

The Atomic Weights Commission and Isotopic Abundance Ratio Determinations

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Abundance Ratios and Atomic Weights and their Applications"
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The Atomic Weights Commission and Isotopic Abundance Ratio Determinations*

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Introduction

Values of the atomic weights of the chemical elements were traditionally determined by chemical means, relative to some standard reference value, e.g., hydrogen or oxygen. There were national committees on atomic weights in the late nineteenth century. The International Committee on Atomic Weights (ICAW) began at the beginning of the Twentieth Century. The ICAW evaluated the published measurements and recommended values every year for each chemical element. When the International Union of Pure and Applied Chemistry (IUPAC) began operation after the First World War, ICAW became part of IUPAC. It was one part of the Committee on Chemical Elements during the 1920s. When IUPAC became the International Union of Chemistry (IUC) in 1930, ICAW was separated from other parts of the Chemical Elements Committee¹.

The Rise of Isotopes

In the early years of radioactivity at the beginning of the Twentieth Century, many radioactive substances were being found with various atomic weight values. In 1911, Frederick Soddy used his displacement law for alpha particle decay and beta transitions to show the chemical identity² of meso-thorium (²²⁸Ra) and radium (²²⁶Ra). In 1913, he concluded that there were chemical elements with different radioactive properties and different atomic weights but with the same chemical properties and therefore occupying the same position in the Periodic Table of the Elements. He coined the word "isotope" (Greek for in the same place) to account for those radioactive species³.

In 1897, J.J. Thomson had discovered the electron⁴. In 1912, he studied the rare gas neon by sending electrons through neon gas, creating neon ions, which he then accelerated toward a photographic plate⁵. Using electric and magnetic fields operating at right angles to each other to deflect these ions, Thomson found darkening at two locations on the

* This paper is dedicated to Prof. Zhang Qinglian (past member of the International Commission on Atomic Weights) for his seventy years of measurements on isotopic abundance ratios and atomic weights, which aided the Commission in its biennial recommendations on atomic weights.

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plate, corresponding to positions for the ^{20}Ne ion and for the ^{22}Ne ion. Relative intensity of the darkening on the plate was 90% and 10%, respectively, for the two ion beams. This could account for neon's non-integral atomic weight value of 20.2. This was the first time that isotopes of a stable chemical element had been found, in contrast to isotopes of the previously known radioactive elements. Francis Aston, using a mass spectrograph (a variation of Thomson's instrument), began measuring the percentage of each element's isotopes, or the chemical element's isotopic composition. Aston's first compilation of these isotopic abundance values was published in his book on "Isotopes" in 1922⁶.

The binding energy of a nucleus is the difference between the combined masses of the nucleons in the nucleus and the mass of the nucleus itself. A mass spectrographic method for determining the atomic masses dates from Aston's observation⁷ of small divergences of atomic masses from integral values. This difference between the isotopic mass and the mass number was called the packing fraction and is related to the binding energy per nucleon in the nucleus. Measurement of various atomic masses indicated that the binding energy per nucleon varied as a mass number increased. A Table of packing fractions appeared in Aston's Book in 1933⁸.

The atomic masses were determined by the "doublet method"; the mass difference between two atomic or molecular ion fragments having the same mass number and the atomic mass is related to the packing fraction.

Atomic Weights by Physical Measurements

In the First Report⁹ of the Commission on Atomic Weights of the International Union of Chemistry (IUC) in 1931, mention was made of the efforts by Aston to determine chemical atomic weights by micro-photometric measurements of the intensities of the isotope lines in a mass spectrograph. Notice was taken of the close agreement of these results with the chemical results, which indicated that the mass spectrograph was capable of giving results of a high degree of accuracy.

In the 1933 report¹⁰, the Commission on Atomic Weights concluded that atomic weight values derived from mass spectrographic data was still seen to be inferior to the very best chemical methods. In the 1934 report¹¹, Aston stated that niobium (columbium) appeared to be mononuclidic. His mass spectrographic measurement gave an atomic weight value of 92.90. This value disagreed with the Commission's recommended value of 93.3. In the 1935 report¹², the Commission recommended a new atomic weight value of 92.91 for niobium from a new Honigschmidt chemical determination¹³, which had been performed to check on the discrepancy in niobium previously noted by Aston. This is the first time that a physical measurement had an impact on the Atomic Weights Table.

In the 1938 report¹⁴, the Commission changed the atomic weight value for hydrogen, based on the abundance measurements of Bainbridge and Jordan¹⁵, the value for helium, based on the measurements of Aston¹⁶ and on Bainbridge and Jordan¹⁵ and the value of

osmium, based on the measurements of Nier¹⁷ and of Aston¹⁸. These corresponded to the first official atomic weight values that were based on mass spectrometric data.

At the 1951 IUPAC meeting in New York, the Commission¹⁹ revised the following atomic weight values of the elements: aluminum (data of Mattauch and Ewald²⁰), silicon (data of Bainbridge and Nier²¹), phosphorus (data of Motz²²), potassium (data of Bainbridge and Nier²¹), scandium (data of Collins, Nier and Johnson²³) and krypton (data of Bainbridge and Nier²¹). Natural variations in the abundances of the sulfur isotopes led to the range being expressed as an uncertainty on its atomic weight. All of these changes were based on the determinations by physical methods.

At the 1953 IUPAC meeting in Stockholm, the Commission²⁴ changed the atomic weight values of ten elements including, carbon (data of Nier²⁵), gold (estimated from the packing fraction curve), iridium (data of Sampson and Bleakney²⁶), manganese (mass data of Collins, Nier and Johnson²⁷), ruthenium (data of Friedman and Irsa²⁸), sodium (data from nuclear reactions), tantalum (estimated from the packing fraction curve), terbium (estimated from the packing fraction curve), thorium (mass data of Stanford, Duckworth, Hogg and Geiger²⁹), and thulium (estimated from the packing fraction curve).

In 1954, Nier³⁰ performed a review of all published physical measurements of masses and the relative abundances of isotopes. As a result of this study, at the 1955 IUPAC meeting in Zurich, the Commission³¹ changed the atomic weight values of twelve elements including, dysprosium (data of Inghram³²), erbium (data of Hayden, Hess and Ingrham³³), gadolinium (data of Hess³⁴ and of Leland³⁵), hafnium (data of Hibbs³⁶), indium (data of White and Cameron³⁷), nickel (data of White and Cameron³⁷), palladium (data of Sites, Consolazio and Baldock³⁸), platinum (data of Inghram, Hess and Hayden³⁹), rhenium (data of White and Cameron³⁷), samarium (data of Inghram, Hess and Hayden⁴⁰ and of Leland⁴¹), tungsten (data of Williams and Yuster⁴²) and xenon (data of Nier⁴³).

In the first half of the twentieth century, only 3 chemical elements had their atomic weight value determined by mass spectroscopy, hydrogen, helium and osmium. Within the next ten years, an additional twenty-nine elements had their atomic weights determined by mass spectroscopy. One element, sulfur, had a range attached to its atomic weight value on the basis of mass spectrometric measurements. All of these thirty-two atomic weight values were due to relative isotopic abundance ratio measurements.

The ¹²C = 12 Mass Scale

Following Nier's suggestion¹ and the approval of the International Union of Pure and Applied Physics (IUPAP) and IUPAC, the atomic mass scale changed from ^{nat}O = 16 (chemical scale) and ¹⁶O = 16 (physics scale) to ¹²C = 12 (unified scale). At the 1961 IUPAC meeting in Montreal, the Commission⁴⁴ revised all atomic weight values to the new mass scale. The review of all chemical measurements and isotopic compositions from mass spectrometric measurements, along with a new atomic mass table⁴⁵, computed

with least squares methods from all significant experimental data on mass values from nuclear reaction data and mass doublets, led to many changes.

The total number of the 84 chemical elements, whose atomic weight values were based on mass spectrometric determinations increased from 32 elements to 62 elements in this revision. The other 22 elements had values based on chemistry alone (15 elements) or based on the agreement of chemical and mass spectrometric measurements combined (7 elements). Prior to this 1961 revision, only 10 of the 21 mono-nuclidic elements were based on packing fraction data. The other 11 elements shifted with the new atomic mass table. ^{231}Pa , which is not strictly a mono-nuclidic element, would not be considered for a Standard Atomic Weight (SAW) value until the 1969 Atomic Weight Table⁴⁶.

Results on calibrated mass spectrometric measurements were recommended for the first time in the 1961 Report for boron⁴⁷, nitrogen⁴⁸, argon⁴⁹ and chromium⁵⁰. In the 1967 Report⁵¹, five other elements and a better calibration on chromium⁵² had atomic weight values based on calibrated measurements.

Uncertainties, Variations and Footnotes

In the 1961 report, data on the variations in nature of the isotopic abundances from various sources of a particular element led to hydrogen, boron, carbon, oxygen and silicon joining sulfur in having an assigned range due to natural variations listed as an uncertainty. An uncertainty value was assigned to the chemical measurement of silver, which led to corresponding uncertainty values on the atomic weight values of chlorine and bromine, which had been determined by their ratios to silver. Chromium and iron also had uncertainties applied to their atomic weight values due to the experimental uncertainties on their isotopic ratios or to cover a disagreement with the chemical determination. By the 1969 report⁴⁶, uncertainties were assigned to all atomic weight values. At the time of the 1969 report, only fourteen elements did not have atomic weight values directly based on isotopic abundance measurements.

The procedure of the Commission has always been to tie the variations in nature with the uncertainty in the atomic weight value. The problem with this technique is that the Commission has always required that uncertainties on atomic weight values must be symmetric. Unfortunately, the variations of the isotopic abundance ratios in nature are usually not symmetric. To avoid extending the uncertainty in a symmetric fashion about the mean value of the atomic weight, the Commission has chosen to shift the mean value of the atomic weight to the center of the range of variations. As a result, the revised mean value for the atomic weight may not represent any known sample of the element in nature.

Footnotes were introduced in the Commission's tables to account for those specimens which were considered to be geologically exceptional, with footnote "g"; to account for specimens which had modified isotopic abundance sources, with the footnote "m"; to

account for those elements, whose range of abundance values restricted a more precise atomic weight values from being recommended, with the footnote “r”.

The Commission procedure allows the footnote “g” to be used, not for geologically exceptional specimens but for any specimen, which falls outside of the range indicated by the uncertainty on the atomic weight mean value (no matter how slight that difference). This mechanism was introduced by the Commission to avoid the problem of continually changing the atomic weight value to the center of the range of abundance ratios measured, every time a slightly larger range is observed in nature.

There is a problem in the use of the “delta/epsilon” scale of isotope ratio measurements. The variation of the isotopic abundance ratio for a sample compared to a standard is expressed as one part in one thousand (delta scale) or one part in ten thousand (epsilon scale). The difficulty is to find a standard that everyone can relate to. If the standard atomic weight value as discussed above does not relate to any known sample in nature, then the corresponding isotopic abundance ratio for the atomic weight of that element cannot be realized in a measurement. Thus, you cannot directly relate the measurement of some variation in nature of a sample of an element to the uncertainty in the atomic weight value for that element.

All of the above problems are related to one another. The Commission needs to consider how to handle these problems and what type of solution can be found for these problems. There is a need to study the delta/epsilon scale problem to try to find a solution that would be acceptable internationally.

Relative and Corrected Abundance Ratios

The Commission⁵³ treats the measurement of isotopic abundance ratios as “corrected” or “relative”. The corrected abundance ratio measurements can fall into 1 of 3 categories:

fully calibrated measurements, where the mass spectrometer is corrected for bias (linearity and fractionation) by the measurement of synthetic mixtures of isotopes, whose abundance ratios approximate all of the ratios in the mass spectrum of the natural element. Alternately non-linearity could be determined and fractionation could be corrected for by adding a synthetic mixture of isotopes to the sample prior to sample processing and isotopic analysis, the so-called double spike method. Both of these approaches are noted a “C”: a fully calibrated measurement.

calibrated mixtures are used to correct for isotopic fractionation but the measurement fails to fulfill all the requirements for a “C” measurement. This is noted as “F”: a fractionation corrected measurement.

the linearity of the mass spectrometer has been established for the relevant abundance ratios and beam intensities by using synthetic mixtures of isotopes or certified materials

produced by an appropriate Standards Laboratory. This is noted as “L”: a linearity corrected measurement.

relative abundance ratio measurements for which none of the above corrections have been attempted are noted as “N”: a non-corrected measurement.

At the present time, slightly over one quarter of the chemical elements fall into the category of mono-nuclidic elements (plus ^{231}Pa), whose atomic weight values are based on the atomic mass tables. Just under one quarter of the elements are based on relative isotopic abundance ratio measurements and slightly over one half of the elements have atomic weight values based on corrected isotopic abundance ratio measurements. Of these latter elements, about 90% are fully calibrated “C” measurements; about 10% are fractionation corrected “F” measurements and just one element (molybdenum) is linearity corrected “L” measurement.

A Task Group has been formed and is now working to assign uncertainties on isotopic abundance ratio measurements on a more consistent basis by determining which measurements fall into each category and to better estimate and assign the systematic uncertainty component of the uncertainty accordingly.

Evaluations of Isotopic Abundance Measurements

In 1973, the Commission formed a working group, the IUPAC Mass Spectrometric Evaluation Group (IMSEG), to perform an evaluation of all of the isotopic abundance measurements in the literature. It was to recommend natural isotopic abundance values and uncertainties for all elements, the best value for mass spectrometric atomic weights and uncertainties, identify discrepancies between chemical, mass spectrometric and ICAW atomic weight values and to recommend a range of values for isotopic abundances for the stable nuclides of each element that are found to be subject to variations in nature.

In 1975, after a successful IMSEG report from, the Commission created a Subcommittee on the Assessment of Isotopic Composition (SAIC) to continue this work on a more permanent basis. SAIC, as well as its successor, the Subcommittee for Isotopic Abundance Measurements (SIAM) both periodically produce Tables of the Isotopic Composition of the Elements (TICE). The tables include the range of abundance values in nature, footnotes or annotations, a best measurement from a single terrestrial source, the literature reference, any available reference material and a representative isotopic composition.

In 1983, the Commission approved for publication the SAIC evaluation report⁵⁴ “element by element review of their atomic weights”, which provided a historical review of the isotopic abundance ratio measurements and the resulting atomic weight values. In 2003, a Task Group of the Commission published another historical review⁵⁵. This report covered all of the isotopic abundance ratio measurements and atomic weight values reported during the twentieth century.

Discussion

The Commission has studied the range of isotopic abundance values for different sources of elemental isotopic composition. Their procedure selects the mid-point of each range as the point at which the Standard Atomic Weight value will be calculated. The uncertainty on the SAW is required to be symmetric, arguing that users do not know how to treat asymmetric uncertainties and that most computer programs will not accept uncertainties, which are asymmetric. Unfortunately, most variations are asymmetric in nature.

There is a disadvantage to this procedure, since the selected SAW value may correspond to no known natural sample of that element. Another disadvantage is that, as the range of natural variations keep becoming larger, either every infinitesimal change in range must be designated as an exceptional sample geologically, with the footnote "g", or the SAW value must be shifted and the uncertainty value expanded. Either option is illogical and inconsistent with the idea of a standard atomic weight value for an element. In the first case, it downgrades the truly exceptional geological specimen such as OKLO elements and in the second the SAW value keeps getting more uncertain and less well known as time goes on.

The uncertainty on the representative isotopic composition can be significantly larger than the uncertainty on the best measurement for particular elements, even by orders of magnitude. It is 27 times larger for calcium, 29 times larger for carbon, 32 times larger for silicon, 33 times larger for sulfur, 43 times larger for samarium and 270 times larger for gadolinium. In the case of hydrogen, it is a factor of 1400 and that large difference in uncertainties still does not cover the source of tank H₂. All of the above differences do not include geologically exceptional specimen but only normal sources of the element. These large differences are due strictly to the natural variations included as an uncertainty on the atomic weight value.

The measured isotopic abundance variations in nature reported in the literature, usually expressed on a "delta" or "epsilon" scale, are not directly related to the absolute isotopic abundance scale for many of the chemical elements. If an element's atomic weight value does not apply to any known sample of that element in nature, you can not relate the delta measurements to the atomic weight.

Conclusions

Following Thomson's discovery of stable isotopes in non-radioactive chemical elements, the derivation of atomic weight values from mass spectrometric measurements of isotopic abundance ratios moved very slowly. Forty years later, only 3½ % of the recommended values were based on mass spectrometric measurements and only 38% in the first half century. It might be noted that two chemical elements (tellurium and mercury) are still based on chemical measurements, where the atomic weight value calculated from the relative isotopic abundance measurement either agrees with the value from the chemical measurement or the atomic weight value calculated from the relative isotopic abundance

measurement falls within the uncertainty of the chemical measurement of the atomic weight.

Of the 19 chemical elements, whose atomic weight is based on non-corrected relative isotopic abundance measurements, five of these are two isotope systems (indium, iridium, lanthanum, lutetium and tantalum) and one is a three-isotope system (oxygen).

Recommendations

It is recommended that the Commission introduce a modified “guide to the expression of uncertainties in measurements of the International Organization for Standardization” (ISO/GUM) procedure to separately list the statistical uncertainty, the systematic bias of the instrument and the possible variations in the material sources in nature as the GUM type A, type B and type C uncertainties.

It is recommended that the Commission restrict the use of the footnote/annotation “g” to geologically exceptional specimens with truly anomalous values, such as “OKLO”, and perhaps to an element, for which there is only one natural sample that is variant.

The Commission should consider assigning the standard atomic weight (SAW) value to the most common sample of an element or to a reference material.

The Commission should appoint a Task Group to study the “delta/epsilon” scale problem. A Task Group should evaluate the relationship between absolute and relative abundance ratio measurements and make some recommendations for each element on a time frame of two to four years.

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