UNITED STATES DEPARTMENT OF THE INTERIOR

U.S. GEOLOGICAL SURVEY

An X-ray Diffraction Method for Semiquantitative Mineralogical Analysis of

Chilean Nitrate Ore

by

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Open-File Report 94-240

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ABSTRACT

Computer processing of digital X-ray diffraction (XRD) data provide a simple method for determining the semiquantitative mineralogical composition of naturally occurring mixtures of saline minerals. The method herein described was adapted from a computer program by Hosterman and Dulong (1989) for the study of mixtures of naturally occurring clay minerals. The program determines the minerals present in a sample based on a user supplied library of mineral standards, and estimates their relative abundances based on chemical and experimentaly derived factors. The method requires precise calibration of XRD data for the minerals to be studied and selection of diffraction peaks that minimize inter-compound interferences. Results about mineralogical composition for selected saline minerals show good reliability with an overall precision of ± 1 % and an overall relative accuracy of ± 1.5 % based upon absolute differences.

INTRODUCTION

A computer program for determining the mineralogical composition of mixtures of clay minerals (Hosterman and Dulong, 1989) was adapted for the purpose of determining the mineralogical composition of Chilean nitrate ores. This paper details the laboratory study of mixtures of selected synthetic and natural saline minerals, which are among the most common constituents of the Chilean nitrate ores, to determine the percentages of each mineral in such deposits. These ores are complex both mineralogically (Table 1) and chemically (Table 2). The phases used to develop the mineral library used by the computer program were synthetic,

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reagent-grade nitratine [$NaNO_3$], halite [$NaCl$], and niter [KNO_3] and high-purity, natural darapskite [$Na_3(SO_4)(NO_3) \cdot H_2O$], bloedite [$Na_2Mg(SO_4)_2 \cdot 4H_2O$], and gypsum [$CaSO_4 \cdot 2H_2O$], which represent the three major anions--NO_3^-, Cl^-, and SO_4^---in the saline minerals of the Chilean nitrate deposits (Tables 1 and 2). Several other minerals known to be abundant locally such as anhydrite, glauberite, and humberstonite (Table 1) were not included in this study.

By far the most abundant minerals in typical nitrate ore are nitratine and halite. Each of the sulfate minerals listed in table 1 may be dominant locally, but typical ore generally contains two or more sulfate minerals, none of which are as abundant as either nitratine or halite even though the total SO_4 might be greater than either NO_3 or Cl (Table 2). In addition, the typical nitrate deposits contain trace amounts, generally less than 0.1 percent, of the anions IO_3^-, CrO_4^-, ClO_4^- and several borate minerals (Tables 1 and 2). In order for the program to identify the phases in a sample, as well as estimate their relative abundances, careful attention was paid to interference among the diffraction peaks of the various saline minerals and the effect of such interferences on the reliability of the method.

The X-ray diffraction method for semiquantitative mineral analysis of saline mixtures, herein described, was developed for the purpose of obtaining accurate data about mineral composition for direct comparison with chemical analyses of the same mixtures. The method is applicable to natural saline mixtures, which because of incongruent solubility of some mineral componants, cannot be reconstituted by recrystallization from solutions of these mixtures. The relationship between mineralogical and chemical composition is essential to understanding the solution chemistry of complex saline deposits and the development of models for the phase systems in which the nitrate ores formed. Although several partial phase systems involving the major anions of the ores have been studied, no effort has been made to develop an overall model for the deposits such as that for oceanic saline deposits that was first defined on the basis of experimental data by Van't Hoff (see Braitsch, 1971). Such a model is not only important to a better understanding of the nitrate deposits but also would have direct application to the beneficiation of nitrate ores.

SAMPLE PREPARATION

Three groups of samples were prepared for XRD analysis: (1) pure minerals to calibrate for mineral identification (Table 3a), (2) mixtures of known composition analyzed for semiquantitative calibration (Table 3b), and (3) mixtures of known composition analyzed as unknowns (Table 4). Samples were prepared from pure mineral compounds which were dried in an oven at 50°C for 4 hours and then pulverized in an agate mortar and sieved to <200 mesh. Mixtures were prepared by weighing predetermined amounts of the pure mineral compounds to an accuracy of ±0.0005 g, and then mixing these components first in an agate mortar and then in a Spex mill. Two pressed pellets for XRD analysis were made for each sample on a backing of 50 percent each of boric acid and methyl cellulose using a hydraulic press. Each pellet was analyzed once, accounting for two analyses for each sample,

for all but two samples (HAL251, MIX600). To test more accurately for precision, each of two pellets for samples HAL251 and MIX600 was analyzed 5 times, for a total of 10 analyses for each sample (Table 5). The remaining mineral mixtures (Table 4) were analyzed as unknowns and shown as runs 1 and 2 in table 6. Semiquantitative XRD data for calibration mixtures listed in table 3b are shown in table 7.

The XRD analyses were made with a Diano computer-automated X-ray diffraction unit, with a spinning sample holder, at a goniometer speed of 1°/minute. The digitized X-ray data were collected on an IBM PC every 0.02° 2θ, starting at 3.00° 2θ and ending at 60.00° 2θ. Copper K-alpha radiation was used in conjunction with the following optical settings: a 1° soller slit, a 0.1° detector slit, a graphite monochronometer, and a 2° take-off angle.

THE COMPUTER PROGRAM

The computer processing of the X-ray data, which is described in detail by Hosterman and Dulong (1989), is based on algorithmic calculation of the digitized X-ray data representing the diffraction pattern. Briefly, the program smooths the digitized data, determines and subtracts background, identifies and locates peak position and integrates peak intensity, qualitatively identifies the mineral associated with the peaks, and estimates weight percentages of each mineral present.

The user must supply both a sample file containing a digitized X-ray diffraction pattern of an unknown mineral mixture, and a library file consisting of X-ray diffraction data for the minerals likely to be encountered in the sample (Table 8). We have utilized only 6 of the major minerals present in nitrate ore in developing the method--a more comprehensive study of the complex saline ores such as the Chilean nitrate ores will require a library file containing all the minerals likely to occur in amounts detectable by X-ray diffraction. The output of the computer program is a report sheet consisting of 2θ and d-spacing values, relative intensities for each peak, the minerals determined to be present, and estimates of the relative weight percentages of each of these minerals.

The library file utilized in this study (Table 8) contains selected XRD data for the six minerals used to develop and test the method and instrument settings as defined by Hosterman and Dulong (1989). All variables shown on this library file were used for this study and are arranged precisely as required by the program to function, any deviation from this format may cause error. Important variables include the window of acceptibility, the multiplication factor, and the number of major peaks to be used for mineral identification. The window of acceptibility is adjusted to compensate for instrumental precision error by defining the number of degrees 2θ from a peak that the program will search for a positive match for that peak. This is essential for defining major peaks that are in close proximity to each other. The multiplication factor or strength factor along with the mass absorption coefficient for a specific phase are essential to the least-squares calculation of

semiquantitative mineral composition. The strength factor is experimentally derived so that the mineral percentages calculated by the program reflect the known values of the calibration standard for that mineral. The user may choose up to 3 major peaks for mineral identification; however, for the minerals studies here, peaks of sufficiently unique d-spacings were available so that only one peak was needed to identify each mineral.

For this study, the calibration standards used to determine the strength factors are HAL250S, NIT205S, DKT205S, GYP205S, and BLD205S (Tables 3b and 7). Sample HAL250S was prepared as a mixture of 50 percent each of $NaNO_3$ and NaCl, representing nitratine and halite; the other samples were prepared as mixtures of 95 percent $NaNO_3$ and 5 percent of the mineral being standardized. In this way, the calibration assured the greatest accuracy at the 5 percent level, relative to nitratine, for niter, darapskite, gypsum, and bloedite, which commonly are present in low concentrations in the Chilean nitrate ores, and at the 50 percent level for halite, which together with nitratine are generally the most abundant constituents of the nitrate ores.

Pure mineral standards for the six minerals used in this study were analyzed twice each to determine d-spacing and peak intensities used in the library file (report sheets for these analyses are shown in table 9). The resulting XRD data were computer adjusted to a known peak selected from published X-ray data for the given mineral (JCPDS 1986) to compensate for peak displacement. The 2θ values representing the specific peaks used for this adjustment are shown in tables 7 and 9. The computer generated d-spacing and intensity data of the pure mineral standards (Table 9) were then compared with published values (JCPDS 1986), and those peaks that were consistently resolved and identified by the computer program were matched with published d-spacings, and these published values were entered into the computer library file (Table 8). We note here that even though the JCPDS d-spacings used in table 8 and the experimental d-spacings shown in table 9 are generally slightly different, they represent the same respective peaks. The averages of our measured intensity ratios for the two analyses of each mineral were also entered into the library file. The measured intensities were used, rather than published intensities, to compensate for the effects of sample orientation in the pressed powder mounts that could have the effects of enhancing or diminishing peak intensities.

DISCUSSION OF RESULTS

Nitratine. Because nitratine is the most abundant and economically important nitrate mineral in the Chilean nitrate deposits, reagent-grade $NaNO_3$ was used as the primary component of the mixtures used in this study. The analyses of all the samples containing nitratine were computer adjusted to the strongest (100) peak at 2θ = 29.45° (d = 3.03 Å). This peak was also used as the primary identification peak for nitratine. Analyses of seventeen samples containing nitratine as a major

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componant showed an average absolute difference of ± 1.3 % between the actual weighed and computer calculated concentrations of nitratine (Table 10).

Halite. Along with nitratine, halite is generally the most abundant saline mineral in the typical Chilean nitrate ores. To reflect this abundance, reagent-grade NaCl was mixed in equal proportions with $NaNO_3$ for all mixtures containing 3 or more mineral constituents. The XRD analysis of pure NaCl was computer adjusted to the strongest (100) peak at 2θ = 31.70° (d = 2.82 Å). However, because for mixtures of halite and nitratine the computer program could not distinguish between this peak and an overlapping strong peak for nitratine, the next strongest peak for halite at 2θ = 45.45° (d = 1.994 Å) was used as the primary identification peak for halite. This peak does not interfere significantly with any of the strong peaks of the other minerals studied. Semiquantitative XRD analyses of halite in thirteen samples showed an average absolute difference of ± 1.3 % between the actual weighed and computer calculated concentrations of halite (Table 10).

Niter. Among the saline minerals occurring in minor amounts in Chilean nitrate ores, niter is important economically because it is a source of both nitrate and potassium, the latter being utilized in the preparation of a potassium-rich nitrate product that commands a premium price as a fertilizer. However, although niter is widespread in Chilean nitrate ores, it does not occur in all ores, and where it does occur, the amount rarely exceeds 5 percent of the total saline material present. ·

Reagent-grade KNO_3 was used for this study, and the XRD analyses of the pure material were computer adjusted to the strong peak at 2θ = 29.45° (d = 3.03 Å). However, this same peak is the strongest peak for nitratine, and the strongest peak for niter at 2θ = 23.53° (d = 3.78 Å) is interfered with by nearby peaks of moderate intensity for bloedite and gypsum, and was not resolved by the computer if either of these minerals were present in mixtures containing 5 percent niter. Consequently, the nearby peak at 2θ = 23.81° (d = 3.73 Å), having an I/I° = 17, was used as the primary identification peak for niter.

Because of their close proximity, a concern with the identification of niter was whether the computer program would accurately and precisely resolve the peaks at d = 3.73 Å and d = 3.78 Å. It was found that the two peaks were resolved down to the 5 percent level for niter in mixtures, but were not resolved in a mixture containing 1 percent niter and 99 percent nitratine (these results not shown). However, it was found that by decreasing the sampling interval from 0.02° to 0.01° 2θ, the program did resolve these two peaks. Obviously, a slower goniometer speed would be desirable for XRD analyses of minor minerals in mixtures, but such a slower speed would lengthen the time required for an analysis and was not deemed necessary for this preliminary study. Semiquantitative XRD analyses for niter in four samples showed an average absolute difference of ± 2.3 % between the actual weighed and computer calculated concentrations of niter (Table 10).

Darapskite. The nitrate-sulfate mineral darapskite is a widespread, relatively minor but locally abundant constituent of the Chilean nitrate ores. It is one of the most abundant sulfate-bearing minerals because it is stable in solutions from which $NaNO_3$ is precipitating (Ericksen and Mrose, 1970), whereas thenardite [Na_2SO_4], which is abundant in nearly all other continental saline deposits, is not stable in solutions from which $NaNO_3$ is precipitating. Darapskite also presents a problem in beneficiation of nitrate ore because separation of pure $NaNO_3$ requires close control of solution chemistry to inhibit precipitation of darapskite, which is an undesirable contaminant.

The darapskite used in this study is natural crystalline material from the Chilean nitrate deposits. The XRD analyses of pure darapskite were computer adjusted to the relatively minor peak at 2θ = 28.96° (d = 3.08 Å), which was chosen because of several peaks tested, this peak gave the most consistent results, perhaps because it is near the mid-point in the XRD pattern. Nevertheless, the primary identification peak selected for darapskite was the strongest (100) peak at 2θ = 8.58° (d = 10.3 Å). This peak does not interfere with any peaks of the other minerals studied. In an earlier study, it was shown that in mixtures of halite and darapskite, darapskite in amounts of as little as 0.25 percent could be identified in X-ray diffraction patterns (Ericksen and Mrose, 1970). Semiquantitative XRD analyses of three darapskite-bearing mixtures showed an average absolute difference of ± 1.0 % between the actual weighed and computer calculated concentrations of darapskite (Table 10).

Bloedite. Bloedite is a widespread minor, but locally abundant sulfate mineral in the Chilean nitrate deposits. It is one of the principal sources of sulfate and magnesium in nitrate ore (Table 2). The bloedite used in this study consists of pure crystalline material from a 10-15 cm wide vein in the oxide zone of a small silver and base-metal deposit near the town of Sierra Gorda, northern Chile. The XRD analyses of pure bloedite were computer adjusted to the moderately strong peak at 2θ = 20.71° (d = 4.28 Å), whereas the strong (95) peak at 2θ = 19.47 (d = 4.555 Å) was selected as the primary identification peak. A relatively narrow window of acceptability was entered in the library file (Table 8) because of the close proximity of a niter peak at d = 4.58 Å. However, because of this narrow window of acceptibility, a lower percentage of peaks were identified by the computer program in samples containing bloedite than in samples that did not. Semiquantitative XRD analyses of four mixtures containing bloedite show an average absolute difference of ± 1.8 % between the actual weighed and computer calculated concentrations of bloedite.

Gypsum. The routine chemical analyses of Chilean nitrate ores by companies that beneficiate the ores generally report only the saline constituents that are dissolved by hot water. As a consequence, concentrations of relatively insoluble minerals in nitrate ore such as gypsum and anhydrite are reported incompletely by these chemical analyses. Inasmuch as gypsum and anhydrite are among the most widespread and abundant saline minerals in the nitrate deposits, chemical analyses

requiring dissolution by hot water are misleading in terms of both chemical and mineralogical composition. Obviously, XRD analyses offer a relatively simple method for determining the concentrations of such minerals.

The gypsum used in this study is from a naturally occurring large crystal of selenite from Minas Gerais, Brazil. The XRD analyses of this pure gypsum were computer adjusted to the strong (100) peak at 2θ = 11.58° (d = 7.63 Å). This peak was also used as the primary identification peak for gypsum because it did not interfere with peaks of any other minerals studied. The average absolute difference between the actual weighed and computer calculated concentrations of gypsum in four mixtures analyzed semiquantitatively by XRD was ± 1.3 %.

CONCLUSIONS

Semiquantitative mineralogical analysis by computerized X-ray diffraction is a relatively rapid method for determining the concentration of saline minerals in mixtures. The method shows good reliability with an overall precision of better than ± 1 % (1 sigma) and an overall relative accuracy of ± 1.5 % (average for 6 minerals shown in Table 10). The lower limit of concentrations studied were 5 percent, but this can be effectively reduced by decreasing the computer sampling interval.

A primary limiting factor for this semiquantitative method of mineral analysis is peak overlap, which can be minimized in the following ways: (1) the window of acceptibility in the computer library file can be adjusted to better resolve peaks in close proximity; and (2) integration of as many peaks as possible minimizes the net effect that overlapping peaks will have on the semiquantitative determinations. For mineral identification, however, it is recommended that a single peak be used if possible. This peak does not necessarily have to be the strongest peak for a given mineral, but it must be free of interfering peaks of other minerals present, and must have sufficient intensity to be resolved at the lower limit of detectibility for the specific mineral.

The relationship between relative intensity (I/I°) of a given peak for a mineral in a series of mixtures and its actual concentration in those mixtures is not necessarily a straight-line constant, particularly in complex mineral mixtures. Therefore, it is not practical to construct a simple linear regression to predict actual concentrations for a mineral based on relative intensity for a single peak.

The investigation on which this report is based is preliminary and was for the primary purposes of defining and testing the method, which consequently requires refinement for application to naturally occurring saline complexes. Inasmuch as the relative accuracy of the analyses decreases with the amount of mineral present in the sample, it is necessary that the method be calibrated for still lower concentrations, and a detection limit be determined for each of the minerals to be studied.

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Table 1.-- Saline minerals in the Chilean nitrate deposits
      (after Ericksen, in press).

HALIDES

5-628   Halite, $NaCl$

NITRATES

7-271   Nitratine, $NaNO_3$
5-377   Niter, KNO_3
23-1408   Darapskite, $Na_3(SO_4)(NO_3) \cdot H_2O$
21-682   Humberstonite, $K_3Na_7Mg_2(SO_4)_6(NO_3)_2 \cdot 6H_2O$

SULFATES

5-631   Thenardite, Na_2SO_4
11-647   Mirabilite, $Na_2SO_4 \cdot 10H_2O$
19-1187   Glauberite, $Na_2Ca(SO_4)_2$
19-1215   Bloedite, $Na_2Mg(SO_4)_2 \cdot 4H_2O$
33-882   Kieserite, $MgSO_4 \cdot H_2O$
8-467   Epsomite, $MgSO_4 \cdot 7H_2O$
33-311   Gypsum, $CaSO_4 \cdot 2H_2O$
6-226   Anhydrite, $CaSO_4$

IODATES AND CHROMATES

28-221   Lautarite, $Ca(IO_3)_2$
26-1405   Brüggenite, $Ca(IO_3)_2 \cdot H_2O$
   *   Hectorfloresite, $Na_9(IO_3)(SO_4)_4$
25-132   Dietzeite, $Ca_2(IO_3)_2(CrO_4)$
15-365   Tarapacaite, K_2CrO_4
27-380   Lopezite, $K_2Cr_2O_7$

BORATES

12-419   Ulexite, $NaCaB_5O_6(OH)_6 \cdot 5H_2O$
12-420   Probertite, $NaCaB_5O_7(OH)_4 \cdot 3H_2O$
8-116   Ginorite, $Ca_2B_{14}O_{23} \cdot 8H_2O$
35-646   Hydroboracite, $CaMg(B_6O_8(OH)_6)_2 \cdot 3H_2O$
18-669   Kaliborite, $KHMg_2B_{12}O_{16}(OH)_{10} \cdot 4H_2O$
   **   Iquiqueite, $Na_4K_3Mg(CrO_4)B_{24}O_{39}(OH) \cdot 12H_2O$

*   Ericksen and others (1989)
**   Ericksen and others (1986)

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Table 2.-- Chemical composition (wt.%) of water-soluble
        saline constituents in typical nitrate ores and
        high-purity veins of the Chilean nitrate deposits.
        From Ericksen (1981).

	1	2	3	4	5	6
NO_3	6.35	6.66	12.11	17.67	72.15	32.63
Cl	3.87	7.07	7.15	11.11	.07	21.84
SO_4	6.6	12.43	14.23	3.47	.10	8.30
IO_3	.061	.068	.05	.004	---	.04
ClO_4	.035	.027	.02	---	.04	.25
BO_3	.54	.92	.13	.11	---	.27
Na	6.2	8.9	9.2	13.8	26.7	26
K	.56	.61	.46	.95	---	.32
Mg	.15	.73	1.06	---	.01	.93
Ca	1.15	2.27	1.61	1.06	.05	.32
H_2O	1.74	1.08	1.03	1.87	1.52	3.4
Insoluble	72	59	47	50	0	4

(1) Average grade of 5.97 million metric tons of ore
treated at Oficina Pedro de Valdivia, July 1, 1935-June 30,
1936.

(2) Average grade of 6.27 million metric tons of ore
treated at Oficina Maria Elena, July 1, 1951-June 30, 1952.

(3) Average grade of hand-selected nitrate ore treated by
Oficina Alemania during the month of February, 1951.

(4) Sample of high-grade nitrate ore from Oficina
Humberstone.

(5) High-purity $NaNO_3$ ("caliche blanco") vein at Oficina
Lautaro.

(6) High-purity "caliche blanco" vein at Oficina Santa Fé.

Table 3.--Minerals and mineral mixtures utilized for calibration: (a) pure synthetic and natural minerals used to calibrate for mineral identification, and (b) mineral mixtures used as semiquantitative calibration standards.

Table 3a.

Sample Number	Mineral calibrated
NAN100	NITRATINE
HAL100	HALITE
NIT100	NITER
DKT100	DARAPSKITE
GYP100	GYPSUM
BLD100	BLOEDITE

Table 3b.

Sample Number	Mineral calibrated	Composition (wt %)
HAL250S	HALITE	50% HALITE, 50% NITRATINE
NIT205S	NITER	5% NITER, 95% NITRATINE
DKT205S	DARAPSKITE	5% DARAPSKITE, 95% NITRATINE
GYP205S	GYPSUM	5% GYPSUM, 95% NITRATINE
BLD205S	BLOEDITE	5% BLOEDITE, 95% NITRATINE

Table 4.-- Mineral mixtures (wt %) analyzed to test accuracy and precision of semiquantitative X-ray diffraction method.

TWO PHASE MIXTURES

SAMPLE NUMBER	ACTUAL COMPOSTION
HAL205	5% HALITE, 95% NITRATINE
HAL210	10% HALITE, 90% NITRATINE
HAL220	20% HALITE, 80% NITRATINE
HAL240	40% HALITE, 60% NITRATINE
HAL251	50% HALITE, 50% NITRATINE
NIT210	10% NITER, 90% NITRATINE
DKT210	10% DARAPSKITE, 90% NITRATINE
GYP210	10% GYPSUM, 90% NITRATINE
BLD210	10% BLOEDITE, 90% NITRATINE

THREE PHASE MIXTURES

NIT305	5% NITER, 47.5% NITRATINE, 47.5% HALITE
NIT310	10% NITER, 45% NITRATINE, 45% HALITE
DKT305	5% DARAPSKITE, 47.5% NITRATINE, 47.5 % HALITE
GYP305	5% GYPSUM, 47.5% NITRATINE, 47.5% HALITE
GYP310	10% GYPSUM, 45% NITRATINE, 45% HALITE
BLD305	5% BLOEDITE, 47.5% NITRATINE, 47.5% HALITE
BLD310	10% BLOEDITE, 45% NITRATINE, 45% HALITE

SIX PHASE MIXTURE

MIX600	32.5% NITRATINE, 32.5% HALITE, 10% NITER, 10% GYPSUM, 5% DARAPSKITE, 10% BLOEDITE

Table 5.-- Tests for reliability of computer calculated mineral compositions based on X-ray diffraction analyses of a 2-phase and a 6-phase mineral mixture. Five determinations were made on each of two sample aliquots (pellets 1 and 2). All values are in wt. percent.

Sample: MIX600

	analysis number	Mineral (actual wt %)					
		Nitratine (32.5 %)	Halite (32.5 %)	Niter (10 %)	Darapskite (5 %)	Gypsum (10 %)	Bloedite (10 %)
pellet 1	1	31	29	13	7	10	11
	2	29	29	15	7	10	11
	3	31	29	11	7	10	12
	4	31	29	13	7	9	11
	5	30	28	15	7	9	11
pellet 2	6	33	28	12	7	8	11
	7	32	29	11	7	9	12
	8	30	29	15	7	8	11
	9	31	30	12	7	8	12
	10	31	30	12	7	8	12
	average	31	29	13	7	9	11
	high	33	30	15	7	10	12
	low	29	28	11	7	8	11
	range	4	2	4	0	2	1
	std. dev.	1.1	0.7	1.6	0	0.9	0.5
	absolute[1] difference	1.5%	3.5%	3%	2%	1%	1%

Sample: HAL 251

	analysis number	Mineral (actual wt %)	
		Nitraline (50%)	Halite (50%)
pellet 1	1	51	49
	2	51	49
	3	50	50
	4	50	49
	5	49	51
pellet 2	6	48	52
	7	48	52
	8	50	50
	9	50	50
	10	49	51
	average	50	50
	high	51	52
	low	48	49
	range	3	3
	std dev	1.1	1.2
	absolute difference	0 %	0 %

(1) Difference between the average composition as determined by x-ray diffraction and the actual composition of the mixture.

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Table 6.--Test for reliability of computer calculated mineral compositions based on X-ray diffraction analyses of known 2 and 3 phase mineral mixtures. Two aliquots of each sample was analyzed (runs 1 and 2). [AVG, average of two analyses; ACT, actual mineralogical composition; DIFF, absolute difference between average of two analyses and actual composition].

Minerals Present (wt. %)

SAMPLE NUMBER	NITRATINE					HALITE					NITER				
	RUN1	RUN 2	AVG	ACT	DIFF	RUN 1	RUN 2	AVG	ACT	DIFF	RUN 1	RUN 2	AVG	ACT	DIFF
HAL205	93	93	93	95	2	7	7	7	5	2	0	0	0	0	0
HAL210	89	89	89	90	1	11	11	11	10	1	0	0	0	0	0
HAL220	77	77	77	80	3	22	23	23	20	3	0	0	0	0	0
HAL240	57	58	58	60	2	43	42	43	40	3	0	0	0	0	0
NIT210	89	88	89	90	1	0	0	0	0	0	11	11	11	10	1
NIT305	47	47	47	47.5	0.5	47	47	47	47.5	0.5	6	6	6	5	1
NIT310	40	43	42	45	3	45	43	44	45	1	14	14	14	10	4
DKT210	90	91	91	90	1	0	0	0	0	0.	0	0	0	0	0
DKT305	46	45	48	47.5	1 5	48	49	49	47.5	1 5	0	0	0	0	0
GYP210	90	90	90	90	0	0	0	0	0	0	0	0	0	0	0
GYP305	47	46	47	47.5	0 5	47	48	48	47.5	0.5	0	0	0	0	0
GYP310	44	43	44	45	1	44	45	45	45	0	0	0	0	0	0
BLD210	91	90	91	90	1	0	0	0	0	0	0	0	0	0	0
BLD305	46	47	47	47.5	0.5	47	47	47	47.5	0.5	0	0	0	0	0
BLD310	42	42	42	45	3	44	45	45	45	0	0	0	0	0	0

SAMPLE NUMBER	DARAPSKITE					GYPSUM					BLOEDITE				
	RUN 1	RUN 2	AVG	ACT	DIFF	RUN1	RUN2	AVG	ACT	DIFF	RUN 1	RUN 2	AVG	ACT	DIFF
HAL205	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HAL210	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HAL220	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
HAL240	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NIT210	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NIT305	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
NIT310	0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
DKT210	10	9	10	10	0	0	0	0	0	0	0	0	0	0	0
DKT305	6	6	6	5	1	0	0	0	0	0	0	0	0	0	0
GYP210	0	0	0	0	0	10	11	11	10	1	0	0	0	0	0
GYP305	0	0	0	0	0	6	8	6	5	1	0	0	0	0	0
GYP310	0	0	0	0	0	12	11	12	10	2	0	0	0	0	0
BLD210	0	0	0	0	0	0	0	0	0	0	9	10	10	10	0
BLD305	0	0	0	0	0	0	0	0	0	0	7	6	7	5	2
BLD310	0	0	0	0	0	0	0	0	0	0.	14	13	14	10	4

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Table 7. -- Semiquantitative X-ray diffraction data for 5 prepared mixtures of saline minerals listed in table 3b. These results are the averages of analyses of two sample aliquots.

Standard Mixture HAL250S[1]

Peak #	d	I	I/I°	2θ	Index[2]
1	3.896	293	0.6	22.80	4
2	3.253	1152	2.5	27.40	3
3	3.030	22119	48.1	29.45	4
4	2.813	45953	100.0	31.79	3 4
5	2.527	447	1.0	35.49	4
6	2.303	1511	3.3	39.08	4
7	2.117	656	1.4	42.68	4
8	1.987	9645	21.0	45.62	3
9	1.940	297	0.6	46.79	not identified
10	1.889	3847	8.4	48.14	4
11	1.873	691	1.5	48.56	4
12	1.692	373	0.8	54.16	3
13	1.645	416	0.9	55.85	4
14	1.622	2687	5.8	56.72	3 4

Composition determined by computer program:

		Index
Halite	50.0%	3
Nitratine	50.0%	4

(1) XRD pattern has been adjusted to 2θ = 29.45° by reducing by 0.034° 2θ; 13 peaks have been identified out of 14 found, which accounts for 99.7 percent of the total measured intensity.

(2) Index number denotes order in which minerals are listed in the library file (Table 7).

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Table 7. (cont.)

Standard Mixture NIT205S[3]

Peak #	d	I	I/I°	2θ	Index	
1	3.889	1457	2.4	22.84	4	
2	3.768	1149	1.9	23.59	6	
3	3.727	326	0.5	23.85	6	
4	3.030	60300	100.0	29.45	4	6
5	2.797	12087	20.0	31.97	4	
6	2.758	220	0.4	32.43	6	
7	2.640	809	1.3	33.92	6	
8	2.625	153	0.3	34.12	not identified	
9	2.528	2011	3.3	35.47	4	
10	2.302	7588	12.6	39.10	4	6
11	2.187	299	0.5	41.25	6	
12	2.150	183	0.3	41.98	6	
13	2.117	2594	4.3	42.68	4	
14	1.938	1519	2.5	46.83	6	
15	1.889	12674	21.0	48.12	4	6
16	1.873	2809	4.7	48.56	4	6
17	1.645	1657	2.7	55.85	4	
18	1.622	1308	2.2	56.72	4	

Composition determined by computer program:

		Index
Nitratine	95.1%	4
Niter	4.9%	6

(3) XRD pattern has been adjusted to 2θ = 29.45° by reducing by 0.014° 2θ; 17 peaks have been identified out of 18 found, which accounts for 99.9 percent of the total measured intensity.

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Table 7. (cont.)

Standard Mixture DKT205S[4]

Peak #	d	I	I/I°	2θ	Index	
1	10.438	2860	4.9	8.46	1	
2	5.929	406	0.7	14.93	not identified	
3	4.131	445	0.8	21.49	1	
4	3.886	2022	3.5	22.86	3	
5	3.517	699	1.2	25.30	1	
6	3.455	411	0.7	25.76	1	
7	3.186	1161	2.0	27.98	1	
8	3.030	58259	100.0	29.45	3	
9	2.860	976	1.7	31.24	1	
10	2.795	13679	23.5	31.99	3	
11	2.567	429	0.7	34.93	1	
12	2.528	2814	4.8	35.47	1	3
13	2.301	9179	15.8	39.12	3	
14	2.118	3163	5.4	42.66	3	
15	2.056	228	0.4	44.01	1	
16	1.938	1512	2.6	46.83	1	
17	1.889	15749	27.0	48.14	3	
18	1.873	3226	5.5	48.56	3	
19	1.644	1967	3.4	55.87	3	
20	1.621	1591	2.7	56.74	1	3

Composition determined by computer program:

		Index
Nitratine	95.0%	3
Darapskite	5.0%	1

(4) XRD pattern has been adjusted to 2θ = 29.45° by reducing by 0.014° 2θ; 19 peaks have been identified out of 20 found, which accounts for 99.7 percent of the total measured intensity.

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Table 7. (cont.)

Standard Mixture GYP205S[5]

Peak #	d	I	I/I°	2θ	Index	
1	7.645	4408	7.5	11.57	7	
2	5.913	489	0.8	14.97	not identified	
3	4.285	1074	1.8	20.71	7	
4	3.893	1506	2.6	22.82	4	
5	3.797	756	1.3	23.41	7	
6	3.030	58392	100.0	29.45	4	
7	2.868	471	0.8	31.16	7	
8	2.797	15456	26.5	31.97	7	4
9	2.676	146	0.3	33.46	7	
10	2.528	1888	3.2	35.47	4	
11	2.303	7144	12.2	39.08	4	
12	2.117	2492	4.3	42.68	4	
13	1.940	974	1.7	46.79	not identified	
14	1.889	16998	29.1	48.14	7	4
15	1.873	3014	5.2	48.56	7	4
16	1.645	1601	2.7	55.85	7	4
17	1.622	1415	2.4	56.72	7	4

Composition determined by computer program:

		Index
Nitratine	95.0%	4
Gypsum	5.0%	7

(5) XRD pattern has been adjusted to 2θ = 29.45° by addition of 0.006° 2θ; 15 peaks have been identified out of 17 found, which accounts for 98.8 percent of the total measured intensity.

Table 7. (cont.)

Standard Mixture BLD205S[6]

Peak #	d	I	I/I°	2θ	Index
1	6.010	562	0.9	14.73	not identified
2	5.866	181	0.3	15.09	not identified
3	4.548	1236	2.0	19.50	6
4	4.269	234	0.4	20.79	6
5	3.889	1676	2.7	22.84	3
6	3.791	203	0.3	23.45	6
7	3.276	1355	2.2	27.19	not identified
8	3.243	302	0.5	27.48	6
9	3.030	61854	100.0	29.45	3
10	2.961	415	0.7	30.15	not identified
11	2.795	16208	26.2	31.99	3
12	2.725	178	0.3	32.83	6
13	2.643	290	0.5	33.88	6
14	2.527	2081	3.4	35.49	3
15	2.302	8294	13.4	39.10	3 6
16	2.268	171	0.3	39.70	6
17	2.117	3064	5.0	42.68	3 6
18	1.938	1697	2.7	46.83	not identified
19	1.888	17363	28.1	48.16	3
20	1.873	3061	4.9	48.56	3
21	1.855	245	0.4	49.07	6
22	1.777	252	0.4	51.36	6
23	1.661	176	0.3	55.25	6
24	1.644	1838	3.0	55.87	3
25	1.621	1466	2.4	56.74	3
26	1.602	224	0.4	57.48	6
27	1.585	63	0.1	58.15	not identified
28	1.581	58	0.1	58.33	not identified

Composition determined by computer program:

		Index
Nitratine	95.1%	3
Bloedite	4.9%	6

(6) XRD pattern has been adjusted to 2θ = 29.45° by addition of 0.026° 2θ; 21 peaks have been identified out of 28 found, which accounts for 96.5 percent of the total measured intensity.

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Table 8.--Computer printout of the library file of X-ray diffraction data required for mineral identification and calculation of composition of mineral mixtures. The format as shown here is arranged precisely as needed by the computer program of Hosterman and Dulong (1989) to function. See Hosterman and Dulong (1989) for a detailed description of the library-file format.

```
DARAPSKITE[1]              29.0[2].700[3]  33[4] 500[5] 1[6] 10000[7]

10.3[8] 100[9]  TT[10]4.13   7    FF 3.59    2    FF 3.53    4    FF
  3.46   9    FF 3.27    3    FF 3.19    2    FF 3.08    1    FF
  2.955  1    FF 2.856   8    FF 2.667   1    FF 2.57    8    FF
  2.534  1    FF 2.438   2    FF 2.421   1    FF 2.378   1    FF
  2.341  1    FF 2.251   1    FF 2.177   1    FF 2.1     1    FF
  2.06   5    FF 2.013   1    FF 1.938   1    FF 1.912   1    FF
  1.7737 4    FF 1.7481  1    FF 1.7284  2    FF 1.7053  1    FF
  1.6732 1    FF 1.6294  1    FF 1.5954  1    FF 1.5804  1    FF
  1.54   1    FF
HALITE                    74.6 .500   5    200   1    5000

  3.258  3    FF 2.821 100    FF 1.994  20    TT 1.70    1    FF
  1.62   5    FF
NITRATINE                 16.9 .700  10    200   1    10000

  3.890  2    FF 3.03  100    TT 2.81   20    FF 2.53    3    FF
  2.311  9    FF 2.125   4    FF 1.898  21    FF 1.880   4.·  FF
  1.652  2    FF 1.629   2    FF
NITER                     62.5 .300  23    500   1    840

  4.66  14    FF 4.58    4    FF 3.78  100    FF 3.73   17    TT
  3.033 70    FF 2.763  34    FF 2.707  11    FF 2.647  87    FF
  2.409  4    FF 2.367   3    FF 2.332   5    FF 2.292   1    FF
  2.192 27    FF 2.159  17    FF 2.071   9    FF 2.050  16    FF
  1.947 34    FF 1.888   2    FF 1.750   4    FF 1.733   1    FF
  1.707  3    FF 1.688   4    FF 1.585   5    FF
GYPSUM                    69.5 .500  25    200   1    3300

  7.63 100    TT 4.283  12    FF 3.799  15    FF 3.065  18    FF
  2.873  4    FF 2.789   1    FF 2.685   4    FF 2.597   1    FF
  2.534  1    FF 2.495   1    FF 2.452   1    FF 2.406   1    FF
  2.219  3    FF 2.074   3    FF 2.048   1    FF 1.992   1    FF
  1.899  6    FF 1.879   1    FF 1.812   3    FF 1.779   3    FF
  1.684  1    FF 1.645   1    FF 1.621   3    FF 1.600   1    FF
  1.584  1    FF
```

(Table 8 continued on next page)

table 8 (cont.).

BLOEDITE   50.1 .200   41   500   1   1300

4.555	100	TT	4.442	1	FF	4.281	20	FF	4.126	6	FF
3.981	4	FF	3.860	4	FF	3.800	13	FF	3.333	17	FF
3.289	89	FF	3.252	30	FF	3.091	1	FF	3.055	1	FF
2.971	25	FF	2.724	36	FF	2.687	8	FF	2.644	26	FF
2.586	12	FF	2.518	1	FF	2.454	2	FF	2.420	1	FF
2.314	7	FF	2.297	6	FF	2.271	9	FF	2.194	4	FF
2.170	10	FF	2.141	3	FF	2.113	7	FF	2.062	1	FF
2.025	11	FF	1.933	13	FF	1.921	6	FF	1.901	2	FF
1.858	7	FF	1.779	15	FF	1.753	2	FF	1.723	2	FF
1.711	1	FF	1.665	8	FF	1.661	9	FF	1.601	4	FF
1.567	6	FF									

END OF LIBRARY   99999
    .5[11]1.0[12].02[13] 3.00[14] 60.00[15] 0[16]
       [17]0263   0656   0694   1200   1275   4000
[18]1.5,3.5,4.0

Explanation for italicized footnotes--

1. Mineral name
2. Mass absorption coefficient
3. Window of acceptibility
4. Number of peaks
5. Minimum intensity
6. Number of important peaks
7. Multiplication factor
8. d-spacing
9. Relative intensity
10. Code describing peak importance: TT, most important;
    FF, less important.
11. Instrument factor (0.5)
12. Goniometer speed in degrees 2-theta/minute (1.0)
13. 2-theta counting interval (0.02)
14. Lowest 2-theta angle (3.00)
15. Highest 2-theta angle (60.00)
16. Code for chemistry calculation (0=no)
17. Digital counts number where doublets are expected (0263,
    0656, 0694, 1200, 1275, 4000)
18. T1, T2, T3 peak tests (T1 = 1.5, T2 = 3.5, T3 = 4.0)

Table 9. -- X-ray diffraction data for 6 pure mineral standards (Table 3a) utilized to calibrate for mineral identification. These results are the averages of analyses of two sample aliquots.

Standard Number   NAN100[1]

Peak #	d	I	I/I°	2θ
1	3.893	1890	1.8	22.82
2	3.030	104273	100.0	29.45
3	2.797	20584	19.7	31.97
4	2.582	2402	2.3	35.47
5	2.301	8894	8.5	39.12
6	2.117	3414	3.3	42.68
7	1.889	22254	21.3	48.14
8	1.873	3451	3.3	48.56
9	1.644	1962	1.9	55.89
10	1.621	1547	1.5	56.76

∴

(1) XRD pattern has been adjusted to 2θ = 29.45° by addition of 0.127° 2θ.

Table 9. -- (cont.)

Standard Number   HAL100[2]

Peak #	d	I	I/I°	2θ
1	3.260	1505	2.4	27.33
2	2.820	62438	100.0	31.70
3	1.990	11997	19.2	45.45
4	1.954	37	0.1	46.42
5	1.697	434	0.7	53.99
6	1.624	2762	4.4	56.63

(2) XRD pattern has been adjusted to 2θ = 31.70° by reducing by 0.241° 2θ.

Table 9. -- (cont.)

Standard Number   NIT100[3]

Peak #	d	I	I/I°	2θ
1	4.672	1261	13.5	18.98
2	4.595	307	3.3	19.30
3	3.778	9365	100.0	23.53
4	3.734	1553	16.6	23.81
5	3.030	6561	70.1	29.45
6	2.760	3140	33.5	32.41
7	2.703	986	10.5	33.12
8	2.643	8125	86.8	33.88
9	2.403	386	4.1	37.39
10	2.360	291	3.1	38.09
11	2.327	441	4.7	38.65
12	2.287	80	0.9	39.36
13	2.187	2525	27.0	41.25
14	2.152	1575	16.8	41.94
15	2.066	795	8.5	43.79
16	2.045	1506	16.1	44.25
17	1.936	3212	34.3	46.89
18	1.881	176	1.9	48.34
19	1.754	360	3.8	52.09
20	1.744	99	1.1	52.43
21	1.701	276	2.9	53.86
22	1.682	331	3.5	54.52
23	1.577	510	5.4	58.47

(3) XRD pattern has been adjusted to 2θ = 29.45° by reducing by 0.095° 2θ.

Table 9. -- (cont.)

Standard Number   DKT100[4]

Peak #	d	I	I/I°	2θ
1	10.521	108084	100.0	8.40
2	4.147	7682	7.1	21.41
3	3.594	2549	2.4	24.75
4	3.529	4514	4.2	25.21
5	3.466	9210	8.5	25.68
6	3.268	2836	2.6	27.27
7	3.191	2141	2.0	27.93
8	3.080	1480	1.4	28.96
9	2.950	775	0.7	30.27
10	2.870	8408	7.8	31.14
11	2.664	1438	1.3	33.61
12	2.574	8494	7.9	34.82
13	2.530	1084	1.0	35.45
14	2.435	2602	2.4	36.88
15	2.413	674	0.6	37.24
16	2.374	495	0.5	37.86
17	2.334	391	0.4	38.55
18	2.247	856	0.8	40.10
19	2.176	286	0.3	41.47
20	2.096	361	0.3	43.12
21	2.057	5614	5.2	43.98
22	2.006	810	0.7	45.15
23	1.933	713	0.7	46.97
24	1.905	902	0.8	47.69
25	1.768	4866	4.5	51.66
26	1.745	652	0.6	52.38
27	1.725	2472	2.3	53.03
28	1.713	1541	1.4	53.45
29	1.700	720	0.7	53.87
30	1.626	581	0.5	56.55
31	1.603	462	0.4	57.42
32	1.587	205	0.2	58.06
33	1.556	531	0.5	59.35

(4) XRD pattern has been adjusted to 2θ = 28.96° by reducing by 0.121° 2θ.

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Table 9. -- (cont.)

Standard Number   GYP100[5]

Peak #	d	I	I/I°	2θ
1	7.635	48464	100.0	11.58
2	4.287	5729	11.8	20.70
3	3.798	7052	14.6	23.40
4	3.058	8593	17.7	29.18
5	2.865	1957	4.0	31.20
6	2.777	330	0.7	32.20
7	2.675	1832	3.8	33.47
8	2.592	377	0.8	34.58
9	2.527	205	0.4	35.49
10	2.485	269	0.6	36.11
11	2.447	199	0.4	36.69
12	2.399	154	0.3	37.46
13	2.209	1467	3.0	40.82
14	2.073	1630	3.4	43.62
15	2.033	266	0.5	44.53
16	1.984	479	1.0	45.70
17	1.891	2645	5.5	48.07
18	1.873	671	1.4	48.58
19	1.805	1383	2.9	50.51
20	1.773	1231	2.5	51.50
21	1.676	72	0.1	54.72
22	1.653	271	0.6	55.55
23	1.612	1296	2.7	57.10
24	1.593	99	0.2	57.82
25	1.576	323	0.7	58.53

(5) XRD pattern has been adjusted to 2θ = 11.58° by reducing by 0.02° 2θ.

Table 9. -- (cont.)

Standard Number   BLD100[6]

Peak #	d	I	I/I°	2θ
1	4.565	14162	100.0	19.43
2	4.464	165	1.2	19.87
3	4.288	2861	20.2	20.70
4	4.133	778	5.5	21.48
5	3.989	555	3.9	22.27
6	3.868	544	3.8	22.97
7	3.802	1883	13.3	23.37
8	3.334	2462	17.4	26.72
9	3.288	12636	89.2	27.10
10	3.250	4259	30.1	27.42
11	3.088	195	1.4	28.89
12	3.050	164	1.2	29.25
13	2.966	3593	25.4	30.10
14	2.728	5069	35.8	32.80
15	2.682	1075	7.6	33.38
16	2.645	3646	25.7	33.87
17	2.581	1625	11.5	34.73
18	2.514	98	0.7	35.68
19	2.450	230	1.6	36.65
20	2.414	87	0.6	37.21
21	2.308	1049	7.4	38.98
22	2.293	911	6.4	39.26
23	2.269	1272	9.0	39.69
24	2.188	617	4.4	41.22
25	2.167	1476	10.4	41.64
26	2.135	466	3.3	42.31
27	2.107	933	6.6	42.89
28	2.055	136	1.0	44.02
29	2.020	1527	10.8	44.82
30	1.927	1855	13.1	47.12
31	1.918	778	5.5	47.36
32	1.899	266	1.9	47.86
33	1.853	952	6.7	49.11
34	1.777	2181	15.4	51.37
35	1.746	205	1.5	52.36
36	1.716	267	1.9	53.34
37	1.697	176	1.2	53.99
38	1.671	1102	7.8	54.91
39	1.660	1230	8.7	55.30
40	1.596	545	3.8	57.71
41	1.560	896	6.3	59.16

(6) XRD pattern has been adjusted to 2θ = 27.10° by reducing by 0.128° 2θ.

Table 10. Summary of tests for reliability of computer calculated mineral compositions based on X-ray diffraction data for mixtures of saline minerals (Tables 5 and 6).

Mineral	Relative Accuracy Average Absolute Difference	Precision Average Standard Deviation (1 sigma)	Number of Sample Aliquots Analyzed	Number of Analytical Determinations
Nitratine	1.3%	1.1	34	50
Halite	1.3%	1	26	42
Niter	2.3%	1.6	8	16
Darapskite	1.0%	0	6	14
Gypsum	1.3%	0.9	8	16
Bloedite	1.8%	0.5	8	16