Fluocerite and its alteration products from the Afu Hills, Nigeria

M. T. Styles and B. R. Young

Institute of Geological Sciences, Exhibition Road, London SW7 2DE

ABSTRACT. Eluvial pebbles from the Afu Hills, Nigeria, are largely composed of fluocerite, Ce_{0.45}La_{0.45}Nd_{0.11}Pr_{0.02}Th_{0.06}Ca_{0.02}F_{3}, with minor monazite. The fluocerite shows two types of alteration, to bastnäsite, and to bastnäsite-(La) with cerianite. Electron microprobe analyses are given for fluocerite, monazite, bastnäsite and bastnäsite-(La). The refined cell parameters of the fluocerite are \( a = 7.130 \pm 0.001 \), \( c = 7.298 \pm 0.001 \) Å. Strongest lines are 3.204 Å (100), 3.65 (45), 2.059 (45), 2.010 (45) and 3.565 (40). Space group \( P 6_2/mcm, Z = 6, D_\text{calc} = 6.12 \text{ g cm}^{-3}, \omega = 1.613, \epsilon = 1.609 \text{ both } \pm 0.002, \text{ uniaxial negative.} \)

For bastnäsite \( a = 7.131 \pm 0.002 \), \( c = 9.786 \pm 0.004 \) Å, for cerianite \( a = 5.460 \pm 0.005 \) Å.

SPECIMENS containing the rare earth minerals described in this paper were originally sent for analysis in March 1961 to the Atomic Energy Division of the Geological Survey of Great Britain by Mining and Technical Services Ltd. Optical and XRD studies by Mr J. E. T. Horne and Mr R. K. Harrison showed that they contained fluocerite with subordinate cerianite, and an XRF analysis showed predominant rare earths with minor Th, Fe and traces of Ca, Sr, and Pb.

The sender's description stated that the specimens were found 'in the course of ground sluicing over albite-pegmatites bearing tin and subsidiary columbite, on the property of United Tin Areas of Nigeria, near Odeigi, Afu Hills, Nigeria'. Attempts were made to determine the exact location and possible source of the specimens, but despite the co-operation of Vectis Tin Mines Ltd. (previously United Tin Areas of Nigeria), Mackay and Schnellmann Ltd. (the consultants to United Tin), the Nigerian Mining Corporation and the Nigerian Geological Survey Department these attempts have been unsuccessful.

A detailed study of these unusual specimens has now been carried out by electron microprobe and X-ray diffraction.

Petrography

A polished thin section (G 152, Sliced Rock Collection, Institute of Geological Sciences) of a pebble approximately 1.5 cm in diameter has been studied in detail. It consists largely of fluocerite (and its alteration products) and a small amount of monazite with a dark rusty brown limonitic vein along one side. The fluocerite occurs as prismatic crystals up to 5 mm in length with widespread alteration along cracks and the basal cleavage. It is colourless to very pale yellow, uniaxial negative with low birefringence and refractive indices \( \omega = 1.613, \epsilon = 1.609 \text{ both } \pm 0.002 \) (Horne and Harrison, 1961).

The nature of the alteration of the fluocerite varies from point to point. Along the basal cleavage, fine cracks, and in some small patches, it is altered to a fine-grained (c. 1 μm) aggregate of a light yellowish-brown mineral with high birefringence, identified as bastnäsite. The fluocerite and bastnäsite are both cut by wider veins of a dark reddish-brown material that is composed of aggregates of mostly submicron crystals, which also appear brown in cross-polarized light with no discernible extinction or birefringence. These dark-brown aggregates were shown to be bastnäsite-(La) and cerianite with rare thorite.

Monazite occurs adjacent to the fluocerite and is pale yellow and traversed by numerous cracks and patches of dark-brown alteration products. The limonite vein is composed largely of goethite and quartz, an alumino-silicate, probably kaolinite and rare cassiterite.

Mineral compositions

Mineral compositions were determined using a Link Systems energy-dispersive X-ray analyser on a Geocan electron microprobe. The techniques used are described in detail in the appendix.

Fluocerite. Six fluocerite grains were analysed; they are unzoned and show only slight variation from grain to grain. The average composition is given in Table I along with the ionic formula which shows close agreement to a stoichiometric fluoride composition. A chondrite-normalized RE diagram
**Table I. Electron microprobe analyses of fluocerite**

<table>
<thead>
<tr>
<th>La</th>
<th>Ce</th>
<th>Pr</th>
<th>Nd</th>
<th>Sm</th>
<th>Ca</th>
<th>Th</th>
<th>F*</th>
</tr>
</thead>
<tbody>
<tr>
<td>20.85</td>
<td>37.25</td>
<td>3.23</td>
<td>8.18</td>
<td>0.35</td>
<td>5.51</td>
<td>3.84</td>
<td>25.28</td>
</tr>
<tr>
<td>1.707</td>
<td>3.027</td>
<td>0.261</td>
<td>0.645</td>
<td>0.012</td>
<td>0.141</td>
<td>0.189</td>
<td></td>
</tr>
</tbody>
</table>

* By difference.
1. Fluocerite, mean of seven analyses.
2. Number of ions on basis of eighteen fluorines.

(Leedly chondrite, Masuda et al., 1973) is given in fig. 1 which shows the strong enrichment in LRE. Early analyses of fluocerite did not give determinations of individual RE; however, if the analysis given here is recalculated on the same basis it gives a similar composition with the ratio of Ce-group (Ce+Th) to La-group (La+Pr+Nd) approximately 4:3, (see Palache et al., 1951, p. 49). A compilation of more recent analyses of fluocerite with individual RE determined is given in Table II.

Fig. 1. Chondrite-normalized RE abundances for fluocerite (stars), monazite (squares), bastnäsite (triangles), bastnäsite-(La) (diamonds) and cerianite (circles). This shows that all fluocerites are strongly enriched in LRE but the proportions vary as is shown by the sum of La + Ce + Pr which varies from 70 to 96% of the total RE content. The analysis with the

**Table II. Lanthanides in fluocerite, at % (ΣRE = 100%)**

<table>
<thead>
<tr>
<th>1</th>
<th>2</th>
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<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
<th>9</th>
<th>10</th>
</tr>
</thead>
<tbody>
<tr>
<td>La</td>
<td>20.3</td>
<td>17.9</td>
<td>28.5</td>
<td>27.3</td>
<td>31.5</td>
<td>30.1</td>
<td>31.2</td>
<td>39.1</td>
<td>25.0</td>
</tr>
<tr>
<td>Ce</td>
<td>44.0</td>
<td>48.3</td>
<td>39.4</td>
<td>48.4</td>
<td>48.0</td>
<td>34.4</td>
<td>45.4</td>
<td>47.2</td>
<td>68.0</td>
</tr>
<tr>
<td>Pr</td>
<td>6.5</td>
<td>5.7</td>
<td>4.9</td>
<td>3.2</td>
<td>5.7</td>
<td>4.6</td>
<td>3.1</td>
<td>3.2</td>
<td>1.3</td>
</tr>
<tr>
<td>Nd</td>
<td>18.2</td>
<td>20.7</td>
<td>13.9</td>
<td>12.3</td>
<td>12.8</td>
<td>11.4</td>
<td>10.1</td>
<td>9.3</td>
<td>5.1</td>
</tr>
<tr>
<td>Sm</td>
<td>6.5</td>
<td>4.0</td>
<td>2.3</td>
<td>3.1</td>
<td>1.4</td>
<td>0.5</td>
<td>0.8</td>
<td>1.2</td>
<td>0.6</td>
</tr>
<tr>
<td>Eu</td>
<td>0.1</td>
<td>---</td>
<td>0.5</td>
<td>---</td>
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</tr>
<tr>
<td>Gd</td>
<td>2.6</td>
<td>3.0</td>
<td>4.2</td>
<td>1.9</td>
<td>0.4</td>
<td>0.3</td>
<td>---</td>
<td>---</td>
<td>0.1</td>
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<tr>
<td>Dy</td>
<td>1.2</td>
<td>0.4</td>
<td>3.1</td>
<td>1.0</td>
<td>0.1</td>
<td>---</td>
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</tr>
<tr>
<td>Ho</td>
<td>---</td>
<td>---</td>
<td>0.6</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td></td>
</tr>
<tr>
<td>Tb</td>
<td>0.4</td>
<td>tr</td>
<td>1.9</td>
<td>0.4</td>
<td>0.1</td>
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</tr>
<tr>
<td>Tb</td>
<td>0.2</td>
<td>---</td>
<td>0.3</td>
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<td>---</td>
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<td>---</td>
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<td></td>
</tr>
<tr>
<td>Yb</td>
<td>---</td>
<td>1.8</td>
<td>0.3</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td></td>
</tr>
<tr>
<td>Lu</td>
<td>---</td>
<td>---</td>
<td>0.3</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td></td>
</tr>
<tr>
<td>La + Ce + Pr</td>
<td>70.8</td>
<td>71.9</td>
<td>72.8</td>
<td>78.9</td>
<td>85.2</td>
<td>88.1</td>
<td>88.8</td>
<td>89.5</td>
<td>94.3</td>
</tr>
</tbody>
</table>

1. Arkhangel’skaya (1970); av. of 2, metasomatic, alk. granitoids, E. Siberia.
2. Popova and Bazhenova (1976); greisen, Far Eastern USSR.
3. Feldman et al. (1973); granite, northern Tien-Shan.
4. Vetoshkina et al. (1980); amazonite pegmatite, Ploska Mt., Kola Peninsula.
5. Gurov and Gurova (1974); metasomatic, Ukraine.
6. Styles and Young; eluvial, Nigeria.
7. Heinrich and Gross (1960); granite pegmatite, Teller Co., USA.
8. Semenov and Barinetskii (1958); hydrothermal alteration of granite, N. Kingizya.
**Table III. Electron microprobe analyses of monazite, bastnäsite, and bastnäsite-(La)**

<table>
<thead>
<tr>
<th></th>
<th>1</th>
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<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
</tr>
</thead>
<tbody>
<tr>
<td>La&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>16.63%</td>
<td>0.984%</td>
<td>21.65%</td>
<td>1.758%</td>
<td>55.04%</td>
<td>4.407%</td>
</tr>
<tr>
<td>Ce&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>38.49%</td>
<td>2.265%</td>
<td>37.97%</td>
<td>3.090%</td>
<td>nd</td>
<td>—</td>
</tr>
<tr>
<td>Pr&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>3.68%</td>
<td>0.212%</td>
<td>3.13%</td>
<td>0.249%</td>
<td>4.35%</td>
<td>0.348%</td>
</tr>
<tr>
<td>Nd&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>9.69%</td>
<td>0.515%</td>
<td>4.094%</td>
<td>0.692%</td>
<td>11.49%</td>
<td>0.884%</td>
</tr>
<tr>
<td>Sm&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;3&lt;/sub&gt;</td>
<td>0.41%</td>
<td>0.026%</td>
<td>0.40%</td>
<td>0.036%</td>
<td>nd</td>
<td>—</td>
</tr>
<tr>
<td>CaO</td>
<td>nd</td>
<td>—</td>
<td>0.65%</td>
<td>0.212%</td>
<td>1.72%</td>
<td>0.551%</td>
</tr>
<tr>
<td>ThO&lt;sub&gt;2&lt;/sub&gt;</td>
<td>1.28%</td>
<td>0.092%</td>
<td>nd</td>
<td>—</td>
<td>nd</td>
<td>—</td>
</tr>
<tr>
<td>P&lt;sub&gt;2&lt;/sub&gt;O&lt;sub&gt;5&lt;/sub&gt;</td>
<td>28.87%</td>
<td>3.926%</td>
<td>nd</td>
<td>—</td>
<td>nd</td>
<td>—</td>
</tr>
<tr>
<td>CO&lt;sub&gt;2&lt;/sub&gt;</td>
<td>6.000%</td>
<td>6.000%</td>
<td>6.000%</td>
<td>6.000%</td>
<td>6.000%</td>
<td>6.000%</td>
</tr>
</tbody>
</table>

**Composition**

- *Monazite.* The analysed monazite grains show a slight variation from grain to grain and slight zoning to more Ce-rich rims. An average analysis is given in Table III col. 1, which shows that it has a similar RE content to the fluocerite but is relatively depleted in La (Fig. 1). It also has a much lower Th content, 1.13% compared with 3.84% in fluocerite. The textural relations suggest that the monazite and fluocerite are primary minerals and these differences in RE and Th represent the differing chemical affinities of the two minerals.

- *Bastnäsite.* The light yellowish-brown bastnäsite shows only a slight variation from point to point on the thin-section and an average composition is given in Table III, col. 3. (These analyses represent aggregates of several grains as the grain size is smaller than the electron beam.) The RE distribution is very similar to that of fluocerite (Fig. 1) although the total RE content is lower due to the presence of CO<sub>2</sub>. Th was not detected (< 0.4%) and contrasts with 4% in fluocerite.

- *Dark-brown alteration products.* Analyses of the dark-brown alteration products show a wide range of compositions varying from Ce-rich to La-rich (Table IV). The grain size is generally much smaller than the electron beam and in consequence a mixture of several mineral grains have been analysed. However, two analyses of single minerals were obtained, first of a dark-brown Ce-free bastnäsite-(La) (Table III, col. 5) and secondly of a thorite. The analyses of these mineral aggregates are plotted on a diagram of Ce vs. La (Fig. 2) which shows that they lie on a mixing line between an La-rich phase of approximately the same composition as the analysed bastnäsite-(La) and a Ce-rich phase containing approximately 65% Ce. The mineral cerianite (CeO<sub>2</sub>) was detected on the X-ray diffractometer charts. If it is assumed that all the La is in the bastnäsite-(La) the composition of the Ce-rich phase can be calculated. The greatest

---

**Fig. 2.** La vs. Ce for dark-brown alteration products (circles), average fluocerite (star), average monazite (square) and average bastnäsite (triangle).
uncertainty in the calculation is the distribution of Th. A plot of Ce vs. Th (fig. 3) shows that, apart from two which have much higher Th, the analyses lie along a straight line. In addition, the two anomalous analyses contain high Si (Table IV, col. 5) and also lie away from the line on the Ce vs. La diagram. These results suggest that most analyses contain a Ce-rich phase with a Ce:Th ratio of approximately 8:1 and that two analyses also contain thorite.

A calculated composition of the Ce-rich phase, cerianite, is given in Table IV, col. 6. This is only intended as an approximation as there are several possible sources of error in the calculation including: errors in the matrix correction of the microprobe data since the detected X-rays are produced by several different phases rather than one homogeneous phase as assumed by the matrix correction program; and variation in the composition of the minerals in the aggregates, e.g. the variation in Ca shows that the La-rich aggregates have insufficient Ca to form bastnäsite-(La) of the analysed composition, whilst in contrast, the Ce-rich aggregates have excess Ca, suggesting either that Ca enters the cerianite (Ca 1.0%) or that a small amount of a Ca mineral is also present.

The survey of bastnäsite compositions (Fleischer, 1978) does not give Ce-free compositions and it is thought that this is the first reported occurrence of naturally occurring bastnäsite-(La), with a composition close to the end member.

**X-ray data**

The d-spacings for fluocerite, bastnäsite, and cerianite were determined from diffractometer charts taken with a Cu-Kα radiation at room temperature (around 20°C) and a scanning speed of 1/8° 2θ/min. Lead nitrate (α = 7.8558 Å at around 20°C) was used as an internal standard.

The powder data were provisionally indexed by comparison with published data (JCPDS Data Cards 2-529, 4-593 and 11-340) and the fluocerite and bastnäsite data were refined using the US Naval Laboratory least squares program for refining cell parameters.

The fluocerite has cell dimensions a = 7.130 ± 0.001, c = 7.298 ± 0.001 Å, space group P63/mcm, and its density corresponding to these cell dimensions and the unit-cell contents (Table I) is 6.12 g cm⁻³. The powder data are listed in Table V.

The bastnäsite was heavily contaminated with fluocerite and cerianite and it was not possible to obtain very accurate powder data since the peaks were weak and several coincided with cerianite or fluocerite peaks, but the data obtained closely match those for bastnäsite from Mountain Pass, California (Glass et al., 1958), and give cell dimensions a = 7.131 ± 0.002, c = 9.786 ± 0.004 Å.

**Figure 3.** Th vs. Ce for dark-brown alteration products. Symbols as fig. 2.

**Table IV. Electron microprobe analyses of dark brown alteration products**

<table>
<thead>
<tr>
<th></th>
<th>1</th>
<th>2</th>
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<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
</tr>
</thead>
<tbody>
<tr>
<td>La₂O₃</td>
<td>32.83</td>
<td>23.16</td>
<td>17.36</td>
<td>6.65</td>
<td>2.70</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CeO₂</td>
<td>27.74</td>
<td>40.46</td>
<td>52.18</td>
<td>67.49</td>
<td>60.80</td>
<td>80.5</td>
<td>1.705</td>
</tr>
<tr>
<td>Pr₂O₃</td>
<td>3.20</td>
<td>2.92</td>
<td>3.86</td>
<td>1.76</td>
<td>1.79</td>
<td>2.5</td>
<td>0.058</td>
</tr>
<tr>
<td>Nd₂O₃</td>
<td>9.27</td>
<td>7.33</td>
<td>8.79</td>
<td>6.48</td>
<td>5.29</td>
<td>7.5</td>
<td>0.160</td>
</tr>
<tr>
<td>Sm₂O₃</td>
<td>nd</td>
<td>nd</td>
<td>0.35</td>
<td>0.40</td>
<td>0.46</td>
<td></td>
<td></td>
</tr>
<tr>
<td>CaO</td>
<td>0.48</td>
<td>0.59</td>
<td>1.30</td>
<td>0.95</td>
<td>0.95</td>
<td>0.69</td>
<td></td>
</tr>
<tr>
<td>ThO₂</td>
<td>2.86</td>
<td>4.74</td>
<td>4.97</td>
<td>6.41</td>
<td>11.23</td>
<td>9.5</td>
<td>0.131</td>
</tr>
<tr>
<td>SiO₂</td>
<td>—</td>
<td>—</td>
<td>1.00</td>
<td>—</td>
<td>—</td>
<td>4.45</td>
<td></td>
</tr>
</tbody>
</table>

nd Not detected.

1-4. Cerianite-bastnäsite-(La) mixtures.
5. Cerianite-bastnäsite-(La) mixtures with thorite.
7. Number of ions on basis of four oxygens.
The cerianite peaks were broad, indicating a variable composition. The cerianite has a mean cell dimension \( a = 5.460 \pm 0.005 \text{Å} \).

**Discussion**

The fluocerite-monazite pebbles show that the alteration of fluocerite takes place by two distinct processes. The first is carbonation to form the fluorocarbonate bastnäsite as is described from most of the reported occurrences of fluocerite. This involves very little mobilization of the RE as shown by the very similar RE distribution patterns and the Ce-La diagram (fig. 1 and 2). In this particular instance the Th in the fluocerite is not incorporated in the bastnäsite and presumably forms a separate Th phase, possibly thorite or thorianite (ThO₂). A simplified chemical reaction is

\[
\text{Ce}_2\text{La}_{1.5}(\text{Nd,Pr,Th})_0.5(\text{CO}_3)_4\text{F}_4 + \text{fluid} \rightarrow \text{La}_{1.5}(\text{Nd,Pr})_0.5(\text{CO}_3)_2\text{F}_2 + 2(\text{Ce}^{4+},\text{Th})\text{O}_2 + \text{fluid.}
\]

\[
\text{Ce}^3+\text{La}_{1.5}(\text{Nd,Pr,Th})_0.5F_{12} + \text{fluid} \rightarrow \text{Ce}_{2}\text{La}_{1.5}(\text{Nd,Pr,Th})_0.5(\text{CO}_3)_2\text{F}_4 + \text{fluid}.
\]

The cerianites have approximately the same Ce/Th ratio as the fluocerite (fig. 3) perhaps indicating that the first reaction is more common as it does not require separate Th phase to be recombined in exactly the same proportions which precludes mobility of chemical components during alteration.

Further evidence that Ce and La can behave differently during alteration is shown by the analyses given by Chistyakova and Kazakova (1969), Table II, cols. 9 and 10, where there is a marked change in the Ce/La ratio from 0.78 in the fresh sample to 2.75 in the altered sample. The Ce-rich altered sample is similar in composition to some of the cerianite-rich dark-brown alteration products described in this paper.

**Acknowledgements.** We thank Mr R. K. Harrison for bringing these interesting specimens to our notice and for helpful discussion and assistance during the course of the
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and for bringing to our attention many of the analyses given
in Table II. This paper is published by permission of the
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455-9.
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Palache, C., Berman, H., and Frondel, C. (1951) Dana’s
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Vetoshkina, A. M., Gordienko, V. V., Elina, N. A., and
(in Russian).

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APPENDIX

Microanalysis techniques. The microprobe analyses
were made using a Link Systems energy-dispersive (ED)
X-ray analyser on a Cambridge Instruments Geoscan
electron microprobe. An accelerating voltage of 15 kV, a
specimen current of 5 × 10^{-9} amps and an electron beam
focused to approximately 5 μm were used throughout.
The ED X-ray detector has a resolution of around 165 eV
(FWHM) for Co-Kα under the analytical conditions used

with an input count of approximately 3.5 × 10^{3} counts per
second and a dead time around 25%. All measurements
were made using a 100 sec live time counting period.

Data processing was carried out using the Link Systems
‘ZAF4 FLS+’ program and calibration for RE was made
using synthetic single crystals of LaB_{6}, CeO_{2}, PrAlO_{3},
NdAlO_{3}, and SmAlO_{3}. Tests have shown that the ED
system can adequately deal with the overlapping spectra
of the RE L lines and the transition metal K lines. The
limit of detection for RE in overlapping spectra is
approximately 0.35 wt. % element for the elements
reported here. The detection limits for the heavier RE are
slightly higher due to the less efficient excitation and it is
preferable to use a higher accelerating voltage.

The glass REE3 produced by Drake and Well (1972)
was analysed using the same instrumental conditions as
the minerals and the results are given in Table VI. The
average of four points, widely spaced on the glass
specimen, approximately 1.5 mm in diameter (col. 1), is in
close agreement with the stated composition (col. 5). One
point on the glass was also analysed four times to give an
indication of the precision of the analyses and the results
are given in col. 3. One of these four analyses has a high
total, 101.9% (probably due to beam intensity drift) and
this causes the standard deviations to be higher than
might be expected; if the analyses are normalized to 100%
before the standard deviation is calculated the RE
standard deviation would be approximately 0.15. This is
probably a better indication of the precision of the
analysis of RE using an E.D. spectrometer.

It should also be noted that the concentrations of Y and
Ce in col. 3 and col. 1 are significantly different. It has been
found that the RE glasses of Drake and Well are not
homogeneous on a micro scale and that when they are
analysed using a finely focused electron beam (5 μm) the
concentration of RE and Y can vary by ±10% (relative), 0.5 wt. % oxide from the given concentrations. The bulk
composition is very close to the given composition (cols.
1 and 5).