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REPRODUCIBILITY AND THERMAL STABILITY
OF COAL-TAR PITCH

W. E. Smith
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UNION
CARBIDE

OAK RIDGE Y-12 PLANT
OAK RIDGE, TENNESSEE

*prepared for the U.S. ATOMIC ENERGY COMMISSION
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**REPRODUCIBILITY AND THERMAL STABILITY
OF COAL-TAR PITCH**

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ABSTRACT

Two aspects of a coal-tar pitch were studied: (1) reproducibility of the properties of as-received pitch as a function of the sampling period, and (2) changes in the properties that occur on heating the pitch for extended periods at elevated temperature. Reproducibility studies were conducted on twelve samples which had sampling histories that divided them into two groups of six samples each. Data indicated little variability of properties within a sample group, but significant variability between the two groups.

A single lot of coal-tar pitch was analyzed as a function of residence time in an impregnator at 235° C. Properties reflecting increases in molecular size underwent gradual changes with increasing heating time, but the overall changes after 88 days did not appreciably alter the properties or utility of the pitch.

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SUMMARY

An analytical study was designed: (1) to determine the reproducibility of a coal-tar pitch over an extended production period, and (2) to determine the behavior of the pitch as a function of its residence time in an impregnator at 235° C. Six samples (5 lots) of the pitch that were in inventory at the beginning of the program were analyzed. Six additional samples (6 lots) were received from the vendor over a period of six months and at a rate of one sample per month. The 12 samples were analyzed for: (1) molecular weight and distribution, (2) softening point, (3) viscosity, (4) benzene insolubles, (5) elemental composition, and (6) coke yield. Nuclear magnetic resonance (NMR) methods were used to determine the distribution of protons between the aromatic and aliphatic constituents. Collectively, these data indicated: (1) significant differences in properties of samples that were already in inventory relative to those that were received from the vendor during the course of the study, but (2) little variability between samples within a respective group.

A single lot of coal-tar pitch was evaluated under impregnation conditions by analyzing samples as a function of residence time at 235° C. Data collected after a residence time of 88 days indicated: (1) no change in coke yield, (2) a slight increase in softening point, and (3) a more significant increase in benzene insolubles.

INTRODUCTION

This study is part of a continuing effort in carbon precursor technology at the Oak Ridge Y-12 Plant^{(a)(1-8)} and consists of an evaluation of coal-tar pitch with respect to reproducibility over a given production period and to thermal stability on continuous heating at elevated temperature. Twelve pitch samples (11 lots) were included in the reproducibility study which consisted of a collection of analytical data and statistical treatment of these data. The thermal stability study consisted of monitoring properties of a pitch sample on continuous heating at 235° C.

Pitch products derived from the destructive distillation of coal have been and continue to be used extensively in industrial extrusion and molding operations, and serve as a source of carbon in furnace and foundry operations. Coal-tar pitches are recovered as residuals after the distillation of light-to-heavy oil fractions from coal tar with approximately two thirds of the coal tar recoverable in pitch form. In general, coal-tar pitches have: (1) relatively high coke yields, (2) relatively low contents of ash and sulfur, (3) a variable content of free carbon, and (4) are usually classified as soft (mp, 50 - 70° C), medium (mp, 70 - 90° C), or hard (mp > 90° C).⁽⁹⁾

Factors which influence the properties of coal-tar-pitch materials include: (1) inherent properties of the coal, (2) conditions used to produce coal tar from coal, and (3) conditions used to produce pitch from coal tar. Data on currently known coal reserves indicate significant differences in composition and physical state for coals in different geographical locations.

Two classifications of coal tars that are produced by the destructive distillation of coal are: (1) retort gas tar which contains 20 - 50 percent free carbon, and (2) oven gas tar which contains 12 - 20 percent free carbon.⁽¹⁰⁾ The three primary methods that are used to distill oil fractions from coal tar with the accompanying production of pitch residuals are: (1) distillation to a hard, high-softening-point pitch (150° C or higher) and subsequent addition of tar distillates to produce pitch with the desired softening point, (2) distillation under vacuum with steam until pitch of the desired softening point is obtained, and (3) distillation in a horizontal still (without steam or vacuum) until pitch of the desired softening point is obtained.⁽⁹⁾

Though neither of the methods used to prepare the coal tar nor those used to produce pitch from coal tar seem amenable to precise control, mere standardization of procedures would allow some degree of control. However, the variability in the inherent properties of the feedstock materials (coal and coal tar) would probably be reflected in the properties of the pitch, since currently used processing would not tend to compensate for this variability. The product evaluated in this study is a vacuum-distilled pitch with a softening point of 85 - 90° C. A description of the materials and methods used in the study are included in Appendix A.

(a) Operated by the Union Carbide Corporation's Nuclear Division for the US Atomic Energy Commission.

REPRODUCIBILITY AND THERMAL STABILITY OF COAL-TAR PITCH

REPRODUCIBILITY STUDIES

Some applications utilizing pitch require a high degree of control over material properties and processing conditions. Since the time table in moving from concept to production is usually in terms of years, materials used in high-quality assurance systems must be reproducible throughout the usage period. For this reason, a study was designed to determine the reproducibility of a typical coal-tar pitch (CP 277-15 V) over a period of one year. A single sample (one drum) was to be produced and analyzed each month. After receiving six samples on such a schedule, the program was terminated because the pitch was no longer available except by special order in quantities of 10,000 pounds or more. In addition to these six samples, six samples (5 lots) that were already in inventory when the sampling program began were analyzed. Collectively, 12 samples (11 different lots) compose this sample group.

Because of the chronology of the samples, the six samples that were in inventory prior to initiation of the sampling study were designated as Group I; the remaining six samples that were procured monthly from the vendor were designated as Group II. Production data associated with the Group I samples could not be ascertained, but were estimated to be 12-24 months prior to production of the Group II samples. In analyzing the data, efforts were made to determine the variability within the two respective groups as well as between the two groups. Data collected during the study are summarized in Table 1. Analytical methods used in this study are described in Appendix A. Individual data points within Groups I and II all fall within two standard deviations of the respective means (95% confidence limits) calculated for the properties measured. How much of this variability is associated with the analytical methods is not known. However, differences in properties between groups were large, as can be observed by comparing the mean values for Groups I and II in Table 1. A test of significance (Student "t" test) at the 95 percent confidence level was used to compare the mean values between Groups I and II. Those properties that do show a significant difference are indicated in Table 1 by an asterisk. As indicated, the Group II samples contained less oxygen but more sulfur and ash than did the Group I samples. In addition, the Group II samples had higher softening points, more insolubles, and higher coke yields than the Group I samples. Molecular-size-distribution data show that the Group II samples had a lower content of high-molecular-weight species (Fraction 4) than the Group I samples, but had a correspondingly higher content of an intermediate species (Fraction 2).

In summary, coal-tar-pitch materials that were received from the vendor in 1972 were quite different from those received at an earlier (~ 1970) date. It should be stated, however, that samples received during the six-month period in 1972 were quite reproducible. No explanation can be offered for the differences in the 1970 and 1972 products.

THERMAL STABILITY STUDIES

Pitch materials are commonly used to impregnate carbon structures in order to enhance the bonding in the matrix and/or to densify the structure. Thus, to use liquid impregnation techniques requires that the impregnant be a low-viscosity fluid under processing conditions.

Table 1
PROPERTIES OF COAL-TAR PITCH
(Type CP 277-15 V)

Sample Number ⁽¹⁾	Date	Softening	Viscosity ⁽²⁾ (sec)	Average	Chloroform	Benzene	Composition in Terms of GPC Fractions ⁽⁴⁾ (%)				Oxygen	Nitrogen	Sulfur	Ash	H _A [*] (5)	N ⁽⁶⁾	f _a (7)	Coke Yield (%)
	Received	Point		Molecular	Insolubles ⁽³⁾	Insolubles	Fraction 1	Fraction 2	Fraction 3	Fraction 4	Content (%)	Content (%)	Content (%)	Content (%)				
	From the Vendor	(° C)	Weight	(%)	(%)													
Sample Group I ⁽⁸⁾																		
15V-L111-D1	(8)	84.4	17.8	327	13.9	13.7	7.9	51.1	23.9	17.1	1.34	1.04	0.31	0.12	0.80	1.15	0.94	40.8
15V-L111-D7	-	86.1	18.2	326	15.3	12.8	8.3	50.4	24.1	17.2	1.34	1.02	0.39	0.11	0.81	1.16	0.94	40.4
15V-L014-D1	-	86.0	18.5	361	15.7	13.9	10.3	47.4	22.8	19.4	1.19	0.94	0.47	0.12	0.81	1.19	0.94	41.8
15V-L105-D1	-	87.0	18.5	354	13.9	13.0	8.8	52.0	22.0	17.3	1.07	1.07	0.37	0.13	0.81	1.13	0.94	40.7
15V-L112-D1	-	89.8	15.8	-	16.0	27.3	7.3	48.0	24.5	20.1	1.23	1.03	0.45	0.16	0.79	1.20	0.93	41.2
15V-L0024-D1	-	85.0	21.8	335	20.3	20.9	8.6	48.3	24.8	18.4	1.14	1.18	0.43	0.13	0.78	1.18	0.93	44.9
Average		86.4	18.4	341	15.8	16.9	8.5	49.5	23.7	18.2	1.22	1.05	0.40	0.13	0.80	1.17	0.94	41.6
Standard Deviation		1.9	1.9	16	2.4	5.9	1.0	1.9	1.1	1.3	0.11	0.08	0.06	0.02	0.01	0.03	0.01	1.7
Sample Group II																		
15V-L2082-D1	7-72	90.5	23.3	349	20.0	26.8	8.9	50.8	24.3	16.9	0.89	1.11	0.50	0.24	0.84	1.14	0.95	44.8
15V-L2092-D2	8-72	89.2	18.1	339	22.7	24.6	12.3	55.0	20.3	12.4	1.02	1.20	0.58	0.27	0.83	1.30	0.95	45.8
15V-L2008-D1	9-72	90.2	17.2	341	20.2	23.0	12.1	56.3	18.8	12.8	0.82	0.93	0.45	0.25	0.75	1.27	0.92	44.5
15V-L2105-D1	10-72	90.0	20.8	340	24.5	22.2	8.7	61.0	24.0	6.4	0.83	1.02	0.55	0.28	0.78	1.29	0.93	45.9
15V-L2116-D1	11-72	91.0	30.4	355	19.9	19.5	6.8	57.7	22.2	13.2	1.00	0.96	0.57	0.28	0.85	1.14	0.96	45.9
15V-L2122-D1	12-72	90.5	21.2	431	21.3	17.0	5.9	55.4	23.0	15.7	0.89	1.08	0.45	0.27	0.79	1.37	0.93	43.3
Average		90.2	21.8	359	21.4	22.2	9.1	56.0	22.1	12.9	0.91	1.05	0.52	0.26	0.81	1.25	0.94	45.0
Standard Deviation		0.6	4.7	36	1.8	3.5	2.6	3.4	2.2	3.6	0.08	0.10	0.06	0.02	0.04	0.09	0.02	1.0
Properties Showing Significant																		
Difference (at 95% Confidence		*			*			*		*	*		*	*				*
Level) Between Means of																		
Groups I and II. (9)																		

(1) Unless otherwise specified, analyses were performed by methods described in Appendix A.

(2) Determined at 200° C; expressed in Saybolt-Furol seconds.

(3) Chloroform insolubles removed prior to analysis for average molecular weights and molecular-size distributions (via GPC).

(4) See Figure 2 for the division of the molecular size distribution (GPC) scan.

(5) Fraction of protons on aromatic carbon; determined from NMR spectra (from Williams⁽¹¹⁾ and Clutter⁽¹²⁾).

(6) Number of carbon atoms per alkyl substituent; determined from NMR spectra (from Williams⁽¹¹⁾ and Clutter⁽¹²⁾).

(7) Molar ratio of aromatic to total carbon; determined from NMR spectra (from Williams⁽¹¹⁾ and Clutter⁽¹²⁾).

(8) Date received for the samples in Group I were not determinable, but were 12 - 24 months prior to the receipt of samples in Group II.

(9) Determined using Student t test.

For pitch materials, this property may be obtained by dissolving the pitch in a suitable solvent, or by operating at temperatures sufficient to maintain the pitch as a low-viscosity fluid. While the former is necessary when an elevated temperature is a constraint, the latter is preferable if a maximum amount of pitch is to be deposited per impregnation cycle, such as would be desirable in densification procedures. For this reason, pitch materials are often kept in a molten state over long periods of time, and the constancy of pitch properties under these conditions becomes an important factor in the utility of the pitch as well as in the quality control of the impregnated structures.

Coal-tar pitch (CP 277-15 V; Lot 111; Drums 4 through 7) was evaluated under impregnating conditions covering an 88-day-use cycle. Approximately 220 gallons of pitch were initially placed in an impregnator which was maintained at 235° C for the duration of the study. No pitch was added during the 88-day period, which included approximately 50 impregnation cycles each of which was accompanied by evacuation and pressurization sequences. Samples of pitch were taken for analysis after 0, 18, 34, 48, 63, and 88 days. Data were obtained by the techniques listed in Appendix A and are reported in Table 2.

Some of the properties listed in Table 2 underwent graduated changes during the 88-day period, but overall property changes were relatively small for a heating period of this length. Figure 1, which better illustrates these changes, shows moderate increases in softening points, viscosities (at 200° C), average molecular weights, and benzene insolubles. Such changes would result from loss of volatile constituents and/or from polymerization reactions. Values for benzene insolubles could be influenced by an accumulation of particulate carbon derived from dust on the parts that are submerged in the pitch. As indicated in Figure 1, coke-yield values were quite scattered, but it could be concluded that the cook time had little effect on these values. The GPC scan in Figure 2 shows a typical molecular-size distribution for 15 V coal-tar pitch and indicates the divisions used in analyzing the GPC data. Gradual changes in molecular-size distributions with increasing cook time are noted in Figure 3. Values of the fractional composition corresponding to the largest (Fraction 4) and smallest (Fraction 1) molecular sizes increased slightly during the test period, while the two fractions of intermediate molecular sizes decreased. These data suggest that both decomposition and polymerization are occurring simultaneously as would be expected when energies are sufficient to disrupt bonds.

In summary, these data suggest that this coal-tar-pitch sample underwent only gradual changes in properties with increasing time at 235° C, and further suggest that such a material would have a reasonably long pot life at this impregnating temperature before property changes were sufficient to require replacement of the impregnant.

Table 2
 PROPERTIES OF COAL-TAR PITCH AS A FUNCTION OF RESIDENCE TIME IN THE IMPREGNATOR AT 235° C

Sample Number ⁽¹⁾	Residence Time (days)	Softening Point (° C)	Viscosity ⁽³⁾ (sec)	Average Molecular Weight ⁽⁴⁾	Benzene Insolubles ⁽⁵⁾ (%)	Composition as per GPC Fractions (%) ⁽⁶⁾				F _a ⁽⁷⁾	H _A ⁺ ⁽⁸⁾ (%)	Coke Yield ⁽⁹⁾ (%)
						1	2	3	4			
15V-L111-PI-1	0	88.2	19	360	15	8	51	24	17	0.93	78	41
15V-L111-PI-2	18	88.3	19	360	15	8	52	23	17	0.92	76	41
15V-L111-PI-3	34	90.4	22	360	15	8	49	25	18	0.94	80	42
15V-L111-PI-4	48	90.5	23	342	21	8	50	24	18	0.92	75	40
15V-L111-PI-5	63	91.0	25	388	19	9	49	24	19	0.92	76	41
15V-L111-PI-6	88	93.0	26	376	28	9	48	23	20	0.95	81	40

(1) Approximately 220 gallons of coal-tar pitch (Cp 277-15V, Lot 111, Drums 4 through 7) were placed in the impregnator. No pitch was added during the study period. Approximately 50 impregnation cycles occurred during the study period, each cycle consisting of an initial evacuation and a subsequent pressurization (80 psi N₂).

(2) Ring and Ball, ASTM-D-36-66.

(3) Saybolt-Furol viscosity at 200° C; reported as Saybolt-Furol seconds.

(4) Determined by vapor pressure osmometry; chloroform insolubles (13 - 17%) removed prior to analysis.

(5) Per ASTM D-2317.

(6) Gel permeation chromatography (GPC) scans were divided into four elution fractions and the area under the curves were determined. Chloroform insolubles (13 - 17%) were removed prior to analysis. The fractions correspond to elution counts of 45 - 48, 42 - 45, 39 - 42, and 32 - 39, respectively.

(7) Molar ratio of the aromatic carbon to the total carbon in the sample. Determined using NMR techniques (from Williams⁽¹¹⁾ and Clutter⁽¹²⁾).

(8) Fraction of protons on aromatic carbon, determined by integration of NMR spectra (from Williams⁽¹¹⁾).

(9) Conradson, ASTM-D-189-65.

Figure 1. PROPERTIES OF COAL-TAR PITCH AS A FUNCTION OF THE RESIDENCE TIME IN THE IMPREGNATOR AT 235° C. (Type CP 277-15 V)

Figure 2. GEL PERMEATION CHROMATOGRAPHIC SCAN SHOWING THE MOLECULAR SIZE DISTRIBUTION OF TYPE CP 277-15 V PITCH.

Figure 3. COMPOSITION OF COAL-TAR PITCH BY GEL PERMEATION CHROMATOGRAPHIC ANALYSIS AS A FUNCTION OF THE RESIDENCE TIME IN THE IMPREGNATOR AT 235° C.

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APPENDIX A

EXPERIMENTAL PROCEDURES

Materials

The coal-tar-pitch samples evaluated in this study were a product of Allied Chemical Company, and represented various lots of a pitch designated CP 277-15 V.

Methods

Descriptions of the analytical procedures used in this study are as follows:

1. Softening points were determined by the ring-and-ball method as per ASTM D-36-66. Visual observations of the melting processes involved use of a Fisher-Johns melting-point block.
2. Viscosities were measured at 200° C by the Saybolt-Furol viscosimeter as per ASTM D-88-56.
3. Average-molecular-weight data were obtained using a Hewlett-Packard Model 302 vapor pressure osmometer. Sucrose octaacetate was used as a standard. Prior to analysis, chloroform insolubles were removed by a standard procedure (see Appendix B). Samples were analyzed as tetrahydrofuran solutions.
4. Benzene insolubles were determined as per ASTM D-2317.
5. Gel permeation chromatography (GPC) data were obtained using a Waters Associates Model 200 GPC with columns packed with Styragel (styrene-divinylbenzene polymer). Prior to analysis, chloroform insolubles were removed by a standard procedure (see Appendix B). Samples were analyzed as tetrahydrofuran solutions.
6. Oxygen and nitrogen contents were determined by neutron activation analysis using a Kaman Model 711 neutron generator and beryllium as a standard.
7. Analysis of the sulfur contents involved combustion in a bomb followed by a gravimetric determination of sulfur as barium sulfate.
8. Ash contents were determined by firing samples to 900° C in air and weighing the residuals.
9. Nuclear magnetic resonance (NMR) studies were conducted using a Bruker HFX-15, 90-megaHertz spectrometer. Tetramethylsilane was used as an internal standard. Samples were analyzed as five percent solutions in carbon tetrachloride.
10. Coke yields were determined by the Conradson method as per ASTM D-189-65.

APPENDIX B

PROCEDURE FOR REMOVING INSOLUBLES FROM PITCHES

The following procedure was used to remove insolubles from CP 277-15 V pitch prior to GPC and average-molecular-weight determinations:

1. Weigh two 0.5-gram aliquots of powdered pitch into 500-milliliter boiling flasks having ground-glass joints.
2. Add 200 milliliters of chloroform to each flask.
3. Using water condensers, reflux for 1 1/2 hours.
4. Transfer to 400-milliliter beakers and evaporate to dryness at room temperature.
5. Add 200 milliliters of chloroform to the dried samples and again evaporate to dryness at room temperature.
6. Add 200 milliliters of chloroform to the dried samples and agitate thoroughly.
7. Filter the samples through a tared, fine-porosity-fritted glass funnel using Teflon for the seal between the funnel and vacuum flask.
8. Rinse each beaker and funnel with 50 milliliters of chloroform.
9. Dry the fritted funnels containing the insolubles at 50° C under house vacuum for 30 minutes, cool in a desiccator, and weigh. Calculate percent chloroform insolubles, reporting the average of the two aliquots.
10. Transfer the filtrate to 400-milliliter beakers and evaporate to dryness at room temperature under house vacuum.
11. Use liquid nitrogen to break up the dry, chloroform-soluble pitch.
12. Transfer to glass bottles and dry overnight at room temperature under house vacuum.

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