Identifying and Quantifying Water Species in Spacecraft Materials using QTGA Analysis: Implications for Molecular Contamination Modelling and Bake-Out Efficiency

Contamination, Coatings, Materials and Planetary Protection Workshop 2023

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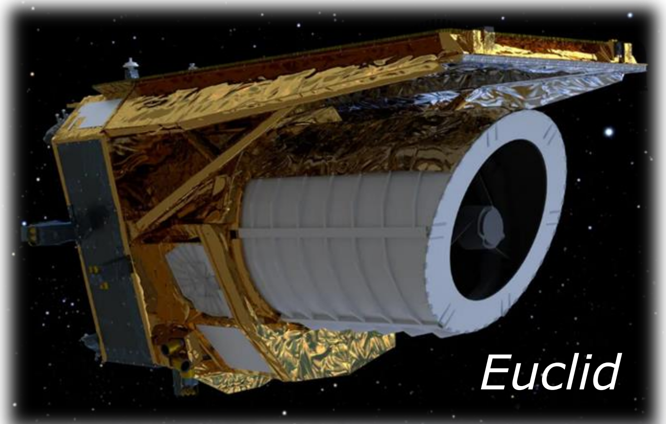
Motivation

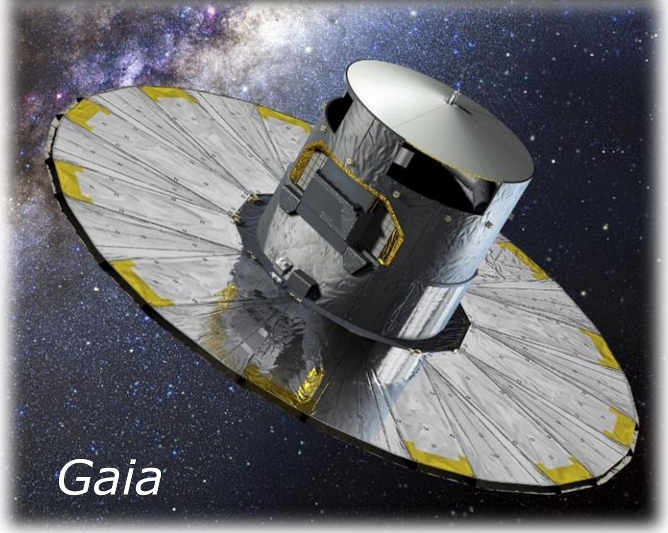
Water is the most common contaminant for spacecraft operating in near-cryogenic conditions (e.g. Euclid, Gaia). Common materials that act as carriers of water are multilayer insulation materials (MLI), honeycomb structures (CFRP) and paints.

Understanding the outgassing effects is critical to take necessary mitigation measures, e.g. prepare decontamination plans. Euclid's decontamination phase lasts 18 days (without subsequent calibrations)!

Water as a contaminant behaves differently than organic contamination. Water re-absorption on ground is unavoidable and cleaning/bakeout is not as useful as in case of organic contamination.

It is important to estimate the water/contaminant ratio of each material prior to mission launch to truly understand the contamination effects during the entire mission lifetime.





QTGA analysis to distinguish water contamination

Dynamic outgassing testing allows for estimation of contamination source's outgassing potential and its temperature dependence. Contamination effects significantly vary depending on surface temperatures.

We propose a semi-empirical approach to differentiate fast outgassing (short residence time) from slower outgassing (longer residence time) using a post-processing technique acquired from the decontamination data using an additional step during standard testing.

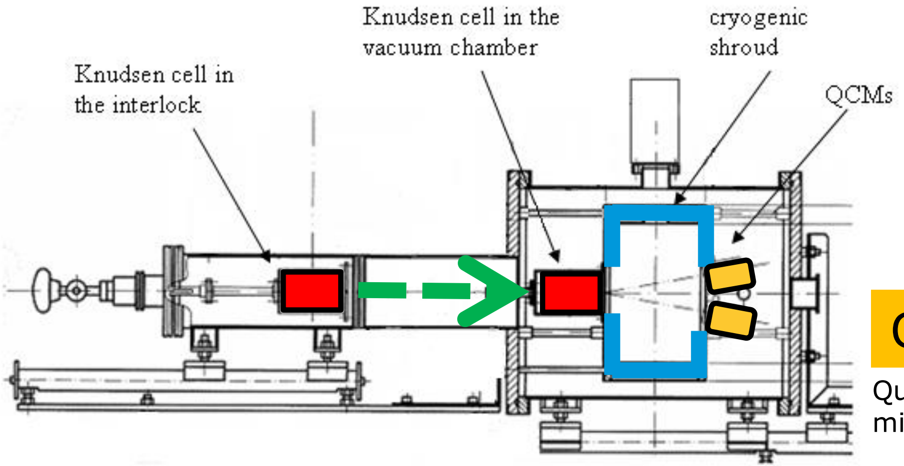
1. Dynamic outgassing test, with at least two QCMs:
   - Cryo QCM: Operating at -175°C
   - Cold QCM: Operating at -75°C
2. QTGA analysis after each outgassing step
3. Assigning specific peaks to two groups: 1) water and 2) contaminants
4. Calculation of water/contaminants ratio
5. Comparison with ex-situ measurements (microbalance, FTIR, RGA, GSMS)

Materials tested

1. Acktar Fractal – black inorganic coating: common coating for optical baffles
2. Norcoat – thermal protection material
3. CFRP assembly – structural material (complex geometry)

Step 1: Dynamic outgassing test

Sample temperature is increased from 25°C to X°C by steps of 25°C every 24h. The number of steps and the duration at the max. test temperature is adjusted based on the real application temperature and the max. permitted test temperature of the sample.



QCMs

Quartz crystal microbalance

KC

$T_{max} \cong 450°C$

Liquid N_2

$\bar{T} \cong -175°C$

Standard: ECSS-Q-TM-70-52A

$$P < 5 \times 10^{-6} mbar$$

$T_{QCM,1} \cong -170°C$ } **TML** Total mass loss

$T_{QCM,2} \cong -25°C$

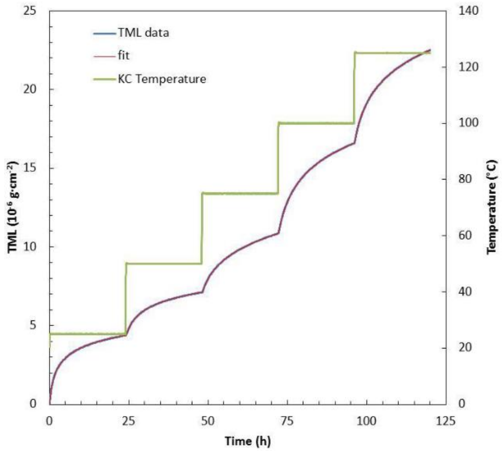
$T_{QCM,3} \cong -75°C$

$T_{QCM,4} \cong -50°C$

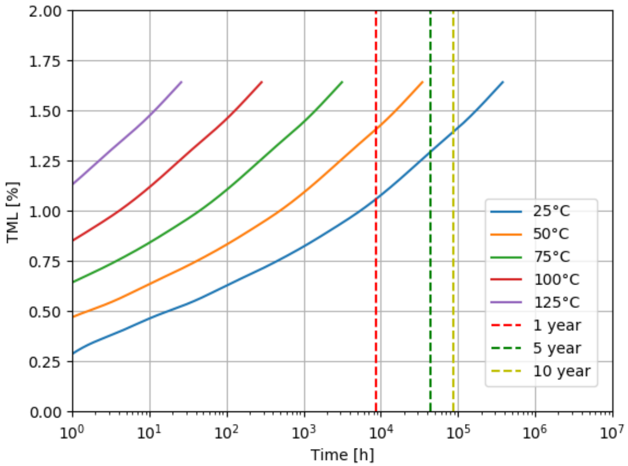
} **CVCM** Collected Volatile Condensable Material

Parameters generated by standard outgassing test

Long-term outgassing modelling is based on a step-wise dynamic outgassing test. The experimental data collected over few days is extrapolated to many year mission through a power



Conversion of accelerated test results to long-term outgassing predictions



1) Determine from one step to another the acceleration outgassing rate as temperature is increased
2) Establish the temperature dependence of the acceleration factors
3) Extrapolate the time, from each outgassing step to desired prediction temperature
4) Standard approach is lacking a focus on reemission behaviour of contaminants

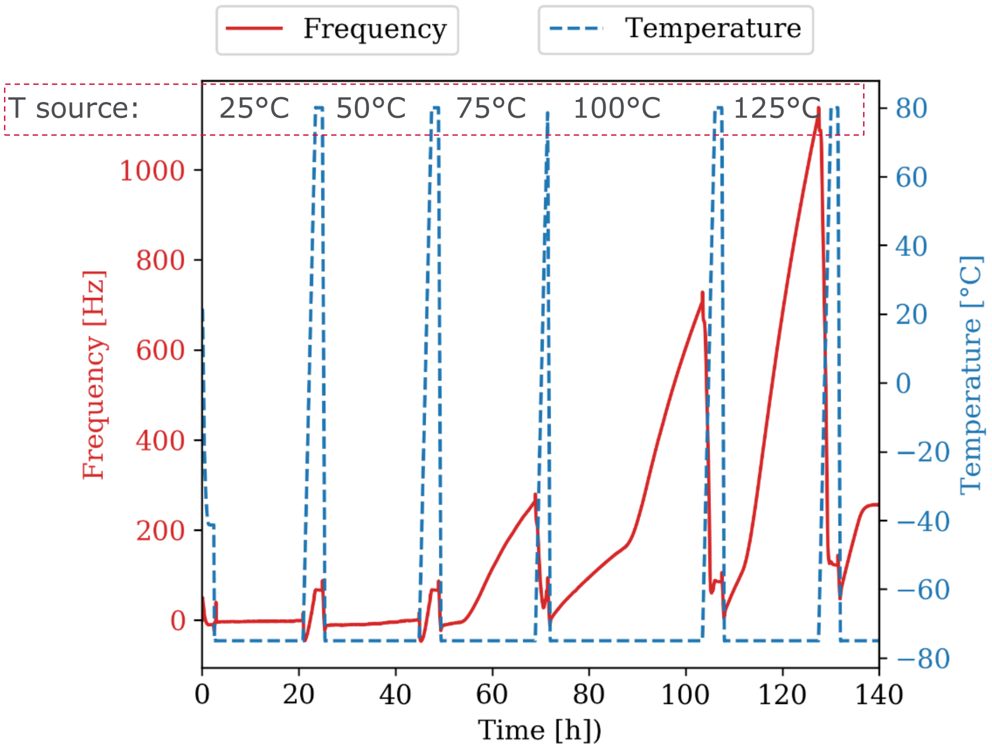
$$\text{TML}\ (T) = \sum_i W_{0,i} \cdot (1 - e^{-t/\tau_{T,i}})$$

$W_{0,i}$ – initial mass derived experimentally

$\tau_{T,i}$ - time constant derived experimentally

Step 2: Modified standard outgassing test

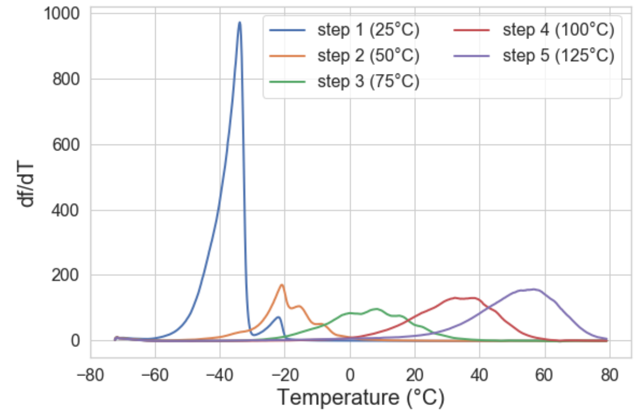
Decontamination of a QCM set to specific temperature, after every outgassing step



- Standard as per:

Kinetic outgassing of materials for space ECSS-Q-TM-70-52A, ESA-ESTEC (2011)

- QTGA analysis performed on the QCM at -175°C and -75°C
- QTGA performed after each outgassing step (T_{source} = 25,50,75,100,125°C)
- Reemission step presented as 1st derivative (peaks)



Step 3: Assigning peaks

Residence times of water at cryogenic temperatures is assumed infinitesimally long compared to cold conditions (-75°C) -> We are working with an assumption that water is only recorded by the cryoQCM.

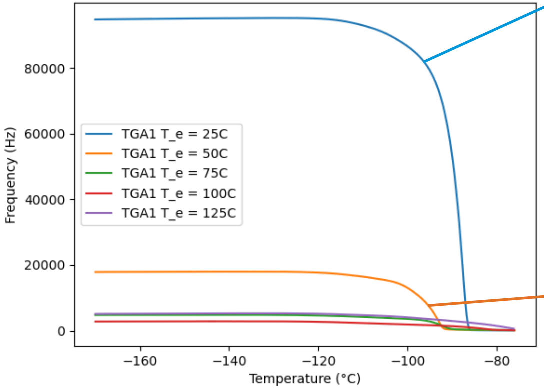
$$\tau_{water}(T) = \tau_0 * e^{\frac{E_a}{RT}}$$

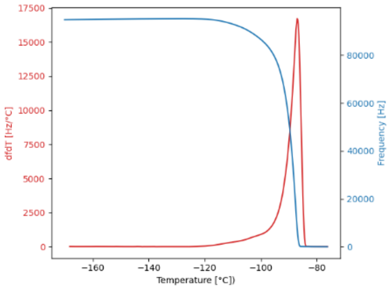
$$\tau_0 = 10^{-13}\ [\mathrm{s}],\ E_a = \frac{46kJ}{mol}$$

Outgassing rate per temperature:

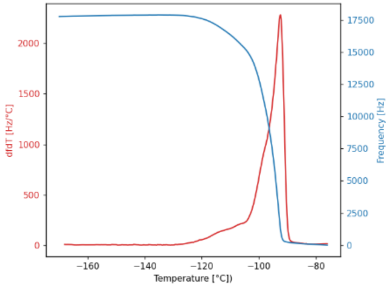
$$\tau_{water}(-175°C) = 10^{10}\ [\mathrm{s}]$$

$$\tau_{water}(-75°C) = 0.1\ [\mathrm{s}]$$





Outgassing step 25°C



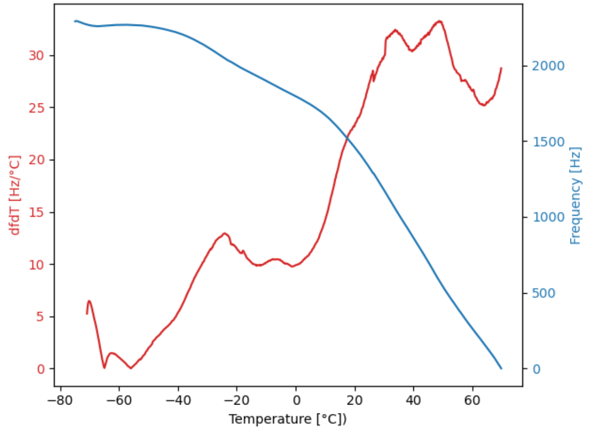
Outgassing step 50°C

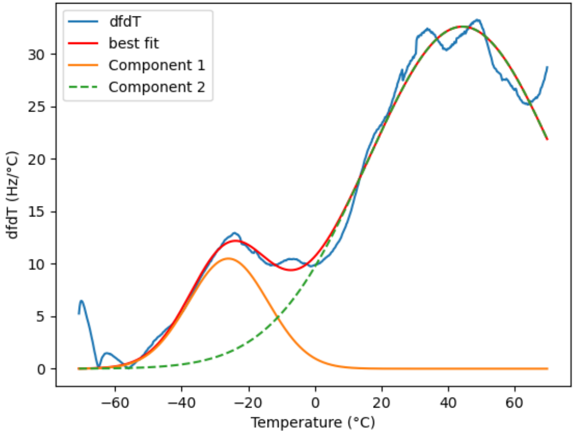
(Other steps not shown)

DTGA curve shows the maximum change per temperature and can be used to fit a series of pseudo-species which represent outgassing components.

Step 4: Calculation of water/contaminant ratio

1. Sum the peak areas of all peaks assigned to water
2. Sum the peak areas of all peaks assigned to a contaminant
3. Calculate the ratio

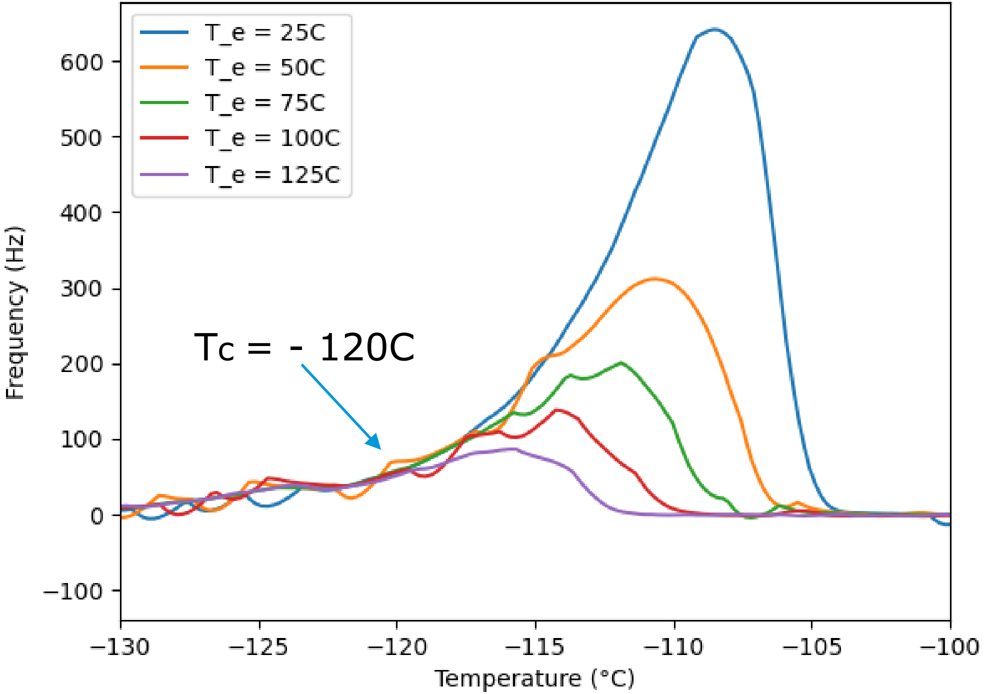




DTGA can be fitted with several peaks, representing chemical species. Note: these are pseudo species, the fit is a mathematical operation.

Simple case: Acktar Fractal Black – only water contamination

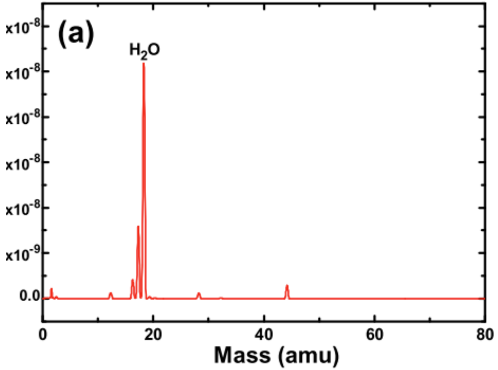
Emission temperature of the outgassing source influences the reemission behaviour of water, but the captation temperature (Tc) is constant.



Increasing the emission temperature of the outgassing source shifts the water peak towards lower temperatures. This is probably an effect of multiple deposited layers reemiting one after another.

Supporting information for the peak to represent water:

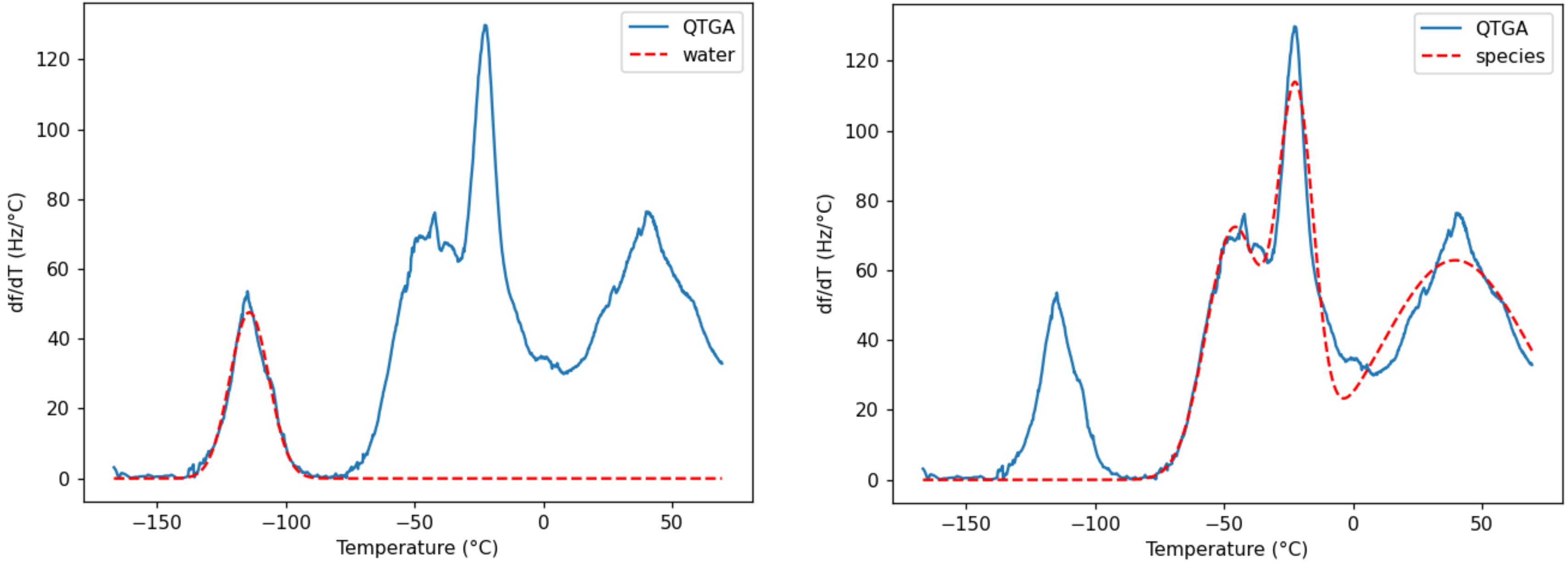
1) No deposit on QCM at −75°C
2) RGA analysis for this material



Source: Salomon, Y. M., Gouzman, N. A. S. I., & Grossman, G. L. E. (n.d.). QUALIFICATION OF ACKTAR BLACK COATINGS FOR SPACE APPLICATION . 1, 2–8.

More complicated: water/contaminants mixture

Data below is showing QTGA curve obtained at the end of the outgassing test (after all 5 outgassing steps). For the tests operating at pressures <10-5 Torr, it can be assumed that water sticks completely below -100°C, so we assign that peak to water. All other peaks above −100°C are assigned to a contaminant family.



Step 5: Comparison with ex-situ measurements

Comparison of the QTGA method with external microbalance to ensure reliable data acquisition and postprocessing.

3 very different materials analyzed:

	QTGA	Microbalance
Sample: Acktar Fractal Black		
Water	**100%**	**99.9%**
Contaminant	**0%**	**0.01%**
Sample: Norcoat		
Water	**86%**	**85%**
Contaminant	**14%**	**15%**
Sample: CFRP		
Water	**80%**	**70%**
Contaminant	**20%**	**30%**

Complex geometry error?

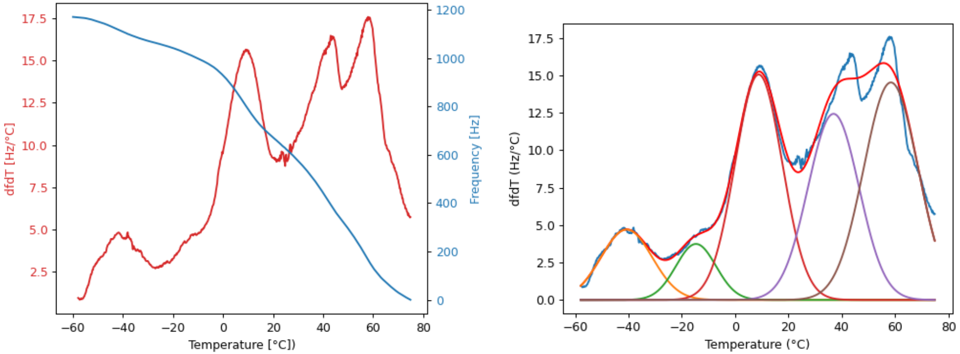
Results from the QTGA analysis are in a very good agreement with the ex-situ measurement using microbalance. Also, the microbalance measurement confirms that all outgassed water is reabsorbed.
We suspect that complex geometry of the test specimen can introduce error.

Step 5: Comparison with ex-situ measurements

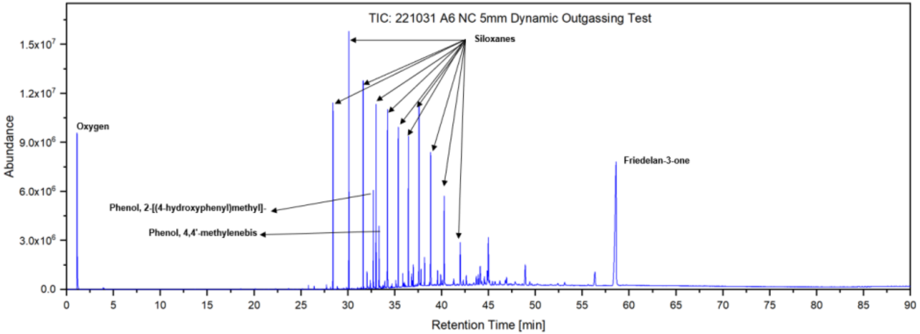
Complementary methods, such as FTIR, GCMS reveal the nature of remaining contaminants.

What is the remaining organic contamination?

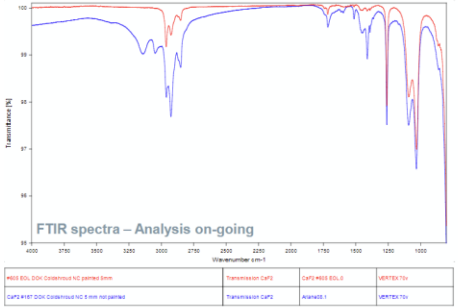
	QTGA	Microbalance
	Sample: Norcoat	
Water	**86%**	**85%**
Contaminant	**14%**	**15%**



QTGA analysis shows several species (at least 5) reemitting in a broad temperature range.



GSMS confirms that most of the contaminant is inseparable siloxanes with varying molecular weight.



FTIR also confirms that most of the contamination is of silicone origin.

Summary

Results of the presenting testing approach can be used to:

- Better understand the dynamics of the contamination process (What is the amount of water escaping into space through a certain aperture?)
- Break down the outgassing process to basic physics and supply data to water transport modelling between surfaces (Input for a simulation software)
- Simplify contamination assessment for missions where water is not a contaminant of concern (Using the sicking coefficient approach and subtracting TML of water, CVCM levels can be more accurately forecasted. Not covered here please see reference [1].
- The assessment of vacuum bakeout's effectiveness in mitigating or avoiding risks can be improved. It is also possible to calculate the efficiency of a bakeout process.

[1] Suliga, A., Ergincan, O., & Rampini, R. (2021). Modeling of Spacecraft Outgassed Contamination Levels by Thermogravimetric Analysis. Journal of Spacecraft and Rockets, 1–7. https://doi.org/10.2514/1.a35020