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Cold-Water Method of Separation of Bitumen  
from Alberta Bituminous Sand<sup>\*</sup>

By

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L. E. Djingheuzian, A.R.S.M., M.Inst.M.M., M.C.I.M. \*\*

The writer of this submission has a great admiration for Mr. S. M. Blair, whose study of the problem posed by Alberta bituminous sands, a study embodied in his monumental report, places this problem on the eve of a successful solution. It is through his dynamic efforts that we are assembled here today to initiate, and of this the writer is sure, another great romantic chapter in the history of Canadian mining, which chapter will go far in advancing the cause of our civilization and democratic tradition.

When the writer, who is only a newcomer into the fold of Alberta bituminous sands, thinks of such veterans as Dr. K. A. Clark, W. M. Ball, Dr. D. S. Pasternack, the late McClave, a glorious band of scientists and technologists who were later joined by W. E. Adkins and who untiringly worked for years on developing methods for processing economically the bituminous sands, indeed he considers it a privilege to be permitted to offer his modest contribution before this distinguished audience.

One of the processes which Mr. Blair describes in his

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report is the cold-water process of separation of bitumen from Alberta bituminous sand as worked out at the Mines Branch in Ottawa on a small pilot-plant scale. The idea of using cold water, being in contrast to the use of hot water, is probably inherent in the hot-water method and this, the writer believes, is what caused initiation of the experimental work on cold-water separation of bitumen in the laboratories of Abasand Oils Limited.

The laboratory work having indicated the feasibility of cold-water separation, a cold-water separation pilot unit was installed by the General Engineering Company of Toronto, who were managers of the plant of Abasand Oils Limited. This pilot unit consisted of a mixer to which the bituminous sand together with water and light oil, the latter having properties of kerosene, was fed at around 70° to 80°F. The mixer was followed by a Geco flotation cell, the floated diluted bitumen from the cell going to settling chambers. The results obtained with this pilot unit were stated to be so good that the whole hot-water plant of Abasand was converted to the cold-water process. Unfortunately, before this cold-water plant, expected to handle upwards of 500 tons of bituminous sands in 24 hours, was started, it was destroyed by fire in 1945. In this fire practically all the records of the preliminary investigations were lost.

When, after the fire, Abasand Oils Limited was unable to continue the experimental work, laboratory research was started at the Mines Branch in Ottawa. This laboratory work was carried out by H. L. Beer and subsequently by P. V. Rosewarne and A. A. Swinnerton under general supervision of Dr. T. E. Warren. A trial semi-pilot run was made by H. L. Beer and a 200 lb. per hour pilot plant was designed and erected.

## Cold-Water Method

### Investigations and Results

Pilot plant investigations using cold-water method of separation of bitumen, for which 150 tons of bituminous sand was obtained from the Horse River property of Abasand Oils Limited, near McMurray, were started on April 19, 1949, and completed on May 19, 1950.

The purpose of these pilot plant investigations was to establish cold-water separation of bitumen from Alberta bituminous sands as an ore dressing process, to work out the operational technique, and to establish the most economical flowsheet possible.

To do this it was necessary to proceed in a systematic way, each week's run being made under constant conditions so as to obtain more reliable data. The performance of each machine had to be evaluated, basing these evaluations on the results of weekly runs. The characteristics of each procedure tried had to be determined with a view to deciding on a machine or a re-arrangement of the flowsheet which might be an improvement on the previous one. Sampling procedure had to be most carefully standardized. In addition, the samples had to be assayed within 24 hours, for which purpose a special section was set up at the Division of Fuels for fast analysis of the samples. A close check was kept on the controlled additions of the alkaline reagents and wetting agents, and effects of those additions studied.

In pilot plant investigations the following definitions have been used:

Diluent = petroleum distillate, which added to the feed would combine with liberated bitumen,

Oil = combined bitumen and diluent,

Crude oil or finished product = #1 thickener overflow,

Classifier sands = mineral matter eliminated by the classifier from the feed,

Tails = #2 thickener underflow,

Total tails = classifier sands + #2 thickener underflow,

Classifier extraction = oil overflowed by the classifier and pumped into #1 thickener,

Recovery = oil overflowed by #1 thickener and delivered to the refinery,

Actual recovery = bitumen input less total oil loss in terms of bitumen,

Hence:

$$\% \text{ sands eliminated by classifier} = \frac{\text{Dry classifier sands}}{\text{Mineral matter in feed}} \times 100$$

$$\% \text{ classifier extraction} = \frac{\text{Oil input} - \text{Oil in classifier sands}}{\text{Oil input}} \times 100$$

$$\% \text{ indicated recovery} = \frac{\text{Oil input} - \text{Oil in total tails}}{\text{Oil input}} \times 100$$

$$\% \text{ actual recovery} = \frac{\text{Bitumen input} - \text{Oil (in bitumen terms) in total tails}}{\text{Bitumen input}} \times 100$$

The bituminous sands can be regarded as, essentially, a compacted mass of sand particles held together by bitumen which forms a film around each particle. When a light oil having the properties of kerosene is added to the bituminous sands at the same time as the bitumen film around sand particles is ruptured or scoured off by some rubbing action, bitumen dissolves in the light oil, forming a compound oil with a specific gravity of less than 1. When this process takes place in water, the heavier than

water sand particles, liberated from oil, fall to the bottom of the vessel and the lighter oil rises to the surface. Thus, fundamentally, the cold water process is a gravity concentration process in which the valuable constituent of the ore rises to the surface and the gangue sinks to the bottom.

In the preliminary stages of pilot plant investigations the process consisted of the four following steps:

- (1) Disintegration of bituminous sands by means of a low-discharge pebble-mill, kerosene in amounts approximately equal to the bitumen content of the sands being fed into the pebble-mill together with the sands. Soda ash was also added to the feed.
- (2) Separation of combined bitumen-kerosene, designated as oil, from sands in an agitator.
- (3) Classification of agitator discharge, by means of a rotary classifier, into sands and oil slurry.
- (4) Settling the oil slurry with cone classifiers and in tanks.

A product suitable for the heat-treatment plant (refinery), as designed by the Division of Fuels, was obtained.

The optimum temperature ranges were determined as lying between 73° and 81°F. Below 73°F. the separation became less efficient and incomplete. When the temperature approached 83°F. the sands discharged from the rotary classifier started becoming dirty and more free oil ran over with them, while higher temperatures resulted in still dirtier sands discharge. Thus the temperature control of the classifier overflow was the key to the

efficient operation of the plant with high bitumen recoveries.

In these preliminary runs the indicated recoveries of oil were over 97 per cent, and samples of crude oil, taken with a dipper from a tank after overnight settlement, averaged 76.4 per cent oil, 1.9 per cent mineral matter, and 21.7 per cent water. The requirements of the refinery for the crude oil, as specified by the Division of Fuels, were "not less than 65 per cent oil and not more than 5 per cent mineral matter and 30 per cent water", and a crude oil suitable for treatment in the refinery was always obtained when settling was allowed. However, in a big plant settling meant a very large tankage capacity, which from the ore dressing point of view did not appear to be attractive. Hence, it was decided that, while keeping the recoveries as high as possible, all the efforts should be directed towards obtaining a crude oil of sufficiently high grade without the necessity of prolonged settling.

It turned out that this was a wise decision. As the investigations progressed, it was found that settling caused an excessive oil loss through the formation of an oil emulsion termed sludge which settled at the bottom of the tanks. When attempts were made to adapt the ore dressing technique, already worked out, to the recovery of oil from the sludge they proved to be failures.

Djingheuzian and Warren say<sup>(3)</sup>:

"The magnitude of this loss was not known at first and it was not taken into consideration in calculating the recovery of oil. In

the final arrangement of apparatus, a different settling procedure eliminated sludge formation.

"During the preliminary runs, the equipment was progressively altered and the final apparatus and operating scheme were as follows: The low-discharge pebble mill was satisfactory, as initially used for the mixing and disintegration step. With a charge of 0.5 lb. of pebbles per lb. of bituminous sand per hour, the oil was separated with very little size reduction of the sand. The agitator was also satisfactory. The vertical centrifugal pumps were found to cause froth formation and were replaced by suction pumps with a positive action. The rotary classifier was replaced by a 14 in. by 8 ft. duplex rake classifier. The settling operation was carried out in two thickeners. The classifier overflow was pumped into the first of these, which was 4 ft. in diameter and 4 ft. deep. The overflow from the first thickener was the finished product of the hydrometallurgical section of the plant (cold-water separation plant). The underflow from the first thickener, which still carried appreciable amounts of oil, was pumped to a second thickener 4 ft. in diameter and 3 ft. deep. The underflow from the second thickener was sent to waste and the overflow returned to the first thickener. The settling tanks were removed in order to avoid prolonged settling which was always attended by sludge formation. The revised flowsheet is shown in Fig. 1".

The combination of the machines shown in Fig. 1, with the use of soda ash and suitable wetting agents, eliminated the necessity of settling the thickener overflow, and a crude oil of a grade sufficiently high to be suitable as a direct feed to refinery was produced in a continuous process.



The Flowsheet

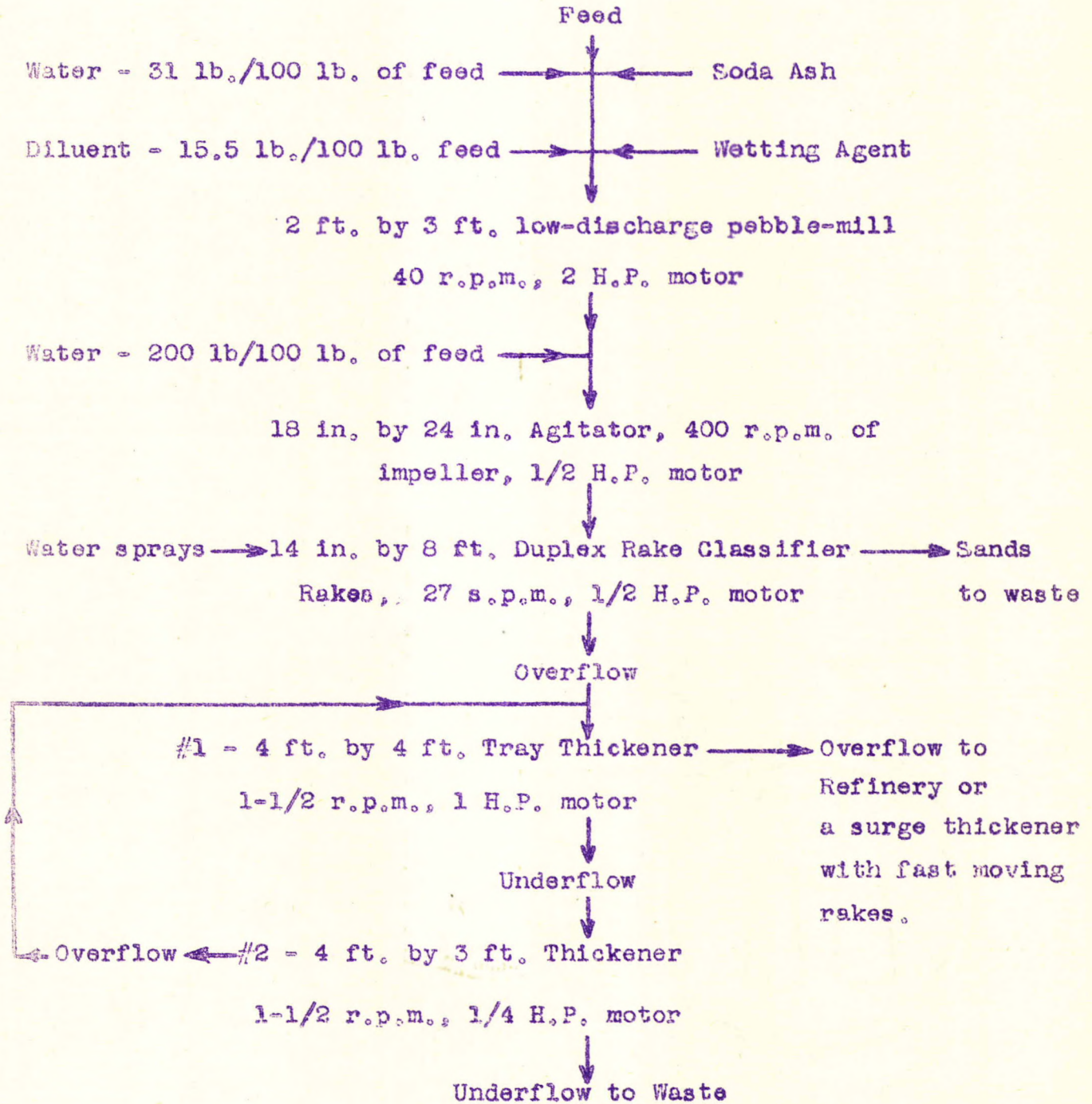


Fig. 1

TABLE I

| Bituminous Sands treated, lb. | Rate of Feed, lb./hr. | Soda Ash, lb./ton | Wetting Agent, lb./ton | Aver. Temp., °F. | pH of C.O.F.* | Per Cent Bitumen in Tar Sands | Viscosity of Combined Oil, Centipoises | Per Cent Sands Eliminated by Classifier | Per Cent Oil in Class. Sands | Series of weekly |  |  |
|-------------------------------|-----------------------|-------------------|------------------------|------------------|---------------|-------------------------------|--|---|------------------------------|------------------|--|--|
|                               |                       |                   |                        |                  |               |                               |  |   |                              |                  |  |  |
| 6550                          | 238.1                 | 1.46              | Span 80<br>0.041       | 77.4             | 9.5           | 15.8                          | 103.7                                  | 92.8                                    | 0.41                         |                  |  |  |
| 4050                          | 238.1                 | 1.45              | Rosin Amine,<br>0.041  | 77.2             | 9.2           | 16.2                          | 103.7                                  | 89.6                                    | 0.47                         |                  |  |  |
| 6600                          | 300.0                 | 1.56              | Span 80<br>0.031       | 78.3             | 9.3           | 14.9                          | 103.7                                  | 88.3                                    | 0.38                         |                  |  |  |
| 3100                          | 215.0                 | 1.51              | Span 40<br>0.041       | 77.0             | 9.1           | 15.9                          | 103.7                                  | 82.0                                    | 0.26                         |                  |  |  |
| Series of weekly              |                       |                   |                        |                  |               |                               |  |   |                              |                  |  |  |
| 4450                          | 170.0                 | 0.68              | Span 80<br>0.062       | 79.0             | 8.6           | 15.4                          | 336                                    | 87.7                                    | 0.26                         |                  |  |  |
| 5700                          | 215.8                 | 0.89              | Span 80<br>0.045       | 81.5             | 9.0           | 16.5                          | 336                                    | 92.3                                    | 0.35                         |                  |  |  |

\* Classifier Overflow

| Per Cent. Extraction of Oil by Classifier | Per Cent Oil in #2 Thick. U.F.-Tails | Tails to Waste, lb. | Per Cent Indicated Recovery | Per Cent Actual Recovery | Loss of Bitumen, lb./ton | Loss of Diluent, lb./ton | #1 Thickener Overflow Per Cent |      |       |
|---|--------------------------------------|---------------------|-----------------------------|--------------------------|--------------------------|--------------------------|--------------------------------|------|-------|
|   |                                      |                     |                             |                          |                          |                          | Oil                            | M.M. | Water |
| runs with kerosene                        |                                      |                     |                             |                          |                          |                          |                                |      |       |
| 98.6                                      | 0.12                                 | 14,960              | 97.8                        | 95.6                     | 7.2                      | 6.8                      | 71.3                           | 2.1  | 26.6  |
| 98.6                                      | 0.17                                 | 11,000              | 97.1                        | 94.2                     | 9.4                      | 8.9                      | 71.5                           | 1.3  | 27.2  |
| 98.8                                      | 0.12                                 | 18,040              | 97.7                        | 95.4                     | 6.9                      | 7.4                      | 75.6                           | 2.2  | 22.2  |
| 99.3                                      | 0.10                                 | 10,120              | 98.2                        | 96.4                     | 5.7                      | 5.6                      | 75.6                           | 1.7  | 22.7  |
| runs with Bitumount diluent               |                                      |                     |                             |                          |                          |                          |                                |      |       |
| 99.2                                      | 0.18                                 | 20,240              | 96.5                        | 93.0                     | 10.6                     | 10.5                     | 67.5                           | 3.0  | 29.5  |
| 99.0                                      | 0.12                                 | 22,880              | 97.5                        | 95.0                     | 8.1                      | 8.5                      | 70.9                           | 2.7  | 26.4  |

Table I gives the results obtained with the last six weekly series of runs, the first four being made to test the effectiveness of different wetting agents using kerosene as a diluent, and the last two using as a diluent, a heavier-than-kerosene petroleum distillate obtained from the experimental plant operated by the Alberta Government at Bitumount, Alberta.

Several observations, which also sum up the results obtained from numerous daily runs, can be made from a study of the above table:

- (1) In every series of runs, with high overall recoveries maintained, a thickener overflow was obtained which could be fed directly into the refinery.
- (2) The optimum amounts of soda ash required per ton of tar sands are governed by pH of the classifier overflow. Whenever this pH dropped below 9.0, the recoveries started to fall off.
- (3) The beneficial effects of all three wetting agents on the treatment of tar sands were firmly established. Indications were that Span 40 gave the best results, followed by Span 80 and Rosin Amine D Acetate.
- (4) The indicated recoveries of oil by the use of the Bitumount diluent were practically the same as with kerosene, and, more important, the grade of the thickener overflow was suitable as a refinery feed so that settling was unnecessary.

Since the diluent to be used in a large commercial operation must be a derivative from the bitumen itself,

it was essential to ascertain whether the ore dressing technique, already worked out with kerosene as a diluent, would apply also when using heavier oils directly derived from bitumen. For this purpose, six barrels of an oil derivative from Alberta bituminous sands were obtained from the Bitumount plant in Alberta.

Notes on the Effect of Clay in Bituminous Sands and the Effect of Reduced Water Additions to the Mill Circuit on Oil Recoveries

As it is well known the Alberta bituminous sands carry varying amounts of clay.

The observations made at the Mines Branch on the effect of clay on oil separation and recoveries in cold water process were similar to those made already by Clark and Pasternack in the hot-water separation process<sup>(1)</sup>. Those, who are interested, are referred to this publication as well as to a paper by Djingheuzian and Warren<sup>(3)</sup>, and the Mines Branch Research Report No. MD70<sup>(2)</sup>.

Investigations were also carried out on the effect of reduced water additions on oil recoveries. Though it was found that for the highest recovery of oil a total water to feed ratio of 3:1 by weight was the optimum ratio, there was sufficient evidence to show that the best economical balance would be obtained with water to feed ratio of somewhere between 2.5:1 and 2:1.

Summary and Conclusions

- (1) A flowsheet with standard ore dressing machinery was developed which, using Bitumount diluent and small amounts of soda ash and wetting agent, obtained an actual recovery of 95 per cent of bitumen from Horse River sands.
- (2) The use of soda ash is imperative. When its use was discontinued, the result was always low recoveries of oil, even if the pH was over 9.
- (3) The optimum ranges of temperature were found to be between 73° and 81°F, though good recoveries were also obtained at as low a temperature as 70°F and as high as 83°F.
- (4) The optimum pH required appeared to be just over 9.
- (5) To obtain the best recoveries the mineral matter in bituminous sands had to be reduced in the pebble-mill from 5 to 7 per cent minus 200 mesh to 11 to 15 per cent minus 200 mesh. This grind was the measure of efficient loosening and scouring of the bitumen off the sands.
- (6) Increases in clay contents affected thickener recoveries adversely. However, since the best separation of oil in the classifier took place with a certain content of clay in the mill feed, there appears to be an optimum content which will assist in effecting the highest overall recoveries.
- (7) Crude oil of a grade suitable for refinery treatment was produced consistently in a continuous process.

Commercial Application of the Cold Water Method to  
Processing of Alberta Bituminous Sands

It is evident that commercial exploitation of bituminous sands is possible only on a large scale. However, the operation of the Mines Branch pilot plant cannot be used as a basis for the design of a mill for treating 20,000 cu. yards ( $\approx$ 33,700 tons) per 24 hours mill because the operation of the pilot plant does not give sufficiently conclusive data on mill capacities for large-scale calculations. It is suggested that the operation of a 500 ton per 24 hours pilot mill will furnish the necessary data for the design of a 20,000 cu. yard mill. Accordingly, a flowsheet for a 500 ton mill was designed.

The cost of the equipment for this 500 ton mill, together with motors F.O.B. Waterways, Alberta, will come to around \$140,000 (1950 prices) or, it might be said that the cost of the 500 ton mill erected on site will be roughly \$300,000. This will also include the cost of the mill laboratory.

Fig. 2 shows this flowsheet for the 500 ton mill.

Flowsheet.

Total installed horsepower: 201.

Feed = 500 tons of bituminous sands per day.

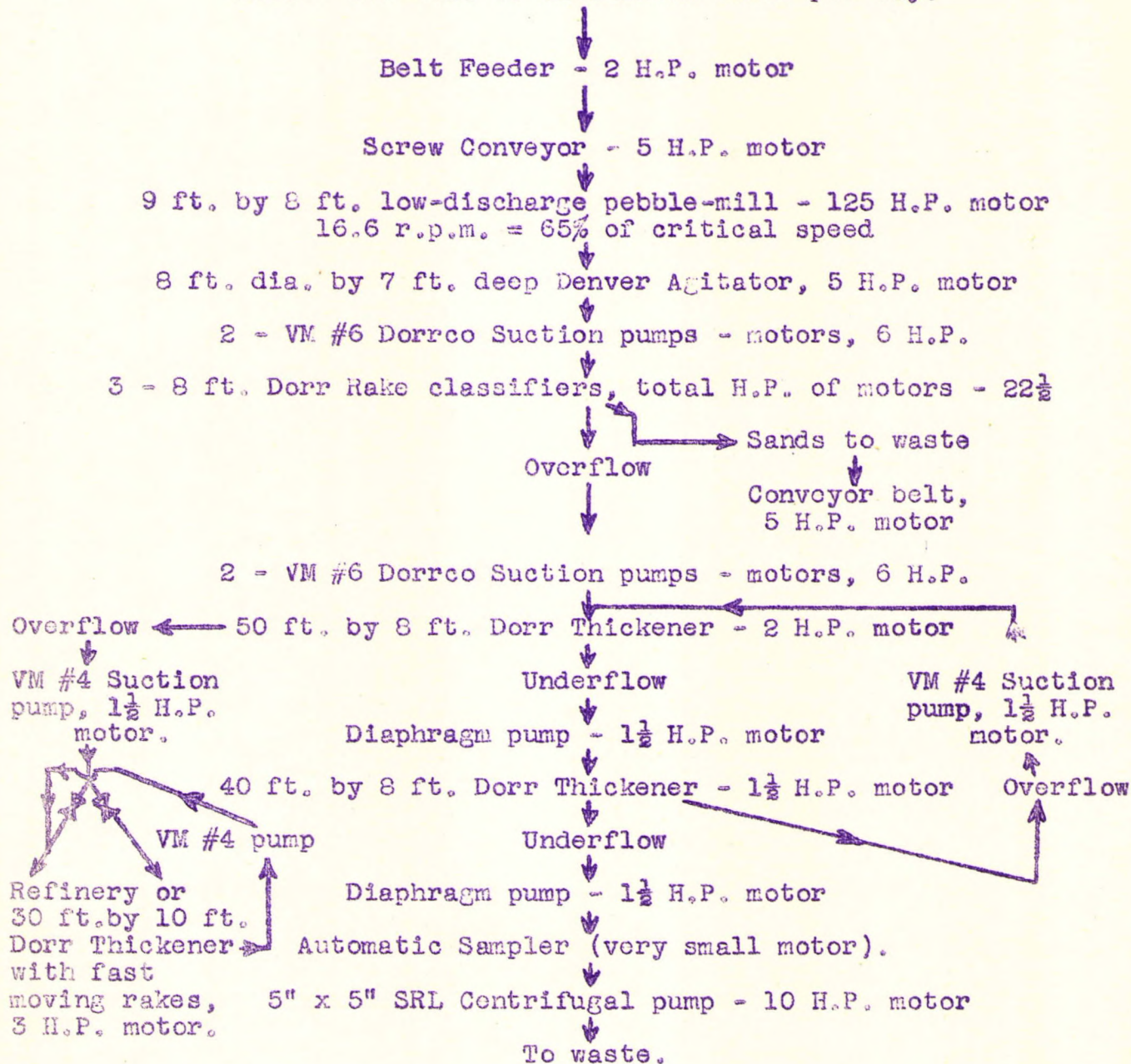


Fig. 2

While conducting his survey, Mr. Blair asked the writer whether he could prepare for him an estimate for a 20,000 cu. yards per 24 hours cold-water separation plant, together with an estimate of personnel required to operate the plant. The writer told Mr. Blair that with the present data on hand such an estimate could, at the best, be only his best guess, hence, a very rough affair. However, on being assured that his estimate would be treated as such, which the writer is very pleased to say is the case in Mr. Blair's report, he, the writer, prepared a rough estimate for Mr. Blair, which he is appending in a slightly revised form. The slight revisions were necessary when more information became available with the issuance of Mr. Blair's report.

Estimated Capital Cost of the Plant

|   |              |
|---|--------------|
| 1. Machinery, complete with electrical equipment,<br>F.O.B., Waterways, Alberta . . . . .   | \$6,000,000  |
| 2. Add 100 per cent for costs for the erection<br>of buildings and installation of<br>machinery, and also costs for trans-<br>portation of machinery and materials<br>from Waterways to the site of<br>construction . . . . . | \$6,000,000  |
| <hr/>   |              |
| Total estimated capital cost . . . . .  | \$12,000,000 |
| <hr/>   |              |



A Preliminary Rough Estimate of a Cold Water Separation Plant,  
treating 20,000 cubic yards of Alberta Bituminous Sands per  
24 hours with an estimate of Personnel required to operate  
the Plant.

Skeleton Flowsheet

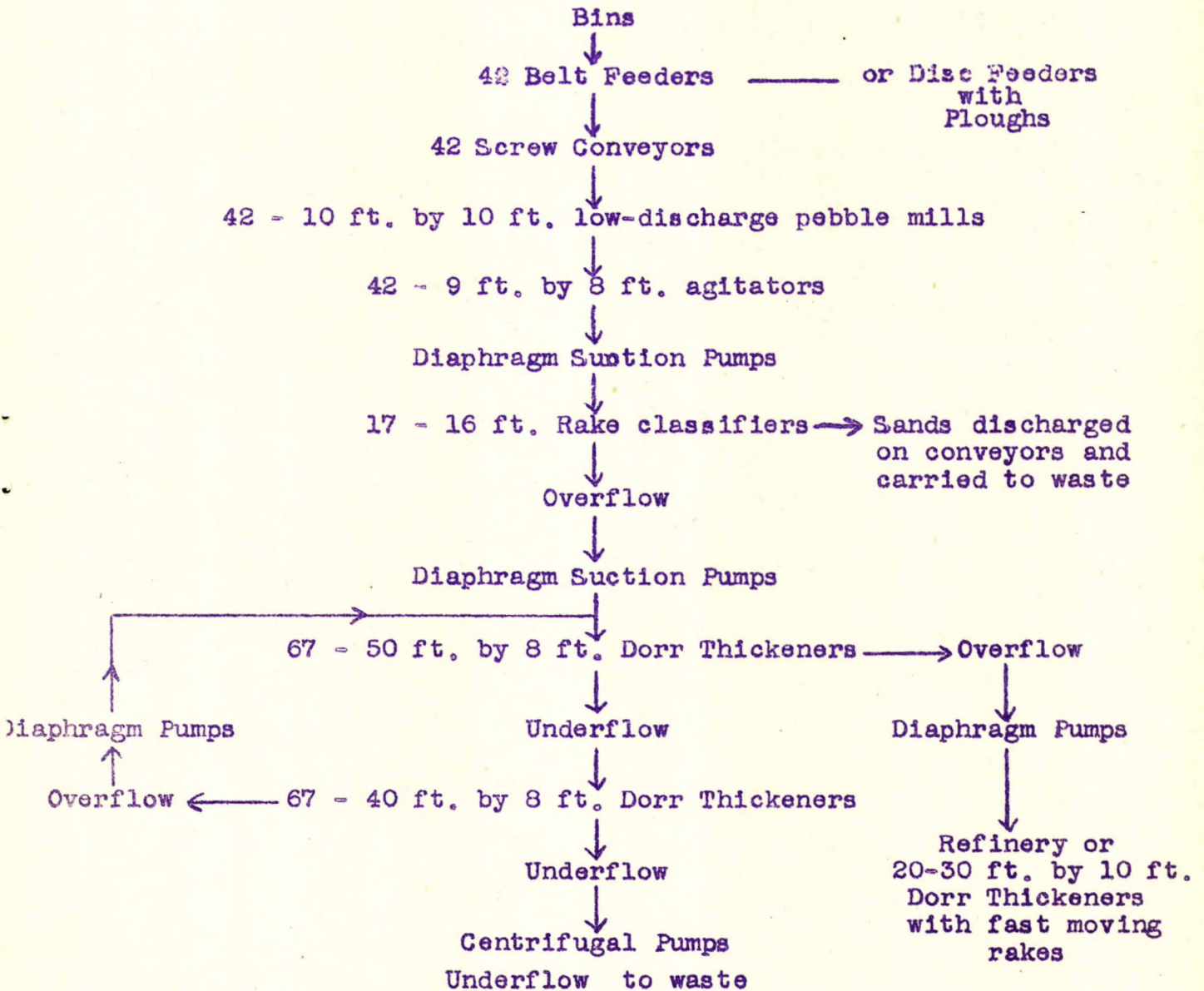


Fig. 3

In the above estimate of the cost of machinery, since there was no time to obtain proper specifications and quotations for some of the machines from the manufacturers, the writer had to base prices of these machines on his past experience and to bring them to 1950 levels.

The cost of erection of buildings, installation of machinery, etc., was estimated at 100 per cent of the machinery cost. This was based on the cost of the erection of buildings, installation of machinery, etc., for the Bitumount plant which cost was 98 per cent of the total cost of machinery, F.O.B., Waterways. However, it must be added that within the writer's experience, the costs of building, etc., at the mining camps of Northern Ontario and Quebec have been around 125 per cent of the cost of machinery, F.O.B., manufacturing points.

The flowsheet is based on the following assumptions:

(1) Tonnages handled by pebble mills are directly proportional to the pebble charge. In other words, at 0.6 lb. of pebbles per lb. of feed per hour, a 10 ft. by 10 ft. mill will handle 471 cu. yards or 785 tons of bituminous sands per day.

If, on the other hand, the capacity of the mill varies as the diameter to 2.4 power, which is the capacity of cylindrical mills when grinding paints, then the capacity of a 10 ft. by 10 ft. mill will be 1471 tons, or around 872 cu. yards of bituminous sands per day. This, by cutting down the required number of 10 ft. by 10 ft. mills to 23, will cut down the capital cost and, by cutting down labour, power, etc., will cut down operating costs also.

(2) Rough calculations indicate that a 16 ft. Dorr rake classifier will handle 2000 tons or around 1185 cu. yards of bituminous sands per day. It is, of course, expected that classifier capacities must be first determined by the operation of a 500-ton pilot mill.

In actual practice, it would be better to use a smaller classifier for each individual pebble mill. The advantages of this method are obvious, since this would eliminate the necessity of controlling the amount of feed to each classifier.

(3) A 50-ft. by 8-ft. Dorr thickener will handle a minimum feed, equivalent to the original 500 tons or 300 cu. yards of bituminous sands per day. Thickening would thus require sixty-seven 50-ft. thickeners. However, it is expected that a 50-ft. thickener would handle more feed, and if this be so, then less than sixty-seven thickeners will be required to handle a feed equivalent to 20,000 cu. yards per day. This will also cut down the number of secondary thickeners required, thus effecting more reduction in capital and operating costs.

It might be asked here why not use thickeners of larger diameter? A 100-ft. thickener, for instance, has 4 times the capacity of a 50-ft. thickener, but, by using a 100-ft. thickener, the time of contact is correspondingly increased and this might contribute towards sludge formation.

From the foregoing estimate and discussion, it can be seen that the above flowsheet gives the picture of the cold-water separation plant in its most expensive form. However, it must be emphasized that no design of a 20,000 cu. yard plant can be con-

templated before the capacities of the various machines are established by the operation of a 500 ton or 300 cu. yard per 24 hours pilot unit. There is little doubt in the writer's mind that with the data obtained from the operation of this pilot unit, the flowsheet given in this report can be vastly improved and costs reduced.

Basing the estimate of personnel on the flowsheet given in this report, the following is the proposed list of superintending staff and operating labour for a 20,000 cu. yard per 24 hours plant for cold-water separation of bitumen.

Personnel

- 1 - Mill Superintendent
- 1 - Assistant Mill Superintendent
- 1 - Research Engineer
- 4 - Laboratory Assistants
- 3 - Mill Shift Bosses
- 1 - Grinding Engineer
- 3 - Thickener Shift Bosses
- 18 - Pebble Mill Operators
- 18 - Pebble Mill Helpers to look after agitators, feeding of pebbles, etc.
- 6 - Classifier Operators
- 21 - Primary Thickener Operators
- 15 - Secondary Thickener Operators
- 3 - 30-ft. Thickener Operators

Mill Office Staff

- 1 - Mill Statistician (preferably a young graduate in ore dressing)
- 1 - Draftsman
- 1 - Stenographer
- 2 - Clerks

Maintenance labour is not included in the above estimate of the personnel. However, for the purposes of estimating costs, the maintenance charge per year may be assumed to be 5 per cent of the capital cost. Actually, this will be a very high figure, since in ore dressing installations, because of the robustness of ore dressing machinery, the maintenance cost is much lower.

The personnel estimate also does not include labour, in case it is required, for feeding bituminous sands from the bins onto belt feeders. The feeding of sands was a problem that the writer had neither opportunity nor means of studying at the Mines Branch.

Since feeding arrangements appear to be a definite problem, it must be listed as a separate step in the process and given a thorough study at the 500-ton pilot plant. However, for the purposes of this preliminary estimate, it is suggested that, provisionally, feeding methods used at Bitumount should be considered in order to arrive at some idea as to the extent that the feeding process is going to influence the costs.

Commenting further on the above estimate of the

personnel, it must be said here that it is fully expected that this estimate be checked. At the same time, no estimate can be considered correct till time studies are made when operating a 500-ton pilot mill.

To illustrate the point just an example will suffice: At Chino Mill, Nevada Consolidated Copper Co., one man per shift takes care of 21 thickeners, ranging from 20 to 75 ft. in diameter. Whereas, in this estimate, the writer assigned 10 thickeners to a man per shift, his chief reason for this being that the overflows of the thickeners in cold-water separation of bitumen must be controlled very closely. In addition, the writer, to make this control as foolproof as possible, had to create three additional jobs, namely, those of thickener shift bosses. It is possible that the operation of a pilot plant might show that a man can take care of more than 10 thickeners and, at the same time keep the overflows under close control. However, till complete data are obtained from the pilot plant operations, care should be taken that, for the purposes of this estimate, every possible factor/<sup>which</sup> might influence the costs be taken into consideration.

#### References

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