



National Institute of Standards & Technology

Report of Investigation

Reference Material[®] 8599

Henderson Molybdenite

This Reference Material (RM) is intended for use in standardizing ¹⁸⁷Re-¹⁸⁷Os absolute age measurements on molybdenite (MoS₂) ores. RM 8599 is supplied in a glass bottle containing approximately 10 g of MoS₂ powder.

Reference Age Value: A reference age for the Henderson Molybdenite is provided in Table 1. A reference value [1] is a noncertified value that is the best estimate of the true value; however, the value does not meet NIST criteria for certification and is provided with associated uncertainties that may reflect only measurement precision and may not include all sources of uncertainty.

Information Mass Fraction Values: The information values for Re and ¹⁸⁷Os provided in Table 2 are noncertified values with no reported uncertainties. An information value [1] is considered to be a value that will be of interest to the RM user, but insufficient information is available to assess the uncertainty associated with the value.

Expiration of Value Assignment: RM 8599 is valid, within the measurement uncertainties specified, until **31 December 2020**, provided the RM is handled and stored in accordance with the instructions given in this Report of Investigation (see "Instruction for Handling, Storage, and Use"). This report is invalid if the RM is damaged, contaminated, or otherwise modified.

Maintenance of RM Value Assignment: NIST will monitor this RM over the period of its validity. If substantive technical changes occur that affect the value assignment before expiration of this report, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

This Report of Investigation was prepared by R.D. Vocke, Jr. of the NIST Analytical Chemistry Division.

Technical aspects of the preparation, analysis, and distribution of this RM were coordinated through the Applied Isotope Research (AIRIE) Program at Colorado State University (Fort Collins, CO, USA) by H.J. Stein, R. Markey, J.L. Hannah, J.W. Morgan, and A. Zimmerman and the Radiogenic Isotope Facility (RIF) at the University of Alberta, (Edmonton, Alberta, Canada) by R.A. Creaser and D. Selby and through the NIST Analytical Chemistry Division by R.D. Vocke, Jr.

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Revision History on Last Page

INSTRUCTIONS FOR HANDLING, STORAGE, AND USE⁽¹⁾

Storage and Handling: Store under normal laboratory conditions. The molybdenite powder should be kept capped in its original glass container in a dry environment when not being used.

Preparation and Analysis: The molybdenite in this RM was taken from the Henderson mill in the Front Range of the Rocky Mountains, Colorado. The Henderson ore body is a Climax-type stockwork porphyry molybdenum deposit of Oligocene age associated with a granitic intrusive complex. This molybdenite deposit was chosen because the ore was milled on site and only Henderson ore has ever been processed in that mill. The molybdenite ore is nearly mono-mineralic (hexagonal polytype 2H₁) and emplacement of the ore body is thought to have taken place over a geologically short period of time [2,3]. The deposit is young and contains relatively low concentrations of Re and therefore modest amounts of radiogenic ¹⁸⁷Os. The bulk sample of milled molybdenite has a particle size of ≈46 μm (≈300 mesh). After cleaning and purification, the sample was mixed, split, and bottled. Additional geological details are provided in [4].

The reference value listed in Table 1 was obtained by combining data from 47 independent isotope dilution measurements utilizing two different mixed Re-Os spikes at two expert labs using solid-source negative thermal ionization mass spectrometers. Details of the experimental procedures and results are published in [4]. No evidence of isotopic heterogeneity was found and the expanded uncertainty for the age can be treated as reflecting random measurement error as well as the random variation between labs and methods.

Table 1. Reference Age Value for RM 8599

Age (Ma) ^(a)	U (Ma) ^(b)	Data Source
27.66	± 0.10	[4]

^(a) The ¹⁸⁷Re decay constant (λ ¹⁸⁷Re) used for the age calculation is $1.666 \times 10^{-11} \text{ a}^{-1}$ [5].

^(b) The expanded uncertainty for the age, computed as $U = ku_c$, is $k = 3.18$ times the standard uncertainty of the mean, u_c , of the means of the reported values in [4], treated as four independent draws from a normal distribution by lab and method. The coverage factor, k , was obtained from the Student's t distribution with 3 degrees of freedom and a confidence level of 95 %.

The information values listed in Table 2 were obtained by combining data from 47 independent isotope dilution measurements utilizing two different mixed Re-Os spikes at two expert labs using solid-source negative thermal ionization mass spectrometers. Details of the experimental procedures and results are published in [4]. Statistically speaking, these data sets effectively have only two pieces of information concerning the uncertainty of the Re and ¹⁸⁷Os amounts due to between-lab variation. Because the action controlling the uncertainty is the variation between labs, the two different spikes used give information on the within-lab variation with changes in the spike, but that variation is inconsequential when compared with the between-lab differences. The two different spike methods become, in effect, additional replicates that confirm the within-lab variances but give no information concerning the between-lab issue. For this reason, the amount mass fractions for Re and ¹⁸⁷Os are presented as information values.

Table 2. Information Mass Fraction Values for RM 8599

Re (mg/kg)	¹⁸⁷ Os (μg/kg)
11.2	3.236

⁽¹⁾ Certain commercial equipment, instrumentation, or materials are identified in this report to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by National Institute of Standards and Technology nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

REFERENCES

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- [2] Seedorff, E.; Einaudi, M.T.; *Henderson porphyry molybdenum system, Colorado: I. Sequence and abundance of hydrothermal mineral assemblages, flow paths of evolving fluids, and evolutionary style*; *Econ. Geol.*, Vol. 99, pp. 3–37 (2004).
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- [5] Smoliar, M.I.; Walker, R.J.; Morgan, J.W.; *Re–Os isotope constraints on the age of Group IIA, IIIA, IVA, and IVB iron meteorites*; *Science*, Vol. 271, pp. 1099–1102 (1996).

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