



Outgassing of space materials

Screening tests for COTS components proposed for space applications

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I. INTA-OUTGASSING LABORATORY, BRIEF OUTLINE





INTA Public Research Organism - Ministry of Defence

OUTGASSING LABORATORY

ESA -VALIDATED OUTGASSING TEST HOUSE → TEST SUPPLIER FOR AEROSPACE FIRMS

Created in 1970 in collaboration with ESRO -ESTEC MATERIALS SECTION (1968)

TESTS FOR EXTERNAL:



TESTS FOR INTERNAL:

INTA SPACE PROJECTS : RAMAN, ATHENA, MAPS MARS, SOPHI, NEWTON..

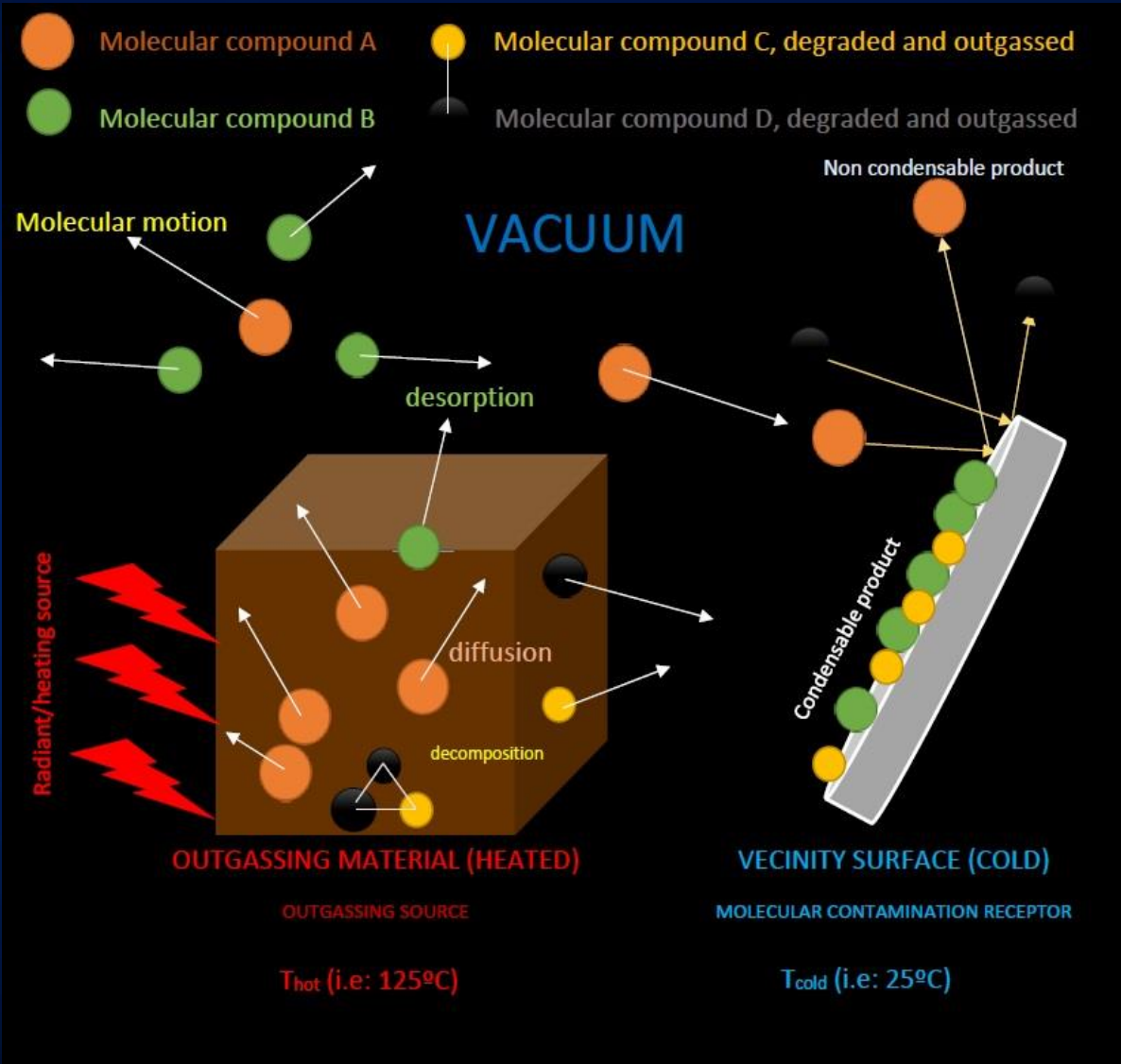
II. OUTGASSING EFFECT DESCRIPTION

What is Outgassing....

Release of volatile molecular compounds from a material, contained in it or adsorbed on the surface, due to the exposure to space vacuum environment. It is a process activated and accelerated with the increase of temperature. It is the main effect provoked by the extreme vacuum (from 10^{-5} - 10^{-7} mbar low orbits, i.e: ISS, to $< 10^{-13}$ mbar deep space)

Space environment:

- Temperature
 - Vacuum
 - Radiation
 - Space debris
 - Micrometeorites
 - Atomic Oxygen
- > OUTGASSING EFFECT.
(synergistic effect)



Outgassing mechanisms:

Mechanism	Time dep.	E_a (Kcal / mol)
Desorption ($T^a \uparrow$)	$t^{-1} - t^{-2}$	1 - 10
Diffusion ($T^a \uparrow \uparrow$)	$t^{-1/2}$	5 - 15
Decomposition ($T^a \uparrow \uparrow \uparrow$)	n/a	20 - 80

- **Desorption** : molecules adsorbed on the material surface.
- **Evaporation/sublimation**: change of physic state of material to gas phase.
- **Diffusion**: random motion within the material of concentrated molecular species.
- **Decomposition**: chemical reaction on material, generation of outgassing species \rightarrow diffusion \rightarrow desorption \rightarrow release to vacuum (outgassing).
- **Permeation**: molecular flux can permeate a material, diffuse, and later be released after to vacuum space.

Outgassing mechanisms, analytical approach

Desorption

$$\frac{dm^\alpha}{dt} = -\frac{m^\alpha}{\tau_d^\alpha(T)}$$

- with dm^α / dt : weight loss per surface unit of species α ($\text{g}\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$)
 m^α : contaminant α available mass per surface unit ($\text{g}\cdot\text{cm}^{-2}$)
 $\tau_d^\alpha(T)$: outgassing time constant of species α (s)
 T : temperature (K)

The desorption time constant is thermally activated (Arrhenius law):

$$\tau_d^\alpha(T) = \tau_{d0}^\alpha e^{-E_d^\alpha / RT}$$

- with E_d^α : desorption activation energy of species α on this material (kJ/mol)
 τ_{d0}^α : Arrhenius pre-exponential factor (s)
 R : the gas constant (0,00831 kJ/mol·K)

Diffusion

$$\frac{dm}{dt}(t, T) = Cm \frac{\exp^{-E_s / RT}}{\sqrt{t}}$$

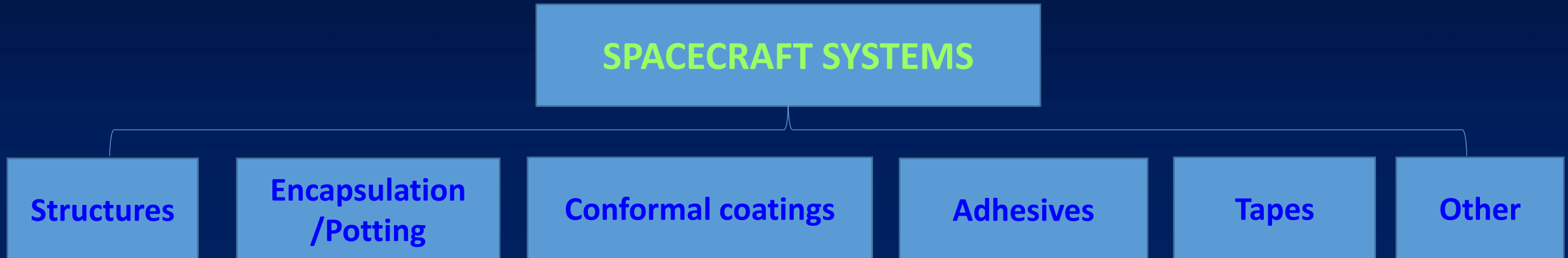
$$\frac{\Delta m}{m} = 2C \exp^{-E_s / RT} (t_2^{1/2} - t_1^{1/2})$$

$$f(x) = D \frac{dn(x)}{dx}$$

- with f : contaminant flux through the material ($\text{g}\cdot\text{cm}^{-2}\cdot\text{s}^{-1}$)
 n : the contaminant concentration in the material (g/cm^3)
 D : the diffusion coefficient (cm^2/s)
 x : the depth coordinate throughout the material (cm)

III. OUTGASSING SOURCES-MATERIALS

Materials outgassing, main sources....



- **Polymer or macromolecular materials**, comprising **low relative molecular weight molecules**, that can contain concentration of volatile variety of mixed chemical compounds in a complex formulations. Weakness of the linkages between molecular structures in polymers also make them prone to outgassing.
- **Certain metallic materials** can also outgass, such us **Zn** that can sublime under vacuum at heating (150°C) or **Cd** (100°C), but at lower order of magnitude compared with polymers, metallic depositions can be critical and must be avoided also.

Electronic and microelectronic materials /COTS

- Plastic materials made of **complex compounds**. Formulations can include i.e : epoxy resin, silicones, urethanes, hardening compounds, accelerators, fillers, flame retardants, couplers, stress relief additives, mold release additives, coloring, and ion-getters among others. LCP with metal treatments.
- Typically **proprietary formulation** of a specific parts resin (i.e: encapsulating), and various types of additives, which provide the desired properties (i.e: for the packaged device).

Main factors determining outgassing levels, Material processing

Material is not validated itself, validation of particular processing for specific application is required for space use or concrete mission.

- Chemical nature of materials.
- Formulation.
- Manufacturing process.
- Curing parameters / process and other treatments (i.e. baking).
- Environmental conditions/storage.
- Preparation/application methods.

typical chemical compounds outgassed /deposited

epoxy, silicone, polyamide , dyciandiamide, polyurethane, acrylic, fluorocarbons, polycarbonates, acetals, ester compounds, hydrocarbons, water vapour , etc..

- Residual concentration of monomers non reacted after polymerization process.
- Concentration of volatile additive or agent compounds present in the polymer (flame retardant, plastificier, estabilizer agent, hardeners, etc.).
- Contaminants, compounds resulted from chemical reactions, absorbed or trapped during material processing.
- Excess of humidity (water vapour could be critical contaminant)

IV. INFLUENCE OF OUTGASSING IN SPACECRAFT

Detrimental effects of outgassing on materials and components

- **Degradation of functional properties of materials** (i.e : outgassing of functional additives).
- **Molecular or chemical contamination on component surfaces, affecting the performance** (i.e: degradation of optical, thermal, electrical properties).
- **Perturbation of the spacecraft medium** (i.e: voltage breakdowns, arcs, heat transfer problems)

V. OUTGASSING TEST AND BAKE OUT IMPLEMENTATION

Outgassing testing

Screening outgassing tests

Std. ref.: ECSS-Q-ST-70-02C / ASTM E 595

Implementation of **general criterium for material acceptance/rejet**, in the materials selection step for space application, and reject those with high levels of outgassing. The general standard limits are:

- **1,00% Total mass loss (TML/RML).**
- **0,10% sample mass, for Collected Volatile Condensable Material (CVCM).**
- **More stringent values can be used, for specific critical cases (i.e: in optical applications, CVCM < 0,01%---→ “Ultra Low OutgassingTM requirements” < 0.1 % TML and < 0.01 % CVCM).**

The test is performed at one standard temperature of reference: **125°C** sample, and **25°C** for collector plate for condensables, on samples in the order of **100-300 mg** mass.

Outgassing testing

Kinetic /dynamic outgassing tests

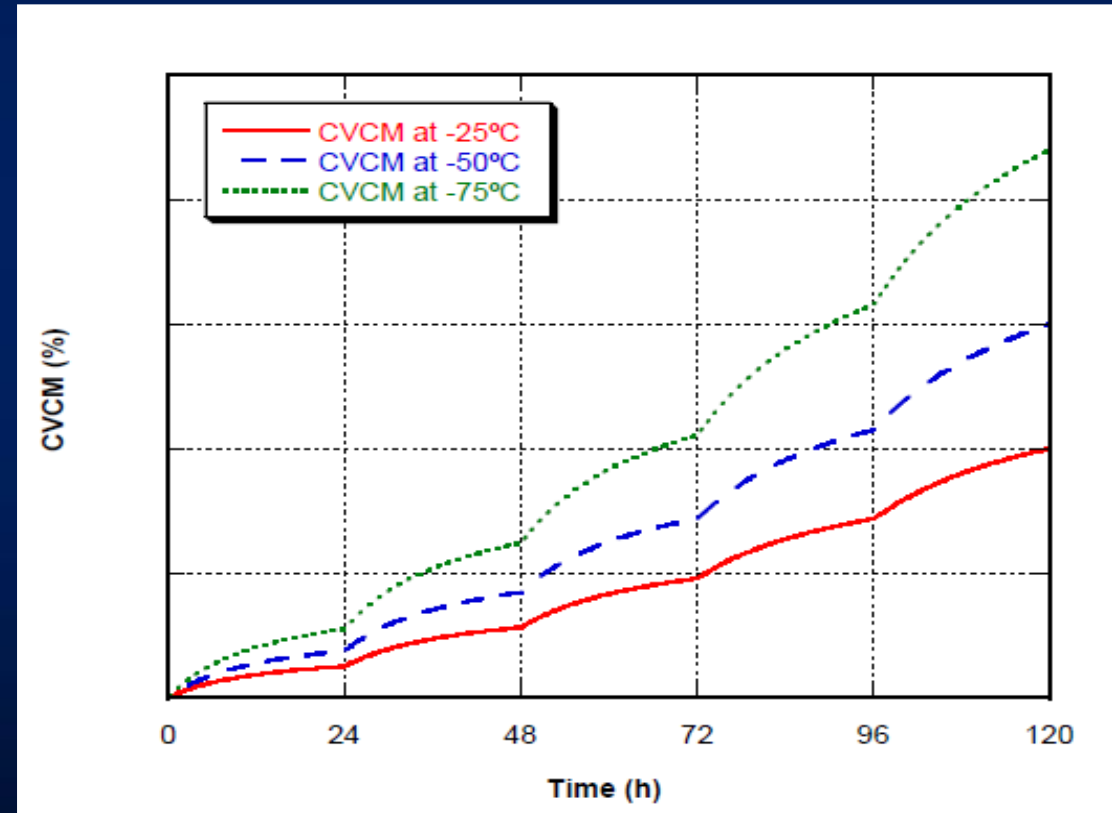
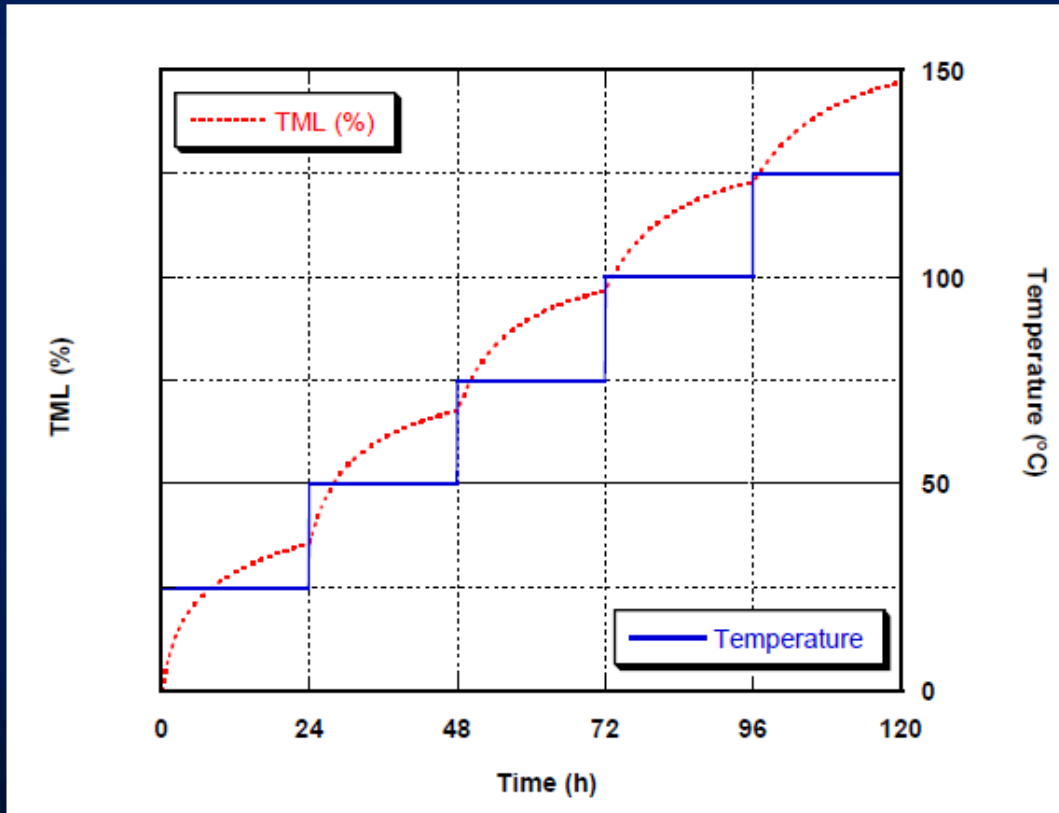
Std. ref.: ECSS-Q-TM-70-52A / ASTM 1559

Mesurement of **outgassing/deposition rate characteristics** with time, under selected values of different heating sample and cold condensable collector temperatures → **outgassing long term predictions**. The method include the use of Vacuum balances / **QCM** Quartz Crystal Microbalances (ESA / ASTM configurations).

Data from this tests allow to compute contamination models and simulate contamination processes in the designed systems, as well as provide further information of outgassing characteristics more than simple data obtained from screening tests.

Outgassing testing

Kinetic /dynamic outgassing tests: standard method ESA



Outgassing testing

Bake out under vacuum

The purpose of this activity, is to evacuate the excess of possible volatile compound of the material or molecular contamination on hardware to **reduce or eliminate the potential outgassing** or in a cleaning process for the removal of residual contamination after solvent application (i.e: IPA) or humidity excess. A material or component is heated under vacuum for a certain period of time (normal practice is between to heat **70-125 °c** under mid – high vacuum , **$10^{-2} - 10^{-5}$ mbar** , for **72 h** or more).

It can be done also in controlled atmosphere.

VI. STANDARD TEST METHOD APPLICATION: ECSS-Q-ST-70-02C / ASTM E 595

Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

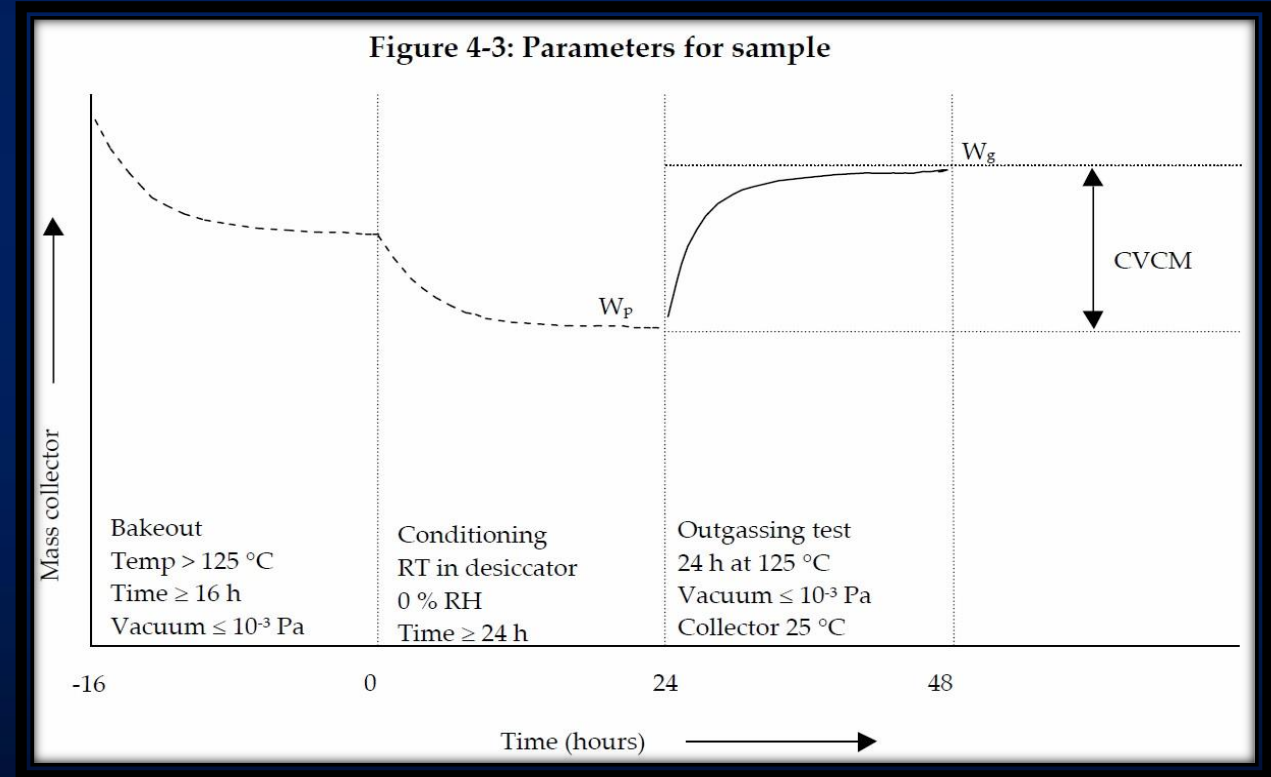
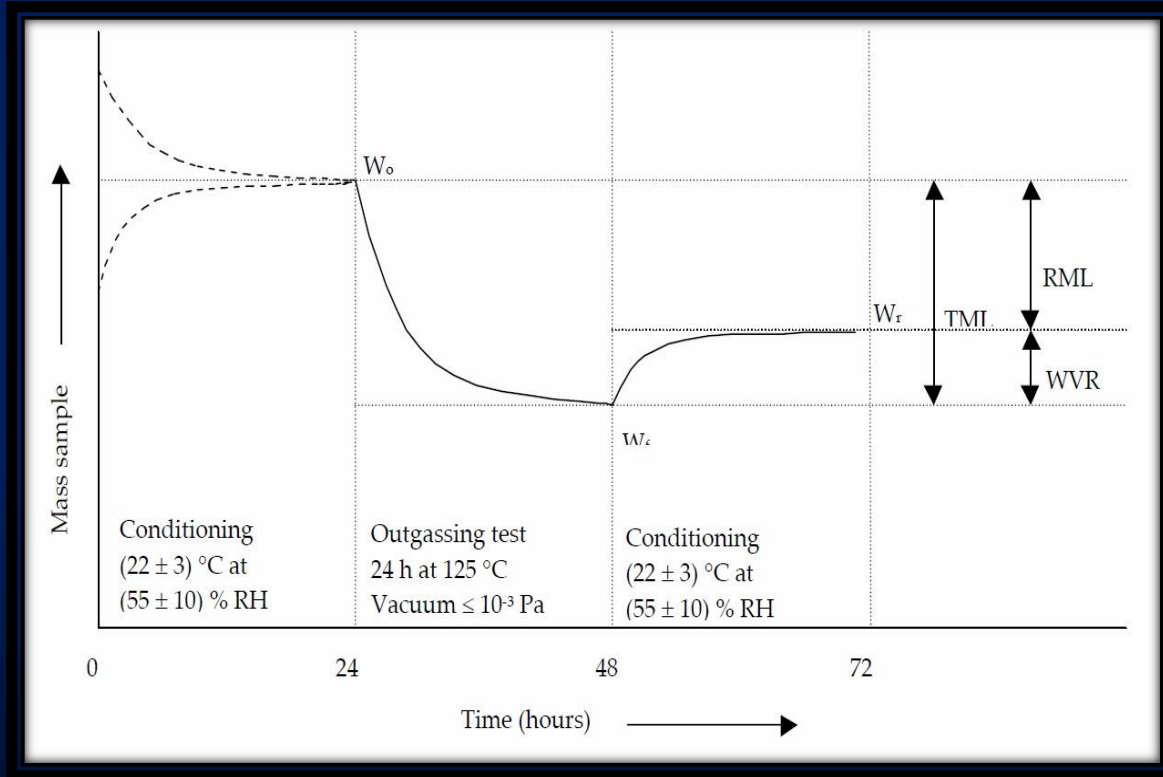
Test standard conditions and data obtained

- **SAMPLE MASS: 100-300 mg (metal parts excluded)**
- **SAMPLE TEMPERATURE: 125°C.**
- **COLLECTOR COLD PLATE TEMPERATURE: 25°C.**
- **TEST DURATION: 24h.**
- **VACUUM PRESSURE: high vacuum range, 10^{-6} - 10^{-7} mbar.**
- **TEST DATA: TML%, RML%, WVR%, CVCM% , in % of total mass sample tested.**
- **Application: materials < 50°C operating temperatures (in general). Determination of outgassing properties of material, not intended for outgassing simulation**

In order to control the balance of water vapour absorbed/desorbed, samples are conditioned before and after the test at **22°C , 55 HR%, 24h.**

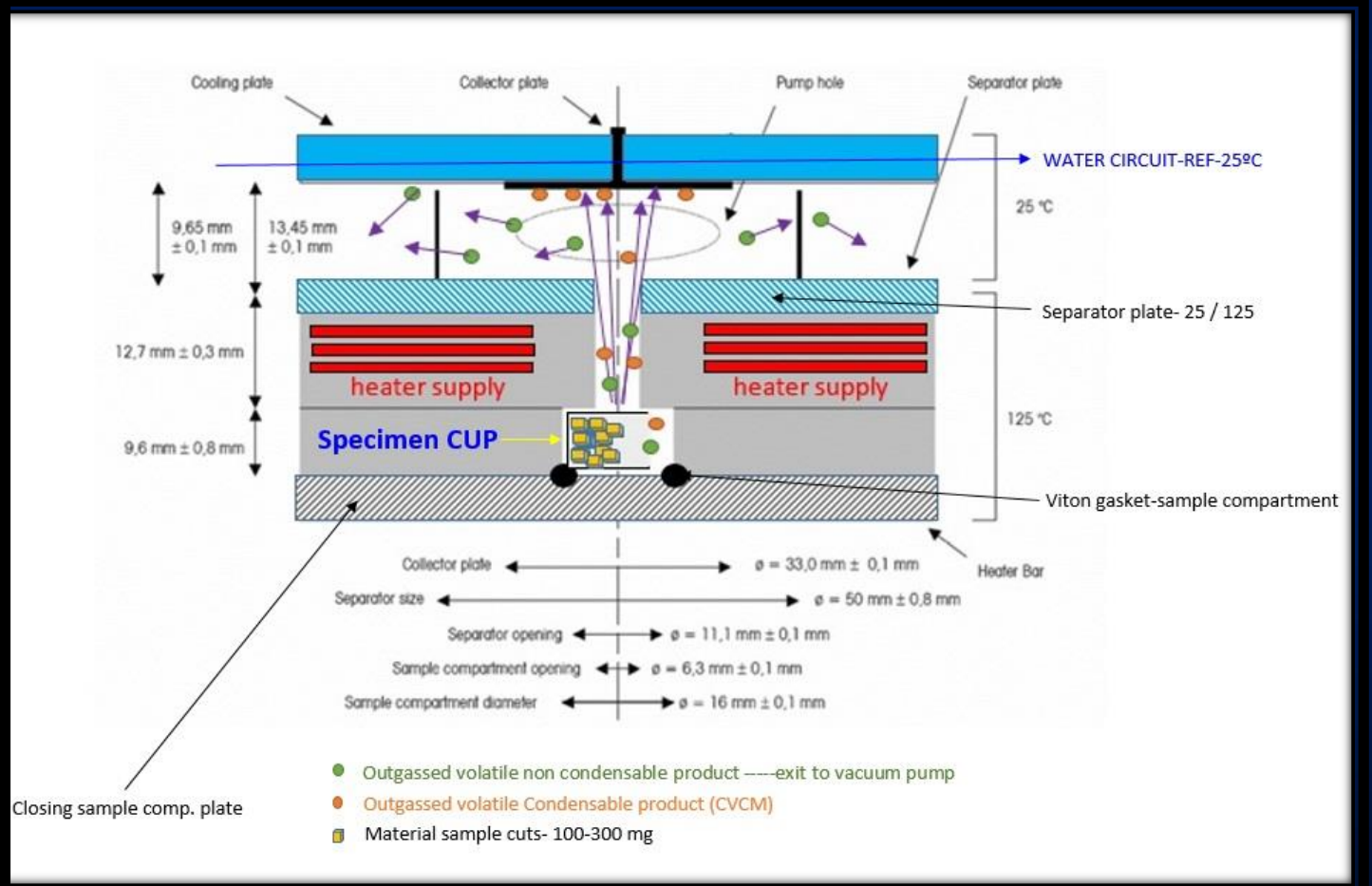
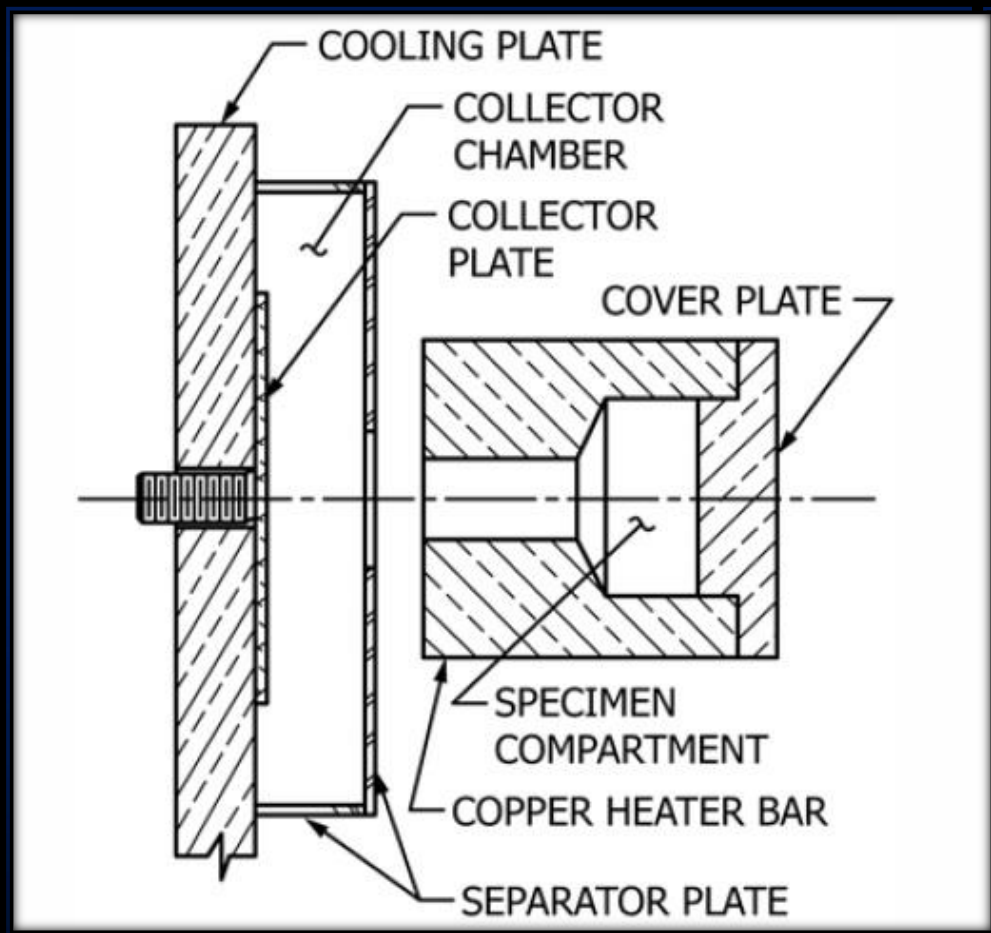
Screening outgassing test: ECSS-Q-ST-70-02C / ASTM E 595

Test process illustration




Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

Test sample configuration



Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

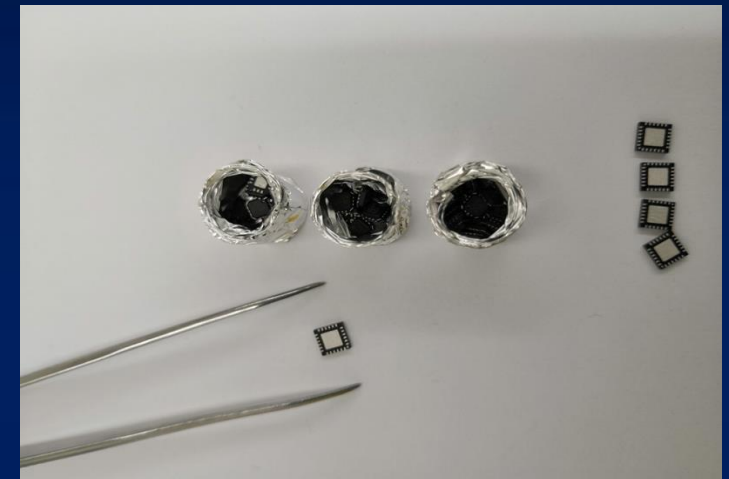
Sample basic preparation

TYPE OF SAMPLE	GNRL. PREPARATION 100-300 mg /cup 	EXAMPLE / REMARKS
BULK MATERIALS	Small cuts 1-2 mm	Epoxies, silicones, etc
PAINTS / UNIFORM COATINGS	Rolls 1 cm wide	Aeroglaze paints, primers, varnishes, sol-gel coats, etc.
FILMS/FOIL/PLATE MATERIALS/TAPES	Rolls, cuts 2-3 mm side	Kaptons, polymer plates, etc.
CABLE MATERIALS	Small cuts 1 cm	Space cable/Insulations.
ELECTRONIC COMPONENTS /COTS	Direct test, for micro components or small components (20-40 mg per unit). Small cuts, plastic parts bigger components that can not be placed directly inside the cups.	COTS, connectors, electric material in general. Plastic material is extracted from connectors or E-devices, and tested with cutting as bulk materials.



3 specimen cups

Ø 10 mm x h 10-12 mm



VII. OUTGASSING TEST FACILITIES



Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

Outgassing test facilities : vacuum technology application



MODEL DMAVAC –Horizontal heating –cooling plates
Developped by INTA

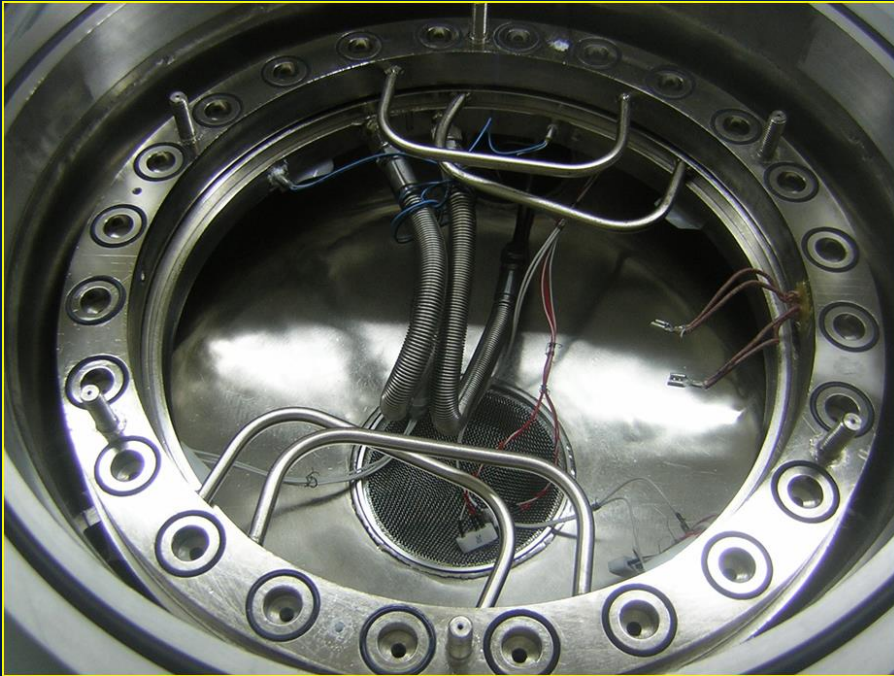


MODEL ESA –Vertical heating –cooling plates
ESA – design

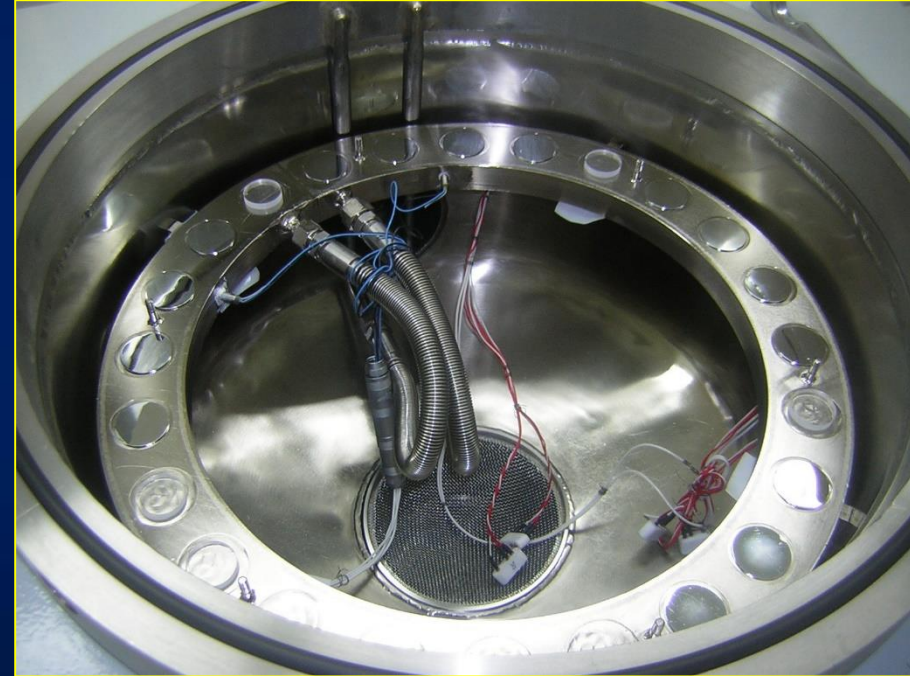
Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

Outgassing test facilities

DMAVAC system –vacuum chamber



Heating plate- 24 sample cup compartment
Room T^a – 200°C



Cooling plate- 24 collector positions
25°C

Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

Outgassing test facilities

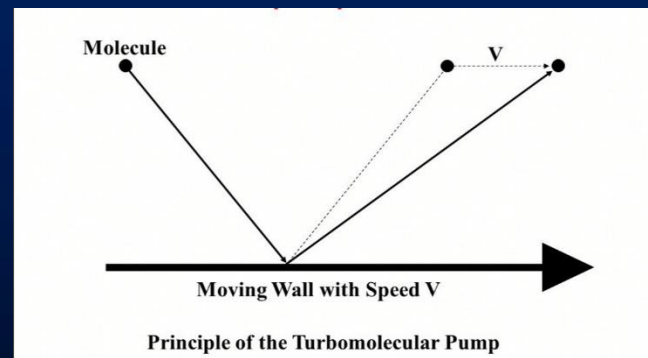
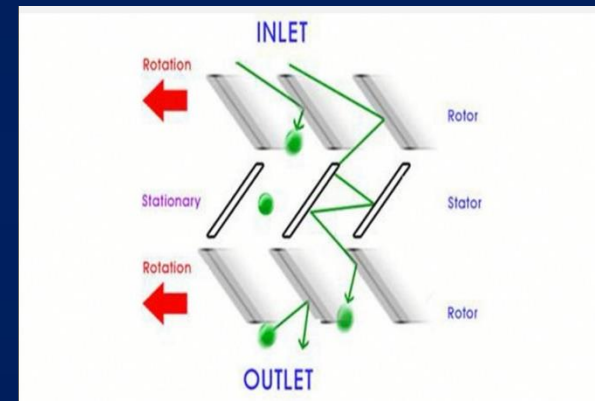
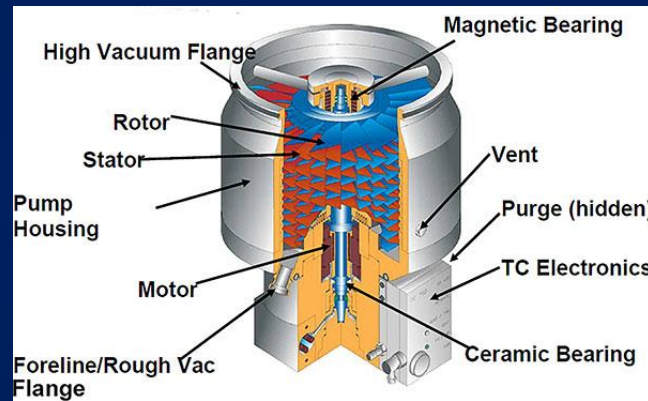
Examples of condensable products produced by outgassed samples (25°C)



Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

Outgassing test facilities

HIGH TEST VACUUM GENERATION: Turbomolecular pump



Mass air suction up to 300 mbar, and kinetic displacement of residual molecules with blade impact under high rpm (830) Hz = 49800 rpm.

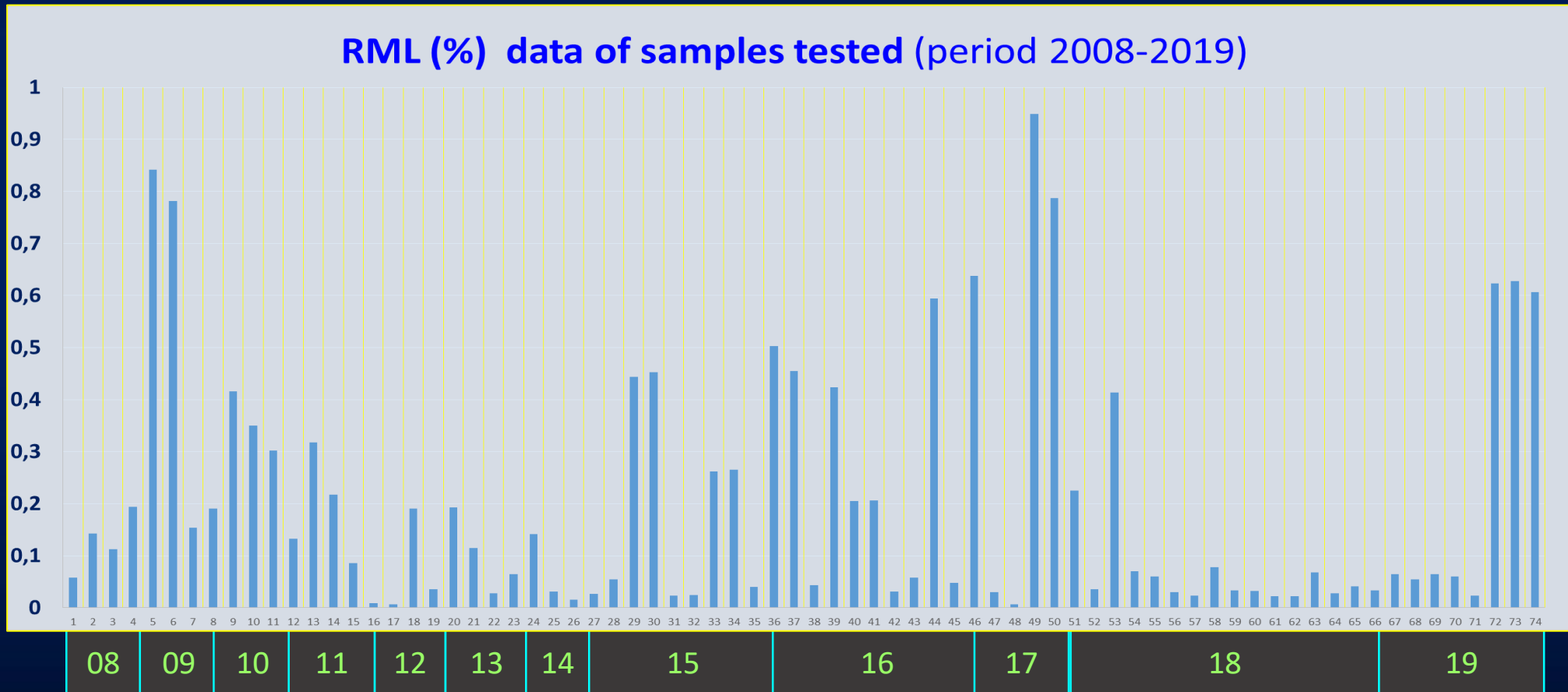
VIII. OUTGASSING TEST DATA AND EVALUATION OF E-COMPONENTS AND COTS PROPOSED FOR SPACE APPLICATIONS

Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

Test experience on several E-components / COTS materials

RML (%) data of samples tested (period 2008-2019)

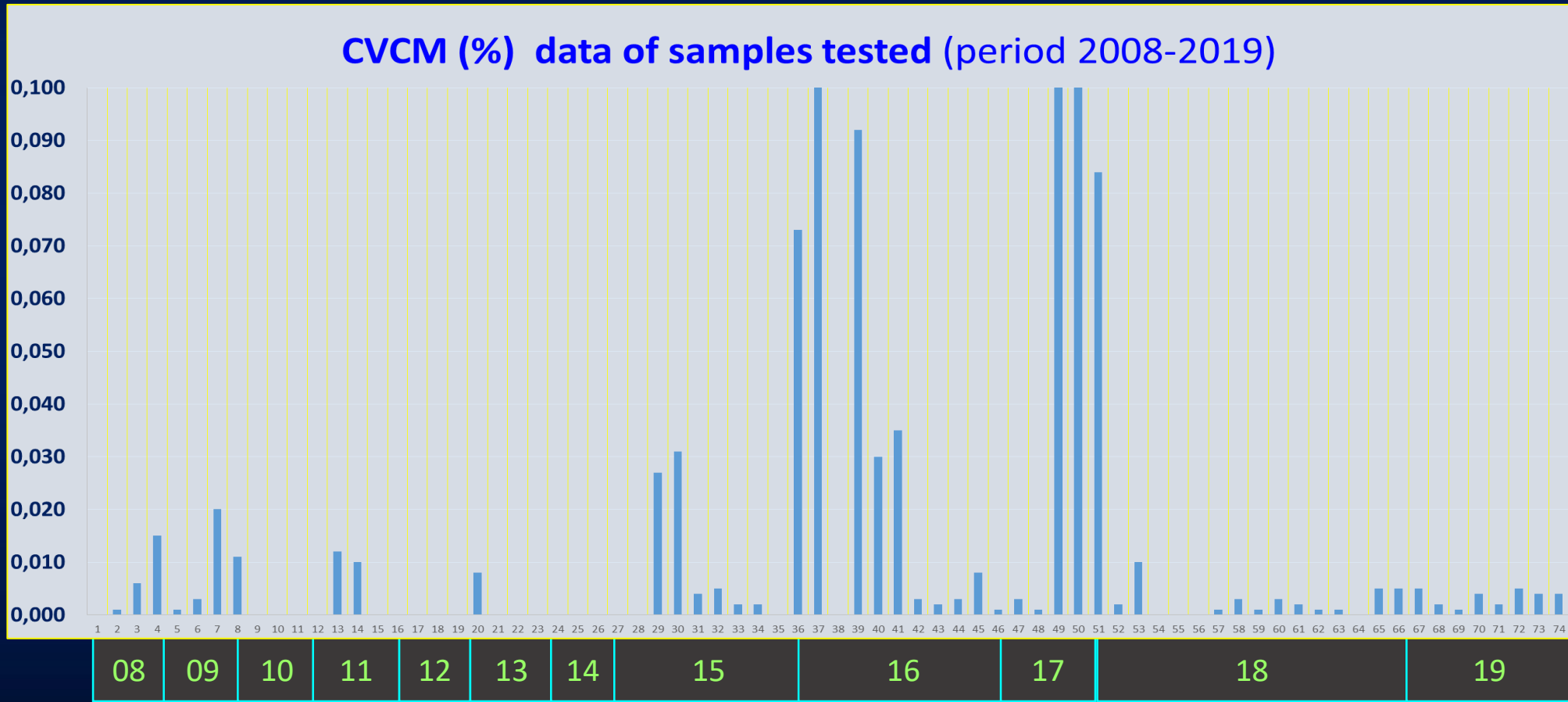
RML%



population Item tested

Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595
Test experience on several E-components / COTS materials

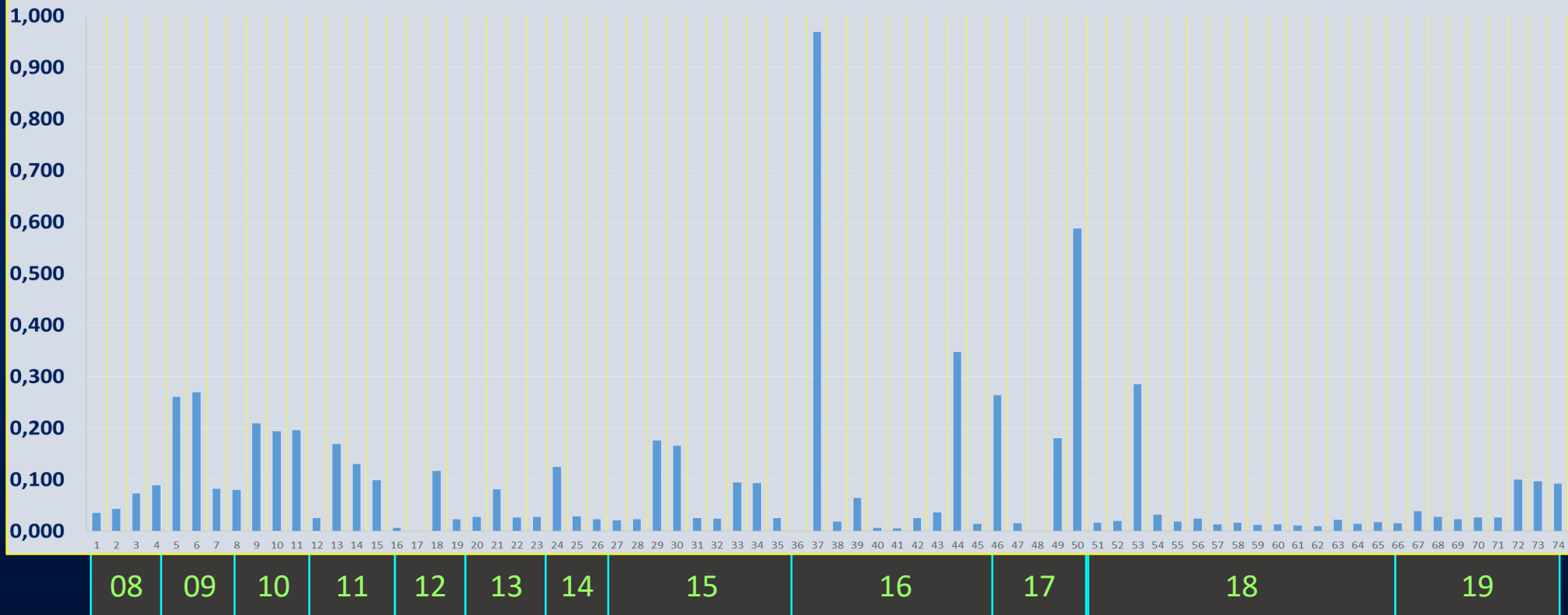
CVCM%



Year / population Item tested

Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595
Test experience on several E-components / COTS materials

WVR (%) data of samples tested (period 2008-2019)

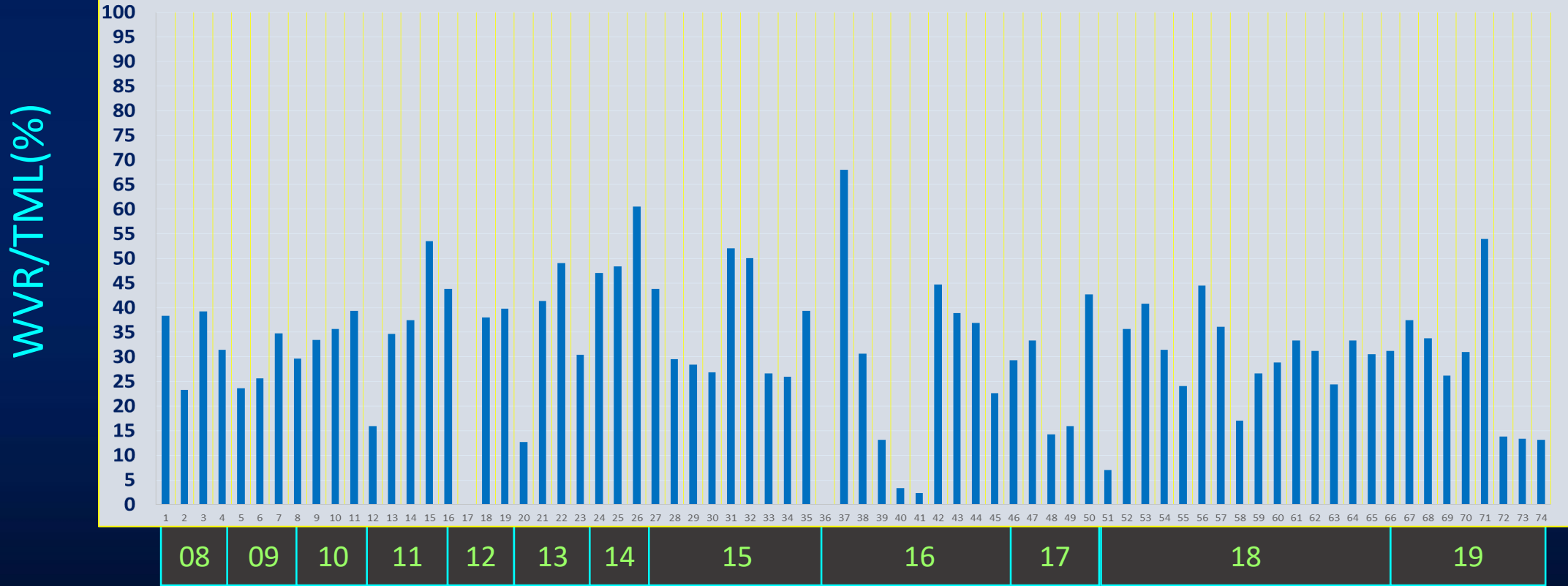


WVR%

Year / population Item tested

Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595
Test experience on several E-components / COTS materials

WVR/TML (%) data of samples tested (period 2008-2019)

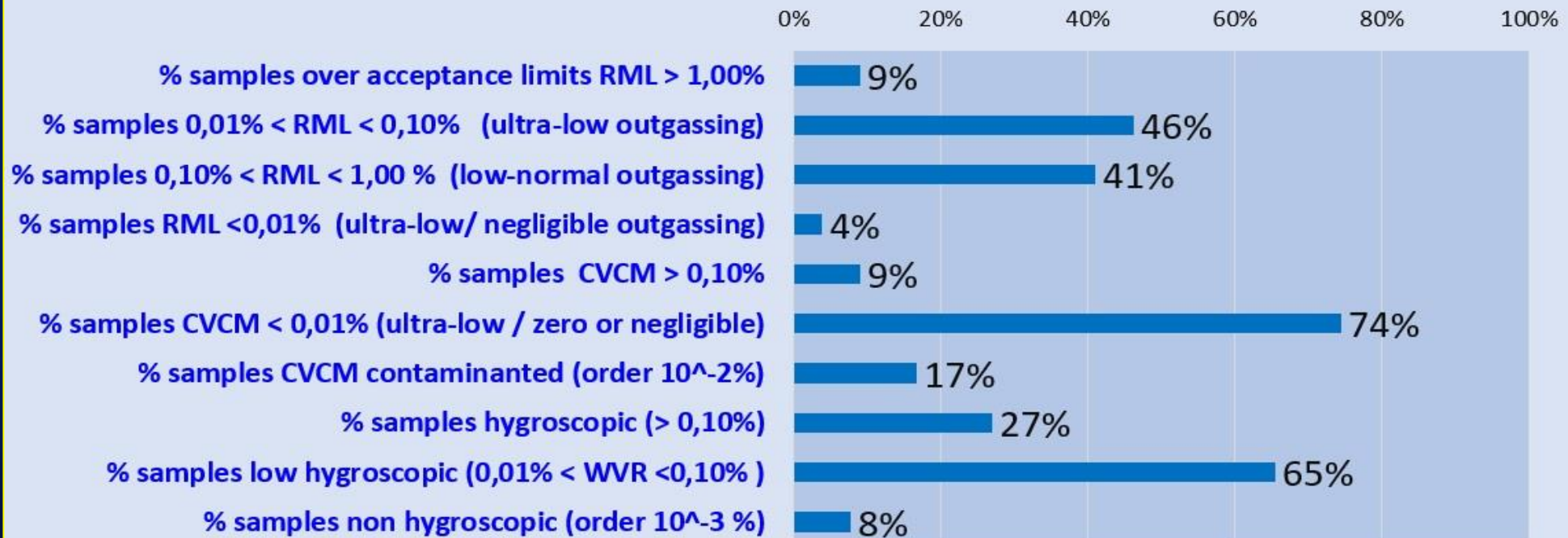


Year / population Item tested

Screening outgassing test ECSS-Q-ST-70-02C / ASTM E 595

Test experience on several E-components / COTS materials

% Samples from total tested vs Outgassing characteristic (period 2008-2019)
E-components / COTS



THANKS FOR YOUR ATTENTION !!

