

Chapter 2

Materials and Methodology

Abstract Two borehole cores extracted between Old House Rocks and Black Rock, East Falkland, were studied using a combination of detailed sedimentary logging and geochemical analyses. The former involved grain and clast studies while the latter included X-ray fluorescence (XRF) and reflectance scanning using an Avaatech XRF core scanner. Logging data collected incorporated details on grain composition, size, shape, sorting, orientation, colour, sedimentary structures and fossils as well as a detailed account of variations in clast characteristics. A full scan of the original hand-drawn log is presented in the data postscript at the end of this book, and a digital version may be found online as a SPRINGER EXTRA. The high-resolution log was generated at a scale comparable to the X-ray fluorescence and reflectance data collected by the core scanner to encourage subsequent, more detailed climatic interpretations. The core scanner measured variations in element concentrations downcore and three reflectance parameters: lightness, red:green ratio and yellow:blue ratio. Total Organic Carbon (TOC) measurements on loose fragments throughout core DD090 were also taken. Cyclicity observed in the XRF and reflectance data was compared alongside Milankovitch orbital cycles through age modelling, thereby helping to constrain the time frame spanned by the deposits.

Keywords Sedimentary logging · XRF · Reflectance · Clast content · Total Organic Carbon · Age modelling

2.1 Overview

Detailed sedimentary logging was combined with high-resolution X-ray fluorescence (XRF) and reflectance scanning of two borehole cores, DD029 and DD090, to allow precise stratigraphic correlations and detailed sedimentary and climatic reconstructions. The cores used in this study, DD090 and DD029, have incomplete recovery in the diamictite sections ($\sim 10\text{--}20\%$) but were selected because they captured the contact between the Fitzroy Tillite and the Hells Kitchen Member and because they appeared to have the most stratigraphically complete sequence through the Hells Kitchen Member, which is crucial to understanding the climatic transition. Table 2.1 summarises the sections of cores DD029 and DD090 that were available and used in this study.

Table 2.1 Measured thicknesses of the three main units in cores DD029 and DD090 and the parts involved in this study

Core	Total core length (m)	FT available (m)	FT studied (m)	HK available (m)	HK studied (m)	BRMa available (m)	BRMa studied (m)	BRMb available (m)	BRMb studied (m)
DD029	227	222.8–181.3	181.3–190	181.3–176.4	181.3–176.4	176.4–170	176.4–170	0	0
DD090	395	395–392	395–392	392–383.5	392–383.5	383.5–375	383.5–375	153.8–161.8	155.8–156.7, 157.9–158.7

Fitzroy Tillite (FT), Hells Kitchen Member (HK) and Black Rock Member (BRMa and BRMb). Subdivision of core here is that of Stone (BGS report 2011)
Note: Thicknesses indicated are not equivalent to stratigraphic thicknesses

2.2 Logging

A 2:1 stratigraphic log was produced by recording millimetre-scale changes in lithology along the length of both cores. Log depths were corrected for the drilling angle and the dip of the beds (Fig. 2.1). Scanned images of the original logs and a digital version are presented in the appendix.

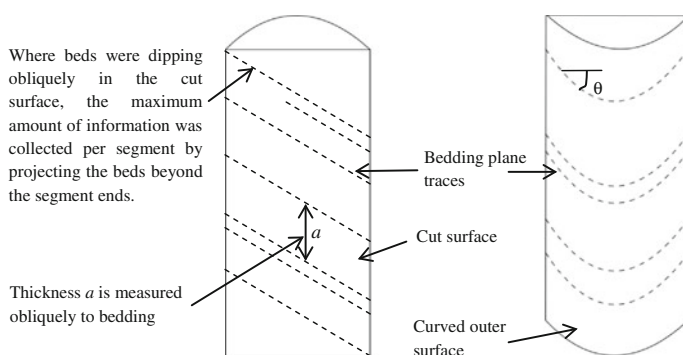


Fig. 2.1 Technique for measuring bed thickness

Box 2.1 Conversion of Apparent Thickness (a) to True Stratigraphic Thickness (t)

The true thickness of a bed (t) is the perpendicular distance between adjacent bedding planes. However, because the beds were dipping and the drilling angle was oblique to bedding, the measured value ' a ' was multiplied by the cosine of the maximum angle the beds made with the horizontal (θ), measured on the curved surface, to find ' t ': $t = a \cos \theta$.

2.3 Reflectance Scanning

Reflectance scanning was carried out using the Avaatech core scanner in the sedimentary laboratory, University of Cambridge. Preparation of core samples included sanding the upper cut surfaces of the segments to remove the original ink labels and milling lines. Sanding by hand proved the most effective method since pieces were commonly fractured or brittle and had to be handled with care. Prior to scanning, the light in the scanner was allowed to warm up for around 30 min and a blind was drawn to avoid alteration to image brightness.

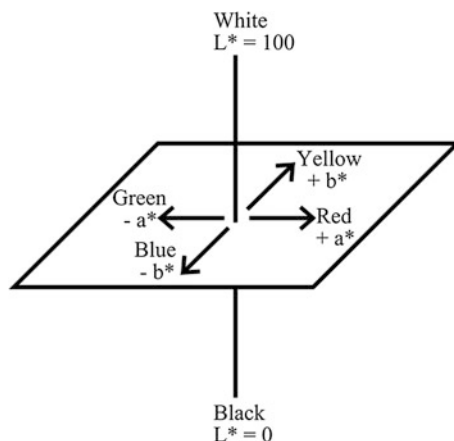


Fig. 2.2 CIE $L^*a^*b^*$ colour scale defined by the Commission internationale de l'Éclairage, HunterLab manual (2008); figure is modified from this manual. L^* characterises lightness scaled from 0 (*black*) to 100 (*white*). The parameters a^* and b^* are chromaticity coordinates that define the colour: a^* ranges from -60 (*green*) to $+60$ (*red*) and b^* from -60 (*blue*) to $+60$ (*yellow*)

The cores were then illuminated by high-intensity light with a broad spectrum across all wavelengths to allow photographs and reflectance data to be collected. Scans were run at lens aperture '8+' against standard colour charts. This process is non-destructive and fairly rapid. Long reflectance measurement bands were then drawn on the images generated, over the same central section of the core where XRF data were also collected to ensure effective comparison later. Colour reflectance values were summarised using the L^* , a^* and b^* parameters (Fig. 2.2). This ratio scheme was used because raw colour is dominated by brightness and so gives less independent information.

The reflectance scanning results have been assimilated onto a depth scale comparable to the log and clast data (Box 2.2) and are shown in the appendix.

Box 2.2: Generation of Depth Scale for Reflectance and XRF Data

Original depth labels on the segments were approximate and marked according to drilling depths rather than true stratigraphic thicknesses. They had been extrapolated from drillers' markings which amounted to a figure for the top and bottom of each core box (each box would hold about 5–6 m) and an insert, where present or legible, every 1–1.5 m marked with the depth figure. When producing a depth scale, the positioning of these labels was initially not corrected for using the dip of the beds because their positions were clearly vague and this adjustment would only have increased implied overlap. Some indication of the position of the labels within a segment was measured though and the depth correction to reach the segment top applied.

Complications also arose because the XRF and reflectance data were collected along the cut surface rather than perpendicular to bedding. Therefore, to make these data sets comparable with the log and clast data, depth control points were inserted into the XRF and reflectance scales for the two cores at the beginning and end of each segment and the depths in-between linearly interpolated. The full procedure is outlined in Appendix A.1.

2.4 XRF Scanning

A continuous record of elemental data was collected non-destructively at the split core surface using the Avaatech X-ray core scanner. Before running samples through the scanner, the X-ray tube was allowed a warm-up time of 30 s and four standards were run twice at the 10 kV energy band; the 10 kV band is the most sensitive measure to fluctuations in counts. The removal of the ink labels during sanding also prevented biases to the signal. The labels were found to cause considerable alteration to the chemistry on trial XRF runs. Non-continuous segments were positioned with sufficiently wide gaps such that the detector could skip between them and avoid damage to its Ultralene window. The level of the core segments was checked using a mini-spirit level, and they were secured where necessary using foam to prevent them being caught by the moving window.

The X-ray source progressively irradiated the cross-core and downcore directions causing fluorescent energy, characteristic of atoms of specific elements, to be emitted. The chemical composition of the sediment was measured as element intensities in total counts. Heavier elements require more energetic photons in order to fluoresce, so both cores were run on three different energy levels (10, 30 and 50 kV) to activate different sets of elements and provide reliable element intensities (Fig. 2.3). A computer registered the energy of every photon, allowing for the creation of a spectrum. A spectrum interpretation software package, WinAxilBatch, was then used to interpret spectra quantitatively by assigning distributions of photons (counts) at different wavelengths to elements. Each metre segment was run over 24 h collecting two replicates at an interval of 1:25, a step size of 2.5 mm, a downcore slit size of 2.5 mm and a slit size across core of 12 mm; a level of resolution comparable to the logs. The data were then normalised to aluminium (Box 2.3). Standards were run to check the consistency of results and machine behaviour. The complete XRF data sets from both cores are presented in the appendix on the same depth scale transformation as the reflectance data.

Box 2.3: Normalisation

In variable settings, such as lakes (the arguable depositional setting here; see Chap. 4), the strength of the XRF-scanning method lies in an analysis of the relative variations of the different elements compared to a major conservative element to obtain an environmentally relevant signal (Löwermark et al. 2011). Without such normalisation, biological or diagenetic processes can influence elemental counts and interpretations would be at risk of becoming a mirror of organic matter (or carbonate) variations (Löwermark et al. 2011). Even with an absolute calibration, changes in flux rate of particular elements and dilution effects are extremely difficult to separate. While normalisation does not compensate for the effects of sediment composition, it does help to compensate for some effects of surface irregularity (Weltje and Tjallingi 2008). Therefore, the relative signal strength was used as an indication of compositional change and interpreted in terms of climatological and sedimentological change. Normalisation also enables presentation of data from elements with very different XRF count rates. Log ratios accommodate the non-linearity of the relationship between relative intensities and concentrations. Table 2.2 evaluates possible elements which may be used for normalisation and the rationale behind the choice of aluminium.

Table 2.2 Reasoning behind the choice of aluminium for normalisation summarised from Löwermark et al. (2011)

Element	Comment on suitability
Fe or Mn	Unsuitable because of their redox sensitive nature
Na, K, Sr or Mg	Easily dissolved and weathered out so unsuitable
Ti	Ti is an important component in many minerals, is abundant and not very active biologically. However, Ti is enriched in heavy minerals and in aeolian dust and is therefore used as a proxy for high current regimes or enhanced wind activity. Also, Ti content is highly influenced by the composition of the original protolith
Al	Normalisation to Al helps reduce problems associated with surface irregularities, which tend to decrease counts of most elements. Al is abundant and little affected by diagenetic and biological processes such as redox reactions. As it is a major structural component in the sand, silt and clay that enters basins, normalisation of the other elements against Al allows the changes in proportion of the individual elements in the lithogenic component of the sediment relative to each other over time to be indicated in terms of aeolian, terrestrial and biogenic inputs

2.5 Clast Data

Details on clast composition, size, frequency, sorting, roundness, sphericity and orientation were collected from core DD090 in order to better understand sediment source area and ice sheet flow patterns and processes. Frequency was recorded as a

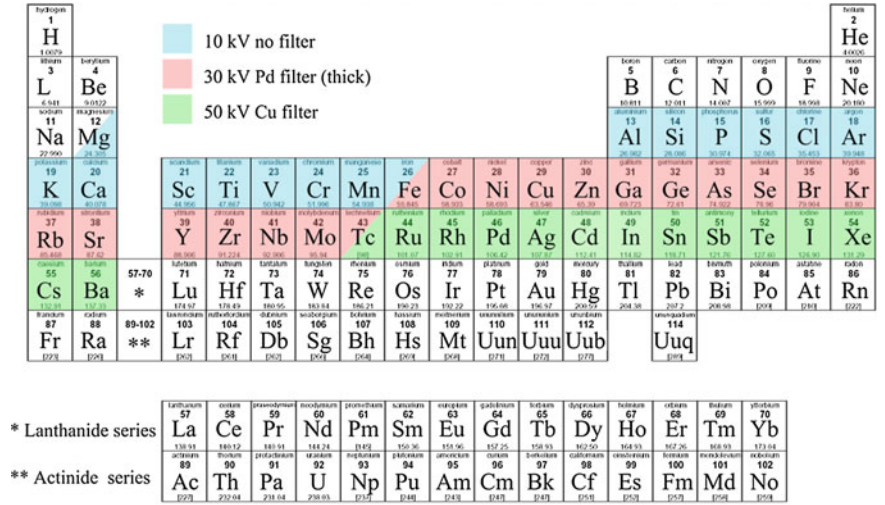


Fig. 2.3 The range of elements that are activated for the different instrument settings (information from Xelerate Documentation, University of Delft, Bloemsma (2012)) are highlighted on the periodic table. Elements between ¹³Al and ⁹²U could be detected and included in the deconvolution

percentage while sorting, sphericity, roundness and orientation were ranked on scales 0–2, 0–10, 0–10 and 0–10, respectively; where the higher number in each case corresponds to a higher degree of the variable in question. The comparator charts used are provided in Fig. 2.4. Plotted data from the clast analysis are presented in the appendix.

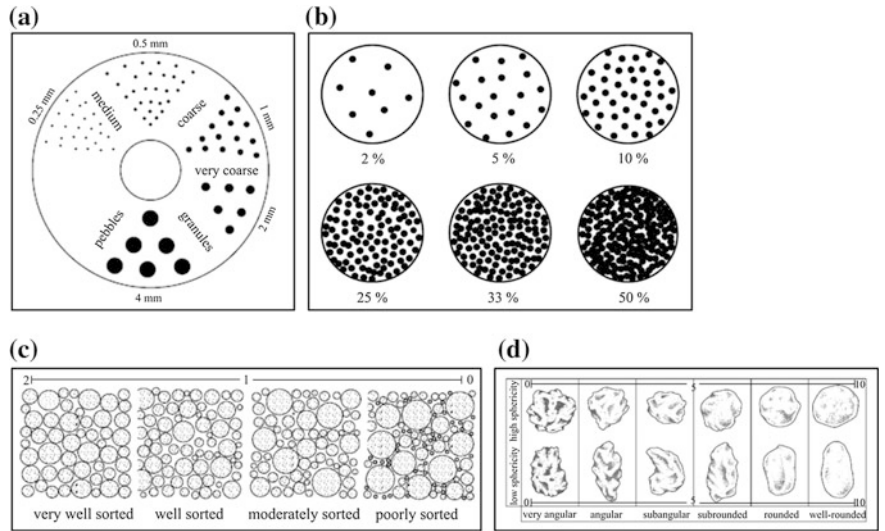


Fig. 2.4 Comparator charts used for estimating clast **a** size, **b** abundance, **c** shape (including *roundness* and *sphericity*) **d** sorting. Figure compiles and modifies relevant diagrams from Tucker (2003)

2.6 Total Organic Carbon

Nine loose fragments from core DD090 were collected for Total Organic Carbon (TOC) measurements: one from the diamictite, five from the Hells Kitchen Member and three from the Black Rock Member. The samples were ground to a powder before being placed in an oven at 60 °C to remove any moisture. They were weighed after 4 and 8 h to ensure constant weight. Once their mass stopped declining, they were added to separate beakers containing ~15 ml 1 M HCl to remove inorganic carbon present as carbonate. TOC content was then determined by dry combustion. This procedure collected information on ^{13}C isotopes also. The mass of the samples before and after drying and bathing in acid is shown in Table 6.1.

2.7 Development of an Age Model

The XRF and reflectance data were used to construct a relative age model, and the existing absolute dates from other localities across Gondwana informed judgements of when the transition took place in the Falklands. Spectral analyses and wavelet analyses were used to look for prominent periodicities in the depth domain and from these observations, a tentative relative age model was developed and further wavelet analyses undertaken (see Chap. 4). Although absolute timescales become increasingly imprecise with increasing age (Arthur and Garrison 1986), it was hoped that biostratigraphy in the form of micropalaeontology might play a supporting role in constraining the time frame over which the icehouse to greenhouse transition took place. However, studies of similar Falkland Island material by Mike Stephenson of the British Geological Survey (personal communication) suggest a lack of suitable spores/fauna in the cores for absolute dating.

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