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Determination of the Relative Electron Density at the Be Nucleus in Different Chemical Combinations, Measured as Changes in the Electron-Capture Half-Life of ^{7}Be

H. W. Johlige, D. C. Aumann, and H.-J. Born
Institut für Radiochemie, Technische Hochschule München, 8046 Garching b. München/Germany

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Differences in the decay constant of ^{7}Be in various states of chemical combination were measured using the differential-ionization-chamber technique. The results are:

$$\lambda(\mathrm{BeO})-\lambda(\mathrm{BeF_2})_{\mathrm{amorph}}=(1.130\pm0.058)\times10^{-3}\lambda(\mathrm{Be}),$$

$$\lambda(\mathrm{BeO})-\lambda(\mathrm{Be_4O(CH_3COO)_6})=(-0.724\pm0.057)\times10^{-3}\lambda(\mathrm{Be}),$$

$$\lambda(\mathrm{BeO})-\lambda(\mathrm{BeBr_2})=(1.472\pm0.063)\times10^{-3}\lambda(\mathrm{Be}),$$

$$\lambda(\mathrm{Be_4O(CH_3COO)_6})-\lambda(\mathrm{BeF_2})_{\mathrm{amorph}}=(1.852\pm0.082)\times10^{-3}\lambda(\mathrm{Be}),$$

$$\lambda(\mathrm{BeO})-\lambda(\mathrm{Be(C_5H_5)_2})=(0.795\pm0.074)\times10^{-3}\lambda(\mathrm{Be}),$$

$$\lambda(\mathrm{BeO})-\lambda(\mathrm{Be^{2+}(OH_2)_4})=(-0.374\pm0.077)\times10^{-3}\lambda(\mathrm{Be}),$$

$$\lambda(\mathrm{Be(C_5H_5)_2}-\lambda(\mathrm{Be^{2+}(OH_2)_4})=(-1.169\pm0.106)\times10^{-3}\lambda(\mathrm{Be}).$$

The decay constant of ^{7}Be is proportional to the electron density at the nucleus. These results can therefore be used to establish a scale for the relative electron density at the Be nucleus in different chemical combinations.

I. INTRODUCTION

^{7}Be undergoes orbital-electron capture with a half-life of 53 days. The capture probability, and hence the decay constant of ^{7}Be is proportional to the electron density at the nucleus and varies with the chemical state of the atom. Experiments to determine the difference in the decay rate in Be, BeO, and BeF_2 have been performed by groups in Berkeley,[1,2] Brookhaven,[3] and France.[4] The results of these experiments are listed in Table I, together with the results of our measurements.

The measurements presented in this paper were undertaken to learn more about the influence of electron rearrangement by bonding on the decay constant of ^{7}Be. The method might be useful in giving some insight into the electron density at the Be nucleus in different chemical surroundings. The results could be interpreted by comparison with the results of quantum-mechanical calculations of such electron densities.

II. EXPERIMENTAL PROCEDURE

A. Method of Measurement

The small differences $\Delta\lambda$ in the decay constants of two different beryllium compounds were measured by the differential-ionization-chamber method first used by Rutherford.[5] The two essentially identical ionization chambers were constructed similarly to those used by Bainbridge, Goldhaber, and Wilson,[6] who also discuss at length the method, the calculation, and the standard deviations. We measured the ion currents by means of a vibrating-reed electrometer, Cary Model No. 31 (Applied Physics Corporation, Manovia, California), utilizing the rate-of-drift method. Our apparatus is described in detail by Johlige.[7]

The two samples which are to be compared give rise to the ionization currents $J_1(0)$ and $J_2(0)$ at time $t=0$. At time t, the ionization currents will be

$$J_1(t)=J_1(0)\,e^{-\lambda t}\,,$$

$$J_2(t)=J_2(0)\,e^{-(\lambda+\Delta\lambda)t}\,,$$

where λ and $(\lambda+\Delta\lambda)$ are the decay constants of ^{7}Be in the two sources. The difference current at time t is

$$\Delta i(t)=J_1(0)\,e^{-\lambda t}-J_2(0)\,e^{-(\lambda+\Delta\lambda)t}\,,$$

$$\Delta i(t)=J_1(0)\,e^{-\lambda t}-J_2(0)\,e^{-\lambda t}+J_2(0)\,e^{-\lambda t}\times\Delta\lambda\times t\,,$$

provided that $\Delta\lambda \ll 1$.

When the sources are interchanged with respect to the chambers, the ionization currents are averaged and systematic errors minimized. The sensitivities of the two chambers were very nearly equal. The differences of the chamber sensitivities were measured to be close to zero, varying between 10^{-3} and 3×10^{-3} over the duration of a run and for the different pairs of the experiment. Good care has to be taken that no change in the position of the sources with respect to a chamber takes place after the measurements have been started.

To simplify the analysis, $\Delta i(t)$ is multiplied by $e^{\lambda t}$:

$$\Delta i(t)\,e^{\lambda t}=J_1(0)-J_2(0)+J_2(0)\times\Delta\lambda\times t\,.$$

This equation can be fitted by a least-squares analysis to the linear equation $y=a+bx$, where

$$a=J_1(0)-J_2(0)=\Delta i(0)$$

and

$$b=J_2(0)\Delta\lambda\,.$$

In the experimental run, the measurement of $J(t)$ versus t gives the quantities λ and $J_2(0)$, the initial current of the source with decay constant $\lambda+\Delta\lambda$. The value of λ and the measurement of $\Delta i(t)$ versus t are fitted to the least-squares solution of the straight line $y=a+bx$. The slope of this line is $b=J_2(0)\Delta\lambda$. Together with the value of $J_2(0)$ and λ, the desired result $\Delta\lambda=b/J_2(0)$ or $\Delta\lambda/\lambda=b/J_2(0)\lambda$ can be calculated.

B. Preparation of the Sources

The ^{7}Be was supplied by the New England Nuclear Corporation (Boston, Massachusetts) in carrier-free solution in 0.5*N* HCl. It is very important that the sources are radioactively pure, because otherwise a critical error may be introduced. The carrier-free ^{7}Be was therefore purified by the radiochemical procedure which has been described by Aumann.[8] The only observable radiation from ^{7}Be is a γ ray of 0.477 MeV. No particle radiations are emitted. After the purification, the β activity was less than 1 β ray in 10^7 0.477-MeV γ rays and therefore entirely negligible.

γ-ray spectroscopy with a Ge(Li) detector showed only the 0.477-MeV γ ray. To check further for the few radioactive contaminants which

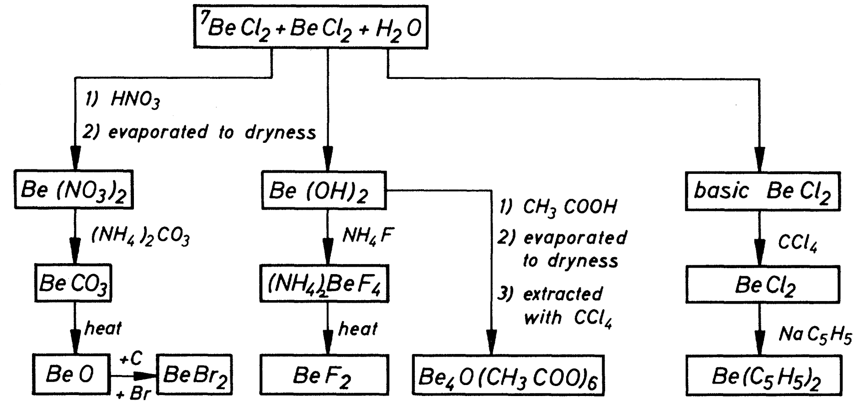

FIG. 1. Schematic diagram of the synthetic methods for the preparation of the beryllium compounds.

TABLE I. Differences in the decay constant of ^{7}Be in different chemical compounds.

Source pair	(Refs. 1,2) ($\times 10^{-3}$ λBe)	(Ref. 3) ($\times 10^{-3}$ λBe)	(Ref. 4) ($\times 10^{-3}$ λBe)	This work ($\times 10^{-3}$ λBe)
$\lambda(\mathrm{BeO}) - \lambda(\mathrm{BeF_2})_{\mathrm{hexag}}$	0.69 ± 0.03	0.609 ± 0.055		
$\lambda(\mathrm{BeO}) - \lambda(\mathrm{BeF_2})_{\mathrm{amorph}}$				1.130 ± 0.058
$\lambda(\mathrm{Be}) - \lambda(\mathrm{BeF_2})_{\mathrm{hexag}}$	0.84 ± 0.10	0.741 ± 0.047		
$\lambda(\mathrm{Be}) - \lambda(\mathrm{BeF_2})_{\mathrm{amorph}}$			1.2 ± 0.1	
$\lambda(\mathrm{BeO}) - \lambda(\mathrm{Be})$	-0.15 ± 0.09	-0.131 ± 0.051		
$\lambda(\mathrm{BeO}) - \lambda(\mathrm{Be_4O(CH_3COO)_6})$				-0.724 ± 0.057
$\lambda(\mathrm{BeO}) - \lambda(\mathrm{BeBr_2})$				1.472 ± 0.063
$\lambda(\mathrm{Be_4O(CH_3COO)_6}) - \lambda(\mathrm{BeF_2})_{\mathrm{amorph}}$				1.852 ± 0.082
$\lambda(\mathrm{BeO}) - \lambda(\mathrm{Be(C_5H_5)_2})$				0.795 ± 0.074
$\lambda(\mathrm{BeO}) - \lambda(\mathrm{Be^{2+}(OH_2)_4})$				-0.374 ± 0.077
$\lambda(\mathrm{Be(C_5H_5)_2}) - \lambda(\mathrm{Be^{2+}(OH_2)_4})$				-1.169 ± 0.106

do not emit β rays, an indirect procedure was used similar to the one described in Ref. 3 [a precipitation of $\mathrm{Fe(OH)_3}$ with $8N$ NaOH was made before the precipitation of ZnS and CuS, and the precipitate was combined with the sulfide residues]. The γ-ray spectra showed that any contaminants emitting γ rays of energies differing by 0.005 MeV or more from the 0.477-MeV γ ray of ^{7}Be did not exceed the negligible amount of 9×10^{-7} of the ^{7}Be activity of the sources.

One aliquot of the purified carrier-free ^{7}Be solution in $0.5N$ HCl was transferred into a small quartz capsule. The small hole in the capsule was stopped up with paraffin, taking care that no air bubble was left in the liquid. The closed capsule was immobilized in Araldit in the source holder. This source of carrier-free ^{7}Be in $0.5N$ HCl will be called $\mathrm{Be^{2+}(OH_2)_4}$, indicating that in this solution the beryllium should exist as $[\mathrm{Be(OH_2)_4}]^{2+}$.

Although not very much is known about the stability of carrier-free beryllium solutions,[9] one may conclude from the available data about the radiocolloidal properties of beryllium that a carrier-free solution of ^{7}Be in $0.5N$ HCl should be stable. But this assumption cannot be proved; therefore, the possibility that slight changes took place in the distribution of the ^{7}Be atoms throughout the sample (for example, by absorption on the walls) should be kept in mind.

Sufficient inert $\mathrm{BeCl_2}$ was added to the carrier-free $^7\mathrm{BeCl_2}$ solution. All beryllium compounds were prepared from aliquots of this solution. Figure 1 shows schematically how the beryllium compounds were prepared. The procedures for preparing BeO, $\mathrm{BeF_2}$, $\mathrm{BeBr_2}$, and $\mathrm{Be_4O(CH_3COO)_6}$ were taken from Braur[10] with slight modifications. The $\mathrm{Be(C_5H_5)_2}$ source was prepared according to a method of Fischer and Hoffmann.[11]

The sources were placed into the source holders with great care to provide protection against mechanical and chemical changes during the experiment. The solid sources were pressed into the source holders by means of a brass stopper to prevent any mechanical movement during the measurements. The cylindrical source holders were made of stainless steel and had a wall thickness of 0.5 mm. They fitted exactly into the re-entrant source thimbles of the ionization chambers and could be replaced to better than 10^{-3} cm in the thimbles. $\mathrm{BeF_2}$, $\mathrm{BeBr_2}$, and $\mathrm{Be(C_5H_5)_2}$, which are hygroscopic or sensitive to air, were brought into the holders under dry argon or nitrogen. The brass stopper was spread with adhesive (Uhu-Plus), squeezed into the source holders with a few hammer strokes to immobilize the powder, and kept under pressure till the adhesive was hardened.

The activities of the individual beryllium sources were roughly matched $[\Delta i(0) < 0.01 J_2(0)]$ by using the differential ionization chamber as a balance. One of the sources in one of the chambers was al-

TABLE II. Half-life determination for ^{7}Be.

Author	Half-life of ^{7}Be (day)
Segrè and Wiegand (Ref. 1)	52.93 ± 0.22
Kraushaar, Wilson, and Bainbridge (Ref. 3)	53.61 ± 0.17
Bouchez *et al.* (Ref. 4)	53.00 ± 0.40
Wright *et al.* (Ref. a)	53.50
This work	53.52 ± 0.10

[a] H. W. Wright, E. J. Wyatt, S. A. Reynolds, W. S. Lyon, and T. H. Handley, Nucl. Sci. Eng. 2, 427 (1957).

ready encapsulated, the other source in the second chamber was still open, so that the amount of the beryllium compound in the source holder could be changed by putting in or taking out a little.

The difference current should be as small as possible at the beginning of the experiment. Therefore the single currents of the encapsulated sources have to be matched closely to each other. This was achieved by placing the two encapsulated sources in the source-holder thimbles, fixing the position of one source, and varying the position of the other source until the difference ionization current was smaller than $5\times 10^{-4}J_2(0)$.

III. RESULTS

The decay constant λ of ^{7}Be and the initial ionization current $J(0)$ at $t=0$, which enters directly into the calculation of $\Delta\lambda/\lambda$ from the slope of the $e^{\lambda t}\Delta i(t)$ plots, were obtained by measuring the decay of the BeF_2, BeO(II), BeO(III), and $Be(C_5H_5)_2$ sources. The decay curves $J(t)=J(0)e^{-\lambda t}$ were fitted to the data by a least-squares procedure. The final result was

$$\lambda = 0.012\,951 \pm 0.000\,012 \text{ day}^{-1}.$$

The average value of λ corresponds to a half-life of 53.52 ± 0.05 day. This value agrees well with other measurements which are shown in Table II.

Table III summarizes the results of the least-squares analysis of the data for the eight sets of sources. $\lambda = 0.012\,951 \pm 0.000\,012$ day^{-1} was used in calculating $e^{\lambda t}\Delta i(t)$. The given errors are standard deviations (based on external consistency). Figures 2–6 are plots of $y=\Delta i(t)e^{\lambda t}=\Delta i(0)+J_2(0)\times\Delta\lambda t$ for five sets of the sources listed in Table I. The solid lines represent the least-squares fit of the data for which the values of $\Delta i(0)$ and $J_2(0)\Delta\lambda$ are given in Table III.

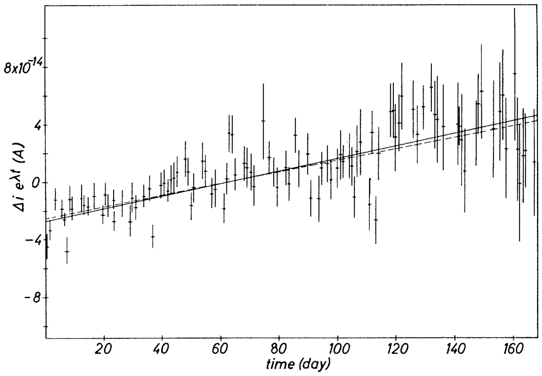

FIG. 3. $e^{\lambda t}\Delta i$ versus time for the $Be_4O(CH_3COO)_6$-BeO(I) source pair.

For three sources, called A, B, and C, the following relation exists between the respective values of b of the three possible combinations of the sources A-B, C-B, and A-C:

$$b_{(A-B)} = b_{(C-B)} + b_{(A-C)}\,.$$

The values of b of the three pairs BeO-BeF_2, $Be_4O(CH_3COO)_6$-BeO, and BeF_2-$Be_4O(CH_3COO)_6$ and of the three pairs $Be^{2+}(OH_2)_4$-$Be(C_5H_5)_2$, $Be^{2+}(OH_2)_4$-BeO, and $Be(C_5H_5)_2$-BeO follow this relation satisfactorily within the distribution of the errors (Table III, column 3).

A constraint-fitting using a least-squares procedure was now imposed on these values of b to get a more accurate knowledge of each of these values.[12] The slopes were adjusted by the constraint that the sum of the values of the pairs C-B and A-C must equal the value of b of the pair A-B. The results for b of the constraint fitting are shown in Table III (last column). The dashed lines in Figs. 2, 3, and 5 are plots of $y=a+bx$ with these adjusted slopes b.

$\Delta\lambda/\lambda$ was calculated from the adjusted slopes using the relation $\Delta\lambda/\lambda = b/J_2(0)\lambda$. Table I summarizes the results.

Another test for checking the over-all performance of the apparatus was applied by comparing two identical sources. Zero slope should be measured within the errors if everything is working

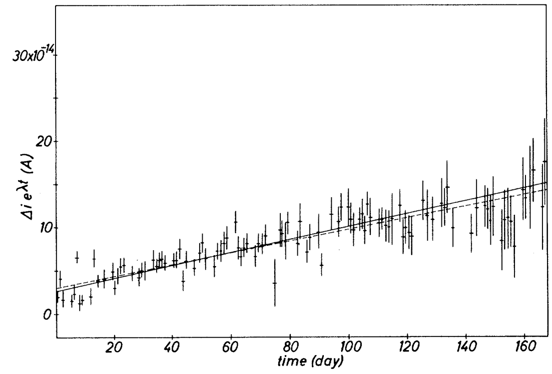

FIG. 2. $e^{\lambda t}\Delta i$ versus time for the BeO(I)-BeF_2 source pair.

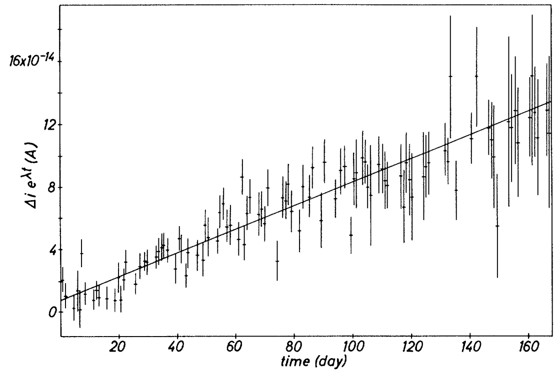

FIG. 4. $e^{\lambda t}\Delta i$ versus time for the $BeBr_2$-BeO(II) source pair.

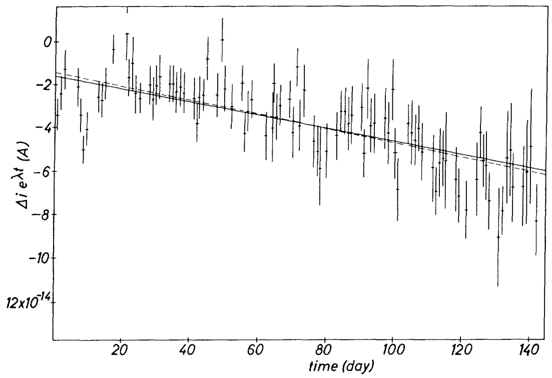

FIG. 5. $e^{\lambda t}\Delta i$ versus time for the $Be(C_5H_5)_2$-BeO(III) source pair.

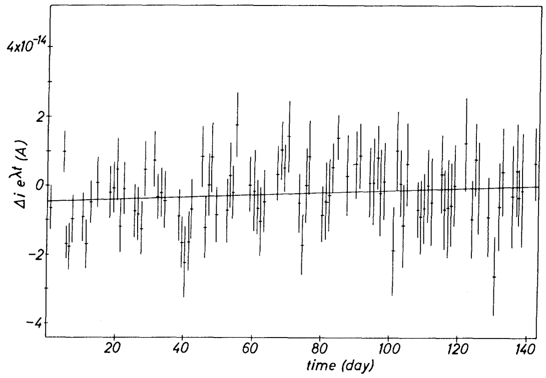

FIG. 6. $e^{\lambda t}\Delta i$ versus time for a pair of sources of identical composition (BeO).

properly, if $\lambda = 0.012\,95$ day^{-1} is correct, and if contaminants are absent (or balanced). Figure 6 shows that essentially zero slope was recorded for two BeO sources.

IV. DISCUSSION

The value for $\lambda(BeO) - \lambda(BeF_2)$ found in this work is about two times larger than the value measured by Leininger, Segré, and Wiegand[2] and Kraushaar, Wilson, and Bainbridge.[3] Both groups prepared their BeF_2 by converting $Be(OH)_2$ into BeF_2 with anhydrous hydrofluoric acid. The structure of their BeF_2 was found to be hexagonal with constants $a = 4.72$ Å and $c = 5.18$ Å. In this work BeF_2 was prepared by thermal decomposition of $(NH_4)_2BeF_4$. This method yields a vitreous BeF_2 which has a structure like the BeF_2 investigated by Warren and Hill.[13] The Be atoms are surrounded tetrahedrally by four F atoms at a distance of 1.60 Å. Therefore the different structure of the BeF_2 used might explain the different results. The Be-F distance in the amorphous BeF_2 is smaller than in the hexagonal BeF_2. The power of the fluorine atoms to attract electrons should be smaller in the hexagonal structure, and thus the electron density at the Be atom should be larger than in the vitreous BeF_2. But, on the other hand, one could also reason that in the vitreous form of BeF_2 the electron density could be higher at the Be nucleus because of squeezing in of valence density due to shorter interatomic distances.

That the first assumption could be right is indicated by comparing the results for $\lambda(Be) - \lambda(BeF_2)$ found by Bouchez *et al.*[4] with the results of Segré and Wiegand[1] and Kraushaar, Wilson, and Bainbridge.[3] The value found by Bouchez *et al.* is higher, and they too prepared their BeF_2 by decomposition of $(NH_4)_2BeF_2$. If the result of Kraushaar, Wilson, and Bainbridge[3] for $\lambda(BeO) - \lambda(Be) = (-0.131 \pm 0.051)\times 10^{-3}\lambda(Be)$ is added to the result of Bouchez *et al.*[4] for $\lambda(Be) - \lambda(BeF_2)_{\text{amorph}} = (1.2 \pm 0.1)\times 10^{-3}\lambda(Be)$, one finds a value for $\lambda(BeO) - \lambda(BeF_2)_{\text{amorph}} = (1.069 \pm 0.151)\times 10^{-3}\lambda(Be)$. This value compares more favorably with the result of this work for $\lambda(BeO) - \lambda(BeF_2)_{\text{amorph}} = (1.130 \pm 0.056)\times 10^{-3}\lambda(Be)$.

Because of the decreasing electronegativity in the sequence $E_F > E_O > E_{Br}$, one could assume that the Be atom in BeF_2 should be left with fewer electrons on the average than in BeO or $BeBr_2$. Therefore one would expect $\lambda(BeBr_2) > \lambda(BeO) > \lambda(BeF_2)$.

TABLE III. Results of the least-squares analysis of the data for $e^{\lambda t}\Delta i$ versus t, and $J(t)$ versus t. (The errors are standard deviations based on external consistency.)

Source pair	$\Delta i(0) \pm \sigma(\Delta i)$ $(10^{-4}\ A)$	$b \pm \sigma(b)$ $[b = J_2(0)\Delta\lambda]$ $(10^{-18}\ A/d)$	$J_2(0) \pm \sigma(J_2(0))$ $(10^{-11}\ A)$	b (after constraint fitting) $(10^{-18}\ A/d)$
$BeO(I) - BeF_{2\,\text{amorph}}$	2.679 ± 0.176	688 ± 29.0	4.307 ± 0.005	630 ± 32.8
$Be_4O(CH_3COO)_6 - BeO(I)$	−2.753 ± 0.180	447 ± 29.6	4.306 ± 0.005	403 ± 31.8
$BeF_{2\,\text{amorph}} - Be_4O(CH_3COO)_6$	−2.507 ± 0.215	−1108 ± 35.7	4.307 ± 0.005	−1033 ± 45.7
$BeO(II) - BeBr_2$	0.811 ± 0.181	752 ± 30.0	3.949 ± 0.003	
$Be^{2+}(OH_2)_4 - Be(C_5H_5)_2$	0.732 ± 0.240	535 ± 39.4	3.355 ± 0.004	507 ± 46.2
$Be^{2+}(OH_2)_4 - BeO(III)$	−0.551 ± 0.230	125 ± 36.9	3.353 ± 0.004	162 ± 33.6
$Be(C_5H_5)_2 - BeO(III)$	1.643 ± 0.194	−315 ± 29.4	3.351 ± 0.004	−345 ± 31.7
$BeO(III) - BeO(IV)$	−0.467 ± 0.159	31.7 ± 29.6	3.182 ± 0.004	

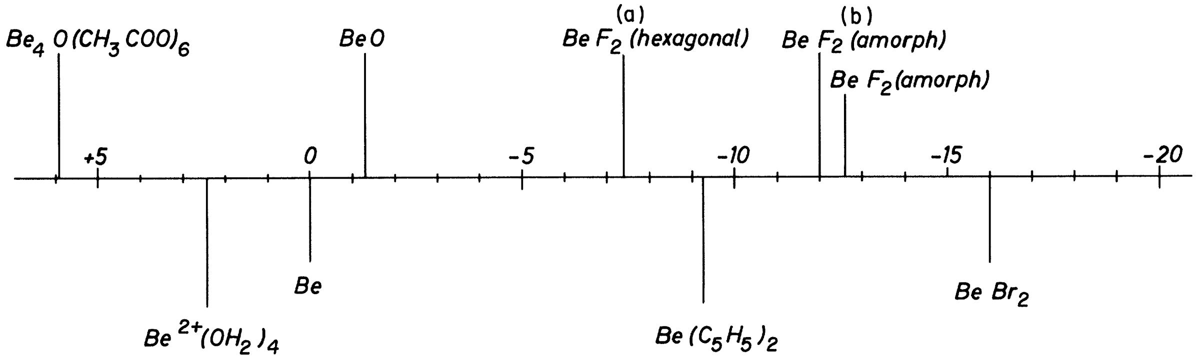

FIG. 7. Differences of electron densities at the Be nucleus in various compounds (BeX) of Be: $|\psi_0|^2_{Be} - |\psi_0|^2_{BeX}$ in units of $10^{-4}\,|\psi_0|^2_{Be}$. (a) Measurement of Ref. 3; (b) measurement of Ref. 4.

We find, however, $\lambda(BeO) > \lambda(BeF_2) > \lambda(BeBr_2)$. This means that the results cannot be interpreted only in terms of the electronegativities of the atoms with which the Be is combined. Lattice conditions play an important part, too, in the variation of λ and therefore in the variation of the electron density at the Be nucleus. Akishin *et al.*[14] and Semenenko and Naumova[15] found that $BeBr_2$ consists of continuous chains of $BeBr_4$ tetrahedra linked together by opposite edges. The Be-Br distance in crystalline $BeBr_2$ is given as 2.10 Å.[15]

The Be nucleus is surrounded by four oxygen atoms in BeO, $Be_4O(CH_3COO)_6$, and $Be^{2+}(OH_2)_4$. In the lattice of $Be_4O(CH_3COO)_6$ the Be-O distance of the four oxygen atoms surrounding the Be atom is 1.65 Å, as reported by Bragg,[16] Morgan and Astbury,[17] and Pauling and Sherman,[18] but three of these oxygen atoms belong to the acetate group. The Be^{2+} ion in aqueous solution appears to be surrounded by four water molecules in which the oxygen atoms are directed towards the Be^{2+} ion. BeO forms a wurtzite structure in which each Be atoms is surrounded by four oxygen atoms at a distance of 1.64 Å.[19] The measurements show that even in the compounds BeO and $Be_4O(CH_3COO)_6$ the electron density at the Be nucleus is not the same, although in both cases the Be nucleus is surrounded by four oxygen atoms at the same distance. Thus the influence of the atoms or group of atoms with which the oxygen atoms are combined is observable.

In dicyclopentadienylberyllium, $Be(C_5H_5)_2$, the Be atom is situated between two cyclopentadienyl rings. It can occupy two positions between the rings[20] at a distance of 1.48 or 1.98 Å from the plane of the rings. One might assume that the electron attracting power of the two cyclopentadienyl rings should be smaller than one of the oxygen atoms in BeO, $Be_4O(CH_3COO)_6$, and $Be^{2+}(OH_2)_4$ or of the fluorine atoms in the BeF_2. One might even expect that the electron density at the Be atom could be increased by the π electrons of the two rings. But the change of the decay constant shows that the electron density at the Be atom is larger in the oxygen-containing compounds and even in the hexagonal form of BeF_2. Obviously the π electrons of the rings do not reach down to the Be nucleus, and the electrons of Be are drawn away by the ring systems.

All these attempts to interpret the results are very crude. Perhaps one should not try to interpret these results with the known facts about beryllium compounds, e.g., their structure, the electronegativity of the atoms, etc., but should use the results as information about the electron density near the Be nucleus in different chemical combinations.

The results show that the decay constants of ^7Be in different chemical compounds decrease in the following sequence:

$$\lambda(Be_4O(CH_3COO)_6) > \lambda(Be^{2+}(OH_2)_4) > \lambda(Be) > \lambda(BeO) > \lambda(Be(C_5H_5)_2) > \lambda(BeF_2) > \lambda(BeBr_2)\,.$$

Since the decay constant λ of ^7Be is proportional to the electron density at the Be nucleus, $|\psi_0|^2_{Be}$, these results permit the establishment of a scale (which is shown in Fig. 7) for the relative electron densities at the Be nucleus in different chemical surroundings. The results of the measurements of Kraushaar, Wilson, and Bainbridge[3] and Bouchez *et al.*[4] are also plotted in Fig. 7.

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