Outgassing of Space Materials at Low Temperature

CCMPP-2021

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CNES

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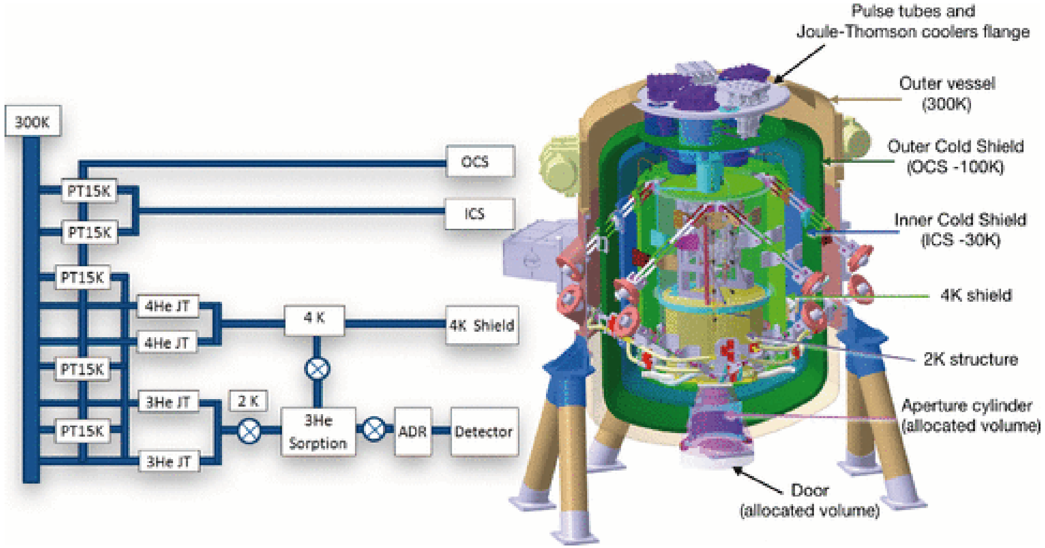
Table of contents

Introduction

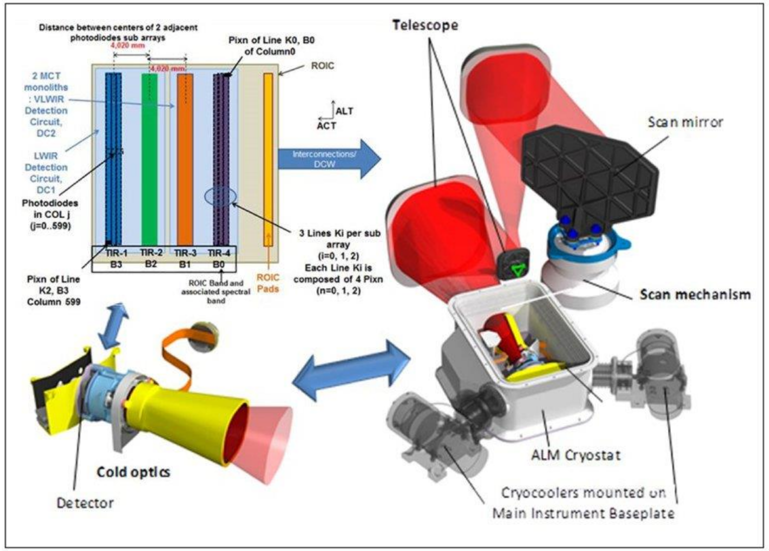
Increase of cryogenic missions

Cryogenic temperature ➔ sensitive surfaces ➔ performance affected by contamination



The Athena X-ray Integral Field Unit (X-IFU)

Pajot, F., Barret, D., Lam-Trong, T. *et al.*, The Athena X-ray Integral Field Unit (X-IFU). *J Low Temp Phys* 193, 901–907 (2018)



Trishna

Buffet . L, Gamet P., Maisongrande P. *et al.*, The TIR instrument on TRISHNA satellite: a precursor of high resolution observation missions in the thermal infrared domain, *Proc. SPIE 11852,* International Conference on Space Optics - ICSO 2020, 118520Q (2021)

MicroCarb, MTG, CO2M, LSTM, CHIME and others…

Outgassing at cryogenic temperature ➔ intuition to be negligible ➔ no experimental data

Introduction

Objectives: to quantify the outgassing at cryogenic temperature

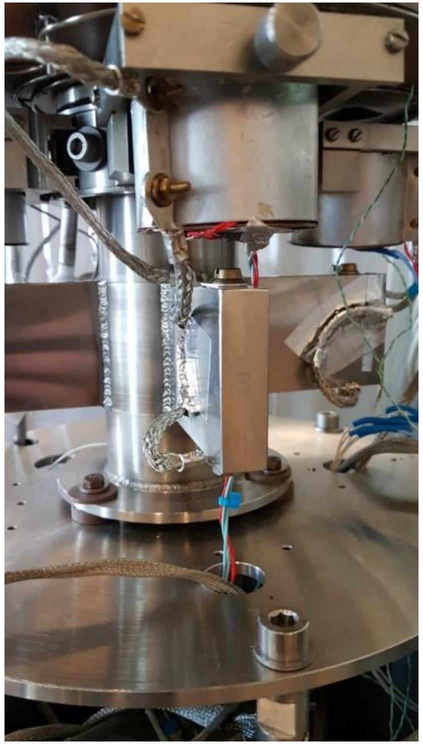
Optimization of CNES facilities

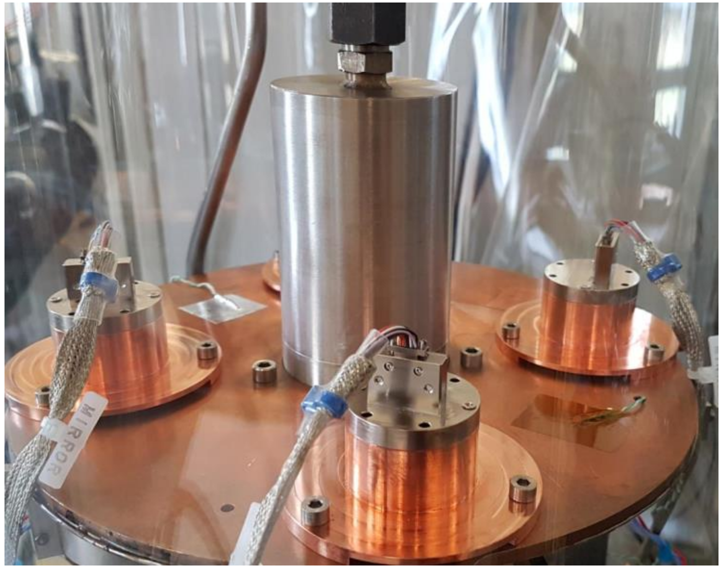
Measurements of outgassing kinetics of widely used space materials → Cooling of materials, protocol modification…

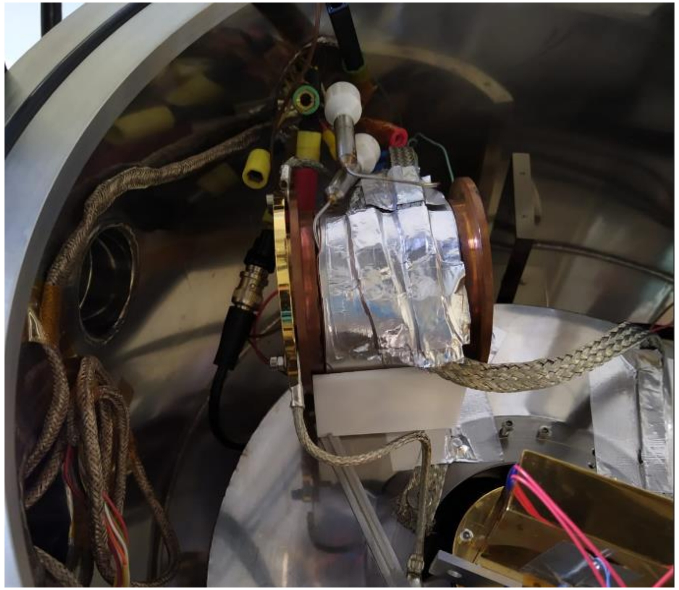
Identification of the chemical nature of outgassed molecules → RGA analysis, modification of oven location…

Prediction of outgassing at low temperature → Fitting with Arrhenius law…

Optimization of CNES facilities



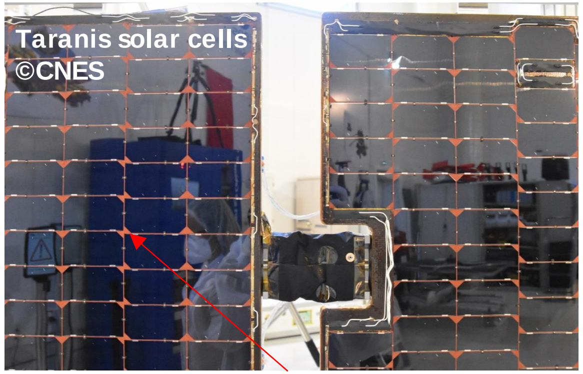




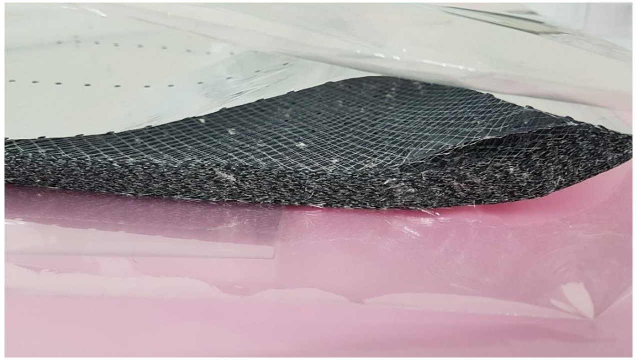
Optimization of thermal control to cool down the oven at cryogenic temperature

Optimization of oven temperature and location
➔ RGA sensitivity

Materials and protocol



RTV-S 691 adhesive



Cryogenic MLI (52 sheets + PE spacers)

Parameters:

- About 12 g of material
- NDK® CQCM, S = 2.38.10^8 (Hz/g)/cm²
- T_{CQCM} = -165 °C
- $T_{material}$ = -75 °C to 25 °C

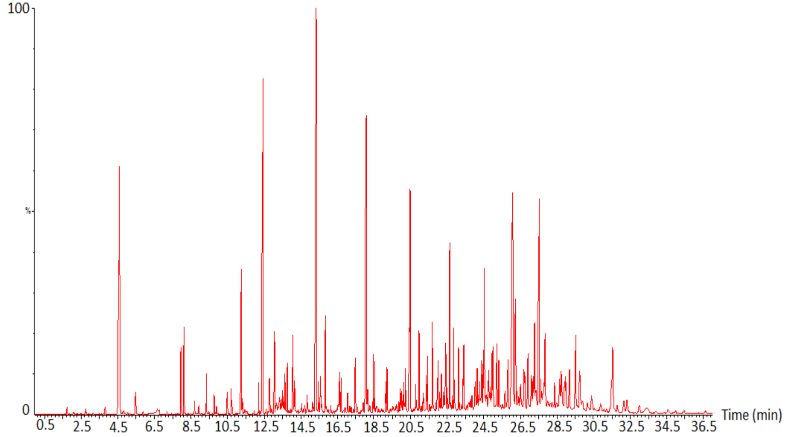
Protocol:

- Samples are loaded into ovens
- GN_2 flushing during a certain duration then LN_2 cooling at ambient pressure
- At a certain temperature, vacuum system was switched on
- Cooling till -165 °C
- Start of the temperature stages and RGA analysis

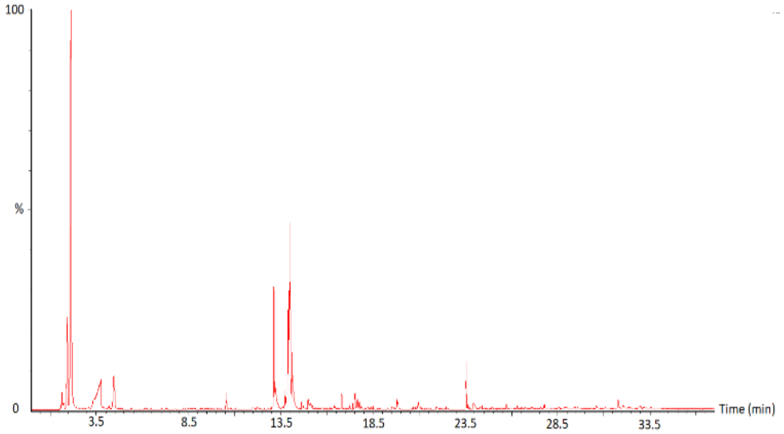
TDGCMS

Gas chromatography (GC) analysis performed with a Clarus 500 chromatograph (PerkinElmer Elite 5-MS column with dimensions of 60 m × 0.25 mm × 0.25 µm) coupled to a TurboxMatrix 300 TD thermal desorber (TD) and a Clarus 500 mass spectrometer (MS)

Parameters: 200 °C, solvent delay of 1 min, ambient pressure



RTV-S 691 adhesive chromatogram



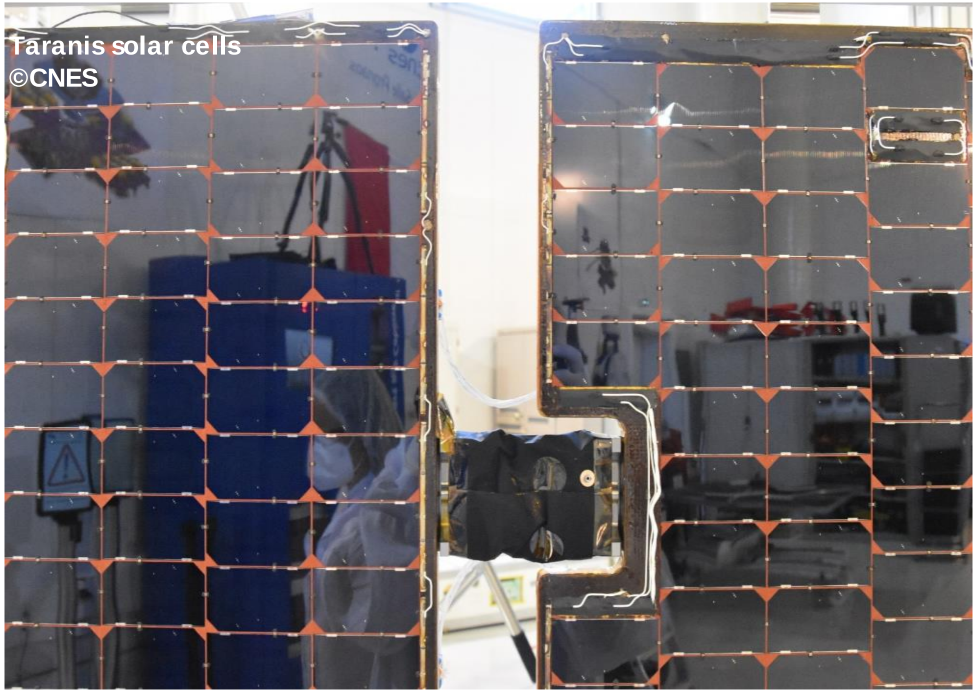
Cryogenic MLI chromatogram

- benzene at retention time (tr) = 4.6 min
- toluene (tr = 7.9 min)
- cyclic and linear siloxanes (tr = 12.4, 15.3, 18.1, 20.5, 26.1, 27.5 min, and so on)

- benzoic acid, phenyl ester (tr = 23.5 min)
- alcohols such as 1-heptanol, 6-methyl- (tr = 13.9 min)

Measurements

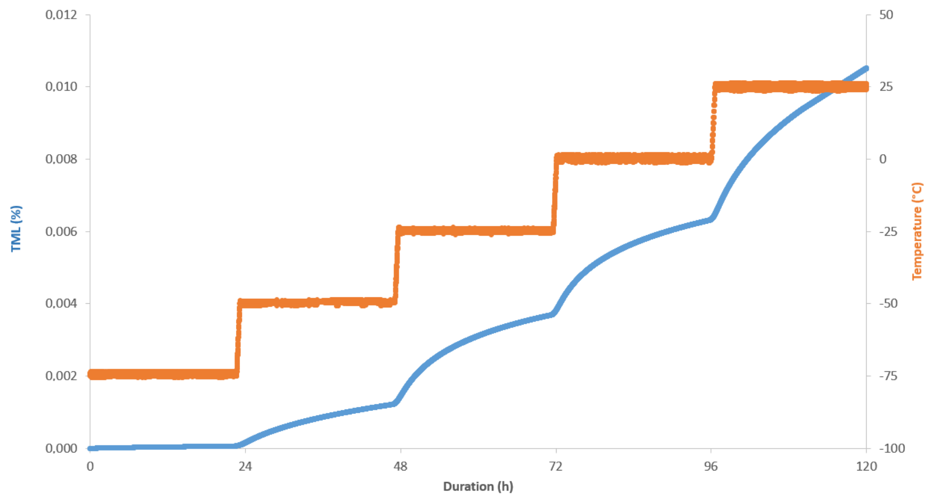
RTV-S 691 adhesive

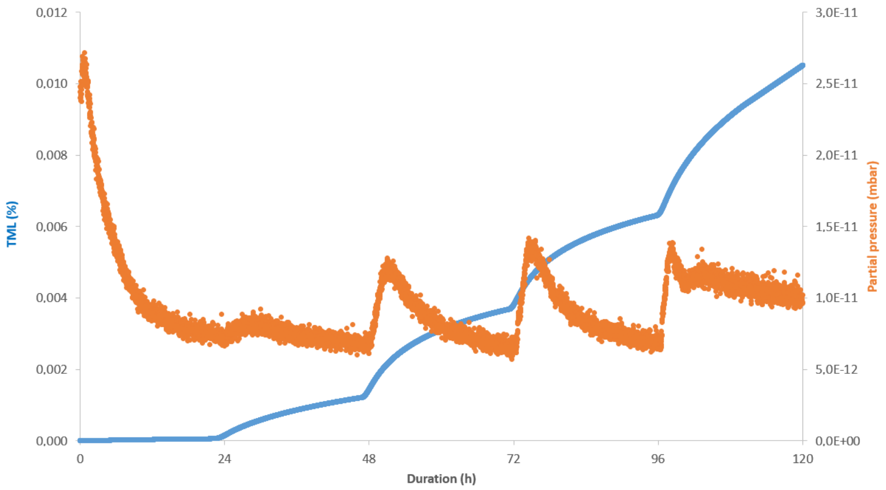


Measurements

RTV-S 691 adhesive: outgassing and RGA analysis (water desorption)

12 g of material, $T_{RTV\text{-}S691}$ = -75°C to 25 °C, T_{CQCM} = - 165 °C





Outgassing starts from -50 °C (0.001 %)

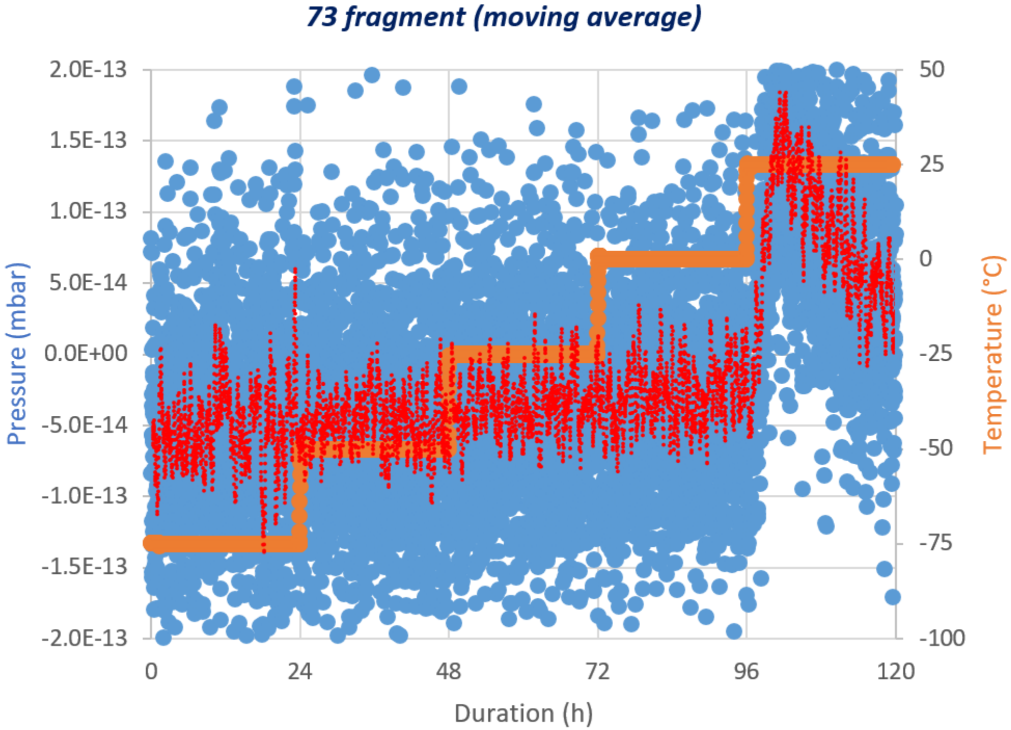
Water starts to desorb from -50 °C and regularly up to 25 °C

$\Delta P_{18\_-50°C} = 2.0.10^{-12}$ mbar ; $\Delta P_{18\_-25°C} = 6.0.10^{-12}$ mbar ; $\Delta P_{18\_0°C} = 7.5.10^{-12}$ mbar ; $\Delta P_{18\_25°C} = 7.0.10^{-12}$ mbar

Measurements

RTV-S 691 adhesive, RGA analysis

Siloxane fragment: SiC_3H_9



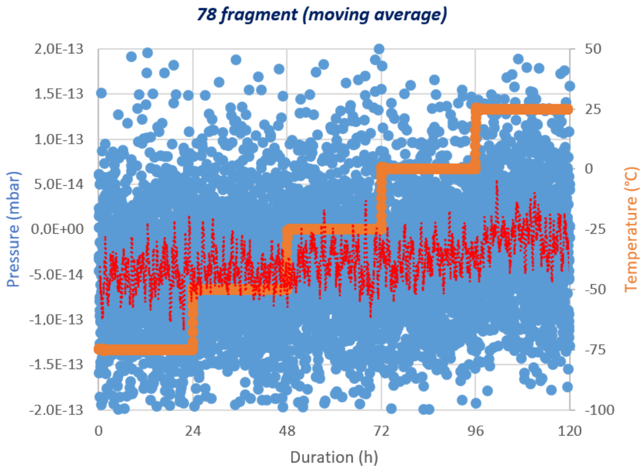
Silicone fragment might start to desorb from 0 °C but significantly from 25 °C

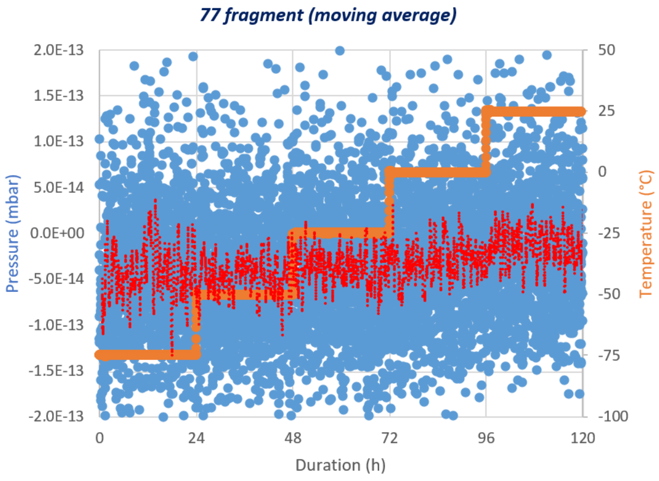
$\Delta P_{73\_25°C} = 2.5.10^{-13}$ mbar

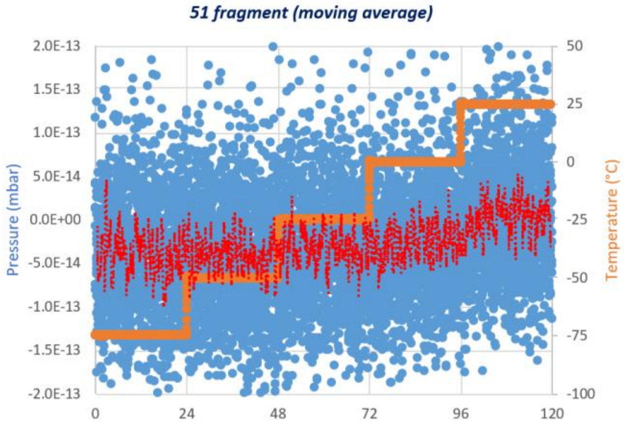
Measurements

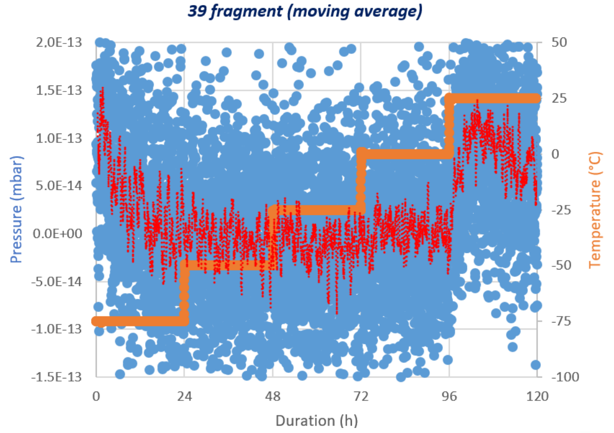
RTV-S 691 adhesive, RGA analysis

<u>Benzene fragments:</u> C_6H_6, C_6H_5, C_4H_3, C_3H_3









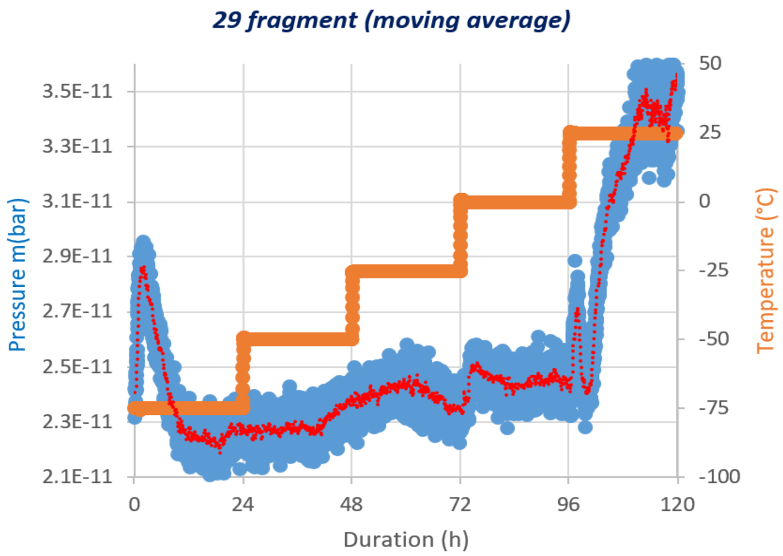
Benzene fragments might start to desorb from 0 °C

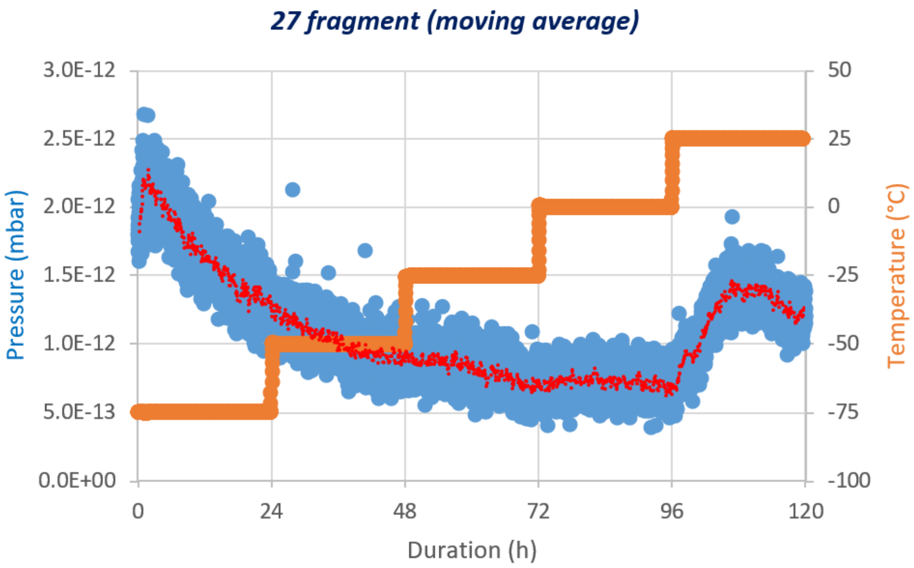
$\Delta P_{39\_25°C} = 1.5.10^{-13}$ mbar

Measurements

RTV-S 691 adhesive, RGA analysis

C_xH_y fragments: C_2H_5, C_2H_3



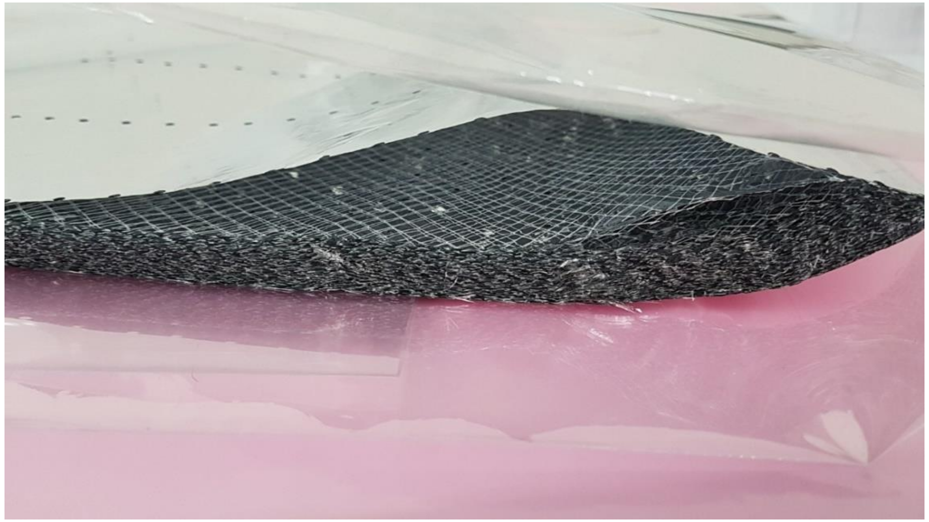


Low mass C_xH_y fragments start to desorb from -75 °C but significantly from 25 °C

$\Delta P_{29\_-75°C} = 5.5.10^{-12}$ mbar ; $\Delta P_{29\_25°C} = 1.1.10^{-11}$ mbar ; $\Delta P_{27\_25°C} = 1.5.10^{-12}$ mbar

Measurements

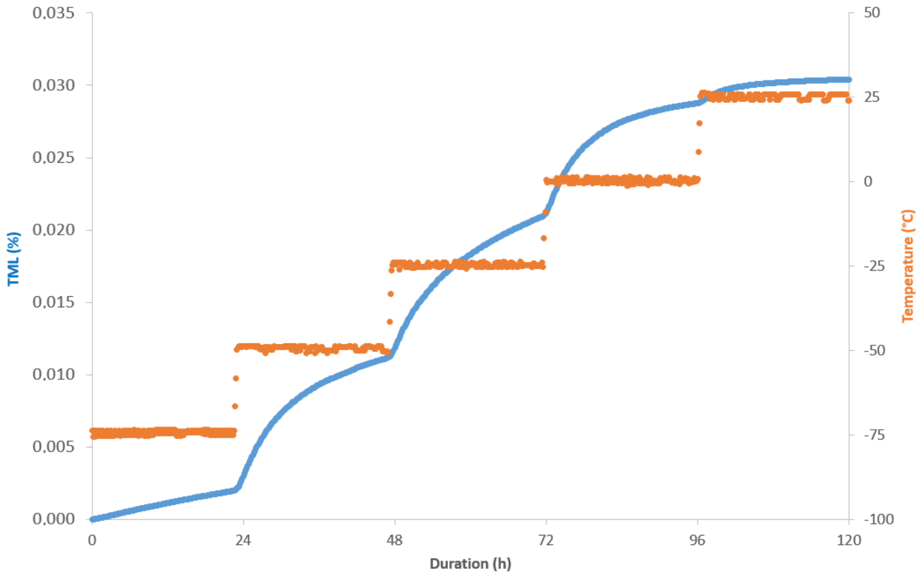
Cryogenic MLI

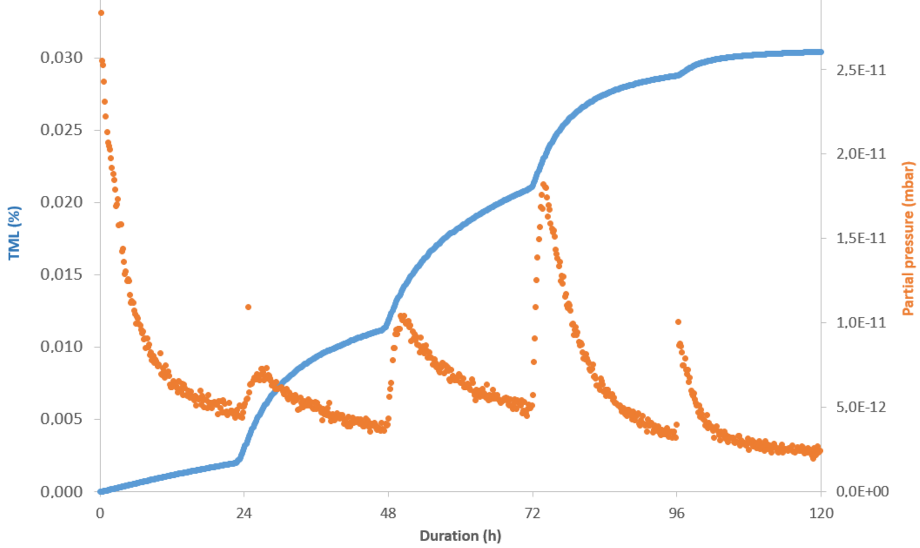


Measurements

Cryogenic MLI: outgassing and RGA analysis (water desorption)

12 g of material, T_{MLI}= -75°C to 25 °C, T_{CQCM} = - 165 °C





Outgassing starts from -75 °C (0.002 %)

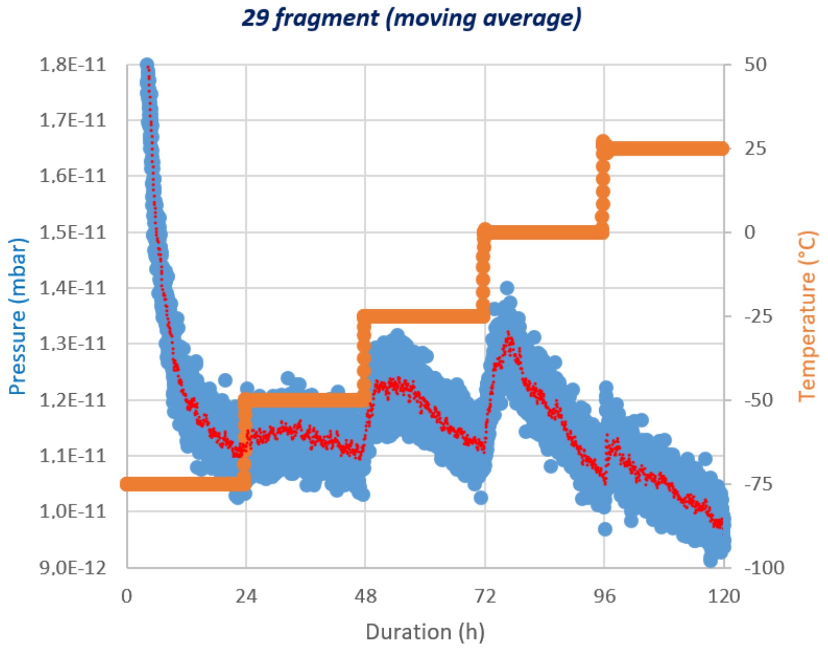
Water starts to desorb from -50 °C and regularly up to 25 °C

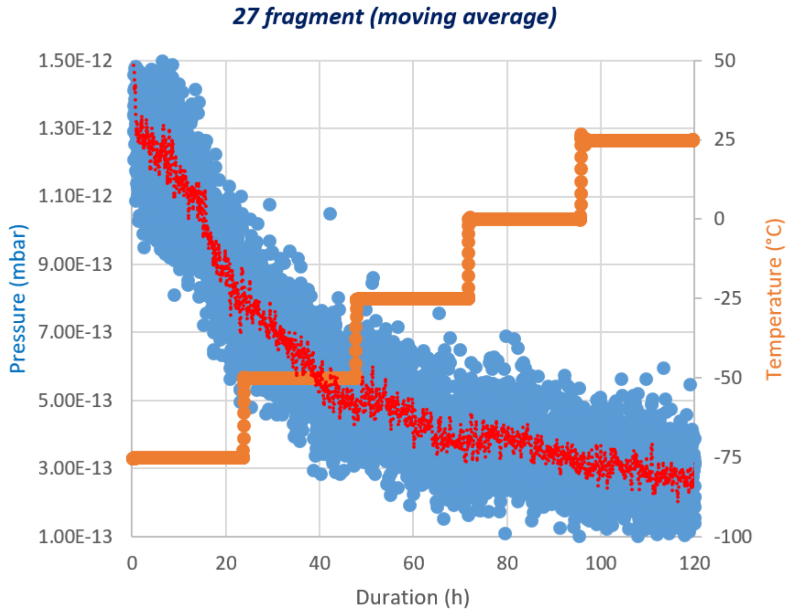
$\Delta P_{18\_-50°C}$ = 2.0.10^{-12} mbar ; $\Delta P_{18\_-25°C}$ = 6.0.10^{-12} mbar ; $\Delta P_{18\_0°C}$ = 12.0.10^{-12} mbar ; $\Delta P_{18\_25°C}$ = 7.0.10^{-12} mbar

Measurements

Cryogenic MLI, RGA analysis

C_xH_y fragments: C_2H_5, C_2H_3





Low mass C_xH_y fragments start to desorb from -50 °C

$\Delta P_{29\_-50°C} = 5.0.10^{-13}$ mbar ; $\Delta P_{29\_-25°C} = 1.0.10^{-12}$ mbar ; $\Delta P_{29\_0°C} = 2.0.10^{-12}$ mbar

Fitting

LP/VB5E ➔ computation, simulation and database

- Computation part ➔ first order desorption law, consideration of 5 outgassed chemical species with decreasing volatility, linear temperature slope

$$\frac{d\mu_{(t)}}{dt} = k_{i(t)}\left(\mu_i - \mu_{i(t)}\right) \text{ with } \mu_{i(t0)} = 0$$

$$\mu_{(t)} = \sum_{i=1}^{n} \int_{t_0}^{t} \left(\mu_i - \mu_{i(t)}\right).\left(1 - e^{k_{i(t)}\mathrm{dt}}\right)$$

$$k_{i(t)} = A_i\, e^{\frac{-E_i}{RT(t)}}$$

➔ to find the coefficients (μ, E, A)1 to 5 from the multi-step test

- Simulation part enables to simulate outgassing graphs for a given material using coefficients (μ, E, A)1 to 5 with different temperature profiles
- Database ➔ to be re-used for other computation

Fitting

Coefficient values after fitting of each experimental curve for cryogenic MLI and RTV-S 691 adhesive

		Cryogenic MLI	RTV-S 691 adhesive
Exponential 1	μ (%)	1.10^{-3}	0
	E (kcal / mol)	1.6	1
	A (1 / s)	3.10^{-3}	9.10^{-4}
Exponential 2	μ (%)	9.10^{-3}	1.10^{-3}
	E (kcal / mol)	15	17
	A (1 / s)	2.10^{10}	2.10^{12}
Exponential 3	μ (%)	10.10^{-3}	2.10^{-3}
	E (kcal / mol)	17	19
	A (1 / s)	7.10^{10}	1.10^{13}
Exponential 4	μ (%)	8.10^{-3}	3.10^{-3}
	E (kcal / mol)	18	21
	A (1 / s)	3.10^{10}	3.10^{12}
Exponential 5	μ (%)	3.10^{-3}	6.10^{-3}
	E (kcal / mol)	18	21
	A (1 / s)	2.10^{9}	8.10^{10}

Fitting is pretty good ➔ same order of magnitude for μ (cf. experimental data)

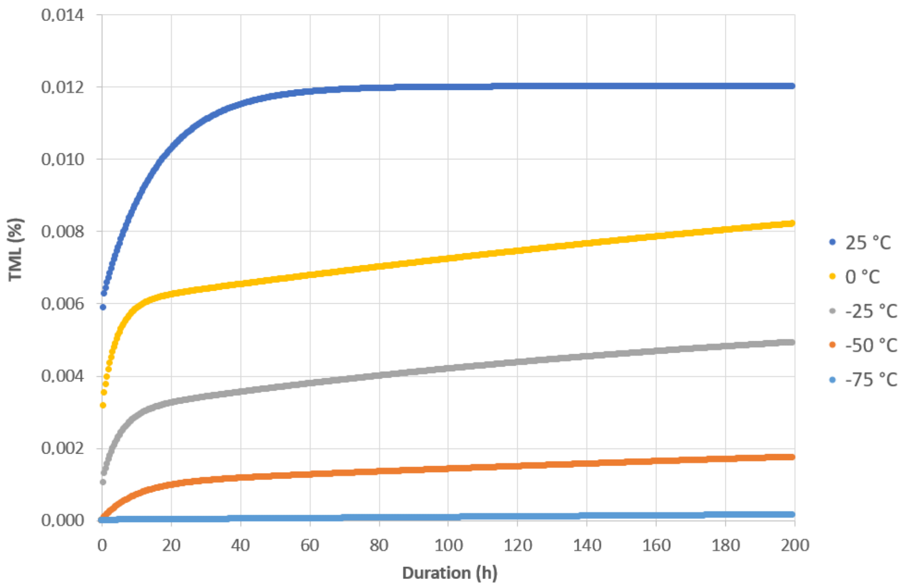
Activation energy below 20 kcal/mole ➔ desorption (< 10 kcal / mol) and diffusion (< 15 kcal / mol)

John J. Scialdone. (1982), Characterization Of The Outgassing Of Spacecraft Materials, Proc. SPIE 0287, Shuttle Optical Environment.

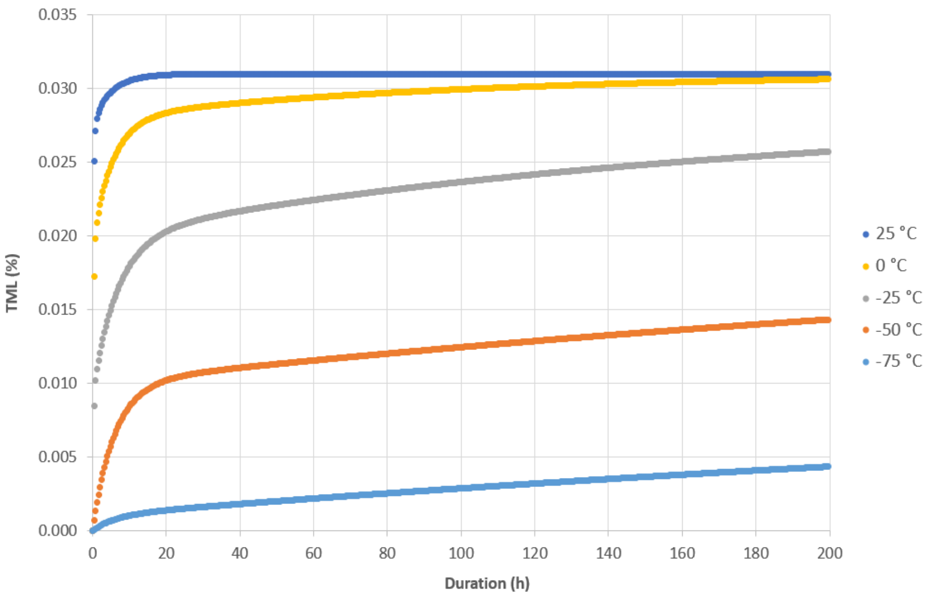
Prediction

Prediction from coefficients (μ, E, A)1 to 5 found with fitting

RTV-S 691 adhesive



Cryogenic MLI



Outgassing is negligible at -75 °C but starts at -50 °C
Models might need to be reworked

Conclusion

- CNES facilities were modified and are now ready to perform tests for different materials at cryogenic temperature.
- Outgassing starts at -75 °C (mainly water) and at -50 °C for some small solvent fragments but outgassing remains dominated by water desorption.

Temperature	-75 °C	- 50 °C	- 25 °C	0 °C	25 °C
TML for RTV-S 691 adhesive (%)	0	0.001	0.002	0.003	0.004
TML for cryogenic MLI (%)	0.002	0.01	0.01	0.007	0.001

- RGA data processing could be improved to decrease S/R ratio.
- Models and predictions need to be reworked and validated at low temperature to be realistic.
- Some materials have already been tested (epoxy glues, composite and other MLI) and each material exhibits its own behavior at low temperature.
- A paper is currently being written.

Thanks!