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Calibration of the isomer shift of the Mössbauer isotopes ^{127}I and ^{129}I[1])

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Abstract. The influence of the chemical environment on the electron capture decay rate of ^{125}I was measured with a differential ionization chamber. Together with Mössbauer data the isomer shift calibration constants for ^{127}I and ^{129}I were obtained. The change of the nuclear radius during the Mössbauer transition in ^{129}I was found to be $(\Delta R/R)_{129} = +(7.2 \pm 1.7) \times 10^{-4}$. The half life of ^{125}I is $T_{1/2} = 59.666 \pm 0.016 d$.

I. Introduction

The isomer shift δ, as measured by the Mössbauer effect involves the product of an atomic quantity, the electron density change at the nucleus $\Delta\rho(0)$ which is influenced by the chemical environment, and a nuclear quantity, the change of the nuclear radius ΔR during the Mössbauer transition:

$$\delta = \frac{4\pi}{5} Ze^2 R \Delta R \Delta\rho(0) = C\Delta\rho(0). \tag{1}$$

In order to evaluate $\Delta\rho(0)$ from isomer shift data, one has to know the "calibration constant" C of the corresponding Mössbauer transition. In the present paper an experiment to determine C (or ΔR) based on the measurement of the influence of the chemical environment on a long-lived electron capture decay is described.

II. The Mössbauer isotopes ^{127,129}I and the decay of ^{125}I

The isomer shifts of ^{127,129}I have been extensively measured. They show the systematics indicated in Fig. 1. An increase of δ in ^{127}I (δ_{127}) corresponds to a decrease in δ_{129}. From this observation and equation (1) it follows that the ΔR values of the two isotopes have different signs. An early analysis of the isomer shift data [1] revealed

$$\frac{(\Delta R/R)_{127}}{(\Delta R/R)_{129}} = -0.73 \pm 0.03. \tag{2}$$

The slope of the line plotted in Fig. 1 corresponds to this ratio.

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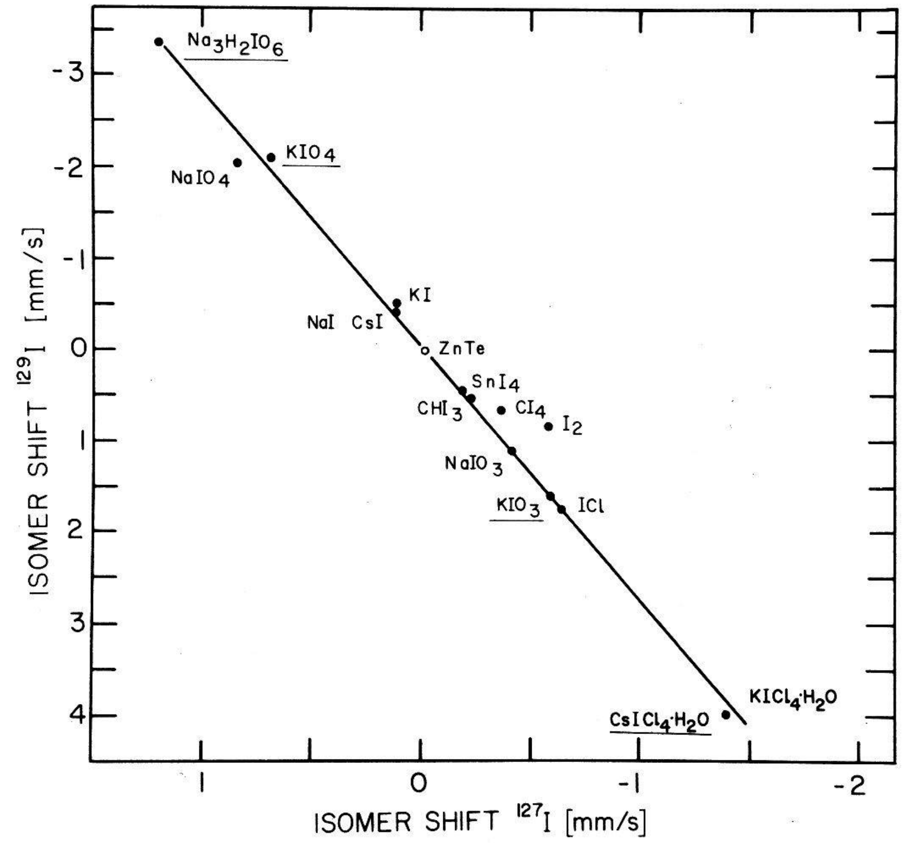

Figure 1
Plot of isomer shifts of isoelectronic pairs of ^{127}I and ^{129}I compounds.

For the absolute $(\Delta R/R)$ values only estimates based on calculated electron densities exist

$$\begin{aligned}(\Delta R/R)_{129} &= +4.2\times10^{-4}\ [2]\\ (\Delta R/R)_{129} &= +5.0\times10^{-4}\ [3].\end{aligned} \tag{3}$$

From the scattering of the various reported $\Delta R/R$ values the error is estimated to be at least 30%.

An experimental determination of the calibration constant would therefore be very desirable. In a continuation of earlier isomer shift calibration measurements [4, 5] we tried to determine $\Delta R/R$ via the influence of the chemical environment on the radioactive lifetime. Since a determination of $\Delta R/R$ via the lifetime of the converted Mössbauer transitions seemed inpractical, a method based on the chemical influence on the electron capture decay rate was used.

The decay probability for electron capture is given by [6, 7]

$$\lambda = \text{const} \sum_i \rho_i(0)(E_0 - E_i)^2, \tag{4}$$

where E_0 is the transition energy and E_i the binding energy of the captured electron. The relative decay constant variation due to the different electronic structures can thus be written as

$$\frac{\Delta\lambda}{\lambda}=\frac{\left[\sum_i \rho_i(0)(E_0-E_i)^2\right]_{II}-\left[\sum_i \rho_i(0)(E_0-E_i)^2\right]_I}{\left[\sum_i \rho_i(0)(E_0-E_i)^2\right]_I}. \tag{5}$$

For ^{125}I equation (5) reduces to

$$\left(\frac{\Delta\lambda}{\lambda}\right)_{125}=1.553\,\frac{\Delta\rho_{\text{tot}}(0)}{\rho_{\text{tot}}(0)}, \tag{6}$$

where $E_0=147$ keV [8] and $E_{1s}=33.2$ keV were used. This expression combined with (1) gives

$$\left(\frac{\Delta R}{R}\right)_{129}=\frac{5}{4\pi}\,\frac{\delta_{129}1.553}{Ze^2R_{129}^2\rho_{\text{tot}}(0)}\left(\frac{\lambda}{\Delta\lambda}\right)_{125}. \tag{7}$$

Since the lifetime measurement on ^{125}I determine an absolute value of $\Delta\rho(0)$ characteristic for any iodine isotope, the isomer shift and the lifetime experiment do not need to be done on the same isotope. The total electron density at the nucleus was extrapolated from Mallow et al. [9] to be

$$\rho_{\text{tot}}(0)=2.65\times10^5\,a_B^{-3}=1.78\times10^{36}\ \text{m}^{-3}. \tag{8}$$

In the experiment described here the isomer shift values δ_{129} were taken from the literature. The corresponding $\Delta\lambda/\lambda$ values were measured with a differential ionization chamber first described by Rutherford [10].

III. The differential ionization chambers

Small differences in λ occurring between two chemically different sources of the same isotope were first measured by Segrè [11] and Daudel [12] with the help of the differential ionization chamber. The chamber used in the present experiment is shown in Figs. 2, 3. The ionization chamber with the positive high voltage measures the positive ionization current

$$I_+(t)=I_+(0)\exp(-\lambda t). \tag{9}$$

The current in the negative chamber is

$$I_-(t)=I_-(0)\exp(-(\lambda+\Delta\lambda)t). \tag{10}$$

Thus, in a good approximation ($\Delta\lambda \ll \lambda$), the current measured by the electrometer is

$$\Delta I(t)=\exp(-\lambda t)(\Delta I(0)-I_-(0)\Delta\lambda t). \tag{11}$$

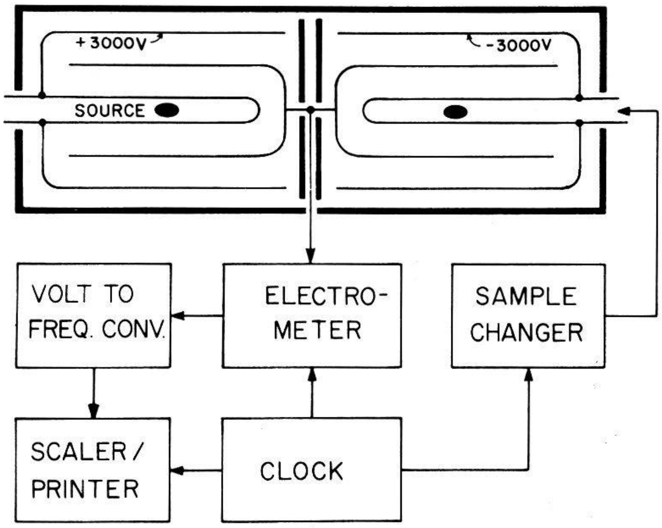

Figure 2
Diagram of the differential ionization chamber.

In the actual measurement the source strength and therefore the currents are equal within 5×10^{-3} and one may write

$$\Delta I(t)\exp(\lambda t)=\Delta I(0)-I(0)\Delta\lambda t. \tag{12}$$

The difference current multiplied for convenience by $\exp(\lambda t)$ increases linearly with time with a slope proportional to $\Delta\lambda$.

In order to measure lifetime differences in the order of 1×10^{-4} with an accuracy of a few percent, the chamber had to be built with high precision. Some of the mechanical characteristics are: The two parts of the chamber were built

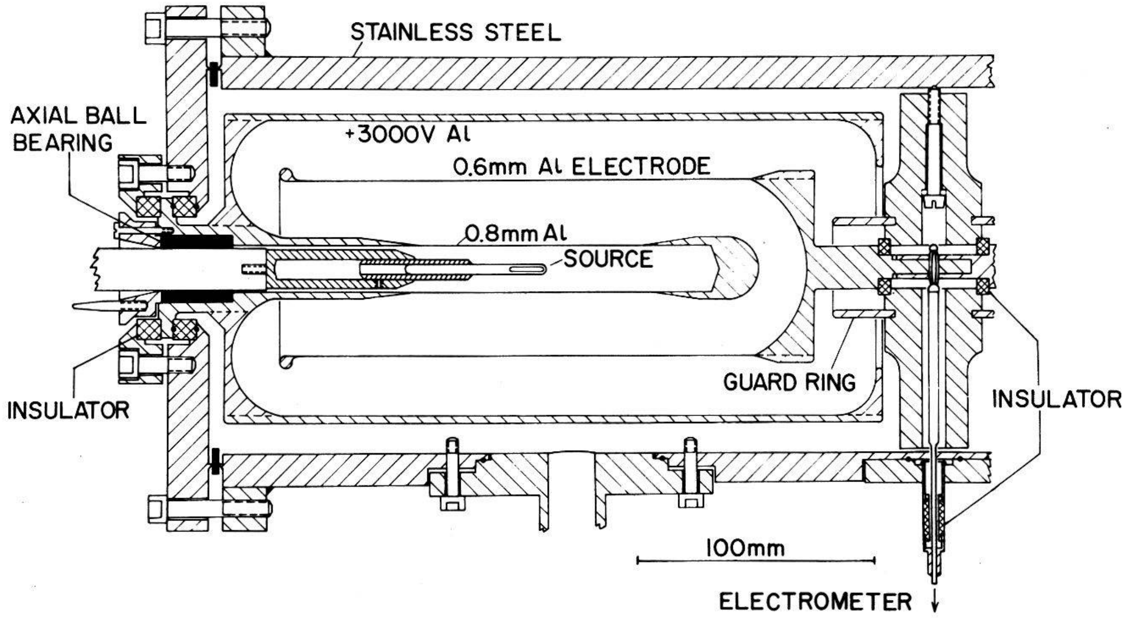

Figure 3
Cross section of the left half of the differential ionization chamber. The chamber is filled with 15 atm of argon. The sources are exchanged pneumatically.

symmetrically with an interconnected fillgas volume. The aluminium electrodes were machined with high accuracy to be thin and symmetrical. For better conduction (surface Al_2O_3 layer) they were first 2 μm copper- and then 2 μm gold plated. Only metal seals were used throughout. As insulators on both sides lapped Al_2O_3 rings ($\rho > 10^{16}\ \Omega$ cm) were used. The collecting electrodes were insulated from ground by two high resistance glassed ceramic rings. The electrometer was connected via an insulated feedthrough ($R > 10^{17}\ \Omega$) to the electrodes. Each half chamber had two active volumes on both sides of the collecting electrode. Care was taken that the electric field in the active volume was fairly uniform, especially that there were no regions with low fields and no corners with high fields. In the assembly great care on cleanliness was observed. The stainless steel parts were electro-polished. All parts were cleaned with aceton, warm soap solution and distilled water. The assembly done, the chamber was evacuated for several days to $<5 \times 10^{-6}$ Torr at a temperature of 150°C. The chamber was carefully checked for leaks both evacuated and under the operating pressure of 15 atm using a He leak detector. After this the chamber was several times flushed and evacuated and then filled with 99.9999% pure argon (L'air Liquide) with an overpressure of 15 atm. Within experimental errors of $<2\%$ no decrease of the gas pressure could be detected over a period of 2 y. The chamber including the critical parts of the electronics was then mounted in a temperature-stabilized cabinet situated in the bomb-shelter of the Institute. The chamber temperature recorded during the whole measurement was 22.00 ± 0.05°C. One of the sources was mounted fixed in one half of the chamber. 6 additional sources mounted on a sample changer were pneumatically changed every two hours during the total running time of the experiment. The reproducibility in the positioning of the sources in the chamber was better than 0.01 mm.

For the current measurements the vibrating reed electrometer CARY 401 was used in the "rate of charge" mode. By this method a calibrated charge collecting capacitor $C_C = 19.87$ pF gets charged by the current to be measured. A simplified diagram (Fig. 4) indicates the essential features of this measuring mode. With a collecting time of typically 1 h the method allows measurements in the range of 10^{-14}–10^{-17} A. To extend the range to 10^{-12} A an additional high insulating glass capacitor of 2000 pF connected between the input and the

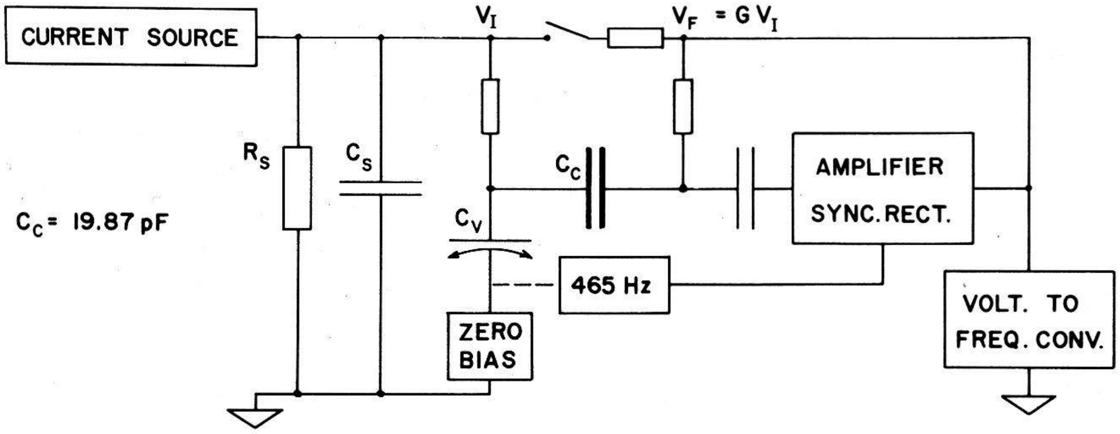

Figure 4
Diagram of the current measurements by the rate of charge method with a vibrating reed electrometer.

feedback terminal was used in the initial phase of the experiment. By the use of the "guarded" input circuit errors due to the stray capacitance C_S, insulator leaks R_S, and changes of the electric field strength due to the charging of the electrodes are reduced by the open loop gain G of the instrument. The circuit functions are as follows: The current I to be measured charges the charging capacitor C_C, the vibrating reed capacitor C_V, and the stray capacitance C_S (capacitance of chamber and connectors)

$$Q = V_I(C_S + C_V) + C_C(V_I + V_F). \tag{13}$$

With the gain $G = V_F/V_I \cong 1000$ one may write

$$Q = V_F\left[C_C + \frac{C_S + C_V + C_C}{G}\right] \cong V_F C_C. \tag{14}$$

V_F is measured at the output of the instrument. The current to be measured is

$$I = \frac{dQ}{dt} = C_C \frac{dV_F}{dt}. \tag{15}$$

The voltage V_F was measured via a voltage to frequency converter whose output was integrated by a scalar from t to $t+\Delta t$ and from $t+\Delta t$ to $t+2\Delta t$ with the corresponding number of counts N_1 and N_2. From these two numbers the current is calculated to be

$$I = \frac{(N_2 - N_1)C_C}{k(\Delta t)^2}, \tag{16}$$

where k is the conversion rate of the voltage to frequency converter. The time intervals chosen were $\Delta t = 1800$ s. Each of the six sources mounted on the sample changer was measured twice a day. The sequence which was controlled by a central clock was: change of sample, reset electrometer, wait about 1 hour for the ionization current to stabilize, start of the measurement of N_1 for 30 min, print out and reset of scaler, measurement of N_2, print out and reset of scaler, change of sample etc.

All the sensitive parts of the electronics were temperature-stabilized to within 0.05°C. Great care was taken in proper grounding. As an additional precaution the line voltage was stabilized and filtered.

The current-voltage characteristic of the chamber measured with an ^{241}Am source placed in one half of the chamber has from 500 to 3000 V at 2.5×10^{-10} A a slope of 1.7×10^{-15} A/V. The current reaches 90% of the saturation value already at 30 V. Drifts due to changes of the high voltage power supplies (which were temperature and line voltage stabilized) are certainly negligible. A difference in the sensitivity of the two chamber halves of about 1% was corrected by inserting a thin-walled plexiglass tube (~0.5 mm) into the more sensitive chamber half. Once a week the measurements were interrupted to determine the individual source strength. The corresponding current was roughly 10^3 times larger than the difference currents ($\sim 10^{-10}$ A). This current was measured by the guarded high-resistance-leak method, using the built-in resistor $R_i = 10^{10}\ \Omega$. The current is then simply

$$I = \frac{V_I}{R_i} + \frac{1}{G}\frac{V_I}{R_S},$$

where the current through insulator leaks R_S is again reduced by the open loop gain.

The stability of the chamber including the electrometer was checked by the measurement of an ^{241}Am source once a week. After corrections for the ^{241}Am decay ($T_{1/2} = 458$y), the measured current of 2.523×10^{-10} A was constant within 2×10^{-13} A over a period of one year. From this measurement it was concluded that the long term stability of the electronics and the chamber was better than 10^{-3}. Nevertheless, in the determination of the absolute lifetime all the measurements were normalized with respect to ^{241}Am.

IV. Preparation of the sources

It is very important that the sources are radioactively pure, because otherwise a critical error might be introduced due to the energy-dependent absorption in the source material. The ^{125}I was supplied by the New England Nuclear Corporation (Boston) in a carrier-free solution of I_2 in CCl_4. The specified purity was >99% with a maximum of 0.05% ^{126}I ($T_{1/2} = 13d$). The γ-spectrum taken with a Ge (Li) detector at the beginning of the chemical preparations showed no ^{126}I lines within an error of 10^{-3}. During the preparations for the actual measurements ($\sim 60d$) a possible admixture of ^{126}I would be reduced to $\leqslant 5 \times 10^{-5}$. The starting activity was 100 mCi in 4 ml CCl_4. This solution was split into 5 equal parts. To each of these parts 2 ml ether and 100 mg stable I_2 was added. By evaporating the solutions at 75°C elemental I_2 was formed.

The chemical compounds were chosen according to the following criteria: (1) For statistical reasons the range of isomer shifts should extend over a large region. (2) The compounds should be chemically stable and not volatile. (3) Since the radiochemistry had to be done in special glove boxes with the appropriate filtering system at the Swiss Reactor Institute (EIR), the recipes had to be simple and adaptable to small quantities. (4) Since different shipments might contain various amounts of impurities, the starting point for all the compounds had to be the same source material. The compounds chosen were: $Na_3H_2IO_6$ ($\delta_{129} = -3.4$ mm/s), KIO_4 (−2.1), KIO_3 (+1.6), and $CsICl_4$ (+4.0) (see Fig. 1).

$Na_3H_2IO_6$. 125 mg $NaClO_3$ were dissolved in 5 ml H_2O at 45°C and 2 μl HNO_3 (conc.). To this solution the 100 mg radioactive I_2 described above was added. After a short time at 60°C the I-colour disappeared indicating the end of the reaction to IO_3^-. To this solution 140 mg NaOH in 1 ml H_2O was added. Then Cl_2-gas was circulated through the boiling liquid. After neutralization NaOH base was added (pH = 11). The hot solution was then stirred for 20 h. The $Na_3H_2IO_6$ precipitate formed was twice washed with cold water and then dried at 110°C.

KIO_3. The radioactive I_2 was dissolved with 230 mg $KClO_3$ in 0.7 ml H_2O, and 2 μl HNO_3 (conc.) at 45°C. The solution was then evaporated.

KIO_4. To the KIO_3 solution containing 100 mg I_2 195 mg KOH and 1 ml H_2O was added. This solution was then brought to 112°C, where Cl_2 was put through similarly to the preparations of $Na_3H_2IO_6$.

$CsICl_4$. To a solution of 111 mg CsCl, 1.7 ml H_2O, and 0.6 ml HCl (conc.) 100 mg I_2 was added. This solution was heated to 60°C where for ~4 min Cl_2 was circulated through the liquid. The precipitating $CsICl_4$ was then dried for 4 days over P_2O_5 in an dry-box.

All the sources were checked by the X-ray powder method on their structure and purity. It is very important that the sources be protected against mechanical and chemical changes during the experiment. The sources were put into small glass vials of ~2 mm inside diameter and were equalized with the help of the chamber to better than 0.2%. The source material was then covered with fine ground and dried quartz powder. After pressing the powder with a stopper to prevent mechanical movements during the measurements, the glass vials were sealed off. In the cooling process a pressure developed from the glass on the quartz powder. The sealed sources were then mounted on the high precision rods of the sample changer.

For calibration purposes 1.9 mCi ^{241}Am was used. The ^{241}Am obtained from Amersham (England) was in the form of the industrially used 'alpha foil'; ^{241}Am dispersed in a gold matrix was sandwiched between a backing of 0.2 mm silver and a front covering of 0.003 mm gold alloy. This foil was bent around a cylinder and also mounted on a rod of the sample changer. From the ^{241}Am source only the 60 keV γ-rays and the LX-rays (22 keV and less) were used. The α-particles would not enter the sensitive volume of the ionization chamber.

V. Measurement of the ^{125}I lifetime

Once a week the $\Delta\lambda/\lambda$ experiment was interrupted for approximately 6 h in order to determine the absolute source strength of one of the sources ($Na_3H_2IO_6$). These current measurements were done automatically using the high resistance leak method. The output of the electrometer-voltage-to-frequency converter was integrated over 20×3 min. Special care was taken in recording the correct time. During these $I(t)$ runs drifts in the sensitivity of the apparatus could be eliminated by measuring ^{241}Am for comparison. All the measurements were normalized to the lifetime corrected Am-current. The result of a section of these measurements is shown in Fig. 5. Since the range of the electrometer had to be changed a few times during the experiments, the corresponding sections were fitted individually. The half life of ^{125}I determined by a weighted fit of the data points is

$$T_{1/2} = 59.666 \pm 0.016 d. \qquad (17)$$

The error includes the scattering resulting form the fitting of the individual setions. This value may be compared with the value given in the 'Nuclear Data Sheets' [13] $T_{1/2} = 60.14 \pm 0.11 d$, which was obtained by the weighted average of

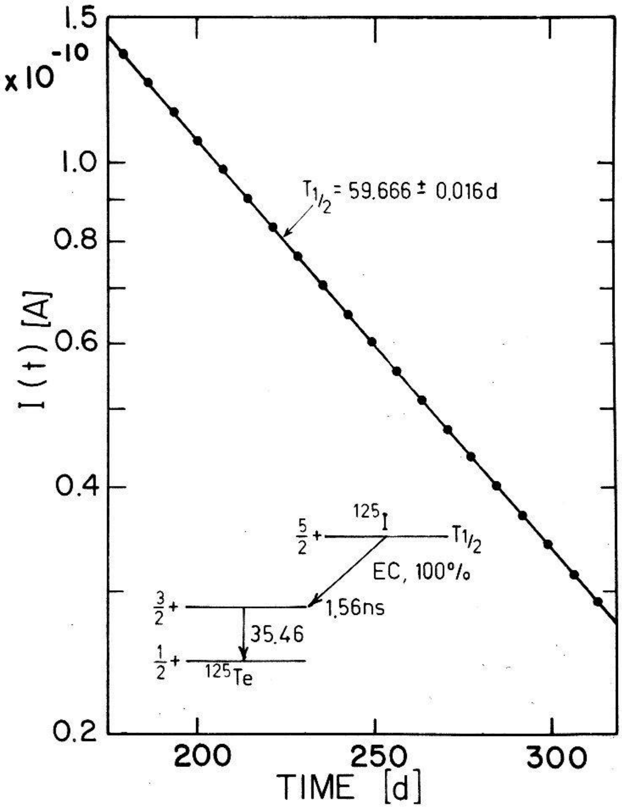

Figure 5
Section of the ^{125}I decay curve. The errors of the experimental points are approximately a factor 10 smaller than the points shown.

the values $60.25 \pm 0.06d$ (H. Lentz, 1964), $59.83 \pm 0.11d$ (S. C. Anspach, 1965), $59.89 \pm 0.18d$ (F. Lagoutine, 1968), and $60.18 \pm 0.17d$ (S. T. Emerson, 1971).

VI. Measurements of the decay constant differences

A $Na_3H_2IO_6$ source (source S0) was permanently installed in one half of the ionization chamber. The sources in the other half were changed every 2 h. These sources with their isomer shifts are [14]

$$\begin{array}{lll} \text{S0, 1, 2:} & Na_3H_2IO_6, & \delta_{129} = 0\ \text{mm/s} \\ \text{S3} & : KIO_4 & , \delta_{129} = 1.5\ \text{mm/s} \\ \text{S4} & : KIO_3 & , \delta_{129} = 5.0\ \text{mm/s} \\ \text{S5} & : CsICl_4 & , \delta_{129} = 7.43\ \text{mm/s} \end{array} \tag{18}$$

In the evaluation the five sources S1–5 were compared with each other. Source 0 served just as a reference source in the measurement. This source was not included in the evaluation due to periodic and coherent drifts of the five current measurements which seemed to indicate that the symmetry of the chamber changed with a diurnal and possible additional longer periods. May be that these drifts were related to the operation of the Institute's Van de Graaff accelerator.

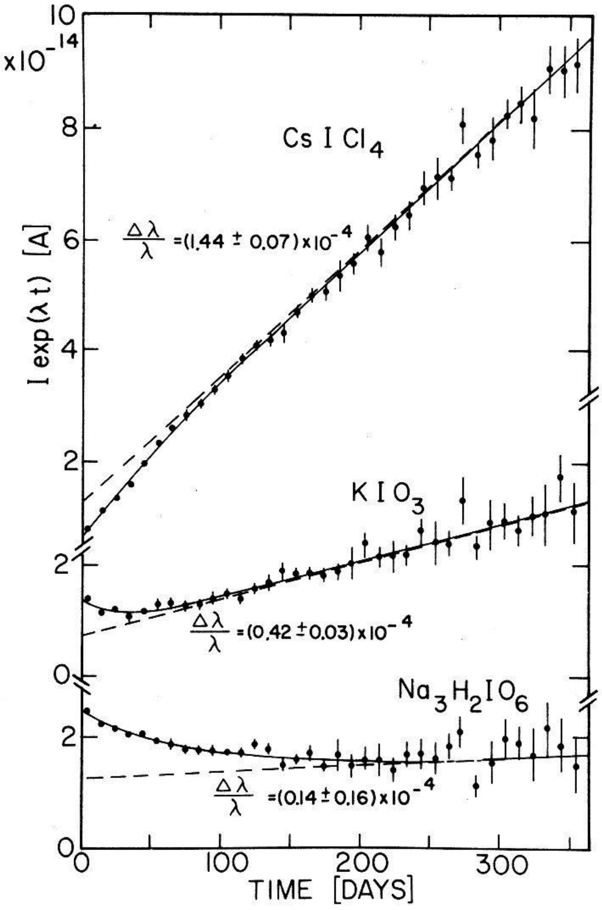

Figure 6
Life time corrected difference current of the differential ionization chamber plotted as a function of time. The three sources were measured relative to $S1 = Na_3H_2IO_6$.

$\Delta\lambda$ was evaluated according to equation (12). The difference current corrected for the decay

$$y = \Delta I_{ij}(t)\exp(\lambda t) = \Delta I_{ij}(0) - \Delta\lambda_{ij} I(0)t \qquad (19)$$

is plotted for some source combinations in Fig. 6. Each of the points indicated is the average over a 10 days measuring period (20 individual measurements). The slope of the lines is proportional to $\Delta\lambda_{ij}$. The current $I(0)$ extrapolated from the life time measurements to an effective time zero was determined to be $I(0) = 1105.5 \pm 0.2$ pA. The absolute value of $I(0)$ is of no importance here, since all the measurements ($I(t)$, $\Delta I_{ij}(t)$) will contain the same relative error. As shown in Fig. 6 a sample dependent exponential behaviour was found superimposed on the expected linear increase $y(t)$. This unexpected exponential behaviour is unambiguously explained by slow chemical reactions and related mechanical motions of the source material within the sealed source vials. By the movement of the source material the self-absorption in the sources will change, which results in changes of the measured source strength. Some moisture or chemical impurities in the source material may be responsible for these slow reactions. It seems reasonable to assume that these chemical effects follow an exponential law. The data for the various source combinations were therefore fitted to

$$y = a + bt + c \exp(-\alpha t). \tag{20}$$

In the evaluation of $\Delta\lambda/\lambda$ a quality factor for the 10 difference measurements was introduced. This weight factor which varied between 2 and 8 was chosen to be approximately inversely proportional to the exponential admixture (e.g. source combination S1–S2 was assigned a weight of 4, combination S1–S6 a weight of 8 – see Fig. 6). The weighted average for the difference measurements is (19)

$$(\Delta\lambda/\lambda)_{125}/\delta_{129} = (1.68 \pm 0.20) \times 10^{-5} \text{ s/mm}, \tag{21}$$

where the error was increased by a factor of 2 in order to include uncertainties due to the somewhat arbitrarily chosen weight factors. This result corresponds to (equation (7), $R_{129} = 6.12 fm$)

$$(\Delta R/R)_{129} = +(6.68 \pm 0.88) \times 10^{-4}. \tag{22}$$

In order to account for the known correlations between the various possible difference currents the total set of data was fitted to

$$y = \sum_{i=j}^{4} \sum_{j=i+1}^{5} \{(A_i - A_j) + Ct(\delta_i - \delta_j) + (B_i - B_j) \exp(-\alpha t)\}. \tag{23}$$

For this fit 11 independent parameters are required. The exponential factor $\exp(-\alpha t)$ was assumed to be the same for all the sources. The weight of the individual points $y_{ij}(t)$ corresponded to the actually measured errors, no quality factor was introduced. Two fits were tried, one with 4 (Fig. 7) and one with 5 sources (source S3 was somewhat inferior). The result is:

$$\begin{aligned} &\text{4 sources: } (\Delta\lambda/\lambda)_{125}/\delta_{129} = (1.05 \pm 0.09) \times 10^{-5} \text{ s/mm} \\ &\text{5 sources: } (\Delta\lambda/\lambda)_{125}/\delta_{129} = (1.22 \pm 0.08) \times 10^{-5} \text{ s/mm.} \end{aligned} \tag{24}$$

The significant deviations between the two results indicate systematic errors. The assumption that the exponent α in equation (23) is the same for all the source is certainly not justified. It is believed that the evaluation of the result by the first method – which does not include correlations – is superior. For the final result we suggest

$$(\Delta\lambda/\lambda)_{125}/\delta_{129} = (1.55 \pm 0.35) \times 10^{-5} \text{ s/mm} \tag{25}$$

corresponding to

$$\begin{aligned} (\Delta R/R)_{129} &= +(7.2 \pm 1.7) \times 10^{-4} \\ C_{129} &= 0.38 \pm 0.09 a_B^3 \text{ mm/s.} \end{aligned} \tag{26}$$

An isomer shift δ_{129} of 1 mm/s corresponds to a change in the electron density of $2.65 a_B^{-3}$ (a_B = Bohr radius).

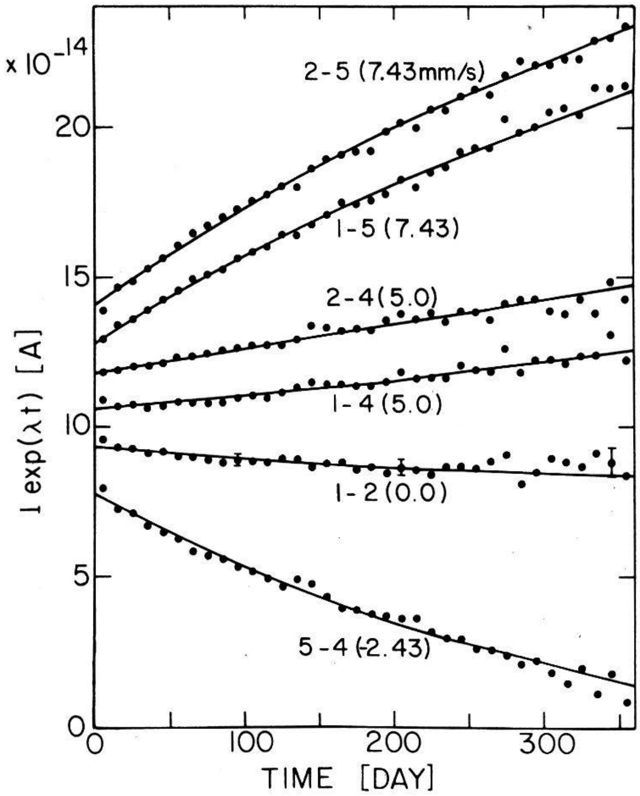

Figure 7
Life time corrected current for all combinations of the sources 1, 2, 4, and 5. The vertical scale for the various curves is arbitrarily shifted. The lines plotted are from a fit to equation (23). The relative isomer shifts are indicated, they are proportional to the slopes of the curves given.

This result must be compared with a recent determination of $(\Delta R/R)_{129}$ by conversion electron spectroscopy. The result obtained by Spijkervet and Pleiter [15] is

$$(\Delta R/R)_{129} = +(4.1 \pm 1.0) \times 10^{-4}. \tag{27}$$

In this difficult experiment the assumption had to be made that only the 5s conversion electrons are influenced by the chemical environment. Beside the source preparation a major difficulty in the experiment was the separation of the various electron lines in the β-spectrum.

Considering the various uncertainties in the two quite different experimental determinations of $\Delta R/R$ we suggest that it is probably fair to take the average of the two values as the present value for $\Delta R/R$.

$$(\Delta R/R)_{129} = +(5.6 \pm 1.6) \times 10^{-4}. \tag{28}$$

Together with the known ratio (2) one gets

$$(\Delta R/R)_{127} = -(4.1 \pm 1.3) \times 10^{-4}. \tag{29}$$

These values are in reasonable agreement with theoretical estimates based on electron density calculations (3).

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