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Peter Webb, Michael Wiedenbeck, Johannes Glodny

# G-Chron 2019 – Round 1

## An International Proficiency Test for U-Pb Geochronology Laboratories -

## Report on the 2019 Round of G-Chron based on Palaeozoic Zircon Rak-17 (Distribution: September 2019)

Scientific Technical Report STR - Data 21/06

www.gfz-potsdam.de

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**Recommended citation**

Webb, P., Wiedenbeck, M., Glodny, J. (2020): G-Chron 2019 – Round 1: An International Proficiency Test for U-Pb Geochronology Laboratories; Report on the 2019 Round of G-Chron based on Palaeozoic Zircon Rak-17 (Distribution: September 2019), (Scientific Technical Report STR - Data; 21/06), Potsdam: GFZ German Research Centre for Geosciences.
https://doi.org/10.48440/GFZ.b103-21061

**The data described in this report are published as**

Webb, Peter; Wiedenbeck, Michael; Glodny, Johannes (2021): Survey questions and responses to the G-Chron 2019 proficiency test. GFZ Data Services.
https://doi.org/10.5880/GFZ.3.1.2021.005

**Imprint**

Helmholtz Centre Potsdam
GFZ German Research Centre
for Geosciences

Telegrafenberg
D-14473 Potsdam

Published in Potsdam, Germany
April 2020

DOI: https://doi.org/10.48440/GFZ.b103-21061
URN: urn:nbn:de:kobv:b103-21061

This work is published in the GFZ series
Scientific Technical Report (STR)
and electronically available at GFZ website
www.gfz-potsdam.de

# G-Chron 2019 – Round 1

## — An International Proficiency Test for U-Pb Geochronology Laboratories — Report on the 2019 Round of G-Chron based on Palaeozoic Zircon Rak-17 (Distribution: September 2019)

Peter Webb[1], Michael Wiedenbeck[2], Johannes Glodny[2]

[1]Formerly of the Department of Environment, Earth and Ecosystems, The Open University, Walton Hall, Milton Keynes, MK7 6AA, UK.

[2]Deutsches GeoForschungsZentrum GFZ, Telegrafenberg, 14473 Potsdam, Germany.

**Abstract**

*Proficiency testing (PT) is one of the few ways for an analytical laboratory to assess data quality under routine operating conditions. Here we report the results of Round 1 of the G-Chron PT programme, which is sponsored by the International Association of Geoanalysts. G-Chron is the first PT scheme devoted to the U-Th-Pb dating of mineral phases, primarily zircon, in geological materials. In this first round of G-Chron a total of 72 geochronology laboratories received the test material "Rak-17", which previously had been characterized by seven well-established isotope dilution TIMS laboratories. A total of 63 of the PT participating laboratories reported data by the 15 December 2019 deadline. Here we both report and assess the measurement results submitted to this round. Our analysis provides a means for participating laboratories to assess their individual performance in relation to the isotope ages assigned, the experimental fitness-for-purpose criteria proposed by the scheme's organisers and the results of similar laboratories participating in this round.*

**Introduction**

In the Earth Sciences the understanding of timing and rates of geological events and processes is of crucial importance. Such information is commonly derived using radioactive decay systems. Due to zircon's resistance to chemical alteration and its propensity to incorporate significant amounts of uranium and thorium into its crystal structure (while largely excluding the incorporation of lead at the time of crystallization), zircon is perhaps the most widely analysed mineral phase for defining the timing of igneous or metamorphic events. However, until now there has been scant information about the overall quality of this ever-expanding dataset. It is anticipated that this PT will provide information about the measurement processes applied by the participating laboratories. When reporting ages based on the $^{238}$U–$^{206}$Pb radioactive decay system uncertainties ranging from less than ± 0.1% (isotope dilution, 1s) to ± 1% (*in situ* methods, 1s) are routinely provided. The goal of the G-Chron programme is to give participating laboratories an opportunity both to test for unrecognized bias in their age results and to provide information on the reliability of their uncertainty assignments. In circumstances where results submitted are shown to be unsatisfactory, it is recommended that those participating laboratories should investigate their procedures for unsuspected analytical bias in their results or unrecognized components in their uncertainty budget, allowing them to take corrective action if it appears warranted.

**The Organisation of G-Chron and its Scheduling in 2019**

Having already established PT schemes in bulk geochemical analysis and microanalysis, namely Geo*PT* and G-Probe respectively, the governing Council of the International Association of Geoanalysts voted in March 2015 to launch a Proficiency Testing (PT) scheme devoted principally to U-Pb zircon geochronology. The intention of this initiative was to produce PT materials consisting of concordant, homogeneous zircon. These materials would be assigned ages based on U-(Th)-Pb isotope dilution results from multiple TIMS laboratories having proven performance capabilities. Suitable material was to be identified, either from scientific publications or from other information sources, and the initial homogeneity testing of candidate zircon populations would be evaluated by ion microprobe. It was intended that the G-Chron PT programme would distribute a single test sample per year. Left-over materials from this work would subsequently be made available to the geoanalytical community. It was agreed that other phases besides zircon could be sent out as supplementary materials on a case-by-case basis during future rounds.

On-line registration for participation in the first round of G-Chron opened in June 2019. The Rak-17 zircon test material was dispatched to participants either at the end of August or during the first few days of September 2019. Each participant received a 0.5 ml screw-top plastic vial that contained a ~100 mg split of nearly pure zircon with a grain size ≤ 1 mm, which was supplied in a Ziploc plastic bag and labelled “Rak-17 Zircon”. A 1-page set of instructions accompanying the test material provided information about the material and the method for submitting results. The deadline for on-line data submission was set for 15 December 2019.

In October 2019 the [www.gchron.info](http://www.gchron.info) website, modelled on the websites of the Geo*PT* and G-Probe PT schemes, was opened for data submission, enabling participants to submit their results. Laboratories were required to provide $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ ages and the associated 1s uncertainties for both. Additionally, analysts had the option of submitting an age result for the $^{208}Pb/^{232}Th$ decay system along with its associated uncertainty. All values were to be reported in units of Ma (millions of years) with at least one digit to the right of decimal point to be provided. Furthermore, analysts were asked to provide metadata on key aspects of their procedures. On 16 December 2019 the on-line data submission portal was closed, at which point 63 laboratories had provided results. All laboratories that registered to take part in this round of G-Chron are eligible to download this report from the G-Chron website.

**Description and Origin of the Test Material**

The starting material for this study was derived from a pegmatitic schlieren, sampled in 2017 from the Skagerrak coast of Norway a few metres above the high tide line. The sampling coordinates were 58°58'41"N; 10°01'54"E, which is roughly 2.3 km south of the town of Stavern, Norway. The nearest geographic feature is the “Rakke Compass”, from which the name Rak-17, along with its year of collection, was derived. Mineralogically the material sampled from the schlieren was similar to its finer-grained host. The coarse-grained material collected contained large amounts of zircon as well as potassium feldspar crystals commonly exceeding 2 cm in size. Petrographically the rock is classified as a hornblende-biotite monzosyenite. It is part of the Permian Larvik Plutonic Complex that is positioned at the southern end of the onshore segment of the Oslo Rift magmatic province. Approximately 150 kg of starting material was collected from a ca. 5 m-long segment of one of the locally more prominent schlieren.

**Preparation of the Test Material**

The material was crushed using a jaw crusher followed by further grain size reduction using a roll mill and sieving, resulting in a < 1 mm grain-size sieve fraction. Zircons were concentrated using a Wilfley table, followed by Frantz magnetic separation operating at 2.2 A current and a side tile of 2°. Further enrichment of zircon involved using heavy liquids employing 1x bromoform, followed by 2x methylene iodide and a final clean-up with distilled water in an ultrasonic bath for 2 hours at room temperature, followed by an acetone rinse. Under a binocular microscope the resulting material was found to be largely dust-free and to consist of ca. 90% zircon. Observed impurities included apatite, feldspar, minor pyroxene and opaque phases. Some of the zircon grains displayed small amounts of adhering phases (mainly feldspar) and many grains showed rust-coloured staining. To remove non-zircon phases a 20-minute room temperature leach of the material was conducted using 20% hydrofluoric acid in an ultrasonic bath, followed by ~5 hours in 6M hydrochloric acid on a ~60°C hotplate, followed by rinsing and sonicating in copious amounts of distilled water and a final cleaning with high-purity ethanol. This resulted in a ca. 10% loss in mass with an end-product estimated at greater than 98% pure zircon, which was largely free of visible surface staining. At the end of this entire procedure 630.1 g of zircon was recovered. Finally, riffle splitters were used to prepare the individual units of material for use in both the ID-TIMS characterization and the G-Chron 2019 Proficiency Test.

**Derivation of the Assigned (Target) Age Values**

The initial testing of this material consisted of mounting numerous grains of Rak-17 in a 25.4 mm diameter epoxy disk along with the 91500 and Temora2 zircon calibration materials; this was done prior to the final splitting. SIMS U-Th-Pb determinations on this mount indicated Rak-17 zircon to be homogeneous at the uncertainty levels provided by the experiment, no evidence for either inheritance or for Pb-loss was found. Subsequently a more detailed SIMS investigation based on an entire 100 mg split – identical to those distributed within the PT programme – was completed; these results are presented in Figure A.

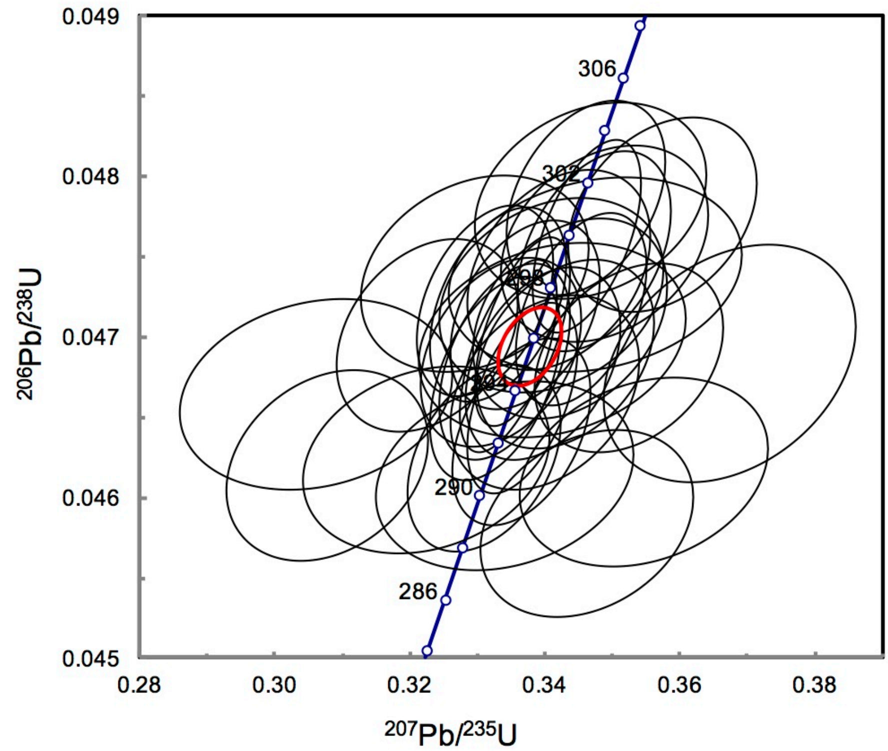


***Figure A:*** *U-Pb concordia plot for 36 determinations on Rak-17 obtained from the Potsdam SIMS instrument. The red ellipse is the mean value for this suite of data. All ellipses represent 1s uncertainties. The concordia curve and age values (in Ma) are based on the decay constants and the* $^{238}U/^{235}U$ *ratio of Steiger and Jäger (1977) without assigning any uncertainties to these values.*

As this material was deemed well suited for use in a U-Pb PT programme, seven isotope dilution thermal ionization mass spectrometry (ID-TIMS) laboratories of proven measurement capability were recruited to provide high-precision geochronology data. These included four laboratories using the EarthTime enriched isotope spikes and three laboratories using independently calibrated in-house spikes. In total, 68 U-Pb isotope dilution analyses were provided for Rak-17 – 35 using a chemical abrasion (CA) pre-treatment and 33 using less aggressive washing protocols. Additionally, one laboratory also reported seven Th-Pb isotope dilution determinations – four based on a CA pre-treatment and three based on less aggressive washing of the material prior to analysis.

While data from these experiments were being reported, an unexpected data pattern began to emerge in which the data from CA-based analyses formed a tight cluster (Figure B), whereas the results from less aggressive pre-treatment methods showed dispersion in $^{206}Pb/^{238}U$ ages reaching 7 Ma. The age cluster for the CA-based ages agreed well with the results of the *in situ* SIMS analyses determined by ion microprobe, though obviously the overall uncertainty from the SIMS results was significantly larger than the total range of the 35 CA ID-TIMS results (Figure B, inset). Importantly, it was found that in nearly all cases the $^{206}Pb/^{238}U$ data obtained from zircons not CA pre-treated were older than those from CA pre-treated material. This is opposite to the pattern that would normally be expected. Hence, this dataset cannot be explained by a simple Pb-loss model; rather it seems that the Rak-17 material may be coated with an anomalous radiogenic Pb component which can only be removed through a harsh cleaning protocol. Furthermore, this suspected extraneous Pb must have a radiogenic composition similar in age to the crystallization age of the Rak-17 zircon population. As this Pb component is apparently on the exterior of the grains only, it would not impact SIMS measurements. We believe this is the reason that the SIMS data both agree well with the CA-defined age and why they show no significant dispersion. We have therefore defined all three assigned or target ages of the Rak-17 material using exclusively the CA-based radiogenic isotope ratios reported by the participating ID-TIMS laboratories.

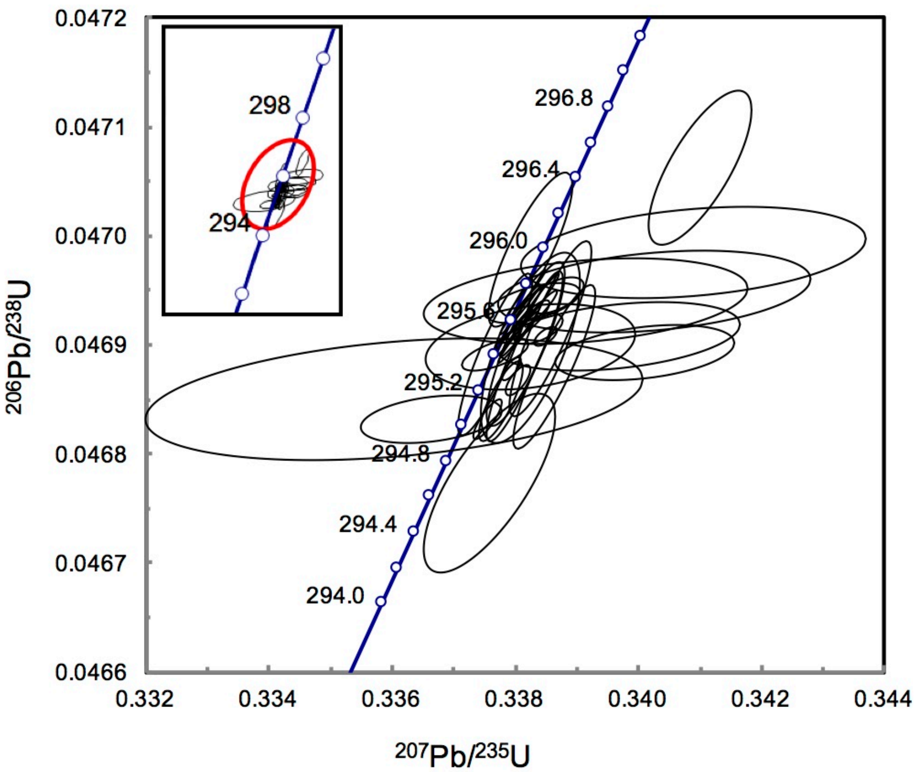


***Figure B:*** *U-Pb concordia plot for 35 determinations by isotope dilution as reported by seven independent TIMS laboratories. Inset upper left: the same data at a compressed scale along with a red ellipse showing the mean result from the SIMS data (see Figure A). All ellipses represent 1s uncertainties. The concordia curve and age values (in Ma) are based on the decay constants of Steiger and Jäger (1977).*

The assigned ages for this proficiency test, derived as robust means and their 1s uncertainties from the 35 CA ID-TIMS determinations for Rak-17 are, as reported in Table 1:

$^{206}Pb/^{238}Pb$ age: 295.56 ± 0.21 Ma

$^{207}Pb/^{206}Pb$ age: 300.34 ± 2.68 Ma

$^{208}Pb/^{232}Th$ age: 293.36 ± 0.80 Ma

Further details about the source data and the estimation of assigned ages will be presented in a separate publication (see below).

These age calculations are based on the radioactive decay constants and isotope ratio values recommended by Steiger and Jäger (1977), noting that these decay constants and the $^{238}U/^{235}U$ ratio in nature were presumed to be exact, with no associated uncertainties being propagated. In the case of $^{206}Pb/^{238}Pb$ and $^{207}Pb/^{206}Pb$ the robust mean ages are based on n = 7 laboratories and in the case of $^{208}Pb/^{232}Th$ the robust mean age is based on n = 4 individual CA ID-TIMS determinations. The associated age uncertainties (in Ma) are derived as half of the difference between (i) the age obtained from the given isotope ratio plus its corresponding uncertainty (1s) and (ii) the age obtained from that same isotope ratio minus its corresponding uncertainty (1s). The differences between the age results for the three chronometers needs to be a subject for future discussion, but we see no evidence in our data to suggest that Pb-loss has affected the Rak-17 material.

**Submission of Results**

Participants were asked to submit results obtained using their routine procedures and to report Rak-17 isotope ages, corresponding uncertainties and key details of procedures employed. Sixty-three laboratories submitted results: the numbers of values reported by each analytical method are given in Table 1. The majority of results were obtained by LA-ICP-MS methods with at least twice as many by Quadrupole and Time-of-Flight (QuadToF) as compared to Sector Field Instruments (SFI). Only one set of results for $^{206}Pb/^{238}U$ and $^{207}Pb/^{206}Pb$ was provided by ID-TIMS and 6 sets by SIMS. In all, 32 laboratories submitted $^{208}Pb/^{232}Th$ ages.

**Review of Results**

The age results contributed by all 63 laboratories are presented in Table 2, ordered according to analytical method and identified by a randomised laboratory code to ensure complete anonymity of participating laboratories. This table lists not only the ages and uncertainties that were submitted but also *z*-scores, explained below, which represent an index of performance for each result relative to the assigned value for each age. Some of the details of procedures employed are also listed in the table, demonstrating in particular that several different reference materials were used, although zircon 91500 was most prevalent. A cause for concern is that a range of $^{206}Pb/^{238}U$ ages were assigned to these reference materials, particularly for the zircon RM named JG-1.

Observations relating to the distribution of results may be made, not only from Table 2, but more easily from Figures 1, 2 and 3, in which each set of submitted data (both age and 1s uncertainty) is ordered by increasing age and colour coded according to the laboratory method used. Additionally, each of these figures shows how submitted results, identified by laboratory code, compare with the relevant Rak-17 assigned age. For the $^{206}Pb/^{238}U$ age, a number of laboratories submitted results in close agreement with the assigned age of 295.56 Ma. However, many results can be seen that diverge by several million years from the assigned age, with ~20% of the results diverging from the assigned age by more than 5 Ma. Figure 1 clearly shows a bias toward $^{206}Pb/^{238}U$ ages that are too young. For the $^{207}Pb/^{206}Pb$ age, the dataset is better centred on the assigned age, but a significant proportion of the results are more than 10 Ma from the assigned value of 300.34 Ma, with a large proportion of the LA-ICP-MS: QuadToF ages hugely discrepant, some to an extent that is far outside their own reported uncertainty estimates. For the $^{208}Pb/^{232}Th$ age, many results from the three methods employed agree reasonably well with the assigned age, but again many of the LA-ICP-MS: QuadToF ages and several of the LA-ICP-MS: SFI ages are hugely discrepant. In general, quoted uncertainties are smallest for isotope dilution, mostly quite small for SIMS, and range from relatively small to very large for LA-ICP-MS: QuadToF, whereas for LA-ICP-MS: SFI they are either quite small or quite large.

The wide variations in the uncertainty estimates by almost all methods makes for difficulties in assigning appropriate target precisions for the assessment of results (see later). Figures 1 to 3 reveal a number of laboratories reporting ages close to the assigned target value but which have large uncertainty assignments. In contrast, some reported ages differ significantly from the target ages, but have much smaller uncertainty estimates. The weak correlation between uncertainty estimates and divergence from the target value assigned suggests that a community-wide re-evaluation of data assessment methods could be beneficial.

*Note:* Our apologies for the inappropriate formatting of data in the final two columns of Table 2. This resulted from data acquisition as text rather than in numeric format; it will be rectified in future rounds.

**Performance Assessment – *Z*-scoring**

In proficiency testing, *z*-scoring provides a convenient way for laboratories to assess their performance in relation to a pre-defined fitness-for-purpose criterion. The *z*-score is a scaled measure of the deviation of the reported measurement from the assigned target value. We calculated *z*-scores for each of the 158 contributed measurement results as follows:

$$z = [\, x_i - x_{pt}] \,/\, \boldsymbol{\sigma}_{pt}$$

where $x_i$ is a reported laboratory result, $x_{pt}$ is the assigned target value (based on ID-TIMS data) and $\boldsymbol{\sigma}_{pt}$ is the standard deviation for proficiency also referred to as the target precision. The target precision to be applied in age dating has no historical precedent, unlike that employed in geochemical analysis. Consequently, for the purposes of this first round of G-Chron we elected to use the median of the reported uncertainties for a particular isotopic age system and for a particular analytical procedure: ID-TIMS, SIMS, LA-ICP-MS-SFI, LA-ICP-MS-QuadToF, and “other”, as the target precision. We should emphasise that as PT programmes applied to isotope ratio determinations is not a well-developed field, with no generally accepted performance criteria, this approach to defining target precisions must be viewed as experimental at this time (see also discussion below). The consequential 13 target precision values applicable for this round of G-Chron are listed in Table A, also in Table 1, and recorded in the legends of Figures 1 to 3. For isotope dilution and “other” methods there were too few data to derive a valid median, therefore in the cases of values listed from those five datasets extreme caution is advised.

***Table A:*** *Listing of target precision ($\boldsymbol{\sigma}_{pt}$) values (to be regarded as experimental at this time) and the limits of 'satisfactory' ranges whereby $-2 \le z \le 2$, for each isotopic system and each analytical procedure. *Values to be regarded with extreme caution.*

| Age Ratio | Analytical Technique | Target Precision (z = 1) | Satisfactory Range (z = ± 2) |
|---|---|---|---|
| | | $\boldsymbol{\sigma}_{pt}$ (Ma) | ± 2$\boldsymbol{\sigma}_{pt}$ (Ma) |
| $^{206}Pb/^{238}U$ | Isotope Dilution | 0.38* | ± 0.76* |
| $^{206}Pb/^{238}U$ | SIMS | 0.75 | ± 1.5 |
| $^{206}Pb/^{238}U$ | LA-ICP-MS: SFI | 2.6 | ± 5.2 |
| $^{206}Pb/^{238}U$ | LA-ICP-MS: QuadToF | 1.47 | ± 2.94 |
| $^{206}Pb/^{238}U$ | Other | 4.2* | ± 8.4* |
| $^{207}Pb/^{206}Pb$ | Isotope Dilution | 0.47* | ± 0.94* |
| $^{207}Pb/^{206}Pb$ | SIMS | 16.5 | ± 33.0 |
| $^{207}Pb/^{206}Pb$ | LA-ICP-MS: SFI | 7.4 | ± 14.8 |
| $^{207}Pb/^{206}Pb$ | LA-ICP-MS: QuadToF | 22.6 | ± 45.2 |
| $^{207}Pb/^{206}Pb$ | Other | 49.95* | ± 99.9* |
| $^{208}Pb/^{232}Th$ | SIMS | 1.75* | ± 3.5* |
| $^{208}Pb/^{232}Th$ | LA-ICP-MS: SFI | 8.0 | ± 16.0 |
| $^{208}Pb/^{232}Th$ | LA-ICP-MS: QuadToF | 4.15 | ± 8.3 |

The z-score value for each age measurement contributed to G-Probe 2019 is listed in Table 2. As is routine in proficiency testing, participating laboratories are invited to assess their performance using the following criteria: *Z*-score results in the range $-2 < z < 2$ are considered to be 'satisfactory', in the sense that no action by the participant is required. 'Satisfactory' ranges are shown graphically in the form of 'error bars' at the right hand sides of Figures 1 to 3. These figures permit participating laboratories to readily assess whether their submitted results are consistent with their own uncertainty estimates (as shown by error bars for individual laboratories) and whether their reported age can be considered 'satisfactory' according to the $-2 < z < 2$ criterion. If the *z*-score for any result falls outside this range, and especially if it is outside the range $-3 < z < 3$, laboratories are advised to examine their procedures and, if necessary, take appropriate action to ensure that their determinations are not subject to unsuspected analytical bias.

A consequence of this approach with recognising a target precision for each particular isotopic age system and each particular analytical procedure based on the median of the reported 1s uncertainties is that the performance of a given laboratory is assessed in relation to the performance of that methodological community. Hence, a given age result obtained by one method may be outside the 'satisfactory' range as provided by its *z*-score, and interpreted as having significant bias, whereas the exact same age result submitted using a different method might have resulted in a 'satisfactory' *z*-score.

We are aware that our experimentally derived target precisions, and therefore the derived *z*-scores, are not entirely adequate in some cases for the assessment of performance. We recommend that when the difference between a laboratory's reported age and the assigned age is greater than twice the uncertainty reported, that the laboratory reviews their results, particularly the possibility of unexpected bias or inappropriate estimation of the associated uncertainty.

**Overall Performance**

A summary of the overall performance of individual laboratories for this round of G-Chron is plotted in a multiple *z*-score chart in Figure 3. In this chart the *z*-score performance for each result is distinguished by symbols that make it easy to identify whether the measurement results were satisfactory or gave *z*-scores that exceeded the action limits (as shown by either the small or large triangle symbols). This chart is designed to help individual laboratories judge their overall performance in this proficiency test. Note, however, that participants should always review their *z*-scores in accordance with their own fitness-for-purpose requirements.

**Future G-Chron Participation**

As this proficiency test has revealed valuable data trends within the geoanalytical community, it is intended that further such rounds of G-Chron will be conducted. Assuming suitable material can be obtained, in conjunction with the ongoing support from expert ID-TIMS laboratories, it is intended that such proficiency tests will be conducted on an annual basis. As zircon is the most widely employed phase for U-(Th)-Pb geochronology research, zircon will remain the focus of future rounds. However, if suitably concordant material from other mineral phases can be obtained then such materials may also be employed in the G-Chron testing programme. News concerning upcoming rounds will be made available via both the *www.geoanalyst.org* and *www.gchron.info* websites.

The benefit from proficiency testing arises from regular participation and laboratories are invited to contribute to Round 2 of G-Chron, the test sample for which is expected to be distributed during late 2020.

**Future Actions Planned for the Rak-17 Material**

Due to the high yield of zircon from the collected rock starting material, over 6,000 units of Rak-17 remain. This material will be made available to the geoanalytical community via *www.IAGeo.com*, the website of IAGeo Limited, which is the trading arm of the International Association of Geoanalysts.

It is intended that a discussion of the results of this proficiency test will be submitted for publication as part of a detailed description of the Rak-17 characterization study. That paper will also explore possible origins of the differing age results between the various radioactive decay systems. Furthermore, the ID-TIMS work reported here was accompanied by parallel determinations on the 91500 zircon, which was used as a traceability material for the Rak-17 characterization. It is intended that these new data on the 91500 zircon will form the basis of an additional manuscript. Finally, this report will be made publicly available on the *www.geoanalyst.org* website at an appropriate time.

**Considerations for Modification of Assessments in Future Rounds of G-Chron**

The use of median uncertainty values to provide target precisions for each method, and the application of the $-2 < z < 2$ criterion for assessing whether data were fit-for-purpose were implemented in this first round of G-Chron with due consideration of procedures employed in the IAG's Geo*PT* whole rock testing programme. In the case of Geo*PT* the existence of the Horwitz empirical relationship (Horwitz, 1982) between the reproducibility uncertainty of quantity measurements and their magnitude provides a convenient fitness-for-purpose criterion. As the G-Chron programme is based on isotope ratios, rather than analyte quantities, there is no equivalent means of assessing expected data quality. An alternative design for defining fitness-for-purpose thresholds was therefore sought. A community-based strategy seemed a plausible alternative – namely using the median of reported uncertainty (1s) values as the target precision, $\boldsymbol{\sigma}_{\mathrm{pt}}$. As different analytical methods have both differing performance criteria and differing sample test portion masses, it seemed prudent that a given dataset should be judged only against the data quality reported by peer laboratories operating similar analytical designs. Hence the listing of individual values for target precision as derived for each method in Table A.

In view of the weak correlation between reported uncertainty estimates and the bias from the assigned target ages, this community-based strategy did not provide the expected outcome. Many results were clearly incongruent, with differences from the assigned ages that were substantially more than twice the laboratory's own uncertainty estimate, but which are nonetheless classified as fit-for-purpose based on the criteria used in this report. Such a result should more appropriately have been flagged as a source of concern and the reporting analyst should be encouraged both to investigate possible sources of bias in their results and to investigate the origin of a possible underestimation of their uncertainty estimates. We aim to resolve this shortcoming for future rounds of G-Chron. Possible solutions might include either a tightening of the target precision, e.g., assigning for a given method only half the median reported uncertainty as the target precision, effectively doubling z-scores, or by finding a more independent means of identifying acceptable laboratory performance e.g., abandoning the use of community-based uncertainty estimates for assessing data quality.

**Acknowledgements**

The authors wish to thank the following for various forms of assistance they have given to this programme: the seven expert TIMS laboratories for their analytical contributions, Thomas Meisel for support in the statistical evaluation of TIMS data, Frédéric Couffignal for providing SIMS data, Juliane Herwig for assistance with mineral separation and Phil Potts for guidance during the early design of the programme.

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**Table 1 - G-Chron 2019 Rak-17, Zircon - Assigned ages and uncertainties (1s), with target precisions for each analytical technique.**

| 206Pb/238U Age | Assigned Age (Ma) | Uncertainty (Ma) | Analytical Techniques | Target Precision (Ma) | Number of Values Reported |
|---|---|---|---|---|---|
| | 295.56 | 0.21 | ID | 0.38 | 1 |
| | 295.56 | 0.21 | SIMS | 0.75 | 6 |
| | 295.56 | 0.21 | LA-ICP-MS: SFI | 2.6 | 17 |
| | 295.56 | 0.21 | LA-ICP-MS: QuadToF | 1.47 | 37 |
| | 295.56 | 0.21 | OTHER | 4.2 | 2 |

| 207Pb/206Pb Age | Assigned Age (Ma) | Uncertainty (Ma) | Analytical Techniques | Target Precision (Ma) | Number of Values Reported |
|---|---|---|---|---|---|
| | 300.34 | 2.68 | ID | 0.47 | 1 |
| | 300.34 | 2.68 | SIMS | 16.5 | 6 |
| | 300.34 | 2.68 | LA-ICP-MS: SFI | 7.4 | 16 |
| | 300.34 | 2.68 | LA-ICP-MS: QuadToF | 22.6 | 36 |
| | 300.34 | 2.68 | OTHER | 49.95 | 2 |

| 208Pb/232Th Age | Assigned Age (Ma) | Uncertainty (Ma) | Analytical Techniques | Target Precision (Ma) | Number of Values Reported |
|---|---|---|---|---|---|
| | 293.36 | 0.8 | SIMS | 1.75 | 2 |
| | 293.36 | 0.8 | LA-ICP-MS: SFI | 8 | 8 |
| | 293.36 | 0.8 | LA-ICP-MS: QuadToF | 4.15 | 22 |

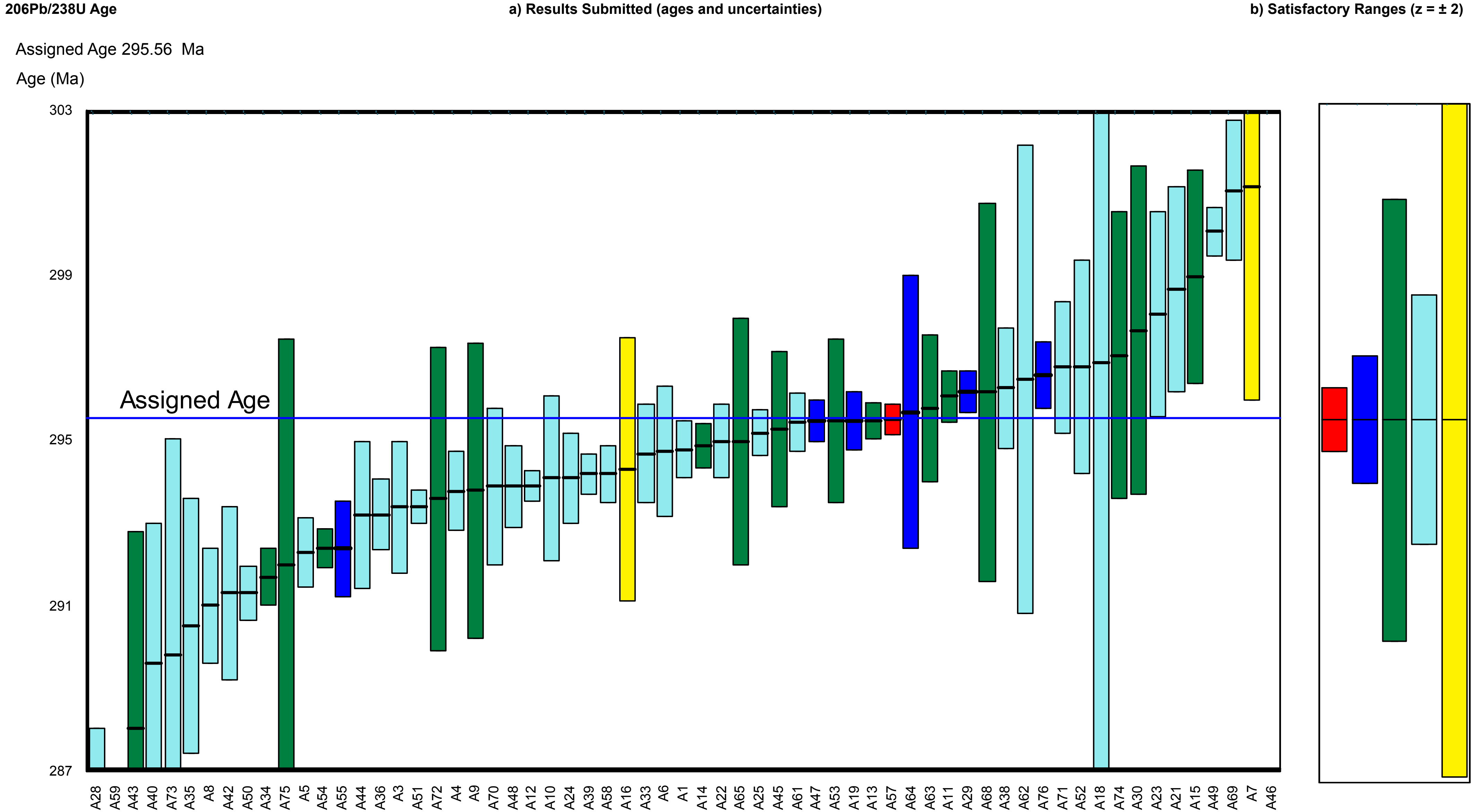
**Table 2 - G-Chron 2019 Contributed data and z-scores for Rak-17, Zircon, ordered according to analytical method and identified by randomised laboratory code. 15/12/2019**

| Lab Code | Method | 206Pb /238U Age (Ma) | 206Pb /238U Uncer -tainty (1s, Ma) | 206Pb /238U Age z-score | 207Pb /206Pb Age (Ma) | 207Pb /206Pb Uncer -tainty (1s, Ma) | 207Pb /206Pb Age z-score | 208Pb /232Th Age (Ma) | 208Pb /232Th Uncer -tainty (1s, Ma) | 208Pb /232Th Age z-score | Total Number of Measure -ments | Number of Reject- ions | Common Pb Correction | ID Spike | RM Name | RM 206Pb /238U Age (Ma) | RM 206Pb /238U 1 St Er of Mean |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A57 | ID | 295.529 | 0.38 | -0.08 | 295.46 | 0.47 | -10.38 | | | | 16 | 10 | 204Pb ancient | ET535 | | | |
| A19 | SIMS | 295.5 | 0.7 | -0.08 | 277.0 | 18.5 | -1.41 | | | | 23 | 1 | other | | Temora2 | 416.8 | |
| A29 | SIMS | 296.2 | 0.5 | 0.85 | 287.6 | 6.6 | -0.77 | | | | 90 | 1 | 204Pb modern | | 91500 | 1062 | 3.1 |
| A47 | SIMS | 295.5 | 0.5 | -0.08 | 301.0 | 14.0 | 0.04 | 292.1 | 1.8 | -0.72 | 32 | 0 | 204Pb ancient | | Temora2 | 416.8 | 1.6 |
| A55 | SIMS | 292.4 | 1.15 | -4.21 | 301.0 | 38.0 | 0.04 | | | | 38 | 1 | other | | BR266 | 559.1 Ma | 0.25% |
| A64 | SIMS | 295.7 | 3.3 | 0.19 | 301.0 | 36.0 | 0.04 | | | | 25 | 0 | 204Pb modern | | 91500 | 1062.4 | 1.9 Ma |
| A76 | SIMS | 296.6 | 0.8 | 1.39 | 295.0 | 14.5 | -0.32 | 291.0 | 1.7 | -1.35 | 20 | 0 | 204Pb modern | | 91500 | 1062.4 Ma | 8.5 Ma |
| A9 | LA-ICP-MS: SFI | 293.8 | 3.6 | -0.68 | 314.9 | 4.9 | 1.97 | | | | 77 | 39 | none | | GJ-1 | 601.86 | 0.37 |
| A11 | LA-ICP-MS: SFI | 296.1 | 0.62 | 0.21 | 305.5 | 11.7 | 0.70 | | | | 30 | 0 | none | | 91500 | 1062.4 | 3.1 (2s) |
| A13 | LA-ICP-MS: SFI | 295.51 | 0.44 | -0.02 | 295.8 | 3.7 | -0.61 | | | | 73 | 0 | none | | 91500 | 1065 | 1.3 |
| A14 | LA-ICP-MS: SFI | 294.9 | 0.55 | -0.25 | 309.2 | 1.3 | 1.20 | | | | 30 | 0 | none | | GJ-1 | 605.95 | 0.5 |
| A15 | LA-ICP-MS: SFI | 299.0 | 2.6 | 1.32 | 326.9 | 7.4 | 3.59 | 292.0 | 11.0 | -0.17 | 68 | 35 | none | | Plesovice | 339.4 | 2.6 |
| A30 | LA-ICP-MS: SFI | 297.7 | 4.0 | 0.82 | 293.8 | 6.0 | -0.88 | | | | 600 | 150 | other | | Plesovice | 337.13 Ma | 3 % (10 Ma. aprox |
| A34 | LA-ICP-MS: SFI | 291.7 | 0.7 | -1.48 | 314.0 | 6.5 | 1.85 | 283.3 | 1.3 | -1.26 | 35 | 0 | none | | GJ-1 | 601.7 | 1.0 |
| A43 | LA-ICP-MS: SFI | 288.0 | 4.8 | -2.91 | 308.8 | 5.1 | 1.14 | | | | 34 | 0 | none | | GJ-1 | 604.5 | 1.1 |
| A45 | LA-ICP-MS: SFI | 295.3 | 1.9 | -0.10 | 310.7 | 11.7 | 1.40 | 297.7 | 3.7 | 0.54 | 25 | 1 | none | | GJ-1 | 601.95 | 0.2 |
| A53 | LA-ICP-MS: SFI | 295.5 | 2.0 | -0.02 | 305.0 | 33.6 | 0.63 | | | | 6 | 0 | none | | GJ-1 | 600.62 | 2.4 |
| A54 | LA-ICP-MS: SFI | 292.4 | 0.48 | -1.22 | 740.0 | 25.5 | 59.41 | 262.2 | 1.8 | -3.90 | 30 | 3 | none | | 91500 | 1062 | 4.7 Ma |
| A63 | LA-ICP-MS: SFI | 295.8 | 1.8 | 0.09 | 302.6 | 3.5 | 0.31 | | | | 17 | 0 | none | | 91500 | 1065.4 | 0.3 |
| A65 | LA-ICP-MS: SFI | 295.0 | 3.0 | -0.22 | 299.9 | 28.0 | -0.06 | 292.6 | 15.9 | -0.09 | 24 | 0 | none | | 91500 | 1061 | 1 |
| A68 | LA-ICP-MS: SFI | 296.2 | 4.6 | 0.25 | 294.1 | 57.3 | -0.84 | 292.0 | 13.8 | -0.17 | 36 | 1 | none | | 91500 | 1062.4 | 2.0 |
| A72 | LA-ICP-MS: SFI | 293.6 | 3.7 | -0.75 | 305.0 | 25.1 | 0.63 | 280.3 | 9.1 | -1.63 | 120 | 48 | other | | GJ-1 | 600.4 | 6.9 |
| A74 | LA-ICP-MS: SFI | 297.1 | 3.5 | 0.59 | 310.0 | 27.6 | 1.31 | 297.3 | 6.9 | 0.49 | 30 | 1 | none | | 91500 | 1062 | 0 |
| A75 | LA-ICP-MS: SFI | 292.0 | 5.5 | -1.37 | 0.0 | 0.0 | | | | | 56 | 0 | none | | 91500 | 1063 | 0.39 |
| A1 | LA-ICP-MS: QuadTo | 294.8 | 0.7 | -0.52 | 299.0 | 16.0 | -0.06 | 255.4 | 2.6 | -9.15 | 15 | 2 | none | | GJ-1 | 600 | 3 |
| A3 | LA-ICP-MS: QuadTo | 293.4 | 1.6 | -1.47 | 296.0 | 42.0 | -0.19 | | | | 35 | 3 | none | | 91500 | 1062.7 | 6.7 |
| A4 | LA-ICP-MS: QuadTo | 293.79 | 0.97 | -1.20 | 301.0 | 26.0 | 0.03 | 295.9 | 5.3 | 0.61 | 63 | 1 | none | | GJ-1 | 600.3 | 3.1 |
| A5 | LA-ICP-MS: QuadTo | 292.3 | 0.85 | -2.22 | 277.0 | 15.0 | -1.03 | | | | 20 | 1 | none | | GJ-1 | 600 | 600 |
| A6 | LA-ICP-MS: QuadTo | 294.76 | 1.59 | -0.54 | 355.4 | 30.2 | 2.44 | 293.78 | 2.47 | 0.10 | 30 | 0 | none | | 91500 | 1065 | 2.38 |
| A8 | LA-ICP-MS: QuadTo | 291.0 | 1.4 | -3.10 | 311.0 | 18.0 | 0.47 | 294.0 | 3.1 | 0.15 | 71 | 2 | none | | 91500 | 1062.4 Ma | 4.1 Ma (2 standar |
| A10 | LA-ICP-MS: QuadTo | 294.1 | 2.0 | -0.99 | 235.0 | 45.1 | -2.89 | | | | 30 | 29 | none | | Temora2 | 417 | 2.9 |
| A12 | LA-ICP-MS: QuadTo | 293.92 | 0.37 | -1.12 | 288.1 | 7.4 | -0.54 | | | | 35 | 0 | none | | GJ-1 | 601.8 | 0.4 |
| A18 | LA-ICP-MS: QuadTo | 296.9 | 11.7 | 0.91 | 568.5 | 478.1 | 11.87 | | | | 30 | 1 | none | | Other | 523 | 5 |
| A21 | LA-ICP-MS: QuadTo | 298.7 | 2.5 | 2.14 | 292.0 | 25.0 | -0.37 | 290.6 | 4.8 | -0.67 | 22 | 0 | 204Pb modern | | GJ-1 | 608.5 | 0.4 |

**Table 2 - G-Chron 2019 Contributed data and z-scores for Rak-17, Zircon, ordered according to analytical method and identified by randomised laboratory code. 15/12/2019**

| Lab Code | Method | 206Pb /238U Age (Ma) | 206Pb /238U Uncer -tainty (1s, Ma) | 206Pb /238U Age z-score | 207Pb /206Pb Age (Ma) | 207Pb /206Pb Uncer -tainty (1s, Ma) | 207Pb /206Pb Age z-score | 208Pb /232Th Age (Ma) | 208Pb /232Th Uncer -tainty (1s, Ma) | 208Pb /232Th Age z-score | Total Number of Measure -ments | Number of Reject- ions | Common Pb Correction | ID Spike | RM Name | RM 206Pb /238U Age (Ma) | RM 206Pb /238U 1 St Er of Mean |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| A22 | LA-ICP-MS: QuadTo | 295.0 | 0.9 | -0.38 | 296.9 | 22.6 | -0.15 | | | | 20 | 1 | other | | 91500 | 1065 | 2.98 |
| A23 | LA-ICP-MS: QuadTo | 298.1 | 2.5 | 1.73 | 297.1 | 14.8 | -0.14 | 290.1 | 6.9 | -0.79 | 65 | 2 | none | | 91500 | 1065 | 5.7 |
| A24 | LA-ICP-MS: QuadTo | 294.1 | 1.1 | -0.99 | 248.0 | 25.0 | -2.32 | | | | 15 | 3 | none | | 91500 | 1062.4 | 2 |
| A25 | LA-ICP-MS: QuadTo | 295.2 | 0.55 | -0.24 | 319.0 | 9.0 | 0.83 | 258.2 | 2.0 | -8.47 | 30 | 3 | none | | GJ-1 | 600 | 3 |
| A28 | LA-ICP-MS: QuadTo | 283.0 | 5.0 | -8.54 | 407.9 | 110.0 | 4.76 | | | | 30 | 8 | none | | 91500 | 1065.4 | 6.4 |
| A33 | LA-ICP-MS: QuadTo | 294.7 | 1.2 | -0.59 | 279.0 | 24.0 | -0.94 | 298.6 | 2.0 | 1.26 | 45 | 1 | none | | 91500 | 1065 | 8.6 |
| A35 | LA-ICP-MS: QuadTo | 290.5 | 3.1 | -3.44 | 244.0 | 36.0 | -2.49 | 272.0 | 12.0 | -5.15 | 27 | 0 | none | | 91500 | | |
| A36 | LA-ICP-MS: QuadTo | 293.21 | 0.86 | -1.60 | 0.0 | 0.0 | | | | | 45 | 10 | none | | GJ-1 | 600.4 | 0.174 |
| A38 | LA-ICP-MS: QuadTo | 296.29 | 1.47 | 0.50 | 296.04 | 4.21 | -0.19 | | | | 32 | 2 | none | | 91500 | 1065 | 1.79 |
| A39 | LA-ICP-MS: QuadTo | 294.2 | 0.5 | -0.93 | 224.0 | 15.0 | -3.38 | | | | 30 | 1 | none | | 91500 | 1062.3 | 1.2 |
| A40 | LA-ICP-MS: QuadTo | 289.6 | 3.4 | -4.05 | 281.8 | 35.4 | -0.82 | 289.2 | 5.6 | -1.00 | 14 | 1 | none | | 91500 | 1062.4 | 0.4 |
| A42 | LA-ICP-MS: QuadTo | 291.3 | 2.1 | -2.90 | 309.0 | 54.0 | 0.38 | 286.0 | 4.2 | -1.77 | 30 | 10 | none | | 91500 | 1062Ma | Â±10Ma |
| A44 | LA-ICP-MS: QuadTo | 293.2 | 1.8 | -1.61 | 288.0 | 37.0 | -0.55 | | | | 50 | 3 | none | | 91500 | 1058.4 | 8.9 |
| A46 | LA-ICP-MS: QuadTo | 305.7 | 1.8 | 6.90 | 417.0 | 51.0 | 5.16 | 309.3 | 5.1 | 3.84 | 24 | 2 | none | | 91500 | 1062.5 Ma | 4.4 Ma |
| A48 | LA-ICP-MS: QuadTo | 293.9 | 1.0 | -1.13 | 349.0 | 22.0 | 2.15 | 293.5 | 2.4 | 0.03 | 58 | 33 | none | | GJ-1 | 601.92 | 0.7 |
| A49 | LA-ICP-MS: QuadTo | 300.1 | 0.6 | 3.09 | 357.8 | 15.8 | 2.54 | 300.1 | 0.6 | 1.62 | 40 | 0 | none | | 91500 | 1062.4 | 3.5 |
| A50 | LA-ICP-MS: QuadTo | 291.3 | 0.65 | -2.90 | 277.0 | 6.0 | -1.03 | 277.6 | 1.15 | -3.80 | 85 | 0 | none | | 91500 | 1065 Ma in the : | 0.28 |
| A51 | LA-ICP-MS: QuadTo | 293.4 | 0.4 | -1.47 | 385.3 | 39.4 | 3.76 | | | | 48 | 6 | none | | 91500 | 1063 | 0.5 |
| A52 | LA-ICP-MS: QuadTo | 296.8 | 2.6 | 0.84 | 291.0 | 7.1 | -0.41 | | | | 25 | 0 | none | | 91500 | 1065 | 1 |
| A58 | LA-ICP-MS: QuadTo | 294.2 | 0.7 | -0.93 | 304.0 | 18.0 | 0.16 | 292.6 | 2.3 | -0.18 | 25 | 0 | other | | 91500 | 1063.51 | 1.85 |
| A59 | LA-ICP-MS: QuadTo | 285.6 | 0.7 | -6.78 | 301.0 | 6.0 | 0.03 | | | | 42 | 1 | none | | Plesovice | 337 | .17 |
| A61 | LA-ICP-MS: QuadTo | 295.47 | 0.71 | -0.06 | 309.0 | 16.0 | 0.38 | 293.0 | 2.0 | -0.09 | 44 | 6 | none | | GJ-1 | 600.46 | 0.701 |
| A62 | LA-ICP-MS: QuadTo | 296.5 | 5.7 | 0.64 | 672.1 | 334.6 | 16.45 | 317.9 | 20.5 | 5.91 | 25 | 0 | none | | 91500 | 1064 | |
| A69 | LA-ICP-MS: QuadTo | 301.1 | 1.7 | 3.77 | 331.0 | 42.0 | 1.36 | 305.1 | 4.3 | 2.83 | 19 | 1 | none | | | | |
| A70 | LA-ICP-MS: QuadTo | 293.9 | 1.9 | -1.13 | 309.3 | 42.0 | 0.40 | 292.5 | 5.0 | -0.21 | 27 | 5 | none | | 91500 | 1062.4 | 6.6 |
| A71 | LA-ICP-MS: QuadTo | 296.8 | 1.6 | 0.84 | 301.9 | 6.3 | 0.07 | 301.3 | 4.1 | 1.91 | 30 | 2 | 204Pb ancient | | 91500 | 1064 Ma | 10 Ma |
| A73 | LA-ICP-MS: QuadTo | 289.8 | 5.24 | -3.92 | 1.1 | 1.1 | -13.24 | 281.42 | 11.29 | -2.88 | 20 | 1 | other | | 91500 | 1065 | |
| A7 | Other | 301.2 | 5.2 | 1.34 | 284.4 | 32.6 | -0.32 | | | | 16 | 3 | none | | | | |
| A16 | Other | 294.3 | 3.2 | -0.30 | 323.7 | 67.3 | 0.47 | | | | 314 | 0 | none | | | | |

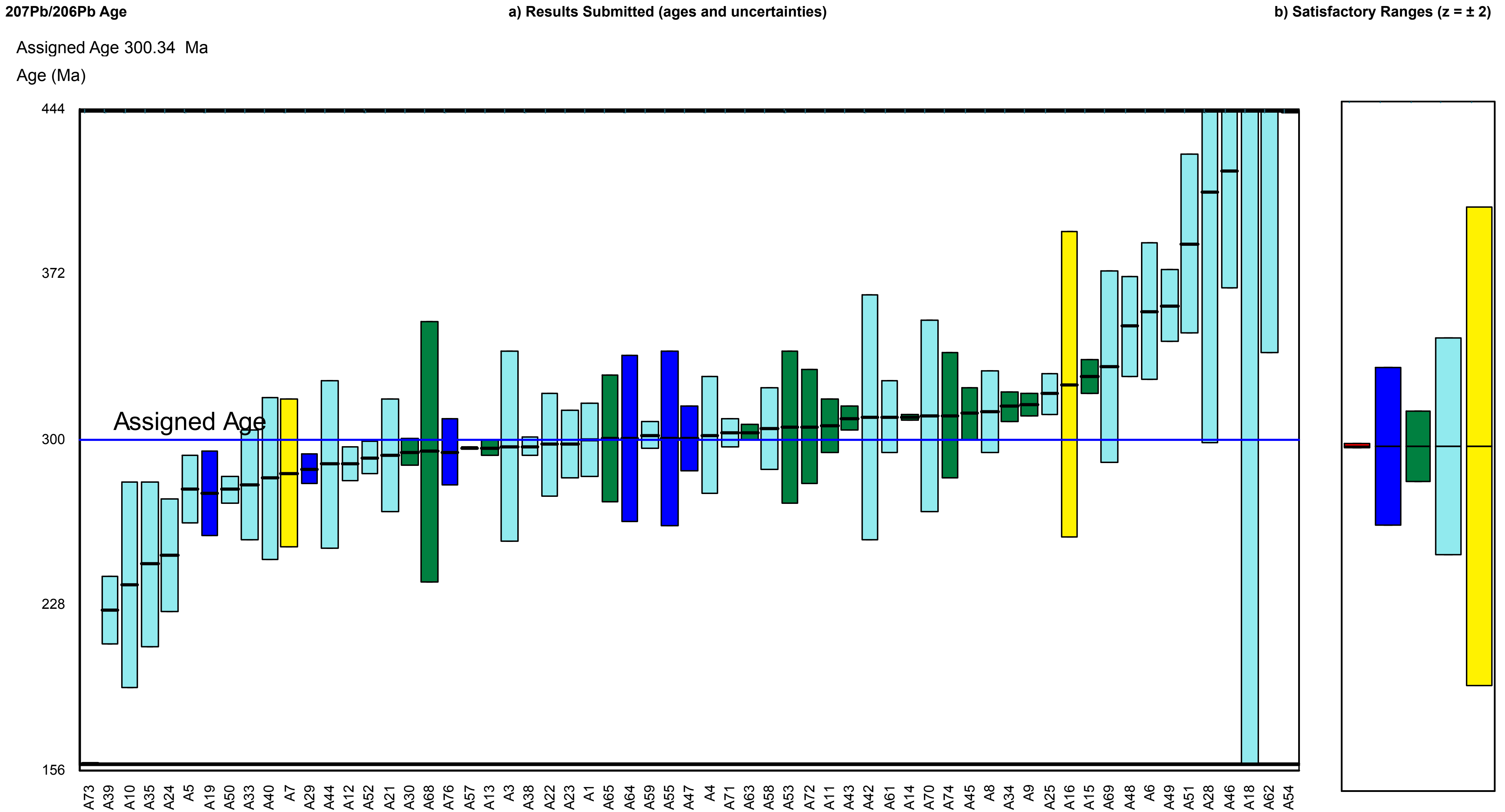
**Figure 1: - Distribution of contributed 206Pb/238U age results for G-Chron 2019 in relation to the assigned age, specified target precisions (z = 1) and corresponding satisfactory ranges (z = ± 2) for Rak-17, Zircon**




Data plotted in a) according to method, as shown by colour, with reported uncertainties (1s) shown as error bars. Satisfactory ranges (z = ± 2) represented in b) for each method.
Target Precision (z = 1) values (Ma) specified below, with colour code defined for each analytical method.

| Isotope Dilution | SIMS | LA-ICP-MS: SFI | LA-ICP-MS: QuadToF | Other |
|---|---|---|---|---|
| 0.38 | 0.75 | 2.60 | 1.47 | 4.20 |

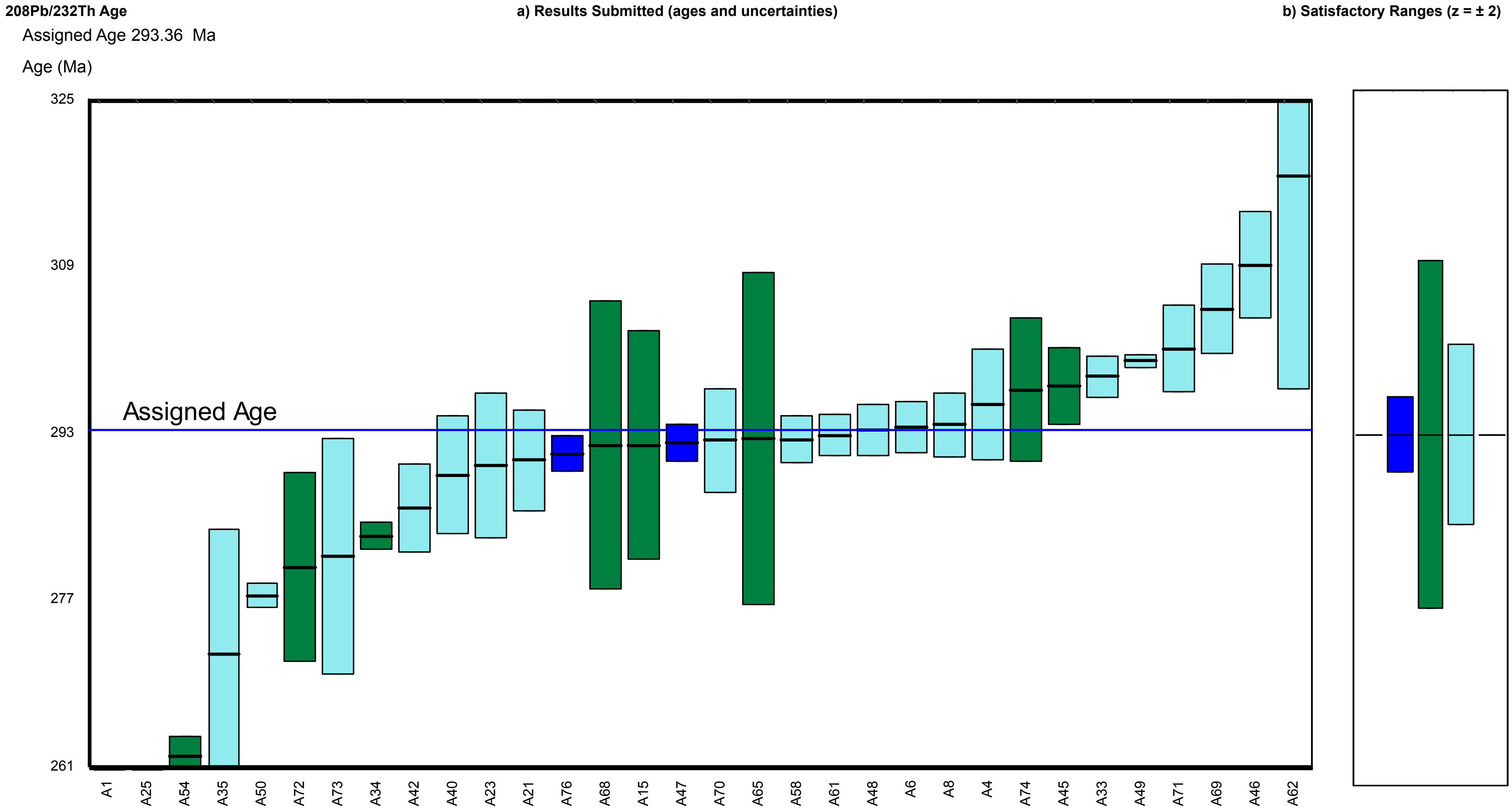
**Figure 2: - Distribution of contributed 207Pb/206Pb age results for G-Chron 2019 in relation to the assigned age, specified target precisions (z = 1) and corresponding satisfactory ranges (z = ± 2) for Rak-17, Zircon**




Data plotted in a) according to method, as shown by colour, with reported uncertainties (1s) shown as error bars. Satisfactory ranges (z = ± 2) represented in b) for each method. Target Precision (z = 1) values (Ma) specified below, with colour code defined for each analytical method.

| Isotope Dilution | SIMS | LA-ICP-MS: SFI | LA-ICP-MS: QuadToF | Other |
|---|---|---|---|---|
| 0.47 | 16.50 | 7.40 | 22.60 | 49.95 |

**Figure 3: - Distribution of contributed 208Pb/232Th age results for G-Chron 2019 in relation to the assigned age, specified target precisions (z = 1) and corresponding satisfactory ranges (z = ± 2) for Rak-17, Zircon**




Data plotted in a) according to method, as shown by colour, with reported uncertainties (1s) shown as error bars. Satisfactory ranges (z = ± 2) represented in b) for each method. Target Precision (z = 1) values (Ma) specified below, with colour code defined for each analytical method.

| Isotope Dilution | SIMS | LA-ICP-MS: SFI | LA-ICP-MS: QuadToF | Other |
|---|---|---|---|---|
| | 1.75 | 8.00 | 4.15 | |

**Multiple Z-Score Chart for G-Chron 2019**

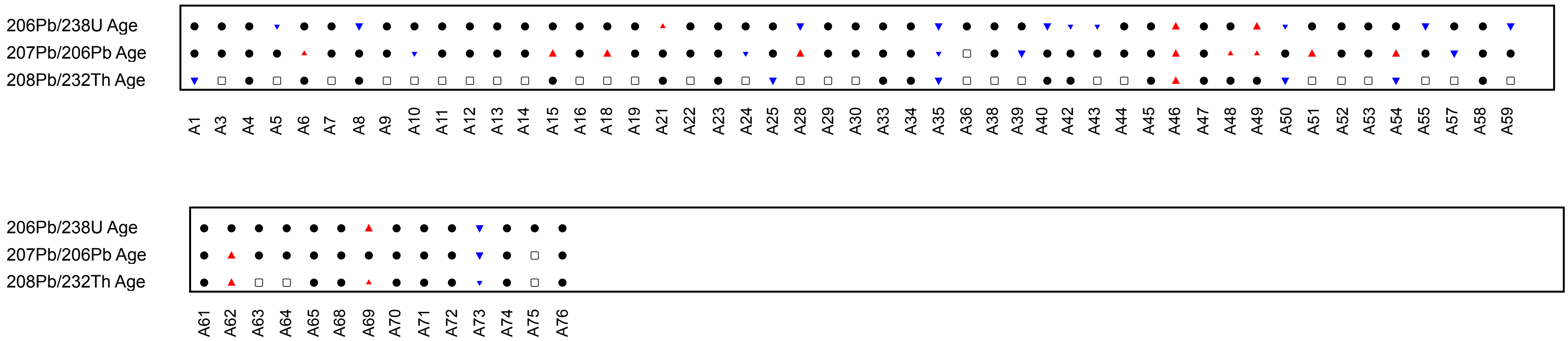
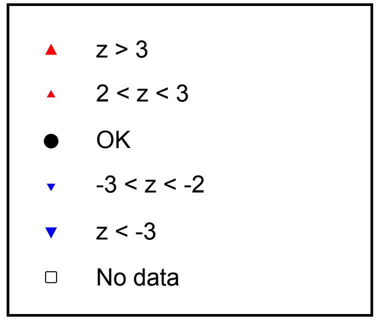



Figure 4: G-Chron 2019 - Rak-17, Zircon. Multiple z-score charts for laboratories participating in the G-Chron 2019 round. Symbols indicate whether or not an elemental result complies with the -2<z<+2 criteria (see key).



ISSN 2190-7110