GALEITE AND RELATED PHASES IN THE SYSTEM
Na$_2$SO$_4$-NaF-NaCl

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Abstract

The composition of galeite, a newly described mineral found as small crystals with gaylussite in drill cores from Searles Lake, California, may be represented by the formula that has been used for schairerite, Na$_2$SO$_4$·Na(F,Cl). Two analyses of galeite and one new analysis of schairerite are reported. The Cl/F ratio is probably 1/4 in galeite and 1/6 in schairerite. The diffraction symmetry of both minerals is $\bar{3}12/mP$. The cell dimensions are:—galeite a 12.17, c 13.94 Å, Z=15; schairerite a 12.17, c 19.29, Z=21. These minerals are distinct from the artificial Na$_2$SO$_4$·NaF which is rhombohedral or pseudorhombohedral with a_h 27.85, c_h 24.50, Z=144. Indexed x-ray powder diffraction patterns have been tabulated for all four known phases on the join Na$_2$SO$_4$·NaF − Na$_2$SO$_4$·NaCl. Refractive indices are:—Na$_2$SO$_4$·NaF ω 1.439±0.001, ϵ 1.442±0.002; schairerite ω 1.443 ±0.001, ϵ 1.445±0.001; galeite ω 1.447±0.001, ϵ 1.449±0.001. Morphologically galeite and schairerite are generally distinguishable and new angle tables are presented for each. Syntactic intergrowths of galeite and schairerite are considered to be polycrystals. Twinning on (0001) in Na$_2$SO$_4$·NaF and schairerite and on ($\bar{1}\bar{1}2\bar{2}$) in schairerite has been established both by goniometric and x-ray observations. When heated to 500° C. or more schairerite, galeite and sulfohalite break down to Na$_2$SO$_4$·NaF, Na$_2$SO$_4$ and NaCl. Textural differences in the products support the assumption that schairerite has a lower Cl/F ratio than galeite.

Discovery

Galeite was first noticed by Mr. Leroy J. Bailey in examining a core from a test hole drilled in Searles Lake, California, by the American Potash and Chemical Corporation. An excerpt from the core log of Test Hole L-NN at a depth of about 113 feet reads: "The lower six inches of this section contains several 0.5 to 1.0 cm spherical aggregations of microscopic crystals, hexagonal in form with an extreme development of the basal pinacoid. These crystals averaged 0.02 to 0.4 mm across the axis and approximately 0.005 mm thick. They are water-clear, transparent, and apparently belong to the sulfohalite and schairerite group." This material was submitted to one of us (G.S.) and in correspondence with Dr.

Leo Briggs, Assistant Director of Research of the American Potash and Chemical Corporation in December, 1949, the possibility that this might be a new mineral was considered and the name galeite, in honor of Mr. W. A. Gale, was suggested.

HISTORY OF THIS INQUIRY

Soon after 1949 galeite was recognized in cores from more than 20 other wells and test holes of the American Potash and Chemical Corporation, scattered over much of Searles Lake. In some places the galeite was in minute crystals like those of the initial find (Fig. 1A), but in others it was found in clusters of slightly larger crystals with barrel habit (Fig. 1B). Some of this material was sent by D.L.S. to A.P. in August, 1951, and after a few months' delay the distinctness of the material was confirmed by single crystal x-ray examination. The senior author having been previously concerned with the related sulfohalite (Pabst, 1934) and having done some unfinished work on schairerite, the three present authors agreed to combine their studies after conferring with other interested colleagues. A preliminary report of our work in which the name galeite was proposed appeared only after several years (Pabst *et al.*, 1955). Most of the later phases of the work were carried out in Berkeley and have been subject to many delays.

ACKNOWLEDGMENTS

OCCURRENCE AND ASSOCIATIONS

Galeite has been found only in drill cores which have penetrated into the "lower salt" or "lower structure" of the Searles Lake basin. In many cores it is found in ovoid white aggregates consisting very largely of microscopic galeite crystals (Fig. 2A and 2B). These are embedded in so-called muds which are grayish to buff. The muds consist partly of saline minerals, some being mostly gaylussite in microscopic euhedral crystals. The minerals most closely associated with galeite, besides gaylussite, are northupite, less commonly tychite, and often trona, the principal constituent of the "lower salt." Galeite aggregates appear to be limited to one horizon usually encountered at depths of 93 to 123 feet. This is generally below the burkeite strata of the "lower salt" (Haines, 1959, Fig. 7).

Galeite was not recorded in the published core logs of the U. S. Geological Survey from Searles Lake (Smith and Pratt, 1957; Haines, 1959) and schairerite was recorded only in drill hole GS-22, depth 98.5–98.9 feet. The description of this reads in part "faint fine very light gray laminae in lower 0.4 ft with scattered nodules, averaging 5 mm in length, of white finely crystalline pulverulent schairerite*." This description suggested galeite to the senior author (A.P.) and Mr. Haines kindly furnished comparable material from two other drill holes with unpublished core logs with the following descriptions:

Hole GS-17, M2 unit, 107.3–107.4 feet depth:

"Black (N1) to light olive-brown (5Y5/6) to greenish-gray (5GY6/1) to very light gray (N8) locally finely laminated clay with 40 per cent disseminated anhedral colorless gaylussite crystals averaging 3 mm in length; single 3/4 inch pocket of white (N9) finely crystalline pulverulent massive schairerite*".

Hole GS-41, M2 unit, 93.4–93.9 feet:

"Black (N1) clay with scattered 2- to 3-inch pockets of disseminated radiating colorless trona blades 10 to 40 mm in length; a few round to oval pockets 2 to 20 mm in diameter of very pale orange (10YR8/2) finely crystalline pulverulent massive schairerite*. Clay 89 percent, trona 10 percent, schairerite 1 per cent."

Crystals of the material designated schairerite in these two samples were examined by two-circle goniometer and by single crystal x-ray diffraction and found to be galeite. The material from hole GS-17 is tabular like that shown in Fig. 1A and the galeite from hole GS-41 has a barrel habit similar to that shown in Fig. 1B. The asterisks in the above quotations designate minerals identified optically by immersion methods. As the refractive indices of schairerite differ from those of galeite by only about 0.004 the two might easily be confused. The "finely laminated clay" referred to in the GS-17 description consists largely of northupite in octahedra about 0.1 mm on edge and of euhedral gaylussite crystals

0.02 mm or less in maximum dimension. "(The term "clay" as used in the core logs and throughout this report refers only to particle size and not to mineral composition.)" (Haines, 1959, p. 143).

Sulfohalite has been recorded at various levels in both the upper and lower salts in a dozen or more holes for which core logs have been published (Haines, 1959; Smith and Pratt, 1957). It was not encountered in the present study. Most Searles Lake sulfohalite occurs at lesser depths, often with halite or hanksite which are not generally found with schairerite or galeite.

A   B
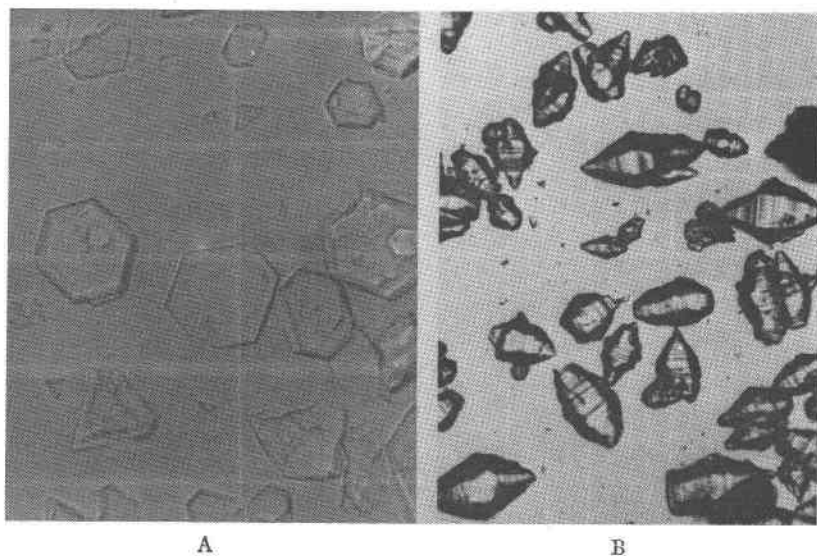
Fig. 1. A. Galeite from test hole MG-8. 290×.
B. Galeite, source not identified. 42×. Photographs by Al Gonzales.

Foshag (1931) described schairerite from an unknown depth in a drill hole at Searles Lake which "penetrated the salt body for a depth of 125 feet." It was judged to be from "one of the deeper samples." Foshag's schairerite occurred as disseminated crystals "measuring less than two millimeters." In the course of the present study many schairerite crystals from a depth of 121′ 2″ in hole LS were examined. These attain a length of nearly one centimeter with a habit shown in Fig. 3A, mostly with satellites of galeite. Other material from hole LS and from hole LP and core samples of uncertain origin yielded more single crystals of schairerite, many of the larger ones skeletal or partly chalky and of a habit like Foshag's Fig. 1, a single steep rhombohedron only. Numerous galeite crystals up to one mm long, always of pyramidal or barrel habit, and

various types of schairerite-galeite polycrystals, were obtained from the same sources.

Summarizing: galeite is found in friable clusters or aggregates, as single crystals, or as parts of polycrystals, all restricted to the lower part of the "lower salt" of Searles Lake and invariably associated with gaylussite. The galeite of tabular habit has been observed only in aggregates of very fine grain. Galeite of pyramidal habit occurs in clusters and also in single crystals or polycrystals. Galeites of the two habits have not been found together.

CHEMICAL COMPOSITION

In Table 1 are set forth all available analyses of galeite and of schairerite. The two analyses of galeite agree well and support the formula

TABLE 1. ANALYSES OF SCHAIRERITE AND GALEITE FROM SEARLES LAKE, CALIFORNIA.

	Schairerite	Schairerite		Galeite	Galeite	
	A	B	C	D	E	F
Na	35.77	36.20		36.60	36.66	
K	0.13	0.55		tr.	tr.	
SO_4	50.01	52.24		50.99	50.80	
Cl	3.44	2.85		3.80	3.90	
F	8.08	7.98		7.70	8.07	
rem.	1.55	0.12		0.10	0.50	
sum	98.98	99.94		99.19	99.93	

Same analysis adjusted to $(Na, K) + SO_4 + F + Cl = 100$

	A	B	C	D	E	F
Na	36.72	36.27	36.98	36.94	36.87	36.74
K	0.13	0.55		tr.	tr.	
SO_4	51.33	52.33	51.51	51.46	51.09	51.17
Cl	3.53	2.86	2.72	3.83	3.92	3.99
F	8.29	7.99	8.79	7.77	8.12	8.10
F:Cl	4.38	5.21	6	3.78	3.86	4
$F+Cl:SO_4$	0.98	0.92	1	0.97	1.01	1

A. Analyst, W. F. Foshag (1931). rem. is CaO 0.30, $(Fe, Al)_2O_3$ 0.15, insol. 0.20, ig. loss 0.90.

B. Analyst, Laura E. Reichen. rem. is insol. 0.12.

C. $Na_2O_4 \cdot Na(F, Cl)$, F:Cl=6.

D. Analyst, D. L. Sawyer. Analysis reported as Na_2SO_4 75.39, NaCl 6.27, NaF 17.43. rem. is ig. loss 0.10.

E. Analyst, R. Klemen. rem. is insol. 0.47, H_2O 0.03. Spectrographic examination by G. M. Gordon showed Ca 0.03%, Sr 0.02, K 0.6, Li 0.005.

F. $Na_2SO_4 \cdot Na(F, Cl)$, F:Cl=4.
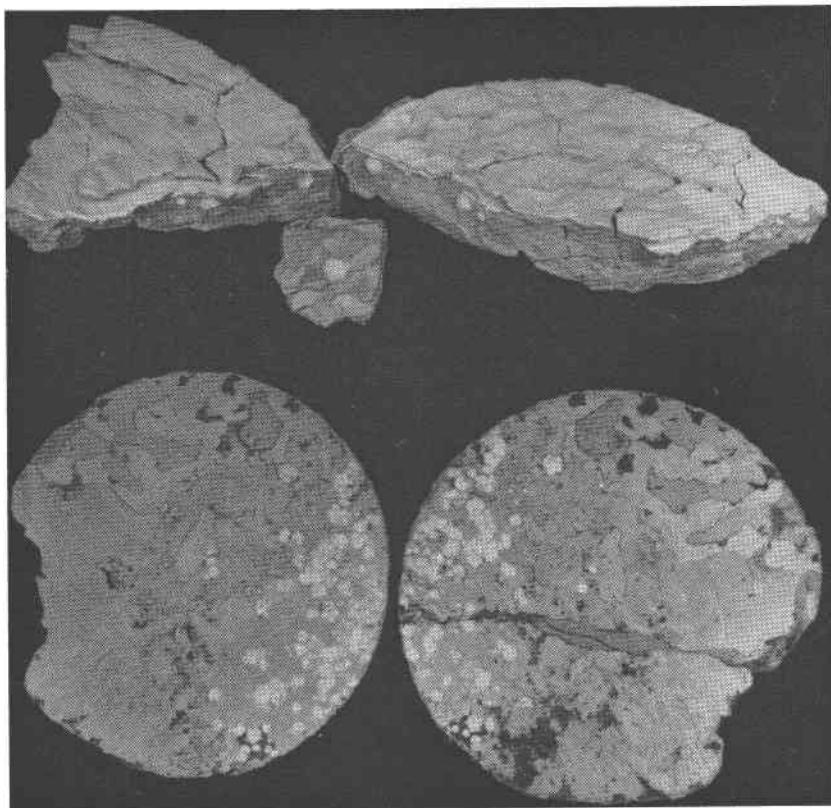
FIG. 2. Pieces of two 8-inch cores from Searles Lake, California, showing white, ovoid, aggregates of galeite.

A. (above) Well ✕-9.
B. (below) Test hole MG-8.
Photographs by Al Gonzales.

$Na_3(SO_4)$ (F, Cl). The F:Cl ratio of these analyses is just under 4, suggested as the ideal ratio for schairerite in Dana's System of Mineralogy, 7th ed., vol. II, p. 548, 1951. The two schairerite analyses, the new one made in connection with this study on selected crystals from holes LS and LP, do not agree so closely but do indicate that the ratio of F:Cl is higher in schairerite than in galeite. The ideal compositions with F:Cl equal to 4 and to 6 are inferred from cell dimensions and relations to the structure of sulfohalite. Fleischer's (1956) comment, referring to the preliminary description of galeite, that "the mineral is therefore dimorphous with schairerite," may require revision.

Fig. 3. 10–20° axonometric projections of schairerite and galeite.
A. Schairerite with galeite outgrowth.
B. Schairerite-galeite polycrystal. Forms labelled on schairerite part only.
C. Galeite of "barrel habit," simplified and idealized. See text.
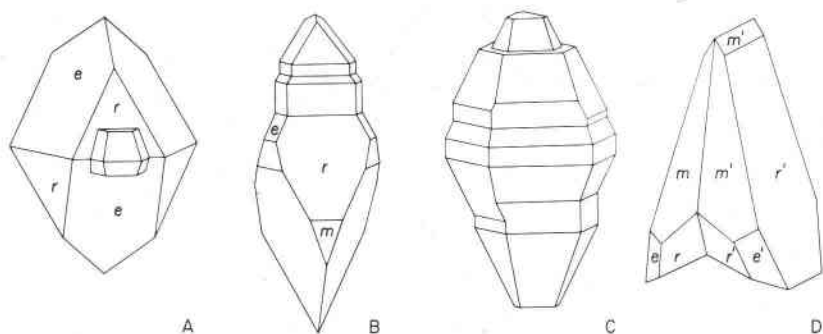D. Schairerite twin on (1̄122). Original shown in upper right quadrant of photograph, Fig. 5.

OPTICAL PROPERTIES

The optical properties of galeite and some related materials are set forth in Table 2. Refractive indices were determined both by observation on small single crystals mounted for goniometry or x-ray diffraction using techniques similar to those described by Fisher (1960) and by immersion of fragments and very small crystals using light from a sodium vapor lamp and liquids freshly calibrated at constant temperature on a refractometer reading directly to 0.0005. The birefringence of galeite and schairerite is very low so that it cannot be adequately expressed in terms of the measured values of ω and ϵ if the uncertainties of these are of the order of 0.001 or more. The birefringence was determined directly on numerous mounted crystals in air and white light and by Berek compen-

TABLE 2. REFRACTIVE INDICES OF MULTIPLE SALTS IN THE SYSTEM Na₂SO₄-NaF-NaCl

		$Na_2SO_4 \cdot NaF$	Schairerite	Galeite	Sulfohalite
New Data	ω	1.439 ± 0.001	1.443 ± 0.001	1.447 ± 0.001	
	ϵ	1.442 ± 0.002	1.445 ± 0.001	1.449 ± 0.001	$n\ 1.455 \pm 0.001$
	$\epsilon - \omega$		0.0012 ± 0.0002	0.0012 ± 0.0002	
Quoted Data	ω	1.436[1]	1.440[2]		$n\ 1.454 \pm 0.002$[3]
	ϵ	1.439	1.445		

[1] Foote & Schairer (1930a, p. 4206).
[2] Foshag (1931, p. 138).
[3] Larsen (1921, p. 139).

sator in Na light on immersed crystals of measured thickness. The small difference in the indices of galeite and schairerite and the identity or near identity of their birefringences were checked by simultaneous observations on crystals of the two and by observations on polycrystals.

CELL DIMENSIONS, DENSITY, CELL CONTENT AND SPACE GROUP

In Table 3 are listed the cell dimensions of $Na_2SO_4 \cdot NaF$, a synthetic material, schairerite, galeite and sulfohalite as newly determined from

TABLE 3. CELL CONSTANTS OF MULTIPLE SALTS IN THE SYSTEM Na_2SO_4-NaF-NaCl

	Sulfohalite	Galeite	Schairerite	$Na_2SO_4 \cdot NaF$
Space Group[1]	$Fm3m$	$P\bar{3}12/m$	$P\bar{3}12/m$	$R\bar{3}2/m$
a	10.065Å (10.10)[2]			
a_h	7.117 ($a/\sqrt{2}$)	12.17	12.17 (12.14)[3] (7.06)[4]	27.85
c_h	17.43 ($a \cdot \sqrt{3}$)	13.94	19.29 (19.23) (19.37)	24.50
a_h'	7.117	7.026	7.026	6.912
c_h/n	2.905	2.788	2.756	2.722
Density (meas.)	2.505	2.605	2.612[5]	2.67
Density (calc.)	2.505	2.610	2.627	2.675
Cell Content				
$Na_2SO_4 \cdot Na(F, Cl)$	6 (hex. cell)	15=3×5	21=3×7	144=16×9

[1] The most highly symmetrical of the possible space groups only is indicated in each case.

  a_h' is for the pseudocell discussed in text.

  c_h/n, the value of n will be apparent from the statement of the cell content for each phase.

  All quoted cell dimensions were originally reported in kX units and have here been converted to Å for comparison with the newly determined values.

  The uncertainty of all the newly determined cell dimensions is less than 1/1000.

  The uncertainty of the newly determined densities is of the order of 0.01.

[2] Pabst (1934).

[3] Frondel (1940, p. 352).

[4] Wolfe and Caras (1951).

[5] Foshag (1931, p. 135).

quartz calibrated Weissenberg patterns and shrinkage corrected precession patterns, together with earlier data on schairerite and sulfohalite. The cell given for $Na_2SO_4 \cdot NaF$ is only a pseudocell associated with a rhombohedral lattice and no statement as to the space group is made. The diffraction symmetry of schairerite and of galeite is the same, $\bar{3}1mP \ldots$, the planes of symmetry coinciding with $\{10\bar{1}0\}$. No choice has been made among the indicated space groups. $p\bar{3}12/m$ is suggested by the morphology of schairerite whereas the morphology of many galeite crystals would conflict with this. Attempts at obtaining satisfactory etch figures on

schairerite were unsuccessful. Speculations as to the structures suggest that the space group is probably not $p\bar{3}12/m$ or $p312$ for either mineral. The space group of sulfohalite has been established by the concurring structure determinations of Watanabé (1934) and of Pabst (1934).

Determination of the cell content is unequivocal in each case except for the doubt that remains as to the F:Cl ratio for schairerite and galeite. The calculated densities for these are based on the ideal ratios 6:1 and 4:1 respectively. The newly measured densities were, in each case, determined by suspension in liquids simultaneously checked by Westphal balance.

MORPHOLOGY

Most of the single crystal x-ray work was carried out on crystals that had been measured by two-circle goniometer. Over 50 crystals were handled in this way. In view of the space group indications, $p\bar{3}12/m$ or one of its trigonal subgroups, for both schairerite and galeite, the rhombohedral (or pyramidal) forms which are invariably of only one order are to be designated as second order, $\{hh\bar{2}hl\}$, forms to conform to the choice of the smallest hexagonal cell. The relations will be clear from Fig. 4. This point is generally not covered in crystallographic textbooks. These usually suggest that where crystals show rhombohedrons of only a single order they are to be considered as of the first order without exception (Pabst, 1957 p. 380).

Foshag designated the principal rhombohedron, r, on schairerite $\{10\bar{1}1\}$. Referred to the hexagonal cell newly measured, previously found by Frondel (1940) and called "inconsistent with morphology," this form is to be designated $\{11\bar{2}1\}$. Only the habits reported by Foshag (1931, Figs, 1 to 4) and minor variants thereof were observed on simple schairer-

$D_{3d}^l - P\bar{3}12/m$ $D_{3d}^3 - P\bar{3}2/m1$

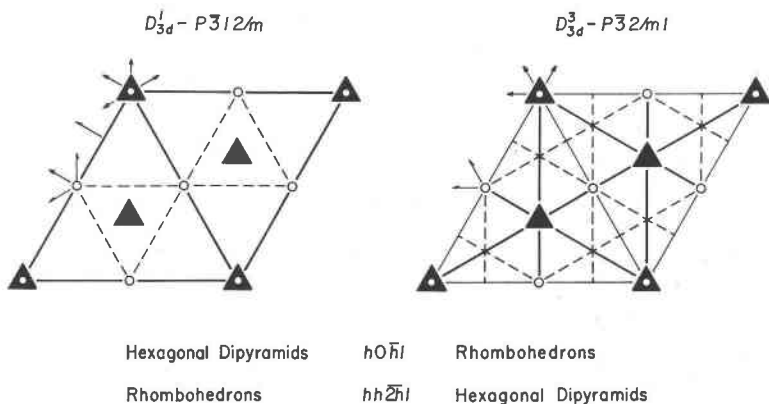| Hexagonal Dipyramids | $h0\bar{h}l$ | Rhombohedrons |
| Rhombohedrons | $hh\bar{2}hl$ | Hexagonal Dipyramids |

FIG. 4. Projections of the symmetry elements in two space groups onto (0001) with correlation of the appropriate designations of first and second order forms.

TABLE 4. STANDARD ANGLE TABLE FOR SCHAIRERITE

Hexagonal—P; $\bar{3}12/m$, $31m$ or 312; $a:c=1:1.585$; $p_0:r_0$ 1.830:1

forms		ϕ	$\rho = C$	M	A_2
c	0001	—	0° 00′	90° 00′	90° 00′
a	11$\bar{2}$0	0° 00′	90 00	90 00	60 00
r	11$\bar{2}$1	0 00	72 29$\frac{1}{2}$	90 00	61 31
e	2$\bar{1}\bar{1}$2	60 00	57 45	42 55	32 15

ite crystals (Fig. 5, lower right quadrant). All observed forms are included in the revised angle table (Table 4). The matrix for transforming Foshag's setting to that of the structure cell is $1\bar{1}\cdot0/12\cdot0/00\cdot1$ and the inverse matrix is $\frac{2}{3}\frac{1}{3}\cdot0/\frac{\bar{1}1}{33}\cdot0/00\cdot1$.

The cell reported by Wolfe and Caras (1951) corresponds to Foshag's setting. In any rotation or oscillation patterns taken on an axis parallel to the edge between the rhombohedron and base of schairerite all layer lines other than 3n are very weak. If these are not observed one is led to the cell of Wolfe and Caras which may be considered a strongly marked pseudocell. Similar relations exist in galeite.

Though, as for schairerite, only $hh\bar{2}hl$ forms have been observed on galeite, its habits are more varied and complex. The minute tabular crystals (Fig. 1A), occurring only in friable aggregates, are {0001} plates bounded, where measurable, only by {11$\bar{2}$0}, {11$\bar{2}$1} or both. The slightly larger and apparently more common type of galeite, occurring both in friable aggregates and as scattered single crystals, is irregularly bipyramidal or rhombohedral. About 20 such crystals were measured. They are always striated as seen in Fig. 1B and in the lower left quadrant of Fig. 5. {11$\bar{2}$1} is invariably present, mostly accompanied by vicinal faces, and usually {0001} can be seen. The $hh\bar{2}hl$ zones generally yield almost a continuum of signals of varying strength over the range of ρ from about 66° to about 114°. {11$\bar{2}$0} may be absent or represented only by vicinal faces. On about half the crystals {2$\bar{1}\bar{1}$1} appears together with {11$\bar{2}$1}, in

TABLE 5. STANDARD ANGLE TABLE FOR GALEITE

Hexagonal—P; $\bar{3}12/m$, $31m$ or 312; $a:c=1:1.145$; $p_0:r_0=1.323:1$

forms		ϕ	$\rho = C$	M	A_2
c	0001	—	0° 00′	90° 00′	90° 00′
a	11$\bar{2}$0	0° 00′	90 00	90 00	60 00
r	11$\bar{2}$1	0 00	66 25	90 00	63 42
t	2$\bar{1}\bar{1}$1	60 00	60 25	39 52	27 35

some cases in about equal development. Signals were repeatedly recorded at angles close to ρ for $\{77 \cdot \overline{14} \cdot \overline{6}\}$, 69° 29′, while occasional measurements suggest $\{22\overline{4}1\}$, $\{33\overline{6}4\}$, $\{33\overline{6}\overline{2}\}$, and others as possible forms. Only the certainly identified forms, in order of prominence $\{11\overline{2}1\}$, $\{2\overline{1}11\}$, $\{0001\}$ and $\{11\overline{2}0\}$, are shown in Fig. 3C and included in Table 5.

The System Na_2SO_4-NaF-NaCl

Figure 6 shows the compositions of crystalline phases in the system Na_2SO_4-NaF-NaCl plotted in mole proportions. The only multiple salts known have a 1:1 ratio of Na_2SO_4 to Na halides. Marignac (1859) accidentally discovered $Na_2SO_4 \cdot NaF$, correctly stated the composition and reported crystal measurements. Wolters (1910), in a study of fusion relations in the system, found only this one double salt, determined its melt-
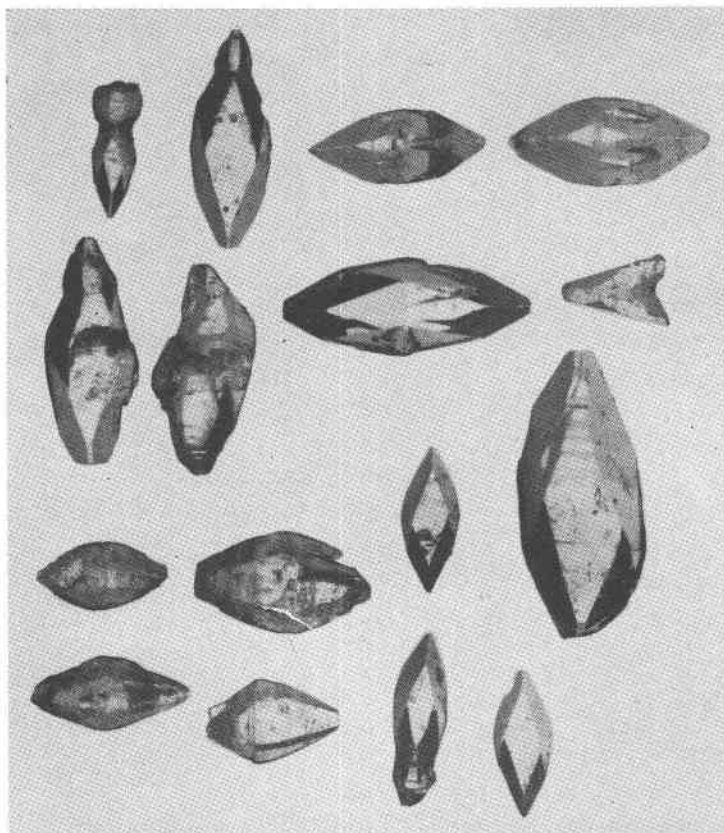

Fig. 5. Lower right quadrant: Four schairerite crystals. Lower left quadrant: Four galeite crystals. Upper left quadrant: Four polycrystals. Upper right quadrant: Four schairerite twins. Largest crystal is 2 mm long. Photograph by V. G. Duran.

ing point to be 781° C. and recognized it to be uniaxial positive. He con-
sidered it to be truly hexagonal only above 105° C. and pseudohexagonal
with mimetic twinning at ordinary temperature. Foote and Schairer in
their work on the system Na_2SO_4-NaF-NaCl-H_2O (1930a & b) reported
optical properties (Table 2, this paper) and goniometric measurements
confirming the work of Marignac. In order to fit the goniometric results
to the cell dimensions shown in Table 3 it is necessary to halve the c/a
ratio given by Foote and Schairer or corresponding to Marignac's desig-
nations, as indicated herewith:—

	Marignac (1859)		Foote and Schairer (1930a)	This work
c/a	1.76	(2×0.88)	1.76 (2×0.885)	0.880
R	$10\bar{1}1$		$10\bar{1}1$	$02\bar{2}1$
$S^{\frac{1}{2}}$	$01\bar{1}2$		$01\bar{1}2$	$40\bar{4}1$
S^2	$02\bar{2}1$		—	$10\bar{1}1$
$S^{\frac{4}{5}}$	$04\bar{4}5$		—	$80\bar{8}5$
			$03\bar{3}2$	$30\bar{3}1$

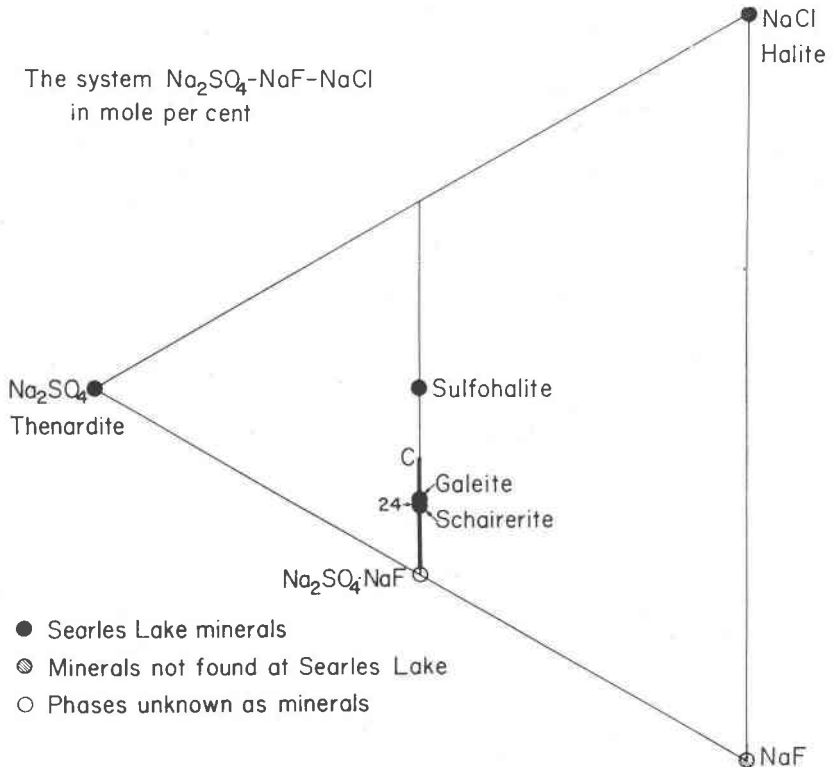Several of the crystals of the double salt kindly supplied by Dr. Schairer

FIG. 6. The system Na_2SO_4-NaF-NaCl in mole per cent.

were measured goniometrically preparatory to x-ray examination. Only the base and rhombohedra were observed, $\{02\bar{2}1\}$ and $\{40\bar{4}1\}$ being invariably present. These become $\{01\bar{1}2\}$ and $\{10\bar{1}1\}$ if referred to the conspicuous pseudocell of $Na_2SO_4 \cdot NaF$ mentioned below.

Neither Wolters nor Foote and Schairer found any phase corresponding to sulfohalite. Foote and Schairer (1930b, p. 4215) did report that "in the quaternary system the double salt $Na_2SO_4 \cdot NaF$ has a limited part of its fluoride replaced by chloride." In Fig. 6 the heavy line from Na_2SO_4 $\cdot NaF$ to C corresponds to Foote and Schairer's line ABC (1930b, Fig. 3) representing the range of replacement and the numeral 24 indicates the point along this line corresponding to the double salt with maximum Cl/F ratio formed from solutions at 35° C. (Foote and Schairer, 1930b, Table 1). The compositions of schairerite and of galeite indicated along this line close to point 24 correspond to the analyses by Foshag and by Sawyer respectively.

Foshag tried to reconcile his morphological description of schairerite with that of Foote and Schairer's chlorine-free double salt and labelled a drawing of the latter "Schairerite, artificial" (Foshag 1931, Fig. 5). Results reported here show that schairerite as well as galeite must be considered a phase distinct from $Na_2SO_4 \cdot NaF$. However, the cell dimensions of these and of sulfohalite are all related, as can be seen from the data in Table 3 and from Fig. 7 in which the cells are shown together with a common origin and corresponding orientations. Sulfohalite, which is cubic face centered, is shown by the hexagonal cell that would correspond to one primitive rhombohedral unit. The base of this cell corresponds to the base of the pseudocell of schairerite and of galeite previously referred to in the comments on the work of Wolfe and Caras. $Na_2SO_4 \cdot NaF$ has an even more marked pseudocell of this sort. All layer lines on a axis rotation or oscillation patterns of this material for which $n \neq 4$ are recognizable only upon especially long exposure. The edge of this base or pseudobase of all four phases increases in length with increasing Cl content. Furthermore the c axes can be considered to be simple multiples of a basic spacing, c/n, which likewise increases with increasing Cl content.

STRUCTURAL RELATIONS AND IDEAL FORMULAS

Only the structure of sulfohalite is known (Watanabé, 1934; Pabst, 1934). The picture of this structure in Strukturbericht (III, 118) is faulty, a fact noted on ASTM card 3–0345. A correct picture of the structure is shown in Fig. 8A. Figure 8B shows the structure of sulfohalite within the hexagonal unit pictured in Fig. 7. The basic spacings whose multiples are c for the several phases correspond in sulfohalite to the distance between planes of Cl and planes of F.

That all of these structures are closely related is indicated not only by

the dimensional relations but also by the results of experiments on trans-
formations produced by heating schairerite, galeite and sulfohalite and
by various features of the single crystal x-ray diffraction patterns. In
Table 6 the lines observed on c axis rotation zero layers in $Na_2SO_4 \cdot NaF$,
schairerite and galeite are tabulated for comparison with the zero layer
of a sulfohalite [111] rotation pattern. The hexagonal indexing for the
sulfohalite spots applies also to the pseudocells of the other materials. The
stronger spots on all of the patterns can be indexed on this base and their
observed intensities are comparable. The projections of the structures on
this base must then be closely similar.

A mere variation of the stacking sequence in sulfohalite would, how-
ever, be insufficient to account for the differences. A sulfohalite cell, as
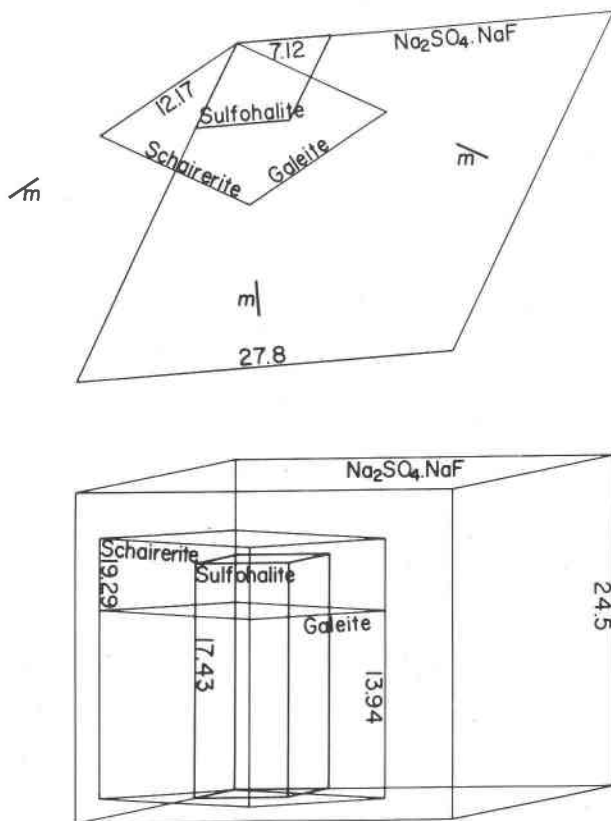drawn in Fig. 7 or Fig. 8B, contains an even number, 6, sulfate groups,

FIG. 7. Outlines of unit cells of multiple salts in the system Na_2SO_4-NaF-NaCl
with common origin and corresponding orientations.

TABLE 6. COMPARISON OF $hki0$ REFLECTIONS FOR $Na_2SO_4 \cdot NaF$,
SCHAIRERITE, GALEITE AND SULFOHALITE

$Na_2SO_4 \cdot NaF$			Schairerite and Galeite			Sulfohalite			
hk	I	d	hk	I	d	hkl	hk^1	I	d
44	10	3.481	30	9	3.513	$2\overline{2}2$	11	10	3.558
			22	4	3.042				
82	2	2.631							
			32	1	2.374				
			41	1	2.300				
12·0	1	2.010	33	2	2.028	$22\overline{4}$	30	2	2.055
88	10	1.741	60	10	1.757	$4\overline{4}0$	22	9	1.779
			43	1−	1.733				
			52	1	1.688				
			61	1	1.607				
12·6	1	1.519							
			44	1−−	1.521				
			71	2	1.396				
16·4	6	1.315	63	5	1.328	$42\overline{6}$	41	6	1.345
			55	1	1.217				
12·12	8	1.160	90	6	1.171	$6\overline{6}0$	33	8	1.186
			82	1+	1.150				
			74	3	1.093				
24·0	9	1.005	66	9	1.014	$44\overline{8}$	60	10	1.027

[1] Hexagonal indexing for sulfohalite referred to unit shown in Table 3 and Fig. 7

apices of the SO_4 tetrahedra in successive layers normal to "c" being in alternate orientations. Such a succession is not possible if the number of SO_4 groups in the cell or pseudocell is odd. Nevertheless, the cell dimensions do suggest 5 layers within the cell normal to c in galeite with a F:Cl ratio of 4:1 and 7 layers in schairerite with a ratio 6:1. In Fig. 9 some properties of the four phases have been plotted as functions of the F:Cl ratio, the data for galeite and schairerite being entered at the points just suggested. The fit of the data to loci representing linear variation with mole proportions is imperfect but the departures are not such as to conflict with the suggested ideal compositions.

POWDER PATTERNS

In Table 7 the powder patterns of the four phases have been tabulated together. Calculated spacings are given only for observed lines because the total number of possible spacings within the range covered is very large for the phases with larger cells. Indexing has been determined by detailed checking against indexed single crystal patterns. In addition in-

dexing of the $Na_2SO_4 \cdot NaF$ and schairerite powder patterns was confirmed by direct superposition of powder patterns on c-axis rotation patterns recorded in the same cassette.

ASTM card 2–0668 is for $Na_2SO_4 \cdot NaF$, referred to erroneously as "artificial schairerite" and indexed on the basis of the dimensions given by Wolfe and Caras (1951), a triple confusion. The pattern, with the exception of a few lines, corresponds to that given for $Na_2SO_4 \cdot NaF$ in Table 7. The name, cell dimensions and indexing of the card are in error.

Sulfohalite $4\left[Na_6(SO_4)_2ClF\right]$
$a_0 = 10.07Å$ Fm3m   Type H5$_8$
T. Watanabé 1934   A. Pabst 1934

$a_h = a_0\sqrt{2}$

$c_h = a_0\sqrt{3}$

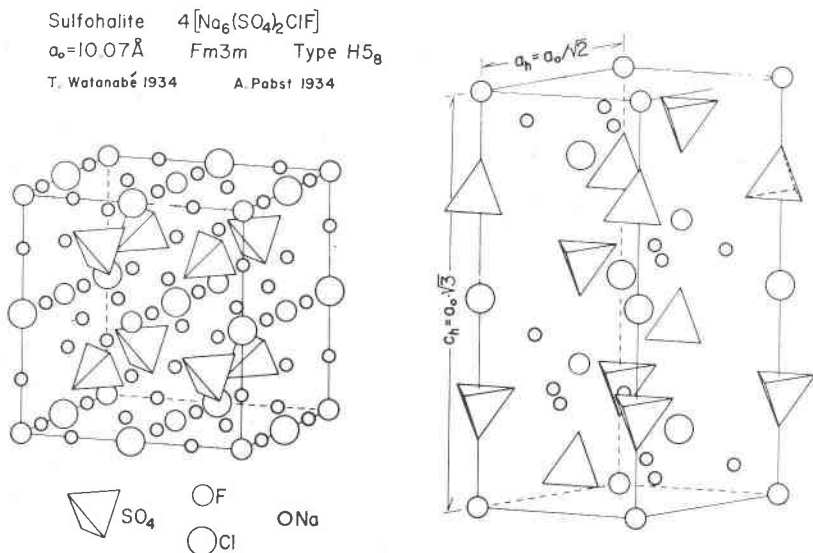SO$_4$ OF   ONa
                Cl

FIG. 8. The crystal structure of sulfohalite. Left, one isometric unit cell. Right, the same structure referred to hexagonal axes defined in Table 3.

COMPLICATIONS IN $Na_2SO_4 \cdot NaF$

Material examined included crystals provided by Dr. Schairer, crystals grown from fusion of composition $Na_2SO_4 \cdot NaF$, sintered material produced from a preparation with a 1:4 ratio of Cl:F, as well as crystals grown from fusion or solution of such a preparation. The sintered, fused and dissolved preparations with 1:4 ratio of Cl:F all yielded a mixture of phases, largely $Na_2SO_4 \cdot NaF$ with some NaCl and Na_2SO_4. X-ray patterns of the principal phase can be fully indexed on the basis of a hexagonal cell with a 27.85, c 24.50 and with systematic absences required by a rhombohedral lattice and an approximation to the Laue symmetry $\overline{3}2/m$, indicating the space groups $R\overline{3}2/m$, $R3m$ or $R32$. Since the lattice dimensions of crystals grown in the presence of NaCl do not differ significantly from
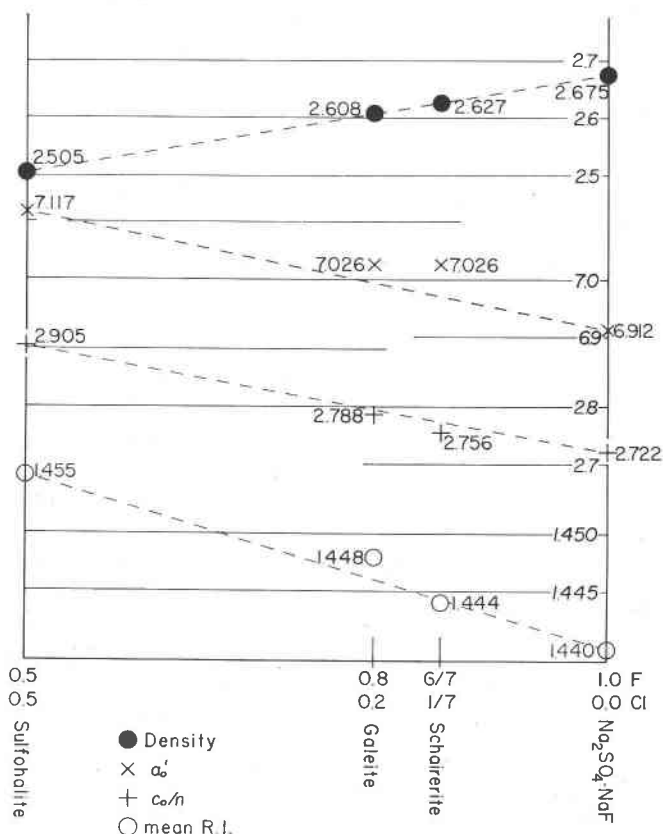
FIG. 9. Some physical constants of multiple salts in the system Na₂SO₄-NaF-NaCl plotted as functions of the mole proportions of F or Cl.

crystals obtained from a fusion of the composition $Na_2SO_4 \cdot NaF$, it is assumed that none of the crystals contained more than a trace of Cl.

The Laue symmetry $\bar{3}2/m$ involves the plane symmetry *6mm* in *hki*0 patterns. Several such patterns, including some obtained from Schairer's crystals, four of which were examined by Weissenberg and precession methods, showed slight departures from this symmetry in the intensities of the spots. Such departures might be attributed to the effects of absorption or crystal missetting, but in the *hki*0 precession pattern obtained from a fragment of a large crystal from a group grown very slowly from aqueous solution with 1:4 ratio of Cl:F at room temperature the departure is pronounced. The pattern clearly has the symmetry *2mm*, though patterns from two smaller crystals of the same group scarcely show the effect. The structure then cannot have a Laue symmetry higher

TABLE 7. X-Ray Powder Diffraction Patterns of Multiple Salts in the System Na₂SO₄-NaF-NaCl

Na₂SO₄·NaF

hk·l	I	$d_{obs.}$	$d_{calc.}$
40.4	4	4.30	4.30
04.5	7	3.79	3.80
30.6, 60.3	½	3.56	3.64, 3.61
52.4			3.53
44.0	8	3.48	3.48
62.1, 21.7	1	3.34	3.31, 3.30
41.6, 44.3	½	3.20	3.23, 3.20
08.1	7½	2.99	2.99
80.2	2	2.93	2.93
08.4	10	2.71	2.71
44.6, 82.0	2	2.64	2.65, 2.63
80.5	7½	2.57	2.57
22.9, 71.6, 64.4, 82.3	1	2.52	2.54, 2.53, 2.52, 2.50
18.5, 26.7, 41.9, 56.3	2	2.44	2.45, 2.42, 2.42, 2.42

Schairerite

hk·l	I	$d_{obs.}$	$d_{calc.}$
00.2	½	9.6	9.65
11.1	½	5.77	5.80
11.2	½	5.14	5.15
11.3	2	4.44	4.42
00.5	½	3.87	3.86
11.4	7	3.79	3.78
21.2	1	3.70	3.68
30.0	8	3.52	3.51
11.5	½	3.26	3.26
30.3, 21.4	½	3.08	3.08, 3.07
22.0	1	3.04	3.04
22.1	6	3.01	3.01
22.2	½	2.91	2.90
11.6, 30.4	1	2.84	2.84, 2.84
00.7, 22.3	10	2.76	2.76, 2.75
10.7, 31.3	½	2.67	2.67, 2.66
22.4	6	2.58	2.57
31.4	½	2.51	2.50

Galeite

hk·l	I	$d_{obs.}$	$d_{calc.}$
11.0	½	6.07	6.08
11.1	1	5.59	5.58
11.2	3	4.58	4.58
11.3	7	3.68	3.69
30.0	8	3.52	3.51
30.2	2	3.14	3.14
22.0, 11.4	4	3.04	3.04, 3.02
22.1	6	2.97	2.97
30.3, 22.2	10	2.79	2.80, 2.79
10.5, 31.2	1	2.70	2.69, 2.69
22.3, 11.5	7	2.55	2.55, 2.54
31.3	2	2.48	2.47
00.6	2	2.32	2.32
40.3, 22.4	3	2.29	2.29, 2.29
41.1	1½	2.27	2.27
41.2, 30.5	2	2.18	2.18, 2.18
32.3	½	2.15	2.15

Sulfohalite

hkl	I	$d_{obs.}$	$d_{calc.}$
111	2	5.81	5.81
200	2	5.03	5.032
220	10	3.56	3.56
311	4	3.03	3.03
222	8	2.91	2.91
400	6	2.51	2.516
331	½	2.30	2.31
420	2½	2.249	2.251
422	1½	2.054	2.055
333, 511	5	1.939	1.937
440	6½	1.779	1.779
531	½	1.700	1.701
600, 442	1	1.677	1.678
620	½	1.591	1.591
622	1	1.518	1.517
444	1	1.452	1.453
711, 551	½	1.410	1.409
642	1	1.346	1.345
731	1	1.310	1.310

TABLE 7. (continued)

	Na₂SO₄·NaF				Schairerite				Galeite				Sulfohalite		
hk·l	I	d_obs.	d_calc.	hk·l	I	d_obs.	d_calc.	hk·l	I	d_obs.	d_calc.	hkl	I	d_obs.	d_calc.
92.1	1	2.37	2.37	00.8	2	2.41	2.41	41.3	3	2.06	2.06	800	½	1.259	1.259
10.0.2			2.37	22.5	1	2.39	2.39	22.5			2.06	733	½	1.232	1.230
08.7	3	2.28	2.28	41.1	1	2.29	2.28	33.0	1	2.03	2.03	820	½	1.221	1.221
84.1			2.27	22.6	2	2.21	2.21	33.2	3	1.949	1.948	822	2	1.185	1.186
60.9			2.25	30.7	1	2.17	2.17	41.4	1	1.922	1.920	662	1	1.154	1.155
22.7	1	2.24	2.25	41.4	½	2.08	2.08	33.3	2	1.861	1.859	840	1½	1.126	1.125
10.0.4			2.24	22.7	2	2.046	2.042	22.6	4	1.848	1.846				
20.11	1½	2.19	2.20	33.2	½	1.981	1.978	60.0	6	1.758	1.757				
81.7			2.20	33.3	2	1.939	1.934	33.4			1.753				
83.5			2.19	22.8	2	1.893	1.890	11.8	1	1.674	1.675				
80.8			2.15	33.4	1	1.871	1.870	22.7	4	1.668	1.666				
44.9	6	2.15	2.14	42.4	½	1.846	1.841	33.5	2	1.641	1.640				
84.4			2.14	33.5	½	1.797	1.795	42.5	½	1.629	1.621				
12.0.3	3½	1.953	1.952	60.0			1.757	52.3	½	1.585	1.586				
1.11.5,	½	1.922	1.923	00.11	7	1.760	1.757	22.8	2	1.513	1.512				
94.5				22.9			1.752								
08.10	3	1.903	1.901	10.11	½	1.726	1.730	44.2	3	1.489	1.486				
0.10.8			1.895	11.11	½	1.684	1.685	60.5			1.485				
82.9			1.892	41.8	½	1.667	1.664	44.3	2	1.448	1.446				
0.11.7	1	1.854	1.858	33.7	2	1.633	1.634								
12.0.6	2	1.802	1.803	22.10			1.629								
88.0	7½	1.743	1.741	33.8	½	1.552	1.552								
75.10	½	1.679	1.681												
12.0.9	1	1.617	1.617												
08.13	3	1.596	1.598												

than $2/m$ though the lattice is dimensionally rhombohedral. Wolters (1910) considered $Na_2SO_4 \cdot NaF$ to be only pseudohexagonal at ordinary temperature with transition to hexagonal at 105° C. Single crystal x-ray examination at elevated temperature would be required to confirm this.

A different remarkable feature was found in three of Schairer's crystals. In addition to the lattice just discussed, patterns obtained from these crystals show another weaker set of sharp spots corresponding to a rhombohedral lattice that can be referred to hexagonal axes coincident with those of the principal lattice but with a one fourth that of the main crystal and c equal to 65.4 ± 0.1 Å, within the limits of uncertainty corresponding to $8/3 \times 24.50 = 65.33$ Å. Spots attributable to this lattice were first noticed on a c-axis rotation pattern and subsequently found and indexed on the $000l$ and $40\bar{4}l$ rows in precession patterns.

POLYCRYSTALS

Large crystals of schairerite from a depth of 121′ 2″ in Hole LS were found to have satellites of galeite attached at the sides. Such satellites are present on nearly every one of a lot of over fifty crystals, mostly 5 to 10 mm long, from this source. In favorable cases the galeite satellites can be recognized as such from the ρ angles of their principal surfaces, 66° 25′, contrasting with ρ $11\bar{2}1$, 72° $29\frac{1}{2}′$, of the schairerite faces to which they are usually attached, Fig. 3A. Their identity was confirmed by single crystal x-ray diffraction on detached outgrowths and later by oscillation patterns with the crystals adjusted so that only selected sections were irradiated. For this a small collimator (0.37 mm diam.) was used.

Subsequently many other combinations of schairerite and galeite were observed in material from hole LS and other sources. Four examples are shown in the upper left quadrant of Fig. 5 and an idealized version of one of the more common types in Fig. 3B. Such syntactic intergrowths are appropriately referred to as polycrystals in the sense of Donnay and Donnay (1953). In many cases they can be recognized goniometrically. This was repeatedly checked by oscillation patterns, sometimes 3 or 4 being taken on different parts of a polycrystal.

The most common polycrystals are largely schairerite with a tip of galeite. Some schairerites have both tips and lateral outgrowths of galeite and some polycrystals are about equally divided between the two. The mere appearance of an outgrowth at the tip of a schairerite crystal is, however, no sure indication of galeite structure. Several apparent polycrystals were measured on which the angles indicated both parts to be schairerite and this was confirmed by x-ray examination. One such pseudopolycrystal is included among the four schairerites pictured in the lower right quadrant in Fig. 5.

In many cases the boundary between the schairerite and galeite parts of polycrystals can be detected not only goniometrically but also by the abrupt though small change in refractive indices. However, transitions do occur. The great majority of galeite crystals show a continuum of diffraction in the c^* direction which can often be noticed in the vicinity of strong spots even on oscillation patterns exposed only twenty minutes, such as were used in the survey of many polycrystals. Figure 10 shows the $22\bar{4}l$ rows from precession patterns of galeite showing but little con-
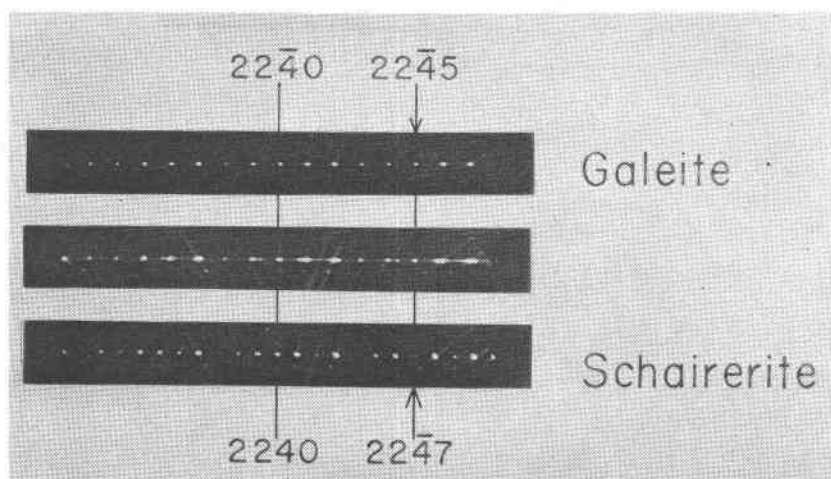

FIG. 10. $22\bar{4}l$ rows from $hh\bar{2}hl$ precession photographs of three crystals. Cu-Ni; $\bar{\mu}=30°$. About 0.54 original size.

tinuum, of schairerite and, between these, galeite with pronounced continuum in which can be seen some schairerite spots. Many galeites show the continuum even though no schairerite spots can be seen so that the continuum must be attributed to stacking disorder within the galeite and is not or not necessarily associated with a recognizable polycrystal. The c^* streaking in galeite patterns might be considered to be connected with variations in the c repeat distance. If this is determined by the Cl/F ratio, the streaking would be symptomatic of local variations therein.

Both tabular and bipyramidal or rhombohedral galeites commonly show the c^* continuum in greater or less degree, but it has not been observed in patterns from a single one of the simple schairerites examined. It seems probable that the striking difference in the morphology of these two closely related minerals, striated surfaces on galeite yielding a near-continuum of signals on the optical goniometer in the $hh\bar{2}hl$ zone and smooth simple forms of schairerite, is an outward expression of a characteristic structural difference.

Though usually only $\{11\bar{2}1\}$ can be surely identified on galeite, many other signals can be recorded, often in multiplets with intervening continuum. On several specimens clear $\{11\bar{2}1\}$ reflections were recorded at both galeite and schairerite angles though distinct segments assignable to the one or the other were not recognizable. These specimens yielded superposed schairerite and galeite diffraction patterns, in a few cases of nearly equal intensity, and may be considered polycrystals with a complex interlocking or interlayering of the parts.

Twins

Referring to $Na_2SO_4 \cdot NaF$, Foote and Schairer (1930a, p. 4206) reported that "twinning on the base is common." Two of the four crystals selected for study from the lot supplied by Schairer are twinned in this manner as indicated by morphology and confirmed by x-rays. One of these crystals is the one in which the superlattice with $c = 65.4$ is most conspicuous. The basal twins are twins of the rhombohedral lattice or
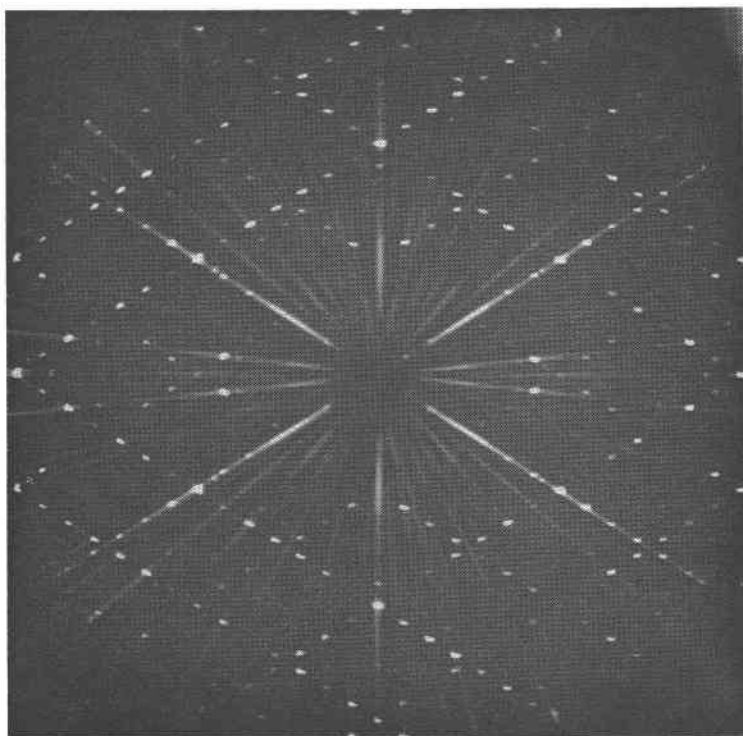

FIG. 11. $hh\bar{2}hl$ precession photograph of schairerite twin on $(\bar{1}1\bar{2}\bar{2})$.
Cu-Ni; $\bar{\mu} = 30°$. About 0.84 original size.

pseudolattice and distinct from any possible multiple twinning relating the lower symmetrical units mentioned under "Complications in Na₂SO₄ ·NaF."

Foshag (1931, p. 136) referred to "a curious habit" of schairerite "perhaps the result of twinning on the plane *c*—shown by crystal 7 (Fig. 4)." During the current work many crystals of this habit were found and several are shown in the upper right quadrant of Fig. 5. It was established by pairs of oscillation patterns taken at the opposite ends of such crystals that they are indeed basal twins. Schairerite basal twins with galeite tips were also found and confirmed goniometrically and by *x*-rays.

Purely by chance another type of twin was found among the schairerite crystals. Though only a single good example was observed this is of such perfection that it can be taken as adequate to establish the existence of another type of twinning in schairerite. Fig. 3D is a drawing of this and the original is included among the twins to be seen in the upper right quadrant of the photograph, Fig. 5. The composition plane, which is also the twin plane, is ($\overline{1}1\overline{2}2$). The labelling of Fig. 3D shows all of the common schairerite forms except the base. In the drawing only a few of the faces in the reentrant angle of the twin can be seen. By making 2-circle goniometric observations in two settings, one that shown in the drawing and another with the *c* axis of one part of the twin as rotation axis, it was possible to identify 9 faces on each part of the twin. The 18 faces of the whole twin correspond to the full development of the three forms, *r*, *e* and *m* with 6 faces each, just half of the faces of each form being developed on each part of the twin.

A zero layer *x*-ray photograph with the precession axis parallel to the composition plane and the edge *r* : *m* shows duplicate $hh\overline{2}hl$ reciprocal lattice planes at an angle of 64° 30′, just twice the complement of $\rho 11\overline{2}2$, to each other. The angle $11\overline{2}2 \wedge \overline{1}1\overline{2}5$ is 89° 53′ on the basis of $c/a = 1.585$ and an effectively orthogonal pseudocell arises in the superposed reciprocal lattice planes, the points $11\overline{2}\cdot 12$, 0007, $11\overline{2}9$, etc. of the one coinciding with $33\overline{6}8$, $22\overline{4}3$, $33\overline{6}1$, etc. of the other. If c/a were $\sqrt{10}/2 = 1.581$, the superposition would be perfect. The slight departure of 7′ cannot be surely detected on the film. The minimum change in lattice dimensions to make $c/a = 1.581$ would require $a = 12.185$ and $c = 19.265$, not far from the values reported but just beyond the presumed limits of error.

In view of the fairly common occurrence of basal twinning in Na₂SO₄ ·NaF and schairerite and of the structural relations it seemed reasonable to expect similar twinning in galeite and sulfohalite. No such twins were found though several galeite crystals that appeared as though they might be twinned were examined by paired oscillation patterns with this in view. Failure to find basal twinning in galeite does not necessarily mean

that it does not exist or even that it is less common than in schairerite. Due to the irregular striated development of galeite such twinning would not be readily detectable from morphology, whereas the perfection and simplicity of schairerite crystals of which many hundreds were inspected in selecting material for analysis makes the regognition of twins easy.

Effects of Heating

The work of Wolters (1910) showed the existence of two ternary eutectics in the system Na_2SO_4-NaF-$NaCl$; at 620° C. between NaF, NaCl and $Na_2SO_4 \cdot NaF$ (hereafter referred to as ds.), and at 594° between Na_2SO_4, NaCl and ds. Since no phases corresponding to minerals of ternary composition in this system were observed by Wolters it was to be expected that sulfohalite, galeite and schairerite would each break down to a mixture consisting of the components of the 594° eutectic upon heating.

Two crystals of sulfohalite, four each of galeite and of schairerite and three polycrystals were used for heating experiments intended not to establish equilibrium relations but only to observe the changes caused by heating and indications of the temperatures and rates at which they occur and, especially, to determine the differences in the behavior of the several minerals upon heating. The single crystals were heated to various temperatures between 177° and 595° C. for periods varying from half an hour to 15 hours. In most cases the same crystal was heated several times at successively higher temperatures, being remounted, examined microscopically, x-rayed and, when its condition permitted, remeasured on the 2-circle goniometer, after each heat treatment. To reduce the large number of x-ray exposures needed for carrying out such a program oscillation patterns were mostly used. In the case of these materials, which all have the diffraction symmetry $\bar{3}2/m$, 60° c-axis oscillation patterns over appropriate ranges include one example of each type of spot and are more surely interpretable than disoriented rotation patterns which would be obtained from imperfectly oriented crystals. All of the crystals used could initially be adjusted to perfection by optical goniometry but after several heatings or after heating to the higher temperatures perfect adjustment by goniometric or microscopic means was impossible.

No changes were detected in any of the three minerals upon heating to 177° for periods up to 15 hours. All were completely changed to the expected combination of phases upon heating to 510° for 3 hours or possibly a lesser period, the changes being complete within a half hour or less at 560°. No effect of annealing on the original state of disorder in galeite was detected prior to the breakdown of the crystals. Both galeite and schairerite retain their shape, though dulled and slightly granulated at

the surface, upon heating even to 595°. Heating of sulfohalite was not carried beyond 510°.

The weight percentages of the phases ideally formed when the changes produced by heat are complete are:

	$Na_2SO_4 \cdot NaF$	Na_2SO_4	NaCl
sulfohalite	47.86	36.94	15.20
galeite	78.59	15.17	6.24
schairerite	84.23	10.89	4.48

taking the Cl/F ratios of the latter two to be $\frac{1}{4}$ and $\frac{1}{6}$ respectively. In all cases the NaCl and Na_2SO_4 formed are represented in the diffration patterns only by smooth powder rings with no suggestion of preferred orientation. The NaCl, though smaller in amount, is more easily detected, especially in the early stages, because of its simple pattern of a few strong lines. The Na_2SO_4 is in form III though a little thenardite may also be present in some cases.

The condition of the ds. formed varies and is more or less characteristic of the starting material. That formed in heated sulfohalite yields powder rings with only a slight indication of any tendency towards formation of larger crystals and no consistent orientation. The ds. formed from galeite or schairerite is always at least in part in the form of a single crystal in common orientation with the initial crystal while more or less of the ds. is in disoriented crystals represented by streaked segments of powder arcs. In no case did the ds. formed from galeite or schairerite give smooth powder arcs. The tendency towards formation of a single oriented crystal of ds. upon heating is much greater for schairerite than for galeite. In the case of one schairerite crystal, heated directly to 595° without preliminary heat treatment, the transformation yielded a ds. single crystal giving a *c*-axis oscillation pattern just as perfect as one from a pure crystal of ds. modified only by the smooth powder rings of the other products.

The difference between the results in galeite and in schairerite is well exemplified by the changes in polycrystals. In the course of the changes the initially clear crystals become milky or porcellaneous and the surface dulled. One polycrystal after heating for 3 hours at 395° was found to be completely porcellaneous in its galeite part while still partly clear at the schairerite end. The paired oscillation patterns taken with the polycrystal in this condition show remnants of the initial single lattices in each part but only streaked powder arcs of ds. in the galeite part whereas the schairerite part yields ds. single crystal spots, mostly in good orientation. Upon completion of the transformation in this same polycrystal by heating one half hour at 560° the differences are most striking. The galeite part

now yields only powder arcs, those for the ds. discontinuous, whereas the schairerite end gives a ds. single crystal pattern only slightly disorganized plus faint arcs for the other products. These observations support the earlier assumption that the Cl/F ratio is significantly lower in schairerite than in galeite.

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