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Infrared Absorption in Microcline

By

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with 6 Tables and 7 Text-figures

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ABSTRACT: Infrared absorption spectra appearing on the microcline specimens collected from the Ishikawa pegmatite deposit in Fukushima Prefecture, Japan, have been prudently inspected especially by means of polarised radiation. The results thus obtained have not been so much agreeable with the data given previously for the fine-grained or synthesized specimens when examined under non-polarised radiation and even with those attained for particular orientation in contrast to the present work with polarised radiation along several directions on some specifically oriented thin-plates.

CONTENTS

I. INTRODUCTION

With reference to infrared absorption caused by several kinds of interatomic vibration in certain structures, a majority of excellent works have so far been brought to light by many authors. SAKSENA (1940, 1942, 1944, 1945 and 1961) as well as SAKSENA and NARAIN (1949) gave a very significant clue to estimation of the vibration frequencies concerned with the interatomic forces, LAUNER (1952) described presence of the regularities in the infrared absorption bands for the silicate minerals with various structures, SIMON and McMAHON (1953) referred to dichroism in infrared absorption, LAVES and HAFNER (1956 and 1962) dealt with infrared absorption of microcline, sanidine, adularia and so forth together with the mixtures of plagioclases, HAFNER and LAVES (1959) alluded to that attained for the series of potash feldspars, DACHILLE and ROY (1959) proposed its use for checking co-ordination, STUBIČAN and ROY (1961 a, b) clarified the relation of isomorphous substitution to infrared absorption and some others, WOLFF (1963 and 1965) put forth the behaviors of OH in the oriented flakes of kaolinite with inclined infrared radiation and in several clay minerals, DUKE (1964) demonstrated the infrared activity of Si-O tetrahedra due to the surrounding ions, and LÉONARD, SUSUKI, FRIPIAT and KIMPE (1964) manifested usefulnees of infrared procedure on discriminating Al co-ordination.

In addition, the works enlightened by DONNAY and DONNAY (1952), GOLDSMITH and LAVES (1954), LAVES and GOLDSMITH (1955), BAILEY and TAYLOR (1955), MEGAW (1956, 1961 a, b, c and 1962), MACKENZIE (1957 and 1959), McCONNELL (1960), JONES and TAYLOR (1961), MACKENZIE and SMITH (1962), and BROWN and BAILEY (1964) etc. are also to be mentioned owing to their contributions, though to more or less extent, to the present theme.

The stretching, bending or, some other kinds of, vibration relative to the interatomic bondings such as Si-O, O-O, O-Si-O, Si-O-Si, Si-O-Al, Al-O, O-Al-O and the likes have thus been designated respectively as each individual whereas three-dimensional inspection for such a complicated structure as discernible in that of microcline with miscellaneous alinement of electric vectors remains yet to be carried out. So, this study will preliminarily be developed along a scheme to find out, at least, difference in infrared absorption taking place on several orientations of the specimen referring to its structure determined already.

II. EXPERIMENTS

The mineral specimens subjected to infrared absorption spectroscopy are those taken out from a pegmatite mass occurring in Mt. Ishikawa well-known for production of some radioactive minerals, white in color but faintly striped with thin-layered alternation of grayish bands arranged seemingly parallel to the basal plane, vitreous in luster, relatively well-defined along pinacoid, prism, base and rarely orthodome in crystal habit and often twinned mainly after Carlsbad rule. Microscopic examination of the specimens in question exhibits the sporadically disposed inclusions of perthitic texture though in a little amount, while their x-ray diffraction patterns obtained at random for all parts and even in each stripe imply nothing but a meaningless trace of albitic composition (surely far less than some % in amount) and untraceable evidence for triclinicity. The thin-plates, 15μ in thickness, prepared through polishing the specimens with pulverized alumina are held carefully within the metallic frame, $0.7 \times 2.5\text{cm}^2$ in dimension, equipped normal to incident infrared radiation. In the cases of, for comparison, using the pulverised specimens the metallic ring holding KBr tablet mingled with the powder in a very few quantity is inserted into the receiving window in place of the frame.

The spectroscope provided for experiments is of the improved IR-27C type, constructed according to the authors' special order by Shimazu Man. Co. Ltd., annexed with the automatically moving recorder and various other instruments, and capable of being operated from 5000cm^{-1} to 400cm^{-1} in wavenumber by means of KBr prism kept in a monochromater chamber at constant temperature and humidity. A filter attached to the apparatus, though useful and necessary for cutting either a part of oblique radiation or obscurity caused by increasing dispersion with broadening slit below the range of less than 1000cm^{-1}, has not been operated so as to avoid discontinuity arisen in recording.

Exposing the oriented thin-plates cut parallel to the planes such as (001), (010), (100), (110) and (20$\bar{1}$) against the incident infrared radiation with definitely confined vibration and rotating the vibration plane per each interval of 15°~30° in angle, variations in status and intensity of absorption (transmission) have been pursued for scrutiny with recording speed of 1μ/min in wavelength or 5000~400cm^{-1}/23min in wavenumber.

III. RESULTS

The results obtained through the operations described above are illustrated in the figures and presented numerically in the tables. Basing on that the interatomic forces or electric vectors including dipoles and others are disposed regularly in the structure, it is accountably deducible that the infrared absorption behaviors are to be characteristic not only on each orientation of the specimen but also along each direction even in its similarly oriented section and that the data given for the fine-grained specimens, if impossible to prepare such thin-sections as are large enough in size for manipulations, are of course effectual for discriminating certain kinds of structures from one another but essentially different from those attained with use of polarised radiation. In the present case it is to be remarked that infrared transmission intervening between the range 1300~1000cm^{-1} in wavenumber, though to be clarified in the near future, has reached minimum in intensity owing to thickness of the section and instead that its details in the range of less than 1000cm^{-1} have become far more clear than ever known, as will be referred to in the following.

A. PRELIMINARY EXAMINATION WITH NON-POLARISED RADIATION

The infrared absorption spectra have been preliminarily examined for the pulverised specimens (*cf.* Fig. 1) and the oriented sections (*cf.* Fig. 2) within the range of 5000~400 cm^{-1} in wavenumber. Differences in transmission within the range of 778~417cm^{-1} are arranged in Table 1.

1. 5000~1000 cm^{-1}

Those appearing on (001), (010), (100), (110) and (20$\bar{1}$) are recognized most intensely at 1779cm^{-1} and 1613~1567cm^{-1} while those on the former two planes are found splitted into a number of bands and those on the latter three only into some parts. Those at 1300~1000cm^{-1} are, though very much significant for the stretching vibration of Si–O and for other main bondings, too conspicuous to be distinguished from one another.

2. 1000~500 cm^{-1}

As for the pulverized specimen, that at 769cm^{-1} is weaker in intensity than that at 730cm^{-1} while on the oriented sections both are respectively branched into more than two parts and either the former on (010), (100) and (110) or the latter on (001)

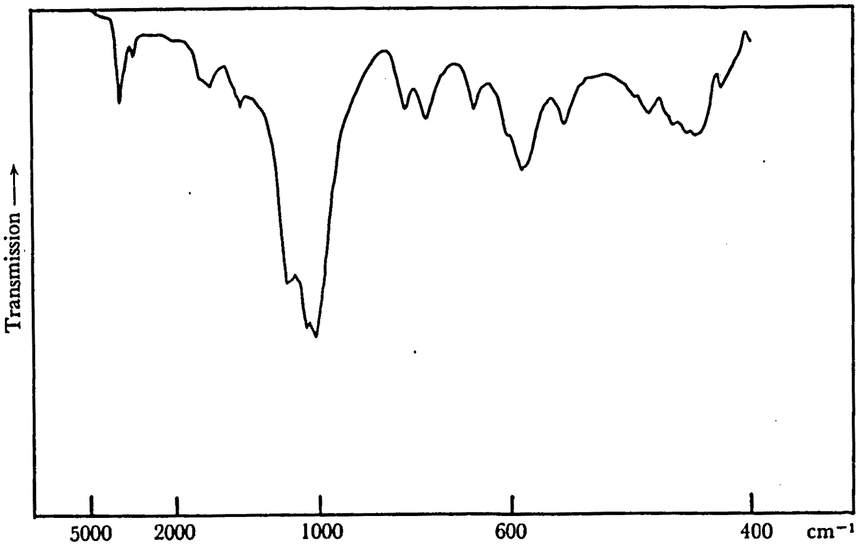

FIG. 1 Infrared absorption spectra for the pulverised specimen

and (20$\bar{1}$) are rather marked compared with those in the other cases. That observed near 604cm^{-1} as uncertain shoulder in association with those at 649cm^{-1} and 585cm^{-1} as well as that appearing at 585cm^{-1} as knick in case of the pulverized specimen are separated respectively into more than two bands, variable at 649cm^{-1} and invariable at 610cm^{-1}, for all of the oriented sections. That revealing at 585cm^{-1} in case of the powder is ascertainable nowhere on the oriented sections. This may be ascribed to emergence of certain vectors resulted from synthesis of each one alining at random especially in the former. Those splitted into more than two at 574～565cm^{-1} are apparent on (100) and (110) and very faint on (001) and (20$\bar{1}$) but not discernible on (010) and in the powder. That confirmed at 540cm^{-1} in the powder is divided into more than two with almost similar intensity at 546～536cm^{-1} on the oriented sections.

3. 500～400cm^{-1}

That seen at 476cm^{-1} in the powder is represented as ambiguous shoulder on (001) and (010) but never or almost not observable on the other planes. That discerned at 465cm^{-1} in the powder is dismembered into more than two at 469～461cm^{-1} clearly on (001) and (010) but slightly or as shoulder on the remainders. That detected at 448cm^{-1} in the powder corresponds to those at 455～450cm^{-1} on the oriented sections. Those at 441cm^{-1} and 417cm^{-1} are traceable in both cases.

B. DETAILED INSPECTION WITH POLARISED RADIATION

Variations in infrared absorption (transmission) have been traversed along each direction on the respective sections concerning some representative ranges of wave-

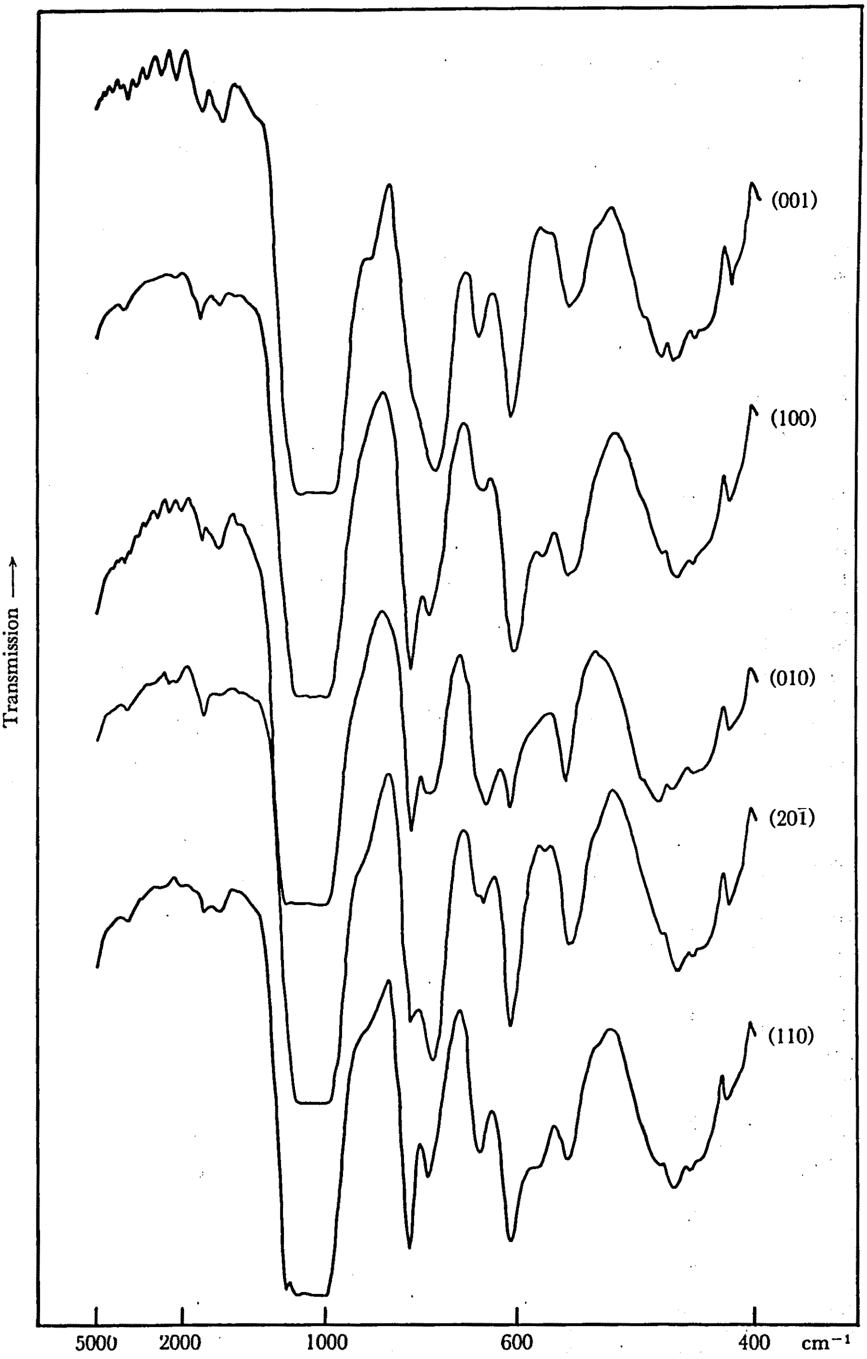

Fig. 2 Infrared absorption spectra on each oriented plane with non-polarised radiation

TABLE 1. DIFFERENCES in INFRARED ABSORPTION FOR RESPECTIVE PLANES

Wavenumber cm^{-1}	Transmission % on				
	(001)	(010)	(100)	(110)	$(20\bar{1})$
778				15	
774		18	10		20
768					
759					
741		25	20	30	
737					
735					
731	9				12
727					
658					
653	35		45	35	45
649					44
646					
642		24			
613					
610	20	23	13		20
608				17	
606					
574				31	
570			32		
567					54
565	56				
546		28	28		
543	42			33	36
541					
538					
536					
476	39	28	*		
469					
467		24			
465	32				
462			33	32	38
461					
455	32	26	28	28	
450				27	30
441	35	30	31	31	33
417	46	38	43	45	44

* uncertain shoulder (used similarly in all of the following tables)

number, as are indicated in the figures 3, 4, 5, 6 and 7 and in the tables 2, 3, 4, 5 and 6.

1. 778～759 cm^{-1}

More than two bands discernible with non-polarised radiation are furthermore subdivided into those at 786, 778, 774, 768 and 759 cm^{-1} probably in relation to arrangement of electric vectors composing the structure.

On (001), the absorption spectra, strikingly different from those on the others, are not separable because of high-grade absorption at 759 cm^{-1} and 741～727 cm^{-1} along the directions of 45°, 60° and 90° from b-axis but that appearing at 786 cm^{-1} is to be noticed in the last case (parallel to ac-plane). On (010), that observed at 774 cm^{-1} reveals variation in intensity (in the range of about 10%), culminating in a maximum at 0° (parallel to c-axis). On (100), that same as the former is also variable (in the range of about 4%), reaching a maximum at 90° (parallel to b-axis). On (100), those recognized at 778 cm^{-1} and 774 cm^{-1} are considerably indefinite in all angles. On $(20\bar{1})$, the corresponding band shows conversion from 778 cm^{-1} to 768 cm^{-1} with intensity (in the range of about 18%), revealing a maximum at 90° (parallel to b-axis).

2. 741～727 cm^{-1}

More than two bands confirmable with ordinary radiation are separable into those at 741, 737, 735, 731 and 727 cm^{-1}.

On (001), almost no directional variations are clarified. On (010), either broadening of the spectra or their shoulders are liable to appear, reaching a maximum in intensity at about 115° (parallel to ab-plane). On (100), the spectra are variable in wavenumber and in intensity (in the range of about 22%), displaying a maximum at 90° (parallel to b-axis). On (110), those indicate increment to 741 cm^{-1} at 0° (parallel to c-axis) and a maximum in intensity (variable in the range of 23%) at 90° (probably parallel to b-axis). On $(20\bar{1})$, those show decrease to 727 cm^{-1} and a maximum in intensity (variable in the range of about 5%) at 0° (parallel to b-axis).

3. 658～642 cm^{-1}

The absorption spectra within this range are branched into those of 658, 653, 649, 646 and 642 cm^{-1}.

On (001), those are remarkably inconstant (in the range of about 30%) and become splitted into two with increase of sharpness and intensity, pointing to a maximum at 90° (parallel to ac-plane). On (010), those bear a trend similar to those in the former case (though in the range of about 10%) and a maximum in intensity at 60°. On (100), those are generally broadened and remarkably variable in intensity (in the range of about 33%), eventuating in a maximum at 0° (parallel to ac-plane). On (110), those are dividable into two setting aside the broadened at 60° and exhibit variable intensity (in the range of about 26%) with a maximum at 150°. On $(20\bar{1})$, those at 649 cm^{-1} reveal a marked variation of intensity (in the range of about 36%) with a

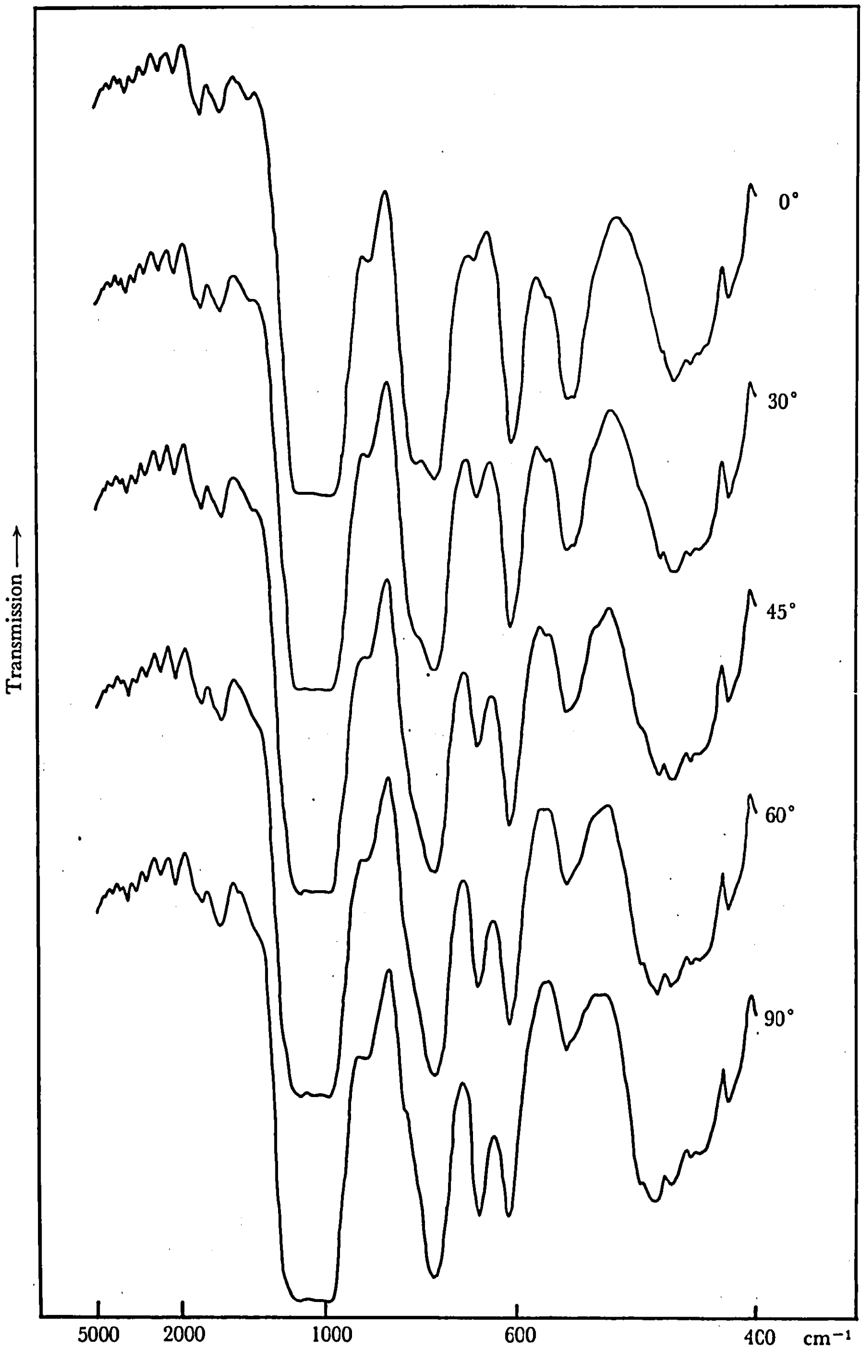

FIG. 3 Infrared absorption spectra along each direction on (001) with polarised radiation

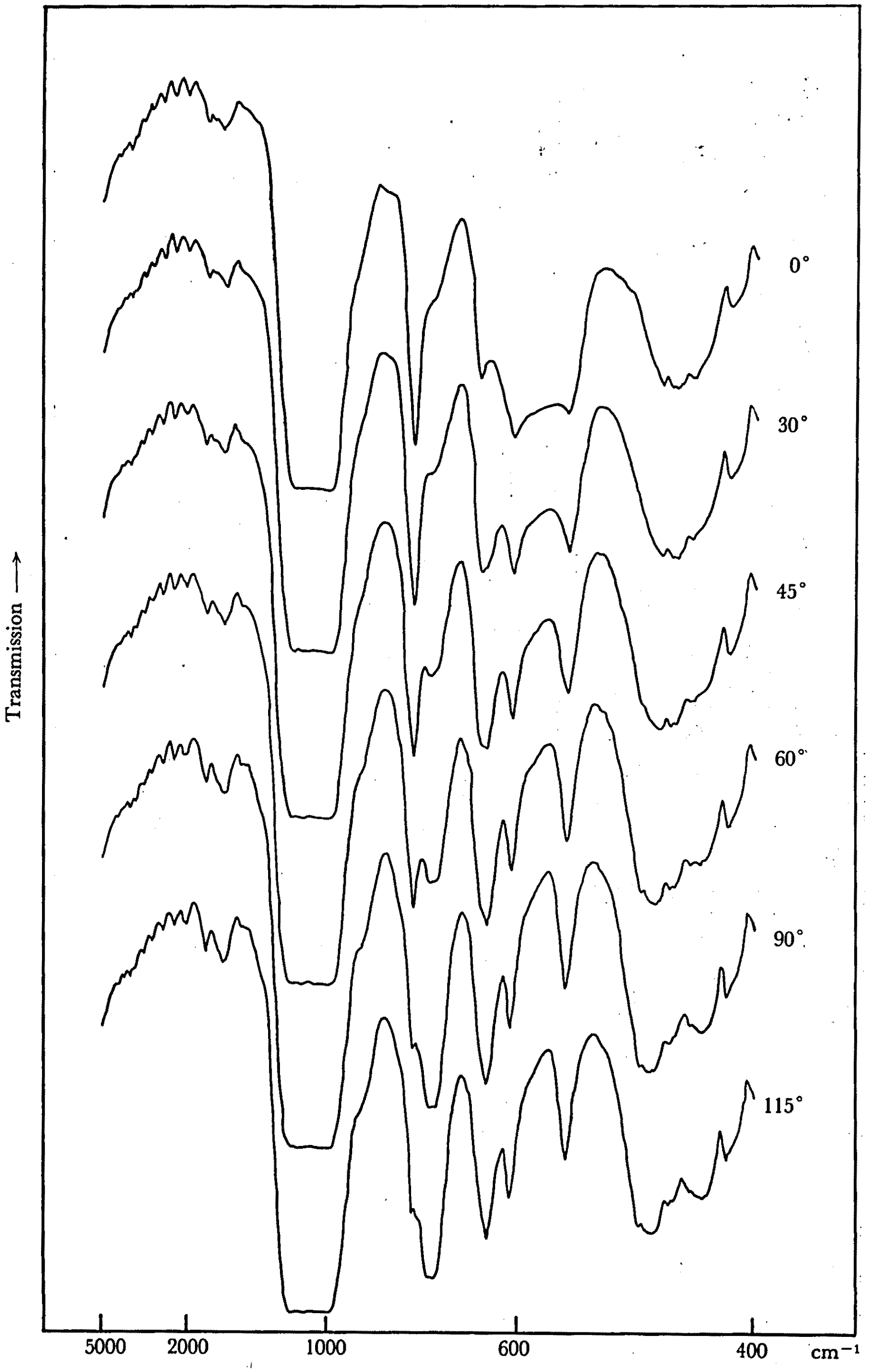

Fig. 4 Infrared absorption spectra along each direction on (010) with polarised radiation

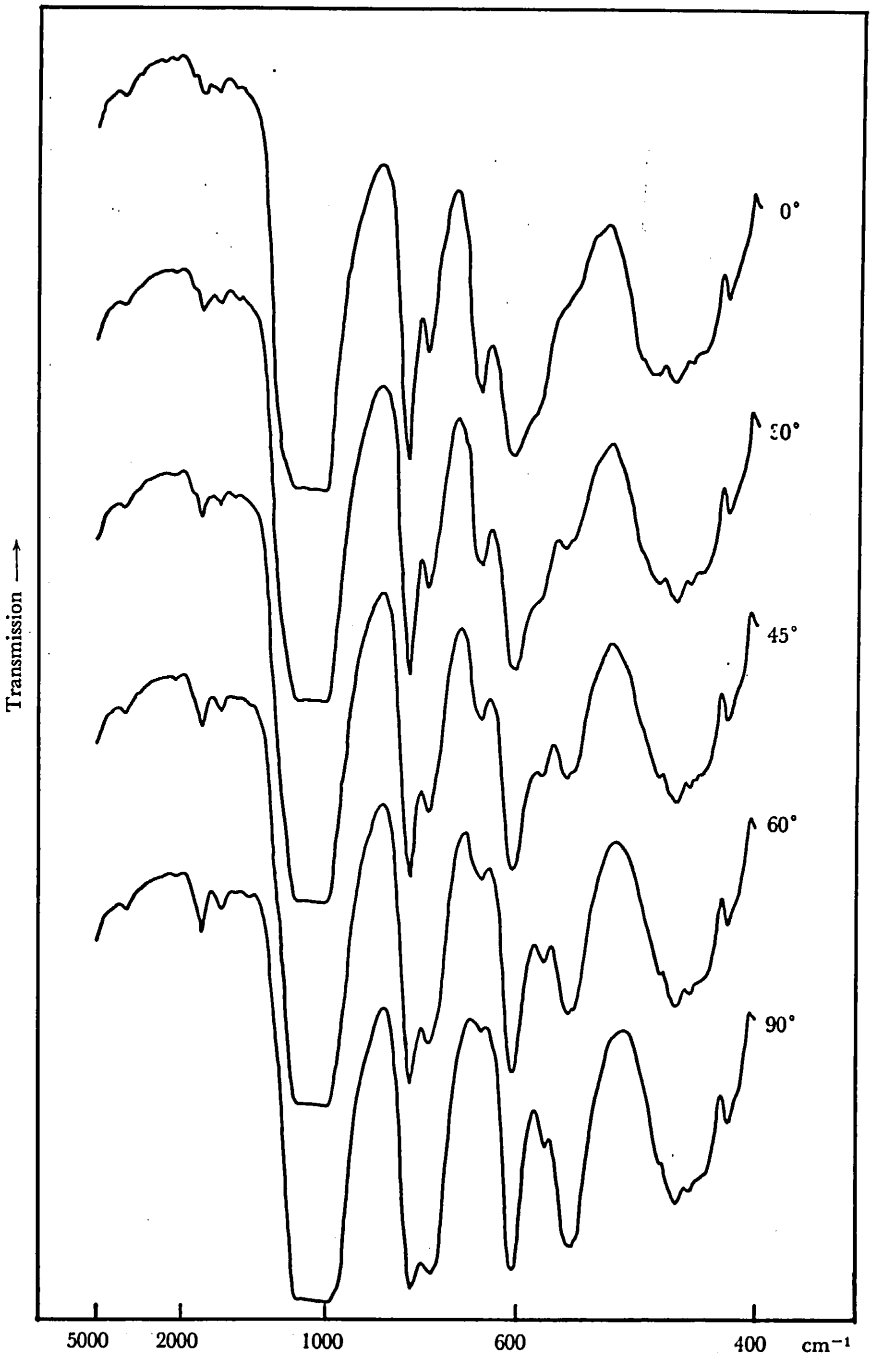

FIG. 5 Infrared absorption spectra along each direction on (100) with polarised radiation

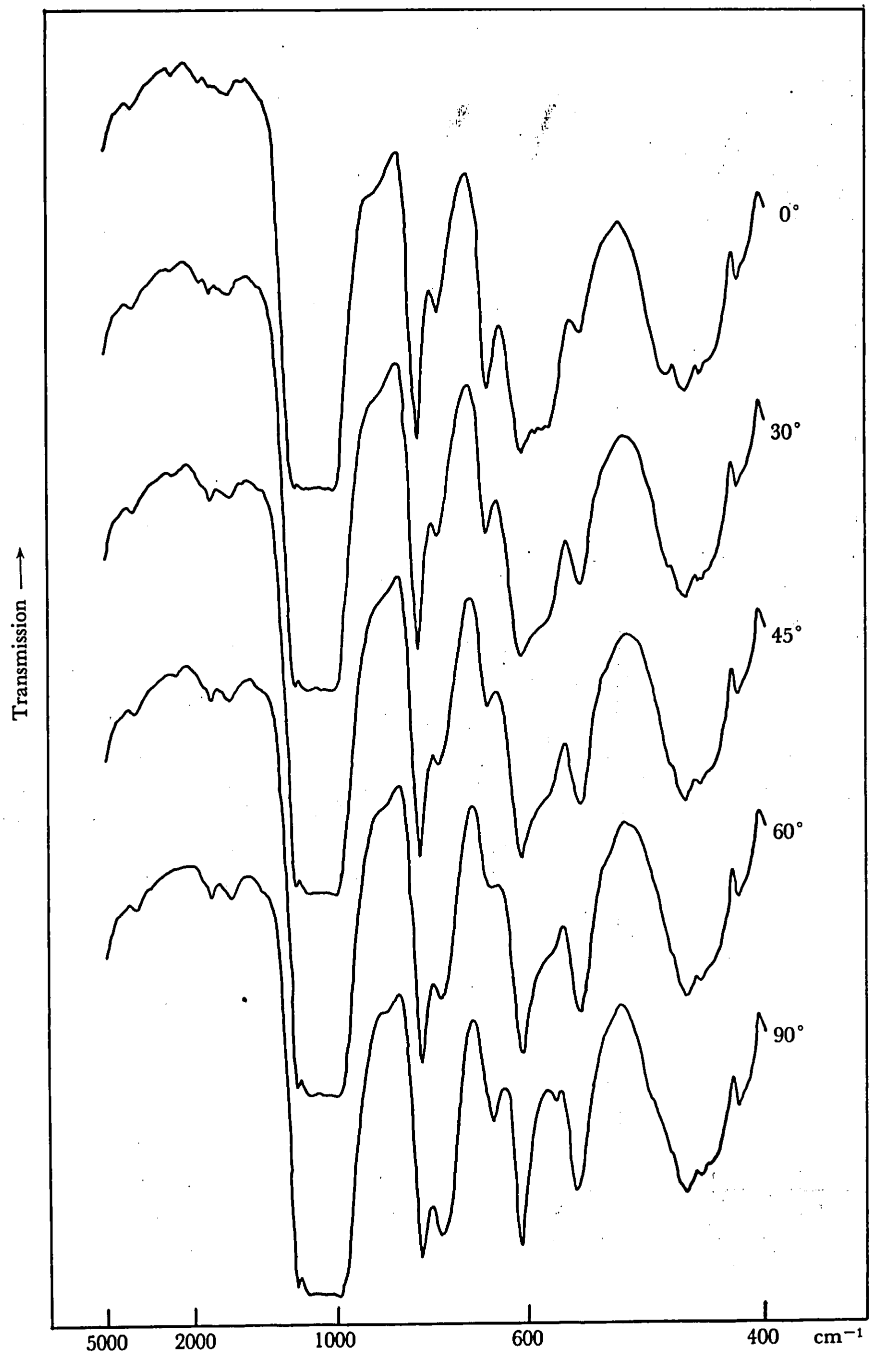

FIG. 6 Infrared absorption spectra along each direction on (110) with polarised radiation

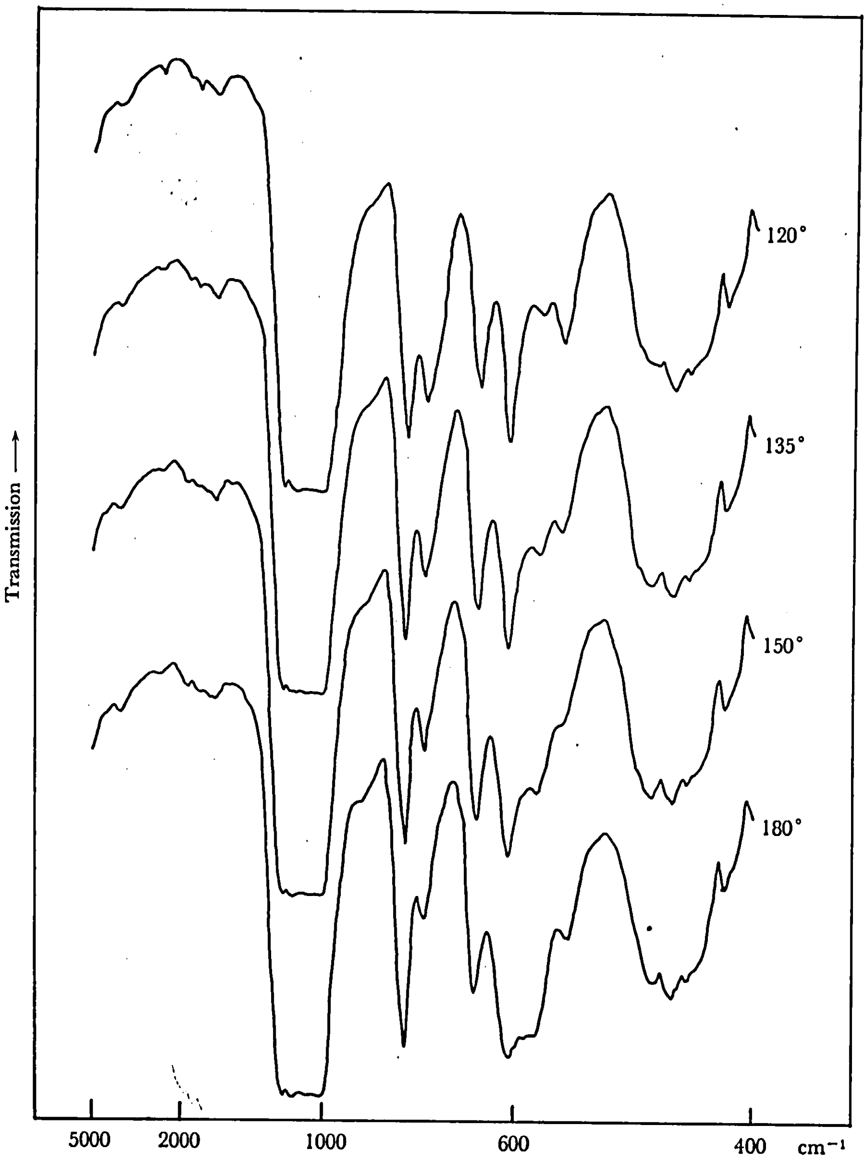

FIG. 6 (continued) Infrared absorption spectra along each direction on (110) with polarised radiation

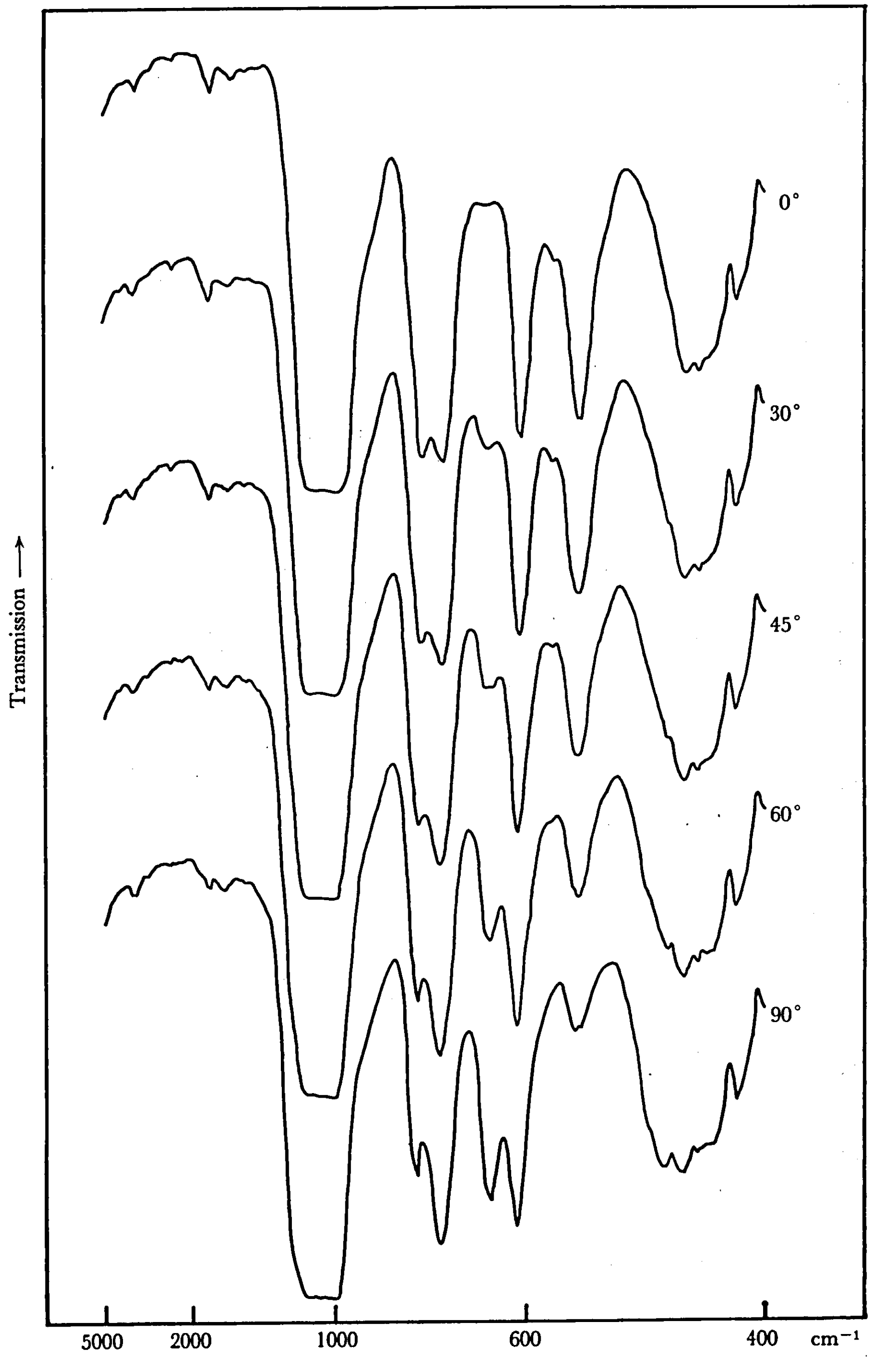

FIG. 7 Infrared absorption spectra along each direction on $(20\bar{1})$ with polarised radiation

TABLE 2. VARIATION OF INFRARED ABSORPTION FOR RESPECTIVE FREQUENCIES ON (001)

Wavenumber cm^{-1}	Transmission % for direction from b-axis				
	0°	30°	45°	60°	90°
778					
774					
768					
759	9	15			
741					
737					
735					
731					
727	6	8	8	8	8
658	50	43			
653			34	26	20
649					
646					
642					
613					
610	14	17	18	18	20
608					
606					
574					
570					
567					
565	56	50	50	55	61
546					
543					
541	22	32	41	47	53
538					
536					
476			37	31	26
469					
467					23
465				25	
462		31	28		
461	31				
455		28	27	27	27
450	26				
441	29	31	31	30	32
417	42	43	42	41	43

TABLE 3. VARIATION OF INFRARED ABSORPTION FOR RESPECTIVE FREQUENCIES ON (010)

Wavenumber cm^{-1}	Transmission % for direction from c-axis					
	0°	30°	45°	60°	90°	115°
778						
774	12	13	16	19	23	24
768						
759						
741	*	*	32	24	12	11
737						
735						
731						
727						
658	26					
653		20	18			
649						
646				15		
642					16	19
613						
610	14	20	23	27	28	27
608						
606						
574						
570						
567						
565						
546	19	24	28	32	36	35
543						
541						
538						
536						
476			26	22	20	21
469				20	19	
467						20
465			21			
462	24	23				
461						
455	24	23	22	22	24	25
450	24	23	23	23	26	26
441	26	27	27	27	28	28
417	40	39	38	35	34	35

TABLE 4. VARIATION OF INFRARED ABSORPTION FOR RESPECTIVE FREQUENCIES ON (100)

Wavenumber cm^{-1}	Transmission % for direction from ac-plane				
	0°	30°	45°	60°	90°
778					
774	11	8	8	7	7
768					
759					
741	32				
737		26			
735			21	15	
731					10
727					
658					
653		31			
649	24		40		57
646				48	
642					
613					
610					
608	12	10	10	10	10
606					
574	*	*	28		
570					
567				32	35
565					
546	*	34			
543					
541			28	22	15
538					
536					
476	31	33			
469					
467					
465	33				
462		27			
461			28	29	31
455		27	24	24	24
450	28		23	23	23
441	30	27	26	26	26
417	43	41	40	39	40

TABLE 5. VARIATION OF INFRARED ABSORPTION FOR RESPECTIVE FREQUENCIES ON (110)

Wavenumber	Transmission % for direction from c-axis								
cm^{-1}	0°	30°	45°	60°	90°	120°	135°	150°	180°
778	15	13					16	16	15
774			12	12	13	16			
768									
759									
741	40								41
737		36	30					33	
735				24		23	28		
731					18				
727					19				
658									
653	25	36	42	46					26
649					40	26	22	20	
646									
642									
613									
610									
608	12	12	12	13	15	15	14	13	
606									
574	*	*	*	*					*
570							33	25	
567					44	40			
565									
546						35	38	*	
543	36			21	26				36
541		26	22						
538									
536									
476					44	34	*	*	
469						31	27	25	
467									28
465	28								
462		30	*						
461				*	*				
455				26	27		26	25	25
450	24	24	23	24	24	25	24	24	24
441	31	28	28	26	28	29	28	28	28
417	47	46	44	44	43	42	42	43	46

TABLE 6. VARIATION OF INFRARED ABSORPTION FOR RESPECTIVE FREQUENCIES ON $(20\bar{1})$

Wavenumber cm^{-1}	Transmission % for direction from b-axis				
	0°	30°	45°	60°	90°
778				24	29
774		14	18		
768	11				
759					
741					
737					
735					15
731			11	13	
727	10	10			
658			46		
653	60				
649		53		35	24
646					
642					
613					19
610			17	18	
608	15	16			
606					
574					
570					
567	54	50	50	54	58
565					
546					
543	18	24	32	44	58
541					
538					
536					
476				*	*
469					
467					30
465					
462					
461		38	36	33	
455					
450	27	27	27	27	27
441	28	28	28	29	33
417	32	31	31	32	34

maximum at 90° (parallel to ac-plane) and are generally sharpened saving the broadened at 0° (parallel to b-axis) and 45°. The characteristic directions shown on the former two planes are considered of significance as a question for analysis of the structure.

4. 613～606 cm^{-1}

The spectra concerned are separated into those at 613, 610, 608 and 606cm^{-1}.

On (001) and (100), almost no variations are observable either in wavenumber or in intensity. On (010), those at 610cm^{-1} are not so much shifted but variable in intensity (in the range of about 13%) with a maximum at 0° (parallel to c-axis). On (110), those at 608cm^{-1} are nearly invariable. On (20$\bar{1}$), those, sharpened but covertible in wavenumber, show a little variation of intensity (in the range of about 4%) with a maximum at 0° (parallel to b-axis).

5. 574～565 cm^{-1}

The spectra concerned are subdivided into those at 574, 570, 567 and 565cm^{-1} and characterized with weakness in intensity in comparison with the others.

On (001), those at 565cm^{-1} are variable not in wavenumber but in intensity (in the range of about 11%). On (010), various figurations are recognizable. On (100), the related ones are, though intense, found splitted into those at 574cm^{-1} as shoulders and those, tiny but sharp, at 567cm^{-1}, indicating a maximum in intensity at 0° (parallel to ac-plane). On (110), those at 574cm^{-1} as shoulders as well as those at 567cm^{-1} and 570cm^{-1} as well-defined ones are confirmable. On (20$\bar{1}$), those at 567cm^{-1} are variable in intensity (in the range of about 8%), resembling those on (001).

6. 546～536 cm^{-1}

The spectra cnocerned are, though difficult to do with due to severe broadening, subdivided into those at 546, 543, 541, 538 and 536cm^{-1}.

On (001), their intensity is remarkable (in the range of about 31%) and, probably because of their difference, reversely proportional to sharpness of figurations, reaching a maximum at 0° (parallel to b-axis). On (010), the spectra at 546cm^{-1} are not fluctuated with respect to wavenumber but reveal variable intensity with a maximum at 0° (parallel to ac-plane). On (100), those bear a tendency shifting to the lower side of wavenumber and to a maximum intensity with approaching to the direction along b-axis. On (110), those indicate either shift to the higher side with increasing angle from c-axis excepting the special case at 180° or variation of intensity (in the range of about 15%) with a maximum at 60°.

7. 476 cm^{-1}

The spectra concerned are characteristically invariable aside the cases of their weakening along certain directions, deriving a difficulty in further inspection of the structural relation.

8. 469～461 cm^{-1}

The spectra concerned are branched into those at 469, 467, 465, 462 and 461cm^{-1}

and characterized with conspicuous variations in wavenumber.

On (001), those observed near 461cm^{-1} at 0° (parallel to b-axis) show increase in wavenumber with increasing angle, accompanying considerable variations of intensity. On (010), those, somewhat sharp at 0° (parallel to c-axis), exhibit more marked broadening and shift to the higher side of wavenumber with increasing angle in association with a little variation in intensity (in the range of about 5%), saving the case at 115° (parallel to ab-plane). On (110), those revealing broadening at 0° (parallel to ac-plane) convert into the simple one at 90° (parallel to b-axis) and display a little variation of intensity (in the range of about 5%) with a maximum at 30°. On (110), those are discernible only as shoulders in the region of lower wavenumber but as the well-defined at 469, 467 and 465cm^{-1} with slightly variable intensity (in the range of about 5%). On $(20\bar{1})$, those kept invariable at 461cm^{-1} become broader with approaching to 90° (parallel to ac-plane) but disappear at 0° (parallel to b-axis), associating variation in intensity (in the range of more than 8%).

9. 455～450 cm^{-1}, 441 cm^{-1} and 417 cm^{-1}

The absorption spectra related to these ranges are surely recognizable but variations to appear characteristically along respective directions on each plane are too ambiguous to be determined.

IV. CONSIDERATION

It has become clear that the infrared absorption behaviors in the cases with the pulverized specimens, with the oriented sections under ordinary radiation and with those along certain directions under polarised radiation are evidently different from one another. Presuming from the structure constructed of the complicated but definite alinements of various electric vectors resulted from several kinds of interatomic forces, no matter whether those may be related to the status of the main compositions or to that of the subordinates, the results obtained above seem to be natural, and it is believed that the data for this kind of experiments are by all means to be restricted to those derived from the orientational experiments, especially if their relations to the inner structure be pursued in detail.

Several representatives in wavenumber have, as referred to already by SAKSENA and some others, been assigned in connection with certain sorts of individual or simplified bondings comprised in relatively simple structures and with a number of minerals nearly without any notice on orientation. However, spacial correlation of the characteristic absorption spectra to all of the interatomic forces controling the whole structure is, though, mainly because of difficulty in preparing the idealized specimens in most cases, remained yet to be put into effect, believed indispensable for analysing the structure in question more concretely.

In such a sense, the present work is still now merely a preliminary one far from completion specifically on account of omitting the experiments within the range of

$1300\sim1000\text{cm}^{-1}$, possible in case with thin-plates of less thickness. Nevertheless, to be added is that a sort of conclusion concerning the respective vibrations on the thin-plates under consideration have been reached and will be reported after careful inspection for general orientations in relation to structural analysis.

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