NOTE ON QUANTITATIVE ANALYSIS BY *X*-RAY DIFFRACTION METHODS

KATHLEEN LONSDALE, *University College, London, England.*

In a recent paper ("Quantitative Mineral Analysis with a Recording *X*-ray Diffraction Spectrometer," *Am. Mineral.*, **32,** 508–517) Howard F. Carl has drawn attention to some of the factors limiting the usefulness of quantitative *x*-ray analysis by photographic methods. Undoubtedly sample preparation is one of the most critical problems whatever method is used. In preparing samples for "working curves" it appears from the paper that quartz powder was mixed with various other powdered materials in a range of concentrations. The record shown in Fig. 1. of the paper, if it is typical, would indicate that the powder used was finely divided, but that the particles were not small enough to show line broadening. It should be pointed out that considerable quantities of *colloidal* quartz, if mixed with other well crystallized materials, could be present and yet elude measurement, especially if well-crystallized quartz were also to be found in the mixture.

Howard F. Carl implies that variations in the *x*-ray output are more serious in the case of the photographic method than in that of the Geiger-counter spectrometer. That is surely not the case, however; such variations are likely to be *less* significant in the photographic method, which integrates the output over an appreciable time period, than in any method depending on the almost instantaneous output from a variable source, even if measurements are repeated several times. The fact that the standard commercial equipment used provided a very steady output, was a recommendation for that particular source of *x*-rays, but not evidence in favor of any particular method of measuring intensity.

The most surprising statement in the paper, however, is that in the case of the recording Geiger-counter *x*-ray spectrometer "there is only a linear relation between intensity and recorder deflection. This numerical value of line height is, under standardized conditions, a direct measure of the actual intensity of the diffracted beam from the specimen." No evidence is offered to show the correctness of this statement. Now it is possible to obtain a calibration curve for any individual recording spectrometer by repeating the same peak with an increasing number of Ni filters either between the *x*-ray tube and the specimen or, if more convenient, between the specimen and the Geiger-counter tube (the results are indistinguishable). Each filter, if 0.0006″ thick, should cut down the intensity by a factor of about 2, or if 0.001″ thick, by a factor of about 3. The accompanying graph (Fig. 1) shows the recorded peaks obtained in this way for 1, 2, 3, 4, 5, 6, 7 Ni filters each 0.001″ thick, and it is very obvious that the ratio of peak heights for n to $(n-1)$

filters is far from constant, as it ought to be if there were a linear relationship of intensity and recorder deflection. The high intensities (corresponding to a full scale deflection) are much lower than they ought to be relative to an intensity of medium value, and the low intensities (such as would be given by a colloidal specimen unless present in considerable quantity) are not recorded at all. A correct linear relationship was only found to exist over a very restricted portion of the graph (about 1–3 units) for any conditions of sensitivity or damping. The instrument ceased to record, even at highest sensitivity, at an intensity which was certainly more than 100 times the minimum intensity observable with a scaling circuit and mechanical impulse counter. Results similar to the above were found with various Geiger-counter tubes and under various conditions, but there is the possibility that different spectrometers may have different calibration curves, and therefore any quantitative analysis, to be of value, should be preceded by the measurement of the curve appropriate to the particular equipment used.

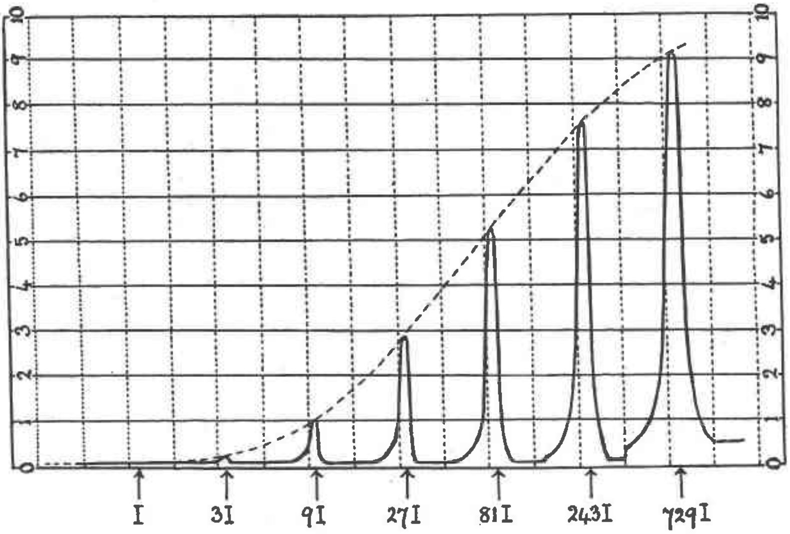

FIG. 1. Calibration curve for recording Geiger-counter spectrometer. For a linear relationship, each peak should be 3 times the height of the previous peak; this is only approximately true in the limited range 1–3 units. Similar curves were obtained under widely varying conditions.

Perhaps it should be pointed out that quantitative analysis by photographic methods equally requires care with regard to calibration of line blackening or density versus x-ray intensity. Mere matching against a "working curve" (that is, a set of lines corresponding to different concentrations of one constituent in a powder mixture) is not sufficient in the case where one or other constituent may be of colloidal dimensions or poorly crystallized.

LETTER TO THE EDITOR

Finland has an excellent and keenly scientific minded Technical Institute, Teknillinen Korkeakoulu. During the war its library was bombed and totally destroyed.

On my recent trip to Finland for the American Friends Service Committee, I discussed the situation with Dr. Martti Levon, Director of the Institute. He said he would welcome gifts of Scientific and Technical Books and Periodicals from America to take the place of those destroyed. In the remarkable efforts for recovery that the Finns are making, the lack of technical library facilities is a very serious handicap. It would be a practical act of friendship to a nation that holds America in high regard if Americans should contribute good technical books and periodicals to this library.

Any such gifts should be marked for the Institute of Technology, Helsinki, and sent to the Legation of Finland, 2144 Wyoming Ave., N.W., Washington, D. C. Dr. K. T. Jutila, the Finnish Minister, will arrange for their being shipped to Finland.

Sincerely yours,
ARTHUR E. MORGAN
Member, *American Friends Service Committee*
Yellow Springs, Ohio

MANUSCRIPT TRANSLATIONS FROM THE RUSSIAN

All geologic publications.—The Committee on Russian Literature of the Geological Society of America has begun the compilation of a list of translations from Russian into English of geologic papers and books; that is, a list of translations extant in manuscript form in the United States. Please send information as to any such translations to the committee chairman or to Geological Society headquarters. The word *geologic* is used here in its widest sense to extend inclusively from geophysics to paleontology.

Current geologic publications.—If information about translations of recent publications is received soon enough, it will be included in forthcoming volumes of the *Bibliography and Index of Geology Exclusive of North America* and the *Bibliography of Economic Geology*, subject to permission from proprietors of translations.

RONALD K. DEFORD, *Chairman*,
Box 1814, Midland, Texas.

To the list of United States triplite occurrences as presented in Table 1, page 521, in the September–October 1947 issue of the *American Mineralogist* (Triplite crystals from Colorado by C. W. Wolfe and E. Wm. Heinrich) there should be added the Rutherford pegmatite near Amelia, Virginia. This occurrence, which was recorded and described by Glass (*Am. Mineral.*, **20**, 759, 1935), was inadvertently omitted from the tabulation.

E. WM. HEINRICH

Correction

Dr. John P. Marble has called our attention to the fact that in our short article entitled "Polycrase in New York State" we omitted to cite his paper entitled "Possible Age of

Allanite From Whiteface Mountain, Essex County, New York" which appeared in the *American Journal of Science*, January 1943. Hence, the polycrase locality in the town of Day, New York, referred to in our paper is the third occurrence of radioactive minerals in the Precambrian of northern New York State.

EDWARD S. C. SMITH AND OSCAR R. KRUESI

The Industrial Diamond Information Bureau, Industrial Distributors (1946) Ltd., St. Andrew's House, 32–34, Holborn Viaduct, London, E.C. 1., publishes monthly a bulletin containing abstracts of articles dealing with properties and industrial applications of diamond together with notices of patents and patent applications in many States. A copy of this bulletin may be obtained, free of charge, on application to the above address.

The first Fall meeting of the New York Mineralogical club was held on Oct. 15, 1947, and was devoted to the collecting experiences of its members during the preceding summer. At this meeting fifteen members delivered talks on their activities which extended from Maine to Brazil, and many interesting specimens were displayed. Some collectors brought in a supply of minerals for distribution to other members.

MINERALS OF ARIZONA by FREDERIC W. GALBRAITH. Second edition (*revised*). Arizona Bureau of Mines, Geological Series No. 17, Bulletin No. **153** (1947); 102 pages. Price one dollar (free to residents of Arizona).

This is an enlargement and revision of Bulletin No. 149 which was issued in 1941 (see *Am. Mineral.*, **27**, 332). With additional information now available through the publication of vol. 1 of *Dana's System of Mineralogy* it was deemed desirable to issue the Bulletin on Minerals of Arizona in a revised and enlarged form. The mineral index now lists 431 names.

W.F.H.

A suggestion has been received that the Journal publish from time to time research projects under investigation and the names of the individuals involved. It was felt that such publication might prevent unfortunate duplication of effort, as such instances have occurred in the past, and further greater cooperation between individuals engaged on the same or similar investigations. The success of the effort will depend largely upon the extent to which this information is made available by the individuals concerned. The editor will gladly publish all data thus submitted.

Recent publications of interest to mineralogists and petrologists include The Pennsylvania State College Bulletin No. 47, Properties and New Uses of Pennsylvania Slate (price $2.00), and Arizona Bureau of Mines, Technology Series No. 42, Bulletin 154, Field Tests for the Common Metals (ninth edition), by George R. Fansett.