S.S.S. 36 C

DEC 12 — ~~Ssln~~ filtrate from main batch centfgd. clear, concd. in vac. to dryness.

Wt. dish + subst. 23.4588 — 23.4626 < after 1 day @ 110 05

" ~~subst~~ dish 23.4005

.0583

DEC 22 — Diss. in H_2O + xs NaOH, neutr., made up to 11.66 cc., centfgd. from trace insol. matter.

OPTICAL ROTATION

l = 1. α = 0, 0. Rdgs.: 1.14 1.16 1.17 ✓

$[\alpha]_D = +234°$

$[\alpha]_D = +234°$

Microkjeldahls on 1 cc.

1N/50 HCl (x1.001)	5.40	5.40	5.00	5.00	Mean: 1.02
" NaOH (x1.015)	4.28	4.34	3.96	4.02	Bl. .22
		1.06		.98	.80

Blanks.

1N/50 HCl (x1.001)	5.20	5.20	Mean:	5.20
" NaOH (x1.015)	4.92	4.89		4.98
				.22

N = .8 x .28 = .224 mg. N in 5 mg.

N = 4.5%

HYDROLYSIS

DEC 29 — 2 cc. dil. to 4, 4 cc. 2N HCl added, b. 7 hrs. overnight in ice-b

DEC 30 — Neutr. w. $NaHCO_3$. 2 cc. samples used for anal.

	I	II	Bl.
cc. $Na_2S_2O_3$	16.48	16.30	20.04
	Mean: 16.39		16.39
			3.65

3.65 x .981 x .318 = 1.14 mg. Cu_2 = 0.6 mg. glue.

Redg. Sugars: 24%

JAN 2 1925 — Hydrol. soln., acidif. w. N HCl, gives brownish ppt w. HPW

Specificity of Original Soln: 1: 3,000,000

S S 37

	Tag Nos.	Tag dates	No Flasks	Precip	Ster
7/9/24	1/188/38	VI - 27	11	7/10	7/11
7/10	1/188/38	VI - 27, 30	4, 6	7/11	7/12
7/14	1/188, 193/384	VI - 3, VII - 8	2, 8	7/15	7/16
7/15	1/193/4	VII - 8	11	7/16	7/17
7/22	1/193/4	VII - 11	9	7/23	7/24
7/23	1/193/4	VII - 11	8	7/24	7/25
7/24	1/193/4	VII - 11	8	7/25	7/26
9/9	1/193/5	VIII - 26	10	9/10	9/11
9/10	1/193/5	VIII - 26	9	9/11	9/12
9/19	1/193/6	IX - 12	10	9/20	9/22
9/23	1/193/6	IX - 12	10 106	9/24	9/26
9/30	1/193/7	IX - 23	10	10/1	10/2
10/1	1/193/7	IX - 23	10	10/3	10/4
10/14/24	1/193/8	X - 6	8	10/15	10/16
10/16/24	1/193/8	X - 6	14	10/17	10/18
10/17/24	1/193/8	X - 6	14 142	10/18	10/20
10/20/24	1/193/8, 9	X - 6, 7	1, 13	10/21	10/22
10/21/24	1/193/9	X - 7	14	10/22	10/23
10/22	1/193/9, 11	X - 7, 15	10, 2	10/23	10/24
10/23	1/193/12	X - 15, 22	12, 2	10/24	10/25

(then HOAc and 1:1 HCl added until max. ppt. formed / 216 flasks
Centfgd., ppt. washed 1" w. H_2O. at
OCT 27 1924 - Treated as 35A, vol. = 5500 l.
Ppt'd. w. ca 7 l. alc. Supernatant -, but 1 l. more
alc added to make sure. OCT 28 - Supernatant
2 siphoned off, ppt. centfgd., taken up again in
H_2O, centfgd., ppt. washed w. H_2O. Soln., at
3 500 cc. vol., ppt'd. w. 400 cc. alc. & let stand
over night in ice-box. OCT 30 - Supernatant, -, siphoned off, ppt.

324 liters

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centfgd. in cold. OCT 31 aft. – Rediss. in ca. 500 cc. H_2O, insol. portion
4 repeatedly centfgd. Main soln, at vol. of 600 cc., pptd. w. 400 cc. alc.
Ppt., which remained fluid, ~~sold~~ taken up in little more H_2O & sald. w. $(NH_4)_2SO_4$
but ppt. in this case contained active material as well, so extd. several times w.
H_2O. Solns, dialyzed over night in running H_2O, concd., & pptd. w. alc. (2° fract. above)
~~active material & main ppt.~~ Main ppt., taken up in cold H_2O, stirred
~~thoroughly~~, dissolved only slightly, so soln., weakly +, added to
(38). Ppt. again homogenized with 2° fract. above & dild. to ca.
250 cc. Set stand on ice over night, centfgd. in cold until
light ppt. fairly solid. NOV 7 aft. – Decanted as well as possible, ppt. taken
5 up again in H_2O, repeatedly centfgd., solns. combined. NOV 12 – Clear
HOAc but opalescent soln. pptd. w. HOAc to max. ppt. Set stand in ice-box.
NOV 13 aft. Centfgd. in cold. ~~Supernatant~~ –, discarded. Ppt. (2 portions)
washed, ea. portion w. 50 cc. cold N/2 HOAc. Centfgd. in cold. Supernatant
like previous one, gives strong purplish red w. I. Ppt. washed again w. 50 cc.
ea. N/2 HOAc. Supernatant –, but still red w. I. NOV 17 – Ea. washed
again w. 100 cc. N/2 HOAc, centfgd. in cold. NOV 18 – Supernatant –, now
gives only very faint color test for glycogen. Poured off, ea. washed
again w. 50 cc. N/2 HOAc. ~~Still~~ faint glycogen test after centfg.
NOV 19 – Ppts. transf. to 1 ~~flask~~, diss. by gradual addn. 10% NaOH
until faintly alk. Repeatedly centfgd. in cold, at vol. of ca.
6 200 cc. NOV 20 – Clear soln., to which em. amt. remaining of (35)
HOAc was added, pptd. by shaking w. HOAc in xs. Centfgd.
NOV 22 aft. – Supernatant, still somewhat active, at vol. of 125 cc.,
7 pptd. w. 50 cc. alc. & let stand in cold. Ppts. combined, diss. w.
N/10 NaOH alc sl. xs. NaOH, & at vol. of 125 cc., pptd. w. xs. alc. Supernatant, still
somewhat active, ~~neutr~~; concd. to sm. bulk, again made alk., ~~ppt~~ w.
~~xs~~ alc. added, but no ppt. Acetone then added, ppts salts & 2
liquid layers. S in salts. These diss. in H_2O & soln. added to that
of the main ppt. in H_2O. DEC 1 – At vol. of 150 cc., 5 g. NaOAc, 5 cc.
8 NaOH, alc. 10% NaOH added, then pptd. w. 200 cc. alc. DEC 2 – Mixt. centfgd.
DEC 3 – Supernatant –. Ppt. taken up in H_2O. Soln. still
yellow. 5 g. NaOAc added. At vol. of 250 cc., after acidification w HOAc,
9 pptd. w. 75 cc. alc. 5 acid came down in very voluminous, gelatinous
form. DEC 5 – Centfgd. Ppt. susp'd. in H_2O, diss'd. w. sl. xs. NaOH.
Neutr. again w. HCl, ~~&~~ 10 g. NaOAc added, at vol. of 250 cc. acidif. w.
10 HOAc HOAc. Heavy ppt. Set stand, centfgd. Supernatant –, poured
off, ppt. taken up in H_2O. DEC 9 – Diss. by making acid to Congo w.

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11 HCl. At vol. of 200 cc. ppt'd. in cold w. 500 cc. cold redistd. alc. Centfgd. in cold. DEC 10 — Supernatant ±, discarded. Ppt. taken up in H_2O + few drops N HCl. Still somewhat yellowish, so 25 cc. 1:1 HCl added, + at vol. of 200 cc., again ppt'd w. 500 cc. alc. Centfgd. + few drops N HCl. Supernatant −, discarded. DEC 11 — Ppt. diss. in ca. 200 cc. H_2O, incl. rinsings, rinsed into 2 collodion bags, dialyzed. agst. running H_2O, then agst. successive changes distd. H_2O + toluene in ice-box until no Cl^- inside or out. DEC 17 — Transf. to centfg. bottles + centfgd. repeatedly. Supernatant: 37A. Ppt. JAN 5 1925 taken up w. redistd. alc., centfgd., then washed 2X w. redistd. acetone. JAN 8 — filtered off, washed w. acetone, dried in warm vacuum desiccator. Yield: 3,2 g.

Active at 1:6,000,000 dilu. — at 1,8000,000.

At 1:400 gives sl. turbidity w. III serum.

Portion b. few min. w. naphthoresorcin + conc. HCl, boiled, dild., extd. w. Et_2O, gives violet color absorbing in yellow — evidence of glucuronic acid. Also gives this test after treatment w. HNO_2.

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Wt. bot + air-dry subst (1) 6.8209 — Dried in vac at T. R. $CHCl_3$.
" " 6.6126
.2083

6.8209 — Diss. w. few drops N HNO_3 + H_2O,
6.6995 — neutr. w. N NaOH until ppted. & redissolved, made up
.0214 — to 20 cc.

6.8015 6.8002 6.7998
6.6975
.1023

MICROKJELDAHLS

2 cc. samples

cc. N/50 HCl (1.001)	5.00	5.00	
cc. N/100 NaOH (1.014)	14.00	15.40	
	8.00	9.00	
	6.00	6.40	6.48
	10.01		10.01
	3.93	3.53	
	.44	.44	
	3.49	3.09	
	4.8% N	4.2% N	4.5% N

N 4.5%

OPTICAL ROTATION

Bl. −0.04 −0.04 −0.05 — Rdgs. +1.46 1.46 1.45 — l = 1

$\frac{1.50 \times 200}{1.023}$ $[\alpha]_D$ = + ~~300~~ 293.3°

$[\alpha]_D$ = +295.3° Ash-free

ASH

Wt. bot + subst 6.6979
" " 6.6899
.0580

Wt. dish + ash 23.4016
" " 23.4012
Wt. ash .0004

Diss. in N HCl, made alk. w. N NH₄OH, gave CaC_2O_4 w. $Na_2C_2O_4$.

Ash 0.7%

Acid Equivalent (Goebel)

2 cc. sample. 5 cc. $AgNO_3$ N/10 × 1.013
2 cc. of centfgd. soln. for titrn.
$\frac{2}{7} \times 25 \times 1.013$ = cc. N/5 $AgNO_3$ for sample = 7.24
cc. KCNS (F×1.03) 6.58 6.50 6.70
.54 cc. used up by 2.92 mg. subst.
= 0.0108 cc. N.

29.2 mg. of 108 cc.

Acid Equiv. 270

Acid Equiv 272 Ash-free

Jun 20 - Repetition of N

Wt. bot D + subst 7.4972 7.4606
7.4606 7.4224
.0366 .0382

cc. N/70 HCl (×.995) 15.00 15.00
in NaOH (×1.015) 5.45 5.175

14.92 14.92
5.53 5.25
9.39 9.67
.11 .11
9.28 9.56

mg. N	1.86	1.91
% N	5.08	5.00
Ash-free	5.12	5.04

N = 5.1% Ash-free

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Amino-N after different reaction periods.
3 cc. soln. dild. to 6. 2 cc. samples for anal.

Blanks.

Shaken 2 min., let stand 0.5 hr., shaken 3 min. $KMnO_4$ 1.5 min.

T	Bar.	Rdg.
22°	764.4	0.21

In other blanks see 37B, Dr. Goebel's book.

Same as above, but let stand 1 hr.

21°	764	0.31
22°	764	0.27

Detns.	½ hr.		−Bl.	Factor	
	23°	764	0.45	0.24	5640
1 hr.	24°	763.7	0.51	0.22	5610

$\frac{.24 \times 5640 \times 100}{1} = 1353$ mg. NH_2N per 100 cc orig. soln.

In 100 cc. .5115 mg.

$NH_2N = 2.6\%$

FEB 24 — In view of result obtained on hydr. of S. S. 39 w. 2N HNO_3, Acid Hydrolysis repeated w. N HNO_3.
3 cc. soln. dild. to 6, 6 cc. 2N HNO_3 added, hr. 4 hrs.

OPTICAL ROTATION

$l=1$	Bl.	+.04	.04	.01	.03	.03
	Rdg.	+.26	.25	.24	.26	

$$[\alpha]_D = \frac{.22 \times 100 \times 8}{1} = +176°$$

Redg. Sugars 2 cc. sample

Bl. 20.05 (Goebel

15.60

$4.45 \times .957 \times .318 = 1.35$ mg. glucose $= 0.68$ mg. glucose

27.2%

B 1 hr. longer

Bl.	+.04	.05	.03
Rdg.	+.21	.21	.22

$l=1 \cdot [\alpha]_D = \frac{.17 \times 100 \times 8}{\quad}$

$= +136°$

(Goebel) Bl. 20.05

16.35

$3.70 \times .957 \times .318$

Hydrolysis of SSSI.

Nov 11

0.5 gms of SSSI dried to const. wt. was dissolved in 50 cc of 75% (by wt) H_2SO_4. Solution was slow, taking about 24 hrs. Solution allowed to stand at room temp.

After 24 hrs 1 cc was removed diluted to 5 cc with water and optical rotation determined.

Time.	α	$[\alpha]_D$
24 hrs.	+.52	+260°
48 hrs.	+.38	+190°
72 hrs	+.13 (.5 dm tube)	+130°

$\frac{.52 \times 250}{.5 \times 1} = +260°$

quite dark.

$\frac{.13 \times 250}{.5 \times .5} = 130$

250 / .52 / 500 / 1250 / .5 | 130.00 / 260.00

Adsorption - Lyfor A undried Alumina

a - test was gotten in supernatants so the system was centrifuged, washed once, & recentrifuged. The washings gave a test. The Alumina was shaken with 500 c.c. N/5 Na_2CO_3 for two hours, centrifuged, washed twice, & reshaken with 500 cc more. The whole supernatants after the first and second extraction (which stood over night) the Na_2CO_3 was neutralized with with HCl and the whole was conc in vacuo, dialyzed in parchment until nearly free of chlorides, & conc. again and finally, after centrifuging away the small amount of Alumina which had in the meantime come out, placed in a collodion bag and dialyzed until free entirely of chlorides, concentrated and ppt from 15 c.c. vol with 50 c.c. 95% alc 1:1 at 0°. washed free of chlorides and with 95% alc & then with acetone. Dried in vacuo. .36 gm, or 72% recovery.

12/1 The supernatants from a third extraction with N/5 Na_2CO_3 gave a + test and were worked up exactly as above.

1/4/25 Adsorbtion on Lyfor I S.S. 37Ad

An experiment was tried in which 10 mg Lyfor I in 10 c.c. water were shaken with .1 g Alumina at PH 5. For a couple of hours. It was found after centrifuging that complete adsorbtion occurred. With a similar weight of Kaolin at PH 8 - after with phenol phthalein, no great amount of adsorbtion took place because a very strong test for S was obtained. The adsorbed stuff was shaken for a period of two hours with about 20 c.c. of half normal sodium carbonate, a small portion removed, centrifuged & tested for S.

1/5/25 .25 gm Lyfor I (prep 37) dissolved in water and diluted with .6 gm of Lyfor A undried Alumina. Worked up to PH 5. After shaking for 3 hrs no test for soluble. Test after standing overnight specific test gotten so .4 g more of Alumina were added. This also failed to bring all S out. PH taken & found to be 6.5 so it was made up to 4. again & after an hours shaking no S could be detected.

1/2/25 The ppt (after cent, was shaken overnight with 500 c.c. of 1/5 normal Na_2CO_3, cent & extracted twice with two lots of 1/5 normal 250 c.c. Carbonate each extraction Carbon 4 hours. These last two fractions were kept separate from first one. The Alumina was washed free of S & extracted overnight a fourth time with 300 c.c. N/5 Na_2CO_3

1/9 - The 1st Alumina extract was neut, conc, & dialyzed in running water. Conc again and dialyzed in dist water till Cl free.

1/28. The last free dialysis was conc to 15 c.c. vol in vacuo and the last S cent off. The water liquors contained a slight amount of S and were added to the 2nd & 3rd extract. The white ppt was washed with acetone & cent. repeated 3 times. Washed with acetone onto a paper & dried in vac. dessicator. Yield from first extract .17 gm.

2/2

Biological titration 1:4 hours 100

5.5.37 B

Analysis of Prep. Lypsi After Adsorption with Alumina.

First extract.

Bottle #1 + Sample	6.7500	Diluted to 16.32 c.c. giving a 1:200 Soln. Aliquot sent to lab was centrifuged for 1½ hrs in cent. centrifuge. A deposit resulted (probably alumina) and the specific rotation taken on this product.
	6.7496	
	6.7496	
" - Sample	6.6680	
	0.0816	
Ash	64	
	.0752	

① Rotation: Zero +.05 Alpha and Zero 1.37, 1.36, 1.37, 1.37

$\alpha = 1.32°$

$[\alpha]_D = 264°$

297

$[\alpha]_D^{20}$ +287 ash-free

Ash

Wt. bot + subst	6.6684	Wt. dish + ash	23.4058
" "	6.6155	" "	23.4016
	.0529		.0042

Ash = 7.9%

② Ammo Nitrogen

	Blanks	Temperature	Barometer (corrected)
	.14 c.c.	22°	771.7
	.14 "	"	
	.13 "	"	
Run 10 min	.20	25.5°	762

Blank + Ammo nitrogen from 2 c.c.c of the above sample in a 1:400 soln

	.34	22°	
	.34	"	"
Run 10 min	.48	25.5°	

c.c. Actual Ammo nitrogen = .20

Factor from table = .5230

$\frac{.20 \times .5730 \times 100}{1} = 11.46$ mg. NH_2 N per 100 c.c. = 500 mg. subst.

2.3

$NH_2N = 2.5\%$

$\frac{.28 \times .5550 \times 100}{1} = 15.54$ mg. NH_2 " " "

3.1

" " = 3.4%

③ Silver Equivalent: 2 c.c. Soln (1:200), 5 c.c. $AgNO_3$ (F = 1.03) cent. and 2 c.c. for titration, or ⅔ of the original.

	1	2	
c.c. KCNS (N/50 F: 1.03) =	6.45	6.55	Average = 6.50
Silver Equivalent	230		

Total nitrogen on 37 standard past. 2 c.c. of a 1:200 ovlen used for each sample

	1.	2.	Blank
c.c. N/50 (F=.998) HCl	5.00	5.00	5.00
" N/100 (F=1.014) NaOH	6.70	6.60	9.30
	6.65		

Blank = .27 c.c. of N/50 Acid.
Sample = 1.62 c.c. of N/50 Acid

Difference = 1.35 c.c. N/50 Acid neutralized

% nitrogen = $\frac{1.35/50 \times .014}{.01 \times 92}$ = 4.10% (ash free)

Reducing sugars with Nitrous Acid. ½ c.c. of a 1:200 solution of 37 Std. (2.5 mg) was pipetted into a sugar tube and 1 c.c. of V. Slyke via $NaNO_2$ added + 4 c.c. glacial acetic acid. Allowed to stand (together with a blank) for ½ hr. then 2½ c.c. of 1 N ~~HNO3~~ added and a blast of air blown ~~through~~ for 10 minutes. Neut with Na_2CO_3 + a regular sugar determ made on residue.

	1	2	Blank
c.c. Thiosulfate	15.35	15.05 } 15.20	20.05
Blank - " xF=.957	= 4.85		
Copper equivalent	1.148 mg		
glucose "	73		
% glucose	29.4% (ash free)		

31.7%
Ash free

S.S.S. 37

Acid Hydrolysis.

JAN 16 1925 — 2 g. finely pulverized substance added to 5 cc. conc. HCl in glass-stoppered flask. Formed a gelatinous mass which dissolved on standing over night. JAN 19 — Trace of C deposited from dark brown soln. All but faint traces S hydrolyzed. Set in ice-box. JAN 20 — No glucosamine HCl ppt. in spite of seeding, so dild. w. a little H_2O to ca. 10 cc. & again seeded. JAN 21 — Still no cryst., so dild. w. H_2O to 20 cc. 1 cc. of this soln. dild. to 10, ~~and fd~~ neutr. w. $NaHCO_3$, used for sugar & rotation. Centfgd. from dust. OPTICAL ROTATION

$l = 1$ Bl.: −0.01 +0.02 +0.02 +0.03 Rdgs.: +2.08 2.07 2.06

$$[\alpha]_D = +\frac{205}{.9 \times .993} = +229°$$

2 cc. dild. to 6. 1 cc. samples taken

Titration of Redg Sugars

$Na_2S_2O_3$ Blanks: 20.2 20.15 20.18 Soln: 16.50 16.35

$$\frac{16.42}{3.75} \times .957 \times .318 = 1.14 \text{ mg. Cu} = 0.6 \text{ mg. glucose}$$

18.1% to redg. sugars.

JAN 22 1 pt. — Remaining 19 cc. of the main soln. b. 2 hrs. to see if hydrol. complete. Apparently evolution of CO_2 on beginning b. Color changed to deep red, then deep brown. 1 g. $SnCl_2$ added, soln. dild. w. H_2O & in hope of eliminating color, H_2S passed through in xs. Very little lightening in color. Filtrate from Sn sulfides bld. on H_2O bath, filtered, then bld. w. bone-black, but w. little effect. Finally concd. in vac. to ca 10 cc. & centfgd. Supernatant A. Ppt. washed w. ca. 5 cc. 1:1 HCl, centfgd, & soln. added to A. Ppt. b. w. ca. 5 cc. H_2O, allowed to cool, centfgd. Soln. B. Ppt. C.

FEB 2 — A centfgd. from sl. amorph. brown ppt., concd. again in vac.

FEB 3 — Centfgd. from " " " ", alc. added to incipient ppt. Centfgd. Ppt. discarded. FEB 4 — Soln. concd. further in vac to ca 10 cc. Treated w. ca. 10 cc. abs. alc. & centfgd. Soln. A_1 seeded w. glucosamine HCl & let stand in ice-box. Ppt. A_2 diss. in ca 2 cc. H_2O, abs. alc. added to incipient turb., let stand in ice-box. FEB 5 — Sm. amt. oil had sepd from A_1. Clear soln. poured from this, = vol. abs. alc. again added, seeded again. Oil from A_1 diss. in A_2, alc. added to soln. until sl. turbid, warmed until clear

SS 38

	Tag Nos	Tag dates	No. Flasks	Precipitation	Stoked
10/24/24	1/193/11,12	X-15, 16	5, 3	10/25	10/27
10/27/24	1/193/12	X -16	14	10/28	10/29
10/28	1/193/11,12	X-15, 16, 20	1, 3, 10	10/29	10/30
10/29	1/193/11	X-20	14	10/30	10/31
10/30	1/193/11,12	X-15, 20, 22	9, 4, 1	10/31	11/1
10/31	1/193/11,12	X-20, 22	1, 14	10/,	11/3
11/5	1/193/194/12,5	X-22, 28	4, 10 94	11/6	11/7
11/6	1/194/5	X-28	14	11/7	11/8
11/7	1/194/5	X-28	14	11/8	11/9
11/10	/194/5,6	X 28, 30	9, 5	11/11	11/12
11/11	194/5,6,7	X-28, 30, X1-1	1, 12, 1	11/13	11/14
11/12	1/194/7	X1-1	14	11/13	11/14
11/18	1/194/12/7,4	X1-1, 4	2-12	11/19	11/20
11/19	1/192/194	X1-12, 1	5, 3, 6 192	11/20	11/21
11/20	1/192/4,5	X1-12, 11	7, 7	11/21	11/22
11/21	1/192/4,5	X1-11, 12	14, 10	11/22	11/22

219 flasks

328.5 l.

NOV 24 — At vol. of 4 l., 1:1 HCl stirred in to max. pptn. Acid to Congo. Centfgd., ppt. washed 2x w. $\frac{N}{40}$ HCl. At vol. of 4700 cc. pptd. in cold w. 3 l. alc. & let stand over night in ice-box.

NOV 25 — Supernatant —, siphoned off, ppt. centfgd. & washed 1x w. $\frac{N}{2}$ HOAc. NOV 26 — Susp'd. in H_2O, diss'd. w. sl. xs. NaOH, centfgd. repeatedly in cold until clear. NOV 29 — Ppt. washed w. H_2O, 10 g. NaOAc added to soln., & at vol. of 700 cc., pptd. w. 350 cc. alc. Set stand in ice-box. DEC 1 — Supernatant —, so centfgd. in 4 bottles. Ppt. in each taken up in 50 cc. $\frac{N}{2}$

HOAc. DEC 2 – Centfgd. in cold. ~~Supernatant~~ –. Ppts. taken up in H_2O, made faintly alk. w. 10% NaOH, centfgd. ~~Soln. + HOAc~~, 10 g. NaOAc added, & at vol. of 500 cc., ppt'd. w. 50 cc. alc. DEC 3 – Much
4 S. in ppt., but also in filt. 50 cc. more alc. added & set in ice-box. After several hrs. portion centfgd. in cold. Test very weak. 25 cc. more alc. added. DEC 4 – ~~Supernatant~~ ±, discarded, ppt. taken up in 250 cc. H_2O, centfgd. DEC 6 – 10 g. NaOAc added, & at vol. of 400 cc., ppt'd. w.
5 125 cc. alc., set in ice-box. Centfgd. ~~Supernatant~~, ±, ~~sucked off, ppt. centfgd.~~ discarded. Ppt. taken up in H_2O. Made faintly alk. to effect complete soln., centfgd. DEC 10 – At vol. of 300 cc. (+10 cc. NaOAc) ppt'd. w.
6 200 cc. alc. Let stand in cold. DEC 11 – ~~Supernatant~~ –, ~~centfgd~~, sl. ppt. Ppt. diss. in H_2O,
HOAc centfgd. DEC 16 – Poured from trace of ppt., & at vol. of ca. 200 cc., ppt'd. by adding 12½ cc. HOAc & mixing thoroughly. Let stand in ice-box. DEC 17 – Centfgd. DEC 24 – Ppt. diss. w. aid of xs. dil. NaOH, soln. neutr. w. N HCl, & 10 g. NaOAc added. At vol. of 250 cc., ppt'd.
7 w. 150 cc. alc., let stand in cold. DEC 26 – ~~Supernatant~~ –, so mixt. centfgd. in cold. DEC 29 – ~~Supernatant~~ poured off, ppt. rediss. in H_2O. JAN 8 1925 – Made acid to Congo w. 1:1 HCl, & at vol. of 250 cc.
8 HCl ppt'd. w. 700 cc. chilled alc. After several hrs. in ice-box, supernatant, weakly +, sucked off, remainder centfgd. in cold. Supernatant (X) + ca. ½ vol. Et_2O gave slight ppt. Main ppt. diss. in H_2O + little N HCl, but soln. thick & opalescent, so NaOH in xs. added. Neutr.
9 JAN 9 – Centfgd., 5 g. NaOAc & 10 cc. N NaOH added. At vol. of 200
NaOH cc., ppt'd w. 250 cc. alc., let stand in cold. JAN 10 – Taken up
10 in H_2O & ppt'n. repeated under exactly the same conditions, as most
NaOH of color had been removed by previous ppt'n. ~~Supernatant~~ ±, discarded, ppt. centfgd. Ppt. from (X) above diss. in H_2O, soln., ++, neutr., added to (39) JAN 12 – Main ppt. diss. in H_2O, centfgd. JAN 13 – Neutr. w. 25%
HOAc HOAc, & at vol. of 200 cc., 12 cc. HOAc added. Thoroughly stirred. Centfgd. JAN 15 – ~~Supernatant~~ poured off, ppt. ~~stirred~~ up w. H_2O, acidif. to
11 HCl Congo w. 1:1 HCl, & at vol. of 225 cc., practically colorless soln. ppt'd. w. 600 cc. chilled redistd. alc. After several hrs., supernatant –, removed by centfg. Ppt. taken up in H_2O. 16th & 17th – Transf. to dialysis bags, rinsed in w. little N HCl, dialyzd agst. running H_2O for several hrs., then ~~agst~~. in ice-box agst. successive changes distd. H_2O until Cl-free inside & out. Let stand in ice-box under H_2O + acetone. FEB 19 – Washed w. acetone, filtered, dried. **Yield: 2.7 g.**

MAR 2 – Since analytical values are quite O.K., 2.6 g. diss.

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FEB 20 — Wt. subst. + bot(11) dried at t. b. $CHCl_3$ 8.6480 8.6418 8.6414 8.6414

Wt. bot + subst. 8.6415
" " 8.5390
.1025
Ash .0004
.1021

Diss. in H_2O + little N H_2SO_4 N NaOH added until bpt. 1" formed redisp., made up to 20.42 cc. Solns. centfgd. from traces suspended matter.

ASH

Wt. bot + subst 8.5890
" " 8.4636
.0754

Wt. Pt dish + ash 23.4010
" " " 23.4007
.0003

Ash 0.4%

(Wt. bot. + subst 8.4637
" " 8.4051
.0586

Wt. Pt dish + ash sulfate 23.4008
" " " 23.4005
.0003)

FEB 26

OPTICAL ROTATION

l=1 0 = −0.02 −0.03 −0.03 Rdgs.: +1.44 1.43 1.44

$[\alpha]_D = +29.4°$ Ash-free

MICROKJELDAHLS 2 cc. samples

	I	II
cc. N/50 HCl (x	3.00	3.30
" N/100 NaOH (x 1.014)	15.79	10.71
	12.81	8.00
	2.98	2.71

b. 3 hrs. HYDROLYSIS w. b. N HNO_3 2 cc. dil. w. 2 H_2O, 4 cc. 2 N HNO_3

Rotation: l=1 0 = −0.07 −.07 Rdgs.: +.19 .19 $[\alpha]_D = +20.8$

Redg. Sugars: 2 cc. sample (Goebel) Blank 20.05
Rdg. 17.90

b. 1 hr. longer

Rotation: l=1 0 = +0.04 .04 Rdgs.: +.28 .32 .32 .33 $[\alpha]_D = +22.4$

Redg. Sugars: 2 cc. sample (Goebel) Blank 20.05
Rdg. 17.45

18.4%

2.60 x .318 x .957 = .79 R_h = .46

Test for Ammonium Salts

2 cc. dil. to ca. 5 cc., 5 cc. 10% aq. Na_2CO_3 added. Aspirated.

cc. N/50 HCl (x	2.00
" N/100 NaOH (x 1.014)	12.90
	9.00
	3.90

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FEB 26 – Stability to approx. 5% NaOH at room temp.
2 cc. soln. let stand + 1 cc. H_2O + 3 cc. 10% NaOH over night.

Stability to htg. w. approx N/10 NaOH.

2 cc. ~~soln~~ + 3 cc. H_2O + 5 cc. N/5 NaOH

cc. N/50 HCl (f)	2.00
" N/100 NaOH (×1.014)	14.61
	10.71
	3.90

Hydrolysis w. HNO_2

4 cc. soln. + 6 cc. 30% $NaNO_2$ + 3.5 cc. HOAc let stand ca. 0.5 hr.
1.2 cc. 18 N H_2SO_4 added & HNO_2 aspirated out for 0.5 hr. FEB 27 – Dil'd. to 20 cc.

ROTATION

l = 2 0 : -0.03 0 -0.02 -0.02 Rdg.: +0.32 .33 .32

$$[\alpha]_D = \frac{+34 \times 10}{2} = +170°$$

Redg. Sugars

cc. $Na_2S_2O_3$ ~~(Blank)~~

	3 cc. sample
Blank (graded)	20.05
Rdg.	16.60
	3.45 × .957 × .318

= 1.05 mg. Cu = .56
= 18.7%

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w. xs. dil. HCl, made faintly alk. to litmus w. NaOH, ppt'd. w. xs. sat'd. $Ba(OH)_2$ (Kahlbaum's fur Analyse) soln. Let stand in ice-box over night.
$Ba(OH)_2$ MAR 3 – Centfgd. repeatedly. Opalescent supernatant still +++. Addn. of even enough $Ba(OH)_2$ to cryst. out on cooling does not ppt. all S. MAR 23 – Ppt. (A) taken up in sl. xs. H_2SO_4. Supernatant B, ppt'd. w. xs. H_2SO_4, centfgd., wsh'd. w. NaOH, concd. to dryness in vac., taken up in washings of H_2SO_4 ppt., again wsh'd., & at vol. of ca. 150 cc., ppt'd w. 100 cc. alc. MAR 24 – Supernatant discarded, ppt. centfgd., stirred w. H_2O, set in ice-box. (A) repeatedly centfgd., ppt. washed w. little dil. HCl & centfgd. Soln. made sl. alk. w. aq. NH_3.

B lost in accident to cold centrifuge.

APR 2 – A solns. + 10 g. NaOAc, at vol. of 250 cc., ppt'd. w. 200 cc. alc. Let stand in cold. APR 6 – Supernatant –, wsh'd. centfgd. APR 7 – Ppt. diss. in ca 150 cc. H_2O & milky soln. ppt'd. by adding HOAc to neutrality, then 4 cc. more. Stirred thoroughly, let stand in cold. APR 8 – Centfgd. APR 20 – Diss in H_2O + sl. xs. dil. NaOH, dil'd to 250 cc. & run through Berkefeld N. Still opalescent, so run through Berkefeld W, now coming through practically clear. Washings opalescent, so added to (44). APR 21 – Concd. in vac. APR 22 – At vol. of ca 150 cc., again ppt'd. w. HOAc, considerable xs. being necessary. Centfgd. Supernatant, –, discarded. Ppt. taken up in dil. HCl, ~~ppt'd w a~~ at vol. of ca. 100 cc., ppt'd. w. 150 cc. redist'd. alc. Let stand in cold ½ hrs., centfgd. Sm. amt. active material ppt'd. from supernatant w. Et_2O, added to main portion. Diss. in ca 75 cc. H_2O + HCl, reppt'd. w. redist'd. alc. Let stand 2 hrs., centfgd., ppt. taken up in H_2O, reppt'd. w. ca. 3 vols. redist'd. alc. Let stand in cold, centfgd. Ppt. taken up in H_2O, not diss'g. this time. Let stand over night. MAY 5 – Xs. redist'd alc. added, let stand, centfgd. in cold. Repeated 1×, then let stand again w. H_2O. MAY 6 – Centfgd., ppt. taken up in H_2O, centfgd. again after several hrs. Repeated. Ppt. again taken up in H_2O, let stand over night. Centfgd. Repeated. Now Cl^- free, so taken up 2 × in acetone, then filtered, sucked off, air-dried in warm place, then in vac. over night. Yield: 2.0 g.

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MAY 11 – Wt. bot (4) + subst. 8.3943 8.3904 8.3886 8.3876 8.3869

8.2305

MAY 27 – Diss. w. dil. HCl + NaOH, made up to 30.72 cc; centfgd. Corr. f. Ash .1564 .0028 .1536

MAY 12

ASH

Wt. bot + subst 8.2307
" bot 8.1534
.0773

Wt. dish + ash 23.3910 23.3909 23.3895
" dish
.0014

Ash 1.8%

OPTICAL ROTATION

l = 1. 0 = 0 +.01 -.01 Rdg. = +1.54 1.55 1.55

$[\alpha]_D = 155 \times 2 = +310°$

$[\alpha]_D$ = +310° (ash-free)

Microkjeldahls

2 cc. samples

cc. N/70 HCl (x0.995) 10.00 10.00 9.95
" " NaOH (x1.015) 7.16 14.44 7.32
7.18 2.63
7.26 .15

2.48 x .2 = .496 mg. N
= 0.5 mg. N

Amino-N (Van Slyke)

JUN 2 – Bar. 761.5 Temp. 29°

Rdgs: Bl. 0.23 0.20 Mean: .215
0.69
.69
.265
.495

$\frac{.495 \times .5441 \times 100}{2}$ = 12.9 mg. NH_2N per 100 cc. soln.

N = 5.0% Ash-free

NH_2 N = 2.6% Ash-free

Action of HNO_2

JUN 1 – 2 cc. soln. + 4 cc. 30% $NaNO_2$ + 2.2 cc. HOAc let stand 0.5 hr. ~~5 cc~~. Cooled w. ice-H_2O, 0.8 cc. 6N H_2SO_4 added, aspirated 1¾ hrs. made up to 25 cc

5 cc samples

Reduc. Sugars

Bl. 19.30 (Gaebel) Rdgs. 16.25 (overrun) 16.30 Mean: 16.25
16.25
3.05 x 1.01 x .318 = .98 mg. Cu = 0.97 mg. glucose = **28.5% glucose**

Rotation

l = 2 0 = -0.01 0 +0.01 Rdgs.: +.18 .15 .16 .18 .16 .17

$[\alpha]_D = \frac{.17 \times 100}{.04 \times 2} = +213°$

b 100
17

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HYDROLYSIS w. 1.4 N HNO_3.

JUN 2 2 cc. dil. to 4, 4 cc. 2 N HNO_3 added, hr 5 hrs. Refrozen alc. in cold.
JUN 3 ~~2 cc. sample~~ Neutr. w. $NaHCO_3$, made up to 10 cc., 2 cc. taken for anal.

Bl. 19.30 (Goebel.) Rdgs: 14.70 14.70
14.70
4.60 × 1.01 × .318 = 1.48 mg. Cu = 0.77 mg. glucose = 38.5 % Redg. Sugars

~~Hydrolysis w. 1/2 H_2SO_4~~

Hydrolysis w. 1/2 H_2SO_4
Wt. bot. + subst 8.1538
" " 8.1260
.0278

?

Jun 23 Repeated by Goebel.

4 cc. dil. to 8, 8 cc. 3 N HNO_3 added.

Time:	4 hrs	5 hrs.	6 hrs.
Sample	2 cc.	2 cc.	2 cc.
$Na_2S_2O_3$ (×1.01)	16.15	15.50	15.20
Cu Eqv.	1.03	1.26	1.35
Glucose	.59	.68	.785
% "	23.6	27.2	28.6

Bl. 19.40

28.6 % Redg. Sugars

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SEP 28 1925 — 1 g. 38A (contg. few mg. left of 37) diss., w. shaking, in 5 cc. conc. HCl, cooling w. ice-H_2O. Material gelatinized & diss. in viscous soln. very slowly, so 5 cc. more conc. HCl added. SEP 29 — Still ++++, so removed from ice-box & let stand at room temp. SEP 30 — A.M. + P.M. ± OCT 1 — Now —. 0.1 cc. withdrawn for redg. mg. deter. 21%. Remainder centfgd. from trace amorphous matter & clear supernatant concd. in vac. at bath temp. not exceeding 45°, taking care not to let concentrate get that hot. Finished on Hg-Vac. pump until very viscous syrup. Ca 25 cc. abs. alc. (redistd.) added. OCT 2nd — Worked up w. abs. alc. until solid, centfgd. & ppt. washed 2× w. sm. amts. abs. alc., then acetone, & dried: (A). 0.1062 g.

Supernatant + alc. washings (B) concd. at about ½ vol. more of insol. subst. sepd. This centfgd. off, washed, w. a little abs. alc., then w. acetone, dried, added to (A). Soln. (B) concd. in vac. to thick syrup, part of which solidified (apparently amorphous, but became syrupy again on standing. Rediss. in abs. alc. & let stand in ice-box. Remained clear, so concd. dry, finally in high vac. OCT 22 — Taken up in 5.5 cc. abs. alc., 5.5 cc. dry Et_2O cautiously added, w. stirring. Ppt. centfgd. off, washed 2× w. 3 cc. ea. time abs. alc.-Et_2O, taken up in redistd. acetone, filtered off, washed. Ppt. (B): 0.44 g. Clear reddish M.L. & washings let stand in ice-box. OCT 27 — Nothing had sepd., so concd. dry in vac., finally in high vacuum. Taken up in 10-12 cc. H_2O, centfgd. from sm. amt. dark brown amorphous material, & lt. brown soln. dild. to 25 cc. (C).

A - gives weak Cl^- test, strong Molisch. After continued drying over $CaCl_2$ + NaOH — practically Cl-free

B - contains much Cl^-.

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Fraction (B)

NOV 18 — 0.33 g. B diss. in 5 cc. H_2O (contg. 26.7 mg. Cl = 27.5 mg. HCl) & treated w. 0.12 g. $AgNO_2$, ∝ to the HCl in the sample, + 0.18 g. in xs., adding 1.2 cc. IV HCl ∝ to the xs. $AgNO_2$. After 4 hrs. 0.3 g. more $AgNO_2$ & 2 cc. IV HCl (slight deficiency) added. 19" ppt. AgCl filtered off, washed well w. H_2O, & H_2S passed into filtrate to remove xs. Ag. Ag_2S centfgd. off, soln. concd. to dryness in vac, finally w. abs. alc., taken up w. little abs. alc., let stand in ice-box. No sepn. of crysts. Dry Et_2O added until faint oily ppt., & mixt. seeded w. crysts. of 2,5-anhydroglucose, but none sepd.

This fraction gives strong naphthoresorcin test.

S.S.S. 38 A

Hydrolysis

OCT 7 — Fraction A (E) Wt. bot. + desicc. dried substr. 7.3542
" " 7.2480
.1062

Dried in vac at room temp. over $CaCl_2$ + KOH 7.3507
7.3506
7.3006
.0500

OCT 20 Diss. in H_2O + made up to 10 cc.

OPTICAL ROTATION

l = 1 Bl. = −0.06 05 07.06 Rdgs: +0.96 .98 .98 .99

$$[\alpha]_D + 2.08°$$

MICROKJELDAHLS 2 cc. samples.

cc. N/70 HCl	10.00	10.00	Bl	10.00	Bl	10.00
" " NaOH (x.934)	8.38	8.40		10.60	10.00	10.60
	10.00				9.90	
	7.84				.10	
	2.16					
	×10					

2.06 × 0.2 = 0.412 mg. N

N Total = 4.1%

Reducing Sugars 1 cc. samples

Blank 18.93 Titrns. 14.65 14.60
14.63
4.30 × 1.025 × .318 = 1.40 mg. Cu = 0.69 mg. glucose

Redg. sugars = 13.8%

OCT 23

Amino N

			cc.			
Barom	761	Bl.	0.15	~~Bar~~ T	Deth.	
T	22.5°		.13	22.0	0.58	
	21.5°				.14	
					.44 × .5643 × 100	
					2	

NH_2 N = 2.5%

= 12.4 mg. NH_2 N per 100 cc.

OCT 28

Hydrolysis

Wt. bot. + substr. 7.3010
7.2935
.0075

6 cc. N/2 HCl added, b. 2 hrs.

Dil. to 7.5 cc., 2 cc. samples taken

NH_2 N = 2.5

Blank 18.93
Deth. 17.70 Dupl. bot

1.23 × 1.025 × 0.318 = 0.4 mg. Cu = 0.29 mg. glucose

Redg. sugars 14.5%

1 cc. soln. left 2 hrs. longer in sealed tube in b. H_2O

	w. 1 cc. conc. HCl	w. 1 cc. 1:1 HCl
Blank (Gebel)	19.00	19.00
	18.35	17.85
	.65 × .2126 Cu = 21% glucose	1.15 × 1.025 × .318 = .375 Cu = .28 28% glucose

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Hydrolysis

Reaction A

B

Amino N

Redg. Sugars after HNO_2 Treatment.

2cc. + 4cc. 30% N_4NO_2 + 2.2cc. HOAc. after ½ hr. 1.5cc. 30% (vol.) H_2SO_4 added, aspirated 1½ hrs. Dil. w. H_2O, made up to 25cc., 5cc. samples taken.

Blank: 18.93

Detn 15.30 15.30

$3.63 \times 1.025 \times .318 = 1.18$ mg. Cu = 0.6 mg. glucose

Redg. Power 30%

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Hydrolysis Fract. B

OCT 23 – Portion dried in vac. at room temp. over H_2SO_4

+ NaOH in bot(12) 8.1698 8.1686 Diss. in H_2O, made up to 20.2 cc.
8.0676
.1010

OCT 26

OPTICAL ROTATION

P=1 Bl. −0.06 .06 .06 Rdgs. +0.85 .88 .87 .89

$[\alpha]_D = +93 \times 2 = +186°$

$[\alpha]_D = +186°$

MICROKJELDAHLS 3 cc. samples

cc. N/70 HCl	10.00	10.00	10.00	
" " NaOH(1.9434)	7.20	7.20	6.72	
			3.28	
		Bl.	10	

3.18 × 0.2 = .636 mg. N in 15 mg.

Total N 4.2%

Amino N

Barom 757 Soln: 0.50
T. 22° 22° 095
Blanks: 0.095 − Bl. .405 × 5613 × 100 / 720 = 11.4 mg. N per 100 cc.
.085
.105

NH_2 N 2.3%

Chlorides

	1 cc. sample		2 cc. sample	
cc. HNO_3-$AgNO_3$ N/20	3.00	7.50	3.00	7.50
" " " " N/50				
" N/50 KSCN(1.084)	6.36	6.89	5.87	6.36
		.61		1.14 × .709 = .808 mg. Cl in 10 mg.

Cl = 8.1%

Redg. Sugars as Glucose

1 cc. samples

Blank	18.93	18.93
Rdgs:	15.55	15.35

(average) 3.58 × 1.025 × .318 = 1.17 mg. Cu = 0.6 mg. glucose

Redg Power 12%

Hydrolysis – 2 cc. dil. 5, 5 cc. 4N HCl added.

2 cc. samples

				Mean	Redg. Power
B 2 hrs:	Blanks:	18.93	18.93	18.93	30%
	Titrn:	15.33	15.38	15.36	
				3.57 ×	

Same as above; Glucose = 0.6 mg.

B 4½ hrs.	Blanks (Goebel)	19.00	19.00	19.00	28%
		15.65	15.75	15.70	
				3.30 × 1.025 × 0.318 = 1.08 Cu = 0.56 gl	

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Titration of $AgNO_2$.

NOV 17 — .1000 g. dis. in H_2O, little $\underline{N}$ HNO_3 added, Fe, cooled, made up to 100 cc. 25 cc. samples

				Con.
cc. NH_4SCN (x1.001)	(1.93)	1.95	1.96	1.95 ∝ 9.75 mg. NaCl
				6.085 " HCl

.100 g. ∝ 0.0243 g. HCl ∝ 0.685 cc. $\underline{N}$ HCl

S. S. S. 38 A Hydrolysis

Fract. C.

OCT 27 – 0.5 cc. main soln. dild. to 5, 1 cc. taken ea. for sugar anal.
Blank: 18.93
Titrn: 18.92

OCT 28 – 0.5 cc. main soln. used

Bl.	18.93		18.93
	15.94	15.86	15.90

3.03 × 1.025 × .318 = 0.99 mg. Cu = 0.52 mg. glucose in 0.5 cc.
26 mg. " in 25 cc.

OCT 30 Hydrol. w. Stronger Acid.
~~Fol Hyd~~ 2 cc. dil. to 4 w. 4N HCl. B. 2 hrs. 1.5 cc. samples

Blanks (boiled)	19.00	
Rdgs.	4.25	4.50

OCT 31 1 cc. hydrolyzed material dild. filtered, washed to 5 cc.
1 cc. samples. = 0.1 cc. orig.

Blanks:	18.93	
Rdgs:	18.00	18.00

∴ in orig soln 250 × 0.25 = 62.5 mg. glucose if hydrol. further

0.93 × 1.025 × .318 = .3 mg Cu = 0.25 mg. glucose in 1 cc.

OCT 30 MicroKj. ½ cc. samples

cc. N/70 HCl	10.00	10.00
" " NaOH (x.934)	9.40	9.35

Wt. of fraction. 2 cc. used

Wt. dish + residue:	14.3564	14.3555
" "	14.3358	14.3358
		.0197

× 12.5

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	No on Fla.	Date on Fl.	No. of Fl.		Precip.	Stoved
11/24/24	1/192/5	XI - 12	14		11/20	11/26
11/25	1/192/193/5,6	XI - 12,19	5, 9		11/26	11/28
11/26	1/193/6	XI - 19	14		11/28	11/29
12/1	1/193, 195/6,3	XI - 19, 20	7, 7		12/2	12/3
12/2	1/195/3	XI - 20	14		12/3	12/4
12/3	1/193, 195/5,3,4	XI - 18, 20, VV	1, 9, 4	84	12/4	12/5
12/4	1/195/3, 4	XI - 20, VV	1, 13		12/5	12/6
12/5	1/195, 196/4,3	XI - 22, 29	3, 11		12/6	12/8
12/8	1/196/3	XI - 29	14		12/9	12/10
12/9	1/196/3	XI - 29	11	137	12/10	12/11
12/12	1/196/4	XII - 6	14		12/13	12/15
12/15	1/196/5	XII - 8	14		12/16	12/17
12/16	1/196/4,5	XII - 6, 8	4, 9		12/17	12/18
12/17	1/196/5	XII - 8	14		12/18	12/19
12/18	1/196/5	XII - 8	13	205	12/19	12/20

168
37
205

DEC 22 – At vol. of 4200 cc., pptd. w. 1:1 HCl to max. turb. Soln. not yet acid to Congo. After ca. 2 hrs. ppt. centfgd. off, washed 1" w. H_2O slightly acidulated with HCl. At vol. of 4700 cc., chilled soln. pptd. w. 3000 cc. ~~abs~~ cold alc. DEC 23 – Supernatant –, centfgd. off (Contains considerable active material – XX) in cold, ppt. washed w. N/2 HOAc. DEC 24 – Relatively slight amt. ppt., after removal of – supernatant, homogenized w. H_2O. DEC 26 – Centfgd. Somewhat colored supernatant, ±, discarded, ppt. again mixed w. H_2O. DEC 29 – rff. Diss. w. dil. ~~ac~~ as. NaOH. X in meantime ~~diss. w. aid~~ again washed w. H_2O & soln. ~~+~~, added to main soln. Ppt. diss. w. aid of NaOH & soln.

307.5 liters

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+, pptd. w. alc. Ppt. redis. as before, reppted. w. alc. Ppt. taken up in H_2O, acidified to Congo w. 1:1 HCl, centfgd. Supernatant, now pale brown, neutralized, ++. Added to main portion. JAN 7 1925 – At
3 vol. of 500 cc., 20 g. NaOAc added, pptd. w. 250 cc. alc., set in cold. JAN 8 – Supernatant, –, mixt. centfgd. JAN 9 – Ppt. taken up in H_2O, centfgd. JAN 10 – 15 g. NaOAc added to clear solns, & at vol. of 450 cc., pptd.
4 w. 250 cc. alc. JAN 12 – Supernatant –, mixt. centfgd. JAN 13 Ppt. taken up in 250 cc. H_2O, centfgd. Clear soln. poured into 2 centfg.
HOAc bottles, mixed into ea. w. ca 25 cc. H_2O, & each then acidified w. 8 cc. HOAc. Thoroughly stirred, centfgd. Ppt. taken up in H_2O, neutr. w.
5 NaOH, centfgd. Soln. + 10 g. NaOAc, at vol. of 200 cc., pptd. w. 125 cc. alc. Let stand in cold. JAN 16 – Centfgd., supernatant –, discarded.
HOAc Ppt. taken up w. ca 300 cc. N/2 HOAc. JAN 17 – Centfgd. Ppt. taken up in H_2O, neutr. w. NaOH. JAN 19 – 5 g. NaOAc & 10 cc. N NaOH added, & at vol. of 200
6 NaOH cc., pptd. w. 250 cc. alc. JAN 20 – Ppt redis in H_2O & reppted. as before. Supernatant +, 50 cc. more alc. added.
7 NaOH JAN 22 – Ppt centfgd. off. Supernatant still +, so pptd. w. 2 vols. alc. & let stand in ice-box. Both ppts combined, diss. in H_2O, centfgd. from dirt.
HOAc JAN 26 – Soln., at vol. of ca 200 cc., acidif. w. 12 cc. HOAc & thoroughly mixed. Centfgd. after several hrs. JAN 27 – Ppt. diss. in H_2O + enough NaOH to make soln. slightly alk. $Ba(OH)_2$ soln. added. No ppt. at 1", but w large excess considerable ppt.
$Ba(OH)_2$ formed. Let stand in ice-box. JAN 28 – Centfgd. Milky supernatant (A) poured from chiefly gummy ppt. (B) and run through Berkefeld N 5 in candle, coming through clear. B washed w. H_2O contg. $Ba(OH)_2$ & washings also run through the filter. JAN 30 Filtrate –. Ppt. washed w. ca. ½ satd. $Ba(OH)_2$ & this run through the filter. FEB 3 – Washings also –. Filter washed through repeatedly w. very dil. HCl. Main ppt. B also diss. by shaking w. xs dil. HCl & Ba^{++} removed from both portions by ppting. w. xs H_2SO_4. Centfgd., neutr.,
8 concd. in vac. FEB 5 – At vol. of ca. 150 cc., pptd. w. 100 cc. alc. Let
NaOH stand in cold. FEB 6 – Supernatant –, centfgd. off. Ppt. taken up in
9 H_2O, centfgd. FEB 7 – Soln. acidified w. 1:1 HCl, vol. ca 125 cc. pptd. w. 100 cc. cold
HCl alc. + 200 cc. abs. alc. redistd. Let stand in ice-box ¾ hrs., centfgd. Supernatant –, ppt. diss. in H_2O soln. diald. 1" agst. running H_2O, then agst. distd. H_2O in ice-box until free from Cl^- & $SO_4^=$. (Contents of bags centfgd., washed 2x w. redistd. acetone, ppts. filtered off, washed w. redistd. acetone, dried. Yield: 0.8 g. + 0.6 g. = 1.4 g.

S. S. S. 39

FEB 14 Tot. Bot (11) + substr 8.5573 8.5524 8.5499 8.5469 8.5455 8.5447
8.5444 8.5437 8.5433
8.4319
.1114
Ash .0018
.1096

Diss. w. x. H_2SO_4, $N/_2$ NaOH added until pH just red, made up to ~~22~~ 22.0 cc.

FEB 18

Ash Detn.

Tot. Bot. + substr. 8.4323
" " 8.3709
.0614

Tot. Pt dish + ash 23.4018
" " " 23.4018
23.4008
.0010

Ash diss. in drop HCl w. greenish color. Soln. gives strong Ca^{++} test w. $C_2O_4^{=}$, showing ash mainly CaO.

Ash 1.6%

OPTICAL ROTATION

l = 1 0 = +0.02 .02
Rdgs: +1.56 1.54 1.52 1.53

$$[\alpha]_D = +304°$$

$[\alpha]_D$ +304° Ash-free

MICROKJELDAHLS

1.5 cc. samples

	I	II	Bl. I	Bl. II
cc. N/50 HCl (x .998)	2.00	2.00	2.00	2.00
cc. N/100 NaOH (1.014)	12.09	13.06	11.36	14.80
	16.01	12.09	8.00	11.42
	1.08	.97	3.36	3.38
Mean: 1.03	3.99		3.99	
	1.04		3.42	
	2.95		.57	
	.57			

2.38 × 0.14 = 0.333 mg. N in 1.5 cc. or in ~~7.5~~ mg.

N = 4.4% Ash-free

Amino N

Shaken 5 min.

FEB 19 – 1:400 Soln S.S.S. 39 2 cc.
Blanks: 0.2 20°, 769 , 0.2 21°, 769.
Rdgs.: 0.43 21.5° " 0.42 21.5° "
Factor .5733 Rdg - Bl. = 0.22 cc.

$$\frac{.22 \times .5733 \times 100}{2} = 6.3 \text{ mg. } NH_2\text{ N per 100 cc. 1:400 soln.}$$

or 100 cc. 250 mgs.

NH_2 N 2.5% Ash-free

HYDROLYSIS w. 2N HNO_3

2 cc. dil. to 4, 4 cc. 4N HNO_3 added, b. 5 hrs.
Made up to 10 cc. 2 cc. for redg. sugars
Bl. 20.05
Rdg 19.35

.70 × .957 × 318 = .213 mg. Cu = .24 mg glucose = 12% Redg. Sug.

OPTICAL ROTATION

l = 1 0 = -.04 .02 .02 Rdgs: .02 .01 .01

B. 2 hrs. longer. Still optically inactive.

S.S.S. 39

FEB 20 – 4 cc. main soln. + 7 cc. 30% aq. $NaNO_2$ + 3.7 cc. HOAc let stand 0.5 hrs. w. occasional shaking. 1.4 cc. 18 N H_2SO_4 added & HNO_2 aspirated out for ½ hr. Rinsed into flask & made up to 25 cc.

OPTICAL ROTATION

$l = 2$ $\quad 0 = -0.04 \; -0.03 \; -0.03$ $\quad$ Rdg. $+ 0.25 \; .27 \; .26$

$$[\alpha]_D = \frac{.29 \times 100}{2 \times .08} = +181°$$

Redg. Sugars 3 cc. samples

Bl. 20.05 $\quad$ Rdg. 15.65 $\quad$ 15.90

15.80

$4.25 \times .957 \times .318 = 1.29$ mg. Cu = 0.65 mg. glucose

$3 \times \frac{20}{25}$ mg. = 2.4 mg. $\quad$ Redg. Sugars = 27.1%

G. 2 hrs.

ROTATION

$l = 2$ $\quad$ Bl. ~~.04 .04 .01 .03 .03~~ $\quad$ Rdg ~~.26 .25 .24~~ .26

Bl. .04 .03 .04 .03 $\quad$ Rdg + .18 .19 .18 .18 (Goebel)

Redg. Sugars

Bl. 20.05 (Goebel) $\quad [\alpha]_D = \frac{.22 \times 100}{2 \times .08} = +138°$ $\quad$ 3 cc. sample

17.30

2.75

18%

$\frac{11}{17} \times 27$

17 | 297 | 17

17

127

119

NH_3 N $\quad$ 7 cc. sample (neutr.) aspirated 1 hr. w. 10 cc. satd. K_2CO_3.

cc N/50 HCl (x .998) 2.00 $\quad$ 4.00

" N/100 NaOH (x 1.014) 13.61 $\quad$ 3.35

7/25 x 20 mg. 5.6 mg. subst. 10.31 $\quad$ .65 x .14 = .09 mg. N in 7 cc.

3.30

NH_3 IV = 1.6%

Acid Equivalent (Goebel)

2 cc. sample $\quad$ 5 cc. $AgNO_3$ N/10 x 1.013 $\quad$ 2 cc. centfgd. soln. for titr.

2/7 x 25 x 1.013 = cc. N/50 $AgNO_3$ for sample = 7.24

cc. " KSCN (x1.03) 6.80

6.60 $\quad$ 6.60

.44 cc. used up by 2.86 mg. subst.

.0088 cc. IV

28.6 g. = 88 cc. N

Acid Equiv

325

NH_3 N $\quad$ 8 cc. sample neutr. w. NaOH, aspirated ½ hrs. w. 10 cc. 40% NaOH

cc N/50 HCl (x .998) 2.00 $\quad$ 4.00

" N/100 NaOH (x 1.014) 15.09 $\quad$ 3.14

11.99

3.10

.86 x .14 = .12 mg. N $\quad$ NH_3 N =

In sample $\frac{8}{25} \times 20$ mg = 6.4 mg. $\quad$ 1.9%

S. S. S. 39

MAR 2 1925 — 1.5 g. air-dry S.S. 39 diss. in 200 cc. hot N HNO_3. Turned pinkish at 1". Soln. b. under rf. for 4 hrs., becoming bright yellow, then greenish yellow. Concd. in vac., finally in warm desiccator to ca. 10 cc. 4 cc. conc. HNO_3 added & let stand over night.
MAR 4 — Few glistening, rhombic crystals on bottom. Mixt. b. few min. then stirred on watch-glass over b. H_2O until dry. Few cc. more 1:1 HNO_3 added, stirred again over b. H_2O. White residue taken up in acetone, crystn. residue remaining insol. Let stand ~~several hrs. @ room temp., then in ice-box overnight.~~ MAR 5 — Acetone had evapd. off. Residue taken up w. more acetone, filtered, washed with several sm. portions acetone. Filtrate (B). Ppt. contained much ash, app. CaC_2O_4, so taken up in 5 cc. N NH_4OH, filtered, washed w. 2 cc. N/5 NH_4OH, 0.3 cc. conc. HNO_3 added. Free acid rapidly sepd. as prisms. Let stand in ice-box. (A). (B) allowed to evap., residue taken up in a little H_2O, filtered, washed w. 50% alc. Filtrate C. B diss. w. sl. xs. dil. NH_4OH, filtered from traces insol. material, acidif. w 0.2 cc. conc. HNO_3. Free acid began to sep. almost immed. C b. to ~~sm. vol.~~ remove alc., cooled, & at vol. of ca. 4 cc., made alk. w. 40% KOH. Heavy crystn. salt immed. sepd. Centfgd. Ppt. C_1. Supernatant C_2. C_1 washed 1" w. ca. 1 cc. H_2O, then b. w. 2-3 cc. dil. HOAc most remained insol. C_2 acidified strongly w. HOAc to get KH saccharate if present. All let stand in ice-box.
MAR 6 — A filtered on sm. Buchner, washed several times w. sm. amts. H_2O, then w. sm. amts. acetone. Yield: 0.21 g., m. & decomps. 216° w. prelim. softn. & blackening. MAR 7 — B filtered off, washed w. H_2O, acetone, dried. Yield: 0.070 g., m. & decomps. 215° w. prelim. softn. & blackening. C_1 filtered off w. aid of 50% alc., washed w. 50% alc., alc., acetone, dried. Yield: .191 g.

C_2 filtered from sm. amt. crystals, probably same as C_1, seeded w. KH saccharate.

Mar. 9 — Remainder of A & all of B recrystd. from b. H_2O. Combined. Yield: 0.11 g., m. 216° decomps., w. prelim. dark. & softn.
NOV 12 1926 — C_2 decanted from small deposit of heavy crystals. These recrystd. from 1 cc H_2O (C_2). ~~Soln~~ Decanted C_2 (C_3) held. & b. w. bone-black. Tested for optr. act. & found to contain strongly dextro rotatory substr. Rotation +.57°. 7 cc. l = 1. Although strongly acid w. HOAc, gives ppt. w. basic Pb acetate. This added until no more permanent ppt.

Oxidation

SSS 39

NOV 22 – C_2 = ca. 12 mg. Slight quinoline-like odor on burning. Intumesces. MICROKJELDAHL on 9cc. % N

C_3 – Basic Pb ppt. (before neutrn.) centfgd. Aff. Ppt. C_3 Supnt. (C_4) treated w. xs. basic Pb ac. & set in ice-box, centfgd., ppt. suspd. in H_2O, decompd. w. H_2S. C_3 – also.

C_3 – after filtrn. & removal of H_2S gives charac. ppt. of CaC_2O_4 w. $CaCl_2$ + aq. NH_3.

C_4 – filtered & H_2S removed by air. At vol. ca 14 cc. in 2 dm. tube. Bl. – 03 .02 .02 Rdgs: +0.11 .09 .08 .09✓

$\alpha = +0.11°$

Concd. dry in vac. & evapd. several times w. H_2O. Yield: 0.025g. Added to corr. portion of (62)

S.S.S. 39

Wt. bot. + subst. 8.1310
" " 7.9241
.2069

C + H by Gartmann.
on oxidation product.

C 33.32
H 4.96

COOH
(CHOH)$_4$
COOH

C_6 = 72.
H_{10} = 10.08
O_8 = 128
210.08

C = 34.27%
H = 4.80% (theory)

MAR 10 – Recrystd. A & B Wt. bot + subst 8.0351 0.1008 g. anh. subst. for combustion
" " 12 7.9241
.111

$CaCl_2$ 63.8079 Naline 55.4795 Guard 45.0685
63.7637 55.3574 45.0653
.0442 .1221 .0032
.0032
.1253

C = 33.9%
H 4.9%

Fraction C_1

Wt. bot (8) + subst. 7.4265 7.4260 7.4258 7.4259
" " " 7.2345 7.2345
.1914

7.4259
7.3259
.1000

Diss. in 6 cc. H_2O by htg, cooling rapidly.

OPTICAL ROTATION

Bl. -0.07 .05 .06 .06 Rdg. -0.03 .05 .05

Rinsed into weighed Pt dish w. H_2O, evapd. dry, ignited w. H_2SO_4.

Wt. dish + K_2SO_4 23.4551
" " 23.4001
.0550

Recrystd. from dil. HOAc.

Wt. bot. (8) + subst 7.3020 7.3005
7.2347 7.3004
7.2350
.0654

Wt. Pt dish + K_2SO_4 23.4325 23.4320
" " " 23.3995
.0330
.0325
.0146

0.44876 Wt. K

% K 22.3%

-COOK
H-C-OH
COOH

36
3
89
39.1
158.1

K = 24.7%

Type I

5'S'41

12/31/24	1/196/6	XII - 24	12	1/2/25	~~1/3~~ 3 4
1/2/25	1/196/6	XII - 24	14	1/3	1/5
1/5/25	1/196/6	XII - 24	14	1/6	1/7
1/6	1/196/6	XII - 24	14	1/7	1/8
1/21	1/202/3	I - 20	68 14	1/28	1/29
1/28	1/202/3	I - 20	14	1/29	1/30
1/29	1/202/3	I - 20	14	1/30	1/31
1/30	1/202/4	I - 21	14	1/31	2/2
2/2	1/202/3,4,5	I - 20,21,23	1,11,2	2/3	2/4
2/3	1/202/5	I - 23	138 14	2/4	2/5
2/4	1/202,204/4,5,3	I - 21,23,28	1,3,10	2/5	2/6
2/5	1/204) 3	I - 28	14	2/6	2/7
2/6 15 14 60	1/204/3	I - 28	14	2/7	2/8
2/10 15 210 12 222	1/204/3,4	I - 28, II - 2,3	1,10,3	2/11	2/12
2/11	1/204/4	II - 3	14	2/13	2/14
2/13	I 1/204/4,5	~~II~~ - 3,4,	208 14	2/14	2/16

FLASKS 222
LITERS 333

FEB 16 1925 — Cryst. in concentrate diss. by warming, cooled again w. ice, & at vol. of 5 l., treated w. 1:1 HCl until blue to Congo. After standing 1 hr. in ice-box, greenish ppt. centfgd. off in cold, washed 1" w. N/100 HCl. At vol. of 5500 cc., ppted. w. 8 l. alc. FEB 17 — Part of ppt. had not settled, let stand 1 day longer while main ppt. worked up by centfg. & washing w. N/2 HOAc. FEB 18 — 2nd ppt. treated same way, both combined, taken up in H_2O, diss. by warm. w. NaOH, & at vol. of 750 cc. ppted. in cold w. 400 cc. alc. FEB 19 — Supernatant —, so mixed. centfgd. in cold. FEB 20 — Ppt. taken up in N/2 HOAc, let stand ca. 2 hrs., centfgd. Ppt.

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diss. w. just enough NaOH, 10 g. NaOAc added, & at vol. of 500 cc.,
4 ppt'd. w. 300 cc. alc. Let stand in ice-box. FEB 21st Supernatant –,
HOAc cmxt. centfgd., ppt. taken up in N/2 HOAc. Let stand over night,
centfgd., ppt. taken up in H_2O, neutr. w. NaOH. centfgd. Made alk. w.
10 cc. 10% NaOH, 10 g. NaOAc added, & at vol. of 330 cc., ppt'd. w.
5 400 cc. alc. Still ++, so 300 cc. more alc. used. Still ++. 200 cc.
NaOH Et_2O added, completing the pptn. Centfgd. Yellow supernatant.
Ppt. taken up in H_2O, centfgd., & at vol. of 260 cc., treated w. 10 g. NaOAc,
6 10 cc. 10% Na_2CO_3, & ppt'd. w. 500 cc. alc. Supernatant –, overnight in
Na_2CO_3 HOAc cold. Ppt. centfgd. off, taken up in 200 cc. H_2O, 12 cc. HOAc added. MAR 2 –
Ppt. centfgd. off, taken up in H_2O, diss. by making slightly alk., centfgd.
from considerable insol. material. MAR 3 – 20 g. NaOAc added.
7 MAR 4 – 5 cc. 10% NaOH added, & at vol. of 230 cc., ppt'd. w. 500 cc. alc.
NaOH MAR 5 – Supernatant –, sucked off, ppt. centfgd., taken up in H_2O.
8 Centfgd. Mar 9 – Cold soln. ppt'd. w. xs. $Ba(OH)_2$ soln. (hot, concd.), stirred,
$Ba(OH)_2$ let stand in ice-box. 10" – Most of color in supernatant. Centfgd.
Ppt. stirred up w. H_2O. MAR 11 – Sl. xs. strong H_2SO_4 added, all lumps
disintegrated, ppt. centfgd. off. Supernatant (A) contained
very little S. Ppt. again worked w. dil. HCl. MAR 12 – This time
S came out & nec. to dil. very viscous soln. to ca. 125 cc. before could
be centfgd. Ppt. washed 2 × w. ca 40 cc. H_2O acidif. w. HCl. Combined
extd. centfgd. from trace $BaSO_4$, soln. made dil. alk. w. NaOH, ppt'd.
9 by adding 13 cc. HOAc. MAR 13 – Centfgd. repeatedly, supernatant –,
HOAc discarded! MAR 14 – Ppt. taken up in H_2O, diss. w. dil. xs. Aqu. NH_3, $(NH_4)_2C_2O_4$
added to remove any Ca present. MAR 16 – Trace of ppt. centfgd. off,
supernatant acidif. to Congo w. 1:1 HCl, & at vol. of ca. 125 cc., ppt'd. w. redistd.
10, 11 250 cc. redistd. alc. Centfgd. Ppt. taken up in H_2O + HCl, reppt'd. w. alc.
HCl Centfgd. Ppt. taken up in 50% alc. Part dissolved. MAR 17th All lumps
smoothed out, made up to 250 cc. w. redistd. alc. Centfgd. Ppt. taken up
in a little H_2O, let stand 0.5 hr., completely ppt'd. by dilg. to 250 cc. w.
redistd. alc. Centfgd. after 1 hr. Supernatant contained much Cl^-. Process
repeated twice, then ppt. taken up in H_2O alone several times, washings
being only feebly ±, finally washings & ppt. Cl^- free. Taken up in ~~acetone~~, ~~ether~~
redistd. acetone several times, filtered off, washed w. acetone, dried.

Yield: 2.5 g. H_2O soln. Active at 1: 8,000,000

MAR 31 – When titrated to alky. w. phenolphthalein, formaldehyde im-
med. discharges color. 10 mg. req. 0.52 cc. N/100 NaOH to bring it back again.
Soln. still sp., but does not give ppt. on taking past former isoelec. pt. w.
acid.

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MAR 27 — Wt. bot (6) + substr. 7.3769 7.3768
7.2184
.1584

Diss. w. H_2O + dil. H_2SO_4, neutr. w. NaOH until just back in soln. again, made up to 31.68 cc. w. saline.

ASH

Wt. bot + substr 7.2187 7.1508 Wt. Pt dish + ash 23.4000
" " 7.1508 7.1038 " " " ~~23.3998~~ 23.4000
.0679 .0470.
Unweighable trace of ash.

No Ash

OPTICAL ROTATION

MAR 31 Blanks: −0.07 .08 .08 .08. Rdgs: +1.40 1.45 1.44 1.43 $l = 1$

$$[\alpha]_D = + \frac{(1.44 + 0.075)}{1} \times 2 = +303°$$

$[\alpha]_D = +303$

MICROKJELDAHLS 2 cc. samples

N/50 HCl (×1.003)	3.00	3.00	6.02
N/100 NaOH (×~~0.998~~ 1.00)	8.11	10.41	2.21
	6.01	8.11	3.81
	2.10	2.30	.55
			3.26

3.26 × 0.14 = .46

Total N 4.6%

AMINO ~~FORMOL~~ TITRATION NITROGEN

APR 3

T	P	Rdg.		Rdg. − Bl.
24°	759.5	0.15 .155 .16	Bl.	Bl.
22°	"	.640	1	.485
20.5°	760	.600	2	.445

per 100 cc soln.

$\frac{.485 \times .5631 \times 100}{2} = 13.65$ mg.

$\frac{.445 \times 5678 \times 100}{2} = 12.63$ mg.

26.28
13.14 mg. per 500 mg.

Amino N (NO_2^- Method) 2.6%

Jun 20 — Repetition of Total N by distn.

Wt. bot E + substr 7.3181 7.2980
7.2980 7.2758
.0201 .0222

cc N/50 HCl (1.995) 15.00 15.00
" " NaOH (1.015) 9.60 9.15

14.92 14.92
9.74 9.29 5.05 4.97
5.18 5.63
.11 .11
5.07 5.52
1.014 1.104

Total N 5.0%

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APR 2 — HYDROLYSIS w. HNO_2

4 cc. soln. + 6 cc. 30% $NaNO_2$ soln. + 3.5 cc. HOAc occasionally shaken for 30 min. 1.2 cc. 18 N H_2SO_4 added & HNO_2 aspirated out for 30 min. Let stand in ice-box.

APR 3 — Made up to 25 cc.

OPTICAL ROTATION

$l = 2$ $\theta = 0.03\ .01\ .02$ Rdgs. $+.26\ .26\ .28\ .26$

$$[\alpha]_D = + \frac{.28 \times 100}{2 \times .08} = +175°$$

Redg. Sugars 3 cc. samples.

Bl. 19.20 (Soda) 19.50 Rdgs. 14.35

19.00

14.35

4.65 × 1.02 × .318 = 1.54 mg. Cu = 0.78 mg. glucose

Redg. sugars 33%

32.5% Redg. Sugars

~~Sug in sample~~

.8 × 3 mg. in sample

Bl. 19.00 Rdgs. 16.00 16.25

16.13

2.87 × 1.02 × .318 = .93 mg. Cu = 0.55 mg. glucose

23%

APR 6 — Started again w. 3 cc. soln. + 4 cc. 30% $NaNO_2$ soln. & 2 cc. HOAc. 0.8 cc. 18 N H_2SO_4 added & HNO_2 aspirated 1¼ hrs. Made up to 25 cc. Had 10 cc. reagent by mistake. 5 cc. sample

Bl. 38.00 38.00 ✓

32.75 32.65

5.35 × 1.02 × .318 = 1.74 mg. Cu = 0.87 mg. Cu

Repeated w. 5 cc. reagent.

Bl. 19.00 19.00

14.30 14.30

4.70 × 1.02 × .318 = 1.52 mg. Cu = 0.78 mg. glucose in 3 mg. sample

Redg. Sugars 26.0%

HYDROLYSIS w. HCl + $SnCl_2$.

APR 7 — 2 cc. soln. + 2 cc. H_2O + 4 cc. 2N HCl + few crysts. $SnCl_2$ B. 7 hrs. under rf. Color remained faint pink. APR 8 — Soln. to 25 cc., H_2S passed in, centfgd., aspirated, 5 cc. taken for sugar anal.

Bl. 19.00 19.00

17.00 17.00

2.00 × 1.02 × .318 = .65 mg. Cu = 0.40 mg. glucose in 2 mg. sample

Redg. Sugars 20.0%

Hydrolysis of SI with HCl and Bromine -

100 mg of SI was heated for 8 hrs with 15 cc 2N HCl and an excess of Bromine. There was a vigorous evolution of CO_2 in the beginning as shown by the ppt of $BaCO_3$ in a trap on the condenser.