



Data report: X-ray fluorescence scanning of sediment cores, IODP Expedition 390/393 Site U1559, South Atlantic Transect¹

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Abstract

During International Ocean Discovery Program (IODP) Expeditions 390C, 395E, 390, and 393, deepwater sediments were recovered from the western flank of the southern Mid-Atlantic Ridge along a crustal flow line at ~31°S. This multidisciplinary experiment allowed the recovery of data fundamental to reconstructing past climate changes as well as variations in ocean circulation, productivity, and chemistry (i.e., fluctuations in the carbonate compensation depth) in the South Atlantic Ocean.

Here, we report semiquantitative elemental results from X-ray fluorescence (XRF) scanning of the sediment package cored at IODP Site U1559 in the South Atlantic Ocean. Located at 15°02.0941'W, Site U1559 is the easternmost site of the South Atlantic Transect and the closest to the Mid-Atlantic Ridge, located on ~6.6. Ma ocean crust. The XRF data are also compared with magnetic susceptibility and natural gamma radiation measured on the R/V *JOIDES Resolution* to assess correlations with the different lithologic units/subunits. At Site U1559, sediments are predominantly nannofossil ooze with varying amounts of foraminifera, which is reflected by the dominant Ca counts. Trends in elemental counts reflect the slight variations in siliciclastic materials within the Pleistocene. Major shifts in elemental counts were observed at the sharp contact between Pliocene–Pleistocene Subunits IC and ID, as well as the Miocene–Pliocene transition.

1. Introduction

The sediments of the western South Atlantic Ocean have received limited attention by scientific ocean drilling since Deep Sea Drilling Program (DSDP) Leg 3, with a consequent lack of precious information to understand the paleoceanographic evolution of the western South Atlantic Ocean. New sites close to those of Leg 3 were drilled during International Ocean Discovery Program (IODP) Expeditions 390C, 395E, 390, and 393 between October 2020 and August 2022 (Figure F1) along the South Atlantic Transect (SAT; Estes et al., 2021; Williams et al., 2021; Coggon et al., 2022;

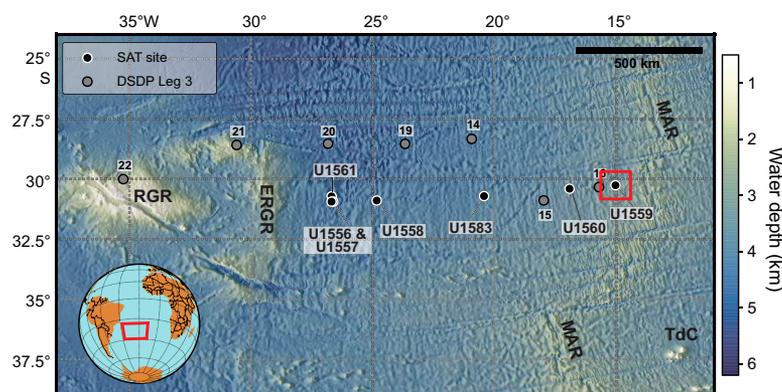


Figure F1. Bathymetric map of South Atlantic Ocean with location of Expedition 390/393 drill sites. Deep Sea Drilling Program Leg 3 sites are also shown. Figure modified from Teagle et al. (2023). Red box = location of Site U1559. RGR = Rio Grande Rise, ERGR = eastern Rio Grande Rise, MAR = Mid-Atlantic Ridge, TdC = Tristan de Cunha.

Teagle et al., 2023). The SAT sites were drilled along a crustal flow line at $\sim 31^\circ\text{S}$ across the western flank of the southern Mid-Atlantic Ridge to address fundamental scientific questions about major shifts in ocean circulation and chemistry, surface productivity, and past global climatic changes and hydrothermal seawater-basalt exchange across the ridge flank. In this framework, the collection of sediment X-ray fluorescence (XRF) data represents a fundamental tool to gain new insights into past environmental change because it is a nondestructive technique to acquire high-resolution semiquantitative elemental data. XRF analysis of sediment cores from Site U1559 was conducted as part of the programmatic scanning of cores recovered during the SAT expeditions to build a robust data set of elemental trends to define lithologic variations (Penkrot et al., 2018; Taylor et al., 2022) and changes in terrigenous versus biogenic inputs through time and space. For comparison with equivalent data sets from sites across the SAT with increasing distance from the Mid-Atlantic Ridge (Figure F1), we refer the readers to the data reports regarding Sites U1560 (Amadori et al., 2024), U1583 (Lam et al., 2024), U1558 (Villa et al., 2024), U1561 (Routledge et al., 2024), U1556 (Wang et al., 2024), and U1557 (Lowery et al., 2024).

Here, we report all quality controlled data produced during XRF scanning for Site U1559, evaluate correlations among elements, and investigate downhole variations in elemental counts of major elements plotted along the splice of Site U1559. The data are integrated with stratigraphy, magnetic susceptibility (MS), and three components of natural gamma ray (NGR) to evaluate variations in deepwater sediment properties through time. Site U1559 is located at the eastern end of the SAT ($30^\circ 15.6336'\text{S}$ $15^\circ 2.0941'\text{W}$; Figure F1) in 3055 m of water ~ 130 km west of the Mid-Atlantic Ridge. The site sits on the youngest crust (6.6 Ma) drilled during the SAT and is close to DSDP Site 16. Three holes were drilled at Site U1559 during Expeditions 390C and 390 (Estes et al., 2021; Coggon et al., 2022; Coggon et al., 2024) and between 43.6 and 58.1 m of sediments was recovered from each hole. The recovered sedimentary package is entirely composed of calcareous nannofossil ooze (with varying amounts of foraminifera) and extends from the Miocene (Messinian) through the Pleistocene (Coggon et al., 2022; Coggon et al., 2024). Relatively small variations in color (from white to pale brown), lithologies, and sedimentary structures, along with age information, were used to define five Subunits (IA–IE) (Coggon et al., 2022; Coggon et al., 2024). An interval of slumped sediment was interpreted at the base of the Pleistocene in Subunit IC because of lighter colored sediments corresponding to a shift in P -wave velocity and containing reworked Miocene fossils.

2. Methods and materials

The archive-half sections included in the splice of Site U1559 were scanned on the fourth generation Avaatech XRF Core Scanner (XRF 2) at the IODP Gulf Coast Repository (GCR) in College Station, Texas (USA).

The instrument is fitted with a water-cooled 100 W rhodium side-window x-ray tube, a Brightspec SiriusSD silicon drift detector, and a Topaz-X high-resolution digital multichannel analyzer. Each archive-half section was scanned at three excitation levels to measure different elements: 10 kV (with no filter) for major and minor elements (including Al, Si, K, Ca, Ti, Mn, Fe, Cr, P, S, and possibly Mg), 30 kV (with a thick Pd filter) for heavier major and minor elements and geologically relevant trace elements (including Ca, Ti, Mn, Fe, Ni, Sr, Rb, Br, Zr, and Zn), and 50 kV (with a Cu filter) for heavier trace elements (including Sr, Rb, Zr, and Ba). The cross-core and downcore slits were set to 12 and 10 mm, respectively. Count times for the 10 and 30 kV scans were set to 6 s, and the count time was set to 10 s for the 50 kV scan. The current was set to 0.15, 1.24, and 0.75 mA for the 10, 30, and 50 kV scans, respectively. The instrument configuration and filter selection correspond to the standard analytical setup used at the GCR, thus allowing comparison with XRF measurements performed on cores at the GCR XRF Core Scanning Laboratory.

Quality control (see below for details) and data analysis were performed using R (R Core Team, 2023; the code is available at <https://github.com/Ravikiran2316/IODP-Exp.-390-393-XRF>), which also produced correlograms and plotted the data versus depth.

2.1. Core preparation

First, section halves were moved out of the GCR cold storage and left for at least 24 h to equilibrate to room temperature. Then, the surface of each section half was gently scraped using a glass slide to provide a flat and fresh surface to scan. To prevent contamination along the section and preserve the stratigraphic integrity, the scraping was performed across the section and the glass slide was cleaned with a Kimwipe between each scrape. In the case of water-rich sediments, several Kimwipes were laid along the section to remove excess water before scraping. Once the section surface had been scraped, a 4 μm thick Ultralene film was carefully placed on the sediment surface and taped to the edges of the core liner to prevent contamination of the XRF detector during measurements. Sections were placed in the XRF scanner, and the horizontality of the surfaces were checked along the section using a bubble level to ensure a good contact between the detector and the core surface.

2.2. Sample selection

Each core section was scanned at a 5 cm resolution, with the first measurement point 5 cm below the top of the section. Data point locations were examined on the core surface using a 3D printed replica of the scanning window of the XRF 2. In disturbed sections (with the occurrence of cracks and/or intervals with drilling disturbance), sample locations were moved to the closest undisturbed spot, trying to maintain the target resolution. In cores recovered with the extended core barrel (XCB) drilling system (Cores 390-U1559A-5X through 8X), measurements were located in the intact and firm biscuits, avoiding any gaps and fractures to ensure good contact between the detector and the sediment surface that in turn could affect the reliability of the measurements.

2.3. Quality control

To ensure consistent data quality from the XRF core scanner, standards (SARM 2 [syenite], SARM 4 [norite], and SARM 45 [kinzigite]) were run at all three excitation levels twice a day. At the beginning of each scanning day, 20 replicates were run at each excitation level to warm up the instrument. At the end, standards were run with no replicates and results were compared to those obtained at the beginning of the scanning day to exclude possible drift during the measurement session.

Raw spectral peaks were processed into peak areas and exported as count data for different elements using the Brightspec XRF spectral processing software bAxil. Quality control (QC) identified erroneous data using two criteria: throughput and argon (Ar) values. First, samples with throughput values <150,000 counts/s on the 10 kV scan were discarded because these values may indicate either the detector did not fully land on the section surface or the occurrence of a void at the sample location. Second, we removed any samples with a positive Ar value because these values indicate measurement of the ambient air without full contact between the detector and the sediments. QC was performed using R (R Core Team, 2023; the code is available at

<https://github.com/Ravikiran2316/IODP-Exp.-390-393-XRF>) to apply a common/standardized data analysis for XRF data from all the SAT sites and create a consistent data set. Complementary data sets have been developed for all the other sites: U1556 (Wang et al., 2024), U1557 (Lowery et al., 2024), U1558 (Villa et al., 2024), U1561 (Routledge et al., 2024), U1560 (Amadori et al., 2024), and U1583 (Lam et al., 2024).

3. Results

3.1. Correlation between elements

A series of crossplots were produced to identify correlations between elements commonly related to pelagic and detrital sources (e.g., Croudace and Rothwell, 2015) (Figure F2). The effectiveness of the applied QC and removal of low-quality data points is reflected in the absence of correlation between aluminum (Al) and Ar (Figure F2C) because Al counts can be influenced by ambient air, which also produces less negative Ar counts. Positive correlations are observed among terrigenous-sourced elements like iron (Fe) and titanium (Ti), Fe and potassium (K), and Ti and silicon (Si). Weak anticorrelation was found between pelagic- and terrigenous-sourced elements like calcium (Ca) and Fe and between Ca and Ti. However, no correlation was observed between Ca and Si (Figure F2D).

Because of the lack of normal distribution in the elemental raw counts, Spearman's rank correlation was performed to quantify correlations among selected elements (Figure F3). Spearman's rank correlation uses a monotonic function to provide a nonparametric measure (coefficient, ρ) of how strongly two variables are correlated. Values of +1 or -1 indicate a perfect positive or negative correlation, respectively, and ρ values equal to zero indicate no association between elements. Unlike Pearson's correlation, Spearman's rank correlation does not assume a normal distribution of the data and does not require linear relationships.

Elements that are generally indicative of productivity, such as barium (Ba) and strontium (Sr), show very weak negative correlation with Ca ($\rho > -0.20$). Si and Ca have a very weak positive correlation of $\rho = 0.15$. In contrast, elements that are typically indicative of terrigenous sources, such as Fe, zirconium (Zr), Ti, K, and Al are positively correlated with Si and Ba; for most of these elements, the correlation with Si and Ba varies from moderate to strong with ρ values between 0.38 and 0.84. The lowest values are observed for correlations between Zr and Ba and between Al and Ba, whereas higher values ($\rho = \sim 0.84$) are reported for Al and Si. Fe and Si also show a well-defined correlation ($\rho = 0.74$).

Sr has no or a weak negative correlation with most elements, except for nickel (Ni), sulfur (S), bromine (Br), and Zr. These correlations are generally weakly positive (ρ spans from 0.19 to 0.37). Only S shows a moderate/good correlation with Sr with $\rho = 0.48$.

Strong positive correlations ($\rho > 0.70$) are observed between Fe and Ti, Fe and Si, Fe and K, K and Ti, S and Br, and Si and Al.

No correlation ($\rho = 0$) is observed for Sr with either Ti or Mn. Very weak negative correlations ($\rho < -0.15$) are shown by Ni with most of the other elements except Sr, S, Ca, and Br, with which it displays a weak positive correlation. Other weak negative correlations are reported between Ca and Zr, Ca and Fe, Ca and Sr, Ba and Sr, Sr and K, and Mn and Br.

The weakest positively correlated values ($\rho < 0.15$) include S and Ni, S and Zr, S and Ba, S and Fe, S and Ti, S and Mn, Br and Ni, Br and Zr, Br and Ba, Br and Fe, Ca and Mn, and Ca and Si.

3.2. Stratigraphic trends

The downhole counts of selected elements (Ca, Fe, Ti, Al, Si, K, and Zr) are plotted in Figure F4 along with whole-round core MS and three NGR components (uranium [U], thorium [Th], and K). XRF elemental counts were compared with lithologic units associated with major geologic epochs inferred from shipboard age models based on microfossil biostratigraphy and interpreted paleomagnetic reversals (Coggon et al., 2022; Coggon et al., 2024).

Overall, the sedimentary package at Site U1559 consists of calcareous nannofossil ooze with foraminifera and is dominated by Ca. Slight variations in siliciclastic and nannofossil content that characterize the different sedimentary subunits are reflected by minor changes in MS (Coggon et al., 2022). In contrast, both NGR components and major element trends do not always show an unequivocal correlation with the lithologic subunits, nor with specific time intervals.

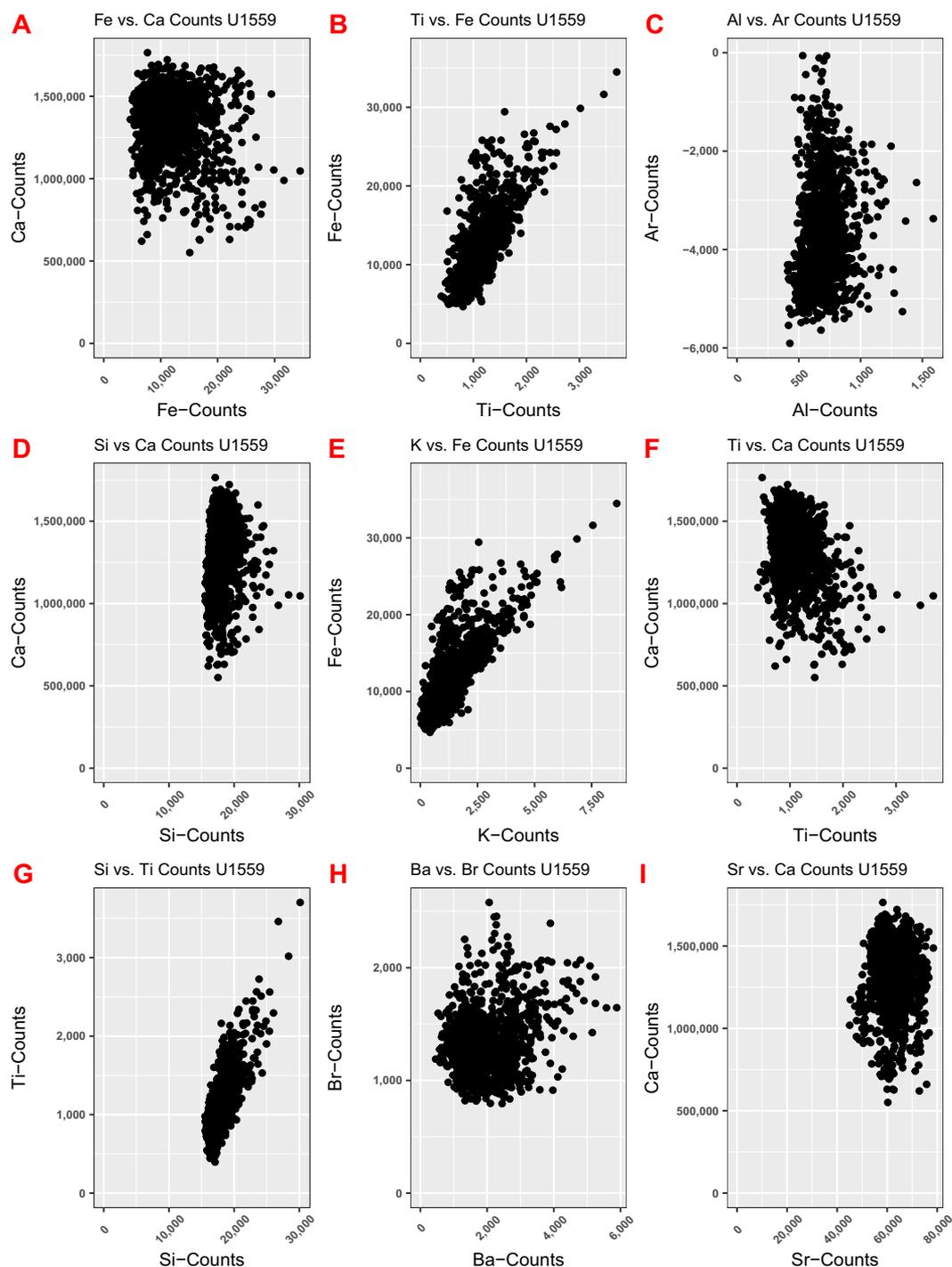


Figure F2. Crossplots of XRF raw counts of common paleoceanographically relevant elements from scanned cores, Site U1559. A. Fe vs. Ca. B. Ti vs. Fe. C. Al vs. Ar. D. Si vs. Ca. E. K vs. Fe. F. Ti vs. Ca. G. Si vs. Ti. H. Ba vs. Br. I. Sr vs. Ca. See Figure F3 for Spearman's rank correlation. Elements used from 10 kV excitation energy: Ca, Fe, Ti, Ar, Al, Si, and K; elements used from 30 kV excitation energy: Ba and Sr; elements used from 50 kV excitation energy: Ba.

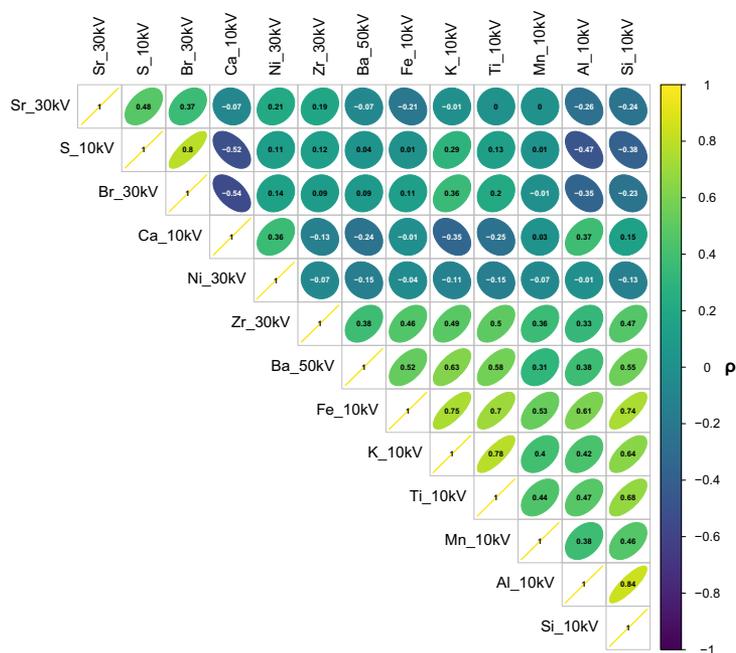


Figure F3. Correlogram for elements above XRF detection limits (>1000 counts/s), Site U1559. Spearman's rank correlation was used to determine correlation between elements. Positive and negative correlations are represented by colors and ellipses (green to yellow = positive correlation [black ρ values]; teal to purple = negative correlation [white ρ values]). Spherical shapes indicate low to absent correlation, whereas more elliptical shapes represent stronger correlation between elements. ρ coefficient values for each correlation are plotted in the center of each correlated element pair. Elements used from 10 kV excitation energy: Al, Si, S, K, Ca, Ti, Mn, and Fe; elements used from 30 kV excitation energy: Ni, Br, Sr, Zr; elements used from 50 kV excitation energy: Ba.

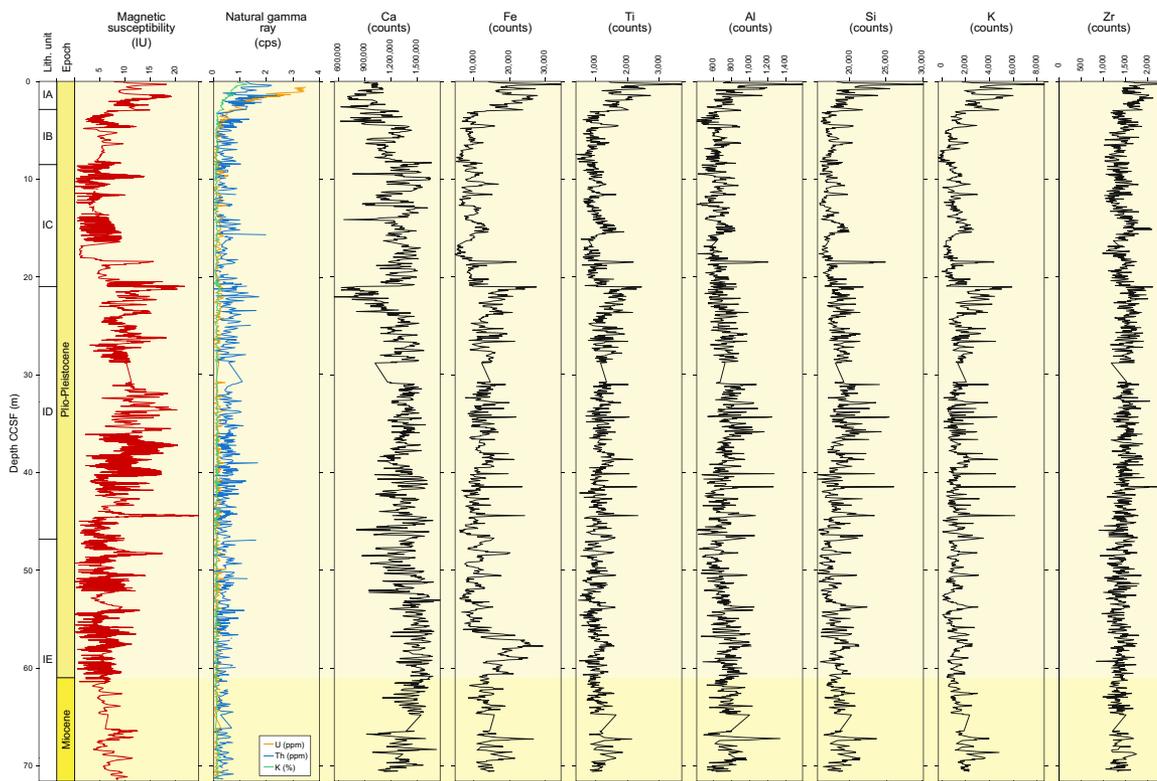


Figure F4. Magnetic susceptibility (whole-round measurements in instrument units), three components of natural gamma ray, and XRF counts of selected elements (10 kV excitation energy: Ca, Fe, Ti, Al, Si, and K; 30 kV excitation energy: Zr), Site U1559. Lithologic units and major geologic epochs are also shown.

The uppermost section of the sedimentary sequence (Subunit IA) has the highest counts for most of the elements (Fe, Ti, Al, Si, K, and Zr), coupled with high MS and NGR components (especially U) with respect to the other subunits. Only Ca shows an inverse trend with relatively low values, reflecting the occurrence of trace siliciclastic materials within Subunit IA (Coggon et al., 2022).

The relative increase in Ca and decreases in the other elemental counts are visible deeper in the Pliocene/Pleistocene Subunits IB and IC (Figure F4). Isolated peaks and increases in Fe, Ti, Al, Si, K, and Zr are observed within these subunits, generally associated with peaks in MS and decreases in Ca. The presence of these features within Subunit IC might be associated with the occurrence of slump deposits (Coggon et al., 2022). However, within Subunit IC, minor variability of elemental counts for some terrigenous-derived elements (e.g., Al, K, and Zr) might be related to low elemental raw counts and relatively small signal-to-noise ratio.

In correspondence to the sharp contact between Subunits IC and ID (~20 m core composite depth below seafloor [CCSF]; Coggon et al., 2022), a strong increase in MS is coupled with a sharp decrease in Ca counts and drastic increases in Fe, Ti, K, and Zr. Downcore below this shift, all elements decrease to mostly stable values, with the exception of Ca counts that show a general increase.

At the Miocene–Pliocene transition in Subunit IE (~55 m CCSF), Fe counts increase sharply. This enrichment is associated with a slight increase in Si, Al, and Ti. In contrast, Ca, K, and Zr remain almost stable.

The lowermost sediments of Site U1559 correspond to the Miocene. Here, MS, NGR components, and the representative elemental counts remain relatively constant. Notable is the occurrence of a secondary peak in Fe, Ti, Al, Si, and K counts at around 68 m CCSF.

4. Data availability

XRF elemental counts at all three excitation levels are included in XRF in **Supplementary material** and archived at PANGAEA (<https://Pangea.de>). The R code used for quality control to remove spurious throughput and Ar values can be found on GitHub (<https://github.com/Ravikiran2316/IODP-Exp.-390-393-XRF>).

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