tion as well as errors in the differential cross section curve for the D(d,n)He³ reaction.⁷ The range of the measurements given here indicated by the solid line extend only over the region in which the D(d,n)He³ reaction has been studied directly by counting He³ particles.⁷ The point at 14 Mev is an independent determination by D. D. Phillips and R. W. Davis.¹⁷ This was obtained with a known flux of 14-Mev neutrons and by use of the same method of calibration of Geiger counters as is described in this report. The dotted portion of the curve in Fig. 2 is an extrapolation between this 14-Mev point and the part of the curve obtained at lower energies. The ordinate scale on the right in Fig. 2 which gives the cross section of the $Cu^{63}(n,2n)Cu^{62}$ reaction is based on the reported value of 0.56 barn for the production of Cu⁶⁶ in normal copper. The uncertainty of this scale is ± 20 percent and is the reported uncertainty of the capture cross section.¹⁴

A linear extrapolation of the points in Fig. 2 gives an apparent threshold of 11.7 ± 0.3 Mev for the Cu⁶³(n, 2n)-Cu⁶² reaction, where the error in the energy is estimated from the uncertainty of the energy scale and the uncertainty of the extrapolation of the points. In a number of runs in which only relative values of activation were measured, independent determination of this threshold in this manner gave an average value of 11.8 ± 0.2 Mev. Within the limits of error, this is in agreement with previously reported determinations of the threshold of about 12 Mev.^{1, 6, 18}

One expects the extrapolation discussed above not to be valid in the case of the (n,2n) reaction. Consideration of the volumes in phase space available for the products of the (n,2n) reactions suggests that the cross sections of these reactions should go to zero as $(E-E_0)^2$, where E_0 is the threshold. The curve in Fig. 2 through the lower energy points is an $(E-E_0)^2$ extrapolation, which gives a threshold of 11.4 ± 0.3 Mev. Correcting this value for the recoil energy of the compound nucleus, one obtains 11.2 ± 0.3 Mev as a neutron binding energy. This agrees within the limits of error with the threshold of the $Cu^{63}(\gamma,n)Cu^{62}$ reaction which has been determined to be 10.9 ± 0.3 Mev by betatron activation.¹⁹

The authors wish to thank the members of the Los Alamos cyclotron group for their aid in this experiment. We wish to acknowledge the suggestion of Dr. J. H. Manley with regard to calibration of the Geiger counters. We are indebted to Dr. Edward Teller for the very helpful discussion of the behavior of cross sections near the threshold. We wish also to thank Dr. R. F. Taschek for his helpful criticism in preparing this report.

¹⁸ R. Sagane, Phys. Rev. **53**, 492 (1938).
 ¹⁹ G. C. Baldwin and H. W. Koch, Phys. Rev. **67**, 1 (1945).

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A Redetermination of the Relative Abundances of the Isotopes of Carbon, Nitrogen, Oxygen, Argon, and Potassium*

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Essentially pure samples of A³⁶ and A⁴⁰ have been produced by thermal diffusion and used for the preparation of synthetic argon isotope mixtures whose A³⁶/A⁴⁰ isotope abundance ratios were very accurately determined. The mixtures were then employed for determining the mass discriminating effects in two mass spectrometers. Carbon, nitrogen, oxygen, argon, and potassium were investigated and new values given for the relative abundances of the isotopes. With atmospheric oxygen as a standard, the conversion factor between the physical and chemical atomic weight scales is 1.0002783 ± 5 . The percentage abundance of K⁴⁰ in potassium is found to be 0.0119 ± 0.0001 percent, a figure of special interest in geophysical problems, and approximately ten percent higher than the present accepted value.

HE general need for more precise information on nuclear constants suggests that a redetermination of isotope abundances in many of the elements may be of value. The existence of nearly pure samples of separated isotopes makes possible the preparation of artificial mixtures of isotopes whose relative abundances may be computed to a high degree of accuracy. The use of such standard isotopic mixtures together with mass spectrometers of modern design makes possible results in which both the accidental and systematic errors are reduced below those previously attainable. The present paper represents the beginning of a study of those elements where it appears redeterminations are of value and can profitably be made.

APPARATUS

Two mass spectrometers of the 60° type were used in the present investigation. One of these has already been described in detail.¹ During the past two years numerous minor modifications have been made in this instrument. The only one of interest here concerns the ion source. Plate J_3 has been eliminated, the space

¹ A. O. Nier, Rev. Sci. Inst. 18, 398 (1947).

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FIG. 1. Mixing bulb for making argon isotope mixtures. G_1 and G_3 , standard tapers 10/30; G_2 , 14/20; S_1 3-mm bore stopcock, S_2 and S_3 2-mm bore stopcocks. The bulb is made in two pieces for ease in measuring the volumes. Both parts were measured by weighing, water being employed for the larger, mercury for the smaller. Corrections for temperature and for buoyancy of the atmosphere were made. V_1 (measured between S_1 and S_2) was 1012.7 cc for Samples A I and A IV and 1013.5 cc for Samples A V and A VI. V_2 (including bore of S_3) was 9.9982 for A I and 11.6991 for A IV, A V, and A VI.

between S and J_1 , J_2 increased to 2 mm and that between J_1 , J_2 , and G, decreased to 2 mm. In the present work the instrument was used with the single collector No. 2. Mass spectra were obtained by varying the voltage on the left grid of V_4 (reference 1, Fig. 6) with a motor-driven potentiometer. Records were taken on a Brown Electronik 4.5-second recording potentiometer. For purposes of identification, the mass spectrometer just discussed will be known as MS1.

The second instrument (to be called MS2) was that employed in the helium and argon work² of the past few years. In the present work the source employed was essentially the same as that used in MS1. The greatest difference was that it contained a small furnace very similar to one employed in some earlier work.³ This permits the study of substances not volatile at room temperature. A further difference was that the gas was led directly into the spectrometer housing rather than into the shield can as in MS1. Perhaps the most important difference between MS1 and MS2 was the fact that MS2 employed a molecular flow gas leak⁴ consisting of a hole 0.001 in. diameter in an aluminum sheet 0.001 in. thick. The reservoir behind this leak had a volume of 5000 cc. Thus in MS2 the gas composition in the ionization region should be the same as in the sample reservoir whereas in MS1 where a viscoustype leak is employed it should be distorted according to the square root of the ratio of the pair of masses

TABLE I. Composition of isotopically separated samples used for making standards.

Stand-	D	an a	Composition (%)					
ard No.	cm oil	°K	Gas	A ³⁶	A^{38}	A40	rities	
AI	63.40		A40	0.00	0.00	99.85	0.15	
	62.95		A^{36}	97.17	0.69	1.69	0.45	
A IV	65.83	299.90	A^{40}	0.00	0.00	99.71	0.29	
	65.83	300.16	A ³⁶	94.78	1.00	3.44	0.78	
A V	44.52	299.41	A^{40}	0.00	0.00	99.32	0.68	
	44.14	299.31	A ³⁶	95.21	1.00	3.46	0.33	
A VI	59.50	298.76	A40	0.00	0.00	99.80	0.20	
	59.50	298.66	A ³⁶	94.99	0.99	3.45	0.57	

² L. T. Aldrich and A. O. Nier, Phys. Rev. 74, 876 (1948); 74, ¹⁵⁹⁰ (1948). ³ A. O. Nier, Phys. Rev. 53, 282 (1938).

⁴ R. E. Honig, J. App. Phys. 16, 646 (1945).

TABLE II. A^{36}/A^{40} ratios ($\times 10^{5}$) for standards.

	Calc. from Table I	Measured Run 1	with MS Run 2	Calc./av. meas.	Recalculated adopted values
A I	953.8	953.7	957.1	0.9983	949 ± 1.9
A IV	1098.6	1108.6	1114.9	0.9882	1105 ± 2.4
A V	1097.0	$1103.2 \\ 1108.8$	1102.1	0.9949	1096 ± 1.5
A VI	1100.4		1106.9	0.9932	1101 ± 1.6
				Av. 0.9937 ± 0	0.0014

under consideration. These assumptions were made in computing the measured A³⁶/A⁴⁰ ratios in Tables III-VII before applying the correction for the "true" isotopic composition of the argon standards. The fact that the subsequent corrections were relatively small in all cases shows that not only did the instruments have small mass discriminating effects but moreover the assumption made for the gas composition in the instruments was very nearly correct if not completely

The electrometer tube grid resistors had a resistance of 10^{11} ohms in each case and as nearly as could be determined were completely ohmic in their performance. Since inverse feed-back amplifiers and self-balancing recording potentiometers were used for measuring and recording the ion currents, linearity problems do not occur in the measuring system.

Argon was chosen as the reference gas for all the measurements reported here. By means of thermal diffusion columns described elsewhere,⁵ some nearly pure A³⁶ and nearly pure A⁴⁰ were obtained. These were mixed in known proportions to give an accurately determined ratio A³⁶/A⁴⁰ near to 0.01. The gas so obtained was then used as a standard for calibrating the mass

TABLE III. Comparison of mass spectrometer readings for standard argon and argon from several sources.

A ³⁶ /A ⁴⁰ ×10 ⁵ A ³⁶ /A		40 ×106	A ³⁸ /A ⁴⁰ ×10 ⁶	
Stand. A V	Airco	Atmos.	Airco	Atmos.
1085	3290	3368	669	681
1090	3326	3341	634	695
1090 1090	3315	3356	627	699
Av. 1090 ± 1.2 Corr. 1096 ± 1.5	$\overline{ 3310.3 \pm 7.3 \atop 3328 \pm 9}$	$\overline{ 3355.0 \pm 5.3}_{3373 \pm 8}$	$\overline{\begin{smallmatrix} 643.3 \pm 8.8 \\ 645 \pm 9 \end{smallmatrix}}$	$\overline{ \begin{smallmatrix} 691.7 \pm 3.7 \\ 694 \pm 4 \end{smallmatrix} }$
Data taken with I Stand, A I	MS1			
937	3333	3361	626	633
939 943 942	3319	3342	625	631
940 940 940	3327	3355	630	
Av. 940.1 ± 0.6 Corr. 949 ± 1.9 Weighted grand	$\overline{3326.3\pm2.8}_{3358}\pm8$	$\overline{ 3352.7 \pm 3.8}_{3384 \pm 8}$	$\overline{ \begin{smallmatrix} 627.0 \pm 1.0 \\ 630 \pm 1 \end{smallmatrix} }$	$\overline{\begin{smallmatrix} 632.0 \pm 0.7 \\ 635 \pm 1 \end{smallmatrix}}$
mean of data				

* Data from MS2 discarded.

⁵ McInteer, Aldrich, and Nier, Phys. Rev. 74, 946 (1948).

Data taken with MS2 36/40×10 ^s (N Stand. A I	(N ¹⁴ N ¹⁵ /N ¹⁴ N ¹⁴)×10 ⁵ Atmos. N ₂		
939	739		
941 942 938 939	738 736		
Av. 939.8 ± 0.5 Corr. 949 ± 1.9	737.7 ± 0.5 735.3 ± 0.7		
Data taken with MS1 Stand. A I			
942 940	737		
940 943 944	725 729		
Av. 941.8 ± 0.5 Corr. 949 ± 1.9	729.7 ± 2.9 728 ± 3		
weighted grand mean of data taken with MS1 and MS2 Unweighted average of MS1 and	735 ±1		
MS2 data	$732 \pm 2^*$		

TABLE IV. Comparison of mass spectrometer readings for atmospheric nitrogen and standard argon.

* The large difference in the P.E. in the two instruments and an examination of the data suggest systematic errors may have been present. It seems safer to assume the data to be inconsistent and adopt the average value disregarding the P.E. in the data from the individual instruments.

spectrometers for systematic errors which depend upon the relative masses of the ions studied.

Figure 1 shows the bulb employed for making the argon isotope mixtures. The following procedure was employed in making gas mixtures: After evacuating the bulb, including the stopcock bores, V_1 was filled with A^{40} to a given pressure carefully measured on an oil manometer. V_2 was filled with A^{36} to approximately the same pressure (the exact value being carefully measured) through G_3 , the temperatures being carefully noted in both cases except in A I where they were assumed to be the same. Mixing is accomplished by attaching G_3

TABLE V. Comparison of mass spectrometer readings for standard argon and for oxygen from several sources.

Data taken with MS2		34/32×10 ⁵		33/32×106	
S	tand. A V	Tank	Air	Tank	Air
	1083 1090	415.3 415.7 411.4	417.5 409.6 407.9	754 754 760	761 757 764
	1068				
Λv. Corr.	$\begin{array}{r} \hline 1080.3 \pm 4.4 \\ 1096 \ \pm 1.5 \end{array}$	$\overline{\begin{array}{c} 414.1 \pm 1.8 \\ 410.7 \pm 2.0 \end{array}}$	411.7 ± 2.0 408.2 ± 2.2	756.0 ± 1.3 752.8 ± 1.6	760.7 ± 1.3 757.5 ± 1.6
Data St	taken with MS1 tand A I				
	942 943 945 946	411.3 412.3 411.1	410.7 410.1 409.4	757 755 757	742 743 742
Av. Corr. Weigl me	944.0 \pm 0.6 949 \pm 1.9 hted grand an of data	411.6 ± 0.3 410.3 ± 0.5	410.1 ± 0.2 408.8 ± 0.5	756.3 ± 0.5 755.1 ± 0.7	742.3 ± 0.3 741.1 ± 0.6
tak and	en with MS1 1 MS2	$410.3\pm\!0.5$	408.8 ± 0.5	755 ±1	749 ±5*

* Data from two instruments assumed inconsistent. Final result unweighted,

to a Toeppler pump. Since the bore of S_3 is included in the measurement of V_2 , no errors are caused by gases trapped in stopcocks.

Table I gives essential data for computing the composition of the standards made. The purity of the gases was measured with a mass spectrometer, known mixtures of pure argon and air being employed to calibrate the mass spectrometer for making the gas analyses. Since highly enriched A^{36} and A^{40} were employed, it was not necessary to know the isotopic compositions to an extremely high accuracy. Nevertheless, any systematic spectrometer errors which existed while measuring the original enriched samples were reduced to negligible proportions by subsequent calibration of the instrument with the standard samples.

The four standard samples were actually made at different times, three different persons being involved. Table II gives the results of the calculations based on the data in Table I. As a check on the reliability of the mixing and calibrating procedures all four of the samples were run on the mass spectrometer MS2 within a period of a few hours, two sets of ten ratios being obtained for each sample. The calculated-to-measured ratios were tabulated and averaged as noted. The difference between 1.0000 and 0.9937 is believed to be due to a mass discriminating effect in the spectrometer. It was gratifying to see the close agreement between the individual calc./meas. ratios. Deviations in these were attributed to errors in the data in Table I and accordingly the original calculated values were recalculated to give the recalculated adopted values used in all of the work which follows. Although all samples were weighted equally in the averaging, extreme care was exercised in preparing and measuring A V and A VI and these may be slightly more accurate than

TABLE VI. Comparison of mass spectrometer readings for standard argon and carbon dioxide from several sources.

Data	A ³⁶ /A ⁴⁰ ×10 ⁵	45/4	4×10 ⁵	46/44	×10 ⁵
	Stand. A I	C No. 2*	C No. 3**	C No. 2	C No. 3
	938	1190	1207	412	412
	938	1206	1196	416	414
	944	1194	1199	412	418
	941				
Av.	940.0 ± 1	1197 ± 3	1201 ± 2	413.3 ± 0.9	414.7 ± 1.2
Corr	$.949 \pm 1.9$	1194 ± 3	1198 ± 2	411.7 ± 1.0	413.1 ± 1.3
Data	taken with MS	51			
	939	1186	1193	410	418
	940	1197	1223	417	416
	942	1196	1194	414	417
	944				
Av	9412 ± 09	1103 ± 7.4	1203 ± 7	$\frac{1137+13}{1137+13}$	417.0+0.4
Corr	949 ± 19	1101 ± 3	1200 ± 7 1201 ± 7	412.3 ± 1.0	4155+06
Weig	tted grand ean of data	1	1201 1	112.0 111	110.0 - 0.0
an	d MS2	1192 + 2	1199 + 2	412 + 1	415 + 1

* Pre-Cambrian limestone, Champlain Valley, Adirondacks, New York. ** Jurassic limestone, Solenhofen, Bavaria. the others. All errors given here and elsewhere in this paper are probable errors unless otherwise noted.

RESULTS

Argon

The synthetic argon isotope mixtures were employed to determine the isotopic composition of naturally occurring argon. Two sources of gas were studied. One was atmospheric argon obtained by passing air over hot lithium metal until essentially all the oxygen and nitrogen were removed. The second consisted of spectroscopically pure argon purchased from the Air Reduction Sales Corporation. In making analyses the procedure was to run the standard followed by each of the unknowns repeating this process at least three times. In each series of runs an extra run was made at the end on the standard. Each of the numbers given is the average of ten determinations, scanning of the spectrum being first in one direction and then in the other to eliminate effect of drift in intensity.

Table III gives the results obtained together with their probable errors. It appears that Airco argon contains slightly less A³⁶ and A³⁸ than atmospheric argon. The data on A³⁸/A⁴⁰ taken on MS2 is discarded in the final computation. The machine was known to have a residual impurity mass 38 peak and although correction was made for it, one can never be certain that the size of a residual peak in an instrument does not change when a gas is introduced. The final adopted values for atmospheric argon correspond to percentage abundances of 99.600, 0.063, and 0.337 for A⁴⁰, A³⁸, and A³⁶, respectively. In earlier investigation the following values have been given for the percentage abundances: Vaughan, Williams, and Tate,⁶ 99.67, ..., 0.33; Nier,⁷ 99.633, 0.060, 0.307; Dibeler, Mohler, and Reese,⁸ 99.57, 0.08, 0.35.

If one assumes the masses of A³⁶ and A³⁸ to be approximately the same as that of A40, which has been given as 39.9751 ± 3.9 and uses a conversion factor of 1.000278 in going from the physical to the chemical scale, one computes an atomic weight of 39.9493 ± 3 where the probable error is due entirely to the uncertainty in the atomic mass determination. The present International value is given as 39.944.

Nitrogen

In this case, room air was admitted directly into the mass spectrometer, no attempt being made to remove the oxygen. Table IV gives the results obtained. The N^{14}/N^{15} ratio found here, 273 ± 1 , is to be compared with 274.5, computed from data of Urey and Murphy¹⁰ on

assumption $O^{16}/O^{18} = 500$, 265 ± 8 given by Vaughan, Williams, and Tate,⁶ and 265, given by Wahl, Huffman, and Hipple.¹¹ With an atomic mass of 14.00754 ± 2^{9} for N¹⁴, and the conversion factor 1.000278 for going from the physical to the chemical scale, one computes an atomic weight of 14.00730 ± 2 for nitrogen. This is to be compared with the international value of 14.008.

Oxygen

As in the case of nitrogen, room air was admitted to the mass spectrometer. No effort was made to remove the nitrogen. Tank oxygen was also investigated. The results are shown in Table V. The close agreement between the 34/32 ratios found in the instruments suggests that the slight difference between atmospheric and "tank" oxygen is real. It is interesting to note that Thode¹² reports a difference of almost exactly the same magnitude and direction. The values 0.004088 and 0.000749 for the $O^{16}/O^{18}/O^{16}O^{16}$ and $O^{16}O^{17}/O^{16}O^{16}$ abundance ratios, respectively, yield the following percentage abundances for atmospheric oxygen isotopes: O¹⁶, 99.759; O¹⁷, 0.0374; O¹⁸, 0.2039. The mean mass number is 16.004452 ± 7 and the conversion factor between the physical and chemical atomic weight scales (if atmospheric oxygen is used as a reference) is 1.0002783 ± 5 . Due to the fact that the O¹⁸/O¹⁶ oxygen isotope abundance ratio is known to vary in nature by some four percent,^{13, 14} care must be exercised in applying the factor given here. If the oxygen from limestone is considered, the factor given here should be nearly correct. On the other hand, if the oxygen from water or iron ores were employed, the O¹⁸ content may be almost four percent lower¹³ and the conversion factor would be only about 1.000268. This computation takes no account of possible variations in O¹⁷ as yet unstudied. It is clear, however, in this example, and others, that the accuracy of measurement has now reached the stage where it is meaningless to speak of precise atomic weights unless the source of material is clearly defined. Recent results of other investigations on the oxygen isotope abundances include, for O¹⁶/O¹⁸: Mecke and Childs,¹⁵ 630; Smythe¹⁶ for oxygen prepared from PbO₂, 503 ± 10 ; Bleakney and Hipple¹⁷ for over 100 samples, average is 500; Murphey¹⁸ for tank oxygen, 500 ± 15 ; for O18/O17: Mecke and Childs, 15 5; Murphey18 for tank oxygen, 4.9 ± 0.2 .

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All data taken with MS2	36/40×10 ⁵ Stand. A I	K ³⁹ /K ⁴¹	K41/K40
Collector slit 0.025 in.	966 949	13.39 13.40 13.50	592 587 579
Av. Corr.	958 ± 6 949 ± 1.9	13.43 ± 0.03 13.37 ± 0.06	586 ± 3 587 ± 4
Collector slit 0.015 in.	945 ± 4	13.50 13.59	570 572
Av. Corr.	945 ± 4 949 ± 1.9	13.55 ± 0.03 13.58 ± 0.04	571 ± 1 570 ± 1
assuming inconsistency	13.48 ± 0.07	578±6	

TABLE VII. Comparison of mass spectrometer readings for potassium (Sylvite, New Mexico) and standard argon.

Carbon

Two of the limestone samples studied earlier by Murphey and Nier¹⁹ were used in the present work. The results are given in Table VI. Due to the fact that the molecule C12O16O17 contributes to the mass 45 ion current, a correction must be made before the C^{13}/C^{12} abundance ratio can be found. The largest variation reported for the O¹⁸/O¹⁶ ratio in nature has been four percent.¹³ However, this ratio appears to be about the same in limestones and the atmosphere. Thus, it may be reasonable to arbitrarily assume that the O¹⁷ in the limestone studied here should not differ by as much as two percent from the value found for it in Table V. Thus, subtracting 0.00075 ± 1.5 from each of the 45/44ratios, one obtains $C^{13}/C^{12}=0.01117\pm 3$ and 0.01124 ± 3 for samples No. 2 and No. 3, respectively. The average percentage C¹³ abundance for the two samples is then 1.108 ± 4 . The close agreement between the present results and the earlier ones of Murphey and Nier19 indicates that any systematic errors present earlier, affecting the absolute answers, must not have exceeded one-half percent. With an atomic mass of 12.003856 ± 19 for C¹² ⁹ and a conversion factor of 1.000278 for going from the physical to the chemical scale, one computes an atomic weight of 12.01160 ± 5 for carbon from limestone. A change of one percent in the C^{13}/C^{12} ratio will change this value by 0.0001. Thus, the greatest uncertainty in the atomic weight of carbon can be attributed to the uncertainty in the isotope abundance ratio in the particular substance under investigation. The present International value is given as 12.010.

Table VI gives also the 46/44 ratio (this is just twice the O¹⁸/O¹⁶) for the CO₂ analyzed. Since the CO₂ was released from the carbonate by treatment with acid in water and conditions were not under control so far as oxygen equilibrium is concerned no particular significance should be attached to the exact value found for the 46/44 ratio beyond the fact that it is approximately correct.

Potassium

In this case a sample of potassium metal prepared from New Mexico Sylvite was introduced in the small furnace of instrument MS2. For calibration purposes argon was allowed to leak into the instrument through a molecular leak. Whereas 75-volt electrons were employed for producing ions in all of the other work reported here, it was found necessary to use 7.5-volt electrons in the potassium study in order to be certain that possible impurities such as A⁴⁰ did not contribute to the very rare K^{40} . The K^{39}/K^{41} ratio was found to be the same for 52.5- and 7.5-volt electrons. The calibration of the instrument with the standard argon sample was made with 52.5-volt electrons. Since the resolution of the K⁴⁰ peak was not quite complete with the 0.025-in. final collector slit used in the spectrometer, a second set of data were accumulated with a slit 0.015 in. wide. The agreement appears satisfactory and the final values adopted for the isotope ratios are shown at the bottom of Table VII. These values correspond to percentage abundances of 6.91±0.04, 0.0119±0.0001, and 93.08 ± 0.04 for K⁴¹, K⁴⁰, and K³⁹, respectively. The most significant thing about the present study is that K40 appears to be about ten percent more abundant than previously^{7, 20-22} believed to be the case. This change will alter the half-life of K40 correspondingly. The K³⁹/K⁴¹ ratio agrees reasonably well with the recent value 13.66 ± 0.1 given by White and Cameron.²³ Since these authors list in their paper all of the previous values given for this ratio, the reader is referred to their Table IV for details. Because of the uncertainty in the exact atomic masses of the isotopes an accurate atomic weight computation does not appear practical at this time.

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