

CHEMICAL COMPOSITION AND PHYSICAL, OPTICAL,
AND STRUCTURAL PROPERTIES OF BENITOITE,
NEPTUNITE, AND JOAQUINITE¹

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ABSTRACT

The chemical composition, density, optical properties, and cell parameters of benitoite, neptunite, and joaquinite from San Benito County, California, are reported. Electron microprobe analyses show that benitoite is homogeneous, stoichiometric $\text{BaTiSi}_3\text{O}_9$. Electron microprobe and emission spectrographic data indicate that neptunite is fairly homogeneous and that its formula is $\text{Li}_{0.8}\text{Na}_{2.1}\text{K}_{0.9}(\text{Fe},\text{Mg},\text{Mn})_{2.0}\text{Ti}_{2.0}\text{Si}_{8.0}\text{O}_{24}$. Joaquinite is quite complicated chemically, containing hydroxyl and 15-20 weight percent rare earth oxides. The density and optical properties for each mineral and cell parameters for benitoite and neptunite are similar to those given in the literature. Weissenberg and precession X-ray diffraction data indicate that single crystals of joaquinite have varying proportions of a monoclinic and an orthorhombic component. The orthorhombic unit cell has parameters $a = 10.48$ (2) Å, $b = 9.66$ (2) Å, and $c = 22.26$ (2) Å; the monoclinic unit cell has parameters $a = 10.51$ (2) Å, $b = 9.66$ (2) Å, $c = 11.82$ (2) Å, and $\beta = 109.5$ (2)°. Structural, chemical, and infrared data suggest the following formula for joaquinite:



INTRODUCTION

The three rare minerals benitoite, neptunite, and joaquinite occur in natrolite veins which cut a glaucophane schist inclusion in a serpentinite body near New Idria, San Benito County, California. Louderbach and Blasdale (1909) described this occurrence in detail; they gave wet chemical analyses for benitoite and neptunite and physical and optical properties for all three minerals. No further chemical data for benitoite have been reported. Benitoite is of crystallographic interest because it is the only known member of the ditrigonal-dipyramidal symmetry class; its structure was determined by Zachariassen (1930). Recently, Fischer (1969) refined the crystal structure of benitoite and confirmed Zachariassen's (1930) results.

Wet chemical analyses of neptunite from this locality were given by Bradley (1909) and more recently by Cannillo, Mazzi, and Rossi

¹ Contribution number 1979.

(1966). A summary of the optical properties and geologic occurrences of neptunite was given by Heinrich and Quon (1963). Berry (1963) and Cannillo *et al.* (1966) reported crystal structure data for San Benito neptunite.

Palache and Foshag (1932) determined the physical and crystallographic properties and chemical composition of California joaquinite. Other occurrences of joaquinite have been discussed by Bell (1963) and Semenov, Bukin, Balashov, and Sørensen (1967). Semenov *et al.* (1967) found 22.59 weight percent $(RE)_2O_3$ (rare earth oxides) in joaquinite from southern Greenland and suggested that there were different minerals of the joaquinite group; they also reported 11.5 and 15.0 percent $(RE)_2O_3$ in two samples of joaquinite from California but did not give complete chemical analyses. All the published work on joaquinite has reported it as orthorhombic. However, J. E. Rowland (written communication from E. H. Nickel, 1970) has found joaquinite from San Benito County with a monoclinic unit cell.

The purpose of this work was to determine the complete chemical composition and other properties of benitoite, neptunite, and joaquinite from San Benito County, California. A secondary objective was to determine whether the electron microprobe techniques used in this laboratory could be employed successfully to analyze such a complex mineral as joaquinite.

ANALYTICAL TECHNIQUES

Electron microprobe analyses were made with a three-channel Applied Research Laboratory model EMX microprobe using the techniques of Bence and Albee (1968) and Albee and Ray (1970). The accelerating voltage was 15 kv at all times; and the beam current, pulse height selection, spot size, and counting time were constant for each element analyzed but adjusted for each element and mineral to obtain maximum counting rates with minimum sample damage and contamination. Simple silicates and oxides were used as standards for the major elements; the rare earth elements of joaquinite were analyzed relative to apatite, thalenite, and monazite. Ba and Ti in benitoite were analyzed relative to Bafeldspar and synthetic rutile, respectively; whereas, Ba and Ti in neptunite and joaquinite were analyzed relative to benitoite, assuming stoichiometry for the benitoite. Previous studies in this laboratory indicate an accuracy for common elements constituting more than about one weight percent of the sample of about two percent of the amount present. Standards and correction parameters for less common elements are not as good, and their abundance in joaquinite somewhat decreases the accuracy for the common elements.

The emission spectrographic analyses are an average of duplicate burns (except for the joaquinite samples, which were only run once because of the small amount of sample available) on a 3.4 m Wadsworth-mount spectrograph with a 15,000 line per inch diffraction grating. A D.C. arc with a 19 amp short circuit was used. The samples were diluted in the proportions: 1.0 sample, 0.1 Na_2CO_3 , 0.5

graphite, and 0.9 quartz. For benitoite and neptunite 25 mg of this mixture were weighed into the electrode and burned to completion; for joaquinite 10 mg were used.

X-ray diffraction powder work was done in a Guinier camera using quartz-monochromatized Cu $K\alpha$ radiation. Single-crystal work was done in Supper Weissenberg and precession cameras with Fe $K\alpha$ and Mo $K\alpha$ radiation, respectively. The unit cell dimensions for benitoite and neptunite were calculated by a least squares technique using previously indexed lines; 44 lines were fitted for benitoite and 34 for neptunite. Indices were calculated for benitoite from the cell parameters of Fischer (1969) and for neptunite from the parameters of Berry (1963).

The densities were determined on a Roller-Smith Berman balance using toluene. For benitoite and neptunite determinations were made on individual grains, weighing from 4–40 mg, and averaged. Because of the limited amount of material available, the density of joaquinite was determined by weighing 2–4 mg samples composed of several grains three times. Indices of refraction were determined with Na light and index oils calibrated with a Zeiss Abbé refractometer. Those indices which were matches with the oils are cited as accurate to 0.001; indices bracketed by two oils are cited as accurate to 0.002.

The infrared spectrum of joaquinite was measured on a Perkin-Elmer 225 infrared spectrophotometer using a KBr pellet, 1.5 mg sample to 400 mg KBr. The spectrum was normalized to that of a similar 400 mg KBr pellet, prepared simultaneously, in the reference beam.

Benitoite, neptunite, and joaquinite were analyzed from five samples from the California Institute of Technology collection. Benitoite was chipped from sample D4, neptunite from samples D2636 and D6794, and joaquinite from samples D2200, D7574, and D4. Joaquinite was taken from sample D6794 by dissolving the enclosing natrolite in concentrated HCl. In order to obtain chemical data consistent with other properties, densities, optical properties, and cell parameters were determined on grains of the same samples that were used for the chemical analyses.

RESULTS AND DISCUSSION

Benitoite

The chemical composition of benitoite is delineated in Table 1. Benitoite may be blue or white to colorless. Both colors occur within single crystals and are separated by distinct contacts. Louderback and Blasdale (1909) suggested a systematic relationship between color change and crystallographic axes, but no systematic and consistent relationship was apparent in the crystals available to us.

The SiO_2 , BaO, and TiO_2 contents of blue benitoite (analysis 1) are virtually identical to those of white benitoite (analysis 2) from the same crystal and closely approach the stoichiometric composition of $\text{BaTiSi}_3\text{O}_9$ (SiO_2 , 43.59 percent; TiO_2 , 19.32 percent; BaO, 37.09 percent). Our results are similar to the original analyses for blue and white benitoite by Louderback and Blasdale (1909) (Table 1, analy-

ses 3 and 4) but do not support their reported differences in TiO_2 and BaO . Standard deviations for the averages of the individual analyses are less than half a percent, indicating both the blue and the white benitoite are homogeneous (see also Figure 1).

Electron beam scans and analytical profiles across color contacts indicate no detectable variation in composition for Ba, Ti, and Si. Emission spectrographic analyses were made to see if differences existed below the sensitivity of the electron microprobe (see Table 1). Although they show only small differences in the trace element contents of the blue and white benitoite, the difference in iron content may be significant.

Table 1. Chemical Analyses of Benitoite, San Benito County, California

| | 1 | | | 2 | | | 3 | 4 |
|-------------------------|------------|----------------------|-------------------|------------|----------------------|-------------------|--------|--------|
| | Microprobe | | E.S. ^a | Microprobe | | E.S. ^a | | |
| | Wt. % | Formula ^b | Wt. % | Wt. % | Formula ^b | Wt. % | | |
| SiO_2 | 43.10 | 2.98 | ---- | 42.62 | 2.97 | ---- | 43.68 | 43.61 |
| Al_2O_3 | d | | 0.2 | d | | 0.04 | ---- | ---- |
| TiO_2 | 19.51 | 1.01 | ---- | 19.44 | 1.02 | ---- | 20.09 | 19.50 |
| FeO^c | d | | 0.05 | d | | 0.01 | ---- | ---- |
| MgO | d | | Trace | d | | Trace | ---- | ---- |
| CaO | d | | 0.1 | d | | 0.006 | ---- | ---- |
| BaO | 37.23 | 1.01 | Major | 37.27 | 1.02 | Major | 36.33 | 37.01 |
| Na_2O | 0.13 | 0.02 | ---- | 0.14 | 0.02 | ---- | ---- | ---- |
| Nb | d | | ---- | d | | 0.14 | ---- | ---- |
| Sr | d | | 0.0035 | d | | 0.0025 | ---- | ---- |
| V | d | | 0.0015 | d | | 0.001 | ---- | ---- |
| Zr | d | | 0.012 | d | | 0.08 | ---- | ---- |
| Total | 99.97 | | | 99.47 | | | 100.10 | 100.12 |

^a Emission spectroscopy analysis by E. Bingham. Looked for but not found: Ag, As, Au, B, Be, Bi, Cd, Ce, Co, Cr, Cu, Ga, Gd, La, Mo, Mn, Nd, Ni, Pb, Pt, Sb, Sc, Sn, Ta, Th, Tl, W, Y, Yb, and Zn.

^b Cation formula proportion determined by normalizing anhydrous oxygen to 9.

^c Total Fe as FeO .

^d Less than the sensitivity of a wavelength scan (~ 0.10 wt. percent).

1. D4, blue color. Microprobe analysis is the average of 6 points.

2. D4, white color. From the same crystal as (1). Microprobe analysis is the average of 6 points.

3. Blue color. Louderback and Blasdale (1909).

4. White color. Louderback and Blasdale (1909).

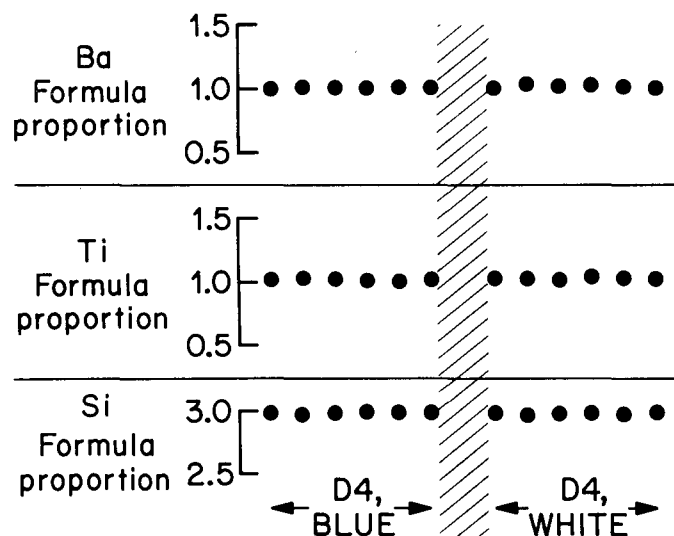


FIG. 1. Scatter diagram for the cation formula proportions of benitoite samples from San Benito County, California. Each point represents a complete microprobe analysis. Formula proportions are determined by normalizing the analyses to anhydrous oxygen = 9.

Benitoite has very intense cathodoluminescence under the electron beam and is commonly used by microprobe operators to check beam size and shape. Many of these operators have found that benitoite contains no detectable elements other than Ba, Ti, Si, and O and have used it as a microprobe standard assuming stoichiometry.

The density of benitoite is 3.64(2) gm/cc; it is uniaxial positive with $\omega = 1.756(1)$ and colorless and $\epsilon = 1.800(1)$ and blue. Louderback and Blasdale (1909) reported $\rho = 3.64\text{--}3.67$ gm/cc, $\omega = 1.757$, and $\epsilon = 1.804$.

From single-crystal work on benitoite from San Benito County, Fischer (1969) reported that benitoite is hexagonal with the space group $P\bar{6}c2$; $a = 6.6410(7)$ Å and $c = 9.7597(10)$ Å. A Guinier X-ray powder pattern gives $a = 6.63(1)$ Å and $c = 9.73(1)$ Å. The d -spacings are in good agreement with the ASTM index.

Neptunite

Chemical data for neptunite are summarized in Table 2. The microprobe analyses are similar to those reported in the literature except that Cannillo *et al.* (1966) reported 1.63 percent Li_2O . It is impossible to analyze for lithium with the microprobe because of its low atomic

number; however, our analytical totals are one percent low, and qualitative emission spectroscopic data indicate 1-2 percent Li_2O . The formula $\text{LiNa}_2\text{K}(\text{Fe,Mg,Mn})_2(\text{TiO})_2(\text{Si}_8\text{O}_{22})$ was suggested for neptunite by Cannillo *et al.* (1966). In the absence of Al, the formula proportions can be calculated from the analyses by normalizing Si to 8. Our analyses normalized in this manner give $\text{Li}_{0.6}\text{Na}_{2.1}\text{K}_{0.9}(\text{Fe,Mg,Mn})_{2.0}\text{Ti}_{2.0}\text{Si}_8\text{O}_{24}$. This formula is also obtained from the analyses by normalizing total cation charges (exclusive of Li) to 47 or total cations (exclusive of Li) to 15. The formula proportion of Li

Table 2. Chemical Analyses of Neptunite, San Benito County, California

| | 1 | | | 2 | | | 3 | 4 | 5 |
|-------------------------|--------------------|----------------------|-------------------|--------------------|----------------------|--------------------|--------|--------|--------|
| | Microprobe | | E.S. ^a | Microprobe | | E.S. ^a | | | |
| | Wt. % | Formula ^b | Wt. % | Wt. % | Formula ^b | Wt. % | | | |
| SiO_2 | 53.96 | 8.00 | ---- | 54.09 | 8.00 | ---- | 53.44 | 52.87 | 52.29 |
| Al_2O_3 | d | | 0.04 | d | | 0.1 | ---- | ---- | ---- |
| TiO_2 | 17.74 | 1.98 | ---- | 17.99 | 2.00 | ---- | 17.18 | 17.83 | 17.35 |
| FeO | 11.71 ^c | 1.45 | 8 ^c | 10.75 ^c | 1.33 | 10 ^c | 11.23 | 11.69 | 11.92 |
| MnO | 1.40 | 0.18 | 1.5 | 1.65 | 0.21 | 1.3 | 1.78 | 0.85 | 2.27 |
| MgO | 1.73 | 0.38 | 1.2 | 2.16 | 0.48 | 1.2 | 1.82 | 1.44 | 1.55 |
| CaO | d | | 0.02 | d | | 0.04 | 0.25 | 1.56 | 0.62 |
| BaO | 0.25 | 0.01 | 0.06 | 0.18 | 0.01 | 0.06 | ---- | ---- | ---- |
| Na_2O | 7.38 | 2.12 | ---- | 7.51 | 2.15 | ---- | 9.14 | 9.56 | 6.81 |
| K_2O | 4.84 | 0.92 | ---- | 4.76 | 0.90 | ---- | 5.39 | 5.08 | 5.58 |
| Li_2O | e | 0.59 ^f | 1-2 | e | 0.54 ^f | 1-2 | ---- | ---- | 1.63 |
| Ag | d | | 0.0003 | d | | 0.0002 | ---- | ---- | ---- |
| Cr | d | | 0.0023 | d | | 0.0048 | ---- | ---- | ---- |
| Co | d | | 0.01 ^g | d | | 0.014 ^g | ---- | ---- | ---- |
| Nb | d | | < 0.01 | d | | < 0.01 | ---- | ---- | ---- |
| Ni | d | | 0.0028 | d | | 0.0038 | ---- | ---- | ---- |
| V | d | | 0.005 | d | | 0.006 | ---- | ---- | ---- |
| Zr | d | | 0.0065 | d | | 0.0054 | ---- | ---- | ---- |
| Total | 99.01 | | | 99.09 | | | 100.23 | 100.88 | 100.02 |

^a Emission spectroscopy analysis by E. Bingham. Looked for but not found: As, Au, B, Be, Bi, Cd, Cu, Ga, La, Mo, Pb, Pt, Sb, Sc, Sn, Sr, Ta, Th, Tl, W, Y, Yb, and Zn.

^b Cation formula proportion determined by normalizing Si to 8.

^c Total Fe as FeO.

^d Less than the sensitivity of a wavelength scan (~0.10 wt. percent).

^e Cannot determine with the microprobe.

^f Calculated by assigning the deficiency in the analytical total from 1.00 to Li_2O .

^g Possible interference.

1. D2636. Microprobe analysis is the average of 12 points on 7 grains.

2. D6794. Microprobe analysis is the average of 7 points on 4 grains.

3. Louderback and Blasdale (1909).

4. Bradley (1909).

5. Cannillo, *et al.* (1966).

in the analyses is calculated by assigning the deficiency of the analytical total from 100 percent to Li_2O ; if the totals are about half a percent high, the formula proportion of Li would be 1.

In the reported analyses (Table 2), iron is given as FeO. Bancroft, Burns, and Maddock (1967) have determined from Mössbauer spectroscopy on neptunite from California that at least 95 percent of the iron is present as Fe^{2+} , corroborating the formula given above.

Figure 2 is a scatter diagram of the cation formula proportions of neptunite for each point analyzed with the microprobe. Although there is a scatter in the Fe, Mg, and Mn contents of single points, the total Fe+Mg+Mn proportion is constant. Electron beam scan photographs also show that neptunite is quite homogeneous.

The density and optical properties of neptunite agree well with those reported in the literature for this and other occurrences. The density is 3.20 (2) gm/cc compared with the values 3.19–3.23 gm/cc listed in Murdoch and Webb (1948). Optical properties of neptunite determined on sample D6794 are as follows: $2V (+) \sim 40^\circ$; optic plane $\parallel (010)$; $y = b$; $Z \wedge c = 17^\circ$; $\alpha = 1.692(1)$, pale yellow; $\beta = 1.702(1)$, yellow orange; and $\gamma = 1.734(2)$, red orange to red brown.

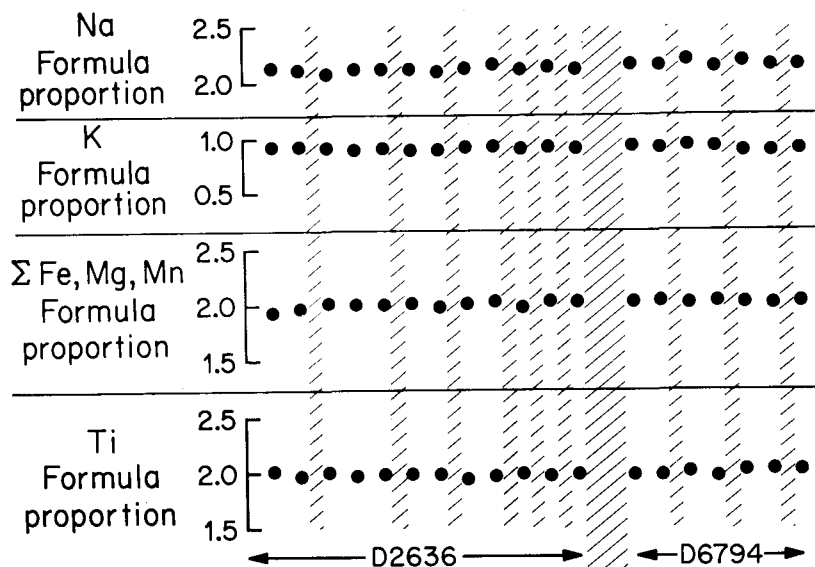


FIG. 2. Scatter diagram for the cation formula proportions of neptunite samples from San Benito County, California. Each point represents a complete microprobe analysis, and analyses for each grain are grouped on the figure. Formula proportions are determined by normalizing the analyses to $\text{Si} = 8$.

Those reported by Larsen and Berman (1934) on California (?) neptunite are: $2V = 49^\circ$, $Z \wedge c = 16^\circ$, $\alpha = 1.690$, $\beta = 1.699$, and $\gamma = 1.736$.

Cannillo *et al.* (1966) proposed that neptunite is monoclinic, space group Cc , but reported no new cell parameters. Guinier powder data on neptunite from sample D6794 give cell dimensions and d -spacings similar to those reported by Berry (1963). Our parameters are $a = 16.48(3) \text{ \AA}$, $b = 12.49(2) \text{ \AA}$, $c = 10.00(2) \text{ \AA}$, and $\beta = 115.4(1)^\circ$; Berry (1963) reported $a = 16.7 \text{ \AA}$, $b = 12.4 \text{ \AA}$, $c = 10.0 \text{ \AA}$, and $\beta = 115^\circ 44'$ from single-crystal precession photographs.

Joaquinite

The density of the joaquinite from sample D6794 is $3.98(5) \text{ gm/cc}$, compared with 3.89 gm/cc given by Palache and Foshag (1932). Our data indicate that joaquinite is biaxial positive with a range in $2V$ from ~ 30 – 55° . The indices are: $\alpha = 1.753(1)$, $\beta = 1.767(1)$, and $\gamma = 1.822(2)$; α and β are colorless and γ is pale yellow. Palache and Foshag (1932) reported $2V = 50^\circ$, $\alpha = 1.748$, $\beta = 1.767$, and $\gamma = 1.823$.

The chemical composition of joaquinite is complex; electron microprobe and emission spectroscopy data are presented in Table 3 with other chemical data on joaquinite taken from the literature. Thirty-eight points on 15 grains of 4 samples were analyzed with the microprobe. Many grains are inhomogeneous, but joaquinite from sample D6794 is quite homogeneous; all of our data on the density and optical and structural properties of joaquinite were determined on grains of this sample.

The infrared spectrum of joaquinite is presented in Figure 3. The sharp bands at ~ 3500 and 3560 cm^{-1} indicate that the joaquinite structure includes significant quantities of crystallographically-ordered hydroxyl groups. The band at $\sim 1610 \text{ cm}^{-1}$ (corresponding to an HOH bending motion) and the broad absorption feature centered at $\sim 3400 \text{ cm}^{-1}$ (the OH stretch) are probably due to water adsorbed during sample handling procedures. The C-H bands around 2900 cm^{-1} are due to organic impurities introduced during sample handling. In this spectrum the lower energy region is dominated by the strong Si-O absorption near 1000 cm^{-1} . However, infrared spectroscopic data on silicate minerals is insufficient to assign this Si-O absorption pattern to a particular type of silicate.

We are unable to determine directly the amount of H_2O that is present in joaquinite because of the small amount of material available and the small size of the crystals. The reported H_2O content is based on the difference between the analyzed oxide total and 100 percent and

may not be very accurate. Qualitative emission spectrographic analyses indicate that Li is present but in an amount less than 1 percent Li_2O . There may be some Fe_2O_3 , although Semenov *et al.* (1967) reported only 0.39 percent Fe_2O_3 in joaquinite from southern Greenland. The presence of these components will affect the value reported as H_2O .

The number of rare earth elements present in joaquinite makes it difficult to do accurate microprobe analyses because their peaks are close together. However, total rare earth oxide concentration determined by electron microprobe and by emission spectroscopic analyses differs by only 0.5 weight percent for sample D6794 and by 2 weight percent for sample D2200. Total rare earth oxide concentration in the 38 microprobe analyses of joaquinite ranges from 15–20 weight percent; this is zero to 8.5 weight percent higher than that reported by Semenov *et al.* (1967) for two samples of joaquinite from California (see Table 3).

Joaquinite is listed in the literature as orthorhombic. However, J. F. Rowland has found a monoclinic crystal from the California locality (E. H. Nickel, written communication, 1970). Single-crystal

Table 3. Chemical Analyses of Joaquinite

| | 1 | | 2 | | 3 | | 4 | | 5 | 6 | 7 | 8 |
|--------------------------------|-------------------|----------------------|-------------------|----------------------|-------------------|----------------------|-------------------|----------------------|-------|-------|-------|--------|
| | Microprobe | E.S. ^a | Microprobe | E.S. ^a | Microprobe | Microprobe | Microprobe | Microprobe | | | | |
| | Wt. % | Formula ^b | Wt. % | Formula ^b | Wt. % | Formula ^b | Wt. % | Formula ^b | Wt. % | Wt. % | Wt. % | Wt. % |
| SiO_2 | 34.97 | 32.00 | 35.17 | 32.00 | 35.17 | 32.00 | 34.93 | 32.00 | 36.4 | ---- | ---- | 33.82 |
| TiO_2 | 11.83 | 8.14 | 12.24 | 8.37 | 11.41 | 7.81 | 11.90 | 8.21 | 30.5 | ---- | ---- | 9.20 |
| Fe_2O_3 | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | ---- | 0.39 |
| FeO | 4.09 ^c | 3.13 | 3.76 ^c | 2.86 | 4.55 ^c | 3.46 | 4.25 ^c | 3.33 | 3.5 | ---- | ---- | 4.78 |
| MnO | 0.00 | ---- | 0.08 | 0.06 | 0.06 | 0.05 | 0.00 | ---- | ---- | ---- | ---- | 0.70 |
| HgO | 0.05 | 0.07 | 0.09 | 0.12 | 0.32 | 0.43 | 0.16 | 0.21 | 0.3 | ---- | ---- | ---- |
| BaO | 22.44 | 8.05 | 22.36 | 7.97 | 21.99 | 7.84 | 22.50 | 8.08 | 24.7 | 18.0 | 18.1 | 21.46 |
| CaO | 0.21 | 0.20 | 0.32 | 0.31 | 0.45 | 0.44 | 0.38 | 0.37 | ---- | ---- | 8.1 | 0.03 |
| SrO | 3.20 | 1.70 | 3.84 | 2.03 | 3.36 | 1.77 | 3.54 | 1.88 | ---- | ---- | ---- | ---- |
| Na_2O | 1.87 | 3.32 | 1.75 | 3.09 | 1.81 | 3.19 | 1.82 | 3.24 | 4.6 | ---- | ---- | 2.41 |
| K_2O | 0.03 | 0.04 | 0.04 | 0.05 | 0.04 | 0.05 | 0.03 | 0.03 | ---- | ---- | ---- | 0.22 |
| Li_2O | e | < 1 | e | < 1 | e | e | e | ---- | ---- | ---- | ---- | ---- |
| ThO_2 | 0.27 | 0.06 | 0.84 | 0.17 | 0.63 | 0.13 | 0.76 | 0.16 | ---- | ---- | ---- | 0.38 |
| Nb ₂ O ₅ | d | ---- | d | ---- | d | d | d | ---- | ---- | ---- | ---- | 2.31 |
| Ce_2O_3 | 10.69 | 3.58 | 9.91 | 3.30 | 11.32 | 3.77 | 11.51 | 3.86 | ---- | 6.8 | 5.6 | 9.40 |
| Pr_2O_3 | 0.21 | 0.06 | 0.21 | 0.06 | < 0.2 | 0.19 | 0.06 | 0.05 | 0.02 | ---- | 0.1 | ---- |
| Er_2O_3 | 0.00 | 0.1 | 0.06 | 0.02 | < 0.1 | 0.03 | 0.01 | 0.02 | 0.01 | ---- | 0.1 | ---- |
| Gd_2O_3 | 0.26 | 0.08 | 0.14 | 0.04 | 0.8 | 0.05 | 0.02 | 0.00 | ---- | ---- | 0.3 | ---- |
| La_2O_3 | 2.14 | 0.72 | 3.0 | 2.52 | 2.9 | 2.82 | 0.95 | 2.99 | 1.01 | ---- | 3.6 | 2.3 |
| Nd_2O_3 | 3.21 | 1.05 | 3.5 | 2.82 | 0.92 | 2.7 | 2.37 | 1.87 | 0.61 | ---- | 3.2 | 2.1 |
| Pr_2O_3 | 1.25 | 0.42 | 1.5 | 1.17 | 0.39 | 0.4 | 1.13 | 0.37 | 1.06 | 0.35 | 1.0 | 0.6 |
| Sm_2O_3 | 0.70 | 0.22 | 0.7 | 0.58 | 0.18 | 0.4 | 0.36 | 0.11 | 0.17 | 0.05 | 0.1 | 0.4 |
| Ta_2O_5 | d | < 0.02 | d | < 0.04 | d | d | d | ---- | ---- | ---- | ---- | ---- |
| V_2O_5 | 0.70 | 0.34 | 0.5 | 0.33 | 0.16 | 0.1 | 0.26 | 0.13 | 0.06 | 0.03 | 0.3 | ---- |
| H_2O | 19.16 | 6.47 | 18.7 | 17.74 | 5.92 | 15.8 | 18.53 | 6.19 | 17.73 | 5.94 | 15.0 | 11.5 |
| F | e | 11.49 ^f | e | 10.75 ^f | e | 10.21 ^f | e | 11.62 ^f | ---- | ---- | ---- | 1.50 |
| $-\text{OH}^2$ | d | ---- | d | ---- | d | d | d | ---- | ---- | ---- | ---- | 0.38 |
| Total | 98.12 | ---- | 98.23 | ---- | 98.32 | ---- | 98.10 | ---- | 100.0 | ---- | ---- | 100.01 |

a Emission spectroscopy analysis by E. Singham. b Cation formula proportion determined by normalizing Si to 32. c Total Fe as FeO. d Less than the sensitivity of a wavelength scan (~0.10 wt. percent). e Cannot determine with the microprobe. f Calculated by assigning the deficiency in the analytical total from 1.00 to H_2O .
 1. D6794, San Benito County, California. Microprobe analysis is the average of 6 points on 5 grains.
 2. D2200, San Benito County, California. Microprobe analysis is the average of 21 points on 7 grains.
 3. D7574, San Benito County, California. Average of 6 points on 2 grains.
 4. D4, San Benito County, California. Average of 5 points on 1 grain.
 5. San Benito County, California. Talache and Footag (1932). Analysis reduced to 100 percent.
 6. San Benito County, California. Semenov, *et al.* (1967).
 7. San Benito County, California. Semenov, *et al.* (1967).
 8. S. Greenland. Semenov, *et al.* (1967).

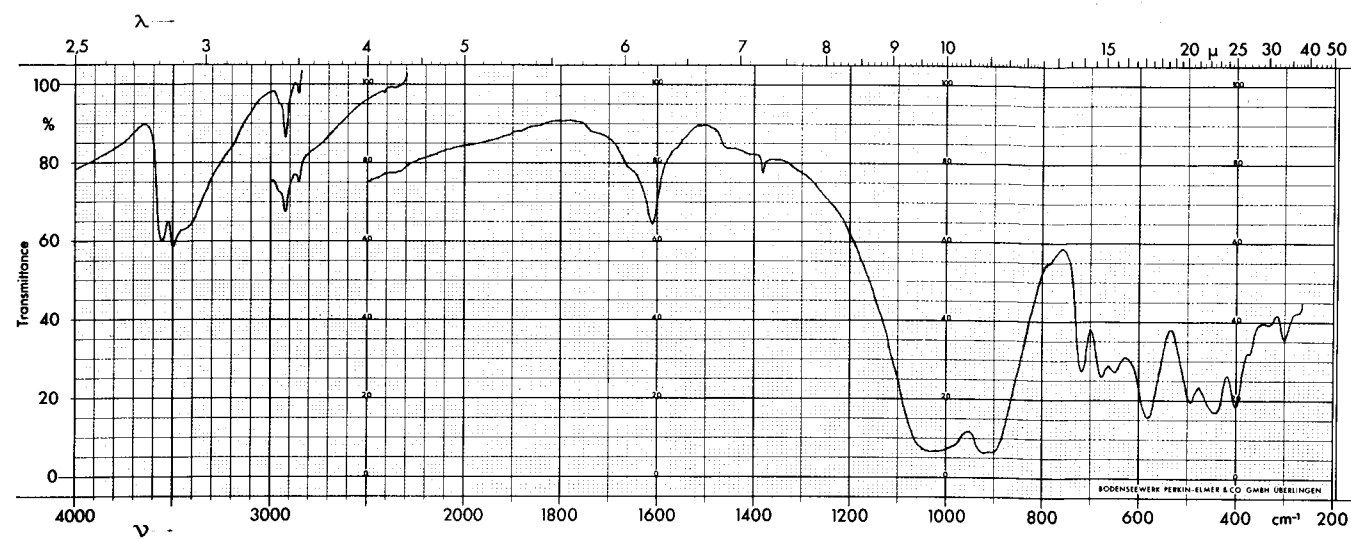


FIG. 3. Infrared spectrum of joaquinite from sample D6794 from San Benito County, California.

