



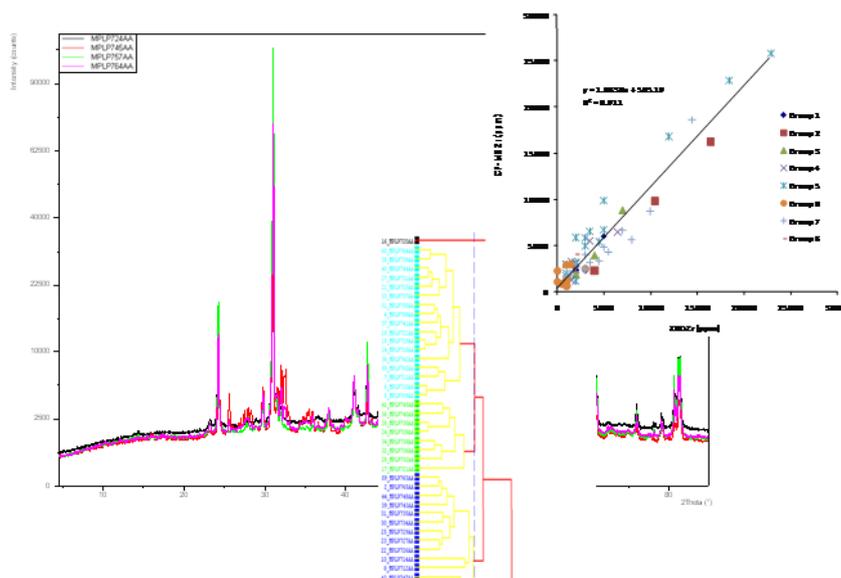
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# Mineralogical analysis of further stream sediments from Nigeria

Mineralogy, Petrology & Biostratigraphy Facility

Internal Report IR/10/083





BRITISH GEOLOGICAL SURVEY

MINERALOGY, PETROLOGY & BIOSTRATIGRAPHY FACILITY  
INTERNAL REPORT IR/10/083

# Mineralogical analysis of further stream sediments from Nigeria

S J Kemp, D Wagner and I Mounteney

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Montage of XRD traces, cluster dendrogram and geochemical cross-plot.

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# 1 Introduction

This report presents the results of mineralogical characterisation of a further suite of sixty stream sediment samples from Nigeria. The samples were submitted for analysis by Dr Roger Key (BGS) and his team as part of the 'Technical Assistance Services for the Geochemical Mapping of Nigeria' project which aims to provide baseline geoscientific information for mineral exploration and environmental management through a study of the distribution of important metallic elements.

Particular interest was expressed in determining the hosts for the elevated levels of Zr in the stream sediments. Full sample details, including Zr geochemical data from inductively coupled plasma-mass spectrometry (ICP-MS) are listed in Table 1.

This study follows a smaller pilot study carried out on a suite of seven stream sediments samples from the same project (Kemp *et al.*, 2009).

## 2 X-ray diffraction analysis

### 2.1 PREPARATION

In order to achieve a finer and uniform particle-size for powder XRD analysis, approximately 5 g portions of the tema-milled material were micronised under deionised water for 10 minutes with 10 % (i.e. 0.5 g) corundum (American Elements, Al<sub>2</sub>O<sub>3</sub>, AL-OX-03-P). The addition of an internal standard allows validation of quantification data and also the detection of any amorphous species in the samples. Corundum was selected as its principle XRD peaks are suitably remote from those produced by most of the phases present in the samples and its mass absorption coefficient is similar to the sample matrix.

The corundum-spiked samples were then spray-dried following the method and apparatus described by Hillier (1999). The spray-dried materials were then front-loaded into a standard stainless steel sample holders for analysis.

### 2.2 ANALYSIS

Whole-rock XRD analysis was carried out using a PANalytical X'Pert Pro series diffractometer equipped with a cobalt-target tube, X'Celerator detector and operated at 45kV and 40mA. The micronised/spray-dried samples were scanned from 4.5-85°2 $\theta$  at 2.76°2 $\theta$ /minute. Diffraction data were initially analysed using PANalytical X'Pert Highscore Plus version 2.2a software coupled to the latest version of the International Centre for Diffraction Data (ICDD) database.

### 2.3 QUANTIFICATION

Following identification of the mineral species present in the sample, mineral quantification was achieved using the Rietveld refinement technique (e.g. Snyder & Bish, 1989) using PANalytical Highscore Plus software. This method avoids the need to produce synthetic mixtures and involves the least squares fitting of measured to calculated XRD profiles using a crystal structure databank. Errors for the quoted mineral concentrations are typically  $\pm 2.5\%$  for concentrations  $>60$  wt%,  $\pm 5\%$  for concentrations between 60 and 30 wt%,  $\pm 10\%$  for concentrations between 30 and 10 wt%,  $\pm 20\%$  for concentrations between 10 and 3 wt% and  $\pm 40\%$  for concentrations  $<3$  wt% (Hillier *et al.*, 2001). Where a phase was detected but its concentration was indicated to be below 0.5%, it is assigned a value of  $<0.5\%$ , since the error associated with quantification at such low levels becomes too large.

**Table 1. Summary of samples submitted**

| Incoming sample number | BGS MPL code | UTM Zone | GRN cell | Easting | Northing | Site lithology (where outcrop)            | Catchment geology                         | ICP-MS Zr (ppm) |
|------------------------|--------------|----------|----------|---------|----------|---|---|-----------------|
| 2                      | MPLP706      | 31N      | SW       | 727691  | 822731   | -   | Migmatitic Gneisses                       | 6460            |
| 3                      | MPLP707      | 31N      | SW       | 732208  | 815530   | -   | Older Granites                            | 4308            |
| 17                     | MPLP708      | 31N      | SW       | 820959  | 806068   | Dark gray schist cut by migmatitic gneiss | Migmatitic Gneisses                       | 2982            |
| 22                     | MPLP709      | 31N      | SW       | 812546  | 814264   | Migmatitic granite gneiss                 | Metasedimentary and Metavolcanic          | 25768           |
| 32                     | MPLP710      | 31N      | SW       | 783761  | 781501   | Biotite schist/quartzite                  | Migmatitic Gneisses                       | 22853           |
| 50                     | MPLP711      | 31N      | SW       | 736069  | 775130   | Augen granite gneiss                      | Older Granites                            | 16789           |
| 78                     | MPLP712      | 31N      | SW       | 790953  | 806052   | Migmatitic gneiss                         | Migmatitic Gneisses                       | 3024            |
| 79                     | MPLP713      | 31N      | SW       | 576176  | 779121   | -   | Metasedimentary and Metavolcanic          | 16241           |
| 85                     | MPLP714      | 31N      | SW       | 607940  | 779750   | -   | Metasedimentary and Metavolcanic          | 2431            |
| 147                    | MPLP715      | 31N      | SW       | 577675  | 771404   | -   | Metasedimentary and Metavolcanic          | 8806            |
| 148                    | MPLP716      | 31N      | SW       | 618978  | 763114   | Granite gneiss                            | Metasedimentary and Metavolcanic          | 5644            |
| 164                    | MPLP717      | 31N      | SW       | 625003  | 768118   | Granite gneiss                            | Older Granites                            | 8729            |
| 168                    | MPLP718      | 31N      | SW       | 516621  | 824537   | -   | Metasedimentary and Metavolcanic          | 6535            |
| 184                    | MPLP719      | 31N      | SW       | 521548  | 822374   | -   | Migmatitic Gneisses                       | 9865            |
| 198                    | MPLP720      | 31N      | SW       | 561327  | 759692   | -   | Mesozoic and Younger strata and sediments | 4033            |
| 225                    | MPLP721      | 31N      | SW       | 551017  | 867734   | Migmatitic gneiss                         | Migmatitic Gneisses                       | 2961            |
| 228                    | MPLP722      | 31N      | SW       | 550114  | 830924   | -   | Migmatitic Gneisses                       | 3054            |
| 253                    | MPLP723      | 31N      | SW       | 509294  | 754516   | -   | Mesozoic and Younger strata and sediments | 1893            |
| 254                    | MPLP724      | 31N      | SW       | 537205  | 782905   | -   | Migmatitic Gneisses                       | 3935            |
| 279                    | MPLP725      | 31N      | SW       | 519730  | 843483   | -   | Migmatitic Gneisses                       | 5832            |

Table 1(continued). Summary of samples submitted

| Incoming sample number | BGS MPL code | UTM Zone | GRN cell | Easting | Northing | Site lithology (where outcrop)           | Catchment geology                         | ICP-MS Zr (ppm) |
|------------------------|--------------|----------|----------|---------|----------|--|---|-----------------|
| 319                    | MPLP726      | 32N      | Minna    | 271295  | 1077098  | -  | Migmatitic Gneisses                       | 3328            |
| 332                    | MPLP727      | 32N      | Minna    | 267314  | 1070102  | -  | Migmatitic Gneisses                       | 3168            |
| 337                    | MPLP728      | 32N      | Minna    | 249690  | 1046574  | -  | Older Granites                            | 849             |
| 365                    | MPLP729      | 32N      | Minna    | 272132  | 1075231  | -  | Migmatitic Gneisses                       | 2360            |
| 368                    | MPLP730      | 32N      | Minna    | 249054  | 1046757  | Crse-grained granite/pegmatite intrusion | Older Granites                            | 1075            |
| 396                    | MPLP731      | 32N      | Minna    | 273528  | 1054128  | Porphyritic granite                      | Older Granites                            | 4973            |
| 423                    | MPLP732      | 32N      | Minna    | 217957  | 1089221  | -  | Older Granites                            | 2513            |
| 474                    | MPLP733      | 32N      | Minna    | 243422  | 1015157  | -  | Mesozoic and Younger strata and sediments | 9817            |
| 513                    | MPLP734      | 32N      | Minna    | 263566  | 1062462  | -  | Older Granites                            | 2560            |
| 539                    | MPLP735      | 32N      | Minna    | 278328  | 1079211  | Biotite granite (coarse-grained)         | Migmatitic Gneisses                       | 4022            |
| 568                    | MPLP736      | 32N      | Minna    | 209679  | 1059161  | Granite-biotite, hornblende, feldspar    | Older Granites                            | 2245            |
| 681                    | MPLP737      | 32N      | Minna    | 269262  | 1058571  | -  | Older Granites                            | 1175            |
| 715                    | MPLP738      | 32N      | Minna    | 213750  | 1077861  | Granite                                  | Older Granites                            | 647             |
| 814                    | MPLP739      | 32N      | Minna    | 178995  | 1090114  | Phyllite                                 | Metasedimentary and Metavolcanic rocks    | 3172            |
| 828                    | MPLP740      | 32N      | Minna    | 299490  | 1097699  | -  | Older Granites                            | 2905            |
| 830                    | MPLP741      | 32N      | Minna    | 290541  | 1103146  | Amphibolite schist                       | Migmatitic Gneisses                       | 1988            |
| 848                    | MPLP742      | 32N      | Minna    | 261352  | 1121494  | Fine- to medium-grained granite          | Older Granites                            | 835             |
| 850                    | MPLP743      | 32N      | Minna    | 317820  | 1086233  | Granite                                  | Migmatitic Gneisses                       | 4810            |
| 851                    | MPLP744      | 32N      | Minna    | 194032  | 1097623  | Schist with a lot of quartz vein         | Zungeru Mylonites                         | 1724            |
| 888                    | MPLP745      | 32N      | Minna    | 284535  | 1104423  | Exposure of pegmatite along stream       | Migmatitic Gneisses                       | 1285            |

Table 1(continued). Summary of samples submitted

| Incoming sample number | BGS MPL code | UTM Zone | GRN cell | Easting | Northing | Site lithology (where outcrop)      | Catchment geology   | ICP-MS Zr (ppm) |
|------------------------|--------------|----------|----------|---------|----------|-------------------------------------|---------------------|-----------------|
| 909                    | MPLP746      | 32N      | Minna    | 321315  | 1080470  | Granite gneiss                      | Migmatitic Gneisses | 1272            |
| 981                    | MPLP747      | 32N      | Minna    | 254937  | 997118   | Grey gneiss                         | Migmatitic Gneisses | 18569           |
| 990                    | MPLP748      | 32N      | Minna    | 188200  | 1092700  | Quartz schist                       | Zungeru Mylonites   | 1853            |
| 1005                   | MPLP749      | 32N      | Minna    | 319785  | 1109491  | Metamorphic gneiss                  | Migmatitic Gneisses | 2393            |
| 1007                   | MPLP750      | 32N      | Minna    | 319754  | 1109495  | -                                   | Migmatitic Gneisses | 3156            |
| 1022                   | MPLP751      | 32N      | Minna    | 324228  | 1122077  | Amphibolite-gneiss                  | Migmatitic Gneisses | 5983            |
| 1024                   | MPLP752      | 32N      | Minna    | 317241  | 1122534  | -                                   | Older Granites      | 5506            |
| 1031                   | MPLP753      | 32N      | Minna    | 321533  | 1108211  | Metamorphic: gneiss                 | Older Granites      | 2373            |
| 1038                   | MPLP754      | 32N      | Minna    | 297224  | 1107902  | -                                   | Migmatitic Gneisses | 2308            |
| 1041                   | MPLP755      | 32N      | Minna    | 298480  | 1109730  | Migmatitic-gneiss (biotite rich)    | Older Granites      | 5869            |
| 1052                   | MPLP756      | 32N      | Minna    | 329040  | 1098319  | Migmatite-gneiss                    | Migmatitic Gneisses | 3000            |
| 1105                   | MPLP757      | 32N      | Minna    | 322963  | 1096533  | Migmatite schists                   | Migmatitic Gneisses | 2300            |
| 1109                   | MPLP758      | 32N      | Minna    | 305985  | 1146366  | Granite: medium- to coarse-grained. | Older Granites      | 2301            |
| 1116                   | MPLP759      | 32N      | Minna    | 327129  | 1116024  | Migmatitic gneiss                   | Migmatitic Gneisses | 1070            |
| 1127                   | MPLP760      | 32N      | Minna    | 330562  | 1079180  | -                                   | Migmatitic Gneisses | 3256            |
| 1142                   | MPLP761      | 32N      | Minna    | 332117  | 1090716  | Pegmatitic gneiss                   | Migmatitic Gneisses | 2270            |
| 1413                   | MPLP762      | 32N      | Minna    | 322657  | 1014466  | Granite                             | Older Granites      | 6713            |
| 1553                   | MPLP763      | 32N      | Minna    | 316180  | 1002689  | -                                   | Migmatitic Gneisses | 1748            |
| 1623                   | MPLP764      | 32N      | Minna    | 321667  | 1003306  | -                                   | Migmatitic Gneisses | 5406            |
| 1633                   | MPLP765      | 32N      | Minna    | 322358  | 1005818  | Biotite gneiss                      | Migmatitic Gneisses | 6621            |

### 3 Results and discussion

The quantitative results of powder XRD analyses are summarised in Table 2.

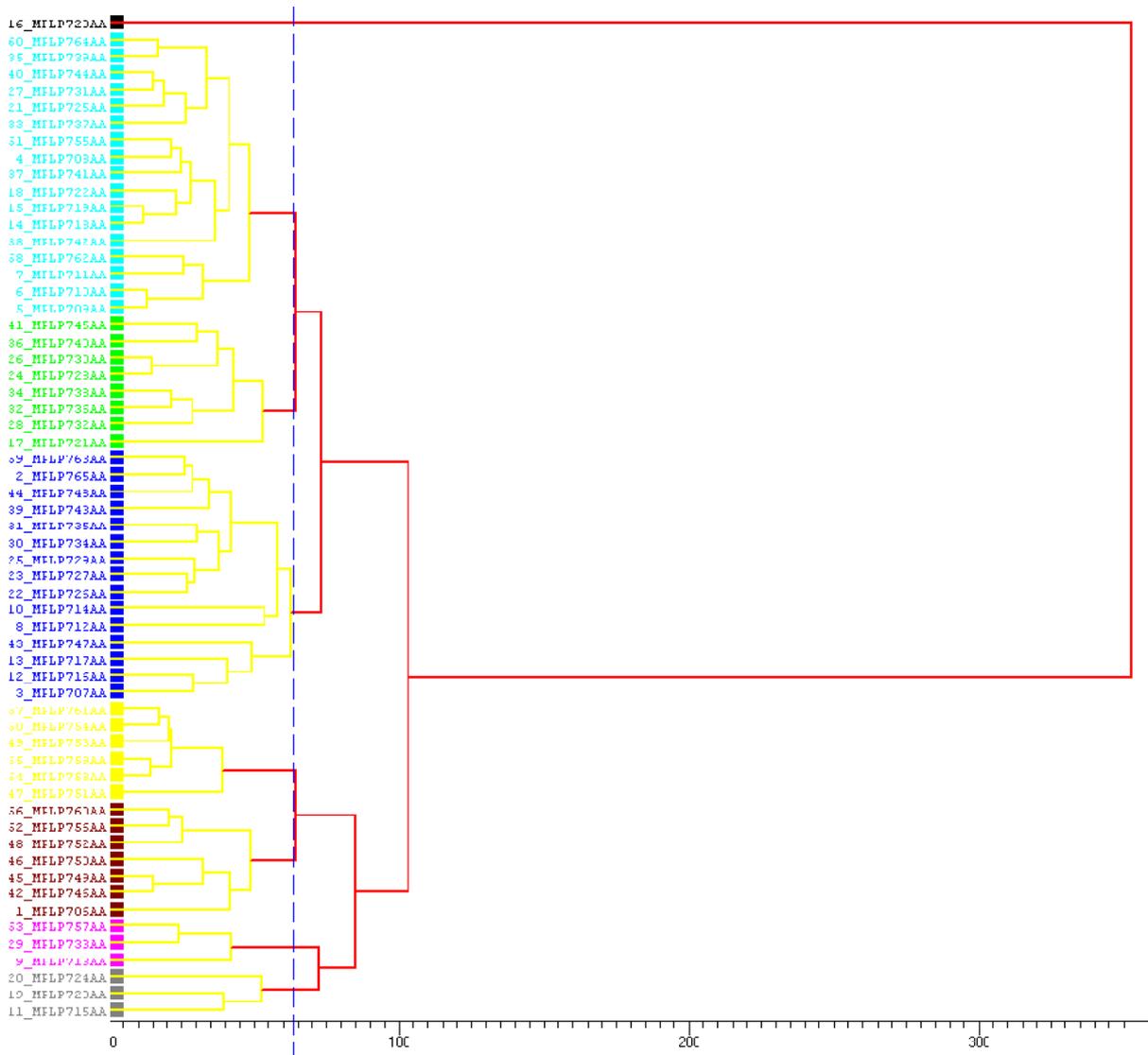
XRD analysis indicates that the samples are predominantly composed of quartz with subordinate amounts of feldspar (plagioclase and K-feldspar), 'mica' (undifferentiated mica species possibly including muscovite, biotite, illite and illite/smectite), 'kaolin' (undifferentiated kaolin group minerals possibly including kaolinite, halloysite etc)  $\pm$  traces of ilmenite, zircon, amphibole, epidote, hematite, monazite, sillimanite, pyroxene and anatase.

XRD analysis (Table 2) suggests that two different plagioclase compositions are present in the samples, one slightly more calcic than the other (plagioclase 1;  $\text{Na}_{0.84}\text{Ca}_{0.16}\text{Al}_{1.16}\text{Si}_{2.84}\text{O}_8$  and plagioclase 2;  $\text{Na}_{0.75}\text{Ca}_{0.25}\text{Al}_{1.26}\text{Si}_{2.74}\text{O}_8$ ). Similarly two different K-feldspar species were identified (K-feldspar 1; microcline and K-feldspar 2; orthoclase).

Cluster analysis of the powder diffraction data, displayed as a dendrogram (Figure 1), indicates eight distinct mineral assemblages:

- Group 1 samples (shown in yellow, sites 1022, 1031, 1038, 1109, 1116, 1142) are characterised by relatively high quartz (62-78%), moderate amounts of 'kaolin' (8-12%), K-feldspar (8-19%) and 'mica' (4-5%) and trace amounts of plagioclase, ilmenite and zircon. No ferromagnesian silicate minerals (pyroxene, epidote, amphibole) were detected in these samples. The catchment geology for these samples are predominantly migmatitic gneisses with occasional older granites. The moderate 'kaolin' concentrations together with a low plagioclase content and a lack of ferromagnesian minerals would suggest that these sediments represent relatively weathered material.
- Group 2 samples (shown in magenta, sites 79, 474 and 1105) are characterised by the highest quartz contents (65-88%) for the sample suite with variable but usually moderate amounts of plagioclase feldspar (nd-10%), ilmenite (2-6%), 'kaolin' (3-10%), K-feldspar (5-7%) and occasional anatase, 'mica', amphibole and monazite. Moderate/high zircon (1-3%) and no ferromagnesian silicate minerals. The catchment geology is highly variable and composed of sedimentary/metasedimentary/metavolcanic/migmatitic gneiss lithologies.
- Group 3 samples (shown in grey, sites 147, 253 and 254) are again characterised by high quartz (66-87%, similar to Group 2), variable but generally high 'kaolin' contents (5-22%), subordinate amounts of K-feldspar (3-5%) and plagioclase (2-4%) and occasional traces of anatase, ilmenite, amphibole and zircon. No 'mica' species were identified in these samples. As for the Group 2 samples, Group 3 samples are also characterised by variable catchment lithologies composed of sedimentary/metasedimentary/metavolcanic/migmatitic gneiss lithologies.
- Group 4 samples (shown in brown, sites 2, 909, 1005, 1007, 1024, 1052 and 1127) are characterised by relatively moderate quartz (56-78%) and K-feldspar (12-16%) contents, variable plagioclase (1-11%) and 'kaolin' (3-12%), subordinate 'mica' (nd-6%)  $\pm$  traces of zircon, pyroxene, ilmenite, amphibole, monazite and sillimanite. With the exception of one sample (site 1024), the catchment geology for these samples is exclusively formed of migmatitic gneisses.
- Group 5 samples (shown in cyan, sites 17, 22, 32, 50, 168, 184, 228, 279, 396, 681, 814, 830, 848, 851, 1041, 1413 and 1623) are most common and are generally characterised by low-medium quartz (34-72%), high K-feldspar (16-36%), moderate plagioclase (3-18%), amphibole (nd-6%), 'mica' (nd-6%), 'kaolin' (nd-4%) and traces of ilmenite,

monazite, zircon (<0.5-5%). The catchment geology for the Group 5 samples is mixed between migmatitic gneisses and older granites with some metasedimentary/metavolcanic input. The higher feldspar and lower 'kaolin' content of these samples suggests that they represent fresher, less weathered material.



**Figure 1. Cluster analysis dendrogram for the stream sediment XRD traces**

- Group 6 samples (shown in green, sites 225, 337, 368, 423, 568, 715, 828 and 888) are characterised by low-moderate quartz (25-55%), high K-feldspar (16-36%), high plagioclase (18-33%) and subordinate 'mica' (3-8%). The samples are unusually characterised by minor amounts of epidote (nd-6%), occasional amphibole (nd-3%) and low zircon concentrations. Group 6 samples are typically characterised by older granite catchment lithologies with occasional migmatitic gneiss. The relatively low quartz and 'mica' content together with high feldspar (K-feldspar and plagioclase) contents are in agreement with the predicted granite-rich catchment. The presence of epidote and occasional amphiboles suggest some metamorphic alteration of the granites.
- Group 7 samples (shown in blue, sites 3, 78, 85, 148, 164, 319, 332, 365, 513, 539, 850, 981, 990, 1553 and 1633) are characterised by moderate-high amounts of quartz (48-

82%), moderate but variable K-feldspar (4-23%) and plagioclase (3-23%) and relatively high ilmenite (2-10%) contents. Moderate amounts of 'mica' (nd-11%) and 'kaolin' (nd-8%) were found together with occasional epidote (nd-7%), sillimanite (nd-5%), monazite (nd-1%), pyroxene (nd-1%), hematite (nd-2%) and zircon (<0.5-3%). The Group 7 samples are therefore similar to those in Group 6 but the Group 7 samples contain more Fe-bearing minerals (e.g. ilmenite, hematite). The catchments for these samples is again dominated by migmatitic gneiss with a few catchments of older granite and metasedimentary/ metavolcanic rocks.

- The single remaining sample (Group 8, shown in black, site 198) is clearly very different to all the other stream sediments. XRD analysis suggest that it has a very quartz-rich mineralogy (95%). Its catchment geology is formed of Mesozoic and younger strata and sediments.

Figure 1 indicates that the above defined sample groupings can be further combined into two general clusters. Groups 1, 2, 3 and 4 show broadly similar mineralogical characteristics of moderate-high amounts of quartz, low-moderate amounts of K-feldspar (microcline, no orthoclase), plagioclase and 'kaolin' together with low/no amounts of 'mica' and ferromagnesian minerals. Zircon concentrations in Groups 1-4 are variable (<0.5-3.3%). The catchment lithologies for Groups 1-4 are also variable but are predominantly formed of migmatitic gneiss.

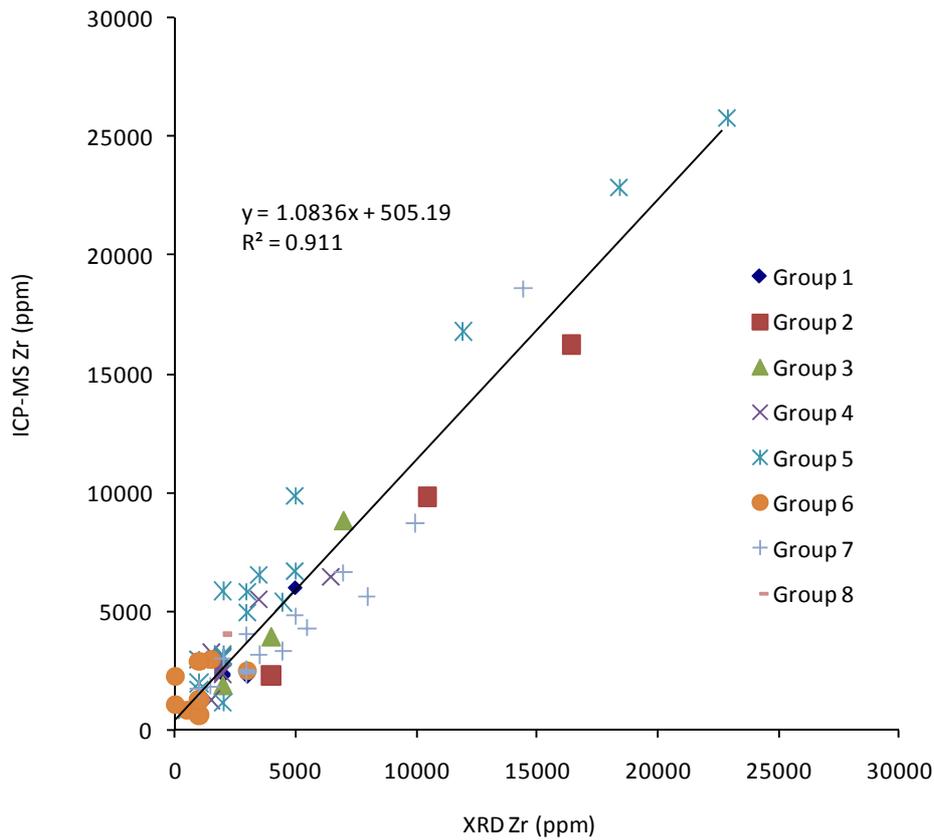
Groups 5, 6 and 7 also show similar mineralogical features and are composed of low-moderate amounts of quartz, high amounts of K-feldspar (microcline and orthoclase) and plagioclase, moderate but variable amounts of 'mica', 'kaolin' and ilmenite together with occasional traces of epidote, amphibole, sillimanite and monazite. Zircon concentrations in Groups 5-7 are variable (nd-4.6%). The catchment lithologies for Groups 5-7 are formed of a mixture of old granites and migmatitic gneiss.

### 3.1 COMPARISON WITH ICP-MS GEOCHEMISTRY

It is interesting and useful to compare the results of mineralogical XRD analyses with the project ICP-MS geochemical data but a detailed interpretation is outside the scope of this limited investigation.

In terms of Zr distribution, the main aim of this study, the only Zr-bearing phase identified by XRD in the sixty stream sediment samples was zircon ( $\text{ZrSiO}_4$ ). XRD concentrations show a good correlation with geochemical data (Figure 2,  $R^2 = 0.91$ ). This concurs with the previous pilot study (Kemp *et al.*, 2009). The lower limit of zircon detection for XRD would appear to be between 500 and 1000 ppm Zr, again similar to the previous study. Zr concentrations derived from the XRD zircon contents therefore confirm that zircon is the dominant host for Zr in the stream sediments. As stated by Kemp *et al.* (2009), more accurate speciation of the heavy mineral fraction and potential identification of further Zr-bearing phases could be achieved if the quartz and feldspar component was removed using heavy media (e.g. Li-polytungstate) separation techniques prior to further XRD analysis.

Figure 2 illustrates that although the highest zircon concentrations are found in samples from Group 5, 7 and 2, each grouping shows highly variable zircon contents. Similarly the catchment lithology for these highest zircon contents varies from metasedimentary/metavolcanic to migmatitic gneiss and older granite. It would therefore appear that the zircon content of the stream sediments is not indicative of any one particular catchment lithology.

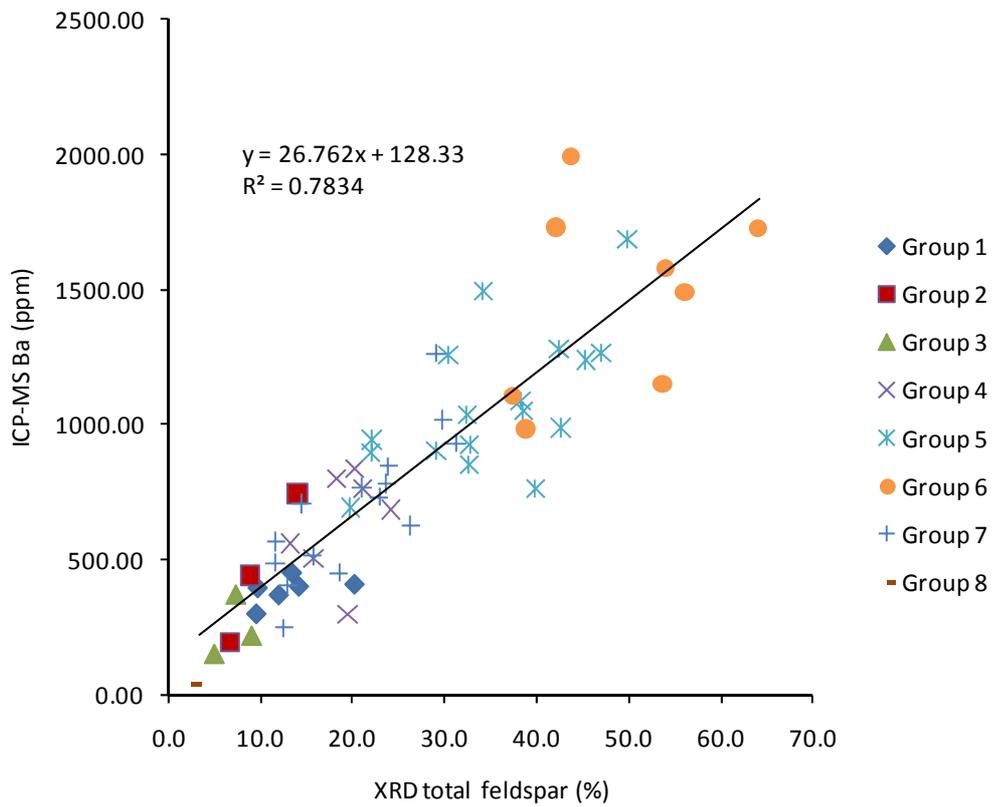


**Figure 2. Correlation plot of XRD Zr (from zircon concentration) and ICP-MS Zr (both ppm) with data grouped according to XRD clusters**

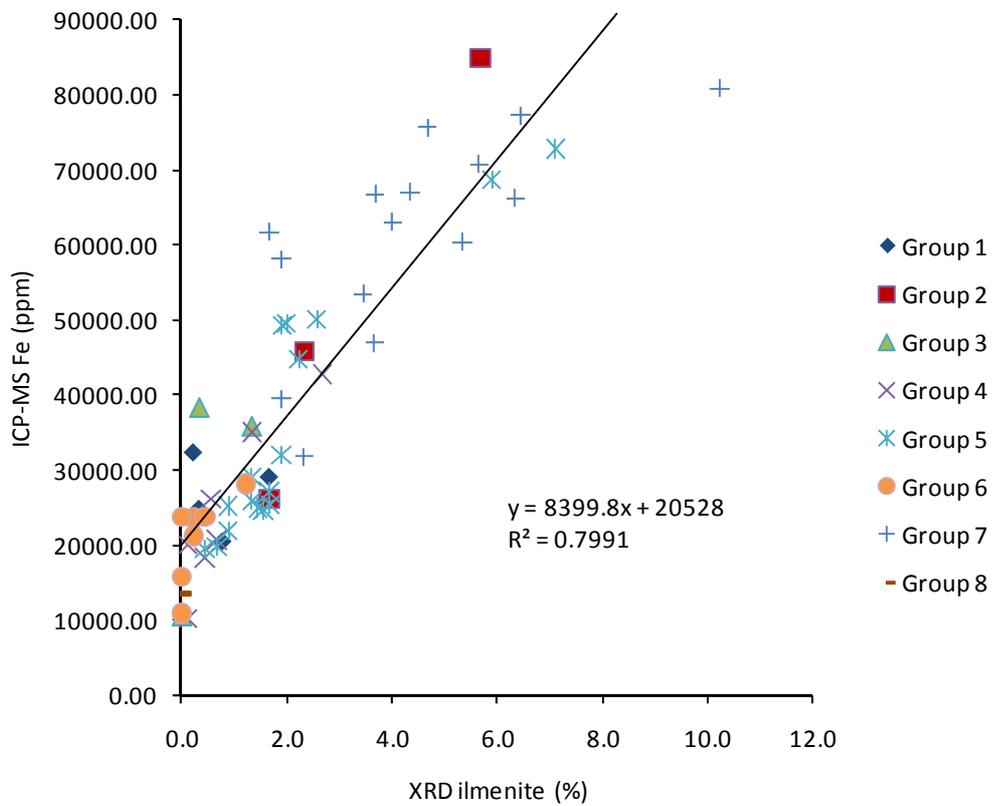
Other interesting correlations between mineralogy and geochemistry include the strong relationship between XRD total feldspar content and Ba with an  $R^2 = 0.78$  (Figure 3). Ba is present in the majority of alkali feldspars and to a lesser degree in plagioclase (Deer *et al.*, 1996). Figure 3 graphically illustrates the difference in feldspar content described in the previous section; Groups 5, 6 and 7 being feldspar-rich and Groups 1-4 and 8 being feldspar-poor.

Ilmenite is the dominant Fe-bearing mineral in the stream sediments, as illustrated by the cross-plot in Figure 4. The Fe-rich nature of the Group 7 samples is very evident from this plot.

Although not illustrated here, it is also noticeable that the XRD-identified monazite concentrations show an excellent correlation with the rare earth element and phosphorus concentrations determined for the samples.



**Figure 3. Correlation plot of XRD total feldspar (%) and ICP-MS Ba (ppm) with data grouped according to XRD clusters**



**Figure 4. Correlation plot of XRD ilmenite (%) and ICP-MS Fe (ppm) with data grouped according to XRD clusters**

Table 2. Summary of quantitative powder XRD analyses

| Site no. | BGS MPL code | Silicates |                |                |               |               |          |           |         |             |        | Oxides, phosphates |          |          |          | Phyllosilicates |          |
|----------|--------------|-----------|----------------|----------------|---------------|---------------|----------|-----------|---------|-------------|--------|--------------------|----------|----------|----------|-----------------|----------|
|          |              | quartz    | *plagioclase 1 | *plagioclase 2 | *K-feldspar 1 | *K-feldspar 2 | pyroxene | amphibole | epidote | sillimanite | zircon | anatase            | hematite | ilmenite | monazite | 'mica'          | 'kaolin' |
| 2        | MPLP706      | 55.7      | nd             | 5.0            | 16.1          | nd            | nd       | nd        | nd      | 2.8         | 1.3    | nd                 | nd       | 2.7      | nd       | 4.7             | 11.7     |
| 3        | MPLP707      | 51.8      | nd             | 7.0            | 22.8          | nd            | nd       | nd        | nd      | 2.9         | 1.1    | nd                 | nd       | 6.3      | nd       | 3.3             | 4.8      |
| 17       | MPLP708      | 58.5      | nd             | 6.5            | 20.8          | 6.8           | nd       | 2.0       | nd      | nd          | <0.5   | nd                 | nd       | <0.5     | <0.5     | 2.7             | 1.8      |
| 22       | MPLP709      | 33.8      | nd             | 17.5           | 23.9          | 5.6           | nd       | 5.7       | nd      | nd          | 4.6    | nd                 | nd       | 1.9      | nd       | 3.7             | 3.4      |
| 32       | MPLP710      | 43.3      | nd             | 15.8           | 21.1          | 8.4           | nd       | 2.3       | nd      | nd          | 3.7    | nd                 | nd       | 1.7      | <0.5     | 3.7             | nd       |
| 50       | MPLP711      | 44.6      | nd             | 6.5            | 24.1          | 7.9           | nd       | 0.9       | nd      | nd          | 2.4    | nd                 | nd       | 7.1      | nd       | 3.3             | 3.1      |
| 78       | MPLP712      | 57.8      | nd             | 8.9            | 12.3          | 2.4           | nd       | nd        | nd      | 5.0         | <0.5   | nd                 | 2.3      | 1.9      | 1.1      | 6.2             | 1.4      |
| 79       | MPLP713      | 65.0      | nd             | 7.2            | 6.8           | nd            | nd       | 2.1       | nd      | nd          | 3.3    | nd                 | nd       | 5.7      | <0.5     | nd              | 9.5      |
| 85       | MPLP714      | 61.7      | nd             | 11.7           | 11.2          | nd            | nd       | nd        | nd      | nd          | 0.6    | nd                 | 1.3      | 1.7      | nd       | 4.6             | 7.3      |
| 147      | MPLP715      | 65.8      | nd             | 4.2            | 4.8           | nd            | nd       | nd        | nd      | nd          | 1.4    | 0.8                | nd       | 1.3      | nd       | nd              | 21.6     |
| 148      | MPLP716      | 56.4      | nd             | 16.4           | 9.9           | nd            | nd       | 3.7       | nd      | nd          | 1.6    | nd                 | nd       | 5.3      | nd       | nd              | 6.7      |
| 164      | MPLP717      | 55.8      | nd             | 14.0           | 15.1          | nd            | 0.9      | 2.0       | nd      | nd          | 2.0    | nd                 | nd       | 10.2     | nd       | nd              | nd       |
| 168      | MPLP718      | 53.5      | nd             | 13.6           | 20.2          | 8.7           | nd       | 1.2       | nd      | nd          | 0.7    | nd                 | nd       | 1.7      | 0.6      | nd              | nd       |
| 184      | MPLP719      | 51.6      | nd             | 14.3           | 22.0          | 6.4           | nd       | nd        | nd      | nd          | 1.0    | nd                 | nd       | 1.4      | 0.6      | 2.8             | nd       |
| 198      | MPLP720      | 94.9      | nd             | nd             | 2.7           | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | nd       | nd       | nd              | 2.0      |
| 225      | MPLP721      | 24.8      | nd             | 32.9           | 16.1          | 15.0          | nd       | <0.5      | nd      | nd          | <0.5   | nd                 | nd       | <0.5     | nd       | 3.8             | 6.7      |
| 228      | MPLP722      | 45.3      | nd             | 14.4           | 30.6          | 4.9           | nd       | 0.6       | nd      | nd          | <0.5   | nd                 | nd       | 0.7      | nd       | 3.2             | nd       |
| 253      | MPLP723      | 77.3      | nd             | 1.8            | 3.2           | nd            | nd       | 1.0       | nd      | nd          | <0.5   | 0.8                | nd       | nd       | nd       | nd              | 15.5     |

Table 3. Summary of quantitative powder XRD analyses (continued)

| Site no. | BGS MPL code | Silicates |                |                |               |               |          |           |         |             |        | Oxides, phosphates |          |          |          | Phyllosilicates |          |
|----------|--------------|-----------|----------------|----------------|---------------|---------------|----------|-----------|---------|-------------|--------|--------------------|----------|----------|----------|-----------------|----------|
|          |              | quartz    | *plagioclase 1 | *plagioclase 2 | *K-feldspar 1 | *K-feldspar 2 | pyroxene | amphibole | epidote | sillimanite | zircon | anatase            | hematite | ilmenite | monazite | 'mica'          | 'kaolin' |
| 254      | MPLP724      | 86.8      | nd             | 1.9            | 5.4           | nd            | nd       | nd        | nd      | nd          | 0.8    | nd                 | nd       | <0.5     | nd       | nd              | 4.8      |
| 279      | MPLP725      | 62.5      | nd             | 12.9           | 16.8          | 2.8           | nd       | 0.8       | nd      | nd          | 0.6    | nd                 | nd       | 1.3      | nd       | 2.4             | nd       |
| 319      | MPLP726      | 72.1      | nd             | 8.3            | 4.2           | nd            | nd       | 1.0       | nd      | nd          | 0.9    | nd                 | nd       | 3.4      | nd       | 5.7             | 4.3      |
| 332      | MPLP727      | 81.8      | nd             | 3.0            | 8.7           | nd            | nd       | nd        | nd      | nd          | 0.7    | nd                 | nd       | 3.7      | nd       | nd              | 2.2      |
| 337      | MPLP728      | 54.9      | 18.4           | nd             | 19.0          | nd            | nd       | nd        | 1.9     | nd          | <0.5   | nd                 | nd       | <0.5     | nd       | 5.5             | nd       |
| 365      | MPLP729      | 66.8      | nd             | 6.9            | 6.1           | nd            | nd       | nd        | nd      | nd          | 0.6    | nd                 | nd       | 6.4      | nd       | 9.8             | 3.4      |
| 368      | MPLP730      | 48.3      | 20.7           | nd             | 18.1          | nd            | nd       | nd        | 3.9     | nd          | nd     | nd                 | nd       | <0.5     | nd       | 7.9             | 0.7      |
| 396      | MPLP731      | 72.0      | 5.9            | nd             | 16.2          | nd            | nd       | nd        | nd      | nd          | 0.6    | nd                 | nd       | 1.6      | <0.5     | 3.7             | nd       |
| 423      | MPLP732      | 33.8      | 26.9           | nd             | 27.1          | nd            | nd       | 2.6       | 6.3     | nd          | 0.6    | nd                 | nd       | nd       | nd       | 2.8             | nd       |
| 474      | MPLP733      | 78.1      | 2.7            | nd             | 6.2           | nd            | nd       | nd        | nd      | nd          | 2.1    | <0.5               | nd       | 2.3      | <0.5     | 4.0             | 4.0      |
| 513      | MPLP734      | 59.1      | nd             | 9.1            | 6.6           | nd            | nd       | nd        | 4.5     | nd          | 0.6    | nd                 | nd       | 4.0      | nd       | 7.9             | 8.2      |
| 539      | MPLP735      | 71.8      | nd             | 6.1            | 5.6           | nd            | nd       | nd        | nd      | nd          | 0.6    | nd                 | nd       | 1.9      | nd       | 9.6             | 4.6      |
| 568      | MPLP736      | 51.1      | 22.3           | nd             | 21.4          | nd            | nd       | nd        | 2.4     | nd          | nd     | nd                 | nd       | nd       | nd       | 2.7             | nd       |
| 681      | MPLP737      | 53.1      | 15.1           | nd             | 17.6          | nd            | nd       | <0.5      | 3.3     | nd          | <0.5   | nd                 | nd       | 2.6      | nd       | 5.7             | 2.0      |
| 715      | MPLP738      | 48.4      | 22.5           | nd             | 19.5          | nd            | nd       | nd        | 5.7     | nd          | <0.5   | nd                 | nd       | nd       | <0.5     | 3.4             | nd       |
| 814      | MPLP739      | 70.8      | 4.9            | nd             | 14.9          | nd            | nd       | 0.9       | nd      | nd          | <0.5   | nd                 | nd       | 2.0      | nd       | 5.1             | 1.0      |
| 828      | MPLP740      | 43.0      | 17.7           | nd             | 35.9          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | nd       | nd       | 3.1             | nd       |
| 830      | MPLP741      | 54.8      | 2.7            | nd             | 35.6          | nd            | nd       | nd        | nd      | 1.3         | <0.5   | nd                 | nd       | 0.9      | <0.5     | 3.8             | 0.6      |

Table 4. Summary of quantitative powder XRD analyses (continued)

| Site no. | BGS MPL code | Silicates |                |                |               |               |          |           |         |             |        | Oxides, phosphates |          |          |          | Phyllosilicates |          |
|----------|--------------|-----------|----------------|----------------|---------------|---------------|----------|-----------|---------|-------------|--------|--------------------|----------|----------|----------|-----------------|----------|
|          |              | quartz    | *plagioclase 1 | *plagioclase 2 | *K-feldspar 1 | *K-feldspar 2 | pyroxene | amphibole | epidote | sillimanite | zircon | anatase            | hematite | ilmenite | monazite | 'mica'          | 'kaolin' |
| 848      | MPLP742      | 57.4      | 8.6            | nd             | 24.1          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | 1.3      | nd       | 4.4             | 4.0      |
| 850      | MPLP743      | 65.4      | nd             | 6.6            | 14.4          | nd            | nd       | 0.7       | nd      | nd          | 1.0    | nd                 | nd       | 4.7      | <0.5     | 4.3             | 2.8      |
| 851      | MPLP744      | 60.6      | 10.2           | nd             | 18.9          | nd            | nd       | 2.2       | nd      | nd          | <0.5   | nd                 | nd       | 2.2      | nd       | 4.4             | 1.2      |
| 888      | MPLP745      | 37.8      | 25.3           | nd             | 30.7          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | 1.2      | nd       | 4.6             | <0.5     |
| 909      | MPLP746      | 70.2      | 4.3            | nd             | 15.9          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | 0.6      | <0.5     | 4.5             | 4.1      |
| 981      | MPLP747      | 48.2      | nd             | 22.9           | 8.3           | nd            | nd       | 2.2       | 6.8     | nd          | 2.9    | nd                 | nd       | 3.7      | nd       | 5.0             | nd       |
| 990      | MPLP748      | 64.5      | 8.0            | nd             | 10.5          | nd            | nd       | 1.2       | nd      | nd          | <0.5   | nd                 | 0.8      | 5.7      | nd       | 6.7             | 2.3      |
| 1005     | MPLP749      | 71.7      | nd             | 3.0            | 15.3          | nd            | nd       | <0.5      | nd      | nd          | <0.5   | nd                 | nd       | 0.7      | nd       | 5.0             | 3.4      |
| 1007     | MPLP750      | 63.3      | 10.7           | nd             | 13.5          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | 1.3      | nd       | 6.2             | 4.6      |
| 1022     | MPLP751      | 71.1      | nd             | 1.1            | 8.4           | nd            | nd       | nd        | nd      | nd          | 1.0    | nd                 | nd       | 1.7      | <0.5     | 5.2             | 11.1     |
| 1024     | MPLP752      | 77.5      | nd             | 0.9            | 12.4          | nd            | nd       | nd        | nd      | nd          | 0.7    | nd                 | nd       | <0.5     | nd       | 3.2             | 4.9      |
| 1031     | MPLP753      | 73.1      | nd             | 1.4            | 10.6          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | 0.8      | nd       | 4.8             | 8.9      |
| 1038     | MPLP754      | 78.1      | nd             | 1.0            | 8.7           | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | <0.5     | nd       | 3.8             | 7.8      |
| 1041     | MPLP755      | 63.9      | nd             | 6.8            | 23.6          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | 0.9      | nd       | 3.8             | 0.6      |
| 1052     | MPLP756      | 69.4      | nd             | 3.7            | 15.8          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | <0.5     | nd       | 5.1             | 5.7      |
| 1105     | MPLP757      | 87.6      | nd             | 1.3            | 5.3           | nd            | nd       | nd        | nd      | nd          | 0.8    | nd                 | nd       | 1.7      | nd       | nd              | 3.3      |
| 1109     | MPLP758      | 61.6      | nd             | 1.2            | 19.1          | nd            | nd       | nd        | nd      | nd          | 0.6    | nd                 | nd       | <0.5     | nd       | 5.3             | 12.1     |
| 1116     | MPLP759      | 72.9      | nd             | 2.0            | 12.2          | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | <0.5     | nd       | 4.1             | 8.3      |

Table 5. Summary of quantitative powder XRD analyses (continued)

| Site no. | BGS MPL code | Silicates |                |                |               |               |          |           |         |             |        | Oxides, phosphates |          |          |          | Phyllosilicates |          |
|----------|--------------|-----------|----------------|----------------|---------------|---------------|----------|-----------|---------|-------------|--------|--------------------|----------|----------|----------|-----------------|----------|
|          |              | quartz    | *plagioclase 1 | *plagioclase 2 | *K-feldspar 1 | *K-feldspar 2 | pyroxene | amphibole | epidote | sillimanite | zircon | anatase            | hematite | ilmenite | monazite | 'mica'          | 'kaolin' |
| 1127     | MPLP760      | 78.4      | nd             | 1.1            | 14.6          | nd            | 0.7      | nd        | nd      | nd          | <0.5   | nd                 | nd       | <0.5     | nd       | nd              | 4.8      |
| 1142     | MPLP761      | 69.4      | nd             | 1.9            | 11.6          | nd            | nd       | nd        | nd      | nd          | 0.6    | 0.6                | nd       | <0.5     | nd       | 4.3             | 11.3     |
| 1413     | MPLP762      | 44.0      | nd             | 8.6            | 31.3          | nd            | nd       | 1.9       | nd      | nd          | 1.0    | nd                 | nd       | 5.9      | nd       | 5.3             | 2.0      |
| 1553     | MPLP763      | 73.0      | nd             | 5.0            | 9.4           | nd            | nd       | nd        | nd      | nd          | <0.5   | nd                 | nd       | 2.3      | nd       | 8.5             | 1.4      |
| 1623     | MPLP764      | 67.2      | nd             | 6.6            | 15.6          | nd            | nd       | 1.0       | nd      | nd          | 0.9    | nd                 | nd       | 1.9      | nd       | 5.3             | 1.6      |
| 1633     | MPLP765      | 53.6      | nd             | 8.8            | 15.1          | nd            | nd       | 1.7       | nd      | nd          | 1.4    | nd                 | nd       | 4.3      | <0.5     | 11.0            | 3.8      |

KEY\*plagioclase 1 =  $\text{Na}_{0.84}\text{Ca}_{0.16}\text{Al}_{1.16}\text{Si}_{2.84}\text{O}_8$ \*plagioclase 2 =  $\text{Na}_{0.75}\text{Ca}_{0.25}\text{Al}_{1.26}\text{Si}_{2.74}\text{O}_8$ 

\*K-feldspar 1 = microcline

\*K-feldspar 2 = orthoclase

nd = not detected

'mica' = undifferentiated mica species, possibly including muscovite, biotite, illite, illite/smectite

'kaolin' = undifferentiated kaolin-group species, possibly including kaolinite, halloysite etc

## 4 Summary

- XRD analyses have been completed on a suite of sixty stream sediment samples from Nigeria.
- XRD analysis indicates similar assemblages to those observed by Kemp *et al.* (2009) which are predominantly composed of quartz with subordinate amounts of feldspar (various species of plagioclase and K-feldspar), ‘mica’ (undifferentiated mica species possibly including muscovite, biotite, illite and illite/smectite), ‘kaolin’ (undifferentiated kaolin group minerals possibly including kaolinite, halloysite etc) ± traces of ilmenite, zircon, amphibole, epidote, hematite, monazite, sillimanite, pyroxene and anatase.
- Cluster analysis of XRD data indicates eight specific mineralogical groups which can be combined to produce two clusters with similar characteristics.
- Groups 1, 2, 3 and 4 show broadly similar mineralogical characteristics of moderate-high amounts of quartz, low-moderate amounts of K-feldspar (microcline, no orthoclase), plagioclase and ‘kaolin’ together with low/no amounts of ‘mica’ and ferromagnesian minerals and variable zircon concentrations. These samples typically correspond to catchments composed of migmatitic gneiss lithologies.
- Groups 5, 6 and 7 are composed of low-moderate amounts of quartz, high amounts of K-feldspar (microcline and orthoclase) and plagioclase, moderate but variable amounts of ‘mica’, ‘kaolin’ and ilmenite together with occasional traces of epidote, amphibole, sillimanite, monazite and variable zircon concentrations. These samples appear to be derived from a mixture of old granites and migmatitic gneiss catchment lithologies.
- The XRD data shows some interesting correlations with previously produced geochemical analyses.
- Zircon was the only Zr-bearing phase identified by XRD in the stream sediments and its concentration shows an excellent correlation with Zr from geochemical analysis. Trace quantities of other Zr-bearing minerals (e.g. baddeleyite) may also be present but are below XRD detection limits. The variable zircon content of the stream sediments suggest that its presence is not indicative of any one particular catchment lithology.
- More accurate speciation of the heavy mineral fraction and potential identification of further Zr-bearing phases could be achieved if the quartz and feldspar component was removed using heavy media (e.g. Li-polytungstate) separation techniques prior to further XRD analysis.

## References

Most of the references listed below are held in the Library of the British Geological Survey at Keyworth, Nottingham. Copies of the references may be purchased from the Library subject to the current copyright legislation.

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