Techniques of field gamma-ray spectrometry

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ABSTRACT. Field gamma-ray spectrometry is a rapid and effective quantitative method of mapping variations of radioelements within igneous intrusions. A field procedure for radioelement mapping demonstrates the value of the method to studies of late-stage magmatic processes during the emplacement of granite intrusions and the determination of their present-day heat productivities. Methods and problems of instrument calibration using both natural and artificial sources are discussed. Calibration based on neutron activation analysis of samples from natural outcrops achieves results comparable with those obtained using artificial sources and has the advantage that it relates directly to field conditions; furthermore it enables secular disequilibrium in the uranium and thorium decay series to be recognized.

THE applications of field gamma-ray spectrometry as a geological mapping and exploration tool are well developed, mainly through the work of the Geological Survey of Canada (e.g. Grasty and Darnley, 1971), the Danish Atomic Energy Commission (e.g. Lovborg, 1973), and the International Atomic Energy Agency (e.g. IAEA, 1974). In Britain, research and development of gamma-ray spectrometry instruments and methods for field use have been carried out by the Institute of Geological Sciences in collaboration with the Atomic Energy Research Establishment, Harwell. These are described for example by Miller and Loosemore, 1972, but perhaps the most important recent application of field spectrometry for natural radioelements relates to the use of towed sea-bed instruments for mapping the continental shelf (Miller et al., 1977).

Related methods of radiometry, using total gamma scintillometry have been applied extensively in Britain since the late 1950s (Miller and Loosemore, 1972) and more recently for the UKAEA sponsored uranium reconnaissance programmes (Bowie *et al.*, 1970; Gallagher *et al.*, 1971) which have resulted in the development of techniques for locating both exposed and concealed mineralization (Michie *et al.*, 1973). Interest in the nature of uranium occurrence in Britain has grown recently, both in the context of uranium mineraliza-

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tion (Simpson *et al.*, 1979) and geothermal studies (Oxburgh *et al.*, 1977; Wheildon *et al.*, 1980), primarily in response to increasing energy needs (Garnish, 1976; Bowie, 1978).

This paper describes techniques of ground-based quantitative gamma-ray spectrometry as applied to British Caledonian granites and in particular to the Loch Doon granite in Scotland. Such granites, many of which contain above-average abundances of lithophile elements including K, U, and Th (Watson and Plant, 1979; Plant et al., 1980), are suited to in situ methods of spectrometry, particularly where there is good exposure. An approach to instrument calibration and its implications is discussed in relation to the mapping of intraintrusion variations of radioelement content. These data are important in characterizing the nature of radioelement occurrence in uraniferous granites (Simpson et al., 1979) and are relevant to problems of petrogenesis (e.g. Adams et al., 1969; Bohse et al., 1974), geothermal studies (e.g. Tilling et al., 1970), and the recognition of potential source regions for uranium mineralization (Stuckless and Ferreira, 1976).

Advantages and disadvantages of field spectrometry

The use of a portable gamma-ray spectrometer for the quantitative measurement of K, U, and Th in surface outcrops offers a number of advantages when compared with the more conventional method of laboratory analysis (e.g. Adams and Fryer, 1964; Lovborg *et al.*, 1971):

(a) In situ analysis provides immediate results. This aspect of field spectrometry makes it a powerful tool for locating uranium deposits (e.g. Bowie, 1972) and even associated metalliferous deposits (Moxham *et al.*, 1965).

(b) A relatively large volume of rock is analysed (Gregory and Horwood, 1961); Lovborg *et al.* (1971) calculated an effective sample size of 30-50 kg, i.e. an order of magnitude larger than that of a conventional hand sample.

(c) On-foot gamma-radiometric surveying facilitates the rapid mapping of geological formations, especially in rough terrain (Lovborg, 1973) and is appropriate for both reconnaissance and detailed mapping of radioelement concentrations (Killeen and Carmichael, 1972).

Possible disadvantages of field spectrometry are:

(a) Quantitative determination of uranium and thorium by gamma-ray spectrometry assumes secular equilibrium between parent isotopes ²³⁸U and ²³²Th with their decay products ²¹⁴Bi and ²⁰⁸Tl respectively (e.g. Adams and Fryer, 1964). However, significant disequilibrium in the ²³⁸U series, which results mainly from the relative mobilities of ²³⁸U itself and its decay products ²³⁴U, ²²⁶Ra, and ²²²Rn, is uncommon in unweathered igneous rocks older than 1 Ma (Lovborg, 1972; Levinson and Coetzee, 1978) and the ²³²Th series may be considered to be in equilibrium in most geological environments (Adams and Gasparini, 1970).

(b) Outcrops must be large enough to subtend a solid angle of detection of 2π (e.g. Killeen and Carmichael, 1972), i.e. quantitative measurements on narrow veins are not possible. Therefore, the method is appropriate for mapping radioelement variations on scales larger than individual small outcrops.

Instrumentation

The instrument used was a commercially available, four-channel gamma-ray spectrometer incorporating a 7.5×7.5 cm² NaI scintillation detector (Geometrics DISA 400). Three energy windows simultaneously record gamma-rays associated with characteristic peaks in the decay spectra of Th, U, and K. These windows are centred on 2.62 MeV (²⁰⁸Tl), 1.76 MeV (²¹⁴Bi) and 1.46 MeV (⁴⁰K), respectively, with widths of 400 KeV for the Th channel and 200 KeV for the U and K channels (for a discussion see Richards and Walraven, 1975); the fourth channel has an energy window recording the total spectrum above 0.1 MeV.

For quantitative spectrometry, the instrument should be sufficiently stable to maintain the limits of the energy windows within about $\pm 3\%$ (cf. Lovborg, 1973; IAEA, 1974) and the amplifier should respond linearly. Stability during field operation was ensured by an adjustment of gain control (see below) and linearity was confirmed at both low and high count rates in laboratory tests. The spectrometer is simple to operate and can be used comfortably in most conditions using a waterproof carrying frame in which case the total weight is about 9 kg; care is required in handling the instrument since the NaI crystal, although well protected, is delicate.

Instrument calibration and implications

(i) Calibration parameters. Field gamma-ray spectrometers are usually calibrated using large sources of known radioelement concentration, either natural outcrops or more often concrete pads doped with Th, U, and K which are designed to simulate the energy distributions of gamma spectra typically emitted from the surface of natural outcrops (Killeen and Carmichael, 1970; Grasty and Darnley, 1971). Such sources must be at least 2 m in diameter and 0.5 m thick (IAEA, 1976). No concrete calibration sources are available in Britain although a simulation model using discrete sources does exist at the UKAEA (Harwell) (Wormald and Clayton, 1976).

Regarding source parameters, the three important prerequisites for quantitative spectrometry are:

(1) Secular equilibrium in the source between parent radioelements (U and Th) and their daughter decay products.

(2) Chemical homogeneity of the source.

(3) Constant source-detector geometry.

Ideally these conditions should be met in both calibration procedures and field use of the spectrometer; however, a more pragmatic approach to *in situ* measurements is often necessary. Therefore, notwithstanding the greater theoretical accuracy of instrument calibration afforded by artificial sources, it is particularly relevant to compare gamma-ray measurements with analyses of typical hand samples (see (iii) below).

Thus, if the spectrometer is *calibrated* against such samples, and, therefore, with reference to the particular characteristics of the geological environment, in situ spectrometric measurements should be equivalent to the analyses of hand samples provided secular equilibrium occurs (see (iv) below). The work described here is based on this procedure and differs from most reported methods of 'field' calibration (e.g. Doig, 1968; Killeen and Carmichael, 1970; Lovborg et al., 1971) in using neutron activation analysis for U and Th in the control samples, thus allowing the recognition of secular disequilibrium in the rock outcrops; the importance of this in calibrating laboratory spectrometers was recognized, for example, by Tammemagi and Smith (1975).

Given the three prerequisites stated earlier, the gamma-ray count rate detected within any one channel of the spectrometer depends primarily on:

(1) The concentration of the parent radioisotope associated with that channel.

(2) The sensitivity of the detector.

(3) The presence of interfering radioisotopes.

(4) The degree of Compton scattering between the energy windows (i.e. the channels).

(5) The background count rate.

(6) The linear attenuation coefficient appropriate to the rock material and the energies of the transmitted gamma-rays.

The equations relating channel count rates to these parameters, for a given rock material, are (cf. Killeen and Carmichael, 1970; Grasty and Darnley, 1971):

$$\mathbf{T}\mathbf{h}' = \mathbf{T}\mathbf{h} - \mathbf{T}\mathbf{h}_{\mathbf{B}} \tag{1}$$

$$\mathbf{U} = \mathbf{U} - \mathbf{K}_1 \mathbf{I} \mathbf{n} - \mathbf{U}_{\mathbf{B}} \tag{2}$$

$$K' = K - R_2 U' - R_3 I h' - K_B$$
 (3)

where Th, U, and K are the raw count rates in the respective spectrometer channels; Th', U', K' are the corrected count rates; Th_B, U_B, and K_B are the background count rates; and R₁, R₂, and R₃ are the stripping ratios (which correct for (3) and (4) above). For a given spectrometer these corrected channel count rates are a function only of the concentrations of the associated parent radioisotope (assuming secular equilibrium) which are:

$$Th (ppm) = Th'/S_{Th}$$
(4)

$$U(\text{ppm}) = U'/S_U \tag{5}$$

$$\mathbf{K} \left(\% \mathbf{K}_2 \mathbf{O} \right) = \mathbf{K} / \mathbf{S}_{\mathbf{K}} \tag{6}$$

where Th (ppm), U (ppm), and K (% K₂O) are the concentrations of the parent elements, and S_{Th}, S_U, and S_K are the respective channel sensitivities. Stripping ratios and channel sensitivities are essentially constant for a given spectrometer. A relatively low-intensity gamma emission at 2.44 MeV due to a ²¹⁴Bi decay (i.e. associated with ²³⁸U) falls just within the thorium channel window and therefore strictly involves a fourth stripping ratio, R₄, in equation (1). Although this correction was applied (cf. Lovborg *et al.*, 1971) it is omitted from this discussion for simplicity.

(ii) Method of determining stripping ratios. Stripping ratios were determined using artificial sources since natural outcrops rarely provide a sufficiently large range of Th/U ratios for reasonable statistics (Killeen and Carmichael, 1970). Stripping ratios depend much less on source parameters (i.e. geological conditions) than do channel sensitivities (Adams and Fryer, 1964) and furthermore, errors in these ratios affect the calibration of the instrument much less than do errors associated with channel sensitivities. The sources, constructed by the Archaeology Department, Oxford University, consist of half-metre cubes of concrete, individually doped with Th, U, and K. Calculation of stripping ratios from measurements on the concrete sources (e.g. Grasty and Darnley, 1971; IAEA, 1976) yielded the values in Table I which differed from those suggested by the manufacturer for the same model.

(iii) Method of determining channel sensitivities and backgrounds. These were determined using field radiometric measurements and analyses of hand samples from natural outcrops. Outcrops selected for combined gamma-ray measurements and sample collection were those which conformed most closely to an ideal source-detector geometry, i.e. the flattest and largest outcrops available. Statistical errors in determining count rates were minimized to < 3% using total counting times of 10 to 14 minutes. In practice five to seven gammaray readings of two-minute duration were taken at different positions (up to 0.3 m apart) on each outcrop surface, to minimize the effect of smallscale inhomogeneities in radioelement distribution and non-flat geometries; very inhomogeneous outcrops were avoided. At each outcrop a 2 to 3 kg sample representative of the freshest rock available was taken as close as possible to the spectrometer readings and always within 1 m. The samples were analysed for U and Th by the neutron activation method (e.g. Brunfelt and Steinnes, 1969), calibrated against both synthetic standards, made from dilutions of pure U and Th oxides with silica, and USGS standards (cf. Plant et al., 1976); K analyses were performed using flame photometry. A total of 34 samples from different outcrops in three Caledonian granites were analysed for calibration purposes: 17 analyses from Cairnsmore of Fleet, 11 analyses from Loch Doon, and 6 analyses from Leinster.

Combining equations (2) and (5), for example, gives the calibration equation for U:

$$U - R_1 Th' = S_U \times U (ppm) + U_B$$
(7)

which defines a linear relationship between stripped count rate $U-R_1Th'$ and sample analysis

 TABLE I. Comparison of calibration constants

 determined for the present spectrometer, with values

 determined by the manufacturer for the same model

 (based entirely on artificial sources)

		Present work	Manufacturer
Stripping ratio	R ₁ R ₂ R ₃ R ₄	$\begin{array}{r} 0.49 \pm 0.07 \\ 0.99 \pm 0.15 \\ 0.63 \pm 0.09 \\ 0.025 \pm 0.004 \end{array}$	$\begin{array}{c} 0.65 \pm \ 0.06 \\ 0.71 \pm \ 0.4 \\ 0.20 \pm \ 0.1 \end{array}$
Channel sensitivity (counts min ⁻¹ ppm ⁻¹)	S _{th} S _u S _k	$\begin{array}{rrr} 8.1 & \pm 0.3 \\ 20.0 & \pm 2.0 \\ 210 & \pm 7 \end{array}$	$\begin{array}{r} 8.0 \ \pm \ 0.2 \\ 18.9 \ \pm \ 0.7 \\ 200 \ \pm 40 \end{array}$

Errors in stripping ratios were calculated from uncertainties in Th, U, and K concentrations within the concrete sources and statistical counting errors; errors in sensitivities are calculated from linear regression fits to the field calibration data.

U (ppm). Therefore, the channel sensitivity S_{11} , and average background count rate U_B , are given by linear regression analysis of stripped count rate with sample analysis. Similar relationships can be written for Th (ppm) and K (% K2O) by combining equation (1) with (4) and (3) with (6), respectively. The thorium calibration line, which in this work includes the 2.44 MeV correction, allows the determination of Th_B , and hence Th', in equation (7). Similarly, Th_B and U_B are used in deducing the potassium calibration line. Calculated channel sensitivities agree well with values quoted by the manufacturer (see Table I) differing by only 2%, 6%, and 6% for the Th, U, and K channels, respectively. Background count rates determined by regression analysis of the field calibration data (cf. Doig, 1968; Lovborg et al., 1969) represent average values for Caledonian granitic terrains. Backgrounds thus determined are more realistic than those measured in low activity environments such as over a deep lake (Killeen and Carmichael, 1970), or those obtained using lead shielding. Such measurements would have provided an independent check on the calculated backgrounds, but were logistically impracticable. A constant linear attenuation coefficient is assumed in the above discussion, and since this parameter depends essentially on rock density (Lovborg, 1972), the assumption is valid for the limited range of rock types studied.

(iv) Discussion of results. The calibration data are plotted in fig. 1. Spectrometric analyses were calculated according to equations (4) to (6), using channel sensitivities and background count rates determined by linear regression of the field calibration data, excepting some rejected data points (see later). The lines of unity are, therefore, equivalent to calibration lines fitted to the accepted samples.

A rejection criterion was considered necessary (cf. Killeen and Carmichael, 1970) in view of the systematic scatter from the obvious linear trends of the calibration data: data points which exhibited a similar degree of scatter from at least two such trends were rejected from all data sets in order to reduce the effect of this scatter. Despite this apparently subjective choice of criterion its validity is demonstrated by close inspection. First, most data points lie close to the unity lines (fig. 1) especially for Th and K. This observation, and the agreement between calculated channel sensitivities and those of the manufacturer, shows that calibrations based on field samples can be comparable with those based on artificial sources (cf. Lovborg et al., 1971). The calibration line to the U data is less well defined, indicating that field calibration sites should be chosen carefully and that U count data must be interpreted with caution. In view of

the agreement with independent results, the calculated calibration constants were used to reduce the field data discussed in (v) below.



FIG. 1. Comparison of field gamma-spectrometric analyses of outcrops with laboratory analyses of hand samples: (a) thorium data, (b) uranium data, (c) potassium data. Spectrometric analyses are calculated with respect to laboratory analyses of the accepted samples, indicated by closed circles; open circles (nos. 1-8) signify data points rejected from calibration calculations. [Note: sample 6 is off-scale in (b)]. Analytical precision for Th and U determinations (neutron activation method) is generally better than ± 2 ppm and ± 0.7 ppm respectively; and for K determinations (flame photometry method) is better than about $\pm 0.1\%$ K₂O. Sample locations are indicated thus: D = Loch Doon granite, F = Cairnsmore of Fleet granite and L = Leinster granite.

Secondly, the rejected data points 1 to 6 (fig. 1) are scattered to a comparable degree on all calibration trends: this discounts secular disequilibrium as a cause of scatter since the K data would be unaffected. Source-detector geometries were similar for all calibration measurements and all samples were comparably fresh; hence, these factors cannot be responsible for the scatter. Interestingly, points 1 to 6 are the results of those samples collected from the marginal facies of the Fleet granite where aplitic and pegmatitic veining is particularly common (Gardiner and Reynolds, 1937; Parslow, 1968). This suggests source inhomogeneity as the most likely cause of scatter, arising from the difference in sample size analysed by the two methods (e.g. Adams and Fryer, 1964). Such systematic scatter might reflect the presence of concealed radioelement-rich veins or microfractures-badly scattered data typically correspond to relatively high gamma count rates.

Points 7 and 8 in fig. 1b (also from the Fleet granite) show a similar degree of scatter in the U calibration data. This is consistent with secular disequilibrium in the ²³⁸U series where parent uranium has been recently remobilized, although the data are equivocal regarding source inhomogeneity especially since U enrichment in fractures and microfractures is well known in Caledonian granites (Gallagher et al., 1971; Simpson et al., 1979). No further data were rejected from the calibration calculations despite some deviation from the calibration trend; therefore, the raw U data must be interpreted with this in mind. Finally, there is no evidence of disequilibrium in the ²³⁸U series due to preferential loss of daughter products in any samples of the three granites since significant reverse scatter is absent.

Using this procedure, the standard deviation between field gamma-ray analyses and neutron activation analyses of hand samples are ± 2 ppm, ± 0.9 ppm, and $\pm 0.2\%$ for Th, U, and K₂O, respectively: these are measures of the difference between methods rather than a quantification of the error in either. The accuracy of gamma-ray determinations of average radioelement concentration within outcrops is probably somewhat better. In addition, field mapping methods using a large number of measurements (see below) can minimize the effect of individual anomalous outcrops, thus allowing the measurement of average radioelement concentration characteristic of a particular location.

Sampling procedure and results of field mapping

Where exposure permits, a sampling interval appropriate to the scale of radioelement variation being investigated and the area of the survey, may be chosen. For example, the Loch Doon granite $(130 \text{ km}^2 \text{ in area, fig. } 2a)$ in south Scotland, which has been extensively exposed by glaciation, permitted an approximately regular sampling grid of 1 km spacing, appropriate to mapping the radioelement distribution trends over the whole intrusion.

In choosing suitable outcrops (usually at least a few square metres in area) and counting times for routine sampling, similar criteria to those adopted for calibration measurements were applied, except that at sampling locations two-minute measurements were made on separate outcrops several metres apart. This procedure minimized the effect of atypical outcrops, thus allowing a reasonably valid determination of the average radioelement concentration at the location to be made. Counting times were extended in the few cases where outcrops with particularly low or variable radioactivity were encountered. Although the use of lead shielding (Lovborg, 1972) or the practice of raising the detector above outcrop (Killeen and Carmichael, 1972) can reduce the effect of poor sourcedetector geometry and inhomogeneity, these techniques were not adopted because the former was impracticable for the extent of the survey area and the latter requires larger outcrop surfaces than those typically measured. Using the above method, a typical sampling rate over the Loch Doon granite was 7 to 10 measurement locations per day, in rough mountainous terrain.

Although seasonal conditions may cause secular changes in gamma-ray flux from both background sources (Killeen and Carmichael, 1970) and even specific outcrops (Lovborg et al., 1978), it is practicable to monitor only changes due to instrumental drift; potentially, this has the most significant effect on gamma-ray measurements. Short-term instrumental drift due to diurnal temperature variations was corrected using an external ¹³⁷Cs reference source to optimize the energy calibration of the instrument at each sampling location, and the instrument was allowed to reach the ambient air temperature at the beginning of each day of field operation. Longer-term instrumental changes were monitored by regular measurements at a base station granite outcrop during each survey, one such outcrop being chosen as a primary base station for all surveys. This practice proved necessary when an apparent change in channel sensitivities occurred due to crystal deterioration during the collection of the calibration data from the three granites. The stripping ratios and channel sensitivities need to be redetermined separately after major changes in instrument characteristics.

Some results from a gamma-spectrometric survey of the Loch Doon granite are illustrated in



FIG. 2. (a) Simplified geological map of the Loch Doon intrusion (based on Gardiner and Reynolds, 1932).
(b) Thorium distribution (in ppm) at outcrop level in the Loch Doon intrusion determined by gamma-ray spectrometry. Data are hand-contoured from 104 sampling locations on an approximately regular grid of 1 km spacing.

fig. 2b which shows the distribution pattern of thorium concentration in surface outcrops; the uranium distribution pattern is similar (Brown *et al.*, 1979). In fig. 2b only 4 of the 104 data points are not fitted by the contours, indicating the good reliability and consistency of the spectrometric method. Ranges of about 9 to 24 ppm (mean 16 ppm) for thorium, 3 to 9 ppm (mean 4.6 ppm) for uranium, and 2 to 4% (mean 2.8%) for potassium from field spectrometric analyses agree well with independent laboratory analyses of hand samples (Hennessy, 1979; Brown *et al.*, 1979). This re-emphasizes the comparability of field spectrometry with conventional sampling methods and its role in augmenting them.

The thorium distribution pattern (fig. 2b) correlates closely with both petrographic and geochemical trends, and thereby reflects the differentiation history of the magma (see Brown *et al.*, 1979; Cassidy, 1979). In addition, the spectrometric survey amplifies the geological map (fig. 2a) by clearly resolving two centres of high radioelement content which perhaps also reflect a differentiation pattern—one that was not detected by earlier petrographic and geochemical data. A primary magmatic distribution for uranium, and by inference thorium (Rogers and Adams, 1969), is suggested by the location of most uranium in primary accessory minerals (e.g. Simpson *et al.*, 1979; Cassidy, 1979) which is consistent with earlier indications that secular disequilibrium in the ²³⁸U series is not significant in this granite since such minerals are relatively resistant to surficial weathering.

Finally, gamma-radiometric surveying is particularly valuable in integrated geothermal studies (e.g. Sass *et al.*, 1968) since the method facilitates rapid and comprehensive mapping of K, U, and Th thereby characterizing radioelement occurrence and abundance, and allowing the calculation of reliable mean values for heat productivity in the surface rocks. For the Loch Doon granite, where a primary occurrence of radioelements is indicated, the average surface heat productivity of $2.6 \,\mu W m^{-3}$ (calculated from the mean Th, U, and K concentrations) may extend to depth, and therefore, represent bulk heat production in the whole granite.

Summary and comments

Field gamma-ray spectrometry is a rapid and effective method of mapping surface distributions of K, U, and Th, particularly over well-exposed granites, such as Loch Doon, which contain above-average levels of radioelements. *In situ* spectrometric determinations of radioelement concentrations are comparable with independent results based on laboratory analyses of hand samples, showing the two sampling methods to be complementary.

Instrument calibration based on laboratory analysis of field samples can be achieved successfully and gives comparable results with a method using artificial sources. Natural calibration sources are available as homogeneous outcrops; the Loch Doon granite, which is characteristically homogeneous on an outcrop scale, was particularly suitable for this purpose. The method has the advantage that it automatically takes account of in situ geological parameters such as the linear attenuation coefficient of the rock, the weathered skin on outcrop surfaces and typical background count rates. It also allows the recognition of secular disequilibrium in the surface rocks thereby checking the validity of quantitative determinations. Despite certain advantages of field calibration methods a set of concrete calibration pads in Britain would provide a valuable alternative calibration and base station facility.

For the three Caledonian granites studied (Loch Doon, Cairnsmore of Fleet and Leinster) secular disequilibrium in the ²³⁸U and ²³²Th series is not especially apparent, although calibration data do suggest a minor degree of disequilibrium in the ²³⁸U series, particularly in the Fleet granite. However, the most erroneous interpretations of spectrometric data are likely to result from inhomogeneity in the source.

Short-term instrument instability and longerterm changes due to instrument wear, which might affect absolute calibration, must be monitored regularly during the field operation of gammaray spectrometers. This is satisfactorily achieved using external energy reference sources (¹³⁷Cs for example) and base station rock outcrops.

Radioelement mapping of igneous intrusions, such as Caledonian granites, using field spectrometry should be complemented by laboratory analyses of hand samples and mineralogical studies of radioelement distribution (especially uranium) in thin section. The combination of these methods provides a powerful means of investigating the controls on radioelement distribution and may be used to interpret late-stage magmatic processes, assess the potential for uranium mineralization and calculate reliable estimates of surface heat productivity.

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