

## STELLAR Science Highlights and Good News stories #2

### A Successful Final Project Meeting and Stakeholder Workshop for STELLAR



A successful M36 final project meeting for the STELLAR project was held at Bushy House in Teddington, England by project partner NPL. Ruth Pearce and Garry Hensey (NPL) welcomed all to the meeting and Paul Brewer – NPL Fellow and Head of Science of the Atmospheric Environmental Science Department provided a short introduction.

This was followed by presentations on work progressed under WP1 by Harro Meijer (RUG), a presentation on WP2 was provided by Stefan Persijn (VSL). Jelka Braden-Behrens (PTB) provided an overview of WP3 and the challenges and discussions with partners around the first phases, and upcoming challenges and discussions towards M36. Garry Hensey presented on the Impact Work progressed under WP4 and provided a guide to the project management status under WP5.

During the work package presentations and discussions, there was further discussion with the consortium regarding the output of the project and future progress towards M36.

For the Workshop, each technical WP Lead provided a ten-minute presentation of highlights from the project to some of the stakeholders. This was followed by a Q&A session.

### STELLAR Training at RUG



Project partner RUG report on the training given to students under STELLAR. A bachelor's student got trained in our lab from November 2021 to February 2022 to fill glass sample flasks with reference air from high pressure cylinders, condition our glass sample flasks to remove any traces of water and measure the isotope composition of atmospheric CO<sub>2</sub> at our Aerodyne dual laser absorption spectrometer. A bachelor's student got trained in our lab from April to July 2021 to prepare CO<sub>2</sub>-in-air samples from pure CO<sub>2</sub> reference materials and measure the samples for their stable isotope composition on our Aerodyne dual laser absorption spectrometer.

A bachelor's student got trained in our lab from April to July 2022 to prepare pure CO<sub>2</sub> samples from calcite reference materials using the phosphoric acid reaction. A bachelor's student got trained in our lab from April to July 2023 to conduct an equilibration experiment of CO<sub>2</sub> and water with the aim to develop pure CO<sub>2</sub> reference material with an oxygen isotope composition linked to the VSMOW scale. Then on the 9th of May 2023 three members of the STELLAR project (Paul Brewer, Heiko Moossen and Nives Ogrinc) were in Groningen to be part of the promotion committee for the defense of Pharahilda M. Steur's thesis with the title 'Using laser absorption spectroscopy for the measurement of  $d^{13}\text{C}$ ,  $d^{18}\text{O}$  and  $d^{17}\text{O}$  of atmospheric CO<sub>2</sub>.

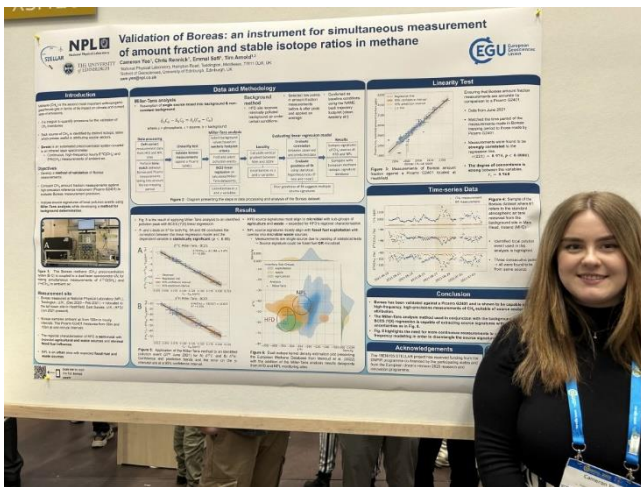
## Preventing drift of oxygen isotopes of CO<sub>2</sub>-in-air stored in glass sample flasks: new insights and recommendations

STELLAR project partners RUG and the Max Planck Institute for Biogeochemistry have made a new project paper available, [Preventing drift of oxygen isotopes of CO<sub>2</sub>-in-air stored in glass sample flasks: new insights and recommendations](#).

It is known that the oxygen isotope composition of CO<sub>2</sub>-in-air, when stored over longer time periods in glass sample flasks, tends to drift to more negative values while the carbon isotope composition remains stable. The exact mechanisms behind this drift were still unclear. New experimental results reveal that water already inside the flasks during sampling plays a major role in the drift of the oxygen isotopes. A drying method to remove any water sticking to the inner walls by evacuating the flasks for more than 72 h while heating to 60°C significantly decreases drift of the oxygen isotopes. Moreover, flasks not dried with this method showed higher differences among drift rates of individual flasks. This is explained through the build-up of H<sub>2</sub>O molecules sticking to the inner walls. Humidity of the air samples in the flasks as well as surface characteristics will lead to differences among flasks. Results also show that permeability of water is higher through Viton O-ring flask seals than through polychlorotrifluoroethylene (PCTFE) shaft seals, and that the stability of flasks sealed with the latter is significantly better over time.

You can find the paper here and over on our [Documents](#) page.

## Validation of Boreas: an instrument for simultaneous measurement of amount fraction and stable isotope ratios in methane



Cameron Yeo of project partner NPL took part in a poster session at the EGU General Assembly 2023 that convened in Vienna, Austria & Online, 23–28 April 2023.

Cameron presented her work on ‘*Validation of Boreas: an instrument for simultaneous measurement of amount fraction and stable isotope ratios in methane*’ as part of the STELLAR project. The poster garnered audience with a large blend of scientists from many disciplines and countries, including the opportunity to discuss the future of projects with current collaborators.

A copy of the poster displayed at the event can be found [here](#) over on our Documents page.



## Metrology for Stable Isotope Ratio Measurements at the 2nd ISO-FOOD symposium

Nives Ogrinc of project partner Jožef Stefan Institute took part in a poster session at the 2nd ISO-FOOD symposium. The poster entitled: *Metrology for Stable Isotope Ratio Measurements* was presented at 2nd ISO-FOOD symposium held 24.4.-26.4.2023 in Portorož in Slovenia

## STELLAR at CCQM – Gas Analysis and Isotope Ratio WG

Ruth Hill-Pearce presented an overview of the STELLAR project at the Meeting of the CCQM – Gas Analysis and Isotope Ratio Working Groups at the Bureau International des Poids et Mesures on the 25 April 2023.

### Analysis of light stable isotopes in environmental and food samples: technical analysis and validation of methods



Nives Ogrinc of project partner Jožef Stefan Institute arranged training entitled: *Analysis of light stable isotopes in environmental and food samples: technical analysis and validation of methods*. The session occurred April 19-21, 2023, at the Department of Environmental Sciences, Jožef Stefan Institute in Ljubljana. The training covered the techniques, methodology, evaluation of stable isotope results and measurements uncertainty in environmental and food samples. The project and its activities were also presented during the session.

### NPL chair 47<sup>th</sup> meeting of the CCQM-GAWG at the BIPM, Paris

Paul Brewer chaired the 47th meeting of the Consultative Committee on Amount of Substance Gas Analysis Working Group (CCQM GAWG) at the BIPM in Paris. This is the first meeting to be held at the BIPM since 2019 and only the second in person (hybrid) meeting to be held since 2020. New proposals were presented by NPL for key comparisons on ethanol in air (CCQM-K93.2023; Panayot Petrov, NPL), carbon capture, utilisation and storage (CCUS; Manohara Gudiyor Veerabhadrapa, NPL) and for a new ongoing key comparison on NO<sub>2</sub> (BIPM.QM-K6) coordinated by the BIPM that NPL has been helping to establish through a Joint Technical Project. Dave Worton presented updates on progress in two key comparisons, CCQM-K26b.2019 ambient SO<sub>2</sub> in air and CCQM-K164 hydrogen purity that NPL is coordinating.

The GAWG meeting was preceded by a joint Isotope Ratio Working Group (IRWG) and GAWG meeting which was co-chaired by Paul Brewer (NPL) where the final report of the first key comparison pilot of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  of pure CO<sub>2</sub> (CCQM-P204) including some of the challenges relating to scale realisation of VPDB and incomplete uncertainty

budgets were discussed. Resolving these challenges is of critical importance for the success of ongoing key comparison BIPM.QM-K3 that will be used to underpin new and improved capabilities for value assignment of isotope ratio for CO<sub>2</sub>. A new joint task group entitled 'Stable Isotope Ratio Metrology for Atmospheric Source Apportionment of Greenhouse Gases' was proposed to strengthened collaborations between the IRWG and GAWGs. The aim of the task group is to facilitate the development of a robust measurement infrastructure for the accurate and real-time measurement of stable isotope ratios for atmospheric greenhouse gases. The focus will be on the stable isotopes of CO<sub>2</sub> and CH<sub>4</sub>. Abneesh Srivastava (NIST) will act as the chair and Christoph Nehrbass-Ahles (NPL) was appointed Executive Secretary. The initial tasks include engaging with stakeholders such as the instrument manufacturers to develop best practice guidelines for the calibration of optical instruments and review the currently employed calibration hierarchies for  $\delta^{13}\text{C-CO}_2$ ,  $\delta^{18}\text{O-CO}_2$ ,  $\delta^{13}\text{C-CH}_4$ , and  $\delta\text{D-CH}_4$ .

At the joint IRWG/GAWG meeting Ruth Hill-Pearce (NPL) also presented an overview of the EMPIR project STELLAR. The STELLAR project is providing isotopic CO<sub>2</sub> and CH<sub>4</sub> reference materials, validation routines and traceability chains for spectroscopic techniques to allow measurements of the isotope ratio of CO<sub>2</sub> and CH<sub>4</sub> to be made in the field. This will allow improved discrimination between natural and manmade sources of GHGs to aid governmental agencies to better attribute emission sources, help demonstrate compliance to national reduction targets and enhance the effectiveness of future abatement strategies.

#### **Outreach And Training Activity work with BRIN**

NPL hosted Muhammed Rizky Mulyana from BRIN - The National Research and Innovation Agency in Indonesia for a 5-month placement in the gas metrology team. Working on activities in WP1, Rizky learned the latest techniques for producing low uncertainty greenhouse gas reference materials and undertook isotopic characterisation of CO<sub>2</sub> by optical isotope ratio spectroscopy. Rizky also learned techniques for characterising impurities in matrix gases including water vapour and took samples of CO<sub>2</sub> and CH<sub>4</sub> reference materials for IRMS measurements.

This outreach and training activity under STELLAR is important so that not just European countries benefit from this research. Rizky hopes to make isotopic gas reference materials for climate monitoring in Indonesia, and now has the background to do that. Future collaborations and bilaterals between NPL and BRIN will be planned in the near future.

#### **Generation of CO<sub>2</sub> gas mixtures by dynamic dilution for the development of gaseous certified reference materials**

STELLAR project partner INRiM have made a new project paper available, [Generation of CO<sub>2</sub> gas mixtures by dynamic dilution for the development of gaseous certified reference materials](#).

The use of Certified Reference Materials (CRMs) is of utmost importance to achieve the comparability and traceability of data, which are essential features of measurement results in environmental and climate fields. The present paper focuses on the generation of gas mixtures at known composition of carbon dioxide at atmospheric amount-of-substance fraction in synthetic air by means of a dynamic dilution system, designed and implemented at the Istituto Nazionale di Ricerca Metrologica (INRiM). The validation of the dynamic system in terms of amount-of-substance fraction is presented. The system was also used to verify the carbon dioxide amount-of-substance fraction of a suite of gas mixtures gravimetrically prepared at INRiM in the framework of the EMPIR Joint Research Project 19ENV05 – STELLAR. Dynamic dilution proved to be an effective tool for the preparation and certification of CRMs for gaseous pollutants (i.e., carbon dioxide, nitrogen oxides) relevant for monitoring environmental pollution and climate changes.

You can find the paper here and over on our [Documents](#) page.

## STELLAR project at ECOFIRA



Fernando Grau in the ECOFIRA and the Air Liquide stand  
Project partner, Air Liquide España, S.A., presented STELLAR within the framework of the [ECOFIRA](#) “Salón del Sector Químico para la Sostenibilidad y Economía Circular”, on October 4-6, 2022, in Valencia.

The STELLAR project was indicated as a research case in Air Liquide’s presentation to the general audience. In addition, Air Liquide disseminated more information about the project at its stand, where a project poster was exhibited.

A copy of the poster displayed at the event can be found [here](#).

## STELLAR at GGMT 2022

Farilde Steur of RUG and Heiko Moossen of MPG presented material on the project at the 21st WMO/IAEA Meeting on Carbon Dioxide, Other Greenhouse Gases and Related Measurement Techniques (GGMT-2022) Wageningen, The Netherlands from 16-21 September.

Farilde presented on the [Long term performance of a dual-laser absorption spectrometer for the measurement of  \$\delta^{13}\text{C}\$ ,  \$\delta^{18}\text{O}\$  and  \$\Delta^{17}\text{O}\$  of  \$\text{CO}\_2\$](#) : results from measurements of stable isotope composition of atmospheric  $\text{CO}_2$  using an Aerodyne dual-laser absorption spectrometer over the period 2020- September 2022. Also, first results from primary reference materials measured on the spectrometer were shown. RUG are working on optimising the production and dilution of primary reference materials to realise a direct link to the VPDB(- $\text{CO}_2$ ) scale for our optical measurements. Heiko provided a presentation on the [Interlaboratory compatibility of  \$\delta^{13}\text{C}\$ -VPDB scale realisation](#).

You can find links to the presentations above, and also over on our [Documents](#) page.

## Successful M27 meeting for STELLAR

A successful M27 meeting for the STELLAR project was held at the University of East Finland Kuopio campus and online by project partner VTT. Ruth Pearce and Garry Hensey (NPL) welcomed all to the meeting and noted that this was our first STELLAR in person meeting and that it was a pleasure to see so many.

This was followed by presentations on work progressed under WP1 by Harro Meijer (RUG), a presentation on WP2 was provided by Stefan Persijn (VSL). Jarvis Nwaboh (PTB) provided an overview of WP3 and the challenges and discussions with partners around the first phases, and upcoming challenges and discussions towards M36. Garry Hensey presented on the Impact Work progressed under WP4 and provided a guide to the project management status under WP5.

During the work package presentations and discussions, there was further discussion with the consortium regarding the output of the project and future progress towards M36.

Hilary Phillips and Mara Soares Silva from the EMPIR Management Support Unit (MSU), provided a talk on exploitation planning and generating impact from STELLAR developments.

### **Reference Materials: gas mixtures to support measurements for climate change studies.**

STELLAR project partner INRiM have made a new project paper available, [Reference Materials: gas mixtures to support measurements for climate change studies](#).

This work aims at presenting some activities carried out at INRiM for the development of new reference materials (RMs) of greenhouse gases to support measurements for climate change studies. Since the preindustrial era, greenhouse gases emissions due to human activities have dramatically increased, mostly due to economic and demographic growth and their concentrations in the atmosphere have grown continuously since 2011, reaching highest levels in 2019. The increase of greenhouse gases concentration in atmosphere due to anthropogenic emissions is the main cause of global warming and carbon dioxide (CO<sub>2</sub>) is one of the main culprits for this phenomenon. In this context, the capability of discriminating between anthropogenic and natural emissions is of utmost importance. The determination of the isotopic composition of CO<sub>2</sub> can support the assessment of the uptake of CO<sub>2</sub> in the environmental compartments, i.e. atmosphere and hydrosphere, and can help to distinguish natural from anthropogenic carbon in the atmosphere. The possibility to establish metrological traceability of the measurements of CO<sub>2</sub> isotopic composition is a key aspect, and greatly relies on the availability of gaseous reference materials having compositions and uncertainties that are fit for purpose. In the past years, INRiM participated in the EMPIR project 16ENV06 SIRS “Metrology for stable isotope reference standards”, and is now taking part in the EMPIR project 19ENV05 STELLAR “Stable isotope metrology to enable climate action and regulation”. INRiM has experience in the preparation of gas standards by primary methods, namely gravimetry and dynamic dilution, which can be applied to the realisation of reference materials of CO<sub>2</sub> at known isotopic composition. The present work describes the preliminary results obtained by INRiM in the preparation of pure CO<sub>2</sub> RMs and at atmospheric amount fraction. Examples of uncertainty budgets and identification of the uncertainty sources are also given.

You can find the paper here and over on our [Documents](#) page.

## Air Liquide present the STELLAR project to IRMS users



(Left) The Air Liquide team: Fernando Grau, Arancha Oca and Antonio Carreira. (Right) The IRMS User Group

Fernando GRAUMARTI  
30 sept 2022 en myNews IBERIA

Participación del equipo R&A en la Jornada de Usuarios de equipos de Espectrometría de masas de relaciones isotópicas (IRMS).

El pasado 29 de Septiembre, el equipo R&A ha participado en un evento organizado por ThermoFischer Scientific y la Universidad de Málaga en donde se citaron 46 usuarios de equipos IRMS pertenecientes a la comunidad científica más importante en España. La ponencia por parte de **+Antonio CARREIRA**, **+Arancha OCA** y **+Fernando GRAUMARTI** aparte de remarcar nuestra amplia gama en el mercado de Isótopos incidió en la vocación de Investigación e **#Innovación** de **#AirLiquide**, como un Proyecto de **#Hidrógeno** isotópico en **#Iberia** con IRNAS-CSIC y la participación en el Proyecto Europeo **STELLAR EMPiR**.



Project partner Air Liquide España, S.A. participated in an event organised by Thermo Fisher Scientific and the University of Malaga, that gathered almost 50 users of IRMS (Isotope Ratio Mass Spectrometry) equipment belonging to one of the most important scientific communities in Spain. The event took place at the University of Malaga in late September.

Air Liquide presented the objectives of the STELLAR project, as well as its role within the project and its main commitment to contributing to the research. A copy of the poster displayed at the event can be found [here](#).

Isotope Ratio Mass Spectrometry is a specialisation of mass spectrometry, in which mass spectrometric methods are used to measure the relative abundance of isotopes in a given sample. Metrology is required to ensure advances in spectroscopy (i.e. IRMS, and Optical Isotopic Ratio Spectroscopy -OIRS) result in field deployable techniques that have reduced uncertainties in order to meet uncertainty requirements set by the World Meteorological Organisation.



## Papers

- *Boreas: A Sample Preparation-Coupled Laser Spectrometer System for Simultaneous High-Precision in Situ Analysis of  $\delta^{13}C$  and  $\delta^2H$  from Ambient Air Methane*  
Chris Rennick, Tim Arnold, Emmal Safi, Alice Drinkwater, Caroline Dylag, Eric Mussell Webber, Ruth Hill-Pearce, David R. Worton, Francesco Bausi, and Dave Lowry
- *Optical Isotope Ratio Spectroscopy – Complementing Isotope Ratio Mass Spectrometry*  
Jelka Braden-Behrens, Heleen Meuzelaar, Joachim Mohn, Javis A. Nwaboh, Stefan Persijn, Francesca Rolle, Michela Segal, Pharahilda M. Steur, Lars Wacker, Kerstin Zeyer, Olav Werhahn and Volker Ebert
- *Generation of  $CO_2$  gas mixtures by dynamic dilution for the development of gaseous certified reference materials*  
Francesca Rolle, Francesca Durbiano, Francesca Romana Pennechi, Stefano Pavarelli, Marco Santiano, Pier Giorgio Spazzini and Michela Segal
- *Reference Materials: gas mixtures to support measurements for climate change studies*  
Michela Segal, Stefano Pavarelli, Francesca Romana Pennechi, Pier Giorgio Spazzini and Francesca Rolle
- *Preventing drift of oxygen isotopes of  $CO_2$ -in-air stored in glass sample flasks: new insights and recommendations*  
Pharahilda M. Steur, Dennis Botter, Hubertus A. Scheeren, Heiko Moossen, Michael Rothe and Harro A. J. Meijer

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**EMPIR**



**EURAMET**

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