Mineralogical studies of the nitrate deposits of Chile.
V. Iquiqueite, $Na_4K_3Mg(CrO_4)B_{24}O_{39}(OH)\cdot12H_2O$, a new saline mineral

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ABSTRACT

Iquiqueite, a new hydrous chromate-borate of sodium, potassium, and magnesium, is a widespread minor constituent in the nitrate fields of northern Chile. It seems to be particularly abundant in the vicinity of Zapiga, Tarapacá province. Associated minerals include nitratite, halite, niter, darapskite, bloedite, glauberite, dietzeite, brüggenite, ulexite, and gypsum. The mineral is named after the city of Iquique (Chile), the major port city for nitrate exports from the 1830s until the 1930s.

The new mineral occurs in nitrate ore as disseminated, thin, yellow, hexagonal platelets, singly or as vermiform aggregates of nearly parallel-oriented platelets; individual platelets are generally 5–50 μm in diameter and less than 5 μm in thickness. Iquiqueite is hexagonal, $P31c$; $a = 11.6369(14)$ Å, $c = 30.158(7)$ Å, Z = 3. The X-ray diffraction pattern has the following six strongest lines (d_{meas}, I, hkl): 10.11(85)(100), 6.04(85)(104), 3.28(85)(207), 3.22(85)(215), 3.02(100)(208), and 2.856(100)(222). Observed forms are c (0001) and m (10$\bar{1}$0). Hardness is ≤2. Cleavage: (0001) perfect, (10$\bar{1}$0) imperfect. The mineral is uniaxial negative with $\epsilon = 1.447(2)$ and $\omega = 1.502(2)$. The measured specific gravity is 2.05 ± 0.09 (pycnometer), the same as the calculated density of 2.05 g/cm³. Iquiqueite is slowly soluble in cold water, and samples for chemical analysis were obtained as residues of cold-water leaching of bulk material. Two microchemical analyses (in weight percent) gave (after elimination of CaO, SO₃, Cl, N₂O₅, and I₂O₅ as components of known or suspected contaminating minerals, and of H₂O⁻ as adsorbed and fluid-inclusion water, and recalculating to 100%): Na_2O 8.2, 7.6; K_2O 10.5, 11.6; MgO 2.9, 3.0; B_2O_3 57.0, 56.3; CrO_3 6.8, 6.7; H_2O 14.6, 14.8. The formula based on these analyses is $Na_4K_3Mg(CrO_4)B_{24}O_{39}(OH)\cdot12H_2O$.

INTRODUCTION

The new mineral iquiqueite (pronounced ē-kē-kā-īt) is a widespread minor constituent of the so-called *caliche amarillo* (yellow nitrate ore) or *caliche azufrado* (sulfurous nitrate ore, in reference to the yellow color), which is relatively pure saline material consisting chiefly of nitratite, $NaNO_3$, and halite, NaCl. Iquiqueite is undoubtedly equivalent to chromloeweite, which was discovered and given this provisional name by Wetzel (1928) for its presumed relation to loeweite, $Na_{12}Mg_7(SO_4)_{13}\cdot15H_2O$. The mineral is less soluble in cold water than most of the other minerals making up the yellow nitrate ore; relatively pure material for this study was recovered as a residue from cold-water leaching of ores in which the iquiqueite occurs as disseminated minute crystals and vermiform crystal clusters. The most brightly colored yellow nitrate ore encountered in this study was found to contain as much as 1% iquiqueite, whereas the pale-yellow material, most widespread in the nitrate fields, either lacks iquiqueite or contains only a small fraction of 1% of iquiqueite. The only naturally occuring, relatively pure iquiqueite of which we have knowledge is a specimen in the collection of the

U.S. National Museum (USNM 95630), containing several grams of the mineral. This specimen, labeled dietzeite, came from Oficina Delaware, Chile. It was not analyzed because we did not wish to destroy even a part of this unique specimen.

Although borate and chromate are known constituents of the nitrate ores of northern Chile, they are not known to occur together as constituents of any other mineral. Furthermore, we have been unable to find any published information about studies of aqueous systems involving chromate and borate, or of synthetic compounds in which both are constituents. Vartbaronov and Bergman (1968) reported that complex compounds formed in melts of the sodium-potassium-chromate-metaborate-borate system, but they were unable to identify those compounds. Our attempts to synthesize iquiqueite—which included crystallization of various mixtures of the components as well as recrystallization from solutions of *caliche amarillo* and of purified iquiqueite—have been unsuccessful.

Wetzel (1923, 1924, 1928) found iquiqueite as tiny columnar to platy crystals embedded in nitrate ore and obtained small amounts of the mineral in residues from leaching of this ore. The amounts were so small that mi-

crochemical tests were inconclusive, and Wetzel was uncertain of the principal constituents of the mineral. Wetzel (1923) first identified the mineral microscopically as dietzeite, $Ca_2(IO_3)_2(CrO_4)$, but later (Wetzel, 1924) made microchemical tests that showed the presence of ferrous iron and sulfate. Finally, Wetzel (1928) found the mineral to contain chromium instead of iron, potassium, and magnesium. He reported the mineral to be trigonal, as is loeweite, which led him to conclude it to be a kind of chromium-bearing loeweite (Wetzel, 1928). The refractive indices reported by Wetzel (1928), $\epsilon = 1.448$ and $\omega = 1.496$, are essentially the same as those determined in the present study for iquiqueite.

Iquiqueite is named for the city of Iquique, Chile, which has been the major port city for nitrate exports from the 1830s, when the first shipments of nitrate went to Europe, until the 1930s when Tocopilla became the dominant port city. The description and name of the new mineral have been approved by the Commission on New Minerals and Mineral Names, IMA. Specimens of *caliche amarillo,* similar to those from which the new mineral was recovered, and a small vial containing about 0.2 g of purified type material have been deposited in the mineral collection of the U.S. National Museum, Smithsonian Institution, Washington, D.C.

OCCURRENCE AND ASSOCIATIONS

The yellow nitrate ore from which the iquiqueite was recovered consists of a relatively high purity saline material associated with veins, layers, and irregular pods or masses of the so-called *caliche blanco* (white nitrate ore). The latter occurs in a form where fractured bedrock is the host and also in saline-cemented alluvium, where the high-purity material was deposited in desiccation cracks and open cavities in pre-existing nitrate ore. In both cases, cracks and cavities were progressively enlarged by the growing crystals as the saline minerals were deposited. The yellow saline material occurs as tabular to irregular masses having gradational contacts with the white saline material, or as wispy or poorly defined yellowish material within the white saline material. In some cases, the yellow saline material occurs along the margins of veins and layers of white material, suggesting late-stage deposition; in others, the yellow material is within the white material, suggesting concomitant deposition.

Iquiqueite was found in samples of *caliche amarillo* from many localities in the nitrate fields of northern Chile, but the only samples that yielded a sufficient amount for chemical analysis were of bright-yellow material from a collection of nitrate ore in the chemical laboratory of the former Servicio de Minas del Estado in Iquique. Unfortunately, the locality or localities from which these specimens came are not known, but they probably came from the nitrate fields in the vicinity of Zapiga (Fig. 1), which are well known for their abundance of *caliche amarillo.*

In addition to nitratite and halite, the bulk *caliche amarillo* from which the analysis samples were obtained also contains niter, KNO_3, darapskite, $Na_3(SO_4)(NO_3)\cdot$
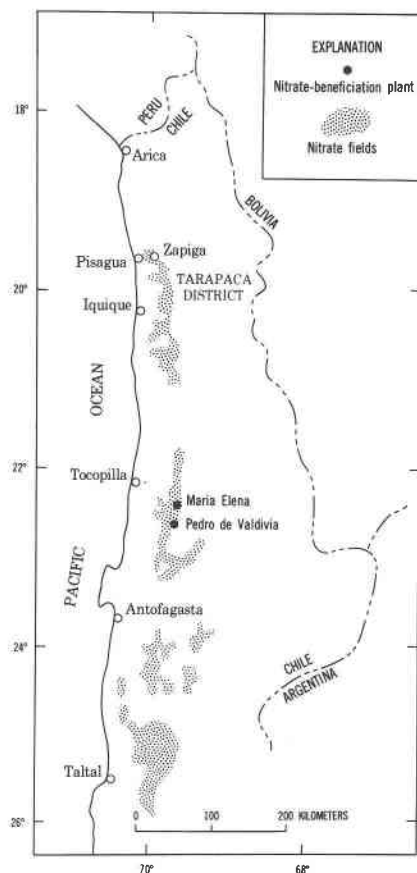

Fig. 1. Index map showing distribution of nitrate fields in northern Chile.

H_2O, bloedite, $Na_2Mg(SO_4)_2\cdot 4H_2O$, and glauberite, $Na_2Ca(SO_4)_2$. The residues obtained by leaching various specimens of *caliche amarillo* have, in addition to iquiqueite, a variety of associated minerals, though not all are present in each specimen. These are dietzeite, $Ca_2(IO_3)_2(CrO_4)$, brüggenite, $Ca(IO_3)_2\cdot H_2O$, ulexite, $NaCaB_5O_6(OH)_6\cdot 5H_2O$, and gypsum, $CaSO_4\cdot 2H_2O$. The leach solutions from *caliche amarillo* are bright yellow, indicating the presence of a highly soluble chromate mineral, probably tarapacaite, K_2CrO_4. In addition, cavities in some samples contain sparse tiny grains of bright-orange lopezite, $K_2Cr_2O_7$.

PHYSICAL AND OPTICAL PROPERTIES

Iquiqueite occurs as thin hexagonal platelets, generally 5–50 μm in diameter and less than 5 μm in thickness; a few are as much as 100 μm in diameter. They occur singly, as overgrowths of two or more crystals, as compound crystals, or as columnar to vermiform aggregates of platy crystals in near-parallel orientation (Fig. 2). In some cases, the crystals in the vermiform aggregates are intergrown with other saline minerals, such as nitratite. The latter dissolve during leaching, leaving slit-like cavities between iquiqueite crystals, but generally they form compact masses
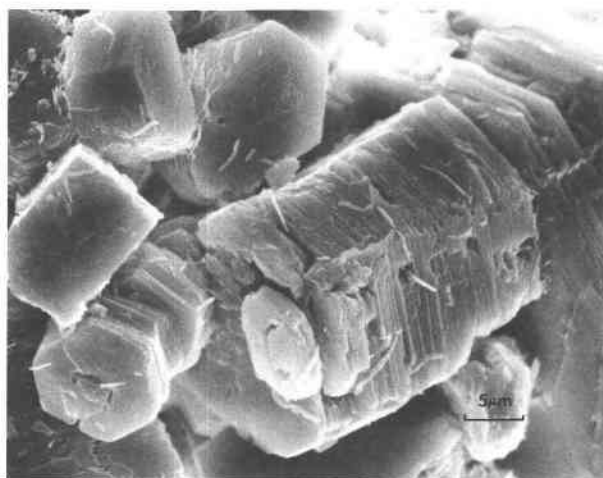
Fig. 2. SEM photograph of columnar aggregates of platy crystals of iquiqueite, showing tubular cavities, some of which are parallel to prism faces. Sparse fibers probably are ulexite.

Table 1. X-ray powder data for iquiqueite, $Na_4K_3Mg(CrO_4)B_{24}O_{39}(OH) \cdot 12H_2O$

h k l*	d_{calc}	d_{obs}	I_{obs}**	h k l*	d_{calc}	d_{obs}	I_{obs}**
0 0 2	15.08	15.11	60	2 1 6	3.04		
1 0 0	10.08	10.11	85	2 0 8	3.02	3.02	100
1 0 1	9.56	9.58	70	0 0 10	3.02		
1 0 2	8.38	8.39	70	3 0 $\bar{5}$	2.935	2.936	40
0 0 4	7.54	7.55	40	2 2 0	2.909	2.910	80
1 0 3	7.12			1 0 10	2.889	2.888	80
1 0 4	6.04	6.04	85	2 2 $\bar{2}$	2.857	2.856	100
1 1 0	5.82	5.82	20	2 1 7	2.854		
1 1 2	5.43	5.44	35	3 1 0	2.795	2.811	20
1 0 5	5.18			5 0 6	2.793		
2 0 0	5.04	5.04	70	2 0 9	2.790	2.791	35
0 0 6	5.03			3 1 1	2.783		
2 0 1	4.97			3 1 2	2.748		
2 0 2	4.78			2 2 4	2.714		
1 1 4	4.61			3 1 3	2.693	2.692	20
2 0 3	4.50			2 1 8	2.679	2.678	20
1 0 6	4.50			1 1 10	2.677		
2 0 4	4.19	4.19	30	3 0 $\bar{7}$	2.649		
1 0 7	3.96			1 0 11	2.645		
2 0 5	3.87	3.86	15	3 1 $\bar{4}$	2.621		
2 1 0	3.81			2 0 10	2.588	2.587	55
1 1 6	3.80			4 0 $\bar{1}$	2.510	2.510	50
2 1 1	3.78			4 0 4	2.390	2.389	65
0 0 8	3.77	3.77	50	4 0 5	2.325	2.324	55
2 1 2	3.69			3 0 10	2.244	2.243	50
2 1 3	3.56	3.56	50	4 0 $\bar{7}$	2.175	2.175	20
2 0 6	3.56			3 1 9	2.146	2.145	15
1 0 8	3.53	3.53	20	3 0 11	2.124	2.128	20
2 1 4	3.40	3.40	70	1 0 $\overline{14}$	2.107	2.105	20
3 0 0	3.36			3 2 $\bar{6}$	2.100	2.103	10
3 0 1	3.34			4 0 9	2.014	2.014	60
3 0 2	3.28	3.28	85	5 0 2	1.998	1.997	25
2 0 7	3.27			2 0 14	1.981	1.980	45
2 1 5	3.22	3.22	85	4 2 $\bar{1}$	1.901	1.901	40
3 0 3	3.19			4 2 2	1.890	1.890	10
1 0 9	3.18	3.18	20	3 1 12	1.869	1.869	50
1 1 8	3.16			5 0 $\bar{8}$	1.777	1.777	45
3 0 4	3.07	3.07	40	4 2 7	1.742	1.742	20

* All possible lines with $\underline{d}$ >2.60 have been listed.
** Intensities estimated visually; average of two films.

Hexagonal, space group P31$\underline{c}$; $\underline{a}$ = 11.6369(14) Å, $\underline{c}$ = 30.158(7) Å.

Guinier-Hägg method, CrKα_1 radiation (λ = 2.28962 Å).

Internal standard: CaF$_2$.

as shown in Figure 2. Many crystals and compact vermiform aggregates are vaguely skeletal, having abundant, irregular to tubular cavities. Such cavities can be seen on cleavage surfaces, suggesting skeletal crystals (Fig. 2), and some open on the prism faces. Others show a dendritic pattern of irregular tubes that may penetrate several crystals in a vermiform aggregate. Inasmuch as the massive nitrate-bearing saline material contains abundant fluid inclusions, it is believed that these cavities once were filled with fluid. Furthermore, iquiqueite is somewhat hygroscopic, and the water (H_2O^-) lost during drying of the analysis samples, 7.0 and 8.1%, was regained within a few hours after exposure to air at room temperature. However, such water does not cause any noticeable change in the physical character of the powdery samples (they do not become sticky or wet, for example), so it seems likely that the water is adsorbed as thin films on crystal surfaces and cavity walls.

Both the single crystals and crystal aggregates are bright yellow, but the powdered mineral has a slightly less intense yellow color. The luster is vitreous and the mineral is brittle. Hardness is equal to or less than 2 (crystals scratch talc but not gypsum). Basal cleavage (0001) is perfect whereas prismatic cleavage (10$\bar{1}$0) is imperfect. The specific gravity, determined on analysis sample no. 1, using the pycnometer method, is 2.05 ± 0.09.

Iquiqueite is optically uniaxial negative; some crystals appear to be anomalously biaxial, showing a small $2V$ (maximum about 10°) believed to be due to slight variations in orientation of compound or superimposed crystals. Indices of refraction, determined on the spindle stage at room temperature with refractive-index liquids measured at the same temperature by a Leitz Jelley microrefractometer, are ϵ = 1.447(2) and ω = 1.502(2).

CRYSTALLOGRAPHY

Many crystals of iquiqueite were examined optically before an individual crystal, minimally large enough for single-crystal precession study and also free of superimposed crystals, was found. Because of the extremely minute size of the selected iquiqueite crystal, precession photographs were taken in CuKα radiation (λ = 1.5418 Å, Ni filter). The results showed iquiqueite to be hexagonal with space group $P31c$ and cell parameters of a = 11.607 Å and c = 30.26 Å. The refined cell parameters, obtained by a least-squares analysis of the powder data, are a = 11.6369(14) Å and c = 30.158(7) Å. The calculated density for the formula $Na_4K_3Mg(CrO_4)B_{24}O_{39}(OH) \cdot 12H_2O$ is 2.05 g/cm³, with Z = 3, the same as the measured specific gravity of 2.05 ± 0.09.

X-ray powder diffraction data were obtained for analyzed sample no. 2 (see Table 2), which was handpicked to remove contaminants, using a Guinier-Hägg camera with CrKα_1 radiation (λ = 2.28962 Å) and with CaF$_2$ (a = 5.46295 Å) as an internal standard; intensities were estimated visually. The observed and calculated powder diffraction data are given in Table 1. Two lines, one at d = 5.50 Å (I = 40) and the other at d = 4.11 Å (I = 20) have been omitted from Table 1. These appeared on the Guinier-Hägg patterns of sample no. 2 but not on Debye-Scherrer patterns of iquiqueite from Oficina Delaware, Chile (USNM 95630); they are believed due to contamination by an unidentified, possibly new, mineral.

Although the crystals of iquiqueite were unsatisfactory for goniometric measurements, it was possible to identify, from precession photographs, the only two crystal forms observed, namely c (0001) and m (10$\bar{1}$0).

CHEMICAL COMPOSITION

Purified iquiqueite for chemical analyses was obtained by covering small fragments of the bright-yellow nitrate ore with sufficient cold water ($\sim 5°C$) and allowing it to stand without agitation for several hours in a refrigerator until the matrix material was completely dissolved; cold water was added, when necessary. The leach-solution was then decanted and the residue washed rapidly, first with cold water and then with methyl alcohol. A final wash was made with ethyl alcohol to remove the vestiges of methyl alcohol and wash water. Using this procedure, about 0.2–0.4 g of purified iquiqueite were obtained per 100 g of the bulk saline material that consisted chiefly of nitratite and halite.

The chemical analyses given in Table 2 were made on two different samples of purified iquiqueite: analysis no. 1, on a 585-mg sample, and analysis no. 2, on a 729-mg sample. Both samples contained microscopically visible impurities. One of the problems in interpreting the analyses was the identification of the constituents that were related to the contaminating phases. The contaminating minerals were identified by microscopic study and by X-ray powder diffraction techniques. Scanning electron microscopy (SEM) scans and energy-dispersive analyses of many crystals and crystal clusters of iquiqueite consistently showed the presence of K, Na, Mg, and Cr; lighter elements present in the chemical analyses are not detectable by SEM. Spectrographic analysis showed the presence of B. Microscopic examination of the analyzed samples indicated that the total amount of the several contaminating minerals was not more than 10–15%. Iodine was not detected by the SEM scans of iquiqueite crystals; therefore, it was assumed that it occurs in the minerals brüggenite and dietzeite that were found associated with iquiqueite. Sulfur in the analysis samples is attributed to associated gypsum and, for sake of simplicity, thenardite, although it is likely that still other sulfate minerals are present. Chlorine is attributed to halite.

The nitrate shown by the chemical analyses, 6.6 and 5.5% N_2O_5 (Table 2), was thought to be a constituent of iquiqueite until late in our study when a microprobe analysis (see below) showed that nitrogen was not present in crystals of iquiqueite. As a consequence, nitrate was eliminated as nitratite, the principal nitrate mineral in the bulk sample from which the analysis samples were recovered. Small amounts of niter and darapskite, which are minor constituents of the bulk sample, might also be present in the analyzed samples, but it is estimated that the amounts are too small to significantly affect the calculated stoichiometry.

The differences in composition shown by the two recalculated analyses (Table 2) are assumed to be analytical errors inherent in chemical analyses of small amounts of
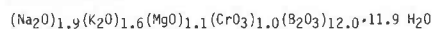
Table 2. Chemical analyses of iquiqueite; analysis no. 1 of 585-mg sample and analysis no. 2. of 729-mg sample

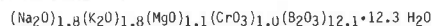|	Analysis no. 1		Analysis no. 2	
		Recalculated*		Recalculated*
	wt. %	wt. %	wt. %	wt. %
Na_2O	10.8	8.2	9.8	7.6
K_2O	8.0	10.5	8.2	11.6
MgO	2.2	2.9	2.1	3.0
CaO	.2		1.2	
B_2O_3	43.4	57.0	39.6	56.3
CrO_3	5.2	6.8	4.7	6.7
N_2O_5	6.6		5.5	
Cl	.1		.2	
I_2O_5	.0		5.3	
SO_3	1.2		2.0	
H_2O^+	11.2	14.6	10.9	14.8
H_2O^-	7.0		8.1	
Total	95.9	100.0	97.6	100.0

* After elimination of CaO, SO_3, Cl, N_2O_5 and I_2O_5 as components of the minerals gypsum, thenardite, halite, nitratite, and brüggenite, and H_2O^- as adsorbed and fluid-inclusion water.

Empirical formulas normalized to CrO_3:

Analysis no. 1:

$(Na_2O)_{1.9}(K_2O)_{1.6}(MgO)_{1.1}(CrO_3)_{1.0}(B_2O_3)_{12.0} \cdot 11.9\ H_2O$

Analysis no. 2:

$(Na_2O)_{1.8}(K_2O)_{1.8}(MgO)_{1.1}(CrO_3)_{1.0}(B_2O_3)_{12.1} \cdot 12.3\ H_2O$

a chemically complex material. Several slightly different formulas can be deduced from each of the analyses, according to the normalization procedure. These differences cannot be easily resolved. As a possible means of resolving the problem, attempts were made to analyze iquiqueite by electron microprobe. An ARL-SEMQ microprobe equipped with a light-element analyzing crystal and an anticontamination cold plate was used. Operating conditions were 10 kV and 0.1–0.5-μA beam current.

The microprobe analyses were complicated by difficulties with sample preparation, specimen–electron beam interaction, and data-correction procedures. The sample did not polish well; a highly polished, flat surface required for accurate microprobe analysis could not be obtained owing to slow dissolution, adhesion of polishing material (alumina), extreme softness, and fine grain size. As a result, surface exposure of grains was poor, with maximum areas of about 100 μm^2 (and usually less) available for analysis. Grains also were thin, and so overlap of the analyzing beam with epoxy around and below the grains was unavoidable. As a result of these analytical complications, only semiquantitative analyses were obtainable with the microprobe, and these were extremely variable owing to sample-surface conditions and thermal effects of the electron beam. However, repeated tests and measurements indicate that the iquiqueite does not contain detectable
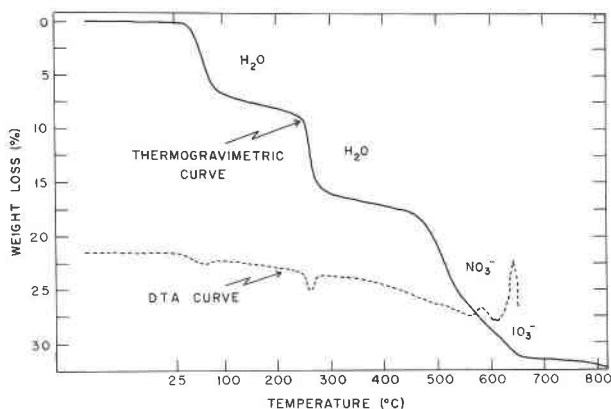
Fig. 3. Thermogravimetric (TGA) and differential thermal analysis (DTA) traces for iquiqueite. The TGA trace shows steplike weight losses due to loss of H_2O^- and H_2O^+ and decomposition and loss of NO_3^- and IO_3^-.

Fig. 4. Infrared spectra for iquiqueite, ulexite, NaCa-$B_5O_6(OH)_6 \cdot 5H_2O$, and synthetic Na_2CrO_4.

N; its presence in the microchemical analysis is due to a contaminant. Ten microprobe analyses, measured under various conditions in which the thermal effect on Na seemed minimal, yielded the following average composition (in weight percent): B_2O_3 41, Na_2O 6.5, K_2O 5.6, MgO 2.8, and CrO_3 7.9. Other analyses yielded similar consistent results for Mg, Cr, and B, but Na and K fluctuated owing to the thermal effect of the beam on the sample. The value for B is not considered accurate because of correction problems.

The problem of the composition of iquiqueite was finally resolved by trial and error in selecting a formula that gave the best fit between the microchemical analyses and the X-ray data, measured density, and refractive indices. The compatibility index—which is the Gladstone-Dale relationship between the chemical composition of a crystalline substance and its refractive indices and density (Mandarino, 1976), and consequently a measure of the accuracy of these parameters—was calculated for several different formulas that were deduced from the microchemical analyses. The final formula selected, $Na_4K_3Mg(CrO_4)B_{24}O_{39}(OH) \cdot 12H_2O$, is based on analysis no. 1 (Table 2), with CrO_3 taken as unity to give the empirical formula shown in the table. The formula $(Na_2O)_{2.0}(K_2O)_{1.5}(MgO)_{1.0}(CrO_3)_{1.0}(B_2O_3)_{12} \cdot 12H_2O$ indicated by the empirical formula gives a calculated density of 2.05 g/cm³, which best fits the measured specific gravity, and a nearly perfect compatibility index of 0.003. An OH^- has been inserted into the final formula to maintain a balance of electrical charges. Recent work on crystal structure of borate minerals has demonstrated that many have part of the oxygen bound in the OH radical, and it is possible that iquiqueite has even more of its oxygen in this form. However, this could be determined only by a study of the crystal structure.

Two other formulas that might be deduced from the analyses (CrO_3 = unity), $Na_3K_3Mg(CrO_4)B_{24}O_{39} \cdot 12H_2O$ and $Na_4K_4Mg(CrO_4)B_{24}O_{40} \cdot 12H_2O$, gave slightly poorer,
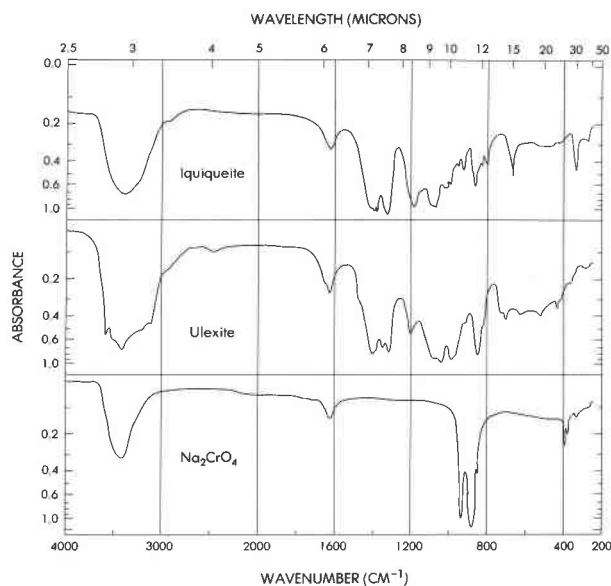
but still superior to excellent compatibility indices (Mandarino, 1979) of 0.012 and 0.031, respectively, based on calculated densities of 2.01 and 2.12 g/cm³, respectively. Still other slightly different formulas that might be deduced by normalizing to MgO give poorer values for density and compatibility indices.

THERMOGRAVIMETRIC CHARACTERISTICS

The thermogravimetric (TGA) trace for iquiqueite (Fig. 3) was made by Priestley Toulmin III and Jane M. Hammarstrom of the U.S. Geological Survey, using a 4.48-mg sample from the same sample used for chemical analysis no. 2 (Table 2). Also shown in Figure 3 is the differential thermal analysis (DTA) curve that was made at the same time as the thermogravimetric curve. These curves were made as the sample was heated from room temperature (25°C) to 800°C in flowing helium (about 85 cm³/min at 1 atm) at a rate of 4°C/min. The total weight loss, as determined by weighing the sample before and after the run, was 1.56 mg; this is equivalent to 32.2% of the sample.

It is assumed that the TGA-DTA sample contained the constituents indicated by chemical analysis no. 2, although not necessarily in exactly the same amounts, and that the thermogravimetric trace reflects partial loss of these constituents as gases. These losses include H_2O, nitrogen oxide, and iodine. As can be seen, the thermogravimetric trace shows three steep segments of rapid, major weight loss, separated by segments of more gentle slopes of less rapid weight loss. The first of the steep segments, at 40–80°C, is due to loss of of adsorbed and fluid-inclusion water (6.8%); the second, at 250–275°C, to loss of water of crystallization (6.2%). The loss between 80°C and 250°C, amounting to 2.2%, probably includes

both fluid-inclusion water and water of crystallization. Additional loss of water of crystallization and perhaps incipient loss of nitrogen oxide takes place between 275 and 470°C, amounting to 2.4%. If all the weight loss between 80 and 470°C was due to loss of water of crystallization, this would amount to 10.8%, essentially the same as the 10.9% H_2O^+ reported in analysis no. 2. The final part of the thermogravimetric trace reflects the decomposition of NO_3^- and IO_3^- in contaminating minerals, chiefly nitratite and brüggenite (Ericksen and Mrose, 1970; Ericksen and others, 1974).

It is concluded that the discrepancies between the weight losses shown by the TGA trace of iquiqueite (Fig. 3) and the composition determined by chemical analysis are due chiefly to an excess of the mineral contaminants $NaNO_3$ and $Ca(IO_3)_2$ in the TGA sample over that reported in the analysis. The other constituents occurring in iquiqueite or contaminating minerals—MgO, CaO, SO_3, Cl, B_2O_3, and CrO_3—are not likely to decompose at these temperatures.

The DTA curve (Fig. 3) shows endothermic peaks at the temperatures of major water loss (70°C and 260°C) and an exothermic peak at about 580°C that may correspond to a chemical reaction during loss of I_2. A final, strong exothermic peak at 645°C follows the major weight losses and, according to Priestley Toulmin III (1979, written comm.), may be due to some type of gas-phase reaction in the sample crucible.

INFRARED SPECTRA

Infrared absorption spectra were made for iquiqueite to ascertain the possible ions present in the mineral, and for other selected borates, chromates, and $NaNO_3$, which contain ions that might be present in iquiqueite. Figure 4 shows the spectrum for iquiqueite and, for comparison, spectra of ulexite and reagent grade Na_2CrO_4. These spectra and others were made under the direction of the late Irving A. Breger of the U.S. Geological Survey, with a Perkin-Elmer Model 621 spectrophotometer, utilizing KBr discs that consisted of a mixture of 300-mg KBr and about 0.6 mg of finely ground sample material.

The infrared spectrum for iquiqueite indicates the presence of a borate ion but does not clearly indicate its structural configuration. Of several infrared spectra made for borate minerals, that shown for ulexite is most similar to that of iquiqueite; this may be fortuitous or may indicate a structural relationship. Tincalconite, $Na_2B_4O_5(OH)_4 \cdot 3H_2O$, hydroboracite, $CaMgB_6O_8(OH)_6 \cdot 3H_2O$, and ezcurrite, $Na_4B_{10}O_{17} \cdot 7H_2O$ show multiple peaks owing to the borate ion in the region of 650–1500 cm^{-1}, which according to published data (Lyon, 1967; Nyquist and Kagel, 1971), is the range of the principal absorption peaks (other than H_2O) of the borate minerals.

The presence of H_2O (water of crystallization and free water) is indicated by the strong absorption peak at about 3400 cm^{-1} (Fig. 4), which is due to O-H stretching vibrations, and the small peak at 1640 cm^{-1}, which is due to H–O–H bending motion (Nyquist and Kagel, 1971).

Unfortunately, the O-H stretching vibration for the hydroxyl ion (OH$^-$) falls within the same range as that for H_2O (see Ryskin, 1974) and consequently would be masked if both H_2O and OH$^-$ were present, as apparently is the case for both iquiqueite and ulexite. The 3400 cm^{-1} peak in the spectrum for Na_2CrO_4 is due to absorbed water; it disappeared in a second spectrum made with the same disk after it had been heated at 110°C for 16 h.

The spectra for NO_3^-, if present as a contaminating mineral, and CrO_4^{2-} would be masked or obscured by the absorption peaks for borate. The NO_3 ion shows a strong broad peak in the range 1280–1520 cm^{-1} (Nyquist and Kagel, 1971). In $NaNO_3$ and KNO_3 it occurs at 1300–1430 cm^{-1}, about the same as the strongest peak for ulexite (Fig. 4) and some other borate minerals. In contrast, the weak absorptions at 925 and 955 cm^{-1} in the spectrum for iquiqueite may be the absorption peaks for CrO_4^{2-}, which in the spectrum for Na_2CrO_4 are at 880 and 940 cm^{-1}, respectively. The moderately strong absorption peak at 330 cm^{-1} in the spectrum for iquiqueite may correspond to the CrO_4^{2-} peak near 400 cm^{-1} in the spectrum for Na_2CrO_4.

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