Comparison of reference standards to global scales

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HIGHGAS STAKEHOLDER WORKSHOP, 9th March 2016, VSL, Delft

International GAW Activities of Switzerland

Empa
Materials Science and Technology

QA/SAC Switzerland

pmod wrc

PMOD/WRC Davos

Empa
Materials Science and Technology

WCC-Empa (O_3, CO, CH_4, CO_2)

GAWSIS

Schweizerische Eidgenossenschaft
Confédération suisse
Confederazione Svizzera
Confederaziun svizra

Federal Department of Home Affairs FDHA
Federal Office of Meteorology and Climatology MeteoSwiss

Empa
Materials Science and Technology

Support to GAW Global Stations Mt. Kenya, Algeria, Indonesia

Support to Ozonsounding Nairobi, Kenya

Schweizerische Eidgenossenschaft
Confédération suisse
Confederazione Svizzera
Confederaziun svizra

Federal Department of Home Affairs FDHA
Federal Office of Meteorology and Climatology MeteoSwiss

Support to GCOS Cooperation Mechanism

Schweizerische Eidgenossenschaft
Confédération suisse
Confederazione Svizzera
Confederaziun svizra

Swiss Agency for Development and Cooperation SDC

CATCOS
Capacity Building and Twinning for Climate Observing Systems

Schweizerische Eidgenossenschaft
Confédération suisse
Confederazione Svizzera
Confederaziun svizra

Swiss Agency for Development and Cooperation SDC

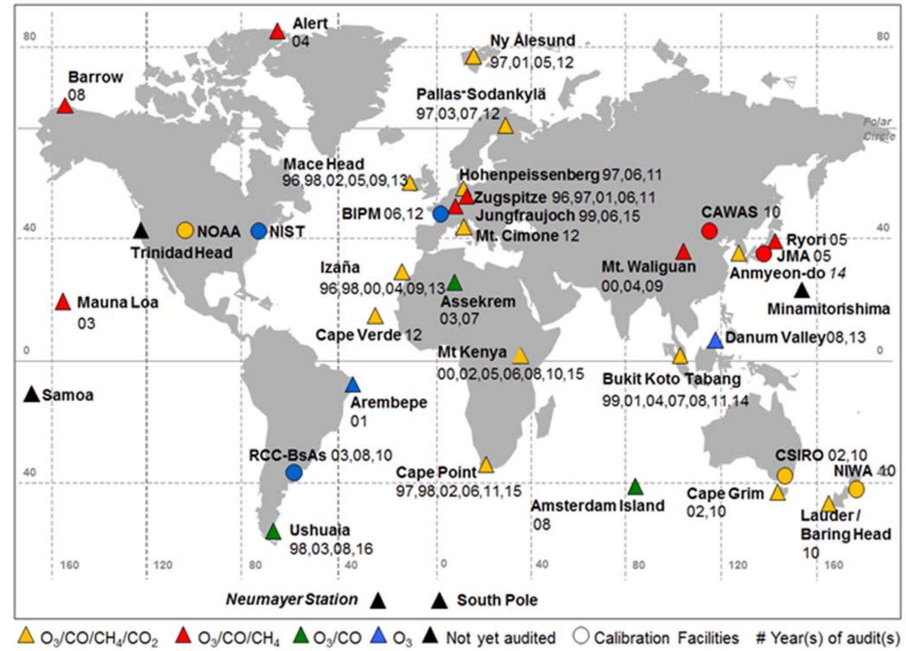
Empa
Materials Science and Technology

PAUL SCHERRER INSTITUT
PSI

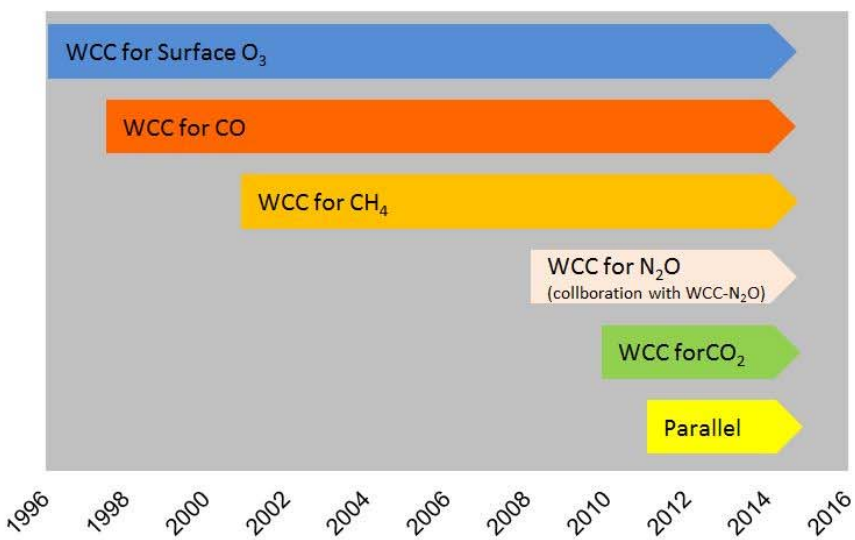
International GAW Activities of Switzerland

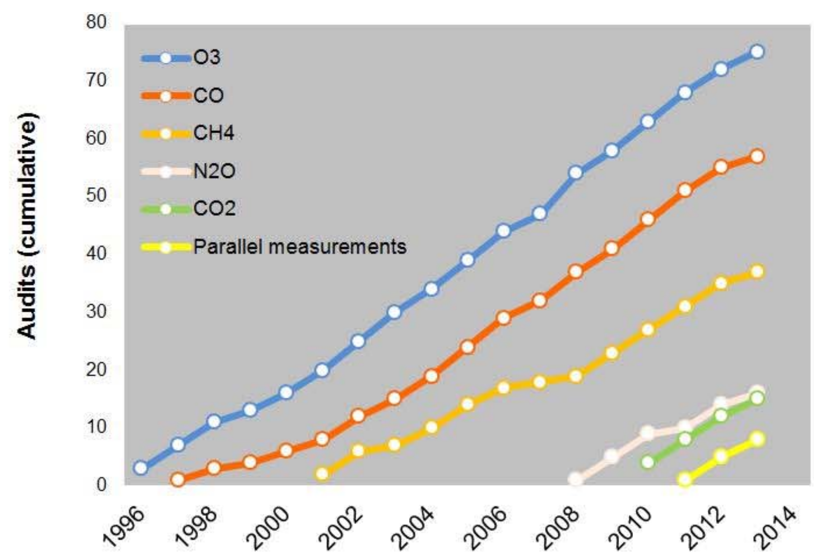
Empa
Materials Science and Technology

WCC-Empa
(O_3, CO, CH_4, CO_2)



Audits by WCC-Empa from 1996 - 2014





Components of the WMO/GAW Programme

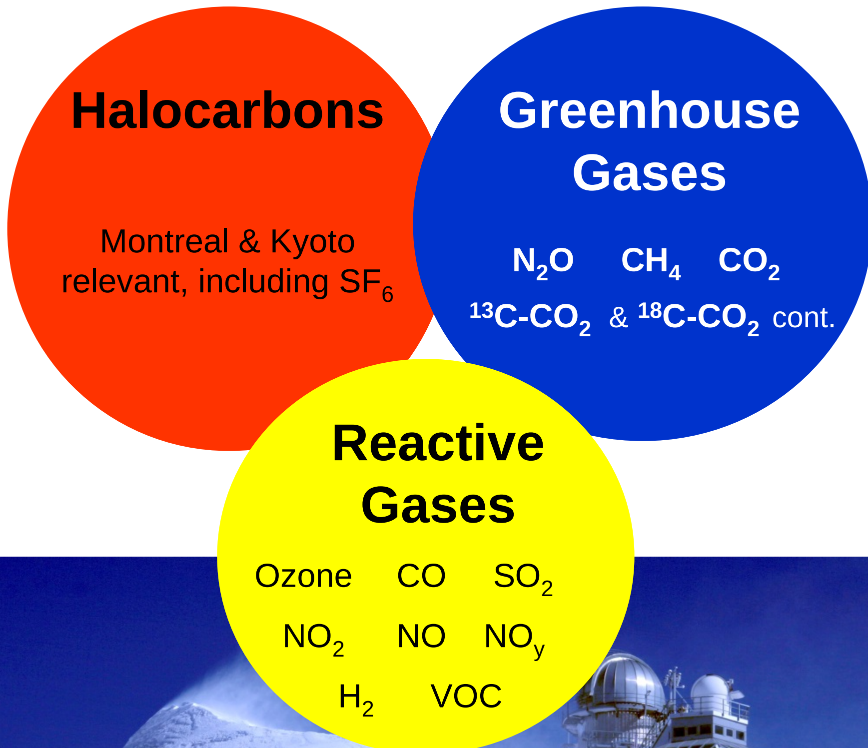
Expert Groups	EPAC SSC	Scientific Advisory Groups *Ozone, UV, GHG, RG, Aerosol, TAD, GURME*	ET-WDC	SAG NRT applications	
Administration Management	WMO/GAW Secretariat		IGACO office *Ozone/UV*		
Central Facilities	QA/SACs	WCCs/ RCCs	CCLs	WDCs	GAWSIS
Observing Systems	Contributing Networks	GAW stations *Global, Regional and Local*	Aircraft Satellite		
Users and applications	Operational Centres	Programmes IGAC, iLeaps..	Systems GCOS, GEO...	Parties to the Conventions UNFCCC, Vienna	Research Projects

NMHSs

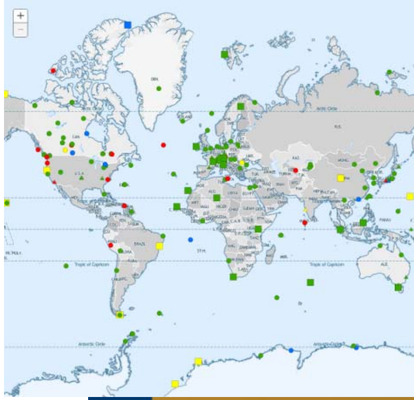
Comprehensive Measurement Programme @ JFJ

Reactive Gases and Greenhouse Gases

More than 70 continuous time-series



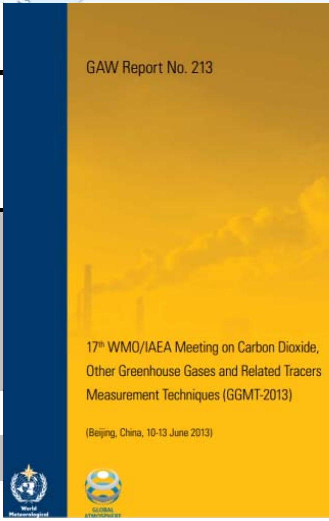
WMO/GAW Greenhouse Gas Measurements

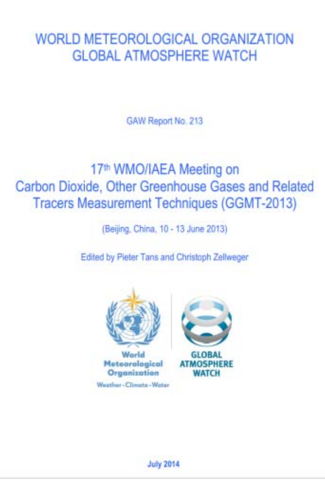


- We need: Comparable data
  (= data that is on the same calibration scale or traceable to SI)
- Compatible data
  (= the absolute value of the difference of any pair of measured quantity values from two different measurement results is smaller than some chosen multiple of the standard measurement uncertainty

Table 1- Recommended compatibility of measurements within the scope of GGMT

Component	Compatibility goal	Extended compatibility goal	Range in unpolluted troposphere	Range covered by the WMO scale
CO_2	± 0.1 ppm (Northern hemisphere) ± 0.05 ppm (South. hemisphere)	± 0.2 ppm	360 - 450 ppm	250 – 520 ppm
CH_4	± 2 ppb	± 5 ppb	1700 – 2100 ppb	300 – 2600 ppb
CO	± 2 ppb	± 5 ppb	30 – 300 ppb	20 -500 ppb
N_2O	± 0.1 ppb	± 0.3 ppb	320 – 335 ppb	260 – 370 ppb
SF_6	± 0.02 ppt	± 0.05 ppt	6 – 10 ppt	1.1 – 9.8 ppt
H_2	± 2 ppb	± 5 ppb	450 – 600 ppb	140 –1200 ppb
$\delta^{13}C\text{-}CO_2$	± 0.01‰	± 0.1‰	-7.5 to -9‰ vs. VPDB	
$\delta^{18}O\text{-}CO_2$	± 0.05‰	± 0.1‰	-2 to +2‰ vs. VPDB	
$\Delta^{14}C\text{-}CO_2$	± 0.5‰	± 3‰	0-70‰	
$\Delta^{14}C\text{-}CH_4$	± 0.5‰		50-350‰	
$\Delta^{14}C\text{-}CO$	± 2 molecules cm^{-3}		0-25 molecules cm^{-3}	
$\delta^{13}C\text{-}CH_4$	± 0.02‰	± 0.2‰		
$\delta D\text{-}CH_4$	± 1‰	± 5‰		
O_2/N_2	± 2 per meg	± 10 per meg	-250 to -800 per meg (vs. SIO scale)	





Outline

- Where are we with respect to the WMO/GAW compatibility goals?
  - Results of the recent WMO/IAEA Round Robin Comparison Experiment
  - Analysis of performance audit results of WCC-Empa

- Examples of a parallel measurements during a WCC-Empa audit

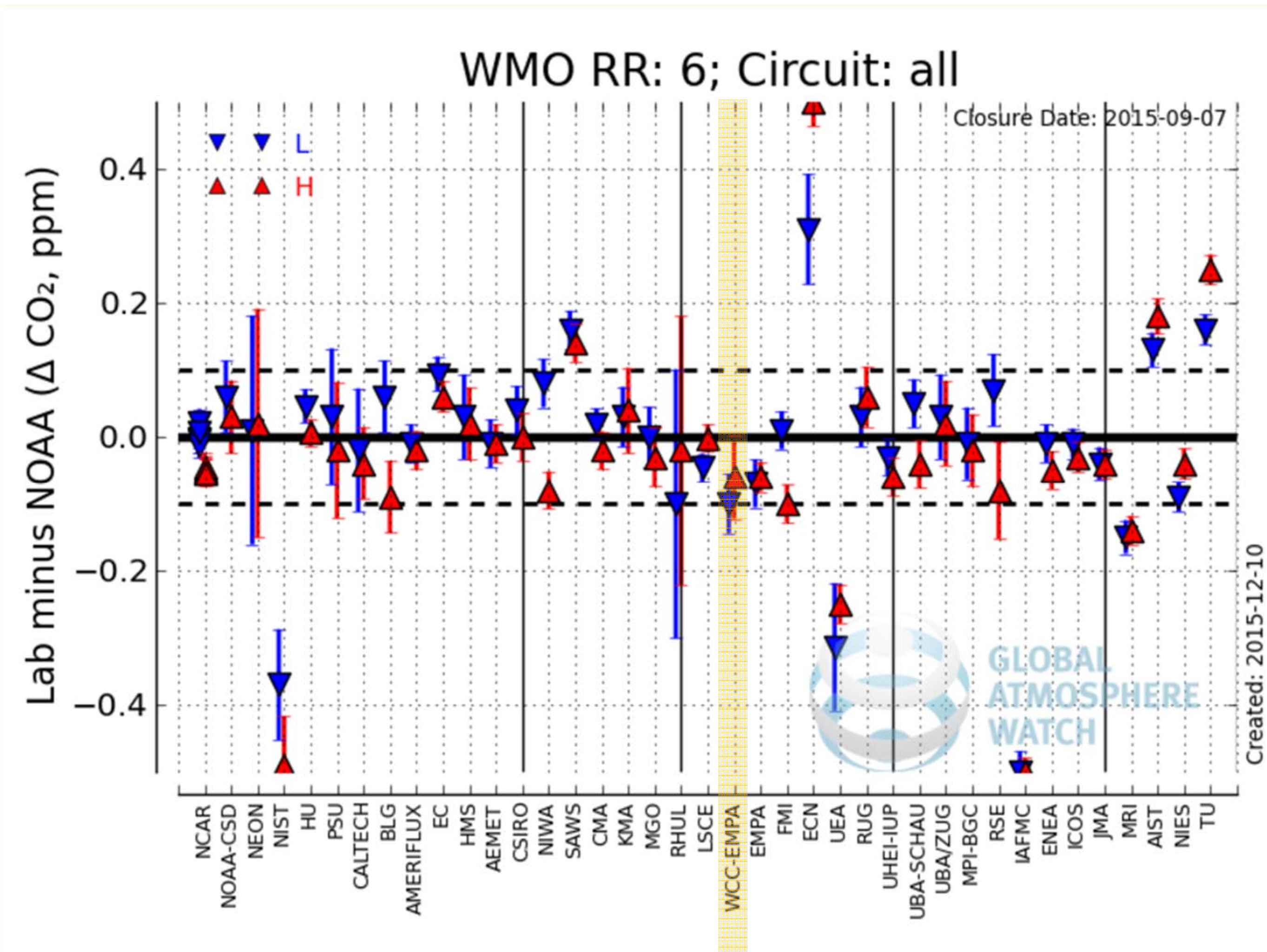
- Our role in the HIGHGAS project

WMO/IAEA Round Robin Comparison Experiment

- The primary goal of the WMO/IAEA Round Robin Comparison Experiment is to assess the level to which participating laboratories maintain their link to the WMO mole fraction scales using normal operating procedures.
- RR took place between January 2014 and September 2015.
- Focus on CO_2 but also comparisons of CH_4, CO, N_2O, H_2, SF_6 and CO_2 isotopes.
- Participation of 48 laboratories (39 reporting for CO_2).
- For species with recent changes in the NOAA scale (CO, CH_4) re-submission of results was possible until 1st March 2016.

Source: http://www.esrl.noaa.gov/gmd/ccgg/wmorr/index.html

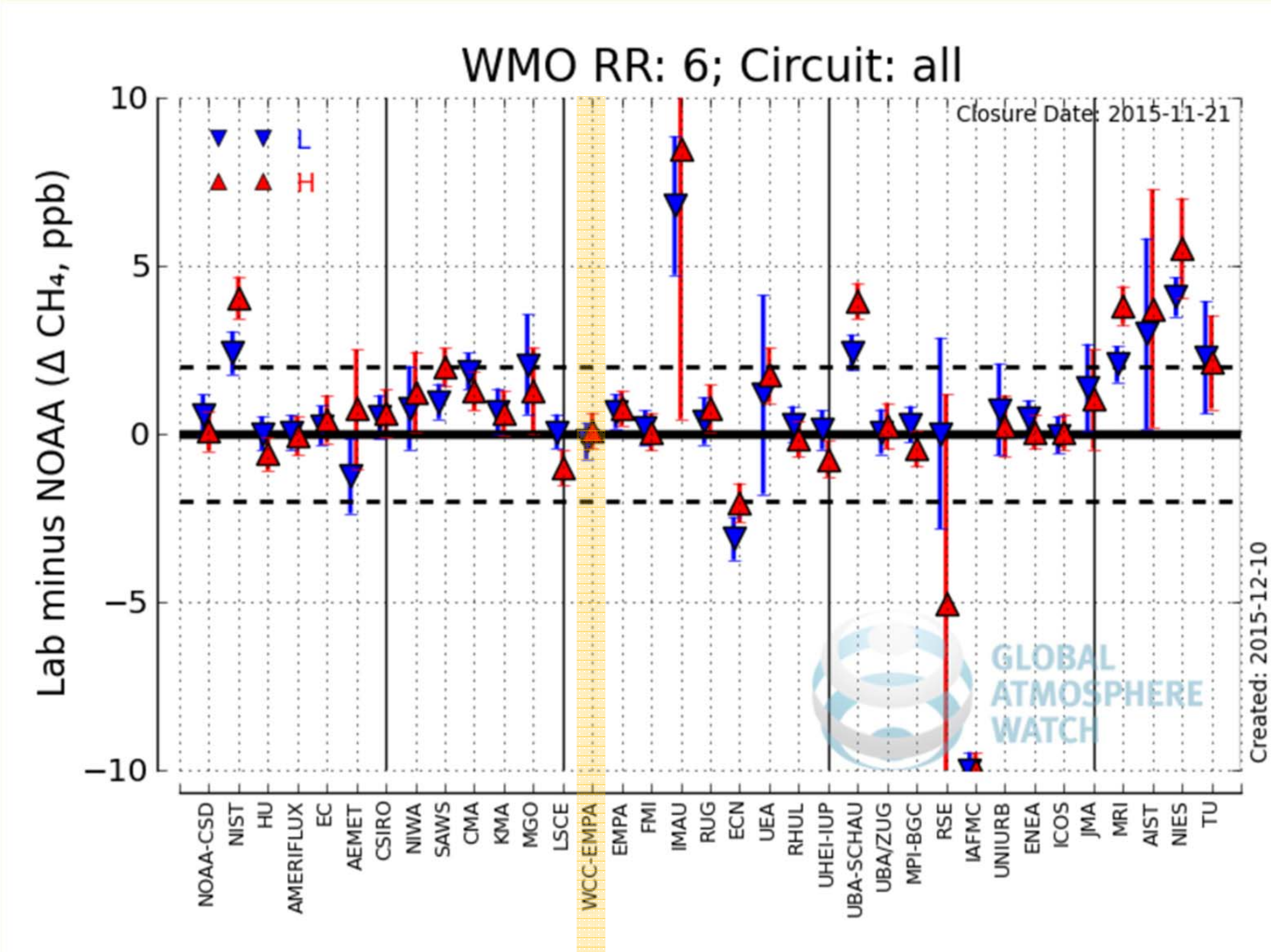
WMO/IAEA Round Robin Comparison Experiment: CO_2



- 79% within WMO goal
- 56% within ½ of goal
- All laboratories were on the WMO-CO2-X2007 calibration scale except Japanese labs (MRI, AIST, NIES, TU) which used their own scales
- NIST ?

Source: http://www.esrl.noaa.gov/gmd/ccgg/wmorr/index.html

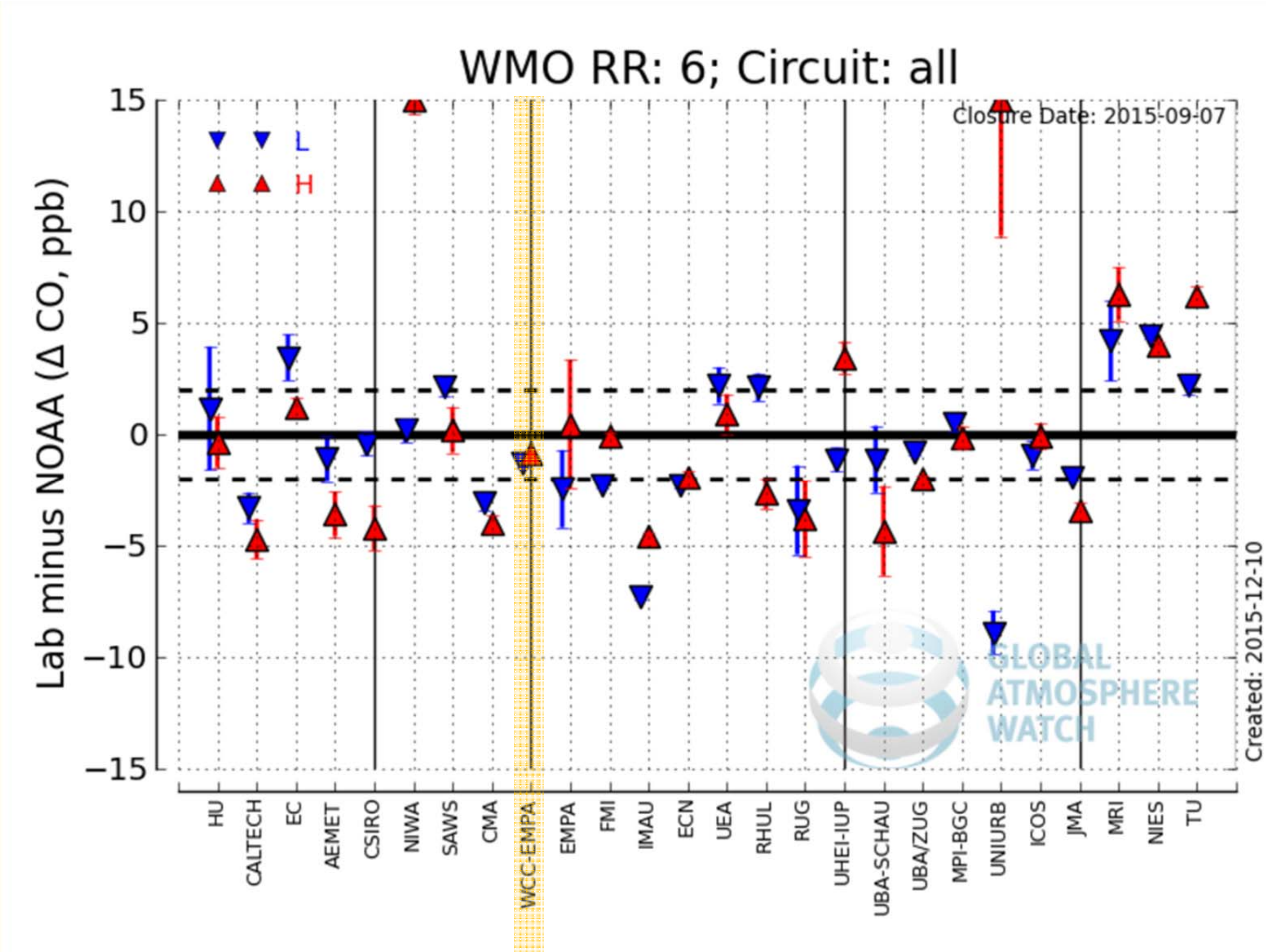
WMO/IAEA Round Robin Comparison Experiment: CH_4



- 71% within WMO goal
- 54% within ½ of goal
- All laboratories were either on the WMO-CH4-X2004 or X2004A calibration scale except Japanese labs (MRI, AIST, NIES, TU)
- These Japanese labs compare better to NIST (which is on X2004 according to reported results) (?)

Source: http://www.esrl.noaa.gov/gmd/ccgg/wmorr/index.html

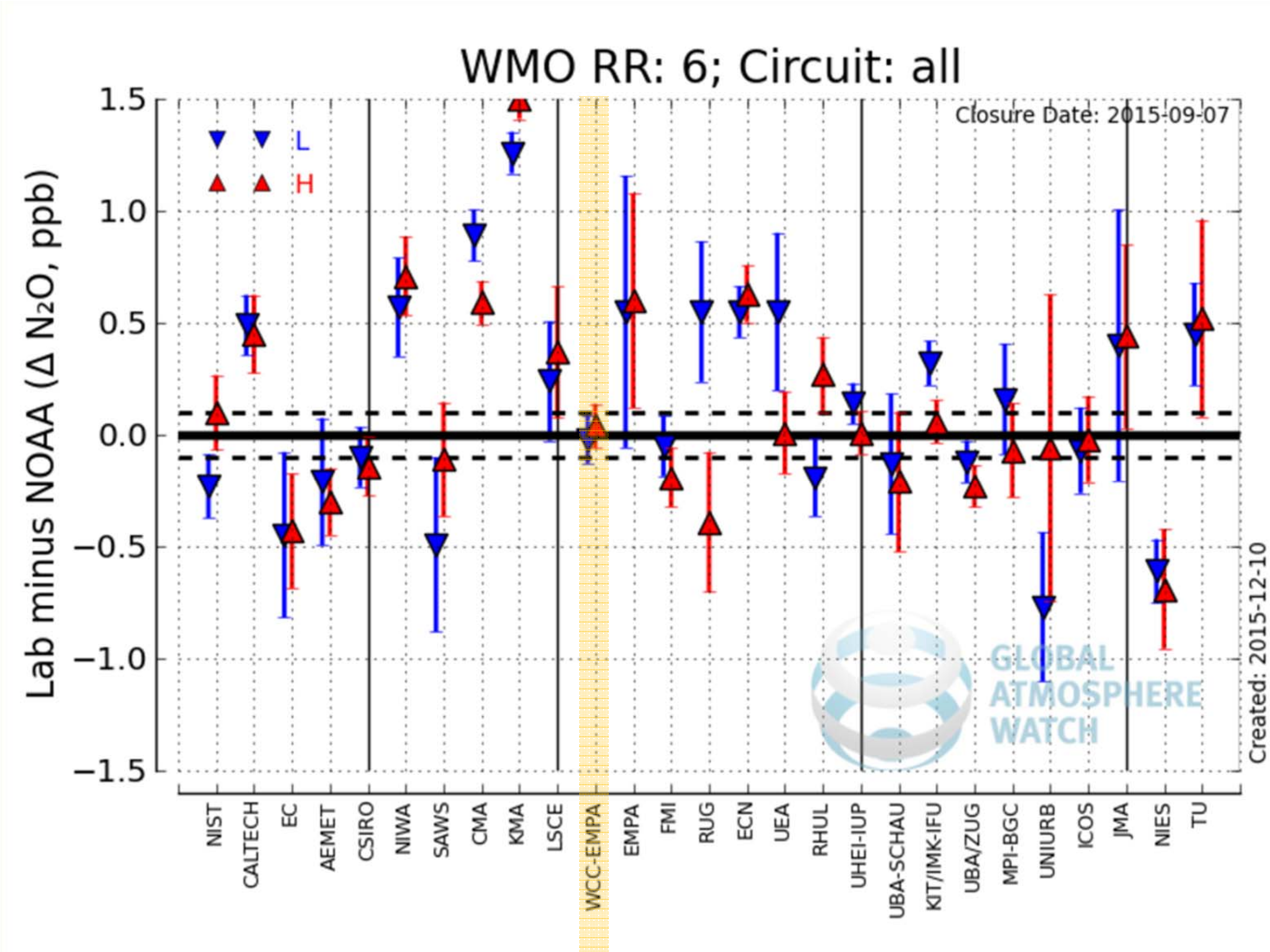
WMO/IAEA Round Robin Comparison Experiment: CO



Source: http://www.esrl.noaa.gov/gmd/ccgg/wmorr/index.html

- 35% within WMO goal
- 12% (2 of 26 labs) within ½ of goal
- All laboratories were either on the WMO-CO-X2004 or X2014 calibration scale except Japanese labs (MRI, NIES, TU) and CSIRO
- The results show that the WMO compatibility goal of 2 nmolmol^{-1} is challenging
- Most likely this is due to issues with standards (drift) and limitations of the analytical techniques
- Update to X2014A on the way

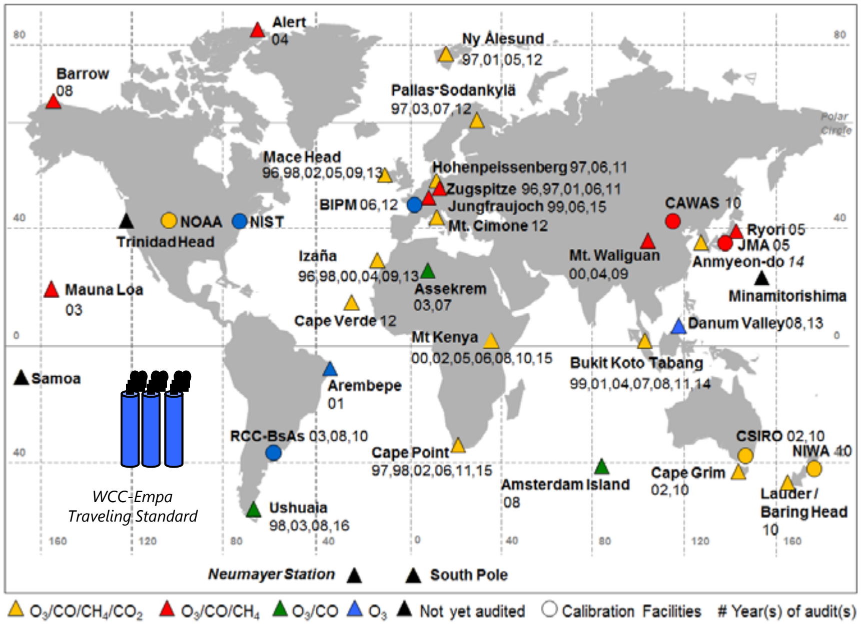
WMO/IAEA Round Robin Comparison Experiment: N_2O

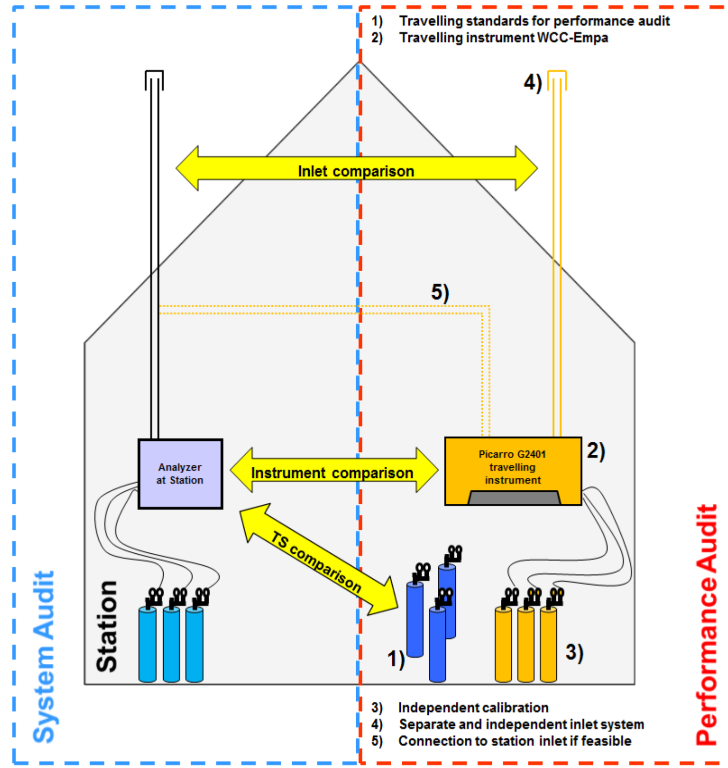


Source: http://www.esrl.noaa.gov/gmd/ccgg/wmorr/index.html

- 11% within WMO goal
- 7% (2 of 27 labs) within ½ of goal
- All laboratories were on the WMO-N2O-X2006A calibration scale except Japanese labs (NIES, TU) which used their own scales
- The results show that the WMO compatibility goal of 0.1 nmolmol^{-1} is challenging
- Most likely this is less an issue of standards but more likely due to limitations of the analytical techniques
- Most labs were using GC/ECD

Audits: Travelling Standards vs. Parallel Measurements

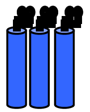




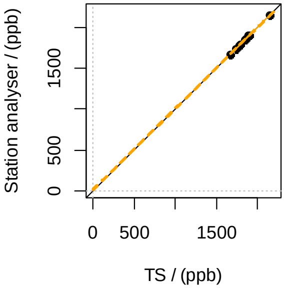
- ☹ Only instrument comparison
- ☹ Snapshot in time
- ☹ Special care might influence results
- ☺ Covers wider mole fraction range
- ☺ Repeatability conditions

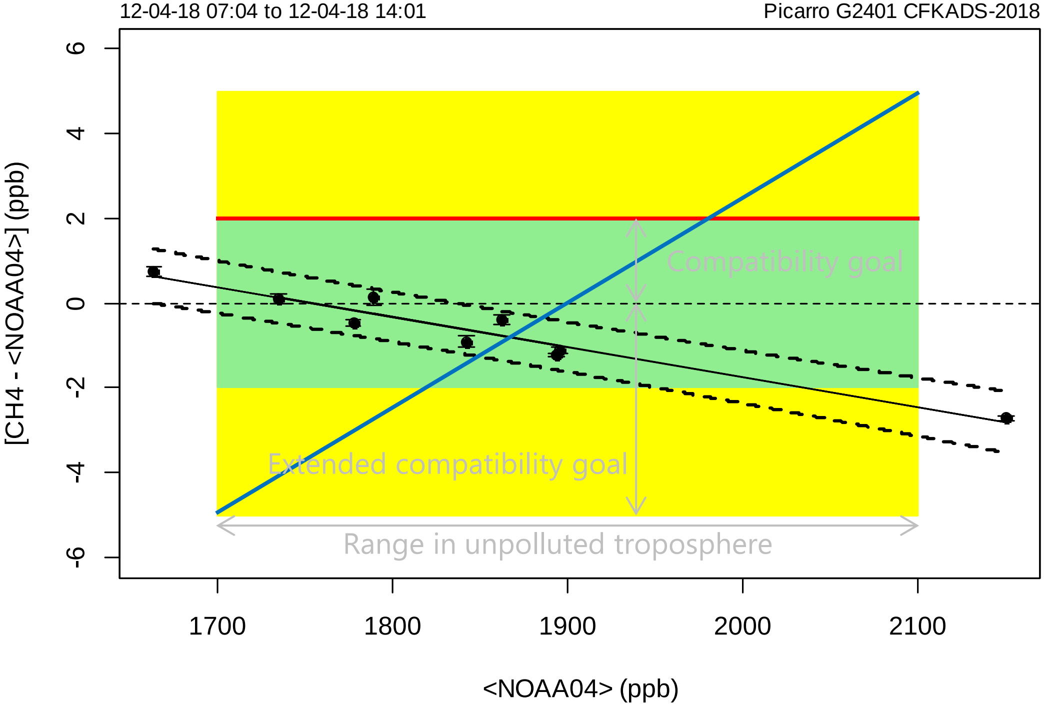
- ☺ Assessment of the whole system
- ☺ Longer time period
- ☺ Less influence by operator
- ☹ Limited to ambient mole fraction range

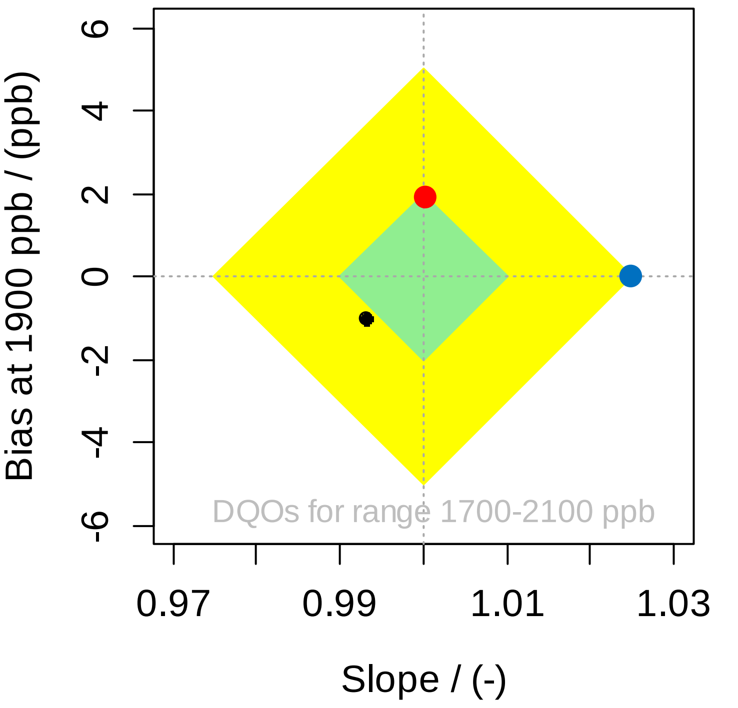
Explanation for the following figures…



WCC-Empa TS

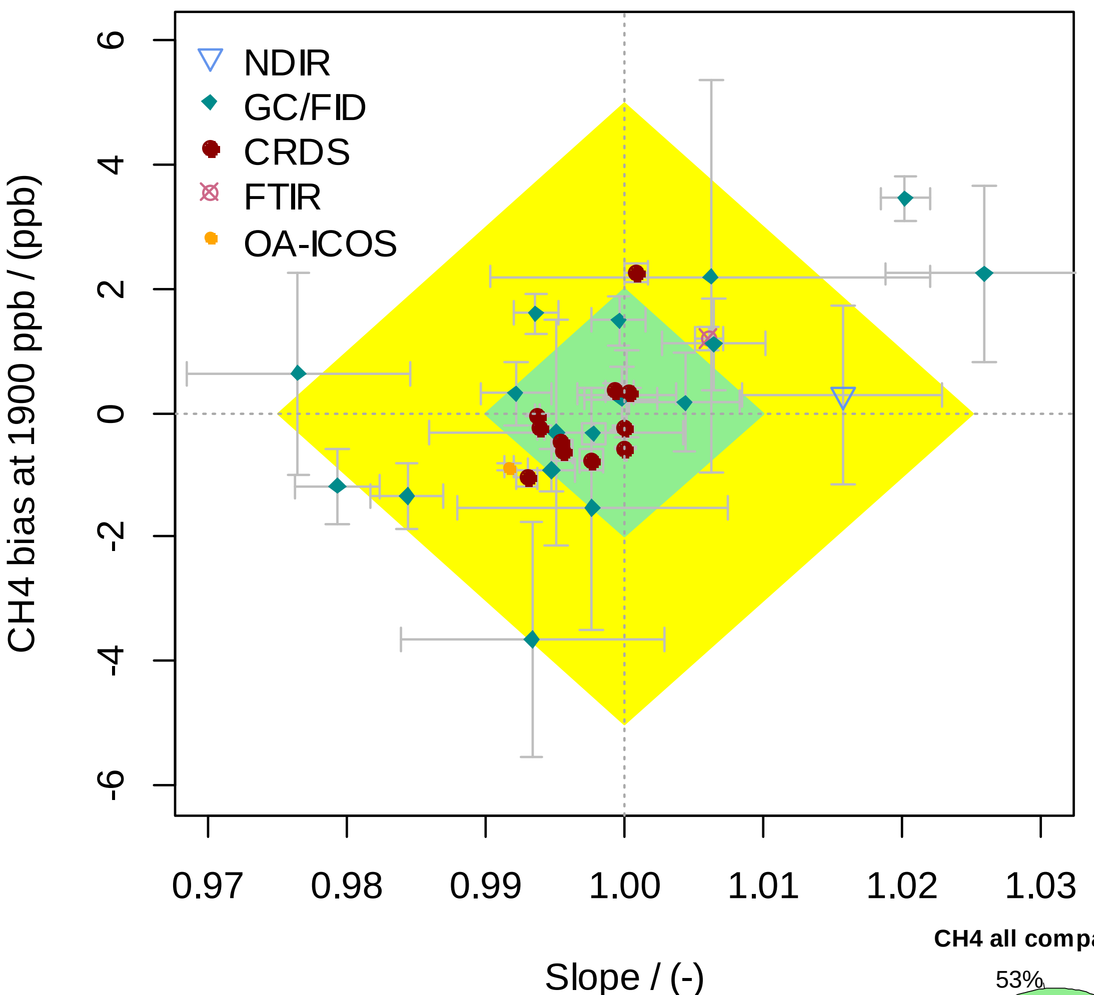




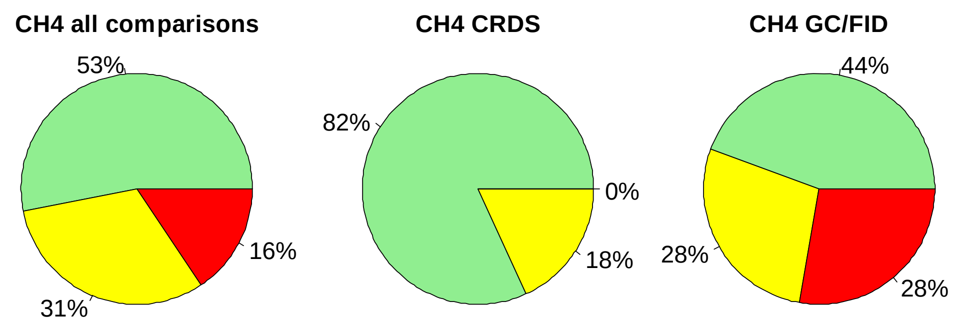


- Intercept = 12.4 ppb
- Slope = 0.993
- Bias @ 1900 ppb = -1.03 ppb

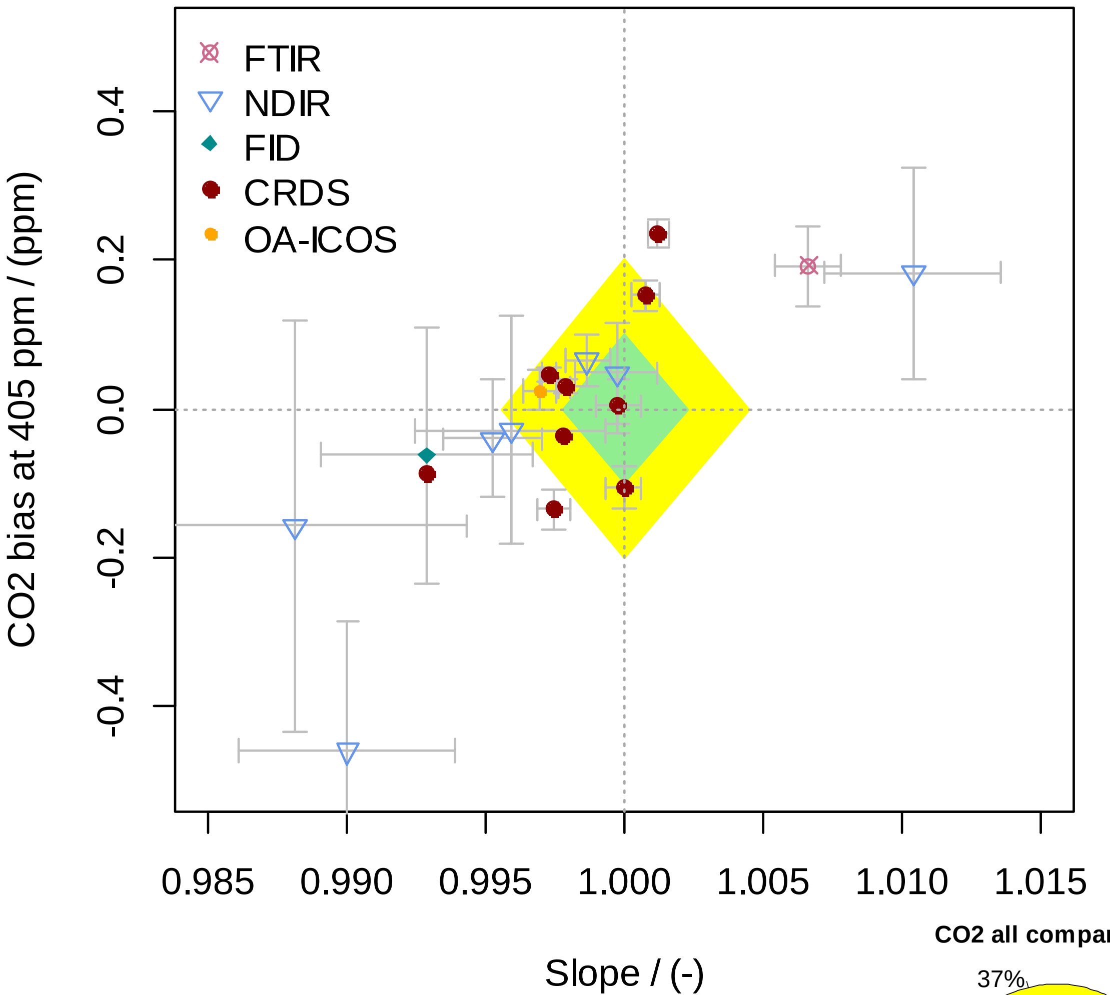
CH_4: Relationship performance – measurement technique



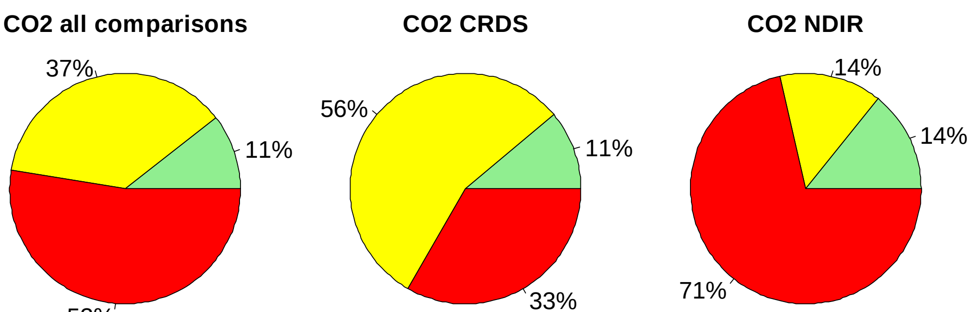
- WCC-Empa audits from 2005 to 2014 were analyzed.
- Two techniques are widely used: GC/FID since the 1970's, CRDS since 2009.
- The analysis of the WCC-Empa audit results clearly demonstrates the progress that has been made with regard to instrument performance.
- Uncertainties are much smaller for CRDS instruments compared to GC/FID due to better repeatability (short term) but also due to long-term stability of the CRDS instruments.



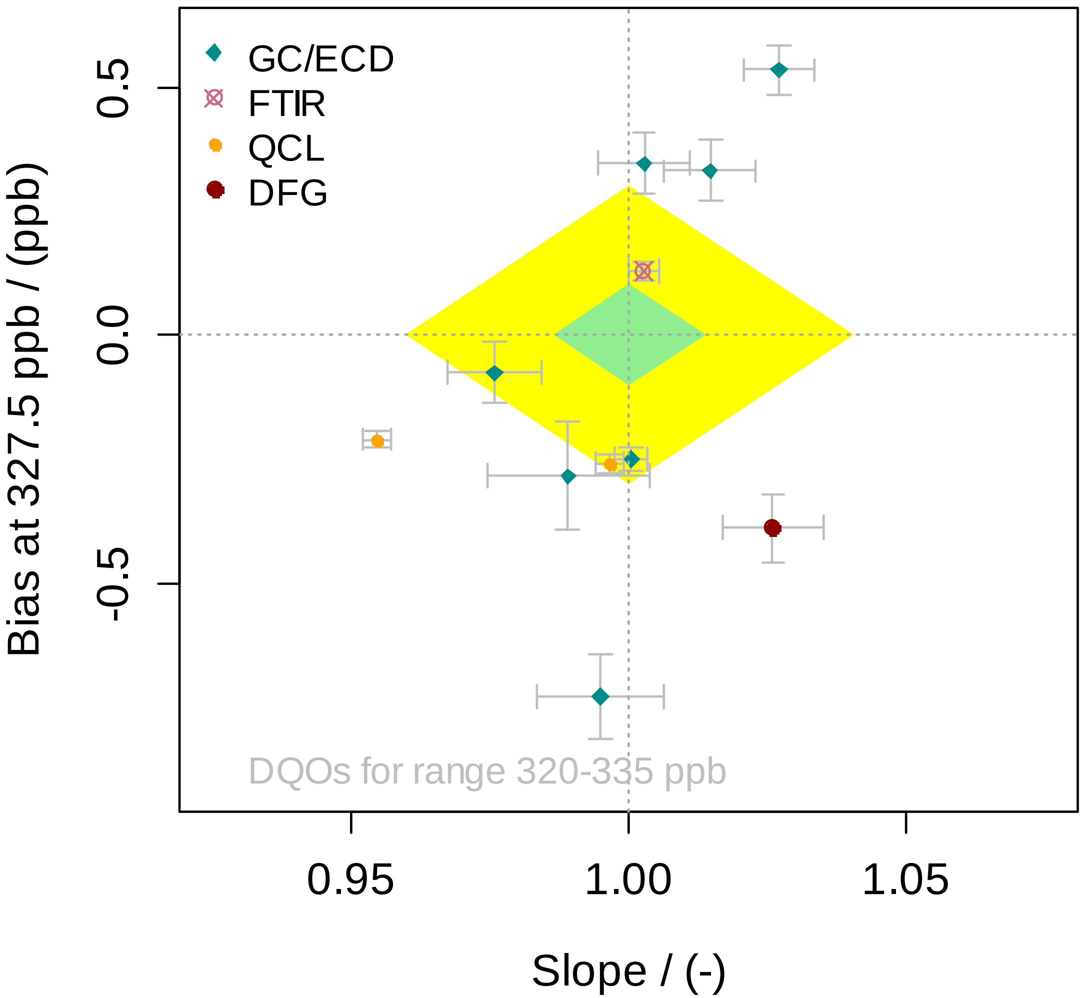
CO_2: Relationship performance – measurement technique



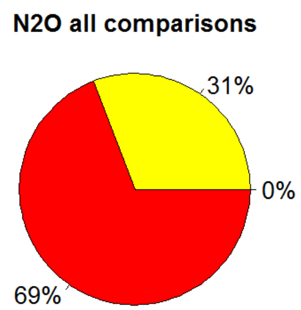
- Audits from 2010 to 2014.
- Two methods are widely used: NDIR since the 1950's, CRDS since 2009.
- The analysis of the WCC-Empa audit results clearly demonstrates the progress that has been made with regard to instrument performance; however, compatibility goal of 0.1 ppm is challenging.
- Uncertainties are much smaller for CRDS instruments compared to NDIR due to better repeatability (short term) but also due to long-term stability of the CRDS instruments.



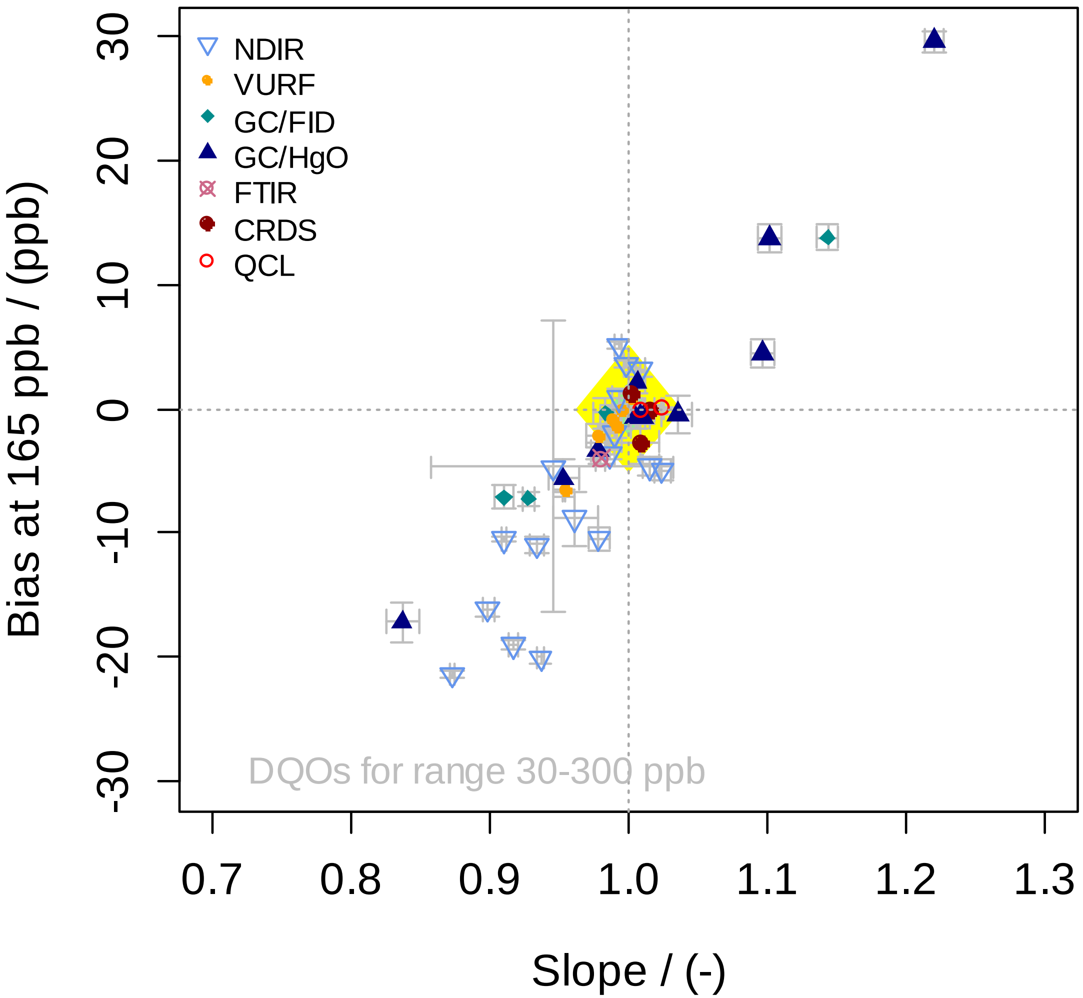
N_2O: Relationship performance – measurement technique



- ... Compatibility goals are very difficult to meet.
- Not enough data from newer techniques to draw conclusions, but large potential of spectroscopic methods (e.g. QCL, FTIR).

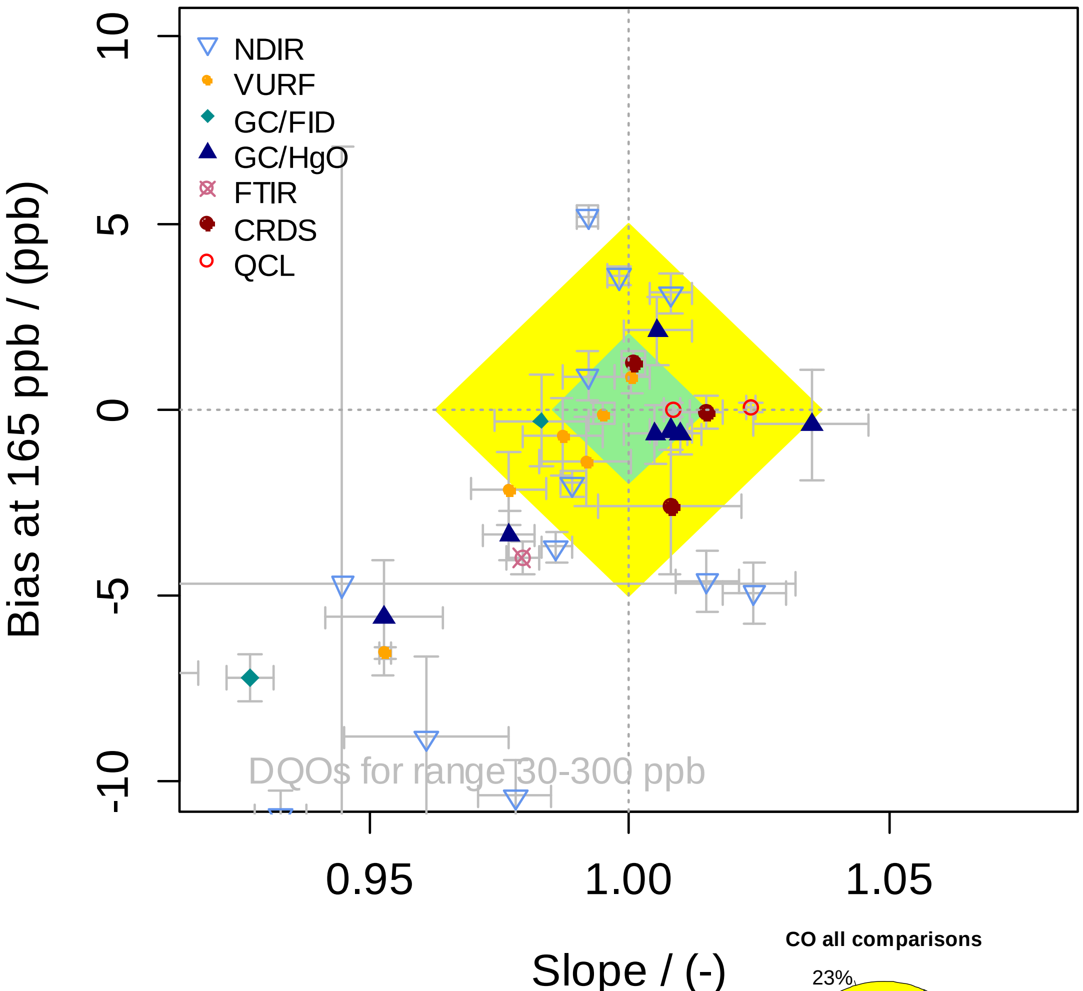


CO: Relationship performance – measurement technique

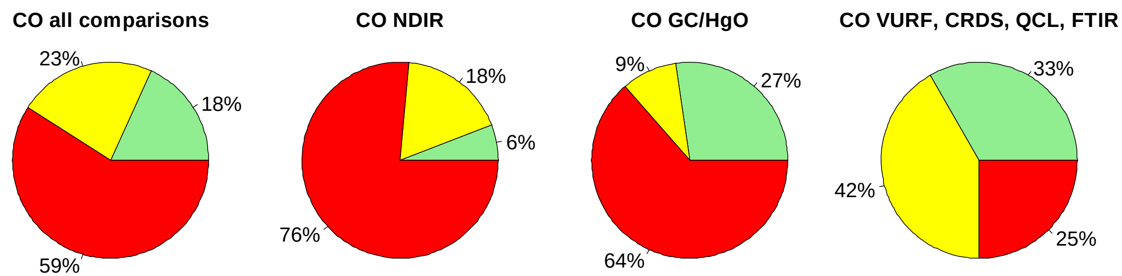


- Audits from 2005 to 2014.
- Two techniques are widely used: NDIR and GC/HgO.
- Many of the audited NDIR and GC/HgO instruments were not able to meet the extended compatibility goal.
- Other techniques, Vacuum UV Resonance Fluorescence (VURF) and GC/FID with methanizer are occasionally used.
- Recently, spectroscopic techniques (CRDS, QCL, FTIR) became available and are increasingly used.

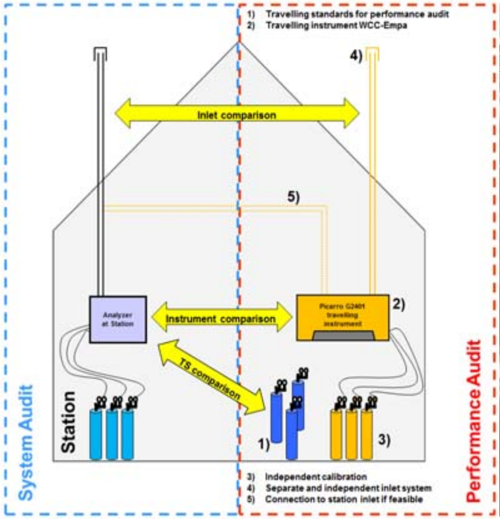
CO: Relationship performance – measurement technique



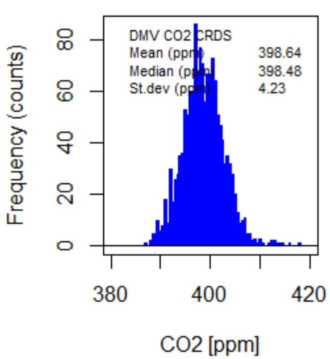
- … in principle, measurements that meet (extended) compatibility goal are possible with NDIR and GC/HgO; however, it is often not achieved due to a number of issues with these techniques.
- More recent techniques perform better: Most VURF and all CRDS and QCL instruments met extended compatibility goals.
- Number of comparisons for CRDS and QCL instruments are still too small for final conclusions, but the potential of these techniques is evident.

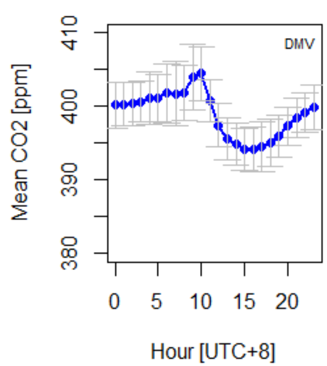


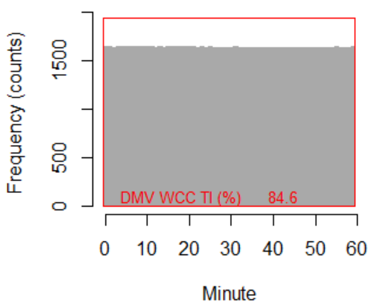
Parallel Measurements: CO_2 @ Danum Valley

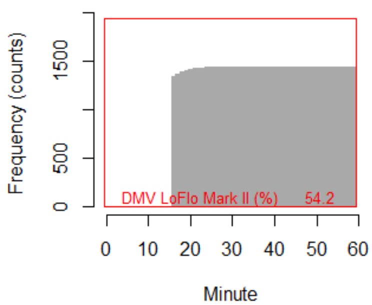


- Station instrument: LoFlo Mark II
- Travelling instrument: Picarro G2401 without sample air drying
- From 2013-12-06 to 2014-02-25
- Two independent inlet lines to same sampling location on top of 100 m tower

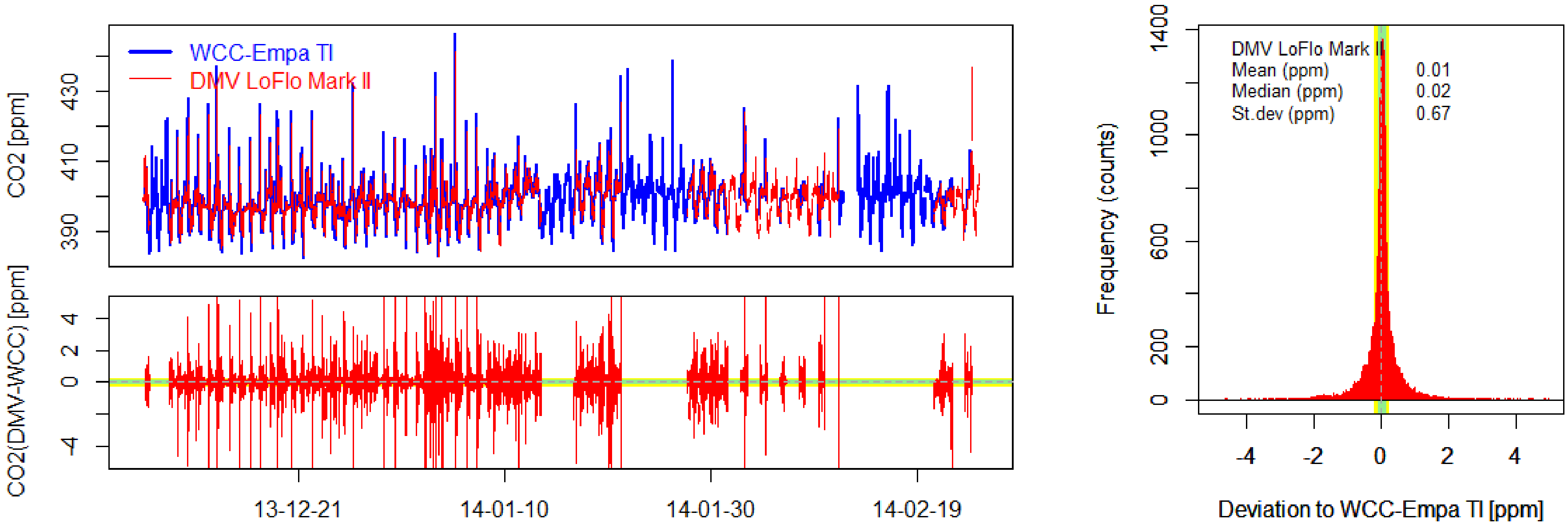






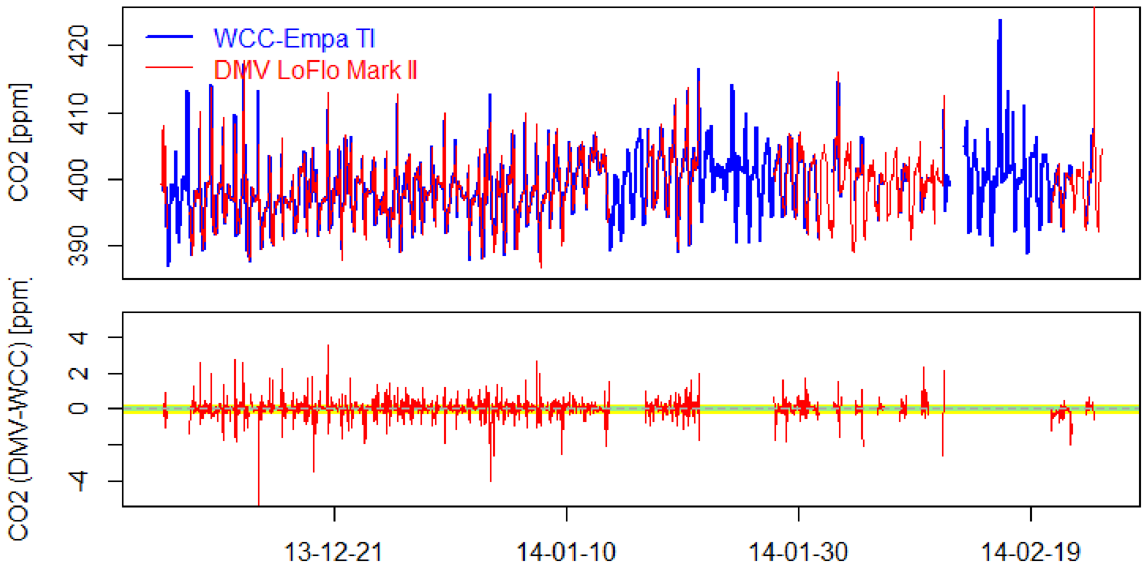


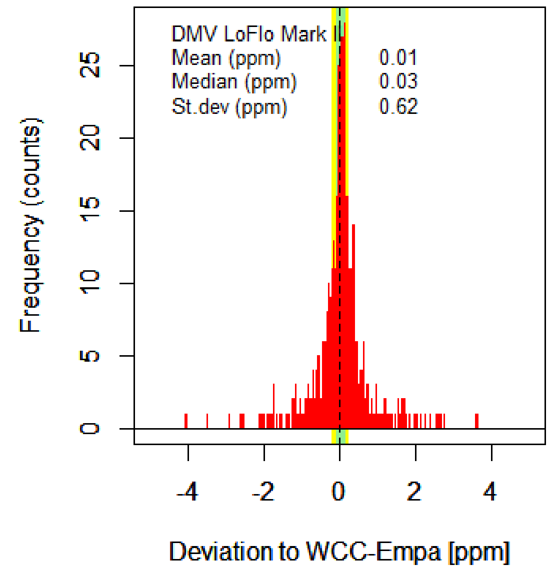
Example: CO_2, LoFlo Mark II @ DMV 1-min



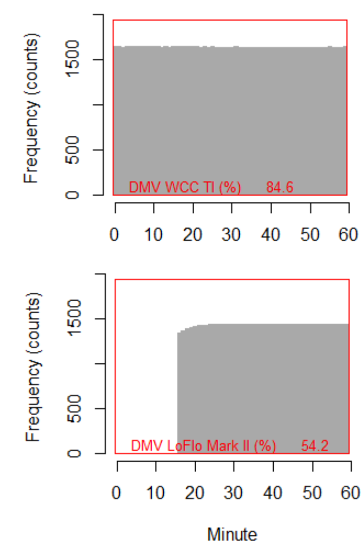
- No offset but large variability of the difference between instruments.
- Relatively high temporal variation, timing (residence time, clock adjustment etc.) and instrument response time is critical.

Example: CO_2, LoFlo Mark II @ DMV 1-h

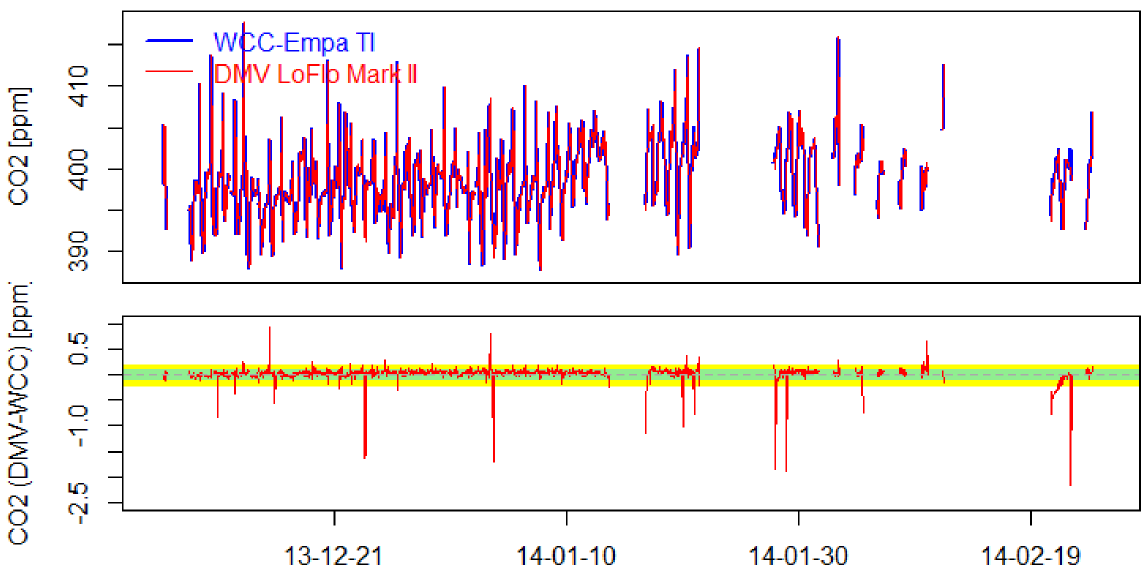


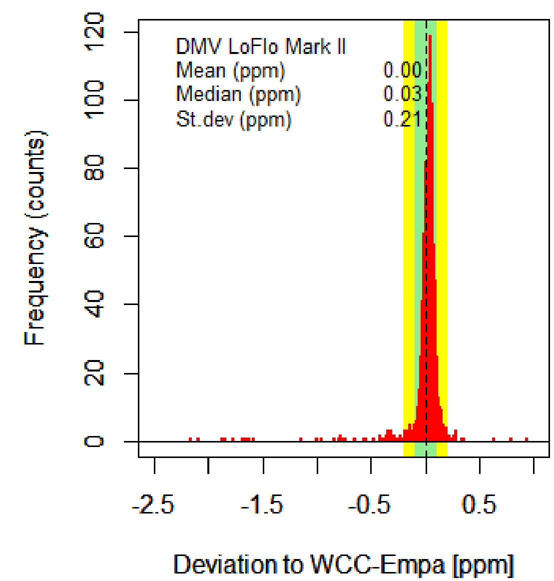


- 1-h averages are usually submitted to the WDCs.
- Data coverage is important because of relatively large CO_2 variability.
- Improvement would be expected when averaged; however, this is not observed here.

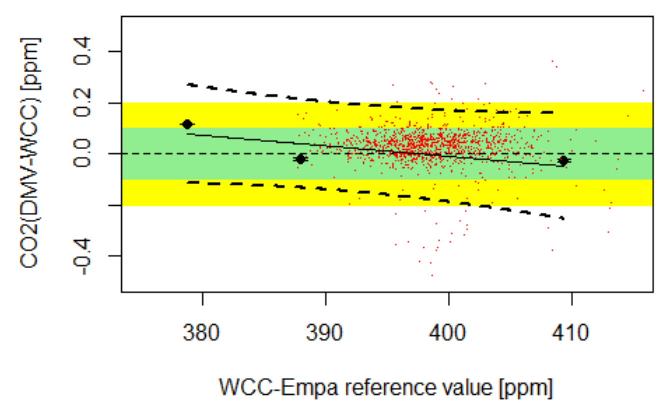


Example: CO_2 @ DMV 1-h concurrent data availability





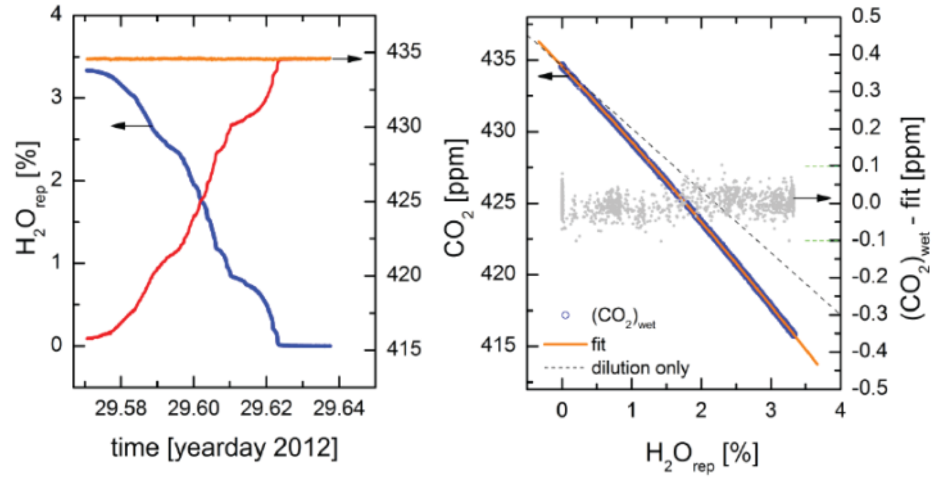
- If only 1-min TI with matching LoFlow data are considered for hourly averages, the agreement becomes much better.
- Data coverage is a important aspect, especially for sites with high ambient variability.



Sample drying …

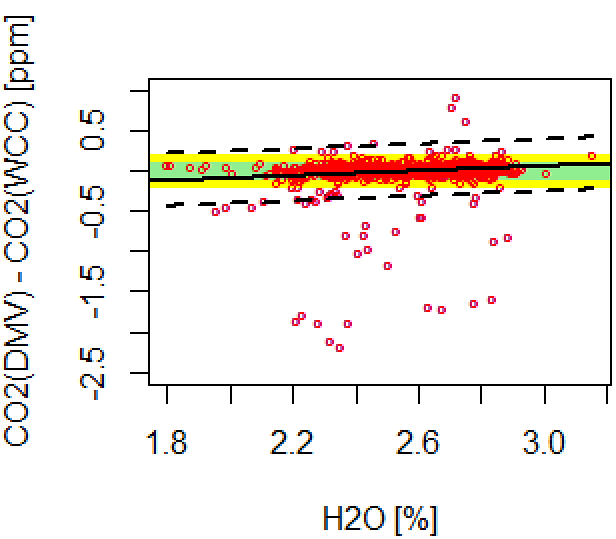
GGMT recommendations:

- 2009: No recommendation, first CRDS instruments commercially available.
- 2011: …we do not recommend correcting CO_2 mole fraction … For CRDS instruments, this recommendation is under review and may be revised in future.
- 2013/2015: Water vapor must either be removed from the sample gas stream, or its influence on the mole fraction determination must be carefully quantified.



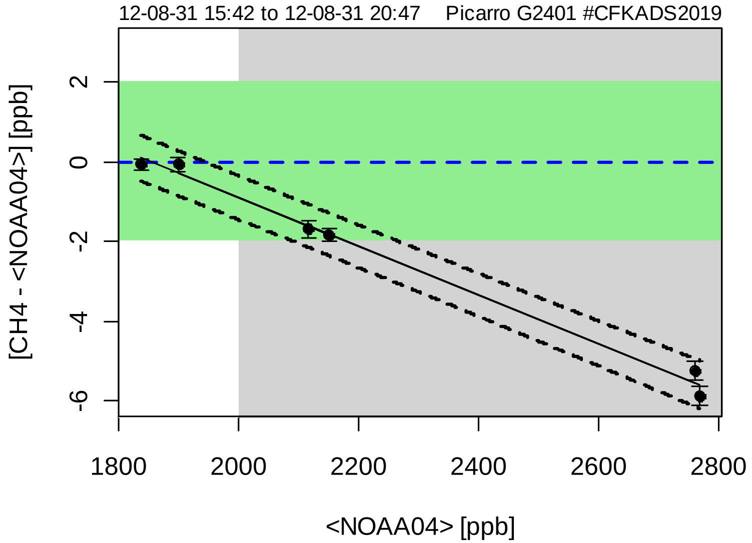
WCC-Empa results from Rella et al. (Atmos. Meas. Tech., 6, 837–860, 2013)

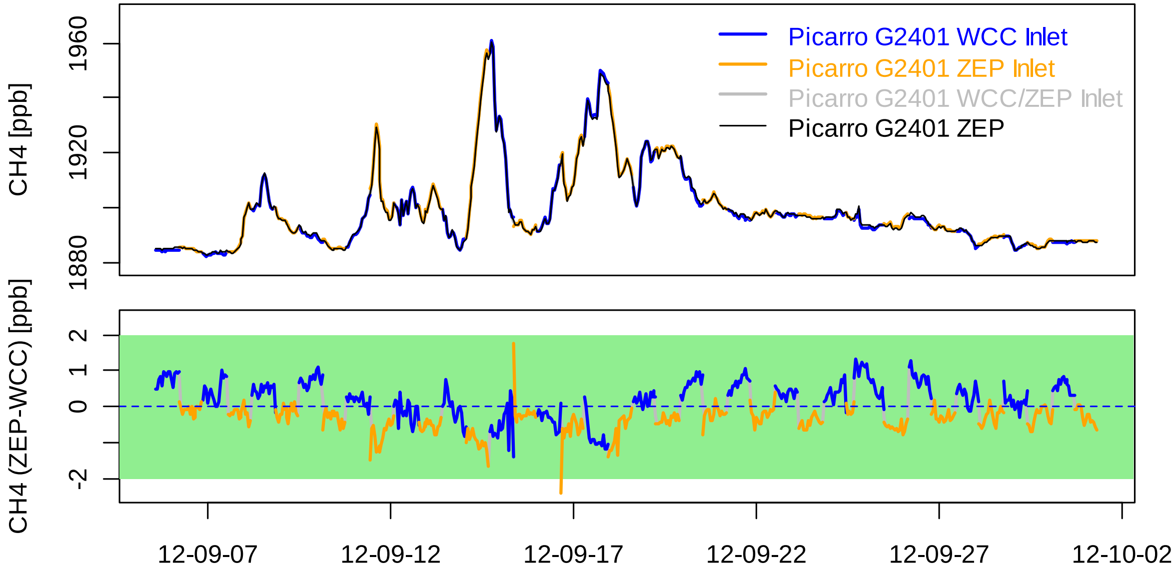
- Results from DMV now show that correction is possible even for conditions with very high humidity.

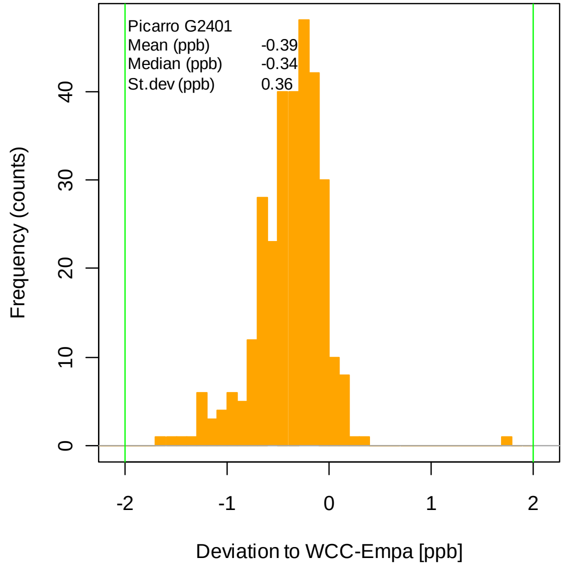


Parallel Measurements: CH_4 @ Zeppelin Mt.

- CH_4 audit Zeppelin 2012 (NILU).
- Small negative bias was confirmed by parallel measurements using the same (ZEP) inlet.

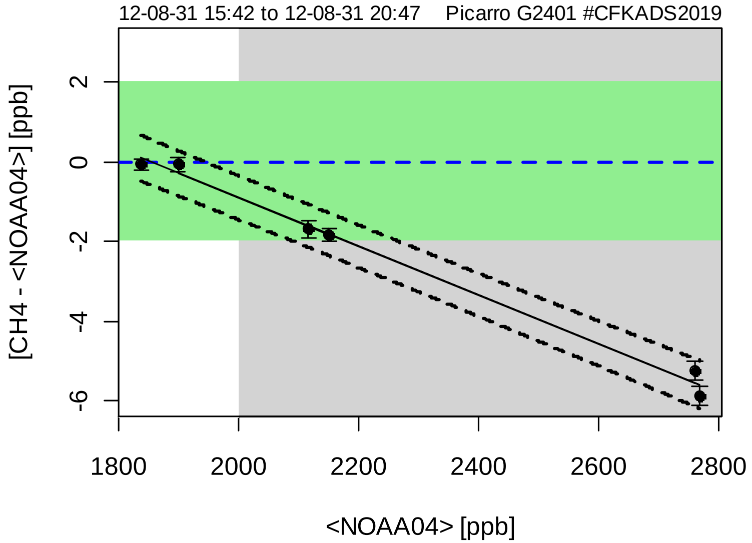


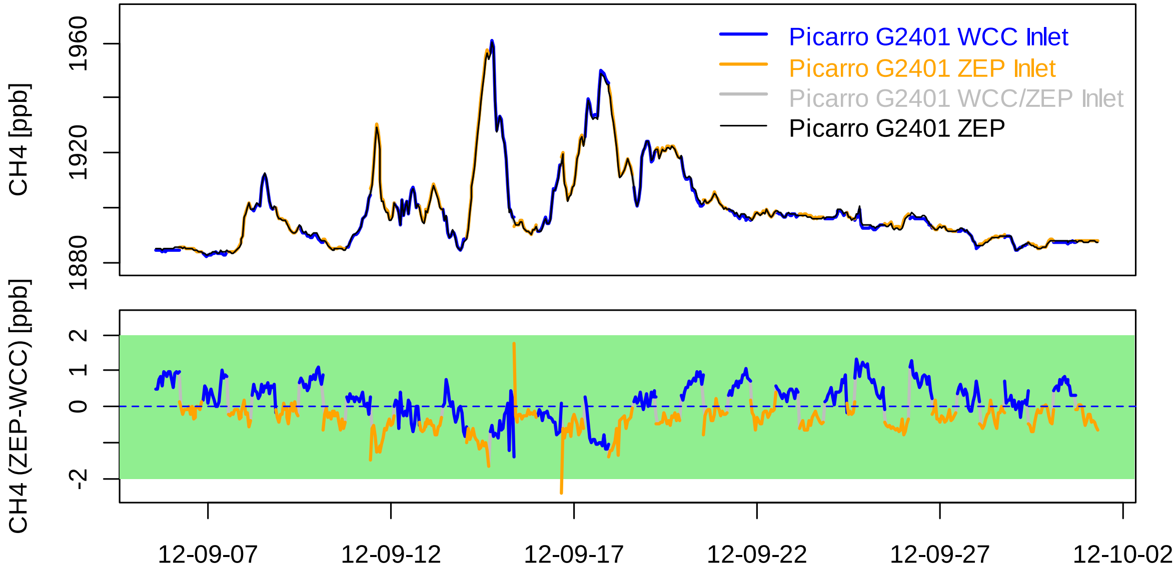


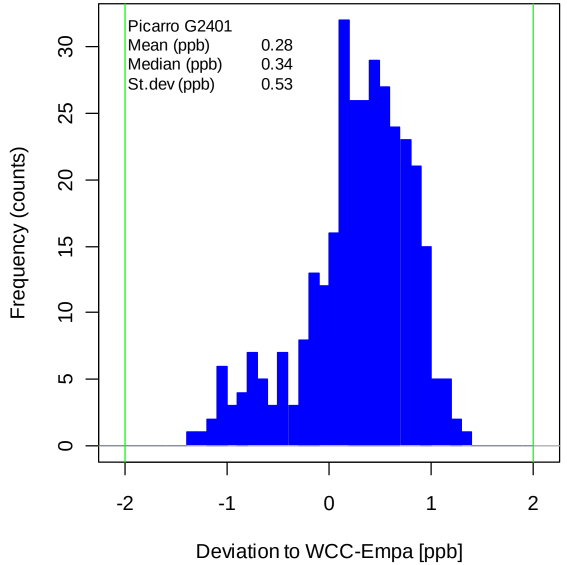


Parallel Measurements: CH_4 @ Zeppelin Mt.

- CH_4 audit Zeppelin 2012 (NILU).
- Small negative bias was confirmed by parallel measurements using the same (ZEP) inlet.
- However, a small positive bias of ZEP was observed with travelling instrument connected to the separate inlet.
- Added value: Indication for a small leak in the ZEP inlet. GC/ECD system with CH_4/Ar carrier gas is emitting high CH_4 into laboratory air.

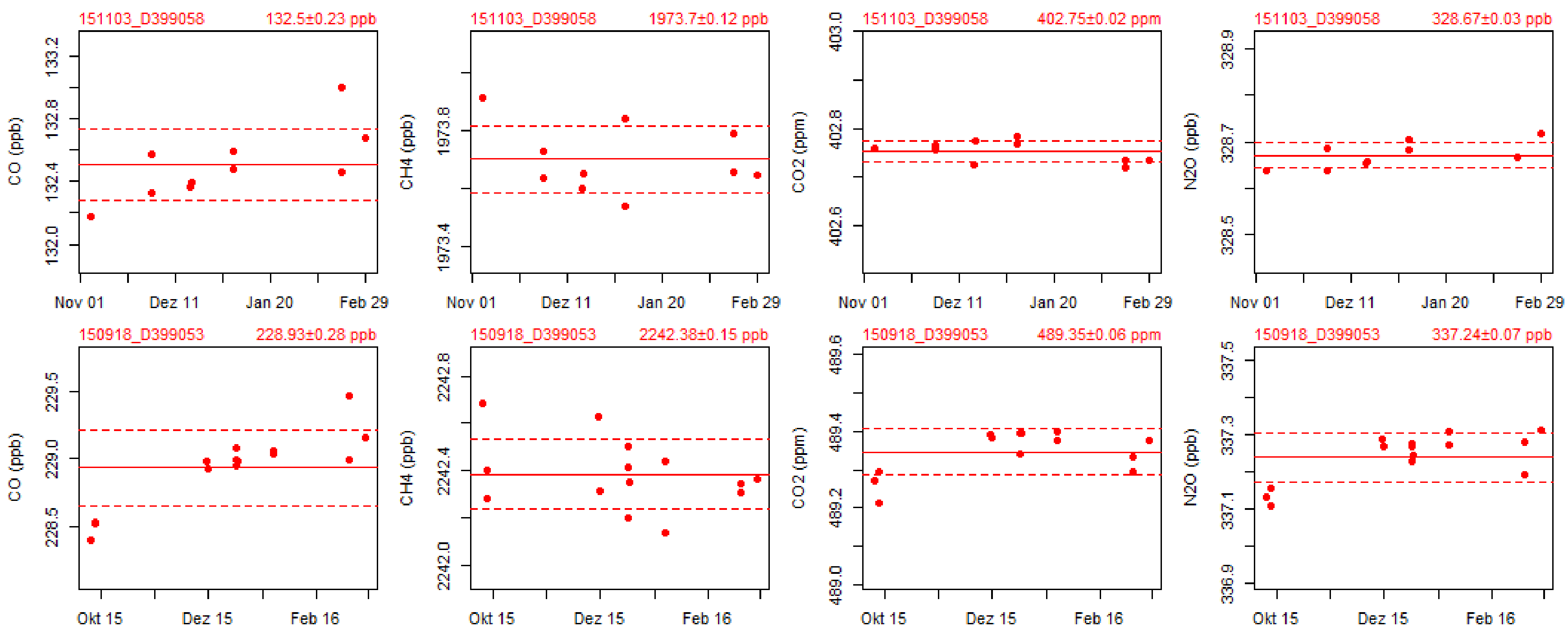






Our role in the HIGHGAS project

- Assessment of the comparability of traceable reference standards to existing standards and scales used by the atmospheric monitoring community.
- For this purpose we prepared a set of air standards with calibration against the WMO/GAW reference scales and monitored for stability.
- In the next few months these standards will be distributed to HIGHGAS partners for comparison against their gravimetrically prepared standards.



Importance of isotopic composition

Example:

d13C-CO_2: natural abundance of $^{13}CO_2$ 1.1% - 4.4 ppm at 400 ppm CO_2
differences in d$^{13}CO_2$ of 25 ‰ translates into 0.11 ppm
equivalent to the WMO compatibility goal for the Northern hemisphere!

	natural abundance	mixing ratio	Deviation from natural abundance 25‰	10‰	WMO compatibility goals
$^{13}CO_2$	0.011	4.4 ppm	0.11	0.04	0.1 ppm
$CO^{18}O$	0.0039	1.6 ppm	0.04	0.02	0.1 ppm
$^{13}CH_4$	0.011	22 ppb	0.55	0.22	2 ppb
CH_3D	0.00062	1.2 ppb	0.03	0.01	2 ppb
$^{15}N^{14}NO$	0.0036	1.2 ppb	0.03	0.01	0.1 ppb
$N_2{}^{18}O$	0.002	0.66 ppb	0.02	0.01	0.1 ppb
^{13}CO	0.011	1.1 ppb	0.03	0.01	2 ppb
$C^{18}O$	0.002	0.20 ppb	0.01	0.002	2 ppb

Impact of chance in isotopic composition > 25 % WMO compatibility goal.

→ Isotope analysis desirable in Empa reference standards and standards prepared by EMRP partners!

all Empa standards (6 x)
one standard per partner and compound

→ Additional C2 cylinder with isotope analysis to support work in WP3

NOAA calibration scales

UNITED STATES DEPARTMENT OF COMMERCE
National Oceanic and Atmospheric Administration
Earth System Research Laboratory
Global Monitoring Division
325 Broadway - David Skaggs Research Center
Boulder, CO 80305-3328

Certificate of Analysis

NOAA Global Monitoring Division (GMD)

Certificate Number: CB11485-A
Issue Date: 20 January 2016
Material: Air, compressed, in an aluminum gas cylinder, nominal pressure 13.6 MPa (2000 psi)
Intended Use: For the calibration of instruments for determining the mole fraction of trace gases in air. Experience has shown that high flow applications may lead to changes in CO_2 mole fraction. For high precision measurement, flow should be less than 0.5 liters per min.
Use and Storage: Cylinders should be used under normal laboratory conditions (room temperature). For storage, we recommend -30 to 40 deg C.
Period of Analysis: December 2015
Prepared by: Thomas Mefford

Cylinder ID: CB11485

Results are based on analysis performed by the WMO/GAW Central Calibration Laboratories (CCL) located at the NOAA Global Monitoring Division (GMD). WMO/GAW mole fraction scales are developed and maintained by GMD in their role as CCL. Results are traceable to the SI unit "amount of substance fraction". Equipment used to develop mole fraction scales and establish traceability to the SI are traceable to national standards for mass, temperature, pressure, and amount of substance fraction (O_2 in N_2). For more information on calibration scales and analysis methods, see http://www.esrl.noaa.gov/gmd/ccl. For isotopic ratios or other informational values, if applicable, see http://www.esrl.noaa.gov/gmd/ccl/refgas.html/.

Results

	Mole Fraction[1]	Reproducibility[2]	Expanded Uncertainty[3]	Unit	Method	Calibration Scale
CO_2	394.30	0.06	0.21	$\mu mol\ mol^{-1}$	NDIR	WMO-CO_2-X2007
CH_4	1844.8	1.0	3.5	$nmol\ mol^{-1}$	GC-FID	WMO-CH_4-X2004A
CO	108.3	0.8	1.1	$nmol\ mol^{-1}$	OA-ICOS	WMO-CO-X2014[4]
N_2O	328.46	0.22	0.5	$nmol\ mol^{-1}$	GC-ECD	WMO-N_2O-X2006A
SF_6	8.74	0.03	0.06	$pmol\ mol^{-1}$	GC-ECD	WMO-SF_6-X2014

[1] mole fraction in dry air, expressed on a WMO/GAW mole fraction calibration scale ($\mu mol\ mol^{-1}$ = ppm, $nmol\ mol^{-1}$ = ppb, $pmol\ mol^{-1}$ = ppt).
[2] expected long-term variation of analysis results assuming no cylinder drift (95% confidence level)
[3] total uncertainty, estimated with coverage factor k=2, (~95% confidence level). Total uncertainty includes uncertainties associated with preparation and analysis of primary standards, as well as scale propagation. Note that we explicitly express the results with the number of significant figures corresponding to the number of significant figures in the reproducibility estimate. This is deliberate, as it provides important information to WMO/GAW end users.
[4] CO mole fractions are currently underestimated by as much as 2 ppb due to known drift in secondary standards. A method to reliably determine drift rates of secondary standards is under development. An update will be announced at a later date.

- Current NOAA calibration scales are:
  CO_2: WMO-CO_2-X2007
  CH_4: WMO-CH_4-X2004A
  CO: WMO-CO-X2014A
  N_2O: WMO-N_2O-X2006A
- Recent changes were made for CH_4 (from X2004 to X2004A), which changed mole fractions in the order of about 0.5 $nmolmol^{-1}$.
- Recent changes were also made for CO (from X2004 to X2014, then to X2014A), which changed values up to >2 $nmolmol^{-1}$ depending on the mole fraction.
- For example, CB11485 (purchased in 2015) changed from 108.3 (X2014) to 110.88 (X2014A) $nmolmol^{-1}$. This change is larger than the WMO/GAW compatibility goal of 2 $nmolmol^{-1}$.
- The reason for these changes is also drift in standards, which has been difficult to quantify.
- There is a clear need for better long-term stability of CO in air standards at ambient mole fractions.

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CO CALIBRATION SUMMARY FOR TANK # CB11485

CO mixing ratios shown are on the WMO CO_X2014A scale.

Scale transfer uncertainties as measured by the reproducibility of calibrations are estimated at 2.6 ppb for measurements by GC (instrument codes R2 and R7) and 0.8 ppb for measurements by VURF and Off-axis ICOS (instrument codes V1, V2, and LGR2). These are 95% confidence intervals and are valid for measurements of mole fractions between 50 – 300 ppb by GC and 30 – 500 ppb by VURF and Off-axis ICOS.

Filling Code **A**

```
    Date      Loc  Inst Pressure   Value    S.D.        Num    Avg     Sdev
--------------------------------------------------------------------------------
 2015-09-23  BLD LGR2     2000    110.61   0.05 .
 2015-10-06  BLD LGR2     2000    111.02   0.05 .
 2015-10-13  BLD LGR2     1900    111.02   0.03 .
                                                        3   110.88     0.23
```

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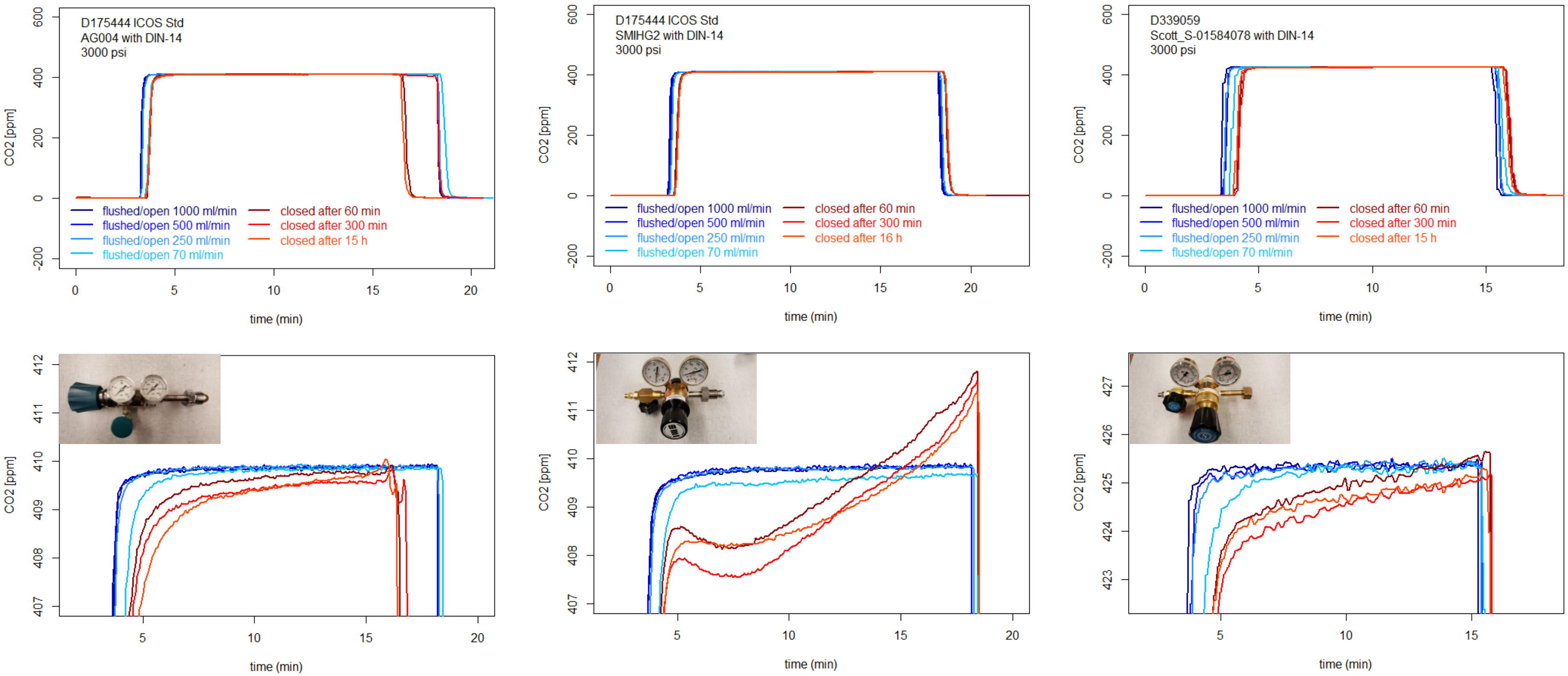
- The reason for these changes is also drift in standards, which has been difficult to quantify.
- There is a clear need for better long-term stability of CO in air standards at ambient mole fractions.

Other things are also important: regulator performance



- Airgas, recommended by NOAA

- Scott Marrin
- Initially purchased for the HIGHGAS project
- Bad performance for CO_2

- Scott Specialty Gases
- Purchased for the HIGHGAS project
- Similar performance as Airgas for CO_2

Conclusions

- Audit approach with parallel measurements AND standard comparisons is optimal.
- Audit results confirm progress in the development of new instruments.
- Very obvious is the improvement for CH_4.
- For CO_2, the WMO/GAW compatibility goals (0.1 resp. 0.2 ppm) are very difficult to achieve, but a clear improvement is seen when CRDS instruments are used.
- The result might look better if a smaller range is considered (especially for CO_2), since stations using NDIR are often focusing on a very narrow mole fraction range.
- CRDS water vapor corrections work also at very high humidity.
- N_2O remains challenging; compatibility goals are very difficult to meet.
- The CO compatibility goals (2 resp. 5 ppb) are very difficult to achieve, but a clear improvement is seen when VURF, CRDS, QCL and FTIR instruments are used.
- Due to clear advantages of the new techniques/instruments, the 'traditional' methods (CO_2 NIDIR, CO GC/HgO and NDIR, CH_4 GC/FID) will disappear at many stations.
- A further advantage of the newer (spectroscopic) techniques is the much larger data coverage (continuous, less calibration required).

Acknowledgments

- Financial support by MeteoSwiss
- Mohd Firdaus bin Jahaya and Marcel Van der Schoot for support during the audit at Danum Valley
- Rainer Steinbrecher (WCC-N_2O)
- Staff at various GAW stations for their support