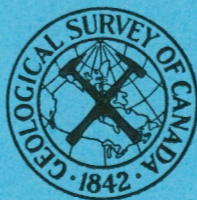


CANADA
DEPARTMENT OF MINES AND TECHNICAL SURVEYS

GEOLOGICAL SURVEY OF CANADA
TOPICAL REPORT NO. 87

INFRARED SPECTROSCOPY - A POSSIBLE
TOOL IN THE DETERMINATION OF
MINERAL CONTENTS IN ROCKS

SYDNEY ABBEY



OTTAWA
1964

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Infrared Spectroscopy
A Possible Tool in the Determination of
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Sydney Abbey

Introduction

Infrared absorption spectroscopy studies the variation in the proportion of infrared radiation which is absorbed or transmitted by substances as the wavelength or frequency of the radiation is varied, and attempts to correlate these variations with the molecular structure of the absorbing substances. Thus the infrared absorption spectrum is a characteristic property, or "fingerprint", of each chemical compound. Since the various absorption bands are related to specific inter-atomic vibrations within a molecule, the spectrum becomes more complex as the complexity of molecular structure increases. Infrared spectroscopy has been used for a number of years as a "fingerprinting" technique in the identification of organic compounds, and as a diagnostic technique for elucidating structure. The relatively recent applications to mineralogy and related fields have been concerned mainly with studies of changes of structure in those cases where other techniques have failed to provide the necessary information. A detailed bibliography in this field is that by Lyon (1962).

Qualitative analysis by means of infrared absorption is based on the same principle as that used in other spectral regions, i.e. the identification of substances by the wavelengths or frequencies of their characteristic absorption bands.

For quantitative analysis, use is made of Beer's Law, which states, in effect, that at a given wavelength, the concentration of absorbing material is proportional to the absorbance, i.e. the logarithm of the reciprocal of the transmittance. Transmittance is defined as the ratio of the radiant power transmitted by a sample to the radiant power incident on it, but in practice it is measured as the ratio of the radiant power transmitted by the sample to that transmitted in an unimpeded beam of equal initial radiant power. In most infrared spectrometers, transmittance is measured by means of a split-beam mechanism (see below).

Beer's Law is an idealized concept, which does not hold in many practical circumstances. Quantitative calculations are simplified where the Law applies; otherwise, calibration curves must be drawn. In analysing a binary mixture, the ideal situation involves strong absorption by each component at a wavelength where the other does not absorb. Such conditions are seldom attained in practice, and it is therefore necessary to assume additivity of absorbances, and to solve simultaneous equations or to apply corrections.

The infrared region, as used in most spectroscopic work, covers the wavelength range from 2 to 16 microns, with some applications requiring extension to about 40 microns. For a variety of reasons, it has become customary in recent years to use "frequencies" rather than wavelengths. Since there are 10,000 microns in a centimetre, a wavelength of one micron corresponds to a frequency of 10,000 "reciprocal centimetres" or " cm^{-1} ". Thus the wavelengths 2, 16 and 40 microns mentioned above, correspond to frequencies of 5,000, 625 and 250 cm^{-1} respectively.

Instrumentation

Infrared spectrometers consist of the same essential components as do those covering other spectral regions i.e. a source of radiation, a dispersing device and a device for measuring transmitted radiation, as well as the usual auxiliary optical and electrical devices.

The most common infrared "light" sources are the "Nernst Glower" and the "Globar". These are both resistance heating elements (the former consisting of oxides of zirconium, yttrium, thorium and related elements, the latter of silicon carbide) which emit useful radiation at temperatures under 2,000°K (Harrison et al, 1948). The major shortcoming of all such "blackbody" radiators is the fact that their maximum intensity lies in the range of one to three microns, while most useful infrared spectroscopic work is done at much higher wavelengths. The limited quantities of radiation available at useful wavelengths is a persistent problem.

Earlier infrared spectrometers invariably used prisms as dispersing devices. These were constructed of rock salt, potassium bromide, or similar materials, the material used being governed mainly by the wavelength range desired. Glass and quartz, being strong absorbers in most of the infrared range, are of little value. Because of difficulties in construction of lenses of suitable materials, focusing and other optical manipulations are generally done by means of mirrors. Recent instruments have shown a strong trend toward replacement of prisms by diffraction gratings, whose major advantage is the reduction in the quantity of radiation lost by absorption.

Thermocouples are the most common form of detectors used in the infrared, mainly because of the wide wavelength range over which they can be applied. Special detectors, such as the Golay Cell (Harrison et al, op. cit) are used in the far infrared.

Most modern infrared spectrometers operate on the double beam principle. Radiation from the source is split by a rotating half mirror into alternating pulses which follow different paths. The "analytical" beam passes through the sample and the monochromator to the detector. The "reference" beam follows essentially the same path, but misses the sample. The detector thus receives a set of alternating pulses of differing magnitudes, which generates an alternating current signal. This current actuates an attenuator (a comb or similar device) which reduces the light transmitted by the reference beam to the point where it is equal to that transmitted by the analytical beam. The movement of the attenuator drives the recording pen along the Y-axis, recording transmittance. Continuous movement of the monochromator varies the wavelength of radiation reaching the detector, and thus provides chart movement parallel to the X-axis. The result is a curve of transmittance vs wavelength.

Aside from the trend toward the use of gratings, improvements in infrared spectrometers have been toward increased sensitivity and spectral resolution, control of scanning speeds, provision for expansion of either axis in the plotting of the spectrum, extended wavelength range, etc. These trends have tended to increase the cost of the instruments. As a result, several manufacturers are now producing alternative "low-priced" instruments, which sacrifice some degree of versatility in order to simplify operation.

Baird-Atomic, Inc., Cambridge, Mass., produces a versatile instrument, Model NK-1, as well as a simpler one, Model NK-3. Beckman Instruments, Inc., Fullerton, California, offers a series of highly flexible instruments, including Models IR-4, IR-7 and IR-9. All of these are similar in design, but the improvements outlined above are evident in going from IR-4 to IR-9. Beckman also produces two more economical models, the IR-5A and IR-8.

The Perkin-Elmer Corporation, Norwalk, Connecticut, have produced another series of increasingly versatile instruments, all of one basic design, evolving from Model 21 through 221, 221-G and 421 to their very latest 521. They also make a lower priced line, including several different models under the name of "Infracord". Unicam Instruments Ltd., Cambridge, England, offer a highly versatile model, their SP-100, and a simpler instrument, the SP-200. Other manufacturers include Applied Physics Corp., Monrovia, California; Hilger and Watts Ltd., London, England; Grubb Parsons and Co. Ltd., Newcastle-upon-Tyne; and VEB Carl Zeiss, Jena, East Germany.

Sample Handling

In scanning the infrared spectra of solid materials, such as rocks and minerals, it is necessary to disperse the sample in a medium which is essentially transparent to infrared radiation. Although a variety of techniques have been used, the pressed halide disc is generally favoured. The sample must first be ground exceedingly fine. Tuddenham and Lyon (1960) suggest that particle size be smaller than the shortest wavelength to be scanned, if spurious readings are to be avoided. A few milligrams of sample are mixed with a 200 to 500-fold excess of a suitable alkali halide (generally potassium bromide), and pressed into discs of varying thickness in an evacuated die. Disc thicknesses are of the order of one millimetre.

History of the Project

In the autumn of 1960, Dr. J.A. Maxwell visited the Kennecott Research Centre, in Salt Lake City, Utah, where a great deal of work was underway in infrared spectroscopy, both as a tool

in mineralogical research and as a method for mineralogical analysis of rocks. While many of their results appeared highly interesting, the tone of some of their publications suggested that they might have been carried away by their own enthusiasm, e.g. "Quantitative Mineralogy in 30 Minutes" (Lyon, Tuddenham and Thompson, 1959); "Infrared Analysis Quick and Easy" (Tuddenham and Zimmerley, 1960).

In the months that followed, exchange of samples and information between Dr. Maxwell and Dr. J.B. Stephens of Kennecott Research Centre, suggested a number of useful possibilities for infrared in the work of the Geological Survey of Canada, but there was little indication of its usefulness in mineralogical analysis of rocks. Samples of pure minerals and mixtures of them, corresponding to some ultrabasic rocks, were prepared by Dr. Maxwell and Dr. C.H. Smith, and were examined in the sales laboratory of one of the manufacturers of infrared spectrometers. Results were inconclusive, largely because the samples were not sufficiently finely ground.

In the spring of 1962, the writer was asked to undertake further work on the infrared project. Accordingly, visits were made to the Fuels Division of the Mines Branch, the Food and Drug Laboratories of the Department of National Health and Welfare, the Plant Products Laboratories of the Department of Agriculture, and both the Pure and Applied Chemistry Divisions of the National Research Council. Instruments seen included the Beckman IR-7, and the Perkin-Elmer 21, 221-G and 421. Useful discussions were held with infrared spectroscopists in all of those laboratories, and also with Mr. (now Dr.) E.A. Schiller, now our Resident Geologist at Yellowknife, who has had considerable experience in the use of infrared in his graduate studies at the University of Utah.

In August, 1962, the writer attended a five-day course on Infrared Spectroscopy Technique, given in the Department of Chemistry, Massachusetts Institute of Technology, Cambridge, Massachusetts. Lectures, delivered by Dr. R.C. Lord and Dr. Dana Mayo, of Massachusetts Institute of Technology, by Dr. Foil A. Miller, of Mellon Institute, and by Dr. E.R. Lippincott, of the University of Maryland, covered the following topics: (a) introduction to infrared instrumentation; (b) optics of infrared spectrometers (two lectures); (c) quantitative analysis (two lectures); (d) factors influencing the performance of infrared spectrophotometers; (e) infrared spectrophotometry (two lectures); (f) sample handling techniques; (g) miscellaneous methods of infrared spectroscopy. Laboratory sessions included experiments on: (1) basic optics of infrared prism spectrometers; (2) infrared spectroscopy with the Perkin-Elmer Model 421 Spectrophotometer; (3) sample-handling techniques; (4) quantitative analysis with the Beckman IR-4 Spectrophotometer; (5) high-resolution and far infrared spectroscopy. In addition to the instruments mentioned, a Baird Model NK-1 was on display, and a number of specialized instruments were in use in the laboratories.

During the course, the writer discussed our problems with Dr. Miller and with representatives of Beckman and Unicam. Afterwards, the Baird and Perkin-Elmer plants were visited, and further discussions held. All expressed interest in trying to analyse samples for us.

Collaborative Tests

Subsequent discussions were held with Mr. Schiller with a view to finding a combination of minerals which would not only be of interest in our work, but which would be amenable to infrared

analysis. Schiller suggested varying proportions of quartz, plagioclase, microcline and muscovite, to give compositions characteristic of granite, quartz monzonite and granodiorite.

Mr. H.R. Steacy provided the pure minerals. Dr. G. Pouliot did a mineralogical examination of the specimens.

The quartz used was a very pure material from Lyndhurst Township, Ontario. The plagioclase was an albite (var. cleavelandite), from Lacorne Township, Quebec, of composition An_2 . The microcline, from Derry Township, Quebec, was the purest available, but mineralogical examination revealed the presence of about 10 per cent albite. The muscovite, from Pied-des-Monts, Quebec, was reported as "very pure".

In addition to the synthetic mixtures of the pure minerals, an actual rock sample was also included. Supplied by Dr. K.R. Dawson under his number DB-560, this rock had both modal and normative analysis readily available. The plagioclase in the rock was reported as having a composition of An_{12} .

It was decided first to reduce the pure minerals and the rock to a fineness of -150 mesh. This was accomplished by repeated 15 minute treatment in a "mixer mill" with steel balls, with screening between treatments. For the muscovite, grinding was facilitated by a preliminary dry treatment in a Waring Blendor at 15,000 r.p.m.

Noticeable iron contamination resulted from this treatment, particularly in the quartz. It was decided to ignore this effect, since the presence of iron would have less influence on the infrared readings than would possible side-effects of attempts to remove it.

Each of the -150 mesh materials was then "homogenized" by tumbling in a half filled vial for one hour, at 60 r.p.m. about an axis at 45 degrees to the axis of the vial.

Portions of the four pure minerals were then weighed into three clean vials, as given in Table I.

Table I

Weights of Pure Minerals Taken for Synthetic Mixtures

<u>Mineral</u>	<u>Mix A</u>	<u>Mix B</u>	<u>Mix C</u>
Quartz, gm.	1.00	0.75	0.50
Plagioclase, gm.	1.00	1.75	2.50
Microcline, gm.	2.50	1.75	1.00
Muscovite, gm.	<u>0.50</u>	<u>0.75</u>	<u>1.00</u>
Total, gm.	<u>5.00</u>	<u>5.00</u>	<u>5.00</u>

After applying a correction for the albite content of the microcline, percentage compositions were calculated for the three mixtures. These are given in Table II, along with the modal and normative data for the rock sample, now designated as "D". All data was rounded to the nearest whole per cent.

The components of each mixture were mixed by tumbling, as before.

Table II

Percentage Compositions of Mixtures and Rock

<u>Mineral</u>	<u>Mix A</u>	<u>Mix B</u>	<u>Mix C</u>	<u>Rock D</u>	
				<u>mode</u>	<u>norm</u>
Quartz	20	15	10	33	34
Plagioclase	25	38	52	35	40
Microcline	45	32	18	29	22
Muscovite	<u>10</u>	<u>15</u>	<u>20</u>	<u>1</u>	<u>0</u>
	<u>100</u>	<u>100</u>	<u>100</u>	<u>98</u>	<u>96</u>

In an effort to reduce particle sizes to the order recommended by Tuddenham and Lyon (1960), 500 milligrams of each mineral, mixture and the rock were ground in turn for 30 minutes in a mullite mortar in a Fisher automatic grinder, in the presence of 9 ml of absolute ethanol. The alcohol evaporated in the course of the grinding, and the resulting finely ground samples were brushed into small vials and again tumbled to ensure homogenization.

Microscopic examination of the finely ground materials by Mr. Schiller indicated the presence of some particles (particularly of the feldspars) which were considerably larger than the 2 to 5 micron size recommended by Tuddenham and Lyon (1960).

100 milligram portions of the finely ground minerals, mixtures and rocks were sent to Baird-Atomic, Inc., Beckman Instruments, Inc., The Perkin-Elmer Corporation and Unicam Instruments Ltd. It was suggested that 2.5 mg of each sample be mixed with 1,000 mg of KBr, and that a 505 mg and a 175 mg disc be prepared. (The two sizes of disc facilitate the choice of absorption bands.) It was requested that as wide a variety of instruments and wavelength ranges as possible be used in recording the spectra, and that attempts be made to determine the mineralogical composition of the three mixtures and the rock.

Results

The samples were forwarded to the collaborating laboratories in October, 1962. Results trickled in over a period of nearly a year. The names of the firms are given in alphabetical order above. They are not identified in the following paragraphs, but are merely described by a code number, in order of increasing quality of results.

Company I

This manufacturer did not acknowledge receipt of the samples, and reported no results. This disappointment was surprising, in view of their earlier interest in the project.

Company II

This firm sent us spectra of our samples, run on both their most flexible instrument and on a more economical model. Unfortunately, they used a KBr /sample ratio of 100/1 instead of the 400/1 which we had requested. Resulting absorbances were excessively high, and it was therefore impossible to resolve some of the useful bands.

These difficulties were pointed out to the company, and they then prepared a second set of spectra, using the correct sample-to-halide ratio. When attempts were made to calculate compositions from these spectra, it was found that their more economical instrument was insufficiently sensitive to show the 2.75 micron ($3,640 \text{ cm.}^{-1}$) band, diagnostic of muscovite, in a sample containing 10 per cent of that mineral. The spectra from their more expensive instrument did not cover that wavelength range at all. They pointed out, with some justification, that the band in question "... arose from unbonded -OH groups and would therefore not be specific for a given mineral ...". However, muscovite was the only one of the minerals present that showed this band, and no other suitable band could be found. Further, although the second set of spectra from this firm were prepared with the correct KBr/ sample ratio, they were not run on discs of differing thickness.

Although no results could therefore be calculated from the spectra received from Company II, they do deserve credit for

their courtesy in providing them.

Company III

This company was the first to reply, and provided some very interesting results. They were so enthusiastic about the work that they asked, by telephone, for permission to publish some of the data. At that point, the correct values were en route to them by mail. Presumably the errors they then discovered were sufficient to keep them from asking, in writing, for permission to publish.

They prepared KBr discs according to our instructions, and examined them on three different instruments. For the final calculations, they used spectra recorded on two of their simpler instruments, set in different wavelength ranges. They assumed (not entirely correctly) that each of the four minerals was "100 per cent pure". They did not assume that the four components must add up to 100 per cent in each mixture.

For muscovite, they used a band which was free from interferences and made their calculations on the basis of Beer's Law.

For quartz, they first made a visual estimation of the sum of plagioclase and microcline, and used this estimate as a correction for interference in a strong absorption band of quartz. After the correction, Beer's Law was again applied directly. Subsequently, they found that the supposed interference of the two feldspars was actually caused by polystyrene contamination from the vials used in mixing the samples with potassium bromide.

For the two feldspars, they first applied corrections for interference of quartz and muscovite in the band used for plagioclase. They then used simultaneous equations based on readings at two wavelengths to determine the two feldspars.

All of these calculations were made on the assumption that Beer's Law was valid throughout. This assumption was probably based on a comparison of absorbances of the two sizes of discs for each sample. Results are listed in Table III.

Table III

Values Reported by Company III

<u>Wavelength microns</u>	<u>Mineral</u>	<u>Mix A</u>		<u>Mix B</u>		<u>Mix C</u>		<u>Rock D</u>		
		<u>Taken</u>	<u>Found</u>	<u>Taken</u>	<u>Found</u>	<u>Taken</u>	<u>Found</u>	<u>Mode</u>	<u>Norm</u>	<u>Found</u>
12.5	Quartz, pct.	20	24	15	21	10	12	33	34	48
13.4	Plagioclase, pct.	25	17	38	37	52	56	35	40	27
30.4	Microcline, pct.	45	51	32	29	18	10	29	22	22
2.76	Muscovite, pct.	10	7	15	16	20	22	1	0	0
		<u>100</u>	<u>99</u>	<u>100</u>	<u>103</u>	<u>100</u>	<u>100</u>	<u>98</u>	<u>96</u>	<u>97</u>

Visual examination of the infrared spectrum of the pure microcline and the pure albite did not suggest the presence of 10 per cent of the latter as an impurity in the former. However, even if the microcline were actually 100 per cent pure, there would be little overall improvement in the results provided by Company III. It was noticed that the muscovite results, which were the best in general, could be further improved by a slight refinement in the technique of taking measurements from the recorded spectra. The objection raised by Company II regarding the band used to determine muscovite must also be borne in mind. However, in the absence of other water-bearing minerals, the error due to variable water content of muscovite would not be too serious, if the muscovite content of the rock were sufficiently low.

More serious are the relatively large errors in quartz determination, particularly in Rock D - the entire analysis of the rock being of little value. On the basis of its constant composition, one would expect better results for quartz than for the highly variable feldspars. The large errors in the results for Rock D might be blamed on differences in composition of the component feldspars of the rock as compared to those in the synthetic mixtures. However, such differences would have a relatively small effect on the quartz determination. Differential grinding of the components of Rock D, resulting in a disparity of particle sizes in the individual mineral components, was suggested by Company III as another possible source of error.

Company IV

This company provided the best results of all, but their report took longer than the others to reach us. They prepared potassium bromide discs according to our instructions, and recorded all spectra on their most versatile instrument.

They first recorded spectra of the pure minerals, the synthetic mixtures and the rock, to obtain an estimate of the ranges of composition to be expected in the samples. Although two wavelength ranges were used, suitable bands were found for all of the components in the 2 to 16 micron range.

For the quantitative work, they prepared synthetic standard mixtures of the pure minerals, covering the concentration ranges estimated in the samples. Duplicate short-range scans were then run over each useable band on each sample and "standard". Calibration curves of absorbance vs weight of component were plotted. The quartz calibration curve showed full adherence to Beer's Law, the muscovite curve showed slight deviation, but the curves of both feldspars showed wide deviation. They suggested that this deviation may have been due to refractive index changes with concentration, a difficulty which might be eliminated by using some other disk matrix, e.g. potassium

chloride in place of potassium bromide.

Results obtained by means of the calibration curves are listed in Table IV.

Table IV
Values Reported by Company IV

<u>Wavelength</u> <u>microns</u>	<u>Mineral</u>	<u>Mix A</u>		<u>Mix B</u>		<u>Mix C</u>		<u>Rock D</u>		
		<u>Taken</u>	<u>Found</u>	<u>Taken</u>	<u>Found</u>	<u>Taken</u>	<u>Found</u>	<u>Mode</u>	<u>Norm</u>	<u>Found</u>
14.4	Quartz, pct.	20	20	15	18	10	12	33	34	45
13.4	Plagioclase, pct.	25	25	38	37	52	53	35	40	29
13.7-X	Microcline, pct.	45	45	32	33	18	17	29	22	34
2.76	Muscovite, pct.	10	10	15	15	20	21	1	0	0
		<u>100</u>	<u>100</u>	<u>100</u>	<u>103</u>	<u>100</u>	<u>103</u>	<u>98</u>	<u>96</u>	<u>108</u>

X - actually used to calculate microcline plus plagioclase

Discussion

While the results obtained by Company IV on the synthetic mixtures were very good, there is much in common between their results and those of Company III, i.e. quartz values show the most striking errors, and both firms obtained rather poor results on Rock D. Possible reasons for these poor results have already been discussed under Company III. Most difficult to explain are the errors in the quartz values, particularly since the two firms used different wavelengths, thus diminishing the likelihood that interference from other components might have caused the high results.

Lyon, Tuddenham and Thompson (1959), in a similar study on the mineralogical analysis of the U.S. Geological Survey Standard

Granite G-1 (Chayes, 1951), also obtained higher results for quartz than expected from previously accepted values. In an attempt at proving the validity of their results, they prepared two synthetic mixtures from pure minerals - one representing the composition they had found by infrared, the other the "accepted" values (from modal analysis). A comparison of the infrared spectra of the two mixtures with that of G-1 tended to confirm the values obtained by Lyon, Tuddenham and Thompson, thereby raising some doubts about the validity of the modal analysis.

With our Rock D, there was a discrepancy between both the mode and the norm, on the one hand, and both sets of infrared results, on the other. Further, the two sets of infrared results were not in close agreement with one another. However, in view of the good agreement between the infrared values from Company IV and the known compositions of the three synthetic mixtures, the question now arose whether the modes or norms are valid standards for judging the quality of infrared results. The three synthetic mixtures were then analysed chemically, using our established "rapid methods". Calculation of the norms from the results was unfortunately complicated by the presence of abnormally high muscovite contents, which were reflected as high water contents. In addition to the norm calculations, a "modified norm" was therefore calculated as follows: since Rock D is known to be almost free of muscovite, its water content was taken as "residual", and applied as a correction to the water found in the three synthetic mixtures. The remainder of the water in each mixture was then calculated as muscovite, whose theoretical potassium content was then subtracted from the total potassium found, before calculating microcline. Tables V, VI and VII compare the values found by the two sets of calculations with the actual mineral contents and the infrared results.

Table V

Norms vs Mineral Contents, Mix A

<u>Mineral</u>	<u>Present</u>	<u>Norm</u>	<u>Modified Norm</u>	<u>IR Results</u>	
				<u>Company III</u>	<u>Company IV</u>
Quartz, pct.	20	20	21	24	20
Plagioclase, pct.	25	32	31	17	25
Microcline, pct.	45	44	36	51	45
Muscovite, pct.	10	-	11	7	10

Table VI

Norms vs Mineral Contents, Mix B

<u>Mineral</u>	<u>Present</u>	<u>Norm</u>	<u>Modified Norm</u>	<u>IR Results</u>	
				<u>Company III</u>	<u>Company IV</u>
Quartz, pct.	15	14	15	21	18
Plagioclase, pct.	38	45	42	37	37
Microcline, pct.	32	36	25	29	33
Muscovite, pct.	15	-	15	16	15

Table VII

Norms vs Mineral Contents, Mix C

<u>Mineral</u>	<u>Present</u>	<u>Norm</u>	<u>Modified Norm</u>	<u>IR Results</u>	
				<u>Company III</u>	<u>Company IV</u>
Quartz, pct.	10	10	11	12	12
Plagioclase, pct.	52	57	54	56	53
Microcline, pct.	18	26	12	10	17
Muscovite, pct.	20	-	20	22	21

These results confirm that, under certain conditions at least, infrared analysis can provide a truer picture of the composition than can normative calculations. Similarly, the work of Lyon, Tuddenham and Thompson suggested that the same may be true as between infrared and modal analysis. X-ray diffraction is another possible technique in this field, but there is not sufficient data available at this time for a valid comparison. It would appear reasonable to conclude that infrared spectroscopy has distinct potentialities in the field of mineralogical analysis, but that much further exploratory work must be done before the full scope of the method can be evaluated.

Conclusions

The superiority of the results provided by Company IV is not necessarily an indication of superior instrumentation, but more likely the result of the greater time which they devoted to the project, and their refined technique. However, Company IV did use their most versatile instrument. For exploratory development work such as we might undertake, it would appear advisable to purchase an instrument which offers the widest possible wavelength range, the highest spectral resolution and the most flexible optical and electronic controls. Should the use of infrared ever become routine, it might then be possible to use a second instrument, of a simpler type, at least for some applications.

The poor results obtained on Sample D indicate some of the lines of investigation that might be followed in undertaking a study of infrared in our work. At least three possible sources of error could be examined: (a) the effect of variability in composition of component feldspars; (b) the effect of other minerals present in minor concentrations; (c) the effect of possible differential grinding in reducing a natural mineral mixture to a very fine particle size.

To study these problems, it would first be necessary to measure variations in spectra with changes of composition in a mineral series, such as the plagioclases. Spectra of minerals separated from various rocks would require examination, by themselves, in combination with others (both major and minor), etc. An interesting experiment would involve comparison of the spectrum of a rock itself with that of a "reconstituted" rock, synthesized from "pure" minerals separated from the rock, under various grinding conditions.

Clearly, much research would be needed, and the services of a competent person required, one with a good background in chemistry, spectroscopy, mineralogy and petrology. As suggested once before, this problem would be suitable for a post-doctoral fellow.

Development work would be necessary for each new rock type introduced, but once such work is completed, routine operations could probably be simplified and expedited. Although "Quantitative Mineralogy in 30 Minutes" (Lyon, Tuddenham and Thompson, 1959) would probably not be attained, at least something of that order of magnitude might be approached. At this stage, there is insufficient data in the literature to estimate how speed, accuracy and applicability of this technique would compare with other techniques. For example, we do not now know which mineral pairs, if any, cannot be distinguished from one another.

It is the writer's considered opinion that infrared spectroscopy should be regarded primarily as a mineralogical research tool, and as a supplement to existing techniques. This data accumulated in such work would be of great value in eventual analytical applications.

To sum up, any decision on potential usefulness of infrared spectroscopy in the work of the Geological Survey of Canada should be based on the understanding that this technique is not new and not revolutionary, but that it has distinct possibilities as an adjunct

to existing techniques, and much additional work will be needed before useful results are forthcoming.

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