SAUNA
Standard Calibration Procedure

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Sammanfattning

Detta dokument innehåller en förbättrad kalibreringsprocedur för SAUNA II och SAUNA III system för insamling och detektion av radioaktivt xenon. Dokumentet innehåller detaljerade procedurer för energi- och effektivitetskalibrering av detektorer (avsnitt 2) samt kalibrering av parametrar i gasprocessen (avsnitt 3): gaskromatograf (GC), överföringsfunktioner samt dödvolym.

Nyckelord: Ädelgas, Xenon, SAUNA, Kalibrering, CTBT

Summary

This document describes an improved standard calibration procedures for SAUNA II and SAUNA III systems for collection and detection of radioxenon. It includes the detailed calibration procedures for the detector energy and efficiency calibration (section 2) and the calibration of the gas processing parameters (section 3) such as the gas chromatograph (GC), transfer functions and dead volume.

Keywords: Noble gas, Xenon, SAUNA, Calibration, CTBT

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1 Introduction

This document describes an improved standard calibration procedures for SAUNA II and SAUNA III systems. It constitutes the report on Task 1 required under the contract No. 2016-1614 between the Preparatory Commission for the CTBTO and the Swedish Defence Research Agency (FOI), “Development of SAUNA Noble Gas System Calibration and Gain Stabilization”.

The objective of Task 1 of the contract is to develop improved standard calibration procedures for SAUNA systems, including a software tool, developed in Task 2 of the contract, to be used when determining the system calibration parameters.

This document includes the detailed calibration procedures for the detector energy and efficiency calibration (section 2) and the calibration of the gas processing parameters (section 3) such as the gas chromatograph (GC), transfer functions and dead volume.

2 SAUNA Detector Calibration Procedure

The detector calibration consists of a set of measurements on solid point sources as well as gaseous sources of xenon and radon, injected directly into the detector using a syringe. The measurements are then analyzed using the software Xeff2 to determine the energy calibration of the beta- and gamma detectors, setting of ROI limits, and to determine ROI efficiencies and interference factors. The result is a set of calibration blocks stored in the SAUNA PostgreSQL database and written to the IMS 2.0 PHD-files resulting from the air sample measurements performed by the system.

2.1 Step by Step Procedure

1. Initial high voltage and gain settings
    a. Set the high voltage at the plateau for the individual PM-tubes.
    b. Place a Cs-137 point source inside the NaI at the center of the beta cell (QC position).
    c. Adjust gain to get the 662 keV gamma peak at channel 230.
    d. Gain match the two beta PM-tubes to get the beta endpoint energy in the channel range 150 – 155.
2. Gamma energy calibration
   Perform individual measurements of point sources (Cs-137, Am-241, Eu-152), each with an activity of ca 1 kBq.
    a. Cs-137, placed inside the NaI at the center of the beta cell (QC position).
    b. Am-241, placed inside the NaI at the center of the beta cell (QC position).
    c. Eu-152, placed on top of the NaI detector.
3. Beta energy calibration
   Perform measurements of a point source (Cs-137), with an activity of ca 1 kBq
    a. Cs-137, placed inside the NaI at the center of the beta cell (QC position).
    b. The Cs-137 spectrum shall be recorded during 5 to 6 hours to yield a beta-gamma diagonal with good statistics.

   Note: the beta energy calibration measurement may be the same measurement as the gamma energy calibration measurement with the Cs-137 source, if the conditions for both measurements are fulfilled.
4. Master QC measurement (applicable for systems with gain stabilization)
    a. Close the lead shield, if open.
    b. Position the QC point source, consisting of Cs-137 and Eu-154, inside the NaI at the center of the beta cell (nominal QC position).
       Note: some systems may be equipped with a QC source consisting of Cs-137 and Eu-152.

   c. Perform a measurement of the QC source during 6 to 7 hours.

5. Detector background measurement.
   a. Close the lead shield, if open.
   b. Make sure that the QC sources are at their parking positions.
   c. Make sure that other sources are not present in the vicinity of the detector. Also make sure that the gas drain of the system, which may contain residual activity, is fed outside the room and that the detector cell is not filled with xenon from sampled air during the detector background measurement.
   d. For present SAUNA II systems (without gain stabilization implemented), start a detector background measurement and collect data during at least three days.
      For systems with gain stabilization implemented, start the data acquisition and collect detector background data during at least four days, including QC measurements and drift correction according to the standard system measurement cycle timing. When the measurement is complete, the resulting individual background spectra shall be added together into one single detector background spectrum.
6. Perform a QC measurement during 1 hour, followed by drift correction (applicable to systems with gain stabilization).

The following steps relate to measurements to determine detector efficiency and inter-isotope interference factors. In the measurements of radio-xenon gas samples the detector, after the radio-xenon has been injected, shall be filled with stable xenon to the nominal xenon volume and carrier gas to the nominal pressure. For laboratory systems, the xenon measurements shall be performed with three different xenon volumes, 1 ml, 3 ml and 5 ml, respectively.

7. **Measurement A**: Perform a measurement of a Xe-133 gas sample with an activity in the range 50 – 100 Bq, as isotopically pure as possible.
8. During flushing and pumping of the cell, perform a QC measurement during 1 hour, followed by drift correction (applicable to systems with gain stabilization).
9. **Measurement B**: Perform a measurement of a Xe-131m gas sample with an activity in the range 25 – 100 Bq, as isotopically pure as possible.
10. During flushing and pumping of the cell, perform a QC measurement during 1 hour, followed by drift correction (applicable to systems with gain stabilization).
11. **(Optional) Measurement E**: Perform a measurement of a Xe-133m gas sample, as isotopically pure as possible.
12. During flushing and pumping of the cell, perform a QC measurement during 1 hour, followed by drift correction (applicable to systems with gain stabilization).
13. **(Optional) Measurement F**: Perform a measurement of a Xe-135 gas sample, as isotopically pure as possible.

14. During flushing and pumping of the cell, perform a QC measurement during 1 hour, followed by drift correction (applicable to systems with gain stabilization).

15. **Measurement C**: Perform a radon gas background measurement. This is done by performing a standard gas background measurement, with a measurement time of at least 2 to 3 hours (applicable to both SAUNA II and SAUNA III systems).

16. Perform a QC measurement during 1 hour, followed by drift correction (applicable to systems with gain stabilization).

17. **Measurement D**: Perform a measurement of a Rn-222 sample. It is important that the length of this measurement is the same as a standard sample measurement, i.e. 11 h 10 min for a SAUNA II system. This is necessary in order to have the same radon to radon daughter ratios in this radon measurement as in a standard sample measurement that includes radon.
    Take radon from a radon source and inject the radon into the evacuated detector cell through the injection port to a count rate of 10 – 20 Bq.
    Add carrier gas to reach atmospheric pressure in the cell. Start the measurement after a time delay equal to the time delay of the standard measurement cycle of the system between the gas injection to the cell and the start of measurement.

The standard radon source used for SAUNA systems is a small, ca 25 ml, plastic gas-tight container containing uranium rich rock. Based on experience, the radon concentration in this type of source is sufficient to give the desired activity in roughly 1 ml gas from the source when it first has been at rest for a few days to a week.

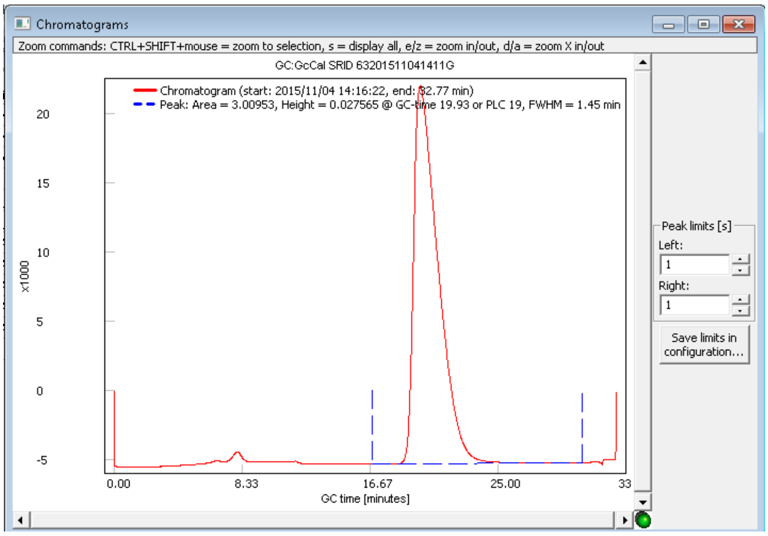
2.2 Detector Calibration Analysis Procedure

When the detector calibration measurements have been performed and the calibration data are available, the analysis of the calibration data is performed using the calibration software, Xeff2. Details on how to perform the analysis is described in reference [1].

3 SAUNA Gas Process Calibration Procedures

3.1 Gas Chromatograph Calibration

A chromatogram is measured using the TCD signal from the gas chromatograph (GC). Peak areas in the chromatogram are proportional to concentrations of eluting substances.



Zoom commands: CTRL+SHIFT+mouse = zoom to selection, s = display all, e/z = zoom in/out, d/a = zoom X in/out

GC:GcCal SRID 63201511041411G

Chromatogram (start: 2015/11/04 14:16:22, end: 32.77 min)

Peak: Area = 3.00953, Height = 0.027565 @ GC-time 19.93 or PLC 19, FWHM = 1.45 min

Peak limits [s]

Left: 1

Right: 1

Figure 3.1. A chromatogram showing the TCD signal, in red, from the SEL63 laboratory system taken from a GC calibration. The xenon peaks retention time is about 20 minutes and the integration limits, in blue, are set to provide a suitable baseline subtraction.

The GC calibration is a semi-automatic procedure (see below) where the system is calibrated several times by injecting a known amount of stable xenon. This is performed using a built-in injection loop with a calibrated volume which is filled from a gas standard containing pure xenon gas.

The resulting xenon volume (in sccm) versus integrated area is fitted to a straight line and the resulting polynomial is used as the calibration of the GC.

The step-by-step procedure for the GC calibration using the installed calibration loop follows below.

3.1.1 Step-by-Step Procedure

The following is the general step-by-step procedure that should be followed but the details and figures are only valid for a SAUNA II system. For other systems deviations in software and hardware might be found and the accurate procedure in the system specific manual should be followed.

Perform at least 5 calibration measurements over the range -50 to +20% of the nominal stable xenon volume for the system.

1. Press the F4 calibration button on the PLC front panel. Check that a new chromatogram is created and running in the GC tab (this may take up to a minute). Make a note in the log book. Also check that a new line is created in the GC Calib tab spreadsheet.

2. Make sure that the SAV is in the “Normal Operation” position, see figure 3.2.

3. Make sure that the valve TV03 is closed. Located on the tube from the Xe bottle regulator, see figure 3.3.

4. Start the calibration pump, to evacuate the calibration loop, see figure 3.2.

5. Open TV01 and TV02, see figure 3.2. They are open when the lever points outwards.

6. Make sure the pump is working by looking at the calibration loop pressure. The pressure is given by SOH sensor PT20 (Cal. loop).

7. Check that the pressure on the xenon regulator is set to wanted pressure covering the range -0.3 to 0.8 bar. For example 0.4 bar gives 1.3 sccm Xe, see figure 3.3. When the pressure needs to be reduced this should be checked while flushing, see step 8.b.
   Info: The area under the xenon peak corresponds to (PT20/1.013)(273/(273+T01)) ml of xenon after normalization to standard pressure and temperature (STP). (The calibrated volume holds 1 ml of xenon gas).*

8. At GC-time 40 minutes flush the calibrated volume with Xe:

   a. Close TV02.

   b. Open TV03 shortly and close it again. Read the calibration loop pressure (PT20) and check that it is within the desired range.

   c. Make sure that TV03 is closed.

   d. Open valve TV02 (with TV03 closed) to evacuate the calibrated volume.

   e. Repeat steps a. to d. at least three times, this will flush the loop.

9. At GC time 55 minutes fill the calibrated volume with xenon by:

   a. Close TV02.

   b. Open TV03.

   c. Close TV01 followed by TV03. Read the calibration loop pressure (PT20).

   d. Log the calibration loop pressure (PT20) and temperature (T01) in the GC Calib tab (or in the spreadsheet GC tab).

   e. Shut off the calibration pump.

10. At about GC-time 60 minutes (see system specific information) turn the SAV to the “GC Calibration” position. This injects the xenon gas sample into the GC.

11. Wait 10 minutes then turn the SAV back to the “Normal Operation” position.

12. A peak in the chromatogram will appear after about 100 – 120 minutes GC-time, depending on system settings. At the time for the xenon peak the flow in drain port PC95 on the STU should be measured to check if the flow has changed. Note the flow in the logbook.

13. The Xe peak area is calculated by the system software and automatically transferred into the GC Calib tab after about 125 minutes GC-time.

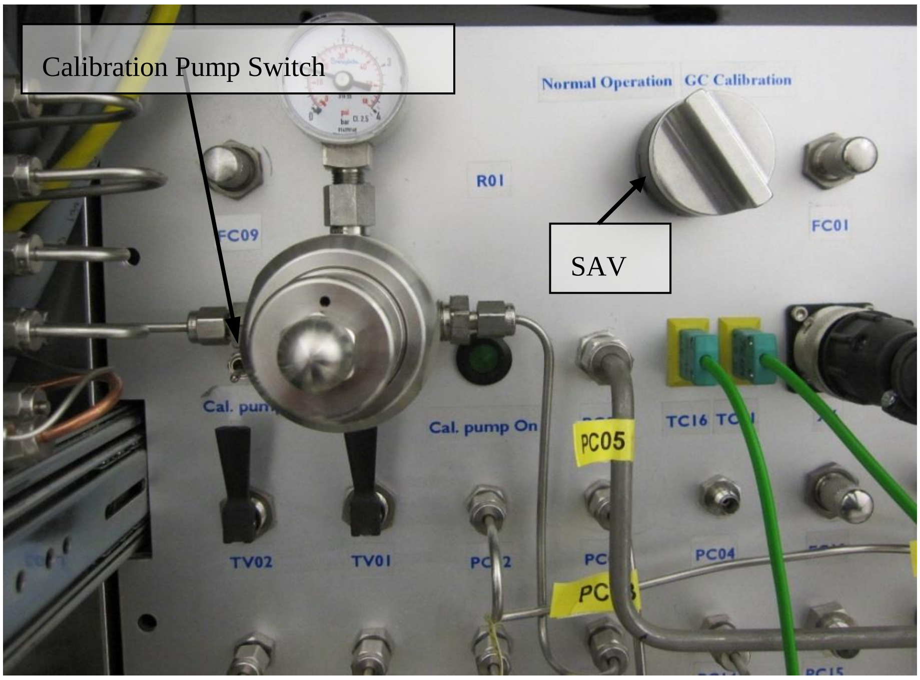


Figure 3.2. The calibration loop (SAV) and the valves TV01 and TV02, located on the back of the PVU. The switch for the calibration pump is located above the valves. The lamp indicating that the calibration pump is operating is seen located to the right of the regulator R01. Note that the indication lamp might be positioned different on older systems.

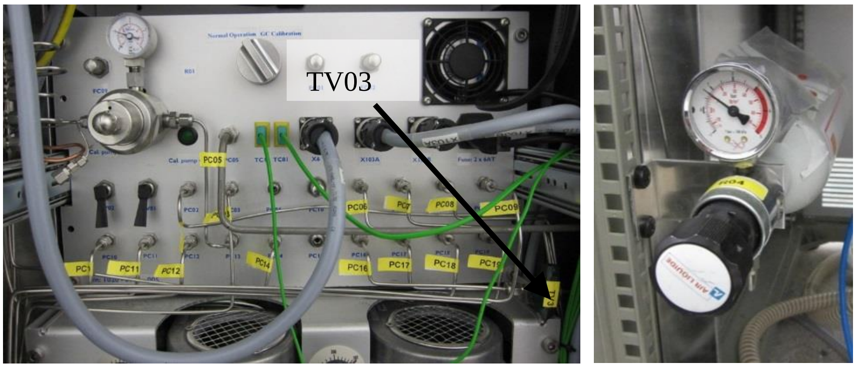


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Figure 3.3. Location of valve TV03 and the Xe bottle with regulator R04. The location of TV03 might differ on older systems. The Xe bottle is located below the SCU in the sampling rack, on older systems this might also differ.

3.1.2 Analysis Procedure

The analysis procedure can be summarized as:

1. Enter all data (loop volume, temperature and pressure, and integrated area) with uncertainties (taken from certificates, see below for details) into the input file for the ROOT script *SAUNA_GC_Cal.cpp*. Most default uncertainties given in the input file should be applicable for any SAUNA system, and an example is given in Appendix B.

2. Run the ROOT script (to be integrated into the system software in the future), *SAUNA_GC_Cal.cpp*. To do this, in ROOT, execute the command:
   .x SAUNA_GC_Cal("indata.txt","outdata.txt")
   where *indata.txt* is the input file and *outdata.txt* the wanted output file.

3. This will give the resulting calibration and confidence intervals as outputs, these should be inserted into the system software (to be implemented in the future).
   Note that the coverage factor for the output is set with the input data, currently the default is k=1.

These steps and calculations are described in detail below.

The amount of xenon, V_{CAL} in sccm (standard cubic centimetres), in each calibration injection is calculated from:

$$V_{CAL} = C_{Xe} V_{INJ} \frac{P_{INJ}}{P_{STP}} \frac{T_{STP}}{T_{INJ}}\,, \qquad (1)$$

where C_{Xe} is the gas purity in the xenon bottle, and V_{INJ}, P_{INJ} and T_{INJ} are the volume, pressure and temperature (in Kelvin) for the loop. The values labelled “STP” are used to correct all measured values to STP, 0 °C and 1 atm.

Calibration measurements should be repeated for at least five different xenon volumes, with a spread of xenon volume covering at least -50 to +20% of the nominal stable xenon volume for the system. The reason for the asymmetry is two-fold: first smaller volumes of xenon are more likely, and might be experienced when the system mal-functions; secondly larger volumes of xenon might saturate the GC detector. For example if a SAUNA II system with a nominal stable xenon volume of 1.4 sccm is to be calibrated the injected xenon volumes should cover the range: 0.7 – 1.7 sccm (if allowed by the pressure sensor). Care has to be taken to never go outside the calibrated range of the pressure gauge in the injection volume. For a SAUNA II system this means that no pressure above 1.7 bar, volumes above 1.6 sccm (corresponding to a 1 ml loop and 1.7 bar pressure STP corrected to 0°C and 1 atm), should be used in the GC calibration . The number of calibrations and the range are just the minimal requirements and a larger set of data will result in a better calibration.

The calibration function is given as a linear fit to the measured xenon volumes and peak areas. The calculation is performed using the ROOT script *SAUNA_GC_Cal.cpp* which performs a weighted linear fit to the data. The resulting fit yields the polynomial coefficients and the confidence intervals over the full range of the calibration, see below.

For a measurement the xenon volume is then given by:

$$V_{Xe} = p_0 + p_1 \times A\,, \qquad (2)$$

where A is the peak area in the chromatogram and p_0 and p_1 the resulting coefficients from the calibration. All uncertainties are handled in the ROOT script, and are described in this document.

3.1.2.1 Uncertainty Analysis

The uncertainty analysis is described in detail in this section. First the uncertainties in the GC calibration measurement data, due to the injected xenon volume and the integrated area, are calculated. Due to the mix of statistical and systematic contributions to the uncertainty in the calibration the resulting uncertainty from the calibration is pre-calculated over the interval of areas where the calibration is valid, and not as uncertainties in the polynomial coefficients resulting from the fit. This uncertainty from the calibration is then added to the uncertainty from the measured area in the last step to calculate the total uncertainty in the measured xenon volume.

The total standard uncertainty, due to the calibration, in the measured xenon volume (V_{Xe}) is given by:

$$u_{cal}(V_{Xe}) = \sqrt{u_A^2 + u_B^2}\,, \qquad (3)$$

where u_A is the total contribution of all type A uncertainties which are handled statistically, see reference [2] and references therein, and reference [3], and u_B all type B uncertainties. The resulting type A uncertainty, u_A, in the calibration is the confidence interval given by evaluating the linear fit.

In the fit only the uncertainty in the peak area is used, since this is the only type A contribution. The uncertainty in the area consists of two parts: the peak integral and the baseline subtraction. The relative standard uncertainty in the peak integral is set to be ±0.7 % from evaluation of system data (1.2 % maximal). Note that all uncertainties throughout this section are given as maximal ranges of the uncertainty and are divided with $\sqrt{3}$, to get the standard uncertainty (k=1), which is then used in all calculations. The standard uncertainty in the baseline subtraction is assumed to be constant, typically resulting in about 0.2 % of the peak area corresponding to 1 sccm of xenon. Standard uncertainty in the injected xenon volume, $u(V_{CAL})$ where V_{CAL} are the measured xenon volumes in the calibration, is set to zero in the fit since all these contributions to the uncertainty are type B and are included after the linear fit is performed.

Uncertainties in the independent variable in the fit are handled using the effective variance method as described in reference [4], p23-24, equation 3.7. The uncertainty in the evaluated values of the fitted functions are determined by the standard error propagation formula taking covariance into account, eq. 3.8 in the reference above, and the uncertainty from the fit is also corrected in the same way for the actual χ^2 values using:

$$u_A = u_A^{fit} \times Q \times \sqrt{\frac{\chi^2}{dof}}\,, \qquad (4)$$

with Q being the Student's t-distribution quantile and dof the degrees of freedom in the fit. The resulting confidence interval, k=1, is used as the type A (u_A) contribution to the standard uncertainty. Note that $u_{cal}(V_{Xe})$ is evaluated for every value of V_{Xe} and the output is given as a table. The range, and step size, for this evaluation is set in the input of the ROOT script and the output should be used to retrieve the final uncertainty for every value of V_{Xe}. In practice the distribution is smooth and a coarse step size can be used.

The type B contributions constitutes of uncertainties in the xenon gas purity, the loop volume, temperature and pressure.

The purity in the bottle with stable xenon is better than 99.998 %, the uncertainty due to this is at least ten times smaller than the largest contribution to the total uncertainty and can thus be neglected.

The standard uncertainty (k=1) in the injection volume $u(V_{INJ})$ is 5.8 %, calculated from a maximal uncertainty of ±10 % which is given from the manufacturer of the loop. Note that for a calibrated loop, used in some laboratory system, the standard uncertainty is 1.2 %.

The standard uncertainty (k=1) in temperature, $u(T_{INJ})$, is 0.19 % for all SAUNA systems, calculated from a maximal uncertainty of ± 1 °C at a constant temperature of 298 K.

The uncertainty in the pressure needs to be handled as an absolute value since the uncertainty is related to the maximum range of the pressure gauge, which makes it complicated to use a relative uncertainty. The uncertainty in the pressure gauge has two components; a maximum deviation, $u(P_1)$, which includes linearity and hysteresis, this is usually 0.5 % of the full range of the pressure gauge for a SAUNA II/III and lab system. The second part is the long-term stability, $u(P_2)$, which is given as the maximal change per year since the calibration of the pressure gauge. This is normally 0.2 % of the full range of the gauge, per year, for a SAUNA system. If this (year) is not known a nominal value of 3 years should be used. The contribution to the standard uncertainty in the xenon volume $(u_{abs}(P_{INJ}))$ from the pressure measurement is calculated by:

$$u_{abs}(P_{INJ}) = C_{Xe}V_{INJ}\frac{1}{P_{STP}}\frac{T_{STP}}{T_{INJ}}\sqrt{u(P_1)^2 + u(P_2)^2}. \tag{5}$$

The reason for calculating the uncertainty from the pressure this way is to avoid using the absolute value for the pressure, which is not known in all points where the calibration is valid, when evaluating the result from the fit and only using the xenon volume which is the output of the fitting function.

All final contributions to the uncertainty in the calibration are calculated as relative uncertainties using:

$$u = \frac{u_{abs}}{V_{Xe}}, \tag{6}$$

where V_{Xe} is given from equation (2) above. The final resulting relative standard uncertainty from the calibration is given by:

$$u_{cal}(V_{Xe}) = \sqrt{u(P_{INJ})^2 + u(T_{INJ})^2 + u(V_{INJ})^2 + u_A{}^2}, \tag{7}$$

with u_A, described above in the beginning of this section, is recalculated as relative uncertainties using the result from the fit and $u(V_{INJ})$ and $u(T_{INJ})$ given as relative uncertainties above. Note that this uncertainty has to be evaluated for every measured area when using equation (2) to calculate the measured value of the stable xenon.

For a measurement, when the calibration function is used to calculate the stable xenon volume, the uncertainty in the measured peak area needs to be combined with the equation above in order to yield the total uncertainty in the measured xenon volume. To do this the uncertainty in the area is recalculated as an absolute uncertainty using the measured area. This results in an absolute standard uncertainty in the measured xenon volume:

$$u_{meas,abs}(V_{Xe}) = \sqrt{u_{cal,abs}(V_{Xe})^2 + u_{abs}(Area)^2 + u_{abs}(Base)^2}, \tag{8}$$

where $u_{abs}(Area)$ and $u_{abs}(Base)$ are the absolute standard uncertainties in the measured area and the baseline subtraction respectively. These uncertainties in the area are scaled to xenon volumes using the relation in equation (2), *i.e* the uncertainties are multiplied with the linear coefficient (p_1). All these contributions are already included in the resulting confidence intervals given as the output of the ROOT script, so for every measured area an associated total uncertainty, $u_{meas,abs}(V_{Xe})$, will already be available.

The calculation of the linear fit is currently implemented in the SAUNA software without taking the uncertainties into account, this routine should be updated using the findings in this report, including the uncertainty calculation. Example data and results are given in Appendix B.

3.1.3 Calibration Check and Service

The standard routine during the yearly service visits is to perform three GC calibrations and check them versus the current system calibration. It is recommended that these measurements are made with an interval of stable xenon volumes corresponding to the system's nominal xenon volume ±20 %.

When validating the system performance the criteria should be that the calibration measurements overlap the calibration confidence interval (k=2). It is accepted that one out of three measurements does not overlap the calibration data when including the expanded uncertainties (k=2). If the measurements do not comply with the calibration the system should be checked for malfunctions since normally a deviation is due to problems with the gas handling system, e.g. leaks, low pressure or flow which might change the calibration or the retention time of the peak. If no such problems can be found the GC should be fully re-calibrated using the routine described above.

If the measurements are found to be in agreement with the calibration it is recommended to include the data points in the calibration and calculate a new polynomial.

3.2 Dead Volume Factor

The dead volume factor for the system is defined as the ratio of the volume inside the detector cell (the detector cell volume) to the total detector volume (detector cell volume plus detector dead volume which includes gas lines and pressure sensors etc.).

The dead volume factor (D_F) is determined by injecting a known amount of air with a syringe into an evacuated cell through an injection port, temporarily mounted on the detector cell inlet between the valve (Sv18 or Sv19) and the gas line to the detector. The pressure difference (*i.e.* the pressure in the cell before and after injection), the detector temperature, the ambient temperature and pressure and the injected volume are used to calculate the total volume. The detector cell volume is given from the drawings of the detector cell.

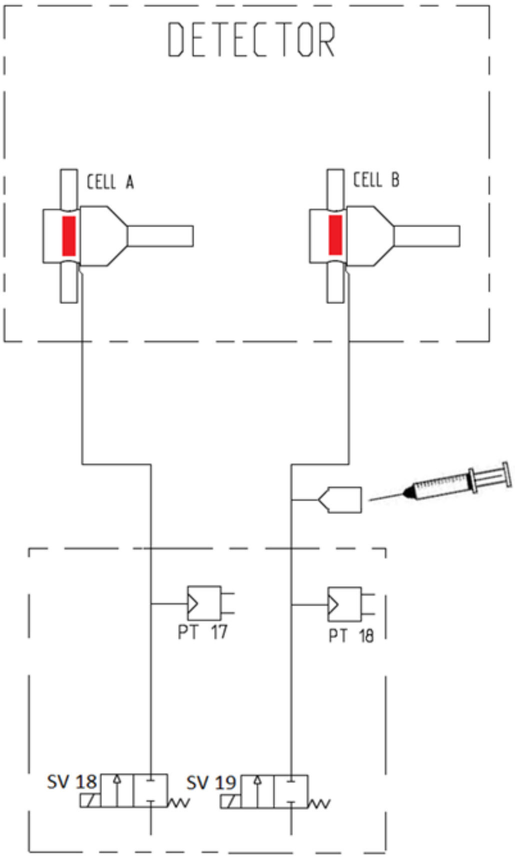


Figure 3.4. Schematic view of the two SAUNA II detectors including gas lines, valves (Sv18 and SV19) and pressure gauges (PT17 and PT18). The active part of the detectors are indicated by the red rectangle but the gas sample fills the volume all the way down to the valves. Note that the SAUNA III detectors connection do not differ from these drawings although a different notation is used. A schematic syringe is shown together with an injection port to illustrate where the injections are made when measuring the dead volume. This port is also used when injecting radio-xenon for calibration of the detector.

3.2.1 Step-by-Step Procedure

Note that if injections with different volumes or syringes are made these needs to be handled separately.

1. Install injection port on the detector gas line, if not already in place.
2. Check that the detector cell and injection port does not leak, by evacuating the cell and evaluating the trend in the detector pressure. This should not exceed 1 mbar/min. It is recommended to complete the rest of the routine in a swift manner to minimize the effect of leaks.
3. Evacuate the detector cell and record the pressure.
4. Fill a syringe, preferably a digital with high precision, with a known amount of air. Use the full volume of the syringe to get the best accuracy. Equilibrate the pressure and temperature with ambient conditions. *Log the syringe volume, room temperature and room pressure.*
5. Inject the air into the detector cell via the injection port. *Log the pressure when it stabilizes (shortly after injection)*. To prevent leakage quickly remove the syringe after injection.
6. Repeat step 4-5 until the pressure in the detector cell reaches ambient pressure. *Do not use this routine with the cell pressure above ambient pressure*.
7. Make sure that at least 5 injections have been made to reach ambient pressure, each resulting in a logged data set. If more data points are needed to get 5 sets of data and the pressure is in the cell is too high, repeat from step 3.
8. All measurement data are recorded in the excel sheet described in detail below.
9. Insert the result in the system software. (Currently the handling of uncertainties is not implemented in the system software, this should be implemented in future versions.)

The Excel sheet *Deadvol_template_v3.xlsx* is used record data from the measurements and to calculate the dead volume factor for each detector. In the excel sheet;

1. In the “Deadvol” sheet in the Excel document, select the type of detector and injection port used in the system from the drop-down lists in cell B5 and B6, the types available are shown below (row 15-18).
2. Copy the sheet “DeadVol meas copy to new sheet!” to a new sheet for each measurement of the dead volume factor made and rename it.
3. Insert the ambient conditions, temperature and pressure with uncertainties in this new sheet, these are used to calculate the standard uncertainties.
4. Insert the volume of the syringe used. Note that if different syringes or volumes are used this is handled as a new measurement series.
5. Insert the pressure measurements in column H (“P, Meas”).

6. The resulting dead volume factor will be calculated automatically and are reported in Cells B19 and B20. If the injection port is left on the detector the value in cell B19 should be used, otherwise use the value in cell B20.

3.2.2 Details of the Analysis in the Excel sheet *Deadvol_template_v3.xlsx*

The initial pressure and temperature in the detector before an injection is given by:

$$P_0 V_0 = n_0 R T_0 \,, \qquad (9)$$

where V_0 is the sum of the detector cell volume and the detector dead volume.

The initial conditions in the filled syringe, normally the syringe is in equilibrium with the ambient conditions so that P_1 equals the atmospheric pressure, is given by:

$$P_1 V_1 = n_1 R T_1 \,. \qquad (10)$$

After injection of the gas and removal of the syringe the conditions in the detector cell is given by equation (11), with n_L representing the amount of gas leaking in during the procedure and T_2 and P_2 the temperature and pressure in the cell after the injection.:

$$P_2 V_0 = (n_0 + n_1 + n_L) R T_2 = \left[\frac{P_0 V_0}{T_0} + \frac{P_1 V_1}{T_1} + n_L R\right] \times T_2 . \qquad (11)$$

Assuming the leak-in rate is negligible ($n_L = 0$) and that the temperature in the cell does not change ($T_2 = T_0$), this gives:

$$(P_2 - P_0)\frac{V_0}{T_0} = \frac{P_1 V_1}{T_1} . \qquad (12)$$

The pressure in the detector cell is measured with the same gauge, which has a smaller relative than absolute uncertainty, before and after the injection, thus it is better to use the pressure difference in the cell: $P_{CELL} = P_2 - P_0$. Inserting this in equation (12) gives:

$$V_0 = \frac{T_0}{T_1} \times \frac{P_1 V_1}{P_{CELL}} \ . \qquad (13)$$

The injection should be repeated to get good statistics and the average of the pressure differences, taken as the difference before and after one injection, used in the calculation of the pressure, $\bar{P}_{CELL}$.

If the temperature in the detector cell is not measured and can be assumed to be equal to the room (syringe) temperature, equation (13) can be simplified. Note that the uncertainty in the temperature is still taken into account according to equation (19).

The dead volume factor is defined as the fraction of the sample that is inside the actual active volume of the detector. If the injection port is left on the system after the dead volume determination it is given by:

$$D_F = \frac{V_{CELL}}{V_0} . \qquad (14)$$

If the injection port is removed D_F will instead be given by:

$$D_F = \frac{V_{CELL}}{V_0 - V_{INJ}} \quad , \qquad (15)$$

where V_{INJ} is the volume of the injection port.

3.2.2.1 Uncertainty Analysis

To get the standard uncertainty in the pressure measurement, first the estimated standard deviation is calculated and multiplied with the Student’s t-distribution factor:

$$s_{est}(P_{meas}) = Q \times \sqrt{\frac{\left(P_{CELL,i} - \bar{P}_{CELL}\right)^2}{n-1}} \,, \qquad (16)$$

where n is the number of measurements, Q the Student’s t-distribution quantile, and $\bar{P}_{CELL}$ the average of the measured difference in pressure.

The standard uncertainty of the mean is given by:

$$u_{abs}(P_{meas}) = \frac{s_{est}(P_{meas})}{\sqrt{n}}\,. \qquad (17)$$

The standard uncertainty in P_{CELL} is given from the relative uncertainty in the pressure gauge, $u(P_{rel})$ and the standard uncertainty in the measurement $u(P_{meas})$.

$$u_{abs}(P_{CELL}) = \sqrt{u(P_{meas})^2 + u(P_{rel})^2}\,. \qquad (18)$$

With the absolute standard uncertainty in V_0:

$$u_{abs}(V_0) = V_0 \times \sqrt{\left(\frac{u(P_{CELL})}{P_{CELL}}\right)^2 + \left(\frac{u(P_1)}{P_1}\right)^2 + \left(\frac{u(V_1)}{V_1}\right)^2 + \left(\frac{u(T_1)}{T_1}\right)^2 + \left(\frac{u(T_0)}{T_0}\right)^2}\,. \quad (19)$$

With the injection port left in place the absolute standard uncertainty in the dead volume factor is given by:

$$u_{abs}(D_F) = D_F \times \sqrt{\left(\frac{u(V_{CELL})}{V_{CELL}}\right)^2 + \left(\frac{u(V_0)}{V_0}\right)^2}\,. \qquad (20)$$

Note that $u(V_{CELL})$ is calculated from the tolerances from the drawings of the detector.

If the injection port is removed the absolute standard uncertainty is instead given by:

$$u_{abs}(D_F) = D_F \times \sqrt{\left(\frac{u(V_{CELL})}{V_{CELL}}\right)^2 + \frac{u(V_0)^2 + u(V_{INJ})^2}{(V_0 - V_{INJ})^2}}\,. \qquad (21)$$

3.3 Transfer Functions

The transfer functions for a SAUNA system are defined as the fraction of xenon transported from one step in the process to the next step.

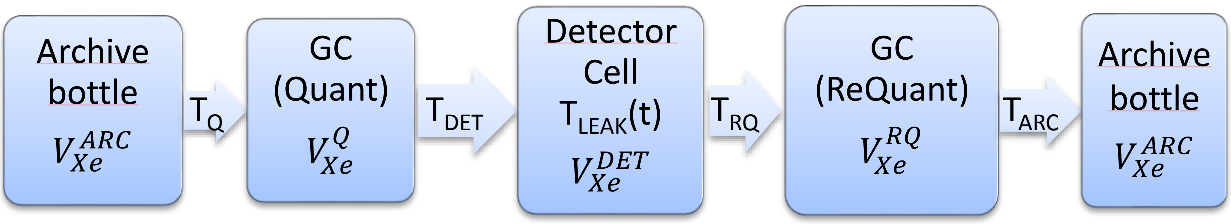


Figure 3.5. The SAUNA laboratory system process. Note that T_{LEAK} is potentially due to leaks or diffusion through the scintillating plastic cell and will be time-dependent, this will be accounted for by measuring the xenon volume when the sample is exiting the detector cell.

The transfer functions can be determined by injecting a known amount of xenon into the system and compare it to the volumes measured in the quantification and re-quantification processes. Xenon can be injected into the system at two points – injection into the archive bottle and injection directly into the detector cell. This results in that the two transfer functions T_Q and T_{RQ} can be measured directly and the two transfer functions T_{DET} and T_{ARC} will have to be determined indirectly knowing T_Q and T_{RQ}.

3.3.1 SAUNA Laboratory System

3.3.1.1 Step-by-Step Procedure

For a laboratory system which is equipped with two bottle positions and two detector cells the transfer functions will have to be measured for both bottle positions and to and from both detector cells ($T_{Q(B1,B2)}$, $T_{DET(C1,C2)}$ and $T_{RQ(C1,C2)}$). If there is a significant difference between the different combinations this will have to be taken into account when calculating the xenon volume.

Measurements shall be performed for relevant xenon volumes, which depends mainly on the detector type but should at least cover the full range of the GC calibration. If the xenon volume dependence is shown to be significant, i.e. deviation more than the expanded uncertainty (k=2), more measurements will have to be performed.

To reduce the risk of cross contamination between measurements blank measurements shall be performed between xenon measurements, these will remove any residual xenon which can be trapped in the system. By evaluating the xenon content in the blank runs an estimate of the cross contamination can be established. The time between measurements have to be long enough to assure that the GC and ATU trap has cooled down before the next cycle, normally this should be handled by the system software.

To perform the transfer function measurements injection ports will have to be installed on both cells and preferably on the archive bottle(s) so that the archive injection can be done without removing the bottle.

Determination of T_Q

T_Q is the transfer function for transport from the archive bottle to the GC and has to be determined for both bottle positions and for relevant xenon volumes.

1. If the system has not been used for a while, or if a xenon measurement without a following blank was last performed on the system: perform a blank measurement by starting a Bottle-to-Cell (BtC) operation, without re-quantification, to drain and 1 min measurement length. This is preferably done with an empty bottle with an injection port that can be used for the injection or as a blank.

2. Verify that the GC and ATU temperatures are below 30 °C

3. Inject the desired xenon volume into the archive bottle, using a syringe, in position 1 via an injection port and fill with carrier gas.

4. Start a BtC operation for bottle position 1 to cell 1 (B1tC1) without re-quantification to drain and 1 min measurement length.

5. Schedule a new blank B1tC1 operation after the first one, to clean the system. Schedule enough time between the measurements to assure that the GC and ATU has time to cool down.

   Measurement cycle: B1 -> C1: Blank -> C2 or drain.

6. Repeat step 2 - 5 for Bottle position 2. The injection can be performed at the same time as injection for Bottle 1 and measurements scheduled to be performed after measurements for B1.

7. Export the results from the GC measurements, fill in the Excel sheet template, *Transfer_template.xlsx* to calculate T_Q.

Determination of T_{RQ}

T_{RQ} is the transfer function for transport from the detector cell to the GC and has to be determined for both detector cells and for relevant xenon volumes.

1. Check that the detector cell has been purged or purge by performing a blank run if necessary.
2. Inject the wanted xenon volume into the detector cell 1 via the injection port (observe the pressure increase in the cell). Inject carrier gas (via syringe or fill up using the system) into the cell until "normal" operational pressure is achieved, usually about 0.8 bar.
3. Start a Cell-to-Bottle (CtB) transfer from cell 1, let the sample go to drain (C1 -> Drain). This needs to be implemented in the operational software, for now this is made manually using the PLC control software.
4. Schedule a blank transfer from cell 1 to drain (C1 -> Drain). This needs to be implemented in the operational software, for now this is made manually using the PLC control software.
5. Repeat step 1-4 for cell 2. The injection can be performed at the same time as for the other detector but need to be scheduled to be performed after the transfer from cell 1 has been executed.
6. Export results from the GC measurements, fill in the Excel sheet template to calculate T_{RQ}.

Determination of T_{DET} and T_{ARC}

The transfer functions T_{DET} and T_{ARC} cannot be determined directly and can therefore only be determined when T_Q and T_{RQ} are known.

1. Make sure that the system has been purged with a blank measurement or perform a blank measurement to purge the system.
2. Connect a clean and evacuated archive bottle to the system.
3. Inject the wanted xenon volume into the archive bottle and fill with carrier gas.
4. Add a measurement cycle that in the case for Bottle 1 is: B1tC1, blank Drain -> C2, C1tB1, C2 -> drain. In the future this should be implemented in the GUI.

This process can be repeated n times without new injections to study continuous losses and to determine T_{ARC}. The continuous loss can be used to check $T_Q \times T_{DET} \times T_{RQ} \times T_{ARC}$ by comparing $V_Q(1)$ and $V_Q(n)$.

$$n\left(T_Q \times T_{DET} \times T_{RQ} \times T_{ARQ}\right) = \frac{V_Q(n)}{V_Q}. \qquad (22)$$

Fill in the measurement data in the Excel sheet *Transfer_template.xlsx* is used to calculate T_{DET} and T_{ARC}.

The values for the transfer functions T_Q, T_{DET}, T_{RQ} and T_{ARC} should be inserted into the system software, this is made in the setup tab in the GUI, and used to calculate the amount of stable xenon in the detector. The system software needs to be updated to incorporate the full uncertainty analysis.

3.3.1.2 Details of the Analysis in *Transfer_template.xlsx*

The transfer functions, T, are defined as the amount of xenon transported from one step in the process to the next step:

$$V_{Xe}^{proc+1} = T_{proc} V_{Xe}^{proc} \qquad (23)$$

The first transfer function for the SAUNA process, T_Q, is then given by:

$$T_Q = \frac{\mathrm{V}_{Xe}^{Q}}{V_{Xe}^{ARC}} \qquad (24)$$

The injected xenon volume – into the archive bottle or the detector cell – can be written as:

$$V_{Xe}^{ARC,DET} = N\, V_{Xe}^{SYR} T_{INJ}\ , \qquad (25)$$

assuming that the total injected volume is divided into equal parts where N is the number of injections, V^{SYR} is the xenon volume in the syringe per injection and $\mathrm{T_{INJ}}$ is the transfer function for the injection into the bottle or cell.

The injected xenon volume is corrected to STP (0°C and 1 atm), changing the equation above to:

$$V_{Xe}^{ARC,DET} = N\, \frac{P_{amb}}{P_{STP}} \frac{T_{STP}}{T_{amb}} V_{Xe}^{SYR} T_{INJ}\ , \qquad (26)$$

assuming that the syringe is in equilibrium with the ambient conditions.

If xenon is injected into the detector cell, the transfer function T_{RQ} can in the same way be given as:

$$T_{RQ} = \frac{V_{Xe}^{RQ}}{V_{Xe}^{DET}}\ , \qquad (27)$$

where the injected xenon volume into the cell is given in the same way as the injected volume into the archive bottle.

The transfer function T_{DET} will have to be determined by injection into the archive bottle, and passing the sample through the GC, into the detector cell and back through the GC again. If the transfer functions T_Q and T_{RQ} are known T_{DET} can then be calculated as:

$$T_{DET} = \frac{\mathrm{V}_{Xe}^{DET}}{V_{Xe}^{Q}} = \frac{\mathrm{V}_{Xe}^{RQ}}{T_{RQ} V_{Xe}^{Q}}\ . \qquad (28)$$

The transfer function T_{ARC} can be determined by looping a sample through the system, back to the archive bottle and then through the system again, giving T_{ARC} as:

$$T_{ARC} = \frac{V_{Xe}^{ARC}}{V_{Xe}^{RQ}} = \frac{\mathrm{V}_{Xe}^{Q}}{T_Q V_{Xe}^{RQ}} \qquad . \qquad (29)$$

This can be done as a continuous loop which, except than provide better statistics, also will give some information about xenon volume dependence for the transfer functions.

When looping a sample through the system the xenon volume in the archive bottle for consecutive loops are given by:

$$V(i+1)_{Xe}^{ARC} = V(i)_{Xe}^{ARC} T_Q T_{DET} T_{RQ} T_{ARC}\ . \qquad (30)$$

And in the same way for the xenon volume measured in the GC at quantification the xenon volume should then be given by:

$$V(i+1)_{Xe}^{Q} = V(i)_{Xe}^{Q} T_Q T_{DET} T_{RQ} T_{ARC}\ , \qquad (31)$$

giving for the n:th loop:

$$n\left(\mathrm{T_Q T_{DET} T_{RQ} T_{ARQ}}\right) = \frac{V(n)_{Xe}^{Q}}{V_{Xe}^{Q}}\ . \qquad (32)$$

Uncertainty Analysis

The relative standard uncertainty of T_Q is given by:

$$u(T_Q) = \sqrt{u(V_Q)^2 + u(V_{ARC})^2}\,, \tag{33}$$

where the uncertainty $u(V_Q)$ is given by the GC-calibration, see above, and the uncertainty of the xenon volume injected into the archive bottle is given by:

$$u(V_{ARC}) = \sqrt{u(V_{SYR})^2 + u(T_{INJ})^2 + u(P_{amb})^2 + u(T_{amb})^2}\,, \tag{34}$$

giving:

$$u(T_Q) = \sqrt{u(V_Q)^2 + u(V_{SYR})^2 + u(T_{INJ})^2 + u(P_{amb})^2 + u(T_{amb})^2}\,. \tag{35}$$

The standard uncertainty for T_{RQ} is given in the same way, where the injected volume into the detector cell is given by the same function as the volume injected into the archive bottle:

$$u(T_{RQ}) = \sqrt{u(V_{RQ})^2 + u(V_{DET})^2} =$$
$$\sqrt{u(V_{RQ})^2 + u(V_{SYR})^2 + u(P_{amb})^2 + u(T_{amb})^2 + u(T_{INJ})^2}\,. \tag{36}$$

The standard uncertainty for T_{DET} is then given by:

$$u(T_{DET}) = \sqrt{u(V_{RQ})^2 + u(T_{RQ})^2 + u(V_Q)^2} =$$
$$\sqrt{2u(V_{RQ})^2 + u(V_Q)^2 + u(V_{SYR})^2 + u(P_{amb})^2 + u(T_{amb})^2 + u(T_{INJ})^2}\,. \tag{37}$$

For T_{ARC} the standard uncertainty is given by:

$$u(T_{ARC}) = \sqrt{u(V_Q)^2 + u(T_Q)^2 + u(V_{RQ})^2} =$$
$$\sqrt{2u(V_Q)^2 + u(V_{RQ})^2 + u(V_{SYR})^2 + u(P_{amb})^2 + u(T_{amb})^2 + u(T_0)^2}\,. \tag{38}$$

3.3.2 SAUNA IMS Type System

3.3.2.1 SAUNA IMS Type System with Re-Quantification Option

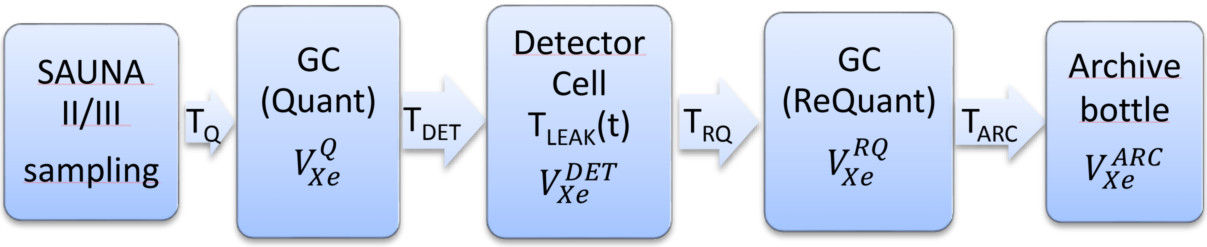


Figure 3.6. The SAUNA IMS system process for systems with re-quantification. Note that T_{LEAK} is potentially due to leaks or diffusion through the scintillating plastic cell, this will be accounted for by measuring the xenon volume when the sample is exiting the detector cell.

For an IMS type system (SAUNA-II/III) with Re-Quantification option the process is quite similar to the one for a laboratory system described above. A difference is that T_Q is the sample yield given by the sampled air volume V_{Sample} in m^3 and V_Q in ml:

$$T_Q = \frac{\mathrm{V}_{Xe}^{Q}}{0.087 \times V_{Sample}} \qquad (39)$$

T_{ARC} can be measured by processing archive bottles from the system in a lab with known transfer efficiencies. Injections of stable xenon into the detector cells and starting the ReQuant operation give T_{RQ}, as in the case of a laboratory system. When T_{ARC} and T_{RQ} are measured T_{DET} can be calculated using data from a number of normal runs.

There is less support in the control software of an IMS type SAUNA for processing samples injected into a cell and transferred to the GC etc. These operations has to be performed by an expert user manually controlling the PLC.

3.3.2.2 SAUNA IMS Type System without Re-Quantification Option

For a SAUNA II system without the re-quantification possibilities, a fixed value for T_{DET} of 0.99 ± 0.04 should be used. This number is based on the average from four laboratory systems, and the uncertainty has been modified by a factor of 2 to account for this not being measured on the specific system. The injection from the GC into the detector cell is identical for all these systems.

3.4 Stable Xenon Calculation

To be able to report the measured activity as activity concentration in the analysis of data from a SAUNA system the amount of sampled air, V_{AIR}, inside the detector needs to be calculated. The corresponding air volume, in m^3, of a sample is given from the amount of stable xenon in the detector and the concentration of xenon in air (87 ppb):

$$V_{AIR} = \frac{\mathrm{V}_{Xe}^{CELL}}{0.087} \qquad (40)$$

where $V_{Xe}{}^{CELL}$ is the amount of stable xenon, given in cm^3 at STP, in the detector. For a SAUNA II/III system, where the re-quantification measurement is only used as a state-of-health check, this is given by:

$$\mathrm{V}_{Xe}^{CELL} = D_F T_{DET} V_{Xe}^{Q} \qquad (41)$$

where D_F is the dead volume of the detector, T_{DET} the transfer function into the detector and $V_{XE}{}^{Q}$ the amount of xenon measured in the GC.

For a laboratory system the re-measurement of the stable xenon volume after the acquisition can also be used to determine the amount of stable xenon in the detector cell. Then the average of the first measurement, given from equation (41) above, and the re-quantification is reported. The xenon volume in the detector cell from the re-quantification is calculated as:

$$\mathrm{V}_{Xe}^{CELL} = D_F \frac{V_{Xe}^{RQ}}{T_{RQ}}, \qquad (42)$$

where $V_{Xe}{}^{RQ}$ is measured in the re-quantification and T_{RQ} is the transfer function from the detector cell to the GC. The standard uncertainty in the xenon volume in the cell is given by:

$$u(\mathrm{V}_{Xe}^{CELL}) = \mathrm{V}_{Xe}^{CELL} \sqrt{\left(\frac{u(D_F)}{D_F}\right)^2 + \left(\frac{u(T_{DET,RQ})}{T_{DET,RQ}}\right)^2 + \left(\frac{u(V_{Xe}^{Q,RQ})}{V_{Xe}^{Q,RQ}}\right)^2} \qquad (43)$$

Since the uncertainties between the two measurements cannot be treated statistically, due to the systematic nature of the uncertainties, it should be estimated from the quadratic sum of the two individual measurements. This will lead to an overestimation and future studies might be performed to reduce this taken into account the correlation between the uncertainties.

$$u(\mathrm{V}_{Xe}^{CELL}) = \sqrt{\frac{u(V_{Xe}^{Cell,Q})^2 + u(V_{Xe}^{Cell,RQ})^2}{2}} \qquad (44)$$

4 References

[1] A. Ringbom and A. Mörtsell, Xeff2 User Manual, Calibration and Gain Stabilization – PM5, 2017

[2] K. Birch, Estimating Uncertainties in Testing, Measurement Good Practice Guide No.36, British Measurement and Testing Association. Teddington, Middlesex: HMSO, 2001

[3] Joint Committee for Guides in Metrology, Evaluation of measurement data - Guide to the expression of uncertainty in measurement, JCGM 100:2008, 2008

[4] A. Axelsson, L. Mårtensson, A. Mörtsell and A. Ringbom, Improvement of the SAUNA Noble Gas System Calibration Procedures, FOI-R - - 3451 - - SE, 2012

Appendix A. Background Information on Detector Calibration

A.1 Detector Background Measurement

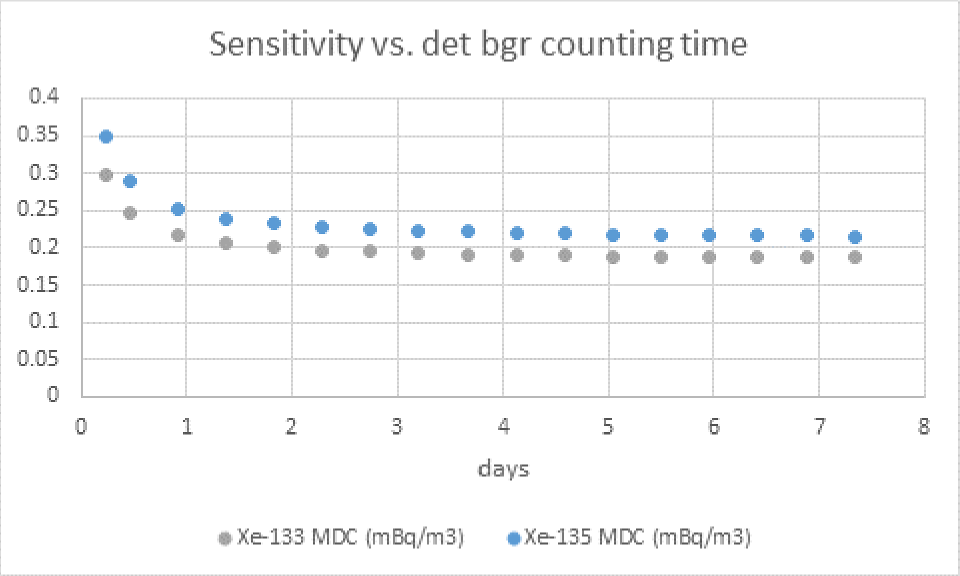


Figure A.1 Sensitivity versus the length of the detector background measurement (11 hour sample measurements are assumed to follow)

Three to four days effective measurement time seems to be a reasonable length of the detector background measurement. Three days effective time, given two 11 hour measurements per 24 hours results in 3.3 days real time. It is recommended that detector measurements are performed during four real time days to give some margin.

A.2 Cross-Talk Between Metastable Isotope ROIs

ROIs for ^{131m}Xe and ^{133m}Xe set according to this prescription can be expected to lead to an interference of ^{131m}Xe into the ^{133m}Xe analysis at a level of about 1 %. At this level of interference, a concentration of 10 mBq/m3 ^{131m}Xe can be expected to cause an additional spurious contribution to the ^{133m}Xe analysis of about 0.1 mBq/m3, which is about the level of the MDC for ^{133m}Xe. A spurious ^{133m}Xe/^{131m}Xe ratio of 0.01 is in the domain that would be screened out as not of Treaty relevance. Consequently, suppressing this effect, which could be done by introduction of an additional interference correction factor or by further increasing the separation of the respective ROI limits, has not been regarded as a priority and is not incorporated in the current implementation of the NCC.

A.3 Some Comments Regarding Spectral Interference Terms that are not Accounted for in the Standard Procedure

The current analysis algorithms for beta-gamma data do not correct the calculation of ^{133}Xe activity for interference from the presence of ^{131m}Xe and ^{133m}Xe, nor the calculation of the activity of any of these isotopes for interference from ^{135}Xe. The calibration procedure described in this document produces data – measurements B, E (optional) and F (optional) – that would allow the determination of spectral interference factors for such corrections, if and when implemented in the standard analysis, or perhaps for *ad hoc* use when warranted by exceptional circumstances.

A previous study, see reference [4], extracted an example of such factors from the equivalent of measurements B, E and F in the current procedure. The method used is described in the study and the description will not be repeated here since it is not suggested to be part of the standard calibration procedure and is not supported by the software developed for it. However, some general comments based on the numbers extracted in the earlier study may be useful for perspective.

Spectral interference from ^{135}Xe

The interference of ^{135}Xe in ROI 6 (^{133m}Xe X-rays) is of the order of 1 %. That is, there will be an extra number of counts in ROI 6 equal to about 1 % of the net counts in ROI 2 (250 keV photons from ^{135}Xe). For ROI 5 (^{131m}Xe X-rays) the interference is an order of magnitude lower. For the entire ROI 4 (all Xe X-rays) it is a few times higher (2.5 % in the example). The ^{135}Xe interference in ROI 3 (81 keV photons from ^{133}Xe) is about 0.5 %. In principle, this carries over into an additional ^{135}Xe interference in the X-ray ROIs (4-10), since the net counts analysis method subtracts an interference term based on net counts in ROI 3 from the gross counts in ROIs 4-10. However, this effect is small (amounting in the most severe case to an approximate doubling of the 0.1 % ^{135}Xe interference in ROI 5) and does not change the order of magnitude reasoning applied here. For the Rn-related interference, however, the “in-direct” interference via ROI 3 is significant (see below).

By comparison, the direct radon-daughter interference terms (the contribution to Xe ROIs as a percentage of net counts in the Rn ROI 1) are generally considerably larger: 30-40 % for ROIs 2 and 3, 5 % for ROI 4 and 0.5 % for ROI 5 (^{131m}Xe). For ROI 6 (^{133m}Xe), the direct Rn interference is similar to the direct ^{135}Xe interference. Considering also the “in-direct” Rn interference in the Xe X-ray ROIs (the interference via ROI 3), the magnitude of the Rn interference in ROIs 5 and 6 becomes more than a magnitude larger than the ^{135}Xe interference.

Another way to evaluate potential interference in ^{133}Xe determination from the presence of ^{135}Xe is to consider the impact of an error in the ^{135}Xe/^{133}Xe ratio, which can be used to time a source event. In estimating the time of a source event from one measurement, a 20 % error in the ^{135}Xe/^{133}Xe ratio will introduce an error in the result of about 3 hours. A 20 % error in the ratio caused by a 2 % ^{135}Xe to ^{133}Xe interference that is unaccounted for requires a “real” ^{135}Xe/^{133}Xe ratio of 10.

Spectral interference from ^{131m,133m}Xe

The interference of both ^{131m}Xe and ^{133m}Xe in ROI 3 (81 keV photons from ^{133}Xe) is of the order of 0.1 %, requiring a “real” ratio of either metastable isotope to ^{133}Xe of more than 100 to cause a 10 % error from this interference in the analysis result ratio. Such values of ^{133m}Xe/^{133}Xe could not be caused by any CTBT-relevant event. Since ^{131m}Xe is longerlived than ^{133}Xe, the ^{131m}Xe/^{133}Xe ratio may theoretically become very high for “old” xenon. However, in practice measuring a ratio of 100 or more would require a very high concentration of ^{131m}Xe for the ^{133}Xe component to be visible. After the Fukushima event in 2011, ^{131m}Xe/^{133}Xe ratios observed in the northern hemisphere reached about 10 before the ^{133}Xe component had decayed to the detection limit.

Appendix B. Test Results

B.1 Gas Process Calibration

B.1.1 Gas Chromatograph Calibration

Data from the SAUNA II system SEX63:

Input file:

```
# SEX63 GC Calibration data (April 2017)

# Mandatory parameters
Vinj    = 1       # Injection loop volume in ml
dVinj   = 10      # Uncertainty in loop volume. Given as maximum
                  uncertainty (specification) range (+/-) in %
dT      = 1       # Uncertainty in loop temperature. Given as maximum
                  uncertainty (specification) range (+/-) in degC.
Pmax    = 2.5     # Full range of pressure gauge in loop (bar).
dP1     = 0.5     # Uncertainty in loop pressure. Given as maximum
                  uncertainty (specification) range (+/-) in bar.
dPlong  = 0.20    # Long term uncertainty in loop pressure. Given as
                  maximum uncertainty (specification) range (+/-) in
bar/year.
Pyear   = 2       # Years since calibration of pressure gauge.
CXe     = 1       # Concentration of xenon in bottle. Not used.
dA_int  = 1.2     # Uncertainty in integration of peak. Currently used
both for calibration and
                   measurement. Full range (+/-) in %.
A_norm  = 6       # Integrated area for 1 ml of xenon, used to scale
baseline subtraction
                  uncertainty. (6 for SEX63 3600 for SEL63)

# Optional parameters
XeVmax  = 2       # Maximum xenon volume for data result. Default =
calculated from data
Npoints = 800     # No of data points out. Default = 800
Tloop   = 295.15  # Temperature in loop, constant for uncertainty
analysis (K). Default = 295.15
Tstp    = 273.15  # Temperature @ STP. Default = 273.15
Pstp    = 1.013   # Pressure @STP 1 atm. Valid if pressures given in
bar. Default = 1.013
k1      = 1       # k range 1 used for expanded uncertainty in report.
Default=1
k2      = 1       # range 2. Default=1


# Measured calibration data (Area, Pressure, Temperature)
#  A          P          T
8.554       1.7         19.3
4.581       0.976       20.5
6.707       1.35        19.7
0.859       0.19        19.5
3.757       0.766       19.1
6.398       1.308       21.7
6.466       1.317       19.9
6.414       1.345       19.6
6.497       1.314       20.6
6.517       1.31        20
6.541       1.326       19.8
6.745       1.353       22.9
6.8350      1.38        23.1
6.8790      1.384       22.7
```

Name	From certificate	Full range	Standard uncer. absolute	Standard uncer. relative (%)
Injection loop volume	10%	1.0	0.023 cm^3	5.8%
Temperature loop	1 °C	N/A	0.58 ℃	0.19%
Pressure	0.5% of full range	2.5 bar	0.0072 bar	N/A- see text
Pressure long-term	0.2% of full range per year	2.5 bar	0.0058 bar	N/A- see text
Area integration	1.2% from data	N/A	N/A	0.58%
Area baseline subtraction	Estimated from data	N/A	0.0096 a.u.	N/A

Raw data from GC calibrations performed on the system in the period 2011-2016, note that the injected volume is from the system software. These are re-calculated when using the ROOT script.

Area	Loop Pressure (bar)	Loop temperature (°C)
8.554	1.7	19.3
4.581	0.976	20.5
6.707	1.35	19.7
0.859	0.19	19.5
3.757	0.766	19.1
6.398	1.308	21.7
6.466	1.317	19.9
6.414	1.345	19.6
6.497	1.314	20.6
6.517	1.31	20
6.541	1.326	19.8
6.745	1.353	22.9
6.8350	1.38	23.1
6.8790	1.384	22.7

Output from ROOT script, only first lines:

```
p0          error_p0    p1          error_p1    Chisquare
1.88E-02    0.00237336  0.183609    5.65E-04    86.4263     0
Area[a.u.]  V(Xe)[sccm] sUnc0[a.u.] Umeas,abs(VXe)[sscm]    conf_neg[sscm]
            conf_pos[sscm]
0.0134878   0.0212835   0.00659122  0.0109256   0.010358    0.0322091
0.0269757   0.02376     0.00657487  0.0109328   0.0128272   0.0346928
0.0404635   0.0262365   0.00655854  0.01094     0.0152946   0.0371784
0.0539513   0.028713    0.00654224  0.01095     0.01776     0.0396659
0.0674392   0.0311895   0.00652596  0.01097     0.0202236   0.0421554
0.080927    0.033666    0.00650972  0.01098     0.0226852   0.0446468
0.0944148   0.0361425   0.0064935   0.01100     0.0251448   0.0471401
0.107903    0.038619    0.00647731  0.01102     0.0276026   0.0496353
```

The result from the fit is shown in the figures below.

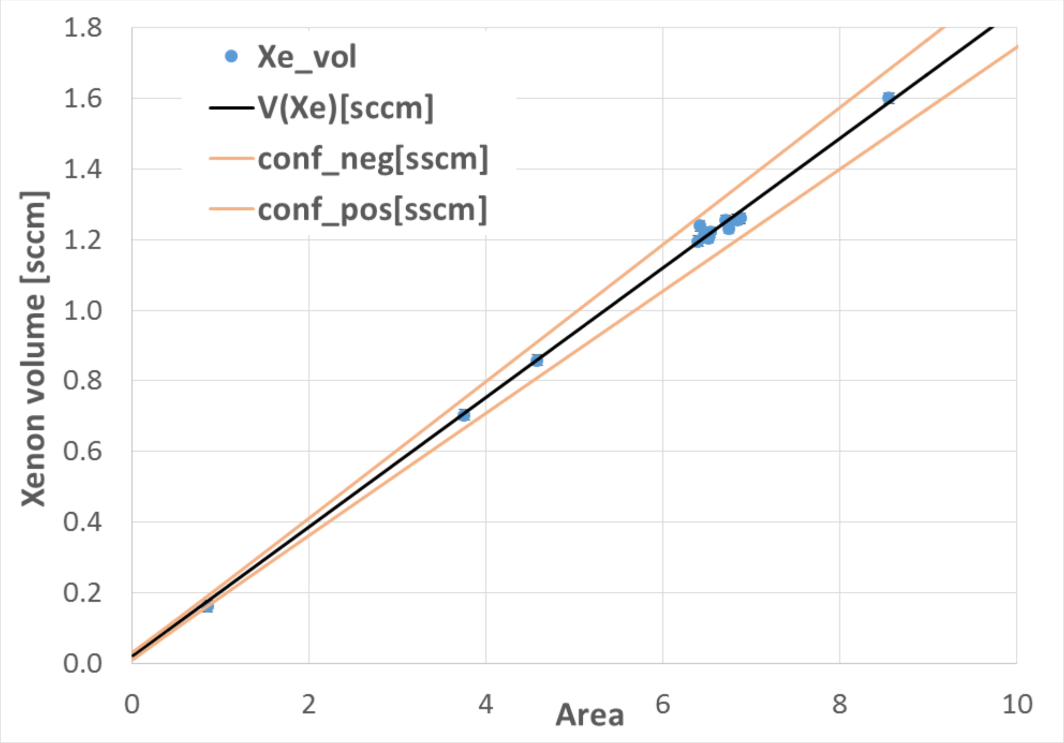


Figure B.1. Calibration data (blue dots) shown together with the resulting linear fit (black) and the upper and lower limits for the expanded uncertainty (brown) at k=1.

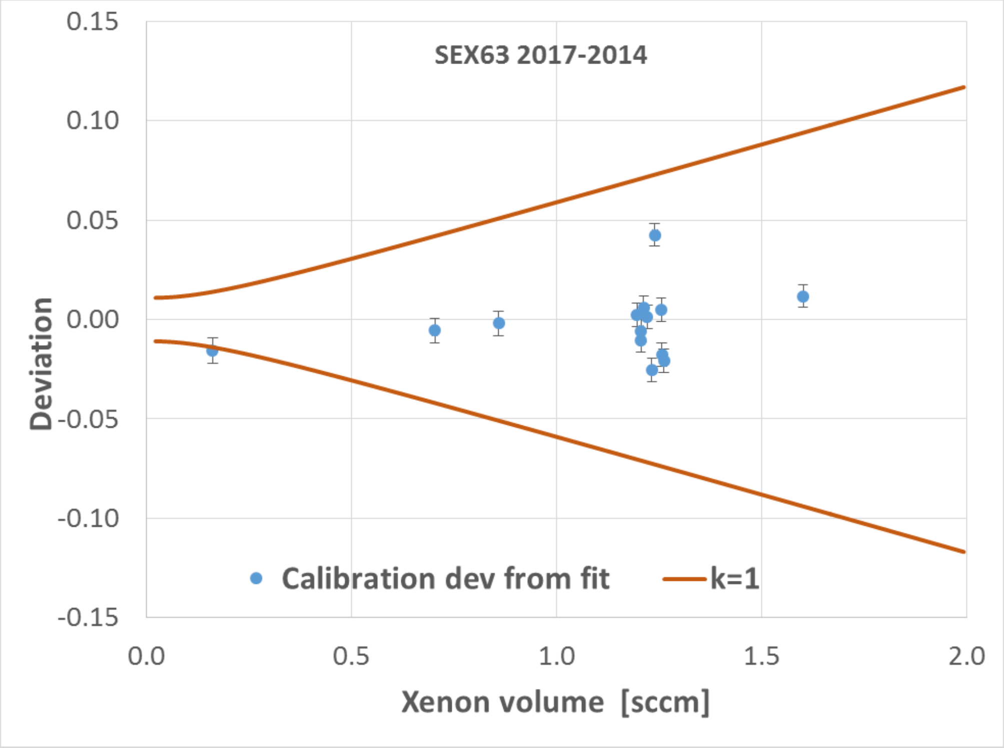


Figure B.2. Residuals from the calibration data and the linear fit (blue dots) shown together with the resulting uncertainties from the fit at k=1 (brown). The error bars represents the standard uncertainties at k=1.

B.1.2 Dead Volume

In the normal procedure at FOI 0.5 ml of air is injected repeatedly using a digital 500 µl syringe (Hamilton), which gives the best accuracy with the current syringes in use, and the pressure is logged.

Name	From Certificate or Tolerance	Full range	Standard uncer. absolute
Pmeas	Given from measurements		
Prel	0.05% of full range	2.5 bar	0.0007
P1	0.005 bar	N/A	0.0025
V1	0.01 ml	0.5 ml	0.0058
T0	1 °C	N/A	0.5
T1	1 °C	N/A	0.5
VCELL, SAUNA II type	0.25 ml	6.24 ml	0.14
VCELL, SAUNA III type	0.25 ml	17.18 ml	0.15
VINJ, old type	Est. from drawings	0.119 ml	0.0134
VINJ new type	Est. from drawings	0.085 ml	0.0053

Example data given from the SEL63 laboratory system equipped with a SAUNA II type detector. Measurement series injecting with a digital 500 µl syringe gives the following results:

Injected vol (ml)	P measured (bar)	P_{CELL} (bar)	Mean P_{CELL} (bar)	Est standard deviation	Standard uncertainty in mean
0.0	0.242				
0.5	0.317	0.0750	0.0740	0.0008	0.00049
1.0	0.391	0.0740			
1.5	0.464	0.0730			
2.0	0.538	0.0740			

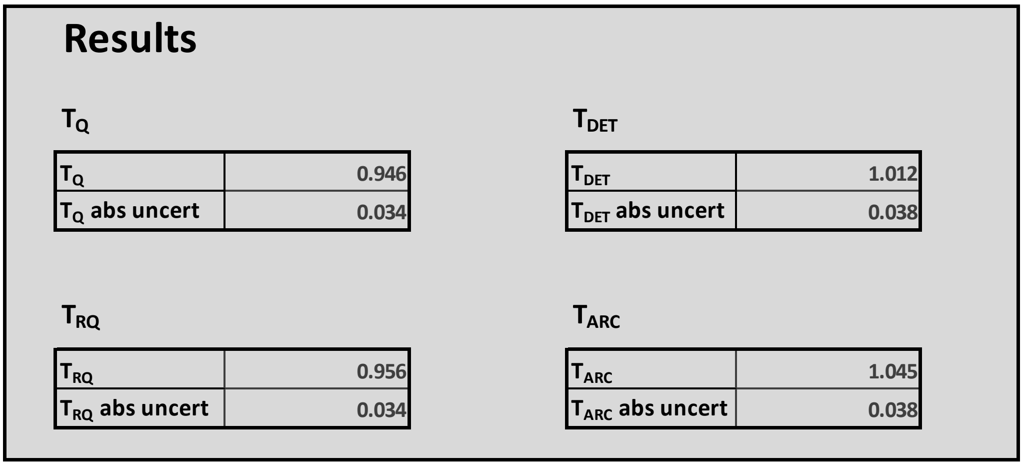
The result is V_0=6.845 with u(V_0)=0.115. An injection port with the new type of septum, with lower volume, gives that the dead volume factor is:

	Result	Standard uncertainty	Relative standard uncertainty
D_F, with injection port left on system	0.911	0.0259	2.8%
D_F, injection port removed	0.923	0.0263	2.9%

So with the injection ports left on the system the dead volume factor for this detector is D_F=0.91 ± 0.03, with a coverage factor k=2 which provides a confidence level of approximately 95%.

B.1.3 Transfer Functions

Example data and results from the laboratory system SEL63.



Results

T_Q

T_Q	0.946
T_Q abs uncert	0.034

T_{DET}

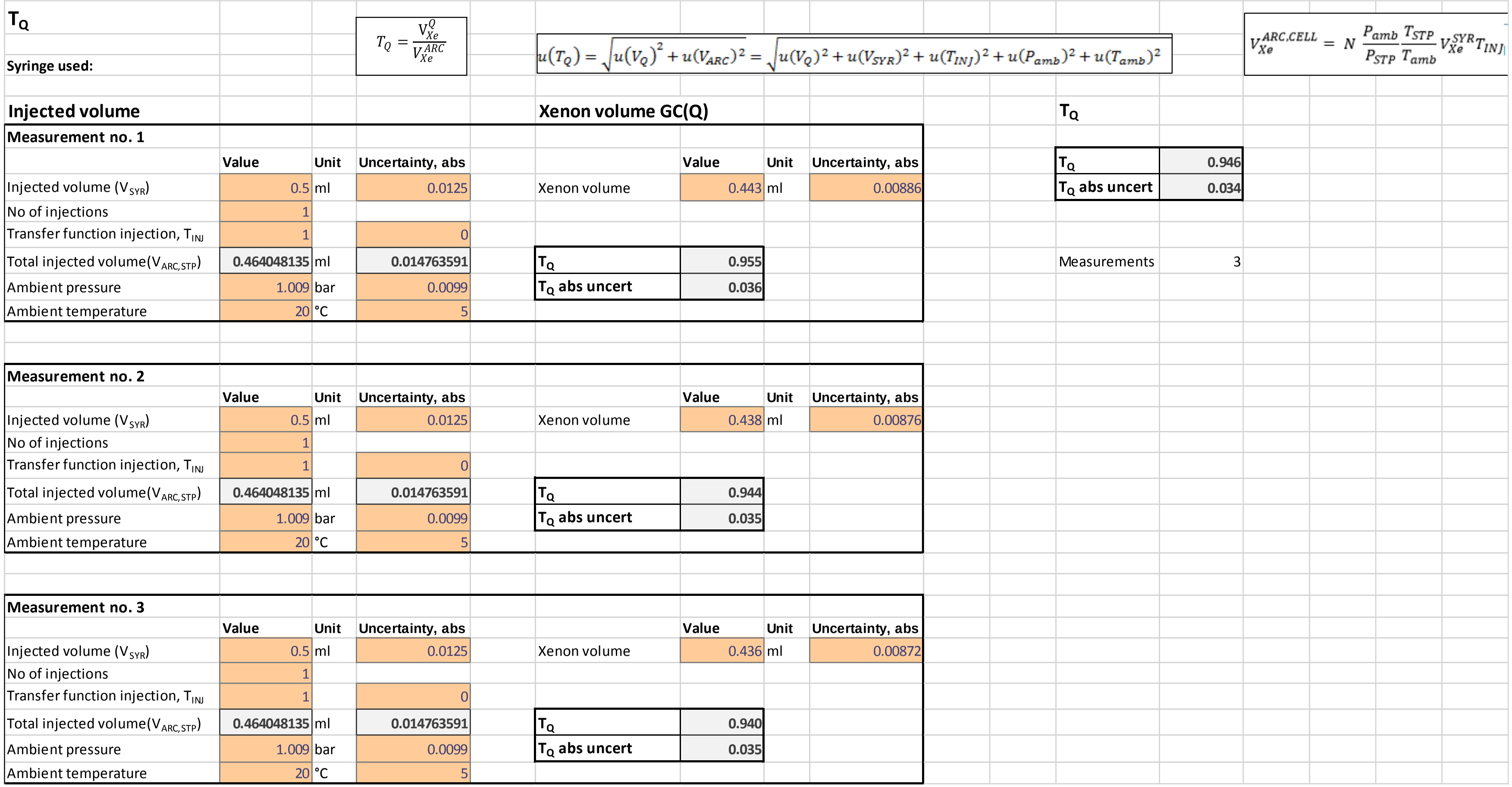
T_{DET}	1.012
T_{DET} abs uncert	0.038

T_{RQ}

T_{RQ}	0.956
T_{RQ} abs uncert	0.034

T_{ARC}

T_{ARC}	1.045
T_{ARC} abs uncert	0.038



T_Q

Syringe used:

$$T_Q = \frac{V_{Xe}^{Q}}{V_{Xe}^{ARC}}$$

$$u(T_Q) = \sqrt{u(V_Q)^2 + u(V_{ARC})^2} = \sqrt{u(V_Q)^2 + u(V_{SYR})^2 + u(T_{INJ})^2 + u(P_{amb})^2 + u(T_{amb})^2}$$

$$V_{Xe}^{ARC,CELL} = N \frac{P_{amb}}{P_{STP}} \frac{T_{STP}}{T_{amb}} V_{Xe}^{SYR} T_{INJ}$$

Injected volume | **Xenon volume GC(Q)**

Measurement no. 1

	Value	**Unit**	**Uncertainty, abs**		**Value**	**Unit**	**Uncertainty, abs**
Injected volume (V_{SYR})	0.5	ml	0.0125	Xenon volume	0.443	ml	0.00886
No of injections	1						
Transfer function injection, T_{INJ}	1		0				
Total injected volume($V_{ARC,STP}$)	0.464048135	ml	0.014763591	**T_Q**	0.955		
Ambient pressure	1.009	bar	0.0099	**T_Q abs uncert**	0.036		
Ambient temperature	20	°C	5				

Measurement no. 2

	Value	**Unit**	**Uncertainty, abs**		**Value**	**Unit**	**Uncertainty, abs**
Injected volume (V_{SYR})	0.5	ml	0.0125	Xenon volume	0.438	ml	0.00876
No of injections	1						
Transfer function injection, T_{INJ}	1		0				
Total injected volume($V_{ARC,STP}$)	0.464048135	ml	0.014763591	**T_Q**	0.944		
Ambient pressure	1.009	bar	0.0099	**T_Q abs uncert**	0.035		
Ambient temperature	20	°C	5				

Measurement no. 3

	Value	**Unit**	**Uncertainty, abs**		**Value**	**Unit**	**Uncertainty, abs**
Injected volume (V_{SYR})	0.5	ml	0.0125	Xenon volume	0.436	ml	0.00872
No of injections	1						
Transfer function injection, T_{INJ}	1		0				
Total injected volume($V_{ARC,STP}$)	0.464048135	ml	0.014763591	**T_Q**	0.940		
Ambient pressure	1.009	bar	0.0099	**T_Q abs uncert**	0.035		
Ambient temperature	20	°C	5				

T_Q

T_Q	0.946
T_Q abs uncert	0.034

Measurements	3

T_{RQ}

Syringe used:

$$T_{RQ} = \frac{V_{Xe}^{RQ}}{V_{Xe}^{DET}}$$

$$u(T_{RQ}) = \sqrt{u(V_{RQ})^2 + u(V_{DET})^2} = \sqrt{u(V_{RQ})^2 + u(V_{SYR})^2 + u(P_{amb})^2 + u(T_{amb})^2 + u(T_{INJ})^2}$$

$$V_{Xe}^{ARC,CELL} = N \frac{P_{amb}}{P_{STP}} \frac{T_{STP}}{T_{amb}} V_{Xe}^{SYR} T_{INJ}$$

$$u(V_{ARC}) = \sqrt{u(V_{SYR})^2 + u(T_{INJ})^2 + u(P_{amb})^2 + u(T_{amb})^2}$$

Injected volume to cell | **Xenon volume GC(RQ)**

Measurement no. 1

	Value	**Unit**	**Uncertainty**		**Value**	**Unit**	**Uncertainty**
Injected volume (V_{SYR})	0.5	ml	0.0125	Xenon volume	0.44	ml	0.0088
No of injections	1						
Transfer function injection, T_{INJ}	1		0				
Total injected volume($V_{DET,STP}$)	0.464048	ml	0.014777839	**T_{RQ}**	0.948		
Ambient pressure	1.009	bar	0.01	**T_{RQ} abs uncert**	0.036		
Ambient temperature	20	°C	5				

Measurement no. 2

	Value	**Unit**	**Uncertainty**		**Value**	**Unit**	**Uncertainty**
Injected volume (V_{SYR})	0.5	ml	0.0125	Xenon volume	0.447	ml	0.00894
No of injections	1						
Transfer function injection, T_{INJ}	1		0				
Total injected volume($V_{DET,STP}$)	0.464048	ml	0.014777839	**T_{RQ}**	0.963		
Ambient pressure	1.009	bar	0.01	**T_{RQ} abs uncert**	0.036		
Ambient temperature	20	°C	5				

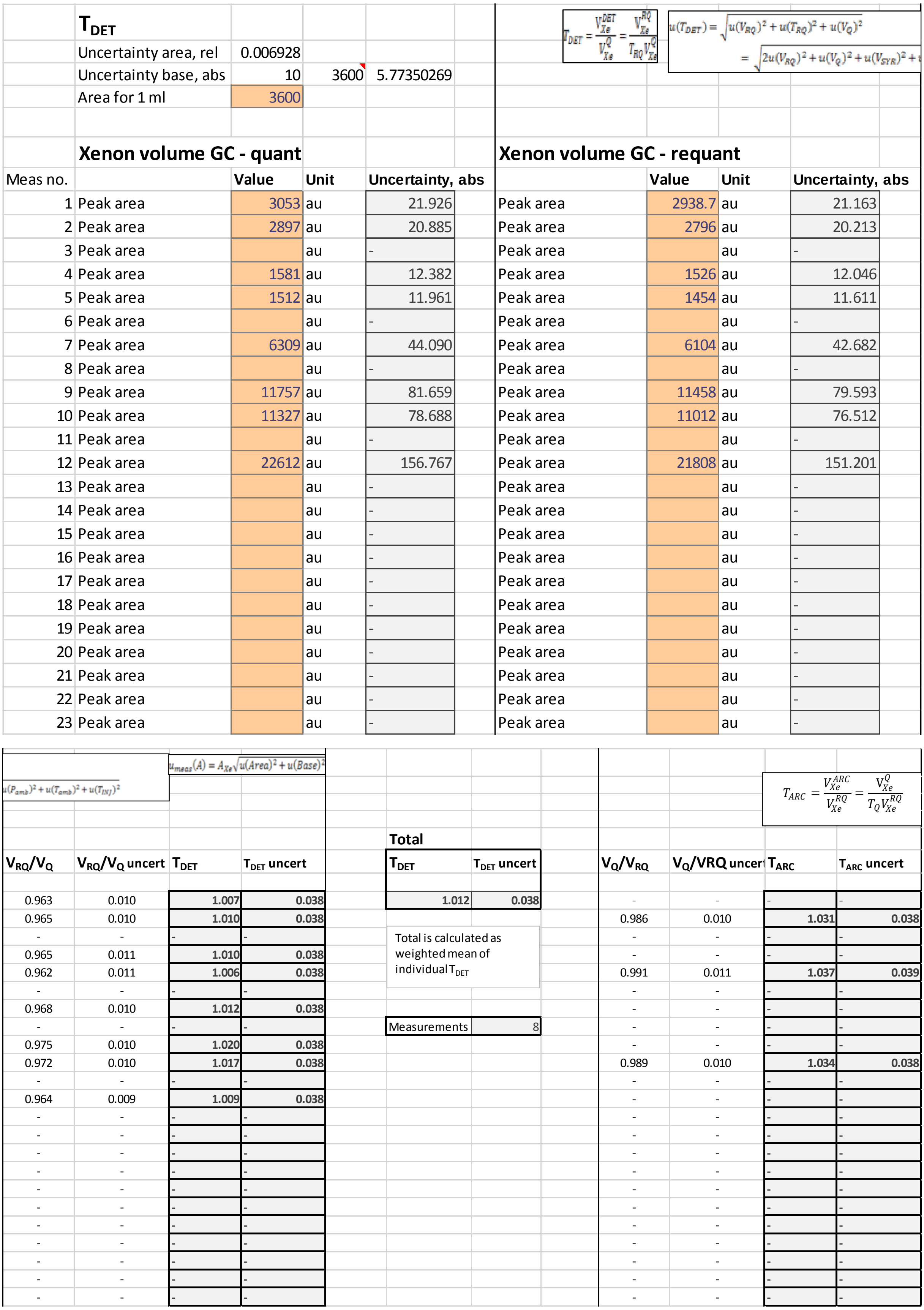
Measurement no. 3

	Value	**Unit**	**Uncertainty**		**Value**	**Unit**	**Uncertainty**
Injected volume (V_{SYR})	0.5	ml		Xenon volume		ml	
No of injections	1						
Transfer function injection, T_{INJ}	1		0				
Total injected volume($V_{DET,STP}$)	0.464048	ml	0	**T_{RQ}**	-		
Ambient pressure	1.009	bar		**T_{RQ} abs uncert**	-		
Ambient temperature	20	°C					

T_{RQ}

T_{RQ}	0.956
T_{RQ} abs uncert	0.034

Measurements	2



T

T_{DET}

Uncertainty area, rel	0.006928		
Uncertainty base, abs	10	3600	5.77350269
Area for 1 ml	3600		

$$T_{DET} = \frac{V_{Xe}^{DET}}{V_{Xe}^{Q}} = \frac{V_{Xe}^{RQ}}{T_{RQ}V_{Xe}^{Q}}$$

$$u(T_{DET}) = \sqrt{u(V_{RQ})^2 + u(T_{RQ})^2 + u(V_Q)^2}$$
$$= \sqrt{2u(V_{RQ})^2 + u(V_Q)^2 + u(V_{SYR})^2 +}$$

Xenon volume GC - quant

Xenon volume GC - requant

Meas no.		Value	Unit	Uncertainty, abs		Value	Unit	Uncertainty, abs
1	Peak area	3053	au	21.926	Peak area	2938.7	au	21.163
2	Peak area	2897	au	20.885	Peak area	2796	au	20.213
3	Peak area		au	-	Peak area		au	-
4	Peak area	1581	au	12.382	Peak area	1526	au	12.046
5	Peak area	1512	au	11.961	Peak area	1454	au	11.611
6	Peak area		au	-	Peak area		au	-
7	Peak area	6309	au	44.090	Peak area	6104	au	42.682
8	Peak area		au	-	Peak area		au	-
9	Peak area	11757	au	81.659	Peak area	11458	au	79.593
10	Peak area	11327	au	78.688	Peak area	11012	au	76.512
11	Peak area		au	-	Peak area		au	-
12	Peak area	22612	au	156.767	Peak area	21808	au	151.201
13	Peak area		au	-	Peak area		au	-
14	Peak area		au	-	Peak area		au	-
15	Peak area		au	-	Peak area		au	-
16	Peak area		au	-	Peak area		au	-
17	Peak area		au	-	Peak area		au	-
18	Peak area		au	-	Peak area		au	-
19	Peak area		au	-	Peak area		au	-
20	Peak area		au	-	Peak area		au	-
21	Peak area		au	-	Peak area		au	-
22	Peak area		au	-	Peak area		au	-
23	Peak area		au	-	Peak area		au	-

$$u_{meas}(A) = A_{Xe}\sqrt{u(Area)^2 + u(Base)^2}$$

$$u(P_{amb})^2 + u(T_{amb})^2 + u(T_{INJ})^2$$

$$T_{ARC} = \frac{V_{Xe}^{ARC}}{V_{Xe}^{RQ}} = \frac{V_{Xe}^{Q}}{T_Q V_{Xe}^{RQ}}$$

V_{RQ}/V_Q	V_{RQ}/V_Q uncert	T_{DET}	T_{DET} uncert	V_Q/V_{RQ}	V_Q/VRQ uncert	T_{ARC}	T_{ARC} uncert
0.963	0.010	1.007	0.038	-	-	-	-
0.965	0.010	1.010	0.038	0.986	0.010	1.031	0.038
-	-	-	-	-	-	-	-
0.965	0.011	1.010	0.038	-	-	-	-
0.962	0.011	1.006	0.038	0.991	0.011	1.037	0.039
-	-	-	-	-	-	-	-
0.968	0.010	1.012	0.038	-	-	-	-
-	-	-	-	-	-	-	-
0.975	0.010	1.020	0.038	-	-	-	-
0.972	0.010	1.017	0.038	0.989	0.010	1.034	0.038
-	-	-	-	-	-	-	-
0.964	0.009	1.009	0.038	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-
-	-	-	-	-	-	-	-

Total

T_{DET}	T_{DET} uncert
1.012	0.038

Total is calculated as weighted mean of individual T_{DET}

Measurements	8

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