

Berichte aus den Zentralen Analytischen Laboren Jena und Heidelberg



Bundesministerium
für Bildung
und Forschung

Aufbau des Flask- und Kalibrierlabors (FCL) Hauptphase 2014/15

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Spurengasmessungen - LGR



LosGatos Research Inc. N₂O/CO Analyzer

- für Tank- UND Flask-Analysen
- => hohe Samplingrate (1 Hz), Wasserdampf-Korrektur
- Geringer Zellendruck (110 mbar) => moderater Probenkonsum
- 1-4000 ppb Messbereich
- 2 Geräte zum Test verfügbar, =>parallel Einbindung in Messablauf mgl.
- **Lieferung August 2015**

		N ₂ O [ppb]	CO [ppb]
Flasks	Rauschen während Messung (60s)	0.09	(0.04)
	Wiederholbarkeit der Messungen (zu versch. Stunden)	0.02	0.03
Targettanks	Rauschen während Messung (3600s)	0.02	0.08
	Wiederholbarkeit der Messungen (an versch. Tagen)	0.03	0.03
GC-System	Rauschen während Messung (24h)	0.11	0.8
	Wiederholbarkeit der Messungen (an versch. Tagen)	0.07	0.4

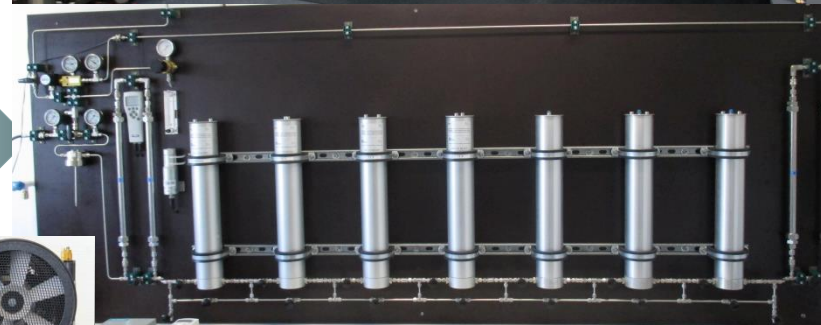
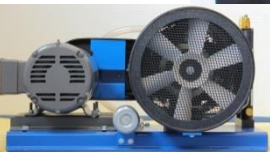
GasStandardHerstellung

Inbetriebnahme 2014
TÜV-Zulassung 2015



$p \sim 5 \cdot 10^{-7}$ mbar
 $T = 100$ °C
 $t < 24$ h

Außen- + Nullluft



Zumischung	CH ₄	CO ₂	N ₂ O	CO
mittl. Abweichung vom bestellten Soll	-0.14%	0.02%	-0.10%	-0.07%

Derzeit Kompromiss aus Genauigkeit der Zusammensetzung und Bearbeitungszeit gesucht.

Vorbereitung Druckgasflaschen

Evakuieren unter Ausheizen,
Restgasanalyse (Quadrupol-MS)

Befüllung mit Außenluft

Betrieb des Kompressors in Bürogebäude[!],
Luftanalyse + Wetterbeob.,
Abreicherung (optional)

Einstellen gewünschter Spurengaskonz.

Spiking-Anlage
(An-/Abreicherung mgl.)

Messung / kalibr. Skalenübertrag

Picarro G2301, GC-System, (ecotech FTIR)

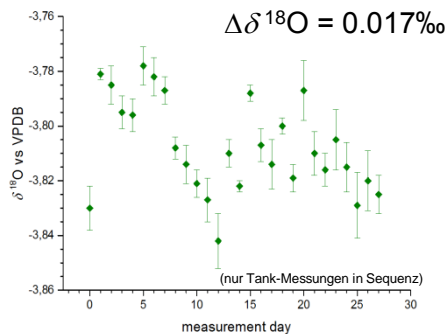
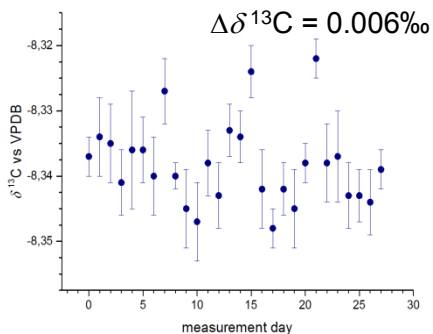
Bilanz bisher:

10 Fl. (2x5er Set) ausgeliefert
15 Fl. zeitnahe Fertigstellung
15 Fl. Spiking
26 Fl. Konditionierung
2 Fl. Re-Kalibration/Neufüllung

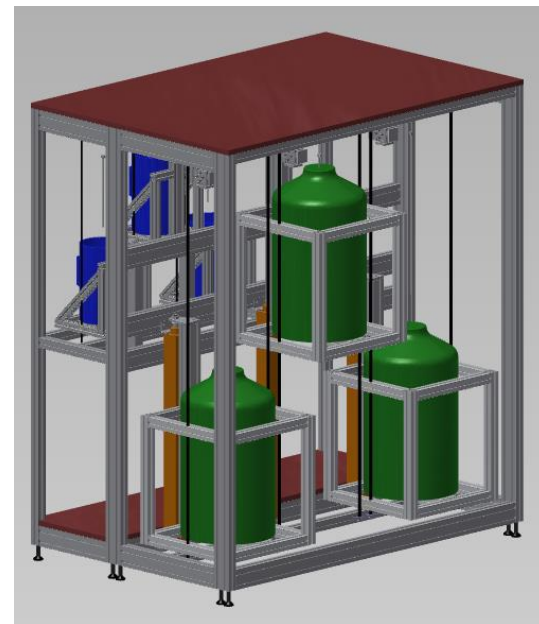
CO₂ Isotopie



- Testläufe für automatisierten Routinebetrieb
- kompletter Betrieb automatisiert
- Dateneingabe nur per Barcode Scanner
- Langzeit-Messwertstabilität muss noch verbessert werden
- Erweiterung der Cryo-Fallen zur Stabilisierung und der Erhöhung des Probendurchsatzes



Messtabilität einer pro Sequenz 1x mitgemessenen Luftprobe



O₂/N₂-Analytik / Flaskkonditionierung



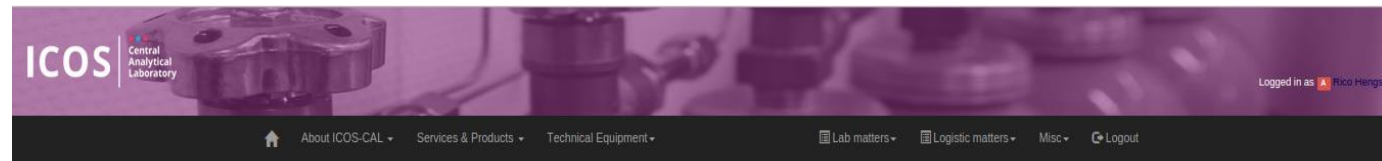
- Massenspektrometer für O₂/N₂ Analytik installiert
- derzeit wird Probeneinlass aufgebaut



- Flaskkonditionierofen fertiggestellt
- wird derzeit zur Befüllung von Test-Flasks genutzt

Optische Methan-Isotopen Analyse von Kalibriertanks soll in Projektskizze für **zukünftiges Projekt** aufgenommen werden

Benutzerzugang und Datenbank



Webapplikation ICOS-CAL

Idean-Design wurde umgesetzt.
Statische Seiten (public) mit Links und
Infos zu den ICOS-CF.

Alle übrigen Seiten und
Funktionalitäten sind nur mit login
nutzbar.

Weiterhin sind versch. Nutzergruppen
definiert. Die Funktionalitäten sind
nutzbar, wenn User den
entsprechenden Nutzergruppen
angehört.

ICOS - CENTRAL ANALYTICAL LABORATORIES

About ICOS

The Integrated Carbon Observation System (ICOS) is a new research infrastructure to decipher the greenhouse gas balance. ICOS provides the long-term observations required to understand the present state and predict future behaviour of climate, the global carbon cycle and greenhouse gases emissions.

Quick facts

- ICOS is on the roadmap of the European Strategy Forum on Research Infrastructures.
- ICOS is in its Preparatory phase (2008-2013), funded by the European Commission under FP7.
- ICOS is a long term (20+ years) observatory.
- 17 European research laboratories participate to the Preparatory phase.
- 17 countries have national focal points to represent their scientific interests.

ICOS mission statement

- To provide the long-term observations required to understand the present state and predict future behavior of the global carbon cycle and greenhouse gas emissions.
- To monitor and assess the effectiveness of carbon sequestration and/or greenhouse gases emission reduction activities on global atmospheric composition levels, including attribution of sources and sinks by region and sector.

ICOS tracks carbon fluxes in Europe and adjacent regions by monitoring the ecosystems, the atmosphere and the oceans through integrated networks.

What is ICOS-CAL?

The new research infrastructure ICOS tracks carbon fluxes in Europe and adjacent regions by monitoring the ecosystems, the atmosphere and the oceans through integrated networks.

The ICOS - Central Analytical Laboratories (CAL) which are located in Germany (Jena and Heidelberg) ensures the accuracy of observational data, thorough quality control and routine testing of air sampling material. The CAL provide reference gases for calibration of in-situ measurements performed at the continuous monitoring stations. CAL also analyze air samples collected at the monitoring stations.

The visit of the following links will give you more information about the two Central Analytical Laboratories:

- the FCL  in Jena and
- the CRL  in Heidelberg.

ICOS-CAL-DB

The ICOS-CAL-Database is the web application of a data retrieval and long-term archive system that

- storage the uncorrected data of the analyzed air samples
- provides the calibrated & corrected data of the analyzed air samples
- supports the shipping logistic of flask between the CAL and the atmospheric stations
- provides a inhouse tracking and a tasks control feature for flasks

ICOS-CAL

- FCL Jena
- CRL Heidelberg

ICOS CENTRAL FACILITIES

- The Carbon Portal
- The Atmospheric Thematic Centre
- The Ecosystem Thematic Centre
- The Ocean Thematic Centre

MISC

- Imprint
- Terms & Conditions
- Acknowledgment

Funktionalitäten (Auswahl):

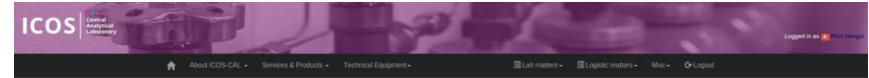
- Registrierung von Tanks, Flasks, Proben, Probennahmen, ...
- Anlegen von Bearbeitungsschritten in der in-house Logistikkette, Zusammenstellen von Logistikprofilen für die Container
- Tracking Container incl. Versand (zwischen beiden CAL's und von CAL zu STATION) von Tanks, Flasks, NaOH-Container

Aktuelle Arbeiten:

- Test Datenaustausch FCL/CRL
- Autom. Preprocessing der Daten Picarro, GC
- Launch der Webapplikation ca. Juni 2015

Perspektive

- Processing Massenspektrometer
- Aufbau analytischer Methoden



SHIPPING #4

Shipping event statuses

Overview (1) Packed (2) Sent (0) Received (4) Abandoned

- The containers are packed into boxes/batch.
- The batch was sent.
- The batch was received.
- The shipping is finished and the shipping is abandoned.

Go to the next shipping step (receiving)

Shipping content

Test Batch 4

The box contains following containers in terms of the current shipping.

1	
2	
3	

Shipping comment

Shipping event => ICOS-CAL-Container-Shipping-Creator-packing
Commentator => Julie Teupers@Stationmember Stat2 | 2014-03-22 07:23:22

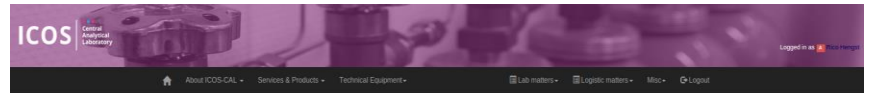
Test comment

Test comment

Shipping event => ICOS-CAL-Container-Shipping-Creator-sending
Commentator => Julie Teupers@Stationmember Stat2 | 2014-03-22 08:03:22

Test comment

Test comment



CREATE A NEW SHIPPING/PACKING EVENT

Form

Sender:

Recipient:

Batch:

Box:

Container:

Comment:

Buttons:

Explanation

A shipping means a transport of objects from a location to another location.
A shipping consists of following objects:

- A shipping consists of at least one batch.
- Each batch consists of at least one box.
- Each box consists of at least one container.

In terms of the locations take notice:

- A sending location can be a station, the recipient location must be a CAL.
- A sending location can be a CAL, the recipient location can be a station.
- A sending location can be a CAL, the recipient location can be another CAL.

A shipping process consists of four steps:

1. Packing
2. Sending
3. Receiving
4. Abandoning



Zusammenfassung

- Alle Arbeitspunkte fortgeschritten
- GC-system einsatzbereit (Flask-/Tankanalysen)
- Flaskkonditionierung einsatzbereit
- GasStandardHerstellung einsatzbereit, angelaufen
=>Optimierung
- Spektroskopische Verfahren Testbetrieb (FTIR verzögert)
- CO₂ Isotopie - Routine Testmessungen und Skalenabgleich mit BGC IsoLab angelaufen
- O₂/N₂ Massenspektrometrie – Probeneinlass wird aufgebaut

Quantifying fossil fuels CO₂ in ICOS - a progress report of the Central Radiocarbon Laboratory

Samuel Hammer, Johannes Lux, Sabine Kühr, Eva Gier, Stefan Lanz,
Manuel Roos, Sanam Vardag, Bernd Kromer and Ingeborg Levin
Institut für Umweltphysik, Heidelberg University

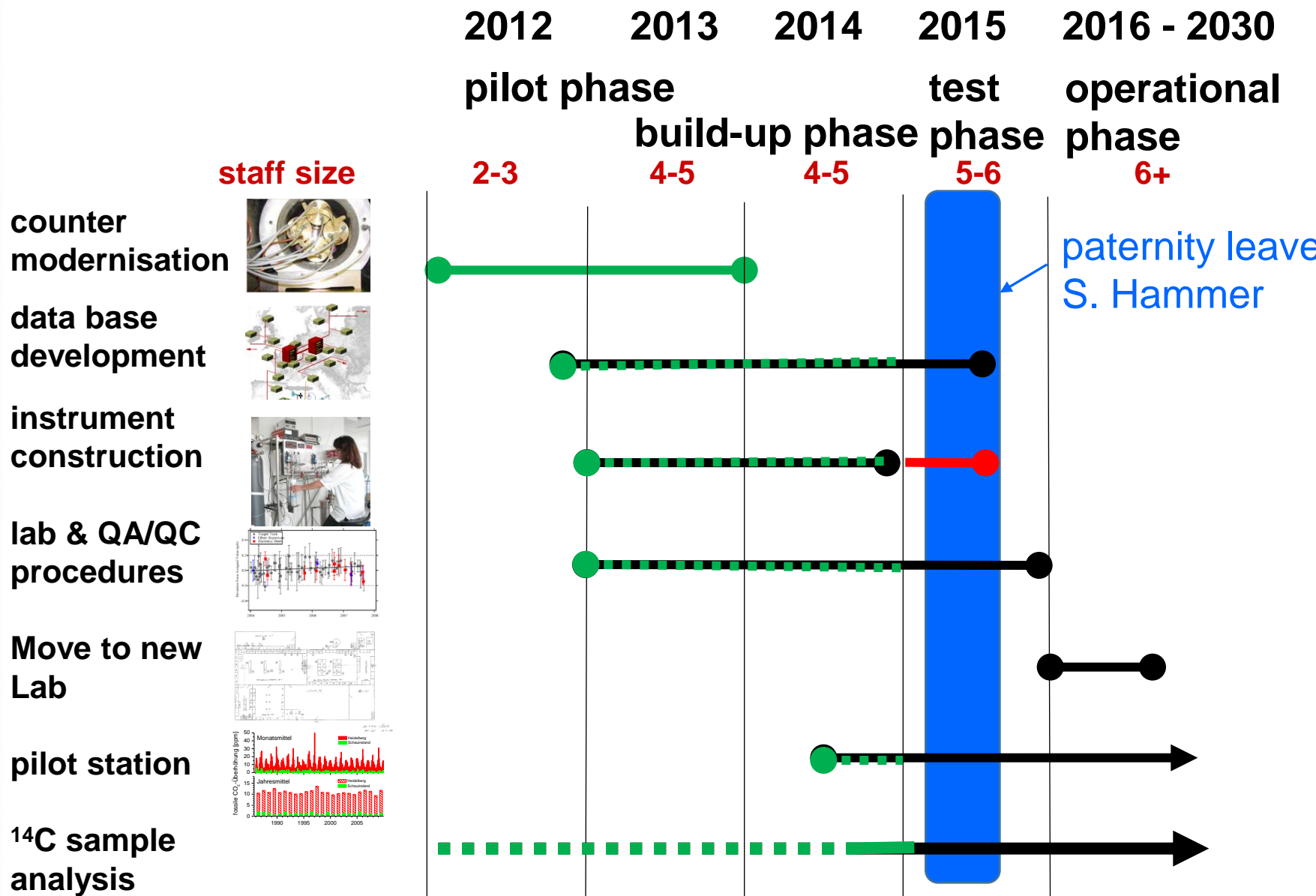
Ronny Friedrich und Bernd Kromer
Klaus-Tschira-Archäometrie-Zentrum

ICOS-D Project Meeting 2015



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CRL implementation plan



The current ICOS $^{14}\text{CO}_2$ network and extensions

Mace Head

Cabauw

HEI

OPE

Trainou

Jungfraujoch

Norunda

Lund

Gartow

Lindenberg

Kresin

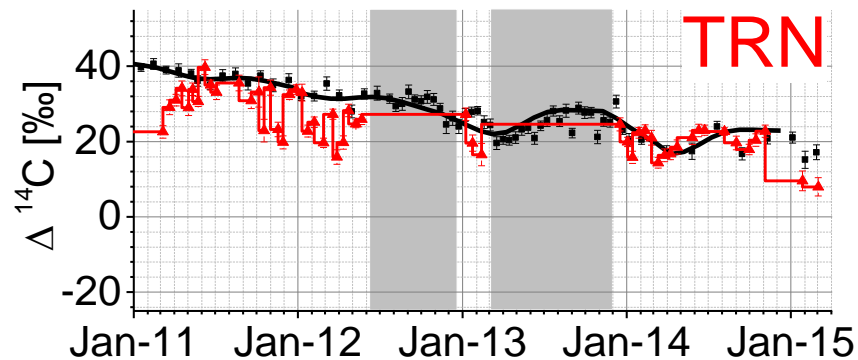
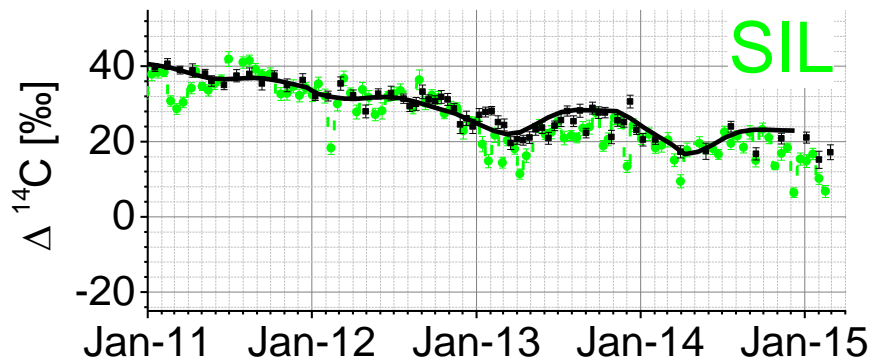
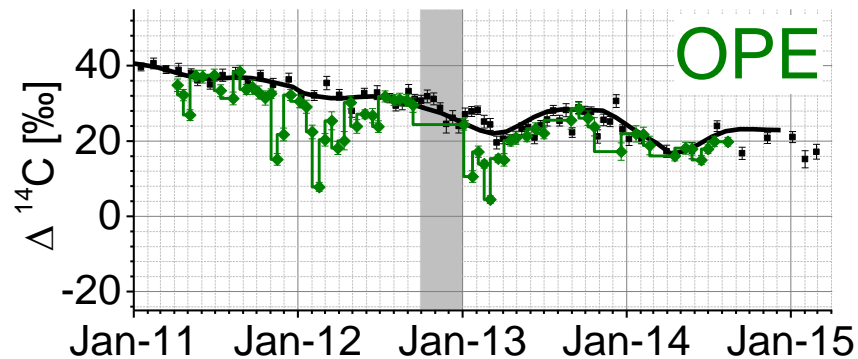
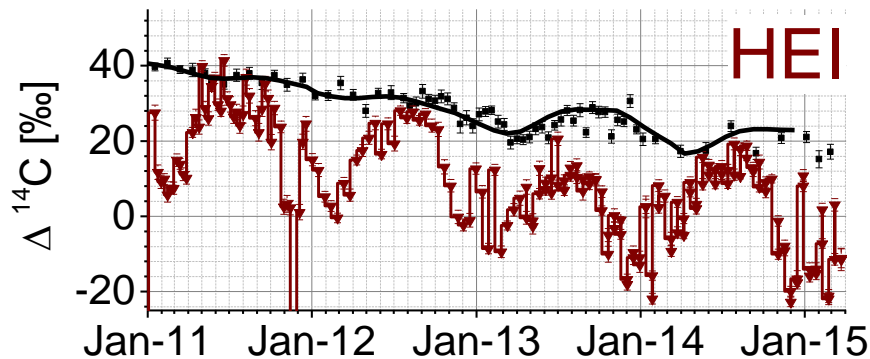
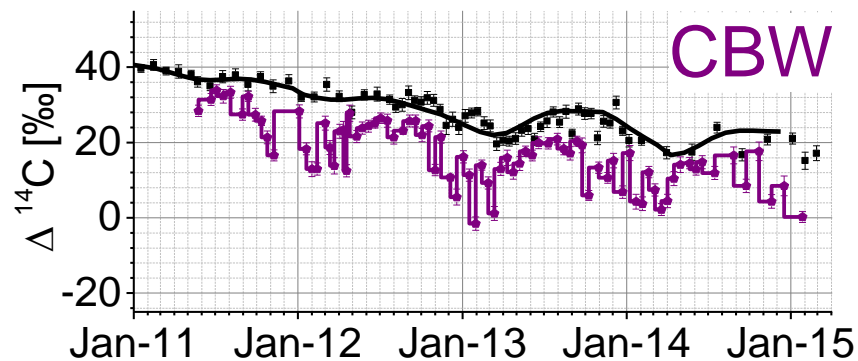
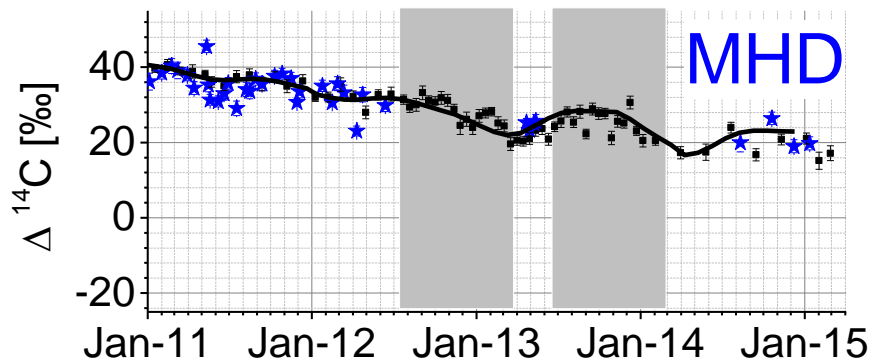
Ochsenkopf

Hohenpeißenberg

Data SIO, NOAA, U.S. Navy, NGA, GEBCO
Image Landsat
Image IBCAO

Google earth

$^{14}\text{CO}_2$ results from integrated sampling

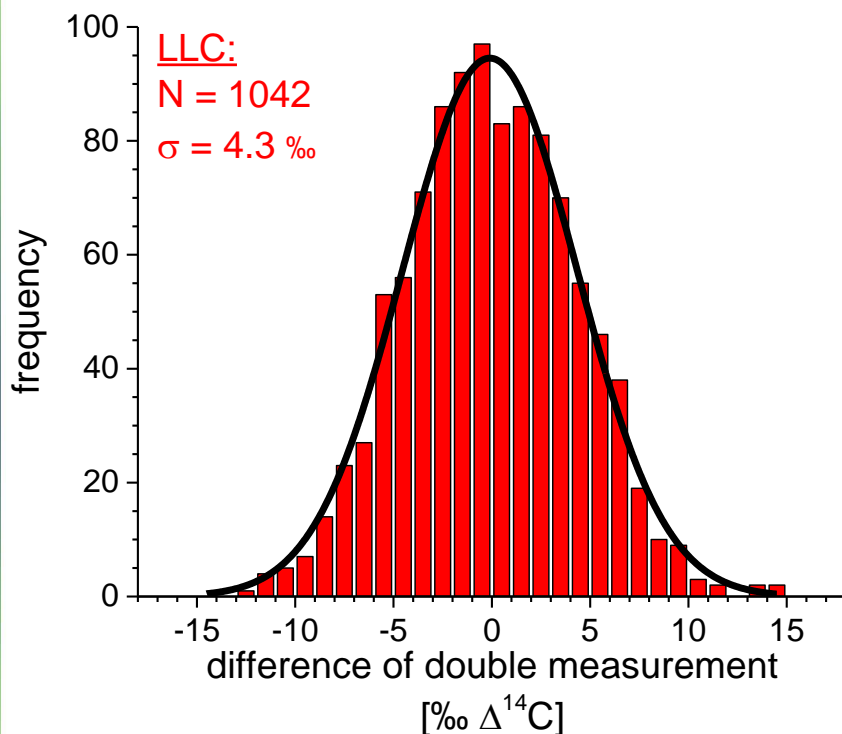


Tasks of the CRL within ICOS

- build conventional $^{14}\text{CO}_2$ sampling equipment, supply and support routine $^{14}\text{CO}_2$ monitoring in the ICOS network
- perform high quality $^{14}\text{CO}_2$ measurement using conventional low level counting (LLC) and AMS
- develop new and enhanced fossil fuel quantification tools and monitoring strategies, tested at the IUP CRL pilot station
- provide a comprehensive and simplified data base system ensuring a high degree of data provenance
- **document the laboratory performance by using a rigorous and fully traceable quality management system**

Repeatability: difference of duplicate measurements

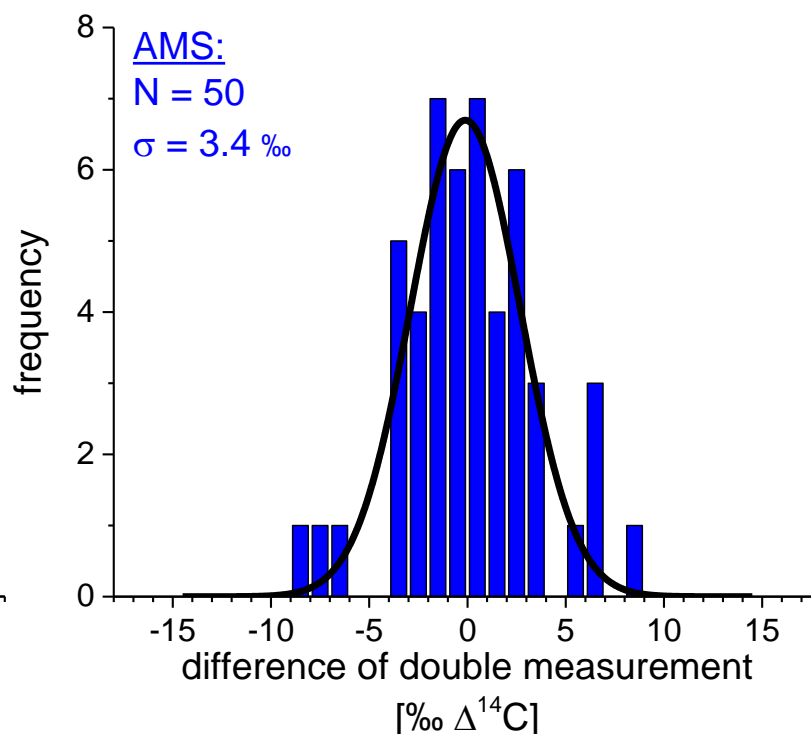
Low level counting (LLC)



Typically two measurements therefore:

$$\sigma_{LLC} = 2.1\text{‰}$$

Accelerator mass spectrometry (AMS)

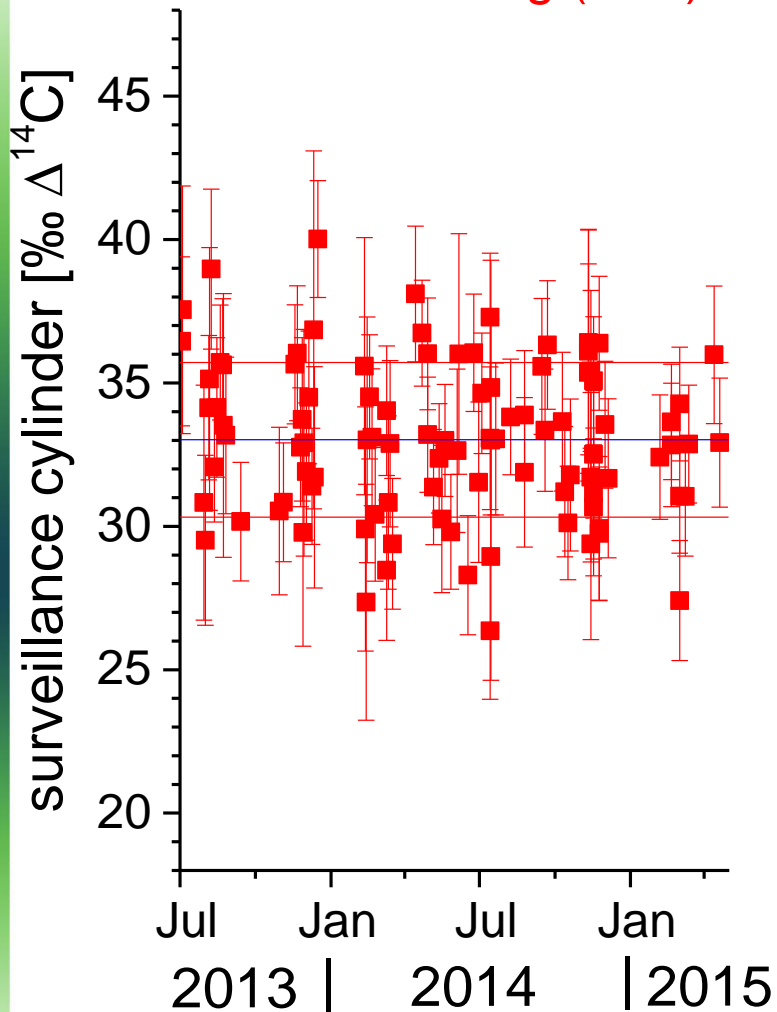


Typically only one measurement therefore:

$$\sigma_{AMS} = 2.4\text{‰}$$

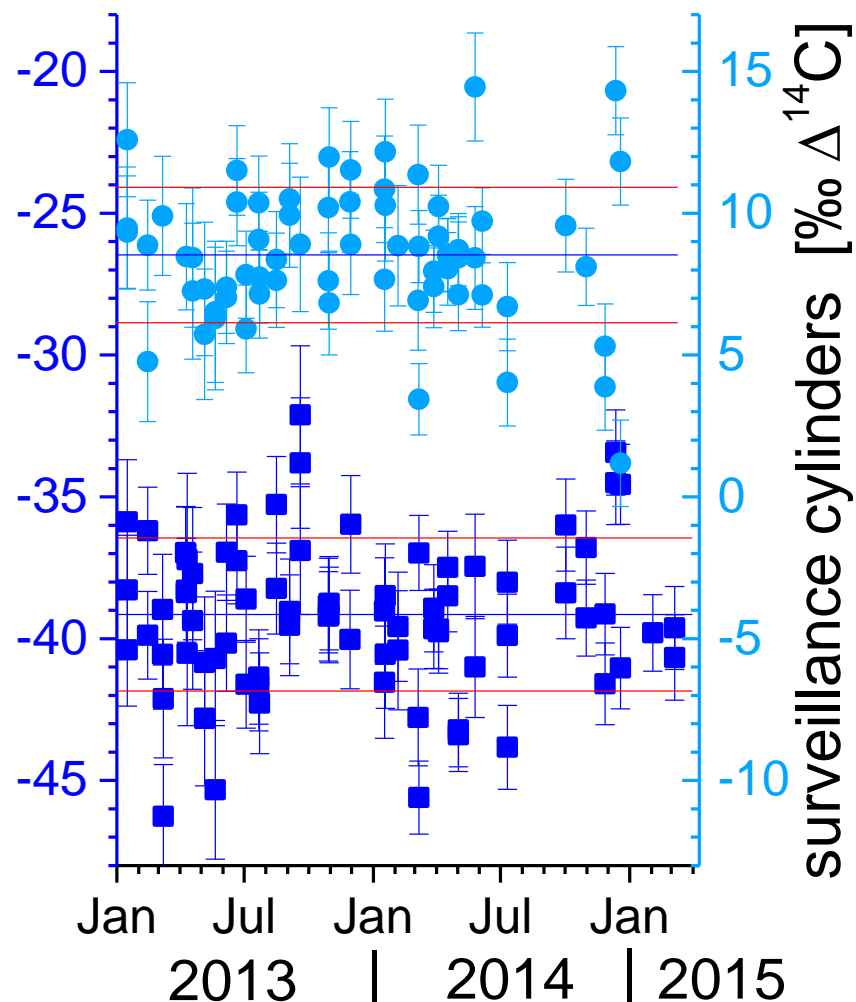
Intermediate measurement precision

Low level counting (LLC)



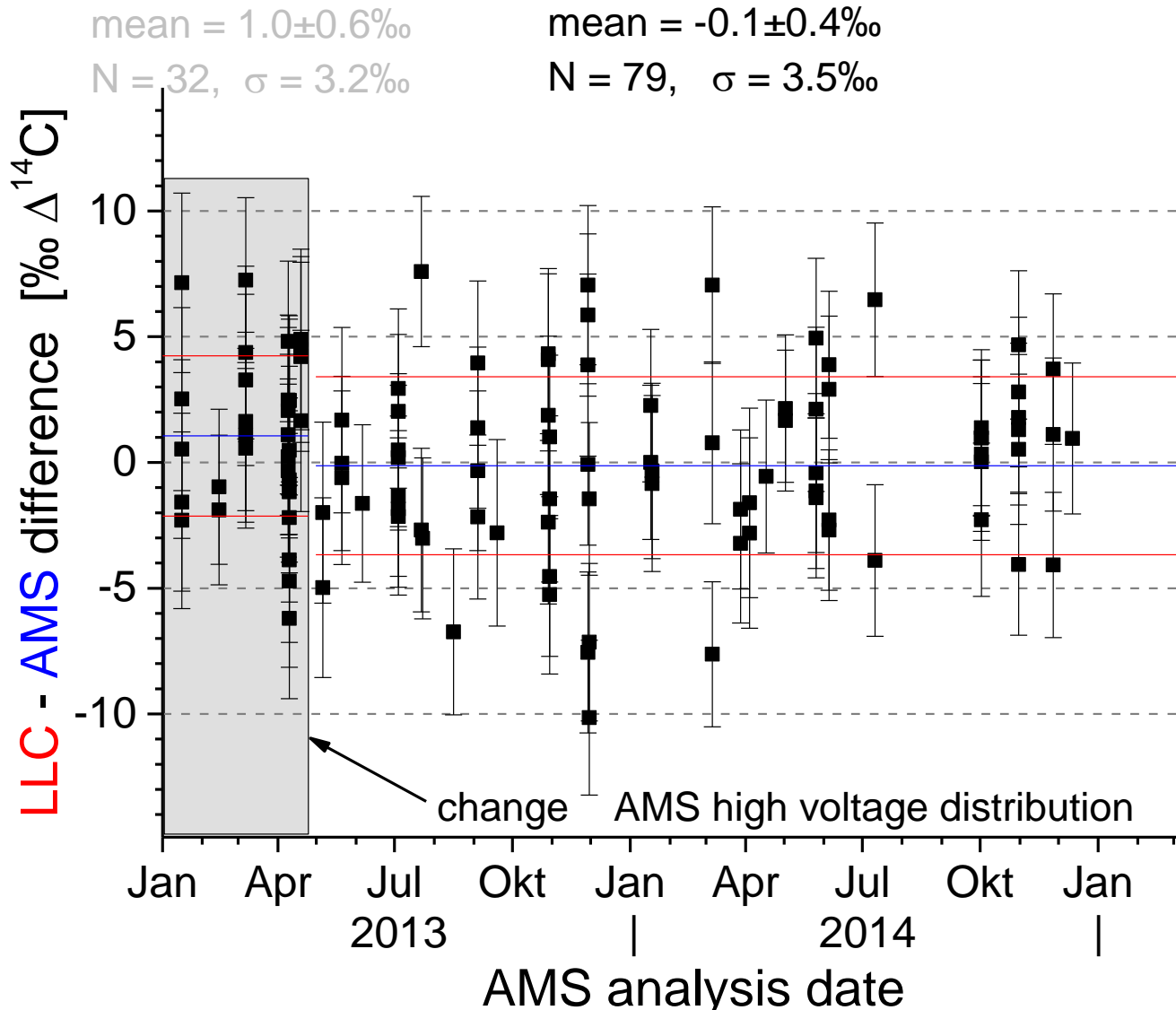
$N = 93$
 $\sigma = 2.7\text{‰}$

Accelerator mass spectrometry (AMS)



$N = 79$ $N = 68$
 $\sigma = 2.7\text{‰}$ $\sigma = 2.4\text{‰}$

LLC – AMS comparison



Summary

- The ICOS CRL has made good progress in providing comprehensive, high quality $^{14}\text{CO}_2$ data for the entire ICOS atmosphere network
- This network is growing and the ICOS CRL is about to become operational with more than 350 ^{14}C samples being analyzed in 2014
- The repeatability for LLC samples is on the order of 2‰ $\Delta^{14}\text{C}$ and 2.4 ‰ $\Delta^{14}\text{C}$ for AMS
- Both measurement techniques show no temporal trend and are in excellent agreement.

