Correlations between the Infrared Spectrum and the Composition of Garnets in the Pyrope-Almandine-Spessartine Series

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Abstract. The i.r. spectrum of 13 analyzed garnets of the pyralspite group has been investigated in the 1400–200 cm^{-1} region, and correlations have been found between the spectrum and the chemical composition. The results include: typical features in the spectrum of the end-members pyrope, almandine and spessartine; relationships between the spectrum and the pyrope percentage in pyrope-almandine solid solutions; and the influence of the CaO (grossularite) amount on the shape of the low-frequency absorption bands. These data allow a semi-quantitative determination of the pyrope percentage in pyrope-almandine solid solutions.

Introduction

A garnet is generally characterized, either by its composition deduced from a chemical analysis, or by some of its physical properties such as the unit cell parameter, the refractive index and the density, whose simultaneous knowledge may lead to the type of the garnet through the use of the well-known Winchell diagram.

If properly carried out, the chemical analysis by the classical wet method is one of the most precise and most reliable methods leading to the chemical composition, but it requires a substantial amount of carefully purified materials, and it is time-consuming.

On the other hand, the use of the Winchell diagram may be misleading, in some instances at least, since there is no unique relationship between the chemical composition and the set of measured physical properties.

The infrared spectroscopy appears to be an attractive potential tool for the investigation of such analytical problems, since the vibrational frequencies depend on the nature of the cations, and thus on the chemical composition of the garnet. Moreover, this method is fairly rapid and requires only samples of a few milligrams.

The i.r. spectra of garnets and their interpretation have already been discussed (Moenke, 1961; Omori, 1971), but these papers are limited to the study of the correlations between the spectrum and the garnet *type* (pyrope, almandine, grossularite ...), and they do not consider the more difficult problem of more precise relationships between the spectrum and the true composition of a garnet which, in most cases, is a complex solid solution between the different end-members.

We shall discuss here the i.r. spectra of natural garnets which are essentially related to the pyrope-almandine solid solutions. Other types of garnets are at present being investigated, and the results will be reported in due time.

Experimental

Origin and Purification of the Samples. All the samples have been chosen in the mineralogical and geological collections of the Musée Royal de l'Afrique Centrale. After grinding, the garnets have been separated from the accompanying rock by conventional methods (magnetic and density separation).

A series of samples were then selected on the basis of their X-ray powder diagram and optical properties. These samples are listed in Table 1, which gives their numbering in the

Table 1. Origin and characterization of the garnets

| G Nr	R. G. Nr	Sample localities	Host rocks	Cell edge: a (Å)	Refractive index
1	G. 12211	Musefu (Kasai, Zaïre)	Garnet sillimanite cordierite granu- lite of charnockite complex	11.510 ± 0.005	1.778 ± 0.001
2	M. 3546	Mbuji-Mayi (Kasai, Zaïre)	Eluvial deposit from kimberlite	11.535 ± 0.005	1.747 ± 0.001
3	M. 12316	Trevenice (Czechoslovakia)	Eluvial deposit from kimberlite	11.520 ± 0.005	1.746 ± 0.001
4.	G. 71183	Mutara (Rwanda)	Granitic gneiss	11.605 ± 0.005	1.805 ± 0.003
5	G. 106826	Carangola (Minas Gerais St., Brazil)	Granulite of the metasedimentary Paraiba serie	11.512 ± 0.005	1.797 ± 0.003
6	M. 10723	Kisenge (Shaba, Zaïre)	Eluvial deposit from the gneissic basement	11.535 ± 0.005	1.800 ± 0.003
7	G. 100413	Stavanger (Norway)	Hornblende granu- lite facies gneiss with cordierite and sillimanite	11.522 ± 0.005	1.801 ± 0.003
8	G. 100212	Storefjell (Norway)	Banded pyroxene gneiss	11.517 ± 0.005	1.781 ± 0.001
9	M. 11252	Shai Hills quarry (Ghana)	Phenocrysts in a garnet-rich gneiss	11.580 ± 0.005	1.776 ± 0.001
11	G. 106719	Alberto Torres (Rio de Janeiro St., Brazil)	Migmatitic granitic gneiss	11.546 ± 0.005	1.804 ± 0.003
12	M. 4824	Kalukangala (Kivu, Zaïre)	Alluvial deposit in gneissic country	11.543 ± 0.005	1.814 ± 0.003
13	M. 1873	Kayove (Rwanda)	Massive garnet of unknown origin	11.530 ± 0.005	1.813 ± 0.003
14	M. 786	Kalu (Kivu, Zaïre)	Alluvial deposit in gneissic country	11.617 ± 0.005	1.799 ± 0.003
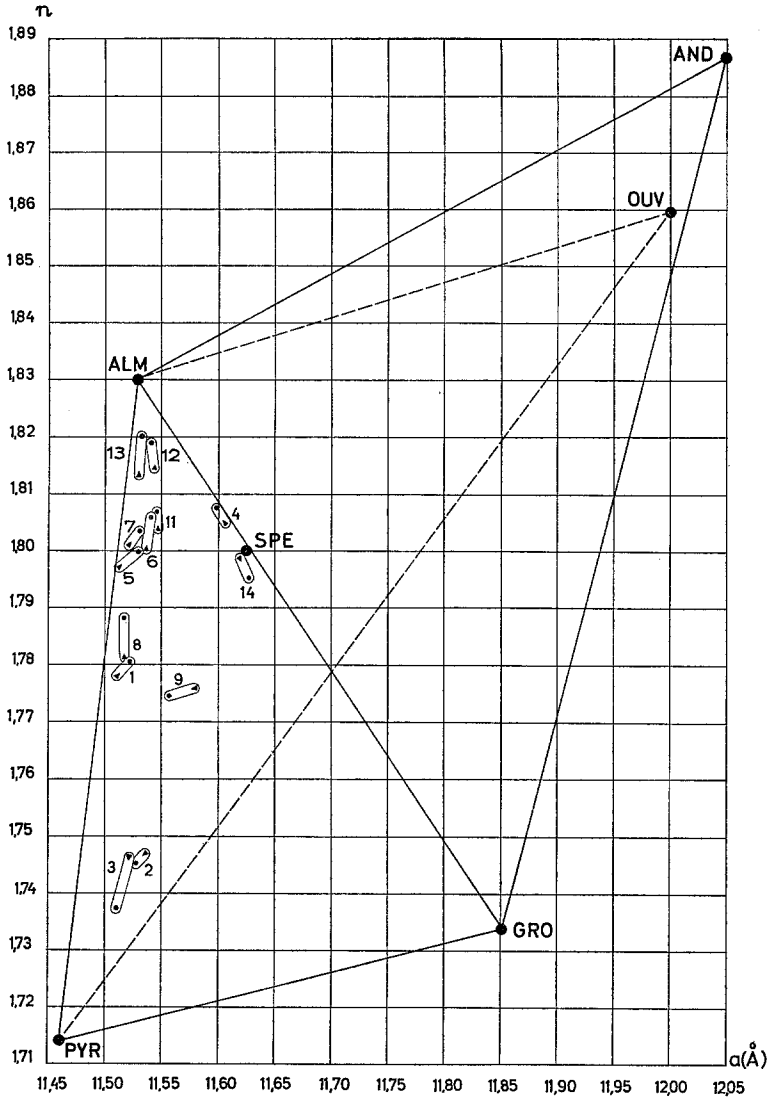
Fig. 1. Localization of the garnets in a Winchell diagram: circles: deduced from the chemical analysis; triangles: deduced from the unit cell parameter a_0 and the refractive index n

present investigation, their catalog number of the Musée Royal de l'Afrique Centrale, their geographical origin, the type of the host rock, the unit cell parameter a_0 and the refractive index n.

Chemical Composition. Some of the garnets [labeled (1) in Table 2] have been analyzed by wet chemical methods; the others [labeled (2)], have been analyzed by X-ray fluorescence, the chemically analyzed samples being used as standards. The analytical results, and the corresponding virtual compositions are collected in Table 2.

These data have been plotted in a triangular diagram where they are compared with the compositions deduced from the combined determinations of the unit cell parameter a_0 and the refractive index n (Fig. 1).

Table 2. Chemical analyses and virtual composition of garnets. (1) analysed by wet chemical methods. (2) analysed by X-ray fluorescence

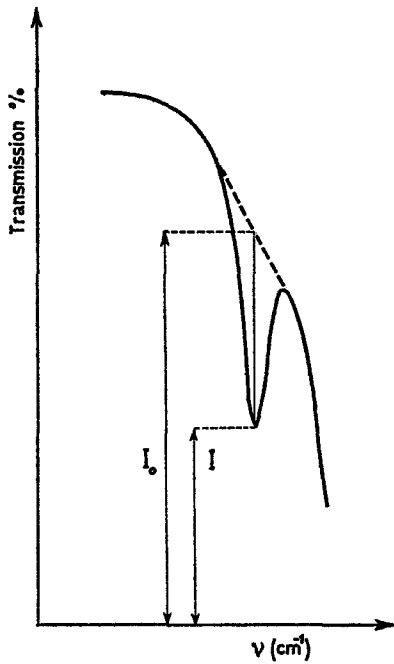| %	G1(1)	G2(1)	G3(2)	G4(1)	G5(2)	G6(2)	G7(2)	G8(2)	G9(1)	G11(2)	G12(2)	G13(1)	G14(1)
SiO_2	38.90	41.40	42.96	37.50	38.51	37.97	36.92	39.04	39.20	39.04	36.43	36.10	36.85
Al_2O_3	21.90	20.00	21.37	20.60	20.32	20.32	20.73	21.51	20.45	18.93	19.83	20.90	20.30
FeO	26.30	7.50	8.96	32.30	33.27	30.86	32.25	29.06	23.30	31.95	36.62	36.80	1.60
Fe_2O_3	—	—	—	—	—	—	—	—	—	—	—	—	0.10
MgO	9.70	21.50	20.11	0.28	5.36	3.16	4.69	8.45	9.80	3.19	1.21	1.33	0.66
CaO	2.20	4.50	4.06	6.80	1.59	1.72	0.76	0.98	5.45	1.63	0.50	0.57	1.52
TiO_2	0.10	0.42	0.47	0.04	0.15	0.12	0.09	0.08	0.22	0.61	0.03	1.21	0.48
MnO	0.35	0.34	0.58	1.85	0.58	4.67	2.08	0.68	1.10	4.54	4.11	2.50	37.80
P_2O_5	0.02	0.02	0.04	0.05	0.04	0.04	0.05	0.02	0.12	0.08	0.20	0.04	0.02
Na_2O	0.08	0.12	—	0.11	—	—	—	—	0.06	—	—	0.05	0.02
K_2O	0.05	<0.01	0.02	0.07	0.04	0.05	0.06	0.03	<0.01	0.06	0.02	0.04	<0.01
V_2O_5	—	—	—	—	—	—	—	—	—	—	—	—	0.12
NiO	—	0.62	—	—	—	—	—	—	—	—	—	—	—
Cr_2O_3	—	3.85	—	—	—	—	—	—	—	—	—	—	—
	99.60	100.28	98.57	99.60	99.86	98.91	97.63	99.85	99.71	100.03	98.95	99.54	99.48
PYR	37.0	73.9	70.9	1.2	21.0	12.9	19.1	32.8	35.7	12.8	5.0	5.6	2.7
ALM	56.2	14.5	17.8	74.5	73.3	71.1	73.8	63.0	47.6	72.1	83.9	86.8	3.7
GRO	6.0	0.8	10.2	20.0	4.4	5.1	2.3	2.6	14.3	4.7	1.5	1.7	4.0
SPE	0.8	0.5	1.1	4.3	1.3	10.9	4.8	1.6	2.4	10.4	9.6	5.9	89.1
UVA	—	10.3	—	—	—	—	—	—	—	—	—	—	—
AND	—	—	—	—	—	—	—	—	—	—	—	—	0.5

Fig. 2. Application of the base line method to the determination of the intensity of the 635 cm^{-1} band

Table 3. Infrared frequencies (in cm^{-1})

G1	G2	G3	G4	G5	G6	G7	G8	G9	G11	G12	G13	G14
998s	995s	995s	985s	993s	990s	990s	995s	990s	992s	990s	990s	975s
969	967	968	957	967	962	965	968	960	962	963	963	945
903	898	898	893	901	898	900	902	894	898	898	900	883
878	871	870	872	877	874	876	878	869	874	875	876	860
	815s	815s										
642b			633	640	638	639	642	640b	638	637	636	632
575	577	575	565	572	568	570	574	568	568	568	567	561
533	533s	530s	520s	529	528s	530	532	525s	527	527	528	520s
515s				515s	515s		515s		512s	510s	510s	508s
478	480	480	476	475	475	475	475	477	474	473	473	470
456	457	458	453	453	453	454	455	454	453	452	451	448
417s		420s		415s								414
382	384	385	385	380	380	380	380	385	380	378	377	382
343				343	344	343	343		344	343	342	350
	336	338	323					330				
330				326	323	324	328		322	320	319	317
241	250	250	235b	240	240	239	240	238b	240	240	239	245
												207

s = shoulder; b = broad band.

Infrared Spectra. Since the garnets under consideration are rather hard, and since the shape and intensity of the i.r. absorption bands are sensitive to the granulometry of the sample, much attention must be paid to the grinding of the sample. The garnets are ground in a tungsten carbide mortar, and the resulting powder is mixed with isopropyl alcohol

(about 20 ml alcohol for about 0.02 g of powder). After 2 hours, the supernatant suspension is submitted to centrifugation. This procedure leads to the separation of a very fine-grained fraction.

The conventional pressed disc technique is then applied to this fraction: 2.5 or 1 mg of powder is carefully mixed with 0.6 g of dry potassium iodide, and the mixture is pressed under vacuum so as to give a transparent disc of 20 mm diameter.

The spectra have been registered with a Beckman IR 12 spectrophotometer in the 1400 to 200 cm^{-1} region. The position of sharp bands may be read to ± 1 cm^{-1}. The intensity of some bands has been determined, either as the surface of the band (integrated intensity) or as the peak intensity by using the base-line method (Fig. 2).

Results

The observed i.r. frequencies are collected in Table 3, and a typical spectrum is represented by Fig. 3.

We shall not consider here the high-frequency region of the spectrum (1000 to 800 cm^{-1}). These bands are essentially related to the antisymmetric stretching motions of the SiO_4 tetrahedra in the garnet structure, and previous studies (Tarte, 1965) have shown that their frequency is essentially related to the unit cell parameter a_0, and not to the presence of specific cations. Likewise, among the numerous bands observed in the 650–200 cm^{-1} region, we shall not consider the strong doublet observed in the 500–400 cm^{-1} region, since the position and relative intensities of these bands are not very sensitive to the changes of chemical composition, at least in the pyralspite family.

Thus, we shall restrict the discussion to the behaviour of the band generally observed near 650–600 cm^{-1}, and to the low-frequency region (below 400 cm^{-1}).

The Behaviour of the Band in the 650–600 cm^{-1} Region

This band is observed in the spectrum of most garnets, its frequency being related to the value of the unit cell parameter a_0 (Tarte, 1965). *But it is missing in the spectrum of typical pyrope*, and this feature has already been considered as a good identification test for pyrope (Moenke, 1961; Tarte, 1965).

We have now found that the *intensity* of this band in other garnets of the pyralspite series depends on the pyrope percentage. This appears clearly in Fig. 4, where we have plotted the intensity of this band (expressed as the logarithm of either the integrated intensity, or of the peak intensity) as a function of the pyrope percentage.

The significance and limitations of this relationship must now be discussed. It is well known that the measurement of the true intensity of an i.r. absorption band is a matter of difficulty, and a detailed discussion of this problem is out of the scope of this paper. We shall simply recall that this intensity may be expressed, either as an integrated intensity over the whole surface of the band, or as a peak intensity giving the transmittance (or the absorbance) corresponding to the maximum of absorption.

From a theoretical point of view, the integrated intensity is certainly more reliable than the peak intensity, since it takes into account the natural width of the band. But its correct measurement is difficult since the limits of the "wings" of a band cannot be defined with precision; this difficulty, and the resulting error on the intensity measurement, is the greatest for the weak bands for which the wings represent an important contribution to the total intensity of the band.
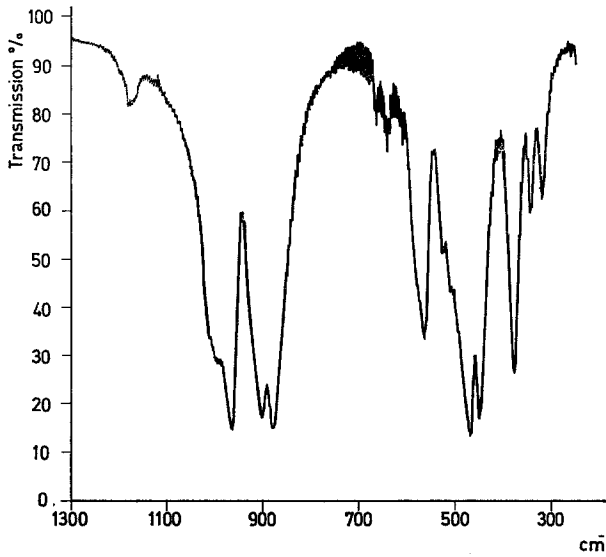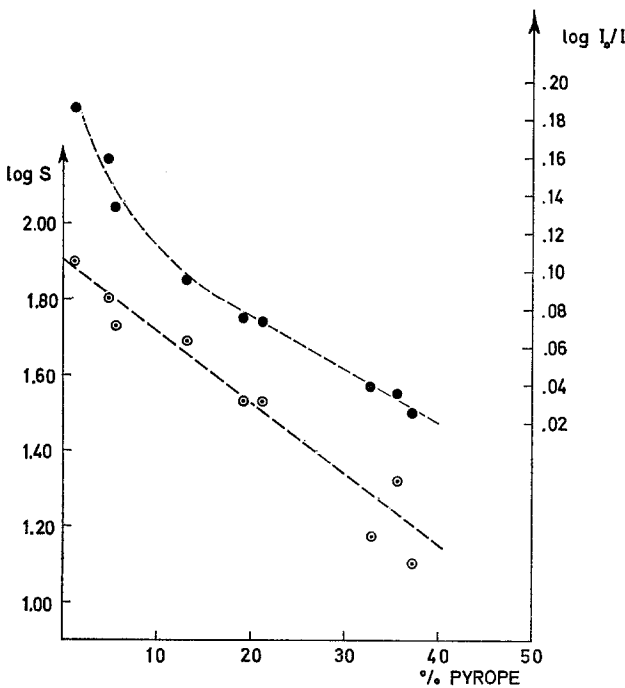
Fig. 3. Typical spectrum of a garnet

Fig. 4. Intensity of the 635 cm^{-1} band as a function of pyrope percentage in pyrope-almandine garnets. Open circles and left hand scale: intensity expressed as the logarithm of the surface of the band (in arbitrary units); full circles and right hand scale: intensity expressed as the logarithm of the I_0/I ratio

Now, if the bandwidth remains essentially constant in a series of compounds, and if relative (and not absolute) intensities only are needed, it is much more simple to measure the peak intensity.

These conditions are fulfilled for the band under consideration, and it is thus expected that both methods (integrated intensity and peak intensity) should lead to similar (although not identical) results.

This is indeed the case (Fig. 4), but it should be noticed that the experimental points related to weak intensities are considerably more scattered for the surface than for the peak intensity; this is probably related to the above-mentioned difficulty, namely the uncertainty on the wing limits for the bands of low intensity.

For this reason, and since in addition the measurements are more easy and more rapid for the peak intensities, this latter method should be chosen in the actual case.

Finally, the limitations of this relationship should be briefly discussed. All the garnets involved in this relation are essentially of the pyrope-almandin type, but may contain a substantial (up to 20%) amount of grossularite (or uvarovite) without detectable alteration of the relation between the intensity and the pyrope percentage. On the other hand, the intensity of this band is significantly greater for spessartine than for almandine, and it is thus probable that the relation under consideration cannot be applied without modification to garnets which contain a more or less important percentage of spessartine.

The Low-Frequency Region (400–200 cm⁻¹)

The Low-Frequency Region ($400–200$ cm^{-1})

Three points will be considered here, namely the broadening of these bands in connection with the CaO amount, the spectra of the end-members, and some characteristic features in the pyrope-almandine series.

1. Broadening of the Bands. The most conspicuous feature in this spectral region is the more or less important broadening of the low-frequency bands in the spectrum of some sample (Fig. 5).

A comparison with the chemical composition (Table 2) immediately shows that this broadening is related to the presence of significant amounts of calcium oxide (4 to 6.80% CaO, corresponding to 10 to 20% grossularite + uvarovite) in the pyrope-almandine series. On the contrary, the absorption bands remain sharp, irrespective of the pyrope/almandine ratio, if the CaO amount is reasonably low: 2.20% CaO (or less) corresponding to 6% grossularite.

This qualitative observation may be put on a semi-quantitative scale by considering the absorption observed in the 350–300 cm⁻¹ region. For low CaO amounts, this absorption is represented by a doublet, the components of which coalesce into a single broad band when the CaO amount reaches 4% or more.

For the garnets with a significant amount of CaO and thus a single band in the 350–300 cm⁻¹ region, it is tempting to consider that the position of this band is related to the CaO percentage. And indeed this seems to be the case (Fig. 6, open circles). But unfortunately, in our actual samples of this type, the pyrope/almandine ratio is also varying in the same manner, and there is an equivalent relationship between the observed frequencies and the pyrope amount (Fig. 6; full circles). This point will be discussed below.

2. Spectra of End-Members. Pure end-members were not available, but 2 samples are reasonably pure almandine (86.8%; Nr. G13) or spessartine (89.0%; Nr. G14). Besides substantial differences in the position of the high-frequency
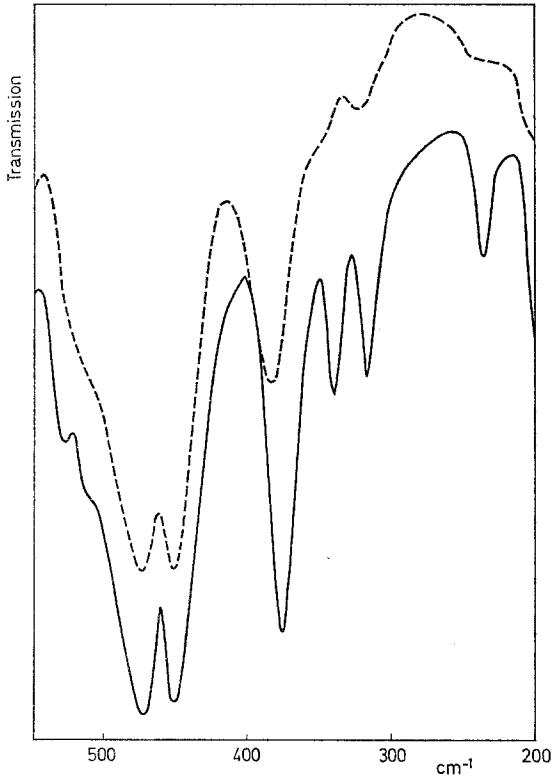
Fig. 5. Broadening of the low-frequency bands in connection with the occurrence of CaO in the pyrope-almandine group. Full line: sample G13 (almandine 86.8%; grossularite 1.7%); dotted line: sample G4 (almandine 74.5%; grossularite 20.0%)
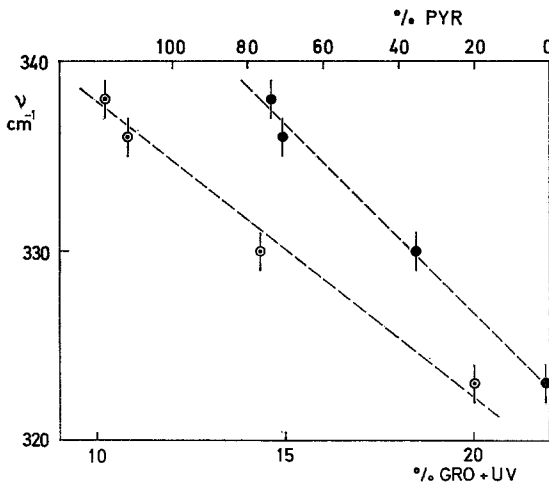

Fig. 6. Position of the unique band in the 350–300 cm⁻¹ region for pyrope-almandine garnets containing significant amounts of CaO. Open circles and lower scale: as a function of the grossularite + uvarovite percentage; full circles and upper scale: as a function of the pyrope percentage

bands (1000–800 cm⁻¹ region, Table 3), there are also significant variations between these 2 garnets for the doublet observed in the 350–300 cm⁻¹ region. The low-frequency component of this doublet has approximately the same intensity and nearly the same frequency for both garnets (319 cm⁻¹ for almandine;
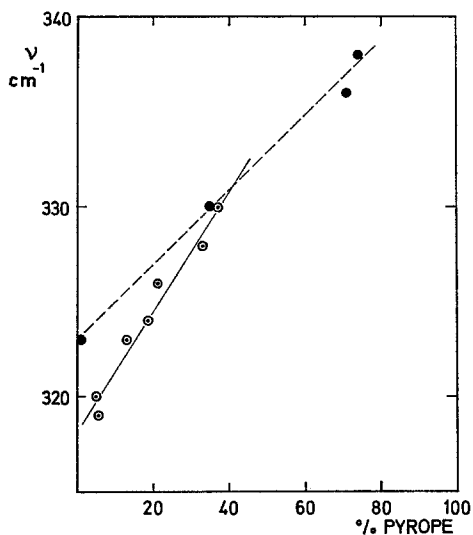
Fig. 7   Fig. 8

Fig. 7. Open circles: relationship between the position of the low-frequency component of the doublet in the 350–300 cm^{-1} region and pyrope percentage for pyrope-almandine with low CaO amount. The relationship for garnets with moderate CaO amount (full circles) is repeated for the purpose of comparison
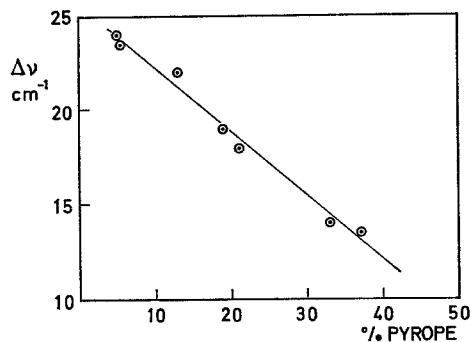
Fig. 8. Relationship between pyrope percentage and the splitting between the components of the doublet in the 350–300 cm^{-1} region

317 cm^{-1} for spessartine); but the high-frequency component has a lower intensity and a higher frequency for spessartine (350 cm^{-1}) than for almandine (342 cm^{-1}). As a consequence, the splitting between the 2 bands is greater for spessartine (33 cm^{-1}) than for almandine (23 cm^{-1}).

We have no really equivalent data on pyrope, since our purest sample is only 73.9% in pyrope. In contrast with the doublet observed in the spectra of almandine and spessartine, the spectrum of the investigated pyrope is characterized by the occurrence of one single band in the 350–300 cm^{-1} region; but since this sample contains a significant amount of CaO, it is actually impossible to determine whether the single band is the result of this CaO amount, or if it is specific for the pyrope garnet.

3. Garnets of the Pyrope-Almandine Series. For these garnets with a fairly low amount of CaO, which exhibit a doublet in the 350–300 cm^{-1} region, the high-frequency component of the doublet is observed at a practically constant frequency of 343 ± 1 cm^{-1}, irrespective of the chemical composition of the garnet and thus of the pyrope/almandine ratio. But the low-frequency component of the doublet is shifted from 319 up to 330 cm^{-1} when the pyrope amount increases from 5 to 37%, with a regular relationship between the frequency and the pyrope percentage (Fig. 7). Since the position of the high-frequency component is practically constant, an equivalent relationship is obtained by plotting the frequency difference between the 2 components as a function of the pyrope percentage (Fig. 8). From

a purely practical point of view, this latter relationship is more convenient than the preceding one, since it is free from any systematic frequency error coming from a calibration defect of the spectrophotometer. This equivalence, however, disappears for garnets which would contain a significant amount of spessartine.

Since the position of the low-frequency band is nearly the same for almandine (319 cm^{-1}) and for spessartine (317 cm^{-1}), it is expected that the relationship between this frequency and the pyrope percentage (Fig. 7) is nearly the same for the pyrope-almandin, and for the pyrope-almandin-spessartine series (except for spessartine-rich samples). But since the splitting between the 2 components is greater for spessartine (33 cm^{-1}) than for almandine (23 cm^{-1}), the relationship presented in Fig. 8 is not valid for garnets which contain a significant amount of spessartine.

Finally, it is worth while to briefly comment on the composition-frequency relationship for the 2 types of garnets investigated, namely the garnets which are poor in CaO and exhibit a doublet, and the garnets which contain a greater amount of CaO and exhibit a single band in the 350–300 cm^{-1} region. It is found that the slope of the frequency-composition relationship is very similar for the 2 types of garnets (Fig. 7). This would suggest that the frequency-composition relationship has essentially the same origin in both cases, and thus that, for the garnets with a significant amount of CaO and a single band in the 350–300 cm^{-1} region, the relationship is predominantly (although not uniquely) determined by the pyrope percentage, and not by the CaO percentage as previously suggested.

Discussion

Besides the possibilities of practical applications, it would be important to understand the theoretical significance of the actual results.

Despite its interest, this point cannot be discussed at length here, because of our insufficient knowledge of the origin of the bands in the garnet spectrum.

Thus, the band of the 650–600 cm^{-1} region has been assigned, either to a bending motion of the SiO_4 tetrahedron (Tarte, 1965), or to a vibration of the octahedral group $A^{III}O_6$ (A^{III} = Al, Cr or Fe) (Omori, 1971). We have good reasons to believe that the first assignment is the correct one, but a discussion of this particular point would be out of place here, and we are planning experiments which should give a definite answer to this type of problem. But whatever the final assignment, neither of them can explain why this band is practically missing (in fact, probably too weak to be observed) in the spectrum of pyrope.

Likewise, the low-frequency bands (below 400 cm^{-1}) have been assigned to vibrations involving the participation of the bivalent cations (Tarte, 1965; Moore et al., 1971); and indeed, this type of assignment is supported by the results reported in the present paper: the fact that some low-frequency bands are observed at a higher frequency for spessartine than for almandine is opposite to the general relationship between the frequencies and the unit cell parameter and suggests some specific influence of the manganese cations on these low-frequency vibrations. But experiments which are actually carried out in this laboratory show that the existing assignments are probably oversimplified, and

that some bands of the low frequency region are probably related to more complex vibrations.

Finally, it should be mentioned that the observed spectral modifications are related (except for the end members) to the formation of solid solutions, and that our fundamental knowledge of the vibrational behaviour of solid solutions is still very poor, particularly in complicated cases such as the garnets.

It is thus clear that a discussion of these points is premature.

Practical Applications

Our results may be applied to the identification and, in some cases at least, to a semi-quantitative evaluation of the composition of a garnet which belongs to the pyralspite family.

It should be first remembered that the position of the i.r. bands in the 1000 to 800 cm^{-1} region allows an immediate discrimination between the pyralspite and the ugrandite family, and that, in this last family, the andradite is immediately identified through its very specific i.r. spectrum (Moenke, 1961; Tarte, 1965). The following discussion will thus be restricted to the garnets of the pyralspite family.

1. End Members. Typical *spessartine* is identified by the following *simultaneous* characters: a relatively low frequency for the 3 strong bands of the 1000–800 cm^{-1} region (typical values are 945, 883 and 860 cm^{-1}, significantly lower than the corresponding frequencies in the pyrope-almandine series) *and* a large separation between the components of the doublet in the 350–300 cm^{-1} region (350 and 317 cm^{-1}). Spessartine containing significant amounts of pyrope or/and almandine should be characterized by higher frequencies in the 1000–800 cm^{-1} region *and* a smaller separation of the doublet in the 350–300 cm^{-1} region.

For typical *almandine*, the 3 strong high-frequency bands are located near 963, 900 and 876 cm^{-1}, and the components of the low-frequency doublet are observed at 342 and 319 cm^{-1}, the splitting being thus smaller than for spessartine.

Nearly pure *pyrope* was not available, but the best samples (71 to 74% in pyrope) are essentially (and immediately) characterized by the lack of an absorption band in the 650–600 cm^{-1} region: this is certainly the most characteristic feature of the pyrope spectrum.

2. Pyrope-Almandine Solid Solutions. Several relationships may be used to determine the pyrope percentage in these solid solutions, but some of them are probably not valid in the presence of substantial amounts of spessartine.

a) The intensity of the band near 635 cm^{-1} depends on the pyrope percentage (Fig. 4). This relation is still valid in the presence of moderate amounts of CaO (up to 6.80%, corresponding to 20% grossularite), but it has 2 disadvantages: first, the band under consideration has a larger intensity in spessartine than in almandine, and it is thus expected that this relationship cannot be used if significant amounts of spessartine are present; and second, from a purely practical point of view, the measurement of the band intensities is more difficult and more constraining than a frequency measurement.

b) The position of the low-frequency component of the doublet in the 350 to 300 cm^{-1} region is shifted from 319 cm^{-1} (5% pyrope) to 330 cm^{-1} (37% pyrope)

(Fig. 7) and, since the corresponding band is observed at 317 cm^{-1} in spessartine, this relation is probably useful in pyrope-almandine-spessartine ternary systems, as far as the spessartine percentage is not predominant.

c) The frequency difference between the 2 components of the doublet is also depending on the pyrope percentage (Fig. 8). But here, since this frequency difference is larger for spessartine than for almandine, the applicability of the relationship in Fig. 8 is restricted to garnets in the pyrope-almandine series and cannot be extended as such to garnets containing significant amounts of spessartine.

Finally, the relationships discussed under (b) and (c) cannot be applied to garnets containing more than 4% CaO (about 10% grossularite) since in this case the doublet is replaced by a single broad band. The existing data do not allow to decide whether the position of this band is determined by the pyrope percentage, or the grossularite percentage, or both. Further studies are needed ot settle this point.

Conclusions

The i.r. spectrum of analyzed garnets of the pyralspite family exhibits some interesting correlations between the chemical composition of the garnet and the frequency or intensity of some specific absorption bands. However, the investigation of additional samples is required for a better knowledge of the reliability and limitations of these correlations.

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