Operating charter for the

Stable Isotopes Analytical Platform (PANISS)

at the CEREGE

Any person using the instruments of the Platform must read and sign the following document. Interns, PhD students and Postdocs need to also have it signed by their CEREGE supervisor (permanent staff).

1. Presentation of the Platform

The CEREGE's stable isotope analysis platform (PANISS) is divided into 5 analytical applications : 1) δ18O and δ13C of carbonates; 2) δ18O and δ17O of silicates and oxides; 3) δ18O, δ17O and δD of water and water vapour, 4) δ15N, δ13C and δD of organic compounds and 5) D47 and D48 (clumped isotopes) of carbonates.

The data acquired will mainly be used to support CEREGE's research into palaeoclimates, water and carbon cycles, archaeology and planetology. PANISS also responds to requests for collaborative analyses or services from French and foreign academic bodies. The platform is also developing a major training activity.

2. Equipment and measurements performed

2.1- δ^{18}O and δ^{13}C analysis of carbonates (rooms 366 & 372)

- Dual inlet mass spectrometer (DI-IRMS : **Delta V Plus**, Thermo Scientific) coupled to an automated carbonate preparation line (**Kiel IV Carbonate Device**, Thermo Scientific) : measurements of δ13C and δ18O on micro-samples of 20-150 µg.
- IRIS laser spectrometer (**Delta Ray** with URI Connect, Thermo Scientific): δ13C and δ18O measurements on samples ≥ 500 µg and of continuous CO2, measurements of δ13C on Dissolved Inorganic Carbon in water.

2.2- δ^{18}O and δ^{17}O analysis of silicates (room 366)

- Dual inlet mass spectrometer (DI-IRMS : **Delta V Plus**, Thermo Scientific) coupled to a manual extraction line of oxygen in silicates, with BrF5 and a CO2 Infra-Red laser (**CO2-laser 30W**, Merchanteck): measurements of δ18O and δ17O on 0.3 - 1.6 mg samples.
- Dehydration line under nitrogen flow and at very high temperature (1100°C) : dehydration and dehydroxylation of amorphous hydrated silica samples.

2.3- δ^{18}O, δ^{17}O and δD analysis of waters (room 372)

- Dual inlet mass spectrometer (DI-IRMS **Delta Plus**, Thermo Scientific) coupled to an automated water sample equilibration line (**H/Device**, Thermo Scientific) : measurements of δD and δ18O on 3- 5 ml samples.
- CRDS laser spectrometer (Cavity Ring-Down Spectroscopy, **Picarro L2140-i**): measurements of δD, δ17O and δ18O measurements on 2 ml water samples and on water vapour.

2.4- δ^{15}N, δ^{13}C and δD analysis of organic compounds (room 357)

- Continuous Flow mass spectrometer (CF-IRMS **Delta Plus**, Thermo Scientific) linked by a **ConFlo III** interface to the following equipment :
    - Elemental Analyser (**Flash EA**, Thermo Scientific) equipped with an autosampler: measurements of %C, %N, δ13C and δ15N on bulk powders.

- Continuous Flow mass spectrometer (CF-IRMS **Delta V Plus,** Thermo Scientific), linked by a **ConFlo IV** interface to the following equipment:
    - Elemental Analyser (**Flash EA IsoLink CN**, option SmartEA, Thermo Scientific) equipped with an autosampler: measurements of %C, %N, δ13C and δ15N on bulk powders.
    - Gas chromatograph (**GC 1310-Isolink II**, Thermo Scientific) equipped with an autosampler (**Triplus** RSH, Thermo Scientific) and a FID detector : measurements of δD, δ13C and δ15N on organic molecules separated by gas chromatography.

2.5- Δ47 & Δ 48 analysis ('clumped' isotopes) in carbonates (Room 362)

- Dual inlet mass spectrometer (DI-IRMS MAT 253+, Thermo Scientific) coupled to an automated carbonate preparation line (**Kiel IV Carbonate Device**) : measurements of clumped isotopes Δ47 & Δ48 as well as δ13C and δ18O.

3. How the platform works and contacts

The platform is under the technical and scientific responsibility of Corinne Sonzogni (CNRS engineer) and Anne Alexandre (CNRS researcher), respectively. In addition, David Au Yang (IRD engineer) is the technical manager for the Silicates application and is also involved in the Water section. Anne-Lise Jourdan (CNRS engineer) is the technical manager for the Clumped section and is also involved in the Organic Compounds section. The 3 engineers manage schedule, installations, maintenance and repairs, order management, visits and training, as well as analytical developments, in collaboration with the scientific referents for the applications (Table 1).

Reference engineers and researchers ensure that the equipment is used appropriately in relation to the stated scientific objectives, carry out the necessary methodological developments and validate the data supplied.

Any analytical projects must be discussed with the scientific referent for the application concerned. Analyses are carried out in collaboration with the lead researcher or the researcher-engineer pair, or as part of a service, subject to the availability of human and analytical resources. Priority is given to degree courses (PhDs and Masters in particular), provided that the project into which the course fits, the (approximate) number of analyses required and the period for obtaining samples have been discussed in advance with the referral team.

The user provides samples ready for analysis (contact the referents for any details) and commit to pay the cost of analysis on the basis of the rates provided.

Table 1 : Contacts for engineers, scientific advisors and permanent users of the platform

Analyses	Nom	Statut	Fonction PANISS	e-mail
All analyses	Corinne Sonzogni	IR CNRS	PANISS Technical Manager	sonzogni@cerege.fr
$\delta^{18}O$, $\delta^{13}C$ Carbonates	Laurence Vidal Clara Bolton Jean-Charles Mazur	Prof AMU CR CNRS IE CNRS	Scientific referent Permanent user Engineer	vidal@cerege.fr
$\delta^{18}O$, $\delta^{17}O$ Silicates	Anne Alexandre Florence Sylvestre David Au Yang	DR CNRS DR IRD IR IRD	PANISS Scientific Manager & scientific referent Permanent user PANISS Engineer	alexandre@cerege.fr sylvestre@cerege.fr auyang@cerege.fr
$\delta^{18}O$, $\delta^{17}O$, δD waters	Christine Vallet-Coulomb David Au Yang	MCF AMU IR IRD	Scientific referent PANISS Engineer	vallet@cerege.fr auyang@cerege.fr
Organic Compounds	Guillaume Leduc Yannick Garcin Anne-Lise Jourdan	CR CNRS CR IRD IR CNRS	Scientific referent Permanent user PANISS Engineer	leduc@cerege.fr garcin@cerege.fr jourdan@cerege.fr
Clumped isotopes	Alexis Licht Anne-Lise Jourdan	CR CNRS IR CNRS	Scientific referent PANISS Engineer	licht@cerege.fr jourdan@cerege.fr

4. Platform operating times

Non-permanent users (students, PhD students and post-doctoral researchers) are welcome on the platform from 9am to 5pm. For security reasons, outside these hours, they must have the authorisation of a referent to be allowed come and work there.

5. Quality control

Approximately 1/3 of the mass spectrometers' operating time is spent on tests or calibrations.

International and laboratory standards are analysed at the same time as the samples. They are used to check that the entire analytical chain is working properly and that the results are accurate and precise, expressed as ‰ vs PDB for carbonates, ‰ or per meg vs V-SMOW for water and silicates and ‰ vs air, PDB or V-SMOW for organic compounds.

Table 2 : standards used by application

Applications	Carbonates	Silicates	Water	Organic compounds	Clumped isotopes
Nb of standards/nb of samples	1/3	1/2	1/4	TBD	TBD
IAEA or international standards Laboratory standards	NBS19	NBS28 Boulangé, MSG60, UWG, San Carlos	VSMOW, SLAP2 Ice, Tap, Sea	n-alcanes, Acetanilide	ETH, NBS19, IAEA-603

Each maintenance operation (changing the filament, modifying the source settings, changing the reference gas cylinder, etc.) is followed by tests and calibrations against IAEA reference standards.

For each application, analysis and maintenance operations are recorded in a laboratory notebook.

6. Validation of results

Results are corrected/standardised/validated by the reference team before being sent to users.

7. Safety

In the event of a problem or if you are unsure of how to proceed, users should contact the PANISS platform engineers, Corinne Sonzogni (office 359), David Au Yang (office 355) and Anne-Lise Jourdan (office 251), or the application's scientific manager (table 1), for assistance.

- When handling liquid nitrogen, lab coat, gloves and goggles must be worn to avoid burns.
- In the event of a chemical or liquid nitrogen burn, quickly put the burnt area under a stream of cold water and then warn the operator.
- The platform's rooms are equipped with leak detectors. If a detector alarm sounds, users must quickly leave the workroom and call for help.
- A first aid kit is available near the silicate dehydration line in room 366.

The silicate oxygen extraction line includes BrF_5 (toxic, harmful, corrosive and explosive) and a class IV IR laser, which is dangerous to use. Users are not authorised to work on the line unless Corinne Sonzogni, David Au Yang, Florence Sylvestre or Anne Alexandre are present nearby. Before any use of the line, safety training is given by the reference team. The BrF_5 safety data sheet and the protocol to follow in the event of a BrF_5 leak are posted at the entrance to room 366-silicates.

Use of BrF5 (toxic, harmful, corrosive and explosive)

Utilisation of a class IV laser

Date and signature of the user	**Signature of the supervisor (CEREGE permanent staff)**