Report on the system and performance audit of Sulfur Hexafluoride

Global GAW Station Cape Point (CPT) South Africa June 2016

Submitted to the World Meteorological Organization by

Haeyoung Lee, Sang-Ok Han, and Sang-Boom Ryoo

World Calibration Centre for SF_6
Environmental Meteorological Research Division
National Institute of Meteorological Sciences
Korea Meteorological Administration
Republic of Korea

WCC-SF_6 Report 2018-1

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GLOBAL ATMOSPHERE WATCH

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1. Summary and recommendations

1.1 General

The first system and performance audits for sulfur hexafluoride (SF_6) by the World Calibration Centre for SF_6 (WCC-SF_6) at Cape Point Global Atmosphere Watch station (CPT) were conducted from 7 to 10 June, 2016.

WCC-SF_6 is responsible for quality assurance measures through audits and inter-comparison experiments. Audits consist of two parts: a system audit and a performance audit. The system audit is more generally defined as a check of the overall conformity of a station with the principles of the GAW system, while the performance audit is a voluntary check for the conformity of a measurement where the audit criteria are the data quality objectives (DQOs) for the specific parameter. In the absence of formal DQOs, an audit will at least involve ensuring the traceability of measurements to the Reference Standards [1]. For SF_6, the DQO is ± 0.02 ppt, while extended compatibility is ± 0.05 ppt [2].

For this audit, WCC-SF_6 used the check list, which refers to [3] and was modified to match the SF_6 system, and the inter-comparison experiment with two different level cylinders.

This report includes the results from system and performance audits and will be distributed to the CPT station, the South Africa GAW country contact, and the WMO/GAW secretariat. The report also will be posted on the WMO GAW webpage.

1.2 System audit of the observatory

The Cape Point GAW station is well operated by the South Africa Weather Service (SAWS) with great facilities for atmospheric monitoring and research. The installation for the ambient air sampling and measurement of SF_6 is good. All systems, including SF_6, are operated with great care. The operator and staff responsible for measurement and data evaluation are very experienced.

Due to its location on the Atlantic coast and its instrumentation systems and facilities, the station is well suited for monitoring activities within the GAW network. It is a very suitable station for other monitoring programs and projects and can conduct a wide scope of activities in atmospheric research.

1.3 Performance audit of the SF_6 measurement

During the audit periods, the individual procedures from operation to data management were considered and generally followed the WMO/GAW requirement.

The gas chromatographic system was mainly suited for background N_2O concentration, not SF_6 measurement. Few analytical conditions were modified to detect both N_2O and SF_6 simultaneously and its improvements were confirmed for the audit periods.

The repeatability and drift tests were conducted as part of the audit. The instrument response showed that repeatability was between 0.14 and 0.28 ppt over 10.5 hours and drift increased during the same period.

The inter-comparison experiment with travelling standard gases was performed as part of the audit. The result was short of the extended compatibility goal as the differences were 0.1 and 0.17 ppt in high and low concentrations, respectively, between CPT and WCC.

1.4 Recommendations

- GAWSIS information about staff and the SF_6 monitoring system should be updated.
- The SF_6 measurement data should be submitted to the World Data Centre for Greenhouse Gases.
- The level of working standards should be adjusted to bracket the target concentrations and compared to laboratory standards regularly.
- Systems such as Mass Flow Controller (MFC) should be improved to assure repeatability and to at least correct the drift.

- A linearity test should be performed with at least 4 standard gases on a regular basis.
- A whole set of lab standard gases should be recalibrated or prepared since their date of production was a decade ago.

1.5 Conclusion

		Inadequate·········adequate						
Site access		□	□	□	□	□	□	■
Facilities	Laboratory and office space/equipment	□	□	□	□	□	□	■
	Air conditioning	□	□	□	□	□	□	■
	Power supply for the station	□	□	□	□	□	□	■
General Management and Operation	Organization	□	□	□	□	□	□	■
	Competence of staff	□	□	□	□	□	□	■
Air inlet system		□	□	□	□	□	□	■
Instrumentation		□	□	□	□	■	□	□
Calibration and Maintenance		□	□	□	□	□	■	□
Standards		□	□	□	□	□	■	□
Data Management	Data acquisition	□	□	□	□	□	□	■
	Data processing	□	□	□	□	□	□	■
	Data submission	■	□	□	□	□	□	□
Documentation		□	□	□	□	□	□	■

Audit completed 10 June 2016
Submitted to WMO 20 April 2018

Scientist of WCC-SF_6 Haeyoung Lee

Head of WCC-SF_6 Sang-Ok Han

Director of the division Sang-Boom Ryoo

2. Introduction

The Korea Meteorological Administration (KMA) has played a role as the World Calibration Centre for SF_6 (WCC-SF_6) since 2012. Under the MoU with the World Meteorological Organization, WCC-SF_6 started conducting the audit.

According to GAW report No. 185, "*System audit*" is more generally defined as a check of the overall conformity of a station with the principles of the GAW system, while "*Performance audit*" is a voluntary check for conformity of a measurement where the audit criteria are the DQOs for the specific parameter.

In this context, the compatibility goal, which is considered DQOs, of SF_6 is ± 0.02 ppt under the background condition and the expended level was ± 0.05 ppt in 2016. The WMO/GAW Central Calibration Laboratory (CCL, NOAA/ESRL) updated the scale NOAA-X2014 as the expanded primary standard levels reflecting non-linearity characteristics of the Electronic Capture Detector.

The Cape Point GAW station is one of the important background stations within the GAW network as it monitors southern hemisphere background concentrations. According to the record, audits were conducted by WCC-Empa (WCC for O_3, CO, CH_4 and CO_2) in 1997, 1998, 2002, 2006, 2011 and 2015, respectively, and by WCC-KIT for N_2O in 2003 and 2011.

In agreement with the South African Weather Service (SAWS), WCC-SF_6 conducted the first system and performance audit for SF_6 at the Cape Point GAW station from 7 to 10 June, 2016.

During the period, the checklist, which was modified from the N_2O audit checklist, was completed in detail and an inter-comparison experiment was conducted using travelling standards (TS) of the WCC-SF_6. Repeatability and instrumental drift were also confirmed with lab standard gases which are tertiary NOAA-X2014 scale.

Finally, WCC-SF_6 appreciates all Cape Point staff and the South African Weather Service for their cooperation in WCC-SF_6 projects.

3. System and performance audit for sulfur hexafluoride

3.1 Description of the site environment

The southern hemisphere GAW Global Station Cape Point (CPT, 34° S, 18° E) is located on the top of a cliff (230 m a.s.l) at the southernmost point of the Cape Peninsula, about 60 Km away from Cape Town. The time zone is UTC +2. More detailed information can be obtained from GAWSIS (https://gawsis.meteoswiss.ch)

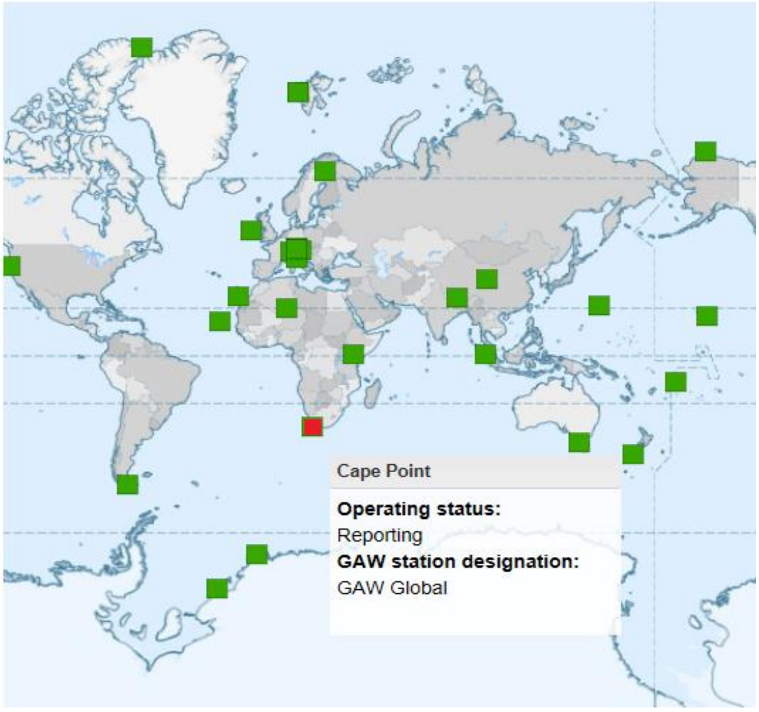


Figure 1. Global GAW stations along with a red square indicating Cape Point Global GAW station (source: GAWSIS, https://gawsis.meteoswiss.ch).

Since the dominant wind direction is SE - S – SW (about 70%), the station is subjected to maritime air from the South Atlantic. The Cape Peninsula has a Mediterranean-type climate in which the summers are generally dry and windy, whilst the winters are cold and wet. When WCC-SF_6 visited the site, a thin layer of a pollution plume under the stagnant condition was observed in the morning. This occurs commonly during the winter period due to a low mixing layer.

Cape Point, which is also a popular tourist destination, can easily be reached by road from Cape Town, which is situated 60 km to the north. From the tourist parking area an electrical funicular railway leads up the hill. Tourists are only permitted to visit Cape Point during daylight hours

3.2 Description of the observatory

Figure 2. Views of the CPT building (left) and the 30 m tower (right) as seen from south to north.

Figure 3. The inside of the CPT main building.

CPT is operated by the South Africa Weather Service with spacious and good facilities. It provides roomy laboratories and a presentation room. It is kept clean and in good shape and all gas cylinders are clean and safe. CPT is the ideal platform for continuous atmospheric research as well as measurement campaigns.

3.3 Staff/operator

The main office is located in Stellenbosch, which is 80 km away from the CPT station. Four people are involved in CPT station monitoring activities including the station manager, two scientists and one IT specialist. Approximately 2 members visit the CPT station two times a week to check and maintain the facility and instruments. When not visiting, all data are monitored remotely in the main office.

The operation and maintenance of the station are well organized with clear assignments of responsibilities (Table 1). Nevertheless, it remains a concern that only few scientists take much responsibility

Table 1. Staff responsible for the trace gas measurements at the Cape Point GAW statsion

Name/duty	**Responsibility**
Casper Labuschagne /Station manager	CO_2, CH_4, CO -CRDS
Lynwill Martin /Scientist	N_2O, SF_6, CFCs, Mercury, Meteorology, CO_2-NDIR
Thumeka Mkololo /Scientist	O_3
Danierd Spuy /IT specialist	IT and Data

Staffs who are involved in the audit on 7 to 10, June are listed below.

WCC-SF_6	Haeyoung Lee	Research Scientist (Auditor)
	Hongwoo Cheo	Technician
Cape Point station	Casper Labuschagne	Station manager
	Lynwill Martine	Scientist
	Thumeka Mkololo	Scientist
	Danierd Spuy	IT specialist

3.4 Monitoring set-up and procedure

3.4.1 Air inlet system

The location of the air intake with rain cap is on the 30 m tower above the roof of the station. About a 40-metre decabon line is connected to the lab through the oilless pump, which is located on the roof deck. The collected air samples go to the dehumidification system, which consists of a – 70 ℃ cooling trap. The cooling trap is only for GC-μECD of N_2O and SF_6. The trap is exchanged twice a week when staff visit the station. After the dehumidification system, the air moves through the sample line with stainless steel, which is connected to the injection valve.

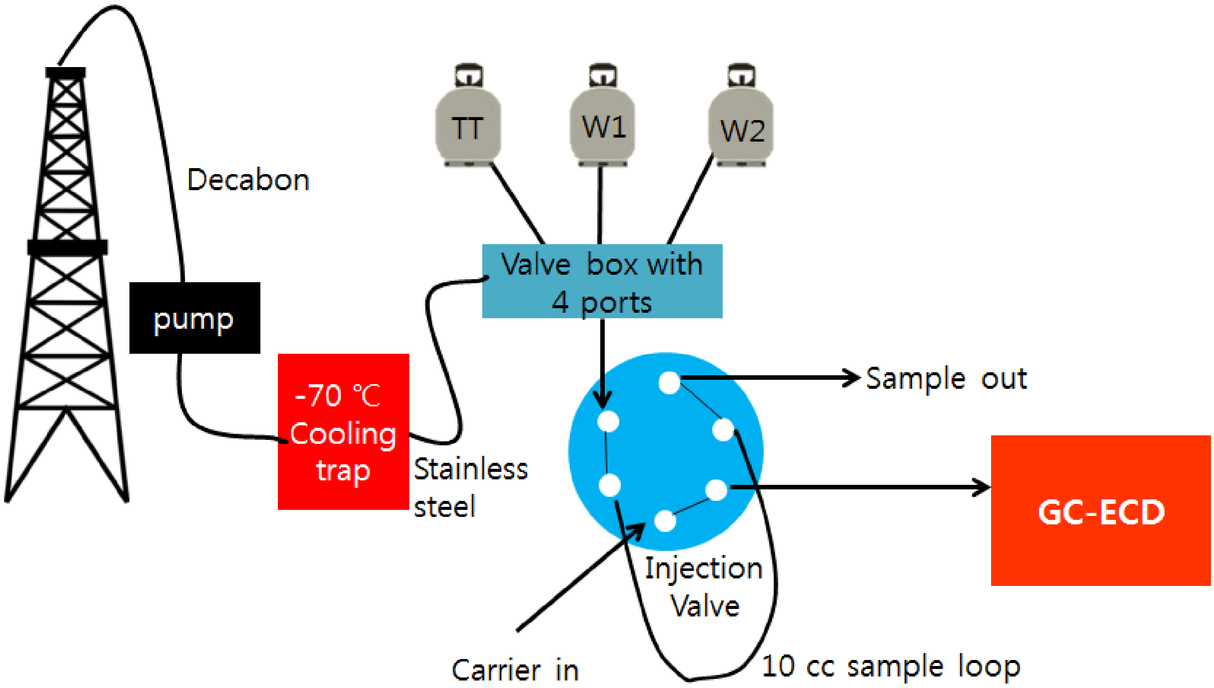


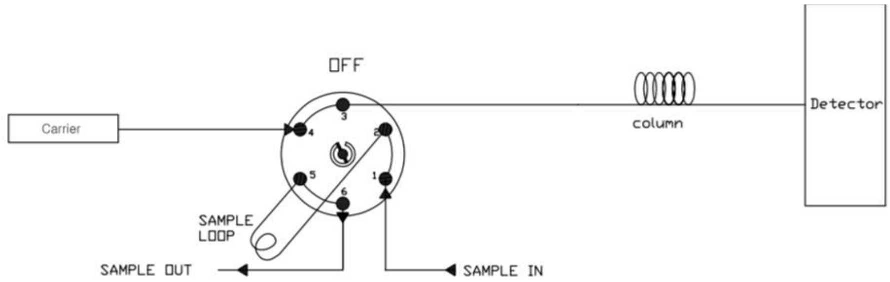
Figure 4. This illustration shows how the air inlet lines up to the instrument under the load position. A more detailed injection valve is described in Figure 5.

3.4.2 Gas chromatography system

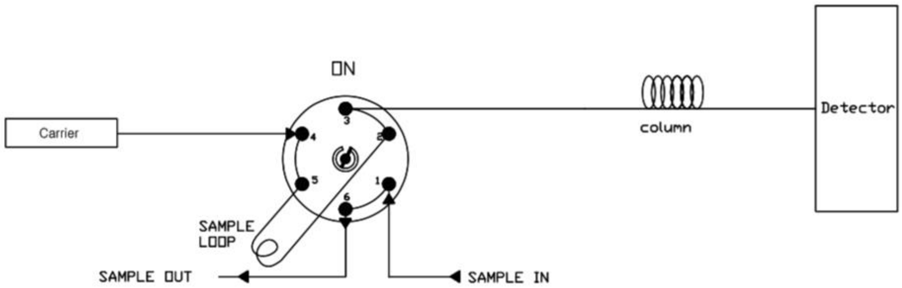
The gas chromatography system for SF_6 consists of a valve box, 6 port-injection valves and GC-μECD (Agilent 6890A). The entire analytical system is fully automated with the possibility to control and monitor the chromatogram after setting the analytical conditions manually.

The stream selection valve: After the dehumidification system, the air is conducted to the valve box through a 1/4" stainless steel line. Working standards, target gases and air sampling line are connected to solenoid valve of the valve box. When the solenoid valve is opened, the air is injected to the valve box for 2 min and then reaches equilibrium inside the valve box for 1 min. The solenoid valves of the valve box follow the sequence schedule.

The injection valve: The valve is located outside of GC and switched electronically. It was set up in 2007 for N_2O mainly with the backflush method using 12 position-injection valves. However, after having problems with it, it was changed to 6 port-injection valve with a conventional method. The current system is shown in Figure 5.



(a) Load position



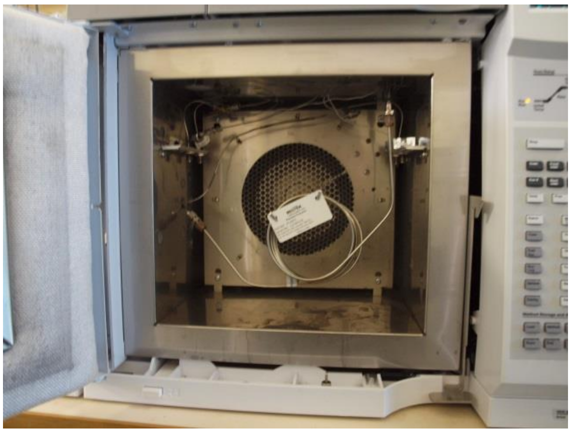
(b) Injection position

Figure 5. Schematic of air flow in 6 port-injection valve system.

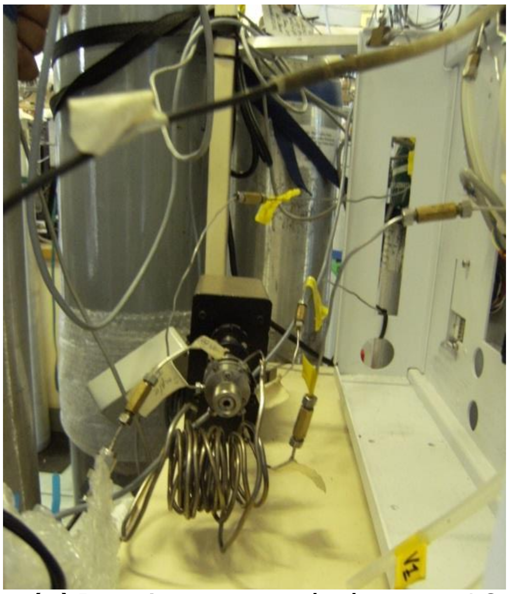
The GC-μECD (Agilent 6890A): It was installed in 2007 and there were minor changes for supplies. During the audit period, the column was changed from Heysep-Q to Porapa Q, which has similar characteristics to Heysep-Q, and a sample loop was also exchanged for a new one (Figure 6). According to the changes, the analytical condition was also adjusted.

Here we see the current analytical condition, which was measured or checked during the audit period (Table 2).

(a) Previous column setting condition (Heysep-Q 8+6 ft, 1/8”)



(b) Current column setting condition (Porapa-Q 12 ft, 1/8”)



(c) Previous sample loop – 10cc

(d) Current sample loop – 10cc

Figure 6. Previous column and sample loop (left panel) and current ones (right panel)

Table 2. Analytical conditions

Analytical condition	
Sample loop size	10 cc
Carrier gas	P-10, 100 ml/min
Make up gas	None
Sample injection flow	50 ml/min
Oven	45 ℃ isothermal
Column	Porapa-Q 1/8” 12ft

After exchanging the column and analytical conditions, a peak area increased, the baseline became steady, and N_2O peaks were separated better than before but still have some difficulties (Figure 7) indicating that retention time should be adjusted.

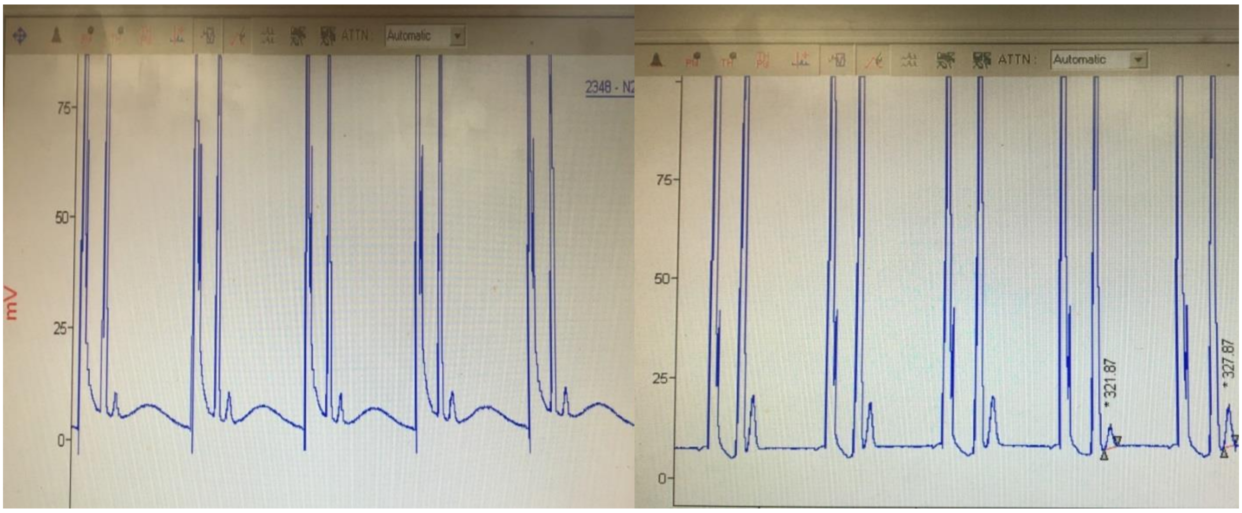


Figure 7. The chromatogram under the previous analytical condition (left) and current one (right).

3.4.3 Recommendation

- Air inlet and dehumidification system are adequate for the measurement of SF_6. However, pump information, such as total flow and brands, is necessary in order to prepare for extra supplies and to plan for the addition of new instruments.
- Since carrier gas flow rate and sample flow rate are checked with a portable flow meter manually and randomly, there is less confidence that gases inject the same amounts for the analysis. During the audit period, different flow rates were confirmed according to the tanks and samples. We highly recommend using the Mass Flow Controller (MFC).
- To separate each peak, more sophisticated adjustments are necessary, such as flow rates and analysis conditions.

3.5 Calibration and maintenance

3.5.1 General

The station has been operated by four staff under the South Africa Weather Service and they visit 2 times a week to maintain the system.

The valve system can be controlled remotely and the data can be monitored at the Stellenbosch office 90 km away from Cape Point station. However, the GC detector and oven cannot be controlled from a remote station.

3.5.2 Sampling and calibration

A leak check: The gas leakages between every connector are checked while visiting the station, and before auditing they confirmed it on 25 April, 2016.

Sequence of analyses and calibration method: The sequence of the working standard (W) and ambient air (A) is: W1-W1-W2-W2-A-A-W1-W1-W2-W2-A-A. It takes 15 minutes for one analysis. Here, W1 and W2 indicate two different levels of working standards. Every 24 hours, target tank (TT) is injected in place of ambient air: W1-W1-W2-W2-TT-A-W1-W1-W2-W2-A-A. This is a routine operation for level-1 calibration.

The level of two working standards and one target tank is around 6 ppt, which is different from the ambient concentrations of around 8 ppt, showing that this calibration method can lead to under- or over-estimated ambient concentrations.

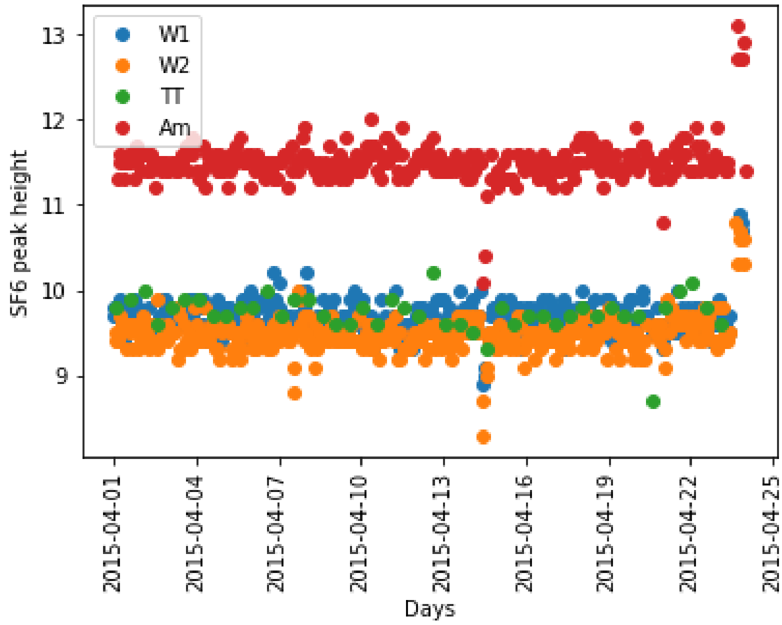


Figure 8. SF_6 peak areas of two working standards (W1 and W2), ambient (Am) and the target tank (TT) from 1 to 5, April.

Linearity and repeatability of measurement: Cape Point station has not tested the linearity of the detector (μECD). This is recommended with 3-point standard gases that are well separated from each other at least [2]. Since the response curve has non-linear characteristics on μECD normally, this test is very important for the calibration.

While conducting the audit, three different lab standard gases were injected for the linearity and repeatability test. The experiment sequence followed as L1-L2-L3-L1… repeatedly over 10.5 hours and each cylinder was duplicated 3 times in 30 minutes. We could have 8 sets according to the sequence.

For the linearity test, each cylinder was corrected using the drift factor. This method is described precisely in [4] and the way to calculate uncertainty is also presented. In this experiment, we didn't consider the uncertainty of the lab standard itself. Three points are inadequate since normally ECD showed non-linear characteristics and there was a big gap between L1 and L3 in this experiment. The residual values were in the DQO, raising the possibility that the detector could have linear characteristics. However this test should be conducted with more standard gases to confirm the linearity again since L2 cylinder, which was between L1 and L3, was a little behind the compatibility goal.

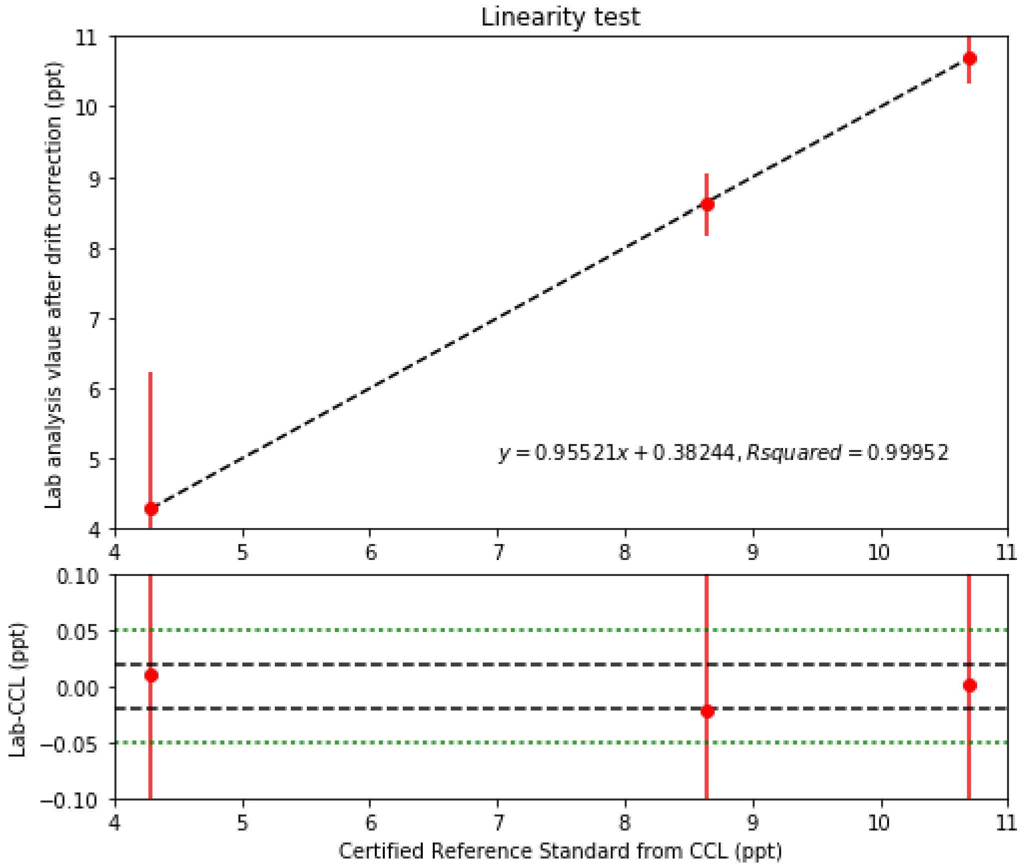


Figure 9. Linear fit of the SF_6 calibration curve (y = 0.99786x + 0.01358, R2=0.99998). Where L1: 4.28 ± 0.06 ppt, L2: 8.64 ± 0.03 ppt and L3: 10.69 ± 0.04 ppt. The cylinder information is listed in 3.6.2, this report.

To see the repeatability and drift, we used the data in which no calibrations and no drift correction were applied. The drift is calculated using a linear regression with the data from 10.5 hours for each cylinder. The slope of the regression represents the drift of the instrument over 10.5 hours and is showed in Figure 10. When we assumed that the first mean value was the same as the certified value, it more decreased at high concentrations.

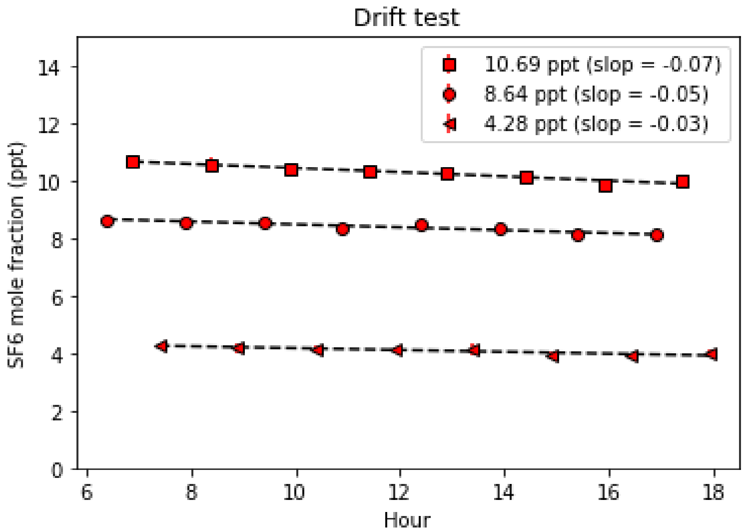


Figure 10. The time series for SF_6 measurements over 10.5 hours. Each plot represents the mean value of 30 minutes.

For the repeatability assessment, we calculated the standard deviation of 30 minutes and 10.5 hours of data. The short-term repeatability was the standard deviation of 30 minutes of data, which was measured 3 times with the same cylinder and suggested the value in Table 3.

Table 3. The Repeatability assessment. Each cylinder was measured 3 times for 30 minutes during 10.5 hours with 8 sets.

cylinder	Repeatability during 30 mins (ppt, N = 3)	Repeatability during 10.5 hours (ppt, N = 24)
L1 : 4.28 ppt	0.01-0.20	0.14
L2 : 8.64 ppt	0.07-0.17	0.20
L3:10.69 ppt	0.02-0.23	0.28

The results from the repeatability and the drift test indicate that GC response was not stable and the calibration should be conducted very often, at least every one hour.

3.5.3 Maintenance

The analysis system is maintained when staff visit the station to check the gas inlet and gas leaks, and bake a column inside of GC every 3 month.

3.5.4 Recommendations

- A linearity test has to be conducted on a regular basis with more than 4 cylinders for the target range.
- According to the drift and repeatability test, the sequence that CPT implements now was appropriate, showing that working standard gases were injected every hour. However, the levels of the standard gases value should bracket the ambient level.

3.6 Standard

3.6.1 Regulators and connections

The pressure regulators are a type of Scott Specialty with two stage brass. The tubing from the cylinders to the valve is made out of stainless steel, 1/16” o.d.

3.6.2 Laboratory standards

Eight laboratory standards were prepared in Aculife-treated aluminum 30 L cylinders from Scott Specialty Gases. They were NOAA tertiary standards with the range nominally from 2 to 10 ppt and converted to the NOAA-X2014 scale according to the webpage of NOAA (www.esrl.noaa.gov/gmd/ccl/refgas.html). For CO_2, CH_4 and CO, it is highly recommended that they be recalibrated every 3 years for precision. There is no certain period for the SF_6 recalibration but it is recommended to recalibrate them for long term stability and precision. Their information is on Table 3.

Table 4. Laboratory standards for SF_6 at the station. Cylinder numbers, year of purchase and mole fractions with standard deviation as reported by the Central Calibration Laboratory. They are in NOAA-X2014 scale.

	Cylinder ID	**Year**	**SF_6** [ppt]	**Unc.** [ppt]
1	CA05050	2002	2.37	0.02
2	CA05081	2002	3.22	0.02
3	CA06213	2005	6.21	0.07
4	CA06267	2005	4.28	0.06
5	AAL071167	2006	5.53	0.02
6	AAL071150	2006	5.97	0.03
7	CA08110	2008	10.69	0.04
8	CA08138	2008	8.64	0.03

3.6.3 Working standards

Working standards were prepared as two cylinders at the station for the routine determination of ambient mole fractions. The cylinders are 30 L type aluminum and high pressure cylinder with ambient air. The comparison of the working standards with laboratory standards is performed for N_2O quarterly but not for SF_6.

3.6.4 Target gas

Target gas was prepared one cylinder at the station for the routine determination of instrumental drift. The cylinder is 30 L type aluminum and high pressure cylinder with ambient air. The comparison of the target gas with working or laboratory standards was implemented as soon as it was filled with ambient air.

3.6.5 Recommendations

- Working standard gases and target gases should be calibrated against laboratory standards regularly not only for N_2O but also for SF_6.

- Since prepared lab standards are over a decade old, it is recommended to recalibrate or prepare new ones according to the remaining pressures.

3.7 Data acquisition and processing

3.7.1 General

Data acquisition of the gas chromatographic signals and parameters is handled by the software "Azur" and "Test Point". It is controlled remotely in the Stellenbosch office. The data is synchronized in the local time zone.

After the drift correction result (refer to 3.5.2), it was handled by a statistical method such as outlier. The instrument logbook is considered part of the data validation procedure and background concentration is selected by statistical method.

Final data validation is implemented by Casper, the manager of the station, and Lynwill, who is in charge of SF_6.

3.7.2 Chromatogram evaluation

Every report of the chromatogram from GC is stored in the drive and peak area and height were used for the data analysis. This information is used for data quality control. Peak integration is performed automatically. The chromatogram can also be reintegrated when it is necessary. Sometimes manual integration is more correct than automatic integration.

The sequence of peaks was O_2, N_2O and SF_6 and the tail of the O_2 peak could overlap the tail of N_2O. However, after the exchanging of a column and analysis condition, peaks were separated well.

The baseline is automatically reset after every injection.

3.7.3 Recommendations

- It is highly recommended to use peak area rather than peak height due to the broadened characteristic of SF_6 with Porapa-Q column.

3.8 Data management and submission

3.8.1 General

The CPT station has its own backup policy and stored all data in NAS-Drive. The final version of the data is stored every 10 years (2000-2009, 2010-2019…) as txt format. The graphical representations of the data are available but not near real time.

Since the data has not been submitted to the World Data Centre for Greenhouse Gases, it does not have a record of calibration history.

3.8.2 Recommendations

- Data submission to the World Data Centre on Greenhouse Gases is highly recommended.

3.9 Documentation

The WMO GAW Measurement guideline and instrument manuals are available to the operators. The field logbooks and instrument logbooks with hand-written type are maintained on the site in an orderly manner. In the logbook, instrumental manipulations, changes, and the reasons for questionable data are included. These logbooks are stored in an alternative location: an NAS-computer drive at the Stellenbosch office.

3.10 Inter-comparison experiment of SF_6 standards

3.10.1 Experimental procedure

Before conducting the audit, WCC-SF_6 sent two travelling standards with two pressure regulators to the Cape Point GAW station. They arrived at the station before the audit.

Table 5. Cylinder information on the SF_6 travelling standards

Manufacture	Luxfer Gas cylinders (UK)
Cylinder #	D379126, D376069
The level of SF_6	2 level between 8 and 10 ppt in natural dry air
Material	Aluminum 10 L cylinders

Two travelling standards for this inter-comparison experiment were listed in table 5. The WCC-SF_6 analysis method is described [4]. For WCC-SF_6, two travelling standards were calibrated against laboratory standards of NOAA-X2014 scale with a two-point analysis method. The levels of standards to certify the traveling standards were selected with a similar level that covers the target range for the calibrations.

For Cape Point stations, two travelling standards were calibrated against working standards, which are traceable to NOAA-X2014 scale with a two-point analysis method as well as W1- sample – W2 –W1 according to the sequence which had been set by CPT. The working standards levels were 8.64 ppt and 10.69 ppt for both travelling standards during the experiment.

Flushing and leak checks were performed before doing this experiment. There were no modifications of the GC system for the inter-comparison experiment. The one cylinder was injected without duplications but the sequence cycle was duplicated.

3.10.2 Results of the SF_6 inter-comparisons

The differences are beyond the extended compatibility goal, ± 0.05 ppt with relatively high standard deviations. As we suggested in 3.5.2, the linearity test should be done for the range between 8 ppt to 10 ppt again. In addition, since random errors can occur, samples should be injected immediately at least two times.

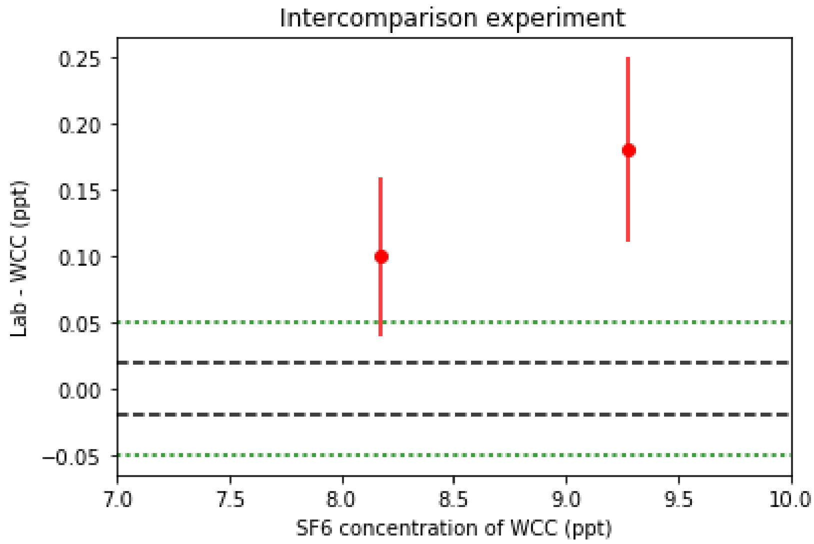


Figure 10. The differences between CPT and WCC. The black dotted line is the compatibility goal (±0.02 ppt) and the green line is the extended compatibility goal (±0.05 ppt)

Table 6. Summary results for the inter-comparison as reported by WCC-SF_6 and CPT.

Cylinder #	WCC-SF_6 (ppt) N=5	Cape Point (ppt) N = 9	Difference (ppt) CPT - WCC
D379126	9.28 ± 0.04	9.46 ± 0.07	0.18
D376069	8.17 ± 0.02	8.27 ± 0.06	0.10

4. References

[1] J.Klausen, H.E.Scheel, M. Steinbacher, 2010: WMO/GAW Glossary of QA/QC-Related Terminology, version 1.0 (last update 2016-05-26 by M. Steinbacher)

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[3] J. Klausen, C, Zellweger, and H.E. Scheel, 2006: The audit questionnaire for system and performance audits of atmospheric trace gas measurement at WMO/GAW sites, Version 1.3-20061214

[4] WMO, 2018: The Calibration Methods of GC-μECD for Atmospheric SF_6 Measurements, WMO/GAW No.239.