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SANTAFEITE, A NEW HYDRATED VANADATE FROM NEW MEXICO*

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ABSTRACT

Santafeite is a new hydrated vanadate found in the Grants uranium district, McKinley County, New Mexico, in the spring of 1951. It has the formula $Na_2O \cdot 3MnO_2 \cdot 6(Mn,Ca,Sr)O \cdot 3(V,As)_2O_5 \cdot 8H_2O$. The mineral occurs as an encrustation of small rosettes of acicular crystals on an outcrop joint surface of Todilto limestone. Physical and optical properties are: perfect (010) and distinct (110) cleavages, very brittle, measured density 3.379, black color, brown streak, subadamantine luster, readily fusible in an alcohol flame to a dull black bead; translucent only in very small fragments, pleochroic from dark reddish brown to yellowish brown, with absorption $X>Y>Z$; $X=c$, $\alpha=2.01$, and distinct dispersion. *X*-ray studies by rotation, Laue, Weissenberg, and powder diffraction methods indicate orthorhombic symmetry, space group $B22_12$, D_2^5, with cell dimensions: $a_0=9.25\pm.02$ Å; $b_0=30.00\pm.02$ Å; $c_0=6.33\pm.02$ Å.

The mineral is named santafeite, after the Atchison, Topeka and Santa Fe Railroad Company, in recognition of its pioneer exploration and development of the uranium deposits in New Mexico.

INTRODUCTION

In the course of a reconnaissance of uranium mineral deposits in the Grants district in the spring of 1951, a small sample of santafeite was collected by Weber from a cliff-face outcrop of the Jurassic Todilto limestone in section 25, T. 13 N., R. 10 W. The locality is about 12 miles north of Grants; approximately one mile northward in McKinley County from the McKinley-Valencia County line, New Mexico.

Preliminary optical, *x*-ray, and semiquantitative spectrographic analysis (which reported significant amounts of silica, alumina, manganese, and vanadium) led to the tentative identification of the mineral as ardennite (abstract, *Am. Mineral.*, **40**, 338, 1955). Through the courtesies of Drs. Hugo Strunz and Clifford Frondel the writers received a sample of ardennite from Salm Chateau, Belgium. Comparison of the *x*-ray powder pattern of the Grants material with the Salm Chateau ardennite showed conclusively that the Grants material was not ardennite. A second, more precise, semiquantitative spectrographic analysis revealed that the unknown mineral was not a silicate, but a hydrated manganese vanadate.

Insufficient material was present in the original sample to permit a

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complete chemical analysis, so the locality was revisited by the writers in the summer of 1954. It was found that the outcrop from which the original sample was taken had been destroyed by subsequent mining operations, and none of the santafeite left exposed in 1951 remained. A second search of most of the cliff edge and surface mine workings in the Todilto limestone between San Mateo road and Haystack Mountain in 1955 was also fruitless. Fortunately, however, an additional 100 milligram sample of santafeite was collected in the summer of 1956 by Mr. Robert Thaden and Dr. Alice D. Weeks of the U. S. Geological Survey, from a shallow open pit along the rim outcrop of the Todilto limestone in Section 26, which adjoins the location of the original sample.

The name santafeite (san-ta-fay-ite) was adopted in recognition of the work of the Atchison, Topeka and Santa Fe Railroad Company, which pioneered in the exploration and development of the uranium deposits of what soon became the most important producing district in New Mexico, and on whose property the mineral was first discovered.

Occurrence

Santafeite was originally found at one point on the cliff-face outcrop of the Todilto limestone, which in this area is a thin-bedded sequence aggregating 8 to 16 feet in thickness (Towle and Rapaport, 1952). A subsequent discovery by Mr. Robert Thaden, and identification by Dr. Alice D. Weeks is noted above. The Todilto limestone is underlain by Entrada sandstone, and capped by what is probably the equivalent of the Summerville formation, succeeded upward by the Morrison formation. The mineral occurred as an encrustation on, and intergrowth with, crystalline calcite coating a nearly vertical joint surface in the limestone. Deposition of both santafeite and calcite was clearly controlled by jointing. Sparse flecks of cuprosklodowskite are present on some of the santafeite crystals, and more conspicuously developed along an irregular surface that roughly parallels the plane of the bedding of the limestone. The occurrence is adjacent to exploratory pits that expose yellow uranium-vanadium minerals. Other minerals that have been identified in Todilto limestone deposits of the area, but not known to be directly associated with the santafeite, include carnotite, tyuyamunite and metatyuyamunite, uranophane, beta-uranotil, uraninite, fluorite, barite, marcasite, pyrite, galena, psilomelane, hematite, and montmorillonite (Gruner, Towle, and Gardiner, 1951; Towle and Rapaport, 1952; Gruner, Gardiner, and Smith, 1954; Weeks and Thompson, 1954).

Physical and Optical Properties

Santafeite occurs as well-defined rosettes of radially oriented acicular crystals (Fig. 1). Individual rosettes range from about 1.5 mm. to 4 mm.

FIG. 1. Santafeite rosettes with associated calcite (c) on joint surface of Todilto limestone.

in diameter. Some of them are triangular in profile along the diameter, suggesting an original spherulitic form from which the upper two-thirds has been broken away. A typical rosette 2.8 mm. in diameter consists of crystals measuring approximately 0.3 mm. along the *a*-axis, 0.07 mm. along the *b*-axis, and 1.4 mm. along the *c*-axis. The small size and fragile character of individual crystals prohibited optical goniometric measurements. Of the forms present, however, the *b*-face is especially prominent.

Cleavages are (010) easy and perfect, and (110) distinct; very brittle; color black; streak brown; luster subadamantine; readily fusible in an alcohol flame to a dull black bead. The density, determined by suspension in Clerici's solution, is 3.379. The mineral is transluscent only in very small fragments, which show pronounced pleochroism ranging from dark reddish brown to yellowish brown, with absorption $X > Y > Z$. Extinction is parallel, with $X = c$, $\alpha = 2.01$ by the immersion method. Dispersion is distinct.

CHEMICAL COMPOSITION

Acting upon the courteous suggestion of Dr. Michael Fleischer, a sample of santafeite was submitted to the Geochemistry and Petrology Branch of the U. S. Geological Survey for spectrographic and microchemical analysis. According to Dr. Alice D. Weeks (letter of April 1, 1957), "The mineral sample looks quite pure and consists of clean, micro-

scopic, prismatic crystals, but the spectrographic analysis shows many trace constituents. Although the mineral appears to be essentially a hydrated calcium manganese vanadate, many of the minor elements must substitute for the major constituents. Several of the minor elements have to be determined in order to make a satisfactory total for the analysis." The results of the semi-quantitative spectrographic analysis are listed in Table 1.

The small amount of the original sample, and the additional 100 mg. of santafeite subsequently collected by Mr. Robert Thaden and Dr. Alice D. Weeks were processed for partial quantitative micro-chemical analysis in the U. S. Geological Survey Laboratory. The results of this analysis are listed in Table 2. At the suggestion of Mr. Robert Meyrowitz, the analyst, a footnote describing the procedures and methods used is at the end of Table 2, so that "one can better evaluate the reliability of an analytical result when the method used for the determination is known. This is especially true in the analysis of small amounts of material," (Robert Meyrowitz, letter of June 10, 1957). The writers believe that the procedures and methods used by Mr. Meyrowitz provide a valuable reference for those who have similar problems in the chemical analysis of small samples of a complex mineral.

The chemical formula of santafeite, calculated from the partial quantitative micro-chemical analysis, is $Na_2O \cdot 2MnO_2 \cdot 6(Mn,Ca,Sr)O \cdot 3(V,As)_2O_5 \cdot 8H_2O$. The Fe_2O_3, CoO, NiO, CO_2, CuO, UO_3, and SiO_2 shown by the chemical analysis to be present in santafeite are considered to be impurities contributed by associated hematite, limestone, and cuproskłodowskite.

Because of the complex nature of the chemical composition, and the small quantity of the analyzed sample, the calculated chemical formula of santafeite may be subject to revision. The analysis shows a total of 96.2 per cent, of which several per cent probably represent impurities. Based on the measured density and the unit cell dimensions of the mineral, the calculated molecular weight of santafeite is 1787.41, assuming that there are two molecules in the unit cell. However, the molecular weight based on the calculated chemical formula of santafeite is only 1449.12. The measured density is 3.379, but some crystals deviate slightly from this value, leading one to suspect that a portion of the discrepancy in the molecular weights may be due to the difference in density of the individual crystals.

X-Ray Analysis

Cell dimensions are $a_0 = 9.25 \pm .02$ Å, $b_0 = 30.00 \pm .02$ Å, and $c_0 = 6.33 \pm .02$ Å, calculated from the $h00$, $0k0$, and $00l$ reflections on the Weis-

TABLE 1. SEMI-QUANTITATIVE SPECTROGRAPHIC ANALYSIS OF SANTAFEITE FROM MCKINLEY COUNTY, NEW MEXICO

Analyst: Katherine E. Valentine, Naval Gun Factory Laboratory, Geochemistry and Petrology Branch, U. S. Geological Survey

Si	.3	Eu	0	Ru	0
Al	.1	F	—	Sb	0
Fe	.3	Ga	0	Sc	.001
Mg	.1	Gd	0	Sn	.003
Ca	.3	Ge	0	Sr	1.0
Na	1.0	Hf	0	Sm	0
K	0	Hg	0	Ta	0
Ti	.01	Ho	0	Tb	0
P	0	In	0	Te	0
Mn	M	Ir	0	Th	0
Ag	0	La	.01	Tl	0
As	.3	Li	0	Tm	0
Au	0	Lu	0	U	.1
B	0	Mo	.001	V	M
Ba	.1	Nb	0	W	0
Be	0	Nd	.03	Y	.003
Bi	0	Ni	.1	Yb	.0003
Cd	0	Os	0	Zn	0
Ce	.1	Pb	.03	Zr	.003
Co	.1	Pd	0		
Cr	.0003	Pr	.01		
Cs	0	Pt	0		
Cu	.1	Rb	0		
Dy	.03	Re	0		
Er	.03	Rh	0		

Figures are reported to the nearest number in the series 10, 3, 1, .3 etc., in per cent. Eighty per cent of the reported results may be expected to agree with the results of quantitative methods.

Symbols used are: — not looked for; 0 looked for, but not detected; M major constituent greater than 10%.

NOTE: The standard sensitivities for the elements determined by the semi-quantitative spectrographic method are those established by the Naval Gun Factory Laboratory, Geochemistry and Petrology Branch, U. S. Geological Survey.

senberg photographs. *X*-ray powder diffraction data obtained through use of a Norelco 114.59 mm. diameter powder camera, and a Nore'co Geiger counter *x*-ray diffractometer, and the calculated indices of reflections are listed in Table 3. The measured *d* values are in fairly good agreement with the calculated values. The space group, designated as

TABLE 2. PARTIAL CHEMICAL ANALYSIS OF SANTAFEITE

(Analyst: Robert Meyrowitz, Naval Gun Factory Laboratory, Geochemistry and Petrology Branch, U. S. Geological Survey, Washington, D. C.

Constituent	%	Footnote	Constituent	%	Footnote
V_2O_5	35.6	h	CoO	0.1	d, g
MnO_2	16.6	i	NiO	0.1	e, g
MnO	13.7	i	CuO	0.5	f, g
CaO	6.2	j, o	UO_3	0.3	n, o
SrO	6.0	k, o	Insol+SiO_2	0.8	b, g
Na_2O	4.1	l, o	H_2O	8.8	q
K_2O	<0.1	m, o	CO_2	0.3	q
As_2O_5	2.2	p	Total	96.2	
Fe_2O_3	0.9	c, g	$H_2O(-)$	0.2	a, g

$B22_12$, D_2^5, was determined according to the condition of non-extinction of reflections shown by Weissenberg photographs. The criteria of non-extinction are: $(h+l)$ is even for hkl, l is even for $0kl$, h is even for $hk0$, $(h+l)$ is even for $h0l$, h, k, and l are even for $h00$, $0k0$, and $00l$ respectively.

Santafeite is highly brittle, tending to break even under very slight pressure. Small grains about 0.2 mm. long and 0.1 mm. thick were selected for single crystal study by Laue, rotation, and Weissenberg methods. Laue photographs were taken with the x-ray beam parallel to each of the three crystallographic axes. The Laue photograph with x-ray beam parallel to the a-axis (Fig. 2A) shows most reflections sharply defined. A photograph with beam parallel to the c-axis is similar to Fig. 2A. A photograph with beam parallel to the b-axis is shown in Fig. 2B, in which both sharp reflections and diffuse streaks are evident. Indices of some of the reflections were obtained from a gnomonic projection of this photograph. An orthorhombic symmetry is indicated for santafeite by the Laue patterns.

Rotation photographs about the a-axis and c-axis (Fig. 2D) show only layer lines of the first kind, with sharp reflections. The rotation photograph about the b-axis (Fig. 2C), however, shows so-called layer lines of the second kind that are characterized in part by sharp spots, and in part by diffuse streaks. Indices of the reflections along the zero layer line, derived by the graphic method, are listed in Table 4. Indices of the diffuse streaks on the zero layer line are 101, 301, 103, and 303. It appears that the diffuse streaks result from layer displacements of santafeite in the ac plane.

TABLE 3. X-RAY POWDER DIFFRACTION DATA: Fe RADIATION, Mn FILTER
FeKα=1.93728 Å

	d, Å, (Meas.)	*d*, Å (Calc.)	*hkl*	I
1	14.88	15.00	020	10
2	7.47	7.50	040	5
3	5.01	5.00	060	1
4	4.56	4.57	210	1
5	4.19	4.20	230	1
6	3.66	3.66	250	3
7	3.39	3.39	260	3
8	3.13	3.14	270	2
9	2.908	2.913	280	3
10	2.788	2.799	052	4
11	2.702	2.704	290	5
12	2.607	2.612	202	3
13	2.600	2.600	341	2
14	2.461	2.467	242	1
15	2.395	2.408	082	1
16	2.311	2.313	400	1
17	2.197	2.199	2 12 0	2
18	2.139	2.135	0 14 0	1
19	2.106	2.111	1 13 1	1
20	2.033	2.036	3 10 1	1
21	1.862	1.867	402	2
22	1.826	1.835	432	2
23	1.817	1.812	442	2
24	1.746	1.748	531	1
25	1.710	1.712	472	1
26	1.667	1.667	0 18 0	4
27	1.656	1.657	2 14 2	2
28	1.579	1.583	004	2
29	1.539	1.542	600	1
30	1.514	1.513	4 15 0	2
31	1.493	1.494	2 19 0	1
32	1.458	1.462	2 17 2	1
33	1.401	1.405	2 18 2	2
34	1.385	1.389	513	2
35	1.366	1.367	5 14 1	1
36	1.205	1.206	791	2

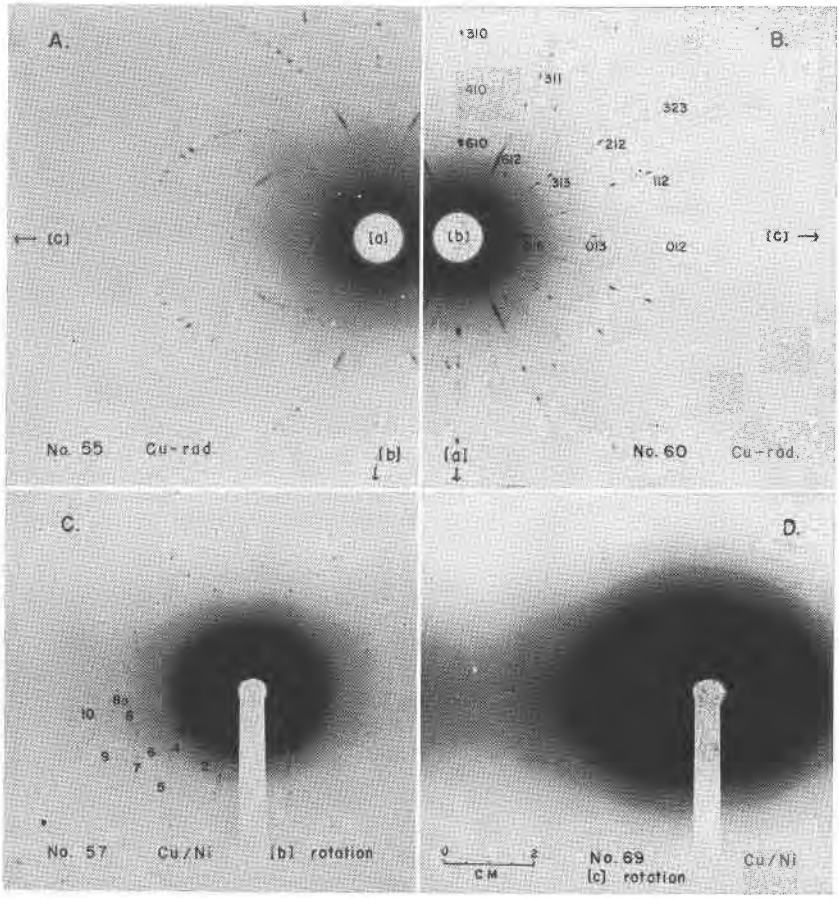

FIG. 2. A. Laue photograph of santafeite, *x*-ray beam parallel to *a*-axis. B. Laue photograph with *x*-ray beam parallel to *b*-axis; distance between crystal and film 6.64 cm. Note sharp spots and diffuse streaks reflections. C. *b*-axis rotation photograph. Note sharp spots and diffuse streaks of layer lines of the second kind. D. *c*-axis rotation photograph; camera diameter 57.3 mm. Scale of all four photographs indicated on Fig. 2D.

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The writers gratefully acknowledge the essential contributions to this report of several persons, all of whom are staff members of the U. S. Geological Survey. Dr. Michael Fleischer, at whose suggestion, and through whose courtesies the analyses were provided. Dr. Alice D. Weeks, who facilitated the analytical work in the Naval Gun Factory Laboratory of the Geochemistry and Petrology Branch, generously sup-

TABLE 4. INDICES OF SHARP SPOT AND DIFFUSE STREAK REFLECTIONS OF THE *b*-AXIS ROTATION PHOTOGRAPH

Nos of layer lines of the 2nd kind	ξ	*hkl* (along zero layer line)		I
		Diffuse reflections	Sharp reflections	
1	.30	101		vs
2	.33		200	s
3	.56	301		m
4	.60		202	m
5	.67		400	w
6	.75	103		w
7	.83		402	s
8	.89	303		w
8a	.98		004	vs
9	1.02		600	vw
10	1.11		602	w

plied additional material for analysis that she and Mr. Robert Thaden collected, and offered many helpful suggestions. Mr. Robert Meyrowitz made the very difficult quantitative microchemical analysis, and provided the footnote outline of the procedure used. Miss Katherine E. Valentine contributed the spectrographic analysis. To these and others who assisted them, the writers extend their sincere thanks.

Dr. Abraham Rosenzweig of the University of New Mexico kindly helped in the space group interpretation.

The selection of the procedures used for the chemical analysis was based on the semi-quantitative spectrographic analysis of the mineral by Katherine E. Valentine, TWS-2841, August 31, 1956.

Footnotes:

(a) Dried to constant weight at 110±5° C.

(b) The "Insoluble+SiO_2" was determined gravimetrically by dehydration in a hydrochloric acid solution of the mineral using a quartz crucible.

(c) Fe_2O_3 was determined spectrophotometrically by the *o*-phenanthroline procedure using an aliquot of the filtrate from the "Insoluble+SiO_2" determination.

(d) CoO was determined spectrophotometrically by the nitroso-R salt procedure (Acetate Medium) as described by E. B. Sandell, "Colorimetric Determination of Traces of Metals, 2nd edition, 1950, page 277, using an aliquot of the filtrate from the "Insoluble+SiO_2" determination.

(e) NiO was determined spectrophotometrically using dimethylglyoxime according to the procedure of W. Oelschläger, *Z. Anal. Chem.*, **146,** 346–50 (1955). An aliquot of the filtrate from the "Insoluble+SiO_2" determination was used.

(f) CuO was determined spectrophotometrically using sodium diethyldithiocarbamate according to the procedure of K. L. Cheng and R. H. Bray, *Anal. Chem.*, **25,** 655–59 (1953). An aliquot of the filtrate from the "Insoluble+SiO_2" determination was used.

(g) The sample size for the $H_2O(-)$, "Insoluble+SiO_2," Fe_2O_3, CoO, NiO and CuO determinations was approximately 18 mg.

(h) The total vanadium calculated as V_2O_5 was determined spectrophotometrically using the hydrogen peroxide procedure. The sample was dissolved using hydrochloric acid. After oxidation by ammonium peroxydisulfate, the precipitate of manganese dioxide formed was dissolved by the addition of hydrogen peroxide. Orthophosphoric acid was used to mask the iron present. The sample size was approximately 4 mg. It was found that the mineral was not completely decomposed by boiling (1+3) H_2SO_4. The mineral was completely decomposed by evaporation to SO_3 fumes.

(i) The total manganese was determined spectrophotometrically using the peroxydisulfate procedure described by E. B. Sandell, Colorimetric Determination of Traces of Metals, 2nd Edition, 1950, pages 433–34. The sample size was approximately 2 mg. The total "available oxygen" was determined as follows: The sample and a weighed amount of $FeSO_4 \cdot (NH_4)_2SO_4 \cdot 6H_2O$ were boiled in a (1+9) H_2SO_4 solution under an atmosphere of N_2. The sample was completely decomposed. After the addition of ortho-phosphoric acid, the solution was titrated with standard 0.03N $K_2Cr_2O_7$ using sodium diphenylamine sulfonate as an indicator, to determine the excess $Fe(SO_4) \cdot (NH_4)_2SO_4 \cdot 6H_2O$. The sample size was approximately 7 mg. The "available oxygen" was arbitrarily calculated as MnO_2 after correcting for that due to V_2O_5. The remaining manganese is reported as MnO.

(j) CaO was determined by flame photometry (wave length = 554 mμ). The sample solution was compared to standard calcium solutions containing approximately the same concentration of sodium, strontium, vanadium, manganese, and iron present in the solution of the sample.

(k) SrO was determined by flame photometry (wave length = 461 mμ). The sample solution was compared to standard strontium solutions containing approximately the same concentration of calcium, sodium, vanadium, manganese, and iron present in the solution of the sample.

(l) Na_2O was determined by flame photometry (wave length = 589 mμ). The sample solution was compared to standard sodium solutions containing approximately the same concentrations of calcium, strontium, vanadium, manganese, and iron present in the solution of the sample.

(m) K_2O was determined by flame photometry (wave length = 765 mμ). The sample solution was compared to standard potassium solutions containing approximately the same concentrations of sodium, calcium, strontium, vanadium, manganese, and iron present in the solution of the sample.

(n) UO_3 was determined fluorimetrically by Grafton J. Daniels.

(o) A hydrochloric acid solution of the sample was used for the CaO, SrO, Na_2O, K_2O and UO_3 determinations. The sample size was approximately 16 mg. A Beckman Flame Spectrophotometer (DU) was used for the flame photometry. The flame was hydrogen-oxygen.

(p) As_2O_5 was determined spectrophotometrically after separation as the bromide according to a modified procedure of J. C. Bartlet, Margaret Wood, and R. A.

Chapman, *Anal. Chem.*, **24**, 1821–24 (1952). The sample size was approximately 1.3 mg.

(q) H_2O and CO_2 were determined by use of modified microcombustion train of the type used for the determination of carbon and hydrogen in organic compounds. The sample was decomposed by ignition at 900° C. in a stream of oxygen. The sample size was approximately 20 mg.

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