THE OPTICAL AND X-RAY PROPERTIES OF THE FERROMAGNESIAN OLIVINE MINERALS WITH CHARTS TO AID IDENTIFICATION

Thesis by

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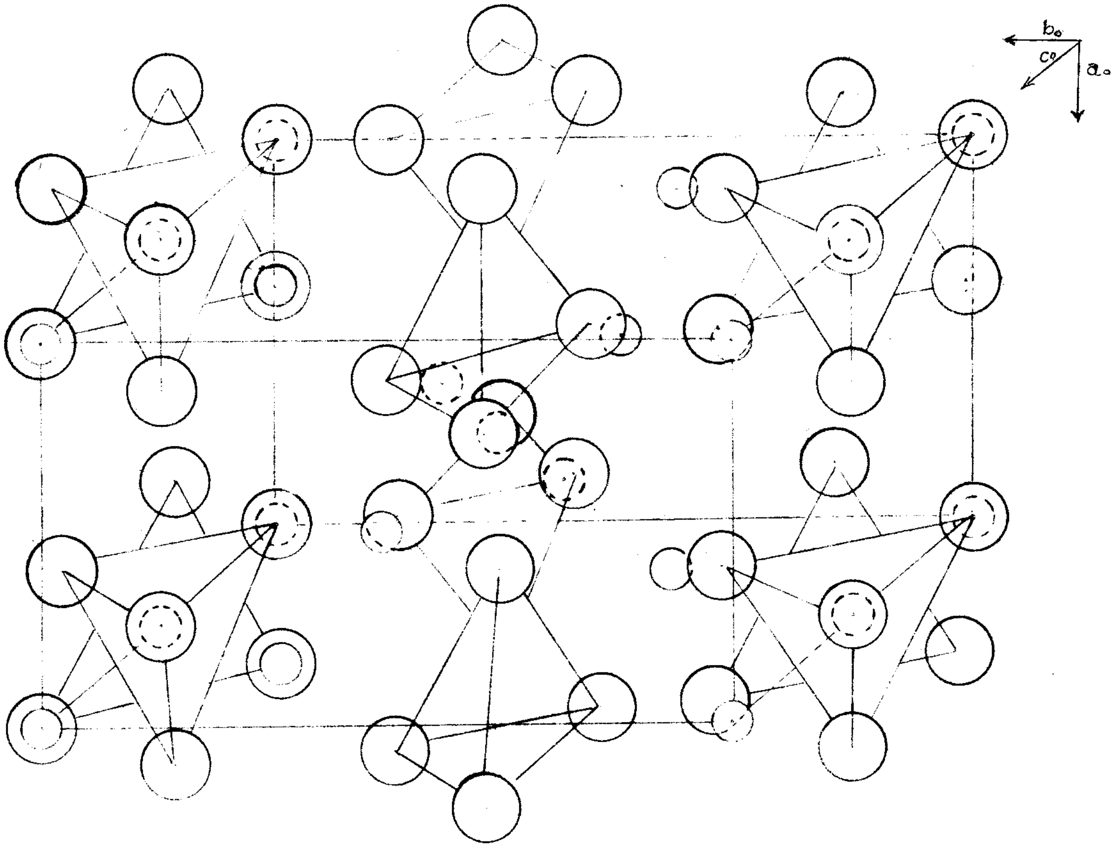

A stereographic illustration of a ferromagnesian olivine (forsterite). Large circles are oxygen atoms. Small circles are magnesium atoms. Silicon atoms are not shown. Basic tetrahedra are indicated by lines between oxygen atoms.

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ABSTRACT

A systematic search of the literature has been attempted to secure all available optical data for the natural ferromagnesian olivine minerals. These optical data have been plotted in graphical form against published chemical compositions, producing a chart relating these properties. The object has been to facilitate identification of these ferromagnesian olivines. The resulting curves of refractive indices, birefringence and 2V have been analyzed for what are considered to be causes of the small deviations from the curves of the same properties of the synthetic ferromagnesian olivines.

A short historical discussion is presented of the work done in determining unit cell dimensions, including the recent studies by Dr. Horace Winchell at Yale University.

Six minerals of the ferromagnesian olivine group have been photographed with X-rays employing the powder technique. The d/n values are given in tabular and graphical form which are usable for the identification of the ferromagnesian olivines.

INTRODUCTION

Purpose

In the last 20 years, more or less, appreciable work has been accomplished in connection with the identification of the olivine group minerals. Much information has been published pertaining to this mineral group.

During the late 1920s, A. N. Winchell[1] published a chart relating the various optical properties used for identification of the ferromagnesian olivine minerals with chemical composition. At that time, certain members of this group had not been found to occur naturally, and as a result a large gap existed in these published data of the refractive indices, specific gravity, birefringence and 2V curves.

Because of the gap these curves were based upon broad extrapolation between a few points. However, recent investigations in the fields of mineralogy, petrology and crystal chemistry have either found the missing minerals or they have been produced synthetically. The number of each reported by a single investigator has always been small, and as a result, pertinent optical data on the entire ferromagnesian olivine group are widely scattered through the literature. It was thought by this writer that if all these new data were collected and a new refined graph produced, from a synthesis

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1. Winchell, A. N., Elements of optical mineralogy,
   3rd Ed., Part II, Description of minerals,
   John Wiley and Sons, N. Y., 1929, p.191.

of these data, areas of greatest uncertainty would be defined and future workers would be saved a great deal of literary research.

X-ray powder photography constitutes an invaluable and widely used tool in the identification of minerals. In the search through the literature, however, only one member of the ferromagnesian olivines was found to have the line data recorded. As these data have not been recorded for the other five members, the second part of the investigation was directed toward this end.

Scope of Work

In the present work investigations have been confined to the ferromagnesian members of the olivine group. Such minerals as larsenite, glaucochroite, monticellite, knebelite, tephroite and picrotephroite are not considered.

The recorded data that follow are of the type that will allow for ready identification of the ferromagnesian olivine minerals. Information related to phase relations, i.e. melting points, etc. have not been included.

Nomenclature

The nomenclature of this group has been added to and modified over a long period of time. The term olivine has now evolved into a group name which includes the ferromagnesian members here discussed, and those minerals that have been excluded from the investigation. Replacing olivine as a compositional term, chrysolite now designates ferromagnesian olivines with 10-30 molecular percent Fe_2SiO_4. Deer and Wager[2] have published a short historical review of the evolution of the classification of the ferromagnesian olivine group and have recommended the following decimal classification which has been followed more recently by Poldervaart[3] and other workers:

	Mol. % Fe_2SiO_4
Forsterite	0 - 10
Chrysolite	10 - 30
Hyalosiderite	30 - 50
Hortonolite	50 - 70
Ferrohortonolite	70 - 90
Fayalite	90 - 100

For convenience in this manuscript, the name olivine is used as a collective term to refer to the six ferromagnesian olivine minerals investigated.

Materials

The materials used in the X-ray studies were secured from two sources. Five of the samples are from the United States National Museum. They are forsterite from Bolton, Mass. (U. S. N. M. 79736); chrysolite from Dreicher Weicher, Eifel, Germany (U. S. N. M. 80728); hyalosiderite from Lumburg, Kaiserstuhl, Baden, Germany (U. S. N. M. R. 3505);

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2. Deer, W. A. and Wager, L. R., Olivines from the Skaergaard intrusion, Kangerdlugssuak, East Greenland, Am. Min., <u>24</u>, 1939, pp.23-25.

3. Walker, F. and Poldervaart, A., Karroo dolerites of the Union of South Africa, Bull. G. S. A. <u>60</u>, April 1949, pp.632-633.

hortonolite from the O'Neil Mine, Monroe, New York (U. S. N. M. 11913), and fayalite from Rockport, Mass. (U. S. N. M. 85726). The ferrohortonolite was kindly contributed by Dr. Sahama and is a portion of the sample used in his recent thermochemical investigations[4] at the Geophysical Laboratory, Washington, D. C. It is from St. Utterviks Hage, Södermanland, Sweden. The materials contributed by Dr. Switzer from the National Museum are samples from areas that have been described in the literature. Although no chemical analyses were attempted it is felt that the data in the various publications are sufficient and allow the calculation of the molecular percent to a close approximation.

The forsterite is from the boltonite that has been described by Smith[5], Brush[6] and Dana[7]. The chemical analyses given for this mineral are calculated to be between 95-97 molecular percent Mg_2SiO_4.

The chrysolite has been described by Dana[8]. From the analysis he has given the mineral has been calculated to

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[4] Sahama, Th. G. and Torgeson, D. R., Thermochemical study of the olivines and orthopyroxenes, U. S. Bur. of Mines, Rept. Invest. 4408, Feb. 1949, p.7.

[5] Smith, J. L., Reexamination of American minerals, Am. J. Sci., II Ser., <u>18</u>, 1854, p.374.

[6] Brush, G. J., On boltonite, Am. J. Sci., II Ser., 1859, pp.387-388.

[7] Dana, E. S., The system of mineralogy of James D. Dana, 1837-1868, John Wiley and Sons, N. Y., 1900, p.451.

[8] Dana, J. D., A system of mineralogy, John Wiley and Sons, 1890, p.257.

contain about 90 molecular percent Mg_2SiO_4.

The hyalosiderite is mentioned in both Dana[9,10] volumes. The first volume does not give a locality, however, and the chemical analysis recorded is not that of a hyalosiderite. The second does give the locality and a correct analysis which has been calculated to be about 35 molecular percent fayalite.

The hortonolite has been described in detail by Brush[11] and Penfield and Forbes[12]. Calculation of the molecular percent indicates it contains 63-68 percent fayalite.

The ferrohortonolite composition is based on the chemical analysis given to the author by Dr. Sahama[13]. It is 94 percent fayalite.

The fayalite is a sample of the material described from Rockport, Mass. by Penfield and Forbes[14] and from the analysis published by them is 100 molecular percent fayalite.

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9. Dana, J. D., op. cit., 1890, p.257.

10. Dana, E. S., op. cit., 1900, p.453.

11. Brush, G. J., On hortonolite, a new mineral of the chrysolite group. Am. J. Sci., Ser. 2, <u>48</u>, 1869, p.18.

12. Penfield, S. L. and Forbes, E. H., Fayalite from Rockport, Mass. and the optical properties of the chrysolite-fayalite group and of monticellite, Am. J. Sci., Ser. 4, <u>1</u>, 1896, pp.129-135.

13. Personal communication.

14. Penfield, S. L. and Forbes, E. H., op. cit., pp.129-135.

Acknowledgement

This investigation has been accomplished under the supervision of Dr. A. E. J. Engel, to whom the author is indebted, not only for his interest, but for his suggestions in regard to the problem in general, the X-ray work, and the writing of the manuscript. The author is also indebted to Dr. Thorgerson G. Sahama of the Institute of Geology, Helsinki, Finland, for sending a sample of the mineral ferrohortonolite used by him in his recent work at the Geophysical Laboratory, Washington, D. C., and Dr. George Switzer of the United States National Museum, who made available the remaining materials used. Thanks are also due to Dr. J. H. Sturdivant of the Institute's Chemistry Department for suggestions to improve the X-ray data and supplying the nickel foil for construction of a filter for the X-ray camera. To Dr. Ian Campbell, gratitude is extended for frequently bringing to the author's attention, literature pertaining to the problem. Appreciation is also expressed for the aid given by Mr. William Oke and Mr. Rudolph von Huene. Mr. Oke did the actual X-ray photography and unselfishly relinquished an evening to construct the nickel filter for the X-ray camera. Mr. von Huene made available his adding machine in the early portion of the X-ray work for calculations and arranged for the author to secure materials from the Institute warehouse.

Dr. Horace Winchell of Yale University is sincerely thanked for graciously forwarding the preliminary, unpublished results of his investigations on the cell dimensions of the olivine minerals.

For the typing and correcting of the manuscript, I thank my wife.

OPTICAL PROPERTIES STUDY

Technique

The optical data used to prepare the chart are a compilation of the data published on the olivines over the past 20 years. The method of finding the source material was to search through the standard bibliographies for any special subject that was in any way related to olivine minerals. The standard bibliographic sources used were: United States Geological Survey Bulletins, 937, 938, 949, 952, 958, (Bibliography of North American Geology, 1939-1947), Bibliography of Geology Exclusive of North America of the Geologic Society of America, Chemical Abstracts of the American Chemical Society, Gmelins Handbuch, and the International Critical Tables of the National Research Council. For the periods 1948, 1949, 1950, a direct search of the periodicals was performed. The search through the literature consumed some 35-40 days within a period of 4 to 5 months and is thought to be rather complete.

After all the data had been collected, those studies which contained chemical analyses were separated, and the molecular percentages of the described minerals were calculated on the basis of pure olivine. These particular data were used as the skeleton or base of the chart. Such data as refractive indices, specific gravity, 2V, and birefringence were plotted against these known molecular percentages and curves drawn.

Results

The results of the compilation of the optical data are shown in figure 1. Some comments about the curves are in order.

It has been suggested by Backlund[15], that if the increase in refractive indices of the pure ferromagnesian olivines are plotted against chemical composition in molecular percentages the result would be straight lines with positive slope in the direction of increasing iron content. It was not until 1935 that this relationship was proven by Bowen and Schairer[16] using synthetic olivines. The possibility of finding any of the members of this series as mixtures of pure ferrous oxide and magnesia occurring naturally, however, is obviously almost impossible. As a result the curves of the optical constants that have been plotted are based on naturally occurring species. In order that the refractive indices of the natural occurring olivines may be compared with the

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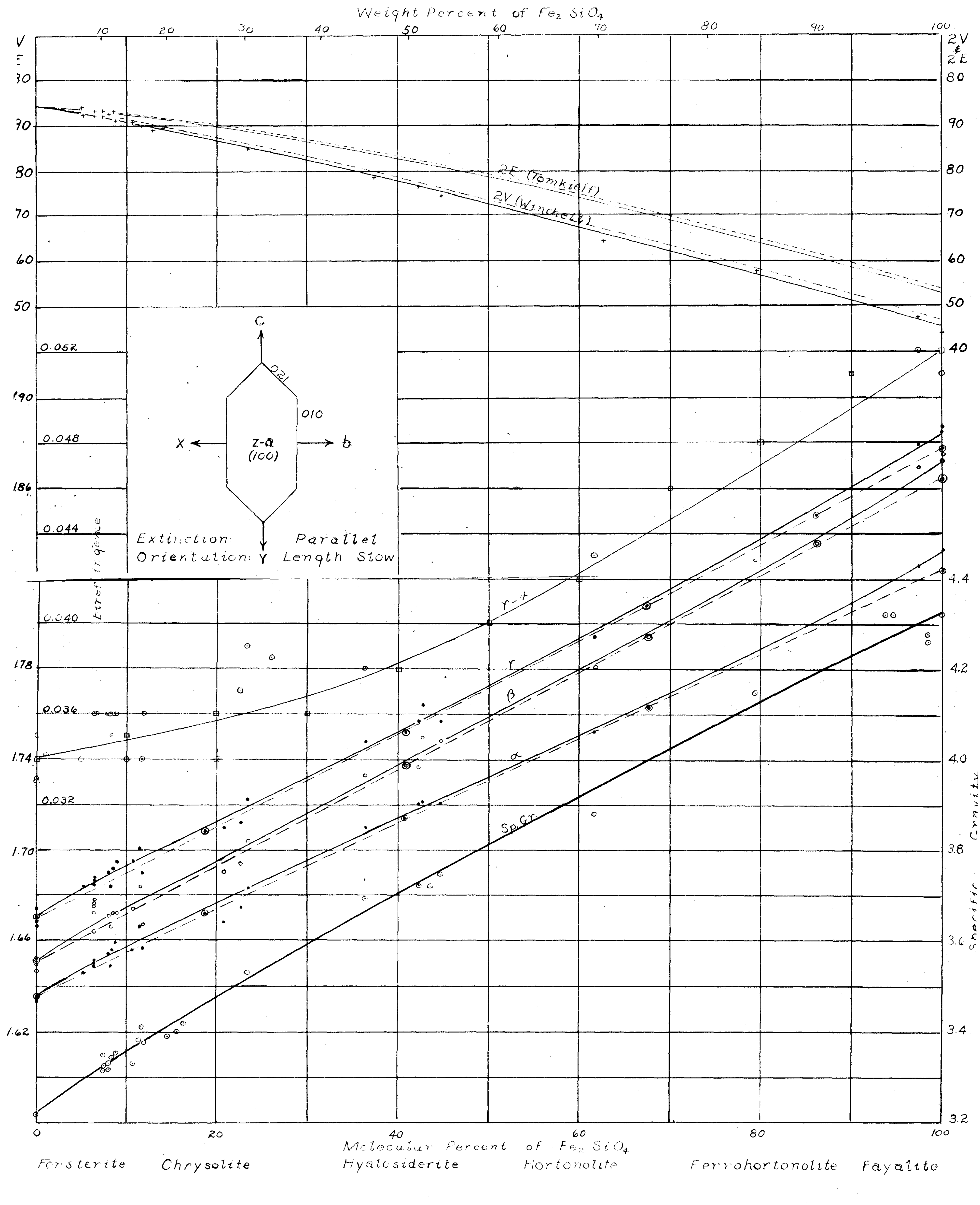
[15] Backlund, Helge, Travaux du Musee Geologique, Pierre le Grand, St. Petersbourg, Tome III, 1909, pp.77-105.

[16] Bowen, N. L. and Schairer, J. F. The system MgO - FeO - SiO_2, Am. J. Sci., Ser. 4, 29, Feb. 1935, pp.151-217.

Fig. 1 Chart illustrating variations in optical properties with change in chemical composition of the ferromagnesian olivine minerals



equivalent indices in the synthetic olivines prepared by Bowen and Schairer[17], the curves of the latter are lightly dashed on the graph. It will be noticed that the curves defined by the plots of the refractive indices of the natural occurring members are not straight lines, and that the deviations of these curves are a little above the curves for the similar property of the synthetic minerals. This condition was also found to exist by Deer and Wager.[18] The cause of these deviations was investigated. A notable feature of the deviations between the refractive index curves of the natural and synthetic olivines is the relative position of the deviations on the curve. The greatest variance occurs where compositions are 10 to 30 and 70 to 100 molecular percent fayalite. The variation in the range of 10 to 30 molecular percent fayalite is not thought to be due to lack of sampling. This sampling factor could well be of some significance in the 70 to 100 molecular percent fayalite range. It is believed by the writer that if more data on the refractive indices of natural occurring olivines were available for the compositional range of 50 to 100 molecular percent fayalite, the deviations would be modified. In the compositional range of 50 to 70 molecular percent fayalite, it is thought the indices of natural occurring olivines would

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17. Bowen, N. L. and Schairer, J. L., op. cit.

18. Deer, W. A., and Wager, L. R., op. cit. p.20.

be higher by a very small amount (possibly 0.002 to 0.004 units). This would tend to make the curves of the plots more nearly straight lines which is probably what they should be. Although naturally occurring ferromagnesian olivines have been found in the compositional range where, heretofore, they have been missing, still more examples of hortonolite, ferrohortonolite, and fayalite are needed to locate the indices curves more exactly through the compositional range represented by these minerals.

All the chemical analyses were studied to determine the kind and relative percentage of contaminating oxides, in order to determine whether there was any correlation between the amounts of any of these oxides and particular changes in refractive indices. No correlation has been found. High ferric oxide content is found both in olivines whose indices are above those of the synthetic olivines, and in those whose indices are below. This was also found true for calcium oxide and manganous oxide. Potassium and sodium oxide are frequently reported. Their total amount is seldom greater than 0.5 to 1.0 percent of the mineral. The small amounts of potassium and sodium oxide occurring in the olivine minerals are probably due to their geologic environment. As a result of magmatic differentiation the iron, magnesium, and calcium are usually an integral part of the denser differentiate, while the potassium and soda are contained in the less dense differentiate. A small amount of the potassium and sodium oxide could be retained by the denser magma. The possibility of any large amounts of these two oxides being retained by ferromagnesian olivines would be still smaller. Another factor which seems to operate against large amounts of potassium in olivine minerals is the size of the atom. The potassium atom has a radius of 1.33Å[19] units as compared with 0.65Å and 0.75Å units for the magnesium and iron atoms respectively. Replacement of large amounts of magnesium or iron by potassium would cause a great amount of distortion in the crystal lattice. As only a limited amount of this distortion can occur for a given mineral, only a limited amount of space is available for the substitution of larger, or smaller atoms. The conditions are different for the sodium atom. Here, the environment must be the controlling factor for the sodium atom is only a little smaller than the calcium atom (0.95Å and 0.99Å units). As the calcium atom substitutes for iron and magnesium rather freely to form other types of olivines (monticellite and merwinite), then it could be expected that the sodium atom would do the same if present when crystallization occurred. The percentage of sodium present in the magma at the time of formation of the olivines must be very small. What effect each of these foreign atoms (Ca, Mn, K, Na, etc.) have upon the refractive indices is not predictable when a great many

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19. All crystal radii used are from Pauling, L., The nature of the chemical bond. Cornell Univ. Press, Ithaca, N. Y., 1948, p.350.

are present in a given olivine. The calcium atom may raise the indices 0.002 units, and the manganese may reduce it 0.004 units, or the opposite may be true, or they both may be additive in their effects.

With a greater number of impurities the variations in refractive indices due to each different atom become more complex to define. A similar conclusion was made by Bowen[20] and Schairer in their study of ferromagnesian olivine melts.

Game[21], in his studies of the olivines from Ubekendt Island, has shown that the mean value of the β index of olivines from flow rocks is of slightly higher value than those from dike rocks. Accordingly, a factor of possible importance, which may influence the deviations in the refractive indices is the type of rock body in which the olivine minerals were associated. Are the indices of olivines in dike rocks different from indices of olivines in stocks, flows, or meteorites? Are the indices of olivines in stocks different from indices of olivines in flows, etc.? A study of the author's compiled data shows the relationship found by Game can not be used as a hard and fast rule. A great number of olivines in dike rocks have β indices that are of higher values than those from flow rocks. For a small number of observations there is no means of predicting by indices values

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[20] Bowen, N. L. and Schairer, J. L., op. cit., p.196.

[21] Game, R. M., Optical properties of olivines from Ubekendt Island, Min. Mag., 26, Mar. 1941, p.12-15.

whether the olivines have been associated with dike or flow rocks. Also, the author's compiled data suggest that olivines from either dike, flow or massive rock bodies have great variations in refractive indices, and as a result can not be used to explain the deviation. Olivines from meteorites also have a large range in indices and do not aid the explanation of the deviation. Boque and Hodge[22] have noted variations in indices due to grain size. This same condition has been observed by Barth.[23] The minerals become more fayalitic as the grain size increases. Barth also observed that in the olivine rich rocks the variety of olivine was more magnesian than in the olivine poor rocks, and that olivine phenocrysts were more magnesian than the associated olivine of the ground mass. Tomkieff[24] reached the same conclusion in his study of zoned olivines. These points have been developed because frequently the author of an investigation on olivine minerals fails to describe fully the occurrence, that is, the rock type, whether the mineral is from the matrix, or is a phenocryst, or whether there was any zoning present. This information is necessary if the

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22. Boque, R. and Hodge, E. T., Cascade andesites of Oregon, Am. Min., 25, Oct. 1940, pp.631-634.

23. Barth, T. F. W., Pacificite, and anemousite basalt, Jour. Wash. Acad. Sci., 20, 1930, pp.60-68.

    Mineralogical petrography of the Pacific lavas, Am. J. Sci., 21, June 1931, Part I, Minerals, pp.337-405, Part II, Rocks, pp.490-530.

24. Tomkieff, S. I., Zones olivines and their petrogenetic significance, Min. Mag., 35, Mar. 1939, pp.229-251.

identification is to serve any petrographic significance. Only a few sources of data noted these details. These few data show no correlation between grain size and refractive indices; some have indices above, and some below, the synthetic olivines.

In addition to the above causes for deviation of the indices of the natural occurring olivines from those of the synthetic olivines there must also be included the human element. In all of the papers examined by the author no mention is ever made of the accuracy of the chemical analyses, i.e., the number of analyses and the deviations of each from the average, assuming that it is the average that is published. This small error added to those discussed above could easily cause the small deviations that occur.

Another factor, introduced by the author, is the calculation of the analyses to pure ferromagnesian olivines. Earlier investigations appear to have based their calculation of molecular percentages on the C. I. P. W.[25] system. The object of calculating the composition to pure ferromagnesian olivines was to reduce all olivines to a common base so that variations caused in the optical properties by the impurities could be examined. As pointed out above, this did not give a satisfactory result, other than agreement with the observations that the impurities, as a whole, increase the refractive

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25. Cross, Whitman; Iddings, J. P.; Pirson, L. V.; and Washington, H. S., Quantitative classification of igneous rocks, Univ. of Chicago Press, 1903.

indices and nothing definite can be said about any individual impurity. The curves, nevertheless, are representative of the refractive indices of the natural occurring olivine minerals.

In reference to the 2V curves, two are shown. The solid line is the curve determined by the author when the value of 2V determined on a Federov stage is plotted against chemical composition. The other curve is that of Winchell.[26] In the region of the low iron members the 2V curve as plotted from the data compiled by the author is in close agreement with the Winchell 2V curve. With increasing iron content there is a small deviation giving a lower value of 2V. This deviation is roughly one degree. As most data that are published are not given with more accuracy than one to two degrees this deviation is not significant. Comparing the author's new curve with a curve published by Boque and Hodge[27] excellent agreement is found. This latter curve has also been plotted using data derived from natural occurring olivines. Thus it is felt the two curves that are shown will give compositions of olivines within one to two percent which is as desirable as could be expected for this type of identification. More accuracy must be secured through chemical

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26. Winchell, A. N., op. cit., p.191.

27. Boque, R. and Hodge, E. T., op. cit., p.633.

analyses. On a chart published by Kennedy,[28] Wahrhaftig calculated a 2V curve based on the refractive indices of Bowen and Schairer's[29] synthetic olivines. This curve has no agreement with that of Winchell, Boque and Hodge, or the curve presented by the author. No equation for calculating the curve has been given and because it is based on synthetic olivines, no real evaluation of the curve can be made.

The dashed 2E curve that is illustrated was calculated by Tomkieff[30] from Winchell's 2V curve. Due to the deviation of the author's 2V and refractive indices curves a second 2E curve has been calculated and plotted using the same equation. It is:

$$\text{Sin E} = \frac{\beta \ \text{Sin V}}{n}$$

β = β refractive index

n = refractive index of the Federov stage (in this case 1.649)

The birefringence curve presented the greatest difficulty to plot. The published values of birefringence when plotted against composition produces a number of random points which appear to have no order. Because of this, the curve that is illustrated is a plot of the differences of the and indices. The curve has a distorted appearance which is caused

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[28] Kennedy, G. C., Charts for correlation of optical properties with chemical composition of some common rock forming minerals., Am. Min., <u>32</u>, 1947, p.567.

[29] Bowen, N. L., and Schairer, J. F., op. cit.

[30] Tomkieff, S. I., op. cit., p.234.

by the scale that is used.

In conclusion, the curves that have been illustrated, aside from the possible causes of deviation, are thought to be rather accurate plots of the optical constants of the olivine minerals.

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Olivine: I From the Hawaiian Islands
II Pure forsterite
Am. Min., 13, November 1928, p.559.
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The younger intrusive rocks of the Kudaru Hills, Nigeria,
Quart. J. Geol. Soc., London, 90, 1934, pp.201-239.
optical constants

Bandy, M. C.
Geology and petrology of Easter Island,
Bull. G. S. A., 48, Nov. 1937, pp.1597-1599.
optical constants with associations

Barth, T. F. W.
The crystallization process of basalts,
Am. J. Sci., Ser. V, 31, 1936, pp.321-351.
minor mention of optical constants, role of olivine in crystallization

Mineralogical petrography of the Pacific lavas,
Am. J. Sci., Ser. V, 21, June 1931, Part I, Minerals, pp.371-381, Part II, Rocks, pp.490-530.
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Cascade andesites of Oregon,
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Crystallization equilibrium in nepheline-albite-silica mixtures with fayalite,
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Zur Petrographie der basischen Eruptivegestein der Campos de Calatrana (Provinz Ciudad Real, Spanien),
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Geology and structure of the Percy Quadrangle, New Hampshire,
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Am. Min., 24, Jan. 1939, pp.18-25.
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Peridotites, serpentines and soapstones of northern Sweden,
Geol. Foren. Forh., Stockholm, 57, 1935, pp.133-260.
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J. Amer. Cer. Soc., Dec. 1943, pp.405-413.
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Gevers, T. W., Mendelssohn, E., Dunne, J. C.
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Trans. Geol. Soc. Sou. Africa, 48, 1945, pp.83-102.
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Handbuch der anorganischen chemie, Achte auflage, Teil A, DerVerbindungen des Eisen, 1929, pp.156-157, Teil B, Der Verbindungen des Magnesiums, 1939, pp.353-358.
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X-RAY PROPERTIES STUDY

Technique

The X-ray diffraction patterns of the olivine minerals were photographed by the usual Debye-Scherrer powder method, using a North American Philips X-ray diffraction apparatus, model 5001. The target material was copper and the radiation used was the CuK$_{\alpha_1}$ K$_{\alpha_2}$ having an average wave length of 1.539Å units. A nickel filter 0.75×10^{-3} inches thick was employed with this target. Exposure time for the iron poor samples was two hours and forty-five minutes. For olivines with high iron content an additional filter was employed in the camera to eliminate the random radiations. This required exposure times to be doubled.

Some special cares had to be observed in the preparation of the samples. The forsterite had to be hand-picked from the rock matrix with the aid of a hand lense. After being powdered a magnet was passed through the sample to remove any magnetite, if present. The material was then examined by means of a petrographic microscope for calcite, a common associate in boltonite. None was seen. There were no calcite lines on the X-ray photograph.

The chrysolite and hyalosiderite presented no impurity difficulties.

The hortonolite was highly desseminated with magnetite. Recovery amounted to only 10 to 20 percent of the original material.

The ferrohortonolite contained an opaque impurity that Dr. Sahama[31] thought was magnetite. His later chemical analysis disproved this as no Fe_2O_3 was recorded. The first sample of this specimen was hand-picked to eliminate the impurity. The resulting photograph was compared with a photograph in which the impurity had not been separated. The impurity did not produce any additional diffraction lines, therefore, separation of the impurity was not accomplished in succeeding samples. The identity of the impurity has not been established.

The fayalite had only a magnetite impurity.

The resulting photographs of the forsterite, chrysolite, and hyalosiderite produced distinct diffraction lines on the film. They had very little "fog", or darkening of the film due to random radiations developing during the X-ray passage through the material. These random radiations are particularly characteristic of materials with high iron content when copper targets are used. The hortonolite, ferrohortonolite and fayalite photographs developed a great deal of "fog" which made the measuring of the lines much more difficult. There is a remarkable increase in the amount of film darkening when the iron content becomes greater than 50 percent. It thus became necessary to try and eliminate this condition. Dr. Sturdivant in the Chemistry Department

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[31] Personal communication.

was consulted and he proposed a nickel foil 0.001 inch in thickness be placed between the sample and the film inside the camera. The nickel filter at the copper target was to be removed.

A piece of 0.008 inch phosphor bronze was to be used for the base and frame of the nickel foil. A strip 0.25 of an inch wide was cut from the phosphor bronze. It was formed into a loop so that the outside diameter of this loop exactly fit the inside diameter of the ridge that protruded from the floor of the camera. To this loop was soldered a piece of nickel foil which was wide enough to fit flush against the camera cover when the cover was in place. Two holes in the filter allowed the collimators to protrude into the camera. The work was done so accurately that the filter had no free play within the camera when the cover was in place.

When the filter was mounted in the camera the exposure times were increased. The increase in the exposure of the minerals to the X-rays could not be allowed for too long a period or the elimination of the film darkening would not be accomplished. The increase in exposure time, however, had to be of sufficient length to produce maximum line intensities. The first exposures were made at twice the previous exposure times, a speed recommended by Dr. Sturdivant. Upon examination of the photographs, the background darkening had been reduced. This first film was compared with those in the chemistry department and the filtering effect was about the same.

The photographs that were produced using this filter have fewer diffraction lines. The very weak lines were eliminated. Compensating for the loss in lines, however, is the increased sharpness of the darker lines. This made for a little more accuracy in the line readings.

The measuring of the lines was done with the North American Philips "Norelco" model reader. Each line was measured four times at various intervals, usually a few days apart. The technique was to measure the distance between similar lines on opposite sides of the collimator holes. In this way any variation in the displacement of either of the lines would be included in the measurement. Only those lines in the forward field of reflection were measured as these are generally the best for identification.

Results

The structure of the olivine minerals has been studied by several investigators[31,32,33,34] and dimensions of the unit cell are

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31. Rinne, F., Bemerkungen und rontgenographische Erfahrungen uber die Umgestaltung und die Zerfall von Kristallstrukturen, Z. Krist., 59, 1934, p.236.

32. Rinne, F., et. al., Die Raumbruppe des Olivins, Z. Krist., 59, 1924, p.548.

33. Bragg, W. L., and Brown, G. B., Structur des Olivin, Z. Krist., 63, 1926, p.63.

34. Bragg, W. L., The atomic structure of minerals, Cornell Univ. Press, Ithaca, N. Y., 1937, p.148.

well known. The minerals are members of the orthorhombic system and are listed under the Schoenflies classification as V_h^{16} . Under the classification of Hermann and Mauguin the symbol would be Pbnm.

The basic element of structure of the olivine minerals is the single silicon-oxygen tetrahedon. In this basic element the silicon atom is surrounded by four oxygen atoms in such a manner that a small tetrahedra is formed. The interatomic distance between the silicon and the oxygen atom is 1.6A units. The basic tetrahedra, when placed so that the oxygen atoms will all be in position of hexagonal close packing, have apices directed alternately in opposite directions along the a and b axes. This produces two layers of oxygen atoms parallel to the (100) surface. In the forsterite member a magnesium atom lies between two tetrahedra so that it is surrounded by 6 oxygen atoms. The valence of the tetrahedron is -1, thus each magnesium requires two tetrahedron to satisfy its valence needs. Figure 2. gives a generalized structural relationship based on the work of Bragg.[35]

The isomorphic substitution of the iron atom for the magnesium atom, due to its larger size (Fe = 0.75Å units and Mg = 0.65Å units), will, of course, cause a change in cell dimensions. The cell dimensions have been studied by many

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35. Bragg, W. L., op. cit., p.149.

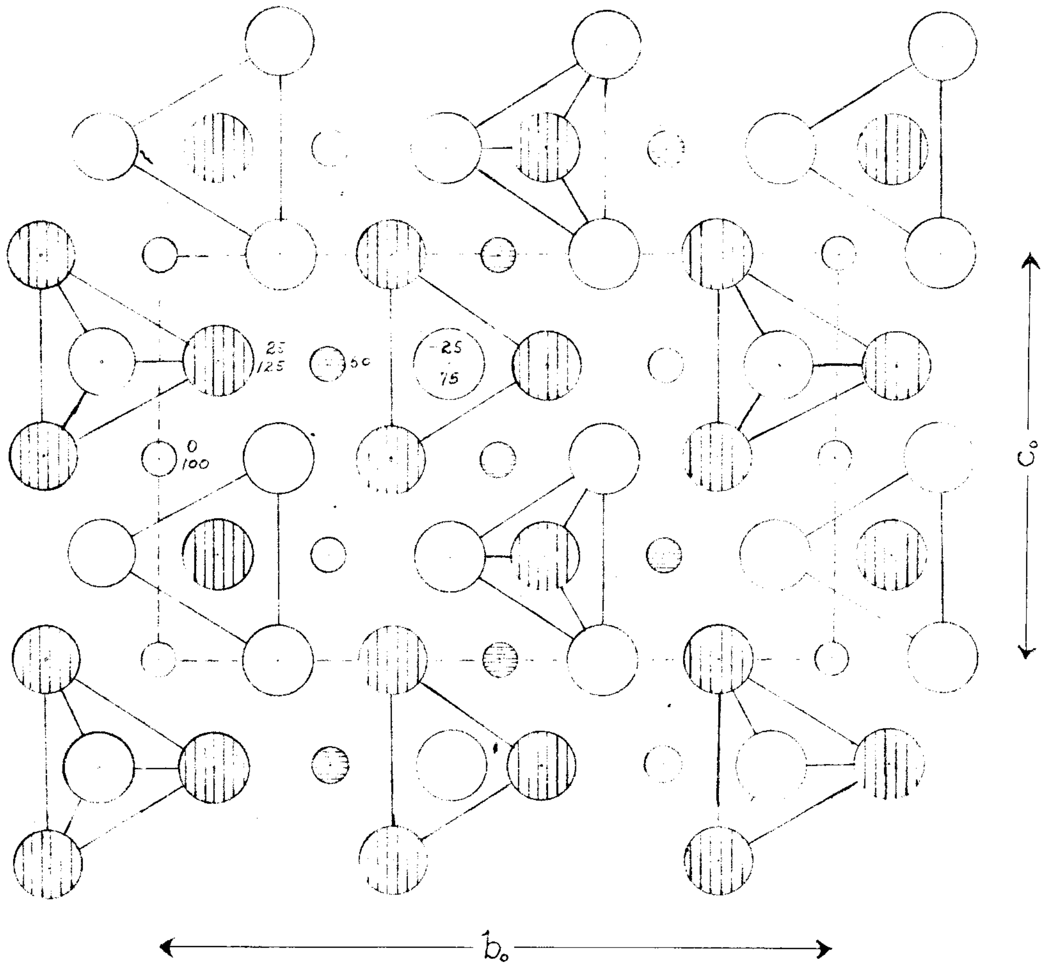

Fig. 2 The idealized structure of forsterite, a ferro-magnesian olivine. Small circles represent magnesium atoms. Large circles represent oxygen atoms of the silicon-oxygen tetrahedron. Silicon atoms are not shown. The a axis is perpendicular to the plane of the paper. Oxygen atoms are in two sheets (plain and shaded). Magnesium atoms lie between six oxygen atoms in two planes indicated by plain and shaded circles.

From W. L. Bragg

investigators. (See footnotes 31 to 34). Recently Winchell at Yale University began a study to measure accurately the cell dimensions of a variety of olivines. The data cited here are the preliminary results of his investigations. Although the results can not be accepted as conclusive a rough approximation is secured of the variation in cell dimensions as a result of the substitution of the iron atom for the magnesium atom.

Other atoms that substitute in the lattice with their crystal radii in Ångstrom units are: Ca 0.99, Mn 0.80, Ni 0.70, K 1.33, Na 0.95, Ti 0.69, Zn 0.74. Except for potassium it is seen that these ions have radii that are not too much different from the radii of the iron and magnesium. The substitution of these ions, especially calcium, zinc, and manganese, for iron and magnesium give other varieties of the olivine group.

It is not difficult to deduce from the values of the crystal radii of the atoms given above that the addition of the ferrous ion for the magnesium ion causes an increase in the size of the unit cell. This distortion of the lattice will, in turn, cause an increase in the interatomic distances. These changes are reflected in the X-ray photographs by the displacement of the lines. Thus, a measure of the distances these lines are displaced will give another means of identifying members of this particular family.

Table II and figure 3. show the d/n values of the several lines in the field of forward reflection. These lines show a continual displacement with only a few minor changes in the general pattern. The relative intensity values of some lines, however, show marked changes. A great many of the lines that are present in the photographs of the first three or four members are not visible in the last two. This may be caused by the nickel filter that was employed. A certain amount of the reduction would be caused by the absorption in this filter. It is felt, however, that a few lines lose intensity due to the characteristics of the minerals. This thought is substantiated by the fact that certain lines that are relatively low in intensity in the photograph of the forsterite become stronger in the photograph of the fayalite regardless of the filter. An example of this is shown by lines 3, 5, 7, 8.

A difficulty encountered in the measuring of the lines was the merging and formation of doublet lines. Doublet lines would be present in the forsterite photograph and observations of this same doublet in the photographs of hyalosiderite through to fayalite would show it merging until in the fayalite photograph the doublet appeared as a single, rather hazy line. Single lines in the forsterite photographs have been observed to split and form doublets in the ferrohortonolite and fayalite photographs.

The d/n values that are listed in the table are the most consistent three of four values calculated. Most of the lines show agreement to 1 part in 1000 which is as accurate as one can expect from the measuring device that was used. The only published d/n values found by the author were those of Clarke[36] and the A. S. T. M. Card Index.[37]

Clarke's data are based on synthetic minerals. His results are slightly different, favoring smaller d/n values than those secured by the author. The data of the author for Mg_2SiO_4 are in many cases lower, and in few cases greater than the d/n values of the A. S. T. M. Card Index.

Lines through plots of the d/n values for a few of the stronger lines are shown in figure 3. It is immediately seen from these curves that the slopes become more positive with increasing iron content. In the low iron members the curves do not vary enough to allow their use for identification. To be able to differentiate the first three members, using d/n values, requires accuracy to four or five decimal places. This is difficult to achieve with the apparatus now in use, unless some modifications are made to eliminate the possible errors. The X-ray technique will allow the

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36. Clarke, C. B., X-ray diffraction data for the compounds in the system CaO - MgO - SiO_2, J. Am. Cer. Soc., 29, 1936, p.28.

37. American Society for Testing Materials
Card index file of X-ray diffraction data.
The American Society for Testing Materials,
Phila, Penna.

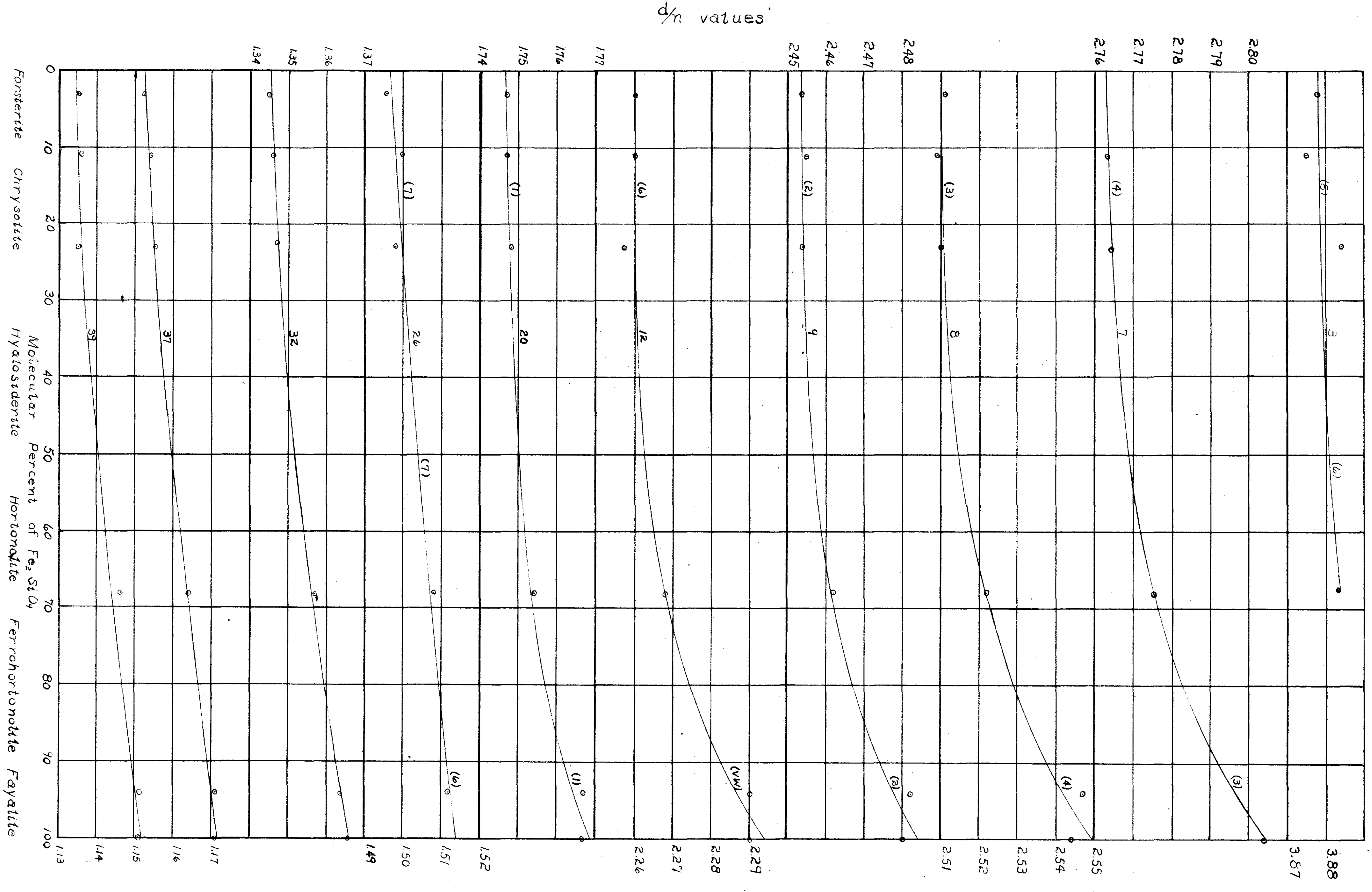

Fig 3 Plot of d/n values for ferromagnesian olivines from powder X-ray photographs. Number indicates line position from collimator hole. Bracketed number is intensity value. (See Table II.)

determination of forsterite from compounds of hortonolite to fayalite composition.

Another interesting feature is the change in slope values of the curves. The lower numbered curves are those resulting from plots of d/n values for diffraction lines close to the collimator punch hole, or those that have low θ values. The numbers increase for d/n values whose lines approach the 90° angle of diffraction. This change in slope is a function of the larger iron atom and the more unsymmetrical planes. Because of these two factors the interplanar spacing becomes smaller more rapidly in the iron rich members relative to the same planes of the magnesium rich members.

Another notable feature of the curves, is the plot of the ferrohortonolite values. Each value of d/n plotted is always equal to or greater than the fayalite value. There are a few instances where this is not true, but in the stronger lines where accuracy is more certain, this relation exists. An examination of the chemical analysis of the ferrohortonolite shows about four and a half percent of manganous oxide. The manganese ion, as noted above, has a radius of 0.80A units. The iron is 0.75Å units. This is only a small difference in atomic radii but it is the only factor allowable for the slight shift of the d/n value as shown. This particular sample has been forwarded to Dr. Winchell for his use in cell dimension work. It will be of interest to see what values he will arrive at for the unit cell dimensions.

The data secured from the olivines examined by the X-ray technique, and the optical constants plotted in the previous section indicate that a more precise determination of iron content in the more magnesian members of the ferromagnesian olivine series can be made using optical methods. This condition may not be true if the North American Philips X-ray reader can be adjusted to eliminate the possible errors.

Besides the standard parallax difficulty there is a very small amount of play in the slider that holds the cross hairs that is used for the reading of the lines. Another disconcerting feature that was noticed a few times concerned the vernier. The first line of the vernier just failed by a hair to aline with a line on the rule and when an examination of the other lines on the vernier scale was made none were found to aline with the rule either. These small sources of error must be eliminated or checked if any close work to determine crystal structure is to be made.

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APPENDIX

Table I

Olivine Cell Dimensions

In Angstrom Units

Mineral	$a_\circ$	$b_\circ$	$c_\circ$	Investigator
Forsterite	4.77	10.26	5.99	Rinne[31]
Forsterite Mg-Fe: 98-2	4.730	10.195	5.990	Winchell*
Forsterite Mg-Fe: 92-8	4.760	10.220	5.990	"
Chrysolite Mg-Fe: 9-1	4.755	10.21	5.985	Bragg[34]
Chrysolite Mg-Fe: 6-1	4.77	10.28	6.00	Rinne[31]
Chrysolite Mg-Fe: 82.5-17.5	4.77	10.245	6.010	Winchell*
Hyalosiderite Mg-Fe: 2-1	4.94	10.56	6.23	Rinne[31]
Hortonolite Mg-Fe: 32-68	4.79	10.335	6.065	Winchell*
Fayalite Mg-Fe: 4-96	4.823	10.482 10.483	6.094	"
Fayalite	4.80	10.59	6.16	Rinne[31]

* Personal communication of unpublished data

Table II

The d/n values of the ferromagnesian olivine minerals as determined from powder photographs. The data of Clarke[38] and the A. S. T. M. Card Index[39] are listed for the forsterite. The symbols employed to define intensities are VS, very strong; S, strong; MS, moderately strong; M, moderate; W, weak; VW, very weak; VVW, very very weak.

Forsterite

Line	I	II	III	Av.	Clarke	A.S.T.M.	I
1	5.082	5.086	5.092	5.087	5.1	5.1	M
2	4.302	4.299	4.293	4.298			VVW-
3	3.876	3.882	3.877	3.878(5)	3.85	3.89(3)	M
4	3.725	3.721	3.720	3.722	3.70	3.72	W
5	3.484	3.480	3.483	3.482	3.48	3.49	W
6	2.990	2.991	2.986	2.989	2.97	2.99	W
7	2.774	2.767	2.784	2.775(4)	2.74	2.77(2)	M
8	2.512	2.510	2.510	2.511(3)	2.49(3)	2.51	S
9	2.454	2.454	2.453	2.454(2)	2.44(2)	2.45	S
10	2.347	2.347	2.345	2.346	2.33		VW

Continued

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[38] Clarke, C. B., op. cit., p.28.

[39] American Society for Testing Materials, op. cit.

Line	I	II	III	Av.	Clarke	A.S.T.M.	I
11	2.307	2.308	2.306	2.307	2.30		VW
12	2.258	2.260	2.262	2.260(6)	2.25	2.26	M
13	2.226			2.226	2.23		VVW-
14	2.157	2.158	2.161	2.159		2.15	W
15	2.030	2.029	2.027	2.029	2.03	2.02	VVW
16	1.944	1.944	1.944	1.944	1.929	1.95	VVW
17	1.872	1.872	1.874	1.873	1.860	1.88	VVW
18	1.819	1.809	1.813	1.814	1.794	1.81	VVW
19	1.786	1.788	1.785	1.787	1.772		VVW
20	1.750	1.747	1.745	1.747(1)	1.733(1)	1.74(1)	S
21	1.668	1.669	1.670	1.669	1.657	1.67	VW
22	1.635	1.635	1.637	1.636			VW
23	1.616	1.617	1.618	1.617	1.620	1.62	VW
24	1.593			1.593	1.603		VVW-
25	1.569	1.568	1.569	1.569	1.575	1.57	VVW
26	1.495	1.496	1.497	1.496(7)	1.483	1.49	M
27	1.477	1.477	1.478	1.477(7)	1.466		M
28	1.433	1.435	1.437	1.435	1.421		VVW
29	1.394	1.392	1.393	1.393	1.382	1.395	VVW
30	1.348	1.348	1.351	1.349	1.338		W
31	1.313	1.313	1.314	1.314	1.302		W
32	1.294	1.294	1.293	1.294	1.282		W
33	1.241	1.241	1.241	1.241			VW

Continued

Line	I	II	III	Av.	Clarke	A.S.T.M.	I
34	1.222	1.221	1.222	1.222			VW
35	1.187	1.187	1.188	1.187			VW
36	1.166	1.165	1.164	1.165	1.179		VW
37	1.152	1.153	1.152	1.152	1.156		VW
38	1.144	1.147	1.148	1.146	1.145		VW
39	1.135	1.135	1.135	1.135	1.114		VW
40	1.099	1.098	1.099	1.099	1.089		VW

Chrysolite

Line	I	II	III	Av.	I
1	5.097	5.092	5.108	5.099	W
2	not visible				
3	3.875	3.874	3.877	3.875(5)	MS
4	3.719	3.719	3.720	3.719	W
5	3.479	3.478	3.481	3.479	W
6	2.994	2.994	2.993	2.994	W
7	2.764	2.763	2.763	2.763(4)	MS
8	2.511	2.508	2.509	2.509(3)	VS
9	2.455	2.453	2.457	2.455(2)	VS
10	2.348	2.347	2.349	2.348	VVW
11	2.312	2.311	2.310	2.311	VVW
12	2.262	2.260	2.258	2.260(6)	MS

Continued

Line	I	II	III	Av.	I
13	not visible				
14	2.151	2.157	2.162	2.157	W
15	2.029	2.031	2.033	2.031	VVW
16	1.947	1.948	1.943	1.946	VVW
17	1.876	1.877	1.879	1.877	VVW
18	1.845			1.845	VVW-
19	1.788	1.789	1.789	1.789	VVW-
20	1.746	1.747	1.748	1.747(1)	VS
21	1.670	1.670	1.671	1.670	VVW
22	1.635	1.636	1.638	1.636	VVW
23	1.617	1.617	1.618	1.617	VVW
24	1.592			1.592	VVW-
25	1.570	1.570	1.571	1.570	VVW
25a	1.512	1.512	1.513	1.512	VVW
26	1.497	1.497	1.497	1.497(7)	MS
27	1.479	1.478	1.480	1.479(7)	MS
28	1.435	1.436	1.437	1.436	VVW
29	1.394	1.395	1.395	1.395	W
30	1.350	1.350	1.351	1.350	W
31	1.315	1.315	1.315	1.315	W
32	1.294	1.295	1.294	1.294	VVW
33	appears to merge with 32				
34	1.224	1.224	1.224	1.224	VVW

Continued

Line	I	II	III	Av.	I
35	1.189	1.188	1.189	1.189	VVW
36	1.167	1.167	1.168	1.167	VVW
37	1.155	1.154	1.154	1.154	VVW
38	not visible				
39	1.136	1.136	1.135	1.136	VVW
40	1.099	1.099	1.098	1.099	VVW

Hyalosiderite

Line	I	II	III	Av.	I
1	5.089	5.120	5.118	5.109	W
2	not visible				
3	3.881	3.886	3.886	3.884(5)	MS
4	3.714	3.718	3.722	3.718	W
5	3.484	3.488	3.495	3.492	W
6	2.986	2.988	2.991	2.988	W
7	2.764	2.763	2.765	2.764(4)	MS
8	2.511	2.508	2.511	2.510(3)	VS
9	2.453	2.454	2.456	2.454(2)	VS
10	2.349	2.349	2.349	2.349	VVW
11	2.309	2.312	2.312	2.311	VVW
12	2.255	2.259	2.258	2.257(6)	MS
13	not visible				
14	2.157	2.155	2.158	2.157	W

Continued

Line	I	II	III	Av.	I
15	2.030	2.029		2.030	VVW
16	1.948	1.947	1.947	1.947	VVW
17	1.859	1.860	1.873	1.864	VVW
18	1.807	1.807	1.808	1.807	VVW-
19	1.788	1.788	1.790	1.789	VVW-
20	1.751	1.747	1.746	1.748(1)	VS
21	1.668	1.670	1.672	1.668	VVW
22	1.635	1.635	1.638	1.636	VVW
23	1.617	1.617	1.619	1.618	VVW
24	not visible				
25	1.570	1.570	1.572	1.571	VVW
25a	1.514	1.513	1.511	1.513	VVW
26	1.496	1.496	1.497	1.496(7)	MS
27	1.480	1.480	1.478	1.479(7)	MS
28	1.436	1.436	1.438	1.437	VVW
29	1.394	1.394	1.394	1.394	W
30	1.351	1.351	1.349	1.350	W
31	1.315	1.315	1.316	1.315	W
32	1.295	1.295	1.295	1.295	VVW
33	appears to merge with 32 in chrysolite				
34	1.222	1.222	1.224	1.223	VVW
35	1.187	1.188	1.188	1.188	VVW

Continued

Line	I	II	III	Av.	I
36	1.166	1.165	1.166	1.166	VVW
37	1.154	1.155	1.155	1.155	VVW
38	not visible				
39	1.135	1.134	1.135	1.135	VVW
40	1.098	1.097	1.099	1.098	VVW

Hortonolite

Line	I	II	III	Av.	I
1	5.126	5.126	5.140	5.131	VVW-
2	not visible				
3	3.889	3.880	3.878	3.883(6)	VW
4	3.713	3.717	3.709	3.713	VVW-
5	3.485	3.490	3.488	3.488(5)	MS
6	2.996	3.005	3.002	3.001	VVW
7	2.776	2.776	2.773	2.775(4)	VS
7a	2.606	2.589	2.618	2.604	VVW-
8	2.520	2.524	2.523	2.522(3)	VS
9	2.462	2.463	2.461	2.462(2)	VS
10	2.370	2.364	2.366	2.367	VVW
11	appears to merge with 10				
12	2.270	2.267	2.268	2.268(6)	VW
13	not visible				

Continued

Line	I	II	III	Av.	I
14	2.160	2.165	2.174	2.166	VVW
15	2.031	2.043	2.059	2.044	VVW
16	1.959	1.974	1.977	1.970	VVW
17	1.896	1.894	1.894	1.895	VVW
18	1.843			1.843	VVW-
19	1.817	1.813	1.816	1.815	VVW
20	1.741	1.757	1.765	1.754(1)	VS
21	1.680	1.681	1.682	1.681	VVW
22	1.653	1.653	1.655	1.654	VVW
23	1.628	1.628	1.631	1.629	VVW
24	1.605	1.605	1.605	1.605	VVW
25	1.579	1.584	1.586	1.583	VVW
25a	1.521	1.519	1.522	1.521	VVW
26	1.508	1.507	1.508	1.508(7)	W
27	1.493	1.493	1.495	1.494(7)	W
28	1.447	1.448	1.445	1.447	VW
29	1.408	1.408	1.410	1.409	VW
29a	1.398	1.395	1.398	1.397	VW
30	1.358	1.357	1.360	1.358	VW
31	1.326	1.323	1.329	1.326	VW
32	not visible				
33	appears to merge with 32 in chrysolite				
34	not visible				

Continued

Line	I	II	III	Av.	I
35	1.193	1.197	1.196	1.195	VVW
36	not visible				
37	1.164	1.162	1.166	1.164	VVW
38	not visible				
39	1.146	1.147	1.145	1.146	VVW
40	1.106	1.106	1.107	1.106	VVW
41	1.084	1.086	1.086	1.086	VVW

Ferrohortonolite

Line	I	II	III	Av.	I
1	not visible				
2	not visible				
3	not visible				
4	not visible				
5	3.525	3.519	3.510	3.518(5)	MS
6	not visible				
7	2.801	2.801	2.797	2.800(3)	MS
7a	2.606	2.608	2.606	2.607	VVW
8	2.552	2.547	2.542	2.547(4)	S
9	2.487	2.481	2.480	2.482(2)	S
10	2.388	2.391	2.387	2.389	VW

Continued

Line	I	II	III	Av.	I
11	appears to merge with 10 in hortonolite				
12	2.291	2.291	2.287	2.290	VW
13	not visible				
14	2.181	2.180	2.183	2.181	VVW-
15	2.05	2.05	2.08	2.066	VVW-
16	1.980	1.979	1.978	1.979	VVW-
17	1.909	1.910	1.914	1.911	VVW-
18	appears to merge with 19				
19	1.830	1.830	1.828	1.829	VVW-
20	1.768	1.768	1.766	1.767(1)	VS
21	1.725	1.720	1.718	1.721	VVW
21a	1.696	1.696	1.696	1.696	VVW
22	1.669	1.670	1.670	1.670	VVW
23	1.643	1.643	1.643	1.643	VVW
24	1.617	1.617	1.617	1.617	VVW
25	1.599	1.597		1.598	VVW
25a	1.526	1.524	1.528	1.526	VVW
26	1.511	1.512	1.512	1.512(6)	MS
27	appears to merge with 26				
28	1.453	1.454	1.455	1.454	VVW-
29	1.426	1.426	1.426	1.426	VVW
29a	1.404	1.404	1.404	1.404	VVW

Continued

Line	I	II	III	Av.	I
30	1.366	1.367	1.367	1.367	VVW
31	1.336	1.336	1.336	1.336	VVW
32	1.283			1.283	VVW-
33	appears to merge with 32 in chrysolite				
34	not visible				
35	1.199	1.200	1.203	1.201	VVW
36	not visible				
37	1.170	1.171	1.173	1.171	VVW
38	not visible				
39	1.150	1.150	1.152	1.151	VVW
40	1.116	1.117		1.117	VVW
41	1.092	1.092	1.092	1.092	VVW

Fayalite

Line	I	II	III	Av.	I
1	not visible				
2	not visible				
3	not visible				
4	not visible				
5	3.510	3.506	3.513	3.510(5)	MS
6	not visible				
7	2.802	2.802	2.806	2.804(3)	S

Continued

Line	I	II	III	Av.	I
7a	2.607	2.609	2.611	2.609	VW
8	2.543	2.545	2.546	2.544(4)	MS
9	2.479	2.481	2.481	2.480(2)	S
10	2.390	2.391	2.391	2.391	VW
11	appears to merge with 10 in hortonolite				
12	2.289	2.290	2.290	2.290	VW
13	not visible				
14	not visible				
15	not visible				
16	1.980	1.972	1.987	1.980	VVW-
17	1.914	1.915	1.918	1.916	VVW-
18	appears to merge with 19 in ferrohortonolite				
19	1.831	1.830	1.832	1.831	VVW-
20	1.766	1.767	1.769	1.767(1)	VS
21	1.695	1.695	1.697	1.696	VVW
22	1.672	1.673	1.673	1.673	VVW
22a	1.643	1.643	1.643	1.643	VVW
23	1.617	1.618	1.619	1.618	VVW
24	not visible				
25	1.521	1.526	1.529	1.525	VVW
26	1.498	1.484	1.515	1.499(6)	MS
27	appears to merge with 26 in ferrohortonolite				
28	1.457	1.454		1.456	VVW

Continued

Line	I	II	III	Av.	I
29	1.424	1.426	1.428	1.426	VVW
29a	1.404	1.404	1.405	1.404	VVW
30	1.367	1.368	1.370	1.369	VVW
31	1.334	1.335	1.336	1.335	VVW
32	1.297	1.282	1.283	1.287	VVW
33	appears to merge with 32 in chrysolite				
34	not visible				
35	1.201	1.202		1.202	VVW
36	not visible				
37	1.171	1.171	1.170	1.171	VVW
38	not visible				
39	1.151	1.151	1.152	1.151	VVW
39a	1.128	1.128	1.130	1.129	VVW
40	1.1161	1.1166	1.1167	1.117	VVW
41	1.093	1.093	1.093	1.093	VVW