



## Radio-xenon spikes as part of the QA/QC programme for IMS noble gas systems

### IMS Operational Manual (CTBT/WGB/TL-11,17/18/Rev.5)

**4.3. Quality Control**  
**176.** The purpose of the *quality control programme* for radionuclide stations is to verify station performance, i.e. to ensure that data produced are of acceptable quality and that the station is working within its certified operational specifications, and to take preventive action to avoid nonconformities or initiate corrective action if non-conformities exist.

**177.** The Technical Secretariat conducts a quality control programme for samples measured in International Monitoring System stations on a periodic and ongoing basis. Samples collected during normal operations are sent from stations to certified laboratories on a periodic basis to verify system calibrations as part of this programme.

### Certification of Noble Gas Equipment (CTBT/PTS/INF.921/Rev.3)

**3.1.2. Detection Requirement for Xenon Isotopes - Essential Criterion**  
The system shall separately identify and quantify the activity of Xe-131m, Xe-133, Xe-133m and Xe-135 in a sample.

**3.1.3. Uncertainty of the Xenon Isotopes Activity Ratios - Essential Criterion**  
The statistical uncertainty (k=1) for isotopic activity ratios shall not exceed the limits listed below when two or more isotopes are present in a sample each with an activity of 10 mBq m<sup>-3</sup>.

- (i) Xe-135/ Xe-133 : 15% or less
- (ii) Xe-133m / Xe-133 : 15% or less
- (iii) Xe-131m / Xe-133 : 20% or less.

### QA/QC through radio-xenon spikes – Basic concept

- The QA/QC programme for IMS noble gas systems requires that the systems are spiked with radio-xenon sources.
- A known mixture of radio-xenon isotopes is produced and. A reference data sheet containing activities, uncertainties (and potential impurities) for all radio-xenon isotopes is provided by the spike supplier.
- The radio-xenon spike mixture is injected into the IMS noble gas system during a normal sampling cycle.
- The total xenon volume, the isotopic activities in the cell, and the isotopic radio-xenon concentration are measured by the noble gas system. The data are also analyzed by the IDC. Ratios of isotope activities are determined.
- After the station measurement the archive bottles containing the spike samples are sent to IMS radionuclide laboratories. The labs reanalyse sample activities, concentrations and Xenon volume in the archive bottle.
- Comparison of data from the supplier with station and lab results supports assessments of:
  - Cross contamination (contamination of one sample to the next through residual activity)
  - Memory effect (residual activity in the detector cell from previous measurement)
  - Quality of detector calibration, interference factors, ratios (for Xe133m and Xe135 by comparison with the spike data sheet)
  - Correct quantification of stable xenon volume
  - Possible gas leakage in the system

## Results of radionon xenon QA/QC exercise 2019

### Lab and IDC analysis results for a 4-isotopes spike

- Results are calculated and compared for isotopic concentrations and activity.
- Deviations and zeta test are used for determination of correlation

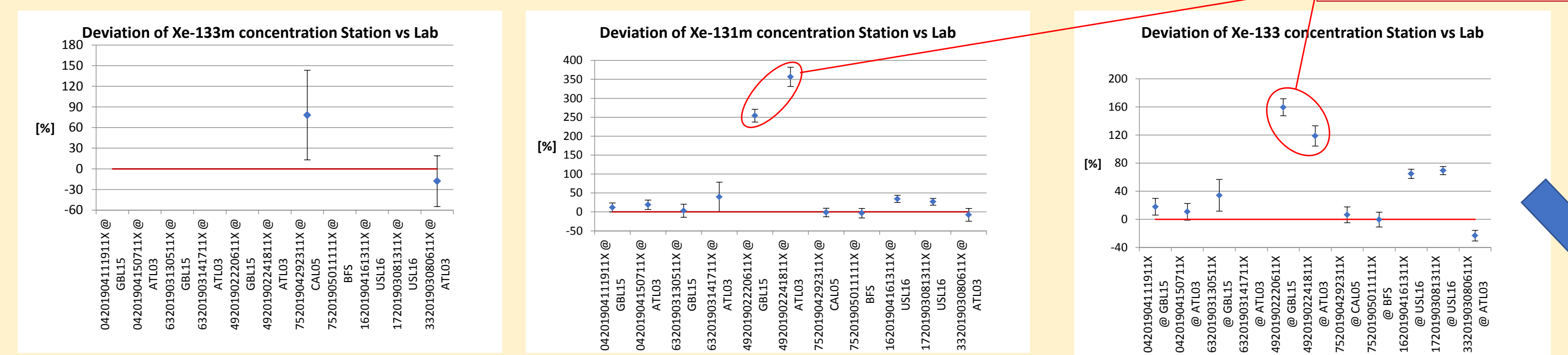
**Example: station spike re-analysis showed good correlation between station, lab, and supplier data sheet**

Spiked SAMPLE	Station (IDC analysis)			LAB			GBL15		LAB vs IDC		LAB vs IDC	
	Conc [mBq/m <sup>3</sup> ]	unc [%]	Conc [mBq/m <sup>3</sup> ]	unc [%]	Conc [mBq/m <sup>3</sup> ]	unc [%]	ξ Test	Δ [%]	ξ Test	Δ [%]	ξ Test	Δ [%]
04201904111911X	218.0	10.1	195.2	6.4	206.5	6.3	0.9	11.6	1.3	17.8		
Xe-131m												
Xe-133	2434.6	10.1	2066.5	6.3								
Xe-133m	39.1	11.3										
Xe-135	6.4	11.5										

**Example: non correlating results of the spike exercise helps to identify station issues:**

Spiked SAMPLE	Station (IDC analysis)			LAB			GBL15		LAB vs IDC		LAB vs IDC	
	Conc [mBq/m <sup>3</sup> ]	unc [%]	Conc [mBq/m <sup>3</sup> ]	unc [%]	Conc [mBq/m <sup>3</sup> ]	unc [%]	ξ Test	Δ [%]	ξ Test	Δ [%]	ξ Test	Δ [%]
49201902241811X	20.96	7.8	4.59	24.2	111.84	7.2	8.28	356.6	4.20	118.8		
Xe-131m												
Xe-133	244.65	12.5										
Xe-133m	12.86	12.4										
Xe-135												

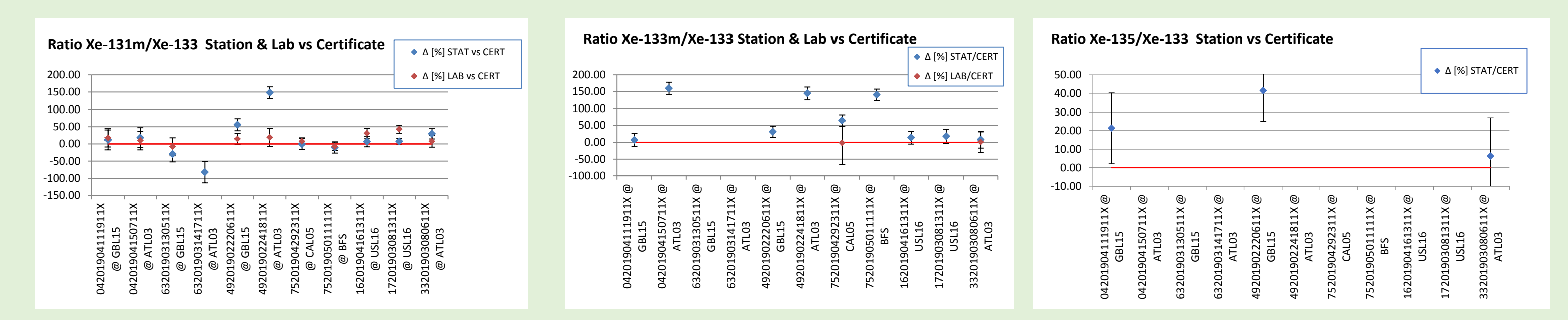
- Good correlation for concentrations for lab versus station, and for ratios between station, lab and spike data sheet
- Large deviation station vs laboratory: Gas leakage in archive system identified - Detector calibration to be checked



### Comparison of ratios between station (IDC), laboratory, and spike data sheet

- Xe-131m and Xe-133 could be measured and re-analyzed by all stations and laboratories
- Xe-133m could be reanalyzed by some laboratories
- Xe-135 was measured by few stations but due to the short half-life it could not be reanalyzed by laboratories

ISOTOPE RATIO	Supplier data sheet			IDC			LAB			Zeta Test		
	Supplier data sheet	IDC	LAB	ξ IDC vs Supp	ξ Lab vs Supp	ξ IDC vs Lab	Supplier data sheet	IDC	LAB	ξ IDC vs Supp	ξ Lab vs Supp	ξ IDC vs Lab
Xe-131m/Xe-133	0.080 (25.1%)	0.090 (14.3%)	0.094 (8.9%)	0.3	0.5	0.4	0.035 (7.8%)	0.086 (14.7%)	0.041 (25.%)	18.0	1.6	3.5
Xe-133m/Xe-133	0.015 (11.3%)	0.016 (15.1%)		0.8			0.021 (7.0%)	0.053 (17.6%)		19.7		
Xe-135/Xe-133	0.0020 (11.3%)	0.0026 (15.3%)		7								



Consistency check between data from spike data sheet, IDC analysis and Lab reanalysis supports the assessment of the system quality and provides information for the investigation of root causes

### Check of cross contamination and memory effect using spike reanalysis

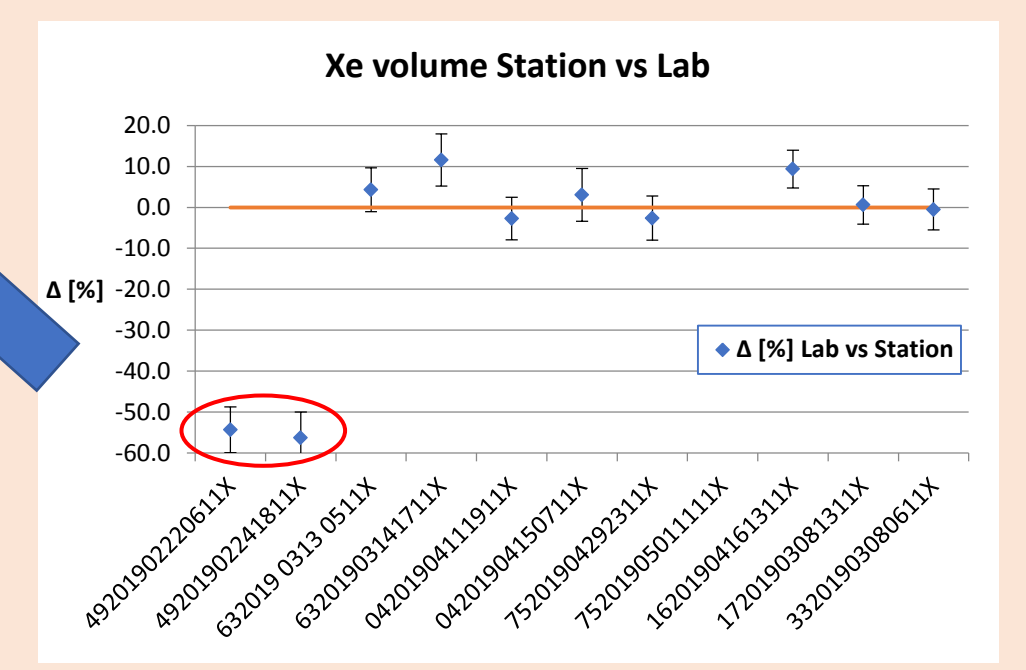
Check of IMS station certification requirement for cross contamination <1%

Noble Gas System	Detector	Sample ID	Cross contamination (requirement < 1%)	Comment
NOX49	NOX49_005	49201902220611X	0.4	ok
NOX49	NOX49_006	49201902241811X	0.3	ok
SEX63	SEX63_004	63201903130511X	na	Very poor statistics
SEX63	SEX63_003	63201903141711X	na	Very poor statistics
AUX04	AUX04_004	04201904111911X	0.1	ok
AUX04	AUX04_003	04201904150711X	0.2	ok
USX75	USX75_007	75201904292311X	0.2	ok
USX75	USX75_008	75201905011111X	0.1	ok
CAX16	CAX16_002	16201904161311X	0.3	ok
CAX17	CAX17_002	17201903081311X	0.5	ok
DEX33	DEX33_03	33201903080611X	< 0.2	ok

Check of IMS station certification requirement for memory effect < 5%

Noble Gas System	Detector	Sample ID	Memory Effect for beta/gamma systems	Comment
NOX49	NOX49_005	49201902220611X	0.12	ok
NOX49	NOX49_006	49201902241811X	0.17	ok
SEX63	SEX63_004	63201903130511X	4.05	rim coated cell
SEX63	SEX63_003	63201903141711X	na	Very poor statistics
AUX04	AUX04_004	04201904111911X	0.09	ok
AUX04	AUX04_003	04201904150711X	0.04	ok
USX75	USX75_007	75201904292311X	0.09	ok
USX75	USX75_008	75201905011111X	0.10	ok

### Stable Xenon quantification



Results/discrepancy between Lab and station Xe-volume measurements at NOX49 (in red circle) support the identification of a broken gas pipe in the archive system with the consequent accumulation of more samples. The issue was not identifiable through routine sample analysis.

### Spiking of a noble gas system

Spike vials and syringe as provided to the station operator

Radioxenon spike in air contained in 12.2ml vial

Extraction of spike from vial into syringe

Injection of a spike into the system's air inlet

Supplier's certificate for radionon xenon spike

SAUNA GUI for marking the spike sample as SPIKEPHD

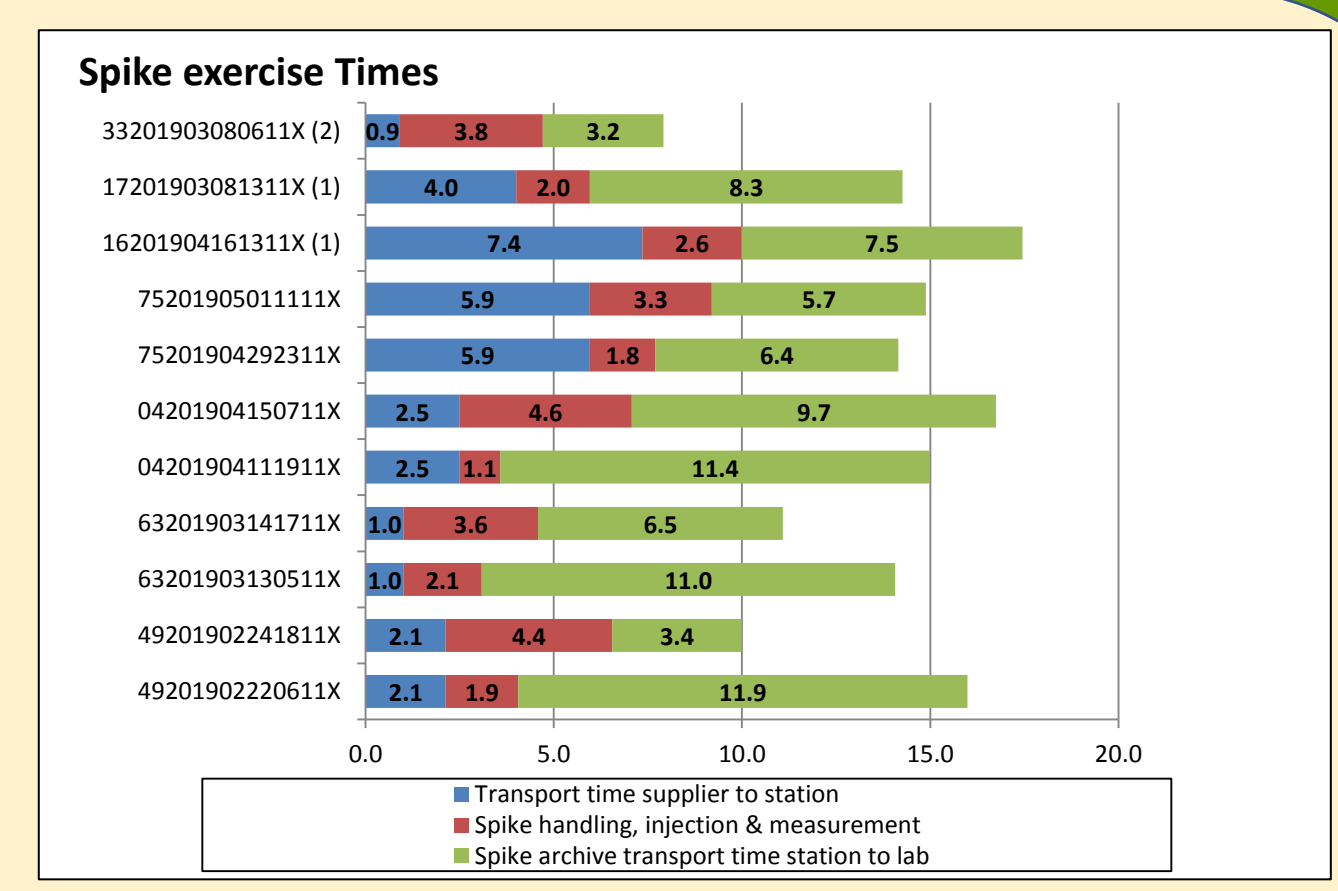
SPALAX GUI for marking the spike sample as SPIKEPHD

IDC Analysis of beta gamma coincidence spike spectrum

IDC analysis of spike by HPGe spectroscopy

### Timing challenge in the spike exercise

- Transport time from supplier to station (avg. 2.3 ± 3.2 d)
- Time for spike handling, injection & measurement (avg. 2.8 ± 1.2 d)
- Time for archive bottle dispatch from station to laboratory (avg. 7.7 ± 3.0 d)



### Procedural steps of spike exercise:

1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
IRIS Request to Station Operator to Spike	IRIS Station Operator Response Confirmation	Request to Lab for Reanalysis	Lab Response Confirmation	Spike Order	Spike Production	Spike Dispatch	Spike Receipt	PRESDN	SPIKE	SEND_SAMPLE Command	SAMPLE_SENT	LABSDN	RLR	Return of archive bottle - TECSDN
PTS	SO	PTS	LAB	PTS	ATL	ATL	SO	PTS	SO	PTS	SO	PTS	LAB	LAB

### Summary, Lessons Learned and Way forward

- Radio-xenon spikes with certified isotope activities are a very important method to perform quality check and assurance for noble gas system's performance
- Comparisons and consistency cross checks between spike certificate, station and laboratory support the assessment of system quality and can provide information for the investigation of root causes of problems
- The spike reference data sheet for all four isotope spikes incl. Xe-133m and Xe-135 enables comparison of the station vs certificate even when the laboratory reanalysis for short lived isotopes is not possible
- Organization of sample production, transport, spike exercise, transfer to lab and reanalysis is a time consuming effort and need to be more streamlined by automated processes
- More certified IMS noble gas laboratories are needed to further enhance the effectiveness of the QA/QC programme which involves up to 40 noble gas stations