Technical Brief

A calcite reference material for LA-ICP-MS U-Pb geochronology

Nick M W Roberts

Troy Rasbury

Randall R Parrish, School of Earth and Environmental Sciences, University of Portsmouth & British Geological Survey, Keyworth, Notts, NG12 5GG, United Kingdom, email: randall.parrish@port.ac.uk

Christopher J Smith

Matthew S A Horstwood

Daniel J Condon

Abstract

U-Pb dating of calcite is an emerging but rapidly growing field of application in geochronology with great potential to inform problems in climate evolution, determining the age of diagenetic cements, structural geology, and the age of geological formations difficult to date otherwise. In this paper we present reliable U-Pb isotope measurements on a calcite that has and will continue to be used as a reference material for in situ LA-ICP-MS dating, and which is suitable to be distributed and used by the geochronological community. We outline procedures for dating carbonates as a general methodology, and present measurements using LA-ICP-MS of this material to demonstrate its suitability for use as a U-Pb dating reference material, in spite of it not quite being the 'ideal' reference material. This material is 253.6±5.8 Ma old, ranges from ~85-98% radiogenic Pb content, and given the instrumental resolution obtainable, is a simple mixture of common lead and radiogenic Pb of a single age without complication arising from, for example subsequent disturbance or admixture of younger or older calcite on the 20-100um scale. As such it is a highly suitable reference material that can facilitate dating of calcite ranging in age from Precambrian to late Neogene age.

Introduction

Laser Ablation Inductively Coupled Plasma Mass Spectrometry (LA-ICP-MS) has greatly facilitated U-Pb accessory mineral dating because of its high spatial resolution and rapid data acquisition, as compared to Isotope Dilution Isotope Ratio Mass Spectrometry techniques (i.e. Thermal Ionisation Mass Spectrometry, TIMS and/or multi-collector ICP-MS). Although it has lower measurement precision (~1% compared to ~0.1% on U-Pb ratio), this suits a wide variety of applications using a very wide range of minerals and materials. The method has also been applied successfully to minerals with lower U (sub-ppm) concentrations than are generally feasible with in situ SIMS methods (< 1ppm calcite, Coogan et al. 2016). Calcite and other carbonate materials (aragonite, dolomite) have been dated by U-Pb via ID-IRMS (see Rasbury and Cole, 2009), but so far have largely been focussed on dating speleothems (e.g. Woodhead et al., 2006; 2012; Pickering et al., 2010). The method is particularly useful for materials with elemental and isotopic compositional heterogeneity on the 1-100 µm scale, (like calcite), and which typically require initial evaluation in terms of dating suitability of samples to find the best material in order to optimize both the dating and the effort and expense involved. The basic method, like that for zircon and other accessory minerals, involves comparing isotope signal intensities of an unknown with that of a reference material whose age or ^{206}Pb/^{238}U ratio is known, in order to determine the age of the sample material of unknown age, a procedure that has been termed U-Pb 'normalisation'. It is clear that with either SIMS or LA-ICP-MS methods, the reference material must be the same mineral (matrix-matched) as that of the unknown to ensure reliability of any date and its precision. However, the lack of matrix-matched carbonate reference materials limits to some extent progress in advancing LA-ICP-MS methods applied to carbonate minerals. One reference material, termed WC-1, is currently being used by several laboratories and in this communication, we present reliable ID-IRMS measurements on this carbonate reference material so that, upon further distribution and use by additional laboratories worldwide, carbonate dating can expand in its application by research laboratories worldwide.

Sample Context – WC-1

WC-1 (Fig. 1) is taken from a ~1 kg hand sample of marine calcite cement that filled a fault-related discordant Neptunian dyke in the Permian (Capitanian) Reef Complex, exposed in the Guadalupe Mountains on the western side of the Delaware Basin of West Texas. Its locality is at BM3647 (1112m elevation) with coordinates 32° 10.62'N and 104° 22.90'W about 0.5 km west of Whites City, New Mexico, USA. According to the International

Chronostratigraphic Chart of 2015 (Cohen et al. 2013, updated), the Capitanian stage ranges from about 265-260 Ma and therefore technically speaking the dyke must be younger than the Capitanian stratigraphy that it ‘intrudes’ which is the Tansil-equivalent reef facies. Overlying the Tansil Formation is the Castile Formation; carbonate of this age was dated by Becker et al. (2002) at 251.5 ± 2.8 Ma using ID-TIMS U-Pb geochronology. This age was re-evaluated by Rasbury and Cole (2007) to be more like 257 ± 3 Ma, using the ^{208}Pb/^{204}Pb as a monitor of alteration and selecting only those aliquots that showed lowest alteration for the isochron age. Underlying the Tansil is the Radar Ash dated at 262.6 ± 0.5 Ma (Nickelson, 2011) by U-Pb on zircons.

The marine cements comprising the dyke were aragonite crystals that have been neomorphosed to calcite. In hand specimen, the botryoidal forms and fibres that make up the botryoids are easily visible. In thin section, the blocky calcite engulfs inclusions that show the original fibres that make up the botryoids. A ca. 6 x 3 cm slab of WC-1 material (Fig. 2) has been sub-sampled and characterised for its U-Pb isotopic composition by ID-IRMS and LA-ICP-MS. Additionally, a region of the slab has been analysed for trace element distribution using LA-ICP-MS. Measurement of four samples from the interior of the Neptunian dyke at the WC-1 locality have a mean ^{87}Sr/^{86}Sr value of 0.706895±0.000048 (Rasbury, unpublished data).

Analytical Methods

ID-IRMS

The ultrasonically-cleaned sample pieces were fully dissolved in ultrapure 4 N HNO3 before being spiked with a mixed ^{205}Pb–^{233}U–^{235}U tracer. Following sample-to-spike equilibration, organic matter in samples was destroyed by addition and evaporation of 0.5 ml of concentrated HNO_3 and 0.5 ml H_2O_2. The precipitate was then re-dissolved in 1 ml 1 N HBr for anion exchange chemistry. Eichrom® anion exchange resin (equivalent to BioRad AG 1 - × 8) was used to separate and purify Pb. Uranium was purified using Eichrom UTEVA resin in nitric acid. The chemistry blanks were < 10 pg for U and < 20 pg for Pb. The ^{207}Pb/^{206}Pb composition of the blank Pb was measured at 0.86±0.08 (2σ).

The separated Pb fractions were loaded individually, with silica gel and H_3PO_4, onto single outgassed rhenium filaments and analyzed using a Thermo Scientific Triton mass spectrometer fitted with an axial MasCom secondary electron multiplier (SEM) at the NERC Isotope Geosciences Laboratory (NIGL) British Geological Survey, Keyworth, UK. Data were obtained in either dynamic single-collector mode (where ^{206}Pb signal was <1M cps), or in a multi-static routine where all isotopes except ^{204}Pb were measured on a Faraday detector and the Faraday-SEM yield was determined in real time using ^{205}Pb which was measured on both detectors. The U separates were run as a oxide in static Faraday mode using the same mass spectrometer. Pb and U reference solutions SRM 981 and U500, were analysed to monitor mass spectrometer performance, ensuring that the SEM linearity, accuracy and reproducibility were better than ± 0.1%. Mass bias for U was corrected internally using the double U tracer.

Data reduction and uncertainty propagation were carried out using customized data reduction spreadsheets following standard parametric statistical methods (Schmitz and Schoene, 2007). The decay constants and U isotope composition used were those proposed by Jaffey et al. (1971) and Hiess et al. (2012), respectively.

LA-ICP-MS U-Pb

U-Pb analysed utilised a Nu Instruments single-collector Attom ICP-MS, coupled to a New Wave Research (ESI) NWR193UC laser ablation system equipped with a TV2 ablation cell. The method is adapted from the standard zircon method described previously in Spencer et al. (2014). Laser ablation parameters for carbonates are a 100 µm static spot, ablated for 30 s, at 10 Hz, with a fluence of 7-8 J/cm^{-2}. Normalisation used a standard-sample bracketing protocol with NIST614 for correction of Pb-Pb ratios and the WC-1 carbonate for correction of U-Pb ratios (as described in Li et al., 2014; Roberts & Walker, 2016). The methodology is feasible with any LA-ICP-MS system (including Q-ICP-MS) using a variety of laser instrumental conditions provided that signal background values and their variation are small and that instrumental sensitivity is sufficiently high (for example, >20,000 cps/ppm U). The use of NIST 614 or NIST612 in the characterisation of this material and indeed in the measurement of any unknowns using WC-1 as a reference material is essential since the Pb isotope composition of the WC-1 calcite is not uniform owing to the somewhat variable admixture of radiogenic Pb 254 Ma old and common Pb. Data are reduced using the Time Resolved Analysis (TRA) functionality in Nu Instruments' Attolab software, with normalisation and uncertainty propagation calculated offline using an in-house excel spreadsheet. Data reduction and propagation of uncertainties follow the recommendations for zircon geochronology outlined in Horstwood et al. (2016).

LA-ICP-MS trace elements

For trace element mapping, the analytical set-up is the same as for U-Pb. Mapping utilises a set of line rasters created with a 100 µm square, translated at 50 µm/s, and ablated at 10 Hz with a fluence of 7-8 J/cm^{-2}. Normalisation uses NIST614 (Jochum et al., 2011) analysed at the beginning and end of the sample map, using ^{44}Ca as an internal standard. Data are reduced and the maps created using the software Iolite v.2 (Paton et al., 2011). A portion of the mapped region was gridded using a 7000 x 100 µm sampling area, with the concentrations for each area being calculated using the standard Trace Element Data Reduction Scheme in Iolite.

Results

ID-IRMS U-Pb

Eight aliquots of WC-1 were taken for measurement by ID-IRMS. These aliquots were sampled across the visible heterogeneous texture and colour of the slab (see Fig. 2), with the aim that the resulting reference ID data will provide a reasonable representation of any heterogeneity that is exists in the material, and that this correlates with the visible colour and texture variation. The resulting data produce a regression in Tera-Wasserburg space (Fig. 3a) with a lower intercept age of 253.4 ± 6.9 Ma (2σ; MSWD = 61). The upper intercept (^{207}Pb/^{206}Pb = 0.77 ± 0.15) overlaps with the expected common-lead composition for this age (0.85) based on Stacey & Kramers (1975), but is not well-constrained due to a lack of analyses comprised of high common lead. Measurements of other hand specimen(s) of this same dyke at the University of Stony Brook had a larger dispersion in ^{207}Pb/^{206}Pb with a resultant upper intercept for initial common Pb of 0.85 ± 0.04. Anchoring the upper intercept to 0.85 ± 0.04 gives a regression with a lower intercept age of 253.6 ± 5.8 Ma (MSWD = 216); this lower intercept age is taken as the reference ‘age’ for WC-1 (see Table 1). The scatter in data cannot be attributed to measurement, for example blank correction, since the ^{207}Pb/^{206}Pb of the blank is very similar with a value of 0.086 ± 0.08 (or less if correlation is taken into account) and because the blank correction is no larger than ~5% of the total Pb in measured samples; this implies some limited heterogeneity within aliquots.

LA-ICP-MS U-Pb

LA-ICP-MS data from eleven analytical sessions, individually normalised to the expected age of 253.6 Ma, are pooled in Fig 2b and reveal an age and uncertainty of 253.6 ± 1.7 Ma (2σ, MSWD = 5.6, n = 214). The data are anchored to an upper intercept of 0.85 ± 0.02. Because these data are self-normalised using the reference age of 253.6 Ma determined by ID-IRMS, the undercertainty of this age is a measure of the dispersion about the linear mixing array between its age and the common Pb composition, as well as a visual indication of the variability in the mixture of radiogenic to common Pb within the material. The measure of over-dispersion of these data, calculated with the weighted mean function in Isoplot (Ludwig, 2003), is 2.7% (2σ). This over-dispersion is only slightly larger than the level of uncertainty in the ID-IRMS data (2.3%), indicating that both datasets are probably a fair representation of the natural heterogeneity of the material with respect to U-Pb. Figure 3 however indicates that the spatial resolution of the laser analyses have highlighted greater variation in the proportion of radiogenic Pb in the material than the ID-IRMS sampling which involves much larger volumes of material being analysed.

Trace elements

A region of the slab analysed for U-Pb was mapped for several trace elements (Fig. 1b, 3). In general it is low-Mg calcite with a mean Mg concentration of ~900ppm though some fractures contain up to 1.47% by weight Mg. Even so, all regions analysed from the slab have <4 mole% $MgCO_3$ and as such are characteristically low-Mg calcite. The region (18 x 5 mm) was chosen as it has visible changes in texture and appearance. The map shows that several trace elements show distinct variability, with darker areas being higher in Mg and Mn, and paler areas lower in Sr. A rare earth element (Tm) and the transition metals (V, Fe, Cu, Zn) are more uniform across the material. A white alteration vein cross-cutting the mapped region is high in Th and the transition metals. Importantly for U-Pb geochronology, the U content is fairly homogeneous across the mapped region, which is consistent with our experience of measuring spot analyses from this material. The concentration data from this mapped region are shown in Table 2. The average Pb, Th and U concentrations are 0.015, 0.015 and 3.70 ppm respectively.

Discussion

Suitability of WC-1 as a reference material

Ideally, a calcite reference material that was as homogeneous as a zircon standard (ie. 91500, GJ-1, etc.) is what is sought, but due to the occurrence of initial common Pb in calcite (in contrast to zircon), and the lack of high-U (hundreds of ppm) calcite with low (<< 1ppm) common Pb content, no such material has yet been identified, though it may be found with more active search.

A number of recent studies have demonstrated that U-Pb ages can be determined using LA-ICP-MS with the WC-1 matrix-matched carbonate material (Li et al., 2014; Harris et al., 2014; Coogan et al., 2016; Methner et al., 2016; Ring & Gerdes, 2016; Roberts & Walker, 2016; Burisch et al., 2017), thus, WC-1 provides a useful and reliable reference material for age determination of calcite of unknown age. WC-1 carbonate is from an abundant source that could be distributed widely, even though the ID-IRMS and LA-ICP-MS data show that there is a marked level of heterogeneity in the material, which thus far appears to be on the level of ca. 10% for ^{238}U-^{206}Pb, ~50% for ^{207}Pb/^{206}Pb, and about 2-3% excess scatter about the line joining the age with the common Pb composition, which is a direct measure of 'age dispersion' within the material. Given the present lack of carbonate reference materials, WC-1 will be a very useful primary reference material to generate LA-ICP-MS U-Pb dates of calcite over a very wide range of age (Precambrian to late Neogene).

Reference material requirements and usage

It is always desirable in LA-ICP-MS dating to have multiple matrix-matched reference materials; in some cases (i.e. variable textures of sample(s), dramatically different U or other trace element contents) this will be required to provide robust validation of ages. The most homogeneous and best materials should be the primary and less perfect but still reliable materials should be secondary reference materials. However, for the time being and until a more ideal calcite reference material is available (see below), we advocate use of WC-1 as the principle calcite reference material for U-Pb normalisation of unknown calcite samples because of its documented age and isotope characteristics, regardless of whether a zircon or glass is used as a primary normalisation monitor. For WC-1, the variability in proportion of common Pb and its discordance implies that the mean value of multiple ^{238}U/^{206}Pb measurements within an analytical session cannot be relied upon for normalisation; a modified approach is therefore required for normalisation that corrects for this common Pb and which relies upon the coherence of the array of WC-1 data about the correct age intersection with the T-W concordia.

Figure 5 shows hypothetical arrays of measured (m) data using a Tera-Wasserburg plot for both the WC-1 reference (R) material and a sample of unknown (U) age, prior to any U-Pb normalisation. The X-intercepts of these arrays can be easily calculated using Isoplot

(Ludwig 2003) as XRm and XUm as shown. The corrected (c) age of the WC-1 reference is shown and a line anchored with the NIST- or zircon-normalised initial common Pb that contains the correct age will have a X-intercept of XRc. To obtain the correct ^{238}U/^{206}Pb value of any unknown, the ^{238}U/^{206}Pb of each measurement on unknowns is simply multiplied by the value given by XRc/XRm. After applying this correction to all measurements of both WC-1 and to all measurements on unknowns and calculating best fit lines, the two measured arrays will shift to coincide with the measured age for the WC-1 standard and the correct age for the unknown sample array, respectively, as shown in Figure 5. Some laboratories adopt a NIST glass or zircon standard as the primary reference for ^{207}Pb/^{206}Pb and ^{238}U/^{206}Pb normalisation to return either the glass or zircon to the correct values, but this will still result in a residual, but smaller offset of the WC-1 calcite array from its correct value; either way the final correction procedure for WC-1 and unknown sample calcites is the same as described above.

In an analytical session, it is highly desirable to have a homogeneous calcite reference material; this allows far fewer analyses per session of the reference material than the case of a reference material with large dispersion and which requires more data to establish its intercept age with acceptable precision. WC-1 is not ideal because of its modest heterogeneity and lack of concordance, but it is reliable nevertheless. The characteristics that would make an even better calcite reference material include a very high proportion of radiogenic Pb (>98%) to ensure virtual homogeneity; U content that is not excessive (to match signal counts between reference material and unknowns); and low common Pb (<<1 ppm). This is likely to be present in calcite with an age sufficiently old to have allowed the radiogenic Pb to thoroughly dominate the Pb isotope composition and render the common Pb almost negligible. For example, had the WC-1 sample been allowed to decay for a further 2 Ga instead of being 254 Ma old, nearly all analyses would return a radiogenic Pb content of >97% and this would as a result be essentially ‘homogeneous’ given the general LA-ICP-MS 2σ measurement uncertainty obtainable. This kind of material should be sought for a future and more ideal reference material, but it has yet to be identified.

Acknowledgements

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Figure Captions

Figure 1. Field photograph of the Neptunian Dyke from Walnut Canyon, with person for scale. These dykes occupy fractures that are roughly parallel with a set of faults that offset the Capitan reef complex of age at least as young as the Tansil formation. The boundaries of the discordant dyke are shown by dotted lines with the crudely bedded Capitan reef facies. Nearby, the reef facies and dykes at this locality are overlain by the Castile Formation which contains an ash 251.5±2.8 Ma old (Bekker et al. 2002).

Figure 2. Top: Photograph of the WC-1 hand specimen; bottom: photograph of the sub-sampled slab analysed for U-Pb and trace elements.

Figure 3. a) ID-IRMS U-Pb data for WC-1 (n=8) anchored at 0.85±0.04 for initial common Pb; b) pooled LA-ICP-MS U-Pb data for WC-1 from eleven analytical sessions (n=214) from the NIGL laboratory. Uncertainties and ellipses are 2σ.

Figure 4. Trace element maps from the sub-sampled slab of WC-1. The concentrations in each scale bar are ppm.

Figure 5. Procedure for normalisation of sample 238U/206Pb using the measured array of WC-1 calcite as a reference.

Table 1. Summary of ID-IRMS-derived reference values; lower intercepts refer to intercepts with the Concordia curve, not the X-axis; all uncertainties quoted at 2σ.

Table 2. Concentrations from a portion of the mapped region of WC-1 based on a 7000 x 100 µm sampling size (n=100).

Table 3. ID-TIMS data for WC-1…….

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Figure 1:

Figure 2.

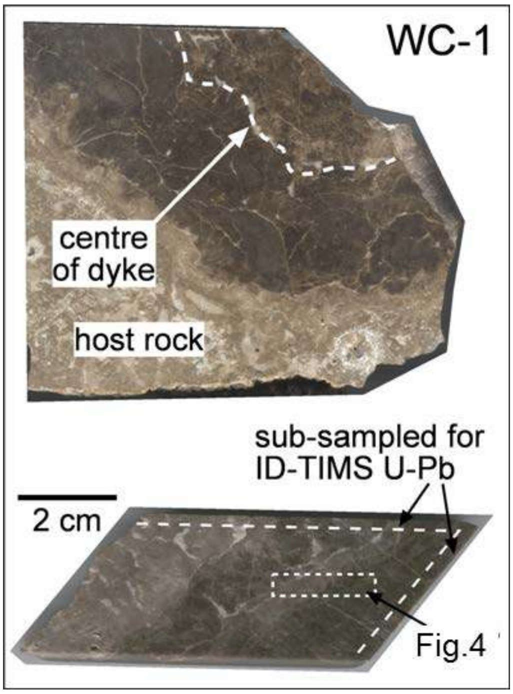


Figure 3:

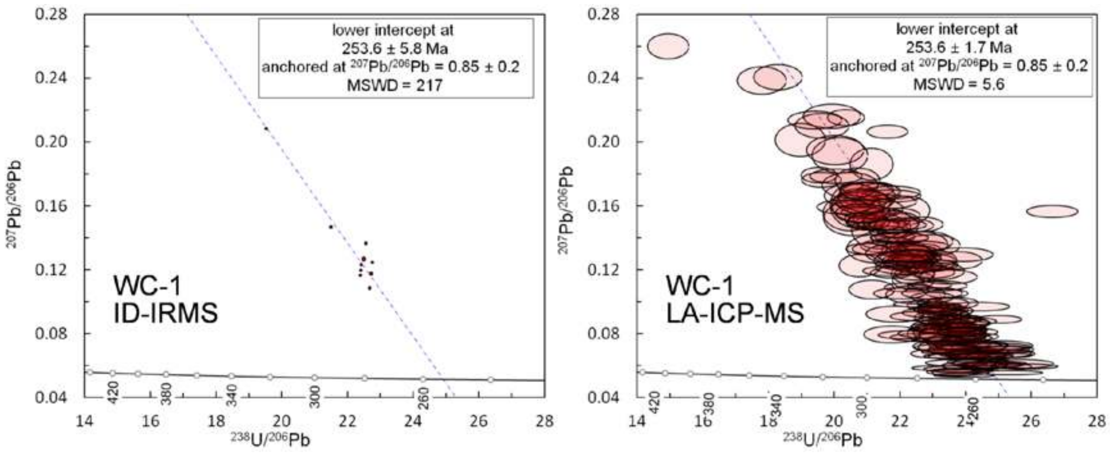
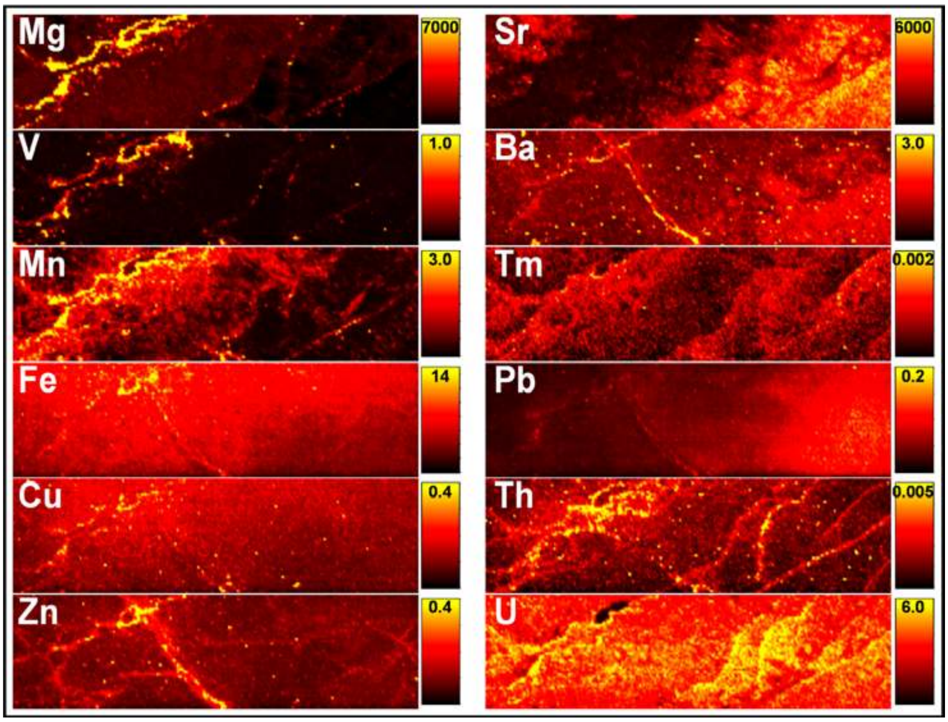


Figure 4:


Mg
7000
V
1.0
Mn
3.0
Fe
14
Cu
0.4
Zn
0.4
Sr
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Ba
3.0
Tm
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Pb
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Th
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U
6.0

Figure 5.

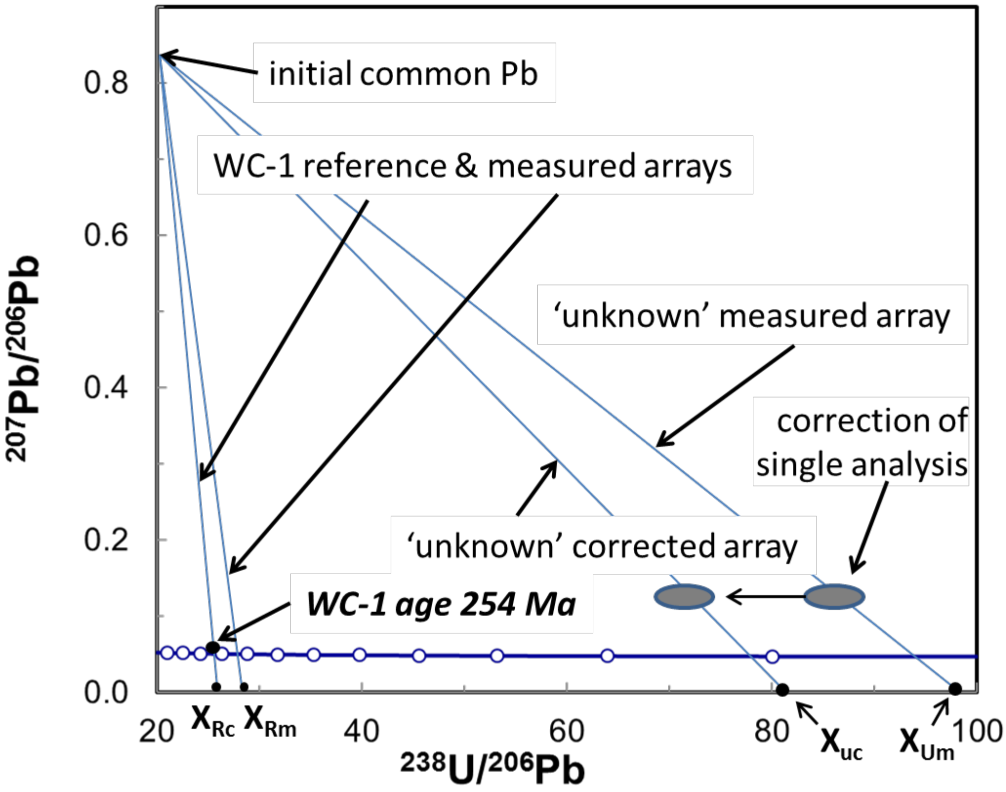
initial common Pb
WC-1 reference & measured arrays
'unknown' measured array
correction of single analysis
'unknown' corrected array
WC-1 age 254 Ma
0.8
0.6
0.4
0.2
0.0
20
40
60
80
100
X_{Rc}
X_{Rm}
X_{uc}
X_{Um}
$^{207}Pb/^{206}Pb$
$^{238}U/^{206}Pb$

Table 1:

Material	Upper intercept ($^{207}Pb/^{206}Pb$)	Lower intercept ($^{238}U/^{206}Pb$)	Lower intercept (Ma)
WC-1	0.85 ± 0.2	24.92 ± 0.58	253.6 ± 1.7

Table 2:

Element	Mean	StDev	Min	Max
Mg (24)	907	1620	158	14700
V (51)	0.067	0.074	0.021	0.610
Mn (55)	0.693	0.461	0.115	2.550
Fe (57)	5.22	1.13	3.10	8.70
Cu (63)	0.075	0.026	0.043	0.183
Zn (66)	0.124	0.019	0.084	0.174
Sr (88)	1606	1144	445	5420
Cs (133)	0.002	0.001	0.000	0.003
Ba (138)	0.610	0.228	0.360	1.800
Tm (169)	0.004	0.002	0.000	0.011
Pb (208)	0.015	0.009	0.005	0.041
Th (232)	0.015	0.008	0.005	0.042
U (238)	3.70	0.60	2.14	5.60