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BENEFICIATION OF KANSAS NUMBER FOUR SALT

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ABSTRACT

The salt industry is of major importance among the mineral industries in Kansas. As a consequence of standard producing methods some grades of mined rock salt have less market potential than others and the present study was designed to investigate means of up-grading a less attractive grade referred to in the industry as no. 4 rock salt. Cleaning tests on Kansas no. 4 rock salt show that washing, flotation, agglomeration tabling, and possibly jigging can be applied to remove shale, clay, and calcium sulfate. Flow sheets and cost estimates are given.

INTRODUCTION

The product of the Kansas salt industry in 1950 was valued at $6,542,250, placing it high among the nonfuel mineral industries of the State. Salt is produced commercially in Kansas by five companies operating mines and plants at Hutchinson, Lyons, and Kanopolis and the reserves of salt in the State are vast, underlying extensive areas in central, south-central, and southwestern Kansas. In mining operations several grades of rock salt are of necessity produced and the demand for the several grades as now generally prepared for the market is not in proportion to the quantity produced. As a result the grade referred to in the industry as no. 4 rock salt is produced in excess. It was the purpose of the present study to discover a method of beneficiation of this grade so as to increase its marketability.

The preparation of Kansas rock salt for market consists simply of crushing the run-of-mine salt and screening it to give various sizes. These size fractions, shown in Table 1, are sold without removing any impurities that may be present. The run-of-mine salt contains small but appreciable percentages of insoluble or nearly insoluble materials consisting primarily of anhydrite and some gypsum with shale and clay. Although impurities are undesirable in all grades of rock salt, they are most objectionable in the fines passing a 9-mesh screen, known as no. 4 salt. A certain amount of no. 4 always results when the salt is crushed to yield the more desirable sizes, and the marketing of this unavoidable product is a problem for the salt producers.

Results of laboratory tests are described in this report. These tests have demonstrated the feasibility of several methods of removing much of the insoluble fraction from the no. 4 salt, thus upgrading its quality to meet the specifications for several uses. By cleaning and possibly also pelletizing, no. 4 rock salt has a greatly expanded market potential.

Acknowledgments.—On March 26 and 27, 1951, a conference was held in the offices and laboratories of the State Geological Survey and the Department of Mining and Metallurgical Engineering with representatives of the salt industry in Kansas. This conference was for the purpose of presenting the preliminary results of this study to the industry and gaining suggestions from them. It was attended by representatives of the following companies: American Salt Company, Barton Salt Company, Carey Salt Company, Independent Salt Company, and Morton Salt Company. Additional conferences were held with representatives of the Regional Meat Inspection Laboratory and the Pure Food and Drug Administration, in Kansas City. Thanks are expressed to these companies and agencies for their assistance and interest in this project.

LABORATORY TESTS

Composition of Samples

The test work was conducted on samples of no. 4 salt supplied by the Independent Salt Company of Kanopolis. This material has a gray appearance (Pl. 1) caused by a thin coating of very fine particles of shale on the white halite (salt) particles. In addition to the very fine particles of shale that give a dirty appearance there are many discrete particles of black shale which are quite free of salt and also particles of salt that have enough shale within the crystal to cause them to be dark in color. Gypsum and anhydrite, which are also present, are not so readily seen because their white color is comparable to that of the halite fragments that constitute most of the material in this grade.

Table 1 contains data about samples of the several rock salt grades of the Independent Salt Company for comparison with their no. 4 grade of rock salt. The comparison shows that the coarsest grade, 0.75 to 0.41 inches, is the highest in brine insoluble impurities (4.6 percent). The next finer grade, 0.41 to 0.18 inches, contained 2.4 percent insoluble material, and the finer grades all contained about 1 percent insoluble material. However, the insoluble content of the no. 4 grade is higher because of a concentration of impurities in this material.

TABLE 1.—*Grades of rock salt produced by the Independent Salt Company* (Samples supplied in June 1950) (All assays in this and following tables prepared by the Geological Survey geochemistry laboratory under supervision of Russell Runnels)

Grade no.	Size Inches	Mesh	Assay, percent insoluble
3	0.75 -0.411		4.6
2	0.411-0.185		2.4
1	0.185-0.131	4-6	1.1
7 special	0.131-0.093	6-8	0.9
7	0.093-0.078	8-9	1.0
4	less than 0.078	less than 9	1.87

Screen analyses and assay data on the mine sample of no. 4 rock salt used in these tests are given in Table 2. They show that the insoluble content declines slightly as the size decreases—from about 1.7 percent in the plus-10-mesh size to about 1.35 percent in the 48 mesh size. In fractions finer than 48 mesh, the insoluble content increases to a maximum of 8.9 percent in the minus-150-mesh fraction. The calculated head assay for the main sample was 2.19 percent insoluble. A direct head assay on a second bag of salt used in testing was 2.14 percent insoluble.

METHOD OF DETERMINING INSOLUBLE

For the determination of the percentage of material not soluble in brine, 25 grams of sample was placed in a beaker with 200 ml distilled water. The mixture was stirred occasionally during a period

TABLE 2.—*Screen analysis and assay data on no. 4 rock salt*

Size, mesh	Weight distribution, percent	Assay, percent insoluble	Insoluble distribution, percent
+ 10	1.5	1.68	1.1
10-14	16.3	1.40	10.4
14-20	18.3	1.44	12.0
20-28	15.9	1.41	10.2
28-35	12.5	1.35	7.7
35-48	8.5	1.36	5.3
48-65	8.7	1.53	6.1
65-100	5.2	2.13	5.0
100-150	5.4	4.37	10.8
less than 150	7.7	8.88	31.4
Total	100.0	(2.19)	100.0

of several hours until all the salt went into solution. The insoluble material was collected by filtration, using a filter paper that had been heated in the drying oven, cooled, and weighed accurately. The paper and its contents were washed free of salt, dried, cooled, and weighed. The difference in the paper's weight before and after use gave the weight of insoluble.

Possible slight errors inherent to this method and in addition the solubility of small amounts of gypsum that may be present probably explain the discrepancies between calculated assay of feeds in the tests and the assay of the head samples. If the calcium sulfate in the sample is present in the form of gypsum this mineral might be dissolved to the extent of more than 1.8 percent of the total sample. Thus, in the head analyses considerable gypsum probably went into solution and was not reported as insoluble. In some of the tests, on the other hand, the gypsum and anhydrite became segregated so that some of the samples did not contain enough to saturate the water with calcium sulfate; while others contained the remainder of the calcium sulfate minerals so that only a small proportion of the total could dissolve. The calculated feed assays would be higher, therefore, under these circumstances than the head assay.

WASHING TESTS

A large part of the insoluble impurities in the no. 4 rock salt is in the very fine sizes as shown by the screen analysis. Dry screening might be used as a means of concentration, but the improvement in the oversize salt probably would not be worth while. It seems that considerable very fine clay and shale sticks to the coarse salt even when it is thoroughly dried before screening. On the other hand, wetting the salt with a saturated brine loosens this fine-grained coating so that it may be removed. In many of the tests settling and decantation was used to remove this fine material, sometimes as a first step and at other times as a scavengering step at the end of a test. Any scheme adopted for the beneficiation of no. 4 rock salt probably should include means of settling and removing salt from clay-laden liquid.

Tests were made to learn the effect of washing time on clay removal and the effect of the degree of agitation. In one test, 500 grams of salt and 2,000 ml of saturated solution were agitated vio-

Table 3.—*Data from washing tests**

Test no.	Time and treatment	Product	Weight distribution, percent	Assay, percent insoluble	Insoluble distribution, percent
4	Agitated gently for 16 min.	salt	94.2	1.01	55.7
		slime	5.8	13.04	44.3
		total	100.0	(1.71)	100.0
5	Soaked for 16 min.	salt	91.2	1.17	62.0
		slime	8.8	7.55	38.0
		total	100.0	(1.72)	100.0
6	Agitated gently for 64 min.	salt	88.0	0.91	55.2
		slime	12.0	5.42	44.8
		total	100.0	(1.45)	100.0
7	Soaked for 64 min.	salt	92.9	0.91	60.7
		slime	7.1	7.72	39.3
		total	100.0	(1.39)	100.0

* In each test, 200 gm salt and 200 cc solution were in contact, after which the charge was screened at 115 mesh.

lently for 16 minutes in a laboratory Fagergren flotation machine. Then the suspension was decanted and the salt was filtered, dried, weighed, and assayed. The salt amounted to 92 percent of the original sample and contained 1.31 percent insoluble. In a parallel test with 45 minutes of agitation, the salt amounted to 85 percent of the original sample and contained 0.79 percent insoluble. Thus, the longer agitation seems to have reduced both the insoluble content and the quantity of cleaned salt, but it is not certain that the manner of decantation was the same. In another test (no. 3, Table 4) 1,000 grams of salt and 1,000 ml solution were agitated in the Fagergren for 3 minutes, then decanted. A second 1,000 ml of solution was added, agitated for 1 minute, and decanted. The decanted solids contained 58 percent of the total insoluble and left a salt product that assayed 1.18 percent insoluble.

Four parallel tests were made using 200 grams of salt and 200 ml solution in each. In two of the tests the salt and solution were gently agitated for 16 and 64 minutes, respectively. In the others, the salt and solution stood without agitation for the same periods of time. At the end of each test, the material was wet screened at 115 mesh to separate the salt from the slime (clay-size particles). Data on these tests (Table 3) show that the longer period of agitation or soaking did not increase the extraction of insoluble material. The longer agitation increased the amount of salt that passed the screen, and in both cases agitation increased the extraction of clay and insolubles by about 6 percent. The unusually low calculated head assays perhaps can be explained by dissolution of calcium sulfate in the large volume of relatively fresh solution used in the screening.

In test 14 (Table 4) a batch of minus-48-mesh salt was divided into two parts for parallel tests. The clay and insolubles were removed from one part by repeated settling and decantation. The other part was treated by flotation with oleic acid as collector for the insoluble material. In this case, flotation removed about 82 percent of the total insoluble, while decantation removed only about 61 percent. Also, the quantity of salt recovered and the quality was better using flotation.

JIGGING TESTS

When the salt is screened in brine, the up-and-down motion of the screen causes a separation of at least some of the dark particles from the salt. Pieces of shale and calcium sulfate collect next to the screen when the sieve is given the proper motion in the solution. In test 25 (Table 4) about one-third of the insoluble was removed from the plus-48-mesh salt by scraping up the layer that collected on the screen during hand screening. The coarse salt product had only 0.56 percent insoluble and did not contain many coarse shale particles. Perhaps a jig with a moving screen could be developed to treat the coarser part of the no. 4 salt. However, tests on the laboratory jigs available were unsuccessful and indicate that those jigs were not designed for this application.

In test 3, deslimed feed was run in a Denver diaphragm jig to give a hutch product somewhat higher in insoluble than the tailings and bed product, but the same sort of results could have been obtained by screening alone. In tests 10 and 11, plus-48-mesh salt was run in a Hartz jig to produce various tailings and bed products. Although there are some differences in the assay of the products, the salt divided so evenly between the products that no worth-while separations were made. In test 14, the Hartz jig was used with a special compartment that permitted the production of three products above the screen. Even though the greatest care was used in this test and the salt was screened into two fractions before treatment, plus-20-mesh and 20 to 48 mesh, the products all have about the same insoluble content. Data on jig tests are presented in Table 4.

Test 3. Test 3 was conducted using a Denver diaphram jig. The feed (1,000 gm) was first deslimed by two decantations. The hutch and bed products were made by recycling tailings for a total of three passes.

TABLE 4.—*Jig tests*

Product	Weight distribution, percent	Assay, percent insoluble	Insoluble distribution, percent
Test 3			
Decanted clay	14.7	9.57	58.2
Jig tailing	11.3	0.98	4.6
Jig bed product	34.7	0.91	13.1
Jig hutch	39.3	1.48	24.1
Total	100.0	(2.42)	100.0
Test 10			
Jig tailing	43.2	0.51	49.8
Bottom of bed	23.0	0.58	30.3
Top of bed	33.8	0.26	19.9
Total plus 48 mesh	100.0	(0.44)	100.0
Test 11			
Bed 1	32.8	0.17	10.4
Bed 2	39.7	0.48	35.6
Jig tailing	27.5	1.05	54.0
Total plus 48 mesh	100.0	(0.54)	100.0
Test 14			
Plus-20-mesh tailing	3.8	0.52	1.0
Plus-20-mesh intermediate	20.0	1.00	9.6
Plus-20-mesh bed	6.0	1.68	4.8
20 to 48 mesh tailing 1	5.3	0.80	2.1
20 to 48 mesh tailing 2	3.5	0.89	1.5
20 to 48 mesh intermediate	19.7	0.87	8.2
20 to 48 mesh top of bed	7.1	0.68	2.3
20 to 48 mesh bottom of bed	6.7	0.74	2.3
Minus 48 mesh (calculated)	27.9	(5.10)	68.2
Total	100.0	(2.09)	100.0
Cleaning minus 48 mesh by settling			
Settled salt 1	30.7	2.03	13.7
Settled salt 2	31.6	3.70	25.6
Decanted clay	37.7	7.34	60.7
Total minus 48 mesh	100.0	(4.56)	100.0
Cleaning minus 48 mesh by flotation			
Float	14.2	31.84	82.0
Nonfloat	82.1	1.13	16.7
Nonfloat decanted clay	3.7	1.94	1.3
Total minus 48 mesh	100.0	(5.54)	100.0
Test 25			
Jig salt product	68.3	0.56	22.1
Jig reject	3.2	6.62	12.2
Minus-48-mesh salt	22.2	1.61	20.6
Decanted clay	6.3	12.42	45.1
Total	100.0	(1.72)	100.0

Test 10.—Test 10 was made using a Hartz jig set for 200 strokes per minute. The feed consisted of 2,000 gm salt agitated and screened at 48 mesh. The oversize was fed to the jig and from there went to the overflow tailings. The bed product divided into top and bottom layers.

Test 11.—Test 11 also used a Hartz jig but was set for 155 strokes per minute. The feed was the same as in test 10 but for this test the oversize was fed to the jig and the bottom part of the bed product was saved and labeled bed 1. The overflow was then rerun to produce bed 2 and tailings.

Test 14.—For test 14 the Hartz jig was used as in test 11 but with a special screen compartment added for the purpose of an intermediate draw-off. The feed consisted of 6,000 gm salt agitated and screened on 20- and 48-mesh screens. Both the coarse fractions (plus-20 and plus-48 size) were run in the jig to produce tailings, intermediate, and bed products. The minus-48-mesh size feed was divided with one-half being cleaned by decantation and the other half cleaned by flotation.

Test 25.—Test 25 was conducted by hand jigging during screening at 48 mesh. Undersized feed was then decanted to remove slime-size material.

FLOTATION AND AGGLOMERATION TESTS

Froth flotation and agglomeration tabling are related processes in that both usually involve the use of reagents to cause certain particles to adhere to air or oil, while others adhere to water. The laboratory work showed that both these processes could be applied to the cleaning of no. 4 rock salt, flotation to the sizes finer than about 48 mesh and agglomeration to the coarser sizes. Data from these tests are given in Tables 4 (test 14) and 5.

As a preliminary test, a charge of washed salt from a previous test was placed in a flotation machine with brine and some oleic acid. The small amount of froth formed carried fine pieces of shale and calcium sulfate and assayed about 19 percent insoluble. Because the larger pieces of shale tended to skin float but could not be removed as a froth, agglomeration tabling and skin flotation were indicated.

In the next test, a charge of untreated salt was agitated to loosen the clay and then floated with oleic acid. This removed insoluble material from the fine sizes which were then screened out as finished product through a 65-mesh screen. The oversize was placed in a flask and decanted to a thick pulp for conditioning with reagents, because in other agglomeration processes this sort of treatment is necessary. Oleic acid, fuel oil, and caustic were added and the mixture was washed on a vanning plaque where the floatable material was removed. The caustic aids in forming soap to coat the particles; and the oil, either fuel oil or clear mineral oil, sticks to the soap-covered particles to make them more water repellant. The results of this test were that the cleaned salt amounted to 89 percent of the feed, and the rejects carried 88 percent of the insoluble in the feed.

In the next flotation test, test 12, the minus-48-mesh material from 4 kg of salt was floated in two stages using caustic and oleic acid. Nearly 90 percent of the insoluble was removed and most likely another stage of flotation would have removed more of it.

Flotation was compared with settling and decantation for removing clay from the minus-48-mesh part in test 14 (Table 4), as mentioned under washing tests. In this test, the fines were divided and one portion was cleaned by decantation only while the other was given a single stage of flotation. Flotation gave a higher recovery of salt with about half the insoluble content of the settled salt.

Several tests were made in which stage flotation removed the clay after which screening at 48 or 65 mesh removed the fine clean salt. This fine salt usually contained some unfloated clay that was decanted as a separate product. The oversize was conditioned in a thick pulp with reagents and washed on a separator made from a laundry wash board. Most of the salt settled in the riffles on the board, and the floatable particles were carried over the board by the flowing solution. The two products so produced were rerun and cleaned on a vanning plaque in various combinations, as shown in Table 5, to give cleaned salt and flotation rejects. The best of these tests, as to quantity and quality of salt, test 13, yielded 83.2 percent clean salt having a calculated assay of only 0.17 percent insoluble.

In the course of the work it was found that calcium ions can prevent the proper use of the reagents under certain conditions. This trouble can be prevented by precipitation or removal of the calcium ions prior to conditioning for agglomeration tabling. Tests 15, 16, and 17, which gave rather poor results, seemed to indicate that something in reused brine interfered with the action of the reagents in conditioning for agglomeration. It seems that the trouble

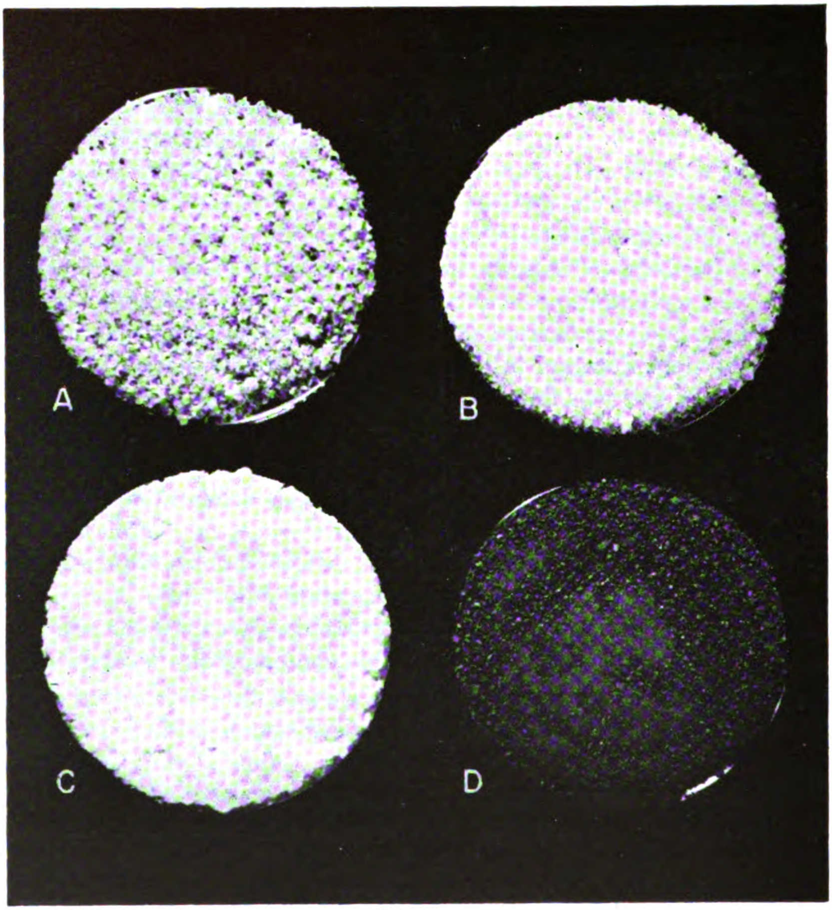

Plate 1. Photograph of samples showing untreated no. 4 rock salt, products produced by agglomeration tabling, and a salt product and reject separated by grinding and flotation. *A*, Untreated no. 4 rock salt; *B*, salt product made by agglomeration tabling in test 23; *C*, salt product made by grinding and flotation in test 28; *D*, first flotation reject from test 28. (Photo by Ada Swineford.)

was from excess calcium ions which precipitated the oleic acid as insoluble soap, or from fine calcium sulfate that used up both oleic acid and mineral oil. This may be overcome by using froth flotation to remove fine gypsum and anhydrite and precipitate soluble calcium before agglomeration, as shown in tests 20 and 21, or by screening out the fine sizes and using a reagent to control the calcium ion, as shown in test 24. Four parallel washboard tests, 18 to

21, were made to check on the effect of calcium-controling reagents and on the effect of the age of the solution. The results of all these tests were good and quite similar although there are minor variations. Calgon (sodium hexametaphosphate) is effective in forming complex soluble ions of calcium and thus is useful in flotation as well as in water treatment. In test 18, with calgon and fresh solution, the quantity of material removed by froth flotation was smaller than in the other tests, but the percentage of insoluble in the froth was much higher. However, the calgon dispersed the clay so that part of it did not float. This resulted in more clay to be decanted, and the over-all loss of salt in the fine sizes was about the same as in the other tests. The use of sodium carbonate in test 19 caused no striking difference from tests 20 and 21 which contained no sodium carbonate. It may have helped give a higher grade salt product, but the difference was slight. Tests 20 and 21 with fresh and used solution, respectively, did not show significant differences.

After conditions had been worked out for satisfactory separations using the washboard separator, tests were made with 5 kg charges of salt using the laboratory Wilfley table to treat the plus-48-mesh fraction. In the first one, test 22, the salt was agitated in a Denver flotation machine and then deslimed by allowing brine to

TABLE 5.—*Flotation and agglomeration tests*

Order of treatment operations	Order of materials additions	Weight or volume*	pH	Product**	Weight distribution, percent	Assay, percent insoluble	Distribution, percent insoluble
		Test 8					
Washed and jigged in test 3							
	Salt from test 3	500					
	Oleic acid	—	—	Float	4.0	18.71	
Floated							
		Test 9					
	No. 4 salt	1000		A. Froth	3.8	37.16	59.8
	Saturated brine	1000					
Agitate 15 min.				B. Minus-65-mesh salt	25.6	0.47	5.1
	Oleic acid	0.132		C. Skin float	7.3	9.18	28.4
Agitate several seconds							
A. Remove froth				C. Plus-65-mesh salt	63.3	0.25	6.7
B. Screen sink at 65 mesh				Total	100.0	(2.36)	100.0
Drain plus-65-mesh to thick pulp and add reagents	Oleic acid	0.44					
	Fuel oil	0.9					
	Sodium hydroxide	0.8					
	Oleic acid	0.44					
C. Shake mixture and separate by skin flotation on a vanning plaque							

TABLE 5.—*Flotation and agglomeration tests—Continued*

Order of treatment operations	Order of materials additions	Weight or volume*	pH	Product**	Weight distribution, percent	Assay, percent insoluble	Distribution, percent insoluble
				Test 12			
Screen at 48 mesh	Salt in tests 10 and 11	4000		A. Float	9.7	28.45	52.4
Agitate minus-48-mesh fraction with brine			7.4	B. Float	5.4	35.34	36.3
				C. Decanted fines	5.6	1.84	2.1
	Sodium hydroxide	0.25†	9.5	C. Salt	79.3	0.61	9.2
	Oleic acid	0.044		Total minus 48 mesh	100.0	(5.27)	100.0
Agitate 5 min.			9.2				
A. Remove froth 4 min.			8.7				
	Sodium hydroxide	0.5	10.1				
	Oleic acid	0.033					
B. Remove froth 2 min.							
C. Decant fines from B sink							
				Test 13			
	No. 4 salt	1000		A. Float	5.1	34.36	64.8
	Saturated brine	1000		B. Float	4.7	4.88	8.5
Agitate 15 min.			7.5	D. Minus-65-mesh salt	20.1	0.22	1.6
	Sodium hydroxide	1.8	9.7				
	Oleic acid	0.132		D. Decanted fines	0.9	4.65	1.6
A. Remove froth				F. Coarse salt sink	49.3	0.16	2.9
	Oleic acid	0.44		G. Float	3.3	11.79	14.4
B. Remove froth			8.7	G. Cleaned salt	8.0	0.15	0.4
C. Screen B sink at 65 mesh				H. Float	2.8	5.18	5.4
D. Decant fines from minus 65 mesh				H. Cleaned salt	5.8	0.17	0.4
Drain C plus 65 mesh to thick pulp and add reagents	Sodium hydroxide	0.4		Total	100.0	(2.70)	100.0
	Oleic acid	0.22					
	Mineral oil	0.44		**Overall results**			
	Sodium hydroxide	0.4		Flotation rejects	15.9	(15.8)	93.1
	Oleic acid	0.22		Cleaned salt	83.2	(0.17)	5.3
E. Shake mixture and wash onto riffled board for skin flotation				Decanted fines	0.9	4.65	1.6
				Total	100.0	(2.70)	100.0
F. To E sink rerun twice on board to remove more float							
G. To E float, clean on vanning plaque to separate float from salt.							
H. To F float, clean on vanning plaque to separate float from salt							
				Test 15			
	No. 4 salt	1000		C. Plus 150 mesh	0.6	14.30	3.0
	Saturated brine	1000		C. Minus 150 mesh	57	32.04	60.3
Agitate 15 min.			7.2	D. Plus 150 mesh	0.4	12.45	1.6
	Sodium hydroxide	4.0	9.9	D. Minus 150 mesh	0.8	30.75	7.7
	Oleic acid	0.44		E. Plus 14 mesh	6.9	0.92	2.1
A. Remove froth			9.5	E. 14-20 mesh	19.4	1.12	7.2
	Oleic acid	0.176		E. 20-28 mesh	18.7	0.96	5.9
B. Remove froth			9.1	E. 28-35 mesh	11.3	0.83	3.1
C. To A float, screen at 150 mesh				E. 35-48 mesh	7.2	0.61	1.5

D. To B float, screen at 150 mesh				E. 48-65 mesh	5.6	0.62	1.2
E. To B sink, screen from 150 to 14 mesh				E. 65-100 mesh	3.6	0.52	0.6
				E. 100-150 mesh	2.7	0.86	0.8
				E. Minus 150 mesh	17.1	0.89	5.0
				Total	100.0	(3.02)	100.0

Test 16

Washed, screened, and jigged in test 14				A. Float	35.6	1.08	42.8
	20-48 mesh intermediate from test 14	615		A. Sink	64.4	0.80	57.2
				Total	100.0	(0.90)	100.0
Prepare as a thick pulp and add reagents	Sodium hydroxide	1.63 (5 cc, 10%)					
	Oleic acid	0.36 (5 drops)					
	Mineral oil	0.72 (10 drops)					
	Oleic acid	0.21 (3 drops)					
A. Shake well and wash onto riffled board							

Test 17

Washed, screened, and jigged in test 14				A. Float	27.1	1.66	50.7
				B. Sink	72.9	0.60	49.3
	20-48 mesh intermediate from test 14	436		Total	100.0	(0.89)	100.0
Prepare as a thick pulp and add reagents	Sodium carbonate	2.28 (5 cc, 10%)					
	Sodium hydroxide	1.83 (4 cc, 10%)					
	Oleic acid	0.50 (5 drops)					
	Mineral oil	1.0 (10 drops)					
	Oleic acid	0.3 (3 drops)					
A. Shake well and wash onto riffled board							

Test 18

	No. 4 salt	1000		A. Float	1.9	52.13	35.0
	Fresh saturated brine	1000		B. Float	2.7	17.80	17.6
	(Fresh solution used throughout)			C. Float	2.5	8.43	7.5
				D. 48-65 mesh	5.6	0.37	0.7
Agitate 10 min. Dilute for flotation			7.0	D. 65-100 mesh	3.6	0.20	0.2
	Calgon	0.5		D. 100-200 mesh	5.7	0.24	0.5
	Sodium hydroxide	2.0	9.6	E. Minus 200 salt	8.9	0.22	0.7
	Oleic acid	0.22		E. Decanted fines	7.7	2.79	7.8
A. Remove froth 1 min.				F. Coarse salt sink	35.6	0.16	2.1
	Oleic acid	0.22		G. Coarse salt sink	19.7	0.31	2.2
B. Remove froth 1 min.			9.4	G. Float	6.1	11.56	25.7
	Oleic acid	0.22		Total	100.0	(2.77)	100.0
C. Remove froth 1 min.			9.4				
D. To C sink, wet screen at 48, 65, 100, and 200 mesh							

TABLE 5.—*Flotation and agglomeration tests—Continued*

Order of treatment operations	Order of materials additions	Weight or volume*	pH	Product**	Weight distribution, percent	Assay, percent insoluble	Distribution, percent insoluble
				Overall results			
E. Decant fines from C minus 200				Flotation and agglomeration rejects	13.2	(18.00)	85.7
Drain D plus 48 mesh to thick pulp and add reagents	Calgon	0.1					
	Sodium hydroxide	0.8		Combined salt products	79.1	(0.23)	6.5
	Oleic acid	0.22		Decanted fines	7.7	2.79	7.8
	Mineral oil	0.44					
	Oleic acid	0.22		Total	100.0	(2.77)	100.0
F. Shake well and wash onto riffled board for skin flotation. Sink was collected and washed onto the board again. Then the sink was cleaned on a vanning plaque							
G. Combined floats from F cleaned twice on vanning plaque							
				Test 19			
	No. 4 salt	1000		A. Float	5.2	28.77	56.8
	Fresh saturated brine	1000		B. Float	3.7	3.80	5.3
	(Fresh solution used throughout)			C. Float	3.5	1.91	2.6
				D. 48-65 mesh	5.3	0.24	0.5
Agitate 10 min. Dilute for flotation			7.2	D. 65-100 mesh	3.1	0.21	0.3
	Sodium carbonate	1.0	8.0	D. 100-200 mesh	5.3	0.19	0.4
	Sodium hydroxide	1.2	9.5	E. Minus 200 salt	9.3	0.18	0.6
	Oleic acid	0.22		E. Decanted fines	2.1	2.14	1.7
A. Remove froth 3 min.			9.2	F. Coarse salt sink	26.2	0.19	1.9
B, C, D, and E, same operations and reagents as in test 18			8.3 at end of	G. Coarse salt sink	29.0	0.19	2.1
				G. Float	7.3	10.00	27.8
				Total	100.0	(2.62)	100.0
Drain D plus 48 mesh to thick pulp	Sodium carbonate	0.2					
	Sodium hydroxide	0.8					
	Oleic acid	0.22		Overall results			
	Mineral oil	0.44					
	Oleic acid	0.22					
Test completed as in test 18				Flotation and agglomeration rejects	19.6	(12.34)	92.5
				Combined salt products	78.3	(0.19)	5.8
				Decanted fines	2.1	2.14	1.7
				Total	100.0	(2.62)	100.0
				Test 20			
	No. 4 salt	1000		A. Float	3.8	38.61	52.3
	Fresh saturated brine	1000		B. Float	3.7	4.34	5.6
	(Fresh solution used throughout)			C. Float	3.4	2.77	3.3
				D. 48-65 mesh	6.9	0.40	1.0
Agitate 10 min. Dilute for flotation			7.4	D. 65-100 mesh	3.7	0.36	0.5
	Sodium hydroxide	1.0	9.5	D. 100-200 mesh	5.5	0.35	0.7
	Oleic acid	0.22		E. Minus 200 salt	8.3	0.35	1.0
A. Remove froth 3 min.				E. Decanted fines	1.3	4.05	1.8

B, C, D, and E, same operations and reagents as in test 18				F. Coarse salt sink	37.9	0.33	4.4
				G. Coarse salt sink	18.3	0.35	2.2
Drain D plus 48 mesh to thick pulp and add reagents	Sodium hydroxide	0.8		G. Float	7.2	10.79	27.2
	Oleic acid	0.22		Total	100.0	(2.84)	100.0
	Mineral oil	0.44					
	Oleic acid	0.22		Overall results			
Test completed as in test 18				Flotation and agglomeration rejects	18.1	(13.91)	88.4
				Combined salt products	80.6	(0.34)	9.8
				Decanted fines	1.3	4.05	1.8
				Total	100.0	(2.84)	100.0

Test 21

	No. 4 salt	1000		A. Float	4.4	35.84	56.8
	Old saturated brine	1000		B. Float	3.1	4.11	4.5
	(Old solution used throughout)			C. Float	2.5	2.20	2.0
				D. 48-65 mesh	6.4	0.34	0.8
Agitate 10 min. Dilute for flotation.			7.95	D. 65-100 mesh	3.4	0.29	0.4
				D. 100-200 mesh	5.2	0.29	0.5
	Sodium hydroxide	0.5	9.5	E. Minus 200 salt	10.3	0.39	1.4
	Oleic acid	0.22		E. Decanted fines	0.5	4.10	0.8
A. Remove froth				F. Coarse salt sink	35.6	0.27	3.4
B, C, D, E, F, and G, same operations and reagents as in test 20			8.2 at end of C	G. Coarse salt sink	21.3	0.33	2.5
				G. Float	7.3	10.20	26.9
				Total	100.0	(2.78)	100.0
				Overall results			
				Flotation and agglomeration rejects	17.3	(14.49)	90.1
				Combined salt products	82.2	(0.31)	9.1
				Decanted fines	0.5	4.10	0.8
				Total	100.0	(2.78)	100.0

Test 22

	No. 4 salt	5000
	Saturated brine	—
Agitate 10 min.		
A. Classify at about 100 mesh by causing cell to overflow		
Drain coarse salt to thick pulp and add reagents	Sodium hydroxide	—
	Oleic acid	—
	Mineral oil	—
	Sodium carbonate	—
	Calgon	—
B. Stir well and try to separate by tabling. No separation resulted. Test abandoned.		

Test 23

	No. 4 salt	5000		A. Float	7.8	26.81	70.9
	Saturated brine	—		B. Minus-48-mesh salt	29.6	0.20	2.0
Agitate 20 min.			7.4	C. Float	5.4	9.66	17.9
	Sodium hydroxide	1.6	9.5	D. Float	2.2	3.91	2.9

TABLE 5.—*Flotation and agglomeration tests—Concluded*

Order of treatment operations	Order of materials additions	Weight or volume*	pH	Product**	Weight distribution, percent	Assay, percent insoluble	Distribution, percent insoluble
	Oleic acid	0.22		D. Salt concentration	35.8	0.31	3.8
A. Remove froth				D. Salt middling	19.2	0.39	2.5
	Oleic acid Pine oil	0.22 0.044		Total	100.0	(2.94)	100.0
Remove froth				Overall results			
	Oleic acid	0.22					
Condition several minutes, then remove froth			8.2	Flotation and agglomeration rejects	15.4	(17.5)	91.7
B. Screen A sink at 48 mesh				Combined salt products	84.6	0.29	8.3
Drain B plus 48 mesh to thick pulp and add reagents	Sodium hydroxide Oleic acid Mineral oil Oleic acid	0.8 0.22 0.44 0.22		Total	100.0	2.94	100.0
C. Stir well and feed to Wilfley table operating with brine							
D. Rerun C sink on table to give concentrate, middling, and float products							
		Test 24					
	No. 4 salt	5000		B. Salt concentrate	24.6	0.28	2.8
	Saturated brine	—		B. Salt middling	25.0	0.30	3.0
Agitate 10 min.				C. Salt concentrate	12.5	0.43	2.2
A. Screen at 48 mesh				C. Salt middling	1.5	0.96	0.6
Drain A plus 48 mesh to thick pulp and add reagents with stirring	Sodium carbonate	2.0		C. Float	4.6	14.08	26.2
	Sodium hydroxide	0.8		D. Decanted fines	1.9	34.07	26.6
	Oleic acid	0.22		E. Float	1.8	38.12	28.0
	Mineral oil	0.44		F. Float	0.4	26.62	4.4
	Oleic acid	0.22		G. Float	1.6	4.64	3.0
B. Stir well and feed to Wilfley table to produce concentrate, middling, and float-containing products				H. Float	1.3	0.95	0.5
				I. Float	0.6	1.31	0.3
				I. Minus-48-mesh salt	24.2	0.24	2.4
C. The float-containing product was rerun three times to give concentrate, middling, and float products				Total	100.0	(2.46)	100.0
				Overall results			
D. Decant A minus 48 mesh several times to remove fines				Flotation and agglomeration tabling rejects	10.3	(14.95)	62.4
Place D settled salt in flotation cell and add reagents			8.0				
	Sodium carbonate	0.2					
	Sodium hydroxide	0.14	9.45	Combined salt products	87.8	(0.31)	11.0
	Oleic acid	0.044		Decanted fines	1.9	34.07	26.6
E. Remove froth				Total	100.0	(2.46)	100.0
	Oleic acid	0.044					
F. Remove froth							
	Pine oil Oleic acid	0.0088 0.044					
G. Remove froth							
	Oleic acid	0.044					
H. Remove froth							
	Pine oil Oleic acid	0.0088 0.044					
I. Remove froth			7.5				

Test 26

	No. 4 salt	1000		A. Decanted fines	14.0	6.77	6.5
	Fresh saturated brine	1000		B. Float	5.4	19.77	73.9
Agitate 10 min.				C. Float	10.0	1.43	9.9
A. Decant three times to remove fines				D. Float	6.2	0.69	3.0
Grind settled salt 20 min. in pebble mill				D. Salt concentrate	64.4	0.15	6.7
				Total	100.0	(1.44)	100.0
Place in flotation cell and add reagents			7.15				
	Sodium hydroxide	1.0	9.5				
	Oleic acid	0.36					
B. Remove froth							
	Oleic acid	0.36					
C. Remove froth							
	Oleic acid	0.36					
D. Remove froth			8.35				

Test 27

	No. 4 salt	1000		A. Float	4.9	45.68	67.4
	Saturated brine	500		B. Float	6.8	8.36	17.0
Grind in pebble mill 8 min. and screen out minus 48 mesh. Repeat, grinding oversize for 6 min. Repeat, grinding 5 min. Repeat, grinding 4 min. (All passed 48 mesh)				C. Float	5.1	5.92	9.1
				D. Float	7.1	0.92	1.9
				E. Float	6.1	0.59	1.1
				F. Float	2.5	1.15	0.87
				F. Salt concentrate	67.5	0.13	2.6
				Total	100.0	(3.34)	100.0
Place ground salt in flotation cell and add reagents			7.5				
	Sodium hydroxide	0.6	9.0				
	Oleic acid	0.18					
A. Remove froth							
	Oleic acid	0.18					
B. Remove froth							
	Oleic acid	0.09					
C. Remove froth							
	Oleic acid	0.09					
D. Remove froth							
	Oleic acid	0.18					
E. Remove froth			7.3				
	Dodecylamine acetate	0.2					
F. Remove froth							

Test 28

	No. 4 salt	1000		A. Float	4.5	23.94	59.3
	Saturated brine	500		B. Float	4.8	6.25	16.3
Grind as in test 27				C. Float	7.6	1.32	5.4
Place ground salt in flotation cell and add reagents			7.9	C. Salt concentrate	83.1	0.42	19 0
	Dodecylamine acetate	0.2		Total	100.0	(1.84)	100.0
	Pine oil	0.05					
A. Remove froth							
	Dodecylamine acetate	0.2					
	Pine oil	0.1					
B. Remove froth							
	Dodecylamine acetate	0.2					
C. Remove froth			7.9				

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* Salt in grams, solution in cc, reagents in pounds per ton of feed.

** Assays and calculated values for products obtained from various operations listed in left-hand column.

† Reagent weights based on 4 kg. of feed.

enter through the air inlet and overflow the cell while it was running. This separation was at a size somewhat finer than 65 mesh. The oversize was placed in a pail and mixed with the same proportions of caustic, oleic acid, and mineral oil as had been used before. When no flotation took place, soda ash and calgon were added; but conditions were not improved and the test was abandoned. The stick used in stirring became coated with a butterlike deposit of calcium sulfate, calcium soap, and mineral oil all stuck together. In another test the same effect was obtained when plus-48-mesh salt was treated without a calcium-controling agent. The addition of sodium carbonate as in test 24 or the precipitation of the calcium ion by oleic acid during froth flotation as in test 23 seems to be necessary for satisfactory agglomeration tabling.

In test 23 the salt was agitated in the Denver cell and then given a three-stage float as in the washboard tests. The minus-48-mesh salt was then screened out and a little clay decanted from it and added to that flotation product. The plus-48-mesh fraction was conditioned with caustic, oleic acid, and mineral oil. It was then fed to the Wilfley table which was operated with brine in place of water. The over-all results of this test were a salt yield of 84.6 percent containing 0.29 percent insoluble material and a rejection of 91.7 percent of the insoluble in the feed.

Test 24 was on a sample of no. 4 salt produced some months after the sample used in the other tests. No difference in behavior was noted. In this test the 5 kg of salt was agitated in brine and then screened at 48 mesh. The minus-48-mesh fraction was decanted to remove part of the clay and then treated in the Fagergren machine by stage flotation to remove the remaining insoluble material. The plus-48-mesh material was conditioned using a generous amount of soda ash as well as the other reagents. The conditioned salt was run on the table several times to produce clean salt products and a float containing the calcium sulfate and shale. The over-all results of this test were a salt yield of 87.8 percent containing 0.31 percent insoluble and a rejection of 89 percent of the insoluble in the feed.

In the tests so far described an effort was made to separate the impurities from the salt without reducing the size of the particles. This required the use of agglomeration tabling for the sizes too coarse for flotation. Some tests were made in which the salt was ground until it was finer than 48 mesh followed by flotation. In tests 26 and 27 the salt was ground and floated using caustic and oleic acid. The quality of the cleaned salt from these tests was better than from any other test, but the yield was relatively low, being about 67 percent.

In test 28 a cationic collector, dodecylamine acetate, was used in the flotation step. This reagent seems to be more selective than the fatty acid because the cleaned salt amounted to 83 percent of the total. It contained 0.42 percent of material insoluble in brine, but the true water insoluble was only 0.103 percent. This indicates that the amine did not collect the calcium sulfate so completely as the fatty acid, but that it did collect the shale and clay particles.

BRIQUETTING AND BRINING TESTS

One of the difficulties with no. 4 salt is that it packs in bins or containers and fails to flow in the manner of the coarser grades. Another trouble is reported to be that it fails to go into solution when placed in a tank with water. Presumably the clay accumulates on top of the salt layer and hinders contact between salt and water. These difficulties might be overcome if the salt were made into lumps strong enough to withstand handling and contact with water. Some trials were made along this line.

A lump of the dried minus-48-mesh salt from test 12 was placed in water. After about 4 hours most of the lump was in solution, but the part of it that remained was strong enough to withstand rapid stirring without breaking.

Briquettes were made by filtering the cleaned salt as dry as possible and then compacting it in a cupel mold. Fine salt and mixtures of fine and coarse salt were easily formed into briquettes which, when dry, were quite strong. These briquettes would dissolve rapidly and without disintegration, but briquettes made from coarse salt alone were weak when dry and disintegrated on contact with water.

To compare the brining characteristics of various samples, parallel tests were made in which 150 grams of salt and 300 ml of water were placed in a beaker. At intervals the solution was stirred gently so as not to disturb more than the surface layer of salt. Twice part of the brine was removed and replaced by water. After standing over night, all of one sample was in solution. Then the salt remaining in each beaker was filtered, dried, and weighed to determine the amount dissolved. The results showed that the following weights of salt dissolved under similar conditions: briquetted cleaned salt, 149 grams; loose cleaned salt, 131 grams; no. 2 rock salt, 128 grams; and uncleaned no. 4 salt, 103 grams. This might mean that removing most of the insoluble material from the no. 4 salt would overcome the present difficulties of putting it into solution.

ECONOMIC CONSIDERATIONS

The laboratory tests have shown that the quality of no. 4 rock salt may be improved by cleaning operations and that the degree of improvement depends on the treatment method. Washing with saturated brine will remove up to half the insoluble impurities while a combination of washing, flotation, and agglomeration tabling will remove about 90 percent of the insoluble material. The selection of a treatment process will depend upon the quality of finished product desired, the improvement in value of that product, the first cost of the treatment plant, and the operating cost. In general, a higher quality product will cost more to produce than one of lower quality.

A product produced by the cleaning of no. 4 rock salt might be used for domestic water softeners, meat-curing brines, food pickling brines, and for certain purposes where the insoluble matter would not remain in the product, as well as many of the purposes

TABLE 6.—*Chemical analyses of selected salt products from tests* (Expressed as percentage of total sample by weight) (Analyses by geochemistry laboratory under supervision of Russell Runnels)

	Test 23, flotation and agglomeration tabling		Test 25, decantation and hand jigging			
Element	Minus 48 mesh	Plus 48 mesh	Minus 48 mesh	Plus 48 mesh	Test 27 cleaned salt	Test 28 cleaned salt
Water insoluble	0.13	0.09	0.30	0.22	0.074	0.103
Calcium oxide (CaO)	0.13	0.25	1.83	0.53		
Magnesium oxide (MgO)	0.010	0.015	0.010	0.005		
Sulfur trioxide (SO_3)	0.23	0.13	2.49	0.79		
Water soluble as Fe_2O_3	0.00004	0.00042	0.00006	0.00011		
Remainder as NaCl	99.50	99.51	95.37	98.46		
Calculated constituents:						
$CaSO_4$	0.32	0.22	4.23	1.29		
$CaCl_2$		0.27	0.18			
$MgCl_2$		0.04	0.02			
$MgSO_4$	0.03	.		0.03		
Na_2SO_4	0.03			0.02		
NaCl (by difference)	99.49	99.38	95.27	98.44		

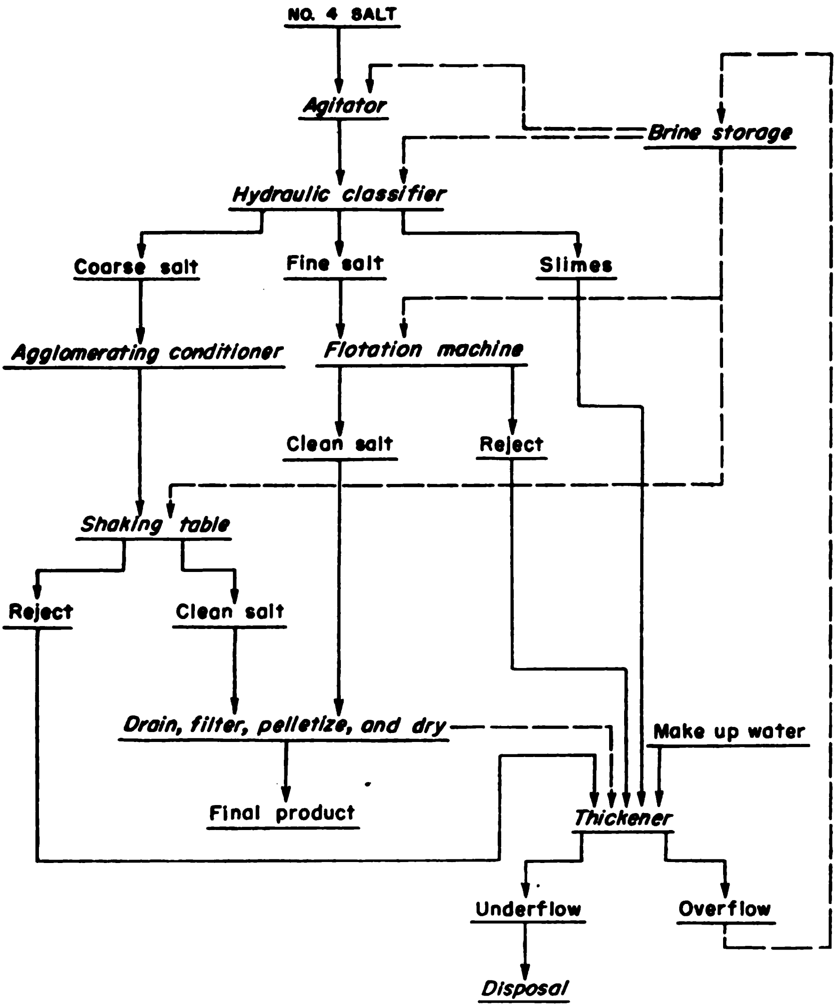

Fig. 1.—Proposed flow sheet for cleaning no. 4 salt by using flotation and agglomeration tabling.

served by the coarser grades of rock salt. For use in water softeners the low insoluble content would be attractive, but for maximum usability it should be made into lump form. This would probably not be difficult to do using pitted rolls to form the lumps and a grate drier to harden them. Such a product should be better for most purposes than the coarse rock salt because of less impurities. The salt cleaned by flotation or agglomeration tabling meets the chemical specifications for brining use in the food industries as shown by data in Table 6, and probably would be approved for such use by the authorities. Brine made from salt cleaned by agglomeration tabling had a pH of 7.0, indicating that it did not contain any of the caustic reagent. A sample of the same salt gave a negative test for organic matter when charred with sulfuric acid, thus indicating that it retained none of the oleic acid or oil reagents.

Two proposed flow sheets are shown; these follow the general procedures used in laboratory testing. Figure 1 is a flow sheet for cleaning of the salt without grinding. The process consists of agitating the no. 4 salt with brine to loosen the clay and shale coatings followed by hydraulic classification to separate it into coarse and fine sizes and to remove the very fine waste material. The coarse salt is then treated by agglomeration tabling to separate clean salt from waste material. Flotation of the fine salt cleans it likewise. Other parts of the diagram show the flow of brine and the thickening of the rejects to recover brine for reuse. The cleaned salt products could be combined, filtered, and dried as in standard salt-plant practice or manufactured into pellets or lumps for special purposes.

Figure 2 is a flow sheet for grinding the salt in closed circuit with a classifier followed by removal of the slimes with a hydroseparator and flotation of the coarser impurities. The product from this process would probably have to be formed into lumps for market. However, the very fine salt should be easy to compact and dry into hard pieces.

The cost of a plant to treat 200 tons per day of no. 4 salt by either process is estimated to be about $50,000 to $75,000. The cost of reagents in the fatty acid flotation and agglomeration tests ranged from 24 to 44 cents per ton, and for the amine flotation test the cost was 55 cents per ton of feed salt.

A plant intended to treat the salt by agitation and desliming only would have a lower first cost and would cost less to operate. However the washed salt would contain more than 1 percent insoluble material and might not pass the Federal inspection agencies for certain purposes. If it were made into lump form, it should be better than the coarse rock salt grades for many purposes. Such a plant might cost as little as $20,000. Very possibly washing of the salt or jig-screening would prove to have optimum economics as a treatment method.

SUMMARY AND CONCLUSIONS

The tests show that the insoluble material in the no. 4 rock salt consists predominantly of anhydrite and some gypsum with shale and clay. These impurities are distributed throughout the entire size range, but there is a concentration of these impurities in the fine sizes. Most of the insoluble material can be removed from the salt by the treatment processes described, the quantity removed depending on the process used.

Rock salt of this grade that has a gray color can be made whiter and about half of the insoluble substances can be removed by washing the salt with saturated solution. This could be a simple and in-

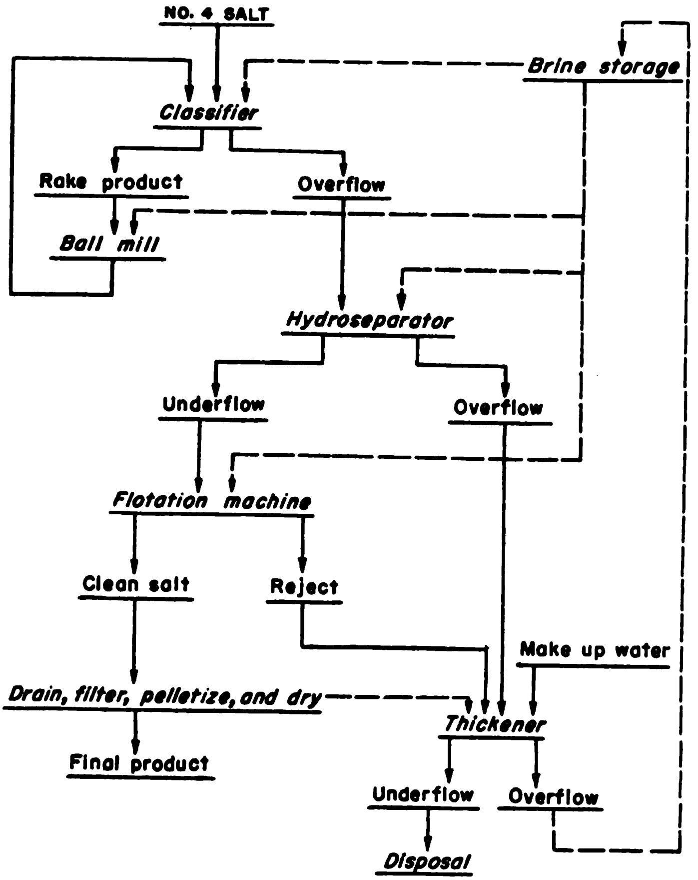

Fig. 2.—Proposed flow sheet for cleaning no. 4 salt by grinding and flotation.

expensive procedure that would yield a white salt containing more than 1 percent insoluble material as fragments of shale and gypsum.

Although tests with the jigs that were available were unsuccessful, there is reason to believe that a jig could be made to clean the coarser fractions of the salt. Such a jig might screen out the fines at the same time, and they could be treated by washing or flotation. This procedure, if worked out, should produce a salt product with a lower insoluble content and fewer black pieces of shale.

The tests showing the best results for the removal of insoluble materials were those using flotation and agglomeration with oleic acid, mineral oil, soda ash, and caustic as reagents. These processes are more complicated although still relatively simple and inexpensive, and they consistently give salt products carrying less than 0.4 percent insoluble material.