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Technical note

Geochemical and isotopic (O-Fe-Sr-Nd) characterization of reference materials relevant to environmental impact assessments

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This study presents multi-faceted characterization of riverine and marine reference materials (RM) relevant for undertaking environmental impact assessments associated with mining or anthropogenic activities. These include composite stream sediments (JSd-2, JSd-3), marine sediments (MESS-3, HISS-1), a banded iron formation (FeR-4) and a basalt (BHVO-2). Whole rock major and volatile element contents (C, H, S) contents were determined using X-ray fluorescence and an elemental analyzer, respectively. Following hotplate digestions 47 trace elements were determined via solution induction coupled plasma mass spectrometer (ICP-MS). Oxygen isotope compositions (δ^{18}O) were measured using an isotope ratio-MS. Stable Fe (δ^{56}Fe) and radiogenic ^{87}Sr/^{86}Sr and ^{143}Nd/^{144}Nd isotope compositions were measured using multi-collector ICP-MS. These results demonstrate that caution should be applied when selecting a sedimentary RM given some suffer from significant heterogeneity (e.g. HISS-1) across multiple parameters including volatile and trace element contents and stable and radiogenic isotope compositions. Due to the potentially diverse source components of siliciclastic sediments (i.e. inherited heterogeneity), when conducting environmental impact assessments across certain settings (e.g., riverine; estuarine; marine), a wider uncertainty window should be applied before definitively ascribing subtle differences to exogenous contamination.

1 Introduction

Reference materials (RM) play a critical role in elemental concentration and isotopic characterization studies because they are essential for monitoring the data quality of unknown measurements (i.e. accuracy and precision) and validating measurement procedures (Flanagan, 1986; Jochum et al., 2005, 2011). Therefore, RM with rigorously and objectively determined compositions are widely utilized in geochemical, geochronological, petrogenetic and sedimentary provenance studies, as well as in biological, environmental and forensic studies (Jochum and Enzweiler, 2014; Li et al., 2019; Horan et al., 2020; McCormack et al., 2021). Magmatic and especially basaltic rock RM (e.g. BHVO-2) are widely measured and have been extensively characterized for a range of parameters (Raczek et al., 2001, 2003; Weis et al., 2005, 2006; Sossi et al., 2015; McCoy-West et al., 2020). However, there remains a sparsity of trace element or isotopic data for sedimentary materials (Chauvel et al., 2011; Fiket et al., 2017; Jo et al., 2021), which generates a gap in suitable quality assurance RM for environmental source tracing studies.

Using well characterized RM is particularly important when applying novel techniques or expanding into less common or more complex matrices. Consequently, the use of basaltic RM is not ideal for quality assurance purposes with sedimentary samples as the latter are often more varied and complex (e.g. clay/carbonate fractions), and often with a greater organic component, meaning digestion procedures and chemical separations may behave differently and require modification. Additionally, the larger particle size (≥120 µm) of sedimentary RM has made them of interest for elemental and isotopic studies as it can result in variable dissolution leading to heterogeneity in analytical results (Ghidan and Loss, 2010; Revels et al., 2015; Kumkrong et al., 2021). Therefore, understanding the extent of this heterogeneity and how it will impact interpretation of data from unknown samples is of high importance. Hence, it is critical to have well characterized matrix-matched sedimentary RM for method validation and quality control.

Normally, RM studies focus on precisely characterising one or two specific parameters (Inagaki et al., 2006; Ghidan and Loss, 2010; Choi et al., 2013; Druce et al., 2020; Mahan et al., 2020; Lu et al., 2021), or a suite of trace elements (Jochum et al., 2016; Fiket et al., 2017). Here, a holistic approach was undertaken to simultaneously characterize a wide range of geochemical parameters (major, trace and volatile element contents along with O-Fe-Sr-Nd isotope compositions), across a suite of RM that encompass the variability seen in settings relevant to environmental source tracing applications (i.e. potentially contaminated riverine/marine sedimentary reservoirs). Radiogenic Sr and Nd are well-established provenance tracers (e.g. Charalampides and Manoliadis, 2002), whereas O and Fe can fingerprint environmental contaminants from anthropogenic activity (i.e. metal oxides in primary ore phase or byproduct/wastes). Thus, this combination of geochemical parameters is incredibly powerful, as it bridges both stable and radiogenic isotopes, and could potentially be utilized to quantifying the distribution of Fe-rich material in a marine setting, such as occurred following the Fundao tailings dam collapse in 2015 (e.g. Valeriano et al., 2019; D'Azeredo Orlando et al., 2020).

2 Materials and Methods

2.1 Materials

Reference materials in this study include: two United States Geological Survey (USGS) igneous rock standards (BHVO-2 and G-2); two Geological Society of Japan (GSJ) terrestrial stream sediments (JSd-2, JSd-3); two National Research Council of Canada (NRC) ocean floor marine sediments (HISS-1 and MESS-3); and a banded iron formation standard from CCRMP (FeR-4) given its markedly different matrix (e.g. >35 wt% Fe_2O_3) to the other sediments. Depending on the distributor, RM have different particle sizes based on the sieve mesh size that was used during preparation, which can affect the homogeneity of the powder. The finest RM herein are USGS igneous RM (e.g. BHVO-2) at ≤74 µm (200 mesh; Flanagan, 1967). The sedimentary materials all have larger particle sizes with the NRC RM (e.g. HISS-1, MESS-3) finer than 125 µm (≤120 mesh; Berman et al. (1997) and the GSJ composite sediments RM (e.g. JSd-2, JSd-3) even coarser at ≤149 µm (100 mesh; Terashima et al., 1990).

2.2 Methods

Detailed description of the analytical techniques is provided in the Supplementary Material. Briefly, major and volatile elements were measured at the University of Tasmania using X-ray fluorescence (XRF) and an elemental analyzer (EA), respectively. Trace elements were determined using a triple quadrupole inductively coupled plasma mass spectrometer (TQ-ICP-MS) following a multi-stage $HF-HNO_3-HCl$ hotplate digestion of 100 mg aliquots at James Cook University (JCU). Oxygen isotopes were measured using an isotope ratio mass spectrometer (IRMS) at Queens University. Chemical separations were performed at JCU with Fe, Sr and Nd separated via column chromatography using well established procedures (Fig. S1, Table S1 in the Supplementary Material, and subsequently measured using multicollector ICP-MS at both JCU and Macquarie University, MQU). Reference materials were digested and processed in a minimum of six batches/analytical sessions. This approach ensures that results (and uncertainties) herein reflect long-term repeatability (i.e. measurement precision achievable using the same analytical setup; Barwick, 2023).

3 Results and Discussion

3.1 Accuracy and precision of concentration measurements

3.1.1 Major and Volatile Element Data

Major element analyses were fully blinded (i.e. analysts unaware of sample origin or context) for six RM with the data providing consistent results with published values (Govindaraju, 1994; Chauvel et al., 2011; Jochum et al., 2016). For RM (BHVO-2 and JSd-2), measured SiO_2, Al_2O_3, Fe_2O_3, MnO, MgO, CaO, K_2O values are accurate to within <±3 % of previous results (Chauvel et al., 2011) with all other elements generally better than ±5 % (Table DR1; McCoy-West et al., 2025).

Volatile elements (C, H, S) in five RM were analyzed blinded to constrain instrument repeatability across different sediment types (Table DR2). The average values of C, H and S obtained herein are within uncertainty of previous determinations (Govindaraju, 1994; Imai et al., 1996; Kubota, 2009). Carbon and H contents of from replicate measurements, run two years apart, generally produced similar results within <±0.15 % for C, and <±0.05 % for H. An exception are the C contents of the marine sediment RM, with MESS-3 having an average of 2.07 ±0.25 % ($n = 7$; 2 SD) and HISS-1 being extremely variable with three determinations between 0.41–0.48 % and one with 1.27 % C (Fig. S2). Focusing on the RM with S concentrations high enough to quantify (Fig. S2c), it can be observed that MESS-3 reproduces relatively well with a S content of 0.10 ±0.08 % ($n = 7$; Table DR2). In comparison, duplicate analyses of stream sediment JSd-2 are highly variable ranging from 0.85 to 1.50 % S ($n = 4$). The observations herein are consistent with the heterogenous distribution of sulfide phases (i.e. "nugget effect") due to the small aliquot size (≤5 mg) utilized during EA measurements. Similarly, Alard et al. (2023) noted the difficultly of making reproducible S concentration measurements in a wide range of RM due to nugget effects. For S the larger aliquots

(c. 1 g) required to make XRF glass disks produce results consistent with the mean of the EA analyses and the previous determinations (Fig. S2).

3.1.2 Trace Element Data

The average composition of BHVO-2 measured herein is in excellent agreement (i.e. ±5 %) with previous determinations (Raczek et al., 2001; Albut et al., 2018; Chauvel et al., 2011) and the recommended values (Jochum et al., 2016) provided for all elements (Table DR3), except Sb ±6 %, Mo and Ta ±10 %, and W at ±19 %. These data are also precise with the average repeatability (±% RSD) better than ±5 % for the majority of elements, with only 5 more difficult to measure elements which are susceptible to memory effects (Mo, Sb, W, Ta and Bi; e.g. McGinnis et al., 1997; Wang et al., 2015; Braukmüller et al., 2020) having repeatability >10 % (Fig. 1a). For the REE (La to Lu), average repeatability (% RSD) and accuracy is very good; within ±4 % RSD, attesting to the robustness of REE analyses herein.

The remaining suite of sedimentary RM herein spans a wide compositional range (e.g. stream and marine sediments). Recommended values do not exist for these RM, with most studies only reporting concentrations for a limited selection of the 47 elements herein (Table DR3). HISS-1 especially is lacking several elements (i.e. n = 7), while the other RM often lack published values for certain elements including Bi, In, Tl, W, Mo. A further complication is that previous determinations of these RM often vary widely by up to 40 % for some elements making assessing accuracy difficult. Notwithstanding this complication, the determinations herein generally agree within ±10 % for the majority of elements for most sedimentary RM (Govindaraju, 1994; Imai et al., 1996; Dulski, 2001; Kumakura et al., 2004; Waheed et al., 2007; Bau and Alexander, 2009; Révillon and Hureau-Mazaudier, 2009; Chauvel et al., 2011; Shaheen and Fryer, 2011; Sampaio and Enzweiler, 2015; Fiket et al., 2017; Outridge et al., 2017; Roje, 2019), although some exceptions do exist (see Supplementary Material for details).

Siliciclastic sedimentary RM are inherently more heterogenous than basaltic rocks (e.g. BHVO-2) as they are aggregates from disparate sources (e.g. minerals and rock fragments). Figure 1 shows these RM have worse repeatability than BHVO-2, with most elements from a conventional element suite (i.e. first row transition metals, large ion lithophiles and REE) generally to within ±5 % RSD or ±10 % RSD. However, substantial anomalies do occur with Cu, Zr, Nb, Mo, Cd, Sn, Sb, Ta, W, Tl, U all having repeatabilities worse than ±15 % RSD for one or more RM. These poor repeatabilities between digestions, compared to basalt, suggests that nugget effects and/or other sources of heterogeneity. The repeatability of RM HISS-1 is particularly bad compared to the other RM, with > 20 % of the elements (n = 11/47) having repeatability worse than ±15 % RSD. When on focusing on the REE the markedly poorer repeatability is striking with 85 % of the REE (n = 12/14) having repeatability worse than ±12 % RSD, whereas for all other RM most REE have average repeatability ≤±5 % RSD (Fig. 1).

3.2 Precision and accuracy of isotopic measurements

3.2.1 Oxygen Isotopes

No previously published $\delta^{18}O$ data could be found for the sedimentary RM herein. Oxygen (O) isotope analyses were conducted in at least 15 different analytical sessions, across two years. At QFIR the widely utilized NBS 28 (NIST RM 8546) was measured repeatedly and produced an average composition of $\delta^{18}O$ = 9.60 ±0.30 ‰ (n = 18; 2SD; relative to V-SMOW; Fig. S3), which is within uncertainty of previous determinations (Reed, 1992; Baker et al., 2000; Hou et al., 2003). Alkali basalt OREAS 24 P was also analyzed as a secondary check on measurement repeatability and produced an average value of $\delta^{18}O$ = 7.30 ±0.25 ‰ (n = 17; Fig. S3; Table DR5). Consequently, the long-term repeatability of silicate RM for $\delta^{18}O$ is ≤±0.30 ‰. All the sedimentary RM possess worse repeatability than that established from the primary RM (Fig. 2a). Focusing on the repeatability of RM with >3 duplicates, it is clear both marine sediments (e.g. HISS-1, MESS-3), possess significant heterogeneity, which is potentially compounded by the small sample size used for these analyses (<5 mg; see Section 2.2).

3.2.2 Fe Isotopes

Iron isotope analyses were conducted in at least 7 separate analytical sessions (Table DR6). To monitor long-term instrument performance, an in-house High Purity Standards 1000 µg/mL Fe solution (Lot# 2122826) was measured repeatedly. Data obtained at both JCU and MQU are within uncertainty of each other (Fig. S4a) with an average of $\delta^{56}Fe$ = -0.307 ±0.046 ‰ (n = 51; relative to IRMM-524B; Table DR4). Additionally, the Durham University Fe-Wire solution was also analyzed and produced a $\delta^{56}Fe$ value of 0.241 ±0.037 ‰ (n = 13), within uncertainty of previous determinations (Fig. S4b; Gerrits et al., 2019). An additional confirmation of the quality of the Fe isotope data is that despite varying over a wide range, all analyses fall within uncertainty of the theoretical line of mass dependence (Fig. S5), confirming no unresolved analytical artefacts affect this dataset. Previously published Fe isotope determinations are not available for most of the RM. Therefore, BHVO-2 must be used to validate the accuracy of the RM processed through chemistry. Replicate measurements of BHVO-2 ($\delta^{56}Fe$ = 0.095 ±0.047 ‰; n = 4; Fig. 2b) are within uncertainty of previous determinations (Craddock and Dauphas, 2011; Millet et al., 2012; McCoy-West et al., 2018). Duplicate analyses (n = 4–6) of the other RM, have $\delta^{56}Fe$ varying by ≤±0.05 ‰, except for HISS-1, which is only marginally worse at ≤±0.07 ‰ (Fig. 2b), showing Fe is less affected by heterogeneity.

3.2.3 Sr Isotopes

Strontium isotope analyses were conducted in at least 7 different analytical sessions (Table DR7). To monitor

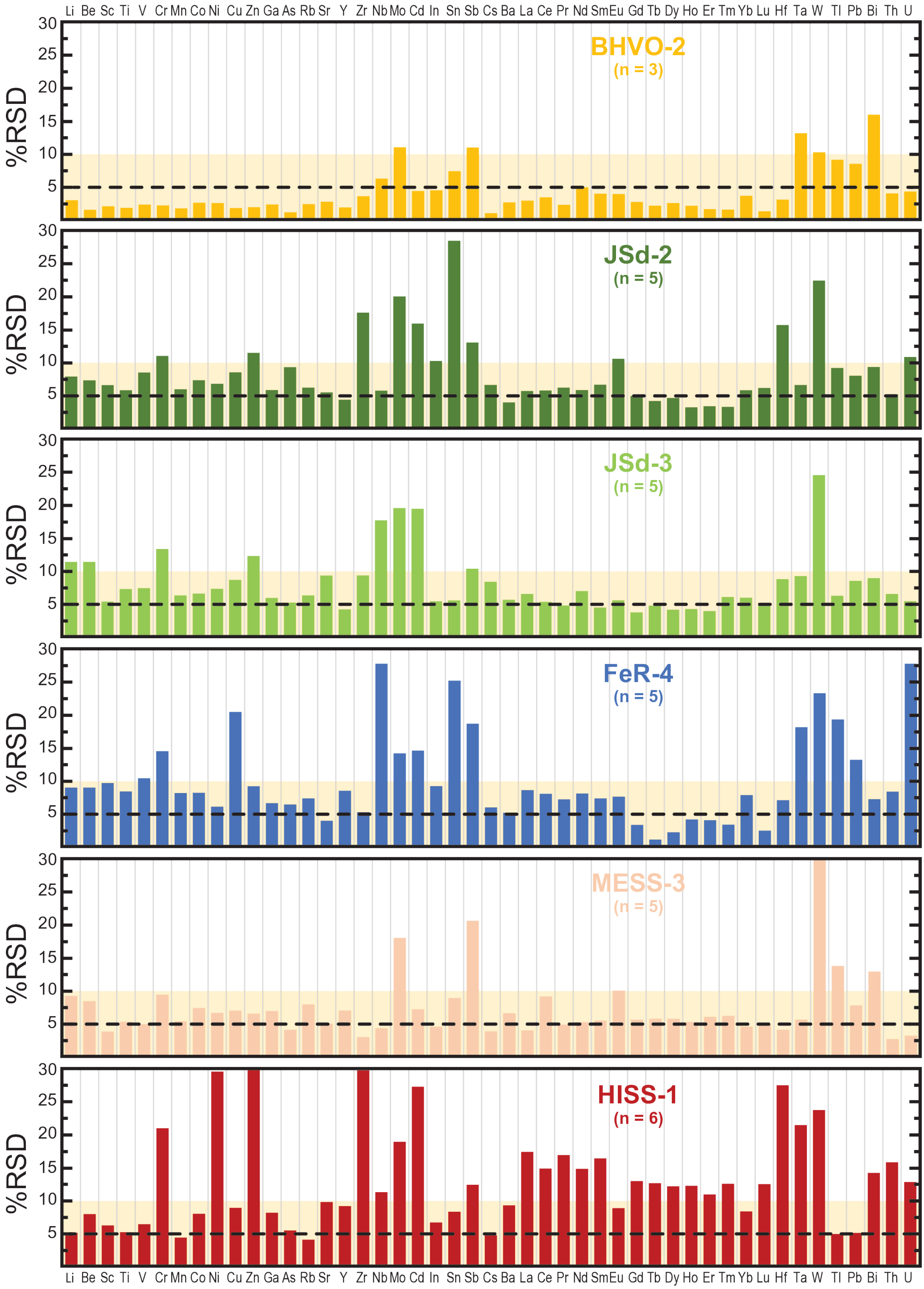

Figure 1. Long-term repeatability of all trace elements investigated here depending on the analyzed reference material. Notice that depending on the type of rocks (e.g. basalt or sediment), the repeatability changes consistent with inherent heterogeneity of the original material. Marine sediment HISS-1 has consistently worse repeatability than other RM even for elements that are conventionally considered easier to measure (e.g. REE). Several elements are also susceptible to nugget effects, or difficult to measure at low concentrations due to memory effects, contributing to poor repeatability spikes.

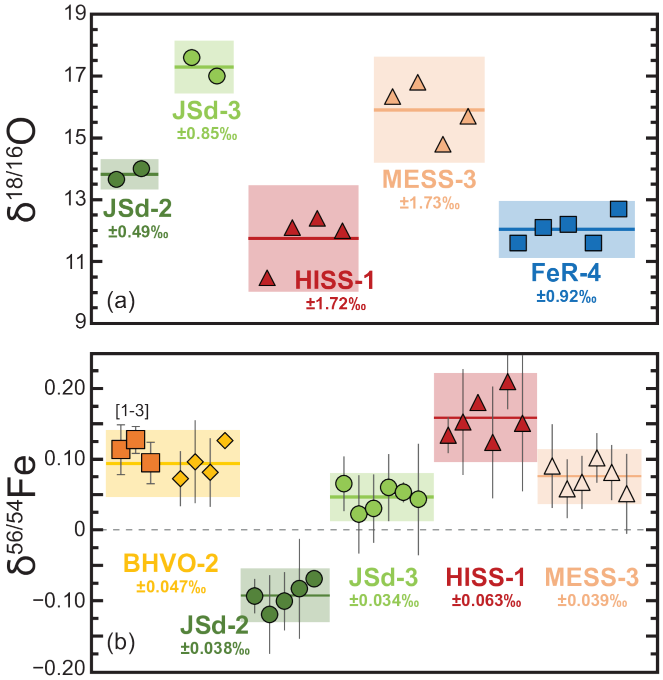

Figure 2. Long-term repeatability of O isotope measurements (a) and Fe isotope measurements (b). Uncertainty bars are measured 2 standard deviation values, with shaded fields representing average value (line) ±2 standard deviations. For Fe due to the small number of replicate measurements (i.e. <3) for some of the digestions calculated 2 SD on duplicates can fluctuate being either large or small, however, the mean value remains close to that observed in the other digestions. FeR-4 is not shown due to its significantly heavier Fe isotope composition. Orange squares represent a selection of published values for BHVO-2: [1] Craddock and Dauphas (2011), [2] Millet et al. (2012), [3] McCoy-West et al. (2018).

long-term instrument performance the in-house High Purity Standards 1000 µg/mL Sr solution (Lot# 1305924) was measured repeatedly. Data obtained at both JCU and MQU are within uncertainty of each other with an average of ^{87}Sr/^{86}Sr = 0.707551 ±24 (uncertainty in ppm; n = 39; Fig. S6a). Duplicate digestions of BHVO-2 have an average ^{87}Sr/^{86}Sr of 0.703469 ±34 (n = 6), within uncertainty of previously determinations (Weis et al., 2006; McCoy-West et al., 2010; Jo et al., 2021). Analyses of granite G-2 and stream sediment JSd-2 are also within uncertainty of published values (Table DR4). Whereas the Sr isotope compositions of some other RM (JSd-3 and HISS-1) show significant variability in Figure 3. Despite this intra-replicate variability, HISS-1 remains within uncertainty of the previous determination by Jo et al. (2021), whereas the ^{87}Sr/^{86}Sr of JSd-3 is resolvably different (Table DR4). Given that exogenous Sr contamination is excluded (high Sr concentration of Sr = 58.6 µg/g, and negligible Sr blanks), this suggests heterogeneity between different RM splits.

3.2.4 Nd Isotopes

Neodymium isotope analyses were conducted in at least 6 separate analytical sessions (Table DR8). To monitor long-term instrument performance the inter-laboratory solution Mona (where Mona is a Monash University High Purity Standards 1000 µg/mL Nd solution Lot# 1601901) was measured repeatedly. Data obtained at both JCU and MQU are within uncertainty of each other, with an average of ^{143}Nd/^{144}Nd = 0.511555 ±10 (n = 40; Fig. S6b), and within uncertainty of double-spike measurements (McCoy-West et al., 2020; Kaufmann and McCoy-West, 2025). Duplicate digestions of BHVO-2 are highly repeatable with an average ^{143}Nd/^{144}Nd = 0.512996 ±8 (n = 4), within uncertainty of previous determinations (Weis et al., 2006; McCoy-West et al., 2010; Chauvel et al., 2011; McCoy-West et al., 2020). Additionally, measurements of a range of terrestrial and marine RM (JSd-2, JSd-3, HISS-1) are also within uncertainty of published values (Chauvel et al., 2011; Jo et al., 2021). This confirms the Nd isotope measurement technique implemented here is accurate, with no unresolved isobaric interferences from Sm. Five of the six RM that were replicated (n = 4–5) have repeatability's (±2 SD) ≤23 ppm, with three RM (BHVO-2, JSd-2, MESS-3) showing excellent long-term repeatability (≤15 ppm; Table DR4). Only HISS-1 displays significantly worse repeatability (^{143}Nd/^{144}Nd = 0.511869 ±36; n = 5; Fig. 3b), consistent with the variability observed in this RM for a range of parameters.

3.3 Heterogeneity of sedimentary reference materials

During preparation of RM significant care is taken to produce a fine homogenous powder from large quantities of material. However, the effectiveness of this process is a function of the grain size and inherent variability of the constituents of the original rock (i.e. variable proportions of crystals, sulfide or metallic nuggets, or rock fragments). Given siliciclastic sediments are comprised of potentially disparate and chemical distinctive material in variable proportions, they possess a higher chance of intra-measurement heterogeneity which should be considered when seeking to interpret anomalous measurements.

If a RM or sample possesses significant variability in multiple parameters obtained through different analytical techniques it is more likely to be the result of inherent heterogeneity than an analytical artefact. Marine sediment RM HISS-1 appears heterogenous from both an elemental and isotopic perspective, with excess variability in volatile elements (Fig. S2), numerous trace elements (Fig. 1), stable oxygen isotopes (Fig. 2) and radiogenic Sr and Nd (Fig. 3). Previously, Choi et al. (2013) observed poor repeatability of U & Th concentrations, while Ghidan and Loss (2010) observed Zn isotope heterogeneity. During preparation, HISS-1 was only milled to a grain size of ≤125 µm (coarser than other RM) thus more intra split/digestion heterogeneity is not unexpected. This also provides a mechanism to explain the variability. For example, the significantly worse repeatability of the REE (Fig. 1) is probably due to incorporation of variable proportions of carbonate (which strongly incorporate REE; Zhong and Mucci, 1995) and detrital silicates in the different digestions. These two endmembers would also possess distinctive radiogenic and

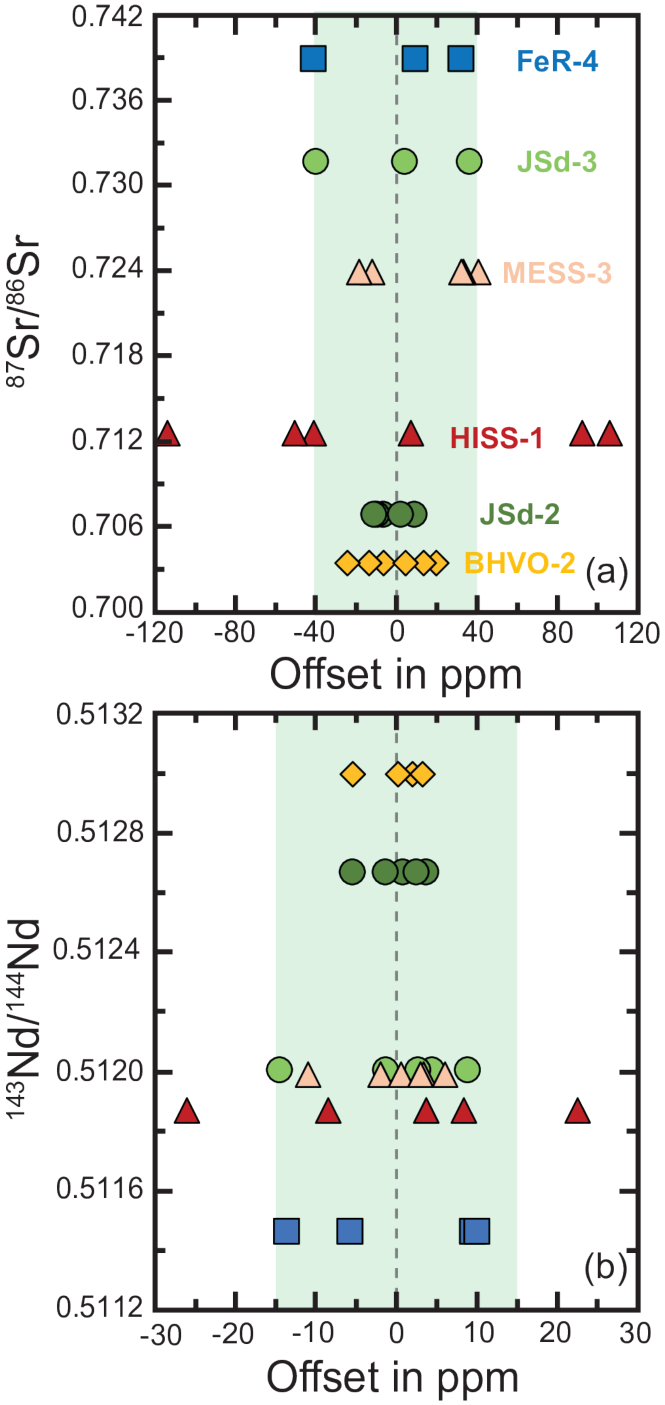

Figure 3. Comparison of the $^{87}Sr/^{86}Sr$ (a) and $^{143}Nd/^{144}Nd$ (b) isotope compositions of RM and the repeatability of replicate digestions of each RM. The offset in parts per million (ppm) is calculated relative to the average composition of all replicates measured herein. The shaded bar approximately represents a long-term repeatability of each isotopic measurement at ±40 ppm for $^{87}Sr/^{86}Sr$ and ±15 ppm for $^{143}Nd/^{144}Nd$, with the majority of duplicates falling within this range except some digestions of HISS-1.

stable isotope compositions (i.e. marine versus detrital components) contributing to the greater diversity observed in these parameters for HISS-1 (Figs. 2–3). Consequently, we attribute the worse repeatability of HISS-1 to inherent heterogeneity, rather than any analytical issues in this dataset.

Marine RM MESS-3 also displays substantial O isotope heterogeneity (Fig. 2a) but appears homogenous within analytical uncertainty for the other elemental and isotopic parameters investigated here (e.g. Figs. 2–3). Despite their larger particle size (≤147 µm) the riverine GSJ RM (e.g. JSd-2 and JSd-3) are presumably less affected by heterogeneity issues than HISS-1 due to being sourced solely from a continental source (i.e. no heterogeneity imparted from the marine realm). Independent of the parameter under investigation (i.e. trace elements or isotopes) sedimentary RM have worse repeatability metrics (% RSD) than BHVO-2 (Figs. 2–3). We attribute these poorer repeatability metrics (% RSD) to several factors: 1) the intrinsic heterogeneity of siliciclastic sedimentary materials (i.e. variable source component proportions, e.g. carbonate or organic matter proportions); 2) coarser particle size of some RM which leads to a greater potential for intra-digestion heterogeneity; and 3) the potential for intra spilt differences between bottles distributed by the manufacturer (e.g. significant Sr isotope discrepancy for JSd-3 between herein; Jo et al., 2021).

This dataset also provides several important observations for future geochemical studies: 1) the characterization of multiple parameters synchronously on the same material is helpful to resolve analytical problems from intrinsic sample variability; 2) to disentangle sample heterogeneity issues from analytical artefacts, it is highly recommended to run well-characterized RM (e.g. BHVO-2) alongside less-well constrained RM; and 3) when attempting to resolve compositional differences between siliciclastic sedimentary materials due to their intrinsic heterogeneity more variability, should be expected and therefore wider uncertainty envelopes applied (i.e. ±3 SD).

4 Summary

We undertook a multi-pronged approach (XRF, EA, TQ-ICP-MS, IRMS and MC-ICP-MS) to characterize siliciclastic sedimentary riverine and marine RM relevant to environmental impact assessments. The combination of major, volatile, trace and stable (O-Fe) and radiogenic isotope (Sr-Nd) data from the same RM allows analytical artefacts to be distinguished from inherent sample heterogeneity. When working with siliciclastic sedimentary samples the intrinsic natural variability needs to be considered when interpreting repeatability metrics and thus wider uncertainty envelopes would be prudent when making interpretations. Quality assurance is of highest criticality during environmental applications, given results are often used in the determination of contaminant provenance (and therefore liability) which can have wide reaching implications.

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Data, code, and outputs availability

All analytical data is available in the EarthChem Library (McCoy-West et al., 2025, https://doi.org/10.60520/IEDA/113997). Main text figures are available for download in the online version of this article.

Competing interests

The authors declare no competing interests.

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