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Precision and reproducibility of cosmogenic ^{10}Be and ^{26}Al measurements assessed from a multi-laboratory compilation of duplicate and reference material analyses

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Abstract.

To quantify reproducibility and precision of cosmogenic ^{10}Be and ^{26}Al analyses, we analysed a suite of liquid and quartz reference materials, together with duplicate preparations of unknown research samples. Comparison among these materials allows us to constrain uncertainties associated with AMS measurement, chemical extraction, and quartz purification, and to assess whether additional variability is introduced at successive stages of the analytical process. Samples were prepared in three chemistry laboratories at the Australian Nuclear Science and Technology Organisation (ANSTO), the University of Wollongong (UOW), and Imperial College London, and subsequently analysed by accelerator mass spectrometry (AMS) using the 6 MV SIRIUS accelerator at ANSTO. All three laboratories showed intra-laboratory variability below the routine 2% analytical uncertainty, while measured values were consistent with the nominal values of the reference materials. These results indicate that both methodological reproducibility and inter-laboratory variability are also within the analytical uncertainty, and provide no evidence for additional uncertainty associated with processing quartz relative to liquid reference materials. The implications for cosmogenic-nuclide research are that: (1) the reproducibility and precision of cosmogenic ^{10}Be and ^{26}Al analyses are typically 2–3% when sufficient counting statistics are achieved; (2) at lower isotope concentrations, counting statistics and/or background corrections may limit the achievable measurement uncertainty, although the resulting variability remains consistent with the reported analytical precision; and (3) precision of approximately 1% is achievable for high-concentration liquid reference materials, as demonstrated by Be-KN-5-4 and Al-KN-4-3, but has not yet been demonstrated for quartz samples.

1 Introduction

Cosmogenic ^{10}Be and ^{26}Al have become important tools for quantifying the timing and rates of processes that shape the Earth's surface, providing constraints on surface exposure, erosion, and landscape evolution over timescales of thousands to millions of years (Schaefer et al., 2022). Robust interpretation of these applications depends critically on the accuracy and precision with which cosmogenic ^{10}Be and ^{26}Al concentrations are measured. Analytical uncertainties that may affect the accuracy and/or

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precision of the results can be broadly divided into two sources: those associated with the chemical extraction of cosmogenic isotopes and subsequent sample preparation, and those arising from the accelerator mass spectrometry (AMS) measurement itself. Additional uncertainties are introduced when measured isotope concentrations are converted to exposure ages or erosion rates. These include, for example, uncertainties in production rates (Kubik et al., 1998; Balco et al., 2009; Stroeven et al., 2015; Kelly et al., 2015; Corbett et al., 2017; Fenton et al., 2019, 2022), scaling factors (Dunai, 2000; Desilets et al., 2006), atmospheric air pressure (Stone, 2000; Staiger et al., 2007), geomagnetic variations (Pigati and Lifton, 2004), solar modulation (Bard et al., 1997), and the ^{10}Be half-life (Nishiizumi et al., 2007; Chmeleff et al., 2010; Korschinek et al., 2010). These additional sources of uncertainty are not considered here; instead, we focus specifically on uncertainties introduced during sample preparation and AMS measurement.

The reproducibility and precision of AMS measurements are typically assessed through a combination of in-house quality-assurance procedures (e.g., Arnold et al., 2010; Rood et al., 2010; Xu et al., 2015), and interlaboratory round-robin exercises (e.g., Merchel and Bremser, 2004; Nishiizumi, 2004; Nishiizumi et al., 2007; Merchel et al., 2012; Braucher et al., 2015; Nishiizumi, 2022). These assessments generally involve repeated measurements of either ion-source-ready reference materials or liquid reference materials that require only minimal processing prior to analysis. The observed variability among replicate measurements is then compared with the quoted analytical uncertainty of individual measurements and with the accepted values of the reference materials. Previous studies indicate that AMS measurement uncertainties can approach 1% when sufficient counting statistics are achieved, with measured values consistent with the nominal values and uncertainties of the reference materials (Nishiizumi, 2022). However, interlaboratory agreement is not always as good, and larger systematic differences between laboratories have been reported (Merchel et al., 2012).

AMS measurement uncertainty, however, does not capture uncertainties introduced during sample handling and chemical preparation. To address this limitation, several geological reference materials have been developed, including CRONUS-A and CRONUS-N (Jull et al., 2015), CoQtz-N (Binnie et al., 2019), and more recently, UVM-A (Corbett et al., 2019, 2024). These materials can be processed independently in satellite laboratories before being submitted to AMS facilities for measurement, allowing the contribution of sample preparation to the overall analytical uncertainty to be evaluated. By comparing quartz reference materials (e.g., CRONUS-A, CRONUS-N, and CoQtz-N) with a liquid reference material (UVM-A), the contributions of different stages of the analytical workflow to the total uncertainty can be assessed. Reported standard deviations for ^{10}Be analyses of CRONUS-A and CoQtz-N are 2.9% and 3.8%, respectively, while those for ^{26}Al analyses of the same materials are 4.9% and 10.4% (Jull et al., 2015; Binnie et al., 2019). For the liquid reference material UVM-A, the reported standard deviations are 4.1% for ^{10}Be and 4.9% for ^{26}Al (Corbett et al., 2024).

Although existing reference materials therefore provide important constraints on AMS measurement uncertainty and on the reproducibility of ^{10}Be and ^{26}Al extraction by wet chemistry, they do not capture the full analytical workflow or all conditions encountered in routine research samples. In particular, quartz purity is also critical, as discussed by Merchel et al. (2019) and Corbett et al. (2022), because incomplete removal of contaminant phases may affect measured isotope concentrations. Extending the technique to samples with lower isotope concentrations introduces further challenges, including background correction and potential non-linearity when isotope concentrations in samples and standards span three orders of magnitude or more.

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Suitable reference materials may also be unavailable at such low concentrations, making it difficult to independently assess measurement accuracy and precision. Consequently, there remains a need to evaluate analytical performance across the full sequence from quartz purification and chemical extraction to AMS measurement, and to determine whether the uncertainties observed in routine research samples are consistent with those inferred from reference materials and AMS quality-assurance measurements.

To address these limitations, we present an internally consistent assessment of analytical uncertainties in ^{10}Be and ^{26}Al analyses conducted across three sample-preparation laboratories at Australian Nuclear Science and Technology Organisation (ANSTO), the University of Wollongong, and Imperial College London. All samples were measured using the 6 MV SIRIUS accelerator at ANSTO (Pastuovic et al., 2016; Wilcken et al., 2017, 2019, 2022), thereby minimising variability associated with differences between AMS facilities and allowing uncertainties introduced at different stages of sample preparation to be compared directly. Our assessment combines several types of quality-assurance material, each designed to constrain a different component of the analytical workflow: secondary standards to quantify AMS measurement uncertainty; geological reference materials (CRONUS-A, UVM-A, and CoQtz-N) to assess uncertainty introduced during chemical extraction and sample preparation; and duplicate research samples to evaluate additional variability associated with quartz extraction and purification, and with lower isotope concentrations. Together, these datasets allow us to test whether uncertainty increases progressively through the analytical workflow and whether the reproducibility and precision achieved for routinely processed quartz samples are consistent with those inferred from reference materials and AMS measurements alone.

2 Methodology

2.1 Accelerator Mass Spectrometry

A key principle of AMS methodology is that samples are measured relative to a standard reference material with a known or assumed isotopic composition (Fifield, 2025). This approach enables correction for measurement losses, provided that samples and standards behave similarly within the spectrometer and that isotopic fractionation is sufficiently small for the measured isotope ratios of both samples and standards to deviate from their true values by the same proportional factor. This normalisation factor may vary over the course of a measurement or may be related to the sample beam current, with different AMS laboratories employing different normalisation strategies.

The dominant sources of ion loss and isotopic fractionation during AMS measurement are negative-ion formation in the ion source and beam transmission through the accelerator as ions are converted from negative to positive charge states. Additional ion-optical losses may be caused by various apertures or restrictions in the beam path, and ion-detection efficiency. By far the largest losses arise from inefficient negative-ion formation in the ion source, with ionisation yields of $\sim 3\%$ reported for BeO^- (Rood et al., 2010) and approximately an order of magnitude lower for Al^- (Auer et al., 2007). Extraction of AlO^- can substantially increase the ionisation efficiency; however, measurement of ^{26}Al then requires additional suppression of the interfering isobar ^{26}Mg, for example using a gas-filled magnet (Miltenberger et al., 2017) or ion-laser interaction mass spectrometry (Lachner et al., 2021). These approaches are not considered further here.

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All AMS measurements presented in this study were performed using SIRIUS, a conventional 6 MV Pelletron accelerator at ANSTO (Pastuovic et al., 2016; Wilcken et al., 2017, 2019, 2022). The transmission of $^{9}Be^{3+}$ through the accelerator as a function of the $^{9}BeO^{-}$ current extracted from the ion source is shown in Figure 1A for a range of samples measured repeatedly at different times during a run. Each data point represents a single measurement, typically lasting 1–10 minutes. Individual samples are generally measured 3–10 times. As sputtering progressively consumes the sample, the ion-source output decreases, resulting in measurements that populate the transmission curve relatively uniformly up to approximately 15 µA. Figure 1A shows that beam transmission remains approximately constant up to a BeO^{-} current of about 10 µA, above which transmission begins to decrease as the beam becomes too large to pass cleanly through the accelerator terminal. To maximise measurement stability and avoid a current-dependent normalisation factor between the measured and true $^{10}Be/^{9}Be$ ratios, the maximum BeO^{-} current from primary standards is therefore limited to approximately 10 µA. This reduces the potential for current-dependent isotopic fractionation within the accelerator. The spread in beam transmission shown in Figure 1A reflects two main factors. First, some variability in transmission occurs between individual samples. Second, the relationship between transmission and BeO^{-} current varies slightly for each analysis time. The anomalously low transmission values observed at low ion currents generally occur towards the end of sample consumption, when nearly exhausted samples produce broader and more distorted beams from the ion source.

A practical way to assess the stability of the normalisation factor across a range of ion-source outputs is to measure a sample with a known $^{10}Be/^{9}Be$ ratio repeatedly until it is exhausted (Figure 1B). Alternatively, samples with a known $^{10}Be/^{9}Be$ ratio can be diluted so that they have progressively smaller Be masses, thereby producing lower beam currents relative to the primary standard (Figure 1B; replotted from Wilcken et al., 2019). Both the diluted samples and those measured to exhaustion are shown in Figure 1B as a function of relative BeO^{-} current, normalised to the average output of the primary standards measured alongside them ($\sim$ 10 µA). Neither the diluted samples nor those measured to exhaustion show any systematic deviation from the known reference value beyond the analytical measurement uncertainty. This demonstrates that, over the range of beam currents investigated, the normalisation used in our methodology is not measurably dependent on ion-source output.

2.1.1 ^{10}Be measurements

BeO is mixed with Nb at a 1:4 mass ratio (1:1 molar ratio) before the samples are pressed into Cu sample holders. Approximately 10 µA of BeO^{-} is extracted from the sample. The accelerator is operated at 6 MV with an Ar gas stripper, and the 3+ charge state is selected for analysis. Transmission from BeO^{-} to Be^{3+} is approximately 35%, depending on the sample and ion-source output (Figure 1A). ^{10}B interference is suppressed using a passive absorber cell before ^{10}Be ions are detected with a multi-anode gas-ionisation chamber. Measurements are normalised to the Nishiizumi KN-5-3 standard, with a nominal value of 6.320×10^{-12} (Nishiizumi et al., 2007).

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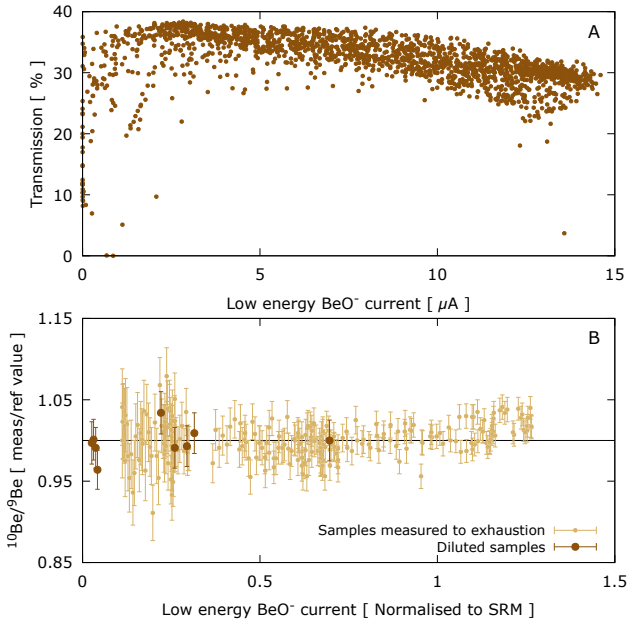

Figure 1. (A) Transmission of $^{9}Be^{3+}$ through the accelerator as a function of beam current, shown for a compilation of standard reference materials and unknown research samples. Lower-ratio samples were measured for longer periods, in some cases to exhaustion, whereas higher-ratio samples required much shorter measurement times. (B) The measured ^{10}Be/^{9}Be ratio relative to the known reference value as a function of beam current, shown for both samples measured to exhaustion and diluted samples producing lower beam currents relative to the standard reference material (SRM). The average SRM beam current was approximately 10 μA, and the 1:1 line is shown for reference. Data for the diluted samples are replotted from Wilcken et al. (2019).

2.1.2 ^{26}Al measurements

Al_2O_3 is mixed with Ag powder at a 1:2 mass ratio (1:1 molar ratio) before the samples are pressed into Cu sample holders. The accelerator is operated at 4 MV with an Ar gas stripper, and the 3+ charge state is selected for analysis. Transmission from Al^- to Al^{3+} is typically 36%–38%. Ions are detected using a multi-anode gas ionisation chamber with a 75 nm thick SiN membrane as the detector window. Measurements are normalised to the Nishiizumi KN-4-2 standard, with a nominal value of 3.096×10^{-11} (Nishiizumi, 2004).

2.2 Sample preparation

All samples measured as part of this work followed standard preparation procedures at one of three chemistry laboratories: ANSTO, the University of Wollongong, or the CosmIC laboratories at Imperial College London.

Primary and secondary standards were prepared at ANSTO from the Nishiizumi 2007 solutions KN-5-3, KN-5-4, and KN-6-3 for ^{10}Be, and the 2004 solutions KN-4-2, KN-4-3, and KN-5-2 for ^{26}Al (Nishiizumi, 2004; Nishiizumi et al., 2007). The

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standards were precipitated, washed, dried, and ignited to oxide before being mixed with Nb or Ag for Be and Al, respectively, and pressed into Cu sample holders.

Samples prepared at the University of Wollongong followed the procedures detailed in Codilean et al. (2023). For quartz samples, including the CoQtz-N reference material and the unknown duplicate samples, low background ^{9}Be carrier was added, targeting 200–300 µg Be per sample. Low background ^{27}Al carrier was added only where required to achieve a minimum of 1.5 mg of total Al (i.e., native Al plus carrier) per sample. The quartz samples were then dissolved in concentrated HF and dried to completeness. The residues were refluxed in aqua regia to destroy insoluble fluorides, evaporated, and redissolved for subsequent anion and cation exchange chromatography. For samples analysed for ^{26}Al, Al was separated from Be by pH-dependent precipitation prior to cation-exchange chromatography. Following chemical separation, Be hydroxide gels were converted to oxides using a propane/butane torch, whereas Al hydroxides were converted to oxides by heating in an ashing furnace at 1000°C. For the liquid reference material UVM-A, approximately 1.5 mL of material was added directly to 100 mL of concentrated HF and dried to completeness. The subsequent chemical separation followed the procedure described in Codilean et al. (2023) to obtain Be and Al hydroxides, which were converted to oxides as described above.

Samples prepared at the CosmIC laboratories at Imperial College London followed methods similar to those described in Corbett et al. (2016). Typically, approximately 0.5 g of CRONUS-A and 4–30 g of pure quartz were dissolved for duplicate samples prior to Be and Al extraction and purification. Low-background ^{9}Be and ^{27}Al carriers were added, targeting ∼260 µg Be per sample and ∼2.5 mg of total Al per sample. Quartz was dissolved using a mixture of concentrated HF and HNO_3. Dissolved samples were then evaporated, fumed with $HClO_4$ to remove residual fluorides and convert the samples to chloride form, and passed sequentially through anion and cation exchange columns to isolate Be and Al. Hydroxides were converted to oxides by heating in an ashing furnace at 750°C.

3 Results

To isolate sources of analytical uncertainty and assess reproducibility and precision, we examined three types of quality-assurance materials and approaches that were progressively more representative of the full analytical procedure: liquid reference materials, quartz reference materials, and duplicate samples.

3.1 Liquid reference materials

Liquid reference materials are prepared in bulk, with individual samples subsequently taken as aliquots from the parent solution. The parent solution can be assumed to be homogeneous, so variability between aliquots is not expected to reflect sample heterogeneity. The extent of subsequent sample preparation differs between the two liquid reference materials used in this study. The Nishiizumi dilution series (Nishiizumi, 2004; Nishiizumi et al., 2007) requires only minimal preparation, whereas UVM-A (Corbett et al., 2019, 2024) undergoes the full chemical separation procedure. Consequently, the spread in measured values and deviations from the nominal value reflect different combinations of sample-preparation, contamination, and measurement uncertainties, depending on the reference material.

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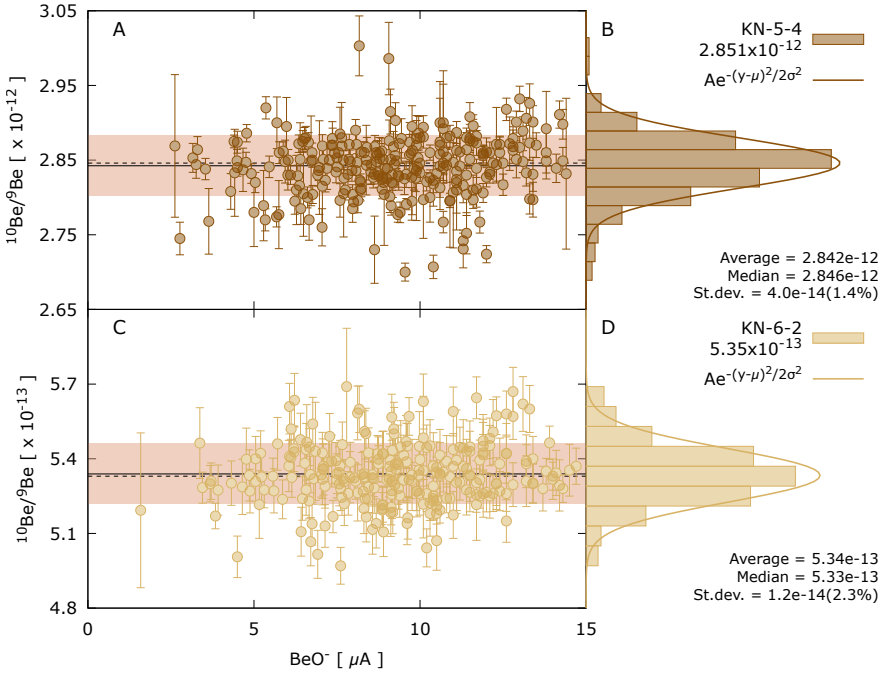

Figure 2. Measurements of KN-5-4 (A & B) and KN-6-2 (C & D) between 2016 and 2025, normalised to KN-5-3. Measured ^{10}Be/^{9}Be ratios show no dependence on beam current over ∼3–15 µA (A & C), and their distributions are consistent with fitted Gaussians (B & D). Solid lines indicate the mean, dashed lines the median, and shaded areas the standard deviation. Mean and median values agree with the nominal values of KN-5-4 $= 2.851 \times 10^{-12}$ and KN-6-2 $= 5.35 \times 10^{-13}$. Standard deviations are 1.4% and 2.3%, respectively, with the lower spread for KN-5-4 reflecting its typically smaller measurement uncertainty.

3.1.1 Secondary standards

Standard practice in AMS laboratories is to include secondary standards in each measurement batch for quality assurance and to guide the choice of normalisation scheme where time-dependent or variable normalisation is required. To assess the long-term performance of SIRIUS, measurements of routinely used secondary standards between 2016 and 2025 were compiled for both ^{10}Be and ^{26}Al. For ^{10}Be, KN-5-4 and KN-6-2 have nominal ^{10}Be/^{9}Be ratios of 2.851×10^{-12} and 5.35×10^{-13}, respectively, and are shown in Figure 2. For ^{26}Al, KN-4-3 and KN-5-2 have nominal ^{26}Al/^{27}Al ratios of 1.065×10^{-11} and 1.812×10^{-12}, respectively, and are shown in Figure 3.

For ^{10}Be, 270 measurements of KN-5-4 and 249 measurements of KN-6-2 show no correlation between ^{10}Be/^{9}Be ratio and BeO^- current (Figure 2A&C). Their distributions are normal (Figure 2B&D), with mean values of 2.842×10^{-12} and 5.34×10^{-13} and standard deviations of 1.4% and 2.3%, respectively. The ^{26}Al standards show the same behaviour: 136 measurements of KN-4-3 and 122 measurements of KN-5-2 show no correlation between ^{26}Al/^{27}Al ratio and Al^- current (Figure 3A&C), and their distributions are normal (Figure 3B&D), with mean values of 1.068×10^{-11} and 1.812×10^{-12} and

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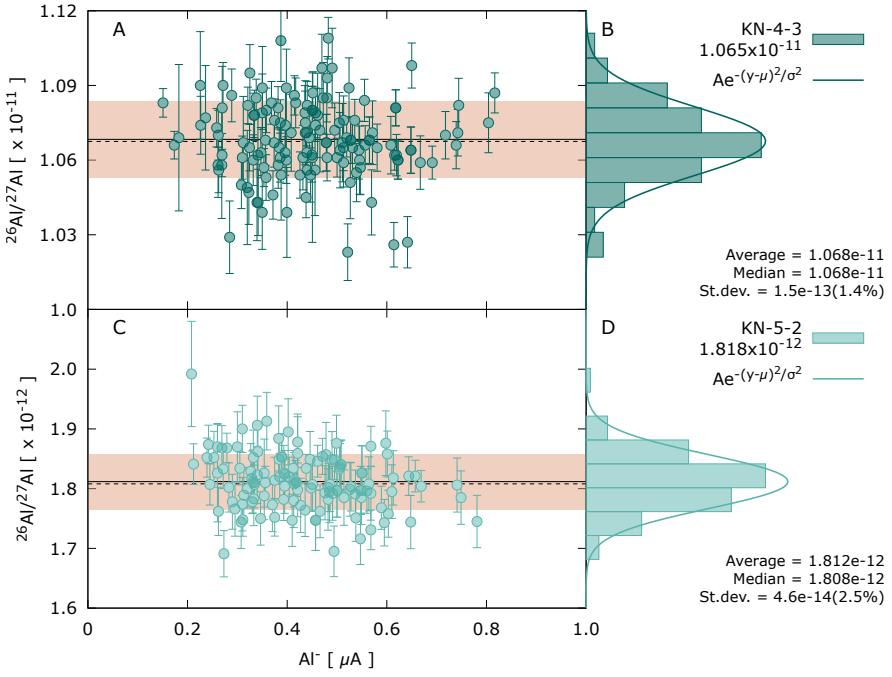

Figure 3. Measurements of KN-4-3 (A & B) and KN-5-2 (C & D) between 2016 and 2025, normalised to KN-4-2. Measured ^{26}Al/^{27}Al ratios show no dependence on beam current over ∼0.2–0.8 µA (A & C), and their distributions are consistent with fitted Gaussians (B & D). Solid lines indicate the mean, dashed lines the median, and shaded areas the standard deviation. Mean and median values agree with the nominal values of KN-4-3 $= 1.065 \times 10^{-11}$ and KN-5-2 $= 1.812 \times 10^{-12}$. Standard deviations are 1.4% and 2.5%, respectively, with the lower spread for KN-4-3 reflecting its typically smaller measurement uncertainty.

standard deviations of 1.4% and 2.5%, respectively. For all four secondary standards, the measured mean ratios agree well with the nominal values, and the observed spread is consistent with the individual measurement uncertainties.

3.1.2 UVM-A

UVM-A is a homogeneous, pre-spiked liquid reference material developed at the University of Vermont for quality assurance of cosmogenic ^{10}Be and ^{26}Al sample preparation and AMS analysis (Corbett et al., 2019, 2024). It was produced by dissolving 267.60 g of the CRONUS-A quartz reference material (Jull et al., 2015) in concentrated HF, combining the resulting solutions, and adding ^{9}Be and ^{27}Al carrier. Following evaporation and treatment with $HClO_4$, the material was redissolved in 1% HNO_3 and diluted to a total volume of 4 L, which was subsequently split into 30 mL bottles for distribution. The UVM-A material analysed in this study was taken from two of these 30 mL bottles. The most recent study of UVM-A, based on repeated preparation and analysis over five years at the University of Vermont, reports preliminary consensus values of $(1.45 \pm 0.06) \times 10^{-13}$ for ^{10}Be/^{9}Be ($n = 96$) and $(4.47 \pm 0.22) \times 10^{-13}$ for ^{26}Al/^{27}Al ($n = 27$), where uncertainties represent one standard deviation (Corbett et al., 2024).

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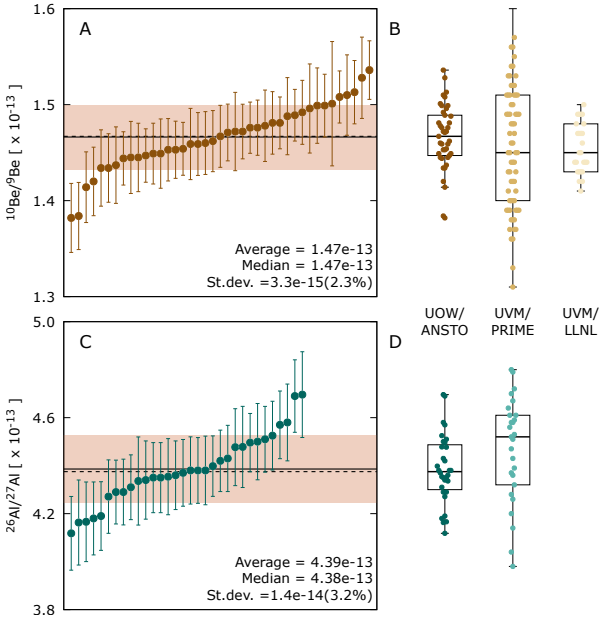

Figure 4. Measurements of the UVM-A reference material for ^{10}Be/^{9}Be ratios (A & B) and ^{26}Al/^{27}Al ratios (C & D) from 2022 to 2026, normalised to KN-5-3 for ^{10}Be/^{9}Be and to KN-4-3 for ^{26}Al/^{27}Al. The average (solid line), median (dashed line), and standard deviation (shaded area; $\pm 1\sigma$) are shown. Box plots compare our measurements with the earlier distribution reported by Corbett et al. (2024).

For $n = 41$ analyses of the UVM-A liquid reference material, the average ^{10}Be/^{9}Be ratio is 1.47×10^{-13}, with a median of 1.47×10^{-13} and a standard deviation of 3.3×10^{-15} (2.3%). The UOW / ANSTO data are in good agreement with the nominal value of $(1.45 \pm 0.06) \times 10^{-13}$ reported by Corbett et al. (2024). The slightly smaller standard deviation of our data, 2.3% compared with 4.2% reported by Corbett et al. (2024), may reflect differences in measurement precision between AMS laboratories. The values reported by Corbett et al. (2024) were measured using accelerators at the Purdue Rare Isotope Measurement Laboratory (PRIME) and Lawrence Livermore National Laboratory (LLNL). Individual UVM-A measurements at PRIME consistently have larger analytical uncertainties (average of 3.9%) than those at LLNL (2.1%) or ANSTO (2.4%), and also show a larger spread, with a standard deviation of 7.0×10^{-15} compared with 2.6×10^{-15} at LLNL and 3.4×10^{-15} at ANSTO (Figures 4A&B). We analysed $n = 32$ samples of UVM-A material for the ^{26}Al/^{27}Al ratio (Figures 4C&D), obtaining an average of 4.39×10^{-13}, a median of 4.38×10^{-13}, and a standard deviation of 1.4×10^{-14} (3.2%). These results compare well with the nominal value of $(4.47 \pm 0.22) \times 10^{-13}$. The standard deviation of our measurements is again slightly smaller, at 3.2% compared with 4.9% reported by Corbett et al. (2024).

3.2 Quartz reference materials

Quartz reference materials used in this study include CRONUS-A and CoQtz-N (Jull et al., 2015; Binnie et al., 2019). Compared with liquid reference materials, these solid materials encompass a more complete analytical workflow, beginning with

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the weighing and carrier addition, and dissolution of quartz for each measurement. Consequently, the spread of the measured values incorporates variability introduced by additional processing steps and / or quartz impurities, which can be significant in some cases, as previously demonstrated by Corbett et al. (2022). To provide additional redundancy in our analysis, a bulk dissolution of CoQtz-N was also prepared, effectively converting the quartz reference material into a liquid reference material and allowing direct comparison between individual quartz dissolutions and the bulk dissolution.

3.2.1 CRONUS-A

CRONUS-A is a quartz reference material derived from exposed sandstone collected from an Antarctic outcrop at 77.8830°S, 160.9431°E, and 1612 m elevation (Jull et al., 2015). More than 20 kg of material was prepared at the University of Vermont by crushing and grinding the sandstone, followed by sieving to retain the 250–800 µm fraction. The quartz was purified by two overnight etches in 6N HCl under heated sonication, magnetic separation to remove susceptible minerals, and three successive etches in a heated (70°C) 1% HNO_3–HF mixture, with the acid replaced every 24 h. A final 72 h etch in 0.5% HNO_3–HF was followed by repeated rinsing with deionised water. This procedure removed non-quartz mineral phases and meteoric ^{10}Be from grain surfaces. As part of an inter-laboratory comparison exercise conducted within the broader CRONUS-Earth project (Phillips et al., 2016), the average and standard deviation of $n = 29$ CRONUS-A ^{10}Be concentration measurements, based on samples prepared in 13 laboratories and measured at 8 AMS facilities, were $(3.42 \pm 0.10) \times 10^7$ atoms g^{-1} of quartz (Jull et al., 2015). Similarly, $n = 13$ CRONUS-A ^{26}Al analyses of samples prepared in 7 laboratories yielded an average and standard deviation of $(1.43 \pm 0.07) \times 10^8$ atoms g^{-1} of quartz (Jull et al., 2015).

Between 2021 and 2025, $n = 23$ ^{10}Be and $n = 6$ ^{26}Al measurements of CRONUS-A quartz were performed on aliquots prepared at Imperial College London (ICL), with one CRONUS-A aliquot processed alongside each batch of samples as part of routine quality assurance practice in the CosmIC laboratory. The measured ^{10}Be concentrations have an average of 3.49×10^7, a median of 3.51×10^7, and a standard deviation of 8.5×10^5 (2.4%). These values are consistent with those reported by Jull et al. (2015) (Figures 5A&B). The standard deviation of our measurements (2.4%) is slightly smaller than the 2.9% reported by Jull et al. (2015), likely because our dataset primarily reflects intra-laboratory variability, whereas the earlier inter-comparison also incorporates variability between laboratories. For ^{26}Al, the measured concentrations have an average of 1.43×10^8, a median of 1.44×10^8, and a standard deviation of 4.1×10^6 (2.8%). As with ^{10}Be, the ^{26}Al concentrations are consistent with those reported by Jull et al. (2015), while the standard deviation of our measurements is again slightly smaller (Figures 5C&D).

The CRONUS-A results are also consistent with the ^{10}Be and ^{26}Al concentrations inferred from our UVM-A measurements (Figures 5B&D). For ^{10}Be, the inferred concentrations have an average of 3.47×10^7, a median of 3.49×10^7, and a standard deviation of 8.3×10^5. For ^{26}Al, the corresponding values are 1.42×10^8, 1.41×10^8, and 4.6×10^6, respectively. Nuclide concentrations for UVM-A were calculated using the quartz and carrier masses reported by Corbett et al. (2019). Blank corrections were applied using background ratios of 4.14×10^{-15} for ^{10}Be/^{9}Be and 2.1×10^{-16} for ^{26}Al/^{27}Al. These background values correspond to procedural blanks prepared during production of the UVM-A reference material and reported by Corbett et al. (2019). This approach does not account for any additional contamination that could contribute to the ^{10}Be/^{9}Be ratio of UVM-A aliquots during sample processing at UOW or during AMS measurement. However, procedural blanks processed at

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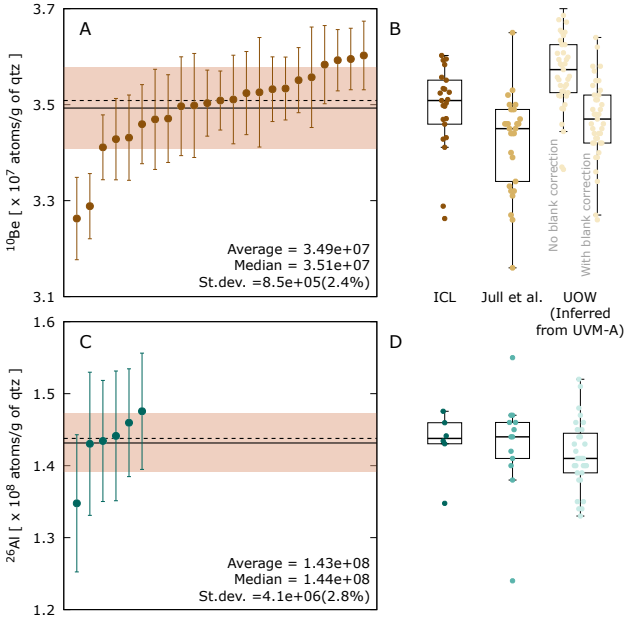

Figure 5. Measurements of CRONUS-A reference material for ^{10}Be (A & B) and ^{26}Al (C & D) concentrations from 2021 to 2025, normalised to KN-5-3 and KN-4-2 respectively. The average (solid line), median (dashed line), and standard deviation (shaded area; $\pm 1\sigma$) are shown. Box plots compare our measurements with those reported in Jull et al. (2015), and values inferred from the UOW / ANSTO UVM-A data. For completeness, we show both blank-corrected and uncorrected ^{10}Be values. For ^{26}Al, the effect of the blank correction is negligible.

UOW indicate that the ^{10}Be background is derived from the carrier itself, with no detectable ^{10}Be introduced during laboratory chemistry or AMS measurement (Codilean et al., 2023; Wilcken et al., 2022). The blank corrections applied above are therefore appropriate for the comparisons presented here.

3.2.2 CoQtz-N

CoQtz-N is a quartz reference material derived from a single boulder of vein quartz collected from a lag deposit mantling a low-relief surface near Hakos, Namibia (Binnie et al., 2019). The material was initially broken up at the University of Cologne, where fragments containing visible impurities, including black tourmaline, were discarded. The remaining quartz was crushed and dry sieved to retain the 250–710 µm fraction. This fraction was then sent to the University of Edinburgh, where it underwent three successive etches in an ultrasonic bath using a 2% HNO_3–HF mixture. Approximately 25 kg of purified quartz was subsequently returned to the University of Cologne, homogenised, and divided using an eight-way rotating cone splitter. The preliminary consensus concentrations reported by Binnie et al. (2019) for CoQtz-N are $(2.53 \pm 0.09) \times 10^6$ atoms g^{-1} of quartz for ^{10}Be and $(1.56 \pm 0.16) \times 10^7$ atoms g^{-1} of quartz for ^{26}Al, respectively. These values were derived using a statistical approach that explicitly accounts for common sources of variation among measurements associated with

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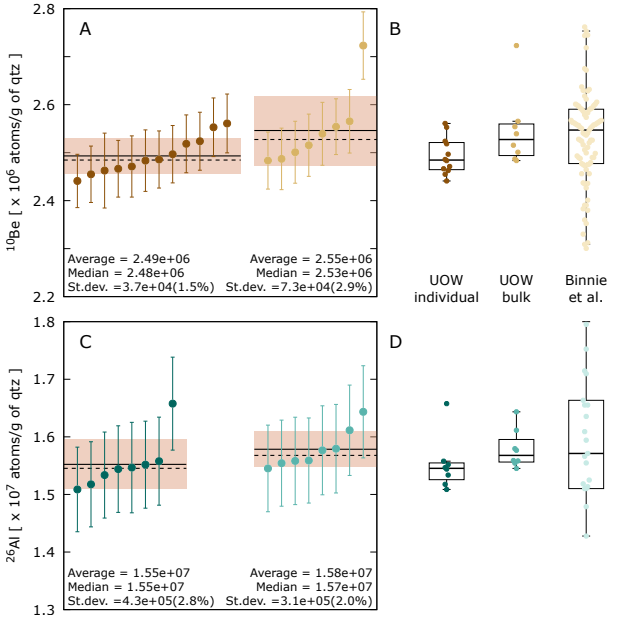

Figure 6. Results for individual and bulk dissolution of CoQtz-N quartz reference material for ^{10}Be (A & B) and ^{26}Al (C & D) normalised to KN-5-3 and KN-4-2, respectively. The average (solid line), median (dashed line), and standard deviation (shaded area; $\pm 1\sigma$) are shown. Both individual and bulk dissolution results are consistent with earlier results by Binnie et al. (2019) (B & D).

sample-preparation laboratories and AMS facilities, rather than treating all measurements as independent, and are reported with 95% confidence intervals.

We performed $n = 12$ individual CoQtz-N dissolutions for ^{10}Be and $n = 8$ for ^{26}Al at UOW. To complement these individual dissolutions and enable comparison with liquid reference materials, we also prepared bulk dissolutions using sufficient carrier to produce 8 aliquots for ^{10}Be and ^{26}Al analyses, respectively. Results from both the individual and bulk dissolutions are shown in Figure 6, together with the earlier measurements reported by Binnie et al. (2019).

The ^{10}Be measurements from individual CoQtz-N dissolutions have an average of 2.49×10^6, a median of 2.48×10^6, and a standard deviation of 3.7×10^4 (1.5%) atoms g^{-1} of quartz. These values are in good agreement with the measurements from the bulk dissolution, which have an average of 2.55×10^6, a median of 2.53×10^6, and a standard deviation of 7.3×10^4 (2.9%) atoms g^{-1} of quartz. The larger standard deviation of the bulk-dissolution measurements (2.9% compared with 1.5%) appears to be driven by a single measurement and is therefore unlikely to reflect systematically greater variability. This is also contrary to the expectation that a bulk dissolution should reduce variability between aliquots by eliminating differences associated with individual quartz dissolution (Figure 6A). Both the individual- and bulk-dissolution results are consistent with the nominal CoQtz-N values reported by Binnie et al. (2019) (Figure 6B). The population of $n = 8$ ^{26}Al measurements from individual CoQtz-N dissolutions has an average of 1.55×10^7, a median of 1.55×10^7, and a standard deviation of 4.3×10^5 (2.8%) atoms g^{-1} of quartz. These values agree within uncertainty with the measurements from the bulk dissolution, which have an average

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of 1.58×10^7, a median of 1.57×10^7, and a standard deviation of 3.1×10^5 at/g (2.0%) atoms g^{-1} of quartz. As with the ^{10}Be measurements, the ^{26}Al results are also consistent with the nominal CoQtz-N value (Figure 6C&D).

3.3 Duplicate samples

The limitation of liquid and quartz reference materials is that each captures only part of the complete analytical workflow used to determine ^{10}Be and ^{26}Al concentrations. In addition, reference materials are often selected from relatively high-concentration samples, which facilitates measurement and minimises complications associated with blank correction. Consequently, their performance may not be representative of the analytical uncertainties encountered for typical research samples. To quantify analytical reproducibility across a broad range of isotopic concentrations, from 10^3 to 10^6 ^{10}Be atoms g^{-1}, we compiled duplicate measurements from research samples processed independently on two occasions. The dataset consists predominantly of ^{10}Be measurements, with only a limited number of ^{26}Al duplicates. Because quartz extraction, purification, and etching are laborious, clean quartz was generally prepared as a single batch and then divided for duplicate processing, rather than being independently extracted and purified twice from the original sample material. Fully independent quartz extraction and purification were performed only where insufficient clean quartz was available for the second aliquot.

Duplicate samples therefore provide a pragmatic estimate of reproducibility across much of the analytical workflow and over a concentration range representative of research samples. However, they constrain reproducibility rather than accuracy, because agreement between duplicates does not demonstrate proximity to the true value. Furthermore, where duplicate measurements show greater dispersion than expected from statistical uncertainties alone, identifying the underlying cause can be difficult because the dataset encompasses a wide range of sample types, concentrations, and other analytical variables.

In Figure 7, the collection of $n = 61$ samples analysed in duplicate for ^{10}Be and $n = 5$ for ^{26}Al is presented as a function of isotopic concentration. Agreement between the two independent analyses of each sample is expressed as a figure of merit, calculated as the difference between the two results normalised by their combined analytical uncertainty. The ^{26}Al dataset is too small to support robust conclusions but is included for completeness. Of the ^{10}Be sample pairs, $n = 26$ were processed at UOW and $n = 35$ at ICL (Figure 7). All analyses were background corrected, and the mean background corrections are shown in Figure 7A and D for ^{10}Be and ^{26}Al, respectively. The ^{10}Be concentrations span approximately three orders of magnitude, from 10^3 to 10^6 atoms g^{-1} of quartz (Figure 7C). Analytical uncertainties range from approximately 2–3% for samples with concentrations above 10^5 atoms g^{-1} of quartz to 60% for a sample with a ^{10}Be concentration of approximately 10^3 atoms g^{-1} of quartz (Figure 7B).

The figure-of-merit comparison for the duplicate ^{10}Be analyses (Figure 7C) shows no strong dependence on isotopic concentration across the approximately three-order-of-magnitude concentration range. The results are broadly distributed about zero, with no pronounced systematic bias, although both the mean and median are slightly negative at -0.219 and -0.180, respectively. The standard deviation of the duplicate ^{10}Be results is 1.2, slightly higher than the value of 1 expected if the observed dispersion were fully accounted for by the reported analytical uncertainties. However, given that the dataset spans three orders of magnitude in ^{10}Be concentration and includes samples requiring background corrections of up to 60%, this modest excess dispersion does not, by itself, indicate a significant analytical problem. For the ^{26}Al duplicates, the mean, median, and

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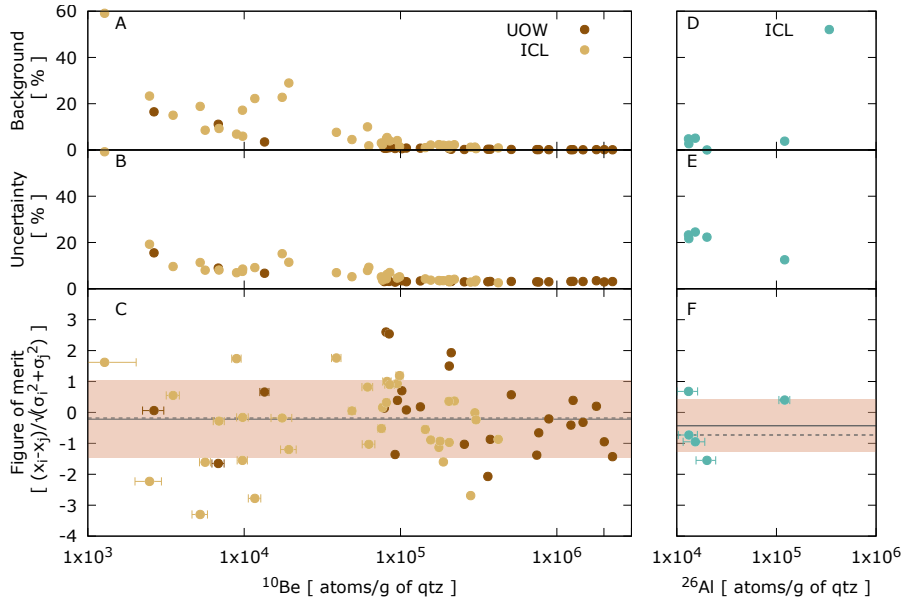

Figure 7. Results of duplicate analyses for ^{10}Be (A, B, C) and ^{26}Al (D, E, F), normalised to KN-5-3 and KN-4-2, respectively. The mean (solid line), median (dashed line), and standard deviation (shaded area; $\pm 1\sigma$) of the figure of merit are -0.219, -0.180, and 1.2 for ^{10}Be, and -0.430, -0.730, and 0.84 for ^{26}Al, respectively. Background corrections are shown in A & D, analytical uncertainties in B & E, and the figure of merit as a function of isotope concentration in C & F. Horizontal error bars in C & F represent the combined analytical uncertainties of the duplicate measurements.

standard deviation are -0.430, -0.730, and 0.84, respectively; however, because of the small sample size ($n = 5$), these results are not discussed further.

4 Discussions

We have presented a comprehensive comparison of quality-assurance materials for ^{10}Be and ^{26}Al that collectively constrain uncertainty across progressively larger portions of the analytical workflow. Liquid reference materials primarily test AMS measurement and, in the case of UVM-A, chemical separation also; quartz reference materials additionally incorporate variability associated with quartz dissolution and sample preparation; and duplicate research samples extend this assessment to materials spanning a much broader range of isotopic concentrations and analytical conditions. The combined results for ^{10}Be and ^{26}Al are summarised in Figure 8, allowing these different levels of analytical complexity to be compared directly. For established reference materials, measured values are normalised to their nominal (or consensus) values, whereas duplicate samples are divided into three populations according to isotopic concentration and normalised to the mean of the two independent measurements. This comparison provides the basis for assessing whether analytical variability increases as progressively more stages of the complete workflow are incorporated.

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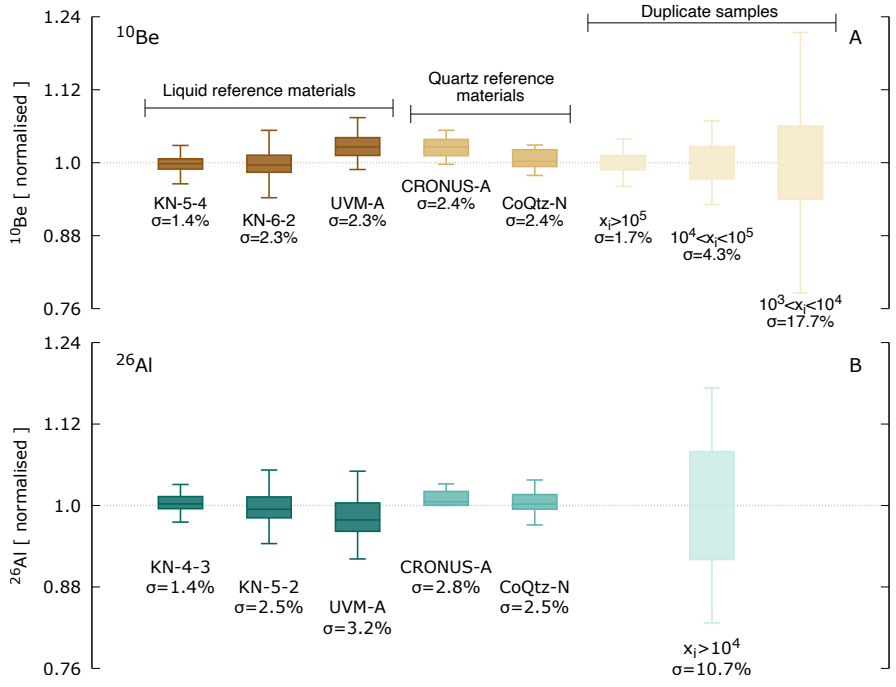

Figure 8. Compilation of the ^{10}Be and ^{26}Al results for all quality-assurance materials. Duplicate ^{10}Be samples are divided into three groups according to isotope concentration: $x_i > 10^5$ atoms g^{-1}, $10^5 > x_i > 10^4$ atoms g^{-1}, and $x_i < 10^4$ atoms g^{-1}. For established reference materials (KN-series, UVM-A, CRONUS-A, and CoQtz-N), results are normalised to the nominal or consensus value, whereas duplicate samples are normalised to the mean of the two independent measurements. For CoQtz-N, results from individual and bulk dissolutions are combined into a single population. The relative standard deviation is shown for each population.

For ^{10}Be, the standard deviations of the liquid reference materials are 1.4%, 2.3%, and 2.3% for KN-5-4, KN-6-2, and UVM-A, respectively, compared with average measurement uncertainties of 0.8%, 1.8%, and 2.4% (Figure 8A). The smaller dispersion of KN-5-4 is consistent with its higher measurement precision, whereas the standard deviations of KN-6-2 and UVM-A are comparable to their typical analytical uncertainties. The quartz reference materials show similar behaviour: CRONUS-A and the combined CoQtz-N population both have standard deviations of 2.4%, compared with average measurement uncertainties of 2.4% and 2.5%, respectively. The close agreement between the dispersion of UVM-A (2.3%) and CRONUS-A (2.4%) is particularly notable given that UVM-A was produced from dissolved CRONUS-A quartz, and indicates that the additional variability associated with processing quartz is not resolvable above the analytical uncertainty. More generally, the close correspondence between observed dispersion and measurement uncertainty for both quartz reference materials indicates that, for these materials, the additional processing associated with quartz dissolution and chemical extraction introduces no resolvable additional variability. This result is consistent across preparation laboratories, with CRONUS-A processed at ICL and CoQtz-N at UOW showing comparable dispersion. Importantly, this behaviour is not necessarily expected for all quartz reference materials. Corbett et al. (2022) reported substantially greater variability in repeated analyses of CRONUS-N, with a

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relative standard deviation of 6.3% for ^{10}Be and dispersion approximately twice that expected from the mean AMS analytical uncertainty. They attributed this excess variability to heterogeneity in CRONUS-N arising from small amounts of non-quartz mineral phases, which disproportionately affected the measured ^{10}Be concentrations. The contrast with our CRONUS-A and CoQtz-N results therefore highlights the importance of quartz purity and reference-material homogeneity: where these are adequately controlled, our data provide no evidence that extraction of ^{10}Be from relatively high-concentration quartz samples introduces significant additional uncertainty beyond the analytical measurement itself.

We divided the ^{10}Be duplicate samples into three groups according to isotope concentration: $> 10^5$ atoms g^{-1}, 10^4–10^5 atoms g^{-1}, and $< 10^4$ atoms g^{-1} of quartz. For samples with ^{10}Be concentrations above 10^5 atoms g^{-1}, the average analytical uncertainty is 2.3%, compared with an observed standard deviation of 1.7%, indicating no resolvable variability beyond that expected from the individual measurement uncertainties. For samples with ^{10}Be concentrations in the range of 10^4 to 10^5 atoms g^{-1}, the average analytical uncertainty increases to 4.4%, while the observed standard deviation is 4.3%. The close agreement between these values again indicates that the observed dispersion is adequately explained by the reported analytical uncertainties, which at these concentrations are increasingly dominated by counting statistics. For the lowest-concentration duplicate samples ($< 10^4$ atoms g^{-1}), the combination of poor counting statistics and larger background corrections results in an average analytical uncertainty of 12.2% and an observed standard deviation of 17.7%. If interpreted as independent variance components, this excess dispersion corresponds to an additional uncertainty of approximately 12.8% beyond the reported analytical uncertainty. However, this apparent excess is strongly influenced by a single sample with a ^{10}Be concentration of approximately 10^3 atoms g^{-1} and a background correction of approximately 60%. Excluding this sample reduces the standard deviation from 17.7% to 10.4%, bringing the observed dispersion into agreement with that expected from the analytical uncertainties alone. Thus, there is no clear evidence for an additional source of analytical variability at low concentrations, although the limited precision and sensitivity to large background corrections make such samples inherently more difficult to assess.

Comparison of the liquid reference materials (KN-6-2 and UVM-A), quartz reference materials (CRONUS-A and CoQtz-N), and the highest-concentration duplicate samples ($x_i > 10^5$ atoms g^{-1}) shows a consistent precision averaging around 2.3% (Figure 8A). Importantly, the observed dispersion does not increase as progressively more stages of the analytical workflow are incorporated, from liquid reference materials through quartz reference materials to duplicate research samples spanning a range of lithologies. This indicates that, where ^{10}Be concentrations are sufficiently high, the additional processing steps associated with quartz preparation and chemical extraction do not introduce a resolvable source of variability beyond the AMS measurement uncertainty. At concentrations between 10^4 and 10^5 atoms g^{-1}, precision decreases to approximately 4%, primarily because of poorer counting statistics. Below 10^4 atoms g^{-1}, analytical precision deteriorates substantially as both counting statistics and background corrections become increasingly important, although the duplicate data provide no compelling evidence for additional process-related uncertainty once the most extreme low-concentration sample is excluded.

For ^{26}Al, the overall pattern is similar to that observed for ^{10}Be. The high-ratio liquid reference material KN-4-3 has a standard deviation of 1.4%, comparable to its average measurement uncertainty of 1.3%, and indicating no resolvable variability beyond the AMS measurement uncertainty. For the lower-ratio liquid reference materials KN-5-2 and UVM-A, and the quartz reference materials CRONUS-A and CoQtz-N, the observed standard deviations are generally between approximately

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2.5% and 3.2%, and are comparable to their respective analytical uncertainties. UVM-A has a standard deviation of 3.2%, CRONUS-A 2.8%, whereas the combined CoQtz-N population has a standard deviation of 2.5%. As for ^{10}Be, the similar dispersion observed for liquid and quartz reference materials provides no evidence that quartz dissolution and the subsequent column chemistry required to isolate Al introduce a resolvable additional source of uncertainty. The comparable distributions obtained for quartz reference materials processed at ICL and UOW further suggest that intra-laboratory variability at both laboratories is similarly small relative to the measurement uncertainty. The ^{26}Al duplicate dataset is limited to only $n = 5$ samples, most with concentrations of approximately 10^4 atoms g^{-1} of quartz, and is therefore too small to subdivide meaningfully by isotope concentration. The low concentrations also limit counting statistics, resulting in relatively large analytical uncertainties. Nevertheless, the observed dispersion of the duplicate measurements is comparable to their typical analytical uncertainty of approximately 14.7%, suggesting no clear evidence for additional process-related variability, although the small sample size prevents further meaningful interpretation of the ^{26}Al duplicate results.

5 Conclusions

We assessed the reproducibility and precision of ^{10}Be and ^{26}Al analyses using liquid and quartz reference materials together with duplicate research samples spanning a broad range of isotope concentrations. The principal conclusions are:

- High-ratio liquid reference materials demonstrate that AMS measurements of ^{10}Be and ^{26}Al can achieve precision approaching 1% when counting statistics are sufficient. Measurements of KN-5-4 (^{10}Be) and KN-4-3 (^{26}Al) are consistent with their nominal values, with standard deviations of 1.4% for both isotopes.

- For liquid and quartz reference materials with isotope ratios more representative of routine research samples (approximately 10^{-13} to 10^{-12}), analytical precision is typically 2–3%. Importantly, quartz reference materials show no resolvable increase in variability relative to liquid reference materials, indicating that quartz dissolution and chemical separation do not introduce significant additional uncertainty when sufficiently pure, homogeneous material is analysed. CRONUS-A and CoQtz-N results are consistent with their nominal values and with the dispersion expected from their analytical uncertainties.

- Duplicate ^{10}Be samples demonstrate that this level of precision extends to routine research samples when isotope concentrations exceed 10^5 atoms g^{-1} of quartz. At concentrations of 10^4 to 10^5 atoms g^{-1}, precision decreases to approximately 4%, primarily because of counting statistics. Below 10^4 atoms g^{-1}, low counting statistics and increasingly important background corrections can increase analytical uncertainties to approximately 10–15%. Nevertheless, the observed dispersion of the duplicate measurements is generally consistent with the reported analytical uncertainties. The ^{26}Al duplicate dataset is too small ($n = 5$) to support broader conclusions; however, the observed precision of approximately 10% is consistent with the analytical uncertainties of the individual measurements.

- Samples were prepared in three laboratories (ANSTO, UOW, and ICL) and measured using the same AMS facility. Although inter-laboratory reproducibility cannot be quantified directly because no single reference material was inde-

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pendently processed in all three laboratories, the results from each laboratory are consistent with the nominal reference values and show no evidence for laboratory-specific variability exceeding routine analytical uncertainty.

- Taken together, these results indicate that cosmogenic ^{10}Be and ^{26}Al concentrations can typically be measured with approximately 2–3% reproducibility and precision when counting statistics are sufficient. At lower isotope concentrations, achievable precision is increasingly controlled by counting statistics and background correction rather than by additional variability introduced during sample preparation. Precision approaching 1% is achievable for high-concentration liquid reference materials, but has not yet been demonstrated for quartz samples.

Data availability. All data supporting the findings of this study are included in the Supplementary Material accompanying this article.

Author contributions. Conceptualization: KMW; AMS analyses: KMW, RHF; Sample preparation at ANSTO: KS, SK; Sample preparation at UOW: ATC, RHF; Sample preparation at ICL: AHR, DHR; Writing original draft: KMW; Writing, review, and editing: all authors.

Competing interests. The contact author has declared that neither they nor their co-authors have any competing interests.

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