CNRS *Centre National de la Recherche Scientifique*
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OUTGASSING TEST OF MAPSIL 213 VACUUM GLUE

Code:
VIR-TRE-PIS-3400-119

Date: 21/04/1997

CHANGE RECORD

Issue/Rev	*Date*	*Section affected*	*Reason/ remarks*

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Approved by:		

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In this note we briefly report the outgassing measurements performed on Mapsil 213 glue for UHV. The measurement method is described in detail in VACPISA 025.

1 - System performances

We performed a baking of the test chamber at 250 ^{0}C for some days and we got:

t(h)	T(^{0}C).	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
after	25	6.1×10^{-10}	2.5×10^{-10}	7.2×10^{-9}

The main components of outgassing after baking were H_2, H_2O, N_2/CO, CO_2. The internal surface of the chamber is 3200 cm^2.

2 - Measurement of the outgassing rate of Mapsil 213

The experimental sample was a two component silicon glue, Mapsil 213, manufactured by MAP, France, which has been proposed for gluing the VIRGO baffles. The sample was prepared by mixing 10 parts of base and 1 part of hardener, 4 hours staying at room temperature and 4 hours polymerization at 100 ^{0}C. The exposed surface was 344 cm^2.

We monitored the evolution of outgassing (time is measured from beginning of the test through the whole paper):

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
1.25	25	4.9×10^{-6}	1.3×10^{-6}	7.2×10^{-5}
0.5	25	3.2×10^{-6}	8.2×10^{-7}	4.8×10^{-5}
1	25	2.9×10^{-6}	7.1×10^{-7}	4.4×10^{-5}
25	25	2.6×10^{-6}	5.5×10^{-7}	4.1×10^{-5}
43.5	23	2.6×10^{-6}	5.2×10^{-7}	4.2×10^{-5}
44.5	23	2.6×10^{-6}	5.2×10^{-7}	4.2×10^{-5}
66	23	2.1×10^{-6}	4.1×10^{-7}	3.4×10^{-5}
71.5	23	2.1×10^{-6}	4.2×10^{-7}	3.4×10^{-5}

The spectrum taken after 71.5 hours is shown in Fig. 1.

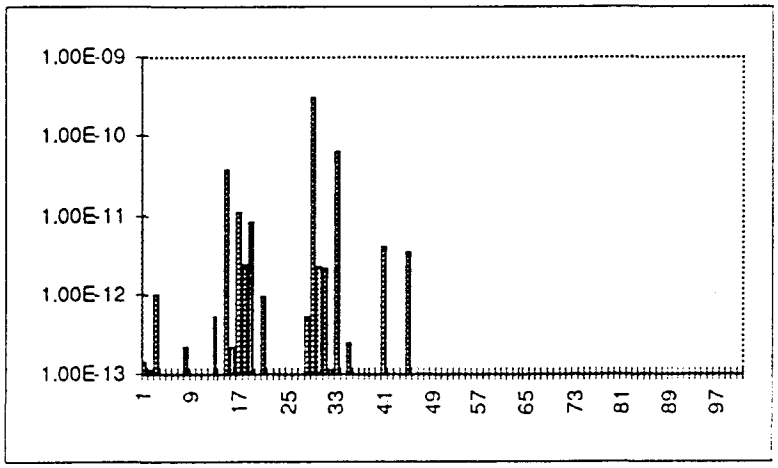

Fig. 1 Outgassing spectrum after 71.5 hours pumping at room temperature

We checked that the peaks at 32 and 40 were not due to a leak.

We set temperature at 50 °C for 67 hours and monitored the evolution of outgassing:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
72	50	7.6×10^{-7}	1.4×10^{-7}	1.2×10^{-5}
73	50	7.7×10^{-7}	1.5×10^{-7}	1.2×10^{-5}
139.25	50	4.8×10^{-7}	8.6×10^{-8}	7.9×10^{-6}

A spectrum taken at 139.25 hours is shown in Fig. 2.

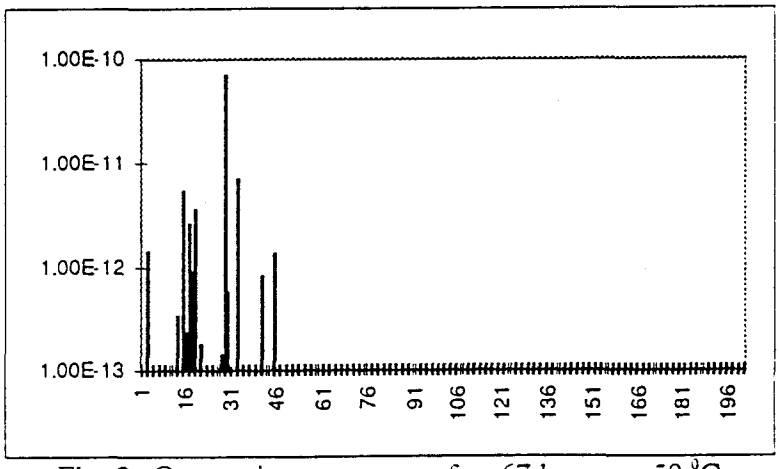

Fig. 2 Outgassing spectrum after 67 hours at 50 °C

We set temperature at 80°C for 51 hours and we measured:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
139.75	80	5.2×10^{-7}	9.6×10^{-8}	8.5×10^{-6}
140	80	4.2×10^{-7}	7.7×10^{-8}	6.9×10^{-6}
144.5	80	4.1×10^{-7}	7.1×10^{-8}	6.8×10^{-6}
185.5	80	2.1×10^{-7}	3.4×10^{-8}	3.5×10^{-6}
191.25	80	2.0×10^{-7}	3.3×10^{-8}	3.3×10^{-6}

The spectrum measured at 185.5 hours at 80 °C is shown in Fig. 3.

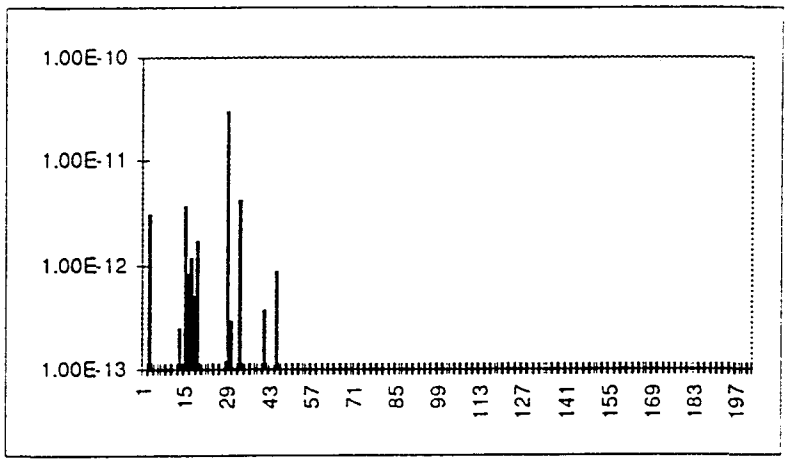

Fig. 3 Outgassing spectrum after 46 hours at 80 °C

We set temperature at 100 °C for 92 hours and monitored the evolution of outgassing:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
191.75	100	3.1×10^{-7}	5.7×10^{-8}	5.1×10^{-6}
192.25	100	3.0×10^{-7}	4.9×10^{-8}	5.0×10^{-6}
193	100	2.9×10^{-7}	5.1×10^{-8}	4.8×10^{-6}
193.75	100	2.8×10^{-7}	5.4×10^{-8}	4.5×10^{-6}
210	100	1.2×10^{-7}	2.7×10^{-8}	1.9×10^{-6}
211.5	100	1.1×10^{-7}	2.5×10^{-8}	1.7×10^{-6}
216.5	100	5.6×10^{-8}	1.5×10^{-8}	8.2×10^{-7}
283.5	100	2.1×10^{-8}	8.0×10^{-9}	2.6×10^{-7}

A spectrum measured at beginning of heating is shown in Fig. 4. A spectrum taken after 92 hours at 100 °C is shown in Fig. 5.

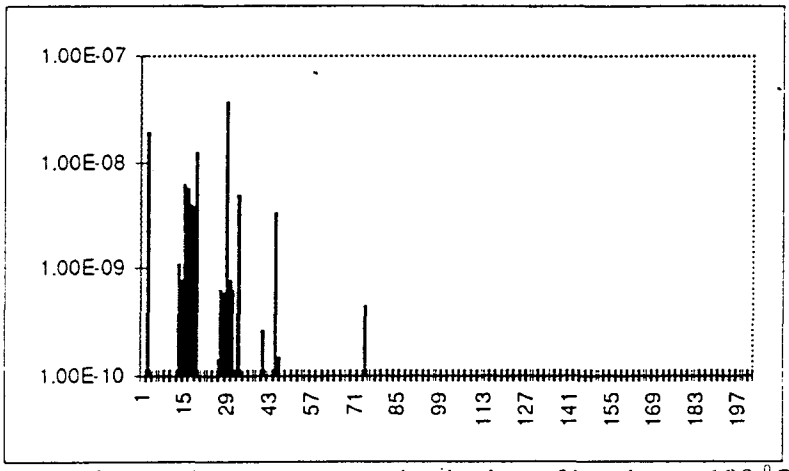

Fig. 4 Outgassing spectrum at beginning of heating at 100 °C

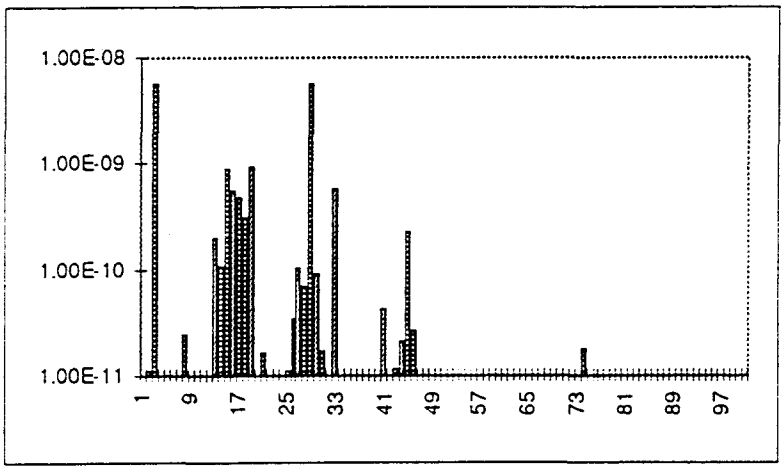

Fig. 5 Outgassing spectrum after 92 hours at 100 °C

A new peak at 73 has appeared.

We set temperature at 150 °C for 172 hours and monitored the evolution of outgassing:

t(h)	T(°C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
284	150	1.7×10^{-7}	1.1×10^{-7}	1.2×10^{-6}
285	150	1.4×10^{-7}	1.0×10^{-7}	8.0×10^{-7}
287	150	1.4×10^{-7}	1.2×10^{-7}	4.0×10^{-7}

288	150	1.1×10^{-7}	1.0×10^{-7}	2.0×10^{-7}
305.5	150	1.3×10^{-8}	3.3×10^{-8}	4.0×10^{-7}
311.25	150	1.0×10^{-8}	2.8×10^{-8}	3.6×10^{-7}
332	150	1.8×10^{-8}	6.1×10^{-9}	2.4×10^{-7}
354	150	4.2×10^{-9}	1.3×10^{-8}	1.8×10^{-7}
359.5	150	4.3×10^{-9}	1.3×10^{-8}	1.7×10^{-7}
383	150	3.3×10^{-9}	1.1×10^{-8}	1.5×10^{-7}
384	150	3.4×10^{-9}	1.1×10^{-8}	1.5×10^{-7}
450	150	5.5×10^{-9}	3.0×10^{-8}	4.9×10^{-7}
456	150	2.3×10^{-9}	8.3×10^{-9}	1.2×10^{-7}

A spectrum taken at the beginning of heating is shown in Fig. 6. A spectrum measured after 172 hours at 150 °C is shown in Fig. 7.

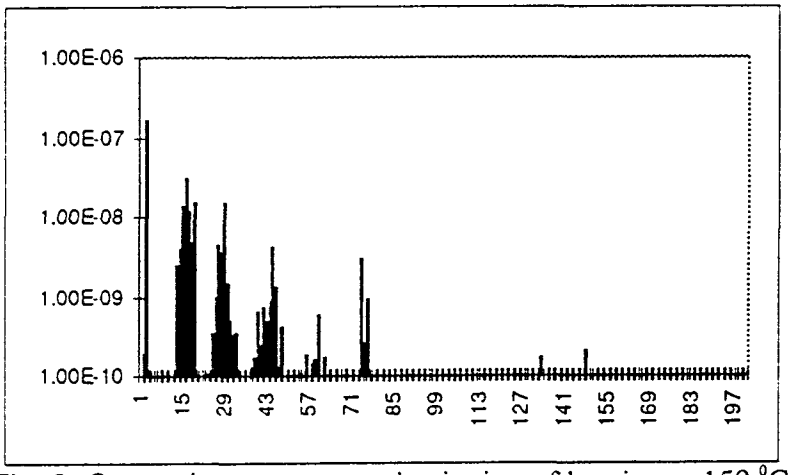

Fig. 5 Outgassing spectrum at beginning of heating at 150 °C

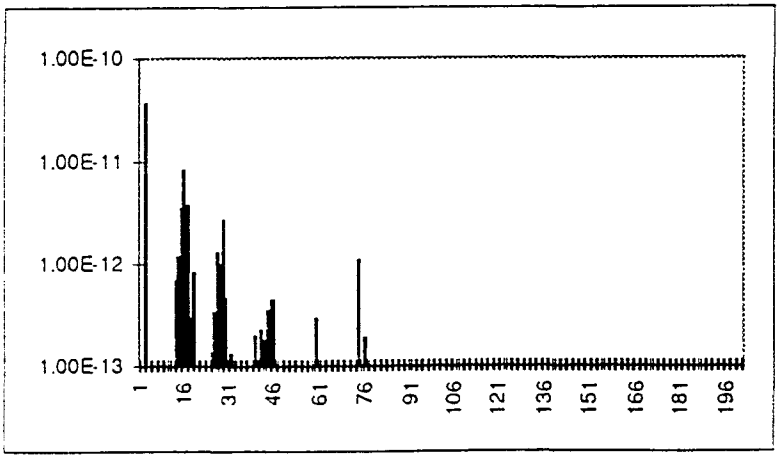

Fig. 5 Outgassing spectrum after 172 hours at 150 °C

We switched off the heating and we measured:

t(h)	T(^{0}C)	p_1(mbar)	p_2(mbar)	Q(mbar l/s)
456.25	130	5.6×10^{-9}	1.6×10^{-9}	8.0×10^{-8}
456.5	110	3.3×10^{-9}	9.8×10^{-10}	4.6×10^{-8}
456.75	100	2.4×10^{-9}	6.7×10^{-10}	3.5×10^{-8}
457	90	2.0×10^{-9}	5.4×10^{-10}	2.9×10^{-8}
473	25	3.0×10^{-10}	1.2×10^{-10}	3.6×10^{-9}
479.5	25	3.1×10^{-10}	1.2×10^{-10}	3.8×10^{-9}
498.5	25	2.7×10^{-10}	1.1×10^{-10}	3.2×10^{-9}
503	25	2.9×10^{-10}	1.4×10^{-10}	3.0×10^{-9}
522.5	25	2.5×10^{-10}	1.1×10^{-10}	2.8×10^{-9}
546	25	2.4×10^{-10}	1.0×10^{-10}	2.8×10^{-9}

The spectrum measured after cooling down is shown in Fig. 8.

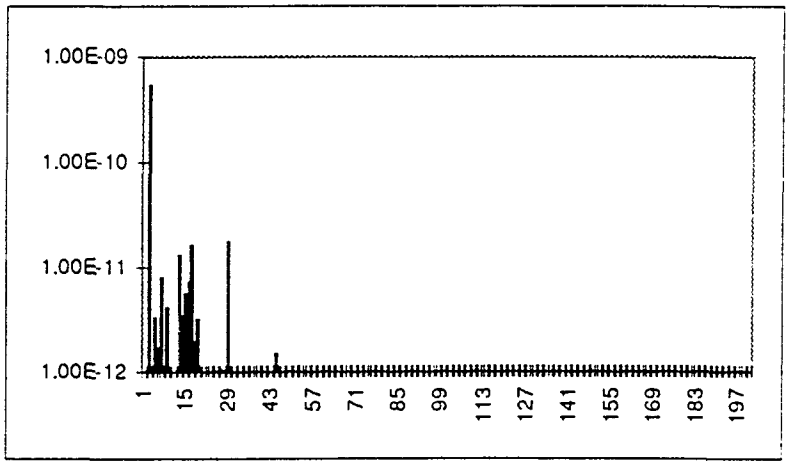

Fig. 8 Outgassing spectrum after thermal cycle

There are no more organic fragments.

The outgassing rate evolution is summarized in Fig. 9.

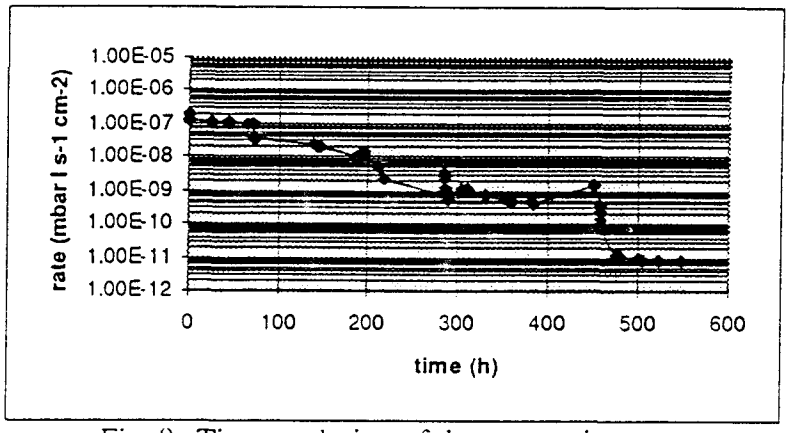

Fig. 9 Time evolution of the outgassing rate

3 - Discussion

The outgassing rate attained after the thermal cycle was 8.1×10^{-12} mbar l s^{-1} cm^{-2}. During heating at 100°C and above some organic peaks appeared, especially around 73/75, which seems typical of silicon organic components. The glue should not be used in the bottom part of the tower and should not undergo baking in situ. Further investigation are needed about the aging and the mechanical strength.