

The isotopic composition and atomic weight of lanthanum

John R. de Laeter*, Nino Bukilic

Department of Applied Physics, Curtin University of Technology, G.P.O. Box U 1987, Perth, WA 6845, Australia

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Abstract

The isotopic composition of lanthanum has been measured with high precision using a thermal ionization mass spectrometer, equipped with a Daly collector, whose linearity was verified by measuring an isotopically certified reference material for potassium (NIST 985), whose isotopes span a wide range of isotope ratios. The abundance sensitivity of the mass spectrometer in the vicinity of the measured LaO^+ ion beams was examined to ensure the absence of tailing effects and interfering isotopes. The isotope fractionation of the lanthanum isotopes was estimated by reference to the isotopically certified reference material for lead (NIST 981) and the fractionation of oxygen isotopes determined from LaO^+ measurements. These procedures are essential because of the extremely low isotope abundance of ^{138}La . An accurate determination of the abundance of ^{138}La is required in order to calculate the atomic weight, and because it is the parent nuclide of two geochronometers, ^{138}La – ^{138}Ba and ^{138}La – ^{138}Ce . The magnitude of the rare odd–odd neutron-deficient isotope ^{138}La is a key nuclide in p-process nucleosynthetic calculations. The measured isotopic composition has been corrected for isotope fractionation to give $^{139}\text{La}/^{138}\text{La} = 1125 \pm 3$, which gives isotope abundances for ^{138}La of 0.000888 ± 0.000002 and ^{139}La of 0.999112 ± 0.000002 . The isotope abundances and relative atomic masses of the two isotopes give an atomic weight of La of 138.905461 ± 0.000003 .

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1. Introduction

Lanthanum has the lowest atomic number of all the rare earth elements, and consists of two isotopes ^{138}La and ^{139}La . Its name is derived from the Greek word “lanthanein” which means “to be hidden or to escape notice”, because it “hid” in Ce ore and was difficult to separate from that rare earth mineral. In addition to the then known La isotope of mass 139 [1], a new isotope of mass 138 was discovered in 1947 [2]. Thermal ionization mass spectrometry (TIMS) was used to measure the isotopic composition of La by means of LaO^+ ions emitted from a La_2O_3 sample deposited on a W filament. The possibility of isobaric interferences from ^{138}Ba and ^{138}Ce was monitored, but no evidence of these nuclides was observed. Measurements of the linearity of the detector system showed a linear response to within 0.2%, and the authors

estimated that the sum of all the systematic errors were less than 1% [2]. The isotope abundances of La were measured to be $(0.089 \pm 0.001)\%$ for ^{138}La and $(99.911 \pm 0.001)\%$ for ^{139}La , to give a $^{139}\text{La}/^{138}\text{La}$ ratio of 1123 ± 12 [2]. ^{138}La is a member of three adjacent isobaric elements— ^{138}Ba , ^{138}La and ^{138}Ce . According to Mattauch’s rule of nuclear stability, which forbids the existence of three stable isobars whose atomic numbers differ by unity [3], it was suspected that ^{138}La was radioactive, though Inghram et al. [2] were unable to confirm this. However, the weak radioactivity of ^{138}La was demonstrated in 1950 [4]. In fact, ^{138}La decays by electron capture to ^{138}Ba and by beta decay to ^{138}Ce with a half-life of 1.1×10^{11} years [5].

In 1956, a two stage mass spectrometer with high abundance sensitivity was used to search for possible naturally occurring isotopes of low abundance in a number of elements, including La [6]. A solution of La_2O_3 in dilute HCl was deposited on a Ta filament in the ion source, and La^+ and LaO^+ peaks were observed. The La isotope ratio had to

* Corresponding author. Tel.: +61 8 9266 3518; fax: +61 8 9266 2377.

E-mail address: J.DeLaeter@curtin.edu.au (J.R. de Laeter).

be corrected for an isobaric ^{138}Ba interference. The resulting $^{139}\text{La}/^{138}\text{La}$ ratio was 1120 ± 20 , but it is neither clear as to the level of uncertainty quoted, nor whether the linearity of the mass spectrometer was evaluated [6]. No evidence of any other naturally occurring isotopes of La was observed.

In the 1980s, two geochronological decay schemes, based on ^{138}La as the parent nuclide, were developed. The first geochronometer involves the decay of ^{138}La to ^{138}Ce . Using this decay scheme, the age of a gabbro from the Bushveld, in South Africa was measured to be 2390 ± 480 Ma using a partial disintegration constant of $2.58 \times 10^{-12} \text{ year}^{-1}$ [7]. It was found that Ba was present as an isobaric interference at mass 138, so that the Ce isotope ratios were measured as the oxide to avoid this potential interference. Since La and Ce are both rare earth elements, this geochronometer can be used in combination with the Sm–Nd geochronometer in isotope geochemistry. Constraints on the use of the La–Ce geochronometer include an accurate knowledge of the beta decay disintegration constant, the low abundance of ^{138}La , and its long half-life [7]. The age of two Archaean granites from the Yilgarn craton in Western Australia were determined by the Rb–Sr, Sm–Nd and the U–Pb techniques. The three decay schemes gave ages, which were in good agreement. The beta decay disintegration constant for ^{138}La was calculated by assuming the La–Ce age was identical with the mean age determined by the other three decay schemes to give a value of $(2.33 \pm 0.24) \times 10^{-12} \text{ year}^{-1}$ [8].

The second geochronometer involves the decay of ^{138}La to ^{138}Ba . Experimental constraints of this decay scheme are imposed by the low elemental abundance of the rare earth element La, the low isotope abundance of ^{138}La , and the relatively high abundance of ^{138}Ba . Despite these difficulties, minerals from the Amitsoq gneiss gave a metamorphic age of 2408 ± 24 Ma determined by the ^{138}La – ^{138}Ba isochron technique using a half-life for the electron capture decay constant of $4.4 \times 10^{-12} \text{ year}^{-1}$ [9]. This age is in good agreement with that from Sm–Nd systematics on the same gneiss, of 2410 ± 54 Ma [9].

The emergence of these two geochronometers led to the re-measurement of the isotopic composition of La [10]. Johnson–Matthey La_2O_3 reagent was mounted in a VG-54-38 double focussing TIMS equipped with a Faraday cup. The abundance sensitivity of the instrument was more than adequate for the experiment [10]. The $^{139}\text{LaO}^+$ ion beam was approximately 5×10^{-11} A, and the isotope fractionation was calculated by taking the sample to extinction. Cerium and Ba were monitored for possible isobaric interferences, but there were little interferences from these elements. Oxygen isotope ratios were determined using PrO^+ ions and these values were used to correct the measured LaO^+ ion beams. The $^{139}\text{La}/^{138}\text{La}$ ratio was determined to be 1108 ± 1 at the 2σ level [10]. In 1992, another determination of the isotopic composition of La was made in which the isotope fractionation of the raw LaO^+ ratios were estimated by normalising to the O isotopes, in order to detect small isotope shifts in this two isotope element [11]. A $^{139}\text{La}/^{138}\text{La}$ ratio of 1101 ± 1 at

the 2σ level was reported. The unusual nucleosynthetic origin of this odd–odd isotope makes it a good candidate to find evidence of early solar irradiation, and meteoritic material has been analysed to search for such proton or photon-induced effects, unfortunately without any positive results [12].

The “best” measurement of La from a single terrestrial source is given as $^{138}\text{La} = 0.0009017 \pm 0.0000005$ and $^{139}\text{La} = 0.9990983 \pm 0.0000005$ to give a $^{139}\text{La}/^{138}\text{La}$ ratio of 1108 ± 3 [13]. The “best” measurement for La bears the notation “N” in the Table of the Isotopic Compositions of the Elements, which implies that the selected measurement is neither a calibrated measurement, nor has it been demonstrated that linearity and isotope fractionation effects have been satisfactorily assessed [13]. The dilemma in assessing these mass spectrometric measurements is that the data fall into two distinct sets—an average $^{139}\text{La}/^{138}\text{La}$ ratio of 1122 ± 12 for [2,6], and an average of 1105 ± 1 for [10,11], with a “best” selected value of 1108 ± 3 [13].

Various nucleosynthetic scenarios have been proposed to account for the bulk p-process content of stellar nucleosynthesis, which produces the stable neutron-deficient nuclides heavier than Fe, and these mechanisms have recently been reviewed by Arnould and Coriely [14]. The odd–odd neutron-deficient heavy nuclide ^{138}La is among the rarest Solar System species but, in spite of its small abundance, ^{138}La is under-produced in all p-process calculations, so that an accurate knowledge of the isotope abundance of ^{138}La is required [14]. The under-production of ^{138}La in p-process calculations is due to the unfavourable balance between its main production by $^{139}\text{La}(\gamma, n)$ ^{138}La and its mass destruction by $^{138}\text{La}(\gamma, n)$ ^{137}La [14]. The present project has measured the isotopic composition of La using a VG 354 TIMS for which the linearity and abundance sensitivity have been confirmed, and the isotope fractionation estimated.

2. Experimental

2.1. Laboratory standard

A laboratory standard solution of La was prepared by dissolving spectroscopically pure $\text{La}_2(\text{CO}_3)_3$ (Johnson Matthey Chemicals Ltd., Laboratory Number G 51113) in ultrapure 1 M HCl. This solution had a concentration of $2000 \mu\text{g La/g}$ of solution, which enabled $10 \mu\text{g}$ of LaCl_3 to be loaded routinely for TIMS analysis on the two side filaments of a triple Re filament assembly. Large La metal and LaO^+ ion beams could be obtained using this ion source configuration. Unfortunately, small Ba ion beams were observed when La^+ ions were analysed, and despite attempts to remove this isobaric element, small traces were always present. A correction could be made for the ^{138}Ba in the measured mass 138 abundance using the measured $^{136,137}\text{Ba}$ ion beams, but in practice, the uncertainties introduced by this procedure mitigated against its use. Therefore the LaO^+ ion beams were used to measure the $^{139}\text{La}/^{138}\text{La}$ ratio where isobaric interferences were not detected. An adequate time

period had to be allowed to enable the Daly collector to recover after measuring the large mass 155 peak before measuring the mass 154 peak. Care also had to be taken to ensure that the baseline zero corrections were appropriate.

After gently outgassing the laboratory standard sample overnight in the VG 354 TIMS, the temperature of the centre filament was slowly increased to approximately 1100 °C at which temperature a $^{139}\text{LaO}^+$ ion beam of approximately 5×10^{-13} A was observed with only a small current through the two side filaments. The sample was evaporated from the two side filaments and ionized on the hot centre filament. The resulting ion beams were stable for hours of operation without appreciable decay. Before data collection was commenced, the mass region in the vicinity of the LaO^+ peaks was scanned at high sensitivity to ensure that no evidence of any interfering isotopes – particularly those of Ba or Ce – were observed. The presence of Ba was monitored by observing the $^{135,136,137}\text{Ba}^{16}\text{O}^+$ ion beams at $A = 151, 152$ and 153 , respectively. If there was any evidence of BaO^+ isotopes present, no data were collected. The effect of a $^{138}\text{Ba}^{16}\text{O}^+$ contamination on the $^{138}\text{La}^{16}\text{O}^+$ ion beam is to significantly reduce the $^{139}\text{La}/^{138}\text{La}$ isotope ratio, because ^{138}Ba is approximately 6.4 times larger than ^{137}Ba , and hence reduce the atomic weight. The isotope ratios for masses 154–157 were measured with respect to mass 154 using a Daly collector in the peak-jumping mode. A number of replicate analyses were measured.

2.2. Linearity

In order to measure the isotopic composition of La with high accuracy, it is essential to establish the linearity of the mass spectrometer detecting system over a wide range, since the magnitude of the ^{138}La and ^{139}La ion beams differ by a factor in excess of 1100 [10]. A NIST Isotopically Certified Reference Material (ICRM) 985 for K was obtained to check the linearity of the VG 354 TIMS used in this experiment. This ICRM comprises highly purified KCl whose absolute isotope abundance ratios have been measured on two mass spectrometers both of which were calibrated using synthetic mixtures prepared from nearly isotopically pure separated ^{39}K and ^{41}K isotopes [15]. The absolute isotope data for NIST 985 are listed in Table 1.

2.3. Abundance sensitivity

Another important consideration is to demonstrate that the mass spectrometer has sufficient mass resolution to

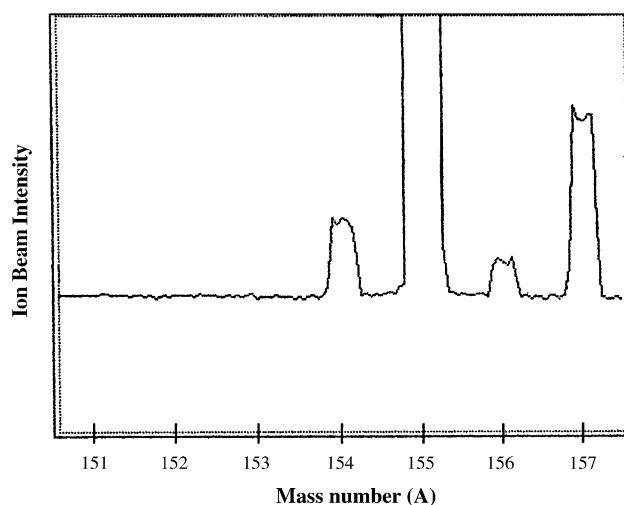


Fig. 1. The mass spectrum of LaO^+ generated by the VG 354 TIMS in the mass region $150.5 \leq A \leq 157.5$, showing the abundance sensitivity of the mass spectrometer in this mass region. The $^{138}\text{La}^{16}\text{O}^+$ ion beam at $A = 154$ is over 1100 times smaller than the ($^{139}\text{La}^{16}\text{O}^+ + ^{138}\text{La}^{17}\text{O}^+$) beam at $A = 155$. The mass peaks at $A = 156$ and 157 are primarily due to $^{139}\text{La}^{17}\text{O}$ and $^{139}\text{La}^{18}\text{O}$, respectively. The mass spectrum shows no evidence of interfering ion beams.

ensure that two adjacent ion beams can be measured without “tailing” effects. This is especially important in situations where the magnitude of the isotope ratio is large and the high abundance ion beam has the higher mass number A . This is the situation when measuring the isotopic composition of La. The abundance sensitivity is also dependent on the vacuum conditions in the mass spectrometer at the time of data collection. It is imperative that the ion source and mass analyser vacuum conditions are carefully monitored. The pressures in the ion source and mass analyser at the time of data collection were 5×10^{-8} Torr and less than 10^{-9} Torr, respectively. Fig. 1 is a mass scan from $150.5 \leq A \leq 157.5$ at the filament current conditions at which the LaO^+ ion beams were measured. The scan demonstrates that the abundance sensitivity for the LaO^+ beams is sufficient to enable the tailing of the peak at m/z 139 to be ignored, and that there is no evidence of BaO or CeO molecular ions.

2.4. Isotope fractionation

A TIMS isotope analysis invariably introduces a time-dependent isotope fractionation into the raw measurements, with the ion beams of the lighter isotopes being enhanced with respect to the heavier isotopes, at least in the early stages of the analysis. Thus, isotope ratios can exhibit variations both within runs and between successive mass spectrometric analyses of the same sample. An accurate assessment of the isotopic composition and the atomic weight of an element must therefore involve a correction for isotope fractionation, which will have the effect of decreasing the $^{138}\text{La}^{16}\text{O}/(^{139}\text{La}^{16}\text{O} + ^{138}\text{La}^{17}\text{O})$ ratio, and thereby slightly increasing the atomic weight. A number of techniques have

Table 1
Calibrated K absolute isotope abundance ratios for NIST 985, together with the isotope ratios measured in this project

	$^{39}\text{K}/^{41}\text{K}$	$^{40}\text{K}/^{41}\text{K}$
Calibrated NIST ratios	13.8566 ± 0.0063	0.0017343 ± 0.0000061
Measured ratios	14.1016 ± 0.0012	0.0017449 ± 0.000005
Corrected ratios	13.8566	0.0017297 ± 0.000009

Uncertainties are at the 95% confidence level.

been adopted in an endeavour to assess the magnitude of isotope fractionation. If an element possesses four or more isotopes, the double spike technique can be used to identify chemical or instrumentally induced mass fractionation, or to use the internal normalisation technique to normalise one ratio to a “standard” value and adjust the other ratios accordingly with elements with three or more isotopes.

However, in the case of elements with only two isotopes these techniques are not applicable. It is possible to take the sample to extinction and integrate the magnitude of the two ion beams over this period of time, or decrease the effect of isotope fractionation by measuring heavier isotopes such as the LaO^+ ions rather than the metallic species. The procedure of calculating the square root of the masses of the two isotopes as an estimate of the isotope fractionation can be adopted, or one can gauge its value by examining the isotope fractionation of an ICRM in the same mass vicinity. In the case of La, a convenient ICRM is NIST 981 for Pb—the calibrated isotope ratios of which are given by Catanzaro et al. [16]. The VG 354 TIMS used in this experiment is routinely used to analyse NIST 981 as part of an ongoing project on Pb in the environment, and the isotope fractionation is $0.24 \pm 0.06\%$ per mass unit for the Daly collector [17]. It must be pointed out that the magnitude of the isotope fractionation of Pb may not be identical to that of La, but it is nevertheless a reasonable approximation, and is in good agreement with the square root of the mass correction factor, provided the Pb fractionation value is adjusted to take into account the mass of the Pb isotopes as compared to the mass of the LaO isotopes. An alternative approach, which is applicable in the case of La, is to calculate the isotope fractionation of the mean of the $^{18}\text{O}/^{16}\text{O}$ ratio from the measured $^{139}\text{La}^{18}\text{O}/^{139}\text{La}^{16}\text{O}$ ratios, compare this with the presently accepted value of $^{18}\text{O}/^{16}\text{O}$ of 0.002055 [13], and use this correction factor to account for the isotope fractionation in the La isotopes [11].

3. Results and discussion

A sample of NIST 985 was dissolved in ultrapure 1 M HCl and loaded in identical fashion as for the La laboratory standard. The measured isotope ratios are given in Table 1. The isotope fractionation in the measured value of $^{39}\text{K}/^{41}\text{K}$ has been evaluated with respect to the calibrated value of 13.8566 to give a deviation of 0.884% per mass unit. This isotope fractionation has been applied to the measured $^{40}\text{K}/^{41}\text{K}$ ratio to give a value of 0.0017297, which is in agreement with the calibrated value within experimental uncertainties. The assumption has been made that the measured $^{39}\text{K}/^{41}\text{K}$ value is unaffected by non-linearity effects since the magnitude of the isotope ratio is 13.8566, and the VG 354 mass spectrometer is routinely used to measure NIST ICRMs for Sr and Pb with similar isotope ratio magnitudes, without difficulty.

A more stringent linearity test is to compare the $^{40}\text{K}/^{39}\text{K}$ ratio of NIST 985 (0.00012516) to the corresponding

measured value (0.00012374). If we apply the same isotope fractionation value of 0.884% to the measured $^{40}\text{K}/^{39}\text{K}$ ratio we derive a value of 0.00012483, which is again consistent with the NIST 985 value within experimental uncertainties. Expressed in the inverse format, the $^{39}\text{K}/^{40}\text{K}$ value from NBS/NIST 985 is 7990 as compared to the measured, but isotope fractionation-corrected value of 8011. Thus, the detecting system of the VG 354 mass spectrometer used in this experiment is linear over nearly four orders of magnitude, and confirms that the $^{139}\text{La}/^{138}\text{La}$ ratio of approximately 1100 can be measured with confidence, at least as far as linearity is concerned.

In order to correct the LaO^+ ratios for O, the isotope ratios of PrO^+ were measured under identical mass spectrometric conditions, as were the LaO^+ ratios. Praseodymium is a mono-isotopic rare earth element of mass 141, and is therefore close to La on the Periodic Table, so that it is an ideal element to use to measure the relevant O ratios. A spectroscopically pure sample of Pr was taken into solution in ultrapure 1 M HCl and loaded in TIMS in an identical manner to La. The measured PrO^+ ratios could be used to correct the LaO^+ for O. In point of fact, the O corrections are negligible in terms of the other experimental uncertainties. Each replicate analysis comprised at least ten blocks of 10 isotope ratios, and the means of these $^{139}\text{La}/^{138}\text{La}$ ratios are given in Table 2 together with the mean of the nine replicate analyses and the mean isotope fractionation-corrected value. The mean of the nine replicate analyses of the $^{139}\text{La}/^{138}\text{La}$ ratios was corrected for isotope fractionation as discussed above. The square root of the mass correction factor gave an isotope-corrected value of 1125; the Pb NIST 981 correction factor 1125; and the $^{18}\text{O}/^{16}\text{O}$ correction factor 1125. The ^{138}La and ^{139}La isotope abundances derived from this corrected $^{139}\text{La}/^{138}\text{La}$ isotope ratio of 1125 ± 3 are given in Table 3 together with the corresponding isotope abundance data by previous authors.

If the relative atomic masses of Audi and Wapstra [18] are used together with the corrected isotope abundances of ^{138}La and ^{139}La as listed in Table 3, an atomic weight of 138.905461 ± 0.000003 can be calculated. This result is in

Table 2
The measured isotope ratios $^{139}\text{La}/^{138}\text{La}$ for a number of replicate analyses of La in the VG 354 TIMS where the uncertainties are at the 2σ level

Sample	Date of analysis	$^{139}\text{La}/^{138}\text{La}$ ratio
1	21/12/04	1117 ± 6
2	21/12/04	1121 ± 8
3	21/12/04	1123 ± 10
4	02/02/05	1118 ± 6
5	02/02/05	1120 ± 4
6	09/02/05	1122 ± 6
7	09/02/05	1125 ± 6
8	09/02/05	1120 ± 6
9	16/02/05	1121 ± 2
Mean (raw value)		1121 ± 2
Mean (fractionation-corrected)		1125 ± 3

The uncertainties of the mean values are at the 95% confidence level.

Table 3

Comparison of the isotope abundances and atomic weights of La in this work as compared to other authors

^{138}La (%)	^{139}La (%)	Atomic weight	References
0	100	138.906349	[1]
0.089 ± 0.001	99.911 ± 0.001	138.90546 ± 0.00001	[2]
0.089 ± 0.0015	99.911 ± 0.0015	138.90546 ± 0.00002	[6]
0.09016 ± 0.00005	99.9098 ± 0.00005	138.905393 ± 0.000001	[10]
0.09074 ± 0.00006	99.90926 ± 0.00008	138.905556 ± 0.0000012	[11,12]
0.0888 ± 0.0002	99.9112 ± 0.0002	138.905461 ± 0.000003	This work

The uncertainties associated with the values of other authors are as stated in their respective papers, whereas the uncertainties associated with our work are at the 95% confidence level. Relative atomic masses of 137.907108 and 138.906349 for ^{138}La and ^{139}La , respectively, have been used to calculate the atomic weights.

excellent agreement with the presently accepted standard atomic weight of La of 138.9055 ± 0.0002 [19]. Unfortunately, this new determination of the isotope abundance of ^{138}La does little to assist in resolving the disparity in the under-production of ^{138}La by p-process nucleosynthesis [14]. Also listed in Table 3 are the atomic weights of La calculated from the listed isotope abundance data, using the relative atomic masses of Audi and Wapstra [18].

The Commission of Isotopic Abundances and Atomic Weights (CIAAW) of the International Union of Pure and Applied Chemistry is responsible for the production of Standard Atomic Weight Tables. In its 1961 report [20], CIAAW recommended a value of $A_r(\text{La}) = 138.91$, based on two chemical determinations and the average of two mass spectrometric determinations [2,6]. In 1969, CIAAW [21] recommended $A_r(\text{La}) = 138.9055 \pm 0.0003$, based on the same experimental data, but taking into account the small influence of any isotope fractionation on the atomic weight of this nearly mono-isotopic element. In 1985, CIAAW decided to reduce the uncertainty of $A_r(\text{La})$ to 138.9055 ± 0.0002 [22]. This value is the presently accepted value of the standard atomic weight [19].

4. Conclusions

The isotopic composition of La has been determined by a VG 354 TIMS whose linearity has been demonstrated over nearly four orders of magnitude using the K ICRM NIST 985, together with good abundance sensitivity in the mass region $154 \leq A \leq 157$. No evidence of isotope interferences in the mass region of La was observed, particularly from Ba isotopes. The magnitude of the isotope fractionation of the La isotope ratios was estimated from the isotope fractionation of the measured O isotopes, by comparison with the Pb ICRM (NIST 981), and from the square root of the mass correction factor. The uncertainty of the isotopic composition of La has taken into account the uncertainty of the magnitude of this correction. Replicate analyses of the La laboratory standard give an isotope fractionation-corrected $^{139}\text{La}/^{138}\text{La}$ ratio of 1125 ± 3 with the isotope abundances of ^{138}La and ^{139}La being $(0.0888 \pm 0.0002)\%$ and $(99.9112 \pm 0.0002)\%$, respectively. Although the exact magnitude of the isotope

fractionation cannot be precisely determined, it has little effect on the atomic weight of La because of the disparity in the abundances of the two isotopes. The atomic weight of La has been calculated to be 138.905461 ± 0.000003 , which is in good agreement with the currently accepted atomic weight $A_r(\text{La}) = 138.9055 \pm 0.0002$. The early work of Inghram et al. [2] is in excellent agreement with the present work, and the detailed analysis of their experimental uncertainties is a model for mass spectrometrists to follow.

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