 g + 9.914 g H₂O found & in 2 de tube is ±.

Now took the 6.75g salt powder finely & put R.F. $\frac{1}{2}$ L with 50 cc abs. alcohol & filter
off hot and got 4.23g insol xalle with respt 220-230°: fairly pure
~~5 dl. 97% pure~~ ~~Boric [keep]~~, with 1.37g corrip ~~by~~ ^{self} ~~from~~ ^{ext} No 9.

The separation of these 2 bodies can undoubtedly be best accomplished by
dissolving each in eq. and H₂O to 20x with ether or even gum in open dish etc.

[18] Dextrose + 33% NaOH; April 8 08 ^{May 24-08} ~~May 1-08~~ ^{March 9th 1906} [Oct 2 + 27]

250g Dextrose in 500cc hot H₂O added ^(20'') = lot sol. of 6.25g 97.1% NaOH in 1.03 L H₂O +
temp. rose to 82° then heat Bubb 4 $\frac{1}{4}$ hrs, and 2.82g 1982 or NaCl (about 20% excess)
distill off in vacuo - 2 lots of 250g Dextrose each. Extract 4x R.F.
with ether using 1.0 L ether in all and got a total of 217.3g ^{brown} gum. Now ext. salt
residue 1x with ~~1.0 L~~ ^{2.5g (see below)} ~~ether~~ ^{ether} 2.25g ^{brown gum}. Now ext. salt residue 1x with
2 L alcohol and got a residue ^{tar + NaCl} which in subseq. ext. with acetone ether took
out 2.5g leaving: only 7.0g ^{tar + NaCl} (discarded).

The ether extract ^{194.7g} gum digested 9 hrs Bubb with 150g pure ZnO₃ + H₂O 2 L etc
gave 11.9g insol ZnO₃ and 144.0g ^{xx} Zn lactate 3 H₂O corrip to 87.12g d l lactic
acid; the 194.7g gum digested 8x in cold with 0.5 L ether a very large amt. about
87.8g ^{much in slaking} ^{8x in cold with 0.5 L ether} ^{50g soluble in cold ether} ZnO₃ etc but gave 0xx of Zn lactate 3 H₂O + got back 174g gum; this
was treated with 30g ZnO₃ etc but gave 0xx of Zn lactate 3 H₂O + got back 174g gum; this
was dissolved in 182 cc H₂O extd 9x with ether; ag. sol. distilled off gave 100g gum +
ether sol. solution gave 69.2g mobile gums which better with 48g ZnO₃ etc gave 68.2g
Zn lactate 3 H₂O, in 3 crops, corrip to 41.25g d l lactic acid. The 100g lot ^{ether} ⁱⁿ
100 cc H₂O again 13x with other gave 37.55g in ether and 60.25g left in H₂O solution
+ the 37.55g gum in ether gave with ZnO₃ etc a total of 17.36g Zn lactate 3 H₂O (in crops)
corrip to 10.5g d l lactic acid. Combine gummy Zn salts ^{after by H₂O} ^{got back 50.0g}
gum which dissolved easily except 4.5g in 900 cc ether and the 4.5g when digested with
100 cc acetic ether left 3g hard tar + Zn salts (discarded) ^{back on} ^{got working up} ^{50g gums}
a total of 47.6g ^{very mobile} gum (very mobile). Therefore dissolved the 217.3g + 50g gums
sol in ether as follows: (1) d l lactic 87.12 + 41.25 + 10.5g = 138.87g ^{1/2 1/3}
(2) 47.6g lactic + 13g ^{very} butyric gum ^{valley} (3) 60.25g ⁴ gums [total = 246.72g].

Now took ~~the 144.0g gum used in cold ether (see above)~~ ^{the 144.0g gum used in cold ether (see above)} + the 106.9g gum left insoluble,
after ext. 87.8g gum by ether from orig. 194.7g gum, + ext. digest R.F. 1x with 500
cc lactic ether, cool, decant & got 44.2g insol gum and 51.5g soluble gum ^{some} ⁱⁿ ^{each}
acetic ether must have been sticking to my 194.7g gum. The 51.5g sol. gum was ext.
R.F. 8x with 1 L ether and found 34.5g had dissolved leaving 17g insol gum which
digested with 85 cc acetic ether cool & hot & then gave

The University of Chicago

8.5 g sol. and 8.5 g insol gums. Unite the 8.5 g sol. gums with the 34.5 g
 other extract and the 47.6 g + the 137.8 g other other extract and get
 81.5 g gums; this was now shook 7x in cold with 1 L ether and foll. ant. succ.
 dissolved 13.0 g; 12.5 g; 9.0; 6.0; 5.0; 4.0; 3.0 g - or acc. det total ant. = 54.35 g (40)
 the portion insoluble acc. det = 24.25 g (5).
 C gums; the 44.2 g gums used in remnants of acetic ether and 8.5 g gum 2.00. now
 combined, dissolved in H₂O, filtr from tar & det wt acc. as 51 g; now acetyl late got 85.6 g which
 digested 2x with 0.75 L ether left 13.0 g insol tar (discarded) and in by dry is further sol.
 gums got back 42.65 g C gums (6); total of (4%) (5%) of 6% = 121.25 g which with totals
 of (1%) (2%) + (3%) = 246.72 g makes 367.97 g of saccharin gums from 500 g d. glucose
 or average of 73.6 g gum for every 100 g d. glucose. N.B. much less
 tar found than in case of d. galactose because less dissol. into
 C₂H₄O₂. C gums (6) were digested R.7. with 400 cc acetic ether, cool, decant and had left (acc det)
 42.65 g of C gums (6) diff sol in acetic ether; titration made up to 250 cc stake 5 cc (i.e. 0.291 g) and
 14.55 g insol gums (6) diff sol in acetic ether; titration made up to 250 cc stake 5 cc (i.e. 0.291 g) and
 found total of 19.3 cc $\frac{2}{10}$ K₂H₄ (thm C₂sacch = 17.97 cc) i.e. that rest of ag. sol. with 37.3 g Buein &
 got 58.4 g Buein salts which dissolved in 60 cc hot alc & let stand for a large amount of ~~precipitate~~
 Buein, 200 g, up 130-145; the alc. filtr crop I gum 30.4 g salt which xx 40 cc alc slowly
 in dishes 12.15 g up 150-160. The alc. filtr crop I gum 30.4 g salt which xx 40 cc alc slowly
 gum cakes and needles and cubes (latter 6.6 g & 6.6 g) the gums soluble in 400 cc acetic ether (labeled) to be
 C₂ gums easily soluble in cold acetic ether: the gums soluble in 400 cc acetic ether (labeled) to be
 29.0 g and on ext these 5x with 1 L ether R.7 found foll. ant. succ. dissolved 3.5 g; 4.5 g; 3.5 g; 3.0 g;
 and 0.5 g or total ant. acc. det = 16.1 g; made up to 250 cc stake 5 cc (i.e. 0.322 g) and found
 27.35 cc $\frac{2}{10}$ K₂H₄ needed (thm for C₂sacch = 24.29 cc) i.e. that rest of ag. sol. with 52.8 g Buein
 got 70.2 g salt which xx from 70 cc alcohol slowly gum 22.8 g crop I Buein salts up 140-5
 of d. saccharinate of Buein - as these xx from 10 cc H₂O + 100 cc alc in wooly needles as well as
 in transp long plates. Ale. filtr. gum 44.6 g salt which xx from 50 cc alcohol (with see)
 the gums used in ether (5x R.7) now det acc as 12.4 g (very thick). make up to 200 cc stake 5 cc i.
 0.31 g and found 22.3 cc $\frac{2}{10}$ K₂H₄ needed (thm for C₂sacch = 19.14 cc) C₂sacch = 23.49 cc
 i.e. that rest of ag. sol. with 34.27 g Buein and got 52.7 g salt which xx 60 cc alc gum
 16.9 g ep. d. saccharinate of Buein in transp long plates depts. 140-150; these xx
 from 100 cc H₂O + 100 cc alcohol in volum. wooly needles looking exactly like saccharinate of Buein.
 The alc. filtr, crop I, gum 30.5 g salt which xx from 40 cc gum 18.5 g, crop II, up 140-155.
 the alc. filtr, crop I, gum 18.1 g salt which was xx from 20 cc alcohol

[24]

This by Hrode found to contain 4.0g wood Brucine
lyt. rat. sat. f. l. gave 29.5g xx Brucine salt
20.03cc 10% H₂O

24.25g of gums 151, made up to 500 cc stake 5cc (i.e. 0.2425g) and found 20.03cc 10% H₂O
needed (theory for 5 saccharin = 18.37cc) i.e. but not of q. l. with 78.12g Brucine &
for 115.5g xx Brucine salt - which xx from 130 cc alcohol gun slowly 15.95g, crop I,
of c.p. ~~metasaccharin~~ of Brucine in transp Slabs kept 140 - 150° which xx from Hrode
alcohol (quickly) in volum. opaque, ~~metasaccharin~~ al. filt. list of q. l. gave 87.5g Brucine salt which xx
from 90 cc alcohol gave 33.17g crop II kept 140 - 150° late filt. crop II gave 53.5g Brucine
salt which xx 60 cc alcohol gave 33.17g crop III kept 140 - 150° late filt. crop III gave 44.7g salt
(6a) ~~metasaccharin~~ etc. (obt. about 33.65g) took crop I 20.0g lot from 160 cc p. 23. 4g with Neohale
5.55g crude thick gum; this with 6 cc alc + 5.5 cc ph. hydrogen gave 28 hrs d.t. 7.73g cum. o. ph. lyd
upt 110° - 120° which xx from 15 cc alcohol gun opaque heavy nodules 4.26g up to 122 - 60°
or xx these from 30 cc alc (too much) got 2.4g c.p. br. in dark blattings. Krustan - identical
with ~~metasaccharin~~ from dry those + 7.5% NaOH (see Na. 5) as proved by getting
[2] w as - 30.7 since 10.967g + 25.9077g Hro i.p. = 4.06; d = 1.011 (pt) & found

α in 1 de tube = -1.26.
(6) took crop I from (5) = 15.95g and crop I from (6) = 16.9g ∴ 32.85g that with NaOH alc &
9.1g gum (alc extr) containing no trace of Saccharin as shown by diss in su. and aceton etc.;
take gum and divide it into 2 parts of 5, 11 and 15. (1) take 5 ∴ 1.8g & got with ph. lyd
etc 2.22g crude hydr or after xx from 5 cc alcohol got 1.28g xx nodules up to 120 - 60° which
again gave 1.06g ap. ph. lydr up to 124 - 60° with [2] w = -30.66; 0.9994g salt & 23.6439g Hro ∴
p = 4.057; d = 1.011 (pt) and found [2] in 2 de tube = -2.51.

(2) 1/5 of 9.1g from ∴ 7.2g approx gum with CaCO₃ etc a total of 6.63g only d xx Ca salt,
in 2 crops (in xx needles) diff sol in cold Hro; the xx salt contains su. and adhering Hro ∴
lost 0 on Hro in vials but lost
0.0262 Hro at 100 - 110°. crop I 3.84g lost 0 on Hro in vials = 2.79g (Hro in vials) = 2.79g.
crop II (K₂CO₃) = 1.4115g & dried 100 - 110° + loss Hro = 0.0254g.
crop II still contains 1.8% Hro; p. 23. took 1.4115g & dried 100 - 110° + loss Hro = 0.0254g.
0.3399g only dr. salt II gave 2x quantity 0.0457g CaO = 7.64% theory for
Ca(C₆H₁₁O₆)₂ = 10.05%.

[2] w = -23.25; 1.02325g only dr. salt and 25.24605g Hro ∴ p = 3.894 d = 1.011 (pt)
found α in 2 de tube = -1.83.
N.B. the total amount of ~~metasaccharin~~ from (5) & (6) was 20.0 + 22.8g + 16.9g + 10.5g + 15.95g
+ 33.17g = 109.32g crop to 33.65g ~~metasaccharin~~ in above expts used only 20.0g &
15.95g and 16.9g lots (= 52.85g). The other salts were united with other (gummy) of the salts
(see below) & treated with CaCO₃ etc (see p. 26).
54.35g lot (40), was now extr 6x R 7 with 400 cc ether + found 7.19g; 7.78g; 6.75g; 4.75g; 3.8g; 3.2g
since dissolved by diff - or acc. del total = 35.2g leaving by acc. del 18.8g wood gum;
60.25g lot (13) was extr 5x with 400 cc ether (found 11.6g; 9.95g; 6.55g; 4.75g; 2.9g dissolved by diff
a total and acc. del = 38.8g, leaving acc. del 18.8g lot ∴ = 49.7g gum.

The University of Chicago

Treated the other sol. portions: 35.2g with 40cc alc + 40cc phlyd r got 0.2 day, O.T.; + the 38.8g lot with 45cc phlyd + 45cc alc O.T. 40 hrs got 15.7g xx 13 dioxy butyne (plumby) resin up to 120°-135°. Combined the gummy hydrog was from both lots 83.64g + 76.8g + 112g H₂O + 1600 cc H₂O etc got back 64.0g gum, leaving acc det 17.0g insol gum. took out acc det 42.25g very mobile gum, leaving 9.39g = 24.100° with 32g NaOH + 150 cc H₂O got back the 40.7g gum (see bottom p. 24) was back 100° with 32g NaOH + 150 cc H₂O got back 39.5g gum + then ext 2x R.F. with 1 L ether was resolved acc det into 15.05g soluble gum and into 23.0g insol gum. Now unite the other sol. gums 42.25g + 15.05g = 57.3g + the insol gums 17.0g + 23.0g = 40.0g gum (6) (a) The 57.3g gums made up to 1 L H₂O 5cc (0.2865g) found 27.32cc $\frac{2}{10}$ K₂H₂O (they for C₄ sacch = 28.1cc). ∴ mol ag. sol. with 214.2g Buein and got 291g salts which xx 200cc alc gave 80.88g, crop I, made 13 dioxy butyne Buein, depth 190°-200°; alc. filter gave 193.5g salts which from 200cc alc gave 53.5g more 13 dioxy butyne Buein crop II, up to 188° then got 126g salts which gave 30.35g crop III up to 140°-176° which contains much free Buein; dissociated ∴ add 30.35g for again bale filter and treat with NaOH etc and got back 26.1g gum (accalc other ext) which was very mobile then dissolved it in 26g H₂O and ext 20x by ether and got other ext 9.65g very mobile and from solution 11.7g gum. titrations 9.65g lot made up to 200cc took 5cc (0.24125g) and found 23.54cc $\frac{2}{10}$ K₂H₂O needed (theory for C₄ sacch = 23.65g); titration like a lactone ∴ treat with 36.17g Buein etc got 48.0g salts which xx from 50cc alc got 13.1g salts (the like lactone) 13 dioxy butyne Buein (in hand). Sept 26-08. Filtered off 13.1g salts which xx from 32cc alc got 6.58g salts (Buein salt det 180-190°). alc. filter det off 24.3g salts which xx from 25cc alc got 0 (disorder). ∴ the 11.7g gum made up to 250cc took 5cc (0.234g) took 19.2cc $\frac{2}{10}$ K₂H₂O (the like lactone) (they for C₄ sacch = 17.73cc) ∴ treat rest of ag. sol. with on B.W.B. to know if lactone + 13 dioxy butyne Buein but noticed prod. no loss got back 11.1g gum; to depolymerize the 11.1g gum on 200cc alc and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and found 18.17cc $\frac{2}{10}$ K₂H₂O needed (they for C₄ sacch = 17.6cc ∴ O.K.) ∴ treat rest of ag. sol. in present in gum and treat it B.W.B. 9.3g gum; this made up to 200cc took 5cc (0.2325g) and extract in open dish on

back from aq. solution 58.5g ~~Brucin~~ salts of ~~X~~ gums probably.
The ~~q.~~ salt from crop I gave 100g ~~gummy~~ Brucin salts which gave xx more de
20.18g ~~crop II~~ ~~not~~ 144-180° ~~leaving~~ :: 79.82g ~~gummy~~ salts.

Now combined gummy ~~xxx~~ 4 ~~Brucin~~ gums as follows; 20.18g + 79.82g and
44.6g [from ~~tot~~, see p. 23] and 44.72g [from (5) see p. 24] :: total amt = 189.32g ~~thru~~
with NaOH etc got (see other extn) back 44.45g gum; to get rid of lactic & oxybuty,
lacton take this gum ~~trump~~ in open dish in Buns. adding H₂O from time to time
until exact wt of ~~Brucin~~ gums left as 38.5g (on hand in de) Sept 26 08
added to this the 9.5g gum ~~in~~ bottom p 25 :: 48g (14.4%)

G gums now combine xx gummy Brucin salts from (5) and (6) as follows
crop I
xx 58.5g ~~salts~~ from 40.0g lot, see top p. 26 from (3) & (4) originally
xx 29.5g ~~"~~ = crop II (5) ~~from~~ Brucin - (p. 24) (orig 33.17g)
xx 30.4g ~~"~~ from most diff sol. G gums (14.55g), crop I bring out.
xx 18.1g ~~"~~ " easily sol G gums 12.4g (6) p 23 bottom
xx 10.5g ~~"~~ " " " 12.4g (= crop II)
xx 22.8g ~~"~~ = crop I from other extn from (6) (see p 23).
xx 8.72g ~~"~~ = crop II (5) (p. 24 top).

for 47.6g sol (2)
see No. 23

178.52g :: = total amt ~~which~~ with NaOH etc gave 41.95g gums (see other extn)
This treated in H₂O sol ~~with~~ 17.0g CaO₃ took up 12.4g (chem for G seeds = 13.18g)
from conc aq. solution soon slowly gave xx etc of ~~Ca~~ ~~phosphate~~ of Brucin
on hand after letting stand all summer; got 10.1g Ca salts crop I; ~~got back~~ ~~from~~

(19) Galactose + 33% NaOH, April 9 + 24 + May 22 - 08. 300g d. galactose in
600 cc hot H₂O (+inse c. 1/2 cc conc H₂O) added to 750g 97% NaOH in 123 g cc H₂O (t 65°)
lune 1/2 hr & temper. rose to 85°; less Buns 4 1/2 hrs - add 338.4g 19.82% NaOH (excess about 25g)
& distill off excess. Ext. 3X with 2L aetic ether got 265.0g ~~gum~~ ~~then~~ 1X with 1L
alcohol got only 5.5g gum + NaOH (discarded). The 265g gum (?) Had present acetocellulose
now ext 2X with 3L ether and found 133g ~~gum~~ (by diff) & this gum treated
with ZnO₃ etc gave ~~only~~ no Zn lactate 3 H₂O XX :: precip by H₂SO₄ and ~~water~~
gum with ether ext (see below). The insol gum 265g - 133g :: 132.0g was now ext
5X more with 1L ether and found a total of 57g (78 last time) went in (by diff) leaving
81.5g insol still mobile gum :: extract this R.F. with 500cc aetic ether,
cool & decant from 41.0g insol tarry gum. The soluble ~~gums~~ gave 30.0g gum
which ext 6X with 2L ether took out 20g whereas the most part 108g
was full of XX etc of Metasaccharine ~~etc.~~ On bleating this ~~very thoroughly~~
with 250cc cold ether & decanting found about 1/2 dissolved but this treated with
ZnO₃ etc also gave no XX of lactate of Zn :: ppt by H₂O combine with
got back a total of 115g acid gums (2)

[27]

The University of Chicago

The other ether extracts not used for July 03 material were now winter found =
55.5g & this on extr 5x with 1L ether took up acc. det 34.15g (b); insol portion was
 now (acc. det) = 20.0g & gum by acylation 33g gum of which 3.5g were used in lot other
 (i. discant) & extr by dry dist of sol. gums 17.95g gums. (c) Acylation of the 41g +
 10g lots (see p 26) gave 76g gum which extr. 2x R.F. 1L ether left 20.0g insol
 and tar (discant). The sol portion by 2x hydrolysis gave 33.3g gum (d).
 (a) + (b) + (c) + (d) make a total of 200.4g Saccharins from 300g d. galactose i. much
 more tar formed than in case of Dextrose because less disac. into lactic or etc; also
 much lactic & 1,3 Dioxibutyric acid gums carried off by operations used.
C₃ & gums (a) = 115g; disac. in 115cc H₂O extr 18x by ether and found both lactic & 1,3-
 -butyrolacton went in; got 56.95g in ether and 53.7g in H₂O solution. The 56.95g
 gum again dissolved in H₂O extr 20x by ether gave 46.7g ether sol. gums and 8.8g insol
 gums left in H₂O unite with 53.7g lot i. 62.5g gums II. The 46.7g lot I (gum with
 Zuber 3 etc only 22.6g in lactate 3H₂O (correct to 13.67g lactic acid); not on hand. (see det)
 The 62.5g gum (II) on extracting 4x R.F. with 400cc ether took out 38.5g gum (see det)
 much with 43cc ph. hyd & 43cc alc gave a total of 29.58g 1,3 Dioxibutyric gum
 hydrolyzed up 125-130° unite with 15.7g lot from Dextrose [No 18] & work up for c.p.
 13 Dioxibutyric acid gum [see No 20] which extr 1x with 1L ether dissolved 24.5g
 back 27.0g gum (acetic ether extr) which extr 1x with 1L ether, taken out by ether,
21.35g (acc. det) mobile gum & left 20.0g insol gum.
 The insol portion of the 62.5g gum, which 38.5g had been taken out by ether,
 was acc. det as 24.2g; this gum needed with 24cc 15.82% HCl (100° 3 hrs) the gum back
 23.6g of same thick gum i. no depolymerization of 1,3 Dioxibutyric gum. On treating
 the 23.6g gum with 20g NaOH & 60cc H₂O 4 hrs 100° the gum back 24.3g fairly mobile gum
 which on extracting 3x with 300cc ether R.F. dissolved 8.55g; 3.9g & 2.3g prop-
 or by acc. det at 100° of 15.6g very mobile gum; this with ph. hyd 18cc gave 18cc sol
 stand of 24 hrs gave 100% the hydrolyzed gum back 14.0g gum; the insol. portion
 acc. det = 8.25g and on extracting the 14.0g gum with 300cc ether found 7.2g (acc. det)
 was in leaving 6.8g insol gum which was united with the 8.25g lot and acc. det
 as 16.25g; the portion easily soluble in ether 7.2g was now united with the 21.35g
 mobile gum above & combined lots det acc. as 28.55g; this made up to 500cc &
 take 5cc (i. 0.2855g) and found 27.1g gum & 13 Dioxibutyric gum up 178-188°
 Bismarck got 139.5g Bismarck salts which 2x 140cc alc gave 40.75g
32.0g & 32.6g of same body up 178-188°
 Male. filter now gum

52.0 g gummy salts - obviously mainly lactic + 13 Dexty butyric Buicin.
 16.25 g lot, made up to 500 cc + take 10 cc ($\therefore$ 0.325 g) from 27.14 cc $\frac{n}{10}$ KOH
 needed; theny C₆ saccharin = 24.72 C₄ sacch = 31.86 cc)
 $\therefore$ that rest with 52.4 g Buicin & 4 g 68.5 g Buicin salts which by xx gave only
 32 cc $\frac{n}{10}$ KOH needed

needed; (theny is sheeham
 ∴ that rest with 52.4g Buicin etc. for 68.5g Buicin salts
 summing salts or dissociated Buicin ∴ nearly 6g more.
 34.15g gum slb/ made up to 500cc & take 5cc ∴ 0.3415g found 32 ccⁿ K₂H needed
 (theny 4 sach = 33.48cc) ∴ that with 124.9g Buicin etc. for 169.6g salts which ^{20cc etc} XX
 gum rapidly ~~62.37g~~ ^{62.37g} crop I ^{and} 13 dry & boty ne Buicin rest 180-1850 (cont some
 woody fibre salt & 220° melting salt); also fills for 100g salts & from this for xx salts
 containing much Buicin (crops II & III).
 0.359g and found 29.2 ccⁿ K₂H needed.
 56.4g Buicin etc. for 81.7g salts which
 34.52g, which known also

(Theory for 4 sachets -
 gave roughly 62.37g crops + 220 melting salts); also fills gave 100g salts
 woody fibre salt + 220 melting salts (crops II & III).
 containing much Bismuth (crops II & III).
17.95g gum (C) made up to 500 cc & take 10 cc $\therefore$ 0.359g and found 29.2 cc $\frac{n}{10}$ lit needed.
 (Theory for 5 sachets = 27.2 cc) $\therefore$ treat with 56.4g Bismuth etc for 81.7g salts which
 XX wcc alcohol gave a large amt of woody fibre salt, 34.52g, which known also
 of Bismuth $\therefore$ much dissociation.
 33cc alcohol and let stand when some got solid with
 "total = 13.67g + 134 + 587g which
 1 new Metas contains same amount
 1 + 00 04 +

17.95g gum(c) made up to
Theory for C₆Succ = 27.2 cc) ∴ mat with ~~the~~ fiber salt,
xx wcc alcohol gave a large amt of woody fiber salt,
contains much Buicin ∴ much dissociation.
C₆Gums 33.3g(d). disorder in 33cc abs'd alcohol and let stand when soon got solid with
Metasaccharin xx etc [got 8.9g] + 1.65g subsequent dist off gave 24.3g film which
was degraded R.F. with 1 L acetic ether solution gave 0 in CR. for a week ∴ distill off &
the acetic ether solutions gave 0 dissolved ace del a lot of 24.6 cc N KOT
fresh ext 4x with 1 L ether dissolved ace del a lot of 24.6 cc N KOT
flake see (∴ 0.30625g) and found 37.8g Buicin got 53.85g xx salts

C₆ gums 33.3 g (d). Dissolve + 1.65 g in 140-4 ml of water. For a week...
 Metasaccharin x x etc. [got 8.9 g in 140-4 ml of water] in C.R. for a week...
 was degraded R.F. with 1 L of acetic ether when only 2.7 g tar...
 insoluble (i.e. discard); the acetic ether solution was dissolved in a total of 24.6 cc of 10% KOH
 got back 23.0 g gum which ext 4x with 1 L ether dissolved and found 24.6 cc of 10% KOH
 (i.e. 23.2 cc) i.e. trial with 37.8 g Buicin got 53.85 g x x salts
 this made up to 200 cc of acetic ether 1.14 g

insoluble (i. discant) 23.0 g gum which expt
got back 12.25 g gum; this made up to 200 cc state
necessary (then for C₅sacch = 23.2 cc) i. trial with 3% of
which xx 60 cc alcohol gum wavy fibrous salt containing much Bism.
The portion insoluble in ether, 10.75 g by diff, gum with alc & diethyl ether 11.45
more Metasaccharin xx the mpt 140-4; now got distilling off solvent 9.0 g emite
C₅ gum which made to 250 cc state 20 cc (i. 0.36 g) and found 24.12 cc for K₂H needed
Bismach = 22.23 cc. Namron treat gum left with 22.81 g Bismach & got 32.55
Bismach = 7.5 g gum (washed with 5% H₂O) (x) what is it in act. dose look
xx from 5 cc H₂O & 75 cc alc & got 10.17 g Bismach 13.5 cc up
be got xx by rubbing or standing in cone
eans of separating it

No 19

Nov 11 + 25

(29)

Nov 12 76

Dec 7

Dec 9

May 22

The University of Chicago

✓ Galactose 33%, K₂H continued from p 28/11. The combined 62g C gums (see bottom p 28) dissolved in 70cc alcohol and 150 cc ether was now worked up
(C) 24g pples gum and also 38g soluble gum. latter was then further worked by
degummed with 90 cc acetic ether R.T. & white into 24.9g soluble gum A and
into 12.55g insol. gum B. Gum A was freed from C₃ by gums by dissolving in 90 cc
H₂O & to 20x with other which looked 9.6g C₃ by gums (discarded) leaving 13.9g
gum A in which by dissolving in 4cc alc ate no α Metasaccharin could be found.
Titration of gum A taking 5cc after making up to 200 cc (∴ 0.3475g) took 27.02 cc $\frac{2}{10}$ K₂H
contraining part of sol. with 41.52 g Buein got 58.3 salts much xx from 60cc alc gum
17.5g crop I xx up to 130°-170° (water soluble). 10.68g I am gum B (see below) xx
130°-140° wavy film. 40cc alc gum B. 3.27g Metasaccharin Buein up to 130°-140°
150° alc. 1.59g wavy film salt up to 130°-140°
180°-200° of xx needles of 13.05g Buein salts left which since in rexx there
after it was given a 2nd crop, 5.68g, up to 180°-200° of xx needles of 13.05g Buein salts left which since in rexx there
Now: had (mol 159g wavy film salt) about 51.05g Buein salts left which since in rexx there
gum disor. Buein was heated with H₂O for 2 hrs on rexx these from 50 cc 97% alcohol got a
rapid separation of heavy opaque xx needles 14.05g, up to 130°-150° consisting mainly of
of β Metasacch. Buein as is shown by following: on rexx these from 100 cc 97% alcohol got
7.7g of c.p. body up to 135°-137° with [α]_D²⁰ = -24.087 (p = 3.997): the alc filtrate from crop I
contained 3.25g salts which were broken by nigger. The alc. filtrate from the 7.7g c.p. salt &
mainly of α + β galactos metasacch. Buein. 14.07g
cont: 6.35g salts was now united with a corresp. alc. filtrate from Fr. B (see below) which
contained 7.26g salts where gum 13.5g salts up to 100° (cont: α body); a corresp. fraction from
crude β galactos metasacch Buein up to 100° (cont: α body); a corresp. fraction from
(see below) 6.82g was now united with those from rexx from 48 cc 97% alc got 10.47g
xx nodules with soft 110° and [α]_D²⁰ = -17.18 i.e. 0.8091g salt + 19.4034g H₂O p = 4003
and found α in 2nd tube = -1.39 (d = 1.011 at 20°): a mixture of about 60% α and 40% β galactos
metasaccharin (part, see later).
Fr C, 24g; got next from 250cc flask 5cc (∴ 0.46g) took 30.29 cc $\frac{2}{10}$ K₂H (then C lost 28.4 cc)
which made up to 250cc flask 5cc (∴ 0.46g) took 30.29 cc $\frac{2}{10}$ K₂H (then C lost 28.4 cc)
∴ that not with 58.48g Buein got 87.4g salts where xx from 90 cc alcohol gum 25.7g
crude β galactos metasacch Buein up to 130°-145° + 3.09g more crop II same salt.
The alcoholic filtrate ∴ about 50g "broke by nigger": hot and probably mainly α + β galactos metasaccharins.
Fr B 12.55g, gum 0.87g galactos metasaccharin xx up to 138°-142° when got back 23g gum
11.7g gum which made up to 200cc flask 5cc (∴ 0.2925g) took
21.46 cc $\frac{2}{10}$ K₂H ∴ that not with 33.0g Buein got
47.9g xx salts which

xx from 50 coaledred got entirely solid with 10.68 g crop I β galactometasacch. Burein
mpt. Δ pt. $130^{\circ}-150^{\circ}$ (note see 28'). The alcoholic filtr from crop I cont. $2.2.3$ g salt - was
note with 12 g Burein salts obtained from xx of 21.95 gms and a woody film salt (see above) and also with 14.6 g
Burein salts obtained from rexx of crude $\alpha + \beta$ galactometasaccharine Burein (see above) and heat
in all: a total of 67.8 g salts (after hydrolysis with H₂O etc) which xx from 97% alcohol gave 18.6 g
crude β galactometasacch. Burein mpt. Δ pt. $130^{\circ}-150^{\circ}$ - x on rexx then from 150 cc about 97% alc
got 11.34 g op. β body down $135^{\circ}-7^{\circ}$ with $[\alpha]_D^{25} = -24.82$; alc fltr cont 7.26 g salt - note (see above).
The alc. fltr from the 18.6 g crop I gave back 47 g salts which xx from 50 cc 97% alcohol gave
13.95 g crop II mpt $100^{\circ}-110^{\circ}$ obviously a mixture of $\alpha + \beta$ galactometasacch. Burein. The alc. fltr
13.95 g crop II mpt $100^{\circ}-110^{\circ}$ obviously a mixture of $\alpha + \beta$ galactometasacch. Burein. The alc. fltr
13.95 g crop II mpt $100^{\circ}-110^{\circ}$ obviously a mixture of $\alpha + \beta$ galactometasacch. Burein. The alc. fltr

[illegible][illegible]

another expt. after 5 hrs run leaving a loss of 1.2 g. gum was still very mobile. $\therefore$ both of these Shylinn parasaccharin λ total of 57.83g; xx in
 Proprs of c.p. β Galactosaccharin = Kilian's parasaccharin λ total of 57.83g; xx in
 salt from 19 June 9 (24 hrs) as follows 3.17g + 14.4g + 4.7g + 10.17 + 25.7g. Total of 57.83g; xx in
 + 20 cc alcohol and got (in 24 hrs) 30.67g hard inc. opaque nodules depth 130-137 mm rex x there from
 15 cc H₂O + 15 cc alcohol got 24.13g ap. β Burein Salt in heavy opaque nodules depth 130-135 mm [d] λ =
 - 25° 22 is. took 0.6453g subrt and 15.4903g H₂O (p = 4.00 and found λ in 1 dc tube = -1° 02; d = 1.011 art.
 On working up orig. another liquor as well as there obt from crys of 30.67g lot got 3.48g more of ap. β salt only.
 these 27.61g small grains with Ba salt & H₂O (55) a total of 17.6g Burein (anhydrous) + no distilling of H₂O in vacuo
 extra salt residue with alcohol (total inc of traces of Burein), pass CO_2 through aq. sol. effective then 80% etc
 $\therefore$ p = 3.96 and found λ in 1 dc tube = 0.07; [d] λ = only -1° 49. Now ppt Ba by Soxhlet
 gum left 3x with alcohol (1x 400cc 2x 200cc) (200 cc del 1x sweep over H₂O) total 5.95g and
 β Galactosaccharin gum made this up to 148.6g with H₂O filling etc. $\therefore$ p = 4.0 and found
 λ in 2 dc tube = -4° 24 [d] λ = -49° 95; d = 1.011 art.

α Galacto metasaccharin (ph. leydigii) mpt 130-115 of gum 94.06 has $[\alpha]_D^{20} = +34.38$ i.e.
work 0.4485g mpt + 10.7676g H₂O; $\rho = 4.0$ d: 1.0113 2) found d in 2 de tube = +1.59.
in a mpt tube dissociates with softening between 190-210 giving very quietly sly chlm
+ then melt 250-260 (The powd. salt melted almost completely between 180-195).
Found $[\alpha]_D^{20} = -8.41$; i.e. work 1.3843g mpt + 33.2317g H₂O; $\rho = 3.999$ d = 1.0113
and found α in 2 de tube = 0.68. This salt is practically insoluble in absd alc
whereas the corresp β galacto metasacch Bumein is easily sol in hot absd alc w/o heating
the separation of the 2 isomers can not be accomplished by means of sly chlm & 2g
(See later). Chinin salt of α body (cf. Kiliani) made from 15 pure lectn & 2g
(absd alc).
Chinin (3 hrs in Bums) + found salt is very sol. in H₂O; xx from 6 cc 96.5% absd alc & gum.
very slowly opaque warts made up of xx needles; 20g, mpt 144 with $[\alpha]_D^{20} = -90.46$, i.e.
took 1.1865g salt + 28.4631g H₂O; $\rho = 4.002$ d = 1.011 det 20° and found α in 2 de tube = -7.32
Ba salt of α galactosaccharin has $[\alpha]_D^{20} = +27.4$; i.e. took 0.9936g mpt 144° sheen Bums for
5 1/2 hrs with excess BaCl₂ filter t det wt of ag. filter as 43.9306g w/ only 2 Ba salt calc from
lectn taken must be 1.519g; $\rho = 3.458$ d = 1.011 (est) and found α in 2 de tube = +1.91.
As the nature of the impurity present in the 57.83g crude β galacto metasacch. Bumein
mentioned in p 30; got out in all 27.61g of β Bumein salt. The alc. filtrates contain
still about 30g Bumein salts which ~~was~~ were examined as follows. On xx then for absd alc
for a large amt of mpt, once grouped crystals made up of them plates mpt 110° which
come out very slowly ~~from H₂O~~ from H₂O absd alc but quickly from absd alc and on
pure. free from particles of β galacto metasacch. Bumein - got 17.25g in all which on rexx
behaves exactly like a honey. body can not be moved. This is a mixture of α + β
galacto metasacch. Bumein salts (in ratio of about 3:2). Thus the 17.25g xx with dept 110° give
on rexx from 3 cc H₂O + 70 cc absd alc got 9.3g crystal nodules with dept 110°. This gum $[\alpha]_D^{20} = -18.05$
on xx from 70 cc mpt absd alc gum 4.00 found α in 2 de tube = -1.46.
i.e. took 1.0547g salt + 25.3221g H₂O; $\rho = 4.00$ found α in 2 de tube = 5.0g Bumein, ex by 3 x 0.10g
Now take 8.32g of this salt and treat with 3.0g BaCl₂ H₂O (in H₂O) got 5.0g Bumein, ex by 3 x 0.10g
then add H₂SO₄ etc 6 gpts Ba and got 1.0g gum (acetic ether ex tr) which at once gave
xx on deat of colour; got 0.41g α metasacch. taken mpt 140-1° Q.E.D; the alc. filter
cut about 1.0 to 1.5g gum of β galacto. metasacch. taken (dis carded).
The remainder of the Bumein salts (in the absd alc filtrates), about 13g, was united with
corresp. salts mentioned in p. 30.
The smaller woody fibre salt Bumein mpt 180° is a mixture of α Isosaccharin &
 β Isosaccharin. Took foll. lots of woody fibre salt: from galactose slant (11 lbs. 3)
4.44g + 2.57g + 1.45g + 3.18g + 3.12g + 5.2g = total 19.96g and 6.2g from 2. galact + 5.2g Bums (7.63.08)
i.e. total = 26.16g; digest there R.F. 2 3/4 hrs with 150 cc absd alc, end of filter shag (diff. diff. 2g)
mpt 180-195. On rexx this exp from 6.5 cc H₂O + 700 cc absd alc got 7.56g heavy opaque nodules made up of
rexx with mpt dect 185°. This gum $[\alpha]_D^{20} = -24.2$ i.e. 0.9972g + 23.8873g H₂O; $\rho = 4.006$; d = 1.011 est.
The salt gum with 10g H₂O & 5.16g rad. dy Bumein so. of 19.82g H₂O etc.
and found α in 1 de tube = -0.98. The salt gum with H₂O to 42.69g; $\rho = 3.982$ gum in 1 de tube + 1.31; $[\alpha]_D^{20} = +32.55$.
gum was now treated in H₂O sol. with 10g pure CaCl₂ for 6 hrs Bums and in conc ag. filtrate got in 2 cups 0.68g
Ca & isosaccharin etc xx. This with 0.225g mpt 2 H₂O etc gum 0.32g xx α isosaccharin mpt 93-94 (keep).
The ag. solution of gummy Ca salt was paper by 0.44g value 2 H₂O etc that broke 0.75g mobile gum which made
up with 17.96g H₂O to $\rho = 4.009$ and found $[\alpha]_D^{20} = +18.5$ i.e. found α in 2 de tube = +15. Gum still contains
some isosaccharin and 2.85g xx Bumein salts which xx from 2 cc H₂O + 70 cc alc

The University of Chicago

gave bulky cone grouped vert. needles, 1.0g, mpt 188°-190° with $[\alpha]_D^{20} = -24.07$ i.e. 0.7581g
 subst + 18.1642g H₂O i.p. = 4.007 found α in 2dc tube = -1.075. Now took all of salt subst - 4.36g
 the (melted) type for back 0.65g gum which with 1.2g Chlorin then Burns etc gum 2.0g salt which xx
 from 4cc H₂O and revealed gum 0.15g Chlorin & saccharinate mpt 202°-4° D.E.D. ale. filtr see below
 The orig. ale. filtrate from the 1377g used Brucin salts (p. 32) gave on dist off ale in mews 12.2g salts
 which xx from 30 cc abs alcohol 2.0g of 13.8gxy buty re Brucin xx the (discolored filtrate).
 2nd lot wooly fibr salt from d. galactose + 33% NaH as follows (May 21 1908) 8.52g + 3.5g + 3.88g
 + 5.87g i. total = 21.75g; xx this from 10 cc H₂O + 60 cc abs alcohol and got 6.86g mwt
 needles mpt 160°-175°; this exp was now boiled out 1 1/2 hrs R.T. with 150 cc abs. alcohol and got 6.86g mwt
 wooly fibr salt dept 181° & this rexx from 3.5g cc H₂O + 60 cc abs. ale gum very slowly opaque disks (heavy)
 mews up of needles with dept 182°: 4.98g with $[\alpha]_D^{20} = -22.72$ i.e. 1.0946g subst + 26.241g H₂O i.p. = 4.006
 and found α in 2dc tube = -0.92. Now took all of salt subst with 0.7g NaH got 3.32g mwt, dry Brucin and
 add 3.5g 1981% H₂O re got 1.13g gum (acetic ether extr) with $[\alpha]_D^{20} = +21.87$ i.e. mews up with H₂O filter to 28.23g
 found α in 2dc tube = +1.77 (p = 4.003). The ag. sol. of gum was filtered 6 hrs with 0.6g pure CaCO₃ and mews
 ag. filtr got 0.33g xx of Ca & saccharinate. The rest mews up of a very sol. Ca salt which got solid on
 scratching after mews of H₂O to get 1.0g which dissolved easily except traces in cold H₂O + mews up it up to
 23.34g i. p = 4.285 found α in 1dc tube = +0.44: $[\alpha]_D^{20} = +10.16$. The Ca salt was now heated with 0.3g
 x H₂O (pure CaCO₃) got back 0.65g gum (acetic ether extr) which mews up to 14.77g c. H₂O gum + 0.29
 in a 1dc tube: $[\alpha]_D^{20} = +7.06$ (p = 4.056). The gum was now boiled in H₂O sol with found gly chlorin stick
 up 1.32g of pure 1.8g gummy Gly chlorin salt which diss easily in 5cc abs. alcohol of gum xx in scratching
 standing up & got back gum 1.3g & sacchar Chlorin mpt 202-4° D.E.D. The rest of the
 H₂O & 20 cc abs alcohol gum 0.29 g. & sacchar Chlorin mpt 202-4° D.E.D. The rest of the
 Chlorin salt was xx, 1.4g by dissolved easily in 5cc 96.5% alcohol so came out in standing (p. 13.8gxy buty re)
 Chlorin). (p. 13.8gxy buty re) and a comp. ale. filtrate from xx the 13.77g xx from H₂O gum 2.58g
 The ale. filtr from the 4.98g salt (i. 1.88g) was mews in all about 8g salt, & heated with BaH₂ & H₂O got
 xx the with $[\alpha]_D^{20} = -24.07$ etc (see p. 32 bottom) with $[\alpha]_D^{20} = +12.68$ i.e. mews up c. H₂O to 24.01g found α in 2dc tube =
 back 1.36g gum (acetic ether extr) with $[\alpha]_D^{20} = +12.68$ i.e. mews up c. H₂O to 24.01g found α in 2dc tube =
 +1.03. This gum now heated with 2.6g Chlorin etc gum 0.5g of ap. & sacchar Chlorin
 mpt 202-204° (mews in alcohol). Rest of salt about 3.4g mews with abur salts; total amt = about
 7.7g; these gum with BaH₂ etc 1.7g gum (acetic ether extr) with 2.4g xx needles mpt 190°-210°; the ale
 which dissolved in 50 cc abs. ale (R.T. 6 hrs) on standing got 2.4g xx needles mpt 190°-210°; the ale
 filtrant 3.4g gummy salts easily sol. in 10 cc abs. alcohol
 The rest of the alcoholic filtrates i.e. ale. filtr got by heating the 14.77g from H₂O salt led
 (see above) + semi about 7.1g Brucin salts; as well as the ale. filtr got from xx the 14.77g from H₂O salt led
 tent: i. about 7.6g salt - was mews: i. 14.7g and worked up as given on p. 30 (top). Look up notes

20 Conversion of α Galacto metasaccharin into β at 200° (see 908). 1st expt of Oct 20 07 No 6
 5g α Metasacch ep. 25cc H₂O, 5cc pyridine in m. S.T. 3 1/2 hrs 180°-190° slowly gum burning but no shifting
 to β body as got Brucin 3.2g pure & dry. 2nd expt 2g heated in a tt. with inside temp. ranging from 195°-215°
 for 9 hrs - Now soluble gum which on 4x hydrol. gum back 1.35g Saccharins & this diss in 15cc ale gum
 75cc ether 2.3g soluble gum which on 4x hydrol. gum back 1.35g Saccharins & this diss in 15cc ale gum
 0.23g xx & body mpt 135°-140°; ale filtr dist off and again let stand in ale gum 0.15g mews xx & body mpt 130°-140°
 2.0g for back 0.95g gum which with 2.2g Brucin gum 3.3g salts + these xx from 10 cc 97% alcohol gum
 2.0g from 97% ale pt 1.3g of c.p. β salt in heavy opaque nodules mpt 132° with $[\alpha]_D^{20} = -24.24$
 i.e. 0.9605g subst + 21.6104g H₂O i.p. = 4.0 and found α in 2dc tube is -1.096.
 The ale. filtr from 2.0g exp I contained 1.25g Brucin salts which diss in
 3.5cc 97% ale gum xx of β salt even on long

(34)
standing i. mixture of $\alpha + \beta$ salts. L.E.D.; a sharper separation of $\alpha + \beta$ bodies is by means of
Casalto (styphnine is N.9.).
Nov 19 08. Jan 5 09.
At 160°: α has $[\alpha]_D^{20} = +93.28$.

Casalta (Schuchman is N. 9. 1).
 21° Constants of α dextro saccharin mpt 160°; $[\alpha]_D^{20} = +93.28$.
 Brucine salt, made from 3.0 g ep. Saccharin mpt 160° and 7.3 g Brucine mpt 102.5° salt which
 dissolved in heating in 10 cc n/10 alcohol but on standing ~~over~~ ^{at room temp} gave no x in c.r. On standing off
 in a vial with salt 10 g in 5 cc H₂O + 50 cc alcohol got on standing over night a large amount of
 bulky ~~crystals~~ ^{crystals} made up of fine needles 5.85 g (no x in H₂O) which were mpt 152° and
 gave $[\alpha]_D^{20} = -26.07$; i.e. 1.0668 g subst + 2.5586 g H₂O: $\rho = 4.003$ $d = 1.0112$ and found α in
 2 de tube = -2.11.
 Pleurhydry Drugul (made Dec 4 '05) with mpt 167-9° gave $[\alpha]_D^{20} = +50.3$, i.e. 0.6325 g subst +
 158°
 1 de tube = +2.03.
 Chinin salt, took 15 g of ~~the~~ ^{the} lactic mpt 160°
 all from much alcohol and is

15.25149 H₂O
 22° Another 5.7g of *Dioscorea chinensis* ref 96
 and 2g chinin on Burro 12 hrs for 3.3g xx salt which xx very white
 is but characterized salt of this body. It can be dissolved in 10 cc hot alcohol but undissolved
 xx from 40 cc alcohol xx on standing 11.6g of crystal needles up to 152° (on hand)
Strychnin salt, made from 1g lactin & 1.57g dross. Strychnin salt gave 2.7g xx salt which dissolved
 easily in 5 cc alcohol & on standing 2 days gave no xx salt ∴ too soluble + n.g.
 the [2]nd of the Ca & Na salt in lactin is given as - 6° (? if correct).
 + 5.7g of *Dioscorea chinensis* ref 96; that of lactin is given in lit as +63°
 for 11.85 g xx salt which dissolved in 25 cc hot water ∴ very soluble using small

(22) Crystallization
 Nov 19.08 of Oct 23.06
Brucein salt took 3g lactin and 7.3g transparin in 3 days got 2.5g transparin
 alcohol and in standing got entirely solid with silky needlesumped in 30 cc rectified alcohol; new comes out very slowly first finally in 3 days got 2.5g transparin
 flat needles, which on drying H₂O in mass lost 0.33g H₂O ∴ had left 2.17g
 anhydrous salt with reft. depts. 164°. The anhyd. salt gave $[\alpha]_D^{20} = -26.01$ i.e. 0.9296g subst +
 22.312g H₂O ∴ $\rho = 4.001$ found & in 2dc tube = -2.011 . The anhydrous salt is far more diff.
 soluble in absol alcohol than salt with xx H₂O and separates out in conc. gumped opaque needles, looking
 like undy fibr salt up to 181° in very large amounts.
Stychnin salt, 1g Isosach lacton took up 1.64g Stychnin (8 H₂O, H₂O) gave 2.8g gummy salt which is
 very soluble in H₂O & H₂O xx from 1 cc H₂O + 10 cc alcohol ∴ n.9.
Chinin salt see Kiliani Ber; is very characteristic; took 1g lactin & 2g chinin (100° 3 H₂O H₂O)
 reft clear solution (vol 150 cc) + in standing got a separation of 1.72g fine volum. needles
 reft 202-4°; in conc. al. filtr got 0.57g more of salt. The salt is pract. insoluble in
 hot absol alcohol ∴ very useful for detecting traces (see Nov 19 + 18 + 23). Dic 30-08
(23) Dextrose + 33% NaOH continued from No 18 (Oct 2 + 27-08 Dic 14 30)
 (Jan 8 + 12 May 25-09)
 June 1-09 - Nov 19.08
 Had on hand (a) 31.7g & gum forced from & dextro
 is exht. on Oct 1 1908; consisting of α and β dextro metasach.
 p. anns.

Summary of No 18
metasaccharin 9.25 g by means of Ca salt for analysis of the saccharin
(b) 48.0 g of gum i.e. 38.5 g + 9.5 g dry from gummy Basin salts saccharin mainly 6%
(c) 47.6 g lot (2) mainly lactic + 10% buty in lactic gum; (d) Had also a total of 162.66 g ^{basin} and 13 dry buty
titration of gum C (2) 47.6 g made up to 1 L H₂O sec (∴ 0.238 g) took 24.45 cc $\frac{2}{10}$ KOH
(unchanged). To prove this is a mixture of lactic & 13 dry buty in

The University of Chicago

took 25 cc of aq. sol. of H₂O in which 1.15 gm which mixed with 1.5 cc ph. by 1.5 cc aq. gave sum and of x₂le only two more making much acetic ether in sol.
Now took 100 cc of aq. solution: 4.75 g gum, treated with 19.3 g Brucine etc got 23.9 g xx salts
which xx from 100 cc about aq. gave 9.0 g xx up to 180-190° mainly 13 Dioxo butyric Brucine;
alc. filter gave 4.55 g crop II of a similar salt and then finally got 4.0 g of crop III of cubes
of dl leucine Brucine up to 190-205°. More careful work later showed that lactic acid
was always present with the 13 Dioxo butyric Brucine ^{supra versa} must separate these two
as shown later [see Nos 34+35]. Took 49.1 g xx 13 Dioxo butyric ph. by hydrogen
c.p. 10xy butyrolactone (from Nos 18+19). Took 39 g from previous expt and by hydrolysis 9 hrs/100°
up to 125-131° [15.7 g from Dioxo butyric expt and 29.58 g from galactose expt] which dissolved
with 490 cc H₂O + 35 g 97% NaOH etc. got 26.25 g gum [acetic ether expt] which dissolved
except 1 g brown tar (discard) in 800 cc Ether on gentle warming. Now distill off ether
+ det. wt. gave as 24.25 g: a very mobile oil; on hand in alcohol saved by me and
by Vellula for various expts e.g. oil to dl. malic by HNO₃ + resolution in d & l. forms.
Got 108: took the oil & dissolved it in 20 cc H₂O heated 8 1/2 hrs in B₂ B₃; cool and ex to 20 x
by ether and found 12.4 g 10xy butyrolactone had ~~decomposed~~ gone into ether, -
Gum A 31.7 g was now hydrolyzed 9 hrs 110-120° with 130 cc H₂O got back 29.45 g gum (a).
Gum B 45.0 g 8 1/2 hrs 110-120° with 180 cc H₂O got " 45.0 g gum (b).
B gum was now dissolved in 50 cc alcohol cool and add 100 cc ether shaking and got a separation
into 20.25 g mobile + soluble gum and into 25.0 g pptd gum. The A gum similarly,
treated with 30 cc alcohol then with 60 cc ether gave 12.1 g soluble gum and 17.6 g pptd gum.
The 20.25 g soluble gum was degraded with 100 cc acetic ether + let cool } portions and insol portions
The 12.1 g " " " 60 cc " " + let cool } respectively
and det. wt. of these gave Sol gums A = 14.65 g; insoluble gums B = 17.45 g.
The ends 13 Dioxo butyric Brucine total 162.66 g as follows 16.1 g + 5.77 g + 12.2 g + 80.88 g + 53.5 g + 13.1 g
+ 6.58 g. These with 19.75 g NaOH 97% gave 125.15 g Brucine (anhyd) only 100 cc H₂O and heat 5 hrs 100°, cool & ex to
and got 34.25 g mobile gum (acetic ether expt). On dissolving this in 20 cc H₂O and heat 5 hrs 100°, cool & ex to
and more with 10 cc H₂O then ex to 20 x with ether found (see det 100° 25 min) 19.25 g gum had gone
into the ether most of this see No 19 p 30. The aq. sol. gave back 10.4 g gum after evap in open
dish for 8 hrs soon further heating & evap with H₂O for 6 days of 9 hrs each this lot. went down to
6.45 g. since C₆ gums present (cf. 2 Dioxoaccharin). Treat rest of aq. sol with 20.35 g Brucine and got 26 g xx
salts which xx from 5 cc H₂O + 100 cc alcohol gave slowly 5.25 g xx needles (4 sided with square opacities) &
with up to 210-225° obviously 2 Dioxo butyric Brucine in part. The alc. filter gave 20.2 g
salts which xx from 30 cc alcohol. The gums B, C & C' all white for C₆ lacton practically and
gum was more x₂le from alcohol.
Gums A, B, C' and C (see above). The gums B, C & C' all white for C₆ lacton practically and
gum on treatment with 47.4 g, 64.0 g and 43.2 g Brucine resp. xx of α or of a mixture of α and β Dextromela
sacch. Brucine salts as follows: B 14.93 g crop I. α; crop II 7.95 g but shape xx; crop III 4.9 g dolo as II. α. 5.25 g
C gave 15.7 g salts crop I; 16.56 g crop II but; crop III 15.8 g but; Fraction C' gave
crop I 29.13 g salts; crop II 14.28 g salts; crop III 3.25 g but.

Gum A 14.65g was first dissolved in 12 cc H₂O & x 20x with ether and found in ether
 6.65g C₃ C₄ gums (see p. 39). The H₂O soluble gum back 7.8g gum which
 with 5 cc acetic & x x of Saccharin put in showed no sign of presence of this body; get rid of acetic
 & make gum up to 20 cc & take 10 cc (i.e. 0.39g) & found 28.3 cc 2% K₂H₂SO₄ (then Co test = 28.6 cc)
 is a mixture of C₄ & C₆ gums. Now treat rest of 20 cc sol. with 21.2g Bismut 28.2g x x salts which
 dissolved in 30 cc alc gum slowly 10.35g, crop I, boils up 140°-150° other 4.55g, crop II, same salt;
 crop III 2.55g same salt. p. 39
 The alc filtrate from A B C & C' after giving no more x x le ~~was~~ then united (see below).
 The mixture of α & β Dextro metasacch. Bismut salts above was first supposed to be pure α body i.e.
 took 94.62g as follows 29.13 (C'); 14.93g + 7.95g (B); 10.35g (A) and 15.7g + 16.56g (C) (marked in red ink)
 & met this with an excess of NaOH & got 70.4g Bismut extra & alc got 28.0g gum (acetic ether ext.)
 which boiled with pure CaCO₃ took up 8.25g tag. filter on fine filtering in x x gum no x x le of Ca salt of
 α dextro metasacch. Ca is obviously much β body also present. 2.50g of this gum was now treated
 & got back 27.0g gum (alc ext.) 26.15g gum (acetic ether ext.). 2.50g of this gum was now treated
 with 65.93g Bismut 102.2g salts which x x from 100 cc absol. alc got 46.53g gum & salts crop I
 with 110°-120° with [α]_D²⁰ = -27.3 (i.e. a mixture of 40% α and 60% β). On rexx 45g & crop I from 20 cc
 96.5% alcohol got 15.28g crop a, up 150°-2°, which rexx from 7 cc H₂O and 90 cc alcohol gum 8.65g x x nodules
 made up of needles with up 150-160° and [α]_D²⁰ = -33.2 is pure α dextro metasacch Bismut (D.E.D.).
 The orig. alc. filtrate from crop I (46.53g) gum 47g salts which x x from 50 cc alc gum 15.58g crop II x x le with
 [α]_D²⁰ = -26.98 i.e. mixture of 40% α & 60% β . This gum with 5.35g Bismut & 8 H₂O (H₂O) 10.11g Bismut & ext. ether, alc
 & got 3.6g gum (acetic ether ext.) (x sweep over) which made up to 90.04g with H₂O gum [α]_D²⁰ =
 +16.17. This was at first supposed to be a new body but soon proved to be a mixture of α & β Dextro metasacch.,
 as was first made certain by conversion of 135g of this gum into the Dextro metasacch. salt (2.8g dissolved of H₂O),
 when got 14.5g x x nodules of α body up 180-190° and 2.86g x x salt easily sol in 20 cc absol. alcohol.
 The alc. filtrate from crop a (15.28g) above cut about 30g salts which dissolved easily in 34 cc absol. alcohol and
 on adding a x x of crop β salt got a separation of 11.23g crop b up 120-130° which together with crop II from
 A. 4.55g and crop III & B 4.9g on rexx from 15 cc 96.5% alc gum 6.42g crop c up 130-140° was
 combined and worked up for pure β dextro metasacch. gum [see below]. It was obviously first
 necessary to det. constants of α & β derivs; there was found to be as follows: not abn. reliable
see 105
 α Dextro metasaccharinic lacto- tite derivations - constants. The Ca salt x x well from H₂O
 and has already been found to give [α]_D²⁰ = -23° 25' (see No 18); the hydrazid gum [α]_D²⁰ = -30.69.
 It was obviously first necessary to prove that we have α & β Dextro metasacch. Ca was present in Cabell's gum.
 i.e. took 135.6g of the x x acid, Ca salt (see No 18 p 26 & 24) & this with 4.3g oxalic 2 H₂O alc gum 10.3g
 crude lactin gum; 2.25g of this with 4.55g Chlorine H₂O has H₂O gum 6.75g x x salts which dissolved easily in 20 cc
 absol. alcohol on warming i.e. no α & β Dextro metasacch. present. The main portion, 8g, of the lactin gum
 got x x on standing and got 1.65g cp. lactin up 89°-91° on warming x x with cold alcohol. The body x x
 from 2/3s absol. alcohol in temp. process is too soluble to be handled from this solvent; it is diff.
 soluble in hot acetic ether and dissolves in this solvent in standing or concentrating (dissolve &
 acetic ether). The alc. filtrate on scratching settling alc crop for finally 6.23g crude lactin up 75°-90°
 i.e. body is practically homogeneous & has on into lactin quantitatively. The cp. lactin up 89°-91°
 gum [α]_D²⁰ = +6.02 or 8.02 i.e. 0.9973g sub + 24.0323g H₂O: p = 3.985 d = 1.011 and found α in 2 de tube
 was +0.50 (at once) or after 1 hr standing got in a 1 de tube α = +0.33. The 0.9973g cp. lactin ag.
 sol. was now used in water with 2.8g Bismut & gum 3.5g and salt which x x from 20 cc 97% alcohol gum
 2.53g salt in opaque nodules up 130°-150° (up 130°) & got 14.5°-150°
 Bismut salt gum [α]_D²⁰ = -33° 14' i.e. 1.1466g salt + 27.5162g H₂O: p = 4.0.
 found α in 2 de tube = +2.1