

RAPID MODAL ANALYSIS OF AN AMPHIBOLITE  
BY CALIBRATED X-RAY DIFFRACTION PATTERNS

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BY CALIBRATED X-RAY DIFFRACTION PATTERNS

By

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## ABSTRACT

A coarse grained amphibolite from the metamorphosed rim of the Whitestone Anorthosite was prepared in several ways for the purpose of determining the modal abundance of the constituent minerals by calibrated X-ray diffraction. A simple two component amphibolite consisting of plagioclase and amphibole was chosen and five major methods of mounting the specimens for X-ray diffraction were employed.

It was hoped that a method could be found which would produce randomly oriented, homogeneous samples. A series of such samples each of a different component ratio would then provide a calibration curve from which the mode of a whole rock specimen could be estimated.

The calibrated X-ray charts were produced on Philips scanning X-ray diffractometers.

The inconsistencies in the results indicate that better technical procedures are required.

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## INTRODUCTION

The purpose of this study was to attempt to produce a fast, standardized method of modal analysis, by the use of calibrated X-ray diffraction patterns, which could be applied to metamorphic rocks.

The method has been applied with some success to a series of fine grained felsic rocks by B. D. Tatlock (1966). However it has not been successfully applied to rocks which contain a considerable proportion of lath-like minerals, such as micas, and amphiboles.

The availability of some relatively simple procedure, which could be applied to numerous samples, providing significantly accurate modal analyses of the rocks, would be of great value to petrologists.

The production of series of standard intensity ratio vs modal abundance plots for a mineralogically related series of rocks may allow petrologists to measure trends in suites of rocks. The chemical analyses using X-ray fluorescence, the method commonly used today, are limited somewhat because they are often used to predict the mineral assemblage which would be produced in situations of chemical equilibrium. This may be valid in crystallizing magmas but is less accurate in metamorphic studies and nearly impossible for use with sedimentary rocks.

The method of analysis by X-ray diffraction, however, would produce a mode which would be equally valid when applied to igneous, metamorphic, or sedimentary rocks because it is an accurate reflection of the mineralogical content of the rocks.



The method would represent an improvement in accuracy over modes produced by point counting. The point counting method is dependent on small two dimensional sections of the rocks for estimation of the mineral abundances. If, however, there is a variation in the specimen which is larger than the thin section one could produce a biased mode using a point count. This type of variation is common in many rock types.

The use of X-ray diffraction patterns for producing modes would eliminate many of the difficulties involved in point count produced modes. By analyzing bulk samples rather than two dimensional sections the effects of textural variations are eliminated. Large samples can be analyzed by crushing a whole specimen or group of specimens and mixing them.

By splitting this sample a number of times until a 1 gram portion is reached, an average sample of the unit could be obtained, thus eliminating the textural variations too coarse to be seen in a thin section. Much human error involved in point counting is also eliminated. Minerals which are easily confused such as quartz and oligoclase are objectively analysed thus there is no decision making.

Thus the availability of a procedure which would produce a large number of accurate modal analyses of a particular rock or suite of rocks would allow the petrologist to analyse accurately mineralogical trends. This expanded study would simplify the task of unravelling a genetic picture of the rocks.

One medium grained amphibolite from the metamorphic rim of the Whitestone Anorthosite was donated by R. Mummery. This particular sample was chosen primarily because it contained amphibole and

plagioclase and very little else. The two component system made it convenient to produce standardized graphs of mineral abundance vs diffracted intensity.

Two series of samples containing unknown proportions of plagioclase and amphibole were produced and various methods of mounting and recording were employed to try to produce standardized graphs. A Phillips diffractometer was used to produce most of the calibrated X-ray patterns.

## PREVIOUS WORK

It has been known for many years that the intensity of a diffracted beam is proportional to the surface area which is producing the particular peak. (Klug and Alexander, 1954, p. 292) This surface area is in turn dependent upon the modal proportions of the diffracting material. (Klug and Alexander, 1954, p. 292) It is also well known that each mineral produces its own particular pattern. And thus every rock will also have its own distinctive pattern.

With this in mind various authors, Tatlock (1966) with some felsic rocks, Brindley and Kurtossy (1961) using kaolinite and a matrix, Carroll (1971) using clay minerals, and others, have produced analytical procedures for measuring the modal abundance of crystalline materials in mixed samples.

They give some indication of the difficulties which can arise in producing standard curves representing the true modes in the sample system.

Beyond the mechanical and analytical accuracy of the methods, problems arise due to the physical properties of the system. When comparing the diffraction patterns of mixed systems it is important to be sure that all component fractions have been reduced to nearly identical size. The importance of this can be seen as the finer fractions will show more reproducible intensity measurements which are due to reduction in extinction. (Klug and Alexander, 1954, p. 295) This effect will be increased for crystals which show a preferred orientation. In such cases the intensity of the extinction factor will be greatly affected

by the orientation of the crystals in the sample mount.

The mass absorption factor will cause variations in the intensity of diffraction produced by any one mineral. If the mineral has a high absorption coefficient Tatlock states that "when a mixture contains both a weak and a strong absorber, peaks of the weakly absorbing component appear weaker and those of the stronger absorbing component stronger." (Tatlock, 1966, p. 11) In the plagioclase amphibole system observed in this study the plagioclase is the weaker absorber of the two. Absorption variations should be corrected by using the method of Klug and Alexander (1954, pp. 411-415).

The problem of high background intensity due to iron fluorescence has been solved where the iron content is reasonably low by the use of filters or monochromators in the diffractometer. Point counting done on a thin section of the amphibolite indicated that the iron oxide content was less than one percent. X-ray fluorescence work done on similar samples by R. Mummery indicated an average  $\text{FeO}$  and  $\text{Fe}_2\text{O}_3$  content of about 13%.

## ANALYTICAL PROCEDURE

### A. BASIC ASPECTS

It is extremely important that in the "diffraction analysis of whole-rock powders from automatically recorded patterns that all steps in sample preparation and instrumentation be as nearly standardized as possible" (Tatlock, 1966, p.3).

The various factors which will influence the intensity of an X-ray diffraction peak must be integrated into any study of crystalline material. This is especially important when quantitative results are required.

The instrument must be set up for optimum resolution and intensity for the measurement of modal abundance. The separation of peaks is as important as the well defined peak height in the quantitative analysis. These parameters are controlled by the radiating slit width, the receiving slit width and the area irradiated by the beam. In the study here a  $1^\circ$  irradiating slit and a  $1^\circ$  receiver slit were used. An area of sample larger than the incident beam was used. At the scanning speeds of  $1^\circ 2\theta$  per minute and  $\frac{1}{4}^\circ 2\theta$  per minute used, the amphibole and plagioclase both produced well resolved peaks.

The use of a geiger-counter to produce a calibrated chart measuring intensity of the diffracted beam with respect to angular position (degrees  $2\theta$ ) makes intensity comparisons relatively simple. However, when quantitative results are required the fluctuation in current exciting the X-ray tube may be an important factor. Both the vertical and the

horizontal diffractometers incorporate a high degree of stabilization in the X-ray generator and receiving circuits by the use of special regulating components. The voltage in the circuit was 30 KV. and the current was 15 ma.

The time constant and scale factor were left at 8 and 1. The multiplicity was set at 2 or 4 depending on which analytical method was being used. For any particular series of tests the time constant remained unchanged.

Copper K $\alpha$  radiation filtered through a graphite monochromator was used to produce the calibrated charts.

Thus the variations in instrumentation have been minimized. Standard methods of preparing the specimens are described in the next section.

## B. SAMPLE PREPARATION

The samples were prepared for mounting as follows:

1. A slab was cut from the specimen and reduced in size using a diamond mortar and pestle.
2. The grains produced were then sieved between 50 and 100 mesh sieves and then washed down with acetone to remove any dust.
3. The sample was then checked to see that the most grains contained only one mineral using a binocular microscope. Then a series of runs through the Frantz magnetic separator were made to produce relatively pure plagioclase and amphibole fractions. Each fraction was again run through the separator and the small separate was thrown away. In this way any composite fragments were removed. The separate fractions were checked again for purity using the binocular microscope.
4. Ten samples containing various amounts of plagioclase and amphibole were then carefully weighed into sample bottles and numbered TC4 to TC13, each sample weighing about one gram.
5. The specimens were then powdered to less than 200 mesh size in an agate mortar containing acetone.

A second series of samples TC14 to TC23 were produced in the same fashion except that in Step 5 the sample was carefully ground for three minutes and then carefully sieved through a 200 mesh sieve. This was repeated until all of the sample passed through the sieve.

In this way it was hoped that a more homogeneous sample could be obtained. See Table 1 for data.

The modal weight percent of plagioclase and amphibole was produced by assuming the sample has no other components.

TABLE 1  
SAMPLE WEIGHTS AND MODAL ABUNDANCES

Sample No.	Wt Plag (g)	Wt Amp (g)	X <sub>P</sub>	X <sub>A</sub>
4	0.05625	0.05425	0.78	0.22
5	0.04772	0.01753	0.90	0.10
6	0.01510	0.03855	0.57	0.43
8	0.01168	0.05527	0.42	0.58
9	0.08044	0.05323	0.84	0.16
10	0.07262	0.04606	0.64	0.37
11	0.01594	0.11408	0.32	0.68
12	0.04471	0.03142	0.83	0.17
13	0.02019	0.07851	0.47	0.53
14	0.09937	0.22782	0.60	0.40
15	0.08428	0.14161	0.68	0.32
16	0.17808	0.09444	0.87	0.13
17	0.12737	0.14509	0.75	0.25
18	0.05396	0.25434	0.42	0.58
19	0.17664	0.07440	0.89	0.11
20	0.14631	0.02126	0.96	0.04
21	0.10455	0.08055	0.82	0.18
22	0.08890	0.19789	0.16	0.39
23	0.02674	0.30905	0.23	0.77



Thus

$$X_P = \frac{N_P}{N_P + N_A}$$

$$\text{if } N_P + N_A = N_{\text{TOTAL}}$$

where N is the number of moles of each component.

Similarly

$$X_A = \frac{N_A}{N_{\text{TOTAL}}}$$

A series of ten such specimens can be produced in about fifteen hours if complete separation could be made with the Frantz separator. The process becomes longer if separations must be made using heavy liquids in a centrifuge.

### C. ANALYTICAL METHODS

The analyses were performed on a Philips vertically scanning X-ray diffractometer except for the few done on the rotating mount. These were done on a horizontal scanning Philips diffractometer with a rotating sample mount at the same settings as the vertical diffractometer in the previous methods.

Powders of the pure amphibole, and plagioclase as well as a whole rock sample were scanned from about  $10^\circ 2\theta$  to about  $50^\circ 2\theta$ . From these runs it was determined which peaks would be best for further study. As suggested by Tatlock (1966) the best plagioclase peaks available when using Cu K $\alpha$  radiation are composite peaks at about 3.20 A ( $27.7^\circ 2\theta$ ) and 3.25 A ( $27.9^\circ 2\theta$ ). Since muscovite has a peak near this a correction must be applied to the plagioclase value. However, in the sample used the lack of muscovite precludes the need for any alterations. The best amphibole peak occurs at 8.50 A ( $10.4^\circ 2\theta$ ).

R. Mummery who has analysed the minerals in the whole rock indicates that the amphibole is pargasite and the plagioclase oligoclase. The values of  $I/I_0$  for the peaks chosen for the plagioclase are 100 for the 3.20 A peak and 55 for the 3.25 A peak and the hkl values given are 204 and 220 planes respectively. For the amphibole the  $I/I_0$  value is 70, and the hkl values given are 110.

These peaks are chosen because they are relatively strong and are not interfered with or badly distorted by neighbouring reflections arising from the plagioclase amphibole mixtures. See whole rock tracing in Figure 1.

To try to obtain reproducible results a series of mounting methods

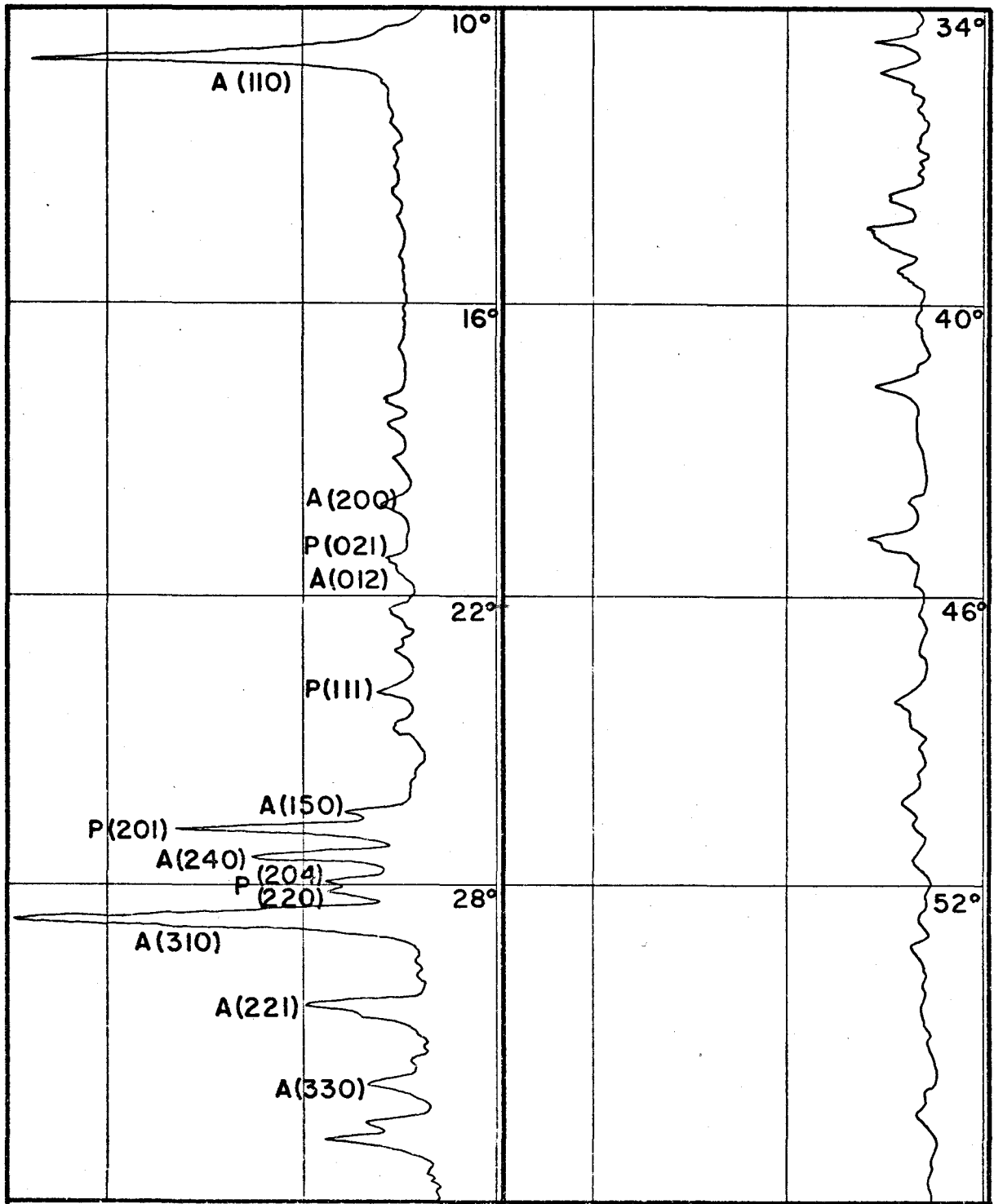


FIGURE I. / WHOLE ROCK TRACE

was used and various scanning speeds and count rates were tried for each method. These methods are outlined below.

### 1. Nail Polish Slurry Method

A small amount of each of the specimens TC1 to TC13, was mounted in a slurry using one drop of nail polish; thus a circular area of sample about the size of a nickel was produced. Each specimen was then scanned at  $1/4^\circ$  20 per minute with the rate constant/multiplicity/time constant at 8/2/1. A pair of scans from about  $10^\circ$  20 to  $12^\circ$  20 and from about  $27^\circ$  20 to  $29^\circ$  20 was made for each sample. Some samples were repeated by irradiating different areas of the sample as in specimen TC4. Others were remixed with additional nail polish and then rerun as seen in specimens TC8, TC11, TC13. The results are in Table 2 and sample traces of TC4 and TC8 are seen in Figures 2 and 3.

One of these samples can easily be produced from the 100 mesh material in 10 minutes and a trace made in about the same time.

### 2. Vaseline Method

For increased randomness a thin film of vaseline was applied to the surface of a number of slides and the sample was sprinkled over this surface. The slide was then shaken to dislodge any loose particles. These loose particles were again sprinkled over the surface. The procedure was repeated until all of the sample has adhered to the vaseline. The samples were then scanned at an increased count, 8/4/1. This increased multiplicity was necessary to produce significant peaks.

Specimens for this method take slightly longer to produce than in the previous method as repeated application was needed.

### 3. Rotating Mount Method

Aluminum discs were made to fit the rotating mount. These discs

TABLE 2  
INTENSITY AND WEIGHT RATIO STATISTICS

Sample	$X_P$	$X_A$	$I/X_P$	$I/I_P$	Area of Plagioclase Peak (sq."")	Area of Amphibole Peak (Sq."")
TC4	0.78	0.22	1.282	.308	3.395	1.030
TC4	0.78	0.22	1.282	.311	3.170	0.985
TC5	0.90	0.10	1.111	.253	2.390	0.605
TC6	0.57	0.43	1.754	2.430	0.615	1.495
TC8	0.42	0.58	2.381	3.333	0.495	1.650
TC8	0.42	0.58	2.381	1.594	1.035	1.640
TC9	0.84	0.16	1.190	.509	1.540	0.785
TC10	0.63	0.37	1.190	1.270	0.945	1.200
TC11	0.32	0.68	3.125	9.340	0.235	2.195
TC11	0.32	0.68	3.125	7.984	0.310	2.475
TC12	0.83	0.17	1.205	.739	1.880	1.390
TC13	0.47	0.53	2.128	4.674	0.430	2.010
TC13	0.47	0.53	2.128	2.383	0.600	1.430
TC14				0.433	1.000	2.305

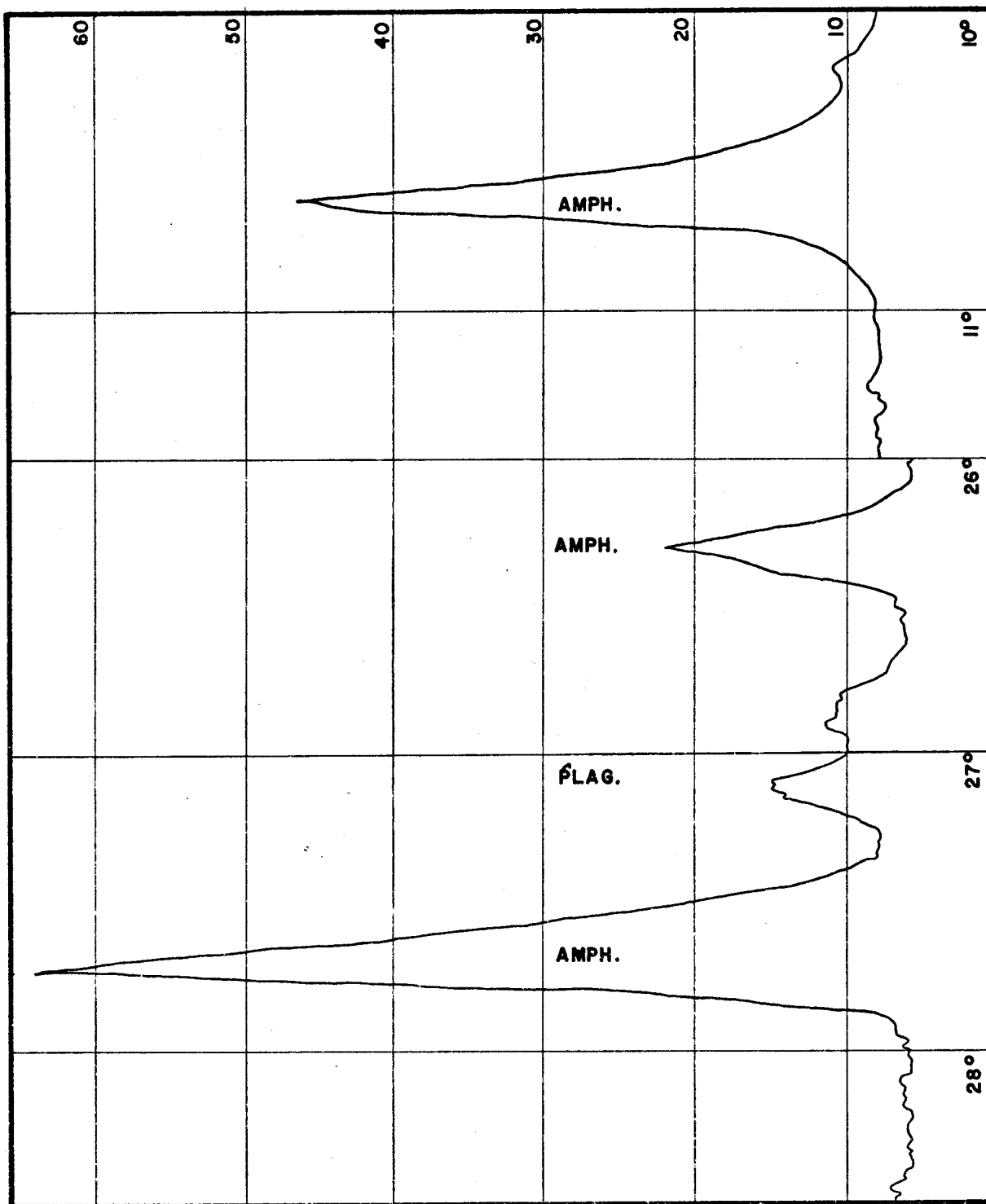


FIGURE 2 / TRACE OF SPECIMEN TC 4

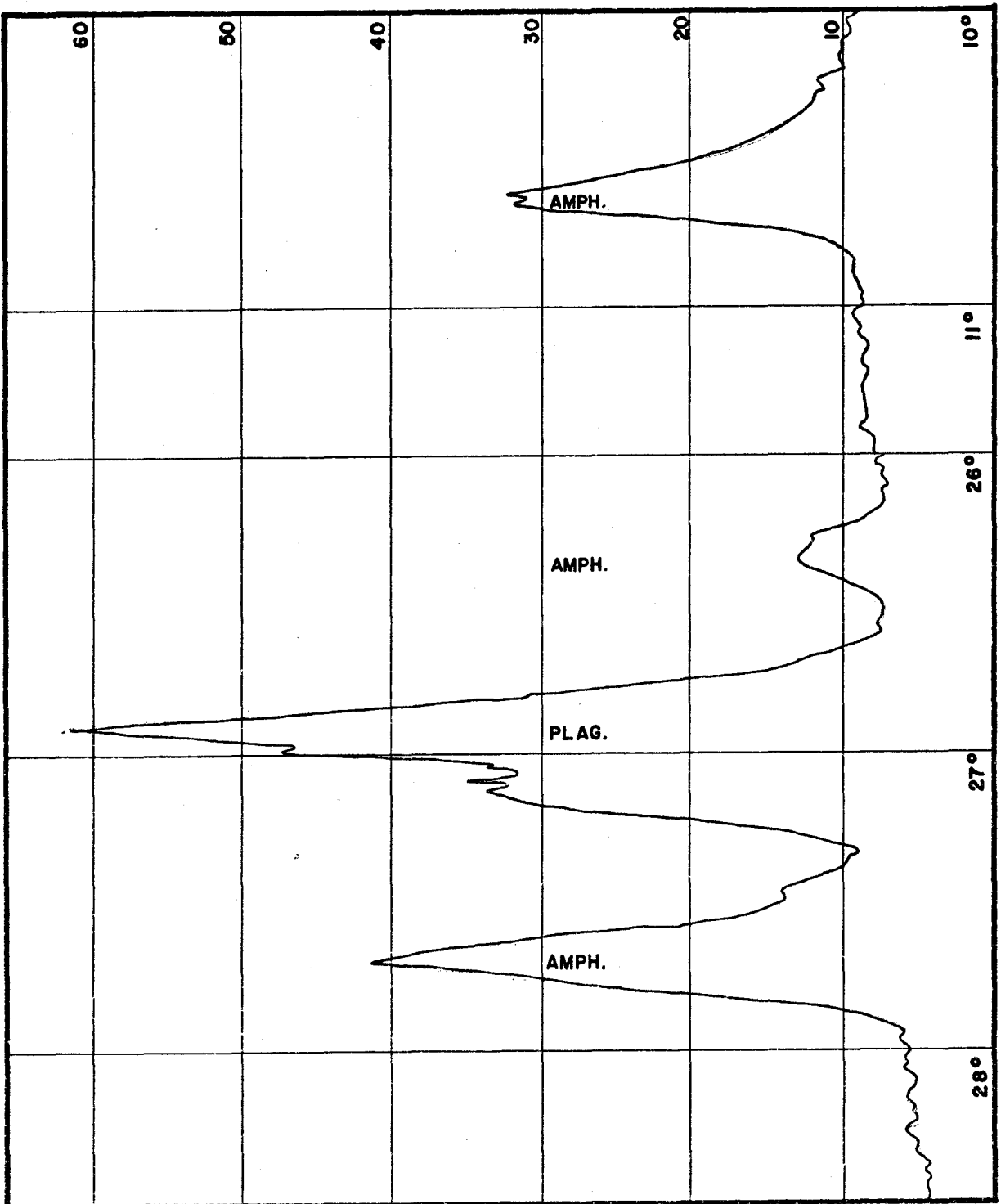


FIGURE 3 / TRACE OF SPECIMEN TC 8

were turned down on a metal lathe to a diameter of about 1/100" smaller than the bore of the rotating mount. Each disc was 3/16" thick. The tight fit in the sample holder keeps the specimen in the same place during rotation. This is very important as the intensity of the reflected beam would vary if the specimen surface was to vary from the plane.

Slurry samples of TC9 and TC11 were produced on the discs as in Method 1. These were scanned while rotating in a Philips horizontal diffractometer. The samples were re-mixed and then rerun to test for reliability.

The time involved in producing the mounted samples here is equal to that in method 1, about 10 minutes.

#### 4. Powder Pack Method

A method of packing the powder in a 1/2" diameter cylinder about 1/8" deep was used. One face of the cylinder had a glass slide placed over it and the powder was tamped in from the back as in McCreery's method (1949). Onto the open face of the cylinder a slip cover was positioned. Then the specimen and slip cover were inverted onto a greased slide. Thus, upon removal of the face slide, the sample could be rotated without disturbing the packing. A series of runs were made on the amphibole peak for TC14 and TC15.

The careful production of large quantities of powder required for this method involves about 30 to 40 minutes per sample.

#### 5. Suspended Mounts

The purpose of these procedures was to produce a specimen in which the grains were not oriented with respect to cleavage faces of the crystals.

The purpose in mounting a specimen in a medium is to provide a



uniform sample for X-raying. This is accomplished by choosing a medium which when ground produces a homogeneous, randomly oriented sample. Within any one of the particles the minerals may be oriented but as long as the shape of the medium is controlling the position and it produces a random sample, then a random orientation of the minerals will occur. See Figure 4.

A portion of specimen TC13 was mounted in Canada Balsam by melting a small chip and adding the sample slowly until a viscous lump had formed. When the mass had hardened it could not be powdered as it stuck together like toffee.

A second set of samples TC16 and TC19 were mounted by the same procedure in epoxy and allowed to harden. The epoxy pellets produced were ground in a diamond mortar with great difficulty. There was some sticking together of the fine material but it was possible to produce a less than 200 mesh sample with repeated grinding. The coolant acetone when used here caused the grains to mat as in the balsam method.

This method was too slow and tedious to be of any value as about two hours of grinding were required to produce one sample.

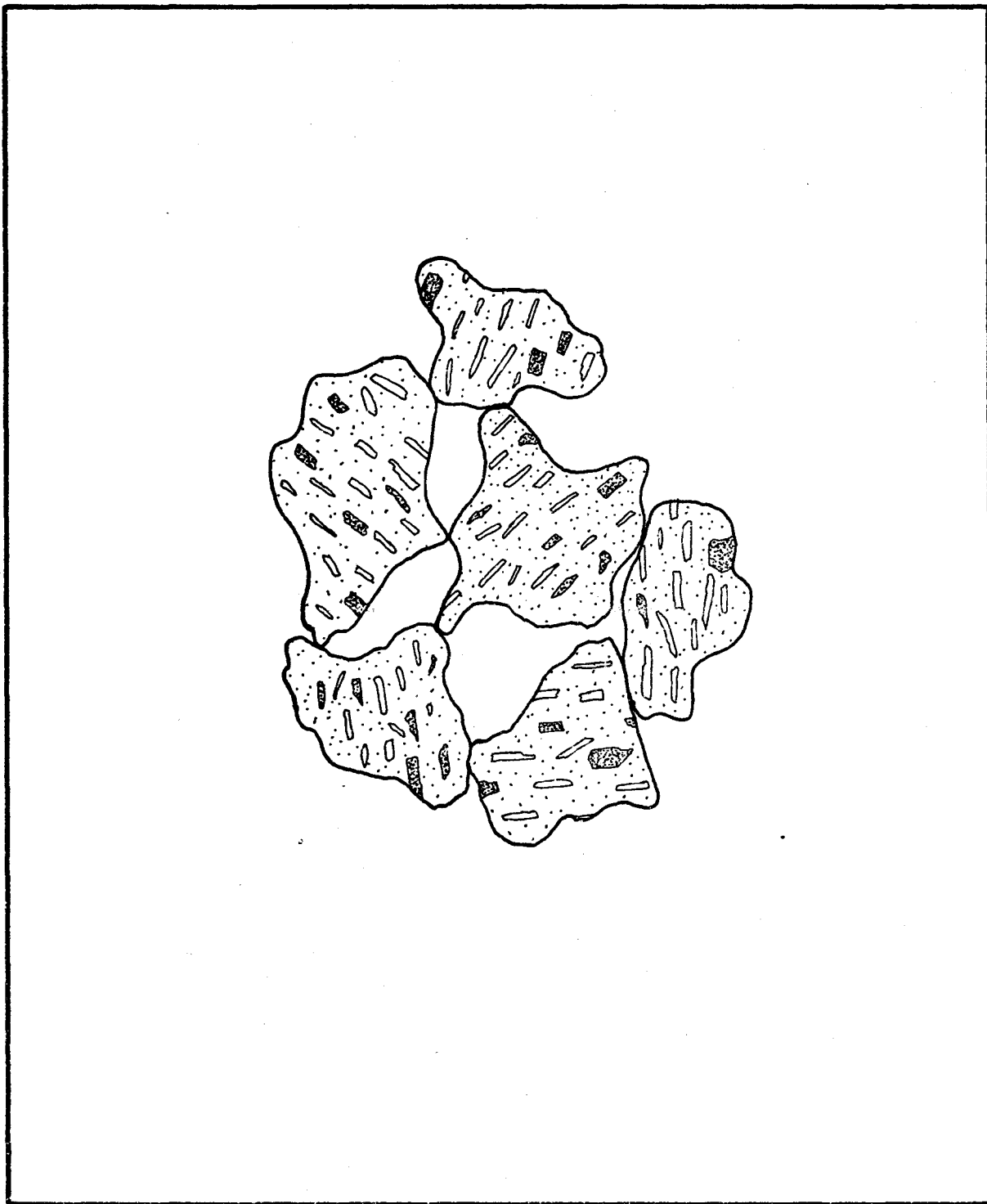


FIGURE 4 / EMBEDDED CRYSTAL SAMPLE

## ANALYSIS AND DISCUSSION

In the majority of studies (for example, Tatlock, 1966) the variation in intensity has been taken as the peak height. Although peak height was used as a rough estimate in some cases, it was felt that producing a base line due to background and measuring the area enclosed by the peaks would increase the accuracy of the method.

It was also suggested that a ratio of intensities, when plotted against the weight ratio of components, would produce more consistent results. The advantage involved in using peak height (or area) ratios is that within one run some of the electronic variations discussed under Basic Aspects of the Analytical Methods will vary. Such things as the divergence of the X-ray beam, the area irradiated, the current supplied to the geiger-counter and others can fluctuate. The measured intensity for any two peaks produced by scanning the same area of a sample twice may vary. The relative intensities of  $I_P/I_A$  however, should remain the same. Thus the problem of constructing universal calibration curves from the measured intensities  $I_Y$  for modal compositions of various  $X_Y$ 's where Y is the component, can be solved by using ratios of intensity and weight. Here the method has become internally calibrated and therefore does not require the factors which determine  $I_Y$  for a particular  $X_Y$  to be constant.

Plots of the area of amphibole/area of plagioclase ( $I_A/I_P$ ) against  $1/X_A$ , where

$$X_A = \frac{N_A}{N_A + N_P}$$

are shown in Figure 5. Also plots of  $I_A/I_P$  against  $X_A/X_{TOTAL}$  may be produced.

It can be shown that the plot in Figure 5 should be a straight line. Where there are only two components:

$$I_A = C_A \cdot X_A$$

$$I_P = C_P(1 - X_A)$$

where  $C_A$  is a proportionality constant for the amphibole,  $C_P$  for the plagioclase.

Thus

$$\begin{aligned} \frac{I_A}{I_P} &= \frac{C_A}{C_P} \frac{X_A}{1 - X_A} \\ &= B \frac{X_A}{1 - X_A} \\ &= B \frac{1 - X_P}{X_P} \\ &= B \frac{1}{X_P} - B \quad 0 < X_A < 1. \end{aligned}$$

From the graph it can be seen that the intensity intercept is  $-B$  and  $B - \tan \alpha$ , where  $\alpha$  is the angle the plotted line makes with the  $X_P$  axis.

Thus theoretically, from this graph the modal abundance of any mixture could be readily found if the ratio of intensities was known.

Also plots of intensity against weight percent of a particular component are frequently used (Klug and Alexander, 1954).

The results obtained from each analytical method and a discussion of each follows:

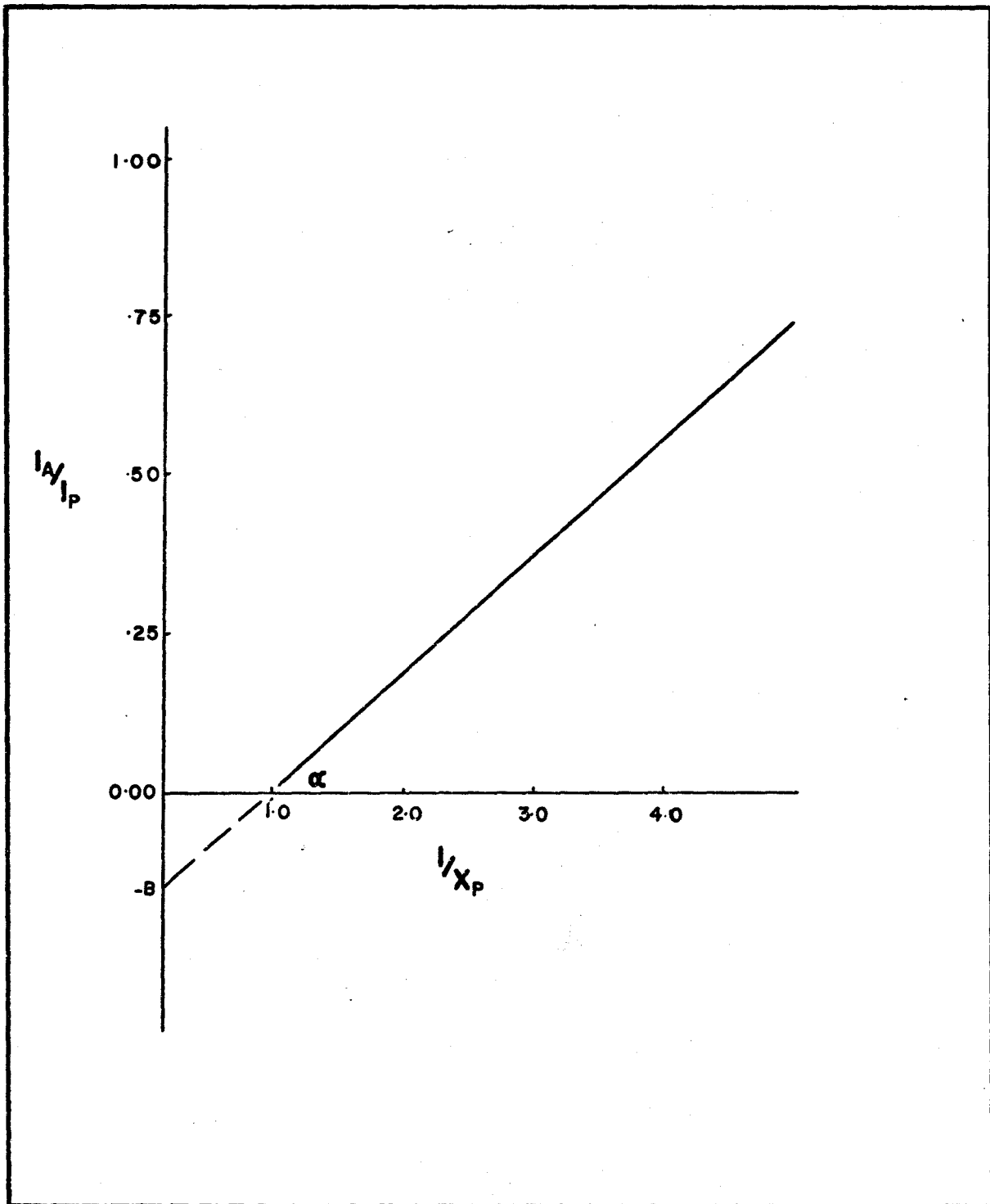


FIGURE 5  $I_A/I_P$  INTENSITY RATIO VS WT. % RATIO

## 1. THE NAIL POLISH SLURRY METHOD

The resulting intensities from each weight ratio are found in Table 2.

The resulting graph (Figure 6) of  $I_A/I_P$  against  $I/X_P$  does not indicate a good linear pattern. Variations within a single sample such as TC9 show that large variations could be produced within each sample. Subsequent runs on these samples indicated that the reproducibility was low. Thus it is felt that the samples are not randomly oriented.

In Figures 7 and 8 the weight percent of each component is plotted against the intensity. In the amphibole plot a linear relation is apparent. But the standard deviation for predicting the weight percent of amphibole is 8.2% as seen in Figure 8. In the plagioclase plot, Figure 7, the correlation between the intensity and abundance is poorer than that for amphibole. This may indicate the nonrandomness is more dependent upon the plagioclase than the amphibole.

The result of the individual plots suggests that the reason the ratio method plotted in Figure 6 fails is that the large errors become multiplicative in ratios, thus indicating that a high degree of precision would be required to use this method.

Thus the problem encountered is in the lack of reproducibility in the sample and thus reliability in the result is low. The following methods are attempts to reproducibly produce randomly oriented homogeneous samples.

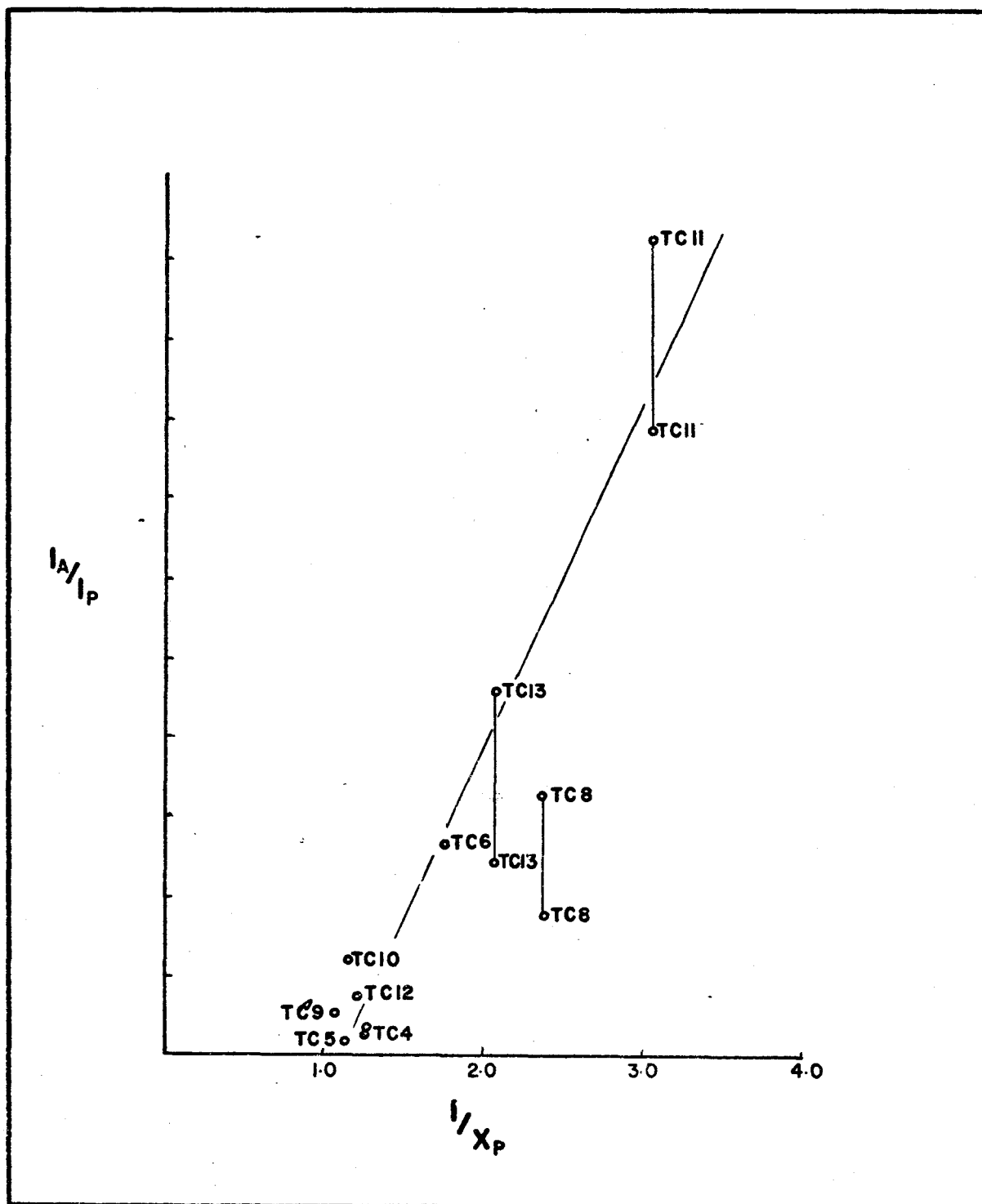


FIGURE 6 / EXPERIMENTAL INTENSITY RATIO VS WT. RATIO

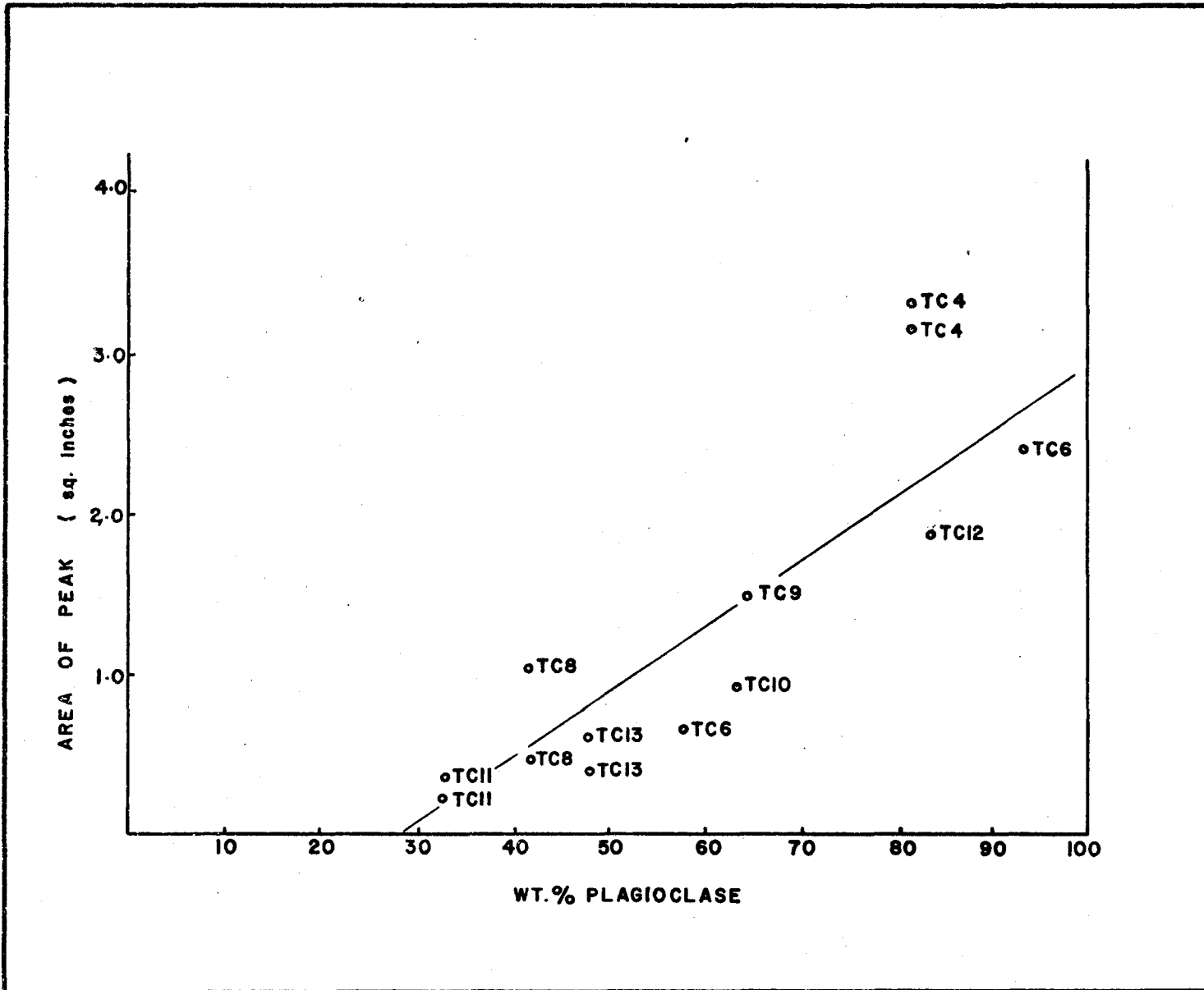


FIGURE 7 / WT.% PLAGIOCLASE VS PEAK AREA



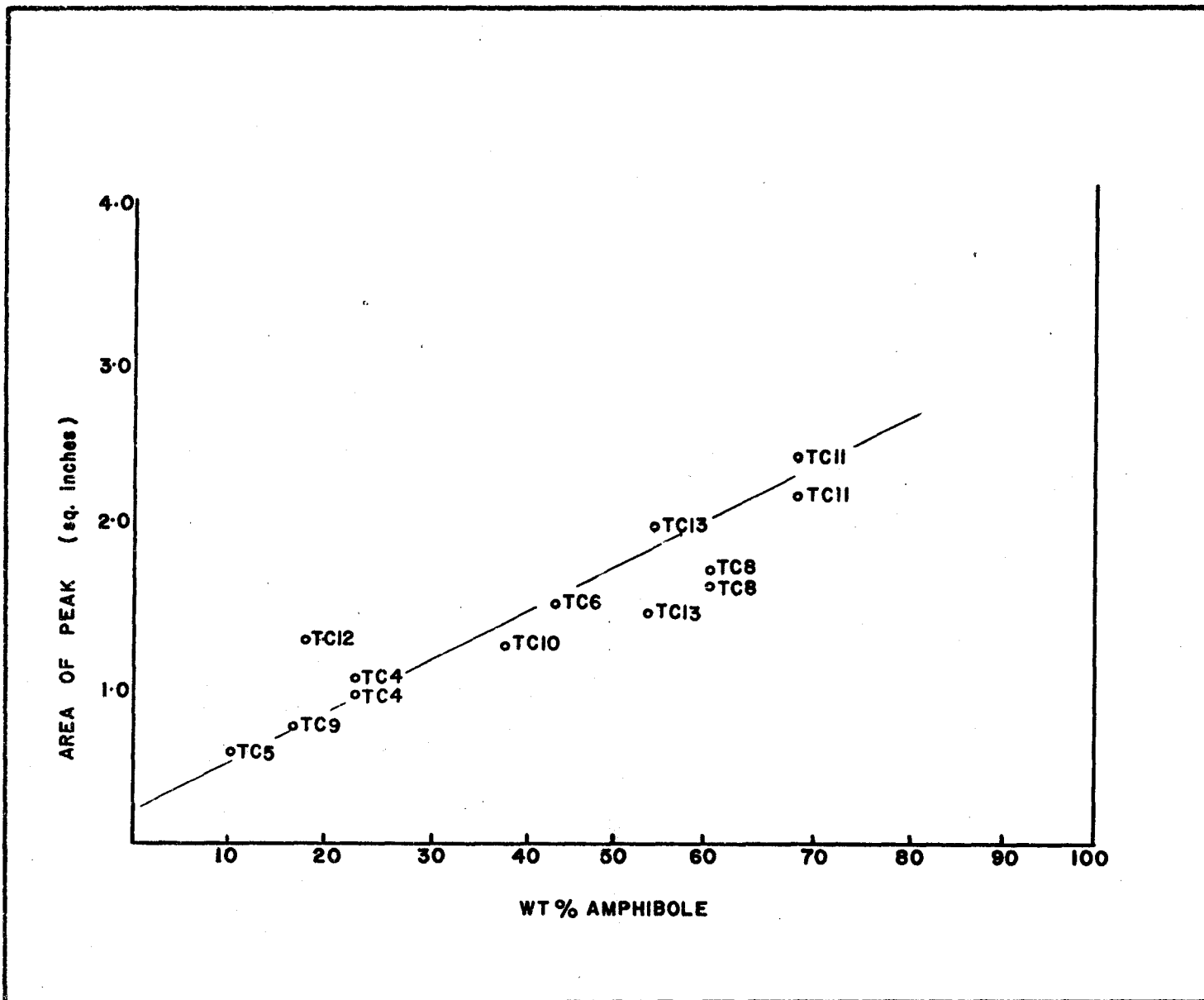


FIGURE 8 / WT. % AMPHIBOLE VS PEAK AREA

## 2. THE VASELINE METHOD

When mounted on a vaseline base the intensity of the diffracted beam is greatly reduced. The effect of this is greatest on materials which have low absorption coefficients, such as the plagioclase. Thus the resulting intensity patterns show greatly reduced peaks for the plagioclase even where the plagioclase is more abundant than the amphibole. Thus low proportions of a poorly absorbing material will not be observed in the diffraction traces.

For these reasons further investigation of this method was not followed.

## 3. THE ROTATING MOUNT METHOD

The series of diffraction traces using the nail polish mount on a rotating stage produce poor results. These results are however fractionally better than those of the previous methods but as before the remixed specimen did not produce a comparable trace.

A series of five scans were made on a rotating specimen; first two runs were made and then the sample was remixed with more nail polish and three more runs were carried out. The resulting ratios of  $I_A/I_P$  were compared. The values of  $I_A/I_P$  of un-mixed samples were relatively good, ranging from 0.77 to 0.81 and 2.09 to 2.24 after re-mixing. It was seen however, that the variation in  $I_A/I_P$  ratio from one series to the next showed a very large deviation, approximately 1.4 from the average ratios.

The rotating stage is meant to compensate for oriented crystals in a specimen. This would seem to indicate that the orientation is rather extreme in these samples.

Further study by this method could not be attempted as the equipment was not available.

#### 4. POWDER PACK METHOD

The methods of mounting powders in a thin lap have failed to produce a randomly oriented sample. An alternative method, that of packing a powdered sample, was used.

The diffracted intensity produced by a crystallographic (hkl) plane is influenced not only by absorption but also the degree of orientation of the crystal faces. For example, if a lath-like crystal such as plagioclase tends to fall preferentially on one of two faces such as a brick does when dropped, then the hkl values which most nearly parallel the face will control the diffracted intensity, while those (hkl) values at acute angles will have only minor effect. Thus the hkl value one desires to use in his measurements may not appear as a strong line even though high values of  $I/I_0$  are predicted for that plane.

The powder packing method is more likely to produce a randomness where all (hkl) surfaces would have an equal area to be irradiated.

The powdered samples mounted in the cylinders were scanned for amphibole only. The amphibole having given the stronger correlation between intensity and abundance, it was felt that the initial study should be done with this mineral. The samples were rotated  $90^\circ$  after each scan until the four positions about the axis normal to the specimen surface had been scanned. Then the specimen was repacked. The scanning procedure in four positions was then repeated. See Table 3 for the results of this method.

The mean value of intensity for the first rotation was 49.5 and for the remixed rotation the mean intensity was nearly double at 95.0.

The results are not reproducible for this method, even though

TABLE 3  
ROTATED SAMPLE SPECIMENS

Sample Number and position*	Amphibole Peak Height	Plagioclase Peak Height	Comments
TC14(1)	off scale	29.5	powder pack runs. (8/1/4). very far off of scale, pro- bably a reading of about 200.
(2)	39.5		
(3)	37.0		
(4)	59.5		
(5)	62.0		
TC14(1)	87.0		sample remixed
(2)	46.3		
(3)	off scale		estimated high 150
(4)	97.0		
TC18(1)	68.5	25.1	sample specimen as TC19 without epoxy(8/1/4)
(2)	54.5	undefined	
TC19(1)	67	off scale	TC19 mounted in epoxy. (8/1/2)-changed to produce significant peaks.
(2)	43	48	broad peaks
(3)	55	32	plagioclase peak poorly defined

\* rotational position about the axis normal to the face of the cylinder

great care was taken in the production and mounting of the samples. This would seem to indicate that sample homogeneity has still not been reached. Figure 9 shows a series of sample traces from the specimen TC15.

#### 5. SUSPENDED MOUNTS

A method of embedding the finely ground sample studies in a material which could be ground down again to the proper size should have produced a sample in which the shape of each particle was unrelated to the shape and orientation of the mineral particles. From this powder randomly oriented powder pack samples could be produced.

As in mounting on vaseline the intensity of the diffracted beam is decreased greatly. With this decrease in height and definition the peaks become wider and overlap with surrounding peaks. Significant peaks were seen only after the count rate was increased. The plagioclase peaks were often too small to measure. This makes the measurements of ratios less accurate. Some data for TC19 and epoxy are in Table 3.

Thus this method is not desirable either.

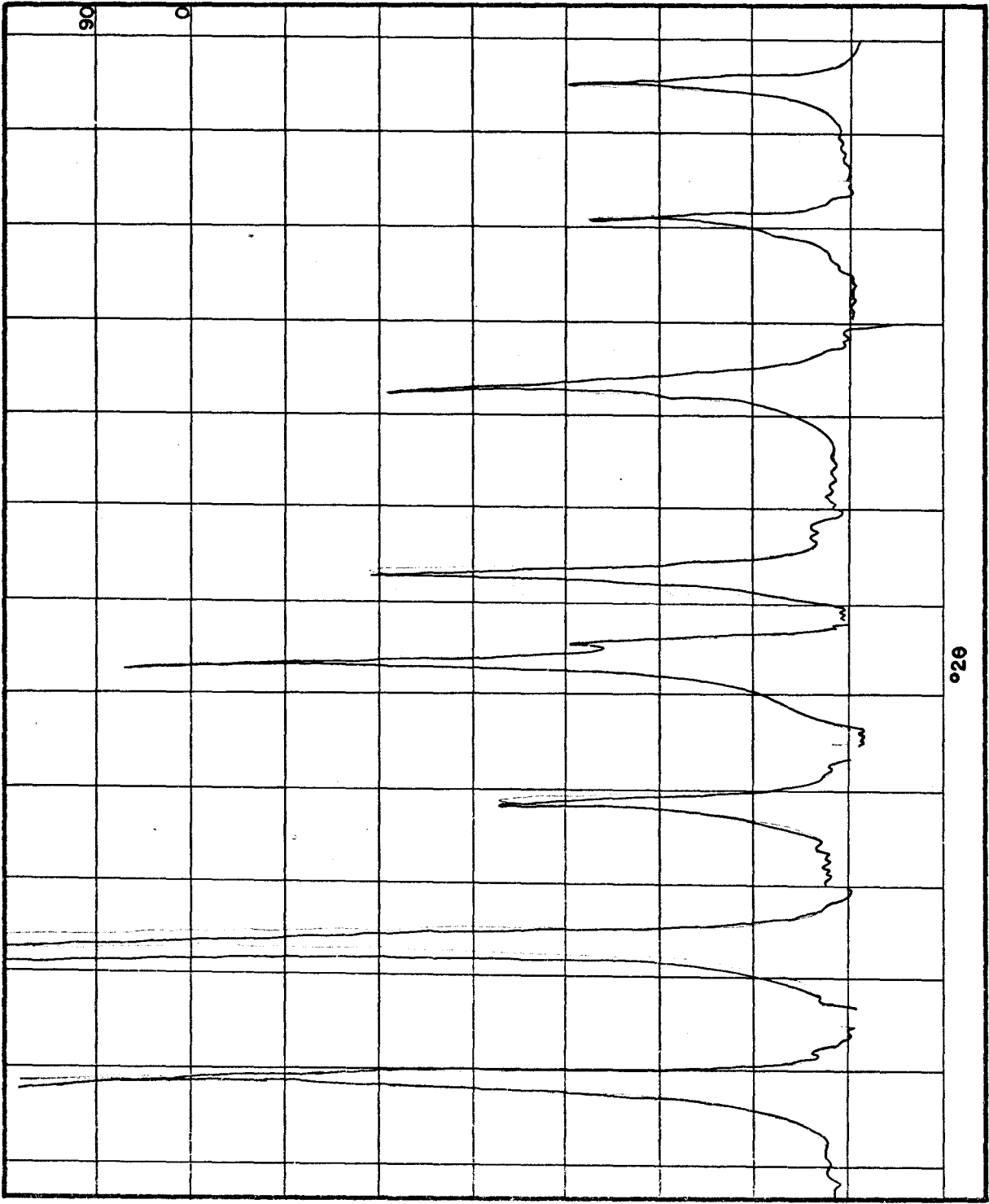


FIGURE 9 / SAMPLE TRACES OF ROTATED AMPHIBOLE

## GENERAL DISCUSSION

In the methods used it was not shown that a random homogeneous sample was formed as the results produced a poor level of reliability. There were however, indications of certain trends.

In Figure 7 the tendency of plagioclase to form better linear approximations where the abundance of the mineral is low can be seen. This effect was likely produced by the separation of similar lath-like particles. As the abundance of the mineral increases more of the laths would be in contact and at some point there will be a shingling effect. The abundance of the component at which this effect became important was variable as indicated by the irregular peaks produced for amphibole in varying weight ratios in the powder packs of Method 4. A hypothetical graph of this is shown in Figure 10. Thus rather than the straight line function indicated in Figures 7 and 8 a more linear function may have been produced.

It was also shown in Figure 4 and by the methods of mounting in epoxy or vaseline that the intensity at which a mineral with a low absorption coefficient becomes significant is of great importance. The slope of the plagioclase line in Figure 7 indicates that at abundances lower than about 30% the plagioclase peak became insignificant and estimates of its abundance could not be made at all.

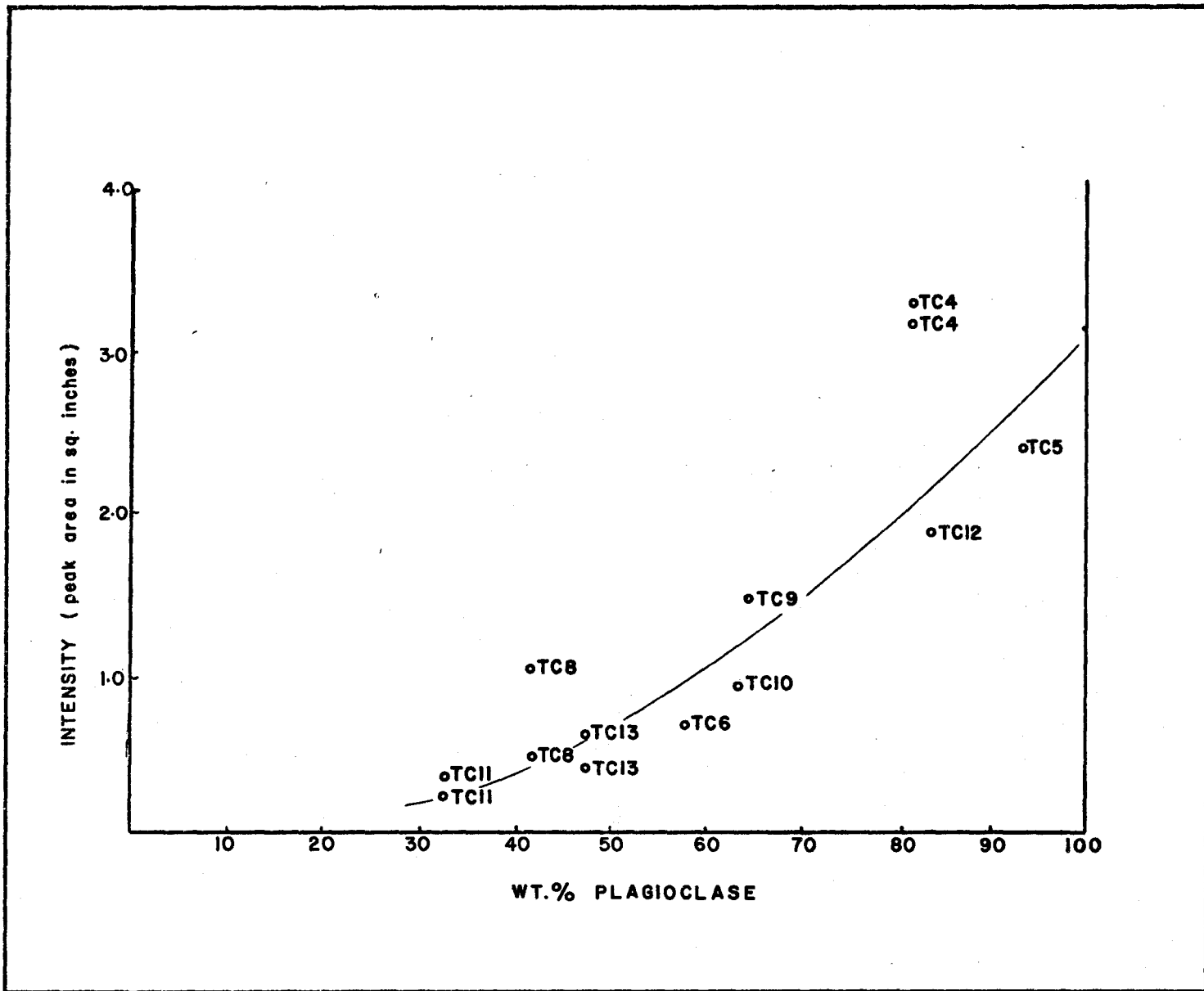


FIGURE 10 / HYPOTHETICAL WT.% VS INTENSITY CURVE



### CONCLUSIONS AND RECOMMENDATIONS

From the study of modal abundances in rocks which contain platy, lath-like minerals by X-ray diffraction, it was found that a technique for the production of random specimens is required.

Problems involving intensity variations could be solved by using ratio measurements. Thus by varying the multiplicity factor significant diffraction traces could be produced for all of the components and accurate ratios formed. This also reduces mechanical variations within the X-ray machinery.

The major difficulty lies in producing randomly oriented homogeneous powders from which standardized curves of mineral abundance vs intensity could be produced. The analysis indicated that the rotating mount and powder packing methods yielded results of better accuracy. Thus, it was concluded that the combination of these two methods may yield results of sufficient accuracy to make estimates of abundances. It was also found that averaging a number of successive still mounts yields results biased toward a particular packing.

The possibility of finding a mounting medium such as the epoxy used here could also produce the randomness required. Some work with low absorption, low melting point glasses may help.

Special attention should also be placed on methods of grinding. It would be best to use an automatic grinder and thus uniformly ground samples could be produced.

The difficulties involved in the study are mostly technical and new and better methods are required.

The author still feels that a fast standardized method of modal analysis by the use of calibrated X-ray diffraction patterns can be applied to metamorphic rocks, and that technical problems can be overcome.

## BIBLIOGRAPHY

- Azaroff, Leonid V. and Buerger, Martin J. (1958) The Powder Method  
New York: McGraw-Hill Book Company.
- Brindley, G. W. and Kurtossy, S. S. (1961) "Quantitative determination  
of kaolinite by X-ray diffraction." American Mineralogist,  
46, 1205-1215.
- Carl, H. F. (1947). "Quantitative mineral analysis with a recording  
X-ray diffraction spectrometer." American Mineralogist,  
32, 508-517.
- Klug, H. P. and Alexander, L. E. (1954) X-ray Diffraction Procedures  
for Polycrystalline and Amorphous Materials. New York:  
John Wiley & Sons, Inc.
- Tatlock, D. B. (1966) "Rapid modal analysis of some felsic rocks  
from calibrated X-ray diffraction patterns." Geological  
Survey Bulletin 1209.