Not for Publication
Presented Before the Division of Gas and Fuel Chemistry
American Chemical Society
Boston, Massachusetts, Meeting, April 5-10, 1959

An Improved Evaluation of Electrode-Binder Pitches Using the Compressive Strength of Test Electrodes

by H. L. Jones, Jr.*, A. W. Simon, and M. H. Wilt
Mellon Institute, Pittsburgh 13, Pennsylvania

Introduction

One of the major uses for coal-tar pitch is as an electrode binder for Soderberg electrodes in the aluminum industry. The accepted method of 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 in the literature on the methods used for preparing and testing carbon electrodes. One method is described,[1] but it involves the preparation of relatively large electrodes (approximately 6 inches in diameter and 8.5 inches long), which necessitates the use of large and expensive equipment for mixing and baking. An improved modification 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 (see Table I) is baked in a graphite mold. The molds are prepared from graphite rods 1-5/8 inches in diameter (National Carbon Company, 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 inches in diameter (#48 drill) through the graphite shell of the mold (see Figure 1). If desired, the molds can be reused several times by cleaning, relining, and punching vent holes in the liner.

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

* Present Address: Atlas Powder Company, Wilmington, Delaware

Table I

Composition of Paste Used in Preparing Test Electrodes

Component	Weight, g	Weight Per Cent
Pitch	262.8	31.1
Calcined Petroleum Coke		
10 to 30 Mesh*	116.7	13.8
30 to 50 Mesh	93.4	11.0
50 to 100 Mesh	110.0	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

* U. S. Standard Sieve Sizes

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 mold is filled to within one-half inch of the top.

The filled molds are then allowed to cool and the paste forms a hard solid. Two grams of petroleum coke particles, 200 to 325 mesh, are placed on top of the solidified paste. 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 tube is wrapped with nichrome heating wire 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 Company cam-type program controller is used to control the baking cycle over the range from 25 to 1000°C. Due to 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 the 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 600-650°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.

Table II

Baking Schedule for Test Electrodes

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

Electrical Resistivity of the 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 g 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 degrees 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 (amp) 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 cc is determined directly by dividing the weight (in grams) by the total volume (in cc's).

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 Company) using a ram speed of 0.05 inches per minute. The results of all the determinations (usually 6 to 8) are then averaged.

Results and Discussion

The preliminary work on developing the method was based on the use of a 46-hour baking cycle. In order to shorten the amount of time involved in obtaining results, baking cycles of 15 and 23.5 hours were tried. The results obtained 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.

Table III

Effect of Length of Baking Cycle on the Compressive Strength of the Electrode

Pitch*	Compressive Strength, kg/sq cm		
	15-Hour	23.5-Hour	46-Hour
L	487	470	481
B	--	295	281
E	--	297	295
H	--	319	335

* See Table IV for pitch properties.

Table IV

Properties of Pitches Used for Preparing Test Electrodes

Pitch Sample	Softening Point, C Cube-in-Air2)	Benzene Insoluble, Wt %3)	Coking Value, Wt %4)	Carbon: Hydrogen Ratio	Characterization Factor No. 1*	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

* Coking value multiplied by the atomic carbon:hydrogen ratio.

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 6 to 8 results gives reproducible results between batches. This is demonstrated in Table VI, where duplicate determinations are given for five different pitches.

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 Bottom	501 455
2	1.41	0.0066	Top Bottom	412 445
3	1.40	0.0071	Top Bottom	428 458
4	1.42	0.0066	Top Bottom	--* 477
Average	1.42	0.0067		454

* Not tested.

Table VI

Reproducibility Between Electrode Batches

Pitch	Compressive Strength, kg/sq cm Batch #1	Batch #2
C	429	422
D	557	541
E	380	408
G	468	454
I	512	472

It has been reported[5)] 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 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/sq cm or 5.5 per cent of the mean of the range of compressive strengths observed. The data of Charette and Bischofberger indicate that their deviation was 19 kg/sq cm or 6.2 per cent of the mean for the range of compressive strengths that they studied.

Summary

In order to evaluate coal-tar pitches for use as electrode binders in carbon electrodes, a method for preparing test electrodes and measuring their compressive strengths was developed. The procedure uses laboratory-size equipment and requires less than 300 g of pitch sample to produce a batch of four electrodes. In addition to compressive strengths, the apparent densities and electrical resistivities of the electrodes are determined. The developed procedure has proved useful in ascertaining the binder quality of a wide variety of coal-tar pitches.

Acknowledgment

This work was carried out by the Coal Chemicals Project sustained by the United States Steel Corporation, to whom the authors are grateful for permission to publish these results.

References

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2. Methods of Testing Coal-Tar Products, Barrett Division, Allied Chemical and Dye Corp., 1950, p. 58.

3. Ibid., p. 27.

4. Am. Soc. Testing Materials, Philadelphia, "Standards", Part 5, D 189-52, 1955.

5. L. P. Charette and G. T. Bischofberger, Ind. Eng. Chem., 47, 1412-15 (1955).

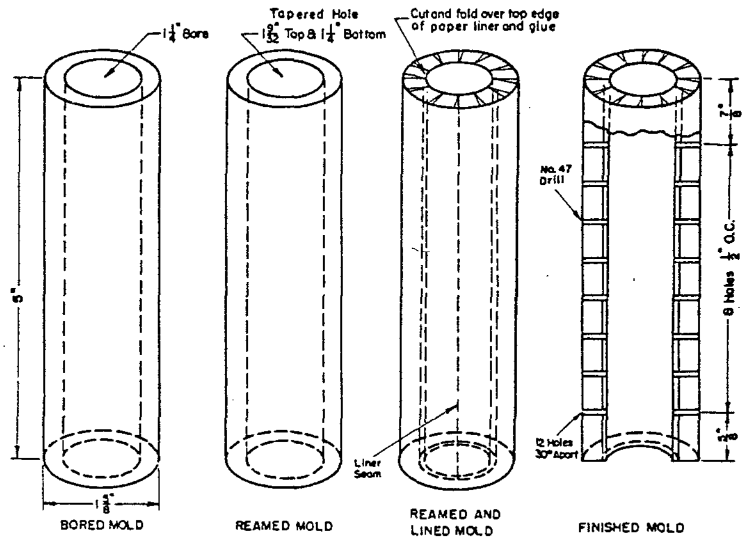

Figure 1. Various Stages in the Preparation of a Graphite Mold

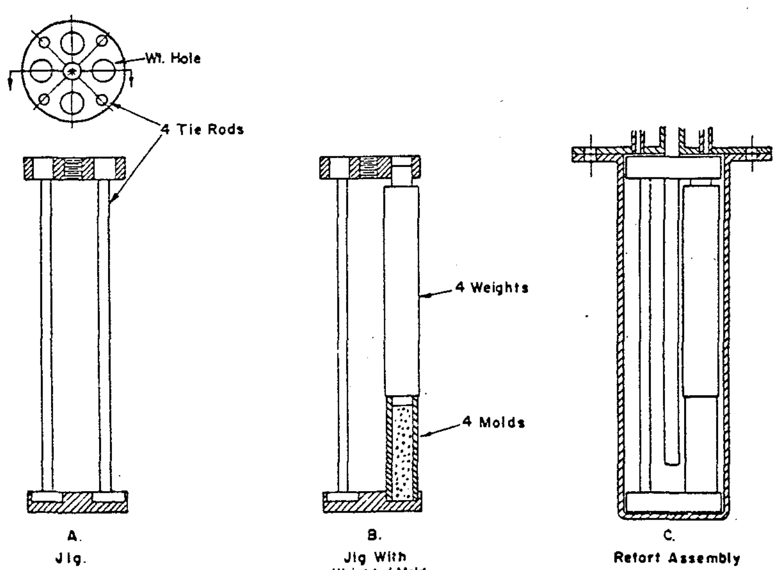

Figure 2. Loading of Filled Molds in Holder and Retort

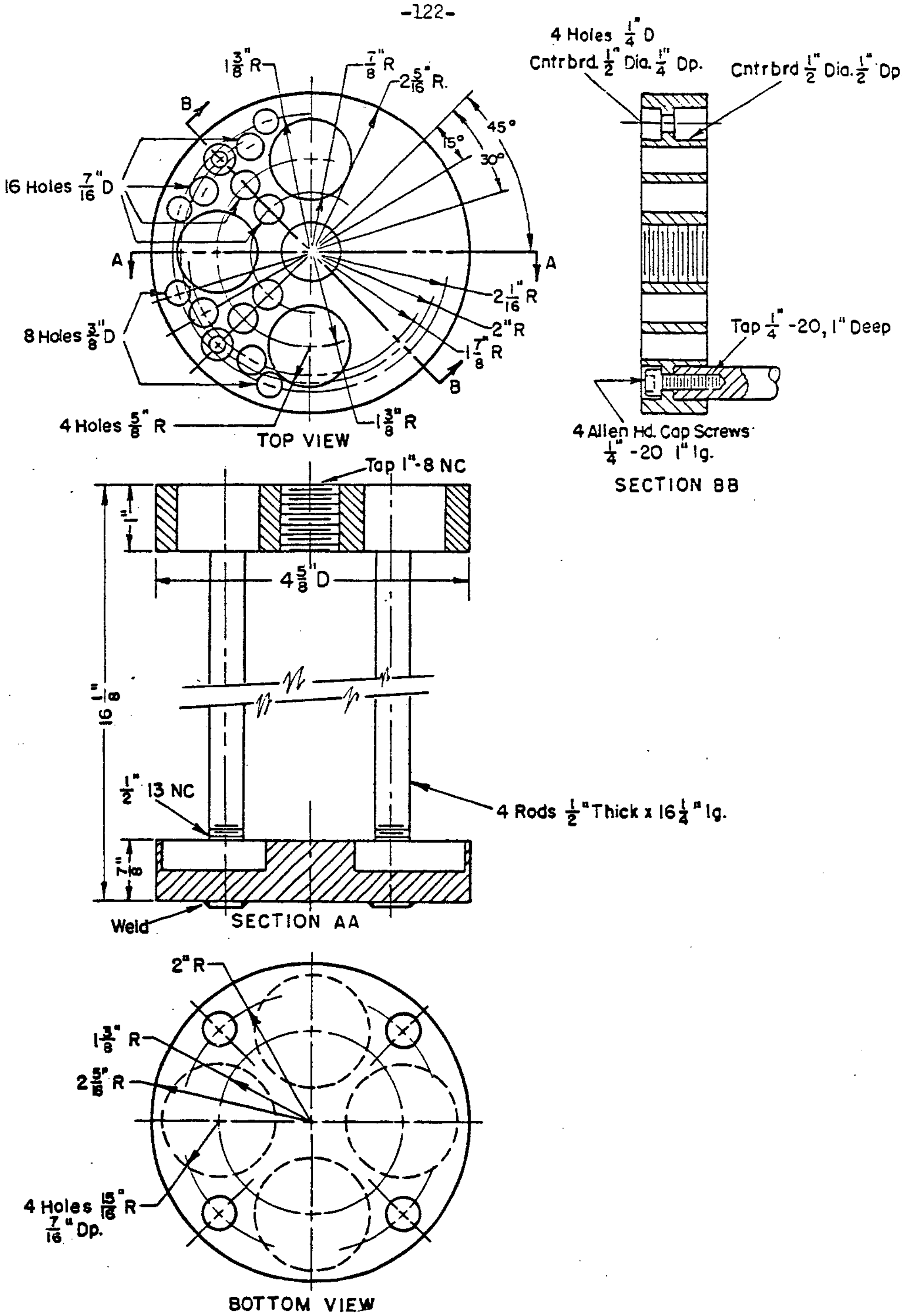

Figure 3. Construction Details of Mold Holder and Weight Guide

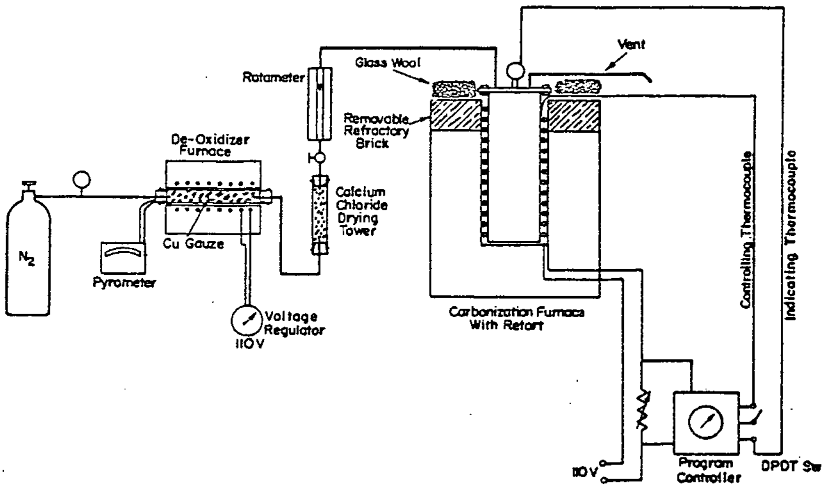

Figure 4. Schematic Diagram of Apparatus for Baking Test Electrodes

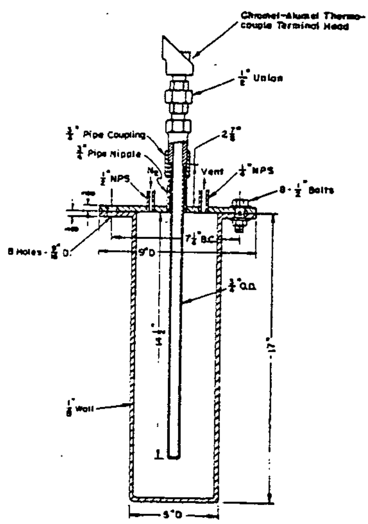

Figure 5. Retort for Baking Test Electrodes

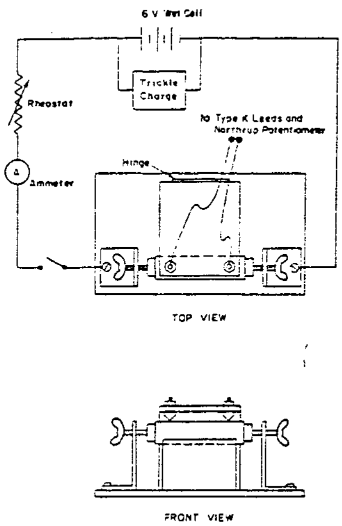

Figure 6. Apparatus for Measuring Electrical Resistivity

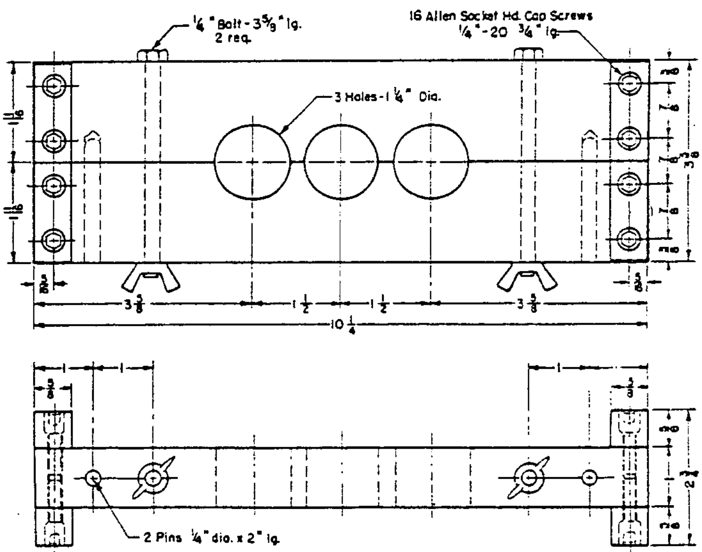

Figure 7. Holder for Grinding Ends of Test Specimens for Compressive-Strength Determinations

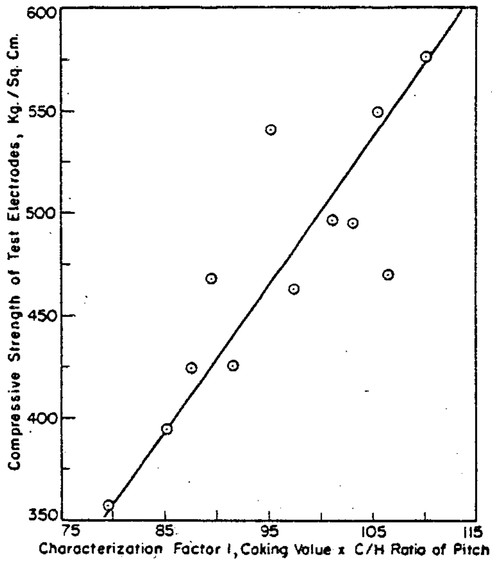

Figure 8. Relationship Between Compressive Strength of Test Electrode and Characterization Factor No. 1 of the Pitch