A blind test of the alanine dosimetry secondary standard of the PTB conducted by the BIPM

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Abstract. In order to verify the reliability of the PTB's secondary standard measurement system for the absorbed dose to water, D_W, based on alanine with read-out by electron spin resonance (ESR), a blind test was conducted. Nine detectors consisting of four alanine pellets each were irradiated in the ^{60}Co reference field of the BIPM. The doses were restricted to the range between 10 Gy and 20 Gy in order to minimize the influence of noise. The values of the delivered doses were not communicated until after the measurement results were reported back to the BIPM.

The absorbed dose to water D_W was measured using a slightly simplified procedure, compared to previous publications, i.e. without construction of a calibration curve. On average, the measurement results are 0.12 % higher than expected from the known difference between the primary standards for D_W of the BIPM and the PTB. Compared to the combined measurement uncertainty of the ratio of determined and delivered dose of approximately 0.45 %, the agreement is good.

Keywords: absorbed dose to water, alanine, EPR, ESR, secondary standard, primary standard, blind test

1. Introduction

Dosimetry using alanine with read-out via electron spin resonance (ESR) is a convenient tool for quality assurance measurements for radiotherapy. The main reasons for this are the good water-equivalence of alanine, the weak dependence on the irradiation beam quality, the non-destructive read-out (in contrast to thermoluminescence detectors) and the comparatively small size of the detectors.

Irradiation induces free radicals in the amino acid alanine [1, 2, 3, 4]. The radicals are stable if the detectors are stored in a dry environment. The fading, i.e. the loss of signal due to recombination of some radicals, is then only of the order of a few parts in 10^3 per year, which makes them suitable for mailed dosimetry [5]. The read-out is usually performed by ESR. Since the reading is not absolute, the ESR amplitude has to be compared to the amplitude of an identical detector irradiated with a known dose†.

† Here and in the following the term "dose" is to be understood as "absorbed dose to water".

Since the 1980s, alanine dosimetry has been used for (mailed) dosimetry for radiation processing, and since around 1996, the National Physical Laboratory (NPL, UK) [6] and others [7] have also used alanine for mailed dosimetry in a more useful dose range for therapy, i.e. with doses lower than 10 Gy. In recent years, advanced therapy modalities such as the cyberknife have also been checked using alanine dosimetry [8]. Within the framework of a quality assurance project in Belgium, a large fraction of the Belgian therapy centres participated in a dosimetry audit using alanine/ESR between 2009 and 2011 [9].

The measurement system used for the audits in Belgium is a copy of the PTB's secondary standard measurement system, including the analysis software. In order to speed up the analysis procedure to cope with the demand, the measurement time spent for the daily calibration of the spectrometer had to be reduced. This was achieved in conjunction with a slight change in methodology [10]. Due to this change, a blind test appeared desirable with the aim of verifying whether this simplification in calibration would yield reliable results in spite of the reduced measurement time. This blind test complements an intercomparison with other National Metrology institutions also carried out in 2011 [11].

2. Materials and Methods

2.1. Procedure for the blind Test

Alanine detectors were sent from the PTB to the Bureau International des Poids et Mesures (BIPM) for irradiation in the BIPM's ^{60}Co reference beam. It was agreed that the doses should be in the range from 10 Gy to 20 Gy, the exact values being communicated only after the PTB measurement results were reported back to the BIPM. The dose range was chosen in order to reduce the influence of noise to a minimum within the measurement range of the PTB's alanine dosimetry system (2 Gy to 25 Gy). Other relevant data such as the time of irradiation and the estimated temperature of the alanine during irradiation had to be communicated before the analysis of the detectors, the latter being the most important influence quantity. In total, nine test detectors were irradiated at the BIPM. Two unirradiated test detectors were sent along as controls.

At the PTB, a set of calibration detectors was irradiated at approximately the same time (± 1 d) as the probes at the BIPM in order to assure that the influence of fading was negligible [12]. The ESR measurements were carried out two weeks after irradiation, on two consecutive days. The measurement results and the associated uncertainties were sent to the BIPM; the results were compared with the BIPM delivered dose values and sent to the PTB.

2.2. Detectors

The detectors were alanine pellets with an addition of approximately 9 % of paraffin as a binder. They were produced by Harwell (UK), batch AL595, with an average mass of

59.5 mg, a diameter of 4.82 mm and a height of 2.6 mm. The bulk density of the pellets was 1.25 g cm^{-3}.

Four pellets were stacked to form one detector and shrink-wrapped in 0.18 mm thick polyethylene foil. For the irradiations, the foil was spanned in a frame made of polymethylmethacrylate. A photograph of such a probe is displayed in Figure 1.

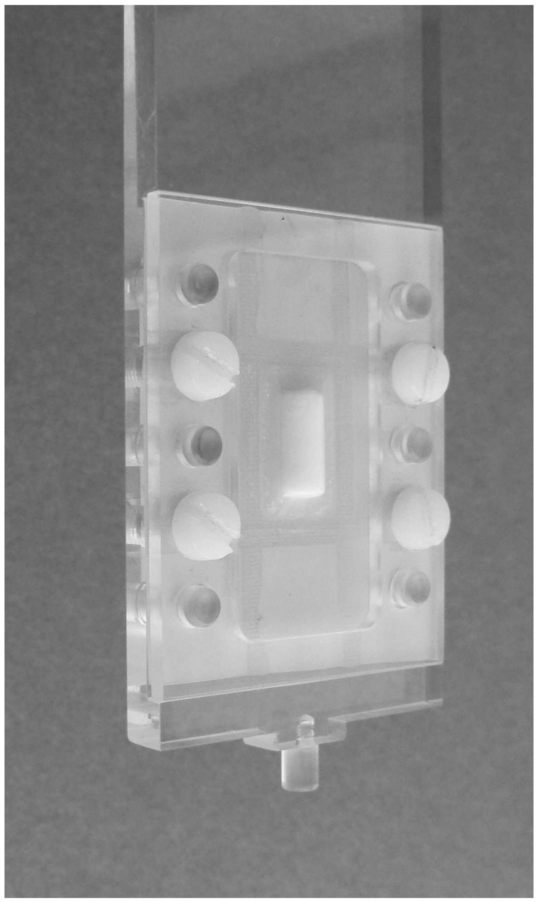

Figure 1. An alanine probe, i.e. the detector, consisting of a stack of four alanine/paraffin pellets, shrink-wrapped in polyethylene foil and spanned in a PMMA frame.

2.3. Irradiations in the Cobalt Irradiation Facility at the BIPM

The PTB alanine detectors were irradiated at the BIPM in the ^{60}Co gamma-ray beam under the reference conditions given in Table 7 of [13]:

- the distance from the source to the reference plane (centre of the detector) is 1 m;
- the field size in air at the reference plane is 10 cm × 10 cm, the photon fluence rate at the centre of each side of the square being 50 % of the photon fluence rate at the centre of the square; and
- the reference depth in the water phantom is 5 g cm^{-2}

The detectors were positioned in the empty water phantom and then the phantom was filled with demineralized water. The water temperature was registered during the irradiations using a calibrated thermistor placed at the reference depth but outside the irradiation field; the water temperature remained stable over the duration of each irradiation to better than $0.2\,^0$C. The mean temperature value for each irradiation was supplied to the PTB. The absorbed dose value is taken from the mean of the four reference measurements made with the BIPM primary standard around the period of the irradiations for the PTB.

The estimated relative standard uncertainties for the BIPM determination of absorbed dose to water are presented in Table 1.

Table 1. Uncertainty budget for the dose delivered by the BIPM

Parameter	$10^2\times$ Relative standard uncertainty†	
	s_i	u_i
Reference absorbed dose to water [13]	0.20	0.21
Dosimeter positioning	-	0.05
Irradiation time	-	0.02
Combined uncertainty of the dose delivered by the BIPM		
Quadratic summation	0.20	0.22
Combined relative standard uncertainty	0.29	

† expressed as one standard deviation.
s_i represents the relative uncertainty estimated by statistical methods, type A
u_i represents the relative uncertainty estimated by other methods, type B

2.4. Irradiation in the Cobalt Irradiation Facility at the PTB

The irradiations for the calibration detectors were performed at the PTB in the ^{60}Co reference beam in a 30 cm × 30 cm × 30 cm cubic water phantom. The geometric centre of the detector was placed at the reference depth of 5 cm where the field size was 10 cm × 10 cm.

The depth was measured with a caliper. The uncertainty of the depth was 0.12 mm, resulting in a contribution to the relative uncertainty of the delivered dose of 0.07 %. The relative uncertainty of the absorbed dose to water as determined with the PTB water calorimeter is 0.2 % [14]. Adding an uncertainty of 0.06 % due to the shutter timing, the relative standard uncertainty of the delivered dose is 0.22 %.

The temperature of the alanine during irradiation is an important influence quantity. As only the temperature of the surrounding water was possible to measure, each detector was placed in the phantom and the temperature was registered with an uncertainty of 0.1 ^{0}C. To ensure thermal equilibrium, the irradiation started 10 minutes after positioning the detectors.

2.5. ESR measurements and analysis

The ESR measurements were made with a Bruker EMX 1327 ESR spectrometer, with an 8 inch (20.32 cm) magnet and an X-band microwave bridge. The high-sensitivity resonator ER 4119 HS was used throughout. The parameters are listed in section 2.4 of a previous publication [15]. All spectra for a given measurement session are recorded with the same receiver gain.

A holder for the alanine pellets made of nested quartz tubes, developed at the PTB, is used which also contains a reference substance provided by Bruker. The ESR spectrum of this reference substance is always registered simultaneously with the spectrum of the alanine pellets.

The data analysis method [10, 15, 16] is based on the assumption that only the *amplitude* of the ESR signal depends on the dose but *not the shape* of the signal. This assumption is realistic for the measurement range of PTB's alanine dosimetry system (2 Gy to 25 Gy).

From a measured spectrum containing the signal contributions from both the irradiated alanine (ala) and the reference substance (ref), the coefficients A^{ala} and A^{ref} are extracted by a least-squares fit of two base functions to the experimental curve. The base functions are determined experimentally from spectra of unirradiated pellets and from spectra of the same number of alanine pellets irradiated in thePTB's ^{60}Co reference field to a comparatively high dose, usually 25 Gy. These spectra have to be measured on the same day as the spectra of the pellets under investigation. For the unirradiated pellets, only the reference substance and the background contribute to the signal. The alanine base function is obtained by subtracting the spectra of the unirradiated pellets from the 25 Gy spectra. Examples for the base functions are displayed in Figure 2. For the blind test, the base functions were constructed from the spectra of eight irradiated and eight unirradiated pellets.

For each pellet, five spectra are acquired and fitted separately using the alanine and the reference base functions. The pellet is rotated by 72 ^{0} after registration of each spectrum, with the aim of averaging over the amplitude variations due to positioning tolerances within the holder [16]. From the fit parameters, *i.e.* from the amplitudes A^{ala} and A^{ref}, the five relative amplitudes

$$A = \frac{A^{\mathrm{ala}}}{A^{\mathrm{ref}}} \tag{1}$$

are calculated and then averaged. Four values A_i ($i = 1 \ldots 4$) corresponding to the four pellets irradiated simultaneously, are averaged to yield the dose-normalized amplitude $\mathcal{A}_D$, which is defined as

$$\mathcal{A}_D = \frac{A_m}{\overline{m}} \cdot \overline{m}^{\mathrm{b}} \cdot D^{\mathrm{b}} \cdot K_{\mathrm{pos}} \cdot K_T. \tag{2}$$

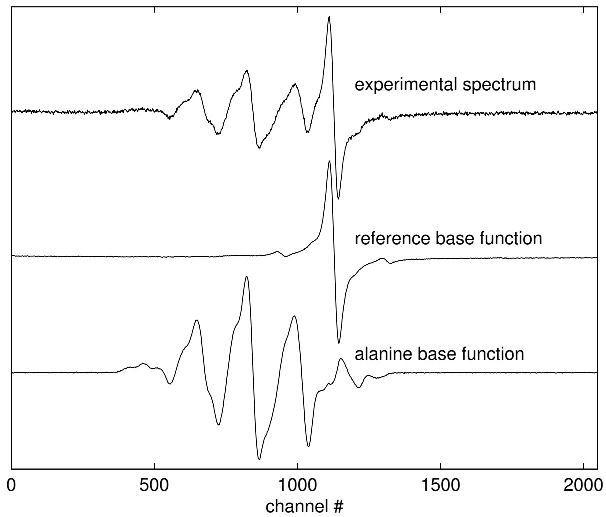

Figure 2. Experimental signal and base functions for the reference method. Top: a typical experimental spectrum containing signal contributions from alanine and the reference substance; middle: spectrum of the reference substance used as a base function to obtain A^{ref}; bottom: pure alanine spectrum used as a base function to obtain A^{ala}

The index b refers to the base function. The meanings of the terms in equation (2) are as follows:

$A_m = \overline{m} \cdot \frac{1}{n} \sum_{i=1}^{n} \frac{A_i}{m_i}$	average mass-normalized ESR amplitude, n is the number of pellets irradiated simultaneously (usually n=4)
$\overline{m} = \frac{1}{n} \sum_{i=1}^{n} m_i$	average mass of the simultaneously irradiated pellets,
$\overline{m}^{\mathrm{b}}$	average mass of the pellets used to construct the (alanine) base function
D^{b}	dose delivered to the pellets used to construct the (alanine) base function
$K_{\mathrm{pos}} = \frac{k_{\mathrm{pos}}}{k_{\mathrm{pos}}^{\mathrm{b}}}$	correction factor for the positioning
$K_T = \frac{k_T}{k_T^{\mathrm{b}}}$	correction factor for the irradiation temperature
	$k_T = 1 - c_T \cdot (T - T_0)$, where c_T is the temperature coefficient

In case two detectors (i.e. n =8 pellets) are used for the construction of the base, the dose D^{b} is the mean of the corresponding two dose values. The reference temperature T_0 for the irradiation temperature correction is $20\,^0$C. The temperature coefficient c_T and its uncertainty have recently been re-determined to be $c_T = (1.82 \pm 0.08) \cdot 10^{-3}\,\mathrm{K}^{-1}$ [17]. Further details can be found in the technical report PTB-Dos-55 [10].

In contrast to previously published work, no explicit calibration curve was determined, the calibration being provided by the normalisation to the base function with a known (cobalt) dose, i.e. $\mathcal{A}_D$ corresponds directly to the determined dose D^{PTB} [10]. The method described here (base only, 8 irradiated, 8 unirradiated pellets) reduces the time required for calibration by two hours per day when compared to measurements using a complete calibration curve [15, 16].

2.6. Uncertainty budget for the dose determined by the PTB

All uncertainties are standard uncertainties and are determined according to the terms of reference stated in the *GUM*, the *Guide to the expression of uncertainty in measurement* [18]. In Table 2, an example uncertainty budget is given for a test dose of 10 Gy.

At least three effects contribute to the uncertainty of the mass normalized amplitude A_m. The first is the repeatability of the amplitude determination. For the chosen parameters and D^{b}=25 Gy, this uncertainty component is equivalent to 40 mGy for a single pellet or 20 mGy for an average over four pellets. This value is independent of dose between 2 Gy and 25 Gy. The second effect is the variation of the individual background signal which amounts to an equivalent of 20 mGy for a single pellet [16]. The third contribution is the intrabatch homogeneity, i.e. the variation of the alanine content within a certain batch which can be quantified by a coefficient of variation of 0.3 % for the batch under investigation. The same estimates apply to the base function amplitudes.

The lower relative uncertainty components for the base detectors compared to the test detectors in Table 2 result from the higher dose of 25 Gy and from the higher number of pellets. For the higher doses, the relative uncertainty due to the amplitude readout repeatability decreases. The limiting components are the intrabatch homogeneity and an additional systematic component of 0.15 % (offset). The latter was deduced from repeat measurements of calibration and test data sets, where the dose calculated without using a calibration line was compared to the known delivered dose‡. It is essentially this offset that is responsible for the fact that the uncertainty of the dose determined without using a calibration curve as described is slightly larger than that for an evaluation using a complete calibration curve.

3. Results and discussion

The results of the measurements are displayed in Table 3 and Figure 3. The first column of Table 3 lists a label which was assigned to the nine detectors. The next two columns display the dose delivered at the BIPM and its standard uncertainty, both in Gy. The fourth column is the dose determined by the PTB in Gy. The next column is the uncertainty of the determined dose, but without the contribution of the primary standard, whereas the following column is the combined uncertainty of the

‡ The data that were used to deduce this figure had been collected over a time span of five years. The actual value of the offset may vary from one measurement session to another; it is related to the actual base detectors chosen, the amount of alanine dust present in the holder arrangement, and probably other parameters.

Table 2. Example uncertainty budget for $D^{\mathrm{PTB}} = \mathcal{A}_D$. A test detector consists of 4 pellets irradiated to a dose of 10 Gy, the base is constructed from 8 unirradiated pellets and 8 pellets irradiated with 25 Gy.

Parameter	$10^2\times$ Relative standard uncertainty†		
	s_i		u_i
Base function detectors:			
Primary standard PTB			0.20
Shutter timing	0.06		
Positioning of the detector			0.07
Irradiation temperature			0.05
Average mass of 8 pellets			0.04
Amplitude (25 Gy, 8 pellets)			
Readout repeatability	0.06		
Individual background	0.03		
Intrabatch homogeneity	0.11		
Offset	0.15		
Test detectors:			
Irradiation temperature			0.05
Average mass of 4 pellets			0.05
Amplitude (10 Gy, 4 pellets)			
Readout repeatability	0.20		
Individual background	0.10		
Intrabatch homogeneity	0.15		
Combined uncertainty of the PTB alanine/ESR measurement			
Quadratic summation	0.34		0.23
Combined relative standard uncertainty		0.41	

† expressed as one standard deviation.
s_i represents the relative uncertainty estimated by statistical methods, type A
u_i represents the relative uncertainty estimated by other methods, type B

determined dose including the uncertainty associated with the primary standard [14]. The last two columns finally show the ratio of determined and delivered dose and its relative uncertainty. The average value of the ratio $D^{\mathrm{PTB}}/D^{\mathrm{BIPM}}$ is 0.9973, the standard deviation related to the scatter of these values is 0.0010. The last line in Table 3 shows the weighted mean of the ratio and its associated uncertainty. For the calculation of the weights, only the uncertainties due to the ESR readout are considered. The uncertainties of the BIPM absorbed dose to water detetermination and of the PTB's primary standard are subsequently added, which results in a combined uncertainty of the weighted mean of 0.0038.

The results are also displayed in Figure 3, where the ratio $D^{\mathrm{PTB}}/D^{\mathrm{BIPM}}$ is plotted as a function of the delivered dose D^{BIPM}. The uncertainty bars represent the uncertainty excluding the uncertainty of the primary standard, designated as u_{ESR} in Table 3. The continuous line indicates the average value of the ratio. The ratio of the D_{W} primary standards of the PTB and the BIPM of 0.9961 [19] is indicated by the thick, dashed line; the standard uncertainty of this ratio of 0.0037 is represented by the thin dashed lines.

The mean value of the ratio $D^{\mathrm{PTB}}/D^{\mathrm{BIPM}}$ is therefore 0.0012 higher than the expected ratio of 0.9961. However, within the limits of uncertainty the agreement is satisfactory. The only disconcerting finding is that the deviation of the mean value of 0.0012 from the expectation is approximately of the same size as the scatter of the nine values (0.0010), i.e. the deviation appears to be systematic; however it is well within the measurement uncertainty. The deviation can not be attributed to accidental irradiation during travel: the controls that had been sent along exhibited a signal that was well below the amplitude uncertainty of 25 mGy.

Table 3. Results of the blind test. Columns from left to right: label assigned to the nine detectors, D^{BIPM} is the dose delivered by the BIPM in Gy, $u(D^{\mathrm{BIPM}})$ its uncertainty, D^{PTB} is the dose determined by PTB, u_{ESR} the uncertainty component of the determined dose without the contribution of the primary standard, $u(D^{\mathrm{PTB}})$ is the total uncertainty of the determined dose. $D^{\mathrm{PTB}}/D^{\mathrm{BIPM}}$ is the ratio of determined and delivered dose and the last column lists its relative uncertainty u_r.

Label	D^{BIPM} in Gy	$u(D^{\mathrm{BIPM}})$ in Gy	D^{PTB} in Gy	$u_{\mathrm{ESR}}(D^{\mathrm{PTB}})$ in Gy	$u(D^{\mathrm{PTB}})$ in Gy	$D^{\mathrm{PTB}}/D^{\mathrm{BIPM}}$	u_r
F 01	12.207	0.035	12.157	0.040	0.047	0.9959	0.0049
F 02	17.269	0.050	17.213	0.052	0.063	0.9967	0.0047
F 03	19.652	0.057	19.606	0.058	0.071	0.9977	0.0046
F 04	14.887	0.043	14.872	0.046	0.055	0.9990	0.0047
F 05	10.122	0.029	10.105	0.036	0.042	0.9982	0.0050
F 06	12.203	0.035	12.177	0.040	0.047	0.9978	0.0049
F 07	14.882	0.043	14.834	0.046	0.055	0.9968	0.0047
F 08	10.119	0.029	10.083	0.039	0.044	0.9964	0.0053
F 09	17.264	0.050	17.213	0.052	0.063	0.9971	0.0047
Weighted mean						0.9973	0.0038

4. Summary and Conclusion

In order to verify the reliability of the PTB's secondary standard dosimetry system based on alanine/ESR, a blind test was conducted. Nine detectors consisting of four alanine pellets each were irradiated in the ^{60}Co reference beam at the BIPM. The doses

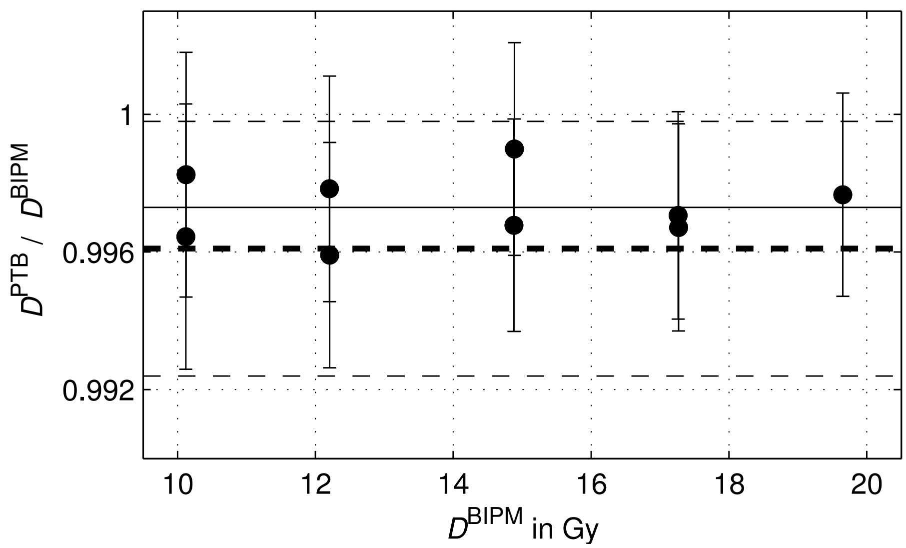

Figure 3. Ratio of determined dose (PTB) to delivered dose (BIPM) as a function of the dose delivered at the BIPM. The uncertainty bars indicate the standard uncertainty of the ESR readout only. The continuous line is the mean of the nine $D^{\mathrm{PTB}}/D^{\mathrm{BIPM}}$ values. Dashed, thick line: ratio of the primary standards for absorbed dose to water of the PTB and BIPM [19]. The thin dashed lines indicate the standard uncertainty of the primary standard ratio.

were restricted to the range between 10 Gy and 20 Gy in order to minimize the influence of noise (the measurement range of the alanine dosimetry system of the PTB is 2 Gy to 25 Gy). The delivered dose values were not communicated before the measurement results were reported to the BIPM.

The absorbed dose to water D_{W} was measured using a slightly simplified procedure, compared to previous publications [15, 16], i.e., without the construction of a calibration curve. On average, the measured dose value was 0.12 % higher than expected from the known difference between the primary standards for D_{W} of the BIPM and the PTB. Compared to the combined measurement uncertainty of the ratio of determined and delivered dose of approximately 0.45%, the agreement is good. As a conclusion, at least for ^{60}Co radiation, the alanine dosimetry system of the PTB appears to yield reliable results within the stated uncertainties. The simplified measurement procedure has been shown to be robust.

Acknowledgments

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