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Determination of effective resonance energy for the ^{193}Ir(n,γ)^{194}Ir reaction by the cadmium ratio method

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ABSTRACT

In this work, the effective resonance energy, $\bar{E}_r$-value for the ^{193}Ir(n,γ)^{194}Ir reaction was measured using cadmium ratio method. A dual monitor (^{197}Au–^{98}Mo), which has convenient resonance properties, was employed for characterization of the irradiation sites. Then analytical grade iridium oxide samples diluted with $CaCO_3$ to lower neutron self-shielding effect stacked in small cylindrical Teflon boxes were irradiated once with a 1 mm thick Cd cylindrical box placed in a thermalized neutron field of an ^{241}Am–Be neutron source then without it. The activities produced in samples during ^{193}Ir(n,γ)^{194}Ir reaction were measured using a p-type HPGe detector γ-ray spectrometer with a 44.8% relative efficiency. The correction factors for thermal, epithermal neutron self-shielding (G_{th}, G_{epi}), true coincidence summing (F_{coi}) and gamma-ray self-absorption (F_s) effects were determined with appropriate approaches and programs. Thus, the experimental $\bar{E}_r$-value was determined to be 2.65 ± 0.61 eV for ^{193}Ir target nuclide. The recent data for Q_0 and F_{Cd} values for $\bar{E}_r$ determination were based on k_0-NAA online database. The present experimental $\bar{E}_r$ value was calculated and compared with more recent values for Q_0 and F_{Cd} for ^{193}Ir. Additionally, the $\bar{E}_r$-values was theoretically calculated from the up-to-date resonance data obtained from ENDF/B VII library using two different approaches. Since there is no experimentally determined $\bar{E}_r$-value for the ^{193}Ir isotope, the results are compared with the calculated ones given in the literature.

1. Introduction

Iridium and other platinum group elements (PGEs: Ru, Rh, Pd, Os, Ir, Pt) have attracted particular interests in industry as catalysts and additives [1]. However, there is an urgent need for accurate analysis methods to establish the element concentrations of these elements during product development and for quality assurance. Neutron activation analysis (NAA) method has a significant role among the various analytical methods due to its selectivity and sensitivity. Especially, k_0-NAA standardization method has become a very common technique, since it has several advantages over the NAA methods, such as there is no necessity for standards for the new materials [1].

In principle, the k_0 standardization method relies on measurement of the ratio between specific activities of the investigated isotope and well defined standards. Simply, the k_0-NAA method is based on effective resonance energy $\bar{E}_r$, Q_0 factors and a few other parameters. An effective resonance energy $\bar{E}_r$ is defined as a weighted average of the true resonance energies, where the contributions of the particular resonances to the resonance integral are used as weights [2]. By the adopted definition, since the $\bar{E}_r$ term represents "*the energy of a single virtual resonance which gives the same resonance activation rate as all actual resonances for the isotope*", the $\bar{E}_r$-value for the isotope is taken as an effective or average value estimated from all the resonances in the epithermal spectrum region. Q_0 is the ratio between the resonance integral and the 2200 m/s cross section (I_0/σ_0). Hence, if k_0-NAA method is used, the accuracy and precision of effective resonance energy $\bar{E}_r$ is a key parameter in the determination of the mass fraction of an analyte contained in the sample. On the other hand, some of the nuclear data obtained with k_0-NAA method are from 1970's to 1980's [3,4].

The literature review shows that the $\bar{E}_r$ values were first calculated in 1979 and 1987 with an analytical expression by the assumption that Breit–Wigner distribution describes any resonance accurately. They were first calculated using resonance data available at that time and have not been updated since then, due to the lack of enough statistical experimental data for comparison. Some of the Q_0 and $\bar{E}_r$ values from the 2003 to 2012 recommended k_0-data sets were adopted from references [5,6].

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In 1984 a multi-channel method was proposed for simultaneous Q_0 and $\bar{E}_r$ experimental determination [7]. However, the experimental re-determination of these factors has been carried out for a few cases and only for a few isotopes. It is seen that some values were experimentally determined once, approximately 30 years ago, and have not been re-determined or updated since then [8].

In this work, the effective resonance energy for the ^{193}Ir(n, γ)^{194}Ir reaction was determined using cadmium ratio method with ^{197}Au and ^{98}Mo monitors. The reason for running the Cd-ratio method is that allows us to eliminate the potential differentiations in the neutron spectrum. This gives us consistent results, provided that the same irradiation and counting conditions apply for every measurement. The approaches for experimental and theoretical determination of the $\bar{E}_r$-value is briefly described as follows:

The reaction rate (R) of the nuclei produced in a neutron field is characterized by the reaction cross section (σ) and the neutron spectrum

$$R = \int_0^\infty \sigma(v) v n(v) dv \tag{1}$$

where $\varphi(v) = v \cdot n(v)$ is neutron flux with neutron velocity v and $n(v)$ the neutron density. This definition can be expressed as the sum of the thermal, epithermal and fast neutron component;

$$R = K\left[\int_0^{E_{Cd}} \sigma(E)\varphi(E)dE + \int_{E_{Cd}}^{E_{epi}} \sigma(E)\varphi(E)dE + \int_{E_{epi}}^{\infty} \sigma(E)\varphi(E)dE\right] \tag{2}$$

where E_{Cd} is 0.55 eV. When the neutron field has a well-thermalized spectrum, fast neutron contribution can be negligible. Therefore, we didn't take into account third term in Eq. (2).

In k_0-NAA method, the thermal to epithermal flux ratio, f and the $Q_0(\alpha)$ are related to each other. The relationship between f-ratio and the Cd ratio to be measured for an isotope, R_{Cd} is as follows [9,10]:

$$Q_0(\alpha) = \frac{f}{F_{Cd} \cdot R_{Cd} - 1}, \tag{3}$$

where α is the epithermal spectrum shape factor represents the measure of deviation from ideal $1/E$-epithermal spectrum and $Q_0(\alpha) = (I_0(\alpha)/\sigma_0)$ with the $I_0(\alpha)$ is the resonance integral corrected for the deviation.

Additionally, the k_0-NAA method also uses the notion of effective resonance energy $\bar{E}_r$ to correct for the influence of non-ideal nature of the epithermal neutron flux distribution. The effective resonance energy, $\bar{E}_r$ is defined as:

$$\bar{E}_{r,i} = \left[(Q_0 - 0.429)/\left(\frac{f}{F_{Cd} \cdot R_{Cd} - 1} - \frac{0.429}{(2\alpha+1)\cdot E_{Cd}^{\alpha}}\right)\right]^{1/\alpha} \tag{4}$$

Effective resonance energy of an investigated isotope can be determined from following equation by taking effective resonance energy of the comparator, thermal and epithermal neutron self-shielding factors, Q_0 factors and Cd ratios for an investigated isotope and the comparator (c) into account [7,11];

$$\bar{E}_r = \bar{E}_{r,c}\left[\frac{Q_0 - 0.429}{Q_{0,c} - 0.429} \cdot \frac{\frac{f}{(F_{Cd,c}\cdot R_{Cd,c} - 1)\left(\frac{G_{epi}}{G_{th}}\right)_c} - C_\alpha}{\frac{f}{(F_{Cd}\cdot R_{Cd} - 1)\left(\frac{G_{epi}}{G_{th}}\right)} - C_\alpha}\right]^{1/\alpha}, \tag{5}$$

where $C_\alpha = \frac{0.429}{(2\alpha+1)\cdot E_{Cd}^{\alpha}}$ and $0.429 \approx 2 \cdot \sqrt{E_0/E_{Cd}} = 2 \cdot \sqrt{0.025/0.55}$

In the case of non-ideal epithermal spectrum, it is clear that, the deviation from the $1/E$ behavior should not be ignored in an irradiation position. Otherwise, this might cause the substantial errors on the analytical results because of the inaccuracy of the essential nuclear parameter $I_0(\alpha)$, which is resonance integral cross section [7].

In Eq. (5) the cadmium ratio, $R_{Cd} = A_{sp}^{-}/A_{sp}^{+}$ can easily be calculated from the measured specific activities of the isotopes as described in our previous studies [12–14]. The thermal self-shielding factors (G_{th}) used in Eq. (5) for Au and Mo-foils can be calculated by the Nisle's approximation [15]. The epithermal neutron self-shielding factors of Au foils calculated by the approximations given in the works of Chilian et al. [16] and Salgado et al. [17]. However, G_{th} and G_{epi} factors for the powder mixtures in the Teflon tubes, were calculated using the procedures for the case of the irradiations in an isotopic neutron field [18,19]. This was a tedious work carried out taking the nuclear data required such as absorption, scattering, total microscopic cross-sections, resonance parameters, etc. from ENDF/B VII [20], and NUDAT 2.6 [21] online libraries. Nuclear data and decay data used in the analyses for effective resonance energy determinations are tabulated in Table 1 together with the calculated neutron self-shielding factors.

2. Theoretically calculation of $\bar{E}_r$-value

In neutron activation, the occurrence of "resonances" in total and capture cross sections is a function of neutron energy. Therefore, the effective resonance energy of an isotope can theoretically be estimated by the following approximate expressions. As the first approach used by Moens et al. [3], one can use:

$$\ln \bar{E}_r = \frac{\sum_i \frac{\sigma_i \Gamma_{\gamma,i} \ln E_{r,i}}{E_{r,i}}}{\sum_i \frac{\sigma_i \Gamma_{\gamma,i}}{E_{r,i}}} \tag{6}$$

where σ_i is partial capture cross-section, $\bar{E}_{ri}$ is the i-th resonance energy and $\Gamma_{\gamma,i}$ is the radiative width at the maximum of the i-th resonance energy. It is assumed in Eq. (6) that resonance self-shielding is negligible, which is the case when using sufficiently diluted samples. The up-to-date resonance data is taken from ENDF/B VII [20]. Fig. 1 exhibits the cross section data for ^{193}Ir as a function of the neutron energy. The $\bar{E}_r$-values calculated from Eq. (6) are also given in Table 3. The main drawback of this approximation is that neutron widths, Γ_n which were neglected in Eq. (6).

The second approach suggested by Jovanovic et al. [4] is that $\bar{E}_r$-value can be calculated by assuming its α-independence according to the term $(1\ \text{eV})^{\alpha} \equiv 1$ that are omitted,

$$\ln \bar{E}_r = \frac{\sum_i w_i \cdot \ln E_{r,i}}{\sum_i w_i} \tag{7}$$

where the weighting factors w_i are given by

$$w_i = \left(\frac{g\Gamma_\gamma \Gamma_n}{\Gamma}\right)_i \cdot \frac{1}{E_{r,i}^2} \tag{8}$$

where $g = (2j+1)/2(2I+1)$ is the statistical weight factor, I and J are the spin of the target nucleus and the resonance state of the neutron captured compound nucleus, Γ_γ is the radiative width, Γ_n is the neutron width, and $\Gamma = \Gamma_\gamma + \Gamma_n$ is the total width of resonance. For the evaluation of Eq. (7) the recent resonance data are taken from the available literature [20], thus yielding the $\bar{E}_r$-values.

3. Experimental methods

The mixture of IrO_2 with a sufficient amount of $CaCO_3$ powder was prepared to reduce neutron self-shielding effects. The mixtures were stacked in small Teflon tubes with a thickness a 1 mm, a radius of 6.5 mm and a height of 6.25 mm. The diluted IrO_2 samples, and Au and Mo foils were irradiated in two sites of the neutron irradiation unit. The irradiation procedure was carried

Table 1
Nuclear data for the isotopes, true coincidence summing correction and self-attenuation factors for the measured gamma-rays.

Target isotope (abundance)	Nuclear reaction	Cadmium transmission factor, F_{Cd}[a]	Effective resonance energy, E_r (eV)[a]	Q_0 ($=I_0/\sigma_0$)[a]S (%)	Half-life[b]	The measured gamma-ray[b]		True coincidence summing[g]	Self-attenuation factor[e]	Thermal neutron self shielding factor G_{th}	Epithermal neutron self shielding factor G_{epi}
						Energy (keV)	Emission probability, γ (%)	F_{Coi}	F_s (From NIST)[c]		
^{197}Au (100%)	^{197}Au(n,γ)^{198}Au	0.992	5.65 ± 0.40	15.7(1.8%)	2.272(16) d	411.802(17)	95.62	0.9986	1.0005	0.999[e]	0.922[e]
^{98}Mo (24.13%)	^{98}Mo(n,γ)^{99}Mo(β)^{99}Tc	1.000	241 ± 48	53.1(6.3%)	65.976(24) h	140.511(1)	4.52(24)	0.9906	1.0011	0.997[d]	0.991[f]
^{193}Ir (62.7%)	^{193}Ir(n,γ)^{194}Ir	1.000	–	12 (2.9%)	19.28(13) h	328.448(14)	13.1(17)	0.9315	1.0095	0.999	0.993

[a] De Corte and Simonits (2003) [27], Kolotov and De Corte (2003) [28].
[b] NUDAT 2.6 [21].
[c] Self-attenuation calculated using mass attenuation coefficients taken from NIST [24].
[d] Calculated by Nisle' approximation, including neutron scattering effects [15] (Gilat and Gurfinkel, 1963).
[e] Calculated by approximation given in [16,17].
[f] Calculated by approximation given in Beckurts and Wirtz (1964) [29].
[g] True coincidence summing correction factors calculated by using GESPECOR Ver. 4.2 software [23] for the present detector-source geometry.

out with two sets of samples. One sample was placed in Cd cylinder box with 1 mm thickness, a radius of 13 mm and a height of 20 mm and the second sample was not placed in such a box. The masses of Au and Mo foils vary between 0.09 and 0.13 g. The samples were always kept at a fixed position during neutron exposure to maintain the same Cd-ratio value while the irradiation process was repeated with use of another set of the foils.

The irradiation unit, details can be found in [22], consists of 37 GBq ^{241}Am–Be neutron source at the center of a water tank surrounded by paraffin and a mixture of paraffin and boron oxide. Fig. 2 shows a schematic view of the experimental set-up used for the work.

The source is positioned in a fixed position between two irradiation channels. One of them is called Site #1 with a pneumatic or "rabbit" sample transfer system. The other is called Site #2 having a location for manual control. In the Site #1 channel, the pneumatic transfer system is delivered by air-compressor using of rubber hoses. The transfer capsules for the pneumatic tube irradiations are made of polypropylene. The lids of the capsules are tapered enough to move in the plastic hose via compressed air without any difficulty.

The Cd-ratios of Au and Mo foils were used to determine the α-shape parameters of the epithermal neutron spectrum distribution obtained from irradiation site #1 and #2. The foils with a purity of 99.95%, a thickness of 0.00508 mm and a radius of 12.7 mm were obtained from Shieldwerx, A Division of Bladewerx LLC, Rio Rancho, NM, USA. From our previous work [22], the neutron flux characterization parameters: epithermal neutron spectrum shape factor, α and thermal-to-epithermal flux ratio, f were found to be $\alpha = 0.045 \pm 0.009$ and $f = 63.6 \pm 1.5$ for the pneumatically driven irradiation position at site #1; $\alpha = 0.038 \pm 0.008$ and $f = 50.9 \pm 1.3$ for the manual use position at site #2.

A p-type HPGe well detector (Canberra, GCW4023) with a 44.8% relative efficiency was used for recording the gamma-ray spectra. The resolution of detector is ≈2 keV at 1332.5 keV for ^{60}Co source. The radius and depth of well are 23 mm and 35 mm, respectively. The electronic system for data collection consists of a spectroscopy amplifier (Canberra 2025) and a 14 bit ADC/MCA analyzer (Canberra Multiport II) with suitable gains setting to cover the required energy range.

A GESPECOR® (Ver 4.2) software based on a Monte Carlo simulation [23] was used to calculate the full-energy peak detection efficiency (ε_p). Background subtraction was always considered for the individual sample spectra. The evaluation of the net counts in the peaks of interest was evaluated by using Genie-2000 software and also checked by manual calculations. For the present counting geometry, gamma-ray self-absorption (F_s) and true coincidence summing (F_{coi}) effects were computed by the GESPECOR® (Ver 4.2) software. Self-absorption correction factors F_s were calculated using mass attenuation coefficients taken from NIST [24].

In our work, the data for specific activities of ^{99}Mo were derived from irradiation of bare Mo foil and Mo foil within a Cd-box. According to the reaction ^{99}Mo (β$^-$) ^{99m}Tc, the nuclide ^{99m}Tc emits the most intensive gamma rays with energy of 140.511 keV. Therefore, this gamma ray was chosen to extract the specific activity of ^{99}Mo due to its intensity. On the other hand, ^{99}Mo itself contributes to the 140.511 keV gamma ray emitted by ^{99m}Tc. Hence, it is also necessary to take branching ratio correction into account in order to obtain more precise results [19]. After that correction, the net specific activity of ^{99}Mo can be obtained using method given by Simonits et al. [25,26].

4. Result and discussion

In this work, a new experimental $\bar{E}_r$-value for the ^{193}Ir(n,γ)^{194}Ir reaction was determined using a reliable cadmium ratio method.

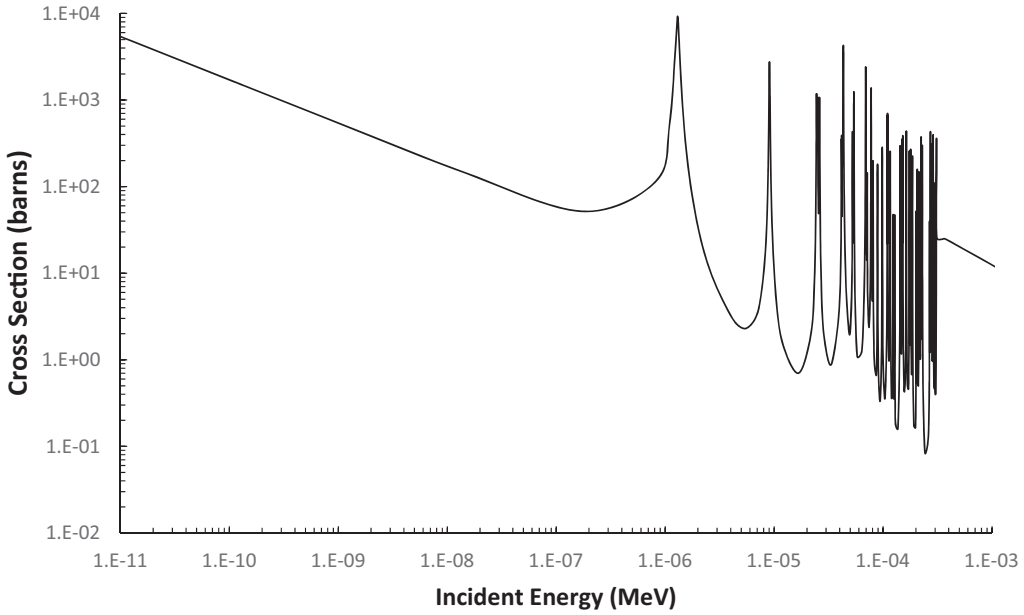

Fig. 1. Cross section versus incident neutron energy for ^{193}Ir.

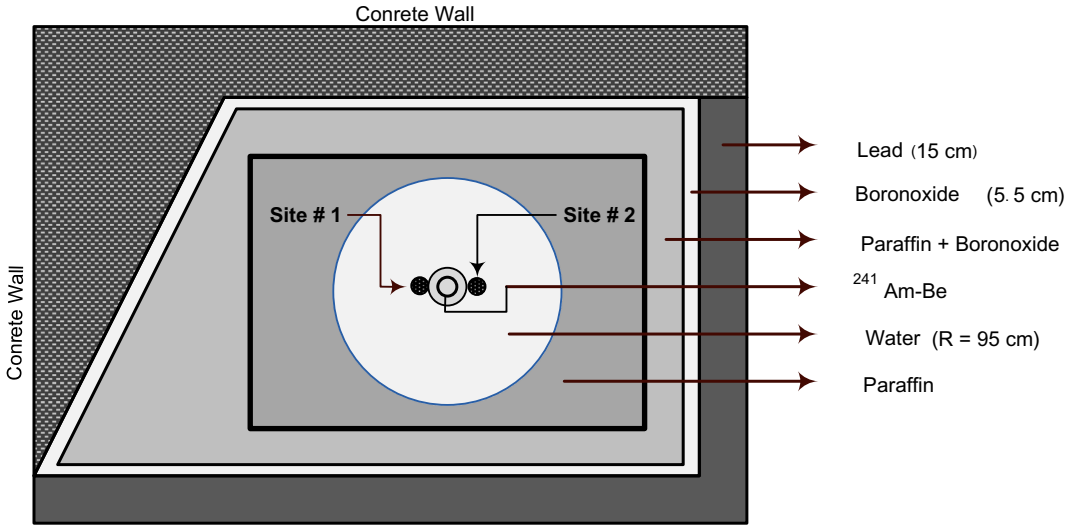

Fig. 2. A schematic view of the neutron irradiation unit with a 37 GBq ^{241}Am–Be source [20].

The experimental $\bar{E}_r$-value determined in this work is given in Table 3 and compared with the present and previous calculated values using two different theoretical approaches. The uncertainty calculation method used in experimental $\bar{E}_r$ determination was outlined in our previous works [12–14]. The main sources of uncertainty in the experimentally determined $\bar{E}_r$-values given Table 3 are mainly due to the α-shape factor (20–21%), the monitor effective resonance energy values (7.1% for ^{197}Au and 20% for ^{98}Mo), and the resonance integral to thermal neutron cross section ratio, Q_0 (1.8% for ^{197}Au, 6.3% for ^{98}Mo and 2.9% for ^{193}Ir). It is not possible to reduce the overall uncertainty of about 20% on the final results for $\bar{E}_r$-values of the isotopes being investigated since the magnitudes of uncertainties on the nuclear data propagated in Eq. (5). According to uncertainty propagation, 20–27% uncertainty is estimated for $\bar{E}_r$-values.

The suggested ratio between the resonance integral and the 2200 m/s cross section value of ^{193}Ir by De Corte and Simonits [27,28] is $Q_0 = 12.0$ as given in the k_0-NAA online database. This Q_0 -value of 12.0 was measured by the Cd-ratio method and it was assumed that $F_{Cd} = 1$. In a recent work [1], the Q_0-value was determined for the ^{193}Ir(n,γ)^{194}Ir reaction by the two-channel method, using five irradiation channels in two different research reactors in Belgium and Canada by Chilian et al. This measured Q_0-value enabled, the first determination of the resonance neutron Cd transmission factor for the ^{193}Ir(n,γ)^{194}Ir reaction and the authors revealed a suspicion that the F_{Cd} might be less than unity. In the work mentioned above, Ir samples were irradiated under Cd cover at both laboratories (SCK-CEN, Belgian Nuclear Research Centre and Ecole Polytechnique, Canada) and the Cd ratios were subsequently used, along with the known Q_0-values, to determine F_{Cd}. For ^{193}Ir(n,γ)^{194}Ir reaction. The authors suggest that the $Q_0 = 13$ and $F_{Cd} = 0.90$ are about 10% different than the literature values. In our work, the experimental $\bar{E}_r$-value was calculated also using values for Q_0 and F_{Cd} for ^{193}Ir from the work of Chilian et al. and a comparison is given in Table 2. It can be seen in Table 2 that the $\bar{E}_r$-values for ^{193}Ir calculated by using $Q_0 = 13$ and $F_{Cd} = 0.90$

Table 2
The measured effective resonance energy values obtained by dual monitors.

Reaction	Experimentally determined effective resonance energy value, $\bar{E}_r$ (eV)					
	Calculated when Q_0 and F_{Cd} values are taken from the k_0 online database [27]			Calculated when Q_0 and F_{Cd} values are taken from Chilian et al. [1]		
	Using ^{197}Au monitor	Using ^{98}Mo monitor	Weighted average	Using ^{197}Au monitor	Using ^{98}Mo monitor	Weighted average
^{193}Ir(n,γ) ^{194}Ir	2.65 ± 0.54	2.58 ± 0.70	2.62 ± 0.43	2.70 ± 0.56	2.63 ± 0.72	2.67 ± 0.44

Table 3
Experimentally and theoretically determined effective resonance energy, $\bar{E}_r$-values.

Reaction	Experimental effective resonance energy value	Theoretically calculated effective resonance energy values			
	This work	This work[a]		Literature	
	$\bar{E}_r$ (eV)	$\bar{E}_r$ (eV) Breit–Wigner[b]	$\bar{E}_r$ (eV) Moens[c]	$\bar{E}_r$ (eV) Breit–Wigner[d]	$\bar{E}_r$ (eV) Moens[e]
^{193}Ir(n,γ) ^{194}Ir	2.65 ± 0.61	2.59	1.68	2.21	1.46

[a] Uncertainties are not given due to no uncertainty quoted in the used data.
[b] Calculated by Eq. (7) using recent resonance data taken from IAEA Evaluated Nuclear Data File (ENDF) data libraries.
[c] Calculated by Eq. (6) using recent resonance data taken from IAEA Evaluated Nuclear Data File (ENDF) data libraries.
[d] Referenced by Jovanovic et al. (1987) [4].
[e] Referenced by Moens et al. (1979) [3].

(suggested by Chilian et al.) are systematically 1.8–2.2% higher than those calculated by using k_0-NAA online database values, $Q_0 = 12$ and $F_{Cd} = 1.00$. The difference between the k_0 literature and the recent extracted values for Q_0 exhibits the requirement for re-evaluation of the effective resonance energy.

The results obtained for $\bar{E}_r$ values were given in Table 2. As can be seen in Table 2, a consistency is revealed between the results obtained by ^{197}Au and ^{98}Mo. The comparator method provides more accurate results due to its capacity to compensate for some systematic errors, which may reflect to the measured values. Two different monitors with known cross sections also allowed to check for the possible differences in the experimental results.

The present experimental result for the $\bar{E}_r$-value was compared with the current and previously calculated theoretical values given in Table 3. In general, the present experimental value was seen to be in good agreement with the calculated ones using the recent resonance data within limits of the estimated uncertainty. However, when Moens' approximation [3] based on Eq. (6) is used to calculate the $\bar{E}_r$-values, the results are not satisfactorily accurate even if the most accurate and currently available neutron resonance data are used as explained in detail in our previous studies [12–14]. This approach is not satisfactorily accurate because of it does not take into account the neutron widths. The experimental results in Table 3 agree with the commonly used calculated values by Jovanovic et al. [4]. It appears that it may be regarded as the experimentally determined $\bar{E}_r$-values rather than the theoretical ones. Since the quantity and quality of the available resonance parameter data for the theoretical calculations of $\bar{E}_r$-values are continuously improved, it is expected that there would be some discrepancies between the older and recently calculated $\bar{E}_r$-values.

Since our experimentally determined $\bar{E}_r$ value is consistent with calculated value from Breit–Wigner formula, when the most recent resonance data are adopted in Eq. (7). Thus, it can be recommended for the calculation of $\bar{E}_r$ of ^{193}Ir. In the light of these findings, it is suggested that the new experimental values of $\bar{E}_r$ for other isotopes, especially for isotopes searched using in k_0-NAA standardization method, should be re-determined.

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