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degli Studi
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Microscopic characterization of materials and devices for underwater environment at CIRCE

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Studies of the KM3NeT materials @ CIRCE

1. Characterization of the 7131 Midel Transformer Oil filling the Vertical Electro Optical Cable (VEOC):
   Evaluation of water infiltration rates;

2. Characterization of solid materials (glasses and plastics):

   - diffusion
   - microfractures

CIRCE (Center for Isotopic Research on Cultural and Environmental heritage)

3MV Pelletron

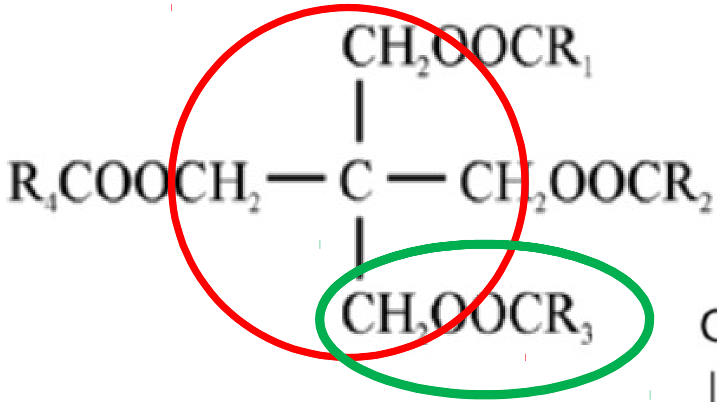
CIRCE (Center for Isotopic Research on Cultural and Environmental heritage)

IRMS lab

MIDEL 7131 MSDS

3. Composition/Information on Ingredients	3.1 Substance **CAS No.:** 68424-31-7. **Description:** Fatty acids, C5-10 (linear and branched), mixed esters with pentaerythritol.

pentaerythritol



organic ester

$$R_1 - \overset{O}{\overset{\|}{C}} - O - CH_2 - R_2$$

R_2 = fatty acid radicals with the structure of $C_{6\text{-}8}H_{13\text{-}17}$

MIDEL 7131 Moisture Standards

The standards relating to moisture content for new and in-use fluids are shown in the Table 1. New MIDEL 7131, as delivered, is manufactured to very high standards with typical moisture content of 50ppm.

Should the moisture content rise above the maximum in-service limit, the same methods and equipment that are used for removing moisture from mineral oil can also be used to remove moisture from MIDEL 7131. For example molecular sieves and vacuum filtration units.

Table 1 - **Standards for Moisture Content**

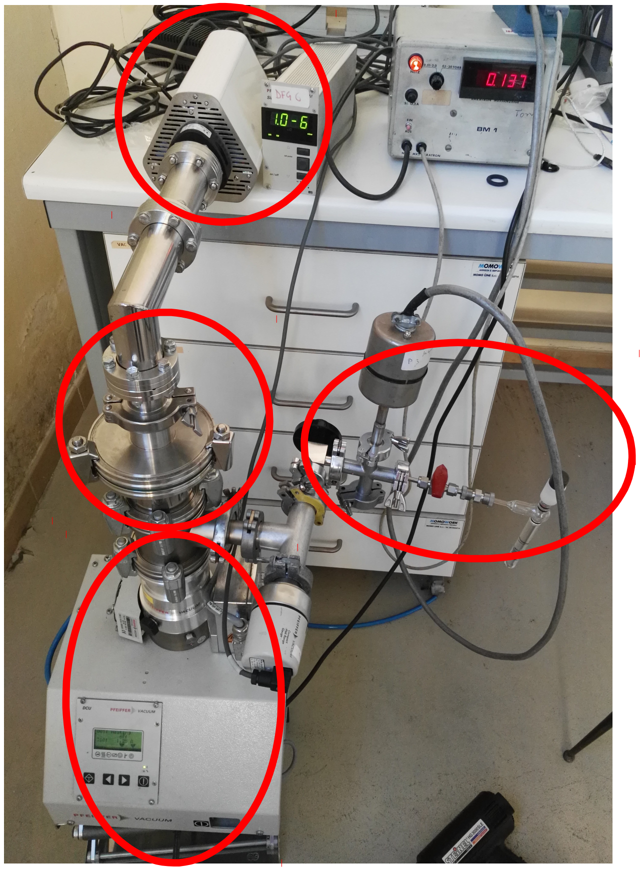
Standard	**Moisture Content**
IEC 61099 - New Esters	max. 200ppm
IEC 61203 - In-service Esters	max. 400ppm
IEC 60296 - New Mineral Oil	max. 30ppm
IEC 60422 - In-service Mineral Oil*	max. 30ppm

Note: The typical value for new MIDEL 7131 is 50ppm

*Equipment at >72.5kV and <170kV

Water absorbtion in oil

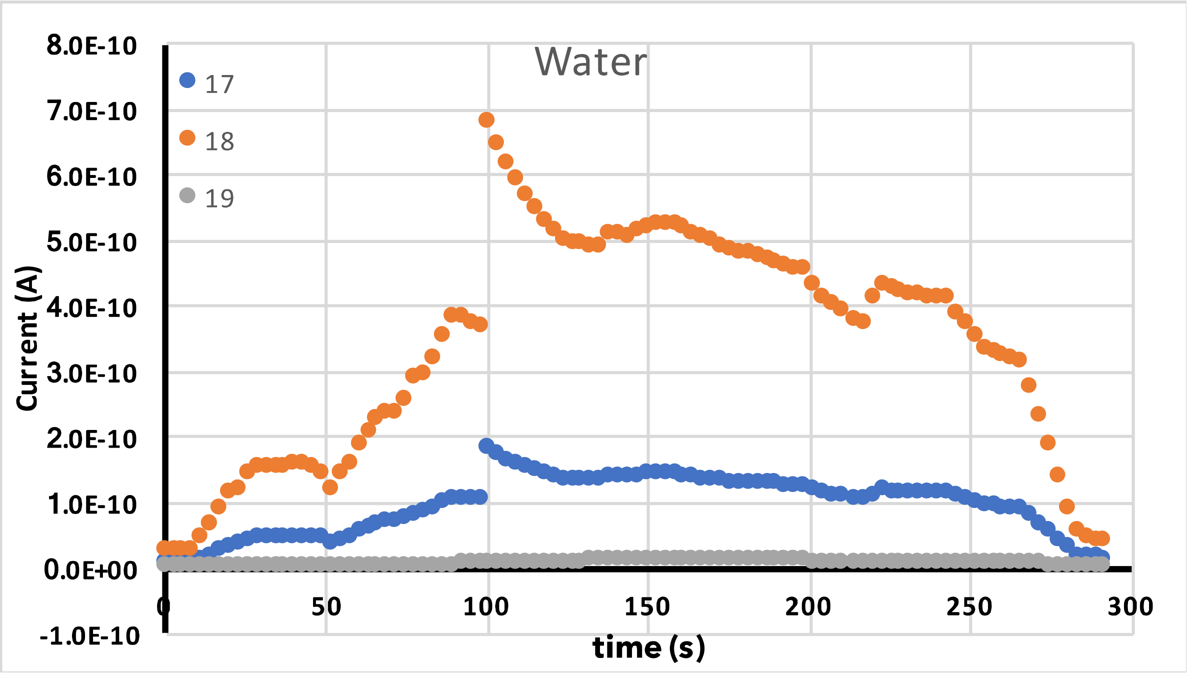
Part 1: Developing a sensitive and cost effective tool for routine QC on 7131 Midel oil moisture content.



Mass spectrometer system:

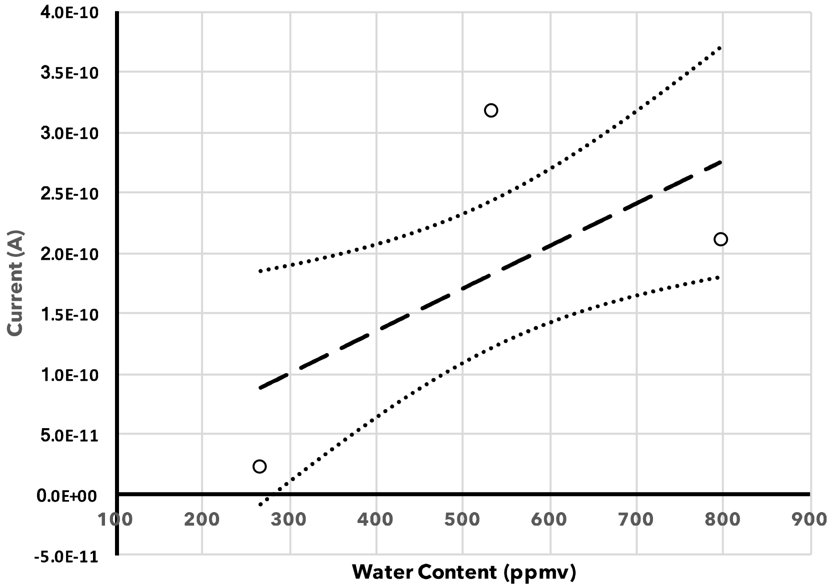
- Compact pumping station (membrane+turbo pumps);
- Inlet system with degassing chamber pressure monitored;
- Compact Mass spectrometer;
- Expansion chamber high vacuum monitored

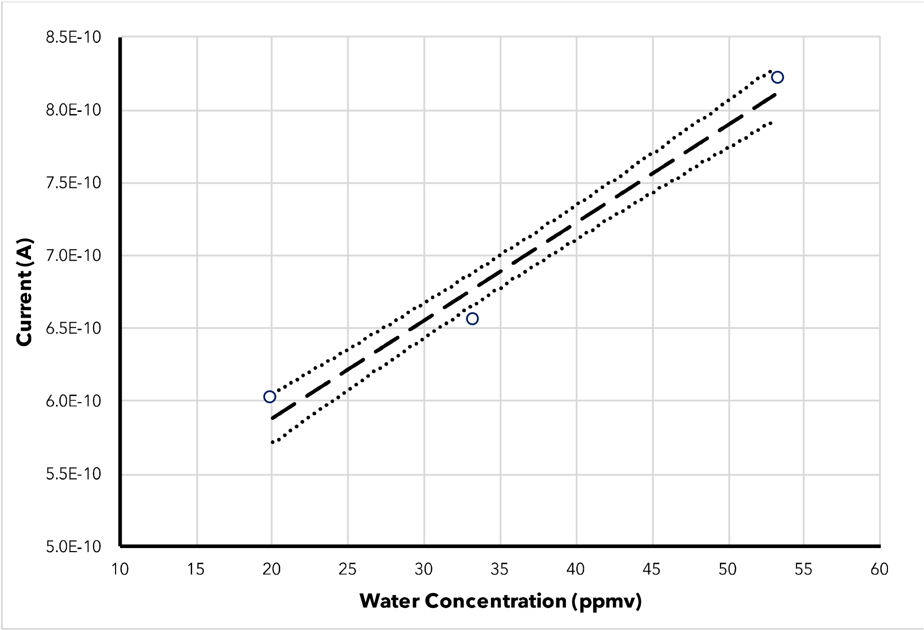
Water absorbtion in oil



Water absorbtion in oil

Calibration





Samples	ion current (A)
sample 2 Oil	6.51E-10

water (ppmv)	unc (ppmv)
29	6

Water absorbtion in oil

In order to aid impregnation of the cellulose it is recommended that MIDEL 7131 be heated to approximately 60°C when filling. At 60°C the viscosity of the fluid is very close to that of mineral oil at 20°C, and a similar impregnation rate has been observed in laboratory testing. It is further recommended that the transformer is filled slowly to aid impregnation and left for at least 24 hours prior to energising for the first time.

Table 1 - **Viscosity Values Versus Temperature**

Temperature °C	Absolute Viscosity mPa s	Kinematic Viscosity mm^2/s
0	229	233
20	73	75
40	28	29
60	13	14

Data quoted above are typical values

Water absorbtion in oil

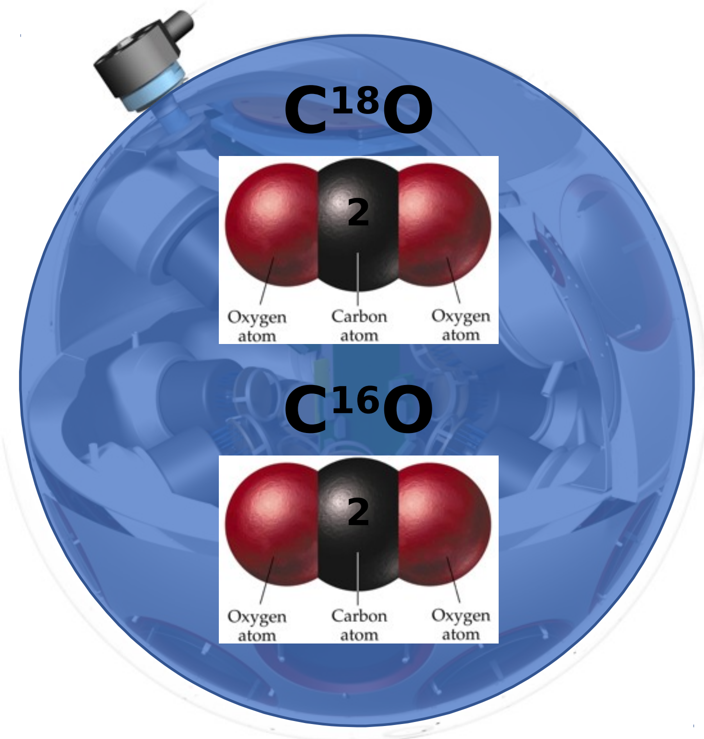
Conclusion – part 1

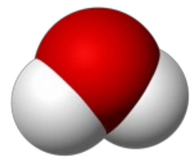
1. Applied oil de-hydration by means of vacuum appears to significantly decrease oil water content (in agreement to procedures suggested by the manufacturer);

2. The definition of a given protocol for oil de-hydration increases reproducibility of water extraction allowing also for oil water quantification with a discrete accuracy;

3. Heating @ c.a. 80°C may significantly affect water extraction (still under investigation) allowing to increase measurement sensitivity.

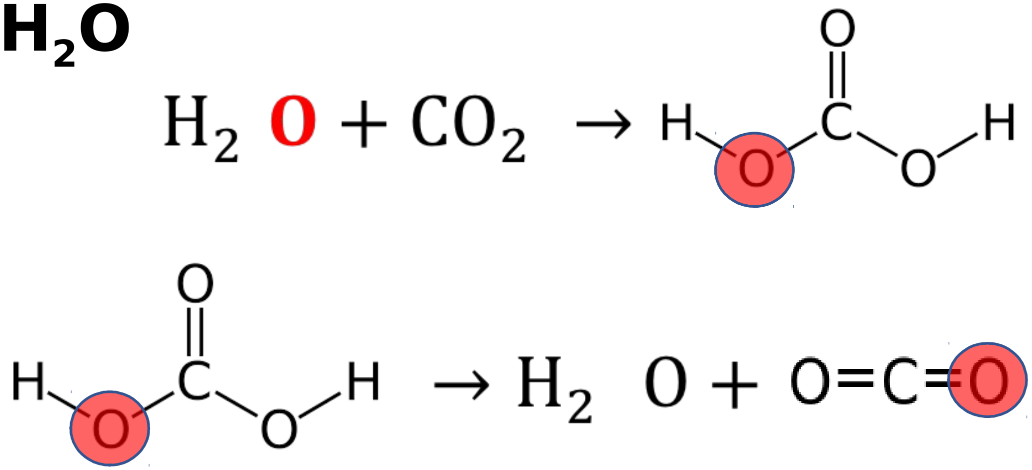
Detection of microfractures using IRMS: isotopic exchange



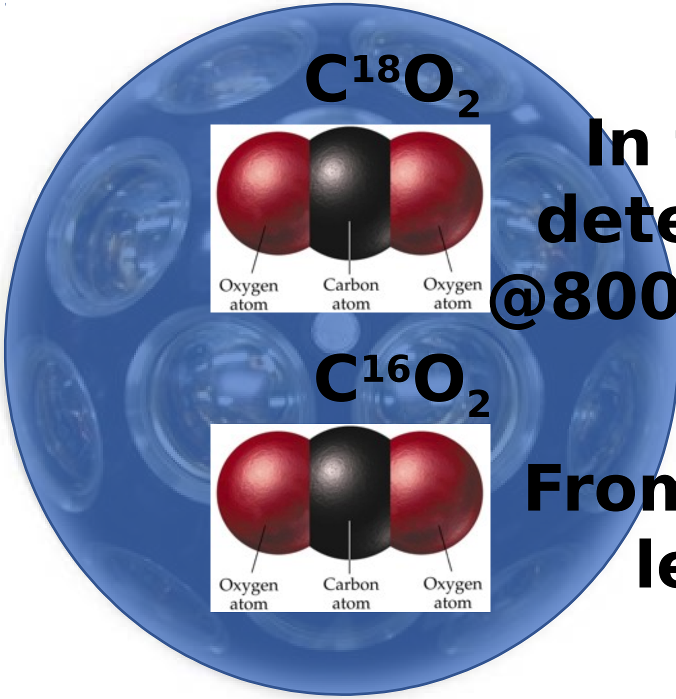


H_2O

$$H_2O + CO_2 \leftrightarrows H_2CO_3$$

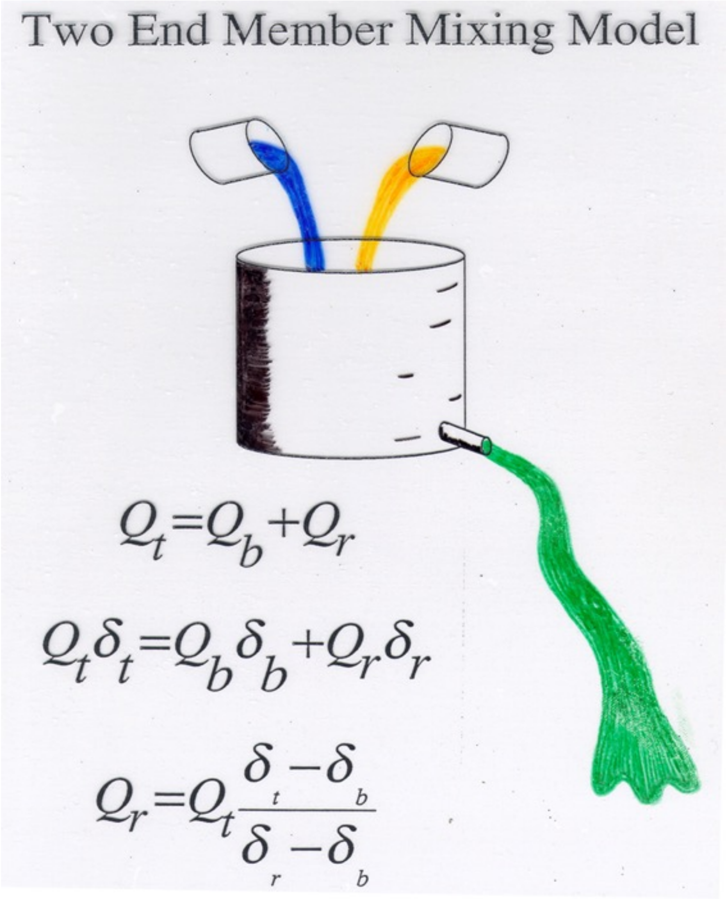


Detection of microfractures: 1. method

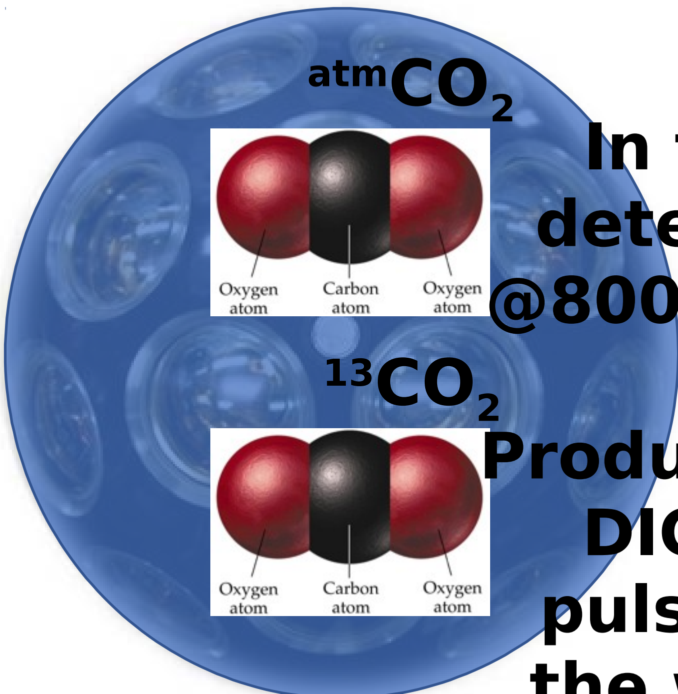


In the detector @800 mbar

From water leaks



Detection of microfractures: 2. method

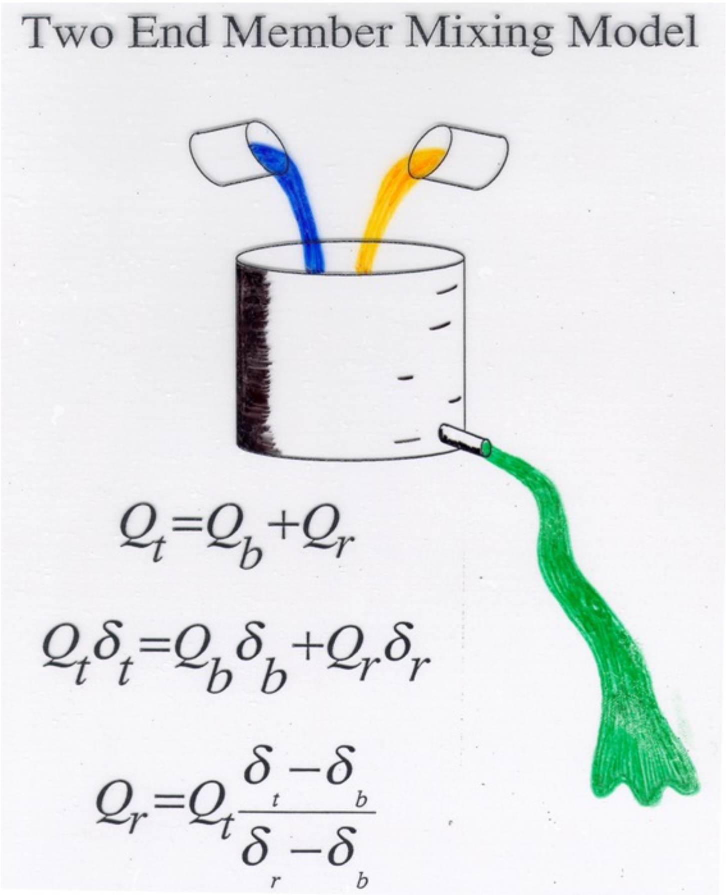


$^{atm}CO_2$

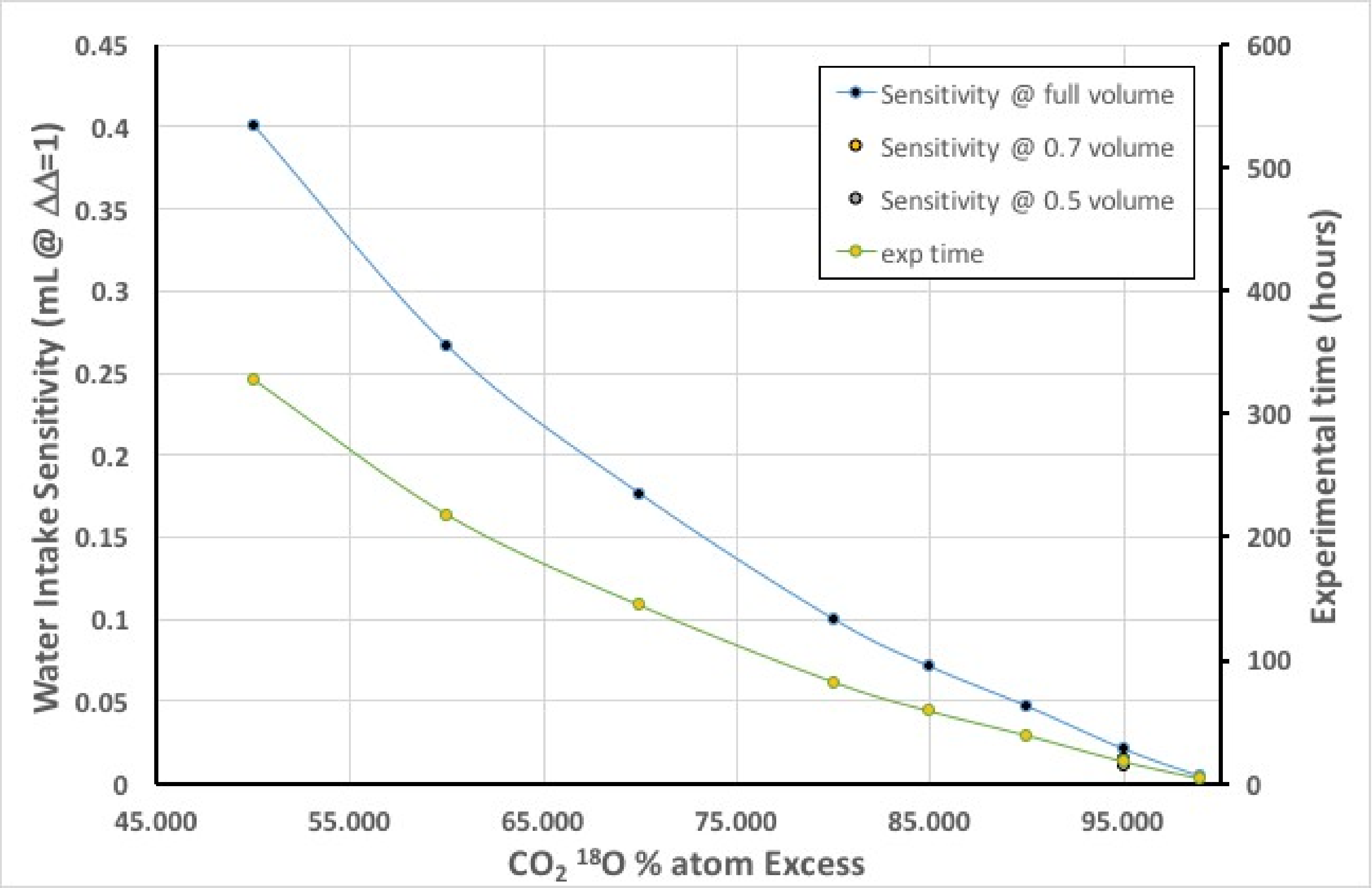
In the detector @800 mbar

$^{13}CO_2$

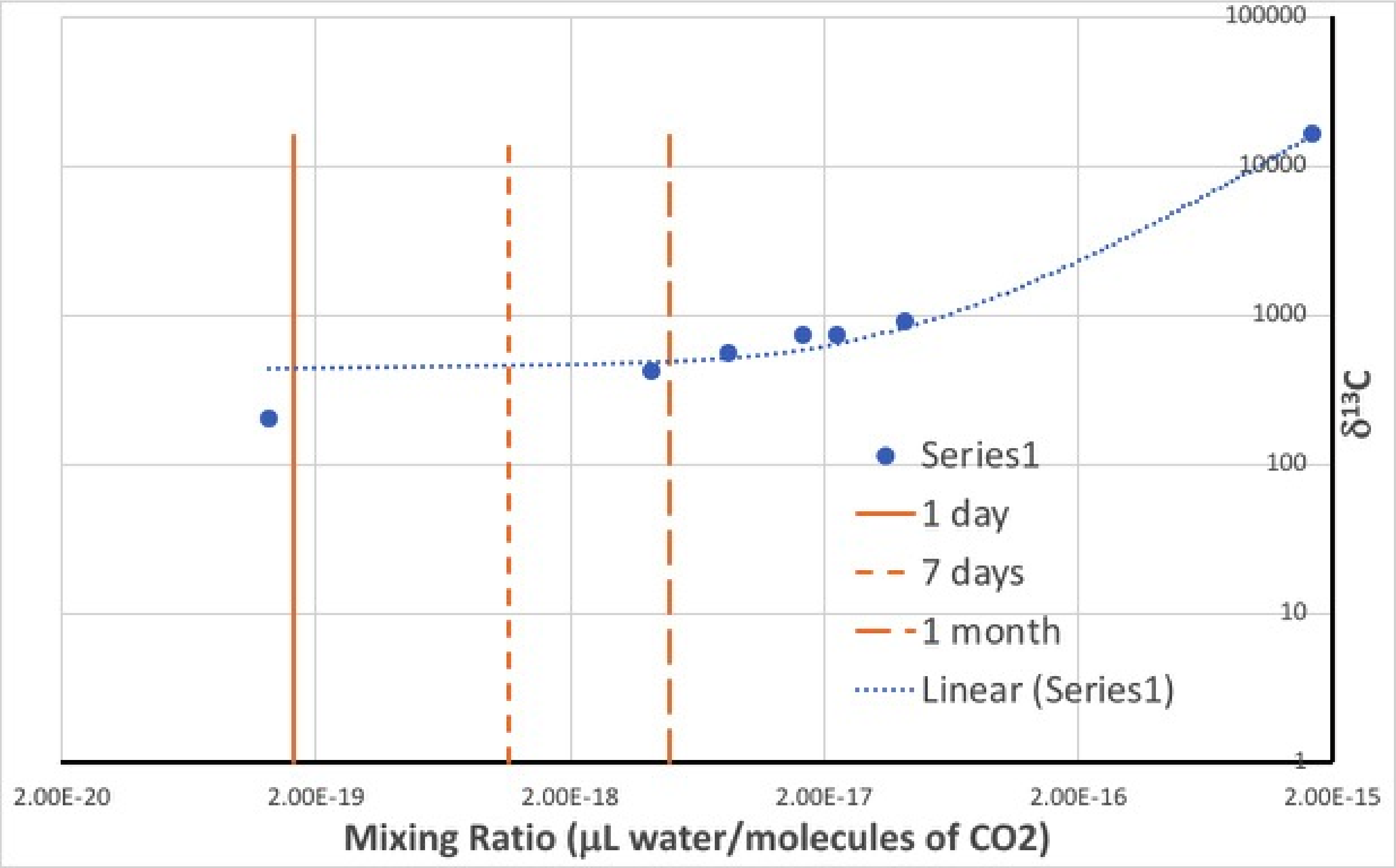
Produced by DIC ^{13}C pulsed in the water



Detection of microfractures: 1. method sensitivity study



Detection of microfractures: 2. method sensitivity study



Water absorbtion in oil

Conclusion – part 2

1. Using IRMS and isotopic tracing is ossible to identify microfractures leading to faluure over a typical period od 10 years in a few weeks;

2. An isotopic traced hyperbaric chamber (d=0.6 m h=1.3 m) will be installed at CIRCE for real size experiments and quality check.

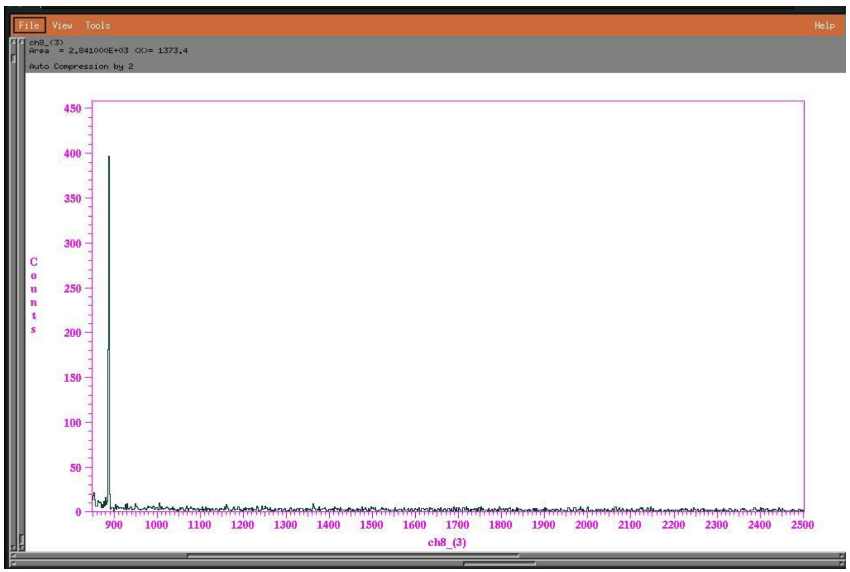
Water diffusion in solids

PIGE and NRA

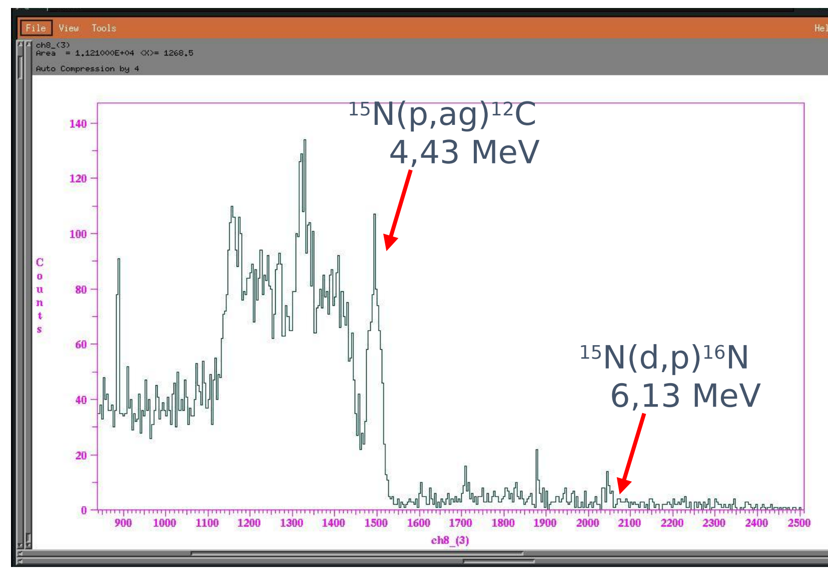
Water diffusion in solids

Test on plastics and glasses

Beam induced background
Runtime 5616 s

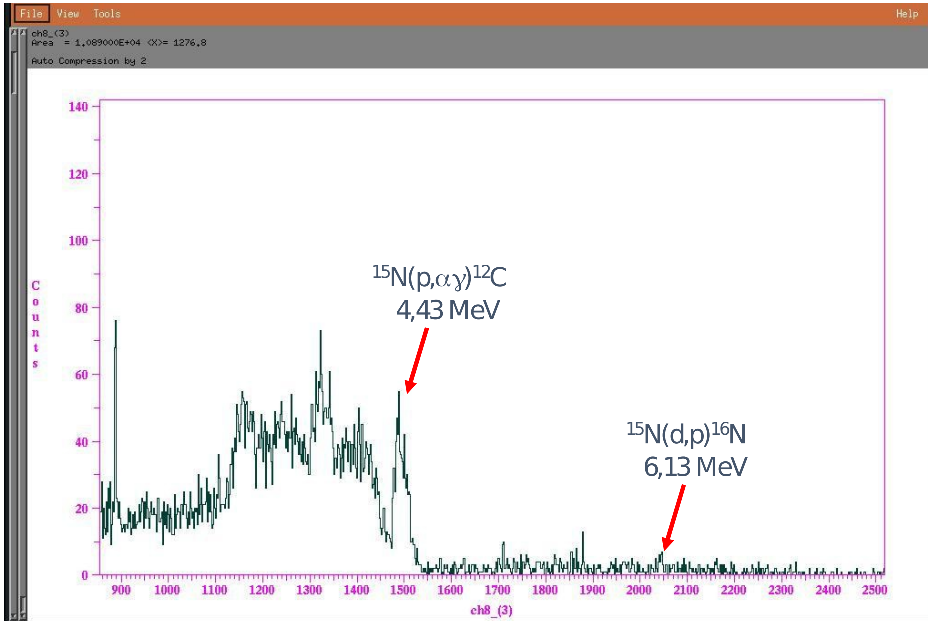


Evonik – Plexiglass
Runtime 900 s

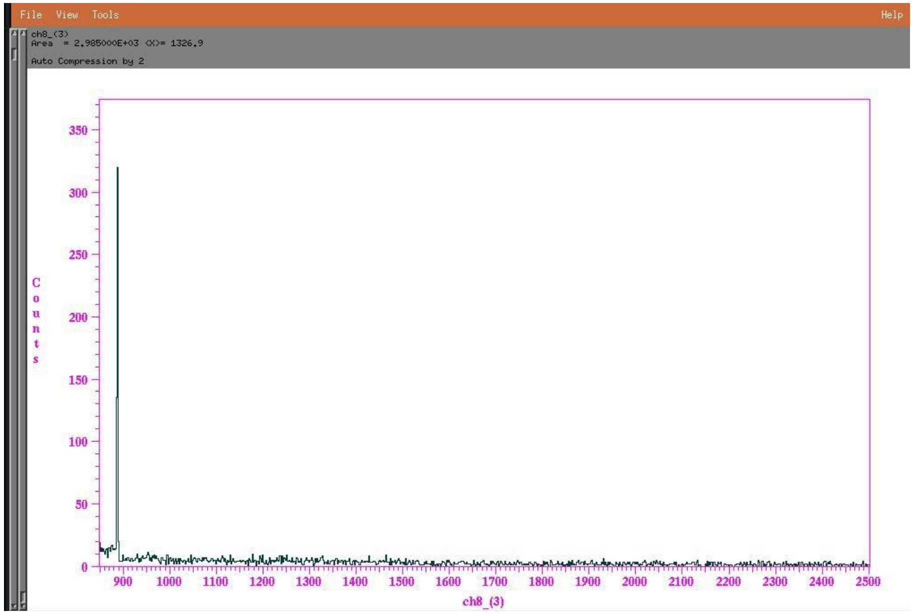


Water diffusion in solids

BOB plastic
Run time 900 s

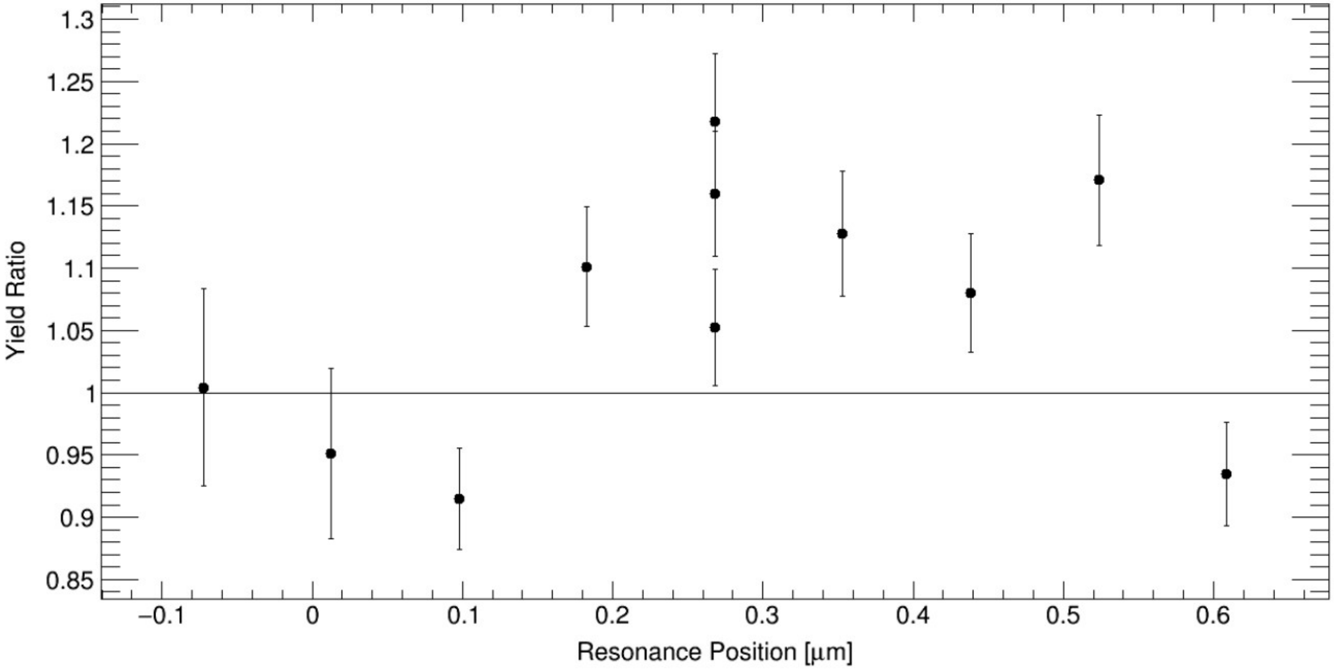


DOM Glass
Runtime 4320 s



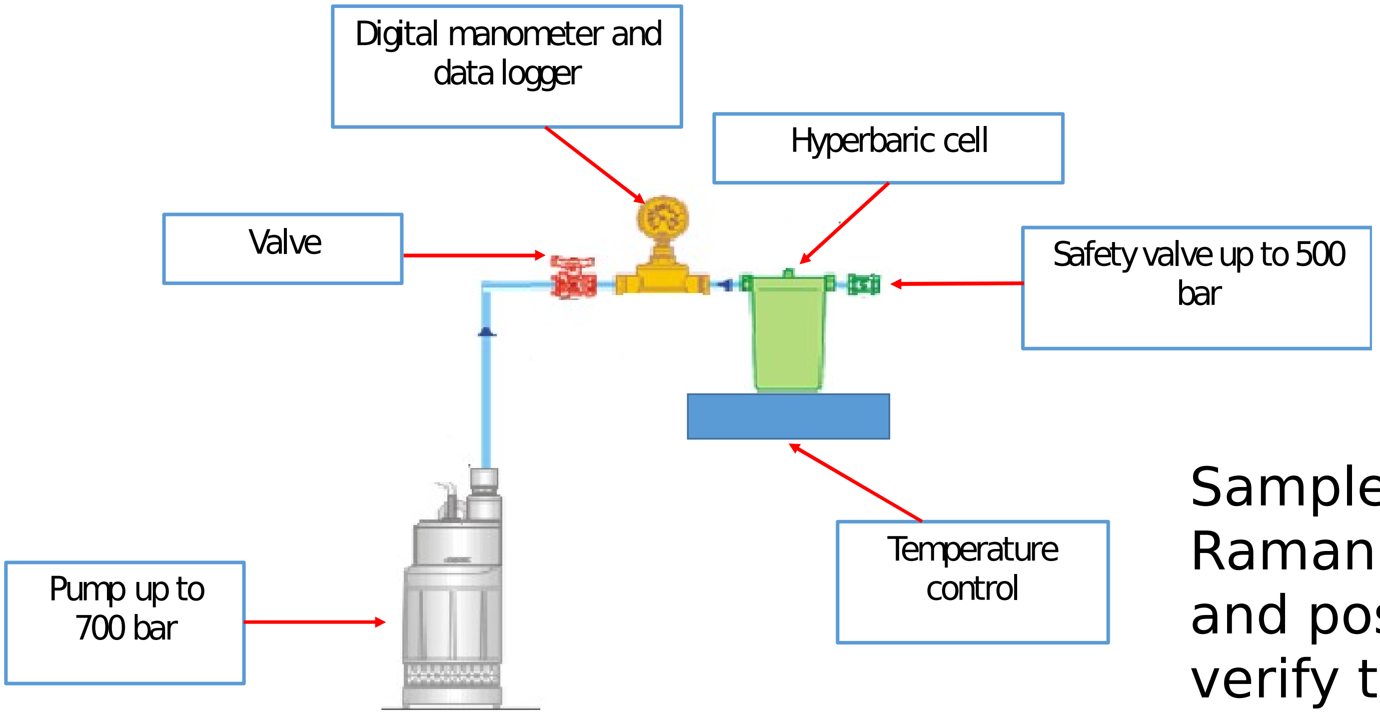
Water diffusion in solids

Measurements on VEOC material: Ratio of yields ARCA-DU3-to-NEW



Water diffusion in solids

Layout of the hyperbaric cell capable to simulate real water infiltration conditions and isotopic anrichment.



Samples will undergo Raman spectroscopy pre and post irradiation to verify their integrity

Conclusion - part 3

1. Null γ background was observed for both $^{15}N(p,\alpha\gamma)^{12}C$ and $^{15}N(d,p)^{16}N$ for the measurement system and DOM glass in the energy range of interest;
2. γ peak appeared for the $^{15}N(p,\alpha\gamma)^{12}C$ @ 4.43 MeV and for $^{15}N(d,p)^{16}N$ @ 6.13 MeV. Being the first significantly higher than the second;
3. These observations indicates how water infiltration rates in glass can be studied with natural water exposures while a greater sensitivity can be obtained by means D_2O on plastic samples.

Conclusion

Isotopic and nuclear methodologies represent ultrasensitive techniques that allow investigating materials and devices over a reasonably short time period gathering enough information to reliably predict the long term behavior