express their appreciation to the corporation's Applied Research Laboratory for permission to use some of the data presented herewith. The contribution of the institute's Research Services Department is also gratefully acknowledged.

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A Laboratory Evaluation of Pitch Binders Using Compressive Strength of Test Electrodes

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A MAJOR USE FOR coal-tar pitch is as an electrode binder for Soderberg electrodes in the aluminum industry. One of the methods for determining whether a pitch is of suitable quality for use in production electrodes is to prepare and bake a test electrode using the pitch and a standard calcined petroleum coke, and then to determine the compressive strength and other physical characteristics of the electrode. However, there appears to be very little published on methods for preparing and testing carbon electrodes. One method (5) involves the preparation of relatively large electrodes (approximately 6 inches in diameter and 8.5 inches long), which requires large and expensive equipment for mixing and baking. An improvement of this method was developed so that readily available laboratory-size equipment could be used to prepare a batch of four test electrodes, 1.25 inches in diameter and 4 inches long.

This procedure was used in a program to relate various properties of a pitch to its electrode-binder characteristics.

EXPERIMENTAL

Preparation of Test Electrodes. The electrode paste, which is made from a mixture of the pitch and petroleum coke particles (Table I), is baked in a graphite mold. The molds are prepared from graphite rods 1-5/8 inches in diameter (National Carbon Co., Type AGX or AJX) which are cut into pieces 5 inches long. A hole 1-1/4 inches in diameter is drilled throughout the length of each piece. The drilled hole is reamed with a tapered reamer so that the inside diameter of the one end is 1-9/32 inches and the other end is 1-1/4 inches. This taper permits easier removal of the electrode after baking. The inside of the mold is then lined by gluing in a layer of Kraft wrapping paper. A template is then used to aid in drilling 96 vent holes 0.076 inch in diameter (No. 47 drill) through the graphite shell of the mold (Figure 1). If desired, the molds can be re-used several times by cleaning, relining, and punching vent holes in the liner.

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The pitch is broken into small pieces and placed in a steam-jacketed, 1-quart sigma blade mixer (Charles Ross and Son, Brooklyn, N. Y.), where it is melted at a temperature which is approximately 30° to 40° C. above the softening point. For pitches with a softening point of about 90° C., 10 minutes at a temperature of 130° C. is usually sufficient. The mixer blades are placed in motion, and the previously heated (120° to 130° C.) petroleum coke particles are added, starting with the coarsest fraction and allowing 5 minutes of mixing between the addition of fractions. After the last fraction is added, the paste is mixed for 5 minutes.

While the paste is being mixed, four of the graphite molds are heated to about 120° C. using an electrical beaker mantle in which the molds rest on a flat graphite plate. The molds are gradually filled in turn with small portions of paste taken directly from the mixer using a metal rod as a tamper to achieve a uniform density. The paste behaves as a heavy fluid after it has been evenly packed in the mold. Each is filled to within 1/2 inch of the top.

The filled molds are then allowed to cool until the paste forms a hard solid. Two grams of petroleum coke particles, 200 to 325 mesh, are placed on top of the solidified paste.

Table I. Composition of Paste Used in Preparing Test Electrodes

Component	Weight, G.	Wt. %
Pitch	262.8	31.1
Calcined petroleum coke		
10 to 30 mesh[a]	116.7	13.8
30 to 50 mesh	93.4	11.0
50 to 100 mesh	110.9	13.1
100 to 200 mesh	75.9	9.0
200 to 325 mesh	58.4	6.9
325 to pan	128.2	15.1
Total	846.3	100.0

[a] U. S. Standard sieve sizes.

Figure 1. Various stages
in the preparation of
a graphite mold
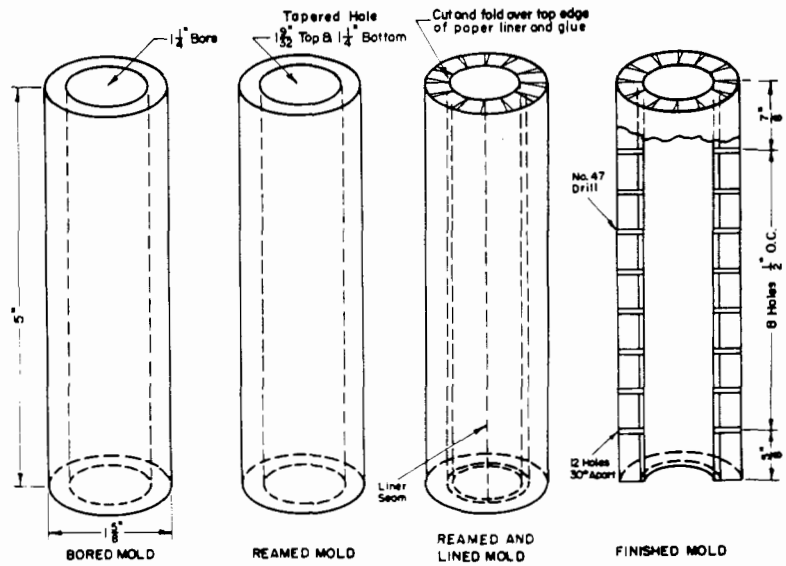
Table II. Baking Schedule for Test Electrodes

Temperature, ° C.	Time, Hours
25 to 200	2.0
200 to 500	12.0
500 to 1000	8.5
Soak at 1000	1.0
Total	23.5

The four molds are placed in a holder (Figures 2 and 3) which is used during the baking. Nine-pound steel weights are placed on top of the paste to simulate somewhat crudely the weight of the unbaked paste above the Soderberg electrode in an actual furnace. The weights are kept in place and guided by the top of the mold holder.

Description of Baking Apparatus. A schematic diagram of the entire baking assembly is given in Figure 4. The electrodes are baked in a specially designed retort fabricated from Type 310 stainless steel (Figure 5). Openings are provided for a Chromel-Alumel thermocouple, the nitrogen purge gas, and an exit tube for the vapors carried out by the gas. The exit is wrapped with Nichrome heating wire and heated to approximately 300° to 350° C. to prevent the pitch vapors from condensing. The furnace used is a Hevi Duty Model 506 electrical crucible furnace with an opening 5 inches in diameter and 9 inches deep. A Brown Instrument Co. cam-type program controller is used to control the baking cycle over the range from 25° to 1000° C. Because of the slow response of the thermocouple inside the retort, a second thermocouple placed near the furnace heating element is used as the controlling point. Controller cams were prepared by trial and error in order to obtain the proper temperature program as measured by the thermocouple inside the retort.
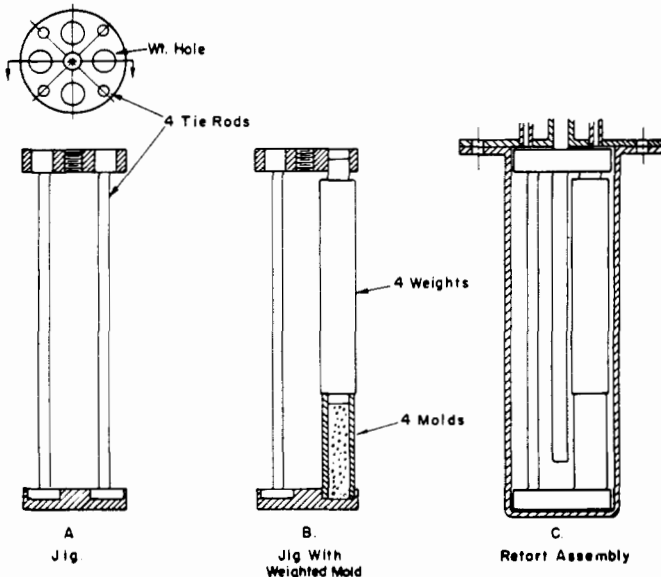
Baking of Electrodes. The holder containing the molds is placed in the retort, and the top is bolted on using a metallic gasket to obtain an airtight seal. Nitrogen which has been passed over copper gauze at 600° to 650° C. to remove traces of oxygen is used as an inert atmosphere to prevent oxidation inside the retort during baking and cooling. The electrodes are baked according to the schedule given in Table II. After the retort has cooled to 150° C., the molds containing the baked test electrodes are removed. The electrodes are carefully removed from the molds by pressing them out with the aid of a hydraulic press.

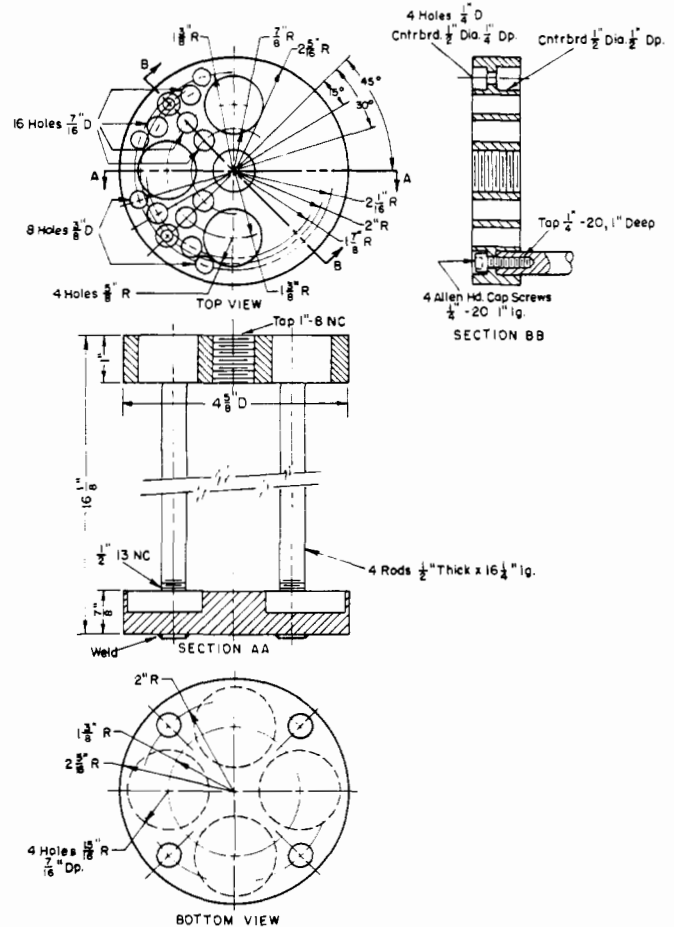Figure 2. Loading of filled molds in holder and retort

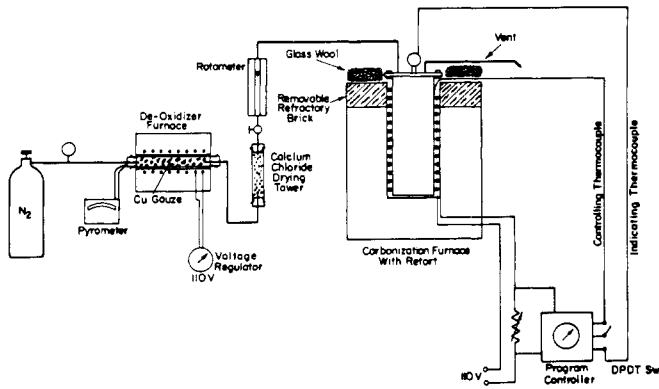Figure 3. Construction details of mold holder and weight guide

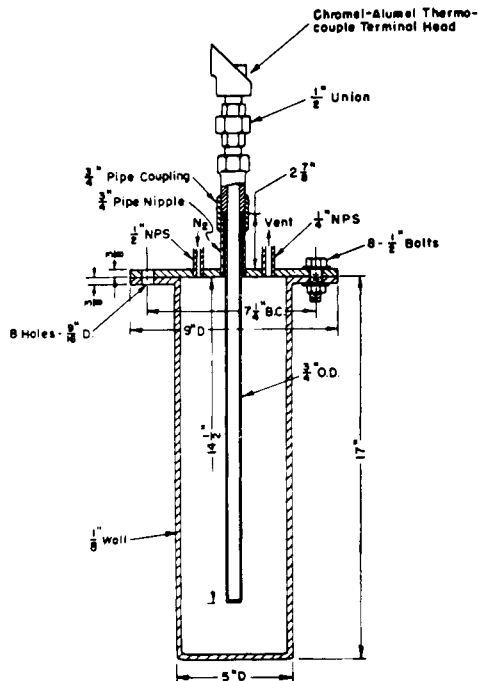Figure 4. Schematic diagram of apparatus for baking test electrodes

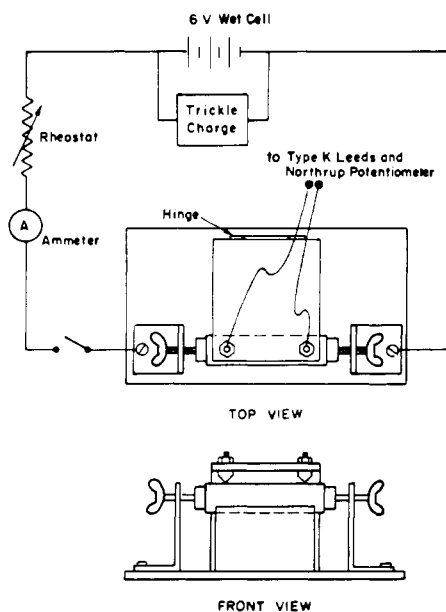Figure 5. Retort for baking test electrodes

Figure 6. Apparatus for measuring electrical resistivity

Electrical Resistivity of Electrode. The test specimen is prepared for the electrical resistivity measurement by facing off each end on a lathe so that the ends are parallel and the distance between them is 3-1/8 inches. The resistivity is then measured with the apparatus shown in Figure 6. The specimen is firmly secured between the brass plates. The fixed brass points (3.0 inches apart) to which the potentiometer leads are attached are placed against the electrode and held firmly in place by a 1000-gram weight. A known current is passed through the electrode by closing the circuit, and the potential drop is measured with the potentiometer. Four readings, 90° apart, are taken around the circumference of the electrode and averaged. The resistivity is then calculated from the following formula:

$$\rho = \text{resistivity (ohm-cm.)} = \frac{EA}{IL}$$

where E (volts) is the potential drop, A (sq. cm.) is the area of the end of the electrode, I (amperes) is the current in the system, and L (cm.) is the distance between the contact points.

Apparent Density. The apparent density of the test specimen of the electrode in grams per cubic centimeter is determined directly by dividing the weight (in grams) by the total volume (in cubic centimeters).

Compressive Strength. Each specimen is then cut on a band saw to obtain two pieces, which after grinding on a surface grinder in a special holder (Figure 7) to obtain parallel ends, yield pieces 1.25 inches in height.

The compressive strength of each piece is determined on a compression-testing machine (Tinius-Olsen Co.) using a ram speed of 0.05 inch per minute. The results of all the determinations (usually six to eight) are then averaged.

RESULTS AND DISCUSSION

The preliminary work on developing the method was based on the use of a 46-hour baking cycle. To shorten the amount of time invloved in obtaining results, baking cycles of 15 and 23.5 hours were tried. The results for four different pitches are given in Table III. The data indicate that, over the range studied, the length of the baking cycle does not have a significant effect on the compressive strength. However, with the 15-hour cycle, the electrodes showed a tendency to develop cracks on the surface. Because of this, the 23.5-hour schedule was adopted as the standard baking period.
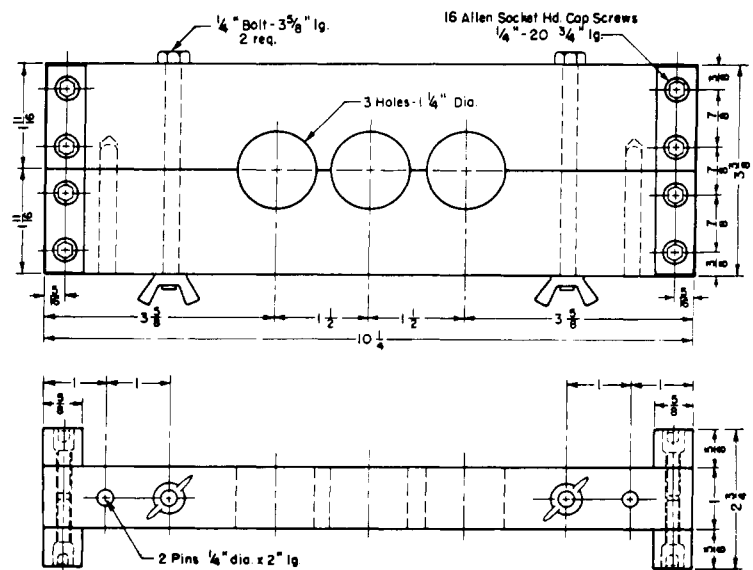

Figure 7. Holder for grinding ends of test specimens for compressive-strength determinations

Table III. Effect of Length of Baking Cycle on Compressive Strength of Electrodes

| | Compressive Strength, Kg./Sq. Cm. | | |
Pitch[a]	15-hour	23.5-hour	46-hour
L	487	470	481
B	...	295	281
E	...	297	295
H	...	319	335

[a] See Table IV for pitch properties.

Compressive strength determinations, electrical resistivities, and apparent densities for a typical batch of test electrodes are given in Table V. Although the compressive strength determinations within a batch show a somewhat high deviation, the fact that the final compressive strength value is the average of six to eight results gives reproducible results between batches. This is demonstrated in Table VI, where duplicate determinations are given for five different pitches.

It has been reported (4) that a correlation existed between the compressive strength of a test electrode and the product of the coking value and atomic carbon to hydrogen ratio (characterization factor 1). This factor has been calculated

Table IV. Properties of Pitches Used for Preparing Test Electrodes

Pitch Sample	Softening Point, °C. Cube-in-Air [2]	Benzene-Insoluble, Wt. % [3]	Coking Value, Wt. % [1]	C-H Ratio	Characterization Factor No. 1[a]	Compressive Strength, Kg./Sq. Cm.
A	86.0	26.2	53.4	1.82	97.2	463
B	88.2	13.0	49.8	1.60	79.7	357
C	88.6	31.7	53.5	1.71	91.5	426
D	89.0	33.2	58.6	1.80	105.5	549
E	89.2	20.2	51.9	1.64	85.1	394
F	90.2	32.6	57.1	1.93	110.2	577
G	90.6	17.5	50.2	1.78	89.4	461
H	91.1	21.2	50.1	1.75	87.7	424
I	93.5	29.7	54.1	1.87	101.2	492
J	94.9	28.0	52.7	1.76	92.8	541
K	103.0	30.8	56.4	1.83	103.2	495
L	106.3	27.9	59.5	1.79	106.5	470

[a] Coking value multiplied by the atomic carbon-hydrogen ratio.

Table V. Characteristics of Electrodes Produced from Pitch G

Whole Electrode			Electrode Sections	
Electrode no.	Apparent density, g./cc.	Resistivity, ohm-cm.	Section	Compressive strength, kg./sq. cm.
1	1.43	0.0064	Top	501
			Bottom	455
2	1.41	0.0066	Top	412
			Bottom	445
3	1.40	0.0071	Top	428
			Bottom	458
4	1.42	0.0066	Top	...[a]
			Bottom	477
Average	1.42	0.0067		454

[a] Not tested.

Table VI. Reproducibility between Electrode Batches

| | Compressive Strength, Kg./Sq. Cm. | |
Pitch	Batch 1	Batch 2
C	429	422
D	557	541
E	380	408
G	468	454
I	512	472

for the pitches used in the present study (Table IV) and has been plotted against compressive strength (Figure 8). The standard deviation about the curve was calculated to be 26 kg. per sq. cm. or 5.5% of the mean of the range of compressive strangths observed. The data of Charette and Bischofberger indicate that their deviation was 19 kg. per sq. cm. or 6.2% of the mean for the range of compressive strengths that they studied.
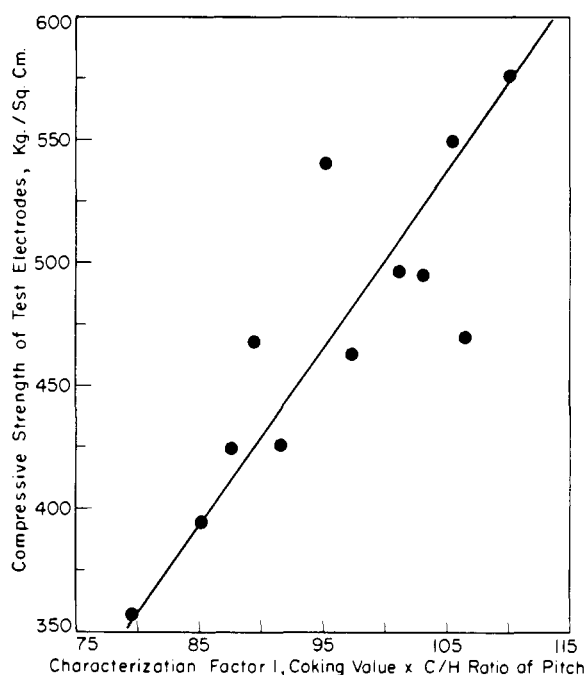

Figure 8. Relationship between compressive strength of test electrode and characterization factor No. 1 of the pitch

ACKNOWLEDGMENT

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