Not for Publication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

STUDIES ON LOW-TEMPERATURE LIGNITE TAR
I. GENERAL TAR CHARACTERISTICS AND PROCESSING

by

H. R. Batchelder, R. B. Filbert, Jr., and W. H. Mink
Battelle Memorial Institute
Columbus, Ohio

BACKGROUND AND HISTORY

A process for the low-temperature carbonization of Texas lignite in dilute suspension was developed by the U. S. Bureau of Mines at Denver, Colorado, in cooperation with the Texas Power & Light Company as part of a program for the more efficient use of this material. Work at Denver was carried through the pilot-plant stage. Promise of this process was such that the Aluminum Company of America installed a prototype carbonizer in combination with the power plant for their aluminum smelting installation at Rockdale, Texas. The boilers for the power plant were designed primarily to handle dried Sandow lignite, but were further designed so that lignite char would be acceptable as an alternate fuel. This permitted the production of the desired amount of tar without regard to the amount of solid fuel required for the boilers. The plant is 360,000-kw capacity, serviced by three boilers, in turn supplied by nine lignite driers. The prototype carbonizer was sized to handle the output of one drier only, with provision for transport of the hot char to the fuel bunkers. In effect, the carbonizer was on a bypass to the main line between the drier and the boiler. If all of the lignite were carbonized, tar production would be of the order of 50 million gallons per year.

The lignite is strip mined and transported by conveyor belt to the driers or field storage. Each drier has a capacity of 50 tons per hour and normally dried the lignite to 2 to 4 per cent moisture. The crushed dried lignite is picked up by recycle carbonizing gas, together with some air admixture, and is carried into the retort. Part or all of the necessary heat may be provided by combustion of tar vapors with process air within the retort. An external furnace is provided to supply what additional heat may be required. After the char is separated, the tar vapors are

condensed and the condensed water decanted. A good review of the carbonization work is given in the Bureau of Mines papers published by Parry et al.*

In 1954, Battelle was asked by Texas Power & Light and Alcoa to undertake studies leading to the exploitation of this tar. The work that will be reported here will cover the processing of this tar, the characterization studies, analysis of products, and the type of reactions of the tar components. No discussion of the utilization of this material is included.

GENERAL PHILOSOPHY OF APPROACH

The information available on low-temperature tars from lignite at this stage was quite sparse and, in many cases, self-contradictory. It was known, however, that the tar was an extremely complex mixture of a variety of hydrocarbon types, as well as compounds of oxygen, nitrogen, and sulfur. Throughout the course of this work, the general philosophy has been that attempts would be made to find uses for the crudest possible fractions, and that subsequent separations and refinements would be undertaken only when the work to that point had shown that further refinement was essential to any attractive utilization.

GENERAL CHARACTER OF THE TAR

The tar produced at Rockdale can be classed as a truly "primary" or virgin tar. The operating temperature of the carbonizer is relatively low. The rate of heating of the lignite and evolution of the volatile matter is rapid, and the vapor residence time of the liberated material is very short. None of the conditions obtain which in a by-product coke oven allow for extensive cracking and rearrangement of the components of the volatile matter. Consequently, the tar character reflects closely the character of the lignite charge.

It is this circumstance which probably contributes most to the complexity of the tar composition. Where extensive thermal reactions are possible as in the coke oven, most of the more unstable compounds are converted to a relatively few simple, highly stable configurations. A substantial part of the nitrogen and sulfur is liberated as ammonia and hydrogen sulfide, and appreciable quantities of hydrogen and methane are formed by the pyrolysis of hydrocarbons.

---

*Parry, V. F., Landers, W. S., Wagner, E. O., Goodman, J. R., and Lammers, G. D., "Drying and Carbonizing Fine Coal in Entrained and Fluidized State", Bureau of Mines Report of Investigations 4954, 1953.

Parry, V. F., "Low-Temperature Carbonization of Coal and Lignite for Industrial Uses", Bureau of Mines Report of Investigations 5123, 1955.

In the lignite tar, where these degradative reactions are at a minimum, a large proportion of the originally evolved compounds probably survive unchanged. Thus, throughout the whole boiling range of the distillates we find paraffinic, naphthenic, olefinic, and aromatic hydrocarbons, with oxygen, nitrogen, and sulfur substitution, and probably the undistilled material was of the same general nature.

EARLY DISTILLATION WORK

In the first approach to this problem, distillation was selected without question as the first means of separating crude fractions from the lignite tar. Because of many references to the instability of this material, particularly at higher temperatures, we used continuous distillation under vacuum with the minimum practical heating time. A series of these experiments showed an expected relationship between the volume of distillate recovered and the softening and/or viscosity of the residues. Comparison of the distillates recovered at various distillation temperatures soon showed that the composition did not vary particularly with the boiling point. In other words, the same classes of compounds were present in a distillate that represented 20 per cent of the primary tar as in a distillate that represented 50 per cent. As we went further with the study of these oils, separation into fractions of various boiling ranges supported this observation very strongly. The evidence was abundant enough to indicate that this constancy of composition extended throughout the tar, and that the undistillable residues were of the same general composition as the light distillates. The properties of the primary tar itself are shown in Table 1, and average composition of the distillate in Table 2.

TABLE 2. AVERAGE COMPOSITION OF LIGNITE TAR DISTILLATE

Compound	Volume Per Cent
Tar Acids	26
Tar Bases	4
Neutral Oils	70

SOLVENT EXTRACTION OF PRIMARY TAR

In studying the products obtained by this distillation of the primary tar, in many cases we found that the heavier portions of the tar had characteristics which were not particularly desirable. However, it was impractical to treat these fractions for the removal of the offending classes of compounds. For example, we had found quite early that a caustic soda treatment of a high-boiling distillate fraction for removal of tar acids was not promising because of emulsion formation and the difficulty of the separations. On this basis, the application of separation means to the primary tar itself appeared to be in order.

TABLE 1.

PROPERTIES OF PRIMARY LIGNITE TAR

Ash, per cent					0.4
C-I, per cent					1.3-1.6
Water, per cent					3-4
Specific Gravity, 25/25 C					0.9867
Viscosity, cp, 28 C					820
Elemental Composition, per cent water-free basis	C	H	N	S	0(diff)
	81.0	8.7	0.7	0.7	8.5
Distillation, ASTM D20-52, weight per cent (dry basis)					
To 170 C					2.5
170-235 C					16.4
235-270 C					11.6
270-300 C					12.6
300-Decomposition Temperature					31.0
Residue at Decomposition Temperature					21.0
Loss					4.9
Residue at 300 C					52.0
Decomposition Temperature, C					337

Batch Shake-Out Experiments

Because of our earlier work on distillation and the work of others on the separation of tar acids, we were led to explore the solvent extraction of the primary tar and tried first a two-solvent system comprising aqueous methanol* and hexane.

It seemed reasonable to expect that, by this means, a residue could be produced from the hexane-soluble fraction which would contain little or no polar materials. At the same time, the high-boiling tar acids and polar materials normally in the residue from the distillation should now be found in, and be recoverable from, the methanol extract. In a series of batch shake-outs, we found that separation could be achieved, and made a start toward defining the preferred conditions for the separation. A series of single batch shake-outs was made to determine the desired temperature of operation, the results of which are shown in Figure 1. As the temperature was increased from 66 to 120 F, it was found that the extent to which insoluble material was dispersed through the methanol phase decreased steadily until, at 120 F, the insoluble material was cleanly separated and was fluid enough so that it could be withdrawn without difficulty. Also, at this temperature, the material loss in the course of the experiment and the yield of insoluble material were markedly less than in the experiments conducted at lower temperatures. Temperatures much beyond 120 F would have required either the substitution of a higher boiling solvent for hexane or the conducting of experiments at higher than atmospheric pressure. Neither seemed desirable at this stage, since there was no reason to anticipate unsatisfactory operation at 120 F.

Another series of single batch shake-outs was made to study the effect of varying the solvent ratio on the products obtained. Increasing either solvent decreased the amount of insoluble material. Increasing the methanol ratio at a constant hexane ratio increased the yield of methanol solubles. The concentration of caustic solubles in the methanol solubles was also increased by increasing the methanol ratio up to a ratio of about 2. Higher ratios, however, resulted in a gradual reduction of the concentration of caustic solubles.

It can be shown that the total recovery of caustic solubles from the primary tar increases with increasing methanol ratio. In the process of recovering more tar acids by using higher methanol ratios, there is included more and more non-tar-acid material in the methanol solution. For maximum purity of recovered materials, low methanol ratios would be used; for maximum removal, high ratios would be used. Practically, the maximum removal in usable purity is the goal.

Because the process is essentially a partition of the tar acid material between the methanol and the hexane, the effect of increasing

* Whenever methanol is used here, it refers to a methanol-water solution, usually 70 weight per cent methanol.

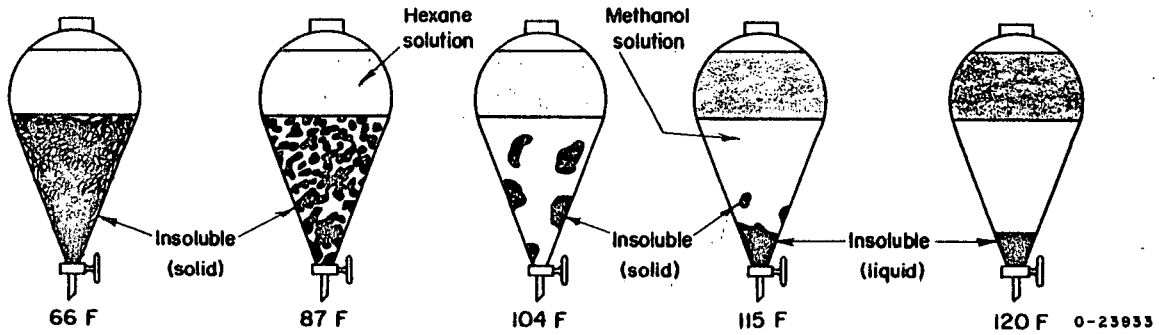


FIGURE 1. EFFECT OF TEMPERATURE ON SOLUBILITY OF TAR IN SINGLE BATCH SHAKE-OUT SOLVENT EXTRACTION

hexane ratio is substantially that achieved by decreasing the methanol ratio, except that the yield of insoluble material is decreased by an increase in either solvent. These preliminary experiments formed the basis for initial operation of the continuous unit.

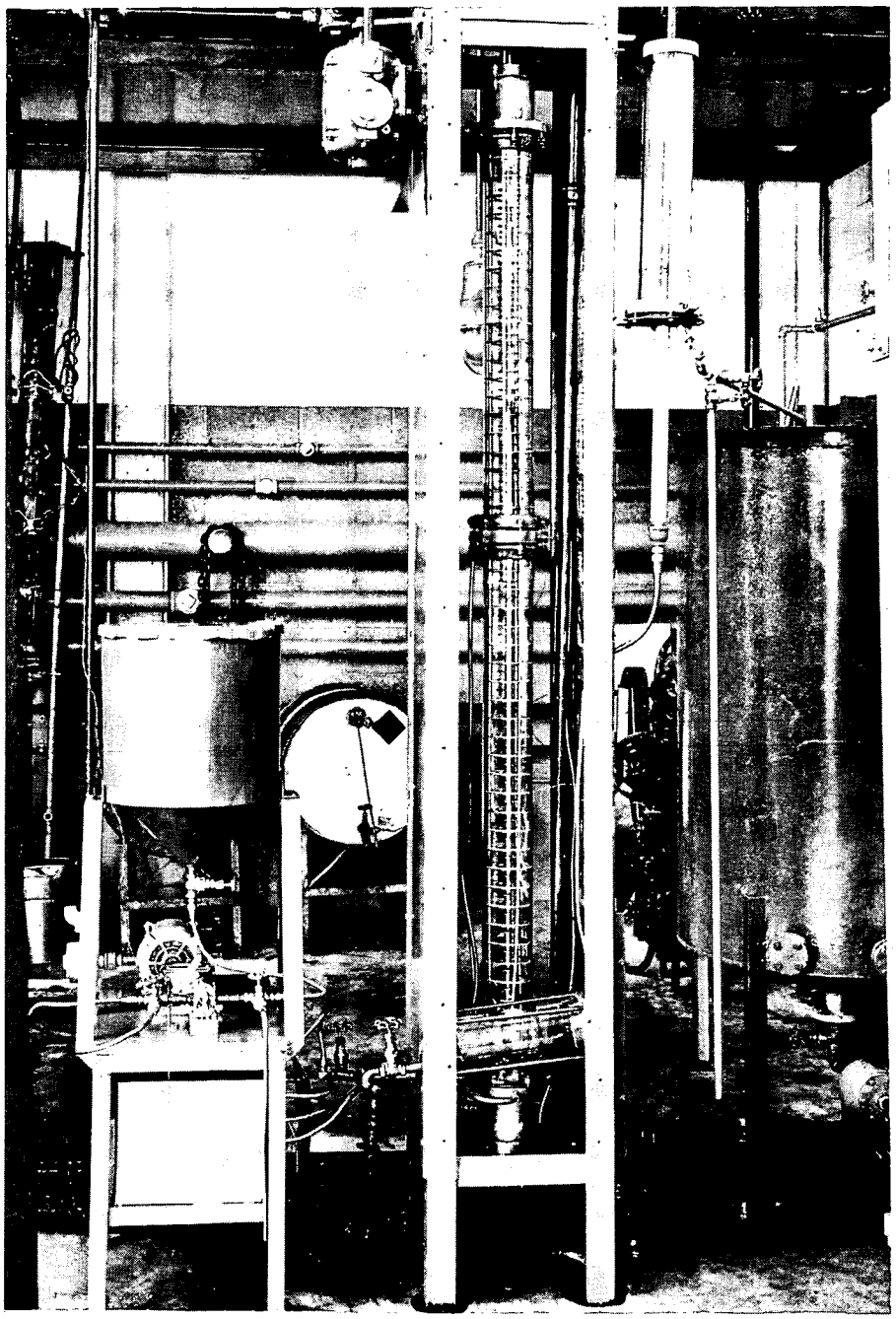
Operation of Rotating Disc Contactor Unit

Description of RDC Unit

The RDC column proper (shown in Figures 2 and 3) is constructed of two 4-inch flanged glass-pipe sections. The top section is 3 feet long and the bottom section is 4 feet long. The stator rings shown in Figure 3 are 3-13/16 inches in OD and 2-3/8 inches in ID. Teflon gaskets are sandwiched between the 20-gage stator rings to seal the ring to the side of the glass pipe. This method of construction was used because the inside diameter of the glass pipe used varies by as much as 1/8 inch. Three holes are punched in the stator rings and gaskets, and 1/4-inch rods inserted through the holes to hold the rings and gaskets. The compartments are formed by using spacers of 1-3/4-inch lengths of 3/8-inch tubing. The rotors are 20 gage and 2-1/8 inches in diameter, and are soft soldered to a 5/8-inch shaft.

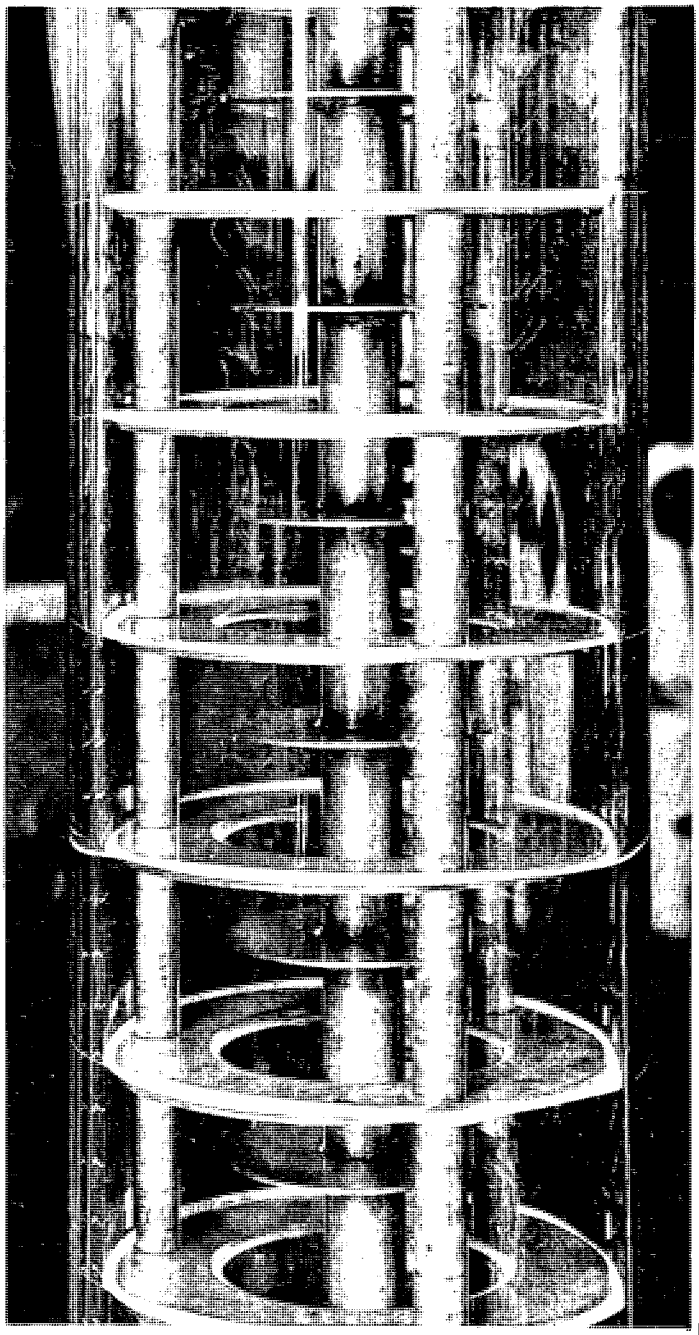
The two glass units are joined together in the center by means of a 1-inch-thick ring. The ring (Figure 2) is drilled and tapped so as to provide an entry for tar feed, a bearing support for the rotor shaft, and a passageway for fluids in the column. The fittings at the ends of the column are similar in construction to the center piece, except that an extension, consisting of a section of 4-inch tubing, forms a settling zone. The extract is removed through a 3/4-inch pipe and fed to a second settling chamber. Extract is taken off the high end of the settling chamber and fed to a variable-height syphon breaker and from there to a storage tank. The variable-height syphon breaker permits balancing the levels of solvent in the column. Tar is fed to the column by means of a small calibrated gear pump operated through a variable-speed drive. Solvents are fed to the column from constant-head feed tanks through rotometers. Both solvents and tar are heated in a hot-water bath prior to entry into the column.

The column is supported in an angle-iron framework which is enclosed on all four sides. The top of the framework is connected to a hood which removes vapors from the column and, at the same time, heats the column by drawing air in through a heat exchanger on the side near the bottom. Raffinate from the column is fed to the solvent-stripping unit, a 100-gallon agitated steam-jacketed vessel. An unlagged 4-foot length of 4-inch pipe filled with Berl saddles provides sufficient rectification to strip solvents from the products. After the raffinate has been stripped, the unit is used to strip the extract. In actual practice, after stripping, the methanol solubles contain 25 to 50 per cent water. This is removed by decanting.



N33341

FIGURE 2. RDC EXTRACTION UNIT



N33343

FIGURE 3. ROTATING-DISK CONTACTOR

Physical Operability of RDC Unit

The first runs in the pilot-plant unit were made to explore the variables which should affect the physical operation of the extraction column, in order that optimum conditions might be used as standard in the subsequent runs. These variables were: rotor speed, feed rate, density difference between phases, and time required for the column to reach equilibrium. Temperature was not considered as an operating variable in the RDC experiments, since an optimum temperature for operation had been established at 120 F through batch shake-out tests as described above. All RDC runs were made at this temperature.

In one run (1:4:4 ratio) feed rates were increased until flooding occurred at a tar feed rate of 2.6 gallons per hour. This corresponds to a total column feed of 350 gallons per hour per square foot of column cross-sectional area. In subsequent runs, the combined flow rate was limited to a maximum of about 270 gallons per hour per square foot.

As the rotor speed was increased from 180 to 215 rpm, the amount of caustic solubles in the methanol solubles increased; but, when the speed was increased further to 270 rpm, an emulsion was formed in the column which made phase separation impossible. All subsequent runs were made at 215 rpm.

Enough difference must be maintained between the density of the raffinate and extract so that the phases may be separated. As each solvent dissolves components of the tar, the density of each solution increases; therefore, the density and density difference will depend not only on the solvents used, but also on the solvent ratios as well. As more water is added to the methanol solution, its density increases so that, for each methanol-water concentration, there is a solvent ratio below which the column will not be operable. These findings are summarized in Figure 4.

For any given methanol concentration, operations to the left of the proper curve will not be feasible because of insufficient difference in density between the two phases. Thus, with 70 per cent methanol, a 1:3:3 ratio is satisfactory, but a 1:5:3 or a 1:1:2 ratio is not. It can be seen from these curves that, for any methanol concentration, there is a hexane ratio above which the column may be operated regardless of the methanol ratio. These curves are not sharp and precise boundaries, but rather the areas of approach to inoperable conditions. These data proved very useful in the selection of solvent ratios for subsequent runs.

Effect of Operating Variables on Products

As with single batch shake-outs, solvent ratios and methanol concentrations can be selected within operable limits for use with the RDC unit so as to produce products of varying quality and in varying yield. For example, the purity and yield (inversely related) of tar acids in the

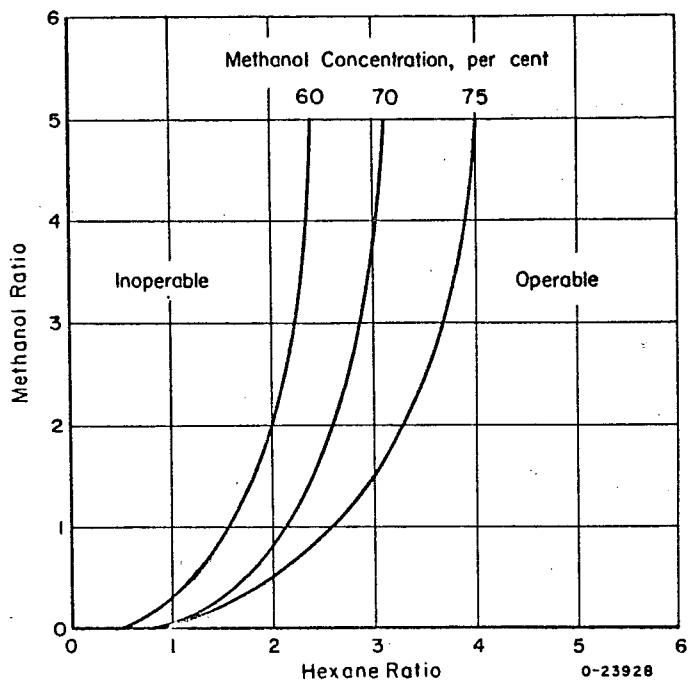


FIGURE 4. LIMITS OF OPERATION OF SOLVENT-EXTRACTION UNIT DUE TO DENSITY DIFFERENCE

methanol extract can be regulated partly by adjusting the methanol concentration. Likewise, the degree of extraction of polar compounds from the hexane-soluble fraction can be adjusted in part by changing the ratios of the hexane-methanol-primary tar streams. The effects of changing these two variables have been investigated within certain ranges in the pilot-plant RDC unit.

The effect of such changes can be followed by determining the fraction of the caustic solubles, originally in the primary tar, which is extracted by the methanol. The total caustic solubles in the tar cannot be obtained directly. However, a number of distillates of various yields were successfully caustic washed and were found to contain about 27 per cent of caustic solubles irrespective of yield or boiling range of distillate. For this reason, the value of 27 per cent was assumed to hold for the primary tar itself.

If the methanol concentration is low, it would be expected that little of the tar would be soluble in it. If, on the other hand, the methanol concentration is high, one would expect that the solubility of the tar, and hence the degree of extraction, would increase. This effect is shown in Figure 5. The position of the curve at 75 per cent methanol indicates that the assumed value of 27 per cent of caustic solubles in the primary tar might be low. If the value of 27 per cent were incorrect, it would have the effect of shifting the curve upward or downward, depending on whether the true value was lower or higher than 27 per cent. It was found in the single batch shake-outs that, whenever the extraction of caustic solubles increased, the concentration of caustic solubles in the methanol solubles decreased. A similar effect for the pilot-plant unit is shown in Figure 6.

The insoluble matter decreased with increasing solvent ratio as in the single batch shake-out tests. However, the column appeared to be more efficient in this separation. At a 1:8:8 ratio, a single batch shake-out resulted in about 4.5 per cent of insolubles while the RDC yielded about 2 per cent. Since Rockdale Run 53 primary tar contained 0.35 per cent of ash, and if it is assumed that char contains 20 per cent of ash, it can be calculated that there was about 1.75 per cent of char in Run 53 primary tar. The curve of Figure 7 appears to approach this value at high solvent ratios.

Figure 8 is a convenient method of showing the effect of solvent ratios for a given methanol concentration. The inoperable zone is defined and the per cent of total caustic solubles extracted is indicated. Because of the few data available, it is not possible to define the shape of the curves showing a constant per cent of caustic solubles extracted as a function of solvent ratios. However, the curve for 70 per cent extraction appears to be approximated by a straight line. The per cent of caustic solubles extracted appears to approach some value around 100 per cent

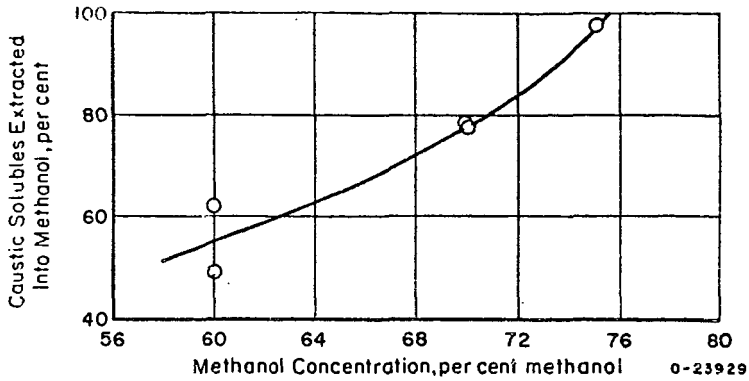


FIGURE 5. YIELD OF CAUSTIC SOLUBLES USING A 1:4:4 SOLVENT RATIO

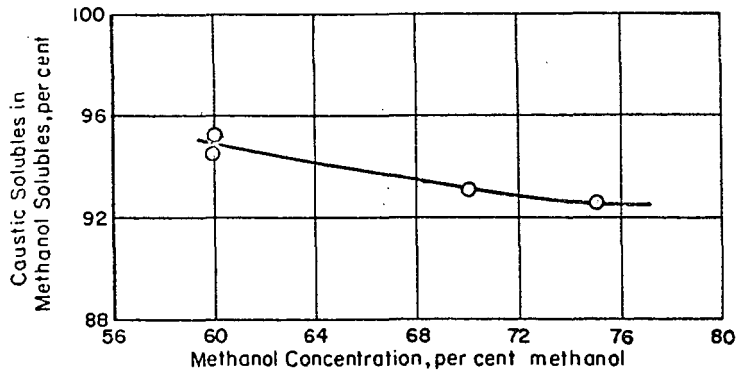


FIGURE 6. CAUSTIC SOLUBILITY OF METHANOL SOLUBLES USING A 1:4:4 SOLVENT RATIO

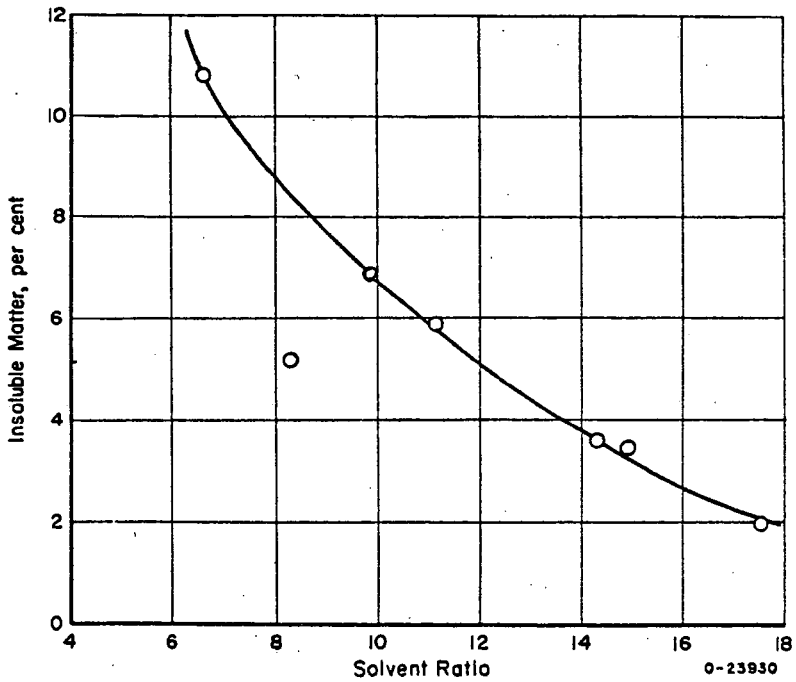


FIGURE 7. INSOLUBLE MATTER AS A FUNCTION OF TOTAL SOLVENT RATIO USING 70 PER CENT METHANOL

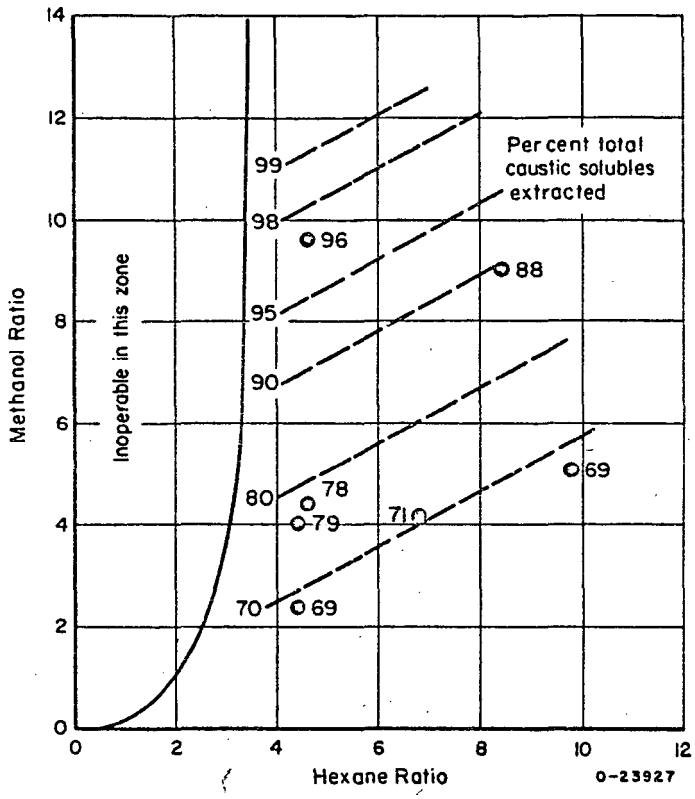


FIGURE 8. PER CENT TOTAL CAUSTIC SOLUBLES EXTRACTED AT VARIOUS SOLVENT RATIOS USING 70 PER CENT METHANOL

asymptotically as the methanol ratio is increased. From the slope of these curves, it is apparent that the methanol ratio has a greater effect on per cent of total caustic solubles extracted than does the hexane ratio under the conditions used.

The relative solubilities of the primary tar in the hexane and methanol phases were determined for 70 per cent methanol at different solvent ratios. These results are shown in Figure 9.

In previous work with the rotating disc contactor, tar feed was introduced at approximately the center of the column. Previous work on solvent-extraction columns has shown that the location of the feed point might have a significant effect on the operation of the column so far as yield and product quality are concerned. For this reason, the rotating disc contactor was altered to permit introduction of tar feed at two locations in addition to the center feed point. The points selected were approximately one-fourth of the distance down from the top of the column and one-fourth of the distance up from the bottom of the column.* Four runs were made in which the feed point was successively changed from center, to top, to bottom, and finally back to center position. There was no significant change in the column variables other than feed-point location. As a result of this investigation, it was found that the total material extracted from the tar by the methanol varied as follows:

Feed Position	Per Cent Extracted
Center	25.6
Top	23.7
Bottom	23.4
Center	25.9

The relatively small change in the percentage extracted as affected by the feed-point location indicates that the column used probably has considerably more stages than are necessary to perform the desired extraction. In future work, however, the present column will be used, with feed introduced at the center position.

Analytical Methods

A 500-ml sample of raffinate is weighed and about 250 ml placed in a tared 500-ml flask. The flask is fitted with a 12-inch Vigreaux column and a thermometer extending to within 1/4 inch of the bottom of

* In the original design of the rotating disc contactor there were 44 stages, with the feed point being at Stage 20, numbering down from the top. Two other feed points were subsequently provided: (1) "Top", Stage 10, and (2) "Bottom", Stage 32.

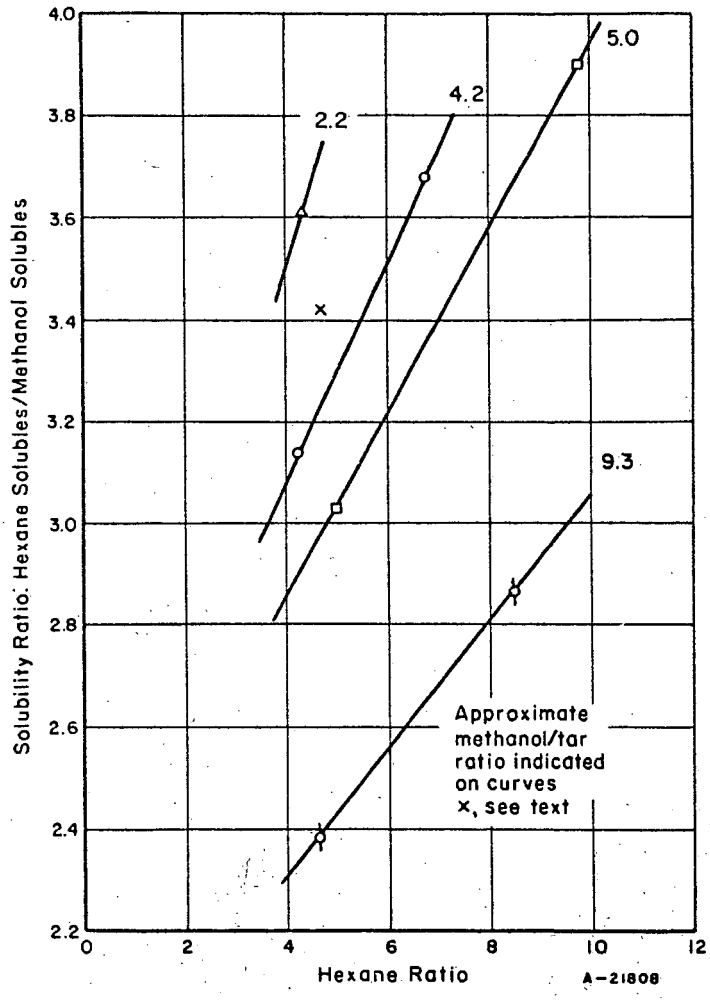


FIGURE 9. RELATIVE SOLUBILITIES AS A FUNCTION OF SOLVENT RATIOS FOR 70 PER CENT METHANOL

the flask. The top of the Vigreaux column is fitted with a side arm connecting to a condenser, and a separatory funnel through which is added the remainder of the raffinate sample during distillation.

The flask is heated with a heating mantle, and the hexane distilled over is discarded. When all the raffinate has been charged to the flask the distillation is continued until the pot temperature reaches 210 C. The flask is then cooled and weighed to determine the weight of hexane-soluble residue.

The extract is handled in the same way as the raffinate except that, after all extract has been added to the flask, distillation is stopped when the pot temperature reaches 95 C. The flask is allowed to cool and 200 ml of benzene is added. The contents of the flask are then transferred to a 500-ml separatory funnel, shaken, and allowed to settle. The water layer is drawn off and discarded and the benzene layer returned to the flask. The separatory funnel is rinsed with methyl alcohol, which is added to the contents of the flask. Distillation is continued until the pot temperature reaches 210 C. The flask is allowed to cool and then is weighed to determine the methanol solubles.

The methanol solubles obtained as indicated in the previous section are washed into a 500-ml separatory funnel with three portions of warm 20 per cent sodium hydroxide solution. The solution is allowed to cool to room temperature and 50 ml of ether added. The mixture is shaken carefully and allowed to settle. The ether layer is transferred to a tared 100-ml flask and heated on a steam bath. After the contents have stopped boiling, a vacuum (using a water aspirator) is drawn until no more boiling occurs. The flask is cooled and weighed to determine the non-caustic-soluble material in the methanol solubles. The per cent of caustic solubles is calculated by difference.

A known quantity of the insoluble phase is charged to a tared flask and heated on a steam bath under vacuum to strip it of solvent. The flask is cooled and weighed to determine the insoluble matter.

SECONDARY PROCESSING OF SOLVENT EXTRACTION PRODUCTS

For the purpose of providing samples for the characterization and utilization studies, hexane solubles were distilled to yield fores, middle oil, and pitch of various softening points. Both batch and continuous methods of distillation were used. Methanol solubles were usually distilled to an arbitrary cut point of 235 C for the same purpose.

ROCKDALE PILOT PLANT

A solvent-extraction pilot plant has been constructed at Rockdale, primarily to provide materials for utilization study on a larger scale. This unit is designed to process about 1000 gallons of primary tar per stream day and to produce five main products. A simplified flow diagram is shown in Figure 10. Tar, aqueous methanol, and hexane--all preheated to 120 F--are fed to the center, top, and bottom of a rotating disc contactor. Rates are controlled by metering pumps. The contactor is 3 feet in diameter by 16 feet high, with 24 contacting stages.

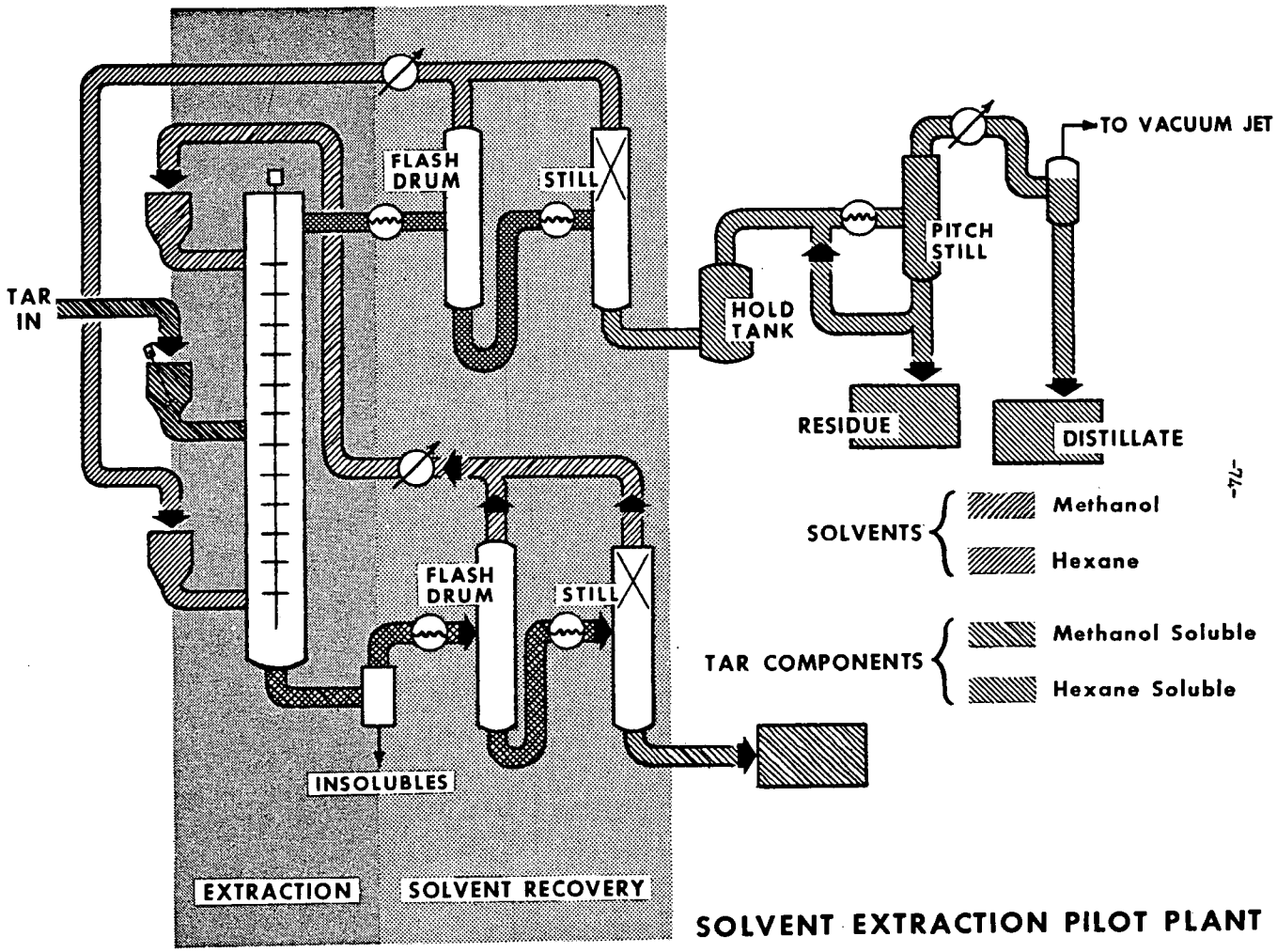
The hexane solution leaves the top of the contactor, passes through a steam heater, then into a flash drum where a substantial portion of the solvent is flashed off. The partially stripped liquid passes through another heater and is fed to a packed tower for the distillation of the remaining solvent. The streams of solvent vapor are combined and passed through a condenser where the water flow is regulated to maintain the proper temperature of the recycled solvent. The solvent-free hexane extract goes to an accumulator tank, then through an electric heater to a vacuum still in which sufficient distillate is taken overhead to yield as bottoms a pitch of the desired softening point. The distillate is topped in another column, not shown on the diagram, to produce a light distillate and a bottoms of the desired flash point and boiling range.

The methanol solution similarly is stripped of solvent in two stages, and the recovered solvent is recycled to the contactor. Adjustment of the water content is necessary because part of that originally present in the solvent feed stream is decanted from the solvent-free material leaving the bottom of the methanol recovery still.

When desired, the methanol-soluble material may be separated into low- and high-boiling fractions in the same column mentioned above for final splitting of the hexane-soluble distillate. Normally, tar acids through the xylenols are taken overhead in this operation. Tables 3 through 7 give typical inspections of the five products resulting from this kind of operation.

ACKNOWLEDGMENT

The lignite tar project was set up in a rather unusual manner. The Texas Power & Light Company and the Aluminum Company of America cooperated in the installation and operation of the carbonizer at Rockdale, and jointly sponsored the start of the research project at Battelle. In addition to this, eleven companies participated in the tar research. In return for nominal support of the research costs, they had access to the results of our work, contributed helpful information and suggestions during the course of the research, and were expected to conduct further work in their own laboratories.



SOLVENT EXTRACTION PILOT PLANT

TABLE 3.

PROPERTIES OF FORERUN, DISTILLING TO 200 C, OF DISTILLATE OF HEXANE SOLUBLES OF PRIMARY LIGNITE TAR

Specific Gravity, 25/25 C					0.8503
Refractive Index, n_D^{25}					1.4754
Viscosity, kinematic cs, 100 F					1.11
Flash Point, F					90
Fire point, F					165
Copper Strip Corrosion					No discoloration
Pour Point, F					<-70
Tar Acids, volume per cent					1.7
Tar Bases, volume per cent					1.2
Neutral Oil, volume per cent					97.1
Paraffins, volume per cent					14
Olefins, volume per cent					41
Aromatics, volume per cent					45
Elemental Composition, per cent, water-free basis	C	H	N	S	O(diff)
	85.4	11.7	0.2	1.6	1.1
Distillation, ASTM D20-52, weight per cent					
To 170 C					26.7
170-235 C					62.1
235-270 C					7.7
270-300 C					2.3
300-355 C					0.8
Residue at 355 C					0.3
Loss					0.1
Residue at 300 C					1.1
Decomposition Temperature, C					>355

TABLE 4.

PROPERTIES OF DISTILLATE, TOPPED TO 200 C, OF HEXANE SOLUBLES OF PRIMARY LIGNITE TAR

Specific Gravity, 25/25 C					0.9325
Refractive Index, n_D^{25}					1.5242
Viscosity, kinematic cs, 100 F					8.13
Flash Point, F					208
Fire Point, F					320
Copper Strip Corrosion					No discoloration
Pour Point, F					66.5
Tar Acids, volume per cent					7.7 ± 2
Tar Bases, volume per cent					1.6
Neutral Oil, volume per cent					90.7 ± 2
Paraffins, volume per cent					15
Olefins, volume per cent					39
Aromatics, volume per cent					46
Elemental Composition, per cent, water-free basis	C	H	N	S	0(diff)
	85.4	10.7	0.4	1.0	2.5
Distillation, ASTM D20-52, weight per cent					
To 170 C					0.1
170-235 C					14.3
235-270 C					20.1
270-300 C					18.1
300-355 C					27.7
Residue at 355 C					18.6
Loss					1.1
Residue at 300 C					46.3
Decomposition Temperature, C					>355

TABLE 5.

PROPERTIES OF RESIDUE OF HEXANE SOLUBLES OF PRIMARY LIGNITE TAR

Softening Point, Ring and Ball, C					68
Penetration, 100 g, 5 sec, 77 F					1
Viscosity, SFS, 350 F					39
C-I, per cent					0.36
Nitrobenzene Insoluble, per cent					1.1
Ash, per cent					0.1
Specific Gravity, 25/25 C					1.064
Elemental Composition, per cent, water- and ash-free basis	C	H	N	S	0(diff)
	87.6	7.6	0.7	0.8	3.2
Distillation, ASTM D20-52, weight per cent					
To 300 C					4.0
300-Decomposition Temperature					13.4
Residue at Decomposition Temperature					82.6
Loss					--
Residue at 300 C					96.0
Decomposition Temperature, C					355

TABLE 6.

PROPERTIES OF LOW-BOILING METHANOL SOLUBLES, DISTILLING UNDER 235 C, OF PRIMARY LIGNITE TAR

Caustic Soluble, per cent	99
Specific Gravity, 25/25 C	0.991
Viscosity, kinematic cs, 100 F	3.17
Sulfur Content, per cent	0.13
Nitrogen Content, per cent	0.67
Distillation, ASTM D20-52, weight per cent	
To 170 C	0.0
170-235 C	81.3
235-270 C	8.8
270-Decomposition Temperature	6.3
Residue at Decomposition Temperature	2.4
Loss	1.2
Residue at 300 C	2.4
Decomposition Temperature, C	280

TABLE 7.

PROPERTIES OF HIGH-BOILING METHANOL SOLUBLES, BOILING OVER 235 C, OF PRIMARY LIGNITE TAR

Specific Gravity, 25/25 C	1.163
Viscosity, SFS, 350 F	12
Softening Point, Ring and Ball, C	40-43
Sulfur Content, per cent	0.99
Nitrogen Content, per cent	1.61
Distillation, ASTM D20-52, weight per cent	
To 170 C	0.1
170-235 C	11.3
235-270 C	12.7
270-300 C	24.1
300-Decomposition Temperature	8.5
Residue at Decomposition Temperature	40.9
Loss	2.4
Residue at 300 C	49.4
Decomposition Temperature, C	325

The companies participating were:

Barrett Division
Celanese Corporation
Grace Chemical Company
Koppers Company, Inc.
The Merichem Company
Oil and Chemical Products, Inc.
Olin Mathieson Chemical Corporation
Reilly Tar and Chemical Corporation
Spencer Chemical Company
Union Carbide Olefins Company
Western Tar Products Corporation

We gratefully acknowledge their assistance and their willingness to have this information presented. We also express our appreciation to the sponsoring companies, Texas Power & Light and Alcoa, for permission to present this information, and for the understanding and assistance we have received in the course of more than five years of a fine relationship.

HRB:REF:WHM/af
January 29, 1959