

d. Mannoic acid

B lactone (+111,1) (91)
γ lactone +51,9 (91) (114)

mp gr (68/91) : Casalt -7,5 (68/91)
Brucein salt -26° 73 (34/74/75) : chinin salt -102° 7,0 2 85 (67/74)

d. Galactonic acid

mp gr (101) - Chinin Strychium + Cinchon in salt (168)
sep from taloric 137 (140) (70) Casalt (275) = +0,37 & 1,23 (93)
β lactone + its hydrate (93)
other d. galactonic acid (275) = -1,5 (189)
Brucein (194) -23,34

d. Glucronic acid

mp gr (69)
β lactone (69/92) +68°
γ lactone (92) (+67° 9)
strych (92) (-10)
Cinchonin +124,6 (38) Casalt +10,63 (61) (60)
Brucein -18,61 (60) Strychium -14,38 (63)
lactone form +29,62 Chinin -94,6 (63)

Taloric acid

ph hydr (139/140)
Brucein salt (196)
strychium
Cinchonin (53)

l. Arabinic

mp gr 94
γ lactone (94) -72,2
ph hydr +14,94 (64)

d. Arabinic acid

Brucein salt -26,33
ph hydr -12°
Casalt (53)

l. Xylonic acid

mp gr (95)
Cinchonin +126,2 (95)
Brucein -18,7 (66)
Ca +11,5 (95)

d. Lyxonic acid

(195)
Brucein -28,52 (658)
γ lactone -27,57 (53) (54)
ph hydr +8,8°
Na salt -12,04 (55/652)
Na salt -2,8

Cane sugar

glycolic acid volatility (57)

l. Idonic (96)

Brucein (170) -24,22 (96)
strychium -24,67
Chinin -109,1
Na salt -

l. Galonic acid

mp gr (96)
Brucein chinin + Strychium (168)
ph hydr (+13,74) (171)
γ lactone (172) -18,67
Na salt +11,51

Recipe

Bone Broth

- 4 Cups Bone
- 2 cups Graham Flour
- 1 pint milk
- 1 cup molasses
- 1/2 cup raisins
- 1 small teaspoonful soda in a little hot water
- 1 table spoon melted butter
- 1 small teaspoonful salt

The University of Chicago

- (1) dl α Bm propionic ethyl ester & dry acetate. 10g dl. ester & 67° at 25 mm & 700 cc absol ether + 13.65g Acetate in cold 24 hrs then at 60° 6 hrs R.T. fract. no action. (probably goes in S.T. 100°)?
- (2) dl α Bm propionic acid & dry acetate. 10g α Bm acid, 100 cc absol ether + 13.65g Acetate; a reaction rapid noticed after a period of incubation due to formation of α Bm propionate & acetic acid. After 1 hr heat R.T. 7 hrs at 50-60° filter & wash well and got 14.77g AgBr (from 1.29 AgBr) & acetic acid. Distill off (Ginsky) ether & steam oil left 100 cc & got 5.91g stiff gum white drops of acetic acid passed over to distillate. Rinse and under carefully make up to 250 cc: take 50 cc ($\frac{1}{5}$) & found 37.53 cc $\frac{N}{10}$ NaOH needed in cold: total amt. of acetic acid present = 1.1265g then for $\frac{1}{5}$ mol = 1.95g $\therefore$ gum was probably acetyl lactic acid. To form this make up to 1 L in H₂O (is difficult) and take 50 cc and found 13.24 cc $\frac{N}{10}$ NaOH needed in cold or heating B.W.B. 10' with excess NaOH etc. NB, a small amt
- took total of 35.98 cc $\frac{N}{10}$ NaOH.
- of the gum did not dissolve in the H₂O: filter ed this off & distillate was as 0.77g thick gum used in H₂O his candle; this was polylactic acid or its acetylated derivative.
- Now take rest of ag. sol. of H₂O soluble gum 5.91g - 0.77g = 5.14g - $\frac{1}{20}$ thd least B.W.B. for 17 hrs R.T. and make up to 2 L & take 100 cc and found 34.8 cc $\frac{N}{10}$ NaOH needed in cold & 35.36 cc $\frac{N}{10}$ NaOH hot: practically complete hydrolysis to lactic acid & acetic acid.
- (3) dl α Bm propionic ethyl ester and Ag₂O. 10g α Bm ester & 7.6g Ag₂O dry about temp 24 hrs then heat to 60° for 6 hrs - practically no action. - (goes in S.T. 100° MC.L)
- (4) Took 2.0g of Ca₂K's dl lactic acid (see Vo 46) 1.21 containing 2.4% excess l. acid, and let stand at 125 cc H₂O + 6.1g BaCl₂ 8 H₂O (1 mol.) for 7 days. add 38.7 cc in H₂O & got back 13.8g gum l. lactic acid by heating 90°-100° for 12 hrs; used gum α D = +5.5 is found in 10 tube = +0.22 β = 4.0
- (5) Took 1.06g l. lactic acid α D = +28.2 (see Vo. 13) and 4.12g BaCl₂ 8 H₂O (2 mols) + 20 cc H₂O and let stand at one week. Pour into 26.1 cc in H₂O & got back 0.72g gum l. lactic acid & 90°-100° for 12 hrs with α D = +29° is found in 10 tube = +1.17 β = 4.0: no change. n.b. this case to
- Jungfleisch & Goldsch is less stable of 2 forms of lactic acid.
- (6) Took 10g α dl Bm propionic acid & 100 cc absol ether + 7.6g Ag₂O; a violent interaction takes place even in cold with much least evolution of gas CH₃ CH₃ CO₂ H₂ + H₂O. after 1 hr heat to 50°-60° for 7 hrs: got 11.65g AgBr Ag₂O. rather fibrous gum 4.85g very thick gum (at 100° 25 mm) & distillate contained traces of acid only. Gum is polylactic acid: proof. Gum was practically used in cold H₂O much: make up to 1 L using approx. 600 cc H₂O & 400 cc absol & take 50 cc. This took 7.4 cc $\frac{N}{10}$ NaOH in cold and 31.87 cc (B.W.B. 10' hot etc) $\frac{N}{10}$ NaOH: at least trilactyl lactic acid but probably a mixture of various polylactyl lactic acids. Now take rest of ag. sol. soluble and least B.W.B. for 17 hrs letting absol distill off (and under) 9 mps. Now make up to 1 L & take 50 cc and found took 24.9 cc $\frac{N}{10}$ NaOH in cold or cold heat 29.88 cc $\frac{N}{10}$ NaOH: not quite complete hydrolysis to lactic acid. Now heat all of rest of ag. sol. with 5.8g ZnO 3 etc for 7 hrs B.W.B. 10' in all 5.8g ZnO 3 etc. 3 H₂O
- comp to 3.5g dl lactic acid Q.E.D.
- (7) Took 2.0g Ca₂K's lactic acid (2.4% excess l. form) and 6.1g BaCl₂ 8 H₂O 1 mol. + 20 cc H₂O and least B.W.B. R.T. for 10 hours and got 13.8g AgBr & then 13.5g lactic acid gum α D = +5.5 is found in 10 tube = +0.22 $\therefore$ no change. Q.E.D. (do. do a previous exp't when it was dry week of. see No 4)

(84) dl lactic acid + acetic anhydride m + .

Now treat the 27.16g residue ~~with~~ ^{with} ~~distilling off~~ ^{distilling off} again in ~~vacuo~~ ^{vacuo} (25 mm 100°)
decided heat evolution (due to presence of some anhydride?) on distilling off again in ~~vacuo~~ ^{vacuo} (25 mm 100°)
got back 25.38g green: this dissolved at once in 25 cc cold H₂O on distilling off again (25 mm 100°)
a small ^{acid} ^{water} ^{soluble} ^{solid} ^{residue} ^{left} ^{is} ^a ^X. Now make up quickly
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8.8200 $\frac{n}{N}$ Nurt in cold ...
A + Hw. 102 Ldl Brown perfume and rose Hw, + 7.68 agro toluene

porosa *bracteata*. 10 } all *Bryophyllum* *aer.*, now at Hn and 1097

all lactic acid at 90° at 25 mm. Took 10g carboxylic acids from 30°-75° to back 8.28 fairly thick but mobile

new solid stepon substance is only very strong. 105 Car 7 K's lactic acid (excess 22.4% lactic) heated in air stream
105-108-109-110-111-112-113-114-115-116-117-118-119-120-121-122-123-124-125-126-127-128-129-130-131-132-133-134-135-136-137-138-139-140-141-142-143-144-145-146-147-148-149-150-151-152-153-154-155-156-157-158-159-160-161-162-163-164-165-166-167-168-169-170-171-172-173-174-175-176-177-178-179-180-181-182-183-184-185-186-187-188-189-190-191-192-193-194-195-196-197-198-199-200-201-202-203-204-205-206-207-208-209-210-211-212-213-214-215-216-217-218-219-220-221-222-223-224-225-226-227-228-229-230-231-232-233-234-235-236-237-238-239-240-241-242-243-244-245-246-247-248-249-250-251-252-253-254-255-256-257-258-259-260-261-262-263-264-265-266-267-268-269-270-271-272-273-274-275-276-277-278-279-280-281-282-283-284-285-286-287-288-289-290-291-292-293-294-295-296-297-298-299-300-301-302-303-304-305-306-307-308-309-310-311-312-313-314-315-316-317-318-319-320-321-322-323-324-325-326-327-328-329-330-331-332-333-334-335-336-337-338-339-340-341-342-343-344-345-346-347-348-349-350-351-352-353-354-355-356-357-358-359-360-361-362-363-364-365-366-367-368-369-370-371-372-373-374-375-376-377-378-379-380-381-382-383-384-385-386-387-388-389-390-391-392-393-394-395-396-397-398-399-400-401-402-403-404-405-406-407-408-409-410-411-412-413-414-415-416-417-418-419-420-421-422-423-424-425-426-427-428-429-430-431-432-433-434-435-436-437-438-439-440-441-442-443-444-445-446-447-448-449-450-451-452-453-454-455-456-457-458-459-460-461-462-463-464-465-466-467-468-469-470-471-472-473-474-475-476-477-478-479-480-481-482-483-484-485-486-487-488-489-490-491-492-493-494-495-496-497-498-499-500-501-502-503-504-505-506-507-508-509-510-511-512-513-514-515-516-517-518-519-520-521-522-523-524-525-526-527-528-529-530-531-532-533-534-535-536-537-538-539-540-541-542-543-544-545-546-547-548-549-550-551-552-553-554-555-556-557-558-559-560-561-562-563-564-565-566-567-568-569-570-571-572-573-574-575-576-577-578-579-580-581-582-583-584-585-586-587-588-589-590-591-592-593-594-595-596-597-598-599-600-601-602-603-604-605-606-607-608-609-610-611-612-613-614-615-616-617-618-619-620-621-622-623-624-625-626-627-628-629-630-631-632-633-634-635-636-637-638-639-640-641-642-643-644-645-646-647-648-649-650-651-652-653-654-655-656-657-658-659-660-661-662-663-664-665-666-667-668-669-670-671-672-673-674-675-676-677-678-679-680-681-682-683-684-685-686-687-688-689-690-691-692-693-694-695-696-697-698-699-700-701-702-703-704-705-706-707-708-709-710-711-712-713-714-715-716-717-718-719-720-721-722-723-724-725-726-727-728-729-730-731-732-733-734-735-736-737-738-739-740-741-742-743-744-745-746-747-748-749-750-751-752-753-754-755-756-757-758-759-760-761-762-763-764-765-766-767-768-769-770-771-772-773-774-775-776-777-778-779-780-781-782-783-784-785-786-787-788-789-790-791-792-793-794-795-796-797-798-799-800-801-802-803-804-805-806-807-808-809-810-811-812-813-814-815-816-817-818-819-820-821-822-823-824-825-826-827-828-829-830-831-832-833-834-835-836-837-838-839-840-841-842-843-844-845-846-847-848-849-850-851-852-853-854-855-856-857-858-859-860-861-862-863-864-865-866-867-868-869-870-871-872-873-874-875-876-877-878-879-880-881-882-883-884-885-886-887-888-889-890-891-892-893-894-895-896-897-898-899-900-901-902-903-904-905-906-907-908-909-910-911-912-913-914-915-916-917-918-919-920-921-922-923-924-925-926-927-928-929-930-931-932-933-934-935-936-937-938-939-940-941-942-943-944-945-946-947-948-949-950-951-952-953-954-955-956-957-958-959-960-961-962-963-964-965-966-967-968-969-970-971-972-973-974-975-976-977-978-979-980-981-982-983-984-985-986-987-988-989-990-991-992-993-994-995-996-997-998-999-1000-1001-1002-1003-1004-1005-1006-1007-1008-1009-1010-1011-1012-1013-1014-1015-1016-1017-1018-1019-1020-1021-1022-1023-1024-1025-1026-1027-1028-1029-1030-1031-1032-1033-1034-1035-1036-1037-1038-1039-1040-1041-1042-1043-1044-1045-1046-1047-1048-1049-1050-1051-1052-1053-1054-1055-1056-1057-1058-1059-1060-1061-1062-1063-1064-1065-1066-1067-1068-1069-1070-1071-1072-1073-1074-1075-1076-1077-1078-1079-1080-1081-1082-1083-1084-1085-1086-1087-1088-1089-1090-1091-1092-1093-1094-1

probably not

(is work! : ? enough)

(13) The resolution of dl lactic acid; took 96.3g Chlorine & 30g CaH₂Ks at last night;
cont. 224% ex ess lactic acid, heated BWWB with H₂O for 7½ hrs got 173g salts (90° vacuum)
which are xx from 126 cc H₂O gave a mass of xx needles of d. lactie Chlorin 29g crop I kept
150-172° containing xx H₂O; ag. filtr distilled off gave 135g salts which xx from 80 cc H₂O gave 17.5g
crop II same salt upr 150-172°. ag. filtr distilled off gave 80g salts which xx from 80 cc H₂O gave only
7.5g crop III l. lactie Chlorin. On crystallizing crops I + II 46g from 160 cc H₂O got 23.11g
crop I (in vacuo) upr 150-173° tent yx H₂O (?) observedly l. lactie Chlorin with [α]_D²⁰ = -12.4°, n_D²⁰
i.e. took 0.9985g + 24.073g H₂O found d in 1 dc tube = -5°.00.
The eq. filtrate from crop I above after adding crop III, gave on xx from 52 cc H₂O, ale 11.06g mon, crop II,
l. lactie Chlorin upr 150-173° solen 4.96g crop III a new same salt i.e. total of 23.11g
11.06g + 4.96g l. lactie Chlorin = 39.13g. On heating this with 15.0g Bar M₂S H₂O (heavy 14.7g) ale
on BWWB got 29.92g Chlorin and 7.87g l. lactie acid gum which was ± near a up to 250cc H₂O.
On weighing taking 5cc (: 0.1574g) this took 16.5 cc $\frac{n}{10}$ NaOH in cold + 16.33 cc $\frac{n}{10}$ NaOH hot : OK.
On taking 25 cc mon of ag. sol. standing with 81.65 cc $\frac{n}{10}$ NaOH ale BWWB + then making salt (Heavt = 0.9146g)
up to 228.646 g with H₂O (: p = 4.0) found d in 2 dc tube = +0.58 or [α]_D²⁰ = +7.17 for l. lactated of Na.
l. lactyl lactic acid: On taking 50 cc mon of abov solution : 1.574g, soluble of H₂O sheet mid used gum
at 90° for 13½ hrs in air stream in vacuo got 1.33g l. lactyl lactic acid which with 31.92g H₂O : p
= 4.0 gave d in 2 dc tube = +2.28 ; [α]_D²⁰ = +28.2. Tubation making up to 250 cc taking 50 cc
of 0.266g took 19.4 cc $\frac{n}{10}$ alkali in cold and a total of 31.53 cc $\frac{n}{10}$ NaOH hot : substance was
about 75% to 80% l. lactyl lactic acid (Should least BWWB) slactyl lactic must be about [α]_D²⁰ = +35°.
l. lactie Brucine. On taking 50 cc mon of abov solution of l. lactie acid; 1.574g, standing with 6.4g Brucine
7.65g xx salt which dissolved in 20 cc hot absd. alcohol & gave white still hot very rapidly transparent
clear as glass, opaque, octahed al & cubical in appearance measurable; 6.15g upr 208-209° with [α]_D²⁰
= -28.84 (? if error in reading); 10.692g + 25.5293g H₂O given d in 2 dc tube = -20.345. Proof on taking
5.0g of op. salt + 0.12g Brucine and 70-100 cc H₂O standing in flask BWWB for 5 hrs, extracted with 8 H₂O x
and then got 1.15g crop I xx salts with [α]_D²⁰ = -28° and then 2.7g crop II salts with [α]_D²⁰ = -26.9°
p = 4.0 acc. det in all cases : variation due to inaccuracy of opt. det. trust to fore Brucine. ..
^{orig.} The eq. filtrates from crops I II + III l. lactie Chlorin on distilling off gave 8.6g salts which xx from 30 cc
H₂O got entirely solid but on adding 50 cc ice H₂O (^{as before}) practically all dissolved; can't be handled in this way
or by use of H₂O cooled according to Jungflossch Goldsch CR. 139, 56 203 Bull 41 222 (1884) of Phloridin
Bull 41, 217 CR. 140 719; 141 iii; 102 515, 637:
i.e. set free gum by 33g Bar M₂S H₂O typ 58.2g Chlorin and 15.38g and d. lactie acid (50° 25 mm only),
made up to 500 cc and take 5 cc found it took 15.4 cc $\frac{n}{10}$ NaOH + 16.1 cc hot. Now took 100 cc of tot
i.e. 3.07g stand with 12.6g Brucine typ 16g salts which xx from 32 cc absd. alcohol gave (2 days o.r.)
10g salts which xx from 50 cc absd. alcohol gave 8.0.1g salt upr 208-210° [α]_D²⁰ = -31.2 both acc. det [Kelp]
got from 40 cc absd alcohol 6.1g salt upr 208-210° [α]_D²⁰ = -31.2 both acc. det [Kelp]
On taking another 25 cc of abov sol. of ande d. lactie acid standing with 80.5 cc $\frac{n}{10}$ NaOH ale
got on dissolving Na salt heavy 0.957g in 22.97g H₂O (: p = 4.0) d in 2 dc tube = -0.18 whence
[α]_D²⁰ = -2.225 ∴ acid is only about 30% dex tro lactic acid (rest is ± acid)

(4)

(14) Intgr of Formose, of Ann 376 p. 107. (a) 347g 35% CH₂O cat. K's + 547g H₂O + 2.92g
Rosen heated B.W.B. 2 hrs then add 153g oxalic acid to get 1 1/2 hrs at 60° 20 mm 22.5g
faintly yellow Formose gum; lost yet. This gave 83.3% sugar by tit. Fehd. sol. (22g gum).
+ treat the rest with Br₂ (15g) in aq. sol. Formed Br₂ all gone after 37 days (22g gum).
(b) made 110g Formose gum acc to Ann 376 p. 107, and acetylated this with 330 cc acetyl chloride 17 hrs
90°-100° got 201g acetylated gum all soluble except 3.6g brown tar into 3.3 L cold ether.
On hydrolysis of the 187g sol acetylated gums 90° 25 mm 3x with 5 pts H₂O gave 108g I;
91g II and 87g gum III. Extraction of 87g crude Formose gum 2x with 2 L ethyl acetate
work out only 11.5g mobile gum A and left 74g insol tarry gum which dissolved in 800cc
alcohol & not 800 cc ether (heating) gave large and used sticky syrup separating out at
first in flakes but transformed on C.P. in to a sweet sticky syrup (discard)
The other alcohol filtrate gave back 54.9g syrup which titr with Fehd sol
contained only 32% the % sugar compared with D. glucose :: discard.
The 11.5g mobile gum A similarly treated contained only 29% the sugar :: discard
obviously much decomposition of Formose sugars takes place in hydrolysis of
acetylated gums -
(c) Made new lot of Formose acc to (a) but heated 8 hrs B.W.B. by adding 153g oxalic acid
and got 220g Formose gum (1/2 hrs 60° 25 mm): much darker than a and giving by titr only
68.9% % sugar i.e. made up to 250cc: 5cc of this made up to 100cc + 16.5 cc of this = 100cc Fehd. Solution =
68.9% D. glucose. Now take rest of sugar 21.5g, & add 153g Br₂ & H₂O (? how much) & make all Br₂
disappear after 3 days still gave after 37 days (disc. cand.).
(d) Euler's method is neg. Took 90g 35% CH₂O + 547g H₂O & 2.0g CaCO₃ heated B.W.B. for 31 hrs
(of Euler Br 39 p) : 2 lots: filtered off 3.0g unol CaCO₃ got only a total of 3.5g residue
mostly paraformaldehyd ind. f. at 90° 25 mm :: no condensation.
(e) Euler's method is neg. Took 60g 35% CH₂O 1 L H₂O + 10g CaCO₃ and heat for flame R.T.
heated for 16 2/3 hrs in a brisic of H₂O & and a coloration; the alkaline solution was
filtered off when distill off H₂O in vacuo (100° and) and got back only a gummy 4x residue, 5.0g,
smelling strongly of CH₂O obviously consisting only of para CH₂O & Ca formate.
(f) repeat (e) using 60g CH₂O (35%) + 1 L H₂O + 10g BaCO₃ (Ca7K's best heat B.W.B. for 16 2/3
hrs: filter distill off solution, much sweet strongly of CH₂O, in vacuo got back only 1.8g
solid mainly para CH₂O + Ba formate
(g) 60g CH₂O 35% i.e. Ca7K's Ph IV and 0.5g H₂O and 2.0g Ph₂H₂ heated B.W.B. nosp.
1 1/2, 2 and 2 1/2 hrs : 3 lots - set sugar in each lot after distilling off at 25 mm 50° flaking up
in alcohol distill using no oxalic to get rid of Ph₂H₂ - got 8.95g Formose I 60-63% sugar.
got 11.8g Formose II 70-5-74% sugar; got 12.8g Formose III 65% sugar.
:: Formose is obviously changed by leaving Ph₂H₂ present and poor yield; also
must be some para CH₂O present in I II & III Formose gums. To purify latter took
Formose I. from steam then solution & then got back 7.3g Formose gum I (ale ext) which
now heated between 74-78% sugar by Fehd. solution.

N.A. Chas hardly reduces Rbl sol. even at 100° but very slowly whereas does reduce it at 30° i.e. the degree controlled by pressure

(as in 9)

(15) (ii) Took 70g ANO 35. 15 minutes stamp went on.
2 hours: take off slab stand on table for 15 minutes stamp went on.
sol after off next day. 2 expts in which I was heated harder & flames under BUBB solution of lead.
As heat mixture on BUBB the Pb part first cakes & then gets black then Pb fraction in small amt. soln
at end of 3/4 hr clears up & changes to a white opalescent solution finally at end of 1 3/4 hrs still
small of ANO is only very faint opt a sweet odor.
73.2 576% Sugar as det by tit.
78- and 80% " " " "

(15) Formose + 33% NaOH
this in H₂O divided into 2 eq. parts. ∴ added 150g
to a bott (450) of 375g NaOH (98.6%) = 6% in H₂O. Temp. rose to 77° in each case. ^{Heavy} 1766g text salt mixture with alcohol 240g water
for 6 hrs and 1725g H₂O (18 NaOH = 4.7069g H₂O used) Heavy 1766g text salt mixture with alcohol 240g water
of 276g Saccharin gum (NaCl) which degraded 3x with 2 L ether LK out 79.65g Saccharin A; then dissolved
in 50% H₂O and gave 49.25g gum A (∴ 4.7g loss) or total loss = 30.4g.
and 3rd exp for 17½ hrs gave 49.25g gum A (∴ 4.7g loss) or total loss = 30.4g.
then ext. mod. Saccharin 3x with 2 L ethyl acetate R.F. got about 81.3g Saccharin B which in H₂O exp 100%
for 24½ hrs and down to 74.9g (∴ 6.4g loss); 3rd exp 17½ hrs gave 68.4g gum B ∴ loss = 2.7g (or total loss = 9.1g).
The mod. Saccharin 2x further extraction with 2 L active ether R.F. gave 26.3g more gum C, and as
salt mixture was sticky it was now ext. with alcohol and gave 80g gum containing tar + sun. and NaCl
and this on further digestion with 2 L ethyl acetate enabled to obtain powder 64.6g dis. cardal whereas the
acetic ether filtrate was mixed with fraction C and this then exp. on B for 24½ hrs wt then was 23.6g + after
2nd exp. of 17½ hrs weight of C fraction was 22.75g and it was now converted with fraction B 68.4g
making a total of 91.15g Saccharin + those with fraction A 49.25g made
total of 140.4g C₆H₅CO₂ gums [59.5g C₆H₅CO₂ gums being dissolved off by
exp. 1

The Agum, 49.25 g, was now extd R.F. 2x with 2 L Ether and 36.5 g and 6 g wup by diff
dissolved leaving (acc det) 7.5 g insol gums. Acc. det of ether exts gum 41.6 g gum (90° 25 mm)
(mobile spent acid w/ in cold). Titration of tooth took 14.2 cc $\frac{N}{10}$ NaOH in cold and total of 36.71 cc
 $\frac{N}{10}$ NaOH lost $\therefore$ must be lactyl lactic acid present.
The other gums 91.15 g extd 4 x R.F. with 2 L Ether took out by diff 15.5 g + 10 g + 9 g + 5 g
wup. and left acc. det 47 g insol C₆ gums. Acc. det of ether exts gum 45.15 g gums (sweet &
mobile in cold) Titration of tooth took 13.5 cc $\frac{N}{10}$ NaOH in cold and total of 38 cc $\frac{N}{10}$ NaOH lost $\therefore$ unite
both ether extracts 86.75 g - 0.87 g used for titr. Stamp Buns with H₂O for 40 hrs got back 69.03 g gum ($\therefore$ loss =
16.85 g) : 2nd wup. for 28 hrs then gave back 65.3 g C₆ gums [loss 3.75 g] or total loss this wup.
60.9 g C₆ gums. On extracting the 7.5 g C₆ gum above with 300 cc ethyl acetate all except
20 g black tar (is caudal) dissolved: similar extraction of the 47 g C₆ gums 3x with 1 L ethyl acetate
took out (by diff) 20 g + 9.0 g + 3.0 g gums leaving (by diff) 15 g insol brown tar which
was discarded. The total weight of all the acetate ether solutions of C₆ gums was now det
as 38.4 g. This was now mixed by digestion with 100 cc ethyl acetate many decants & cool etc
into 18.7 g gums, easily soluble in cold acetate ether, and into 18.45 g diff sol. C₆ gums.
The titration of the 18.7 g gum made up to 250 cc & take 5 cc ($\therefore$ 0.374 g) gum 7½ cc in cold & 26.79 cc $\frac{N}{10}$ NaOH
lost (theory for C₆ = 28.33 for C₆ = 23.1 cc) $\therefore$ most part of sol. with 51.0 g Bunsin got 74.2 g salts which diss
in 150 cc abso. alc gum slowly 14.03 g heavy xx & Bunsin salts up to 185-190° and then 8.55 g wup II some salts up to 180-187°
The alc. filtr gave 49.5 g salts gum which with 18.5 Bunsin de gum back 31.7 g Bunsin and 11.8 g gum (alc ext) see p. 7
Titration of 18.45 g C₆ gums made up to 250 cc & take 5 cc ($\therefore$ 0.369 g) took 5 cc in cold & 21.7 cc $\frac{N}{10}$ NaOH lost (theory
C₆ lactone = 22.8 cc) $\therefore$ most then Bunsin with xx gum. Steychnin + 26.7 g alkalin went in got 50 g gumming
salts which dissolved clear in 100 cc warm abso. alcohol and on cooling some oily salt separates out turnish in
on adding 200 cc more abso. alcohol ($\therefore$ 300 cc in all); decant from there, 15.5 g by diff, wash wall; left free
acids by 6.5 g Bunsin got 7.4 g Steychnin and 5.5 g C₆ gums I (alc ext); titration taking 0.33 g took 17.1 cc
 $\frac{N}{10}$ NaOH: most part with 10 g Bunsin got 16.9 g salts which burnin gum no xx salts, see p. 7
The alc. sol. Steychnin salts 34.5 g, gum with 44 g Bunsin de 17.46 g Steychnin and 11.45 g C₆ gums II (alc ext);
titration of 0.3435 g took 18.7 cc $\frac{N}{10}$ NaOH: most part with 24 g Bunsin got 39 g salts which xx from 80 cc abso
alcohol gum 30.5 g xx salts up to 200-205°
The 65.3 g C₆ gums above made up to 1 L & take 5 cc took 9 cc $\frac{N}{10}$ NaOH in cold & total of 26.15 cc $\frac{N}{10}$ NaOH lost
(theory for C₆ lactone = 24.73 cc) $\therefore$ some lactyl lactic acid still present. Now take rest of aq. sol. Sterch Buns 9 hrs R.F.
and then double off H₂O in steam (booth 60° only) got 63.5 g gum which extd R.F. 8x by 2 L ether took out by diff
27.5 g + 16.0 g + 4.0 g gum : II + III extd acc det = 18.35 g C₆ gums. The part left wup = acc det 14.5 g stiff gums
On dissolving the 31.3 g gum in 60 cc H₂O extd 20x by ether got 18.05 g ether sol. gums mainly illustic acid
and got 13.45 g C₆ gums + in H₂O.
The 18.05 g lost wup Buns. H₂O for 22½ hrs went down to 14.75 g: titration, taking 0.295 g, took 14 cc in cold &
27.53 cc hot (theory for C₆ lactone = 24.35 cc) $\therefore$ wup again Buns. with H₂O for 24½ hrs and got back 10.6 g thick
gum; 3rd wup Buns. for 1 hrs got back 9.0 g stiff gum. Titration of last, taking 0.36 g, took 15.5 cc in
cold & total of 29.6 cc $\frac{N}{10}$ NaOH lost (theory for C₆ lactone = 29.3 cc) $\therefore$ most part with 27.0 g Bunsin got
36.85 g salts which dissolved in 80 cc abso. alcohol gave 7.7 g xx, cup I, up to 190-200°
then only 18.6 g cup I similar salt up to 190°-204°. (alc. filtrate) see p. 7

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The 13.45g C_6 gums I (in H₂O) on titration taking 0.2699 took 6.8 cc in cold total of 22.0 cc $\frac{N}{10}$ NaOH test
 (theory for 8.666 = 20.4 cc) :: treat with 4.20g Baumein $\frac{1}{2}$ got 6.2g xx salts which xx from 180 cc alcohol gum
21.21g crop I salts (18 hrs) up to 195-200 and then 12.15g crop II salts up to 175-185; alc. filter united
 see below.
 The 18.3g C_6 gums II, taking 0.366g, took 9 cc $\frac{N}{10}$ in cold total of 28.75 cc $\frac{N}{10}$ NaOH test :: treated with
 55.0g Baumein $\frac{1}{2}$ got 78g xx salts which from 200 cc alcohol gum 24.55g crop I xx salts up to 195 (18 hrs)
 and then 11.92g crop II salts up to 175-185. Now unite now xx alc. filtrates from C_6 gums I & II & treat
 with 2.0g Baumein $\frac{1}{2}$ got 38.1g Baumein and 7.93g gum (ether extr)
 6.35g gum (alc extr)
 The 14.5g gum (orig 6.35g) left in alcohol on 3x digestion with 2 L ether gum on titration (taking
 0.29g) 20.2 cc $\frac{N}{10}$ NaOH test :: treated with 38.5g Baumein $\frac{1}{2}$ got 53g xx salts which xx from 600 H₂O &
 150 cc alcohol gum 0 :: set free gum by 20g Baumein $\frac{1}{2}$ got 36.83g Baumein and 12.2g gum (alc extr)
 This was digested R.F. with 25 cc alc. ether cool & decant; do. do. with 6.3g lot above using
 15 cc solvent $\rightarrow$ see below.
 The 7.93g gum (ether extr above) on titration, taking 0.3172g, took 25.35 cc $\frac{N}{10}$ NaOH test :: treat
 with 23.5g Baumein $\frac{1}{2}$ got 34g xx salts which xx from 70 cc alcohol gum crop I 14.05g salts
 up to 185-200 - the alc. filtrate and 17g salts + was now united with a crop one from bottom p 6
 (9.0g gum + 27g Baumein etc) got a total of 44.1g salts which with 17.5g Baumein alc gum 29.75g Baumein
 and 9.8g gum (alc extr); this digested with 24 cc ethyl acetate cool & decant in all 4 lots as shown.
 (see note p 6) was digested with 24 cc ethyl acetate cool & decant & got then 20.0g C_6 gums; got from $\frac{N}{10}$ NaOH test
 unite the portions used in ethyl acetate cool & decant & got then 20.0g C_6 gums; got from $\frac{N}{10}$ NaOH test
 (in cold ethyl acetate) 20.5g gums of which on digestion with 0.5 L ether 14.1g alc. det. dissolved,
 leaving as det 5.6g insol C_6 gums :: unite with 20.0g lot in all 25.6g, & found these took
 up in 2 days 4.25g Styphnium $\frac{1}{2}$ got 7.25g gummy salts which turned dissolved in 400 cc
 97.5% alcohol $\frac{1}{2}$ got no insol Styphnium salts on standing recouling :: N.G. :: set free gum
 25.6g (alc extr) by 30g Baumein $\frac{1}{2}$ & alcohol by digestion with 250 cc ethyl acetate into 15.05g soluble gums
 acid into 9.15g gum insoluble in this amt. of solvent; the 15.05g gum was then further
 dissolved by digestion with 500 cc ethyl acetate cool & decant into 8.35g soluble gum and into 6.7g insol
 C_6 gums. Digestion of the 9.15g C_6 gums with Styphnium (7 $\frac{1}{2}$ hr) took up 12.25g Alkaloid and gum
 22.1g salts (50% w/w) on distilling these in 200 cc alcohol & see H₂O got on cooling 7.25g insol
 gummy salts which with 3.5g Baumein gum 3.2g Styphnium and 2.55g C_6 gums which was :: united
 with crop. lot 4.7g, obtained before (p. 6 near middle) :: 7.25g in all. Acylated this with 22 cc anhydrous
 for 8 hrs at 100° $\frac{N}{10}$ NaOH test 10.75g acylated gums of which 3.7g remained as gum used in hot ether. got from other 5.0g
 she 6.7g insol C_6 gums above on titration 0.268g took 17.5 cc $\frac{N}{10}$ NaOH test and was :: treated with 16.5g Baumein
 and gum 24.45g salts which xx from 500 cc alcohol gum 2.6g xx salts up to 180-202 and then no
 more xx salts.
 The alcohol soluble Styphnium salts above, 14.85g, gum back with 7.0g Baumein to 8.53g Styphnium &
 5.5g C_6 gums.
 Titration of the 8.35g gum above, taking 0.334g, took 24.85 cc $\frac{N}{10}$ NaOH test :: treated
 with 23.5g Baumein and got 36.3g salts which xx from 500 cc alcohol gum 9.45g xx salts up to
 180-200, crop I.
 It is obvious from the above that could get no xx derivatives of the
 C_6 gums & that the high melting Baumein salts obtained mainly from
 the C_6 gums were mixtures of all three and all mytho C_6 metacaccharin
 containing also C_4 , C_5 , and C_6 -isocaccharinic acids (all).

The 14.1g gum taken up by 0.5 L ether R₇ (see middle p 7) on weighing 0.282g took up 12 cc $\frac{n}{10}$ NaOH in cold and 24 cc $\frac{n}{10}$ NaOH hot :: some lactic acid still present :: evap BuOH & H₂O for 24 1/2 hrs & got back 11.7g gum: Whiten of 0.234g took 9.2 cc in cold & total of 18.2 cc $\frac{n}{10}$ NaOH hot :: treat with 35.0g BuOH & got in all by xx only one crop 9.15g, mpt 190°-210°, xx salts 140.37g

Summary: Had a total of 128.32g xx BuOH salts mpt 175-210° as follows: 14.03g + 8.55g; 3.05g; 7.7g + 1.86g; 21.21g + 12.15g; 24.55g + 11.92g; 14.05g; 9.15g; 9.45g + 2.60g
The C₅, C₆ gums left on setting free gums from various alc. filtrates etc by BuOH etc removed to following aunts. 8.7g C₆ gums and 28.6g C₆ gums A. Gum A was now freed from C₅ gums by 4X digestion with 0.5 L ether each time & 3.65g, 4.25g, 3.0g + 2.0g by diff succ. dissolved (an ether still in hand) leaving or acc. det of total ether extr = 14.1g C₅ gums (? on hand). The insol. portion, 14.7g by diff., was now united with 8.7g C₆ gums and total amt by acc det found to be 22.9g. This was resolved by 2X digestion cold and decant with 150cc ethyl acetate each time into 10.95g soluble very stiff C₆ gum. The insol. portion again digested 2X with 0.5 L ethyl acetate gave 3.25g stiff C₆ gum and the insoluble portion, 7.8g, was now acetylated thus with 24 cc acetyl anhydride and gave 12.1g acetylated gums from which a digestion got much insol porous brown tar (in ether used) of the 5.53g ether sol acetylated gums finally gave on 1X hydr & H₂O from 10g BuOH & 20g H₂O 3.15g most diff sol. C₆ gums which is in hand but not giving xx salt.

The xx BuOH salts mpt 175-210 total 140.37g; a sep. of these could finally be accomplished by xx from H₂O + 10pts alcohol into (a) BuOH salts mpt 210-225° got 50.55g salts into (b) 43.4g salts mpt 180-210° a mixture of a and c (see note 1c) 40.4g non xx (from alcohol) measuring BuOH salts. Treatment of (a) with 16.0g BuOH etc gave 37.3g BuOH and (b) 9.78g C₆ gum (ether extr (a)) & 0.90g C₆ gum (acetic ether extr). (c) 10.68g C₆ gum (ether extr) mobile in cold & 1.60g C₆ gum (acetic ether extr) stiff gum.

Treatment of (c) with 13.0g BuOH etc gave 25.85g BuOH and 0.643g C₆ gum (ether extr) mobile in cold & 1.60g C₆ gum (acetic ether extr) stiff gum. Gums (a) and (c) ether extr needed up to 250 or 300 cc H₂O was dried & optically. Gum (a), 0.586g, treated with 0.6cc ph hydr & 1cc alcohol of gum no xx salt even when seeded with all three dl erythro & metasacch. phenyl hydr mpt :: discd. Gum c, 0.648g, similarly treated with 0.6cc ph hydr & 1cc alcohol gave no xx salt even when seeded with all three dl erythro & metasacch. ph. hydr mpt :: discd. Now treat rest of gum c 5.84g with 14.33g Chinin and also 1.6g tot persorp. with 5.2g Chinin and got 21.1g xx salts and 8.09g ~~gum~~ soluble in cold absol. alcohol, same body obtained from gum got 0.38g of a salt diff soluble in H₂O, but soluble in cold absol. alcohol, same body obtained from gum a see below :: finally unite. The ag. filtrate distilled off gave 21.1g xx salts which xx from 40cc absol alcohol gave 4.63g mpt 170° with $\alpha_D^{20} = -107.1$ acc. det. The alc. filtrate from crop I, 4.6g, gave 2.59g xx salts which xx from 40cc absol. alcohol, mpt 170° with $\alpha_D^{20} = -107.1$ acc. det. Now unite the 2.54g + 1.57g Chinin salts in cold (ether extr) with $\alpha_D^{20} = +8.5$ i.e. formed with $\alpha_D^{20} = -102.1$. Now distill off H₂O and treat gum with 1cc ph hydr & 1cc alcohol & got after BuOH etc 2.8g Chinin and 1.0g mobile gum in cold (ether extr) with $\alpha_D^{20} = +8.5$ i.e. formed & in 1cc tube (p = 4.0) ± 0.34 . Now distill off H₂O and treat gum with 1cc ph hydr & 1cc alcohol & got 0.5g xx salt 24 hrs or more on ph. hydr mpt 100-110°. The xx this from 2cc alcohol & 10cc ethyl acetate got 0.5g xx salt 24 hrs or more on ph. hydr mpt 120-130° which are obviously all three C₅ metasacch. ph. hydr (dis carb.) obviously a mixture

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on localities filices from all three (Cs) metasand Chiric (4)
I. P. 1 Oct 1 1911

NB3²

with npt 194° = +3.25,
with Id 7% ($\rho = 4.0$) = +3.25,
xx Brouin salt which xx well from 4 pts hot H₂O given
filtrate gave on evap. in vacuo sup. xx residue with abs. alcohol
to leave 2 bodies present. ~~It is a salt & has a sharp m.p.~~
the x rid of the 2.1g Chlorin salt diff sol. in H₂O gave 33.6g xx Chlorin salts which
volatly gave 0.61g xx needles wpt 194° mp I, easily soluble in
abs. alcohol gave 1.17g sp.
3.25 sp III npt

cold H₂O with [α]_D²⁰ = -115.1°
 got 4.02 g more same salt crop II at 194°
 salt m.p. 195° [α]_D²⁰ = -115.1°
 194° subsequently of correct [α]_D²⁰ = -115.1°
 CH₃ glycerin is Chlorin: white crops obtained i.e. 3.25g + 1.17g + 1.15g fairly stiff gum in cold
 2.5g Bull. type 3.74g Chlorin & 1.15g fairly stiff after 24 hrs at 194°
 Traced it with 1 ceph hydr + ice alcohol sept after 24 hrs at 194°
 insoluble in cold water
 by hydrolysis with 3.0g Bull. H₂O
 C₆H₁₂ etc &

on adding the remaining 0.8 g gum soluble in alcohol & p/land off 2.0
 ± 2 CH₃ glyceric acid 0.8 g gum soluble in alcohol & p/land off 2.0
 8ms BMB for back diff soluble in alcohol & p/land off 2.0
 give a xx BMB salt diff soluble in alcohol & p/land off 2.0
 only rest xx from this state: 2 CH₃ glyceric acid 0.8 g gum soluble in alcohol & p/land off 2.0
 on xx this from 24.2 g Formic, water, Nov 22 See No 14, dissolved in 800 cc H₂O closed flask
 1.0 g CuCl₂ 2nd day 5.0 g; dose about 5.0 g for 9 days more and then
 1.0 g for 30 days at on temp closed flask spirally
 1619 gum; hepathe

42.8g insoluble C₆ gums. The 42.8g C₃ C₄ gums were now heated BvB for 12 hrs to convert
all lactate was dissolved in spirit and filtered off from 4.03g mud brown Casals
oil lactate was dissolved in spirit and filtered off from 7.6g gum which
is carbon; the alc. soln gave back 76g gum which
sol. gums of which 32.6g brown fumes tar
95.65g, 1x by 3pts

[Cont as above] acetylated & his stuff in lot other his was by WOG Bacteria
remained until in lot other which by WOG Bacteria
His gun 648 grams were included with the 25.0g G gums previously
Extract this R.F. 4x with 1 L Ethylacetate + hex.
 $\therefore$ total A+B+C = ~~125.5g~~
127

100g sugar $\frac{36}{100}$ equiv to 67g PbO_2 (44/10)

and got resp 20.65g gum I; 12.25g gum II and 12.15g gum III ($\frac{36}{100}$ + $\frac{4}{100}$ exto combined),
the insoluble portion, 31g, acetylated again with 93 cc acetyl anhydride. When some gave 59g acetylated gums of
which 48.9g dissolved in ether soln in 1 $\frac{1}{2}$ hr. with H₂O gave 32g gum soln by 60g BaO₂ at 120

cc H₂O on B.W.B. got finally 24.67g of gums IV (ale extr) all on hand Oct 1-1911
Glycerol + 33% NaOH

(17) d. Fructose + PbO_2 - of Libby de Brugn. Took 25.0g saccharine from Dublin, [27] $\frac{36}{100}$ = 4.0 = -90° 95',
and 650cc H₂O + 2g PbO_2 (from make, see No 12, 1904) heated B.W.B. for 8 hrs shaking time to time.

Solution was strongly colored, and 1.04g oxalic acid (thru 1.045g) filter still off 60° 20 min got
2.50g sugar [N.B. no CO₂ or C₂H₅OH etc.]; this gave 69% sugar as det by tit with Fehd. sol. and

on making up to 500 cc gave [27] $\frac{36}{100}$ = -9° 68' e found 2 in 1 de hite = -0° 48' (C = 5.0)

Repeat using 3X amount of 250g NaOH in 413 cc H₂O (+ 25) time with 50 cc H₂O. Temp rose to 35°, then
H₂O and add to a sol. of 250g NaOH in 413 cc H₂O (+ 25) time with 50 cc H₂O. Temp rose to 35°, then

least B.W.B. for 6 hrs in usual way and 11% of HCl (15 NaOH used = 4.0789g HCl); 1177g thru &
got 78g gum (ale extr). This was extracted 3X with 12 ethyl acetate and got 32.4g gum
The acetic ether extracts were resolved by ether, R.F.

gave 78g gum (ale extr). The acetic ether extracts were resolved by ether, R.F.
pours tar (27.9g dissolved in vacuo) dis. cant. - The acetic ether extracts were resolved by ether, R.F.

etc into 3 fractions A 29.55g mobile C₃ C₄ gums; B 6.65g gum; C 20.25g
B with H₂O Kne etc gave back 6.15g thick gum [27] $\frac{36}{100}$ = +4° 4; C with H₂O Kne etc gave

back 18.95g gum with (p = 3.79) [27] $\frac{36}{100}$ = +2° 64; obviously a fair amt of α and β d. galactose
metasaccharin still present (discard all).

(18) d. Galactose + PbO_2 Glycerol + 33% NaOH. 25g d. Galactose up to 155 + 57 1650 cc H₂O + 2g PbO_2 heated
B.W.B. for 8 hrs shaking time to time; and 1.04g oxalic acid thru still off + 60° 25 min got back 2.50g

Galactose containing 70% sugar as det by tit with Fehd. solution with [27] $\frac{36}{100}$ = +26° 33 (p = 5.0) d in 1 de hite + 10.
Repeat with 3X amount got total Galactose 97g which in vacuo H₂O was poured into a sol of 250g NaOH

in 413 cc H₂O time with 50 cc H₂O. Temp rose from +21° to +46°. Then least B.W.B. for 6 hrs and 1180g
HCl as in 17 etc. got 82.0g gum (ale extr). Ext to 2X with 12 ethyl acetate took out 41.0g gum, and

a third exto with 12 ethyl acetate left 39.5g gum (ale extr). Ext to 2X with 12 ethyl acetate took out 41.0g gum, and
Acetic ether exto was resolved into 17.68g mobile C₃ C₄ gums A; into 9.63g gum B [27] $\frac{36}{100}$ = -2° 33

and into 14.19g gum C [27] $\frac{36}{100}$ = -8° 88; obviously α and β d. Galactose metasaccharin
present in B & C. (discard all).

(19) d. Fructose + CaCO_3 : 10g d. Fructose - 90° 95 from Dublin, 400cc H₂O + 1g CaCO_3 heated B.W.B. for
13 hrs some got yellow; change finally was deep yellowish wh. got 0.64g insol CaCO_3 + titration of sugar

gave 84.7% sugar. after Kne etc got making up to 250 cc α in 1 de hite = -2° 12; [27] $\frac{36}{100}$ = -55° 8.
d. Fructose + BaCO_3 : 10g d. Fructose + 400cc H₂O + 1g BaCO_3 heated 6 2/4 hrs B.W.B.

where all BaCO_3 dissolved gave an acid reaction; deep red coloration; ppt Ba²⁺ by dry K₂Cr₂O₇ stage back
9.55g gum; made up to 250 cc & found 2 in 1 de hite = -10° 53; [27] $\frac{36}{100}$ = about -40°. Sugar 7% by tit

found to be only 71.5%. (discard). action with BaCO_3 much slower than with CaCO_3 !!
d. Glucose and CaCO_3 : 10g glucose CaF_2 but up to 144 = 6° 77g CaCO_3 + 400cc H₂O heated B.W.B.

for 15 hrs; [N.B. 57g BaCO_3 + 10g glucose + 400cc H₂O on B.W.B. for 9 1/4 hrs showed faint coloration only & acidly less
change.]. Noticeably less action than in case of saccharine that yellowish solution and

0.7g insol CaCO_3 removed by filtration.

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(9.7258...)
67-70% Sugar
(21) d. Mammone & Calcoz. 10g d. Mammone mpt 135-140° + 18 Calcoz
mpt 0.35 used Calcoz. spilt by Kne etc. made up to 250cc gave α in 10c tube = -0.16 ∴ [α]_D²⁰ =
-4.0 Titrated with Fehd. solution gave between 80 and 83% Sugar! [$\frac{1}{4}$ eq Calcoz up]

(23) Oxygen formation from hexoses. Took 1 g sugar in 10 cc H₂O & added 3.6 cc of a 10% solution of d. fructose & acetic acid. Oxygen formation begins after 1 hr. A.T. & after 20 hrs. A.T. volume parts ph. by 0 & 50% (wt) acetic acid. was completely solid. Noticeable after 17 hrs. A.T. 17 hrs. A.T. mass of white x x Ale: after 17 hrs

(24) d. Galalox + Pb₂Mn; 10g d. Galalox + 50cc H₂O + 1.0g
oxalic acid ste syt from filtrate [α]_D²⁰ = +19°; distill off final sugar 9.6 g again for 2 hrs
with 1.0g Pb₂Mn + 30cc H₂O then add 1.0g more Pb₂Mn + heat 6½ hrs BWTB + add 1g oxalic Kue etc
syt [α]_D²⁰ = +19°- ? constant
1 - + Pb₂Mn: 10g d. Fuelox + 50cc H₂O + 1.0g Pb₂Mn heated BWTB 5½ hrs + add 0.5g
fuelox + final sugar again for 2 hrs BWTB with 50 cc H₂O + 1.0g Pb₂Mn
Kue etc syt (p = 4.0)

(26) Took 20g d. Glucose, 8g, Calc + 100cc H₂O sheet R.T. for frame
 + phosphate buffer with K₂C₂O₄ + some to small volume supplied by alcohol gun 8.72g Ca salts (in 100cc)
 which gun by injection of 9.9% Ca :: that not 11.5g with K₂C₂O₄ supplied Ca by 3.58g
 oxalate 2H₂O and got 285g gums which completed by 3/4 hrs 100° with 24cc anhydrous gun 6.5g acrylate gums
 sol in ether and 475g used for (this card). Hydrolysis of the 6.5g acrylate gums with 20g Ba(OH)₂ 8H₂O &
 40cc H₂O gun 3.3g gum which K₂C₂O₄ the gun back 2.9g gum $\Delta T_D = +3.045$ made up to 100cc
 I found Δ in 100 tube = +0.10. Titration taking 0.29g, took 12.5cc in cold & total of 23cc hot
 theory for 6 baseline = 17.9cc :: obviously some splitting into
 C₃ C₄ acids, D.C.D.

- The aq. alc. filtrate from the 11.72g Ca salts gave 7.65g neutral sugars ~~which with~~ Knc etc. making up to 100cc gave $[d]_{D}^{20}$ in 10c tube = $+0.03$ $\therefore [d]_{D}^{20}$ = about $+0.8$ and
 given by ttr with Fehd. sol. between 50 and 54.6% Sugar (1g used) ~~Keep solution in 6.5g~~ ^{discarded}
- (27) d. Fructose & CaCO₃ ^{20g d. Fructose + 100g H₂O + 8g CaCO₃ heat R.F. for flame gauge}
 for 35½ hrs. Got by filter 3.43g ^{1.6g up} ~~and~~ CaCO₃. Filter with Knc etc slight spell" given by pphm with
 alc etc 11.06g Ca salts in vacuo crop I + 1.27g crop II $\therefore$ total of 12.33g; these gave 10.9%
 Ca in ignum etc $\therefore$ had not of salts 12.1g with Knc & pphm Ca by 4.15g of alc etc and
 got 7.0g gum which acylated with 2cc anhydrous H₂O gave 6.3g. then sol acylated
 gums and 4.35g used for (this card). Hydrolysis of the 6.3g acylated gums by 20g BuMn
 + 40g H₂O, hrs BuMn gave 3.3g gums (alc extr). This with Knc etc gave 3.0g gum which
 made up to 100cc gave $[d]_{D}^{20}$ in 10c tube = $+0.12$ $\therefore [d]_{D}^{20}$ = $+4.0$ + Saccharose present in
 case of 2 glucose & 2 fructose must be identical. Titration taking 0.3g, look up etc in
 cold and total of 23.83cc $\frac{1}{10}$ NaOH sol (Heavy for 6 lactone = 18.5cc) $\therefore$ C₃ C₄ gums present.
 The alc. filtrate from Ca salts gave 4.85g gums which with Knc etc made up to 100cc
 gave $[d]_{D}^{20}$ in 10c tube = $+0.06$ $\therefore [d]_{D}^{20}$ = about $+1.2$ Titration with Fehd solution.
 shown only between 43% & 47% Sugar ~~Keep solution in 6.5g~~ ^{discarded} ~~19.12~~
- (28) d. Galactose & CaCO₃ ^{20g d. Galactose + 8g CaCO₃, 100g H₂O heated for flame gauge}
 R.F. for 35½ hrs. Got 3.46g ^{1.6g up} ~~and~~ CaCO₃. Filter with Knc etc by pphm gave (in vacuo) a total
 of 14.5g Ca salts (in vacuo) and 10.1% Ca $\therefore$ had not of Ca salts 14.3g with Knc & 4.53g of alc etc and
 got 9.55g gum which acylated 7½ hrs H₂O with 30cc anhydrous H₂O & 52cc H₂O hrs BuMn gave 4.15g gum.
 used for (this card). Hydrolysis of the 8.1g with 20g BuMn & 40g H₂O, hrs BuMn gave 3.65g gums (alc extr).
 This with Knc gave 3.65g gum which made up to 100cc gave $[d]_{D}^{20}$ in 10c tube = -0.07 $\therefore [d]_{D}^{20}$ = -1.09
 obviously a mixture of 2 sp Galactose + 2 sp Saccharose. Titration taking 0.365g, look up etc
 in NaOH $\therefore$ obviously all C₃ C₄ C₅ Saccharose present.
 The aq. alc. filtrate from pphm Ca salts gave 5.0g neutral gums which with Knc etc making
 up to 100cc gave $[d]_{D}^{20}$ in 10c tube = $+0.33$ $\therefore [d]_{D}^{20}$ = $+6.6$ & gave between 37-43% Sugar as
 det. by ttr with Fehd. sol. ~~discarded~~
- (29) Formose & traces PbMn ^{at 70°} Took the 19.35g Formose gum II, see 14, 79.2% Sugar in 40g
 H₂O and add 0.1g PbMn heat 1 hr; add 0.1g (4th) heat 1 hr; add 0.1g PbMn heat 4 hrs; add 0.1g (6th) heat
 and 3rd 0.1g PbMn heat 3 hrs; add 0.1g (8th) 0.1g heat ½ hr and finally add 0.2g more PbMn heat
 2½ hrs & then 7th 0.1g heat 3 hrs. Filter off traces of unresol PbMn. only 0.05g. make up to 250
 11½ hrs longer - Total heating $\therefore$ 2.6½ hrs. Filter off traces of unresol PbMn. only 0.05g. make up to 250
 cc and found 70% Sugar by ttr with Fehd solution to be 67% (very little action)
- (30) Saccharose & traces BuMn 5.0g d. Fructose (for lincin), 10g H₂O + 0.25g BuMn & H₂O ($\frac{10}{175}$ g) & let
 stand O.F. for 4 days: only very faint coloration. Pour cold into 16cc n H₂SO₄ etc & make up to 100cc &
 found $[d]_{D}^{20}$ in 10c tube = -3.52 $\therefore [d]_{D}^{20}$ = -70.4 Incomplete action.

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- (31) d. Glucose + traces BaCl_2 . 5g d. glucose, 10g H_2O , + 0.25g BaCl_2 & H_2O ($\frac{10}{175}$ eq) + let stand O.T. for 5 days and then pour into 16cc n H_2SO_4 etc + get making up to 200 cc & in 1 dc tube = +1°79
 $\therefore [\alpha]_D^{20} = +35.98$ Incomplete action.
- (32) d. Galactose and traces PbO_2 . 20g d. galactose + 40 cc H_2O heated 60-70° for total of 18 hrs adding 0.1g PbO_2 as follows. 0.1g at first; 0.1g after 1 hr; 3rd after 2 hrs; 4th after 6 hrs; 5th after 16 hrs $\therefore \frac{1}{27}$ eq) $[\alpha]_D^{20} = +2.25$ i.e. $[\alpha]_D^{20} = +56.1$ very slow change
 Filler make up to 500 cc & found d in 1 dc tube = +2.25 i.e. $[\alpha]_D^{20} = +56.1$? sweetening? change to d. Talcum only?
 Titration by Fehd. solution gave 79% sugar only!!
- (33) d. Mannose + traces BaCl_2 . do as with No 31 d. glucose 5 days O.T. + get by H_2SO_4 making up to 100 cc & in 1 dc tube = +0.44 $\therefore [\alpha]_D^{20} = +8.8$ - incomplete action.
- (34) d. Mannonic Bismut rotation: Fork pump from Luerline + Fehd solution Feb. 8-'06
 kept 207° & found $[\alpha]_D^{20} = -26.73$ i.e. 0.9124g + 22.3512g H_2O gave d in 2 dc tube = -2°12 (OK)
- (35) d. Fructose and traces PbO_2 . 20g saccharose 40g H_2O heated 25 $\frac{1}{2}$ hrs from 60-70° adding PbO_2 as follows: 0.1g at first; after 5 $\frac{1}{2}$ hrs 0.1g; after 6 $\frac{1}{2}$ hrs 0.1g; after 11 $\frac{1}{2}$ hrs 0.1g; after 14 hrs 0.1g; after 17 $\frac{1}{2}$ hrs 0.5g $\therefore$ total of 1g PbO_2 ($= \frac{1}{13}$ eq). Filler off of much PbO_2 partly PbO yellow make up to 500 cc and found d in 1 dc tube was -1.095 i.e. $[\alpha]_D^{20} = -48.75$. Titration by Fehd. sol. gave between 80.1 and 82.7% sugar $\therefore$ much slower change than in case of d. galactose (32) Not enough to be so fast only!
- (36) d. Fructose + traces BaCl_2 . 20g saccharose in 40 cc H_2O ; add 0.1g BaCl_2 & shake; after 5 hrs add 0.1g more; after 3 hrs longer add 3 $\frac{1}{2}$ 0.1g; after 16 hrs add 0.1g (4th) more; after 3 hrs longer add 5 $\frac{1}{2}$ 0.1g; after 4 hrs add 0.1g. after 16 hrs add 7 $\frac{1}{2}$ 0.1g; after 7 hrs add 8 $\frac{1}{2}$ 0.1g; after 17 hrs add 0.2g more BaCl_2 & H_2O or total of 1.0g BaCl_2 & H_2O and then let stand for one week longer i.e. total of 79 hrs (3 days +). The BaCl_2 was put in solid gets yellow in H_2O but gives practically colorless solution when dissolved. Now treat with 6.3 cc n H_2SO_4 in cold etc make up to 500 cc (i.e. = 4.0) & found d in 1 dc tube = -3°08 lower $[\alpha]_D^{20} = -77^\circ$.
 Sugar Titration by Fehd. sol. gave between 87.5-90% sugar! very little change.
 $\therefore$ Distill off H_2O and dissolve in 40 cc H_2O add 2.0g BaCl_2 & H_2O at once; after 20 hrs add 1.0g more BaCl_2 & H_2O (i.e. in all 3.0g) and let stand 4 days longer or 5 days past in all. add to 19 cc n H_2SO_4 etc making up to 500 cc & found $[\alpha]_D^{20}$ in 1 dc tube = -0°84 $\therefore [\alpha]_D^{20} = -2.14$. Titration for sugar gave between 85-87.3%!! $\therefore$ must have a transformation of saccharose into 6 sugars of d. glucose series only!! but reaction still incomplete i.e. saccharose still in excess - ($\frac{1}{13}$ eq)
 Distill off about 19g, in 40g H_2O O.T. for 5 days with 3.1g BaCl_2 & H_2O + get H_2SO_4 etc making up to 500 cc & in 1 dc tube = -0°28 $\therefore [\alpha]_D^{20} = -7.37$ + Sugar Titration was now between 75 and 77% by Fehd. solution. do as with No 31 d. glucose
- (37) d. Glucose + $\frac{1}{13}$ eq BaCl_2 . 20g glucose, 40g H_2O , and add in cold 2.0g BaCl_2 & H_2O and after 24 hrs. 1.0g more + let stand O.T. for 4 days longer or 5 days in all: pour into 19 cc n H_2SO_4 etc making up to 500 cc and found d in 1 dc tube = +0.63 $\therefore [\alpha]_D^{20} = +15.75$. Sugar titr. was betw. 92.4 + 95.2%!!
 Distill off about sugar, 19.7g, with 3.1g BaCl_2 & H_2O 540 cc H_2O O.T. for 5 days; solution was strongly alkaline & on pouring into H_2SO_4 etc making up to 500 cc found d in 1 dc tube = +0.3 $[\alpha]_D^{20}$ i.e. +7°62; Sugar titration was between 83.17% and 84.7%.

Now distill off streat gum, 19.4 g, in 100 cc H₂O with 15 g Br₂ till dissolved in 1 hr then let stand o.t. for 3 days when draw air thro' solution till Br₂ excess off & then treat with 25 g PbCO₃ + shade heating finally on B.W.B.; filter off in cold (18.15 g PbBr₂ + PbCO₃) streat aq. filtr with AgNO₃ till Ag present or Br⁻ out then ppt Ag ppt by H₂O etc. Make up to 500 cc and found d in 10 cc tube = -0.33 ∴ gums is 1.73 or -8.5.

The 10 cc of solution decolorized slowly in cold 9 cc $\frac{n}{10}$ NaOH to orange pink for $\frac{1}{2}$ hr even at 80° with 11 cc $\frac{n}{10}$ NaOH ∴ can titrate for acid substances present.

Now treat rest of aq. solution at 60° for 11 hrs with 5.0 g powd CaCO₃ and found 2.62 g left insoluble. The aq. filtrate distilled off gave 21.5 g gums + Ca salts which dissolved in 40 cc H₂O and gave 97% spirit to extract gums 11.18 g xx salts (in vacuo) cont.

11.08 g CaCO₃ and giving $[\alpha]_D^{20} = +3.96$ ($\rho = 4.0$). Total wt of salt 11.003 g with 2.74 g oxalic acid & 9.0 g gum with $[\alpha]_D^{20} = +27.78$ ($C = 3.6$), found d in 10 cc tube = +1.0. Titration of 0.36 g took 17 cc $\frac{n}{10}$ NaOH sol (theory for C₆ lactone 20.2 cc) ∴ treat rest of gum with 15.4 g Bismarck and got large amt of a xx salt mpt 145-153° which was subsequently found to consist mainly of d. gluconic acid containing small amounts of d. Mannonic acid.

(38) d. Gluconic Cinculin; optical constants etc - made from Ca F K's d. gluconic acid & Cinculin. Had 12.1 g xx salt mpt 187°: it xx from spirit in transparent flat plates and comes out very completely; it also comes out in large amounts from 2 pts hot H₂O on cooling in hump. flat needles $[\alpha]_D^{20}$ of xx salt from spirit = +124.6 i.e.

1.0909 g + 34.9865 g H₂O ∴ $\rho = 3.024$ gum (d = 1.01) d in 2 cc tube = +7.61. Took 20 g dextrose, 40 g H₂O and add in all corked flask, 5.5 g CaH₂ in approx 0.6 g portions, for 9 days ∴ 9 x 0.6 g = 5.5 g total adding 0.6 g each day.

(39) d. Glucose and CaH₂. Took 20 g dextrose, 40 g H₂O and add in all corked flask, 5.5 g CaH₂ in approx 0.6 g portions, for 37 days longer after all 5.5 g CaH₂ had been added. Then add H₂O to total let stand corked o.t. for 37 days longer after all 5.5 g CaH₂ had been added. Then add H₂O to total volume 150 cc streat B.W.B. for 5 hrs and got 17.1 g gum. On degroting this dx RT with 350 cc ethyl acetate and had 8.5 g insol tarry gum left which was acetylated 8 hrs with 25 cc acetyl anhydride & got 13.65 g acetylated gums of which 8.0 g dissolved in ether leaving rest as usual brown tar. Hydrolysis 1x with 3 pts H₂O gave 5.38 g gum when with 10 g BaH₂ & 20 cc H₂O 7 hrs B.W.B. etc gave 4.13 g gum (alc extn) with $[\alpha]_D^{20} = +15$ i.e. found d in 10 cc tube = +0.62 ($C = 4.13$)

The gums soluble in ethyl acetate evapor on B.W.B. 7 hrs with H₂O + H₂SO₄ gum back 8.03 g stiff gum $[\alpha]_D^{20} = +23.9$ ($C = 4.9$), found d in 2 cc tube = +1.92. White gums ∴ 12.16 g & keep as follows: daily about 1 1/2 gums for 13 days then let stand corked for 33 days. Add H₂O to total volume about 150 cc then heat B.W.B. for 5 hours got 14.35 g gum. This gum contained obviously far more tar than compound expt No 39 with CaH₂ ∴ discarded.

(40) Dextrose and BaH₂. Took 20 g Dextrose, 40 g H₂O, add corked flask a total of 18 g BaH₂ & H₂O as follows: daily about 1 1/2 gums for 13 days then let stand corked for 33 days. Add H₂O to total volume about 150 cc then heat B.W.B. for 5 hours got 14.35 g gum. This gum contained obviously far more tar than compound expt No 39 with CaH₂ ∴ discarded.

entire amount of No 34: the aq. sol. filtr from the above 11.18 g Ca salts gum 8.0 g gum which dissolved except traces. Ca salts (discarded) and dist. alcohol: got finally 7.5 g neutral gums which $C = 3.9$ gum d in 10 cc tube = -0.97 $[\alpha]_D^{20} = -24.87$ Sugar when was 74.70 as det by Feld sol. ∴ much lactic acid present (cf. later).

contains milk sugar see No

(15)

(1 eq = 4.1 g)

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1/3 eq

(41) D. Galactose & CaO_2 . 20 g d. galactose, 40 g H₂O and 5.5 g total CaO_2 added in 0.6 g lots daily for 9 days corked flask: let stand 36 days longer at room temp, then add H₂O to Vol about 150 cc and heat B.W.B. 5 1/2 hrs then add 9.37 g oxalic acid & got 17.45 g gum: on digesting this 2 x R.F. with 450 cc ethylacetate had 12.75 g insol fatty gums which acylated 7 hrs B.W.B. with 37 cc anhydrid gum 22.45 g gums of which 13.45 g dissolved in hot ether (rest gums brown like discart) Hydrolysis 1 x with 3 pts H₂O gum 9.2 g gum still had 7 hrs B.W.B. with 20 g B.W.B. & H₂O 540 cc H₂O & got 7.15 g gum (also extr) with 1 d = -1.9 (c = 3.58) found d in 1 d tube = -0.07 (c = 3.58) The acetic ether extract on evap c H₂O on B.W.B. for 6 hrs then kne etc gum finally 3.85 g stiff gum tit₂ = -1.03 (c = 3.85) found d in 1 d tube = -0.04 (c = 3.85) white gums total = 11.0 g [keep] - obviously a mixture of 2 sps d. galactomista and d. galactose - Saccharin (strongly dextr) brown & d + 12.2 g d. by x mic factone cont No 103

(42) D. Galactose and Ba_2H_2 . Took 20 g d. galactose, 40 g H₂O, and add a total of 18 g Ba_2H_2 & H₂O in 13 days adding: about 1 1/2 g daily: let stand corked for 34 days longer of. add H₂O to 50 cc & heat B.W.B. for 5 1/2 hrs still H₂O & got 15.65 g gum - obviously containing far more fat than corresp. expt with CaO_2 (No 41) : discart.

(43) Maltose & CaO_2 : 20 g Maltose, 40 g H₂O and 5.5 g CaO_2 adding daily about 0.55 g taking: 10 days in all: let stand corked 36 days of. longer when add H₂O to volume 150 cc heat B.W.B. for 5 hours then add 9.37 g oxalic acid & got 16.25 g gum. An extr this with 500 cc resp 300 cc ethylacetate R.F. found 7.05 g insol tar (100% wum) which acylated 8 hrs 100' with 21 cc anhydrid gum 10.95 g gum of which 5.0 g acylated gums only dissolved in hot ether: The latter gum on 1st hydrol. with 3 pts H₂O 3.25 g gum then by 7 g B.W.B. 575 cc H₂O this B.W.B. & got 2.75 g gums tit₂ = +31.3 (c = 2.75) found d in 1 d tube = +0.86. The acetic ether extracts on evap in open dish B.W.B. with H₂O for 7 hrs gum kne etc 8.98 g gums gums tit₂ = +31.04 (c = 3.592) found d in 2 d tube = +2.23 containing much cont No 39

Titration: taking 10 cc: 0.3542 g took 4.7 cc $\frac{2}{10}$ NaOH in cold & total of 23.86 cc $\frac{2}{10}$ NaOH hot (theory C_6 last = 20.17; C_6 lactose = 24.3 cc) white both gums: total = 11.37 g & keep cont No 102

(44) Maltose and Ba_2H_2 . 20 g Maltose, 40 g H₂O and 18 g Ba_2H_2 adding about 1 1/2 g daily for 14 days. Then let stand of. corked for 32 days longer, then add H₂O to volume 150 cc heat B.W.B. 5 hrs add 804 cc & got 14.4 g gum: on digesting this 2 x with 500 cc ethylacetate still had 13.65 g insol tar (100% wum) which acylated 8 hrs B.W.B. with 35 cc anhydrid gum 20.4 g acylated gums which on extr with 1 d ether R.F. took out only 7.4 g acylated gums & these on hydrolysis (incl also gums & discart in ethyl acetate) with H₂O gum 5.7 g gum or after hyd₂ with 10 g B.W.B. & 20 g H₂O this B.W.B. got only a total of 5.0 g gums tit₂ = +3.5 (c = 4.6) 50 g d. galactose 100 g H₂O and 8 g Ba_2H_2 & H₂O flask (No 27 & April 14)

(45) Galactose & Ba_2H_2 . Pour into 57 cc $\frac{2}{10}$ NaOH on B.W.B. : very little acid gums present. stand corked of. in flask for 46 days. Sugar Titration, by Fehd. sol. found between 65 and 70% (used 0.42 g & in 2 d tube = +1.2 : tit₂ = +12. Titration, by Fehd. sol. found between 65 and 70% (used 0.42 g Titration $\frac{2}{10}$ NaOH, taking 20 cc: 1.08 gum): it took 6.2 cc in cold & total of 11.5 cc hot or 14.7 cc $\frac{2}{10}$ NaOH if we large excess of $\frac{2}{10}$ NaOH on B.W.B. : very little acid gums present. Treat rest of gum, 48.5 g, in H₂O solution (Vol 1.2 L) with 5.0 g KNO_3 at 60° for 6 hrs (to slurr!)

and got 3.07g insol CaCO_3 . The aq. filtr. distilled off at 50° was found to have two little Ca salts present to get out by pptn with ~~alc + H₂O~~ + alcohol etc. i. braked all in H₂O solution with 25g Bromine o.f. for 7 days: draws air thru' solution & treat with 42g PbCO_3 & then with Ag_2CO_3 , ~~H₂O~~ etc. & make up gum to 1 L and found d in 2 dextube = -0.25. [d]_D^{20} = about -2.05. Now digest gum in H₂O solution for 10 hrs at 60° with 15.0g powd. CaCO_3 and got 11.45g insol CaCO_3 : aq. filtr. dist. off & dissolved in 50cc H₂O & add 100cc spirit 3X gum; finally 9.32g pptd Ca salts (in vacuo) I; then 9.53g pptd Ca salts II in vacuo;

and then 3.57g pptd Ca salts III in vacuo. The aq. alcoholic filtrates gum ~~26.6g neutral~~ ^{26.6g neutral} which now give [d]_D^{20} = +2.25 and contained as det by titr with Fehd solution 5.8% sugar. This gum ~~26.3g~~ ^{26.3g} standing in alcohol & H₂O & 5cc 50% acetic got a

Seedling gum 135g d. l. euglycose l. sorbose x x l l l up to 153 (on leaves) $\rightarrow$ about 22g. Took 10 of the 26.3g gum: 2.63g in 10cc H₂O tried to it in cold a fresh solution of 5cc ph. hydr in 5cc 50% acetic got a separation at o.f. after 17 hrs of ozon. After 1 week got 183g ozon up to 160-170° well washed with H₂O & then in vacuo. On digesting this with 50cc abs. alc. practically all dissolved but on standing in cold got a separation of 0.1g ? d. galactosazone up to 193°; the alc. sol. ozon 1.73g, was now digested with 50cc C_2H_5 got 14g wood.

fine looking ozon up to 160-170° - obviously? a mixture of d. l. euglycose & l. sorbose ozon. A further exam of this showed it (14g) remained dissolved in 50cc abs. alcohol even on adding 50cc C_2H_5 & let stand: distilled off 1/2 of this solution in B₂W when got 0.42g x x l l l up to 180-190° easily soluble in hot abs. alc. & diff sol in hot H₂O.

The pptd Ca salts I, II, III united = 22.44g; analysis gave 76.35% Ca : obviously sugar present (1); [d]_D^{20} = +12.35. (acc. det.) $p = 4.0$. Rest of salt 22.22g with 5.34g oxalic acid gave 17.3g gum [d]_D^{20} = -5.18 ($c = 3.46$). Titrating this taking 0.311g

gave heavy lump conc. gelled plates up to 130° and dry slowly in vacuo then melted at 138° & solid up to 142° [d]_D^{20} = -20.54 (acc. det. $p = 4.0$). The rest of the acid or salt 6.64g with 3.0g B₂W gave 4.0g B₂W and 2.1g gum (acc. det.) [d]_D^{20} = -44.27 $p = 4.0$: this with 1.5cc H₂O, 2cc ph. hydr & 2cc alcohol gave 2.06g x x pearly plates

up to 200° obviously d. galactonic phenylhydrazine (acc. det.) [d]_D^{20} = -19.04 (acc. det.) they gave with 50% B₂W 7.42g B₂W & 3.85g gum (acc. det.) [d]_D^{20} = -44.27 $p = 4.0$: another way also present. The orig. alc. filtrate from crop I salts gave a 2nd crop of low melting 100-110° salts which were united with the 3.8g just mentioned & these dried in vacuo amounted to 12.48g (acc. det.) up to 100-140° [d]_D^{20} = -19.04 (acc. det.) they gave with 50% B₂W 7.42g B₂W & 3.85g gum (acc. det.) [d]_D^{20} = -44.27 $p = 4.0$: another way also present. The orig. alc. filtrate from crop I salts gave a 2nd crop of

which with 3cc H₂O & 4cc alcohol & 3.5cc ph. hydr o.f. 28 hrs seedling gum only 1.58g made by digest up to 180-185°: obviously much of another acid (?) which is also present with the d. galactonic acid (acc. det. all). (?) [d]_D^{20} = -44.27 $p = 4.0$: another way also present. The orig. alc. filtrate from crop I salts gave a 2nd crop of

(46) $\text{Ca}_2\text{F}_2\text{K}_5$ lactic acid contains 22.4% excess l. lactic acid. Took 3.053g $\text{Ca}_2\text{F}_2\text{K}_5$ acid 1.21, made up to 250cc with 25cc: 0.3053g and found it with 25cc $\frac{1}{10}$ NaOH sol & total of 29.36cc $\frac{1}{10}$ NaOH sol: 8.67g lactic acid. 100cc of the solution + 117.44cc $\frac{1}{10}$ NaOH etc. heated must give 1.31488g Na lactate: make up to with 31.55g H₂O

(47) Lactulose, Br₂, H₂O, & CaCO_3 : whereas d. fructose is practically not affected by Br₂ & H₂O, it is rapidly changed by having also CaCO_3 present. 20g d. fructose, 100g H₂O & 20g Br₂ + 13g CaCO_3 in a flask with capillary tube gums of rapidly CO_2 & Br₂ all gone after 24 hrs but solution still colored deep brown after 6 days o.f. Filter & got 1.57g wood CaCO_3 . Draw air thru' aq. filtrate till it is free of Br₂ & then treat

(7) Can d. galactose $\frac{1}{6}$ eq BuM₂ be oxidized at 7 days by air (?) to ~~lactic~~ lactic acid by xonic
 air? N.B. (17) No

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with 47.5g Pb acetate solution & filler of the wood PbBr₂ former in cold; the eq.
 filtrate was then treated with Ag acetate (about 15g) light red of Br⁻ Pppt with H₂O then with
 13.5g oxalic acid & got 60° vacuo 15.65g gum (alc ext) with $[d]_D^{20} = -33.2$ (C = 3.13).
Tubulin looking 0.939g, took 2.2 cc $\frac{1}{10}$ N NaOH in cold & total of 39 cc liq. i. heat rest of gum
 144g for 5 hrs at 60° with 4.0g powder CaCO₃ & got 3.2g insol CaCO₃ and 1.0g CaCO₄ & 1/2 H₂O by adding
 the neutral ag. filtrate distilled off in vacuo the gum 9.35g neutral gum, $[d]_D^{20} = -44.9$, and
 3.41g pppt Ca salts (in vacuo). Sugar Tubulin of 9.35g gum was between 50 and 54% by Fehling.
 + 1/2 of gum, 1.0g, treated with 2 cc ph. hyd & 2 cc 50% acetic acid at. began to give osazone
 formation only after 4 hrs after 26 gums at. got 0.3g osazone mpt 200-202°.
Analysis of the 3.41g Ca salts gum 10.5% Ca i. heat rest with 1.065g oxalic & got 2.3g brown
 gum (alc ext) which treated with 6.6g Bismuth gum 9.1xx salts but did not work up further (is can)
 Took rest of neutral gum, about 8.0g, in about 40-50 cc H₂O and 4.0g xx BuM₂ & H₂O
 at ord. lamp & let stand at. for 17 hrs then pour into H₂O & got back 7.45g gum (alc ext)
 with $[d]_D^{20} = -41.9$ i. very little change.
Tubulin taking 0.296g, took 2.2 cc $\frac{1}{10}$ N NaOH & 7.15 cc $\frac{1}{10}$ N NaOH liq. i. distill off & heat gum
 7.1g again with 7.0g BuM₂ & H₂O and 70 cc H₂O above temp for 23 hrs pour into H₂O & got back
 6.1g gum with $[d]_D^{20} = -2.46$ i. much change. Now heat gum at 60° with 20g powder CaCO₃
 for 7 1/3 hrs & got 13.3g wood CaCO₃ & some conc ag. filler K₂CO₃ etc got pppt 1.73g Ca salts
 containing 10.62% Ca discarded at this point as could not rotate Ca salt or the 0.95g gum
 obtained from it by 0.436g oxalic etc.

(48) Dextrose 1/6 eq BuM₂ and air. 20g dextrose, 40g H₂O and 3.0g BuM₂ & H₂O (1/2 eq) shown CO₂ free air
 this solution in cylinder with star fish appar. day and night at. for 71 hrs (3 days). Add to 19 cc N H₂O
 etc & got making gum up to 50cc (C = 4.0) & in 100 tube = +10.28 i. $[d]_D^{20} = +32.9$. The amount of sugar present
 as det. by titr with Fehling solution was 9.5% i. i. practically no action!! 17 cc more sugar present
 Titration taking 2.0g or so of solution took only 4.2 cc $\frac{1}{10}$ N NaOH in cold & 9 cc $\frac{1}{10}$ N NaOH in hot. disc card

(49) d Fructose 1/6 eq BuM₂ + air: 20g laevulose, 40g H₂O + 3.0g BuM₂ & H₂O + same CO₂ free air then
 solution in star fish cylinder for 70 hrs etc gum by making up to 50cc etc between 85-90% sugar
 and tit in 20 cc tube = -4.5 i. $[d]_D^{20} = -56.25$. Practically no oxidation!! disc card

(50) Oxid. of alk. sol. of Dextrose by air. 50g dextrose, 100cc H₂O, and 15g BuM₂ & H₂O (1/2 eq) shown CO₂
 present this sol in star fish app for 24 hrs when add 7.5g BuM₂ & H₂O + continue air adding 7.5g more BuM₂
 every 24 hrs for 7 days until total of 67.5g BuM₂ was in, when draw air then sol. for 2 days 48 hrs
 larger open point into 21g H₂O dil etc etc got on distilling in vacuo (3 sweeps over c H₂O) a total
 ag. dist. of 2 L. : 80cc of this (1/2) took 143cc $\frac{1}{10}$ N NaOH + 142.5 cc $\frac{1}{10}$ N NaOH i. have a total of only 5.24g
 H₂O present. Take up resid. gum 45g; in alcohol & got 43.53g sweet gum $[d]_D^{20} = +12.86$
 & mixed up to 1 L. & found 2 cc $\frac{1}{10}$ N NaOH liq. = +0.56: Titration taking 0.435g, took 10cc in cold &
 Titration with Fehling sol. still showed 10 to 20% sugar present!!
 i. heat gum 42.5g with 6.6g BuM₂ & H₂O & 300cc H₂O

and draw Cor free air thro starfish appar heating in bath at 60° for 1 hr and at 70° for 4 hours & then pour into H₂O and get by digesting with gum R.F. with 250 cc ethyl acetate a residue into 20.45g uncol gum and into 15.75g soluble gums (mobile in cold)

The 15.75g lot, made up to 500 cc, gum & in 10 cc tube = +0.17; [27] = +5.04 and 10 cc 1.5315 g
look 28.4 cc $\frac{1}{10}$ NaOH test (12 cc in cold) (theory for C₃ gum = 29.7 cc).
The 20.45g lot, made up to 500 cc, gum & in 10 cc tube = +0.63; [27] = +15.24 and 10 cc 0.409g
look 25.27 cc $\frac{1}{10}$ NaOH test (8.5 cc cold) (theory for C₆ lactone = 23.0 cc). On heating out of aq. sol with C₆ lactone for 17 hrs it took up 27.65 alkaliol gum 47.5g salts-soluble in alc. test; can't have any d. glucose acid present. E.E. 2, but must consist of ~~arabine~~ d. arabine acid and d. lythmie + d. thurmic acids. Set free gum by 25.0g BaH₂ got 26.7g emulsion and 18.9g gum. One gram of this gum heated with 1 cc ph. hydr + 2 cc alc. test gum no xx ph. hydrogen

One gram of this gum heated with 1 cc ph. hydr + 2 cc alc. test gum no xx ph. hydrogen
came on adding other etc. discard and turn over rest of gums to Glattfeld.
(51) dl lacte + l. excess + 7.804. Took 10g CaH₂ lacte 1.21, 86.7% + 22.4% l. excess
+ 3.0g 7.804, 74.8 + 500 cc H₂O & heated R.F. for 8 hrs. Sideshell of H₂O in vacuo 50° & taking up residue in ether got 2.07g uncol 7.804. Other fullale gum gum which heated 8½ hrs BWRB at 15 mm gave 7.42g lactyl lacte acid [27] = +5.93 i.e. made up to 200 cc of form d in 10 cc tube = +0.22. No action

(52) dl lacte acid + 7.804. Took 10g CaH₂ lacte acid + 500 cc H₂O & heated 3½ hrs. Heat open on 54.6 g H₂O & sideshell of fine vacuo, dissolve in 100 cc H₂O & nurse. Heat open on BWRB for 1½ hrs

add to this heated on BWRB a hot solution of 12g BaH₂ & 100 cc H₂O & sideshell of fine vacuo, dissolve in BWRB in a ½ l flask for 6 hrs. Then add 54.6 g H₂O & sideshell of fine vacuo, dissolve in BWRB for 1½ hrs
ether & got 2.55g uncol 7.804. Other fullale gum gum which heated 8½ hrs BWRB at 15 mm gave 7.42g lactyl lacte acid [27] = +5.93 i.e. made up to 200 cc of form d in 10 cc tube = +0.31 no action
and got 5.1g lactyl lacte acid [27] = +6.07 i.e. made up to 200 cc of form d in 10 cc tube = +0.31 no action

(53) d. glucose and alkaline H₂O. Took 25g d. glucose in 810 cc H₂O (3% about) and added a sol of 25.33g 98.7% NaOH, 4.5 mols, in 625 cc H₂O in cold. Sideshell of fine vacuo, dissolve in BWRB for 1½ hrs
and let stand 48 hrs longer. Test shows much H₂O but no sugar. Heat BWRB till H₂O gone (and 147g H₂O (theory 146g) etc and got 8.65g gum non volatile (alc. ex. H₂O). The aq. distillates free of HCl, and a total of 24.62g (i.e. 6.28g) H₂O. Then ext. the 8.65g uncol. gum with 500 cc 400 cc ethyl acetate leaving 2.55g uncol gum + H₂O which acetylated 6 hrs BWRB with 8 cc acetyl anhydride & got 3.7g acetylated gums + H₂O which 2.3g dissolved in ether & other vials on 2x hydrolysis H₂O gave 1.15g gum

R.F. 6.1g dissolved by diff. leaving 2.55g uncol gum + H₂O which acetylated 6 hrs BWRB with 8 cc acetyl anhydride & got 3.7g acetylated gums + H₂O which 2.3g dissolved in ether & other vials on 2x hydrolysis H₂O gave 1.15g gum
3.7g acetylated gums + H₂O which 2.3g dissolved in ether & other vials on 2x hydrolysis H₂O gave 1.15g gum
3.88 salts which xx from 100 cc got alc. test which 2.3g dissolved in ether & other vials on 2x hydrolysis H₂O gave 1.15g gum
(discard) and 1.8g gummy salts which 2.3g dissolved in ether & other vials on 2x hydrolysis H₂O gave 1.15g gum
gum d in 10 cc tube = +1.24 [27] = +36.8. Titration taking 0.25g, ag. sol with 14.6g Burein got 23.15g xx salts
i.e. C₂C₃ by C₆ gum present as proved later, i.e. heated with 2.5g Burein & 0.6g gum with xx salt of Burein
which xx from 500 cc 98% spirit, seeding gum, large amount of salts both before & after drying in vacuo was 167-170
lost 5.6% xx water (0.65g loss) in vacuo except drift of salts both before & after drying in vacuo was 167-170
then higher [27] = -26.33 i.e. 1.0555g + 25.3245g H₂O gum d in 20 cc tube = -2.13. The salt gum with
8.0g BaH₂ & 100 cc H₂O (8 hrs BWRB) got 2.42g Ca salt (keep) & 3.5g d. arabine lactone; could not get sol; arrested
into Ca salt (8 hrs BWRB) got 2.42g Ca salt (keep) & 3.5g d. arabine lactone; could not get sol; arrested

The alc. salts from crop I salts gum 8.1g gummy Burein salts which xx from 25 cc alc. etc. noted by gum 1.58g crop II xx needles

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mpt 200-205° then got 0.15 g crop III salt mpt 150-167°. Meale. filtrate then gave 6.45 g
 gummy Buein salts which with 2.0 g BaH_2 etc gave 3.87 g Buein and 1.17 g gum xx sublimate
 which was therefore counted with the 0.6 g with xx salt on p 18. These were now resolved by acetate
 into 1.2 g gum easily soluble in cold ethyl acetate and into 0.6 g gum diff soluble therein in cold.
 To get rid of glycolic acid in 1.2 g lot (see No 57) it was emp BVB with H_2O for 14 hrs and got back
 0.65 g gum (with xx salt.) with $[\alpha]_D^{20} = +27.95$ i.e. found d in 100 tube ($p = 4.0$) = +1%. $\therefore$ total gum with 0.7 cc ph hydr
 + 2 cc alcohol O.T. for 42 hrs and got 0.29 g ~~xxxxxx~~ d. anionic ph. hydr mpt 200-205°: this proves decisively
 that no ~~anionic~~ d. ribonic acid can be formed in expt Q.E.D. (this case).
 The 0.6 g gum treated similarly with 0.6 cc ph hydr and 2 cc alcohol O.T. for 18 hrs the gum 0.37 g ph hydr
 mpt 180-195° which dissolved in 100 cc alcohol upon cooling 0.18 g xx plates mpt 200-205° from xx these from 3 cc
 of H_2O got 0.19 g xx mpt 214 in flat plates. It is $\therefore$ obvious that main reaction product here is
 d. galactose + alk H_2O_2 . 25 g d. galactose in 810 cc H_2O (about 3%) + add a solution of 25.33 g 98.7% NaOH
 in 65 cc H_2O (4.5 mols); noticed after 4 1/2 standing on table that temp there was +30°: obviously a
 reaction with heat evol. had taken place - H_2O_2 however still present; after 42.5 hours standing added
 140 cc more NaOH (1 mol) that raised 48 hrs O.T. Strong H_2O_2 test but no sugar: leached BVB then H_2O_2 gone
 and add 147 g HCl etc and got 6.99 g gum (ale extr) and found by titr that total amt of H_2O_2 in
 aq. distillates was 25.71 g i.e. 6.56%. The 6.99 g non vol. gum. extr mpt with 500 and 400 cc ethyl acetate
 left 3.2 g lavy gum + NaCl which acylated blue BVB with vocally and gave 4.8 g gums of which 2.88 g
 acylates gums dissolved in ether where on 2 x hyd rolysis gave 1.42 g gum $[\alpha]_D^{20} = +30$ (+1.2 wide tube, $p = 4.0$)
 $\therefore$ treated with 3.1 g Buein got 4.58 salts which gave no xx salts from alcohol.
 The restie other xth of non vol. gums was 3.66 g with $[\alpha]_D^{20} = +16.94$ ($c = 3.66$) d wide tube = +0.62.
 Titration taking 0.1839, took 2 cc in cold and 13.6 cc $\frac{1}{10}$ NaOH lot: that rest with 10.1 g Buein got 14.35 g xx
 salts which xx from 2 cc H_2O and 20 cc alcohol gave, on seeding with Lucas' body, 4.02 g dextro album xx NaOH
 of H_2O d. by xonic Buein (in vacuum) mpt 170-180°; these xx from 4 cc H_2O + 40 cc alcohol gave 1.65 g transo flat
 bulky prisms, mpt 170-175°; these lost 0.15 g xx H_2O (9.09%) on drying in vacuum over H_2SO_4 mpt 175° and
 gave $[\alpha]_D^{20} = -27.1$ i.e. 1.2713 g salt + 30.5337 g H_2O gum d in 100 tube = -1.12.
 Meale. filtr. from crop I salts gave 8.5 g salts which dissolved in 30 cc absol. alcohol gave 3.08 g xx crop II
 mpt 200-208° obviously by Buein salts $\therefore$ discard.
 Took the 4.02 g ~~xxxxxx~~ d. by xonic Buein crop I above got with 1.3 g BaH_2 etc 2.65 g Buein and 0.95 g
 gum $[\alpha]_D^{20} = +42$ ($p = 4.0$) i.e. distill off and treat it with 1 cc ph hydr + 2 cc alcohol and got 0.82 g crude
 hydrazid or by xx from almost 0.5 g x salt mpt 150-162°: obviously
 Meale. filtrate from crop II cont: about 5.6 g salts was now mixed with 4.5 g salts above got with 3.4 g
 Buein etc 5.9 g Buein and 0.50 g gum (ale extr) $[\alpha]_D^{20} = +26.8$ this gave no diff sol. divin salt [forster of
 was obviously lead HCl divin present). evap. of the 1.55 g gum on BVB with H_2O for 14 hrs gave back
 1.2 g gum (discarded).

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(58) l. Xylose + alk H₂O₂. Took 810 cc H₂O₂ (8% about) and dissolve 25 g l. Xylose in it and add a total of 25.33 g 98.7% NaOH in 625 cc H₂O as follows; 9.40 am 1st 140 cc + 15°; at 9.30 + was +20°; cool to 15° and at 10.55 am + was +17°; cool to 15° add 2nd lot of 140 cc NaOH steep lamp between +15 + 20°; at 1 pm add 3rd lot of 140 cc NaOH and at 3.10 pm rest. Finally, added at 5 pm added new lot of 140 cc NaOH solution. The next morning at 8 am all H₂O₂ gone; add 150 cc more H₂O₂ that same for 3 days or when sugar all gone + strong H₂O₂ test: add more H₂O₂ from sheet BWS 2 hrs to destroy it, add 147 g HCl (thru 146 g) etc got in aq. dist a total of 30.298 g H₂O₂ or 79.1%. got 5.8 g gum (ale xtr) or by xtr with ethyl acetate (400 cc) got 3.47 g gum [α]_D²⁰ = -3° 17' (c = 3.47) Titration taking 0.1735 g, took 6.4 cc cold total of 15.79 cc $\frac{N}{10}$ NaOH (hot), i.e. below C₃ & C₄, i.e. that part of gum with 11.8 g Bismarck + got 15.9 g xx salts which dissolved with 50 cc abs. alcohol left large amount insol. minor heavy salt + outstanding got 7.52 g (ale xtr) up to 211°; C₄ salts in large amount; these gum with 3.0 g Bismarck 5.57 g Bismarck and 1.6 g gum (ale xtr) [α]_D²⁰ = -13°; d. glyceric present in as d. erythronic and l. thionine acids. The ale filtrate from on p I gave 6.8 g greenish salts which gave no more xx l. white (see below). The residual gum left insol in ethyl acetate was freed from NaCl by xtr with su. acct. of abs. alcohol and gave this 0.7 g clear gum [α]_D²⁰ (p = 4.0) = +12° 5', obviously d. lyxuric leucine present; on evaporating by 1.9 g Bismarck into Bismarck salts got 0.43 g gum (ale xtr) [α]_D²⁰ = +3° 5' products; obviously contain C₃ acids and got 5.65 g Bismarck + 0.8 g (ale xtr) [α]_D²⁰ = +20° brands C₃, C₃, C₄ gums discarded.

(59) l. Arabinose + alk H₂O₂. Took 810 cc H₂O₂ about 3% & dissolve in it 25 g l. arabinose; then dissolve 25.33 g NaOH 98.7% in 625 cc H₂O + add solution as follows to H₂O₂ - 8.55 am 1st 140 cc + 13°; at 9.40 lamp was +19°; cool to +14°; lamp at 10.50 was +18°; cool to 14° add 2nd lot of 140 cc NaOH steep lamp was +15° + 20°; at 1 pm add 3rd lot of NaOH and at 3 pm rest; finally at 5 pm added 140 cc of new NaOH solution. Next day 8 am H₂O₂ all gone; add 150 cc more H₂O₂ that stand 3 days when H₂O₂ + sugar still present; add 50 cc more H₂O₂ sheet BWS for 2 hrs till no sugar or H₂O₂; add 147 g HCl (thru 146 g) - Found total of 30.99 g H₂O₂ (80.94%) in aq. distillates. Ale xtr gum 5.1 g dark gum which xtr by 400 cc ethyl acetate gum 2.75 g gum [α]_D²⁰ = +11° 52' (c = 2.75) Titration taking 5 cc (or 0.1375 g) took 4.8 cc in cold + total of 12.24 cc $\frac{N}{10}$ NaOH (hot); that part of gum with 9.15 g Bismarck + got 12.1 g xx salts which degraded with 50 cc abs. alcohol left all xx powder got 3.85 salts on p I, up to 207°; these gum with 2.0 g Bismarck 2.6 g Bismarck and 0.82 g gum [α]_D²⁰ = +22°; treated with 0.9 g ph. hydr + 2 cc abs. alcohol and got white other, a semi solid mass; now add 60 cc acetic ether and got 0.3 g ph. hydr up to 155-160° - the xx this from alcohol got a 1/2 hr of rhombic plates up to 210° obviously l. arabinose The active ether filtrate 60 cc distilled off in vacuum gave 0.83 g brown gum which melted in cold with 5 cc acetic ether by scratching at 0.23 g ph. hydr up to 110-120° on xx this from su. acct. hot ethyl acetate got 0.12 g l. erythronic gum & in 1 d cube = -0.12. (Keep prep) The residue left after xtr p. acetic ether was freed from NaCl by xtr with su. acct. abs. alcohol and then got back 0.68 g dark gum [α]_D²⁰ (p = 4.0) = +15° 5'; treatment of this with 1.8 g Bismarck gave nothing definite; mixed with ale. filtr from on p I salts above + got with 3.4 g Bismarck 6.45 g Bismarck and 1.23 g gum (acetic ether xtr) with [α]_D²⁰ = +3° and 0.53 g gum (ale xtr) with [α]_D²⁰ = +14°. The 1.23 g lot crop on BWS for 14 hrs with H₂O went down to 0.99 g xx salt which melted with 2.7 g Chinin de gave 0.4 g diff. sol in H₂O Chinin salt up to 140-170° on xx this from su. acct. with other etc got 0.16 g crude salt up to 160-185°; obviously l. glyceric Chinin D.E.D. (discard)

gave $\alpha/\beta = 1.01$
 II 1055 g salt + 25.4035 g H₂O gave α in 100% water
 (61) Got the α/β of Ca d. gluconate dried in vacuum & made from Ca F. K's prep gum + cal₃
 etc - as + 8.78 whereas a prep of Ca F. K's Ca salt dried in vacuum gave +10.63 m.e. des
 in both cases & lost Ca salt analyzed for Ca O.K. - Different results obviously due to
 errors in making polariscope -
 Ca d. gluconic gum, made from c.p. anhydrous Ca d. gluconate 10.02 g +
 + 1 at 60° only; this gave with 20.48 H₂O: $\beta = 4.0$

[illegible]

(8) Took 4.56g d. glaucous + 5.58g d. ...
which dissolved in 3cc H₂O + we also had slowly pour down ...
on day 22 had mpt 100-105 degt 120°; then lost 0.44g xx H₂O ...
then melted at 110° + degt 130° [Keep prep]
Oct 26 '06 from l. anal. Br + H₂O ...
d = 1.002 dlt

(65°) Constants of d. lyonic acid: $[\alpha]_D^{20} = +14.94$.
 60cc abs. alcohol + 0.3 g reph. 163° with $[\alpha]_D^{20} = -11.2$.
 0.3103 g subst + 7.4985 g H₂O ($\rho = 3.98$) spread d in 100 tube = -0.45.
 lyonic Bismut, which were separately xx from 1 pt H₂O to pts alcohol and
 0.29 xx H₂O in vials + then melted at 171°.

II 2.25g 2nd lot xx clear dry
 (66) Constants of l. xyloic acid: had 2 lots of Bunsen salts one much heavier than
 xx the other from 1st H₂O & 5 pts alcohol & got dark xx platten heavier than
 Anderson 3.7g. I Anderson gave 2.92g xx Teflon air dry which lost 0.31g xx H₂O in vacuo
 d. lxyloic Bunsen. I Anderson gave (27th) $\alpha_D = -18.0^\circ$ d.s. 0.7067g salt + 16.8761g H₂O gave
 & there had reprecip 176° and gave $[\alpha]_D = -0.76$.

(67) d. Mannonic Chirin; took 1.39 g d. Mannonic lactone up to 14.2. 1906 found. Mannon + Borste) found $[\alpha]_D^{20} = +41.93$; digested it with 2.53 g Chirin + got a xx salt very sol. in cold H₂O but xx from m. and alcohol in porous xx needles mp 165° $[\alpha]_D^{20} = -4.14$.
 2.58 g $[\alpha]_D^{20} = -102.1$ is. 1.3902 g salt + 3.3, 2.377 g H₂O gave d in 18c tube = -4.14 .
 167 g Chirin + 0.8 g gum (ale extr) $[\alpha]_D^{20} = +78.0$ (p = 4.0).
 This salt gave with 1.5 g BuM₂ etc 167 g d. Mannonic lactone 11 (mixed ester)
 i must have 2 forms of d. Mannonic lactone 11 (mixed ester)
 BuM₂ etc xx from 5/5 cold H₂O slowly increasing in porous
 = 720 - +50° 93, see March

MB. J. Mammalis Buceina xx from 5 ft cold Has shell
xx Nascher in large antea
J. Mammalis leatone npl 14F-153° [27° = +50° 93, see March
I. Thales it with 4.18 Buceina 5 ft by xx from 5 ft
4.18 Buceina and

~~N.S.~~
 XX Nadeln in large amounts
 Took also 185g d. Mannonic lactone mpt 144° - 150°
 18th 1907, from d. Mannone + Feh. sol. Strates it with 4.1g Borneol 144° - 150°
 & known Nadeln - Set fore gum by 2.0g Borneol 144° - 150°
 " 173° (b = 4.0°) = +68° 1/2 " Now deribill off Hro & heat gum with
 " 173° (b = 4.0°) = +53° 81 (acc det) (b = 4.0°)
 " 173° (b = 4.0°) = +77° 2 acc det

18th 1907, from 2.11.1907
 And large amount from Nadelst. - let you
 1.8 g gum (file x tr) with $\alpha 7^w$ ($\beta = 4.0$) = +68.51 (acc det) ($\beta = 4.0$) det
 also test with 0.55 g xx lactone, cup II, up 145-150° with $\alpha 7^w$ = +53.81 (acc det) ($\beta = 4.0$) det
 then got 0.62 g xx lactone, cup II, up 125-130° with $\alpha 7^w$ = +77.2 acc det
 i. ~~Formed~~ of ~~Mannonic~~ lactone.
 N.B. $\alpha 7^w$ found in 1st case in 12c tube was +2.6 after 28 hrs standing O.T. this went down to
 sol. faintly acc →
 in 12c tube was +3.1 after 28 hrs standing O.T. this went down to
 Mannonic lactone changes quickly

(12) found in 2nd case in 100 " " one from N.B. 3.
 + 1965 sol. was strong by acid table
 with H₂O at. + the other table
 2 stones of J. Mammone conf. Took 50g J. Mammone, 50cc H₂O + 57.75g Brn slt
 + 10g PbO₂ ate Ag₂O H₂O + 40g ag. filtr (moist)
 + 10g PbO₂ ate Ag₂O H₂O + 40g ag. filtr (moist)

1273 found in 2nd case in 100% one from
 + 1065 sol. was strong by acid table N.B. 3.
 with H₂O at 100% + the other one table
 (68) The reactions of J. Mannure's salt. Took 50g J. Mannure, 25cc H₂O + 57.75g Brn slt
 stand it for 7 days: then compare this sol + ppt with 85g PbO₃ etc Ag₂O₃ H₂O + found ag. filtr (moder)
 still gave decided test for sugar: incomplete oxidation: Now digest ag. filtr for 8 hrs at 60° with 20g H₂O
 CaO₃ found 13.7g left used: lost again 8 hrs at 60° + used only 9.1g used CaO₃ left: 3rd leaving 8 hrs to
 gave back 8.17g used CaO₃: 11.83g lost theory of crepeate exd = 13.9g). Distill off the from
 ag. filtr in vacuum, dissolve gum in an. acid H₂O + ppt Ca salts by alcohol + 50.8g Ca salts dried in vacuum
 say. alc. filtr gave 14.2g gum which dissolved in H₂O + alcohol gave 58 gm gum x x Ca salts: total of 56.6g
 salts which x x from 70 cc H₂O gave 45.5 gm x x treated of J. Mannure Ca, 2H₂O (in vacuo) 1273 = -70.52
 the ag. alc. filtr gave back 8.0g gum, 1273 (p = 4.0) + 8.0, 75: much for J. Mannure present: discard after
 proving gum strongly induces fruit salt.
 continuation see No 91
 Took 60g of CaF₂ K's x x d. glycinate of Ca (see (+10%3-

salts which xx fine
 the a.c. filter green back 80% green, 10% yellow
 moving green slowly returns total green.

continuation see No 10

(+10% 3-

(69) The 2 laetines of d. glaucous acid: Took 60g of 0.27 K's xx d. glaucous acid of Ca (see
 may 10 1911) that with 16.8g oxalic acid (Heavy 12018) in H₂O sol 1/2 g finally (le x tr)
 46.68g d. glaucous green (Heavy 50g) which is standing in absol. alcohol leaving discarded
 amount of glaucous green left with mol. Ca salts filtered off: after 3 months got on scratching a
 operation of xxll and after 6 weeks more filtered off 10.25g xxll next 145-150° of d. laetone
 The a.c. filtrate distilled off (50° mean) gave 4.12g green + 1/4 of this comp with H₂O + 0.365g
 HCl on 1000. gave a green only 1/2 of the green + laetone (i.e. is ca. 1/10 th) see No 10
 32.65g green

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Thompson treated this gum (175g) with 4.0g Buein vext 3X Benzol tpt 6.1g salts which xx from H₂O
 alcohol gave 1.2g porous belly needles $\frac{1}{2}$ npt 150-153 (no xx H₂O) $[d]_D^{20} = -16.85$ (acc. det.)
 and then 2.11g xx needles from salt II (no xx H₂O) with npt cfr 125-132 and $[d]_D^{20} = +22.55$ acc. det.
 when finally got from filtrate 1.5g salts which with Buein etc gave 0.95g Buein and
 0.35g gum ($p = 4.0$) with $[d]_D^{20} = +8.0$. crop I salts gum with Buein etc 0.74g Buein & 0.38g gum
 $[d]_D^{20} = +13.025$ whereas crop II gum with 1.5g Buein etc 1.42g Buein and 0.70g gum with $[d]_D^{20} =$
 -2.07 ($p = 4.0$). The last gum was probably d. allonic acid tit gave with 1.27g Chlorine etc
 153g xx salt easily soluble in 6cc abod. alcohol on standing (except 0.5g which was discarded)
 The other 2 gums $[d]_D^{20} = +8.0$ & $[d]_D^{20} = +13.025$ gum also on treatment with Chlorine etc & Saccharin or D-glucose
 easily soluble in small amt of cold abod. alcohol. The d. allonic gum 0.7g treated with Chlorine &
 can be present. discarded these 2 gums. The d. allonic gum 0.77g Chlorine + 0.35g
 sitting out of 0.15g difford. in cold alc salt (see above) was now set free tpt 0.14g
 crude d. allonic gum which treated with 0.5cc ph. hydr & 0.5cc alcohol of 24 hrs gave 0.14g
 xx ph. hydrizing npt 120-180. this was treated with a comp. lot of xx by drying obtained from
 xps with laevulose & CuH₂ (see 1072) total = 1.41g tpt by hydrolysis with 3.0g BaH₂ + 100 cc H₂O
 & 1/2 hr Buein vext 0.8g gum $[d]_D^{20} = +15.0$ ($p = 4.0$) $\frac{1}{2}$ found & in 1/2 cc tube = +0.155g gum (+14.97)
 now treated with 2+3% H₂O & 0.5cc Buein vext 0.8g gum $[d]_D^{20} = +15.0$ ($p = 4.0$) $\frac{1}{2}$ found & in 1/2 cc tube = +0.155g gum (+14.97)
 The 26.3g of neutral sugars + osons left (65% sugar approx) $[d]_D^{20} = -9.77$ gum follow. results:
 2.63g in 100cc H₂O tnd in cold a sol. of 5cc ph. hydr. in 50 cc acetic acid some gum (after 2 hrs t.t.) a sep.
 of ~~oson~~ d. glucosamin: after 1 week standing got by filtration washing well 2.37g and osos npt 170-180
 which on digestion with 100cc abod. alcohol gave 0.82g mod. osos npt 20-80: d. fructose
 must be present; on distilling off the alc. filtrate and 1.55g osos segregating R.F. with 50 cc C₆H₆
 got 0.91g mod. yellow osos npt 160-170° scarcely sol in 50cc hot abod. alcohol nothing came
 out on cooling: adding 50cc C₆H₆ to solution nothing came out on distilling off on Buein etc
 having sol got 0.23g xx npt 180-190° scarcely sol in hot abod. alcohol very diff. sol. in H₂O,
 ? is it the osos of d. pseudofructose or of α & β d. glucose or is latter left in filtrate?
 It is obvious that the osos present must give very soluble (in H₂O) osos (as first)
 comes out at once on mixing the crude neutral gums with ph. hydr. acetate in cold (filic acid)
 (72) d. Fructose & 2g CaH₂ 31 days t.t. (June 15 April 18)
 Took 100g Laevulose (fr. Dublin), wce H₂O and 3.8g CaH₂ that stand t.t. corked for 7 days: pass cor. the sol
 tpt 4.08g mod. CaH₂ 3 (Heavy 5.13g) after heating to 60° then filtering off. aq. filter made up to 1L ($p = 10$)
 gum & in 1 cc tube = -2.51: $[d]_D^{20} = -25.1$ incomplete equilibrium. Now distilled off H₂O & treated again
 with 200cc H₂O + 3.8g CaH₂ for 6 days t.t. pass cor. the sol. as before 4.35g ppd. CaH₂ 3: aq. filter
 made up to 1L gum & = -1.0025 in 1 cc tube: $[d]_D^{20} = -10.25$ still in incomplete equilibrium
 distilled off H₂O in vacuo & treated 3rd time with 200cc H₂O and 3.8g CaH₂ for 18 days t.t. corked.
 got by Cor 3.15g ppd. CaCO₃ & aq. filter made up to 1L gum & in 1 cc tube = +0.23: $[d]_D^{20} =$
 +2.3: O.K. now. Distill off H₂O in vacuo, less in 100cc H₂O tnd 1L spirit that stand & then got rid
 of 18g gum by decantation: the 18g gummy salts (roughly weighed as det. by decant) was then
 dissolved in H₂O & again by spirit and portion ppd. (not weighed) tnd to H₂O sale
 not acc. salts then discarded without det. wt. - whereas aq. alc. filtrate was then
 united with previous aq. alc. filtrate obtained by decanting & the neutral
 gums thus obtained (about 90g+)

were then treated in aq. sol 500cc with 54g Br₂ O.T. for 5 days; then draw them air, add PbO₂, Ag₂O 3 Hrs etc. aq. filtrate free 804") made up to 1 L (c = 10.) then pour & in 1 dec tube = -0.5 or [α]_D²⁰ = -5. The gum was now digested 10 hrs at 60° with 90g CaO 3 & 11.8g then filtered off. Dissolved this in vacuo & dissolved in wace H₂O + add 200cc spirit etc & got finally, in 3 crops, a total of 45.42g in vacuo dried Ca salts; there gave 8.03% Ca and gave [α]_D²⁰ = +4.044 (p = 4.0). Rest of salts then pptd by? got oxide gave 35.7g acid gums which made up to 1 L gave α in 1 dec tube = +0.74 ∴ [α]_D²⁰ = +20.72.

got also a total of 53.0g neutral sugars + 0.95% sugar present as det'd by Feh. Sol was 65-67%, found α in 1 dec tube = -0.95 and % sugar present as det'd by Feh. Sol was 65-67%.

This 35.7g acid gums + 20.72 took up 42.9g cinchonin gave a total of 43.8g d. glucenic cinchonin npt 180-190° this gum with 18g BaBr₂ etc 25.5g cinchonin + 17.4g dried glucenic cinchonin gave 54g CaO 3 spray by xx from H₂O a total of 15.4g sp. d. glucenic Ca salt 4.9g Ca salt

gums which took up 54g CaO 3 spray by xx from H₂O a total of 15.4g sp. d. glucenic Ca salt 4.9g Ca salt

The aq. filtrate then finally gum by pptn with alcohol etc 3.18g Ca salts (in vacuo); these gave [α]_D²⁰ = +9.7 and 9.19% Ca by analysis. Rest 2.96g, treated with 0.857g oxalic acid gum 2.3g form [α]_D²⁰ = +29° ∴ treated it with 5.1g Buecin etc C₆H₅ etc sept 7.6g XX salts which xx from 1.6cc H₂O + 1 bce almost gum 1.1g XX needles of d. Mammone green npt 210° Q.F.D. proving that small amt of d. Mammone acid is carried down with the d. glucenic acid.

Maeq. alc. filtrates from the crude d. glucenic cinchonin (43.8g) gum back 37g gummy salts which by 21g BaBr₂ etc gum 17.08g cinchonin and 17.55g gums with [α]_D²⁰ = +21.65 is. made up to 500cc of found α in 1 dec tube = +0.76. Tihalem taking 10cc took 18.45cc to 10cc H₂O + 100cc alcohol ∴ treated rest with 3.55g Buecin got 55.8g XX salts which xx from 10cc H₂O + 100cc alcohol gum 8.207g crop I d. Mammone Buecin npt 212°; this gave with 3.5g BaBr₂ 5.45g Buecin and 2.5g crude d. Mammone lactone gum which with alcohol etc gum 1.42g xx lactone npt 11/140-150° or on xx this from alcohol got 1.0g lactone npt 149-151° with [α]_D²⁰ = +58.95

The aq. alc. filtrate from crop I d. mammone lactone gum back 46.2g Buecin salts which after desolved in 5cc H₂O and 100cc alcohol got 5.34g (air dry) volun. xalle npt 150-155° mainly d. glucenic acid, 120-130° - the xx this from 9cc H₂O and 80cc alcohol got 5.34g (air dry) volun. xalle npt 150-155° mainly d. glucenic acid, with [α]_D²⁰ = -16.33 (after losing 0.32g xx H₂O over H₂O in vacuo and npt then 153°-165°). This gum with 2.5g BaBr₂ etc 3.1g Buecin and 1.5g gum (alc extn) and [α]_D²⁰ = +33.9 (p = 4.0)

got also a 2nd crop of xalle (b) 5.95g air dry npt 120-145° or after drying in vacuo got 5.7g npt 130-150° with [α]_D²⁰ = -22.15 which gum with 2.5g BaBr₂ etc 3.53g Buecin and 1.67g gum (alc extn) [α]_D²⁰ (p = 4.0) = +13.6.

Finally the orig aq. alc. filtrate from crop I 19.62g salts gum 24.1g salts which desolved in 5cc H₂O and 50cc alcohol got entirely solid with porous xx all crop II air dry, 5.9g, npt 120-140°, desolved in 5cc H₂O these lost 0.21g xx H₂O in vacuo, melted then from 130-160° gum [α]_D²⁰ = +17.8.

BaBr₂ etc 3.38g Buecin and 1.7g gum (alc extn) [α]_D²⁰ = +17.8, or total of 7g were now united & they took up independent 7.84g cinchonin and gum 1.8g salts which xx from all these 4 gums, 1.5g + 1.67g + 2.13g + 1.7g, or total of 7g were now united & they took up 40 cc abt. alc gum a total of 5.1g pure d. glucenic cinchonin xx. followed 180-210 and then no more.