CHEMICAL DIFFERENCES AMONG THE SERPENTINE "POLYMORPHS"[1]

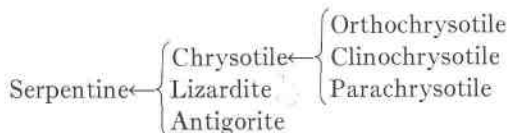NORMAN J PAGE, *U.S. Geological Survey, Menlo Park, California*

ABSTRACT

Existing wet chemical analyses and structural studies of the serpentine minerals, chrysotile, lizardite, and antigorite suggest that these minerals have different chemical compositions and could be identified by composition alone. Triangular composition diagrams, a statistical treatment of the oxide components, MgO, FeO, Fe_2O_3, Al_2O_3, and H_2O, and calculated mineral formulae from chemical analyses all suggest that chrysotile, lizardite, and antigorite are not polymorphs. Antigorite is distinguished by its comparatively low H_2O and high SiO_2 contents. Chrysotile is characterized by a relatively high H_2O and MgO content and by a small ratio of Fe_2O_3 to FeO; while lizardite has high SiO_2 and low FeO contents.

INTRODUCTION

Chrysotile, lizardite, and antigorite compose the serpentine-group minerals. Much discussion has been centered around the nature of the relations among these mineral species and the ultimate question: Are the serpentines polymorphs or are they separate but similar mineral species which can be distinguished by chemical analyses? Though the answer to this question is controversial at present, it is important for the interpretation of alteration phenomena of ultramafic rocks. Although past chemical and crystallographic studies indicate that the serpentine-group minerals have essentially the same structural foundation and similar formulae, the many existing chemical analyses and structural studies have not been consolidated into a cohesive interpretation. These results are synthesized in this paper to show that the serpentine-group minerals, based on the available analyses, are either members of a complex solid-solution series or are separate chemical species, but are not simply polymorphs. A polymorph is a substance that crystallizes in more than one form or crystal structure, all forms or structures having identical chemical compositions.

Unfortunately, in the past, a large number of names have been applied to individual samples of serpentine-group minerals, generally based on textures and appearance of the specimens. Faust and Fahey (1962) provide a comprehensive discussion of this aspect so that it need not be considered here. The best classification of the minerals in the serpentine group is that of Whittaker and Zussman (1956) based on the X-ray diffraction results. This classification is used here as given below:

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[1] Publication authorized by the Director, U. S. Geological Survey.

$$\text{Serpentine} \begin{cases} \text{Chrysotile} \begin{cases} \text{Orthochrysotile} \\ \text{Clinochrysotile} \\ \text{Parachrysotile} \end{cases} \\ \text{Lizardite} \\ \text{Antigorite} \end{cases}$$

The present study was begun at the University of California where most of the work was done; research has been continued at the U.S. Geological Survey.

STRUCTURE OF THE SERPENTINE GROUP MINERALS

Although other structures for serpentines have been proposed, the evidence presented by Warren and Hering (1941), Aruja (1945), and Whittaker (1953) demonstrates that all serpentine minerals, including the fibrous varieties, have a trioctahedral structure analogous to dioctahedral kaolinite. As discussed by Bates (1959) and Deer, Howie, and Zussman (1962), a major factor repsonsible for the different serpentine minerals is the amount of mis-matching involved in joining a brucite-like layer to a tridymite-like layer to form an unit cell of serpentine. At least four possibilities for deriving such structures have been postulated: (1) curving the composite sheets with the tridymite layer forming the concave part of the cylinder, (2) distorting one or both layers, (3) substituting various ions in either layer to adjust the sizes of the layers, and (4) creating cation vacancies in the octahedral layer and maintaining charge balance by removing an equivalent amount of hydroxyl ions. The structure of chrysotile is controlled by (1) and (3) with (1) the dominant mean of matching the sheets (Whittaker, 1956a). Method (4) combined with (1) and (3) is responsible for the structure of antigorite (Kunze, 1961). Another factor in the development of the different structures of serpentine minerals is the possibility of both regular and irregular stacking of the layers.

Chrysotile. Fankuchen and Schneider (1944) suggested, on the basis of small angle X-ray scattering, that chrysotile consists of fibers on the order of 200 Å in diameter packed in trigonal array along the fibers. Since Turkevich and Hiller (1949) first used an electron microscope to demonstrate that chrysotile apparently had a "hollow tube" morphology, abundant evidence based on the electron microscope observations has accumulated (Bates, Sand, and Mink, 1950; Noll and Kircher, 1950, 1951; and Maser, Rice, and Klug, 1960). These observations are supported by the detailed structural studies of Whittaker (1957). In a recent paper Huggins and Shell (1965) reported bulk densities of chrysotile ranging from 2.2 to 2.4 g/cc. Using cylinder diameters of 340 Å and 80 Å as outer and inner diameters, respectively, the theoretical bulk density,

including pore space is 2.19 to 2.25 g/cc. This demonstrates that chryso-tile has hollow or partially filled hollow tubes.

Three varieties of chrysotile are recognized from single-fiber studies: clinochrysotile, orthochrysotile, and parachrysotile (Whittaker, 1953, 1955, 1956a,b,c, and 1957). Ortho- and parachrysotile are orthorhombic while clinochrysotile is monoclinic (neglecting the curvature). In clino- and orthochrysotile, the fibers are curved about the a-axes, and any regular stacking or translational-ordered arrangements would be in a $+a$ or $-a$ direction. By the nature of the fiber, translational order cannot occur in the b direction, so that the atoms at the base of layers would have to be restricted to circumferential grooves. Clinochrysotile (Whit-taker, 1956a) has a two-layered cell in which the successive sheets are offset in the same direction (either $+a$ or $-a$), with alternate layers offset by different amounts. Orthochrysotile (Whittaker, 1956b) has a two-layered cell and has an orthorhombic cylindrical lattice. Alternate layers are offset in the $+a$ and $-a$ direction. The third variety, para-chrysotile, is defined with b as the axis of curvature and has an ortho-rhombic cylindrical lattice. Whittaker (1956c) shows there can be no analogous partner to parachrysotile as there is for clino- and orthochryso-tile.

From Whittaker's study (1956a,b,c) and Whittaker and Zussman (1956), it appears that clinochrysotile is the most abundant, ortho-chrysotile is next most abundant, and parachrysotile is fairly rare or mixed with the other two in small amounts.

Lizardite. Observations under the electron microscope show that lizardite has a platy morphology (Deer, Howie, and Zussman, 1962). Early powder work by Whittaker and Zussman (1956) on samples described but not named by Midgely (1951), suggested that lizardite has a single-layered unit cell and is pseudo-orthohexagonal, individual layers having trigonal symmetry. This was confirmed by the single-crystal analysis of Rucklidge and Zussman (1965). They found that lizardite is disordered in three ways: (1) the crystals are macroscopically bent about more than one crystallographic axis; (2) some layers are rotated by 180°; and (3) some layers are displaced by $+(b/3)$. There are two possible orienta-tions of a lizardite layer which can be combined in three ways to produce stacking arrangements in an individual crystal.

Antigorite. The structure of antigorite, the other distinct serpentine min-eral, has been elucidated by Aruja (1945), Zussman (1954, 1956), Kunze (1956, 1957, 1958, 1961), Zussman, Brindley and Comer (1957) and Chapman and Zussman (1959). Under the electron microscope antigorite

exhibits both a platy and fibrous morphology, but there are no suggestions of cylindrical tubes. Although antigorite is a single-layered serpentine whose c and b dimensions are very similar to lizardite, it's a parameter is large and in many cases approximates 40 Å. On the basis of the single-crystal studies of Kunze (1956, 1958, 1961) and Zussman (1956), the structure involves deficiencies of hydroxyl and magnesium ions. These deficiencies are repeated along the a axes. The resulting structure is then composed of "half-waves joined by means of Mg-bridges" (Kunze, 1956).

The most frequently occurring a parameters for antigorites studied by electron diffraction (Zussman, Brindley, Comer, 1957; Chapman and Zussman, 1959) are 33.7, 35.8, 41.2, and 43.0 Å. Zussman and co-workers found that different grains of one sample yielded values of a of 41, 90, and 110 Å, while other fibrous antigorites had a parameters in the range of 16 to 10 Å (Chapman and Zussman, 1959). Kunze (1961) has treated the variation of a parameters from a structural and theoretical approach and considers that antigorites have compositions between talc and chrysotile which accounts for the octahedral cation and $(OH)^-$ deficiencies. From his structural approach, the various observed values of the a parameter can then be derived theoretically.

Chemical Compositions of the Serpentine Minerals

Althouth the formula normally quoted in mineralogical textbooks for serpentine is $Mg_3Si_2O_5(OH)_4$, or two times this formula, natural serpentines are rarely, if ever, composed only of magnesia, silica, and water. In the general formula $X_6Y_4O_{10}(OH)_8$, X represents the possibility for the substitution of the ions Mg^{2+}, Fe^{2+}, Fe^{3+}, $Co^{?+}$, Ni^{2+}, Mn^{2+}, Mn^{3+}, Cr^{3+}, Cu^{2+}, Al^{3+}, and Ti^{4+} in octahedral coordination; Y represents Si^{4+}, Al^{3+}, B^{3+} (?), and Fe^{3+} in tetrahedral coordination; and OH represents $(OH)^-$, Cl^-, F^-(?), and Br^-(?).

Criteria and selection of analyses. In addition to the ions mentioned above, serpentine analyses frequently show some K^{1+}, Na^{1+}, and Ca^{2+}, that are probably not in the structure but are due to a mineral impurity in the analyzed concentrate. Even the exact amounts of Fe^{2+}, Fe^{3+}, Cr^{3+}, Ti^{4+}, and Al^{3+} in the structure may not always be known because of the fine-grain size of the serpentine and its intimate association with fine-grained spinels or hydrated oxides, such as goethite or lepidocrocite, which makes physical separation of the minerals almost impossible. It is also suspected that in some cases the amount of magnesia reported is incorrect and is really due to the mineral impurity brucite. In considering the analytical results, both structural state and purity require careful

attention and definition. Analyses of serpentines for which there is not sufficient X-ray, optical and textural, DTA and infrared evidence to determine the mineral species are not considered here.

The group of analyses considered here was selected from 248 analyses of serpentine mineral done between 1834 and 1962 as compiled by Faust and Fahey (1962). Their discussion (p. 7, 19) of the sources of the analyses indicates that the literature was well covered and that a wide assortment of types was selected by them. For the present study, analyses (Appendix) made on material apparently free of mineralogical impurities and identified as to mineral type, were chosen. Five new analyses (Page, 1967) were added to this group. Comments concerning each analysis used are given in the Appendix; these comments indicate why every analysis was not used in each of the following figures. All of the analyses were recalculated by computer[1] to mineral formulae, using the hydrogen-equivalent method. Alkalis and $H_2O(-)$ were rejected for the purposes of the calculations.

Compositional variations of serpentine. In Figure 1, the variation in the composition of serpentine is represented as weight percent of a specific oxide versus the normalized number of analyses. The five major components, MgO, SiO_2, FeO, Fe_2O_3, and Al_2O_3, are the only oxides considered. Water is discussed later. The number of analyses out of the 86 for which the amount of the oxides is available, is indicated in the figure.

The frequency diagrams show that serpentines have a fairly limited range of composition. An inspection of the percentage of sample versus weight percent of oxides indicates little chemical difference among the various species. The analyses were recalculated to total weight percentages of the sets of components MgO, SiO_2, H_2O; MgO, FeO, SiO_2; and MgO, FeO, H_2O. The amount of total iron was calculated as FeO and only $H_2O(+)$ was used for the H_2O component except where only total water was determined. Figure 2 shows the recalculated analyses for chrysotiles, lizardites, and antigorites.

On the MgO, SiO_2, H_2O diagram (Fig, 2a), lizardite and chrysotile compositions do not differ significantly. Antigorites tend to be higher in SiO_2 and lower in H_2O and MgO than lizardite and chrysotile but some overlap of the ranges exists which could be caused by errors in separation, identification, or analysis. Lizardites and antigorites tend to have higher total iron contents than chrysotiles as is demonstrated in the diagram for MgO, FeO, SiO_2 (Fig. 2b). Again, antigorites are grouped separately from lizardites. In the compositional diagram for MgO, FeO, H_2O (Fig.

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[1] Jackson (in press) designed the computer program.
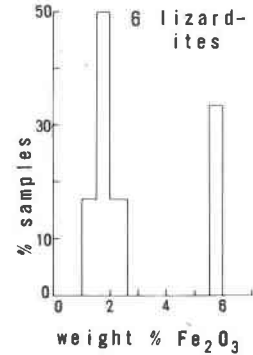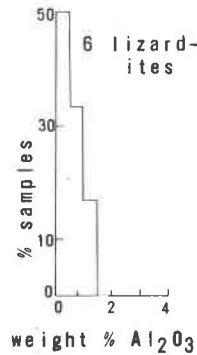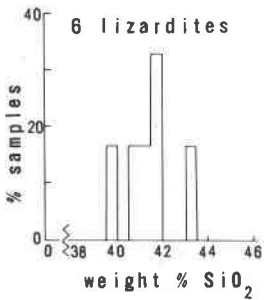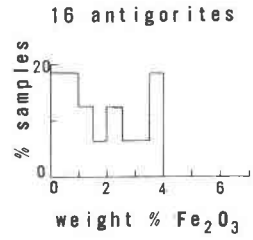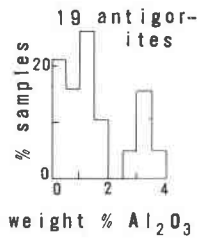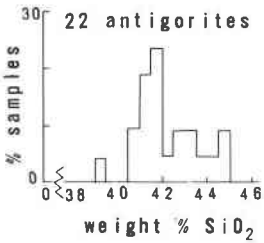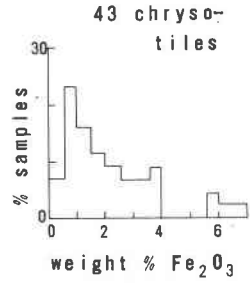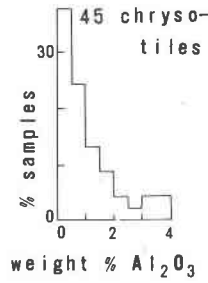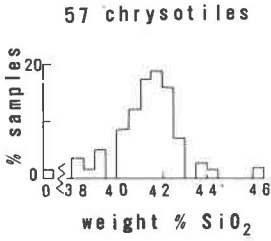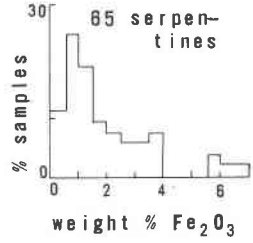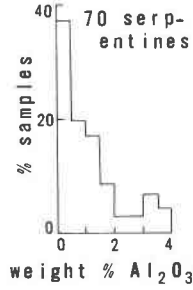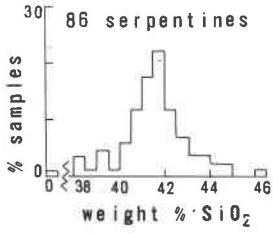
(a)   (b)   (c)
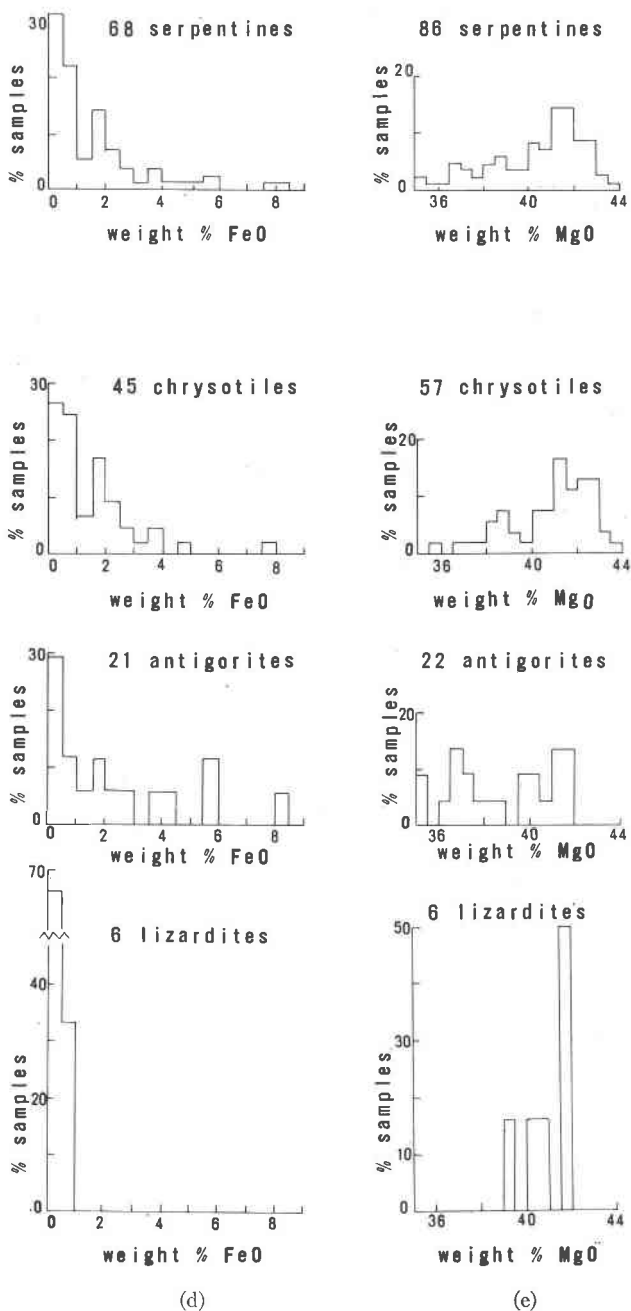
FIG. 1. Histograms for each serpentine mineral group showing the variation in (a) SiO₂, (b) Al₂O₃, (c) Fe₂O₃, (d) FeO, (e) MgO weight percent.

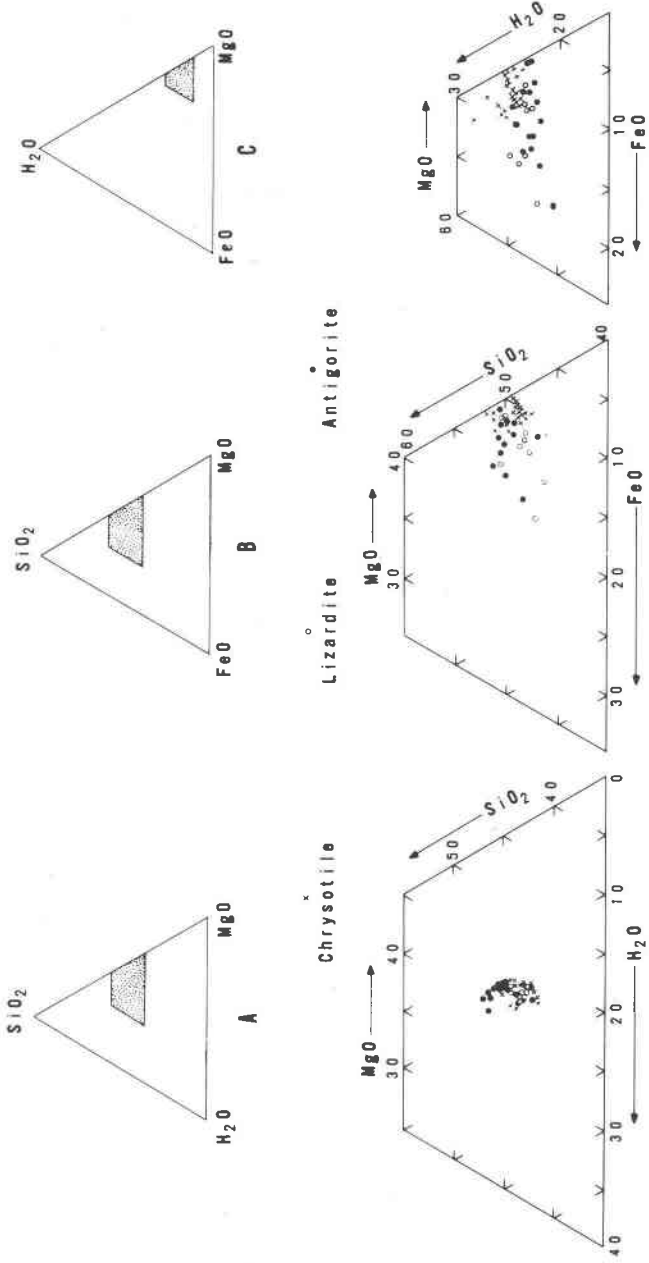FIG. 2. Serpentine mineral analyses projected onto the triangular diagrams (a) MgO-SiO₂-H₂O, (b) MgO-FeO-SiO₂, and (c) MgO-FeO-H₂O. Diagrams from stippled area of index triangles.

2c), lizardite and antigorite analyses plot differently than the chrysotile analyses since they have higher total iron content than chrysotiles. The lizardite and antigorite analyses tend to divide in two groups in which antigorite is distinguished by having a lower water content.

Other methods of plotting mineral analyses make use of calculated mineral formulae. In this study, the hydrogen equivalent method was used to calculate formulae. It is based on the hydrogen-equivalents, that is, on the sum of the anions, O^{2-}, $(OH)^-$, and F^- equals 18. This method for finding the number of ions per formula involves calculating ratios of hydrogen equivalents of oxide weight percents and normalizing these values to twice the sum of anions per unit formula. It is preferred because it uses the $H_2O(+)$ of an analysis. Mineral formulae for the serpentine minerals can also be calculated on the basis of 28 negative charges and assuming that the OH content of the cell is 8 as was done by Faust and Fahey (1962). Originally Page (1966) used this method and came to the same general conclusions regarding chemical compositions and polymorphism, although the number of cations per formulae were different. Since this method neglects the water reported in the analysis, the decision was made not to waste this information in this report.

Figure 3 demonstrates that antigorite and lizardite contain less water than chrysotile and that lizardite and antigorite differ chemically from chrysotile by having lower ratios of $(Fe^{2+}+Mg^{2+})$ to $(Fe^{3+}+Al^{3+})$.

Statistical analysis of serpentine mineral compositions. Given a superior analysis of a serpentine mineral the structural type of the mineral can usually be ascertained. Analyses marked with an asterisk in the Appendix were selected for a statistical treatment. The 52 analyses were assigned to one of three groups, lizardite, antigorite, or chrysotile, by X-ray, DTA, or other information, but not on the basis of the chemical analysis. The weight percent oxides for MgO, FeO, Fe_2O_3, Al_2O_3, SiO_2 and $H_2O(+)$ were treated as variables. These data were analyzed with a computer by a linear discriminant analysis (Program BMD05M, Discriminant Analysis for Several Groups, available from the Stanford Computation Center). The program treats the data (Anderson, 1958) by first comparing the way in which each variable (oxide weight percent) varies within a group and the way in which each variable varies with respect to the entire set of observations (52 analyses of serpentine minerals). The differences in the way in which the group and set vary are used to determine linear equations for the groups. Once these equations are determined they are evaluated for each analysis or observation. Using these functional values for each analysis, a probability is calculated and is used to indicate the likelihood that each observation belongs to any given
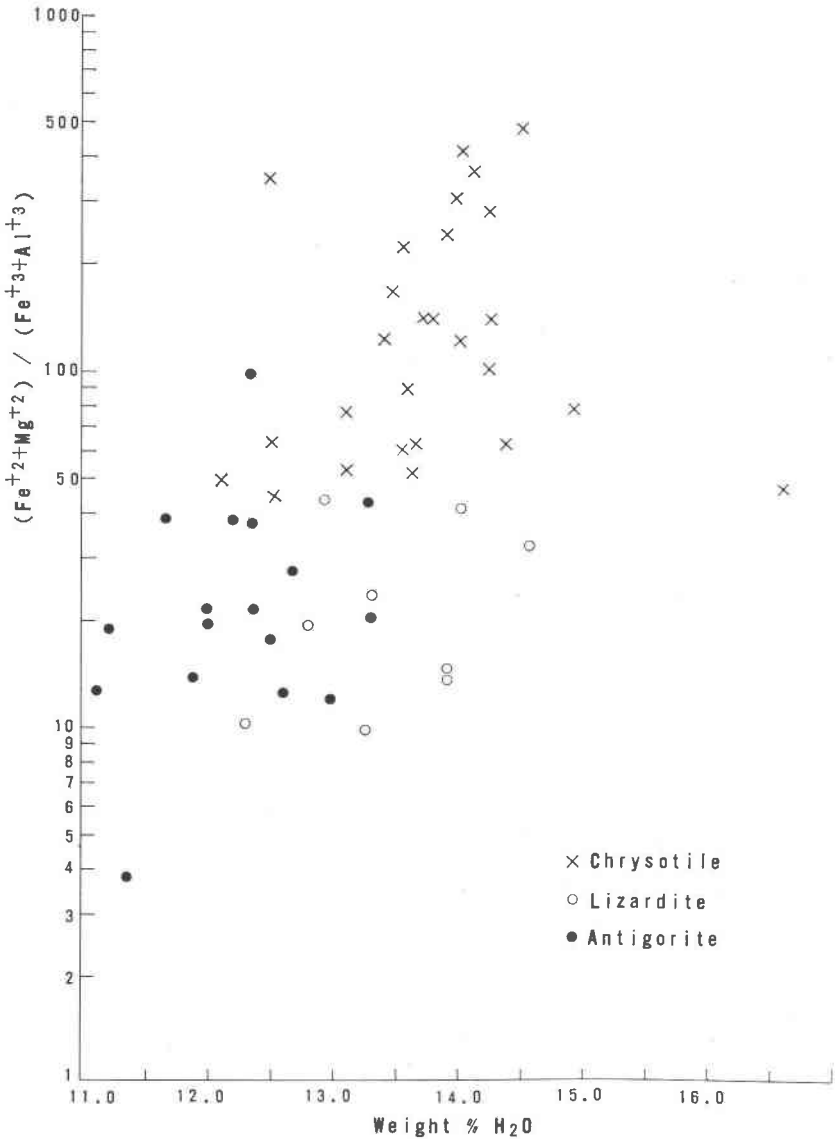
FIG. 3. $(Fe^{2+}+Mg^{2+})/(Fe^{3+}+Al^{3+})$ versus weight percent H_2O for serpentines.

group. Also statistics are calculated which allow one to test whether arithmetic means of the three groups are the same.

The evaluation of the statistical analysis for each individual is that out of 31 chrysotiles (originally grouped together) only two were included

TABLE 1. AVERAGE SERPENTINE COMPOSITIONS AND CALCULATED MINERAL FORMULAE
BASED ON THE HYDROGEN EQUIVALENT METHOD. ANALYSES USED ARE INDICATED
BY AN ASTERISK IN THE APPENDIX

Oxide	Average weight percent		
	Chrysotile	Lizardite	Antigorite
SiO_2	41.530	41.024	42.136
Al_2O_3	0.716	1.395	1.639
Fe_2O_3	0.718	4.100	1.165
FeO	0.624	0.419	3.729
MgO	40.928	39.437	38.369
H_2O+	13.542	13.286	12.098
$\sum$	98.058[a]	99.661	99.136
Formulae			
Tetrahedral ions			
Si^{4+}	3.897	3.834	4.010
Al^{3+}	0.079	0.154	—
$\sum$	3.976	3.988	4.010
Octahedral ions			
Al^{3+}	—	—	0.184
Fe^{3+}	0.051	0.288	0.083
Fe^{2+}	0.049	0.033	0.297
Mg^{2+}	5.724	5.494	5.443
$\sum$	5.824	5.815	6.007
Anions			
O^{2-}	9.524	9.717	10.320
$(OH)^-$	8.476	8.283	7.680
$\sum$ charges on anions	27.524	27.714	28.320
$\sum$ charges on cations	27.524	27.716	28.321

[a] Average H_2O (−) for chrysotile is 1.25 weight percent.

in other groups than chrysotile, for six lizardites only one was put in another group and for 15 antigorites only one was put in another group. If the set of analyses used is representative, then the structural type of serpentine mineral can be found from the mineral analysis. In Table 1 the means of the analyses for each of the three groups is presented. The statistical analysis indicate that the means are not the same at greater than 99.95 percent confidence level.

Besides this type of treatment, all of the analyses were presented randomly to the same statistical program and three groups defined by a random process. These three groups were nearly identical to the groups chosen on a non-chemical basis. Therefore, very little bias was probably introduced by the method of selection of the groups.

RESULTS AND CONCLUSIONS

The chemical differences between the different serpentine minerals as represented by the present available analyses are shown in Table 1 by average analyses and average formulae of chrysotile, lizardite, and antigorite. The features observed here which differentiate the mineral species are the same discussed in the previous section. On the average they are: (1) high SiO_2 weight percent for lizardite and antigorite, (2) low Al_2O_3 contents for lizardite and chrysotile, (3) a large ratio of Fe_2O_3 to FeO for lizardite, (4) low MgO weight percent for antigorite, (5) low H_2O weight percent for antigorite, (6) large numbers of trivalent ions in the tetrahedral coordination for antigorites, and (7) low Fe^{2+} in lizardites.

Structural studies of the serpentine minerals, chrysotile, lizardite, and antigorite indicate that the antigorite structure involves deficiencies of hydroxyl and magnesium ions whereas the other two mineral structures do not involve these. If mineral formulae are calculated on the assumption of 28 negative charges (Faust and Fahey, 1962), the average sum of octahedral cations for the selected chrysotiles is 5.97; lizardite, 5.92; and antigorite, 5.65. This is the reverse of Table 1, but the $(OH)^-$ for antigorite in Table 1 is quite low, 7.680 as compared with the other two species. The actual distribution of cations and anions is probably somewhere in between these two results. Since the structure changes the composition of antigorite from the theoretical end member $Mg_3Si_2O_5(OH)_4$, antigorite, as is now known, is not a polymorph of chrysotile or lizardite.

Recent microprobe studies by Page (1967) suggest that lizardite and chrysotile are separate mineral species and that lizardite is consistently more iron-rich than the associated chrysotile. Present analytical information on serpentines and statistical treatments of the same data suggest that lizardite and chrysotile are not polymorphs. These arguments apply only to the presently available analytical information and as yet no one has managed to obtain an unquestioned serpentine polymorphic transition experimentally (Page, 1966).

This author believes that all serpentine minerals are composed of three sets of solid solutions, one representing lizardite, another chrysotile, and another antigorite, with the possibility of polymorphism or polytypism within each solid solution series.

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APPENDIX: DISCUSSION OF SAMPLES USED IN THE STUDY

The sample numbers listed below are from those analyses of Faust and Fahey (1962) used in this study. Pertinent comments are given below and include the date, whether Fe_2O_3 and FeO, $H_2(-)$ and $H_2O(+)$, and Al_2O_3 were determined, impurities present, and probable serpentine mineral. An asterisk indicates that the analysis was used in the statistical treatment of the present paper.

*6; 1936, 0.50 CaO, probably lizardite.

*10; 1936, H_2O lumped, 0.73 CaO, probably lizardite.

*12; 1936, 0.63 CaO, antigorite.

13; 1936, 0.98 CaO, antigorite.

*15; 1957, antigorite.

*20; 1906, H_2O lumped, 0.72 CaO, antigorite.

*28; 1936, probably lizardite.

29; 1848, n.d. Fe_2O_3, H_2O lumped, antigorite.

*39; 1857, n.d. Fe_2O_3, H_2O lumped, antigorite.

33; 1954, six-layer orthoserpentine.

*35; 1930, n.d. Fe_2O_3, chrysotile.

*37; 1936, chrysotile.

42; 1936, 0.40 CaO, chrysotile.

43; 1936, probably antigorite.

*45; 1957, chrysotile.

*49; 1954, antigorite.

50; 1931, 0.75 total impurity, probably antigorite.

*51; 1947, n.d. FeO, 0.14 Na_2O, probably lizardite.

53; 1885, n.d. Fe_2O_3, n.d. Al_2O_3 probably chrysotile.

54; 1931, n.d. Fe_2O_3, n.d. Al_2O_3, 0.40 total impurity, probably chrysotile.

63; 1931, 0.43 Na_2O and 0.19 K_2O, probably antigorite.

*64; 1957, chrysotile.

*65; 1957, chrysotile.

*67; 1936, 1.41 CaO and 4.54 CO_2, chrysotile.

70; 1906, n.d. FeO, H_2O lumped, probably chrysotile.

71; 1931, n.d. FeO, n.d. Al_2O_3, probably chrysotile.

75; 1930, H_2O lumped, 0.55 total impurity, chrysotile.

76; 1906, n.d. FeO, chrysotile.

*78; 1856, n.d. Fe_2O_3, H_2O lumped, probably antigorite.

79; 1956, chrysotile.

*86; 1930, 0.35 CaO, chrysotile.

89; 1910, lumped FeO-Fe_2O_3, and H_2O, chrysotile.

*91; 1957, chrysotile.

*94, 1910, lumped FeO-Fe_2O_3 and H_2O, chrysotile.

*96; 1936, chinochrysotile.

97; 1930, lumped FeO-Fe_2O_3 and H_2O, average chrysotile.

100; 1954, lumped H_2O, 1.31 CaO, chrysotile.

101; 1891, n.d. Fe_2O_3 lumped H_2O, probably chrysotile.

*102; 1936, n.d. Al_2O_3, labelled antigorite, probably chrysotile.

111; 1930, n.d. Fe_2O_3, 2.90 CaO, chrysotile.

112; 1954, n.d. FeO, six-layer orthoserpentine.

115; 1910, lumped Fe_2O_3-FeO, n.d. H_2O^-, n.d. Al_2O_3, chrysotile.

*120; 1952, antigorite.

*121; 1931, n.d. Fe_2O_3, n.d. H_2O_3, chrysotile.

*129; 1930, n.d. Fe_2O_3, 0.16 CaO, chrysotile.

*132; 1876, n.d. FeO, H_2O lumped, antigorite.

*136; 1818, n.d. Fe_2O_3, n.d. Al_2O_3, antigorite.

*137; 1857, n.d. Fe_2O_3, n.d. Al_2O_3, chrysotile.

*146; 1951, n.d. FeO, H_2O lumped, lizardite.

*147; 1926, n.d. FeO, 1.25 impurities, chrysotile.

152; 1834, n.d. Fe_2O_3, H_2O lumped, chrysotile.

*194; 1936, n.d. Fe_2O_3, n.d. Al_2O_3, 0.58 CaO, chrysotile.

*197; 1931, n.d. Fe_2O_3, n.d. Al_2O_3, chrysotile.

203; 1885, n.d. Fe_2O_3, H_2O lumped, n.d. Al_2O_3, chrysotile.

204; 1897, n.d. Fe_2O_3, H_2O lumped, chrysotile.

205; 1891, H_2O lumped, n.d. Al_2O_3, chrysotile.

206; 1883, Fe_2O_3-FeO and H_2O lumped, chrysotile.

212; 1891, n.d. FeO, H_2O lumped, chrysotile.

217; 1955, n.d. H_2O^-, chrysotile.

*222; 1957, chrysotile.

243; 1947, n.d. Al_2O_3, chrysotile.

248; 1918, n.d. Fe_2O_3, chrysotile.

*250; 1918, n.d. Fe_2O_3, chrysotile.

251; 1918, n.d. Fe_2O_3, chrysotile.

*260; 1890, n.d. FeO, chrysotile.

*F-1; 1956; antigorite.

*F-15; 1962, antigorite.

*F-20; 1956, clinochrysotile.

*F-22; 1962, clinochrysotile.

*F-23; 1962, lizardite.

*F-24; 1962, clinochrysotile.

*F-41; 1962, clinochrysotile.

*F-43; 1962, lizardite.

*F-46; 1937, lizardite.

*F-47; 1937, lizardite.

From Page (1967) the following samples were used: 19-NI-63A, 38-NZ-62-5, 94-NZ-62, 19-NI-63B, and 14-NI-63A.