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Fuels Division

THE COLD WATER PROCESS FOR THE RECOVERY OF THE
BITUMEN FROM THE BITUMINOUS SANDS OF ALBERTA

1. Sampling Procedure and Analytical Methods for the
Control and Evaluation of the Pilot Plant Operation

By

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and H. McD. Chantler

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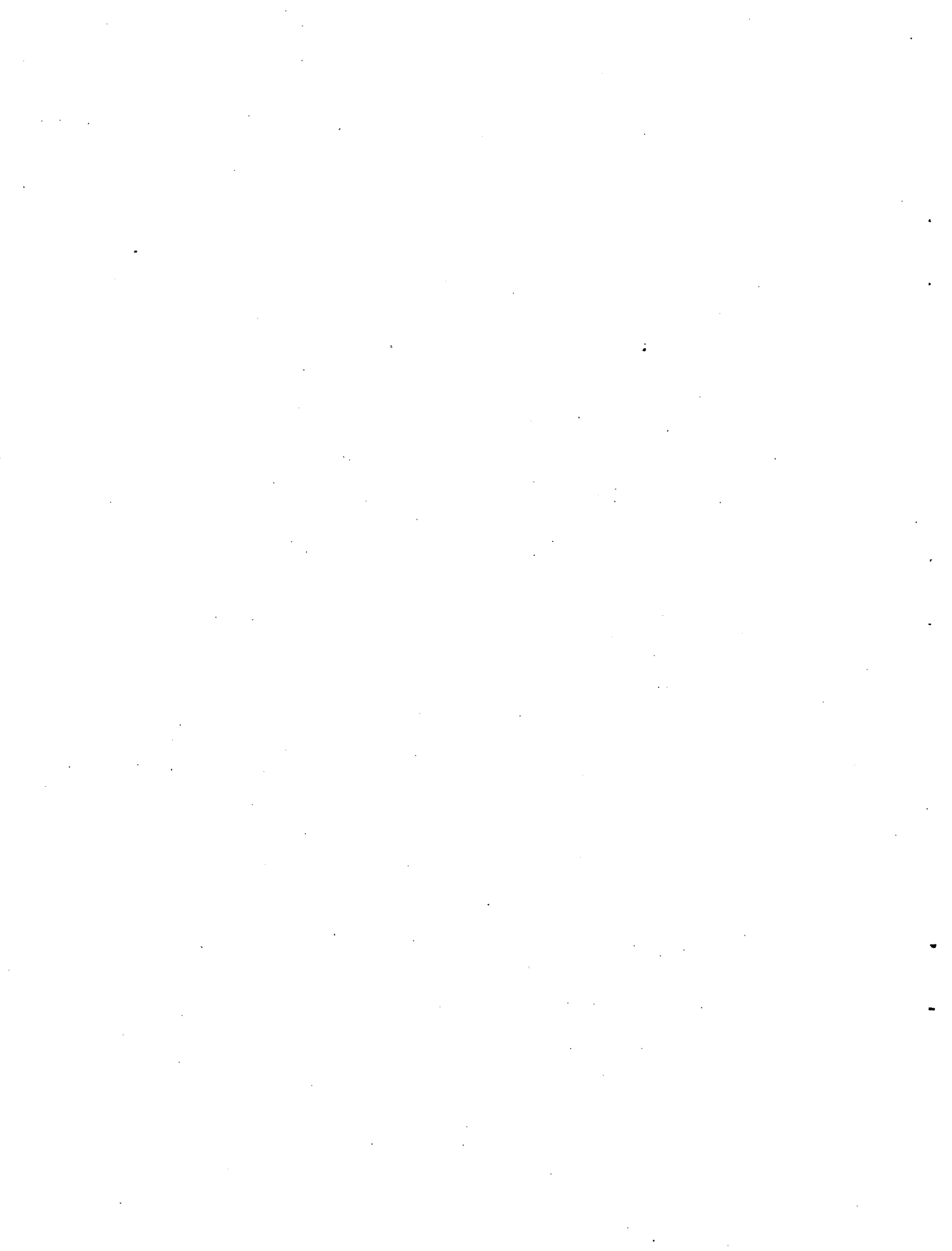
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SUMMARY

This report identifies all the standard analytical methods, and describes the special modifications of these procedures which were used for the control and evaluation of the cold water process for the recovery of bitumen from the bituminous sands of Alberta. Particular emphasis has been placed on the sampling methods used to treat the eight classes of samples submitted by the pilot plant to the laboratory. An attempt also has been made to provide a link between the weekly operating sheets of the pilot plant and the report of the analyses issued by the Fuels Division by explaining the origin of each sample and the numbering system employed for sample identification.



INTRODUCTION

The Mines Branch of the Department of Mines and Technical Surveys started an investigation of the problem of the removal of bitumen from the bituminous sands of Alberta by the cold water process on a pilot plant scale in April 1949. This project was undertaken jointly by the Mineral Dressing and Process Metallurgy Division and the Fuels Division. In view of this joint responsibility the following report was prepared to link the operations of the pilot plant described by L. E. Djinghezian (1), the weekly operating sheets, and the report of analyses issued by the Fuels Division (2). During the study of the cold water process difficulties were encountered in taking some samples of bitumen kerosene, sand, clay, and water, owing to segregation and other factors. The methods employed in taking these samples and in analysing them are described to facilitate future pilot plant studies in this field. A summary is presented of all the various types of samples analysed, as well as the analytical methods employed to evaluate each sample. In the selection of analytical methods the question of their accuracy was carefully examined, but in addition considerable importance was attached to the speed with which the determinations could be made, as it was important to be able to obtain results within twenty-four hours. This speed enabled the results to be interpreted and the necessary changes in the pilot plant operating procedure to be made, with the minimum waste of materials and time.

DESCRIPTION OF THE SAMPLES AND SAMPLING PROCEDURE

In general there were two types of samples taken. The first class, referred to as key samples, were those upon which the material balance of the process depended. The second type of samples, called the control samples, were designed to supply information on the performance of individual pieces of equipment in the sequence of steps which were involved in the cold water separation process. Usually the pilot plant was operated on every working day, and each day's operation was referred to as a mill run. A single experiment to evaluate a given separation sequence or wetting agent required three or more mill runs. Three mill runs were usually the minimum number required for the pilot plant to reach steady operating conditions.

Usually the information sought by analysis was the proportion of oil, water, mineral matter and clay. By oil was meant the extracted bitumen plus diluent. Initially kerosene was used as the diluent, but in a few of the final pilot plant experiments a light oil fraction distilled from Bitumount bitumen was employed. This fraction was referred to as Bitumount diluent. The bitumen and the diluent were always present in equal proportions by weight in all the pilot plant experiments. A description of the physical properties of the diluents which have a bearing on the separation process are given in a subsequent report (2).

For the most part the samples from the pilot plant were received by the Fuels Division Laboratory in two gallon enamel pails. To avoid errors due to segregation the contents were vigorously agitated with a large spatula capable of penetrating the slurry to the bottom of the pail. After thorough mixing a 400 ml. sample was withdrawn and placed in a 600 ml. beaker. Each sample was given a laboratory number prefixed by BX (bitumen extraction sample number). In reporting to the pilot plant, the samples were also identified by the mill run numbers (MR) and part of the mill from which the samples originated e.g. MR-1 Tube Mill Feed. The indexing of the samples has been described in some detail to provide a link between the results reported in the report of analyses (2) and the weekly operating sheets. In order to expand this link so that the analytical results may be understood in the light of the operations of the pilot plant described by Djingheuzian (1), the following brief description of the procedure followed in taking the various pilot plant samples is given. For convenience the key samples are described first followed by the control samples. The number following the name of the sample identifies the sample on the flow sheet shown in Figure I.

FLOW SHEET OF THE COLD WATER SEPARATION PROCESS SHOWING NUMBERED SAMPLING POSITIONS

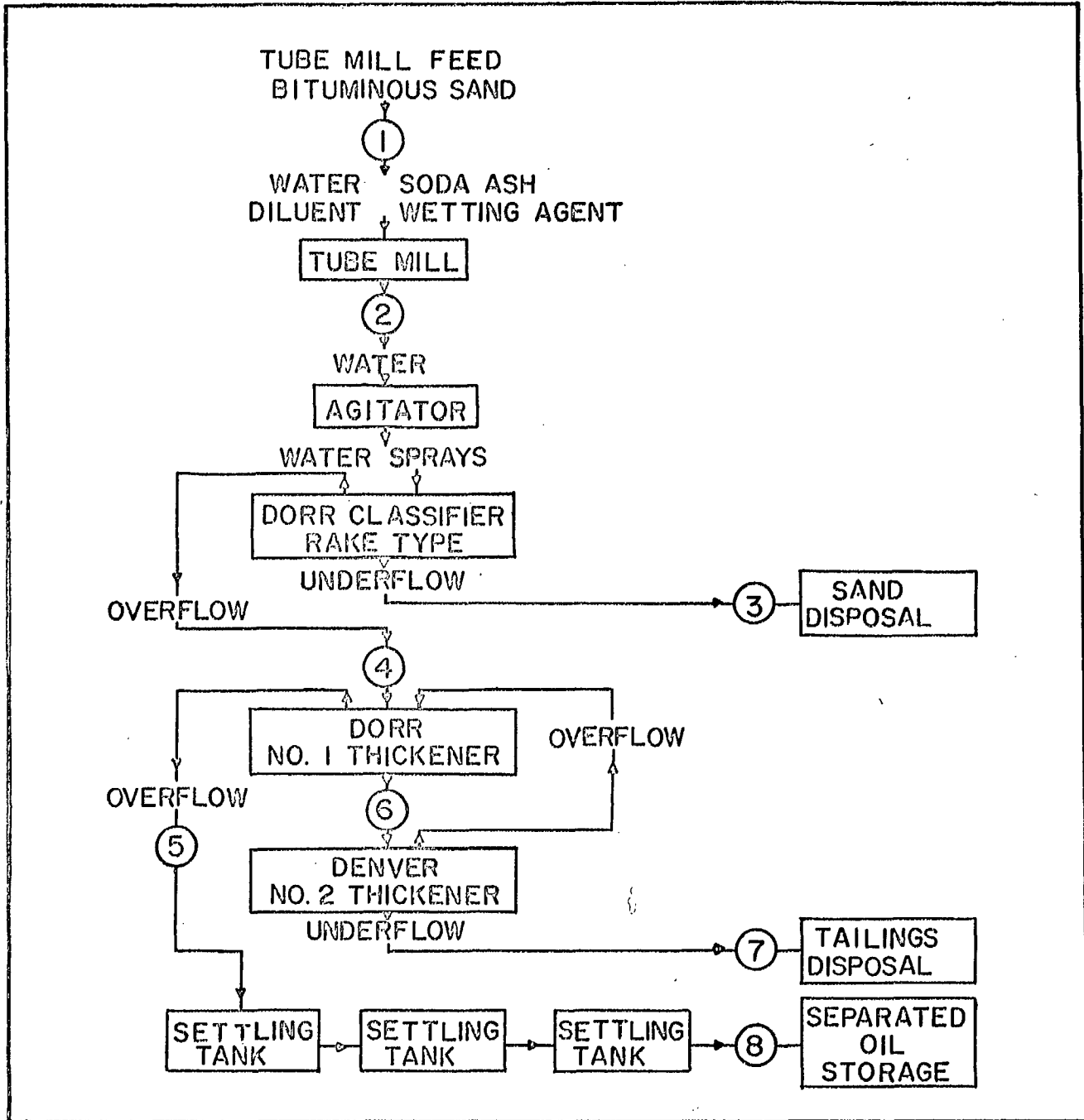


FIGURE 1

Key Samples

Tube Mill Feed - 1

This sample was a composite of the bituminous sand fed to the ball mill during a mill run and was made up by taking a handful of the tar sands from every 50 lb. of the daily mill feed. Owing to the fact that the bituminous sand was taken from a relatively small area in the floor of the Abasand Quarry the content of bitumen in the tar sands was essentially constant and the above sampling procedure was considered adequate to ensure satisfactory material balances. This sample was analysed for oil content, mineral matter and water. A screen analysis was made on a composite sample of the extracted mineral matter from two successive determinations.

<u>Typical Analysis</u>		<u>Identification Numbers of Methods of Analyses</u>
Weight percent		
Oil	15.2	2
Mineral matter	83.6	2
Water	1.2	1

Classifier Sands - 3

This sample was a composite of the sand from the Dorr rake classifier during a mill run, and consisted of accumulated samples of the discharge from the classifier waste discharge lip collected in a pail at half-hourly intervals by the pilot plant operator. This composite sample was analysed for oil, mineral matter and water. A screen analysis was made on a composite sample of the extracted mineral matter from a pre-determined number of successive mill runs.

<u>Typical Analysis</u>		<u>Identification Numbers of Methods of Analyses</u>
Weight percent		
Oil	0.3	6
Mineral matter	74.7	6
Water	25.0	1

No. 1 Dorr Thickener Overflow - 5

This sample was a composite of the oil concentrate from the overflow of the No. 1 Dorr thickener before it entered the settling tanks. Since the thickener was allowed to overflow only at intervals, generally three times a day, samples of the overflow were taken with a 400 ml. beaker at every inch of the rise of the oil in the settling tanks. A steel tape was used to measure the rise of the oil in the tank. This sample was analysed for oil, mineral matter and water.

<u>Typical Analysis</u>		<u>Identification Numbers of Methods of Analyses</u>
Weight percent		
Oil	70.5	9
Mineral matter	2.5	9
Water	27.0	1

No. 2 (Denver) Thickener Underflow or Tailings - 7

This sample was a composite of the No. 2 thickener underflow or the tailings that were discharged into 46 imp. gal. waste barrels. Two samples were taken at the end of the discharge pipeline for each barrel filled. These samples were subsequently analysed for water, mineral matter and oil content.

<u>Typical Analysis</u>		<u>Identification Number of Method of Analysis</u>
Weight percent		
Oil	0.2	7
Mineral matter	0.8	7
Water	99.0	7

Control Samples

The samples, which are listed below and designated as control samples, were analysed to secure a better understanding of the performance of the individual pieces of equipment in the separation sequence. As the initial flow sheet was composed on the basis of limited laboratory experience and intuition, it was important to determine the performance of each piece of apparatus to see if the flow sheet could be simplified and the performance of the individual units improved. The number following the name of the sample again identifies it on the flow sheet of the operation shown in Figure I.

Tube Mill Discharge - 2

This sample was a composite of the discharge from the tube mill. Samples were taken with a pail at half-hourly intervals directly under the steeply inclined launder discharging into the agitator. The laboratory sample consisted of only the settled sand which collected in the bottom of the pail. Usually only a screen analysis of the mineral matter following analytical Methods 3 and 4 was required. However when it was necessary to determine the clay content, samples from several mill runs were combined and analysed by Method 5 to provide reliable average values from mill run numbers 73 to 150.

Classifier Overflow No. 1 Dorr Thickener Feed - 4

This sample was a composite of the Dorr classifier overflow which was the feed to the Dorr No. 1 thickener. Samples were taken with a 600 ml. beaker at half hourly intervals at the end of the discharge pipe line into No. 1 thickener to make a composite sample of 2 gallons representing the mill run. This sample was analysed for oil, mineral matter, and water. When the clay analysis was required one-half gallon samples were taken from each of the 2 gallon composite samples collected during two successive mill runs to provide a representative sample of mineral matter for clay analysis.

Typical Analysis

Identification Number
of Method of Analysis

Weight percent

Oil	2 to 10	7
Mineral matter	1 to 2	7
Water	88 to 98	7

No. 2 (Denver) Thickener Feed - No. 1 Thickener Underflow - 6

This sample was a composite of the No. 1 Dorr thickener underflow or discharge to No. 2 Denver thickener. Samples were taken with a 600 ml. beaker at the end of the discharge pipeline in No. 2 thickener whenever this thickener was being filled. The number of samples taken during the filling period was varied from two to three depending on the level of the slurry in the thickener, in an endeavor to keep the time interval between the samples constant. This sample was analysed for water, mineral matter and oil content.

Typical Analysis

Identification Number
of Method of Analysis

Weight percent

Oil	trace to 3	7
Mineral matter	1 to 10	7
Water	88 to 98	7

Separated Oil Products - 8

This sample was a composite of the final oil product of the pilot plant taken at the pump, during the transfer of the oil from the settling tank to storage. Samples were taken under the discharge tap of the pump with a 400 ml. beaker for every two inches fall of the crude oil in the settling tanks; the fall of the crude oil being measured with a steel tape. This crude oil was analysed for oil, mineral matter and water.

<u>Typical Analysis</u>	<u>Identification Numbers of Methods of Analyses</u>	
Weight percent		
Oil	55 to 90	9
Mineral matter	0.5 to 6.0	9
Water	10 to 40	1

Summary of Samples and Test Methods

For convenience a summary of all the samples taken to evaluate and control the cold water separation process is presented in Table I together with the tests and the identification numbers of the various methods of analysis.

TABLE I

SUMMARY OF SAMPLES AND TEST METHODS

Sample Designation	Test Reported	Identification Number of Method of Analysis
Tube mill feed	Oil content	2
	Mineral matter (M. M.)	2
	Water content	1
	Screen analysis of M. M.	3
Tube mill discharge (mineral matter)	Screen analysis	4
	Aluminium oxide	5
	Clay	5
Dorr classifier sands	Oil content	6
	Mineral matter	6
	Water content	1
	Screen analysis of M. M.	3
No. 1 Dorr thickener feed (classifier overflow)	Oil content	7
	Mineral matter	7
	Water content	7
	Aluminium oxide	8
	Clay	8
No. 1 Dorr thickener overflow	Oil content	9
	Mineral matter	9
	Water content	1
No. 2 (Denver) thickener feed or No. 1 thickener underflow	Oil content	7
	Mineral matter	7
	Water content	7
No. 2 (Denver) thickener underflow or tailings	Oil content	7
	Mineral matter	7
	Water content	7
Separated Oil Product	Oil content	9
	Mineral matter	9
	Water content	1

EXPERIMENTAL

Laboratory Methods of Analyses for Pilot Plant Control

The laboratory methods described in this report consist of modifications of existing analytical methods to meet the requirements of speed and accuracy necessary for pilot plant control. These methods were recorded systematically so that there would be no uncertainties regarding the methods used to obtain the analytical results which will be presented in the second report of this series. A summary of these methods preceded by their identification numbers is tabulated below:

1. Determination of water content.
2. Determination of bitumen (or oil) and mineral matter content.
3. Screen analysis of extracted sand aggregate.
4. Extraction of mineral matter for screen analysis.
5. Analysis of mineral matter for clay content.
6. Determination of oil and mineral matter content by drying and extraction.
7. Determination of water, mineral matter and oil content by evaporation and ashing.
8. Preparation of sample for clay analysis.
9. Determination of mineral matter content by ashing.

This same order is preserved in presenting the description of the various analytical methods. Before passing directly to the detailed

description of the analytical procedures the following explanation may be helpful. It will be noted that four analytical Methods 2, 6, 7 and 9 were employed to determine the oil content of the various samples. This variety of methods was dictated by the wide variations in the proportions of oil, sand and water. It was for this reason that in the section dealing with the description of the samples and sampling procedures, typical analyses were presented, so that the correct analytical method could be chosen by the visual examination of the sample.

1. Determination of Water Content

The method used is essentially the same as the A. S. T. M. Designation D 95-46, (3) which during development (prior to 1921) was known as the Dean and Stark method.

Scope

The purpose of this method is to determine the water content (other than water of hydration) in order to deduct it from the total bitumen and water extract, so that the bitumen content may be reported on a moisture free basis.

The principle of the method consists in the distillation of the water from a flask containing bituminous sand and solvent naphtha and condensing both naphtha and water into a graduated trap in which the volume of the water can be measured. The weight of the sample must be

selected so that the water condensed in the trap does not exceed 10 ml.

Apparatus

Figure 2 shows a convenient arrangement for multiple determinations of this type.

- (1) Glass flask - Pyrex, round bottom, short neck type, capacity 500 ml.
- (2) Trap - Pyrex, with closed end graduated from 0 to 10 ml. in 0.1 ml. divisions, 18 mm. outside diameter.
- (3) Condenser - Liebig type, 400 mm. long with inner tube 9.5 to 12.7 mm. outside diameter.
- (4) Electric heater - 250 (or 500) watts, rheostat control.

Note:

For convenience, glassware may be purchased with standard taper joints - flask, Corning Cat. No. 4320; trap - Corning Cat. No. 3602; condenser - Corning Cat. No. 2360.

Solvent

A. S. T. M. Dilution Naphtha (see Method D 95-46), a petroleum naphtha conforming to the following distillation requirements: "5 percent shall distill at a temperature not below 195° F. (90° C.) nor above 212° F. (100° C.) and 90 percent shall distill below 410° F. (210° C.)". Oil refinery "Dilution Naphtha" with a distillation range 196° to 273° F. has been found to be a quite satisfactory substitute.

Procedure

Fifty grams, (weighed to the nearest 0.1 gram), of sample are placed in the flask and mixed with 150 cc. of solvent by swirling. Heating is so regulated that the distillate falls from the condenser tip at a rate of from 2 to 5 drops per second. The distillation is continued at

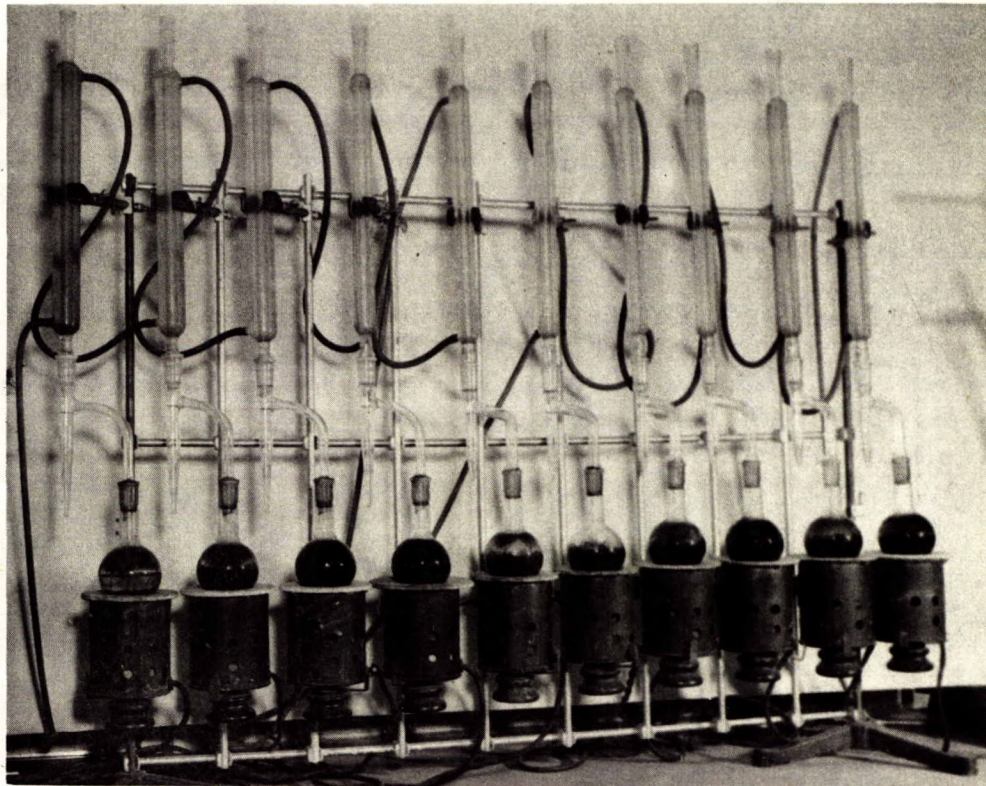


Figure 2 - Multiple Water Determination Apparatus.

the specified rate until no water is visible on any part of the apparatus except at the bottom of the trap.

Between tests it is necessary to clean the apparatus thoroughly to prevent water droplets from adhering to the walls of the trap and condenser. This is accomplished by frequent cleansing with cleaning solution and drying in drying oven.

Calculation

The volume of condensed water measured in the trap multiplied by two (i. e. for a 50 gram sample) is the percentage of water contained in the (air dried) sample tested.

2. Determination of Bitumen (or Oil) and Mineral Matter Content

Scope

This method is suitable for the assay of bituminous sands with bitumen contents above one percent. As a clean sand aggregate is produced, this method may be employed when a screen analysis of the sand is required. In general, this method may be applied to a wide variety of bituminous substances with safety, provided reasonable precautions are taken to ventilate the working area.

Apparatus

Figure 3 shows a convenient arrangement of multiple Soxhlet extraction units.

- (1) Soxhlet extraction unit - large size, similar to Corning Cat. No. 3840.
- (2) Extraction thimbles - Whatman, fat free, double thickness,* filter paper, size 43 x 123 mm.
- (3) Electric heater or hot plate.
- (4) Drying oven (electric).
- (5) Muffle furnace (electric).
- (6) Light metal cans to hold thimbles. See Figure 4.
- (7) Crucibles - 50 ml. capacity.
- (8) Desiccators - for metal cans and crucibles.
- (9) Evaporating lamps or hot plate.
- (10) Analytical balance.
- (11) Miscellaneous glassware - graduated 250 ml. glass stoppered graduates, 25 ml. pipette with rubber suction bulb, etc.

* Double thickness is required to reduce silt correction.

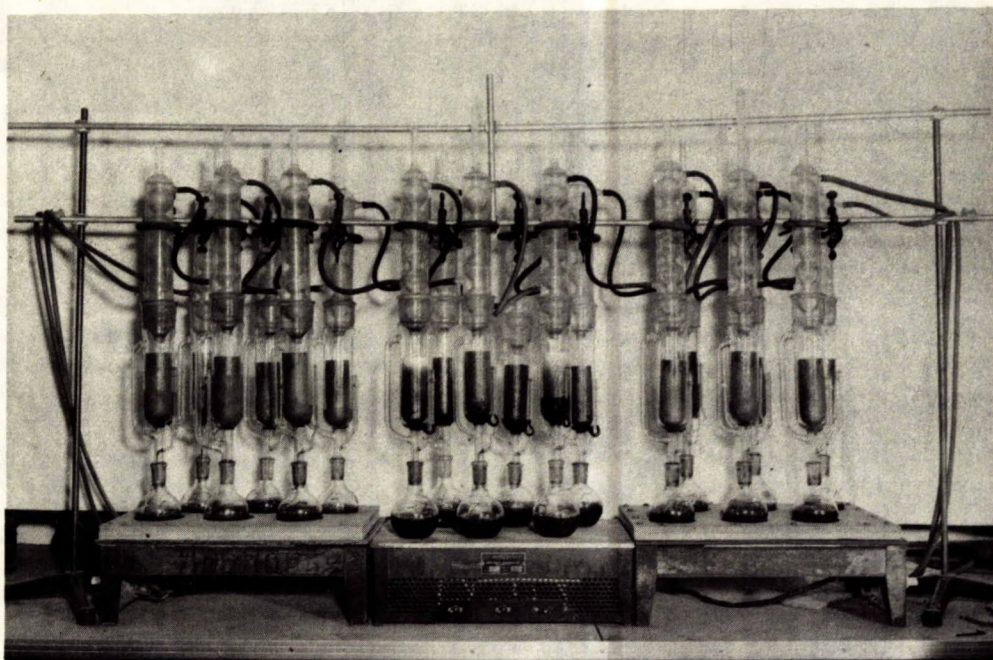


Figure 3 - Multiple Soxhlet Extraction Apparatus

Solvent

Carbon tetrachloride (CCl_4), technical grade, is used at the Fuel Research Laboratories in this extraction for the following reasons: Carbon tetrachloride is not inflammable so the extraction apparatus may be set up on an open bench in a well ventilated room. The relatively high specific gravity of this solvent aids in the suspension of the fine silt coming through the extraction thimbles, making possible a more accurate aliquot of the silt for the estimation of the total mineral aggregate. Carbon tetrachloride is also known to be an excellent solvent for a wide variety of bituminous substances. In general, it is superior to benzene in that it is capable of dissolving a slightly greater percentage of the high molecular weight asphaltic substances.

Procedure

The extraction thimble is placed in an open, numbered, tared metal can, with its lid, and dried in a drying oven at 105 to 110°C. for at least one hour. Then the metal can is removed from the oven, and closed to exclude moisture. After cooling in a desiccator, the metal can is weighed to the nearest 10 mg.

Approximately 50 grams of the sample are then placed in the weighed thimble which in turn is placed in the weighed metal can, the can capped and reweighed in order to determine the weight of the sample. Changes in weight, due to the hygroscopic nature of the extraction thimbles, can be quite large but are readily controlled by

placing the thimble in a light metal can having a closely fitting lid. The aluminum can, shown in Figure 4, weighing approximately 70 grams, has proved satisfactory for prevention of moisture absorption.

The distilling flask of the Soxhlet extractor is filled with 250 ml. of carbon tetrachloride, then the thimble containing the sample is inserted into the upper extraction section of this apparatus. After assembling the apparatus, the boiling rate of the carbon tetrachloride is adjusted to avoid overflowing the thimble. The automatic extraction of the bitumen is continued until the extract surrounding the thimble is colourless. The extraction section of the Soxhlet apparatus is then removed from the distillation flask and the thimble is raised above level of solvent until it has drained. If the drained solvent is colourless the extraction may be considered complete.

The thimble from the extraction section is transferred to its appropriate can and the excess solvent removed under heat lamps in a well ventilated fume hood. When the odour of the carbon tetrachloride is no longer perceptible, the open can containing the thimble and sand residue is transferred to a drying oven and dried overnight at a temperature of 105 to 110°C. After drying, the thimble is removed from the oven, and enclosed in its metal can, which is then cooled in a desiccator for one hour and weighed to the nearest 10 mg.

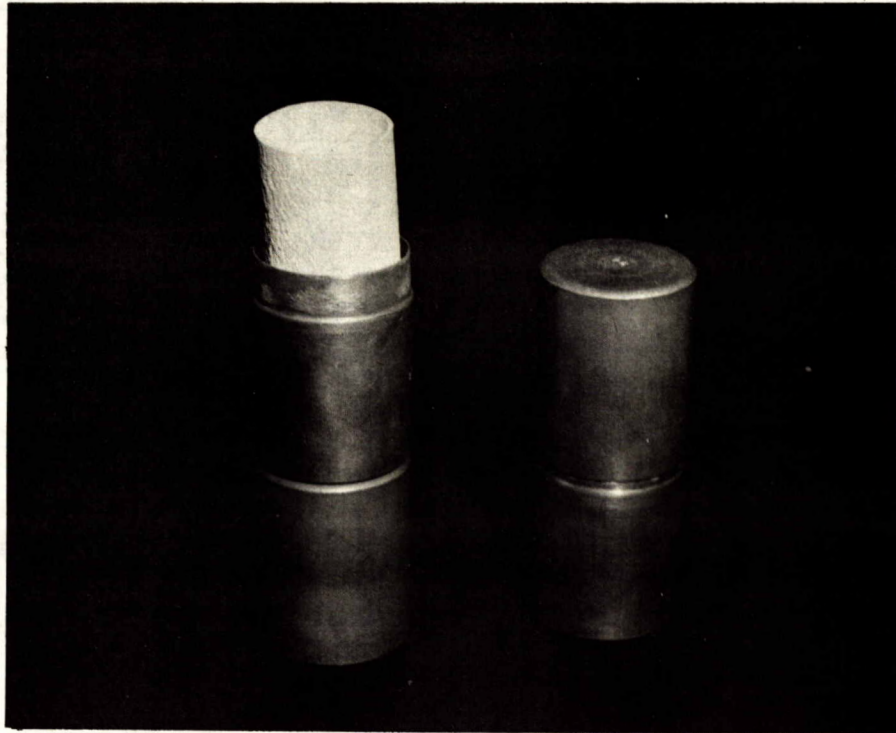


Figure 4 - Metal Holder for Extraction Thimble

The loss in weight of the sample on extraction must be corrected for the fine silt passing through the thimble in the following manner. After the extraction has been completed, the contents of the flask are swirled to bring the finely divided solids into suspension and washed with solvent into a 250 ml. stoppered graduate. After making up the volume to a multiple of 25 ml., the graduate is stoppered and shaken to bring the solids into suspension, and a 25 ml. aliquot is pipetted into a tared crucible and weighed to the nearest 0.1 mg. The solvent is carefully evaporated under heat lamps in a fume hood to avoid spattering. The remaining contents of the crucible are then ashed in a muffle furnace in accordance with the standard procedure for ashing coal or other solid fuel. After removal from the furnace, the crucible is cooled in a desiccator and weighed.

Calculation

Weight of Mineral Aggregate =
Weight of residue in thimble + weight of solids passing
thimble.

Weight of solids passing thimble =
Weight of ash in crucible x $\frac{\text{volume of liquid in graduate}}{\text{volume of aliquot}}$

Percent Mineral Aggregate = $\frac{\text{Weight of Mineral Aggregate} \times 100}{100 - \text{Percent Moisture}}$
(air dried basis)

Percent Bitumen = 100 - Percent Mineral Aggregate
(moisture free basis) (moisture free basis)

3. Screen Analysis of the Extracted Sand Aggregate

Scope

The following method may be applied to determine the size distribution of the extracted and dried sand aggregate between 20 and 200 mesh. Although a sieve analysis of forty grams or so of the sand residue from a fifty gram sample of bituminous sand extracted in a Soxhlet unit will give comparable results on a series of samples, it is recommended that the screen analysis be made on a larger sample obtained by combining the residues from duplicate determinations.

Apparatus

A Tyler Ro-tap shaker machine which is a mechanically operated sieve shaker that imparts to the set of sieves a rotary motion and a tapping action of uniform speed.

Procedure

The procedure followed was that described under A. S. T. M. Designation D 451-40 "Standard Method of Test for Sieve Analysis of Granular Mineral Surfacing for Asphalt Roofing and Shingles"(4). The sieves selected from the set of consecutive sieves, were the 20, 40, 60, 80, 100, 150 and 200 mesh sieves.

4. Extraction of Mineral Matter for Screen Analysis

Scope

This method is applicable to samples containing a relatively large proportion of water such as the tube mill discharge and where

the particle size distribution of the mineral matter is the only quantity sought.

Apparatus

A Soxhlet extractor, suitable hot plate and fume hood facilities.

Procedure

The sample received from the tube mill was allowed to stand to permit the mineral matter to settle. The supernatant water and oil were decanted and a sample of mineral matter sufficient to provide 50 grams of dry sand was withdrawn from the pail and dried under a heat lamp. The dried sample was then placed in a Soxhlet extractor and extracted with about 225 ml. of carbon tetrachloride until the solvent syphoning to the boiling flask was clear. The extracted residue was dried to remove traces of solvent and placed in an envelope prior to conducting the sieve analysis according to Method 3.

5. Analysis of Mineral Matter for Clay Content

Scope

This method may be used to determine the clay content of the extracted mineral matter.

Procedure

Following the sieve analysis of the extracted mineral matter, the material passing the 200 mesh sieve was saved from two to five successive mill runs. The combined sample was analysed for Al_2O_3 content by standard procedures.

Calculation

$$\frac{\text{Al}_2\text{O}_3}{3} \times 3 = \% \text{ clay in mineral matter.}$$

The factor 3 was obtained by assuming all the clay to be in the form of kaolinite $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$.

6. Determination of Oil and Mineral Matter Content by Drying and Extraction

Scope

This method of analysis was used on the sand trainings from the Dorr classifier or where a very large proportion of the sample consisted of sand.

Procedure

About 100 grams of the sample was placed on a watch glass and dried under a heat lamp or in an oven at 105° C. After cooling, a 50 to 60 gram sample was extracted as described in Method 2 except that no aliquot need be ashed, because it was found that the ash correction was negligible.

Calculation

$$\% \text{ Mineral matter (dry basis)} = \frac{\text{Weight of extracted sand} \times 100}{\text{Weight of dried sample}}$$

$$\% \text{ Mineral matter, on wet basis} =$$

$$\% \text{ Mineral matter (dried sample)} \times \frac{(100 - \% \text{ water})}{100}$$

$$\% \text{ Oil} = 100 - (\% \text{ water} + \text{mineral matter (wet basis)})$$

Note: The water content was determined on a 25 gram sample, as described in Method 1.

7. Determination of Water, Mineral Matter, and Oil Content by Evaporation and Ashing

Scope

This method of analysis was used to overcome the difficulties that occurred when the sample contained a large proportion of water which caused rapid segregation. The absolute accuracy of the water and oil determination was not considered to be high owing to the fact that diluent was lost with the water on evaporation. If the evaporation was conducted in a uniform manner and was carefully timed the method was capable of giving as reproducible a result as the sampling procedure warranted.

Procedure

The difficulty of rapid segregation in samples containing large proportions of water was overcome by emulsifying the entire sample by the addition of a suitable wetting agent such as Dupanol G., followed by vigorous agitation with a spatula and the pouring of the sample from one beaker to another. Immediately following this mixing,

a 5 gram sample was placed in a tared No. 2 Coors porcelain evaporating dish and accurately weighed to the nearest 0.1 mg. The sample in the evaporating dish was then dried under a heat lamp. After cooling in a desiccator the evaporating dish was reweighed and the evaporation loss was reported as water. The sample was then ashed by heating over bunsen burners. The weight of the ashed residue was multiplied by the factor 1.1 to correct for the loss in weight of the solids on ignition. This factor was determined experimentally by ashing dry, oil free, sand passing the 200 mesh sieve (5). The corrected weight of the residue was reported as mineral matter. The oil was calculated by difference from the sum of the water and mineral matter. By oil in this report is usually meant extracted bitumen and diluent; however in this method the diluent was largely lost and the oil refers essentially to the bitumen.

Calculation

$$\% \text{ Water} = \frac{\text{Initial sample weight} - \text{Evaporation sample weight} \times 100}{\text{Initial Sample Weight}}$$

$$\% \text{ Mineral matter} = \frac{\text{Residue after ignition} \times \text{factor 1.1} \times 100}{\text{Initial Sample Weight}}$$

$$\% \text{ Oil} = 100 - (\% \text{ Water} + \% \text{ mineral matter})$$

3. Preparation of Sample for Clay Analysis

Scope

This method of sample preparation prior to the clay analysis was necessary when large quantities of water and oil were present in the sample such as in the No. 1 Dorr thickener feed.

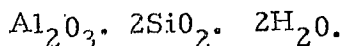
Procedure

Approximately one half gallon of sample was evaporated on a hot plate until all the water and most of the kerosene was expelled. Two successive mill run samples were treated in this manner and when dry they were combined. This mixture was placed in a Soxhlet extraction thimble and extracted with carbon tetrachloride. Care was taken during this extraction, as the thimble had a tendency to become clogged with clay and to overflow. The thimble containing the residue was thoroughly dried and the contents were analysed for Al_2O_3 content by standard wet chemical methods. The clay content was estimated by multiplying the Al_2O_3 content by three and the result was reported to the nearest whole number.

Calculation

$$Al_2O_3 \times 3 = \% \text{ clay in mineral matter.}$$

The factor of 3 in the above relation was based upon the assumption that all the clay consisted of kaolinite (china clay)



9. Determination of Mineral Matter Content by Ashing

Scope

This method was employed when the proportion of oil was large with respect to the water content. Under these circumstances careful drying was necessary before analysis to avoid loss by spattering.

Procedure

A 5 gram sample was placed in a tared No. 2 Coors porcelain evaporating dish and accurately weighed to the nearest 0.1 mg. The sample was evaporated slowly under heat lamps in order to avoid spattering. When evaporation was relatively complete the sample was carefully ashed by heating over bunsen burners. The evaporating dish was cooled and weighed. The weight of residue was then multiplied by the factor 1.1 and reported as mineral matter.

Calculation

$$\% \text{ Mineral matter} = \frac{\text{Residue after ignition} \times \text{factor } 1.1 \times 100}{\text{Initial sample weight}}$$

The oil content was calculated by difference from the sum of the water and mineral matter as follows:

$$\% \text{ Oil} = 100 - (\% \text{ water} + \% \text{ mineral matter})$$

The water content used in the above expression was determined on a 25 gram sample, as described in Method 1.

Miscellaneous Analytical Methods

The diluents employed in the cold water separation process were tested by standard methods of analysis. The dehydrated bitumen after kerosene removal was also analysed by the same methods.

These analytical methods are listed below:

TABLE II

SUMMARY OF MISCELLANEOUS ANALYTICAL METHODS

Flash point (Tag Closed Tester)	A. S. T. M. D 56
Water	A. S. T. M. D 95
Specific Gravity	A. S. T. M. D 1298
Viscosity, Kinematic	A. S. T. M. D 445
Viscosity, Saybolt Universal	A. S. T. M. D 446
Distillation Range	A. S. T. M. D 158
Sulphur Bomb, Method	A. S. T. M. D 129
Color, Union	A. S. T. M. D 155
Pour Point	A. S. T. M. D 97

Distillation, Hempel Method: U.S. Bureau of Mines routine method entitled "The Bureau of Mines Routine Method for the Analysis of Crude Petroleum".

1. The Analytical Method, (6)

Hydrogen Sulphide, Detection of: The presence of hydrogen sulfide was detected by hanging a strip of moistened lead acetate paper in the vapour above the liquid then warming the liquid to drive off occluded gases.

APPENDIX

In order to standardize the form in which the analytical results were submitted by the analysts to the officer in charge, several routine forms were prepared. While these forms are of little general interest they are included so that in the event that research along these lines is resumed the standard forms could be reproduced in quantity with a minimum of delay. Examples of the five standard forms that were used in this project are shown on the following pages.

BITUMINOUS SAND

Water Determination
(A.S.T.M. D95-46)

Date

Sample marked Laboratory No.

Determination No.

Wt. of sample, gm. 50

Vol. of diluent, ml. 150

Vol. of water in trap, ml.

Water in sample, % by wt.

Remarks:

Analyst:

Checked by:

Date:

BITUMINOUS SAND

Date

Soxhlet Extraction with CCl₄

Sample marked	Laboratory No.
Can. No.
Can + thimble + sample gm.
Can + thimble gm.
Sample gm.
Soxhlet No.	
Can + thimble + residue gm.
Can + thimble gm.
Residue gm.	
Aliquot
Crucible No.
Crucible + Ash gm.	
Crucible gm.
Aliquot ash gm.
Total ash gm.
Residue + ash gm.
Residue + ash, % of sample
Average, residue + ash, % of sample
Average, residue + ash, % of sample, (moisture-free basis)	
% of bitumen (moisture-free basis)

Remarks:

Analyst:

Checked by:

Date:

BITUMINOUS SAND

Date

Screen Analysis

Laboratory No.

Wt. of sample gms.

Retained on	20 mesh	gms.	%
	40 mesh	gms.	%
	60 mesh	gms.	%
	80 mesh	gms.	%
	100 mesh	gms.	%
	150 mesh	gms.	%
	200 mesh	gms.	%
	Bottom	gms.	%
	Total	gms.	%

Remarks:

Analyst:

Checked by:

Date:

BITUMINOUS SAND

Date

Ash Test

Crucible No.
Crucible + sample, gm.
Crucible , gm.
Sample (wet). gm.
Crucible + dry sample, gm.
Crucible, gm.
Wt. of dry sample, gm.
Crucible + ash, gm.
Crucible, gm.
Wt. of ash, gm.
	Wt. in gm.	Percent
Oil
M. M.
Water

Remarks:

Analyst:

Checked by:

Date:

BITUMINOUS SAND

Report of Analysis

Date

Laboratory No.

Description of Sample:

.....
.....
.....

Oil Content:

.....

Mineral Matter Content:

.....

Water Content:

.....

Screen Analysis: Retained on Percent

20 mesh

40 mesh

60 mesh

80 mesh

100 mesh

150 mesh

200 mesh

Bottom

Remarks:

Reported by:

REFERENCES

1. Djingheuzian, L. E., "Pilot Plant Investigations on Cold Water Separation of Bitumen from Alberta Tar Sands". Mines Branch, Department of Mines, Canada. Research Report No. MD70.
2. Goodspeed, F. E., Holland, J. G., Draper, R. G., and H. McD. Chantler, Mines Branch, Department of Mines and Technical Surveys, Canada, F.R.L. No. 264.
3. Committee D-2. "A. S. T. M. Standards on Petroleum Products and Lubricants" American Society for Testing Materials. Philadelphia, U. S. A., 1934 to 1950, inclusive.
4. Committee D-4, "A. S. T. M. Standards on Bituminous Materials for Highway Construction, Waterproofing, and Roofing". American Society for Testing Materials. Philadelphia, U. S. A., 1940.
5. Gilmore R. E., and Dyck W. J., "Methods for Assaying Alberta Bituminous Sands" Presented before the Canadian Institute of Mining and Metallurgy - March 20-21, 1944.
6. Smith, N. A. C., Smith, H. M., Blade, O. C., and Garton, E. L. "The Bureau of Mines Routine Method for the Analysis of Crude Petroleum". 1. The Analytical Method. U. S. Bureau of Mines, Bulletin No. 490.

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