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# Monitoring the validity of calibration results - examples of activities performed by an accredited laboratory

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**Laboratory accredited by the Polish Centre for Accreditation, accreditation No AP 155***

*** an actual scope of accreditation No AP 155 is available on the PCA website: www.pca.gov.pl**

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# Introduction

**Monitoring the validity of calibration results** is one of the requirements of the ISO/IEC 17025:2017 standard entitled “General requirements for the competence of testing and calibration laboratories” [1].
This requirement presented in section 7.7.1 of the aforementioned standard is essential in any calibration field, but especially in the calibration of ionization chambers used by medical physicists during dosimetry measurements for radiation therapy centres.

In this work, **we will present the examples of activities** performed by the Secondary Standards Dosimetry Laboratory (SSDL) in Warsaw in Poland **to meet requirement concerning monitoring the validity of our results**. Our laboratory has been accredited by the Polish Centre of Accreditation and has accreditation No. AP 155 since May 2014. In this work, in particular, **we will discuss those aspects of the procedure we have developed that relate to so-called in-house projects**, i.e. those that are carried out on the premises of our laboratory. In addition, in this work **we will highlight the most important aspects of these activities, such as the establishment of acceptance criteria**. Finally, **we will give some results of monitoring the validity of calibration results and analyze these results according to the dispositions established in our procedure**.

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**Examples of activities** performed by the Secondary Standards Dosimetry Laboratory (SSDL) in Warsaw in Poland **to meet requirement concerning monitoring the validity of our results:**

1. intermediate checks:
    a) on the working standard dosimeter - on each day the customer's dosimeter is calibrated using the working standard dosimeter;
    b) on a reference dosimeter - during calibration of the working standard dosimeter;
2. visual check of the equipment which is not a measuring instrument (i.e., measuring bench, water phantom, ionization chamber holder, etc.) - before each use;
3. comparison values of the calibration coefficient determined during calibration of the customer's dosimeter with calibration coefficient used routinely by the user (i.e., the one determined during the previous calibration) – if data is available;
4. Monitoring atmospheric conditions (ambient temperature, relative humidity, atmospheric pressure) on calibration days;
5. metrological control of an additional measuring equipment (i.e. thermometers, barometers, hygrometers) with the frequency established in the current edition of the procedure we have developed;
6. calibration of the same calibration object performed by different members of the SSDL staff in accordance with the currently valid calibration instruction being within the scope of accreditation No. AP 155.

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For each four-year accreditation cycle, the head of the Polish SSDL establishes a **schedule** for monitoring the validity of the calibration results of the previously mentioned in-house projects.

This schedule is recorded on the current issue of the Laboratory Management System form.
This schedule establishes the frequency of execution of each project and the criteria for accepting the results.

The completed form with **schedule is reviewed** by the head of the Polish SSDL or his deputy **at least once a year** (at least every 12 months) **for its suitability** - especially in the case of changes regarding measuring instruments, measuring equipment, personnel performing calibrations and calibration methods.
Records of the review are made by the head of the Polish SSDL or his deputy on the current issue of the Laboratory Management System form called „Review of the schedule for monitoring the validity of calibration results – in-house projects".

Based on the current schedule of activities to monitor the validity of calibration results, **the Polish SSDL staff performs the scheduled activities and prepares records of these activities** on the form called „Report on activities to monitor the validity of calibration results – in-house projects".

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# Intermediate checks on the working standard dosimeter

- checks are performed **on each day the customer's dosimeter is calibrated using the working standard dosimeter**;
- checks are carried out by the employee performing the calibration of the customer's dosimeter;
- the result of each intermediate check is recorded on the current issue of the Laboratory Management System form, called a calibration book;
- Δ parameter is adopted at the Polish SSDL as a measure of the intermediate check result on the working standard dosimeter.

**dosimeter: electrometer with ionization chamber**

The Δ parameter value, in %, is calculated according to the formula (1):

$$\Delta = \frac{|D_{\text{mean}} - D_{\text{cal, x}}|}{D_{\text{mean}}} 100 \qquad (1)$$

where:
$D_{\text{mean}}$ is the mean value of the absorbed dose to water in cGy calculated by the formula (2) [2]:

$$D_{\text{mean}} = \left(\frac{\sum_{i=1}^{n} M_i}{n}\right) N_{D,\text{W}} k_{Tp} \qquad (2)$$

where:
$M_i$ is the charge measured, in nC, with the ionization chamber of the working standard in the *i* successive measurement; ***n***, equal to 10, is number of successive measurements of the charge $M_i$ in a given measurement series; $N_{D,\text{w}}$ is the calibration coefficient of the working standard, in cGy/nC, given in its actual calibration certificate; $k_{Tp}$ is factor to correct the response of an ionization chamber of working standard for the effect of the difference that may exist between the reference and actual environmental conditions;
$D_{\text{cal, x}}$ is the value of the absorbed dose to water, in cGy, calculated on a day of the intermediate check on the working standard taking into account the decay of the $^{60}$Co with a half-life of 5.2711 years, i.e. value recommended by International Committee for Radionuclide Metrology, which was converted to 1925.2 days for our purposes, calculated according to the formula (3):

$$D_{\text{cal, x}} = D_{\text{cal, 0}} e^{-\frac{x \ln 2}{1925.2}} \qquad (3)$$

where:
$D_{\text{cal, 0}}$ is the value of the absorbed dose to water, in cGy, calculated according to Eq. (2) with the use of the reference standard for calibration of the working standard on day “0”; **x** is number of days since day „0” to the day of the intermediate check.

Value of $k_{Tp}$ factor was calculated by Eq. (4) [2]:

$$k_{Tp} = \frac{p_{\text{ref}}}{p} \frac{T_{[K]}}{T_{\text{ref}[K]}} = \frac{p_{\text{ref}}}{p} \frac{273.15 + T_{[°C]}}{273.15 + T_{\text{ref}\,[°C]}} \qquad (4)$$

where:

$T_{\text{ref}\,[°C]}$ = 20 °C or $T_{\text{ref}\,[K]}$ = 293.15 K is the reference temperature; $p_{\text{ref}}$ is the reference pressure specified by the calibration laboratory in its actual calibration certificate, equal to 1013 hPa; $T_{[°C]}$ or $T_{[K]}$ is the temperature measured during intermediated check on given working standard, respectively, in °C or K; ***p*** is the pressure measured during intermediate check on given working standard, in hPa.

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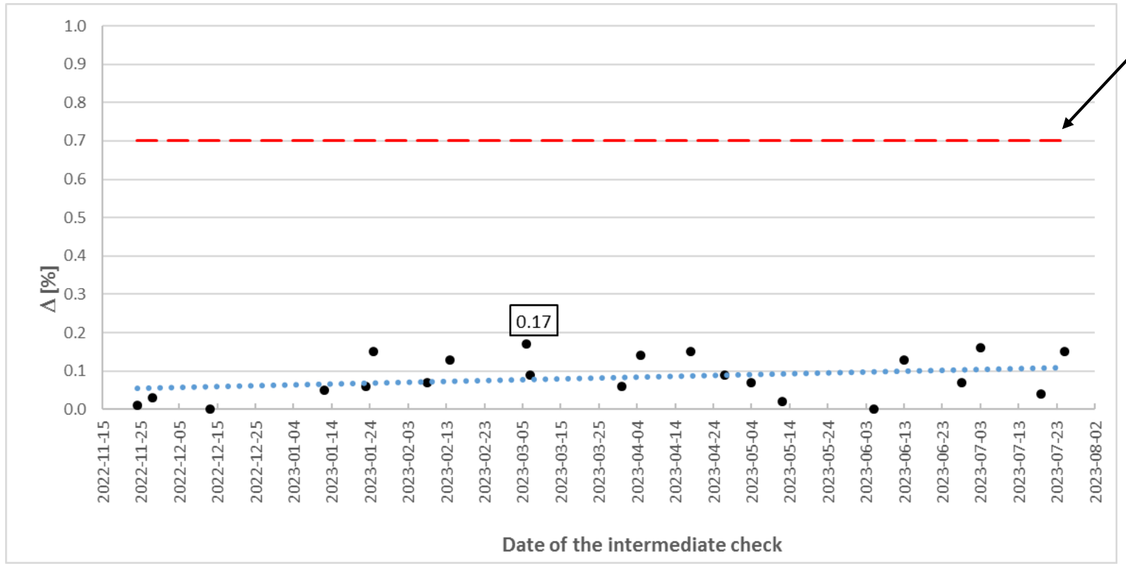
# Intermediate checks on the working standard dosimeter

- the value of 0.7% is adopted as an acceptance criterion of the Δ parameter [3].

**Exemplary results for the working standard dosimeter No. 1**




**The red dashed line represents acceptance criterion: Δ = 0.7 %**

Δ parameter reached values of less than 0.20%, so all the values obtained for this parameter were more than two times smaller than the acceptance criterion.

**Normality**

The assumption was checked based on the Shapiro-Wilk Test (α = 0.05). It is assumed that **residuals' distribution does** follow the normal distribution (*p*-value is 0.7179), or more accurately, we can't reject the normality assumption.

**Fig. 1** **Values of the Δ parameter as a function of time for working standard dosimeter No. 1; the number in the text box is the largest value of Δ parameter.**

**Outliers**

Outliers' detection method: Tukey Fence, *k* = 1.5. The data doesn't contain outliers.

**Table 1 Pearson correlation results**

| Parameter | Value |
|---|---|
| Pearson correlation coefficient (r) | **0.2998** |
| P-value | 0.1753 |
| Covariance | 1.2599 |
| Sample size (n) | 22 |
| Statistic | 1.4054 |

**Table 2 Strength of linear relationship [4]**

| Correlation coefficient value: *r* | Strength of linear relationship |
|---|---|
| **at least 0.8** | **very strong** |
| **0.6 up to 0.8** | **moderatly strong** |
| **0.3 to 0.5** | **fair** |
| **less than 0.3** | **poor** |

Results of the Pearson correlation indicated that at the significance level α = 0.05 there is a **non significant fair positive** relationship between X and Y, ($r(20) = 0.3$, $p = 0.175$).

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# Intermediate checks on the reference dosimeter

- checks are performed **during calibration of the working standard dosimeter** using the reference dosimeter;
- checks carried out by the employee performing the calibration of the working standard dosimeter;
- the result of each intermediate check is recorded on the current issue of the Laboratory Management System form, called a calibration book;
- Δ parameter is adopted at the Polish SSDL as a measure of the intermediate check result of the reference dosimeter.

**dosimeter: electrometer with ionization chamber**

The Δ parameter value, in %, is calculated according to the formula (1):

$$\Delta = \frac{|D_{\text{mean}} - D_{\text{cal, x}}|}{D_{\text{mean}}} 100 \qquad (1)$$

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where:
$M_i$ is the charge measured, in nC, with the ionization chamber of the reference standard in the *i* successive measurement; ***n***, equal to 10, is number of successive measurements of the charge $M_i$ in a given measurement series; $N_{D,\text{w}}$ is the calibration coefficient of the reference standard, in cGy/nC, given in its actual calibration certificate; $k_{Tp}$ is factor to correct the response of an ionization chamber of reference standard for the effect of the difference that may exist between the reference and actual environmental conditions;
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where:
$D_{\text{cal, 0}}$ is the value of the absorbed dose to water, in cGy, calculated according to Eq. (2) with the use of the reference standard for calibration of the working standard on day "0"; **x** is number of days since day „0" to the day of the intermediate check.

Value of $k_{Tp}$ factor was calculated by Eq. (4) [2]:

$$k_{Tp} = \frac{p_{\text{ref}}}{p} \frac{T_{[K]}}{T_{\text{ref}[K]}} = \frac{p_{\text{ref}}}{p} \frac{273.15 + T_{[°C]}}{273.15 + T_{\text{ref}\ [°C]}} \qquad (4)$$

where:

$T_{\text{ref}\ [°C]}$ = 20 °C or $T_{\text{ref}\ [K]}$ = 293.15 K is the reference temperature; $p_{\text{ref}}$ is the reference pressure specified by the calibration laboratory in its actual calibration certificate, equal to 1013 hPa; $T_{[°C]}$ or $T_{[K]}$ is the temperature measured during intermediated check on given working standard, respectively, in °C or K; ***p*** is the pressure measured during intermediate check on given working standard, in hPa.

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# Intermediate checks on the reference dosimeter

- the value of 0.7% is adopted at the Polish SSDL as an acceptance criterion of the Δ parameter [3].

**Exemplary results for the reference standard dosimeter No. 1**

| Date of the intermediate check | Δ Parameter [%] |
|---|---|
| 25 November, 2020 | 0.10 |
| 09 March, 2022 | 0.03 |
| 17 November, 2022 | 0.03 |
| 13 June, 2023 | 0.13 |

Δ parameter reached values of less than 0.15%, so all the values obtained for this parameter were more than four times smaller than the acceptance criterion.

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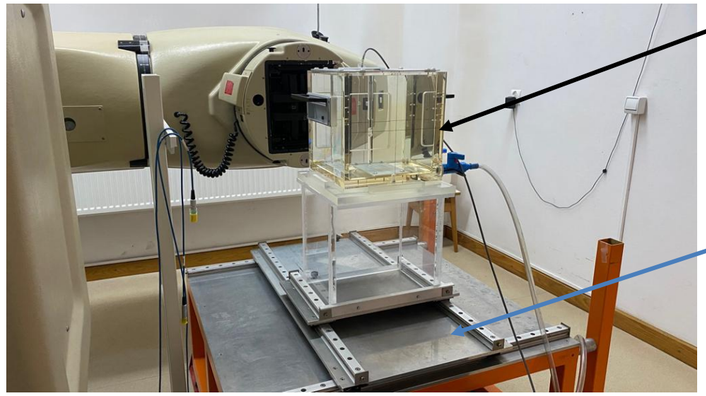
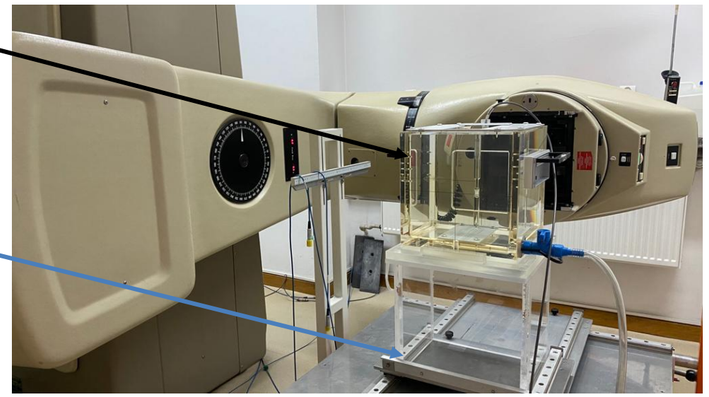
# Visual check of the equipment not being a measuring instrument

- check is performed for the equipment not being a measuring instrument (i.e., measuring bench, water phantom, ionization chamber holder, etc.);
- check is carried out before each use of this equipment by the employee performing the calibration of the dosimeter;
- the results of this check are recorded on the current issue of the Laboratory Management System form, called a calibration book;
- acceptance criterion: no visible damage to equipment not being a measuring instrument;
- results: checks revealed no apparent damage to any equipment not being a measuring instrument during the analyzed period.

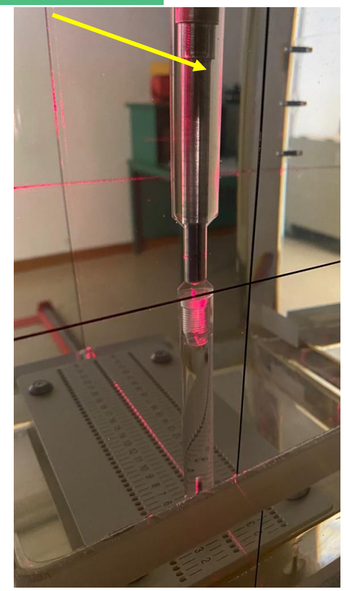


water phantom
PTW-Freiburg,
type 4322

measuring bench



ionization chamber holder
PTW-Freiburg, type 4333/ U13



**Calibration set in a $^{60}$Co gamma ray beam of the Theratron 780E unit at the Polish SSDL**

**Note:**
**The measuring bench allows the water phantom with the ionization chamber to be positioned at an appropriate distance from the radiation source.**
**The ionization chamber holder allows the ionization chamber to be placed in the water phantom so that the reference point of the ionization chamber is at the correct depth in the water phantom.**

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# Comparison values of the calibration coefficient

- values of the calibration coefficient determined during calibration of the customer's dosimeter are compared with the values of the calibration coefficient determined during the previous calibration;
- comparisons are performed after calibration of the customer's dosimeter by the employee performing the calibration of the customer's dosimeter for which data on the value of the coefficient of the previous calibration is available;
- the results of these comparisons are recorded on the current issue of the Laboratory Management System form, called a calibration book;
- acceptance criterion: percentage difference between the calibration coefficients of the customer's dosimeter calibrated in a $^{60}$Co gamma ray beam not greater than ±1,00% (within 2 years).

**Exemplary results**

| Customer's dosimeter | $N_{D,w}$ of 2023 vs. $N_{D,w}$ of 2021 | $N_{D,w}$ of 2022 vs. $N_{D,w}$ of 2019 |
|---|---|---|
| UNIDOS type 10001 with ionization chamber type TM30013 | - 0.17% | 0.13% |
| UNIDOS webline type T10021 with ionization chamber type TM30013 | -0.20% | 0.15% |
| UNIDOS type T10001 with ionization chamber type TM31010 | -0.32% | 0.10% |
| DOSE 1 with ionization chamber type FC65-G | 0.02% | -0.04% |
| UNIDOS type 10001 with ionization chamber type TM34045 | 0.00% | 0.27% |
| UNIDOS type 10001 with ionization chamber type TM34045 | 0.14% | 0.24% |
| UNIDOS webline type T10021 with ionization chamber type TM31022 | -0.24% | 0.11% |
| UNIDOS type 10001 with ionization chamber type TM30001-1 | 0.11% | -0.06% |
| UNIDOS E with ionization chamber type TM31016 | -0.09% | -0.23% |
| UNIDOS webline type T10021 with ionization chamber type TM23343 | 0.24% | -0.07% |
| DOSE 2 with ionization chamber type PPC-05 | -0.46% | -0.19% |
| DOSE 1 with ionization chamber type PPC-05 | -0.23% | -0.15% |

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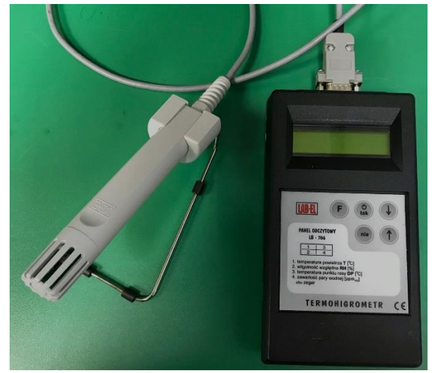
# Monitoring atmospheric conditions

- monitoring atmospheric conditions (ambient temperature, relative humidity, pressure) takes place on each calibration day (at the begining, during and at the end of the calibration);
- the results of these comparisons are recorded on the current issue of the Laboratory Management System form, called a calibration book;
- acceptance criterion:
  - ambient temperature from 15 °C to 25 °C;
  - relative humidity from 20% to 80%;
  - the air pressure from 950 hPa to 1050 hPa.

**Exemplary equipment useful to monitoring atmospheric conditions at the Polish SSDL**


**The digital barometer by VAISALA, type PTB330**


**The digital waterproof thermometer by Elmetron, type PT-401**




**Thermohygrometer by LAB-EL, type LB-706 with a sensor type LB-701**

**Note:**
**Calibration of the customer's dosimeter is carried out only if the acceptance criteria for atmospheric conditions are met.**

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# Metrological control of an additional measuring equipment

- control performed with the frequency established by the Polish SSDL in the current edition of procedure PR22.5_P2 *Management of calibration equipment;*
- the results of this control are recorded by the employee of the Polish SSDL on the current issue of the Laboratory Management System forms, which are a calibration book and a card of the given measuring instrument;
- acceptance criterion:
  - ✓ the difference between the readings of the reference thermometer and the thermometer to be checked should not exceed 0.5 °C;
  - ✓ the difference between the readings of the reference barometer and the barometer to be checked should not exceed 5.0 hPa;
  - ✓ the absolute value of the difference between the readings of the reference hygrometer and the hygrometer to be checked should not exceed 15% of the reference hygrometer reading.

**Note:**
If the absolute value of the difference between the readings of the reference thermometer and the thermometer to be checked exceeds 0.5 °C the thermometer should be marked with the label "Damaged" and should be taken out of service.
If the abolute value of the difference between the readings of the reference barometer and the barometer to be checked exceeds 5.0 hPa the thermometer should be marked with the label "Damaged" and should be taken out of service.
If the absolute value of the difference between the readings of the reference hygrometer and the hygrometer to be checked exceeds 15% of the reference hygrometer reading the hygrometer should be marked with the label "Damaged" and should be taken out of service.

## Exemplary results

**Date of the control: 04 December, 2023 Frequency: every 6 months**

| The reference thermometer | Thermometer No. 1 | Thermometer No. 2 | Thermometer No. 3 | Thermometer No. 4 |
|---|---|---|---|---|
| The readings of the thermometer [°C] | | | | |
| 22.50 | 22.70 | 22.50 | 22.40 | 22.60 |
| 22.50 | 22.60 | 22.50 | 22.35 | 22.55 |
| 22.50 | 22.60 | 22.55 | 22.45 | 22.60 |
| The mean value of the thermometer readings [°C] | | | | |
| 22.5 | 22.6 | 22.5 | 22.4 | 22.6 |
| Corrections [°C] | 0.1 | 0.0 | 0.1 | -0.1 |

**Date of the control: 13 October, 2023 Frequency: every 12 months**

| | The reference hygrometer | Hygrometer No. 1 |
|---|---|---|
| The value of the humidity [%] | 53.4 | 52.5 |
| | 53.4 | 52.5 |
| | 53.4 | 52.5 |
| The mean value of the humidity [%] | 53.4 | 52.5 |
| The absolute value of the difference between the readings of the reference hygrometer and the hygrometer No. 1 [%] | | **0.9 (< 15% of the the mean value of the reference hygrometer readings)** |

**Date of the control: 01 June, 2023 Frequency: every 12 months**

| Barometer | The reference barometer | Barometer No. 1 | Barometer No. 2 |
|---|---|---|---|
| The value of the air pressure [hPa] | 1006.91 | 1007.09 | 1007.0 |
| | 1006.92 | 1007.10 | 1007.0 |
| | 1006.90 | 1007.07 | 1007.0 |
| The mean value of the air pressure [hPa] | 1006.91 | 1007.10 | 1007.0 |
| Corrections [hPa] | | **-0.2** | **-0.1** |

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# Calibration of the same calibration object performed by different members of the SSDL staff

- calibration of the same calibration object performed by different members of the SSDL staff in accordance with the currently valid calibration instruction being within the scope of accreditation No. AP 155;
- frequency: during the periodic comparison between SSDL staff - at least once for each calibration method until the end of the respective accreditation cycle;
- acceptance criterion: the quotient, expressed as a percentage, of the value of the calibration factor *N* determined by employee 1 and the value of the calibration factor *N* determined by employee 2 should be in the range ⟨99.0%; 101.0%⟩.

**Exemplary results**

| Parameter | Employee 1 (called MS) (calibration date: 04 January, 2023) | Employee 2 (called WSK) (calibration date: 05 January, 2023) |
|---|---|---|
| $N_{D,w}$ [cGy/nC] | **5.389** | **5.392** |
| $N_{D,w}^{\text{Employee 1}} / N_{D,w}^{\text{Employee 2}}$ [%] | **99.9** | |

| Parameter | Employee 1 (called WSK) (calibration date: 05 January, 2023) | Employee 1 (called IG) (calibration date: 10 January, 2023) |
|---|---|---|
| $N_{D,w}$ [cGy/nC] | **5.392** | **5.388** |
| $N_{D,w}^{\text{Employee 1}} / N_{D,w}^{\text{Employee 2}}$ [%] | **100.1** | |

**The results met the predetermined acceptance criterion.**

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# Important issues and conclusions

**Failure to accept the results of the analysis of data from the monitoring the validity of calibration results:**

- **requires taking appropriate action to prevent the inclusion of incorrect results in the report of calibration results issued to the calibration laboratory's customer;**
- **is the basis for analyzing the situation in the context of the risk of a non-conforming calibration** and, if necessary, to take the appropriate actions established in the current edition of the procedure relating to this type of work.

Practical examples, presented in this work, can be useful to other calibration laboratories to **meet requirement concerning monitoring the validity of calibration results** and, with minor modifications, can undoubtedly be applied to many areas of calibration.

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# References

[1] INTERNATIONAL ORGANIZATION FOR STANDARDIZATION, General requirements for the competence of testing and calibration laboratories, ISO/IEC 17025:2017, ISO, Geneva (2017)

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[4] Chan YH, Biostatistics 104: Correlation Analysis. Singapore Med J 2003; Vol 44(12): 614-619

**Thank you for your attention.**