THE REFINING OF CRUDE SALTPETRE.

By B. Ganapathi Rao, J. J. Sudborough and H. E. Watson.

A. INTRODUCTION.

The extent and value of the saltpetre industry in India may be gathered from the figures in Table I which gives the quantities of refined saltpetre produced, and also the quantity of saltpetre exported within recent years and the value of the exports.

TABLE I.

Year	Tons of refined saltpetre produced in Bihar, U.P.. and Punjab	Tons exported	Value of exports in lakhs of rupees
1894-95	...	24,400	...
1902-03	...	18,400	...
1907-08	...	18,400	...
1913-14	14,740	13,400	30·8
1914-15	17,223	16,400	42·8
1915-16	20,284	20,700	68 9
1916-17	26,253	26,400	105·5
1917-18	21,511	23,400	92·2
*1918-19	24,500	17,200	55·4
1919-20	17,300	22,100	75·3
1920-21	14,750	12,900	47·9

In 1859 the exports were 35,000 tons, but in 1860 the artificial production of potassium nitrate (saltpetre) from Chilean sodium nitrate and potassium chloride from Stassfurt was started, and from then onwards the exports diminished, until in 1913-14 the minimum of 13,400 tons was reached.

The number of licences granted for preparing crude saltpetre was 31,191 in 1913–14 and 51,830 in 1917–18 and the number of refineries 327 in 1913–14 and 453 in 1917–18.[1]

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* During the years 1918-21 about 700 to 1,000 tons were produced each year in other parts of India.

[1] In Bihar and Orissa the number of refineries in 1921–22 was 219 and in the United Provinces 110.

It is clear that the war stimulated the production, refining and exportation of saltpetre, but that there is now a tendency for the production and exportation to fall.

The chief centres of production in India are the United Provinces, Bihar and the Punjab, but small amounts are also produced in Cashmere, Patiala, Sind, Bengal, Ahmedabad and in the Madras Presidency.

Table II shows the production for the different districts for the years 1917–20.

TABLE II.

Production of saltpetre in cwts.

—	1917-18	1918-19	1919-20
Bihar	125,986	100,888	76,610
Bengal	8,597	5,173	7,044
U. P.	224,472	132,324	87,594
Punjab	138,934	113,236	130,952
Madras	6,857	4,430	3,501
C. P.	524	952	876
Rajputana	4,900	3,600	4,342

These figures indicate a considerable fall in the production of saltpetre in Bihar and the United Provinces since 1917, whereas in the Punjab the production has diminished to a much less extent.

The whole of the crude nitre is obtained by extracting the nitrous earth collected from old, or existing village sites in different parts of India, and detailed accounts of this method of manufacture are given in the following papers :—

1. D. Hooper, *Agricultural Ledger*, 1905, **12**, 17.

2. J. W. Leather and J. N. Mukerji, *Agri. Res. Inst., Pusa, Bulletin* No. 24 (1911).

3. C. M. Hutchinson, *ibid.*, No. 68 (1916).

Hooper (pp. 23–25) gives analyses of 72 samples of nitrous earths and the potassium nitrate content of these varies from 1·17 to 29·6 per cent., but, according to Leather and Mukerji, most of the earths worked do not contain more than 3-5 per cent. Hooper also gives the analyses of 55 samples of crude saltpetre obtained from different districts. The percentage of potassium nitrate in these samples ranged from 26·9 to 79·7 per cent., but the usual value is 45 to 55 per cent. The other constituents determined were :—moisture, insoluble, sodium chloride, sodium sulphate, calcium nitrate and magnesium nitrate—and the amounts of these are as given in Table III.

TABLE III.

Constituents other than potassium nitrate in crude saltpetre.

Moisture	2·1 to 15·2	per cent.
Insoluble	0·5 to 20·8	,,
Sodium chloride	6·0 to 44·7	,,
Sodium sulphate	1·1 to 30·9	,,
Calcium nitrate	traces to 12·3	,,
Magnesium nitrate	traces to 12·2	,,

and Table IV gives complete analyses of six typical samples.

TABLE IV[1].

—	Farukabad		Okera		Mozaffer-pore	Burhanpure
	I	III	I	III		
Potassium nitrate ...	66·07	44·92	53·00	26·86	49·36	68·40 per cent.
Calcium nitrate ...	...	...	2·60	...	3·28	2·60 ,,
Magnesium nitrate ...	2·54	4·80	...	12·24	7·44	2·12 ,,
Sodium chloride ...	21·84	35·38	34·22	34·80	16·82	17·98 ,,
Sodium sulphate ...	3·65	10·00	3·88	11·20	14·60	3·40 ,,
Insoluble matter ...	0·90	1·20	1·10	1·40	1·50	1·70 ,,
Moisture	5·00	3·70	5·20	13·50	7·00	3·80 ,,

In the United Provinces the largest refineries produce from 6,000 to 7,000 maunds of refined nitre per year and the average output is 1,200 to 2,000 maunds, whereas in Bihar and Orissa a large factory produces 3,000 maunds and an average factory 433 maunds.

The methods adopted for the production of the crude material necessitate stoppage during the rainy seasons, so that the refiner can purchase raw material during part of the year only. This means that either the refinery is not run continuously during the whole year,

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[1] Leather and Mukerji, p. 7. Hooper, p. 23.

or the refiner must purchase his year's supplies during the dry season and hold comparatively large stocks of crude nitre during a portion of the year. As the crude nitre is deliquescent this may entail serious losses during the monsoon.

Hooper[1] gives a description of the refineries of Northern India as worked in 1905, and a further account is given by Leather and Mukerji.

In Northern India the method consists in adding the crude nitre to the requisite amount of the boiling mother liquor from a previous operation, heating for half an hour, allowing the insoluble matter and undissolved sodium chloride and sodium sulphate to settle and decanting the dark coloured liquid into wooden crystallising vats. The residue left is known as 'sitta' and contains practically all the common salt present in the crude nitre, but in addition contains much potassium nitrate; and the recovery of this nitrate entails complications in the running of a refinery. The usual plan in Northern India appears to be as follows. The 'sitta' is thrown into heaps in the factory compound and any waste liquors or scrapings from the factory floor are added, and the nitrate is extracted in almost exactly the same manner as the crude nitrate is made from the original nitrous earth.

This means that larger amounts of liquor are produced than are required in the refinery for extracting the nitrate from the crude saltpetre and necessitates the evaporation of some of the liquors from the 'kurias' in which the sitta and nitrous earth are placed for extraction. During the concentration the common salt which separates is removed by perforated ladles and the potassium nitrate crystallised from the residual liquor. The product obtained is highly impure and ranks with the crude nitre brought into the factory (Leather and Mukerji, p. 10).

According to Hooper (p. 34) the crude nitre is treated with a mixture of mother liquor and wash waters from previous operations and the whole is concentrated by boiling in large evaporating pans for three hours, the sitta is removed by means of a large iron spade fixed to a handle 6 feet long. The liquid is removed from the pan to a settling tank by means of an iron scoop and is allowed to stand for about two hours, the scum is removed and the clear liquid syphoned into the crystallising vats. In some factories the sitta is washed and the washings mixed with mother liquors for subsequent extractions. The residue at the bottom of the settling tank contains nitrate and is removed and mixed with the nitrous earth of the factory yard. The

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[1] p. 33.

sitta obtained varies in amount according to the quality of the crude nitre used.

One evaporating pan is capable of dealing with two boilings of 20 maunds of crude nitre each per day, and the crude nitre yields from 37·5 to 57·5 per cent. of refined nitre.

The wooden crystallising vats vary in size, but the usual dimensions are 3' × 5' or 6' × 6' and the length of time required for crystallisation varies from eight to ten days according to the size of the vats. In the U.P. a trellis-work made of bamboo is floated on the surface of the liquor and serves to facilitate the formation of good crystals. When crystallisation is complete the mother liquor is run off, the crystals scraped from the sides and bottom of the vat, then drained and finally washed, either in the vats themselves or by placing in bags and sprinkling with cold water and allowing the wash liquor to drain into an empty tub. Such washings are added to the mother liquors and are used for subsequent extractions.

In South India the method adopted is much the same. In a factory at Dindigul the following procedure is used at the present time. The factory contains one copper pan 4 ft. in diameter and about 2·5 ft. deep heated directly by means of burning spent tan-bark. For the one boiler there are sixteen crystallisers, each consisting of a wooden tub 3 ft. in diameter and 2 ft. deep. The liquor used for extraction is the mother liquor from the crystallising vats plus washings from the residue of common salt and from the refined nitre crystals. If these are not sufficient owing to the prolonged boiling during extraction, water is added. The factory runs for practically eight months in the year, and, as a rule, the mother liquor is changed at the end of a season. As two extractions are made each day and as the crystallising vats take 7–9 days for each crystallisation, the average number of extractions for which the same liquor is used may be taken as about thirty. When the next season begins half the old mother liquor is sprinkled on the earthern floor of the factory and the volume of the remainder made up by the addition of water.

For the extraction the boiler is filled to a depth of 18 inches with the liquor, which is boiled for three hours ; 4 maunds of 80 lbs. each of crude nitre are added and the heating continued for half an hour. If there is any tendency for the liquid to boil over, more of the cold liquor is added. The common salt, sitta, is removed by means of a perforated spade and placed in a conical basket 2 ft. in diameter and 14 inches high. One basket of salt is obtained in each boiling and the liquid is allowed to drain from it back into the boiler. Afterwards the salt in the basket is sprinkled with water in order to wash away as much nitrate as possible, about 5–6 gallons of cold water being used for each basket of salt. The liquor in the boiler is allowed to settle, any scum removed and the clear liquid decanted into the crystallisers where it is allowed to remain from seven to nine days. The mother liquor is then run off, the crystals detached from the sides of the vat and washed with cold water, when they lose the dark yellow colour and become almost white. As each crystalliser yields about 60–70 lbs. of refined saltpetre, the monthly output of such a factory can be taken as about 3,900 lbs. per month for eight months in the year. The fuel used costs from Re. 1 to Re. 1-4-0 per day. The sitta after it has been washed is handed over to the Salt Department for destruction.

In such a factory the sitta is not added to the heaps of nitrous earth in the yard, but each day the floor of the factory is scraped wherever efflorescence is observed and such scrapings are extracted in much the same manner as the crude nitre is extracted from nitrous earth. The product obtained is very impure and is mixed with the purchased crude nitre and refined. The analysis of some products taken from this factory are given in Table XXV (p. 228).

In a saltpetre refinery run on the lines indicated above there appear to be certain defects, and the more important of these are :—

1. In Northern India the large quantities of nitrate retained by the sitta necessitate the collection of this and its extraction in the compound of the factory. Old mother liquors and all materials containing appreciable amounts of nitrate are thrown upon the sitta heap, which is extracted in much the same manner as ordinary nitrous earth in a crude saltpetre factory. The solutions so obtained entail evaporation. There is therefore little or no loss of nitrate in such a factory. Indeed it is claimed that there is actually an increase in the nitrate content of the sitta heap owing to the formation and accumulation of nitrate in the soil of the refinery compound, in other words nitrification. There is no proof of such formation and Leather and Mukerji (p. 14) bring forward certain figures which indicate that little or no nitrification occurs.

2. In most of the factories it appears that larger quantities of mother liquors and wash waters are obtained than can be utilised in dissolving crude nitre and this is probably one of the reasons why the liquor is boiled for three hours either before or after the addition of the crude nitre. This six hours boiling during a day means the evaporation of appreciable amounts of water and hence enhanced costs for fuel consumption. As the fuel generally employed is very cheap (see above, also Hooper, p. 36) this factor does not affect the costs to a very material extent.

3. The products turned out by most of the refineries are in most cases somewhat low grade. Washed crystals rarely contain more than 92 to 96 per cent. of potassium nitrate and often 3 to 6 per cent. of sodium or potassium chloride.

Leather and Mukerji (p. 16) suggest certain improvements in the usual method for refining crude saltpetre. These consist mainly in :—

(*a*) Heating the crude nitre to about 100° with mother liquor in order to dissolve the potassium nitrate.

(*b*) Filtering the hot solution by means of compressed air.

(*c*) Cooling the hot liquid rapidly while it is stirred and so obtaining small crystals.

(*d*) Centrifuging the crystals and washing them with a little water on the centrifuge.

By this process practically no water has to be evaporated and hence there is a saving in fuel. The process, however, has two defects, viz. :—

(*a*) As the residues are not washed at all they contain 15 to 20 per cent. of the potassium nitrate, and this has to be extracted by a method similar to that adopted in the ordinary refinery and this entails evaporation.

(*b*) The nitrate crystals obtained have a purity of only 90 to 93 per cent. They contain appreciable amounts of sodium chloride.

We have carried out several series of experiments in order to work out a process of refining crude saltpetre so that in one operation from a crude product containing only 30–50 per cent. of potassium nitrate refined crystals containing less than 0·2 per cent. of chloride can be obtained, and at the same time the residue consisting of insoluble matter and common salt shall contain less than 2 per cent. of the total potassium nitrate originally present. In addition our object has been to avoid, as far as possible, the production of more mother liquors and wash waters than are necessary for dissolving and washing the usual amount of crude nitre worked up in the factory.

B. EXPERIMENTAL.

1. ANALYTICAL METHODS.

We have used the following methods for the large number of analytical determinations rendered necessary by our experiments.

1. *Nitrate.*—In the great majority of cases the nitrate radicle was estimated by reduction to ammonia with Devarda's alloy in a

strong solution of sodium hydroxide (sp. gr. 1·3). The ammonia was distilled into a given volume of standard sulphuric acid and the excess of acid titrated with 0·1N alkali.[1] The alloy was prepared in the laboratory as described by Scott [2] and the details of the method of analysis were as given by Sutton.[3]

Two other methods were also tried: (*a*) The nitrometer method and (*b*) Reduction with iron and dilute sulphuric acid, but Devarda's method was found to be the most rapid and accurate.

2. *Chloride.*—In the earlier experiments the chloride was determined by titration with standard silver nitrate using potassium chromate as indicator. In the later experimnnts Votocek's method [4] was employed. This consists in titrating the chloride solution, acidified with nitric acid, with standard mercuric nitrate solution, using 0·06 grams of sodium nitro-prusside as indicator when the volume to be titrated is about 250 cc. The appearance of a permanent white precipitate marks the end-point and the method is more accurate than the chromate method especially when only small amounts of chloride are present.

3. *Potassium.*—Potassium was estimated by the chloroplatinate method and weighed as K_2PtCl_6. Lindo Gladding's process of washing the precipitate of chloroplatinate with a 20 per cent. solution of ammonium chloride saturated with potassium chloroplatinate (Scott., p. 351) was used. The modified chloroplatinate method (Scott., p. 350) was found to be extremely convenient for estimating potassium in the case of residues containing small amounts of potassium salts and large amounts of impurities, the removal of which from the precipitated chloroplatinate is often tedious. The method consists in reducing the chloroplatinate to metallic platinum by means of magnesium and hydrochloric acid, filtering and weighing the metallic platinum.[5]

4. *Calcium and Magnesium.*—These were determined in the usual manner: the calcium was precipitated as oxalate and weighed as oxide and the magnesium precipitated as magnesium ammonium phosphate and weighed as magnesium pyrophosphate.

5. *Sodium.*—The sodium was estimated with the magnesium and potassium as sulphate and knowing the percentages of both magnesium and potassium the sodium was calculated by difference.

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[1] Compare A. Devarda, *Chem. Zeit.* **16**, 1952.
[2] *Standard Methods of Chemical Analysis*, second edition, 1917, p. 300.
[3] *Volumetric Analysis* tenth edition, 1911, p. 285.
[4] *Chem. Zeit.*, 1918, **42**, 257, and 271.
[5] Compare Hicks, *J. Ind.Eng. Chem.*, 1913, **5**, 650.

2. SOLUBILITY DETERMINATIONS.

As a guide to the conditions which must be observed when carrying out experiments on refining, we have made a number of solubility determinations.

The complete investigation of a system containing sodium, potassium and magnesium as chlorides, nitrates and sulphates through a wide range of temperatures would be a task of great magnitude and the labour entailed would not be compensated by the results obtained. Consequently we have confined ourselves to studying the system in a few aspects only, in order to obtain approximations to the conditions which exist in the solutions with which we are chiefly concerned.

Leather and Mukerji have discussed the system sodium and potassium, nitrate and chloride at some length[1] and have given numerous experimental results, but unfortunately none above the temperature of 91°. As sodium sulphate is an important impurity in many samples of crude nitre we have made certain determinations in solutions containing this substance and we have also carried out a few experiments with mixtures of nitrates and chlorides at temperatures higher than those mentioned by Leather and Mukerji. The latter are discussed under a separate heading.

a. Experimental Details.

For maintaining constant temperatures two thermostats were used; the one was electrically heated and controlled and could be maintained at a temperature of 25 or 30° within 1/100th of a degree and the second was a gas-heated water bath controlled by means of capsules, and the temperature varied within 0·2°. In the former thermostat there was no mechanical shaking arrangement and consequently the solutions were allowed to remain at the constant temperature for two or three days and were frequently shaken by hand. In the latter apparatus a bottle was immersed in the water and shaken violently by means of a motor, with the result that equilibrium was frequently reached in an hour, particularly at the higher temperatures. Samples for the solubility determinations were, however, not taken until they had been shaken for at least three hours.

At the lower temperatures, when equilibrium was attained, following Pawlewski's method,[2] the solid was allowed to settle and

[1] *Mem. Dept. Agric. India., Chemical series*, 1913, 3, 77.
[2] *Ber.*, 1899, 32, 1040.

some of the clear liquid syphoned off into a small bottle, also immersed in the thermostat, weighed and analysed. At higher temperatures this method gave inaccurate results owing to loss of water by evaporation. Consequently Leather and Mukerji's method of sucking the liquid through a capillary into an evacuated glass tube was adopted and gave very concordant results.

In the simpler systems, a considerable excess of the salts under investigation was taken in order to ensure saturation of the solution; for the more complicated mixtures the quantities of each salt and of the water taken were accurately weighed so that the composition of the solid phase could be calculated. No attempt was made to separate the solid phase and analyse it separately.

b. Values for Solubility.

In Table V. are shown the solubilities in water of the pure salts with which we are chiefly concerned. The figures are the mean values from Landolt's tables recalculated to grams of salt per 100 grams of water.

TABLE V.

Solubility of pure salts in grams per 100 grams of water.

Temp. °C	NaCl	Na_2SO_4	$NaNO_3$	KNO_3
20	35·9	19·2	88·1	31·6
30	36·1	40·5	96·2	46·0
40	36·4	48·2	104·9	64·2
60	37·2	45·6	124·8	110·5
80	37·9	43·5	148·2	169·0
100	39·1	42·6	174·0	246·0

Table VI gives Leather and Mukerji's values for mixtures of sodium chloride and sodium nitrate when the solution is saturated with respect to both salts, for sodium chloride and potassium nitrate when there are three salts in the solid phase and our own values for sodium chloride and sodium sulphate with both salts in the solid phase.

TABLE VI.

Solubility of mixtures of two salts in grams per 100 grams of water.

Temp °C	NaCl	$NaNO_3$	NaCl	KNO_3	$NaNO_3$	NaCl	Na_2SO_4
20	24·8	53·7	37·7	37·3	0·9 (K Cl)	32·3	10·0 (21·5°)
30	23·4	63·4	38·9	49·2	...	32·7	9·1
40	21·8	74·0	38·9	64·7	3·0	33·5	8·2
60	...	...	...	...	...	34·8	7·2
80	...	...	...	...	...	36·0	6·5
91	15·8	141	39·0	218	32·7	37·4	6·3 (97·5°)

From the above it is evident that the solubility of sodium chloride increases with rise in temperature in saturated solutions both of potassium nitrate and sodium sulphate, or in other words, such solutions will deposit sodium chloride on cooling. Moreover, Leather and Mukerji's figures for solutions not saturated with respect to potassium nitrate show that the solubility of sodium chloride may rise to 44 grams per 100 grams of water at 91°. Consequently we may assume that the hot solutions of crude nitre with which we have to deal will ordinarily deposit sodium chloride on cooling.

The solubility of sodium sulphate is greatly lowered by the presence of sodium chloride and decreases with rise in temperature, while as shown by W. Meyerhoffer and A. P. Saunders,[1] the transition point to the decahydrate is lowered to 17·9° by sodium chloride and to 16·3° by sodium chloride and potassium sulphate so that probably no decahydrate will be present in the solutions under consideration.

The mixture, potassium nitrate-sodium sulphate, forms a much more complicated system than any of the above, as, in addition to the double decomposition :—

$$2KNO_3 + Na_2SO_4 \rightleftarrows K_2SO_4 + 2\,NaNO_3,$$

glaserite, 3 K_2SO_4 Na_2SO_4, is formed, and at low temperatures the hydrated Glauber's salt exists. Table VII gives a number of values which have been determined at 25°.

[1] *Zeitsch. physikal. Chem.*, 1899, 28, 462

TABLE VII.

Solubility of mixtures of potassium nitrate and sodium sulphate at 25°.

Substance	Solution. Grams per 100 grams water.					
KNO_3	42·3	41·5	41·9	43·6	43·0	43·2
$NaNO_3$	7·1	13·4	38·5	53·3	55·2	61·0
Na_2SO_4	8·3	8·9	11·5	12·7	12·5	12·5
	Solid phase. Equivalents per 100 grams water.					
KNO_3	0·490	0·416	0·318	0·138	0·210	0·132
$\frac{1}{2}K_2SO_4$	0·080	0·160	0·455	0·620	0·648	0·720
$\frac{1}{2}Na_2SO_4$	0·081	0·114	0·242	0·320	0·300	0·508

Although by no means complete, these figures show that the addition of sodium sulphate to saturated solutions of potassium nitrate results in an increased amount of sodium nitrate in solution and deposition of solid potassium sulphate which probably forms glaserite. The solid phase has been calculated on the assumption that the sodium sulphate is anhydrous. It will be seen that if glaserite is formed, there is in all cases an excess of sodium sulphate. The result is the same if it is assumed that this excess consists of the decahydrate. The reason why this excess does not react with the potassium nitrate to give glaserite and sodium nitrate is not clear and cannot be definitely explained without further experiments, but it is probably connected with the fact that glaserite and sodium sulphate form solid solutions.[1]

Two experiments were carried out at 35° with a large excess of sodium sulphate, the results being shown in Table VIII.

These results are similar to those at 25°.

Measurements were also made at higher temperatures, but in these only slightly more potassium nitrate was taken than the quantity expected to be soluble not allowing for decomposition to sodium nitrate, consequently there was no potassium nitrate in the solid phase and the solutions were not even approximately saturated with respect to sodium nitrate. Sufficient sodium sulphate was taken for some of it to remain insoluble. The conditions thus attained may be regarded as approximating to those which occur during the extraction of crude nitre containing large amounts of sodium sulphate. The results are shown in Table IX.

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[1] K. Okada, *Mem. Coll. Sci., Kyoto*, 1914, 1, 85.

TABLE VIII.

Solubility of mixtures of potassium nitrate and sodium sulphate at 35°.

Substance	Solution. Grams per 100 grams of water.	
KNO_3	59·7	59·7
$NaNO_3$	67·4	68·6
Na_2SO_4	8·9	9·2
	Solid. Equivalents per 100 grams of water.	
KNO_3	0·217	0·208
½ K_2SO_4	0·980	0·988
½ Na_2SO_4	0·477	0·831

TABLE IX.

Solubility of sodium sulphate in almost saturated solutions of potassium nitrate. Grams per 100 grams water.

Substance	45°	60°	80°	97·5°
KNO_3	73·0	92·3	160·0	227·5
$NaNO_3$	8·8	13·8	16·5	24·4
Na_2SO_4	7·1	6·8	6·7	2·8

From all these figures it may be deduced that there is no tendency for sodium sulphate to separate from hot potassium nitrate solutions on cooling.

Experiments similar to these were carried out with mixtures of sodium sulphate, potassium nitrate and sodium chloride, in which the solid phases consisted of mixtures of sodium sulphate, potassium sulphate and sodium chloride, but no potassium nitrate. Some of the results are shown in Table X.

As above, no definite conclusions can be drawn from these figures, but they serve to indicate that in solutions similar to those which are met with in practice, the solubility of sodium sulphate is very slight and decreases with rise in temperature, while the solubility of sodium chloride is a little greater than in pure water, and increases with rise in temperature.

TABLE X.

Solubility of sodium chloride and sodium sulphate in almost saturated solutions of potassium nitrate. Grams per 100 grams water.

Substance	25°	45°	60°	80°	97·5°
KNO_3	43·5	77·4	101·8	179·7	244·8
$NaNO_3$	5·3	3·0	3·2	9·5	17·1
Na_2SO_4	3·4	2·8	2·5	2·5	2·5
NaCl.	35·9	36·5	39·1	40·6	41·6

3. The Reaction between Potassium Nitrate and Sodium Chloride.

It has been shown by Leather and Mukerji that at higher temperatures it is possible to obtain from potassium nitrate and sodium chloride solutions of sodium chloride, sodium nitrate and potassium nitrate not saturated with respect to the latter salt, but in equilibrium with solid sodium and potassium chlorides. Their results are not very comprehensive but they serve to show that there is a possibility of the loss of potassium nitrate if due precautions are not taken. Moreover, it will be shown later that, in the refining process we suggest, there is a tendency for sodium nitrate to accumulate in certain liquors. As, however, many samples of crude nitre contain considerable quantities of potassium chloride, during digestion this salt may react with the excess of sodium nitrate in the liquor and thus prevent an unduly high concentration being attained. The following experiments were carried out in order to ascertain the nature of the equilibrium.

A mixture of potassium nitrate, sodium chloride and potassium chloride was digested in a 1-litre flask with 100 or 150 grams of a mother liquor to which sodium nitrate had been added. 25 grams of potassium chloride and 100 to 150 grams of sodium chloride were added for each 100 grams of mother liquor. The flask was weighed, loosely stoppered and then heated at the required temperature with frequent shaking until equilibrium was attained. Sufficient water (usually 2 grams) was added during the experiment to compensate for any loss of weight by evaporation and the solution was rapidly filtered by suction in a funnel kept at the same temperature as the solution. A small bottle cooled in ice was placed between the pump and the receiver to collect any water which might evaporate during filtration. This water was returned to the solution which was covered and left overnight to crystallise. Potassium and nitrate were estimated in the clear solution and the excess of nitrate over potassium was taken to correspond with sodium nitrate. The addition of water naturally produced a small error in the results, but it was considered that this would be less than the error due to evaporation if no water were added.

The mother liquor used had a composition similar to liquors Nos. 27 and 29 on Table XXII, p. 224 and 25 grams of sodium nitrate was added to each 200 grams of liquor before use.

Table XI gives the results obtained, all concentrations being expressed in grams per 100 grams of water.

TABLE XI.

The equilibrium between potassium chloride and sodium nitrate.

Experiment No.	Temperature	Time of digestion. Hours	Initial conc. of $NaNO_3$	Final conc. of $Na\ NO_3$	Conc. of KNO_3 in hot solution
5	70	8	28·1	12·4	(145)
12	80	8	26·0	10·5	145
3	100	1	25·2	25·3	224
4	100	8	25·2	24·5	225
7	100	8	28·1	24·9	229
11	100	8	26·0	15·1	176
9	100	8	26·0	8·3	146
8	115	8	26·0	20·6	233
6	115	8	28·1	4·3	151
10	115	1	26·0	4·1	151

These figures show that in hot solutions of potassium nitrate, not completely saturated with this salt, but in contact with solid sodium and potassium chlorides, high concentrations of sodium nitrate cannot be attained, as potassium chloride and sodium nitrate readily react forming potassium nitrate and common salt until a given concentration of sodium nitrate is reached. It will be seen that the extent of this reaction depends upon the temperature of digestion and the concentration of the potassium nitrate. The lower the temperature and the less the concentration of the potassium nitrate, the smaller is the final concentration of sodium nitrate. The factor in the latter case appears to be what may be termed the percentage saturation, for in Experiment 8 the actual quantity of potassium nitrate in solution is larger than in Experiment 7, but the proportion to the maximum quantity which could be dissolved is less, consequently the final concentration of sodium nitrate is lower. A similar relation is to be observed in Experiments 12 and 6; while in Experiment 5, the only one in which solid potassium nitrate was present, the concentration of sodium nitrate, in spite of the low temperature, is higher still.

The conditions in Experiment 11 correspond with those under which many of our extraction experiments were carried out. The percentage saturation is about 60. The conditions of Experiment 6 correspond with those of an average refinery and the reason for the small quantity of sodium nitrate in refinery mother liquors is evidently due to the low percentage saturation of potassium nitrate. Experiments 3 and 10 show that the equilibrium is pratically complete within an hour.

4. EXTRACTION EXPERIMENTS.

(*a*) *Raw materials.*

Five samples of crude nitre were used for the extraction experiments. Of these four were obtained from the Coimbatore district and one from Bihar. Analysis gave the results shown in Table XII.

TABLE XII.

Analyses of crude nitres used in the experiments.

—	Sample No. 1 Bihar	Sample No. 2 Coimbatore	Sample No. 3 Coimbatore	Sample No. 4 Coimbatore	Sample No. 5 Coimbatore
K	26·91	16·93	14·14	20·88	20·64
Na	9·92	21·46	23·39	17·91	17·90
Mg	0·89	tr.	0·15	0·56	0·31
Ca	0·00	0·68	0·82	0·19	0·89
NO_3	33·19	22·36	19·91	18·69	20·64
Cl	20·58	34·93	36·72	35·09	34·86
SO_4	2·23	2·70	3·02	2·66	2·10
Insol.	1·09	0·52	0·35	n. d.	n. d.
Moisture	4·87	0·61	1·81	n. d.	n. d.
Total ...	99·68	100·19	100·32	(95·98)	(97·34)

These results may be expressed as percentages of the constituent salts as shown in Table XIII.

TABLE XIII.

Composition of crude nitres used in the experiments.

—	Sample No. 1	Sample No. 2	Sample No. 3	Sample No. 4	Sample No. 5
KNO_3	54·99	36·50	32·86	31·24	34·17
KCl	10·74	5·37	2·76	16·78	14·17
NaCl	21·27	53·37	57·63	42·03	44·89
Na_2SO_4	3·27	1·56	1·56	3·27	0·00
$MgCl_2$	3·47	tr.	0·57	2·20	1·19
$CaSO_4$	0·00	2·31	2·79	0·60	2·99

From this it will be seen that sample No. 1 from Bihar is considerably richer in potassium nitrate than the Coimbatore samples and it also contains the largest quantities of sodium sulphate and magnesium chloride.

It is worthy of note that all these samples contain a considerable proportion of the potassium in the form of potassium chloride, whereas none of Hooper's analyses, some of which have been given in Table IV, indicate the presence of this salt. As Hooper does not state how the analyses were effected, it is possible that potassium was not directly determined, and consequently the results are incorrect.

Samples Nos. 1 and 4 contain appreciable quantities of magnesium salts. The effect of these on the composition of the mother liquor after repeated use is discussed on page 223.

b. Small scale experiments on refining.

General method of procedure. Five pounds of the crude saltpetre were digested in an enamelled iron pan with a given amount of the liquor obtained from a previous experiment and saturated at 25° with both sodium chloride and potassium nitrate. The temperature was raised to that given in the table for the individual experiments and maintained at that temperature for some time, whilst the mixture was slowly stirred. Some 20–30 minutes were required before the given temperature was attained. The undissolved matter was then allowed to settle, the solution syphoned off and the wet residue immediately centrifuged and washed on the centrifuge with 100–300 c.c. of water. About 100 c.c. of the washings was added to the main extract and the whole filtered rapidly on a Büchner funnel with the aid of a filter pump. To the clear liquid 150 c.c. of wash water obtained by washing the pure crystals of nitrate and about 110 to 200 c.c. water were added in order to ensure that common salt crystals were not deposited during crystallisation. The clear warm liquid was kept in a covered bucket for about 36 hours, when the large crystals of potassium nitrate were removed, centrifuged and washed on the centrifuge with about 300 c.c. of water.

Table XIV shows the results obtained.

Notes on the small scale experiments.

Experiment 1.—In this experiment the liquid was not filtered, but allowed to settle and the supernatant solution siphoned off, and the crystals obtained had a brownish colour. Owing to the long time of digestion the water added was insufficient to make up for the loss by evaporation, and cubical crystals of common salt were observed on the surface of the solution. The percentage of chloride in the nitre crystals was relatively high although the crystals were washed four times with small amounts of water. The residue was centrifuged and washed once.

Experiment 2.—The solution, after the digestion was completed, was siphoned off, filtered and 350 c.c. of water added to the filtrate to compensate for evaporation during the operations. The residue was washed twice, each time with 150 c.c. of water and the first washings were added to the main liquor and the whole heated to 60° before crystallisation. The wash waters of the refined nitre amounted to 275 c.c. and the first 175 c.c. of these washings were added to the mother liquor.

Experiments 3 and 4.—Similar to No. 2.

Experiments 5 and 6.—In these two experiments the solution was stirred for about 6 hours during crystallisation in order to obtain small crystals. In Experiment 5 the percentage of chloride in the crystals is very high and is due firstly to the increased evaporation taking place during stirring and hence deposition of sodium chloride crystals together with the potassium nitrate crystals and secondly to the greater difficulty of washing the small crystals as compared with large crystals. In Experiment 6 an attempt was made to reduce the chloride content of the nitre crystals by adding 300 c.c. of water during the stirring and the effect is pronounced. In both experiments during centrifuging of the nitrate crystals the mass on the centrifuge was turned over from time to time in order to expose fresh surfaces to the wash-water.

TABLE XIV.

Small scale experiments.

In all these experiments the crude nitre used was sample No. 1 (for analysis cf. p. 210). In each experiment five pounds of crude nitre were taken and this contained 2·85 lbs. of potassium nitrate and 5 to 7 per cent. of moisture according to the atmospheric humidity.

No. of the experiment	Temperature used for extraction in °C	Weight of liquor used for extraction in pounds	Time of digestion at the given temperature in minutes	Purified nitre				Residue			Weight of mother liquor after extraction in pounds
				Yield of nitre crystals in pounds	Percentage of Cl. in the nitre before washing	Percentage of Cl. in the nitre after 2nd washing	Percentage of Cl. in the final product	Weight of moist residue containing 4–5 per cent. of moisture in pounds	Percentage of NO_3 in the moist residue	Percentage of total nitrate present left unextracted in the residue	
1	55	19·0	120	2·25	...	...	0·61	1·2	1·83	1·26	19·8
2	55	19·8	130	2·75	...	...	0·49	1·5	0·93	0·78	19·5
3	55	20·0	75	2·5	...	...	0·39	1·75	1·0	1·0	19·8
4	55	20·0	45	2·75	...	...	0·35	1·6	1·26	1·2	20·0
5	55	20·0	25	2·75	...	...	2·46	1·5	...	...	20·0
6	55	20·8	25	2·5	...	...	0·43	1·75	...	...	21·3
7	55	20·8	25	2·6	...	...	0·65	1·6	...	...	21·3
8	55	21·3	20	2·75	...	...	1·0	1·6	...	...	21·3
9	55	21·3	20	2·75	...	...	0·42	1·6	...	...	21·3
10	55	21·3	20	2·75	...	0·20	0·08	1·6	...	...	21·3
11	55	20·8	10	2·75	...	...	0·05	1·6	...	...	21·3
12	55	20·8	10	2·3	...	0·14	0·05	1·6	0·7	0·7	22·0
13	55	20·8	10	2·3	...	...	0·05	1·6	1·8	1·7	22·0
14	55	21·0	5	2·3	...	...	0·07	1·6	0·46	0·5	22·0
15	80	10·3	5	2·8	3·5	...	0·15	1·3	2·23	1·6	11·0
16	80	10·0	5	2·8	1·0	...	0·16	1·8	2·60	2·7	10·25
17	80	10·0	5	2·75	0·95	...	0·10	1·7	3·20	3·1	11·75
18	80	10·0	7	2·75	1·60	...	0·12	1·7	1·20	1·2	11·75
19	80	10·0	6	2·75	1·30	0·34	0·12	1·75	1·70	1·7	11·75
20	55	13·5	10	2·7	0·80	...	0·08	1·7	0·90	0·8	14·2
21	80	6·0	10	2·5	...	...	0·10	1·9	3·60	3·9	6·0
22	80	6·0	5	2·7	1·1	...	0·14	1·8	4·30	4·5	6·3
23	80	6·5	5	2·75	1·0	0·34	0·15	2·2	6·0	7·5	6·0
24	80	6·5	5	2·7	...	...	...	2·2	5·8	7·2	6·25
25	80	6·5	5	2·75	...	...	...	1·9	...	...	6·25

Experiments 12–14.—In these experiments the washing of the residue and of the refined crystals was carried out in a more systematic manner so as to reduce to a minimum the amount of wash-water and consequently of the concentration of dilute liquors which is necessary whenever a process entailing washing is used. This question is dealt with fully in connection with the larger scale experiments. Briefly, the wash-liquors were divided into three portions of different concentrations and these were used successively for washing the next batch of solids.

Experiments 15–19.—These experiments were carried out at 80° and slightly different methods of washing the refined crystals were tried. Good results were obtained by mixing the crystals with fresh wash-liquor after once centrifuging and then centrifuging again, but the improvement was not sufficient to warrant the adoption of this process.

Experiments 20–24.—Although the results obtained in Experiments 1-14 are quite satisfactory from the standpoint of yield and purity of the nitrate crystals, and also of the amount of nitrate left in the residue, they cannot be regarded as satisfactory as regards the quantity of liquor required for producing a given amount of pure nitrate. Thus at 55° for every 2·85 lbs. of pure nitrate about 20-22 lbs. of liquor were used in the extraction and the weight of liquid from which this amount of nitrate was obtained was 23-25 lbs. On this basis a factory producing 1 ton of pure nitrate per day would use 9 tons or 1,500 gallons of liquor for crystallisation, and assuming that the crystallising vats have to stand 7 days before the nitrate crystals are removed this entails a crystallising vat capacity of 10,500 gallons. Extracting at 80°, the total vat capacity would be 6,000 gallons. If the amount of liquor required for extraction could be reduced so as to approximate more closely to the theoretical value which, according to Leather and Mukerji's results is approximately 11 lbs. at 55° and 4·5 lbs. at 80° for the quantity of nitrate we used, not only would there be a considerable reduction in the necessary vat capacity but also in extracting pan and filtering capacity and in labour charges connected with the treatment of the liquors.

In Experiments 20–24 the amount of liquor used was reduced, the yield of nitrate crystals was good and the amount of chloride in the crystals was comparatively small; but, on the other hand, in the experiments conducted at 80° the amount of nitrate left in the residue is comparatively high and entails a loss of 4·5 to 7·5 per cent. of the total nitrate present in the crude nitre. This is largely due to the fact that in centrifuging the residue the solution is so nearly saturated with potassium nitrate that crystals of this salt are deposited during centrifuging and are not all removed by subsequent washing. This difficulty has been overcome in the subsequent experiments.

Experiment 25.—A little glue was added in this experiment in order to ascertain if the process of filtration could be eliminated and simple decantation substituted. It was found that filtration was still necessary and the colour of the crystals was not improved.

ANALYSES OF LIQUORS, RESIDUES AND REFINED NITRE CRYSTALS OBTAINED IN THE SMALL SCALE EXPERIMENTS.

TABLE XV.

Analyses of refined nitre crystals.

—	FROM EXPERIMENT 1		FROM EXPERIMENT 2	
	Per cent.	Equivalents	Per cent.	Equivalents
NO_3	59·63	0·961	59·40	0·959
K	37·34	0·955	37·67	0·961
Cl	0·59	0·016	0·49	0·014
Na	0·39	0·016	0·24	0·010
Moisture	2·23	...	2·24	...
Total	100·18	...	100·03	...

TABLE XVI.

Analyses of residues.

Experiment	No. 1		No. 2		No. 4	
	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents
Cl.	49·18	1·385	50·88	1·435	50·10	1·413
NO_3	1·86	0·030	0·94	0.015	1·30	0·021
SO_4	3·65	0·075	3·04	0·063	...	...
K	13·81	0·353	13·20	0·338	14·10	0·361
Na	26·02	1·135	25·96	1·129	...	...
Mg	0·64	0·052	0·44	0·036	0·34	0·028
Moisture	4·92	...	...	...	...	...
Total	100·08	...	...	...	...	...
As salts						
KNO_3	4·3	...	1·8	...	...	...
KCl	23·1	...	23·9	...	...	...
NaCl	59·8	...	61·9	...	...	...
Na_2SO_4	5·3	...	4·5	...	...	...
$MgCl_2$	2·5	...	1·7	...	...	...

TABLE XVII.

Analyses of Mother liquors.

At end of experiment	No. 1		No. 2		No. 4		No. 14		No. 22	
	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents
Cl	11·92	0·335	12·75	0·364	12·89	0·365	12·34	0·348	12·13	0·342
NO_3	11·61	0·187	12·54	0·203	12·15	0·196	14·51	0·234	15·37	0·248
SO_4	0·84	0·018	0·94	0·020	...	...	1·10	0·023	0·99	0·021
K	10·44	0·266	10·81	0·276	10·91	0·279	10·10	0·258	10·31	0·264
Na	5·85	0·256	6·49	0·281	...	...	7·13	0·310	6·44	0·280
Mg	0·15	0·012	0·19	0·016	0·25	0·021	0·55	0·045	0·92	0·076
Total acid radicles	...	0·540	...	0·587	...	...	...	0·605	...	0·611
Total metallic radicles	...	0·534	...	0·573	...	...	...	0·613	...	0·620
As salts per 100g. water										
KNO_3	31·7	...	36·2	...	...	...	43·2	...	47·0	...
KCl	9·7	...	9·5	...	...	...	3·5	...	1·9	...
NaCl	23·4	...	27·0	...	...	...	30·1	...	27·3	...
Na_2SO_4	2·2	...	2·5	...	...	...	2·9	...	2·8	...
$MgCl_2$	0·8	...	1·2	...	...	...	3·9	...	6·7	...
D^{25}_{25}	1·342		1·345		......		1·366		1·380	

It is interesting to note that in the earlier experiments owing to the high percentage of potassium chloride (10·7 in the crude nitre used, and the low temperature of extraction (55°), the mother liquors contain considerable amounts of potassium chloride.

c. Larger Scale Experiments.

As the small scale experiments gave such promising results it was decided to try a series of experiments on a larger scale, using 75 lbs. of crude nitre, and temperatures of 80° and upwards.

Apparatus.

Extraction Vessel.—The vessel used throughout these experiments was a steam jacketted pan of 25 gallons capacity. The amount of crude nitre used had a potassium nitrate content of 23 to 29 lbs. Usually fifteen minutes were required to raise the temperature to the fixed point and the digestion was continued at that temperature for ten minutes with constant stirring.

Filtration.—In the earlier experiments attempts were made to filter the liquid with the aid of a 6″ × 6″ gun-metal filter press using 6 plates. The liquid was transferred to the press by means of buckets and it was found advisable to have these steam jacketted. As the cup leathers in the filter press pumps were quickly destroyed by the hot saturated solution, a considerable amount of the nitrate was lost and a process of suction filtration was adopted for the later experiments.

As first used the suction apparatus consisted of two cylindrical iron drums—an upper and a lower. The upper drum which was steam jacketted was 21″ high by 14″ diameter. In this drum an iron ring 1·5″ wide was fixed about 3″ from the bottom, a layer of cotton wool was placed on this ring and then a false bottom made of sheet iron with $\frac{1}{16}$″ perforations. Over this was a layer of 30-mesh wire gauze and finally a filter cloth. The lower drum was 16″ × 11″ diameter, and was attached to a suction pump. In all the experiments an ordinary filter pump was used for reducing the pressure and the pressure registered was usually about 450 m.m. as compared with an external pressure of 680 m.m. The two drums were connected by a 1 c.m. glass tube fitting into rubber corks in the bottom of the upper vessel and the top of the lower vessel.

When the extraction was complete, most of the residue was removed by means of a perforated ladle and pressed down on the filter cloth. The extract and the remainder of the residue were added and suction started. Filtration began at once and was complete in about 10 minutes. After a number of experiments had been made, the lower drum was made with a conical bottom and a draw-off cock below, so that liquid could be removed readily at any time. The arrangements of the vessels and their relative dimensions are shown in Fig. I.

Washing the residue.—The majority of the experiments were performed with a view to determining the optimum conditions for washing the residue. The fundamental principles which had to be considered were (*a*) the reduction of the quantity of water used to the lowest possible value, (*b*) very complete removal of the nitrate from the residue and (*c*) the adjustment of the process so that when a series of wash liquors was used, the concentration of nitrate in each liquor after use was the same as the concentration of nitrate in the preceding liquor of the series before use.

Of these (*c*) was found to require the most experimental work. The following Table (XVIII) shows the results obtained in Experiment 45.

TABLE XVIII.

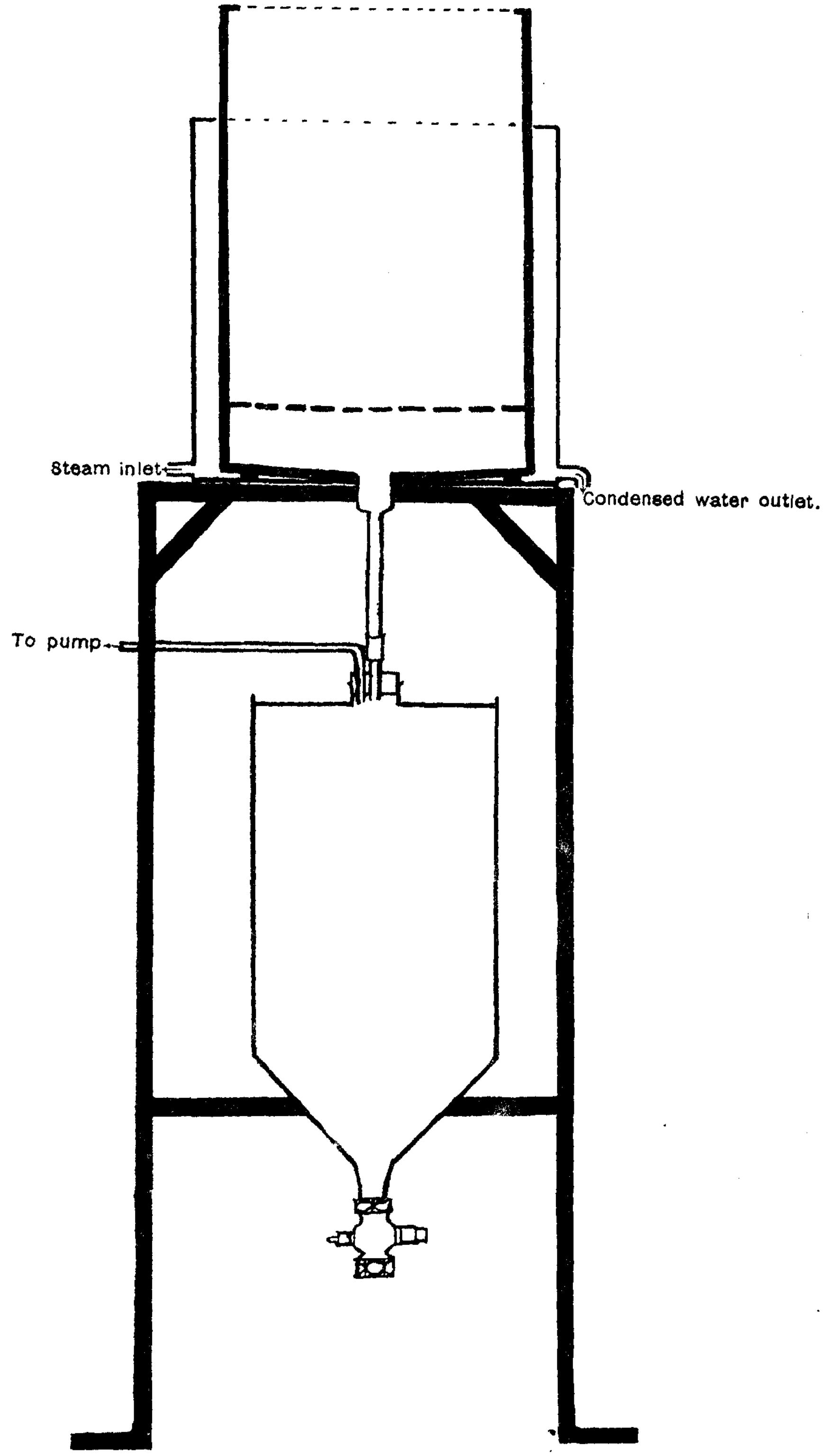
WASH LIQUOR	No. 1	No. 2	No. 3	No. 4
Before use				
Vol. in litres	7·0	7·5	7·5	3·7
Sp. gr. at 80°	1·365	1·312	1·273	water
Per cent. of KNO_3	17·4	12·9	6·6	...
After use				
Vol. in litres	added to main liquor	7·4	7·5	7·7
Sp. gr. at 80°		1·363	1·312	1·270
Per cent. of KNO_3		24·0	12·9	6·2

In the early experiments the wash liquors were divided into six portions in order to observe more closely the changes which occurred. It was found later that three liquors were sufficient.

In one series of experiments a comparison was made between draining as much liquor as possible from the residue before adding the wash liquor and merely removing liquor until the solid residue could be seen and then adding wash liquor and draining again, and it was ound that the latter method gave better results, as the washing was

Figure I.

Apparatus for separating liquor and washing residue.



more even and also less evaporation took place and the risk of deposition of nitre crystals on the residue was eliminated.

The actual amount of liquor mixed with the residue, when the second method was adopted, was found by experiment to be 5·5 to 6·0 litres and in several experiments the volume of wash liquors taken was 6·0 litres, but it was found that using this quantity the sp. gr. and nitre content of the wash liquors did not remain constant as shown in Table XVIII, but increased appreciably after each extraction. The volume was then increased to 7·5 litres and satisfactory results were obtained, and this quantity was used in the later experiments, although naturally it entailed an increase in the volume of the mother liquor and hence evaporation of water in order to avoid an accumulation of such liquor. As wash liquor No. 2 after use deposited small amounts of crystals (KNO_3 + NaCl) on cooling, in one series of experiments the solid was allowed to separate and the mother liquor after heating to 80° used as wash liquor No. 1 in the following extraction. As this liquor had practically the same composition as the main mother liquor it was found simpler to transfer wash liquor No. 2, after use, to the evaporator whilst still hot and concentrate it and to use in place of it, as wash water No. 1 in the next extraction, a corresponding volume of the superfluous main mother liquor.

The final method adopted was as follows :—As soon as the layer of liquid extract had disappeared below the level of the solid residue, 7 litres of hot mother liquor were added and allowed to sink to the level of the solid. The process was repeated twice with 7·5 litres of hot solutions containing about 13·0 and 6·5 per cent. of potassium nitrate respectively [1] and finally 3·7 litres of hot water were added and the solution drained from the solid as completely as possible. These quantities were found to be approximately correct whatever the temperature of digestion. They are roughly proportional to the volume of residue, so that for equal weights of crude nitre a high grade nitre would require less wash liquor than a low grade nitre.

Washing the crystals of refined nitre.—This problem was exactly analogous to that of washing the residue. The best results were obtained by washing on the centrifuge used for separating the crystals from the mother liquor. The weight of crystals was about 20 lbs. and the centrifuge used was rather larger than necessary, consequently the layer of crystals was thin and the results rather irregular. Satisfactory results were obtained by washing with 1·0 litre of each of three liquors of decreasing chlorine content and then with a litre of water or a little less. Table XIX shows typical results for experiment No. 36.

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[1] Compare Table XVIII.

TABLE XIX.

WASH LIQUOR	No. 1	No. 2	No. 3	No. 4
Before use				
Volume in litres	1·0	1·0	1·0	0·9
Sp. gr. at 23·5°	1·316	1·249	1·176	water
Per cent. of Cl.	11·5	5·4	1·3	...
After use				
Volume in litres	1·0	1·0	1·0	1·0
Sp. gr. at 23·5°	...	1·307	1·255	1·212
Per cent. of Cl.	...	10·4	6·2	3·2

In this experiment the Cl. content of the liquors increased rather more than it should have, e.g., liquor No. 3 after use contained 6·2 per cent. of chlorine whereas the percentage should have been approximately the same as in liquor No. 2 before use, viz., 5·4.

The main liquor from the lower drum together with the first seven litres of liquor obtained after adding the first wash-liquor and about one litre of first washings from the refined crystals of the previous experiment were run into the crystallising vessel, an iron drum of forty litres capacity, and, if the volume was still too small as indicated by a mark on the crystalliser, a further quantity of washings from the previous batch of refined nitre was added and the whole allowed to stand for about forty-eight hours.

Amount of liquor used for extraction.—In all these experiments attempts were made to keep the volume of liquor used for extraction as low as possible, viz., 25 per cent. excess of the theoretical amount required to dissolve all the potassium nitrate present in the 75 lbs. of crude nitre.

Tables XX and XXI show the main results which have been obtained. Experiments 26 to 35 were carried out mainly with a view to finding out the best method of washing the residue and the remaining experiments were designed to simplify and generally to improve the process.

TABLE XX.

Larger scale experiments.

Experiment number	Temperature of digestion ° C	Crude Nitre			Liquor used for the extraction lbs.	Residue							Purified nitre crystals				Weight of mother liquor lbs.
		Potassium nitrate lbs.	Moisture lbs.	Residue lbs.		Weight of dry residue after extraction lbs.	Percentage of NO_3 in the residue after washing No.					Percentage of total potassium nitrate left in residue	Weight in lbs.	Percentage yield	Percentage of chlorine		
							1	2	3	6	7				Un-washed	Washed	
26	80	27·3	2·1	45·6	48·0	44·5	2·9	...	...	...	...	7·7	21·5	79·5	1·4	0·10	48·8
27	80	24·0	1·5	49·5	42·0	54·6	5·1	...	...	...	...	18·8	19·0	79·0	0·85	0·17	40·5
28	80	24·0	1·5	49·5	42·0	54·0	...	3·8	...	...	...	13·9	18·5	77·0	1·20	0·15	45·0
29	80	24·0	1·5	49·5	42·0	52·5	3·4	...	...	...	...	12·0	19·0	79·0	0·81	0·13	43·0
30	80	22·5	0·9	51·6	41·0	52·0	3·6	2·4	...	...	...	8·9	19·5	86·0	0·95	0·14	42·0
31	80	22·5	0·9	51·6	41·0	52·3	3·0	2·3	1·7	...	...	6·2	19·8	88·0	1·65	0·22	41·8
32	80	22·5	0·9	51·6	41·0	51·0	...	...	...	0·2	...	0·9	19·0	79·0	...	0·12	...
33	80	22·5	0·9	51·6	41·0	50·0	...	...	...	...	0·18	0·8	20·8	92·0	0·89	0·13	...
34	80	22·5	0·9	51·6	41·0	52·0	...	...	...	...	0·29	1·0	20·9	93·0	1·10	0·16	...
35	80	29·0	0·6	23·4	50·0	22·5	...	...	...	0·4	...	0·6	27·5	94·8	...	0·30	...

TABLE XXI.

Larger scale experiments.

Experiment number	Temperature of digestion °C	Crude Nitre			Liquor used for the extraction		Residue			Purified nitre crystals			Mother liquor	
		Potassium nitrate lbs.	Moisture lbs.	Residue lbs.	Weight in lbs.	Volume in litres.	Weight of dry residue after extraction lbs.	Per cent. of NO_3 in the dry residue	Per cent. of total potassium nitrate left in the residue	Weight lbs.	Percentage yield	Percentage of chlorine (Cl.)	Weight in lbs.	Volume in litres
36	80	21·9	5·0	48·1	41·0	13·6	40·1	0·21	0·6	17·5	79·4	0·34	52·0	17·3
37	80	24·0	4·0	47·0	39·0	...	44·5	0·44	1·3	19·0	79·2	0·32	45·0	...
38	80	24·1	3·1	47·8	39·0	13·2	44·7	0·40	1.2	19·5	78·7	0·07	44·0	14·0
39	80	24·0	3·7	47·3	38·0	12·5	43·0	0·45	1·3	18·5	77·1	0·20	48·0	15·6
40	80	23·8	4·7	46·5	38·0	12·5	44·2	0·53	1·6	19·3	80·6	0·32	44·0	14·2
41	80	23·9	3·9	47·2	38·0	12·5	44·0	0·54	1·6	20·3	84·6	0·12	44·3	14·4
42	80	24·1	3·2	47·7	38·0	12·8	44·0	0·40	1·1	22·0	91·3	0·13	44·0	14·2
43	80	23·1	6·1	45·8	38·0	12·6	43·2	0·23	0·7	20·8	90·1	0·12	45·0	14·6
44	80	24·8	3·3	46·9	38·0	12·6	40·0	0·15	0·4	22·0	88·5	0·28	50·0	16·5
45	80	24·5	4·1	46·4	38·0	12·7	40·0	0·19	0·5	21·8	89·0	0·11	51·0	16·8
46	80	24·1	5·4	45·5	38 0	12·6	38·5	0·22	0·6	20·5	85·0	0·18	53·5	17·8
47	90	24·5	4·0	46·5	32·0	10·9	41·8	0·26	0·7	21·0	85·7	0·24	44·5	14·5
48	90	24·4	4·5	46·1	32·0	10·9	41·5	0·24	0·7	21·0	86·1	0·18	44·8	14·6
49	100	25·0	2·7	47·3	26·0	8·8	43·4	0·32	0·9	22·5	90·0	0·29	35·8	11·6
50	100	24·5	0·3	50·2	29·0	...	48·2	0·21	0·7	21·2	86·5	0·11	36·5	11·9
51	100	24·6	2·1	48·3	26·0	8·7	45·1	0·32	1·0	20·3	82·3	0·19	35·0	11·3
52	100	24·9	2·8	47·3	26·0	8·7	45·8	0·25	0·8	22·5	90·4	0·20	35·0	11·3

(*d*) EXAMINATION OF PRODUCTS.

Mother liquors. In Table XXII are given the results of the analyses of seven different samples of mother liquors obtained in the course of the work. These were taken respectively in Experiments 27, 29, 35, 39, 46, 50 and 52. The liquor obtained in Experiment 27 was a fresh liquor and had not been used in a previous experiment, but in all the subsequent experiments the same mother liquor was used so that at the end of Experiment 52 the liquor had been used 26 times. In connection with these experiments it must be remembered that up to Experiment 35 the whole of the mother liquor from one experiment was used for the following experiment, but from 35 onwards only a portion was used, e.g., in Experiment 45, only 38 lbs. out of a total of 50 lbs. obtained from the previous experiment, while from 44 onwards artificial crude nitre made by mixing refined crystals and residues was employed.

In the lower portion of the table the quantities of the various salts per 100 grams of water have been calculated. For simplicity it has been assumed that all the sulphate is present as sodium sulphate and that calcium and magnesium are in the form of chlorides. It will be noted that the quantity of potassium nitrate fluctuates somewhat, probably owing to the different temperatures at which the crystals were removed and to a certain amount of supersaturation. The quantity of sodium nitrate rises rapidly at first but soon attains an approximately constant value of about 9 grams per 100 grams of water. This is what might be expected from the experiments given on p. 209 but the actual value is somewhat low, e.g., 9 instead of 15, since in the actual extraction experiments the mother liquors had been diluted after the extraction was complete. It follows that it is desirable to use a liquor containing approximately 15 grams of sodium nitrate per 100 grams of water in order to avoid decomposition of the potassium nitrate.

Owing to the use of artificial mixtures containing no magnesium in the later extractions the proportion of magnesium chloride has not increased greatly in the series of experiments, but this would doubtless occur in a factory treating magnesium bearing crude nitre and the increase in concentration of the magnesium chloride would prove the determining factor with regard to the number of times a mother liquor could be used.

Refined nitre.—In Table XXIII the analyses of four samples of refined nitre crystals are given. They show that the Cl content is below 0·3 per cent. corresponding with 0·5 per cent. of sodium chloride. The values for Na are obtained by difference and as they are extremely low cannot be taken as being at all accurate.

It will be noticed that the K equivalent is always higher than that of the NO_3 by about 1 per cent. indicating an approximately constant error in one of the determinations.

TABLE XXII.

Analyses of Mother-liquors obtained after Experiment No.

—	27		29		35		39		46		50		52	
	Per cent	Equivalents	Per cent	Equivalents	Per cent	Equivalents	Per cent	Equivalents	Per cent	Equivalents	Per cent	Equivalents	Per cent	Equivalents
K	7·91	0·202	7·95	0·203	8·64	0·221	6·63	0·169	7·27	0·186	6·66	0·170	7·59	0·194
Na	8·84	0·384	8·22	0·357	8·19	0·356	8·74	0·380	9·15	0·399	8·94	0·389	9·08	0·396
Mg	0·14	0·009	0·18	0·015	0·24	0·020	0·30	0·025	0·31	0·026	0·31	0·026	0·34	0·028
Ca	0·09	0·004	0·20	0·010	0·37	0·018	0·29	0·015	0·19	0·009	0·16	0·008	0·15	0·007
SO_4	0·35	0·007	0·37	0·007	0·32	0·007	0·31	0·006	0·39	0·008	0·36	0·007	0·37	0·007
Cl	12·69	0·358	12·20	0·344	11·69	0·330	12·67	0·357	13·24	0·377	12·53	0·353	12·89	0·364
NO_3	13·76	0·222	15·16	0·245	16·80	0·270	13·61	0·221	15·18	0·244	13·98	0·226	15·55	0·251
As salts per 100g. water.														
KNO_3	36·2	...	36·5	...	41·2	...	29·8	...	34·4	...	30·2	...	36·2	...
$NaNO_3$	3·4	...	4·6	...	8·0	...	7·9	...	8·9	...	8·6	...	9·1	...
NaCl	35·8	...	33·2	...	31·8	...	32·3	...	35·6	...	32·7	...	35·5	...
Na_2SO_4	0·9	...	0·9	...	0·9	...	0·8	...	1·0	...	0·9	...	0·9	...
$CaCl_2$	0·4	...	0·9	...	1·9	...	1·4	...	0·9	...	0·8	...	0·7	...
$MgCl_2$	0·7	...	1·2	...	1·7	...	2·1	...	2·2	...	2·1	...	2·6	...
Temperature at which liquor was removed					25°		24·5°		22°		23·5°		22·5°	
Sp. gr. at 26·5°	1·349		1·358		1·369		1·347 at 19°		1·358 at 22°		1·352 at 19°		1·362 at 20°	

TABLE XXIII.

Analyses of refined potassium nitrate crystals.

From Experiment	No. 28		No. 32		No. 35		No. 52	
——	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents
Moisture	1·23	...	1·11	...	1·23	...	0·30	...
K	38·45	0·983	38·55	0·986	38·34	0·981	38·68	0·989
Na	0·05	0·002	0·02	0·001	0·11	0·005	0·10	0·004
NO_3	59·97	0·967	60·03	0·969	60·17	0·971	60·60	0·977
Cl	0·19	0·005	0·19	0·006	0·29	0·009	0·24	0·007
Total per cent	99·89	...	99·90	...	100·14	...	99·92	...
Acid radicles	...	0·972	...	0·975	...	0·980	...	0·984
Metallic radicles	...	0·985	...	0·987	...	0·986	...	0·993

Residues.—Table XXIV gives the results of the analyses of some residues from the earlier experiments. It will be noticed that some of these contain large quantities of potassium chloride. As however the original crude nitres contained potassium chloride, it may be seen that the quantity of this salt in the residue is only slightly greater than what was present originally. For example in No. 34 the total weight of potassium chloride in the residue is 11·7 lbs. while in the 75 lbs. of crude nitre (No. 5) used there were 10·6 lbs.

Table XXV gives the analyses of a few products obtained from Dindigul and Murungapet (Trichinopoly) factories. The specific gravity of sample No. 8 is high indicating that the crystals were removed at temperatures higher than those employed by us. Sample No. 5 was taken towards the end of the season and the quantity of magnesium chloride is seen to be very large while the potassium nitrate and sodium chloride are correspondingly small in quantity. In both samples the amount of sodium nitrate is small owing to the low concentration of potassium nitrate in the hot extracts.

The analysis of the residue No. 4 shows that the NO_3 has been removed nearly as completely as in our experiments and that potassium chloride is present in small quantities.

The values for the refined nitres are interesting. Sample No. 3, an average sample from the Dindigul factory shows the presence of 12·2 per cent. of Cl, corresponding with 20 per cent. of common salt, but the excess of K over NO_3 equivalents indicates the presence of 4·2 per cent. of potassium chloride. In addition, calcium, magnesium and sulphate were detected.

Sample No. 6, a sample of recrystallised nitre from the same factory the price of which was stated to be Rs. 140 per candy of 800 lbs. as compared with Rs. 80 for the non-recrystallised product, contained 0·62 per cent. of Cl and hence was not so high grade as the products obtained by us in one operation. This sample was free from Mg, Ca and SO_4.

Sample No. 7, a sample of average refined saltpetre from the Murungapet factory contained 6·9 per cent. of Cl and was thus better than the Dindigul sample. Mg, Ca and SO_4 were also detected.

TABLE XXIV.

Analyses of residues.

From Experiment	No. 27		No. 29		No. 32		No. 34	
—	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents	Per cent.	Equivalents
K	5·70	0·146	4·22	0·108	11·56	0·295	12·05	0·308
Na	31·42	1·367	...	...	...	...	27·84	1·211
Mg	...	...	...	...	...	...	0·19	0·016
Ca	1·00	0·050	...	...	...	...	0·35	0·018
SO_4	3·24	0·067	...	...	...	...	3·75	0·078
Cl.	50·43	1·422	...	...	...	...	52·15	1·471
NO_3	5·10	0·082	3·76	0·062	0·23	0·004	0·31	0·005
Insoluble	1·35	...	...	...	...	...	1·62	...
Moisture	1·20	...	...	...	...	...	1·30	...
Total	99·43	...	...	...	...	...	99·56	...
As salts								
KNO_3	8·3	...	6·3	...	0·4	...	0·5	...
KCl	5·4	...	3·4	...	21·7	...	22·7	...
NaCl	78·9	...	...	...	...	...	67·3	...
Na_2SO_4	1·2	...	...	...	...	...	4·4	...
$CaSO_4$	3·4	...	...	...	...	...	1·2	...
$MgCl_2$	...	...	...	...	...	...	0·8	...

TABLE XXV.

ANALYSES OF SAMPLES FROM SOUTH INDIAN FACTORIES.

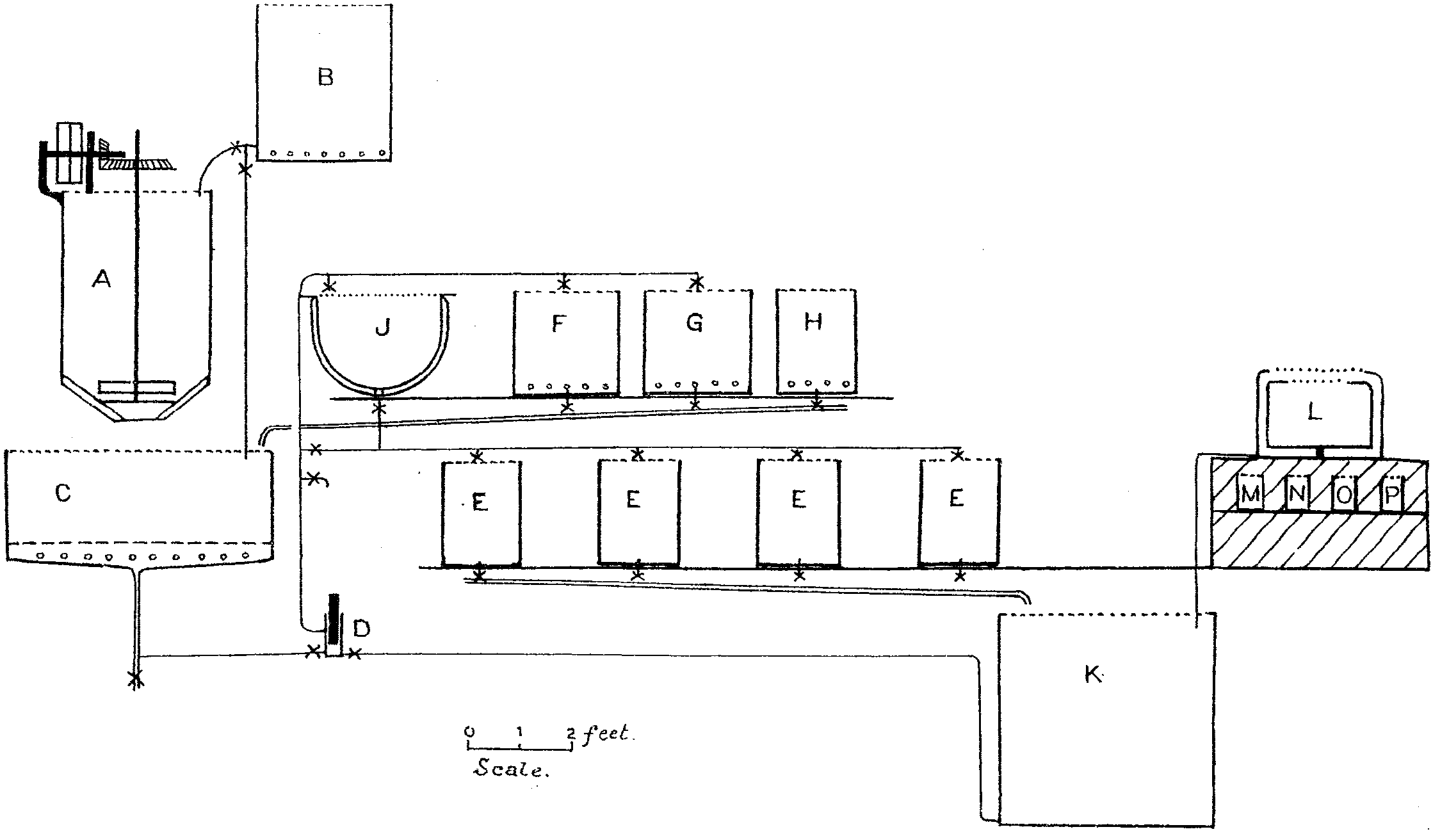
A. *Liquors.*

—	No. 5 from Dindigul Factory		No. 8 from Murungapet Factory	
	Per cent.	Equivalents	Per cent.	Equivalents
K	6·95	0·177	10·06	0·257
Na	3·10	0·135	7·61	0·331
Ca	0·77	0·038	...	...
Mg	2·99	0·246	0·27	0·022
SO_4	0·04	0·001	1·11	0·023
Cl	14·09	0·397	11·25	0·317
NO_3	11·52	0·186	16·16	0·261
As salts per 100g. water				
KNO_3	29·6	...	48·5	...
$NaNO_3$	1·7	...	0·9	...
NaCl	11·2	...	32·1	...
Na_2SO_4	0·1	...	3·0	...
$CaCl_2$	3·5	...	...	...
$MgCl_2$	19·4	...	2·1	...
Sp. gr. at 27°	1·351	...	1·384	...

B. *Residue No. 4 from Dindigul Factory.*

—	Per cent.	Equivalents
K	1·10	0·028
NO_3	0·41	0·007

Fig. II.



C. *Purified nitre crystals.*

—	No. 3 Ordinary refined from Dindigul Factory		No. 6 Recrystallised, from Dindigul Factory	No. 8 Ordinary refined from Murungapet Factory
	Per cent.	Equivalents	Per cent.	Per cent.
Cl	12·18	0·346	0·62	6·90
NO_3	47·51	0·766	...	...
K	32·12	0·822	...	...

C. METHOD OF WORKING A FACTORY.

The annexed drawing Fig. II shows diagrammatically how a factory producing one ton of refined nitre per day could be worked according to the principles investigated in our experiments.

It is assumed that the crude nitre will yield 40 per cent. of refined potassium nitrate and that four extractions can be carried out daily. As the time required for one extraction is 2 to 2½ hours the work could be carried out by one shift of men.

Twelve to thirteen cwt. of the crude nitre is tipped into the vessel *A* which is steam jacketted and provided with a stirrer. Forty gallons of hot mother liquor from tank *B*, which is fitted with a steam coil, is added and the whole heated up to 100° and stirred for some 15 minutes after reaching this temperature. The mass is then run on to the filter C, which is a circular shallow tank fitted with a false bottom covered with filtering material. The space underneath the false bottom can be evacuated to about 500 mm. by means of a pump and contains steam coils for preventing crystallisation of the liquor. The filtrate runs down a pipe attached to a pump, D, which raises it to the evaporator or crystallisers.

These latter consist of iron drums of 22 gals. capacity. Before running in the liquor 1 gal. of the first wash water from the crystals is added to each drum to prevent the separation of common salt from the extract on cooling (cf. p. 205). A crystalliser of this size, when filled nearly full, will yield about 1 cwt. of crystals, so that allowing three days for cooling, 60 vessels would be required. It will be noticed that this allows 5 vessels for each cycle. When 40 gals. of liquor is used for digestion about 80 gals. of solution is obtained which will fill 4 drums. The remaining drum is required for liquors after concentration in evaporator *J*.

As soon as the level of the liquid in the filter *C* falls to the surface of the solid, 28 gals. of hot mother liquor from *B* is run into *C* and the process is repeated with the same quantity of hot liquor from the tanks *F* and *G* in succession. Tank *F* contains wash liquor No. 3 from a previous experiment, it is saturated with common salt, contains roughly 13 per cent. of potassium nitrate and at 80° has a sp. gr. 1·3. Similarly tank *G* contains wash liquor No. 4 from the previous experiment, contains 6 to 7 per cent. of potassium nitrate and has a sp. gr. 1·27 at 80°. *H* contains hot water of which 14 gals. are added when the liquor from *G* has drained to the surface of the solid. After this addition of water the residue is drained as completely as possible.

The main liquor in *D* has a specific gravity of about 1·52 measured hot. As soon as the wash liquors have displaced all the original concentrated liquors the specific gravity will fall, and when the figure 1·40 is reached, the liquor is no longer run to the crystallisers but is pumped to the evaporating pan *J*. When the specific gravity reaches 1·35 the liquor is run to the tank *F* and when it reaches 1·29 to the tank *G*. Although a liquor with specific gravity 1·40 to 1·35 deposits crystals on cooling the quantity obtained from a given bulk of solution is comparatively small and it is better to concentrate it. It will be seen that at each cycle of operations 14 gals. of water is used for washing the residues and as will be mentioned later 5 gals. are required for the crystals so that the total amount of water entering the factory is some 19 × 4 or 76 gals. per day. A certain quantity of water leaves the factory as moisture in the residues; the amount, assuming 8 per cent. of moisture is 24 gals. but this is almost balanced by the moisture in the crude material which amounts to 22 gals. for a 4 per cent. moisture content. A little water is lost by evaporation during the operations so that about 75 gals. per day will have to be boiled off. This can most conveniently be effected by boiling down the first hot wash liquor as mentioned above, together with the excess of mother liquor until the solution is almost saturated with respect to potassium nitrate and decanting from the sodium chloride into a crystalliser. The residue can be added to the crude nitre.

In order to maintain the total volume of mother liquor in the factory at the amount actually required for extracting crude nitre, it is necessary to evaporate a portion of it in *J*[1] in the same manner as the first hot wash liquor after use. As the liquid in *J* evaporates, sodium chloride separates and is removed by a perforated ladle and without being washed is added to the crude nitre in *A* for extraction. The

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[1] This evaporator should be of sufficient capacity to allow of the ready elimination of 75 gallons of water from the liquors during the course of the day (10 working hours).

mount of such salt would be about 250 lbs. per day and should be istributed fairly evenly between the four extractions made in *A* uring the course of the day. As the volume of liquor in *J* diminishes ore mother liquid is added until finally sufficient concentrated liquor .g. containing 180–200 grams of potassium nitrate per 100 grams of ater) has been produced to fill one of the crystallisers. The liquor is en strained into a crystalliser and treated in the ordinary manner. he mother liquor obtained from this crystalliser, after the removal of e deposited potassium nitrate crystals, will be extremely rich in sodium itrate and when added to the main mother liquor in *K* will tend to ring up the sodium nitrate content from 9 grams per 100 c.c. of water o a point nearer the 15 grams per 100 c.c.[1] necessary to prevent loss f potassium nitrate by conversion into potassium chloride during xtraction.

If the crystallisers are approximately of the size given, after unning off the mother liquor into a trough leading to a sump *K*, they ay be carried bodily to the centrifuge *L* and the crystals emptied nto it. After removing mother liquor, the crystals are washed uccessively with 1 gallon of liquid from each of the small vessels *M*, *N*, *O*, *P*, of about 2 gallons capacity. These liquors have been btained by washing previous batches of crystals and the specific gravities are respectively *M* = 1·30, *N* = 1·25 and *O* = 1·18, *P* contains water.

The first wash liquor is collected in the crystalliser which is eturned to its place. The second liquor is collected in *M*, the third n *N* and the fourth in *O*. These are then ready for use with the next batch of crystals.

On this system it will be seen that control of the operations is based on the use of measured volumes and on specific gravity determinations. This can be carried out by unskilled labour. If it is found that the specific gravity of the wash liquors is gradually rising, rather more water must be used for the final wash and *vice versa*.

If the average potassium nitrate content of the crude nitre differs very widely from the assumed figure of 40 per cent., it is advisable to alter the volumes of solutions used, but the figure given is suitable for crude nitres varying considerably from the mean.

If it is found that the specific gravity of the mother liquors is increasing unduly owing to accumulation of magnesium chloride, it

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[1] As there is usually potassium chloride in the crude nitre it will be an advantage if the value 15 grams per 100 c.c. of water is exceeded as then the excess sodium nitrate will react with the potassium chloride forming potassium nitrate until the equilibrium is reached.

will be necessary to eliminate this salt by evaporating portions of the mother liquor nearly to dryness, centrifuging and throwing away the liquor. The solid can be treated as crude nitre. It should never be necessary to throw away the whole mother liquor.

Such a factory could be run by hand power only, although a mechanical drive for the stirrer, vacuum pump and centrifuge would be desirable. A small steam boiler would be necessary, but the capital outlay on the whole factory would be small and the value should be more than rapaid by the increased efficiency of working and the greater purity of the product.

D. GENERAL SUMMARY.

1. The experiments described in this paper show that a high grade, crystallised potassium nitrate containing less than 0·3 per cent. of chloride, equivalent to 0·5 per cent. of sodium chloride, can be produced by a single crystallisation from crude nitres containing 50 or even 30 per cent. of potassium nitrate, whereas the products now manufactured by the single crystallisation process contain from 3 to 20 per cent. of common salt.

2. *Extraction.*—The best process appears to be to digest the crude nitre with the mother liquor from a previous operation and already saturated at room temperature with both potassium nitrate and sodium chloride. It is advisable to use about 25 per cent. more liquor than is required completely to dissolve the actual amount of potassium nitrate present. Good results can be obtained by using any temperature between 55 and 100° for digestion, but to reduce the crystallising vat capacity to a minimum the higher temperatures are advisable, although at these temperatures there is more tendency for double decomposition to take place between the potassium nitrate and the sodium chloride so that sodium nitrate goes into solution and solid potassium chloride remains behind.

For crude nitres containing respectively 50 and 30 per cent. of potassium nitrate the following are the amounts of liquor recommended per 100 lbs. of crude nitre.

Temperature.	55°	80°	90°	100°	
For 50 per cent. nitre	240	90	65	50	lbs.
	18	7	5	4	gals.
For 30 per cent. nitre	145	55	40	30	lbs.
	11	4	3	2	gals.

3. Double decomposition takes place between potassium nitrate and sodium sulphate resulting in the formation of sodium nitrate and potassium sulphate, and further complications arise owing to the formation of the double sulphate, glaserite, $3K_2 SO_4, Na_2 SO_4$. The amount of sodium sulphate present is usually relatively small, although Leather mentions samples of crude nitre containing as much as 30 per cent. The possibility of the separation of sodium sulphate with the potassium nitrate crystals is minimised by the fact that in saturated potassium nitrate and sodium chloride solutions sodium sulphate is more soluble at 25° than at 80 to 100°.

4. When the same liquor is repeatedly used for extraction an equilibrium is finally reached when the amount of sodium nitrate in solution is about 15 grams per 100 grams of water. When that stage is reached digestion of new batches of crude nitre with the liquor will produce no double decomposition between the potassium nitrate and sodium chloride. From the point of view therefore of minimising loss of potassium nitrate it is advisable to use the same liquor for purposes of extraction as long as possible.

If, as stated by Leather, in actual practice the liquor is frequently renewed, e.g., after only a few crystallisations, then each digestion entails loss of potassium in the form of potassium chloride left behind in the residue. In many of the crude nitres potassium chloride is already present and this probably owes its origin to double decomposition which took place during the manufacture of the crude product.

The factor, which in most cases will determine the limit to the use of the extraction liquor, will probably be the magnesium content of the crude nitre. If much magnesium chloride is present this may tend to accumulate in the liquor and contaminate the refined crystals.

5. *Filtration.*—To separate the liquor from the residue which consists largely of common salt some method of suction or pressure filtration is advisable and a process of suction filtration suitable for dealing with several hundredweight of crude nitre is described.

6. *Treatment of residue.*—As the residue contains appreciable amounts of nitrate some method of washing is necessary before this residue is discarded. Three washings, two with hot saturated solutions of common salt containing potassium nitrate and of specific gravity 1·32 and 1·27 measured hot and one with hot water have been found sufficient to remove 99 per cent. of the potassium nitrate.

7. *Crystallisation.*—Hot saturated solutions of common salt containing potassium nitrate deposit sodium chloride as well as

potassium nitrate on cooling. This is not due, as has been supposed to evaporation of the liquid, although if the liquid does evaporate still more sodium chloride is deposited. To prevent this deposition it is necessary to add water, or better a wash liquor from the crystals and containing comparatively little chloride, to the solution before it is allowed to cool.

It has been found that large crystals are easier to free from mother liquor than very fine ones consequently it is not desirable to stir during cooling.

8. *Refined nitre crystals.*—These are best removed from the mother liquor by centrifuging. If they are washed with three liquors containing diminishing amounts of sodium chloride and finally with water the sodium chloride content can be reduced to less than 0·5 per cent. About 85 per cent. of the total potassium nitrate can be obtained as crystals in one operation, and the remainder has to be recovered by concentration.

9. *Evaporation.*—Although it is desirable to reduce the amount of evaporation to a minimum in order to save fuel, a certain amount is necessary if any process of washing is used. In the method of working we propose the evaporation necessary amounts to about 75 gals. of water per ton of refined nitre. This could be reduced somewhat, if considered desirable, by the sacrifice of some of the potassium nitrate in the raw material, and by diminishing the purity of the final product.

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