IUPAC Technical Report

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IUGS–IUPAC recommendations and status reports on the half-lives of ^{87}Rb, ^{146}Sm, ^{147}Sm, ^{234}U, ^{235}U, and ^{238}U (IUPAC Technical Report)

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Abstract: The IUPAC–IUGS joint Task Group "Isotopes in Geosciences" (TGIG) has evaluated the published literature on the half-lives of six long-lived, geologically relevant radioactive nuclides. Where conflicting literature estimates are present, it is necessary to first identify any systematic bias in accordance with metrological traceability and to exclude the biased estimates from further consideration. The TGIG recommends three robust half-life estimates: 49.61 ± 0.16 Ga for ^{87}Rb, corresponding to a decay constant $\lambda(^{87}\text{Rb}) = (1.3972 \pm 0.0045) \times 10^{-11}$ a^{-1}; 106.25 ± 0.38 Ga for ^{147}Sm, and a corresponding decay constant $\lambda(^{147}\text{Sm}) = (6.524 \pm 0.024) \times 10^{-12}$ a^{-1}; 4.4683 ± 0.0096 Ga for ^{238}U, *i.e.* a decay constant $\lambda(^{238}\text{U}) = (1.55125 \pm 0.00333) \times 10^{-10}$ a^{-1}. All cited uncertainties have a coverage factor $k = 2$. For other radionuclides of Sm and U, no unambiguous consensus value can be endorsed at present by TGIG, which limits its evaluation to a status report highlighting unaccounted-for potential sources of bias. The improved repeatability of mass spectrometric measurements has revealed systematic bias

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Dedicated to Norman E. Holden, a long-serving member of the Inorganic Chemistry Division and the Commission on Isotopic Abundance and Atomic Weights. Dr. Holden recently passed away on 18 August 2022, during the proofing stage of this report.

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effects that had been dismissed as subordinate in the past. These issues can only be resolved by future dedicated investigations.

Keywords: ^{87}Rb; ^{146}Sm; ^{147}Sm; ^{234}U; ^{235}U; ^{238}U; decay constants; half-life.

CONTENTS

1 Introduction **1086**
2 Evaluation criteria **1086**
3 Evaluations **1088**
3.1 ^{87}Rb 1088
3.2 ^{147}Sm 1089
3.3 ^{146}Sm 1089
3.4 ^{238}U 1090
3.5 ^{235}U 1090
3.6 ^{234}U 1090
4 Future perspectives **1091**
5 Membership of sponsoring bodies **1091**
References **1092**

1 Introduction

The Task Group "Isotopes in Geosciences" (TGIG) was jointly established by the International Union of Pure and Applied Chemistry (IUPAC) and the International Union of Geological Sciences (IUGS) to recommend consensus half-life values for the long-lived radioactive nuclides that are used in geochronology. So far, it has evaluated the published measurement results for the decay constant and half-life of ^{87}Rb, ^{146}Sm, ^{147}Sm, ^{234}U, ^{235}U, and ^{238}U. The detailed argumentations of each evaluation, and an extensive reference list, are presented by Villa *et al.* [1–3] in a commercial journal widely disseminated in the geochemical and cosmochemical community. The present Technical Report was prepared in order to summarize for the IUPAC membership the conclusions of those three references. Before the actual recommendations are summarized here, it is necessary to explain the procedures applied in the evaluations.

2 Evaluation criteria

The TGIG performed a meta-analysis of data produced by other research groups, with a special emphasis on an independent reassessment of the uncertainty budget. The key difficulty, and the key expertise of the TGIG, is diagnosing and evaluating measurement uncertainties as defined by the binding international conventions ([4], entries 2.9, 2.10, and 2.26). As some measurement results "may be more representative of the measurand than others" ([4], entry 2.9), a significant part of the present evaluation was the assessment, following strict metrological criteria [5], of the measurement uncertainties. Of special concern is the oft-misunderstood difference between precision and accuracy, which follows from Type A and Type B evaluations, respectively ([4], entries 2.28 and 2.29). Type A evaluations pertain to the repeatability of results under the same conditions of measurement; type B evaluations focus on all other sources of uncertainty. The respective uncertainties can be called "measurement precision" and "systematic measurement errors" ([4], entries 2.15 and 2.17). The former (whose informal equivalents are "random error" and "statistical uncertainty") can be improved upon by analytical developments; the latter need to be identified by expert evaluation, if appropriate excluding inaccurate measurements from the database. Throughout this paper, uncertainties resulting from Type A and Type B evaluations are converted to combined standard measurement uncertainties ([4], entry 2.31) and assigned a coverage factor $k = 2$ ([4], entry 2.38).

The continuing improvement of measurement repeatability, due to improved analytical procedures, has laid bare several systematic bias factors that had been deemed negligible half a century ago. This situation requires a decision on how to distinguish acceptable measurements from incorrect ones. One approach is to apply the statistical treatment appropriate for a normally distributed, unimodal random variable, represented by a Gaussian function, to the entire database from the literature, and then optimize the dispersion (quantified by the reduced chi square, χ^2_n, defined as the sum of the variance-weighted residuals normalized by the number of degrees of freedom, in the case of a weighted average the number of measurements minus one) by progressively removing measurements until a χ^2_n value acceptably near the expectation value of 1 is achieved. The progressive removal procedure rejects outliers, irrespective of the existence of experimental flaws. The requirement that χ^2_n be ≤1 is explained by the value of the "probability of fit", p. If $\chi^2_n > 1$, the dispersion of the measured points from the calculated ones (be it by linear regression or by averaging) is larger than the repeatability of the measurement; the data are then said to be overdispersed. This implies that the dispersion of values remains uncorrected for systematic errors, which include, possibly alongside other contributions, "incomplete knowledge of certain physical phenomena" ([5], §D.4). If $\chi^2_n > 1$, this also means that the probability p that the calculation accurately reflects the process that caused the distribution of the measured points becomes progressively smaller. The more typical examples involve treating bimodal distributions as if they were unimodal (*e.g.*, in the case of the ^{146}Sm half-life, see below) and linearly modeling a two-stage geological process as if it were single stage (as can occur in geochronological intercomparisons, see below). Since the probability that the function used for the calculation correctly predicts the observed dispersion is equal to p, the probability that the calculation is incorrect is $1 - p$; for a χ^2_n so high that $p = 0.05$, the likelihood of misidentifying the process controlling the distribution of the data points is 95 %.

An entirely different approach is to identify flawed experiments affected by systematic errors. To this end, one aims to distinguish and evaluate the repeatability of measurements, typically via Type A evaluations ([4], entries 2.15 and 2.26), and the presence of systematic errors, typically via Type B evaluations "based on experience" ([4], entries 2.17 and 2.26). The IUPAC–IUGS Task Group has carried out Type B "expert" evaluations of the measurement protocols, and this is reflected in the present recommendations, which endeavored a recognition of systematic bias factors and the exclusion of inaccurate measurement results in order to estimate the half-lives. An important tenet of Type B evaluations is that if a systematic bias is suspected, the "interval of reasonable values for the measurand" ([4], p. viii) needs to be as wide as to include the highest possible bias. A real-life example of contrasting conclusions when applying Type A and Type B criteria is discussed by Villa *et al.* ([3], Appendix A).

In the opinion of the TGIG, it is essential to highlight the substantial difference between averaging an entire data set as is and only averaging a subset of the data set following a Type B evaluation that removed from consideration those data that are likely biased. Averages are only legitimate in a data set whose results can be modeled as draws from a Gaussian distribution, which the evaluated experimental data sets may not necessarily be. On the other hand, "intervals of reasonable values" as defined by JCGM [4] take into account the true value plus the systematic inadequacy of a given experimental design to actually find it. Estimating the common intersection of all data sets after Type B evaluation can be viewed as a generalization of the concept of weighted average, which is only meaningful for a unimodal random distribution. For those data sets that do not obey a normal, unimodal, random distribution represented by a Gaussian function, the estimate of the coverage interval ([4], § 2.36) must incorporate the external experts' Type B evaluation.

The fundamental lesson inherent in the initial evaluation by Begemann *et al.* [6] is that literature data can sometimes be incorrectly interpreted by the very authors of the experimental paper. Starting from the recognition that if N papers propose N mutually incompatible results, then at least $(N - 1)$ have misjudged their Type B uncertainties, it becomes obvious that even peer-reviewed papers do not necessarily represent Absolute Truth, be it in the form of accurate measurements, or of accurate interpretations, or both. It becomes essential to understand which of the N data sets is flawed and why. A re-analysis and extensive re-calculation of primary data is highly recommended when reading any paper, especially when one intends to subsume and build upon the conclusions of that paper for one's own work.

The determination of half-lives of the comparatively long-lived radioactive nuclides used in geochronology can follow one of three broad experimental designs (*cf.* [6]). (1) Counting decay events of the radioactive parent nuclides. (2) Measuring the artificial accumulation of radiogenic product isotopes in the laboratory. (3) Measuring the natural accumulation of radiogenic product isotopes in minerals of known age (usually by assuming that the ^{238}U–^{206}Pb age of a rock is identical to that obtained using a different parent–product pair on a mineral of the same rock). All three of these designs require a highly precise value for the amount of parent nuclide. If that amount is not accurately determined, a large component of systematic uncertainty is introduced. The third approach, *i.e.* geochronological intercomparison, does not occur in controlled, metrologically traceable conditions and is subject to one crucial assumption: that the mineral chronometers being investigated formed in a "point-like geological event" [6] and have remained ideally impervious to gain or loss of both parent and product nuclides. Any violation of the requirement that the accumulation of product isotopes in the mineral chronometer be unaffected by secondary processes (mineralogical, chemical, thermal, mechanical, etc.) can in principle be detected by a non-isotopic investigation that assesses petrological equilibrium.

In this evaluation, we treat geochronological intercomparisons separately from counting experiments, as the metrological traceability only applies to the analytical part of the geochronological measurements, but not to establishing the equivalence of the two age determinations. Assessing equivalence inherently requires a Type B evaluation ([4], § 2.29), as the intercomparison relies on all mineral phases ($n > 3$, ideally $n >> 3$) that are used to calculate an overdetermined isochron being cogenetic, *i.e.* formed at the same time (or in an unresolvably short time interval) and having remained closed to isotope exchange after formation; the evidence of such ideal "point-like" geological histories can only be established after their measurement. In the present evaluations, the available cosmo- and geochronological intercomparisons were filtered by the requirement that the data define an overdetermined internal isochron having a statistically acceptable dispersion, *i.e.* $\chi^2_n \leq 1$, for all reported isotopic systems. This rigor is justified by the profound difference between "estimating the age of an individual sample" and "establishing a sample as an acceptable age calibrator for universal use." In the former case, most practitioners will accept some data overdispersion, even if $\chi^2_n > 1$ means that the likelihood that the result is accurate and drops below 50 %. When the purpose of a measurement is dating a single sample, any inaccurate assumption only affects the accuracy of that sample. On the contrary, the selection of age calibrators ([4], § 5.13) must be much stricter, as any systematic inaccuracy cascades on the accuracy of all measurements that rely on it. A geochronological intercomparison using natural rock samples as age calibrators is only as good as the least homogeneous and the least pristine of the mineral chronometers in the comparison chain; therefore, the requirements for absence of any secondary modification must be much stricter than for general dating purposes and it is vital that χ^2_n be always ≤1 for the selected overdetermined isochron calculations. Recent years have witnessed a philosophical change of paradigm (*cf.* [3]), which recognizes that the non-isotopic information on petrogenesis is at least as important as mass spectrometer precision. This also ought to apply to the counting experiments, in that the information on material purity, amount of parent nuclide, isotopic composition, and other components of the apparatus is at least as important as counting itself.

3 Evaluations

3.1 ^{87}Rb

The TGIG [1] has reviewed papers covering all three experimental approaches detailed above, namely, counting decay events, artificial accumulation, and natural accumulation (also referred to as geochronological intercomparison). The three independent experimental approaches yielded three very similar values for the ^{87}Rb half-life ([1], Table 1). While in principle, it is possible that each experiment was affected by a bias, experimental artefacts would have had to be of the same magnitude and the same direction to obtain three coincident inaccurate values. As this is quite unlikely, the TGIG proposes that this agreement be viewed as a

very strong indication that each paper had carefully evaluated the presence of potential bias and successfully corrected for it.

The TGIG recommends a value of (49.61 ± 0.16) Ga for the half-life of ^{87}Rb, corresponding to a decay constant $\lambda(^{87}\text{Rb}) = (1.3972 \pm 0.0045) \times 10^{-11}\ \text{a}^{-1}$, with a coverage factor $k = 2$.

3.2 ^{147}Sm

The TGIG [3] has reviewed papers covering two experimental approaches, β counting and geochronological intercomparison, and a first-principles quantum tunneling calculation of nuclear stability. The counting experiments followed several different experimental approaches: liquid scintillation counting, ionization chamber, silicon surface barrier, proportional counter, and CR-39 detector. As these techniques are very different, systematic inaccuracies affecting one of them do not necessarily affect all other ones. After a Type B evaluation with removal of biased experiments, the results of the remaining papers all obeyed a unimodal distribution with $\chi^2_n < 1$, such that a weighted average is legitimate.

The TGIG recommends a value of (106.25 ± 0.38) Ga for the half-life of ^{147}Sm, corresponding to a decay constant $\lambda(^{147}\text{Sm}) = (6.524 \pm 0.024) \times 10^{-12}\ \text{a}^{-1}$, with a coverage factor $k = 2$.

3.3 ^{146}Sm

Samarium-146 has $t_{1/2}(^{146}\text{Sm}) \leq 0.12$ Ga and is extinct in the Solar System, so that it must be synthesized before its decay rate can be counted. The synthesis pathway is traceable in principle, but the only four publications reporting ^{146}Sm half-life measurements do not always provide all the relevant information. While limiting the descriptions of experimental details was commonplace in printed journals until the age of Electronic Supplements, it makes the evaluation of legacy papers nearly impossible. The most problematic result is that the distribution of estimated half-life values is bimodal. Two counting experiments give compatible results in the coverage interval between 57 and 81 Ma, whereas two counting experiments agree in a coverage interval between 93 and 112.2 Ma. Both coverage intervals are given with a coverage factor $k = 2$. All papers state that they have estimated and taken into account all possible biases, but this obviously cannot be true for both contrasting modes of the bimodal distribution of half-life estimates.

A number of studies proposed to evaluate the ^{146}Sm half-life by early Solar System cosmo- and geochronological intercomparisons. A difficulty with such intercomparisons is that they all depend on very extensive assumptions on the ideality of single-stage evolution of the analyzed systems and on the uniform incorporation of one uniform ^{146}Sm/^{147}Sm ratio in the entire Solar System, none of which is amenable to a metrological tracing: there is neither a reference material for an extraterrestrial object with a single-stage point-like history nor for its ^{146}Sm mass fraction, and therefore, no propagation of uncertainty down the traceability chain. No extraterrestrial samples were collected consciously in a geologically controlled context. Mass spectrometric analyses *per se* are traceable to standard reference materials (as are the response functions of the gamma counters of the experiments reviewed by Villa *et al.* [3], Table 3), but assumptions regarding the "point-like" history of early Solar System samples are not (just as the missing experimental details of the counting experiments above are not). Traceability ensures that the measurement is accurate and that any other laboratory, when repeating the measurement under the same conditions, will reproduce the same number within the stated uncertainty. What traceability to an agreed reference material cannot guarantee is that the points in an isochron diagram pertain to a sample in petrologic equilibrium or can it guarantee that the isochron reflects the sample's single-stage formation age.

At this time, the inconsistencies evidenced by Villa *et al.* ([3], Table 3) cannot be pinpointed with certainty to a specific, documented systematic error. In the face of this dilemma, the Task Group must refrain from making a recommendation that satisfies both camps until new, well-documented, and fully metrologically traceable experiments will be performed.

3.4 ^{238}U

The TGIG [2] has addressed the uncertainty budget ([4], entry 2.33) of the ^{238}U half-life and recommends a half-life of (4.4683 ± 0.0096) Ga, *i.e.* a decay constant $\lambda(^{238}\mathrm{U}) = (1.55125 \pm 0.00333) \times 10^{-10}\ \mathrm{a}^{-1}$, both with a coverage factor $k = 2$. It should be pointed out that the counting experiments by Jaffey *et al.* [7] have never been reproduced with similarly high enrichments of ^{238}U and stand out due to their very high precision. As discussed by Villa *et al.* [2], the total uncertainty originally stated by Jaffey *et al.* [7] has been doubled as a conservative estimate, so as to include the systematic uncertainty components suspected by Jaffey *et al.* [7] in the original measurements. The U–Pb ages calculated using the half-life in [7] are conventionally defined as the reference age by the geochronological community [6].

3.5 ^{235}U

The measurement by Jaffey *et al.* [7] of the ^{235}U half-life by γ counting estimated the half-life of ^{235}U as (703.81 ± 1.92) Ma. The combined measurement uncertainty with a coverage factor $k = 2$ of 0.28 % was given by Villa *et al.* [2] after a Type B evaluation. As the precision of mass spectrometric analyses improved, the geochronological community pursued a different strategy, namely, constraining λ_{235} from λ_{238} by geochronological intercomparison of suitable samples ([2], and references therein). The criteria to decide whether a natural sample is fit for intended use as a calibrator ([4], entries 5.12 and 5.13) are not amenable to a metrological assessment and, in fact, have changed over time as geological and petrological knowledge has matured. Past literature often endorsed geochronological sample intercomparisons that are now considered problematic following [6].

Among the potential bias factors are: (1) the isotopic composition of U is not constant in terrestrial minerals; (2) the response function of electron multipliers in high-sensitivity mass spectrometers is not linear; (3) the instrumental mass fractionation in mass spectrometers may systematically discriminate even-mass isotopes from odd-mass isotopes; (4) the instrumental mass fractionation in mass spectrometers deviates from a first-order fit using an exponential law. These factors (as discussed in detail by Villa *et al.* [2]) amount to few parts in 10^4; therefore, they have only been detected and described in the last decade and were undocumented in legacy measurements prior to *ca.* 2010. The Type B evaluation on the presently available data must include the highest possible bias. It yields a combined measurement uncertainty of geochronological intercomparisons that may be no smaller than the [7] combined measurement uncertainties. This discourages a modification of the [7] half-life estimate (with the additional uncertainty derived from potential bias, conservatively proposed by Jaffey *et al.* [7], as discussed in [2]) until all bias factors will have been documented to be about an order of magnitude smaller than the targeted combined uncertainty.

3.6 ^{234}U

Uranium-234 is a comparatively short-lived nuclide, whose presence on Earth is not a residuum of the galactic nucleosynthesis (as are ^{235}U and ^{238}U); it is constantly supplied by the decay of ^{238}U. Its half-life determination by counting is hampered by the extreme difficulty of obtaining a pure ^{234}U sample, which would require a complete (*i.e.*, >99.999 %) separation from ^{238}U, which is four orders of magnitude more abundant. As a result, half-life determinations have only relied on measuring its activity ratio to ^{238}U in selected mineral geochronometers. The selection criterion is the so-called secular equilibrium, *i.e.* the attainment of the asymptotic steady state in which as many ^{234}U atoms decay as are replenished by the decay of ^{238}U.

This selection criterion may have been only incompletely followed in the papers evaluated by Villa *et al.* [2, p. 390]. An indication in this sense is suggested by a different set of (apparently unrelated) experiments endeavoring to estimate the ^{235}U half-life based on geochronological intercomparisons, which made use of old zircon samples that were argued to be undisturbed ever since their crystallization (*cf.* [2, p. 388]) and had,

therefore, achieved secular equilibrium. In the ^{235}U endeavor, the ^{238}U–^{206}Pb and ^{235}U–^{207}Pb ages were required to be ideal and equal. The requirement of undisturbed U–Pb ages also implies that the metrologically traceable measured U isotopic composition was undisturbed, in particular, that the $N(^{234}\mathrm{U})/N(^{238}\mathrm{U})$ number ratio had reached and maintained secular equilibrium. In this instance, the $N(^{234}\mathrm{U})/N(^{238}\mathrm{U})$ number ratio (given as an aside information) was based on metrologically traceable calibrating solutions. However, in the ^{234}U half-life experiments evaluated by Villa *et al.* [2], there is a mismatch (*ca.* 0.17 %) between the latter number ratio and that measured in natural carbonate samples. The attainment of secular equilibrium by the natural carbonate samples is not metrologically traceable and therefore may not reflect a "point-like" geological history. Until the sample characterization will include more thorough tests of petrologic equilibrium, the Type B evaluation must include the complete variation range of the $N(^{234}\mathrm{U})/N(^{238}\mathrm{U})$ number ratio. This results in a provisional coverage interval ($k = 2$) for the ^{234}U half-life of 244.55 to 245.77 ka, corresponding to a coverage interval for the decay constant $\lambda(^{234}\mathrm{U}) = [2.8203, 2.8344]\ \mathrm{Ma}^{-1}$.

4 Future perspectives

The present Technical Report reviews the data available at this time and points out methodological innovations and changes in perspective that have been implemented in the past decade. The implications for future work (covering all aspects addressed here: the nuclear physics measurements and calculations, the choice of ideal geological calibrators, and the evaluation criteria) lie in the necessity to pursue the ongoing efforts to document metrological traceability at all stages of the experimental work. The rigorous treatment of measurement uncertainties during mass spectrometric analyses of small (single-grain or sub-grain) samples is of essential importance. Future work will need to improve the recognition and the understanding of systematic analytical effects that had been considered of minor importance in past decades, as well as their accurate correction. Of equal importance is the recognition and acceptance of the fact that metrological traceability can only apply to measurements of entirely artificial substances (*e.g.* standard solutions), while the choice of a natural material that is fit for intended use as a calibrator must rely on human expertise capable of evaluating the geological/petrological context in its entirety.

5 Membership of sponsoring bodies

The membership of the Inorganic Chemistry Division (Division II) for the period 2020–2021 was as follows:

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