ERRATUM

To the article “Development of a Super-Stable Datum Point for Monitoring the Energy Scale of Electron Spectrometers in the Energy Range up to 20 keV” by D. Vénos, M. Zbořil, J. Kašpar, O. Dragoun, J. Bonn, A. Kovalík, O. Lebeda, N. A. Lebedev, M. Ryšavý, K. Schlösser, A. Špalek, and Ch. Weinheimer, Vol. 53, No. 3, pp. 305–312, July, 2010

Due to various typographical and translation errors, this article is republished below.

IONIZING RADIATION MEASUREMENTS

THE DEVELOPMENT OF A SUPER-STABLE STANDARD FOR MONITORING THE ENERGY SCALE OF ELECTRON SPECTROMETERS IN THE ENERGY RANGE UP TO 20 keV

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UDC 539.16

The long-term energy stability of the 7.5 keV and 17.8 keV conversion electrons of the 9.4 keV and 32 keV nuclear transitions respectively in ^{83m}Kr , emitted by solid $^{83}\text{Rb}/^{83m}\text{Kr}$ sources, prepared by evaporation in a vacuum, is investigated using two different spectrometers. The results obtained indicate the principal applicability of these ^{83}Rb sources for monitoring the stability of the energy scale of electron spectrometers in the 20 keV region at the level of ± 60 meV for at least two months, which corresponds to the requirement of the new KATRIN tritium neutrino project. Investigations are being continued using sources produced by implantation of ^{83}Rb ions into different substrates.

Key words: nuclear transition energy, conversion electrons, electron spectrometer, neutrino mass.

The solution of the problem of the rest mass of the neutrino is one of the most important problems in modern particle physics and cosmology. The accepted value of $2 \text{ eV}/c^2$ [1] for the upper limit of the effective rest mass of the electron

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antineutrino is based on the experimental data of $2.3 \text{ eV}/c^2$ [2] and $2.1 \text{ eV}/c^2$ [3], obtained from an analysis of the form of the β -spectrum of tritium in the region of its endpoint.

The aim of the new KATRIN project (KARlsruhe TRItium Neutrino experiment) [4], which is based on the accumulated experience of earlier projects [2, 3], is to increase the sensitivity to the effective rest mass of the electron antineutrino to $0.2 \text{ eV}/c^2$ (with a 90% confidence level) over a three-year duration of the collection of statistics. One of the most important requirements is the stability of the retarding voltage of -18.6 kV in the KATRIN spectrometer, which enables the β spectrum of tritium to be analyzed close to its endpoint. Calculations have shown that an instability of this voltage of $\pm 60 \text{ mV}$ can introduce a systematic error of $0.007 \text{ eV}^2/c^4$ into the value of the square of the neutrino mass obtained from the measured spectrum. This error makes a considerable contribution to the maximum acceptable systematic error of $0.017 \text{ eV}^2/c^4$ with an expected sensitivity of $0.2 \text{ eV}/c^2$ [4, 5]. Long-term monitoring of the stability of the -18.6 kV voltage within the limits of a deviation of $\pm 60 \text{ mV}$ (i.e., with an error of $\pm 3 \cdot 10^{-6}$) during a period of two months (the planned duration of a single measurement session) is a fairly complex problem for modern techniques. Hence, the stability of this voltage will be verified simultaneously by two independent methods: using a highly accurate voltmeter in combination with a special high-voltage divider [6] and by monitoring the position of a time-stable line of monoenergetic electrons from a natural source in a special monitoring spectrometer (of the same type as the KATRIN spectrometer), which will be connected to a common -18.6 kV voltage source. In this case, any change in the position of the reference line will indicate instability in the common retarding-voltage system.

In previous experiments [2, 3, 7], the narrow line of K conversion electrons of the 32 keV nuclear γ -transition in ^{83m}Kr with an energy of $17824.3 \pm 0.5 \text{ eV}$ [9] (with a natural width of $2.70 \pm 0.06 \text{ eV}$ [8]) was used for the energy calibration of spectrometers. This line is also more convenient than other ones to use in the KATRIN monitoring spectrometer. In [2], ^{83m}Kr on a carbon substrate was used, while gaseous ^{83m}Kr was used in [3, 7]. Since the half-life of ^{83m}Kr is short ($T_{1/2} = 1.83 \text{ h}$), it is not very suitable for long-term monitoring. Moreover, both of the above methods require the use of complex equipment.

A solid source of the parent isotope of ^{83}Rb with a half life $T_{1/2} = 86.2 \text{ days}$ is more suitable for monitoring. The results of measurements of the spectra of the Auger K - and L -electrons of krypton, and also of the conversion K -, L -, M -, and N -electrons of the 32 keV and 9.4 keV γ -transitions in ^{83m}Kr from the decay of the solid ^{83}Rb source, were presented in [10, 11]. The source was made by evaporation in a vacuum onto an aluminum substrate. The result of an investigation of the position of the conversion K -line of the 32 keV γ -transition (K -32) over a period of several months with an error of $\pm 1.1 \text{ eV}$ is given in [12], which does not correspond to the required stability in the KATRIN project. In this connection, we need to recall that no commercial solid-state energy standards of conversion electrons or Auger electrons, having an error of less than 0.1 eV , so far exist. The kinetic energies of these electrons depend directly on their binding energy in the atom. In solid-state sources, the binding energies of the electrons are sensitive to the physicochemical surroundings of the radioactive atoms, which leads to energy shifts in the spectra of both the conversion electrons and the Auger electrons. The same effect is also observed in the photoelectron spectra of stable atoms. This energy shift, which is called a chemical shift, amounts to $\pm 7 \text{ eV}$ and is highly correlated with the valence state of the radiating atom [13–15]. It is also well known that the binding energies of the electrons in solids, as a rule, are $2\text{--}10 \text{ eV}$ less than those for free atoms [16, 17], while for atoms adsorbed on the surface of solids the binding energies are $1\text{--}3 \text{ eV}$ less than those for free atoms [18–20].

Below we present the first results of an investigation of the stability of the measured energies of the conversion lines of the 9.4 keV and 32 keV nuclear γ -transitions in ^{83m}Kr from the decay of ^{83}Rb with an error of less than 0.1 eV using solid sources, prepared by evaporation in a vacuum on different substrates.

Electron Spectrometers. For the accurate determination of the energy of the conversion electron lines using solid sources, it is necessary for the resolving power of the instrument to be several electronvolts. This enables us to distinguish, in the experimental spectrum, peaks of electrons which leave the source without energy loss, so-called no-loss peaks. The natural width of such a peak is determined by the natural widths of the corresponding atomic level and nuclear transition. In our investigations, we employed two different types of electron spectrometers: the differential electrostatic ESA12 [21, 22] of the cylindrical-mirror type with second-order focusing, supplied with a retarding (accelerating) lens and a channel multiplier as an electron detector (Institute of Nuclear Physics, Academy of Sciences of the Czech Republic, Řež near Prague), and an integral spectrometer [23] with the magnetic adiabatic collimator with an electrostatic filter type – the MAC-E-Filter (Institute of Physics, University of Mainz, Germany). The first of the spectrometers operates in the $0\text{--}20 \text{ keV}$ range with a

relative instrument resolution $\Delta E/E = 0.011$ in the “basic” mode and with an absolute resolution $\Delta E = 1$ and 3 eV for electron energies of 2 keV and 7 keV, respectively, in the electron retardation mode (reduced transmission). The second of the spectrometers operates in the 7–35 keV energy range with a limiting value of $\Delta E/E = 5 \cdot 10^{-5}$. The vacuum in the working volume of the ESA12 spectrometer reaches $5 \cdot 10^{-8}$ mbar (5 μ Pa), which is two orders of magnitude worse than in the University of Mainz spectrometer. The limiting vacuum in the second spectrometer is determined by the ability to heat the walls of its vacuum chamber to a temperature of several hundreds of degrees. It should be noted that, after slight reconstruction, the University of Mainz spectrometer will operate as a monitoring spectrometer in the KATRIN project.

The Preparation of Radioactive Sources of ^{83}Rb by Evaporation in a Vacuum. The quality of a radioactive source can be defined as the ratio of its thickness to the mean free path for inelastic electron scattering. The greater this ratio, the lower the probability that the electron will leave the source without a loss of energy. As a result, the intensity of the “no-loss peak,” mentioned above, the formation of which is necessary for the accurate determination of the electron energy from the apparatus spectrum, will be reduced. This is the reason why such close attention is paid to the preparation of radioactive sources in precision nuclear spectroscopy of low-energy electrons. When preparing thin radioactive sources, the most suitable method is by evaporation in a vacuum onto a suitable substrate. It has been used successfully in low-energy ($E < 20$ keV) nuclear electron spectroscopy for the last two to three decades (see, for example [24]).

The radionuclide ^{83}Rb was obtained in the $\text{Kr}(p, xn)^{83}\text{Rb}$ reaction when natural krypton is irradiated with protons with a primary energy of 27 MeV in the U-120M cyclotron of the Institute of Nuclear Physics Řež. The activity of ^{83}Rb reached approximately 100 MBq, and the efficiency of its release from the target in an aqueous solution was about 90% [25]. The solution of rubidium was concentrated by drying under an infrared lamp in a quartz container. The solid residue was dissolved in 0.1 M HNO_3 (0.5–1.5 ml). The solution of rubidium obtained was introduced into a chromatograph column of length 70 mm and diameter 1.5 mm. After this the column was washed with portions of 0.5 cm³ of solutions of HNO_3 with concentrations of 0.3, 0.5, and 1.0 M. The rubidium from the column was eluted with a solution of 2.0 M HNO_3 with a volume of 0.5 cm³. The final solution of rubidium prepared in this way was evaporated to dryness, dissolved in distilled water, and deposited in the boat of the vacuum evaporator.

To prepare sources of ^{83}Rb by the evaporation method we used commercial MED 020 apparatus made by BAL-TEC of Liechtenstein, fitted with an oil-free vacuum system, which enables a vacuum of $3 \cdot 10^{-6}$ mbar to be achieved in the volume of the evaporation chamber. The equipment was only used to evaporate ^{83}Rb . In the majority of cases, we used a tantalum boat, but we also tested molybdenum and tungsten boats. The boats were heated by passing an electric current through them. To choose the optimum evaporation conditions, we measured how the quantity of ^{83}Rb obtained by evaporation depended on the boat temperature. We found that rubidium does not vaporize up to a boat temperature of 300°C. At a temperature of 600°C, several percent of the initial quantity of ^{83}Rb remained in the boat. When preparing sources for measurements of electron spectra, the boat with ^{83}Rb in it was initially heated to a temperature of 200°C for 5–10 minutes, in order to get rid of possible interfering impurities. Here the substrate of the source was protected with a metal screen. When the heating was completed, the screen was moved to one side and the evaporation of the rubidium was carried out at a temperature of 800°C for 30–60 seconds through a mask of diameter 8 mm. After evaporation, the substrate with the ^{83}Rb was clamped in a special copper holder, previously covered with gold, in order to ensure reliable electrical contact.

In addition to the thickness, the quality of the $^{83}\text{Rb}/^{83m}\text{Kr}$ sources was also estimated from the fraction of ^{83m}Kr retained in the source. Unlike the 32.2 keV nuclear γ -transition, which only occurs in the decay of the isomer state of ^{83m}Kr [26], the 9.4 keV transition is generated in three ways: directly from the decay of ^{83}Rb to the 9.4 keV nuclear level of ^{83}Kr ; from the decay of ^{83}Rb to the nuclear levels of ^{83}Kr above 9.4 keV and from the isomer state via the 32.2 keV transition. Consequently, to determine the fraction of the ^{83m}Kr retained in the source it was necessary to measure the ratio of the intensities of γ -quanta with energies of 32.2 and 9.4 keV emitted in the decay of ^{83}Rb .

During the course of the investigations, we varied the main factors which affect the properties of the source, namely: the composition and activity of the initial ^{83}Rb for evaporation (in one case stable rubidium admixture was added), the material of the source backing and the distance from the boat to the source backing. The purpose of this was to achieve reproducibility of the values of the conversion-line energies, measured with different ^{83}Rb sources, a fairly high activity of $^{83}\text{Rb}/^{83m}\text{Kr}$ in the sources, a considerable fraction of ^{83m}Kr retained in the source, and a high “no-loss peak” intensity.

Fig. 1. The L_1 -9.4 conversion line of ^{83}Rb decay, measured on the ESA12 differential electrostatic spectrometer with the apparatus resolution of 3 eV in steps of 0.5 eV (the source was prepared on an aluminum backing).

The distance between the boat and the source backing was varied in the range 2.5–15 mm. We used aluminum foil, oxidized in a natural way, and having a thickness of 50 μm as the source backing or a carbon plate 0.2 mm thick (foil) and 1 mm thick (highly oriented pyrolytic graphite – HOPG). In all, we prepared 29 sources of ^{83}Rb , 16 of which were on an aluminum backing, 12 were on a carbon backing, and one was on an HOPG backing. The activity of the ^{83}Rb in the sources lay in the range 0.4–12.0 MBq, which corresponded to a calculated thickness of 0.02 to 0.5 of a monolayer of ^{83}Rb . Measurements showed that the activity distribution over the surface of the source possesses axial symmetry with a flat maximum at the center of the source [27]. The optimum distance from the boat to the backing was 5 mm. In this way, we achieved a fairly high evaporation efficiency of 25–30%. The fraction of retained ^{83m}Kr in the source was 15–20%. For a closer distance, the efficiency and retained fraction increased, but the intensity of the “no-loss peak” fell considerably. The material and parameters of the above backings had no appreciable effect on the quality of the sources.

It should be noted that even when one would think that the procedures for preparing the sources were identical, in particular the same initial ^{83}Rb activities in the boat, sources with the same properties were not obtained: the structures of the measured conversion electron spectra differed. This confirms the extreme complexity of the problem. The properties of the ^{83}Rb source are obviously very sensitive to the preparation procedure and complete control of this was not possible.

Measurements on the ESA12 electrostatic spectrometer. Investigations of the time stability of the energies of conversion electrons, which arise in the decay of ^{83}Rb , require continuous measurements of electron spectra over a period of many weeks. They were started on the ESA12 electrostatic spectrometer at the Institute of Nuclear Physics Řež. Since this spectrometer was unable to measure the K -32 conversion line, we chose the conversion L_1 -line of the 9.4 keV nuclear γ -transition with an energy of 7.5 keV and a natural width of 3.72 ± 0.19 eV for the investigation [8]. It is necessary to make measurements with an instrument resolution of several electronvolts in order to distinguish the “no-loss peak” in the conversion line. To obtain a statistical error of 30–10 meV in the value of the energy of this line, measurements of the corresponding spectrum were carried out for 3–10 days, depending on the source intensity. In Fig. 1, we show, as an example, the L_1 -9.4 conversion line from a source on an aluminum backing, measured with an instrument resolution of 3 eV in steps of 0.5 eV. On the left of the intense “no-loss peak” one can see a weak broad peak of electrons which have suffered inelastic scattering in the source – “a characteristic energy loss peak.” A spectrum of electrons which have lost energy in shake up/off processes [13] is also superimposed on it. In this connection, it becomes noticeable that, when making measurements with an instrument resolution of tens of electronvolts and/or using a source with a greater effective thickness, the two components of the spectral line will “dissolve” into a single line or the “no-loss peak” will be considerably suppressed, and this prevents an accurate determination of the spectral line energy.

Long-term measurements from 2 to 6 months were carried out using five different sources. In four cases, the sources were extracted several times from the spectrometer in the course of the measurements, stored in air, and then again placed in the spectrometer. We used two different accurate high-voltage dividers in the measurements [28], which, in combination with highly accurate 8508A voltmeters (made by the Fluke Company) enabled us to determine the voltage with a relative error of 10^{-6} .

Fig. 2. Behavior of the energy of the L_1 -9.4 conversion line with time (the measurement conditions are the same as in Fig. 1). The statistical errors in the values of the energy are shown. The continuous line is a linear approximation. The value of the energy drift per month Δ is corrected for the shifts in the divider and the voltmeter.

Fig. 3. The K -32 conversion line, measured on the MAC-E-Filter integral spectrometer (Mainz) with an instrument resolution of 0.9 eV (^{83}Rb source No. 28).

In Fig. 2, we show, as an example, the behavior of the measured values of the energy of the L_1 -9.4 conversion line with time for an ^{83}Rb source on an aluminum backing. The initial activity of the source was 6.6 MBq, and the retained fraction of ^{83m}Kr in the source was 12%. The source was kept continuously in the vacuum chamber of the spectrometer with a residual pressure of the order of 10^{-8} mbar.

For all the sources investigated, the measured value of the energy of the L_1 -9.4 conversion line increased with time (like the graph shown in Fig. 2), and this change can be described using a linear function. After taking into account the corrections for the shift in the dividing ratio of the two high-voltage dividers $((-2.4 \pm 0.4) \cdot 10^{-6}$ and $(-4.0 \pm 0.4) \cdot 10^{-6}$ per month) and the correction for the drift of the voltmeter readings $((0.12 \pm 0.04) \cdot 10^{-6}$ per month) the drift in the measured values of the energy of the L_1 -9.4 conversion line for the ^{83}Rb sources investigated lay in the range $(2.3\text{--}12) \cdot 10^{-6}$ per month. It should be noted that the energy of the L_1 -9.4 line, measured immediately after the ^{83}Rb source was placed in the spectrometer, differed for different sources. This was observed when different procedures were used to prepare the sources and when these procedures, apparently, were identical. The maximum difference in the values of the measured energies of the L_1 -9.4 conversion line was 0.7 eV.

Measurements on the MAC-E-Filter Spectrometer. For measurements in Mainz, we used two sources of ^{83}Rb (marked as Nos. 28 and 29) on carbon foil. The initial activity of ^{83}Rb in the sources was 5.0 MBq (No. 28) and 4.9 MBq (No. 29), while the retained fractions of ^{83m}Kr were 19% and 11%, respectively.

Fig. 4. Behavior of the energy of the K -32 conversion line of source No. 28 with time, investigated on the spectrometer in Mainz over a period of 80 days. The regions with the most stable energy are indicated and the corrected values Δ of the energy drift are given in relative units per month.

Fig. 5. Behavior of the energy of the K -32 conversion line, measured on the Mainz spectrometer with two sources of ^{83}Rb , namely, No. 28 and No. 29, in the 159–196 day period from the beginning of measurements on source No. 28. The linear dependences of the energy on time are represented by the continuous lines; the corrected values of Δ of the energy drift are given in relative units per month.

Two days after source No. 28 was placed in the spectrometer, we made measurements of different conversion lines under different conditions over a period of 80 days. The high transmission of the spectrometer and the efficiency of its electron detector enabled us to measure the necessary part of the spectrum of the K -32 conversion line for three hours to achieve a statistical error in determining its energy at a level of 10 meV. An example of the measured spectrum of the K -32 conversion line is shown in Fig. 3. The measurements were related to the investigation of the energy stability of the K -32 line. In Fig. 4, we show the behavior of the values of the K -32 energy obtained over the whole measurement period covering 80 days. The data were corrected taking into account the drift of the voltmeter readings and the dividing ratio of the special high-voltage divider, amounting to $(-0.30 \pm 0.05) \cdot 10^{-6}$ and $(0.6 \pm 0.1) \cdot 10^{-6}$ in relative units per month, respectively. On the 30th day after the beginning of the measurements, the energies of all the conversion lines investigated abruptly increased by 0.9 eV. This jump

may be caused by two phenomena – an increase in the temperature of the high-voltage divider as a result of breakdown in its cooling system or a spontaneous disconnection of one of the two units supplying the Earth's magnetic field compensation coils. As yet we have been unable to establish the true reason for this sudden jump in the energies of the conversion lines. A detailed inspection of the special divider after the measurements showed that the first reason was the most probable.

As follows from Fig. 4, at the beginning of the measurements the values obtained for the energy of the *K*-32 conversion line of source No. 28 increased, and after a certain time reached “saturation.” On the 31st day, when the technical troubles were eliminated, the energies of the conversion lines did not recover to the initial values. During the period of 31–50 days, the energy of the *K*-32 line decreased smoothly, and in the period from the 60th and 70th days a state of stability began. The smooth changes in the energy of the *K*-32 line are probably due to a change in the work function of the principal electrode of the spectrometer and/or the stability of the ^{83}Rb source. At the end of 70 days the source was heated to a temperature of about 70°C for 10 hours in a separate part of the spectrometer without disturbing the vacuum in order to stabilize the physicochemical composition of the deposited ^{83}Rb layer. After heating, the energies of all the measured conversion lines decreased by 25 meV. In order to compensate for the reduction in the intensity of source No. 28 as a result of radioactive decay of the ^{83}Rb , the electron source and detector of the spectrometer were shifted in opposition to one another, i.e., closer to the corresponding magnets providing the magnetic field of the spectrometer [23]. After this, the intensity of the *K*-32 line recorded by the detector doubled, and its measured energy decreased by 0.36 eV (see the 72–75 day period in Fig. 4). The fact that there is a change in the energy confirms that the adjustment of the position of the ^{83}Rb source and the electron detector in the spectrometer is insufficiently accurate. Despite all these problems, which accompanied the measurements of ^{83}Rb source No. 28, assuming the drift of the energy of the *K*-32 conversion line to be linear, the value determined in the stable behavior regions (the 20–30, 60–70, and 75–83 day intervals) satisfies the energy stability requirement of the *K*-32 conversion line, mentioned at the beginning of this paper, in the range $\pm 1.5 \cdot 10^{-6}$ over a period of one month.

After 84 days of measurements, source No. 29 was added to source No. 28 in the spectrometer in such a way that, using a special outside holder (without disturbing the vacuum in the spectrometer) we could place either of the two sources in the working position. A suppression voltage of +100 V was then applied to the non-operating source in order to shift the spectrum of the conversion electrons emitted by it into the region of the low-energy “tail” of the conversion line of the source being measured, which was at zero potential. In Fig. 5, we show values of the energy of the *K*-32 line of sources Nos. 28 and 29 in the 159–196 day period of measurements when the spectrometer was in a stable state. No problems arose with the vacuum in the spectrometer chamber, and the residual pressure decreased smoothly from $4 \cdot 10^{-10}$ to $3 \cdot 10^{-10}$ mbar. From the linear approximation of the behavior of the values of the energy of the *K*-32 line during this period, we obtained the values of its drift per month $(0.3 \pm 0.5) \cdot 10^{-6}$ and $(0.5 \pm 0.8) \cdot 10^{-6}$ for sources Nos. 28 and 29, respectively, corrected taking into account the shifts of the dividing ratios and the readings of the voltmeter. Both results were within the limits of the relative instability of $\pm 1.5 \cdot 10^{-6}$ per month required for the KATRIN project.

Conclusion. We have presented the results of an investigation of the time-stability of the values of the energies of the L_1 -9.4 and *K*-32 conversion lines of the decay of ^{83}Rb using the essentially different electron spectrometers at the Institute of Nuclear Physics of the Academy of Sciences of the Czech Republic and at the University of Mainz, respectively. We used solid-state sources of ^{83}Rb , prepared by evaporation in a vacuum on different backings, for the measurements. An analysis of the data obtained under relatively stable operating conditions of the spectrometer in Mainz indicates that ^{83}Rb sources, prepared by vacuum evaporation, are applicable in principle for monitoring the stability of the energy scale of the electron spectrometer employed in the KATRIN project with a required error of ± 60 meV (i.e., with a relative error of $\pm 3 \cdot 10^{-6}$) over a period of two months. It also follows from the investigations that, for reliable monitoring of the stability of the energy scale of the electron spectrometer used in the KATRIN project, it is necessary during the measurements to ensure that the work function of the monitoring spectrometer remains unchanged. Instability of the work function is mainly due to the composition of the residual gases in the spectrometer chamber. This is confirmed by the fact that the drift of the L_1 -9.4 line obtained in the spectrometer in Řež is, on average, an order of magnitude greater than the drift obtained in Mainz. Investigations are being continued using sources prepared by implantation of ^{83}Rb ions in various backings.

This research was carried out with the assistance of the Czech Academy of Sciences (Contract No. ASCR IRP AV0Z 10480505), with the support of the Grant Agency of the Czech Republic (Contract No. 202/06/0002) and the Ministry of

Education of the Czech Republic (Contracts Nos. LA318 and LC07050), and also with financial support from the German Agency DFG and BMBF (Universities of Mainz and Münster).

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