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Radioelements

Abstract

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14. Radioelements

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Abstract

Technetium (Tc) ($Z = 43$) and promethium ($Z = 61$) are the only elements below bismuth ($Z = 83$) in atomic number that have no stable isotopes. The discovery of these unusual elements is described, and the physical factors leading to their instability are discussed.

14.1 Introduction

The last of the naturally occurring stable elements were discovered in 1923 ($_{72}\text{Hf}$) and 1925 ($_{75}\text{Re}$). This concluded over a century of rapid discovery of elements found in the earth's crust. Two holes among atomic numbers below $_{83}\text{Bi}$ remained: 43 and 61. Moseley's correlation of atomic number with the energies of transitions involving inner atomic electrons made it clear that the gaps were real.

The steps and missteps in the process of discovering new elements led to caution in accepting a previously unidentified spectroscopic feature as evidence for a new element (Boyd 1959). Chemical characterization was required. For example, "masurium" was proposed for element 43, and "illium" and "florentium" were proposed for element 61 based on atomic spectroscopy of extracts from minerals. In retrospect, it is clear that there was evidence for unusual conditions for these elements. For example, above $_{7}\text{N}$ the only mass numbers of stable isotopes of elements with odd atomic number are also odd, and there is only one stable isobar for each odd A . Molybdenum (Mo) has stable isotopes 92, 94–98, and 100. Ruthenium has 96, 98–102, and 104. Niobium has 93, and rhodium 103. Nothing is left for technetium, which would have the best chance for stability at mass numbers 97 and 99. Similarly, either neodymium or samarium has a β -stable isotope from 142 to 150; nothing is left for promethium.

There is little difference between protons and neutrons with respect to nuclear properties, but our interest

in elements leads to an emphasis on atomic numbers. There are more missing neutron numbers (19, 35, 39, 45, 61, 89, 115, and 123).

The development of the cyclotron and, later, the fission reactor gave the means for a variety of artificial transmutations, but it often was difficult to identify the element and mass number of a radioactive product. In many cases, individual radionuclides could be characterized only by simple features, such as half-life or attenuation of radiations in absorbers, which did not allow discriminating the components of a complex mixture. Chemical evidence was required in order to make definite identification with a particular element.

Except when original observations are relevant, nuclear properties are taken from compilations (Browne et al. 1986; Firestone et al. 1996; Baum et al. 2002).

14.2 Discovery of Technetium

According to the periodic table, the missing element at atomic number 43 should be between manganese and rhenium with regard to chemical properties. The group that discovered rhenium in 1925 (Noddack et al. 1925) claimed to have detected element 43 (“masurium”) in the same minerals. They subsequently were able to isolate weighable quantities of rhenium (more than a gram in 1928), but neither they nor others could confirm detection of element 43.

Technetium was finally discovered by Perrier and Segrè in 1937 (Perrier and Segrè 1937). E.O. Lawrence had bombarded a Mo plate with deuterons (and secondary neutrons) in the Berkeley cyclotron for several months prior to shipping the plate to Segrè’s group in Italy in late December, 1936. Perrier and Segrè began their radiochemical studies in late January, more than 6 weeks after the end of bombardment. On the surface exposed to the deuterons, they found “strong activity, chiefly due to very slow electrons” ascribed to “more than one substance of a half-value period of some months” in addition to ^{32}P (which they could not explain, but did not attribute to reactions with molybdenum).

Subsequent work (Cacciapuoti and Segrè 1937) revealed that most of the Tc activity arose from what are now known to be 61-day $^{95\text{m}}\text{Tc}$ and 90.1-day $^{97\text{m}}\text{Tc}$.

Table 14.1 shows a nuclide chart corresponding to charged particle reactions with Mo. Both (d,n) and (d,2n) reactions are energetically allowed (have energy requirements, Q , that are met by the bombarding particle) and lead directly to Tc. For example, the Q values for $^{95}\text{Mo}(\text{d},\text{n})^{96}\text{Tc}$ and $^{95}\text{Mo}(\text{d},2\text{n})^{95\text{m}}\text{Tc}$ are -3.25 and -4.74 MeV, respectively. The Q value for $^{97}\text{Mo}(\text{d},2\text{n})^{97\text{m}}\text{Tc}$ is -3.42 MeV. In addition, (d,p) reactions with ^{98}Mo and ^{100}Mo produce Mo isotopes that subsequently decay to Tc. Therefore, the bombardments produced many different radioactive products, but after a few months 61-day $^{95\text{m}}\text{Tc}$ and 90.1-day $^{97\text{m}}\text{Tc}$ dominate the activity. Note that both of these are isomeric levels. One, $^{95\text{m}}\text{Tc}$, decays primarily (96%) by electron capture (EC) and subsequent γ -ray emission. The other, $^{97\text{m}}\text{Tc}$, decays by a highly converted 96.6 keV transition, which presumably produced the “slow electrons” reported (Perrier and Segrè 1937).

Table 14.1

Nuclide chart showing stable molybdenum isotopes and radioactive products from deuteron bombardments

^{92}Tc	^{93}Tc	^{94}Tc	^{95}Tc	^{96}Tc	^{97}Tc	^{98}Tc	^{99}Tc	^{100}Tc	^{101}Tc	^{102}Tc
4.2 min	43 min 2.73 h	52 min 4.88 h	61 days 20 h	52 min 4.3 days	91 days 4.2 My	4.2 My	6.01 h 0.21 My	15.8 s	14.2 min	4.4 min 5.3 s
^{91}Mo	^{92}Mo	^{93}Mo	^{94}Mo	^{95}Mo	^{96}Mo	^{97}Mo	^{98}Mo	^{99}Mo	^{100}Mo	^{101}Mo
1.08 min 15.5 min	14.84%	6.9 h 3.5 ky	9.25%	15.92%	16.68%	9.55%	24.12%	2.75 days	9.63%	14.6 min

Stable isotopes are shaded and list natural abundances in percentage. When isomers exist and relative energies of the states are known, the half-life of the isomer is given above that of the ground state. See also Table of Nuclides in this volume’s appendix (My = Ma, i.e., 10^6 years; ky = ka, i.e., 10^3 years).

The chemical identification consisted of three parts: (1) removing ^{32}P , (2) showing that the remaining activity was not due to zirconium, niobium, or molybdenum, and (3) showing that the element was similar, but not identical, to rhenium.

Until recognized for what it was, the ^{32}P was very confusing. Once recognized, it was removed by precipitating magnesium ammonium phosphate.

Zirconium was isolated as a hydroxide, molybdenum as a sulfide, and niobium as sodium metaniobate.

None carried a significant part of the activity.

In solutions containing inactive manganese and rhenium carriers as well as the activity under study, the activity generally followed rhenium. This is consistent with the greater chemical similarities of heavy members of a group in the periodic table as compared to differences among lighter members. However, Tc_2O_7 is more volatile than Re_2O_7 , and the chloride is less volatile. Other chemical properties of Tc are summarized in Sect. 14.5.

14.3 Discovery of Promethium

In principle, ^{61}Pm could have been discovered by techniques similar to those used for ^{43}Tc – bombardment of ^{60}Nd with charged particles from a cyclotron and demonstration that some of the products were isotopes of element 61. Indeed the bombardments were done, and several radionuclides that might have been isotopes of element 61, for example, “cyclonium” (Pool and Quill 1938), were produced, but the essential chemical proof was lacking. Whereas technetium and its neighbors differ considerably in chemical properties, variations among neighboring rare earths are very slight. This chemical characterization was beyond the capabilities of the groups carrying out the bombardments. Conventional chemistry is illustrated by the chemistry used to support the claim of “illinium” (Harris et al. 1926). This report is one (Observations on the Rare Earths XXIII) of a series of studies in which concentrates rich in one or more rare earths were studied by arc spectroscopy. Harris et al. chose a concentrate prepared by methods that were intermediate between those found to enhance ^{60}Nd and ^{62}Sm . The visible and infrared emissions were found to have about 130 lines that were not unique to either Nd or Sm fractions. They found support for their claims in absorption spectra of solutions and L X-ray emission spectra associated with the concentrate.

The chart of the nuclides in the region of promethium is given in Table 14.2. It is obvious now that there is a negligible amount of promethium in natural materials, and if macroscopic amounts were present, the radioactivity would be intense. The isotope with the longest half-life is 17.7-year ^{145}Pm . If it were present at 1 ppm in a 1-g sample, the activity would be 6 MBq.

Table 14.2

Chart of the nuclides in the vicinity of promethium

^{143}Pm	^{144}Pm	^{145}Pm	^{146}Pm	^{147}Pm	^{148}Pm	^{149}Pm	^{150}Pm	^{151}Pm	^{152}Pm	
265 days	360 days	17.7 years	5.53 years	2.62 years	41.3 days 5.37 days	2.21 days	2.68 h	1.18 days	13.8 min 4.1 min	
^{142}Nd	^{143}Nd	^{144}Nd	^{145}Nd	^{146}Nd	^{147}Nd	^{148}Nd	^{149}Nd	^{150}Nd	^{151}Nd	
27.2%	12.2%	23.8% 2.1×10^{15} years	8.3%	17.2%	10.96 days	5.7%	1.73 h	5.6%	12.4 min	
^{141}Pr						2.25%		1.08%		0.419%
100%										
^{140}Ce		^{142}Ce								
88.45%		11.11%								

The columns contain isotones. The fission yields for ^{235}U are given for several mass chains (i.e., isobars situated along the lines shown by the slanted arrows). Stable isotopes are shaded and list natural abundances in percentage. (The upmost cells show the stable and primordial Sm isotopes.) See also Table of Nuclides in this volume’s appendix.

In order to separate adjacent elements and to isolate very small quantities, a new chemistry was needed. This chemistry, ion-exchange chromatography (see Chap. 51 in Vol. 5) with synthetic resins (Walton 1976), was developed during the Manhattan project in order to study the products of nuclear fission, which include several light rare earths. The technique was borrowed in order to identify element 61. By mid-1945, over 30 fission products had been characterized as isotopes of yttrium or one of the lanthanides, but few could be definitely assigned by element and mass numbers. The work that ultimately identified element 61 began with a mixture of radioactive materials that concentrated elements between cerium and samarium. This mixture included a soft β emitter with a half-life of ~ 4 years and a γ emitter

of ~11 days.

Marinsky, Glendenin, and Coryell reported their discovery of promethium in part as follows:

In the comprehensive studies of the radioactive species produced in the fission of uranium it has been found that over thirty are members of the rare earth family (isotopes of yttrium and the group lanthanum through europium). The chemical and physical identification of these was an important part of the research program of the Manhattan Project. Standard oxidative separations and fractional precipitations and the use of radiochemical methods based on chain relations served to distinguish the activities of yttrium, lanthanum, cerium, and some of praseodymium, and those of samarium and europium. The characterization of the sequence praseodymium, neodymium, and element 61 presented very difficult problems that were solved only with the intensification of ion exchange methods originally developed by Boyd and co-workers and applied to the rare earth field by Cohn and co-workers. (Marinsky et al. 1947)

The ion-exchange medium was Amberlite IR-1, a sulfonated phenol-formaldehyde resin, and the eluting agent was 5% ammonium citrate at pH 2.75. The order of elution of the rare earths had been established in a series of experiments with radioactive cerium, lanthanum, europium, and yttrium produced by fission, and praseodymium and europium produced by neutron capture. The lanthanides elute in reverse order of atomic number, and yttrium precedes the fission-product lanthanides. Although the technique was a vast improvement over conventional chemical procedures, the physical form of the resin was quite inferior to modern, uniform beads. Resolution was barely better than the separation between adjacent elements, and there was considerable tailing. It is fortunate that the elution order puts low yield products before higher yield ones; this minimized the interference from tailing.

A sample of the praseodymium group (praseodymium, neodymium, and element 61; most of the cerium, samarium, europium, and yttrium fission products had been removed, cerium by ceric iodate precipitations and the others by potassium carbonate digestions) was absorbed on an Amberlite IR-1 column. The elution curve is shown in Fig. 14.1. The soft β emitter (now known to be 2.62-year ^{147}Pm ; see Table 14.2) appeared in the position expected (by interpolation) for element 61 and became the first isotope identified as the element subsequently named promethium. The ~11-day γ emitter eluted in the position expected for neodymium (10.98-day ^{147}Nd). The praseodymium peak was primarily 13.6-day ^{143}Pr . The yttrium peak was primarily 58.5-day ^{91}Y ; the fission yields of ^{143}Pr and ^{91}Y were high, ~6%, and the yttrium peak represented ~0.001% of the fission yield.

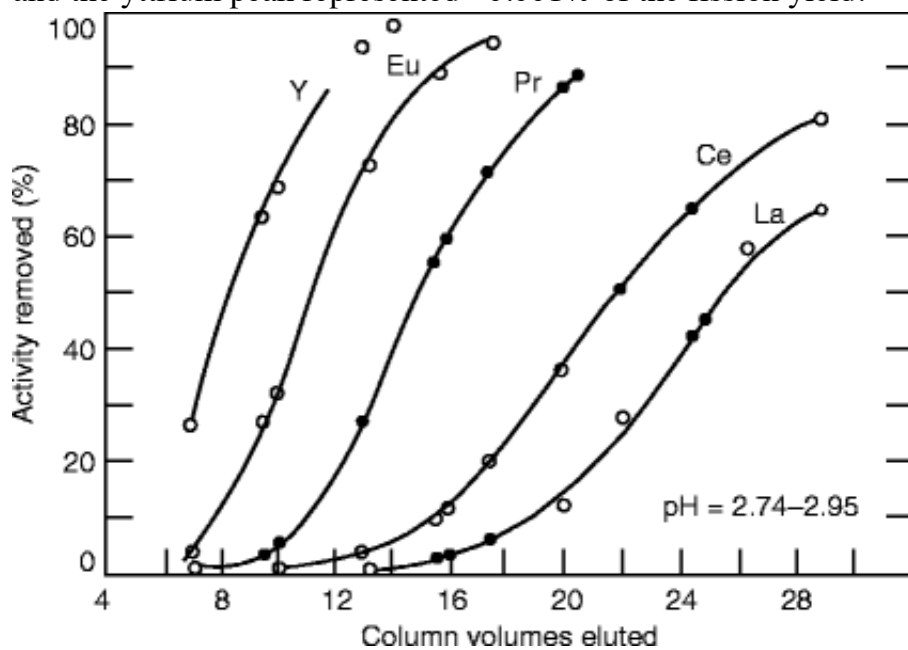


Fig. 14.1

Elution curve for the praseodymium group of fission products from Amberlite IR-1 (Reprinted with permission from Marinsky et al. 1947. © 1947 American Chemical Society)

Subsequent experiments with neutron-activated neodymium, described in the same report, revealed a 1.7-h isotope of neodymium (too short to be isolated from fission with the available columns) and a 47-h

isotope of element 61 (also detected in fission).

Mass number assignments were based on quantitative analysis of the chemically resolved fractions. Fission is known to produce primarily products with neutron/proton ratios similar to the fissioning nuclide (here ^{236}U , two to three neutrons less which accompany the fragments). For $A = 147$, the most likely products are 4-s ^{147}La and 56-s ^{147}Ce , which join their short-lived isobars by quickly cumulating in 11-day ^{147}Nd . Very little ^{147}Pm is produced as a primary fission product; just as in neutron activation of Nd, ^{147}Pm is a product of decay of ^{147}Nd .

The general form of the mass-yield distribution was also known; the yield drops rapidly with increasing mass number above 144. By showing that the cumulative fission yield of the “~4-year” product was about twice that of the “48-h” product, combined with the results of activation measurements, ^{147}Pm and ^{149}Pm were identified by mass number as well as atomic number.

14.4 Why Do Tc and Pm Have No Stable Isotopes?

There is nothing remarkable about atomic numbers 43 and 61, except that they are odd integers and, in terms of nuclear stability, unlucky. In random assemblages of neutrons and protons, there are equal probabilities for even A versus odd A or even Z versus odd Z . However, far fewer odd- Z nuclides are stable. These generalizations can be understood in terms of the symmetry and pairing terms in the semiempirical mass equation (Choppin et al. 2002; Friedlander et al. 1981). The symmetry term is proportional to $(N-Z)^2$ and leads to a parabolic dependence on Z at constant A . The pairing term raises odd- Z odd- N nuclides and lowers even- Z even- N nuclides, producing two parabolas for even- A isobars. The effect is exemplified by the $A = 98$ isobars shown in Fig. 14.2. Both parabolas are centered on $Z = 43.0$, and they are separated by ~2.22 MeV; ^{98}Tc would be at the bottom and, therefore, stable, were it not for the pairing term.

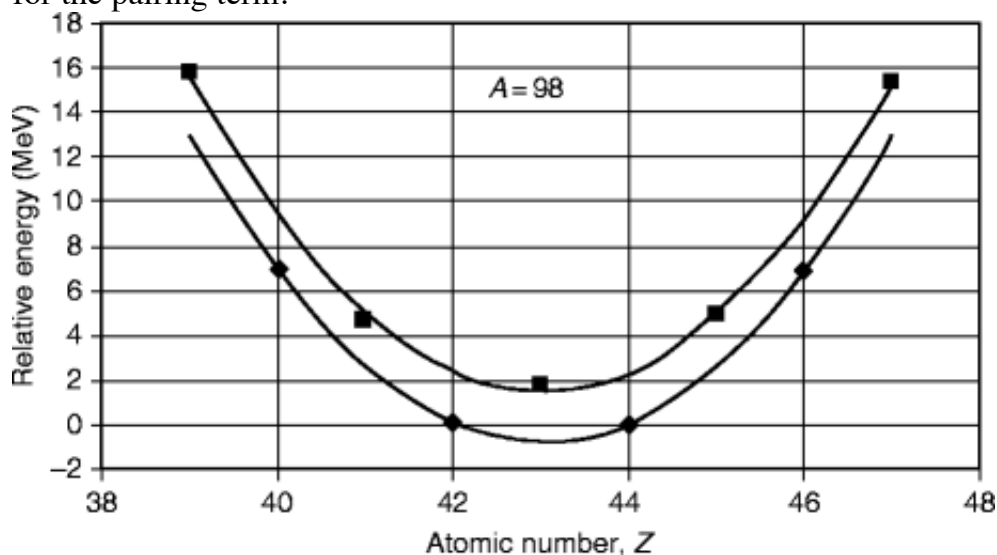


Fig. 14.2

Relative energies of the $A = 98$ isobars. The odd- Z and even- Z elements are represented by squares and diamonds, respectively. The lines are quadratic (parabolic) fits to the data of each type. For mass numbers above ~40, and certainly by $A \sim 98$, Coulomb energies are significant, and stable nuclides have more neutrons than protons. The parabolic minima define $Z_A(A)$, which increases by ~0.45 per mass unit in the vicinity of Tc. Therefore, the minima should be $\sim 43.0 - 0.45 = 42.55$ at $A = 97$ and should be 43.45 at $A = 99$. Both of these are closer to 43 than to the adjacent atomic numbers. If masses were exactly parabolic, both ^{97}Tc and ^{99}Tc would be stable. These are the conditions that lead to two stable isotopes for some odd- Z elements.

Although the masses (energies) are nearly parabolic, as shown in Fig. 14.3, valence neutrons are slightly more stable than valence protons. According to the mass equation, energy differences between adjacent isobars should be linear with atomic number. These energy differences are just the Q values for β decay (β^- and EC). These energy differences are shown in Fig. 14. The upper part gives EC energies for $A = 97$ isobars for comparison with ^{97}Tc , which decays by this mode. The lower part gives β^- energies for $A = 99$ for comparison with ^{99}Tc . In both parts, linear fits to odd- Z and even- Z elements are shown. For both chains, the small positive decay energy for Tc would disappear if the neutron-proton separation were removed.

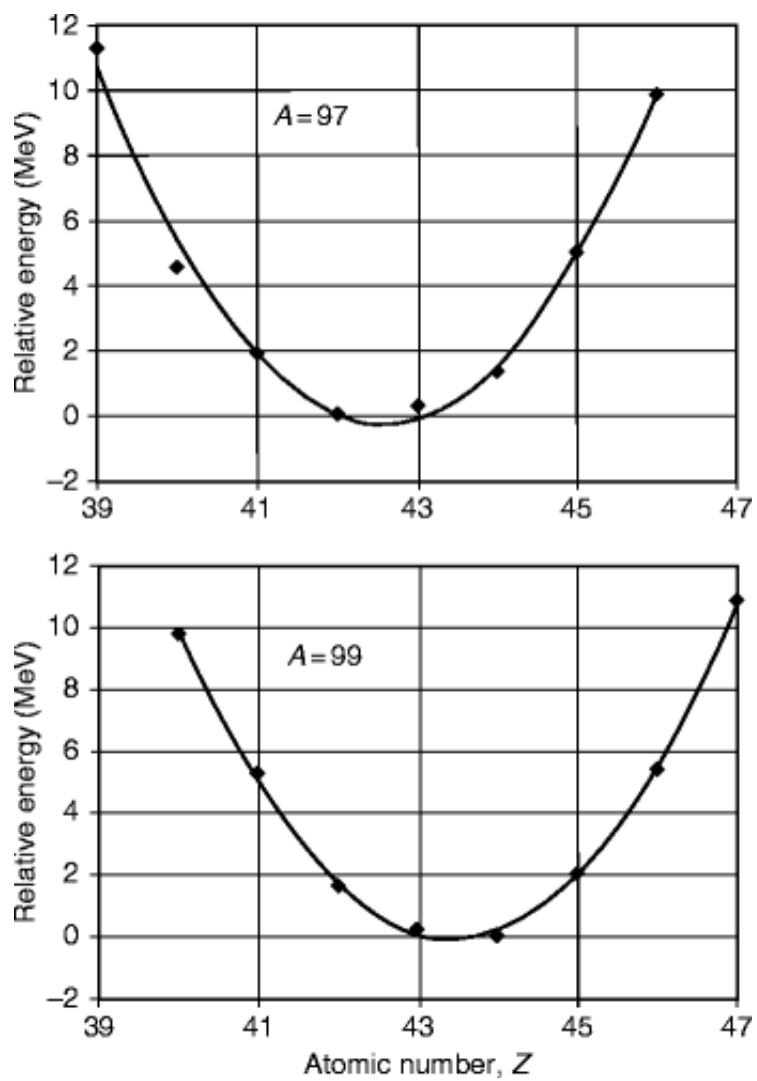


Fig. 14.3
Relative energies of the $A = 97$ (top) and $A = 99$ (bottom) isobars

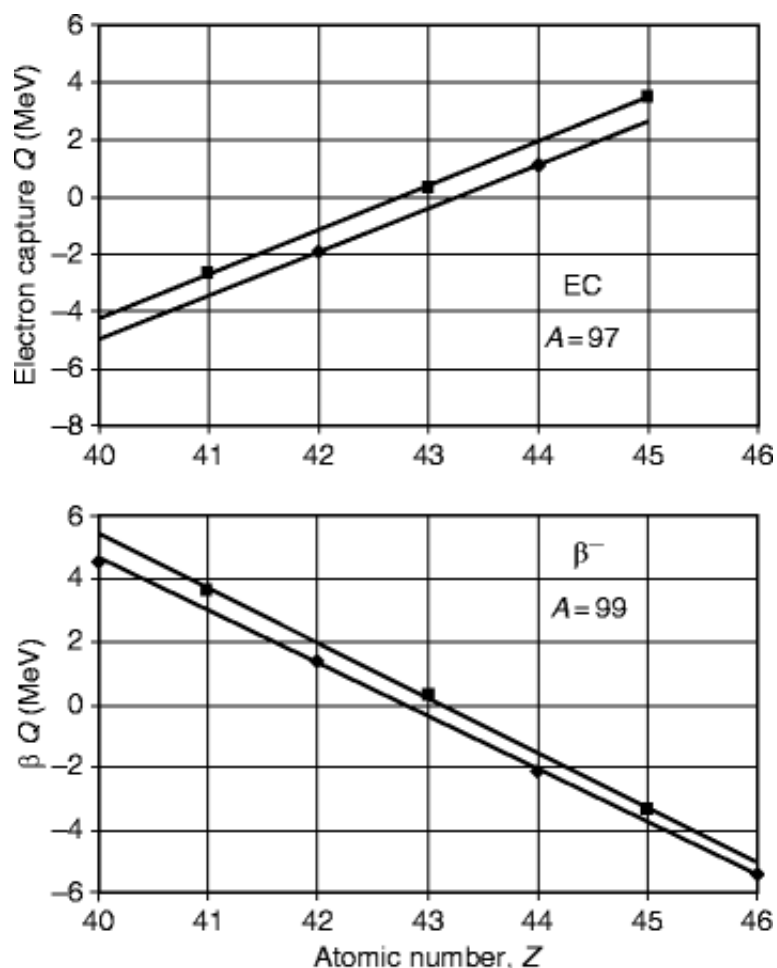


Fig. 14.4

Q values for electron capture (EC) decay of $A = 97$ isobars (top) and β^- decay of $A = 99$ isobars (bottom). The same analysis applies to promethium. Figure 14.5 shows the decay energies for isobars of ^{145}Pm (17.7-year EC decay) and ^{147}Pm (2.62-year β^- decay). Just as for technetium, the most favored isotopes of promethium just miss being stable because of small differences in neutron-proton stabilities.

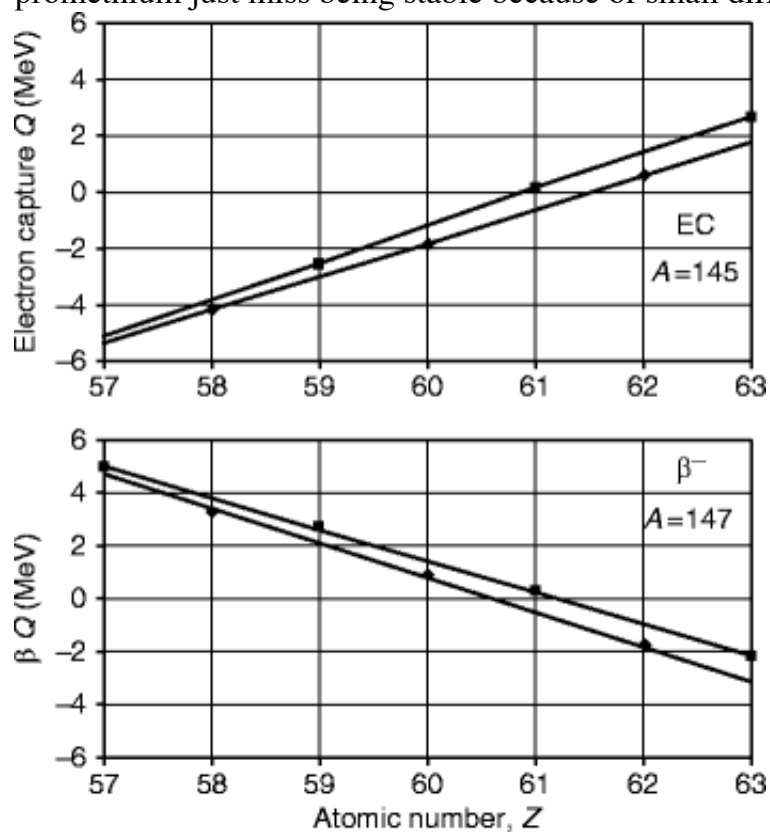


Fig. 14.5

Q values for electron capture (EC) decay of $A = 145$ isobars (top) and β decay of $A = 147$ isobars (bottom)

Although these promethium isotopes have much shorter half-lives than the corresponding technetium isotopes, the decay energies for promethium are actually lower. Decay of the technetium isotopes is hindered by large angular momentum changes.

Because of a small difference in stability of valence neutrons and protons, combined with fortuitous combinations of symmetry and Coulomb energies, technetium and promethium have no stable isotopes. Since Z_A increases ~ 0.4 per mass number, the neutron number must increase by ~ 0.6 per unit A .

Therefore it is expected that all isotones for some odd N would be unstable. As pointed out in Sect. 14.1, there are eight such neutron numbers.

14.5 Chemical and Physical Properties

Neither element is unusual except for the absence of stable isotopes. Of course this means that either pure element can have high specific activity; there are no nonradioactive forms to dilute the activity.

On the earth, the most likely source of macroscopic amounts for technetium and promethium is the reprocessing of irradiated uranium and plutonium from fission reactors. This mechanism produces only a few, and not the longest-lived, isotopes.

For technetium, most of the mass consists of 2.1×10^5 -year ^{99}Tc . Heavier isotopes are short lived, and lighter isotopes are “shielded” by stable isotopes of molybdenum (here “shielded” means not fed by chain decay). The cumulative fission yield of ^{99}Tc is $\sim 6\%$ for both ^{235}U and ^{239}Pu . Because of the long half-life, this isotope contributes little to the total activity of fission products for the first 100 years, but it is the dominant fission product after 1,000 years. There is little energetic γ emission; it is difficult to detect ^{99}Tc in impure matrices without chemical processing. The dominant mode of decay is 293.5-keV β decay to the ground state of ^{99}Ru . Only 0.0016% of the β decays goes to excited states. The only γ -ray is the $\sim 6 \times 10^{-4}\%$ 89.7-keV γ . The most important isotope of technetium is 6.0-h $^{99\text{m}}\text{Tc}$. It is a valuable tool of medical diagnostics because of its chemical versatility and the energy of its γ -ray. This application is described in detail in Chap. 43 of Vol. 4.

For promethium, most of the mass in aged fission products consists of 2.62-year ^{147}Pm , which (except for half-life) has decay characteristics similar to ^{99}Tc . The dominant decay mode is 224-keV β decay to the ^{147}Sm ground state. Only 0.0057% of decays feeds excited states. The most intense γ -ray is the 0.0029% 121.2-keV γ -ray. Because of its high specific activity and the absence of penetrating radiation, ^{147}Pm has been used as a source of energy in thermoelectric converters for remote applications.

Anders (1960) summarized the chemical properties of technetium in his monograph on radiochemistry.

In many respects, the analytical behavior of Tc is similar to that of Re. Both form stable XO_4^- anions that give insoluble salts with large cations; both form volatile heptoxides; and both form acid-insoluble heptasulfides. However, there are subtle differences between the two elements that can cause them to behave very differently under certain conditions. The vapor pressure of Tc_2O_7 is much higher than that of Re_2O_7 at low temperatures (e.g., 10^{-1} mm at 100°C , compared to $\sim 3 \times 10^{-5}$ mm for Re_2O_7). In contrast to rhenium, technetium (VII) is therefore easily lost upon evaporation of acid solutions unless a reducing agent is present or the evaporation is conducted at a very low temperature. Ignorance of these factors has led to a multitude of conflicting statements concerning the volatility of technetium.

The chemistry of promethium must be understood in terms of the trends in properties within the rare earth, or lanthanide, series. The radiochemistry of these elements (plus scandium and yttrium) has been described by Stevenson and Nervik (1961).

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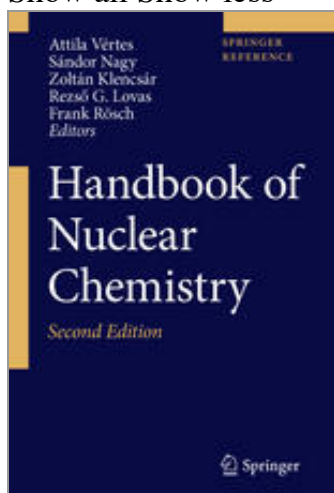
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