GREAT LAKES RESEARCH

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HORSEHEAD INDUSTRIES, INC.

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TO: L. A. Joo' - GLR

CC: Glenn Creamer - RJR
Ron Darling - GLR

FROM: K. D. Church
542-1739

DATE: 6/14/89

SUBJECT: Plant Test Carbonization of Cotton Linters in the GLC Mini-Rebaker at Niagara Fall, NY
Re. P.O. No. 89-21635 (RJR)

The purpose of this trip was to investigate the feasibility of using the mini-rebake furnace to carbonize first cut cotton linters using standard 36"Ø x 144" L electrode double saggers fitted with a lid and a continuous nitrogen purge.

The six saggers were charged with a total of 5,114 pounds of cotton linters (three half-bales per sagger) with thermocouple placement as shown in Figure 1. The lids were put in place and tack-welded for transport. Nitrogen purge tubes were installed at the bottom of each sagger. The saggers were transported to the Niagara Falls plant and returned to GLR via a flatbed truck.

The mini-rebake furnace is a circular, gas fired, furnace 122"Ø x 155" H fitted with a water seal and an internal fan to circulate the furnace atmosphere. The furnace is equipped with an incinerator and the furnace pressure is controlled by a valve in the exhaust duct to the incinerator.

The three double saggers were placed in the furnace as shown in Figure 2. The thermocouples and nitrogen lines were passed through an opening adjacent to the sagger No. 4 position and connected to a strip chart recorder and individual flowmeters for nitrogen supply.

The saggers were purged at 200 SCFH per sagger for 16 hours prior to startup at 0800 on 5/17/89 at which time the flow was reduced to 100 SCFH during the upheat phase of the run. A daily record of temperature readings was kept, Table I, as well as a continuous strip chart recording of the entire run.

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Conthern (E) vs Mini Rebake (NF) → firing curve Δ during cooling

strength most likely occurred during transport ~~1914~~

The cooling time cycle has allowed the annual estimate for the mini-rebake to ↑ 2X to 100,000 #.

6-8 months needed to build & bring a ~~new furnace~~ ~~second rebake~~ on line. - 2-3X ↑ on mini-rebake.
(to take care of annual supplies of in-market brands).

Trip to Morgantar w/ Ron Darling - July?

Composite sample ↑ oxygen 2-6 ↑ C,H,N,O → in depth

Coke → graphite sheets → proposal from Mr. Doi to ABN

Other requirements fulfilled (if CHNO & ash OK) ↑ end ↑ 1989.
~~Would not need~~ Earliest test market would be 4Q'90.
Further scale-up operations; i.e., relocating furnace from NF to E and design of new system @ E are tabled.

FCC III data appear to be good (need to get SA approval?)

VM 2X ↑ Conthern

Freezing test & make rebake

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During the upheat phase of the run the furnace pressure was maintained at approximately 0.5" H_2O and during the cooling phase from atmospheric to 0.2" H_2O.

The firing curve for the run is shown in Figure 3 (plotted from sagger No. 1 temperatures). Comparing Figure 3 and Figure 4 (the firing curve for the GLR furnace) two major differences are observable: (1) The upheat rate between 500°C and 650°C for the mini-rebaker is much slower (20 hours vs. 6 hours), and (2) the cooling time is considerably reduced (24 hours vs. 80 hours). The upheat rate was slowed because the one burner is inadequate (the furnace originally had two burners) for the desired rate and the cooling efficiency is the result of the internal circulation fan. Note that the furnace and saggers were allowed to cool for an extended period (64 hours) before the saggers were removed.

When the required 650°C was reached in all saggers the nitrogen purge was increased to 200 SCFH and maintained at that level until the saggers were below 175°C then reduced to 150 SCFH and reduced to 100 SCFH at 55°C and kept at this rate until removal from the furnace.

The nitrogen purge was maintained while the saggers were sealed and then the supply tubes were disconnected and sealed. A comparison of sagger volumes and nitrogen flows for the GLR furnace and the Niagara Falls furnace is shown in Table II.

Table III is a detailed account of the unloading of the saggers at GLR. As noted there was a considerable amount of oxidation in saggers 2 and 6 and only sagger 1 showed no evidence of oxidation. The oxidation is probably the result of air infiltration during cooling and transport. The pattern of oxidation is shown in Figure 5 and photos of the oxidized material from sagger 2 are shown in Figures 6 and 7. An unusual burning pattern is shown in these photos. The air apparently infiltrated into the bale at a crack and oxidation began inside the bale leaving about a 1" outer shell and progressing to either end and down the sides of the bale leaving an unoxidized outer shell and inner core.

The char yield for this run is shown in Table IV. Included in this list are the 1/2 bale raw weight and resulting char weight, drum number as shipped and the respective sagger number. The overall yield of 24.7% was calculated on the basis of unoxidized material (suspect material not included).

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Samples were taken from top, middle and bottom bales in sagger No. 1 and submitted for the standard analysis and the FCC III analysis. Samples for VM, ash and moisture were taken from middle bales in saggers 3, 4 and 5 and from the bottom bale in saggers 2 and 6. These data are shown in Tables V and VI along with the average data from twelve GLR runs for comparison.

Keith D. Church

Keith D. Church

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Attachments

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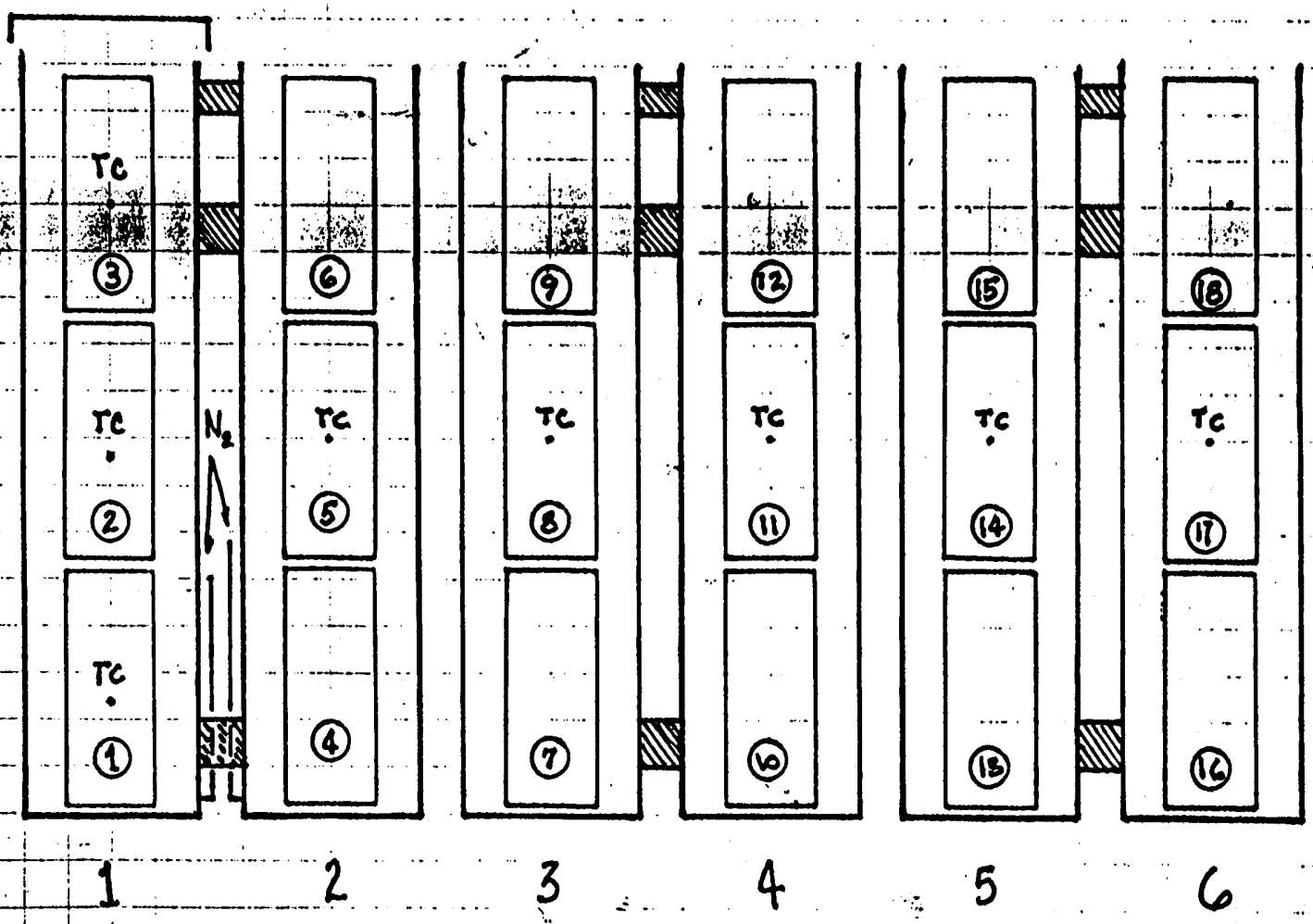
Figure 1

SAGGER LOADING AND TC PLACEMENT FOR MINI-REBAKER RUN



1 = SAGGER NUMBER

① = ½ BALE NUMBER

Tc = THERMOCOUPLE

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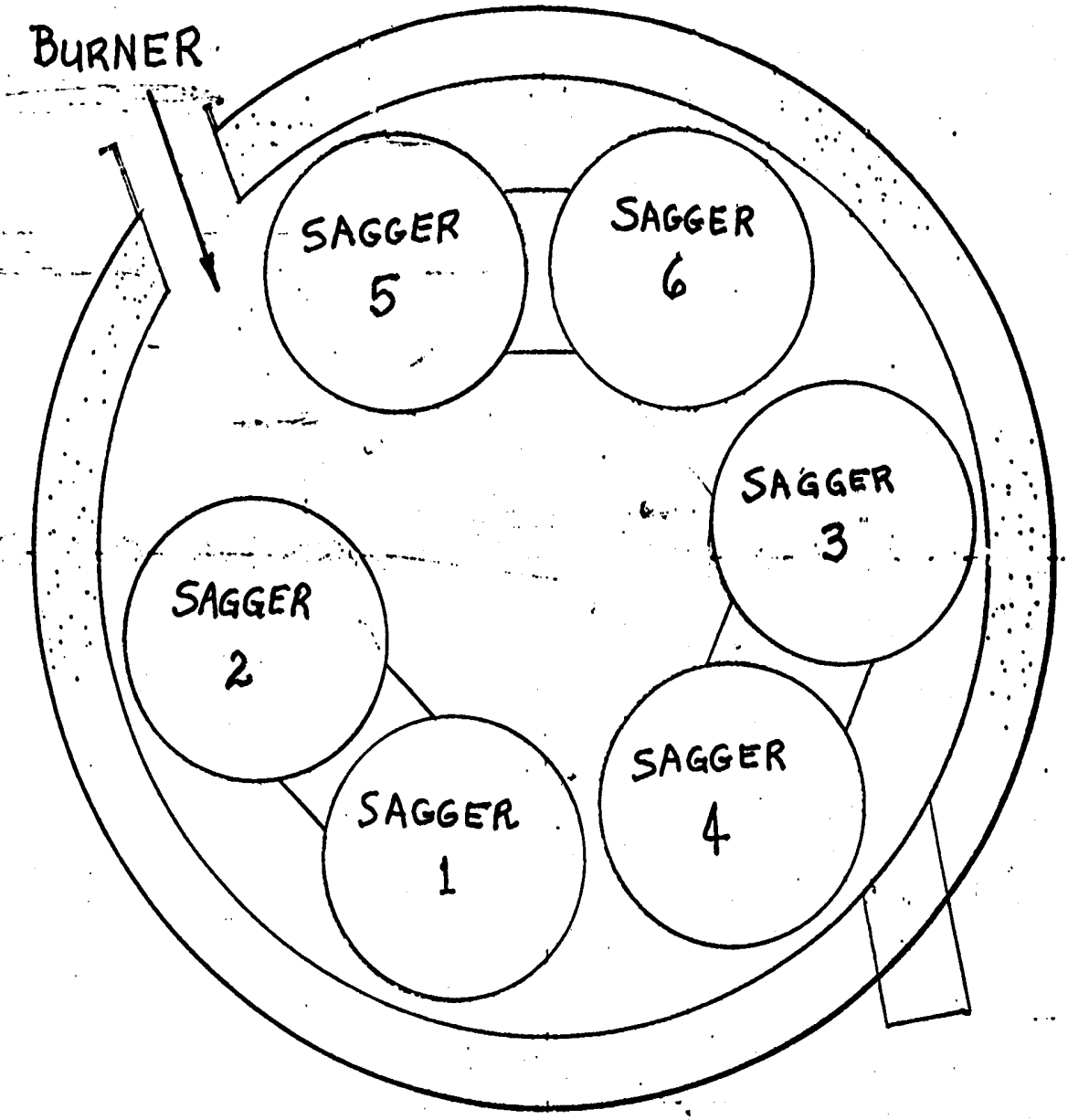
Figure 2

6/V 89



FURNACE LOADING ARRANGEMENT
MINI-REBAKER RUN - NF

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Table I

Cotton Linters Carbonization
Niagara Falls Plant Test
Mini-Rebaker Run - 5/15-22/89

Sagger No.		1	1	1	2	3	4	5	6	Furnace	Nitrogen	
Date	Time	Bot °C	Mid °C	Top °C	Mid °C	Mid °C	Mid °C	Mid °C	Mid °C	Cont. °C	SCFH	
5/15	1699									Room	200	Purging
5/16	0800									Room	200/100	Burner On
	1000	18	15	18	20	20	18	22	22	215	100	
	1200	20	18	20	20	22	18	21	20	275	100	
	1300	21	22	23	22	22	22	20	20	305	100	
	1400	24	26	32	30	22	22	28	28	325	100	
	1500	30	38	43	40	32	30	33	38	342	100	
5/17	0800	425	388	388	420	200	198	355	225	587	100	
	0900	470	440	445	470	250	240	415	-	594	100	
	1000	510	490	498	508	311	310	468	-	599	100	
	1100	530	518	523	526	370	362	500	-	603	100	
	1200	538	532	538	535	410	402	515	-	603	100	
	1300	550	548	550	549	450	448	530	-	604	100	
	1400	556	552	558	552	478	475	540	-	608	100	
	1500	560	558	560	555	500	499	545	-	614	100	
	1600	568	570	568	562	520	518	552	-	622	100	
	2300	605	611	610	600	600	600	598	-	662	100	
	2400	610	620	616	610	606	608	602	-	670	100	
5/18	0800	650	660	655	650	650	650	645	-	730	100	
	0900	668	670	670	668	660	662	655	-	740	100/200	Burner Off
	1000	485	488	485	485	480	480	475	-	460	200	
	1100	360	360	358	358	363	362	352	-	320	200	
	1200	258	252	254	252	280	278	250	-	220	200	
	1300	190	190	190	190	230	230	185	-	180	200	
	1400	171	170	171	172	230	215	170	-	130	200/150	
5/19	0800	55	48	48	52	142	120	45	-	40	150/100	
	0900	52	48	45	51	145	118	45	-	42	100	
	1000	48	45	42	50	142	115	42	-	40	100	
	1630	48	40	40	48	142	115	40	-	40	100	
5/20	1100	42	38	38	45	120	112	38	-	37	100	
5/21	0730	42	40	40	54	162	80	25	-	40	100	
5/22	0800	50	42	52	54	172	52	50	-	40	100	Shutdown Unload

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Figure 3

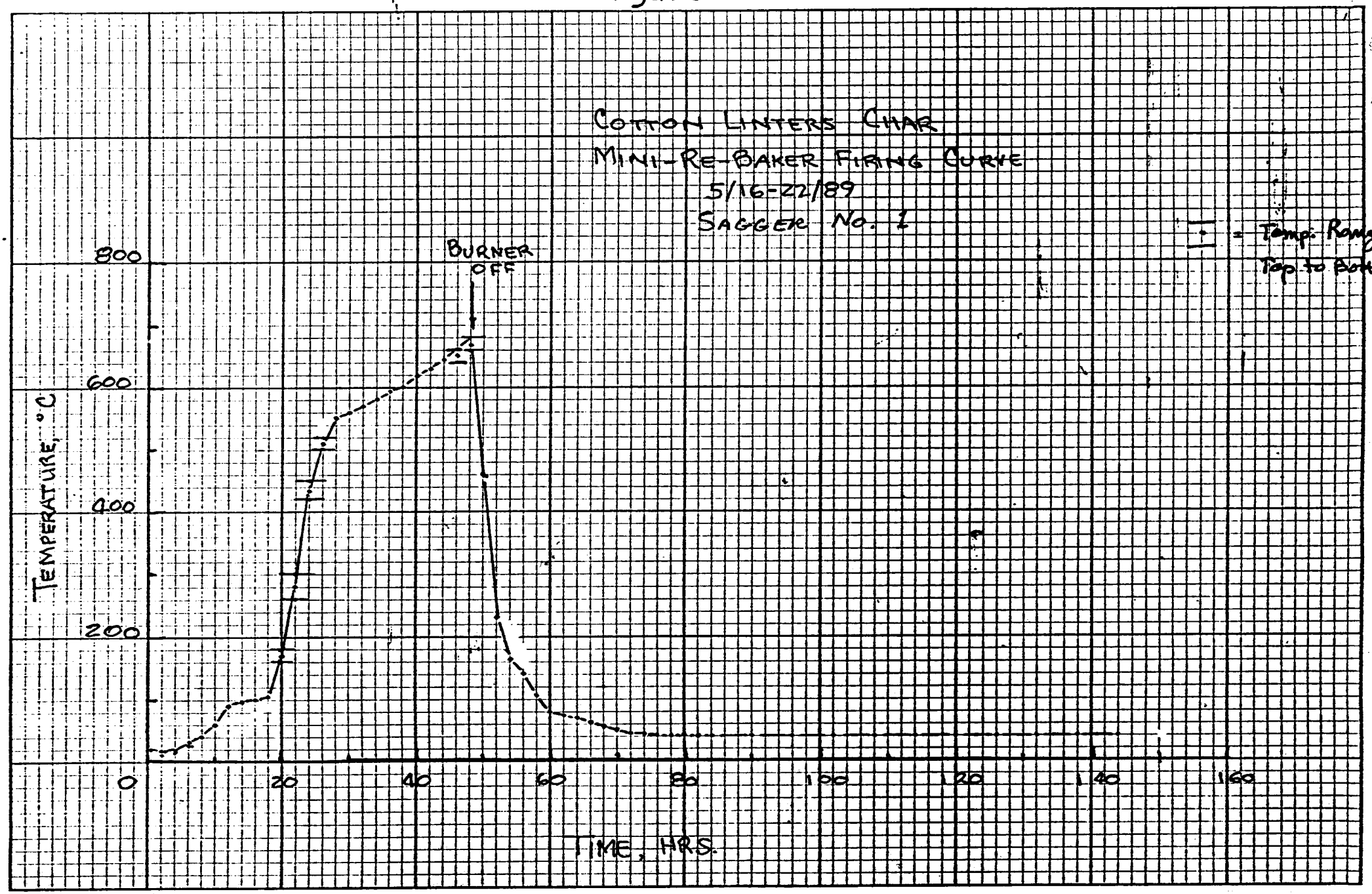

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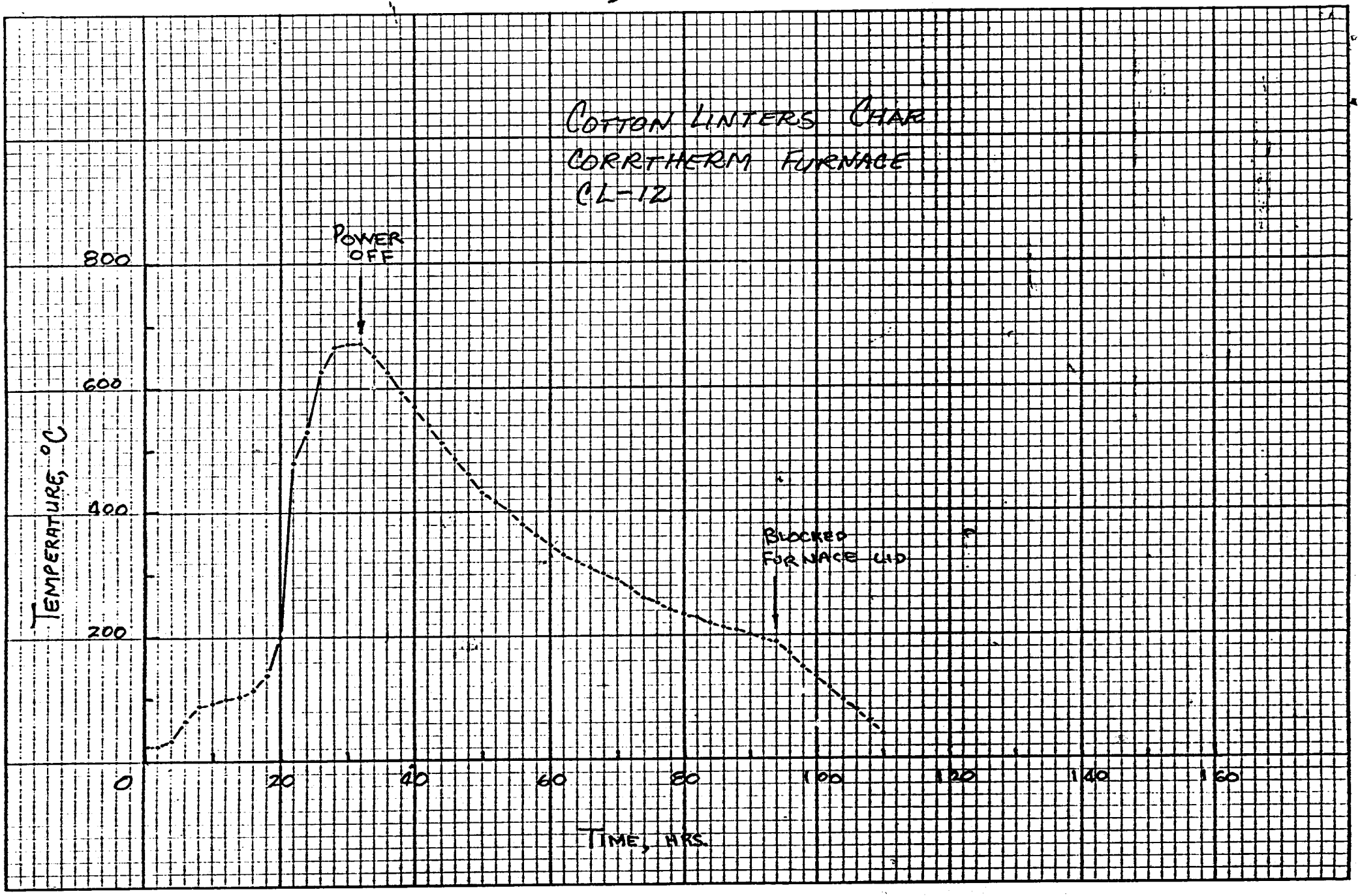
Figure 4



CITADEL© NO. 642 - CROSS SECTION - 10 SQUARES TO INCH

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Table II

Cotton Linters Carbonization
Niagara Falls Plant Test
5/15-22/89

N_2 Flow Rate Calculations

1) Corrtherm Furnace

Sagger = 48" h x 34" x 34" = 32.1 cu. ft.
1/2 CL Bale = 48" h x 25" x 24" = 16.7 cu. ft.
Free Space = 15.4 cu. ft.

N_2 Flow: Purge 2 hr. @ 100 SCFH
Upheat 50 SCFH
Cooling 100 SCFH

2) Mini-Rebaker

Sagger = 146" h x 36"Ø = 86.0 cu. ft.
3 - 1/2 CL Bales = 3 (48" h x 25" x 24") = 50.0 cu. ft.
Free Space = 36 cu. ft.

N_2 Flow: Purge 16 hrs. @ 200 SCFH
Upheat 100 SCFH
Cooling 200 SCFH to 170°C
150 SCFH to 50°C
100 SCFH to 40°C

Charred CL

1/2 Bale 21" x 22" x 47" h = 12.6 cu. ft.

$$\text{Wt.} = \frac{60 \text{ lb.}}{12.6 \text{ cu. ft.}} = 4.8 \text{ lb/cu. ft.}$$

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Table III

Cotton Linters Carbonization
Niagara Falls Plant Test
Mini-Rebaker Run 5/15-22/89

Sagger Unloading and Inspection:

Sagger #1

No evidence of oxidation, all bales look excellent.

Sagger #2

Top Bale - Severely oxidized with outer shell of unoxidized material and core of unoxidized material (pictures).

Middle Bale - Top half of bale severely oxidized with outer shell of unoxidized material and core of unoxidized material (pictures).

Bottom Bale - No oxidation, looks good.

Sagger #3

Top Bale - Partially oxidized, saved center section.

Middle, Bottom Bales - No oxidation, look good.

Sagger #4

Top Bale - Severly oxidized, began at the middle of the bale and progressed toward either end with a shell of unoxidized material. Bale caught fire upon removal from the sagger.

Middle Bale - No internal oxidation, slight amount of ash from top bale.

Bottom Bale - No oxidation, looks good.

Sagger #5

Top Bale - Severely oxidized, began at the middle of the bale progressing toward both ends and down the sides leaving a shell of unoxidized material which was thinner at the point of ignition.

Middle Bale, Bottom Bale - No oxidation, look good.

Sagger #6

Top Bale - Severely oxidized with shell of unoxidized material.

Middle Bale - Still burning when removed from the sagger. Same pattern as previously described.

Bottom Bale - No oxidation, looks good. This bale was sampled at the edge and center for ignition-in-air test.

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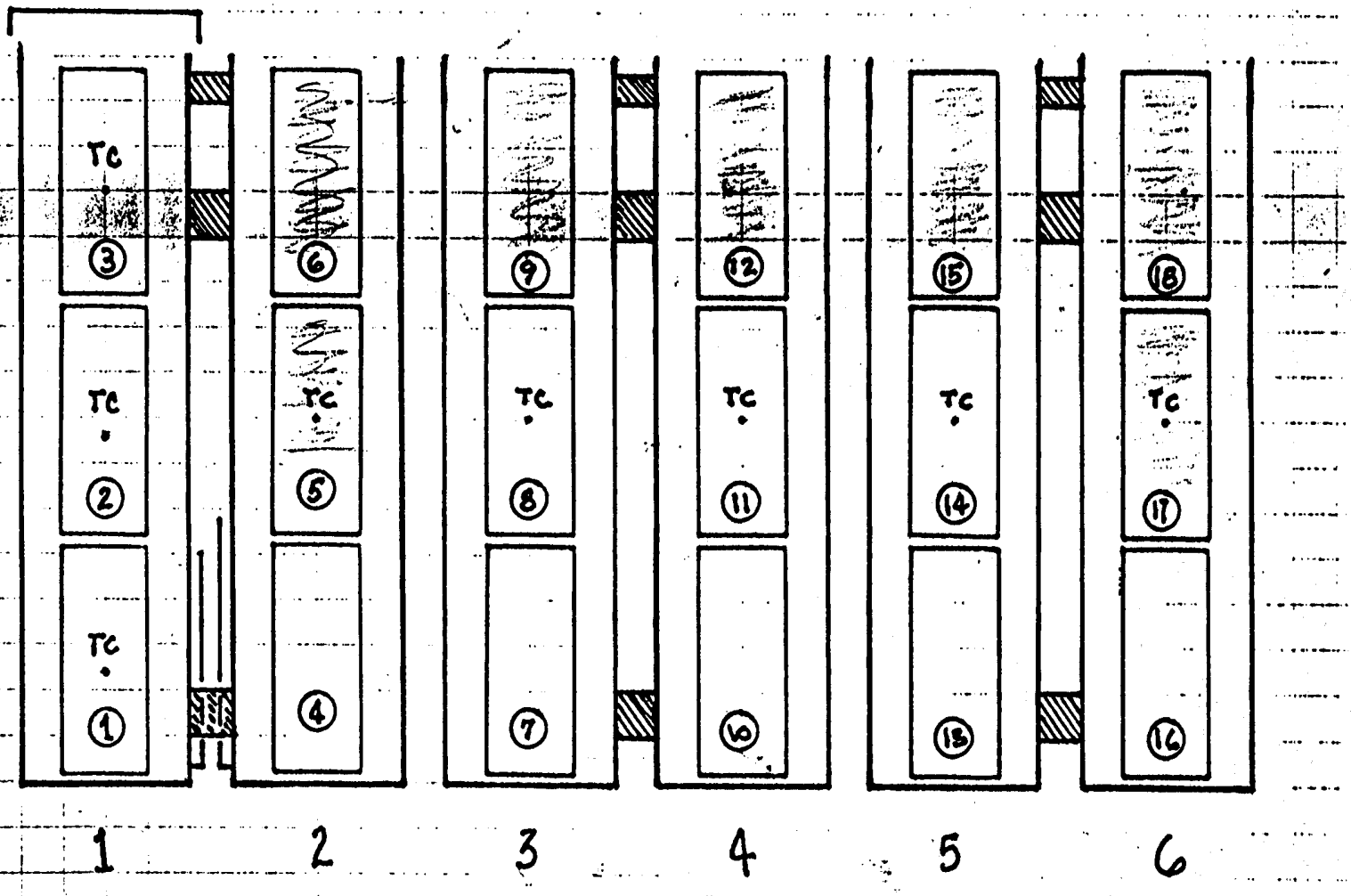
6/1/89

Figure 5

SAGGER LOADING AND TC PLACEMENT FOR MINI-REBAKER RUN



1 = SAGGER NUMBER

① = ½ BALE NUMBER

Tc = THERMOCOUPLE

[ ] = BALES SHOWING OXIDATION

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Figure 6

SAGGER No. 2 TOP

SAGGER No. 2 TOP

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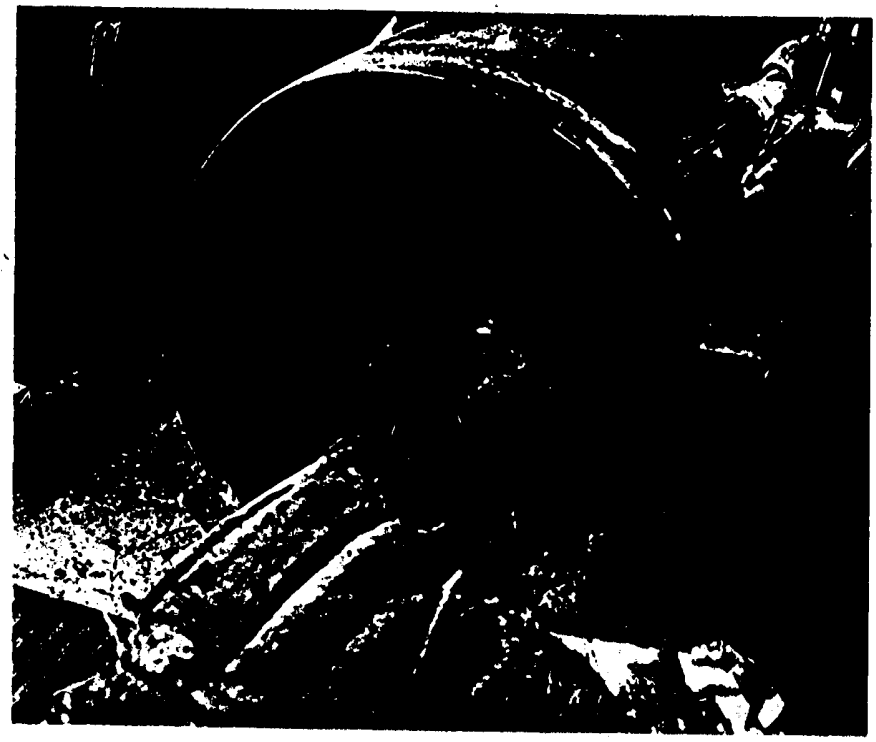
Figure 7



SAGGER No. 2 MIDDLE

SAGGER No. 2 MIDDLE

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Table IV

Char Yield
Mini-Rebaker Run

Sagger No.	1/2 Bale No.	Raw Wt. (lb.)	Drum No.	Char Wt. (lb.)	Yield (%)
1	1	275	6 5	33 37	25.5
	2	300	4 3	36 40	25.3
	3	277	2 1	35 35	25.3
2	4	290	12 11	38 36	25.5
	5	289**	10* 9*	31** 22**	-
	6	270**	8* 7*	23** 26**	-
3	7	253	18 17	30 35	25.7
	8	305	16 15	39 33	23.6
	9	306**	14* 13*	27** 21**	-
4	10	271	24 23	37 32	25.5
	11	319	22 21	41 39	25.1
	12	267**	20 19	- -	-
5	13	308	30 29	33 36	22.4
	14	272	28 27	39 27	24.3
	15	272**	26* 25*	28** 29**	-
6	16	298	36 35	39 34	24.5
	17	240**	34 33	- -	-
	18	302**	32 31	-	-
		5114		991	
	Burned or Suspect	1946	Suspect	207	
		3168 lb.		784 lb. =	24.7%

*Suspect material, exterior oxidation has been removed by brushing and vacuum.

**Weight not included in total for yield calculation.

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7-25 Keith Church

SA # in out 33 m^2/g

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Table V

Cotton Linters Char
Standard Analysis Record

Lot No.	Raw Linters % VM	% Ash	% H_2O	Carbonized Linters % VM	% Ash	% H_2O	% C	% H	% N	% O	BET SA	RD g/cc	Comment
Average	84.1	1.02	5.7	4.06	4.12	1.31	89.8	1.53	0.72	4.01	1.3657	1.50	Corrtherm Data
Sag. 1T													
1M	Composite			5.77	3.85	3.15	89.6	1.94	0.69	3.92	1.7037	1.47	Mini-Rebaker Data
1B													
2B				8.72	3.69	3.04							
3M				8.68	3.83	3.98							
4M				7.45	3.84	4.27							
5M				8.65	3.78	4.26							
6B				8.32	4.10	4.48							
				$\bar{x}$ 8.36	3.85	4.01							

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Composite sample for C,H,N+O for saggers 2-6

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Table VI

Cotton Linters Char
FCC III Analysis

Lot No.	Arsenic	Cyanogen Compounds	Heavy Metals	Higher Aromatic H-C	Lead	Water Extract-ables	Loss on Drying	Residue on Ignition	A & B Identifi-cation	
Average	<3 ppm	Passes Test	<0.004%	Passes Test	<10 ppm	1.22%	1.12%	4.64%	Passes Test	Corrtherm Data
Sagger 1 Composite	<3 ppm	Passes Test	<0.004%	Passes Test	<10 ppm	2.82%	2.77%	4.55%	Passes Test	Mini-Rebake N.F. Run

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