S. S. S. 41 A

APR 1 1925 — 0.3 g. S 41 diss. in H_2O + N HCl, neutr. back w. NaOH.
APR 2 — Made up to 30 cc., treated w. 10 cc. freshly opened, neutralized 40% formaldehyde soln. After 1½ hrs., pptd. w. 100 cc. redistd. alc., sepg. as a jelly. Set in ice-box. After 1½ hrs. centfgd. Ppt. pressed out as well as possible w. rod & diss. in H_2O, yielding acid soln. Neutr. w. 3 drops N NaOH, few more particles dissg. Centfgd. Clear soln. @ vol. of ca. 40 cc., treated w. 4.4 cc. 25% HOAc, S pptg. heavily. Let stand on ice APR 3 — Centfgd. Washed 1" w. N/2 HOAc, 2 x w. 50% Alc., 1" w. redistd. alc. APR 4 — Centfgd., washed 2 x w. redistd. acetone, filtered off, washed w. redistd. acetone, dried. Yield: 0.3 g.

Active at diln. 1:8,000,000.

S. S. S. 41 B

APR 14 — 0.3 g. S 41 diss. as above, made up to 50 cc., made just alk. to phenolphthalein, 10 cc. 40% HCHO soln. (neutr.) added, color adjusted to ca. pH 9. APR 18 — Ca. 1 g. NaOAc added, portion pptd. w. alc. & rapidly centfgd. (B_1). Supernatant treated w. more alc., pptg. further portion (B_2) which was centfgd. off. Supernatants discarded. Ppts. taken up in ca. 10 cc. H_2O ea. & centfgd.
APR 20 — Solns. acidif. strongly w. 1:1 HCl & pptd. w. chilled redistd. alc. After several hrs. centfgd., rediss. in H_2O + HCl, repptd. After ca. 1 hr. centfgd., supernatants discarded, ppts. taken up in chilled 50% alc., then xs. alc. added.

S. S. S. 41 (For soly. detn. see next pg.)

OCT 13 — Wt. bot (10) + subst., dried in vac at t-b. $CHCl_3$ over H_2SO_4. 7.1853
7.1822 7.1824 7.1838
Made up to 10 cc. 7.0630
.1194

15 x
OCT 16 — Portion of dried subst. shaken several hrs. w. redistd. H_2O, let stand over nt. in const. temp. room. Temp. went to 26°. Fans on, then to 19° several hrs. Centfgd. (got warm). 25 cc. Soln. repatted into dishes

Wt. dish III + residue 23.0353 Wt. dish 16 + residue 23.3884
" " " 23.0354 " " " 23.3884

S 41A

APR 6 Wt. bot (4) + subst. (CHCl3 Foven) 8.1713 8.1701 8.0186 .1515

Diss. w. dil. H_2SO_4, run back w. NaOH, made up to 30.3 cc. w. H_2O.

ASH

Wt. bot. + subst 8.0190
" " 7.9561

Wt. Pt dish + ash 23.3999
" " " 23.3999

No Ash

OPTICAL ROTATION

$l = 1$ Bl. = +0.02 .01 .01 Rdg: +1.47 1.46
1.46 1.48 1.47

$$[\alpha]_D = +1.46 \times 200 = +292°$$

$[\alpha]_D = +292°$

AMINO NITROGEN

APR 7 –

t	P	Rdg.	Rdg – Bl.
21°	768.5	0.13	0.14 Bl.
21.5°	"	0.15	
21°		.58	
20.5		.58	

Amino N (HNO2 Method) **2.5%**

$$\frac{.44 \times .5729 \times 100}{2} = 12.6 \text{ mg. N per 100 cc.}$$

" 500 mg.

Total Nitrogen

2 cc samples

cc. N/50 HCl (×1.003)	3.00	6.02	3.00	6.02	6.02
" N/100 NaOH (×1.006)	2.70	2.72	5.25		= 2.37
		3.30	2.70	2.57	3.65
		.50	2.55		.50
		2.80 × .14			3.15 × .14
		3.9			4.4

cc N/50 HCl (×1.003)	3.00	3.00
" N/100 NaOH (×1.006)	2.32	4.70
		2.32

S. S. S. 41

~~Wt. bot (10) + subst 7.0028 Dried in high vac. at 100° over~~
~~" " " 6.9030 H_2SO_4~~
~~.0998~~

NOV 9 Wt. Pt dish (15) + residue 32.5207 32.5201 Sum on bal. dish ( ) + residue 32.3751 32.3748 Sum on bal.
" " " 32.5201 " " 32.3748

35

55 cc. samples used

32.5204
32.5201
.0003

32.3749
32.3748
.0001

S 41 B2

APR 25 • Wt. bot (F) + subst. 7.7537 7.7534 7.7534
" " " empty 7.7082 ~~7.75~~ 7.7082
.0452

APR 25 — Diss. in H_2O + HCl, neutr. w. N/2 NaOH, made up to 10cc., centfgd.

OPTICAL ROTATION

$l = 1$ $0 = 0$, -0.02 -0.02 Rdgs. +1.24 .25 .24

$$[\alpha]_D = +\frac{126}{.452} = +279°$$

APR 27

MICROKJELDAHLS 2cc. samples.

cc. N/50 HCl (×1.022)	3.00	3.00	6.13	6.13
" N/100 NaOH (×1.006)	3.10 ✓	3.30 (Over run)		3.12
				3.01
				.12

2.89 × .07 = .2 mg. N in 1cc.

$\boxed{\text{Total N } 4.4\%}$

$\boxed{NH_2\ N\ 2.5\%}$

Amino N

Bl.: 0.18 cc. @ 26°, 760.5 mm.
Rdg. 0.58 " " " " "
Rdg. − Bl. 0.40

.5529 × .4 × 50 = 11.1 mg. N per 100 cc., contg. 452 mg.

Action of HNO_2.

1cc. + 1cc. 30% aq. $NaNO_2$ + 0.6 cc. HOAc ~~Let~~ stand 0.5 hr.
0.3 cc. 18 N H_2SO_4 added, aspirated 3/4 hr. Made up to 25cc. 10cc. sample
Blank 19.00 cc, Rdgs. 17.45 17.65
17.55
1.45 × 1.02 × .318 = .47 mg Cu = 0.29 mg. glucose

$\frac{10}{25} \times 4.52 = 1.8$ mg. Rdg. sug = 16%

APR 28 — 1cc. + 1.3 cc. $NaNO_2$ soln. + 0.8 cc. HOAc; 0.3 cc. H_2SO_4.
Bl. 19.00 Rdgs: 16.85 16.85
16.85
2.15 × 1.02 × .318 = .7 mg Cu = 0.43 mg. glucose 24%

23.9

See also 39

S.S.S. 41

JUN 3 1925 — 1g. air-dried 41 diss. in 133 cc. hot $\frac{N}{2}$ HNO_3 & soln. b. 5 hrs. Concd. in vac. Jun. 4 — Concn. contd. to ca. 5-6 cc., mixed into sm., wide-mouthed Erlenmeyer w. 4 cc. concd. HNO_3, let stand at room temp., which occasionally rose to 34° JUN 9 — Considerable deposit of cryst.

June 11. Crystals centrifuged. Mother liquors allowed to stand longer after sl. diln. Crystals washed once with 2 c.c. water in cent. tube, cent., super. poured off. Crystals then treated for ½ hr with 5 c.c. water immersed in bail. water bath, only little soln. resulted so conc NH_3 added until soln. resulted. Cent. from a little dist. Clear colorless super. made acid with HNO_3 conc. ½ c.c. and tube put in ice box.

June 12 Cryst filtered on small Buchner and dried in vacuo. m.p. sinter at 206°, melt with decomp at 210°. Snow white 0.130 g. dry stuff recovered. Cryst again suspended in water and put with HNO_3 after NH_3 soln., cooled quickly with stirring. Allowed to stand two days. filtered & dried. m.p. = 210 sinters, 216, melts with dec. Weight: .115 gms.

C & H by Gartmann

Wt. bot + subst	6.5944
" "	6.4941
	.1003

See prepn 39

$CaCl_2$	53.1754	
	53.1317	H = 4.9%
	.0437	
Na-lime	55.9469	
	55.8238	C = 33.7%
	.1231	
Guard	45.5342	
	45.5335	
	.0007	
	.1231	
	.1238	

Theory for $C_6H_{10}O_8$

C = 34.3%

H = 4.8

SEP 24 — Considerable crystn. deposit centfgd. off, washed w. 1-2 cc. H_2O (M.L. A). Cryst. taken up in little H_2O & ss. aq. NH_3 added, but very little soln., as app. too much NH_4NO_3 present. Cryst. diss. in hot H_2O, filtered, soln. cooled, few drops concd. HNO_3 added, app. mucic acid soon crystg. in ice-box.

SS 42

14
3
42
18
60

2/16/25	1/204/5	II - 4	14	2/17	2/18
2/17/	1/204/8	II - 10	14	2/18	2/19
2/18	1/204/8	II - 10	14	2/19	2/20
2/19	1/204/4,5,8,9	II - 3, 4, 10, 11	2, 1, 2, 9	2/20	2/21
2/24	1/204/9	II - 11	70 ~~14~~	2/25	2/26
2/25	1/204,5/9,3	II - 11, 18	14	2/26	2/27
2/26	1/205/3	II - 18	12	2/27	2/28
2/27	1/205/3	II - 18	14	2/28	3/2
3/2	1/205/3	II - 18	14	3/3	3/4
3/3	1/208/3	II - 25	138 ~~14~~	3/4	3/5
3/4	1/205,8/3	II - 18, 25	8, 6	3/5	3/6
3/5	1/205,8/3	II - 18.25	6, 8	3/6	3/7
3/6	1/208/3	II - 25	8	3/7	3/9
3/12	1/209/3	III - 3	14	3/13	3/14
3/16	1/209/3	III - 3	198 ~~10~~	3/17	3/18

168
30

198
99
297

297 liters

~~Total~~
~~Flasks 198~~
~~LITERS 297~~

MAR 19 1925 — Concentrate, at vol. of 4.8 l., pptd. w. 1:1 HCl until acid to Congo. After several hrs. in cold, as much of soln. as possible siphoned off, remainder centfgd. in cold., ppt. washed 1" w. N/100 HCl. 2" washing added to
2 next lot. At vol. of 5 l., pptd. w. 6 l. chilled alc. MAR 20 — Supernatant, almost —, siphoned off, remainder centfgd. Ppt. washed
HOAc w. N/2 HOAc, centfgd. Supernatant, still ++, partially neutr. w. NaOH, 20g.

NaOAc added, & at 300cc, pptd. w. 200 cc. alc. After 1 hr. in ice-box supernatant decanted, ppt. centfgd. off. & added to (43). Main ppt. taken up in H_2O + sl. xs. NaOH, soln. centfgd., & at vol. of ca. 200 cc., pptd.
$Ba(OH)_2$ 1 w. ca = vol. warm satd. $Ba(OH)_2$ soln. Let stand in ice-box. MAR 21
Centfgd. MAR 23 Supernatant –, discarded. Ppt. diss. by taking up in H_2O + enough 1:1 HCl to keep soln. acid to Congo red. Made just alk. w. NaOH & centfgd. MAR 24 – Supernatant ++, further ppt. on making strongly alk. w 40% NaOH. Centfgd. in cold, supernatant
$Ba(OH)_2$ 2 ± discarded, ppt. taken up in H_2O + enough 1:1 HCl to keep reaction acid to Congo. MAR 26 – Centfgd. Supernatant treated w. xs. satd. $(NH_4)_2SO_4$ soln. to ppt. Ba^{++}, then made less acid to Congo w. a few drops concd. aq. NH_3. Let stand in ice-box.

Lost in accident to cold centrifuge.

Portion scraped up from wreckage, extd. w. H_2O. Vol. ca 1.5 l. APR 6 – Concd. in vac. to sm. bulk. Could not be pptd. at isoelec. pt. owing to amt. salts present. APR 7 – At vol. of 125 cc., ~~pptd. w.~~ made alk. w. NaOH, pptd. w. = vol. alc. Let stand several hrs. in ice-box, centfgd. Supernatant –, discarded, ppt. taken up in 100 cc. H_2O, neutr. w. HOAc, then 3 cc. more HOAc added, pptg. some S. APR 8 – Centfgd. taken up in dil. HCl, centfgd., pptd. w = vol. redistd. alc. After ca 1 hr. centfgd., ppt. redis. in dil. HCl, ~~soln.~~ centfgd., pptd. w. alc. as before. Centfgd., ppt. taken up in 50% alc. APR 9 – Ca 2 vols. redistd. alc. added, let stand 1 hr., centfgd..

155' #3

3/20/5	1/209"11/3	5, 9	III - 3, 12		3/21	3/23
3/23	1/211/3	III - 12	14		3/24	3/25
3/24	1/211/3	III - 13	14		3/25	3/26
3/25	1/211/3	III - 13	14		3/26	3/27
3/26	1/211/3	III - 12, 13	4, 10	70	3/27	3/28
3/27	1/211/3	III - 12, 13, 20	3, 5, 6		3/28	3/30
3/30	1/211/3	III - 20	14		3/31	4/1
3/31	1/211/3	III - 20	14		4/2	4/2
4/1	1/211/3	III - 20	14		4/2	4/3
4/3	1/211 "13/3, 4	III - 20, 21	5, 6	140	4/4	4/6
4/6	1/213/4	III - 27	14		4/7	4/8
4/7	1/213/4	III - 27	14		4/8	4/9
4/8	1/213/4	III - 27, IV - 2	14		4/9	4/11
4/9	1/213/4	~~IV - 27~~ IV - 2	14		4/13	4/14
4/13	1/213/4	IV - 2	11		4/14	4/15

207 Plates

310.5 l. in all

APR 15 1925 — Concentrate, at vol. of 4.3 l., pptd. w. 1:1 HCl until ~~acid to~~ Congo. paper turned blue. Let stand 3 hrs., upper liquid siphoned from ppt., ppt centfgd., & ppt. washed 1" w. N/100 HCl. At vol. of 5 l. pptd. w. 7 l alc. APR 16 — Supernatant siphoned off, ppt. centfgd., taken up in N/2 HOAc, let stand 1 hr. in cold, centfgd. Supernatant —, discarded. Ppt. diss. by making susp. faintly alk. w. NaOH & centfg. at vol. of ca 400 cc. Ppt. washed 1" w. H_2O. Combined solns. + 15 g. NaOAc, at vol. of 500 cc., pptd. w. 300 cc. alc.; let stand in cold. Supernatant —, ppt., still dark, centfgd. off,

2

3

ppt. taken up in H_2O & centfgd. at total vol. of ca 300 cc. Repeated centfgn. & standing in cold necessary before clear supernatant (A) obtd. Gelatinous ppt. taken up in H_2O, again centfgd. & ppt washed 1" w. H_2O, combining washings w. this fraction, (B). APR 23 — A, + 7.5 g. NaOAc, at vol. of ca. 250 cc., pptd. w. 150 cc. alc. B, + 10 g. NaOAc, at vol. of 275 cc., pptd. w. 175 cc. alc. APR 24 — Supernatants —, so centfgd. Ppts. taken up in H_2O. (A) clear, (B) centfgd. repeatedly. APR 25 — A, at vol. 170 cc., pptd. w. 90 cc. alc., let stand in ice-box. APR 30 — A supernatant, —, discarded, ppt. taken up in ca 100 cc. H_2O, centfgd., clear supernatant pptd. w. 4 cc. HOAc, let stand in ice-box, centfgd., supernatant, —, discarded, ppt. taken up in ca. 100 cc. H_2O + NaOH to neutrality. Clear supernatant from (B) poured off into centfg. bottles, ppt. again taken up w. H_2O, repeatedly centfgd., adding soln. to previous B solns. Caused formn of ppt. in these. A pptd. w. xs. $Ba(OH)_2$. Let stand in cold, centfgd. Part of crystn. xs. $Ba(OH)_2$ diss. in H_2O, mixt. centfgd., & supernatant discarded, as contd. very little S. Remainder of ppt. diss. w. xs. dil. HCl, Ba pptd. w. Na_2SO_4 soln. Centfgd., ppt. washed 1" w. very dil. HCl contg. little Na_2SO_4, combined supernatants neutr. to Congo w. NaOH. MAY 5 — Pptd. w. ca. = vol. alc., ~~portion~~ centfgd. after ca 3 hrs. Supernatant —, discarded. B solns, repeatedly centfgd. MAY 6 — A ppt. diss. in H_2O + NaOH to faintly alk. reaction, centfgd. from traces $BaSO_4$, & at vol. of ca. 60 cc, pptd. w. 2.5 cc. HOAc. After several hrs. in ice-box, centfgd. MAY 7 — Taken up in H_2O + 1:1 HCl, pptd. w. ca 2 vols. alc. Centfgd. after ca. 1 hr. Process repeated. Ppt. taken up in H_2O, pptd. w. xs. alc., centfgd. again taken up in H_2O, alc. added, finally washed w. H_2O until Cl^- free, then acetone, dried. **0.3 g.**

B & centfgd. residue gave clear soln. Active 1:6,000,000 when enough H_2O was added, again pptg. on addn. of 20 g. NaOAc at vol. of 500 cc. 300 cc. alc. added, let stand in cold ca. 2½ hrs., centfgd. MAY 8 — Ppt. taken up in ca 450 cc. H_2O, centfgd. from sl. insol. portion, pptd. w. 16 cc. HOAc. After several hrs. centfgd. MAY 9 — Bulky ppt. diss. in H_2O + NaOH, 15 g. NaOH added, & at vol. of 300 cc. pptd. w. 175 cc. alc. After several hrs. in cold, supernatant —, mixt. centfgd. repeatedly until ppt. firm. Ppt. taken up in ca. 400 cc. H_2O, centfgd. from traces insol., pptd. w. 12 cc. HOAc. After several hrs. centfgd. MAY 15 — Ppt. taken up in ca. 300 cc. H_2O, neutr. w. NaOH, & soln. pptd. w. xs. hot satd. $Ba(OH)_2$. After several hrs. centfgd. Very little ppt. (B) Supernatant acidified w. HOAc. Worked up as C.

B — Ppt. taken up in H_2O until no more lumps, then N H_2SO_4 added until acid to Congo. Centfgd., & supernatant (milky) neutr. to Congo. MAY 18 — Ppt. washed 1ᵗᵉ more w. very dil. H_2SO_4 & this added to main portion. MAY 19 — Soln. neutr. to litmus, run 1ᵗᵉ through Berkefeld V, then W, & clear soln conc'd. in vac. to 150 cc. Ppt'd. w. 80 cc. alc. MAY 20 — Supernatant — mixt. centfgd. MAY 21ᵗᵉ Ppt. taken up in ca. 150 cc. H_2O, centfgd. from traces insol. material, ppt'd. w. 5 cc. HOAc. Centfgd. MAY 26ᵗᵉ Ppt. taken up in H_2O + 1:1 HCl, & at vol. of ca. 100 cc., ppt'd., w. stirring, w. chilled redist'd. alc. After ca. 1 hr. centfgd. Pptn. repeated 2X. Ppt. diss. in H_2O, ppt'd. w. 1½ vol. alc. MAY 28 — Centfgd. Ppt. taken up in H_2O, alc. added. Finally centfgd. Washing repeated 2X. MAY 29ᵗᵉ Centfgd. Washed 3X w. H_2O, then again until Cl^- —. Washed 2 X w. acetone, filtered off, washed w. acetone, dried. **Yield: 1.4 g.**

Active 1:6,000,000

(C) — Washed w. N/2 HOAc.

C — Deposited some S on standing. Centfgd. Still more in M.L. which MAY 18 — came out on making strongly alk. w. NaOH. After several hrs. centfgd., soln. discarded. Ppt. (D) taken up in H_2O, treated w. xs. N H_2SO_4, centfgd. MAY 19 — Supernatant neutr. w. NH_4OH & at vol. of 150 cc, treated w. 5 cc. HOAc. No immed. ppt. JUN 10ᵗᵉ off — D discarded. C diss. in H_2O + HCl, sl. xs. H_2SO_4 added, centfgd. Soln. neutr. w. NaOH, ppt'd. w. xs. alc., ppt. centfgd. off. Taken up in dil. HCl, ppt'd. w. alc., centfgd. Ppt. taken up in H_2O, 1:1 HCl added (7.8 cc.) to make final concn. 1.3 N, dil'd. to 30 cc. No immed. ppt. but on letting stand in ice-box from 12ʰ to 15ʰ most of S sep'd. as jelly. JUN 15ᵗᵉ Centfgd. Supnt., ++++, deposited relatively little S when treated w. alc. Main ppt. taken up in H_2O, ppt'd. w. alc. Washed by repetitions of process, finally w. H_2O alone, then acetone. Yield: 0.2 gm.

Active: 1:6,000,000.

S.S. 43 A

JUN 4

Wt. bot (10) + subst. 7.1182 7.1180
7.0148
.1032
Less Ash .0012
.1020

	ASH		
Wt. bot + subst	7.0151	Wt. dish + ash	23.3892
" ~~bot~~.	6.9554	" "	23.3885
	.0597		.0007

Ash 1.2%

N.B. Above sample dis in slight excess N/. HCl, diluted to 20.40 c.c. giving 1:200 soln. This soln. was used in all the analyses given below, giving Ash-free results

① Amino Nitrogen Determination

Sample	Temp.	Bar.	Rdg Bl.	Rdg. Sample
—	29.5°	761.7	0.130	—
—	30.0°	761.7	0.130	—
2 c.c.	30.5°	761.5	~~0.590~~	0.590
2 c.c.	30.5°	761.4 (uncorr. temp.)	0 —	0.595
				.13
				.463

% Amino Nitrogen $\boxed{2.49}$ Factor = .5378

NH_2N 2.5%

② Optical rotation. 1 dec. tube.
Zero +.08 +.08 +.09 +.09
Rdg +1.60 +1.60 +1.60

$[\alpha]_D = +303.0°$

③ Total Nitrogen. 3 c.c. above soln. for each analysis (or 15.0 mgs)

		1.	2.	Bl.	Bl.	
9.95	c.c. N/70 HCl (F .995)	10.00	10.00	10.00	10.00	9.95
6.17	" " NaOH (F=1.015)	6.05	6.10	9.75	9.75	9.90
3.78						.05
.05						
3.73	Percent Nitrogen	$\boxed{4.98\%}$				

Total N 5.0

④ Reducing Sugars: 2 c.c. above, 2 c.c. water 4 c.c. N/1 H_2SO_4. Boiled 5 hrs. Add to 20 c.c. and 4 c.c. for each analysis (2 mgs).

	1.	2.	Bl.
(F=1.010) Thiosulphate			

⑤ Reducing Sugars with Nitrite: 2 c.c. soln. (10 mg), 4 c.c. 30% $NaNO_2$, 2.2 c.c. HOAc. Stood ½ hr, 0.8 c.c. 18/N H_2SO_4 added. Soln dil to 20 c.c. 5 c.c. for each sugar analysis (2.5 mgs)

	1.	2.	Bl
(F=1.010) Thiosulphate	15.25	15.15	19.30

" used up. 4.2 c.c.
Cu. Equivalents 1.38 mgs
Glucose " 0.72 mgs
% " 28.8%

Bottle No 12 + Sample 8.2118
" - " 8.1112
Sample 0.1004

1. Ash Crucible + Sample 23.4412
" - " 23.3882 Unweighable trace of Ash.
Sample 0.0530
Crucible after wg. 23.3882.

2. Specific Rotation — : above sample dis. to 20.08 c.c. gives 1:200 soln.
1 dec. tube Zero -.02, -.02, -.02
Rdg +1.48, +1.47, +1.48, +1.48

$[\alpha]_D = +300.0°$

3. Amino Nitrogen —

	Temp	Barom.	Blank c.c.	Sample c.c.
2 c.c. of above soln.	23°	773.2	0.190	~
for each analysis.	23°	773.2	0.190	~
	23°	773.1	~	0.620
	23°	773.1	~	0.620

Factor 0.5708
% Amino Nitrogen = 2.46

4. Total Nitrogen — 3 c.c. above soln.

	1.	2.	Bl.
c.c. N/10 HCl F = .991	10.00	10.00	5
" NaOH F = 1.015	6.05	6.05	4.75

% N. 4.90

5. Reducing Sugars on treating with $NaNO_2$ 2 c.c. above dilution.
4 c.c. 30% $NaNO_2$, 2 c.c. HOAc. stood ½ hr. 0.8 c.c. N/10 NaOH. Ref. 1.5 hrs. Dil 25 c.c. and 5 c.c. for analysis (2.0 mgs)

	1.	2.	Bl.
(F = 1.010) c.c. Thiosulphate	15.25	15.35	19.30

Copper Equivalent 0.97 mgs
Glucose Equivalent 0.57 mgs
% Glucose 28.50

6. Reducing sugars on hydrolysis w. 1.5 N HNO_3 for 5 hrs.
Bl. 19.40 Rdgs. 15.20 15.20
2 cc. dil. to 4 w. H_2O, 4 cc. 3N HNO_3. after 5 hrs. dil. to 20 cc.
5 cc. samples

19.40
15.20
4.2 × 1.01 × .318 = 1.35 Cu
Glucose .71

Redg. Sugars 28.4%

Hydrolysis of Type II S.S.S. and the ammonia liberated.

4 c.c. Prep HBB (1:200 soln) was treated with 8 c.c. 30% $NaNO_2$ + 4.4 c.c. HOAc. Stood ½ hr. and with 1.6 c.c. 50% H_2SO_4. Aspirated 2 hrs, washed into a K flask and ammonia distilled.

	(E=1.02) Acid	alk (F=1.015)		% N liberated
10 min boiling	10.00	8.725	(0.2 Bl)	1.14
15 min more "	10.	9.675		0.38
15 min " "	10.	9.750		0.21
Total in 40 min. of boiling				1.73

A similar expt was tried and the sample, instead of boiling was aerated. Something happened for acid 10 c.c. of acid was used and 15 c.c. of alk was used for end point and a long color range was gone over.

43-C

Bottle 3 + Sample	7.5896
" — "	7.4820
Sample	0.1076

(1) Rotation 1 dec. tube. made up to 21.52 c.c.

Zero +0.16 0.16, 0.17, 0.16

Rdg. +1.66, +1.67, 1.66

[α] +300.0°

(2) Amino N.

Temp.	Bar.	Blank	Sample
26°	760.4	0.130	—
26°	760.2	0.130	—
26°	760.2	—	0.590
26°	760.2	—	0.590
			.13
			.46

Factor = .5512

% Amino N. = 2.58 ✓

(3) Total Nitrogen: 3 c.c. samples (15 mgs)

	1.	2.
c.c. N/10 HCl (F 1.020)	~~6.22~~ 10.0	10.0
" " NaOH (F = 1.015)	6.225	6.20

Blank = 0.15 c.c.

% Nitrogen = 5.00

(4) Ash

Crucible + Sample	23.4448
" — "	23.3882
Sample	0.0564
Crucible after ignition	23.3882

Unweighable trace of ash

(5) Sugar determ with HNO_2 · 2 c.c. Sample → 4 c.c. $NaNO_2$, 2.2 HNO_3

Asp. 2 hrs after standing ½ hr. Dil to 25 c.c. + 5 c.c. formal

	1.	2.	Bl
Thiosulphate (F = 1.01)	16.25	16.30	19.30

Cu Equiv. 0.96 mg.

gl. " = 0.56

% glucose = 28.0

S S H H

4/15	1/213'12/4/3	IV - 3, 7	3, 11		4/16	4/17
4/16	1/212/3	IV - 7	14		4/17	4/18
4/17	1/213"12/4, 3	IV - 3, 7	2, 13		4/18	4/20
4/20	1/212/3	IV - 11	14		4/21	4/22
4/21	1/212/3	IV - 11	14	(70)	4/22	4/23
4/22	1/212/3	IV - 11	12		4/23	4/24
4/24	1/212/3	IV - 18	12		4/25	4/27
4/27	1/212/3, 4	IV - 18, 20	8, 6		4/28	4/29
4/28	1/212/3, 4	IV - 18, 20	8, 6		4/29	4/30
4/29	1/212/4	IV - 20	14	(136)	4/30	5/1
4/30	1/212"16/4	IV - 20, 24	8, 4		5/1	5/2
5/1	1/216/4	IV - 24	14		5/2	5/4
5/4	1/216/4	IV - 24	10		5/5	5/6
5/5	1/216/4	IV - 29	12		5/6	5/7
5/6	1/216/4	IV - 29	12	(96)	5/7	5/8
5/7	1/216/4	IV - 29	14		5/8	5/9
5/8	1/216/4	IV - 29	12 + 2		5/9	5/11

4 flasks	225
Liters	337.5

MAY 12 1925 — Concentrate, at vol. of 4.75 l., ppt'd. w. 1:1 HCl until blue w. Congo. Let stand 3 hrs., upper liq. siphoned off, rest centfgd., ppt. washed 1" w. N/10 HCl. At vol. of 5.75 l., ppt'd. in
2 cold w. 8 l. chilled alc. MAY 13 — Supernatant siphoned off, ppt. centfgd. repeatedly, diss. in H_2O at vol. of ca. 600 cc., ~~centfd~~ neutr. w. NaOH, & centfgd. repeatedly. MAY 14 — At vol. of 700 cc., 20 g. NaOAc added, & soln. ppt'd.
3 w. 350 cc. alc. MAY 15 — Supernatant —. Centfgd. MAY 18 — Ppt. diss. in H_2O, centfgd. from sm. amt. dark ppt. at vol. of 500 cc. ~~15 g. NaOAc~~

MAY 19 & 20 — ~~15 g. NaOAc added, & pptd. w. 175 cc. alc.~~ Pptd. w. 16 cc. HOAc.
HOAc After several hrs. centfgd., supernatant poured off, ppt. taken up in H_2O + enough NaOH for neutrn., centfgd. 15 g. NaOAc added, & at vol. of 400 cc., pptd.
4 w. 200 cc. alc. MAY 21 — ~~Supernatant~~ —, centfgd. Ppt. taken up in ca. 400 cc. H_2O,
HOAc centfgd., soln. ppt. w. 14 cc. HOAc. After several hrs. centfgd. MAY 26 Ppt. taken up in H_2O + NaOH until clear, faintly alk. soln. obtd.
MAY 27 — At vol. of 250 cc., treated w. hot-satd. $Ba(OH)_2$ soln., causing immed. ppt. After several hrs. centfgd. Ppt. (A). Supernatant (B), made more strongly alk. w. 40% NaOH, depositing $Ba(OH)_2$. A taken up w. 250 cc. H_2O + H_2SO_4, centfgd., ppt. washed 1" w. very dil. H_2SO_4, solns. neutr. MAY 29 — Run through Berkefeld V & W successively, coming through clear. Concd. in vac. JUN 5 — At vol. of 175 cc., pptd. w. 100 cc. alc., let stand ca. 2 hrs. in cold, centfgd. M.L. sepd., —, discarded. Ppt. taken up in H_2O, but did not diss. entirely, ∴ reppted. w. alc. & centfgd. " " " 1" in N HCl, then 1:1 HCl added causing pptn. After ½ hr. in cold centfgd. Supernatant, ±, gave no ppt. w. alc., discarded. Ppt. diss. in H_2O (except for few lumps, smoothed out later), reppted. w. 1:1 HCl, centfgd. Reaction evidently not quite right this time, as supnt (A₂) +++. Ppt. (A₁) redis. in H_2O, pptd. w. ~~alc~~ redistd. alc. in cold. JUN 10 — A₁ centfgd., ppt. taken up again in H_2O, little alc. stirred in to reduce sp. gr., & centfgd. from dirt. Soln. pptd. w. chilled alc., centfgd. A₂ treated w. ca. 1½ vols. alc., some subsh pptng., supnt. remaining opalescent.

JUN 11 — A₁ — Ppt. washed again as before, not dissg. entirely. A₂ centfgd. again. Supnt, now clear, discarded. Ppt. diss. in little H_2O, made 1.3 N w. HCl. A₂ pptd. After several hrs. centfgd., supnt. discarded, ppt. diss. in H_2O & added to A₁. This washed repeatedly as before, finally w. H_2O until Cl^- free. Washed w. acetone, filtered off, dried. **Yield: 1.7 g.**

Active at 1:8,000,000.

B. Supernatant (C) poured off, ppt. taken up in H_2O + sea. H_2SO_4, centfgd., ppt washed 1" w. H_2O. Supernatant run through V & W Berkefelds, concd. to sm. bulk, dial'd.

Very little S in B and C.

Saved for next prepn.

544 A

JUN 12 Titrn. of M.L. from 1" HCl pptn. of 44A 1cc. dild. to 10cc.

	1cc. samples		
cc. N/70 NaOH (x 1.015)	8.93	8.93	9.07

1cc. orig. soln. ∝ 90.7 cc. N/70 NaOH ∝ 1.3 cc. N NaOH

Titrn. of M.L. from HCl pptn. of 44A₂ 1cc. dild. to 10cc.

	1cc. samples		
cc. N/70 NaOH (x 1.015)	9.85	9.85	10.00

1cc. orig. soln. ∝ 100.0 cc. N/70 NaOH ∝ 1.4 cc. N NaOH

C & H on 44 A

(Gartmann)

Jun 22 Tot. bot + subst 7.6880
" " 7.5875
0.1005

$CaCl_2$ 53.2216
53.1720
.0496 H = 5.5%

Na-lime 56.1056
55.9471
.1585

Guard 45.5352 C = 43.3%
45.5344
.0008
.1585
.1593

44A

Bottle No B + Sample 7.5390
" - " 7.4254

Weight Sample 0.1136

1. Rotation: diluted to 22.66 c.c. giving a 1:200 soln (calc. as ash free)
1 dec. tube

Zero: -.03, -.02, -.03, -.02, -.02, -.03
Rdg. +1.47, +1.48, +1.48, +1.47

$[\alpha]_D = +300.0°$

2. Ash. Burned with H_2SO_4. Weighed as $CaSO_4$, calc. as CaO.

Crucible + Sample 23.4556
" - " 23.3880

Sample 0.0676

Crucible after ignition 23.3888
Weight $CaSO_4$ 0.0008
% CaO = 0.48%

3. Hydrolysis with HNO_2. 2 c.c. sample, 4 c.c. 30% $NaNO_2$, 2.2 c.c. HOAc. Stood ½ hr. 8 c.c. 18/N H_2SO_4. Asp. 2 hrs. dil to 25 c.c. & 5 c.c. for each analysis (2 mgs)

	1.	2.	Bl.
(F: 1.010) Thiosulphate	16.20	16.30	19.30
Copper Equiv.	0.98 mgs		
Glucose "	0.57		
% glucose	28.5%		

4.

Amino Nitrogen: 2 c.c. above for each analysis.

Temp.	Barom	Blank	Sample
24°	766.3	0.105	—
24°	766.3	0.100	—
24°	766.0	—	0.575
24°	766.0	—	0.575

Factor .5603 % Amino Nitrogen: 2.60%

5. Total Nitrogen: 3 c.c. above dilution for analysis (15.0 mgs)

	1.	2.
c.c. N/70 HCl (F=1.020) =	10.00	10.00
" " NaOH (F=1.010) =	6.125	6.125

Blank = 0.15 N/70 acid

$$\% \text{ Nitrogen} = \frac{\{(10.0 \times 1.020 - 6.125 \times 1.015) - 0.15\} \times 2}{15.0} \times 100$$

= 5.04%

DEC 4 1925 — 1 g 47 6IIIA ~~plec~~ (anhydrous) placed in hydrolysis flask and 10 cc. thionyl chloride run down condenser, w. rotating. DEC 7 — Had gradually turned yellow, but much if not most of gum still undiss. DEC 10 — Mixt. filtered and yellow product washed thoroughly w. dry Et_2O until this no longer gave Cl^- test. Portion of solid, b. w. dil. NaOH, gives marked Cl^- test. Dried in vac. over ~~P_2O_5~~ H_2SO_4 + Na-lime. Yield: 1 g.

Wt. bot (3) + subst: 7.6761, 7.6747, 7.6743

7.4725

.2018

Susp. in H_2O (acid to Congo), drop phenolphth. added, 5.98 cc. N/14 NaOH (×0.966)

S.S.S. 51

H. Discarded

JAN 11 1926 — Ca. 20 g. 47/10BI diss. in 2 l. H_2O, neutr. w. 10% NaOH, 1 l. cold satd. $Ba(OH)_2$ added, let stand over night. JAN 12 off Soln., yellow, decanted from ppt. (A) & this centfgd. Soln. treated w. 3½ l. more $Ba(OH)_2$ soln., giving further slight pptn. (B). Supnt. (C).

A shaken w. xs. dil. H_2SO_4, centfgd. in cold.
B " " " " " " " } Both treated w. few cc. HCl, pptd. w. 3-4 vols. chilled redistd. alc. Ppts. centfgd. off, taken up in redistd. alc., again centfgd., taken up as before, filtered off, washed well w. redistd. acetone, dried over $CaCl_2$ + NaOH in vac.

A: .02 g. Activity: 1:3,000,000
B: 1.2 g. " : 1:3,000,000

No fractionation by $Ba(OH)_2$, even in very dil. soln.

MAR 1 — 5.6 g. 47/10BIV diss. in ca. 30 cc. H_2O. HOAc added, pptn. beginning after ca 2 vols. had been added. In all 3-3½ vols. stirred in. Let stand in cold ca 2 hrs. Centfgd. Ppt. (C). Supnt. + = vol. Et_2O + drop conc. HCl gave more ppt. (D). Let stand in ice-box. C taken up in redistd. alc., isolated as usual. D also.

MAR 24 — Wt. bot (Z) + sample D 8.9467 8.9449 8.9442
" " (Z) 8.5436 8.6184
.3258
.0081
.3257

Diss. in H_2O, made up to 16.29 cc.

Ash

Wt. bot + subst.	8.6184	Wt. dish + ash	14.3120
" "	8.5462	" dish	14.3119
	.0722	" ash	.0001
		" Ca	.00003

Ash 0.04%

OPTICAL ROTATION

l = 2 Bl. = −0.06 .06 .06 Rdgs. −0.93 .92 .91 .93

$$[\alpha]_D = -\frac{92-6}{4} = -\frac{86}{4} = -21.5°$$

$[\alpha]_D$ −21.5°

Na Salt

15 cc. Req. 3.80 cc. N/14 NaOH (×1.025) = 3.89 = 0.278 cc N

Acid Equiv. 1080

l = 2 Bl. = −0.06 .06 .06 Rdgs.: −0.93 .85 .83 .81 .85 .82 .84

$$[\alpha]_D = -\frac{77}{4} \times \frac{18.85}{15} = -24°$$

$[\alpha]_D$ Salt −24°

Furfural Detn

17 cc. above soln. req. 6.0 cc. N/10 $KBrO_3$ = .0288 g. furfural = 10.7%
$\frac{17}{18.85} \times .3000 = .2705$ g .0450 g. pentose = 16.6%

Aq. soln — more viscous than usual.

S S S 51 E, F

APR 1 ff. — 60 g. 47 10B IV lot 2 diss. in 300 cc. H_2O & pptd. w. HOAc after adding 5 cc. 30% aq. $NaNO_2$ + ca 25 cc. HOAc & stirring 0.5 hrs. Ca. 1.5 vols. necessary. After 2 hrs. ~~pp~~ supnt. decanted, ppt. (E) drained, & diss. in H_2O. Supnt. from E & pptd. w. ca 1/4 vol. Et_2O. Ppt. (F) drained as well as possible, dissd. in H_2O. E again pptd. from ca. 200 cc. w. HOAc & let stand. F, at vol. of ca. 50 cc., pptd. w. ca 2 1/2 vols. HOAc, ppt. discarded, supnt. pptd. w. ca vol. Et_2O. E treated w. successive portions redistd. alc. & acetone, washed well w. acetone, dried in vac over $CaCl_2$ + NaOH : 42.3 g. Et_2O ppt., F rediss. in a little H_2O, few cc. 1:1 HCl added, pptd. w. redistd. alc., rediss., repptd. as before, worked up as usual.: 0.7 g.

Activity :	E	1 : 5,000,000	N 0.08%
	F	1 : 3,000,000	N 0.50%

MAY 19 ff. HYDROLYSIS OF S 51E

41 g. 51E diss. in 2 l. H_2O, 85 cc. conc. H_2SO_4 added, then 1 l. cold H_2O, making H^+ N. MAY 20 — B. 4 hrs., let stand over night. MAY 21 ff. Most of H_2SO_4 neutrd. w. hot $Ba(OH)_2$ soln., let stand over night in ice-box, decanted, ppt. washed 1" w. H_2O, centfgd. Supnt. concd. to ca. 1 1/2 l. in vac (some tap H_2O got in owing to pump sucking back), remaining H_2SO_4 MAY 26 — pptd. w. $Ba(OH)_2$, centfgd., ppt. washed w. H_2O, soln. concd. in vac to sm. vol. MAY 27 — Moist, pptd. PbO added, but as this appeared to contain some red lead, mixt. filtered before enough Pb had been added for neutrn. Filtrate + basic $Pb(OAc)_2$ gave no ppt., i.e. dild. & xs. H_2S passed in. Filtered, ppt. washed w. H_2S-H_2O, & filt. & washings mixed. Vol. 1400 cc. 5 cc. sample, 16.6 cc. N/10 $Na_2S_2O_3$ (Macro-Schaffer method) × 1.02. Bl. 49.6 cc. ∴ sugar

$$\frac{33 \times 1.02 \times 6.36}{2.17} = 99 \text{ mg. glucose} = 28 \text{ g. sugar.}$$

Soln. concd. ~~dry~~ in vac. to syrup to remove most of HOAc. MAY 28 — H_2O added & concd. to syrup again. Repeated. Taken up in H_2O, filtered from traces dark ppt., & treated w. sl. xs. basic PbOAc. After 1 1/2 hrs. centfgd. in cold. Supnt. I. Ppt. II. II susp'd. in H_2O, sl. excess HOAc added, & at vol. of ca 200 cc. centfgd. ~~Orange~~ Sm. amt. orange ppt. discarded. Orange supnt. treated again w. PbOAc until no further ppt. After 1 hr. Centfgd. Supnt. IA Ppt. II.

~~I~~ and

Hydrolysis

S 51 E I - Sugar Fraction

MAI 28 rff. - I & IA separately treated w. xs. H_2S, filtered washed filtrates combined, concd. to syrup in vac., taken up in hot H_2O, bone-blacked, without removal of H_2S, again concd. ~~to rem~~ to thick syrup, treated warm w. ca. 2 vols. HOAc, seeded w. few crysts. galactose. After 1 hr. crystn. obvious & increased rapidly on stirring. JUN 1 — Solid cake early broken up w. rod, filtered off in cold, washed w. chilled 2:1 HOAc-H_2O, then HOAc, then redistd. alc., dried. Yield: 8.3 g. Portion dried const. in vac. @ 100° over H_2SO_4 (bot 10) 7.7564 7.7552 7.7544

JUN 3 7.2254

.5290

Dis. in H_2O, made up to 10cc., rotation: Bl. -0.02 .03 .01 Rdg. +6.36 6.36 l=1

$[\alpha]_D = \frac{6.38}{5.290} = +120.5°$

JUN 4 - l=1, Bl. -0.05 .06 .05

Rdg. +3.54 .58 .60 .55 .56

$[\alpha]_D = \frac{3.61}{5.29} = +68.2°$ (probably bact. growth)

JUN 3 — 6.8g. dis. in H_2O, bone-blacked, & crystd. as before, crysts. forming spont. in [aq. syrup before HOAc added. Yield: 5.48.

JUN 3 — 1g. let stand in warm place w. 1:1 HNO_3. After few hrs. crysts. began to sep. JUN 4 — Let stand in cold, filtered, crysts. washed w. H_2O, recrystd. from H_2O, Yield: 0.26g. m., decompo. 214-5° From various M.Ls, additional 0.2 g., m & decomp. 214-5°

JUN 1 Filtrate from 1 "crop galactose + vacuum concentrate of alc. washings, warmed treated w. HOAc until sl. permanent turbidity, filtered through folded filter, again seeded w. galactose. No crystn., so concd. to sm. bulk in vac., taken down again w. H_2O, again treated w. HOAc & seeded. No crysts. JUN 3 — Concd. repeatedly in vac. w. H_2O until little more HOAc evident. JUN 4 — Taken up in hot H_2O, bone-blacked. & at vol. of ca. 350cc. again treated w. xs. basic PbOAc. Heavy ppt. after 1½ hrs. in ice-box centfgd., & filt. IB and ppt. IIA sepd. IB treated w. H_2S, ppt. washed w. H_2S-H_2O, filt. concd. in vac. to syrup, concd. again several times to remove HOAc, flask rinsed w. a little H_2O & syrup treated w. redistd. alc. until sl. perm. turb. Seeded w. galactose, as orcin test still gave brown color. JUN 7 — No crysts. Concd. 2x in vac., taken up in H_2O, bone-blacked, made up to 150cc.

JUN 8 rff. Macro-Schaffer - on 1cc.

Furfural Estn. on 4cc. Bl. 49.6 Rdg. 21.6 $\frac{28.0 \times 1.02 \times 6.36}{2.17} = 8.4$g. sugar

9.0cc. N/10 $KBrO_3$ = 0.0675g. pentose in 224mg. sugar = 30% pentose

Main portion neutrd. w. $Ba(OH)_2$, concd. in vac. to thick syrup, b. out w. 3 successive 190cc. portions 90% alc. Residue IC. Soln. ID. ID portions combined, bone-blacked to remove sm. amt. suspended matter, filtered hot. Sm. amt. gummy material sepg. out on cooling filtered off & soln. concd. in vac. to thick syrup. On seeding w. few

10 cc. dild. F soln. dil. to 20. 1 cc. taken.

20.40

Rdgs: 14.55 14.50 14.40

5.90 x .948 x .318 = 1.78 mg. Cu = 0.85 mg. glucose

.85 x 40 x 50 = 1.7 g. sugar in total soln.

galactose crystals, crystn. started slowly. To hasten this few drops HOAc added & syrup warmed, crystg. very rapidly. Transferred to a dish w. min. amt. hot H_2O, htd. on H_2O bath until only few crysts remained, few drops more HOAc added & set in ice-box. JUN 16 — Solid cake broken up. JUN 17 — ~~Broken up, washed~~ Filtered off on Buchner in cold, washed 1" w. little chilled 50% HOAc, then little 75% HOAc, (filtrate E) dried in vac. over $CaCl_2$ + NaOH. Yield: 2.9 g. JUN 18 — Diss. in a little hot H_2O, bone-blacked, concd. to syrup in vac., ca 2 vols. HOAc added, crystn. commencing quickly. Left over night in ice-box, sucked off, washed w. chilled 2:1 HOAc-H_2O, alc., dried. Yield: 2.1 g.

I E

To E several vols HOAc added, seeded w. a little D, let stand in ice-box w. frequent stirring. JUN 21 — Filtered off, washed w. little 2:1 HOAc & dried. JUN 22 — Yield 0.4 g. Filtrate F, concd. repeatedly w. H_2O in vac, bone-blacked, concd. again, made up in H_2O to 50 cc. 1 cc. dil. to 20, 1 cc. taken.

20.4
4.85 5.35

20.4
5.1
15.3 × .948 × .318 = 4.61 mg. Cu

I C

JUN 17 — Taken up in hot H_2O, let cool, dild. to ca. 75 cc., few drops PbOAc added, & sm. chocolate brown ppt. centfgd. off. Supnt. pptd. w. xs. PbOAc & let stand in ice-box. After 2 hrs. centfgd., ppt. taken up in H_2O, decompd. w. H_2S, let stand in cold. JUN 18 — PbS filtered off, washed w. H_2O, soln. concd. repeatedly in vac., taken up in H_2O, bone-blacked, concd. to syrup, dried in vac. Yield: 2 g. Discarded.

I F

JUN 23 — 5 cc. soln. used for furfural estn. No red color in distillate w. $PhNH_2 \cdot HCl$ paper, but used 18.5 cc. N/10 $KBrO_3$.

Repetition of rotation

Wt. bot + subst. (const). 7.5678

7.1080

.4598

INITIAL $l = 1$ Bl. = −0.04 .05 .05 Rdgs. +5.53 .51

$$[\alpha]_D = \frac{558}{4598} = 121.3°$$

FINAL

0.2 cc. aq. NH_3

Rdgs. 3.10 .14 .15 .13

$$[\alpha]_D = \frac{318}{4598} \times \frac{102}{100} = +70.6$$

Hydrolysis

S 51E

JUN 8 — Dist. bot (10) + recrystd. galactose 7.6926 7.6922
7.1409
.5513

Dis. in H_2O, made up to 10cc.

OPTICAL ROTATION INITIAL

$l = 1$ Bl. +0.06 .06 Rdgs: +6.61 .55 .47 .46 .37 .31

$$[\alpha]_D = \frac{655}{5.513} = +118.8°$$

Drop conc. NH_4OH added, rdgs. taken hourly until const.

FINAL

$l = 1$ Bl. +0.01 .01 Rdgs.: 4.03 .06 .06

$$[\alpha]_D = \frac{405}{5.513} = +73.5°$$

C + H (Gartmann)

Wt. subst. 7.2152
7.1145
.1007

$CaCl_2$ 58.1156
58.0536
.0620

CO_2 54.8729
54.7284
.1445
.0035
.1480

Guard 63.0714
63.0679
.0035

.43573 04884
.17026 79239
99697 99697
.60296 .83820

C = 40.08%
H = 6.89%
found.

72.
12.10
96
180.10

85733 08279
25551 25551
60182 82728

C = 39.98% (theory)
H = 6.72%

Mucic Acid from Orig. Crop Galactose

C + H (Gartmann)

Subst. 7.4262
7.3253
.1009

$CaCl_2$ 58.1139
58.0681
.0458

CO_2 48.4808
48.3574
.1234
.0045
.1279

Guard 63.0782
63.0737
.0045

43573 04884
10687 66087
99611 99611
53871 70582

C = 34.57%
H = 5.08%
found

C = 34.27% (theory)
H = 4.80%

HYDROLYSIS

JUN 21 S 51 E I - D.

Wt. bot (E) + recrystd. sugar 8.1348 8.1346
7.5824
.5522

Diss. in H_2O, made up to 10 cc., readings taken as quickly as possible.

OPTICAL ROTATION INITIAL

$l = 1$ Bl. -0.02 .02 Rdgs: +7.50 .45 .36 .26

$$[\alpha]_D = \frac{752}{5.522} = +136.2°$$

0.5 cc. conc. NH_3 added, mixed

FINAL

Rdgs: 4.09 4.09

$$[\alpha]_D = \frac{4.11}{5522} \times \frac{105}{100} = +78.2°$$

JUN 23 FURFURAL ESTN

Bot + subst 7.5827
" 7.2483
.3344

Distillate gave no red color w. aniline HCl test paper

I E

Wt. bot (Z) + sugar 8.9580 8.9578
8.5435
.4143

Diss. in H_2O, made up to 10 cc., readings as quick as possible.

JUN 23

INITIAL OPTICAL ROTATION

$l = 1$ Bl. - -0.06 .06 Rdgs. + 5.45 .41 .36

$$[\alpha]_D = \frac{551}{4143} = +132.9°$$

0.2 cc. concd. aq. NH_3 added

FINAL $l = 1$ Bl.: same Rdgs: +3.05 3.07 3.05

$$[\alpha]_D = \frac{311}{4143} \times \frac{102}{100} = +76.6°$$

Hydrolysis

S 51E II - Acid Fraction

MAY 28 — II Suspd. in ca. 200 cc. H_2O, satd. w. H_2S, let stand in ice-box over night. Filtered off, washed thoroughly w. H_2S-H_2O, concd. in vac. to syrup, taken up in H_2O, boiled w. boneblack, filtered, & still yellow soln. concd. to sm. bulk in vac. Dried in high vac. at room temp. over P_2O_5. Yield: 5.5 g. Portion dried further

Bot. weighing bot. + subst 16.5265 .....35 ...10 ....03 ....184 @ t.b. $CHCl_3$ ...39 ..5074 ... 02
" " " 15.8058 16.4987 ... 58 ... 38 16.4925 16.4914
16.1585
.3329

JUN 4 & 5 — IIA suspd. in ca 200 cc. H_2O, pptd. w. H_2S, let stand in cold. Filtered, washed, filt., concd. to syrup repeatedly, repptd. w. basic Pb Ac & after 1 hr. in ice-box supnt., after de-leading, added to IB before final concns. Ppt. suspd. in H_2O, pptd. w. H_2S. JUN 7 — Filtered, washed, concd. repeatedly in vac., finally dried in high vacuum. Yield: 3.9 g.

JUN 17 — Ca 3.5 g. diss. in H_2O, treated w. morpline until no more dissolved, filtered, cryst. washed, filt. concd. to sm. bulk in vac., transf. to dish, dried.

JUN 3 — 1 g. crude acid diss. in 20 cc. H_2O (purplish blue to Congo), neutrd. to litmus w. $Ba(OH)_2$, filtered from trace brownish ppt., pptd. w. alc. JUN 4 — Ppt. taken up w. warm H_2O, treated hot w. alc. to incipient turbidity (very little necessary), bone-blacked below b.p., filtered, let stand in ice-box.

JUN 10 — Did not cryst., so pptd. w. more alc. Including this, ca. 5 g. II fraction diss. in ca. 25 cc. H_2O, chilled in ice-H_2O, & satd. $Ba(OH)_2$ soln. added, w. cooling, until no further ppt. Centfgd. in cold & ppt. washed w. 25 cc. chilled half-satd. $Ba(OH)_2$ soln. Ppt. II_1 & supnt. II_2 (filtered from a little ppt. sepg. on standing & adding more $Ba(OH)_2$) both quant. freed from Ba w. H_2SO_4, concd. to dryness in vac.

JUN 14 II_1 : 1.3 g. II_2 2.0 g.

Hydrolysis

S 51 E II

JUN 8 — Wt. bot + dried crude acid, 1st fract. 16.4914
16.1585
.3329

Diss. in H_2O, made up to 15 cc.

OPTICAL ROTATION

$l = 2$ Bl. = −0.01 +0.01 −0.02 −0.01 Rdg. +0.35 .39 .39 .38 ✓

$$[\alpha]_D = \frac{39}{2 \times .3329} \times \frac{15}{100} = +8.8^\circ$$

$[\alpha]_D$ +8.8°

ACID EQUIV. 1 cc. samples

cc. N/50 NaOH (x1.011) w. methyl red 2.32 w. phth. 2.59
.0222 g. N = .047 cc. 1 2.35 N = .052 2.62
Eq. = 473 Eq. = 427

Redg. Power 1 cc. dil. to 10, 1 cc. for anal.

Bl. 20.40
13.75 13.40 ✓
7.00 × .948 × .318 = 2.11 mg. Cu = 0.99 mg. glucose
2.22 mg. in sample

Redg. Sugars 44.6%

JUN 10 — ROTATION after 2 days

$l = 1$ Bl. = +0.02 .02 Rdg. +0.22 .26 .25 .25

$$[\alpha]_D = \frac{+23}{3329} \times \frac{15}{100} = +10.4^\circ$$

II A

JUN 10 Wt. bot (61) + subst 14.8777 ... 33 ... 13 14.8712
14.5648
.3064

Diss. in H_2O, made up to 15 cc.

OPTICAL ROTATION

$l = 2$ Bl. = +0.04 .03 .04 Rdg. − 0.13 .13

$$[\alpha]_D = \frac{-17}{2 \times .3064} \times \frac{15}{100} = -4.2^\circ$$

ACID EQUIV. 2 cc. samples

cc. N/50 NaOH (x1.011) w. phenol red 4.48 4.53 cc.
.0400 g. N = 0.091 cc. Equiv. 450

Redg. Power

1 cc. dil. to 10 cc. 1.5 cc. samples

Bl. 20.40 20.40
— Lost 11.75
8.65 × .948 × .318 = 2.61 mg. Cu
= 1.20 mg. glucose

3.064 mg. in sample

Redg. Sugars 39.2%

Hydrolysis

S 51E

II_1

Tot. bot B + subst 11.0045 .... 24 .... 11.0008
Diss. in H_2O, 10.7858
made up to 15 cc .2150

OPTICAL ROTATION

$l = 2$ Bl. = 0.04 .01 .02 ✓ Rdgs. +0.19 .20 .19

$$[\alpha]_D = \frac{21}{2 \times .2150} \times \frac{15}{100} = +7.3°$$

ACID EQUIV. 3cc. sample Phenol red

N/50 NaOH (x1.011) 4.82 cc. 4.87 N: .097cc for 0.043g. Acid Eq: 443

Redg Sugars
1cc. dil. to 10. 2cc. samples

Goebel) 20.40 20.40
14.07 14.12 14.10
6.3 x 0.948 x .318 = 1.9 mg. Cu = 0.9 mg. Cu 31%
2.87 mg. in sample

II_2

Tot. bot (57) + subst 12.9368 12.9344
12.6101
Diss in H_2O .3243
made up to 15cc.

OPTICAL ROTATION

$l = 2$ Bl. = +0.03 .05 .04 Rdgs. +0.14 .18 .16 .18

$$[\alpha]_D = \frac{13}{2 \times .3243} \times \frac{15}{100} = +3.0°$$

ACID EQUIV.

N/50 NaOH (x1.011) 6.33 6.40 N: 0.128cc. for 0.0649 g. Acid Eq: 507

Redg. Sugars
1cc. dil. to 10. 1.5cc. samples.

20.40 20.40
12.27 12.3 12.28
8.1 x .948 x .318 = 2.44 mg Cu = 1.12 mg. glucose 34.5%

HYDROLYSIS

JUN 21

S 5 E IC – Acid Rec. from Sugar Fract.

Wt. Cdr (57) + subst 13.2763 13.2756

12.7495

.5261

Diss. in H_2O, made up to 15 cc.

1 cc = 0.0351 g.

OPTICAL ROTATION

$l = 2$ Bl = −0.02 −.01 .03 Rdgs. −0.20 .21 .20

$$[\alpha]_D = -\frac{18}{5261 \times 2} \times \frac{15}{100} = -2.6°$$

ACID EQUIVALENT 3 cc. sample

Phenol red. cc N/50 NaOH (x1.011) 3.97 4.01

cc. N 0.08 for .1052 g.

Redg. Sugars 1 cc. dil. to 20 1 cc. samples. 1 cc = 0.00176

Bl. 20.4 20.40

16.65 16.55 16.60

3.80 x .9 x 48 x 0.318 = 1.15 mg. Cu

= 0.59 mg glucose 33.5%

S S S 51 B

Wt. bot (712) + subst. 16.6611 16.6579 16.6577

16.1403

Diss. in H_2O, made up to 25.85 cc. .5174

Ash: .0005

.5169

ASH

Wt. bot + subst	16.1406	Wt. Pt dish + ash	14.3145
" "	16.0340	" " "	14.3141
	.1066	" ash	.0004
		" Ca	.000118

$\boxed{\text{Ash: } 0.1\%}$

OPTICAL ROTATION

$\ell = 2$ Bl.: -.05 .04 .04 Rdgs -0.53 .51 .52 .52

$$[\alpha]_D = -\frac{48}{4} = -12^\circ$$

$\boxed{[\alpha]_D \; -12.0^\circ}$

Microkjeldahls

	①			②	
Air-Dry	5.0148			4.9826	
	4.9826			4.9500	
	.0322			.0326	
cc. N/70 HCl (8/1.025)	5.00	5.13		5.13	5.00
" N/50 NaOH (4/1.011)	3.35				3.40
	3.39 N/70	4.75	N/70	4.82	3.44
		.38		.31	
		.1		.1	
		.28×0.2		.21×0.02	
		= 0.056		= 0.042	

$\boxed{N = 0.2\% \; .15}$

N = 0.17% N = 0.13%

S S S 51 E

APR 8 — Wt. bot (A) + subst. in sac @ 100° over H_2SO_4 8.0244 8.0223
8.0207 8.0206
7.2577
.7629
Ash .0004
.7625

Diss. in H_2O, made up to 25.4 cc.

ASH

Wt. bot + subst 7.2577
" " 7.1060
.1517

Wt. Pt dish + ash 14.3123
" " " 14.3120
" $CaSO_4$ .0003
" Ca .0000 88

Factor .29435

Ash 0.06%

OPTICAL ROTATION

MAY 4 — l = 2 Bl = −0.07.05.07.06 Rdg. −0.69 .70 .70

$$[\alpha]_D = -\frac{64}{6} = -10.7^\circ$$

$[\alpha]_D$ −10.7°

ACID Equivalent

15 cc. soln. + drop phenolphth. req. 7.19 cc. N/4 NaOH (x1.025)
7.37 " " " = 0.526 cc. N.

Acid Equiv. 856

OPTICAL ROTATION of Na Salt

l = 2 Bl. = −0.06.06 Rdg. −0.64.64

$$[\alpha]_D = -\frac{58}{6} \times \frac{22.25}{1500} = -14.3^\circ$$

$[\alpha]_D$ −14.3°

MICROKJELDAHLS 1.5 cc. samples

cc. N/10 HCl (x1.025)	5.00	5.13	5.00
" " NaOH (x1.035)	4.60	4.79	4.65
		.34	
		.15	
		.19 x 0.2 = 0.038 mg. N	

N: 0.08%

FURFURAL Detn.

MAY 17 5.5 cc. soln. req. 4.0 cc. N/10 $KBrO_3$. ∝ 0.0192 g. furfural = 11.6%
0.0300 g. pentose = 18.2%

Pentose 18.2%

Redg. Sugars

1 cc. dil. to 3, 3 cc. 2N H_2SO_4 added, b 4 hrs.
Add to 20 cc. 1 cc. taken
Rdg. 13.0 12.80 Bl. 20.00
12.90
7.10 x 966 x .318 = 2.18 mg. Cu
= 1.02 mg. glucose
= 68%

Redg. Sugars 68%

SSS 51F

APR 8 – Wt. bot (Z) + subst. in vac at 100° over H_2SO_4 9.2600 9.2599
8.6321
.6278
Less Ash .0006
.6272

Dis. in H_2O, made up to 31.36 cc.

ASH

Wt. bot + subst 8.6321
" " 8.5437
.0884

Wt. Pt dish + ash 14.3120
" " " 14.3117
.0003
Wt Ca .000088

Ash 0.1%

OPTICAL ROTATION

MAY 4 – l = 2 Bl. = −0.02 04 03 03 Rdgs. −0.72 .73 .73

$$[\alpha] = -\frac{70}{4} = -17.5°$$

$[\alpha]_D$ −17.5°

Acid Equivalent

20 cc. soln. + drop phthalein req. 6.38 cc. N/14 NaOH (X1.025)
Total added vol. 6.50 (back titrn with phthalein). | = 6.54 cc. N/14 = .467 cc. N.

Acid Equiv. 857

OPTICAL ROTATION OF Na salt.

l = 2 Bl. = −0.04 .04 Rdgs. −0.70 .69 .69

$$[\alpha]_D = -\frac{65}{4} \times \frac{265}{200} = -21.5°$$

$[\alpha]_D$ Na −21.5°

Furfural Estn.

20 cc. above soln. req. 7.25 cc. N/10 $KBrO_3$

$\frac{200}{265} \times .4 = 0.3018$ g. gave 0.0348 g. furfural = 11.5%
.0544 g. pentose = 18.0%

MICROKJELDAHLS

2 cc. samples.

cc. N/70 HCl (X1.025)	5.00	5.13	5.00
" " NaOH (X1.035)	3.85	3.98	3.85
		1.15	
		.15	
		1.00 × .2 = 0.2 mg.	

40

N = 0.5%

Adsorption

SSS 54

JAN 21 1926 — 1 g. 47_{10}, lot 2, diss. in 200 cc. H_2O, 0.5 cc. HOAc added, then 10 cc. specially prepd. $Al(OH)_3$ $\alpha = 0.142$ g. Al_2O_3. Stirred 1 hr., let stand in ice-box overnight. JAN 22 — Centfgd. Supnt. (A) still ++. 10 cc. more $Al(OH)_3$ added, stirred 1 hr., again centfgd. 10 cc. more added, stirred 1 hr. centfgd.

$l = 2$ Bl. = -.06 .06 .04 .04 Rdgs. -.10 .09 .08 .09

Supnt.: $[\alpha]_D = -4 \times \frac{23}{20} = -4.6°$

According to rotation, $\frac{46}{113}$ = 41% still in soln.

Al_2O_3 ppt. washed 1" w. H_2O, ca. 50 cc., still reacting acid & washings added to A JAN 23 — Ppt. B extd. 1 hr. on shaking machine w. 1% aq. Na_2CO_3, centfgd. Extn. repeated, exts. neutr. w. HOAc.

A concd. to sm. bulk, centfgd. from traces $Al(OH)_3$.

JAN 25 eff. — Few cc. 1:1 HCl added, pptd. w. redistd., chilled alc. Centfgd. Taken up in a little H_2O, centfgd., repptd. as before, washed, dried.

B exts. concd. ~~[illegible]~~ in vac., taken up in H_2O, centfgd., copious $Al(OH)_3$ ppt. washed w. H_2O, soln. + HCl pptd. w. alc. Ppt. taken up in H_2O, centfgd., repptd. as before. Taken up in a little H_2O, centfgd. from sl. turb., pptd. by stirring into 10-20 vols. redistd. acetone. Washed w. this & dried over $CaCl_2$ + crushed NaOH in vac.

A: 0.41 g. Both active 1:4,000,000.

B: 0.21 g.

∴ No important fractionation as regards activity

FEB 9 — Repeated w. 10 g. 47_{10} B IV, using 2000 cc. H_2O, 5.0 cc. HOAc, & 400 cc. $Al(OH)_3$ susp. Stirred ca. 4 hrs. FEB 10 — Mixt. centfgd. & $Al(OH)_3$ washed 1" w. 100 cc. H_2O. Supnt. & washings (A), ppt. (B). FEB 11 eff. — A concd. to sm. bulk in vac. B shaken ca 3 hrs. w. 200 cc. 1% Na_2CO_3, centfgd., supnt. acidified w. HOAc. Ppt. extd. again, then 4 × w. 2% Na_2CO_3, neutrg. exts. each time w. HOAc. Exts combined, concd. in vac.

FEB 15 eff. — A centfgd., 1:1 HCl added, & ~~[illegible]~~ vol. of ca. 150 cc., pptd. w. ca. 3 vols. chilled alc. After several hrs. in cold, ppt. ~~taken up in H~~ drained, taken up in H_2O, centfgd. FEB 16 — At vol. of ca 100 cc., few cc. 1:1 HCl added, pptd. w. ca 300 cc. chilled redistd. alc. After 1 hr. centfgd., ppt. diss. in ca 35 cc. H_2O, ca 5 cc. alc. added, stirred, & centfgd. Supnt. pptd. w. alc., isolated as usual. FEB 18 — B centfgd. again from deposited $Al(OH)_3$, concd. to sm. bulk in vac., worked up as in A. Both fractions isolated as usual.

A: 2.35 g. = (C)

B: 3.0 g. = (D)

S S S 54A

Wt. bot (8) + subst 7.6462 7.6454 7.6396 7.6381 7.6366
7.6364
7.3293
.3071
.0001
.3070

Acid Equiv.

Reqs. 4.45 cc. N/14 NaOH (x1.022)
4.55 cc. = .325 cc N NaOH

Less Ash

Made up to 15.35 cc. Centfgd.

Acid Equiv 945

ASH

Wt. bot + subst 7.3293
7.2405
.0888

Factor .2985

Wt. bot. + dish + ash 14.3139
" " 14.3137
.0002
Wt. $Al_2(SO_4)_3$ Wt. Al_2O_3 .00006
" Al .00003

Ash 0.03%

FEB 6

OPTICAL ROTATION

l = 2 Bl. −0.04 .04 Rdgs. −1.00 0.99 1.00

$$[\alpha]_D = -\frac{96}{4} = -24^\circ$$

$[\alpha]_D$ = −24°

S S S 54C

MAR 3 — Wt. bot. (8) + subst. in vac at 100° over H_2SO_4 8.0081 8.0073 8.0066
7.3111
.6955
.0003
.6952

Diss. in H_2O, made up to 25 cc.

ASH

Wt. bot + subst 7.3111
" " 7.2360
.0751

Wt. bot. + dish + ash 14.3128
" " 14.3126
.0002
" Al .00003

Ash 0.04%

OPTICAL ROTATION

l = 2 Bl. = −0.05 .05 Rdgs. −0.71 .70 .69 .71

$$[\alpha]_D = \frac{-65}{2 \times .6952 \times 4} = -11.7^\circ$$

$[\alpha]_D$ Acid −11.7°

Na salt.

l = 2 Bl. = −0.06 .06 Rdgs. −0.70 67 67 68

$$[\alpha]_D = -\frac{62}{8 \times .6952} \times \frac{72}{515} = -15.6^\circ$$

$[\alpha]_D$ Salt −15.6°

Acid Equivalent

15 cc. titrd. w. N/14 (x 1.022) NaOH 5.98 cc. = ~~0.437 cc~~ N NaOH
0.4192 g 6.11 cc = 0.436 cc N NaOH

Acid Equiv. 957

Furfural Estn.

20 cc. above estn. reqs. 9.0 cc. N/10 $KBrO_3$

$$\frac{20}{7} \times \frac{15}{25} \times .6952 = 0.3972 \text{ mg.}$$

0.0432 g. furfural = 10.9%
.0675 g. pentose = 17.0%

Pentose 17.0%

54 B

Microkjeldahls 2 cc. soln.

cc. N/70 HCl (x1.025)	5.50	5.13	5.00
" N/50 NaOH (x1.011)	3.36		3.35
	3.40 N/70	4.76	
		.37	
		.1	

.27 x 0.2 = 0.054

N = 0.3%

54 D

MICROKJELDAHLS 1.5 cc. soln.

cc. N/70 HCl (x1.025)	5.00	5.13	5.00
" " NaOH (x1.035)	4.58	4.75	4.59
		.38	
		.1	

0.19%

.28 x 0.2 x 10/15 = .037 mg. N in 20 mg.

N = 0.2%

HYDROLYSIS

3 cc. dild. to 6, 6 cc. 2N H_2SO_4 added, b. 4 hrs.
Neutrl., made up to 30 cc. = 1 cc. samples

Blank	20.00	20.00	20.00
	10.1	10.2	10.15

9.85 x .966 x .318 = 3.03
= 1.37 mg. glucose

68.5% redg. sug.

SSS 54B

Wt. bot (H) + subst 8.3981 8.3978 8.3964 8.3964
8.2386
.1578
Less Ash .0003
.1575

Acid Equiv. 697

Acid Equiv.
Reqs. 3.10 cc. N/14 NaOH (x1.022)
3.17cc. = 0.226cc. N

Made up to 15.75 cc. Centfgd.

ASH

Wt. bot + subst 8.2386
" " 8.1771
.0615

Factor 0.5303

Wt. dish + ash 14.3137 ✓
" " 14.3131
" $Al_2(SO_4)_3$ .0006
" Al_2O_3 .00018
" Al .0001

Ash 0.16%

FEB 5

OPTICAL ROTATION

l = 2 Bl. −0.02 .03 .03 Rdgs. −0.55 .53 .54 .52

$$[\alpha]_D = -\frac{51}{2} = -25.5°$$

$[\alpha]_D$ −25.5°

SSS 54 D

MAR 3 – Wt. bot ( ) + subst. in vac at 100 over H_2SO_4 9.6937 9.6922 9.6896

Diss. in H_2O, made up to 51.64cc.

9.6889
8.6559
1.0330

1.0330
.0003
1.0327

Wt. bot + subst 8.6559
" " 8.5499
.1060

Wt. dish + ash 14.3130
" " 14.3128
.0002
Wt. Al .00003

OPTICAL ROTATION

l = 2 Bl. = −0.05 .06 .05 Rdgs. −0.52 .51 .51

$$[\alpha]_D = -\frac{46}{4} = -11.5°$$

Ash 0.03%

Na salt l = 2 Bl = −0.05 07.06 Rdgs. −0.56 .54 .53 .53

$$[\alpha]_D = -\frac{48}{4} \times \frac{27.6}{20} = -16.6$$

$[\alpha]_D$ Acid −11.5°

$[\alpha]_D$ Salt −16.6°

Acid Equivalent

20 cc. reqs. 7.58 cc. N/14 NaOH (x1.022) = 7.75 = 0.553 cc. N NaOH

Acid Equiv. 723

Furfural Estn.

25 cc. above soln. reqs. 8.5 cc. N/10 $KBrO_3$

$$\frac{25}{27.6} \times .400 = 0.3623 \text{ mg.}$$

0.0408 g. furfural = 11.3%
0.0638 " pentose = 17.6%

Pentose 17.6%

HYDROLYSIS

3 cc. dil. to 6, 6 cc. 2N H_2SO_4 added B 4¼ hrs.
made up to 30 cc.

1 cc. samples

20.00	20.00	20.00
10.30	10.15	10.20

9.80 × .966 × .318 = 3.01 mg. Cu
1.36 mg glucose

Red, Sug.
68%

SSS 56

MAR 4 1926 — 200g. same G. A. diss. in 1 l. cold 1:1 HCl, let stand 2 days.
MAR 6 — Centfgd., pptd. w. ca 3 vols. chilled alc. After several hrs. this poured off, fresh alc. put on. MAR 8 rff. Rediss. in H_2O, pptd. w. redistd. alcohol. Let stand in ice-box. Supnt. decanted, fresh acetone put on. MAR 12 — Product ground up, filtered off, washed w. acetone, dried in vac. Yield: 70 g.

Wt. bot (8) + subst. in vac at 100° over H_2SO_4 8.9695 8.9618 8.9595 8.9580 8.9564 8.9550 8.9536 8.9529

Diss. in H_2O, made up to 55.8 cc.

	7.8354
	1.1175
Ash	.0015
	1.1160

Ash
0.013%

ASH

Wt. bot + subst	7.8354	Wt. dish + ash	14.3129
" "	7.6453	" "	14.3129
	.1901	" "	14.3124
		" $CaSO_4$	.0008 (factor 0.29435)
		" Ca	.00024

OPTICAL ROTATION

l = 2 Bl. −0.06 .08 .07 .08 .08 Rdg. −0.38 .38

$$[\alpha]_D = -\frac{30}{4} = -7.5°$$

$[\alpha]_D$
−7.5°

Acid Equivalent

20 cc. soln. req. 6.31 cc. N/14 NaOH (x1.025)
6.47 cc. = 0.462 cc. N NaOH

Acid Equiv.
866

OPTICAL ROTATION of Salt

l = 2 Bl. −0.04 .04 Rdg. −0.42 .42

$$[\alpha]_D = -\frac{38}{4} \times \frac{26.35}{20} = -12.5°$$

$[\alpha]_D$ Salt
−12.5°

MICROKJELDAHLS

2 cc. samples
5 cc. acid.

cc N/10 HCl (x1.025)	5.00	5.13	5.00
" " NaOH (x1.035)	4.31	4.48	4.35
		.65	
		.1	

.55 X .2 = .11 mg. N in 40 mg.

N =
0.33%

Furfural Detn.

25 cc. above soln. used. 9.5 cc. N/10 $KBrO_3$.

$\frac{25}{26.35} \times .4 = .3795$

0.0456 g. furfural
0.0713 g. pentose

Pentose
18.8%

SSS 56 A

OCT 15 1926 — Ca. 69 g. 56 diss. in 350 cc. H_2O & pptd. w. 625 cc. HOAc. OCT 16 off. Supnt. decanted off from ppt. (B) and further pptd. w. HOAc (A). (B) diss. in ca 300 cc. H_2O, ~~pptd. w. HOAc~~, redissd. in ca 150 cc H_2O, ~~pptd. w. redistd. alc.~~ HOAc added to initial turbidity, and ppt. (B) centfgd. off. Supnt. pptd. w. more HOAc until further addition produced no more ppt. Ppt. (C). diss. in ca. 150 cc. H_2O, ~~Ppt. diss. in H_2O~~ pptd. w. redistd. alc. + disp HCl. A & B treated same. All rediss. in H_2O, pptd. w. redistd. acetone.

SSS 56 B & C. HYDROLYSIS

NOV 22 — 5.2 g. B and 51.8 g. C, 57 g. in all (desic. dried), diss. in 3 l. H_2O, 85 cc. concd. H_2SO_4 added, & B. 4 hrs. Cooled, & slightly less than calcd. amt. $Ba(OH)_2$ added in hot aqu. soln. NOV 23 off. Supnt. ~~decanted~~ siphoned off, ppt. centfgd. & washed w. H_2O. 2" washing only feebly reducing, so not washed 3rd time. Supnt. & washings concd. in vac. to ca. 600 cc., b. w. bone-black, ~~filtered through~~ asbestos filter, & concd. further in vac. to ca 200 cc. NOV 29 — Basic Pb acetate added, but nec. to add large amt. before pptn. commenced. Soln. 1" supersatd., pptg. sepg. rapidly after large xs. PbOAc present. Vol. 750 cc. Centfgd. after 2 hrs. in the cold & reddish supnt. (I) & pinkish ppt. (II), latter suspd. in H_2O, sätd. sep. w. H_2S. NOV 30 — Each centfgd., ppts. washed 3X w. H_2S-H_2O, when only small amts. redg. sugars left. Solns. concd. to sm. bulk in vac. & evapd. 2X more w. H_2O to remove xs. HOAc. II, at vol. of ca. 75 cc., pptd. w. basic Pb acetate in cold, final vol. being 250 cc., although addn. of another = vol. PbOAc would have brought down more. After 1 hrs. mixt centfgd. & ppt (II) & supnt (I a) separately treated w. H_2S & PbS washed until little redg. sugars left. Solns. concd. in vac., repeating to remove HOAc. I a combined with I. ~~II pptd~~ DEC 2nd — II pptd. 1" more w. PbOAc in xs. discarding a 1" yellow fraction which came out on the acid side. Supnt. from main ppt. contained fract. no redg. sugars, so discarded. Ppt. deleaded as usual, evapd. down in vac., H_2O added, concd., & this repeated 1" more. DEC 3rd — I, at vol. of ca. 75 cc. treated w. PbOAc. ~~in cold~~. Necessary to add ca 700 cc. soln. before ppt. began to form. Centfgd. off after 1 hr. in cold & ppt. (III) & supnt (I) deleaded & worked up separately as usual.

I - Sugar Fraction

SSS 56 B & C HYDROLYSIS

DEC 6 '27 - I taken up in H_2O, bone-blacked, but color deepened. Concd. in vac. to syrup, 0.5 vol. HOAc added, seeded w. galactose. Sugar slowly crystd. After several days, finally in ice-box, sucked off & washed w. little chilled 1:1 H_2O-HOAc. Filtrate (Ia). Cryst. diss'd. in H_2O, bone-blacked, soln. concd., HOAc added, seeded, let stand in ice-box. JAN 11 1927 - Filtered off, washed w. a little HOAc, recrystd. as before.

I a

S S S 56 B & C Hydrolysis

JAN 3 1927 & ff. I a repeatedly concd. in vac w. H_2O to eliminate HOAc, dil'd. w. H_2O to ca 300 cc., bone-blacked, concd. in vac. to sm. bulk & chilled. Pptd. w. ca 2 vols. PbOAc (I a), supnt. pptd. w. l. excess. (I b). Both ppts. worked up as usual & reppt'd. On 2nd pptn I b gave very little, so added to I a, deleaded & evapd. dry as usual. Yield: 1.4 g.

Wt. bot (E) + subst in vac at t. b. $CHCl_3$	7.5900	7.5992
		7.2710
Made up to 10 cc.	.3282	.3282
	.0171	~~.0085~~
	.3111	.3197 Ash-free

ASH

Ba from poor Barie PbAc	Wt. bot + subst	7.2710	Wt. crux + ash 6.7767
	" "	7.2481	" " 6.7747
		.0229	" $BaSO_4$ × .5884 .0020
JAN 14			" Ba .0012

Ash 5.2%

OPTICAL ROTATION

$l = 1$ Rl. = −0.05 .06 .05 Rdgs. + 0.14 .14

$$[\alpha]_D = \frac{19}{.3197} \times \frac{10}{100} = +6.0^\circ$$

Redg. Sugars 0.5 g. dil. to 10, 2 cc. sample

Rl. 19.10
Rdg. 10.60 10.60
8.5 × 1.012 × .318 = 2.73 mg. Cu = 1.31 mg. glucose

Redg Sugars 42%

JAN 18 – III a & I a combined, ca. 3.8 g., freed from Ba w. H_2SO_4 & supnt. b. w. $CaCO_3$, bone-blacked, filt. concd. to sm. bulk in vac., fractionated w. abs. alc. to remove 1st dark portions of the salt.