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EUROPEAN TESTS ON MATERIALS OUTGASSING

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ABSTRACT

With a view to international co-ordination of spacecraft materials, a number of European firms and institutes have performed outgassing tests on identical materials at 125°C in high vacuum. This paper presents the outgassing data obtained with the different types of equipment and discusses both the results and the critical parameters.

INTRODUCTION

The aim of the tests was to facilitate the interpretation of outgassing data from different sources, and to improve both the test methods and the verification of outgassing specifications.

The Micro-VCM Test {1} has been generally accepted as a screening test for materials for spacecraft application. Over 2000 different materials have been tested according to this method, which is specified in ESA/PSS-09/QRM-02T {2}, and ASTM's E-21 Committee is currently preparing a similar specification.

Earlier tests {3} by ESTEC (equipment under contract at INTA, Madrid), Goddard Space Flight Center (GSFC) and Jet Propulsion Laboratory (JPL) with similar micro-VCM equipment according to the original JPL design {1} highlighted a number of discrepancies in the results obtained at the three locations.

Not all of the discrepancies could be explained, and some were probably caused by inhomogeneities in the materials and by the test methods, and probably not by the equipment. There were therefore a number of reasons for performing further outgassing tests with different equipment such as micro-VCM, macro-VCM and vacuum balance.

PARTICIPATING INSTITUTES, EQUIPMENT TYPES AND MATERIALS TESTED

The institutes taking part in the test series and the equipment they used are listed in Table 1.

Seven materials were tested, as listed in Table 2. Items 1 and 2 were supplied by CNES, and the remainder by ESTEC.

Sample size

(a) The three paint samples had been prepared on Al foil (4,4mg/ cm^2) for all institutes except DFVLR, who received their samples on a metal disc of 1 cm diameter (items 1-2-5).

(b) The bulky samples had been cut into smaller pieces(2.5mm cube) at ESTEC before despatch to the participants (items 2-4-6).
  - DFVLR samples (items 3-4-6) had been prepared on metal discs of 1 cm diameter.
  - ESTEC 2 tests were performed on 20 x 20 x 4 mm^3 blocks for items 3-4-6.
  - The CNES samples (items 3-4-6) had been prepared as discs of 30 mm diameter and about 2.5 mm thick.

(c) Rilsan BMNO had a granular structure and the 2.5 mm cubes were tested as received.

TEST PARAMETERS

Tests were conducted on the same data for the two micro-VCM systems and the CNES macro-VCM system. The vacuum-balance tests were undertaken over a period of about two weeks, starting on the same date as the above tests. Except for the MBB tests which were conducted about eight months later.

The test procedure specified in ESA/PSS-09/QRM-02T was followed as closely as the equipment allowed.

The main test variables from above specification, temperature, time and humidity are recorded in Figure 1:

- The materials samples had been conditioned for a minimum of 24 h at $20^{\circ} \pm 1^{\circ}C$ and 65% relative humidity.
- The material samples were weighed just before the test under atmospheric conditions (W_{oa}) or under vacuum (W_{ov}) for the vacuum balance system.
- After pumpdown to 10^{-5} torr, the sample compartment was raised to $+125^{\circ} \pm 1^{\circ}C$.
- The sample heaters were turned off 24 h after $125^{\circ}C$ had been reached and dry nitrogen or inert gas was introduced into the vacuum system at 100-200 torr
- At the moment when the sample temperature had falled to $50^{\circ}C$, further dry nitrogen was admitted up to atmospheric pressure.
- After unloading from the system and after a further cooling down period in a dessicator (about 30 minutes), the samples were

Institute System	Heating-up cooling-down times (h)	Pumping speed $L.S^{-1}$ N_2-20°C	Condensors
ESTEC-1 Micro-VCM	½/2	1.5	2 x chrome, ø 31 mm 1 x NaCl. ø 25 mm
INTA Micro-VCM	½/2	1.5	3 x NaCl, ø 25 mm
CNES Macro-VCM	1½	3.0	2 x quartz, ø 30 mm
MBB 'Macro-VCM' Vacuum Balance	-	60	1 x aluminium, ø 70 mm
DFVLR Vacuum Balance	½/high-frequency heating	-	NO
ESTEC-2 Vacuum Balance	2-3/1 (dummy sample)	25	NO
DERTS Vacuum Balance	-	-	NO
SNIAS Vacuum Balance	-	-	NO

TABLE 1

TABLE 2

No.	Trade name	Manufacturer	Chemical nature	Preparation method
1	PSG-120	Pyrolac	White silicone paint	On Al foil,4,4mg/cm^2
2	PSE-109	Pyrolac	Black epoxy paint	On Al foil,4,4mg/cm^2
3	Silicoset-105	ICI	silicone potting	0.6% CRA - RT cure
4	BSL-203	Ciba	epoxy adhesive	100p/A, 14p/B - RT
5	Chemglaze Z-306	Hughson	Black polyurethane paint	On Al foil, 4,4mg/cm^2
6	Araldite AV-134B/HY994	Ciba	epoxy adhesive	100p/134, 40p/994 22h/RT + 2h/60°C
7	Rilsan BMNO	Aquit. Org.	polyamide 11 grains	A.R.

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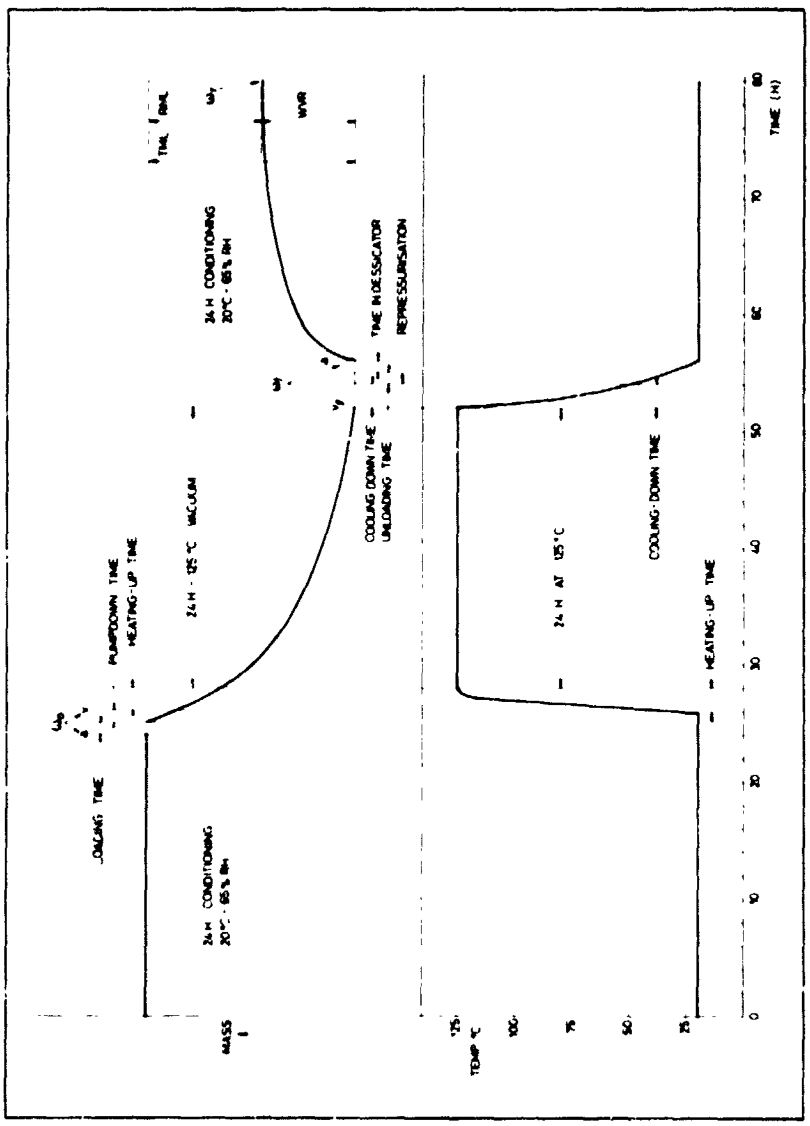

Figure 1

weighed (W_{fa}).

- For the vacuum-balance systems, the sample mass after the tests was determined in vacuum (W_{fv}).
- The samples were re-exposed to 20° ± 1°C and 65% relative humidity for 24 h and then re-weighed (W_r).
- The collectors which were maintained at 25° ± 1°C during the test were weighed just before the test (W_c) and just after (W_c') both times under atmospheric conditions.

MEASURED OUTGASSING DATA

Total Mass Loss	$\frac{W_o - W_f}{W_o}$	x 100% = % TML
Recovered Mass Loss	$\frac{W_o - W_r}{W_o}$	x 100% = % RML
Water-Vapour Regain	$\frac{W_r - W_f}{W_o}$	x 100% = % WVR
Collected Volatile Condensable Material	$\frac{W_c - W_c'}{W_o}$	x 100% = % CVCM

Differences in the total-mass-loss data can be expected as the micro-VCM data and the CMES macro-VCM data are based on the mass measurements under atmospheric conditions.

$$\% \text{ TML}' = \frac{W_{oa} - W_{fa}}{W_{oa}} \times 100\%$$

While the data from the vacuum-balance methods are based on mass measurements in vacuum.

$$\% \text{ TML} = \frac{W_{ov} - W_{fv}}{W_{ov}} \times 100\%$$

Both methods involve uncertainties: the atmospheric method has a number of uncontrolled periods at the end of the test involving cool-down time, unloading time, time in dessicator and weighing time; the vacuum method has the uncontrolled periods at the beginning of the test, involving loading time (the humidity is difficult to control in the vacuum system), and during pump down the mass measurements are disturbed by the 'byoyancy' effects, for which corrections can be made.

At ESTEC, both the atmospheric and vacuum total-mass losses were measured for three materials with two different balances. In this case the vacuum TML was about 5 to 10% higher than the atmospheric TML (Table 3).

TABLE 3

Materials	Atmospheric TML %	Vacuum TML %
(5) Chemglaze Z-306	1.51	1.62
(6) Araldite AV 134	2.01	2.10
(7) Rilsan BMNO	1.29	1.43

RESULTS

Table 4 gives the measured data: % TML, % RML, % WVR and % CVCM (respectively columns 3, 6, 9 and 12) for the seven materials tested by the eight participating institutes. ESTEC 1, INTA, CNES and DFVLR conducted their measurements in triplicate or duplicate (see column 15) and the calculated 1 σ error

$$\{1\ \sigma = \sqrt{(\bar{x} - x)^2/n' - 1}\}$$

is given in columns 4, 7, 10 and 13. Columns 5, 8, 11 and 14 give the ratios of measured values to the average value for all participants.

INTERPRETATION OF RESULTS

Total Mass Loss (TML)

Table 5 gives the ration $TML/TML_{average}$ noted in column 5 of Table 4 in another way. The low TML value for PSG-120 by ESTEC-1 was verified in a second test on a 710 mg sample. The obtained data TML = 0.72%. RML = 0.69% and CVCM = 0.09% were quite close to the earlier ones, the lower CVCM percentages might be explained by the creeping of the silicone products condensed on the collector.

The correlation between the ESTEC-1, INTA and CNES (all having systems based on the same design for routine tests) results is quite good, factors 1.01, 1.01 and 1.03 with 1 σ values respectively 0.12, 0.15 and 0.12.

The MBB average value ($TML/TML_{average}$) is high (1.14) mainly because of the two bulky epoxies BSL-203 and AV-134 (values 1.46 and 1.42). A high initial water sorption by these two materials might explain the high values but, on the contrary, the water vapour regain (WVR) after the test was about half of the average WVR (see table 7). In this case, we should not forget that the MBB tests were made eight months later than the others, so that ageing of the materials might be a factor.

The DFVLR average value of 0.57 is certainly too low, the low outgassing data being caused by a lower sample temperature, as will be explained below.

TABLE 4

Results of European outgassing tests, 1 March 1975

	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17
															SAMPLES		
	Material	Institute	%TML	±1σ	TML/TML_average	%RML	±1σ	RML/RML_average	%WVR	±1σ	WVR/WVR_average	%CVCM	±1σ	CVCM/CVCM_average	No	mg	mg/cm²
1	PSG-120 white silicone paint	ESTEC 1	0.73	0.03	0.88	0.71	0.01	0.84	0.03	<0.01	0.75	0.130	0.01	0.75	1	245	23
		INTA	0.96	0.02	1.16	0.92	0.02	1.08	0.04	<0.01	1.0[illegible]	0.200	0.03	1.16	1	235	23
		CNES	1.02	0.01	1.23	1.02	0.02	1.20	0.00		0.00	0.152	0.03	0.88	2	332	23
		MBB	0.70		0.94	0.71		0.86	0.05		1.25	0.210		1.21	1		23
		DFVLR	0.54	0.01	0.65										2	24	23
		ESTEC 2															
		DERTS	0.95		1.14	0.87		1.02	0.08		2.00				1	252	23
		SNIAS	0.86		1.04										1	709	23
	Average values		0.83			0.85			0.04			0.173					
2	PSE 109 black epoxy paint	ESTEC 1	5.48	0.02	0.94	4.52	0.03	0.85	0.96	0.01	1.16	0.065	0.01	0.22	3	258	8.8
		INTA	5.33	0.13	0.91	4.37	0.13	0.86	0.75	<0.01	0.91	0.105	0.01	0.35	1	170	8.8
		CNES	5.58	0.20	0.95	5.31	0.21	0.99	0.27	0.02	0.33	0.401	0.06	1.35	3	255	8.8
		MBB	5.81		0.99	4.81		0.90	1.00		1.21	0.620		2.60	1		
		DFVLR	4.22	0.26	0.72										2	8	8.8
		ESTEC 2															
		DERTS	8.65		1.48	7.50		1.40	1.15		1.39				1	149	8.8
		SNIAS	5.85		1.00										1	710	8.8
	Average values		5.85			5.34			0.83			0.258					
3	Silcoset 105 silicone rubber	ESTEC 1	2.64	0.08	1.16	2.62	0.01	1.11	0.03	<0.01	0.62	0.342	0.05	0.90	3	230	40
		INTA	2.57	0.06	1.13	2.53	0.07	1.08	0.04	<0.01	0.83	0.262	0.05	0.66	3	303	40
		CNES	2.44	0.16	1.07	2.44	0.16	1.04	0.00		0.60	0.455	0.02	1.15	2	325	110
		MBB	2.60		1.14	2.56		1.09	0.04		0.83	0.48		1.23	1		
		DFVLR	1.25	0.01	0.55										2	142	142
		ESTEC 2	2.15		0.94	2.10		0.89	0.05		1.03				1	1099	166
		DERTS	2.00		0.88	1.8[illegible]		0.79	0.13		2.00				1	294	
		SNIAS	2.60		1.14										1	710	130
	Average values		2.28			2.35			0.05			0.387					
4	BSL 203 epoxy	ESTEC 1	1.15	0.05	1.10	0.64	0.04	0.82	0.31	0.06	1.42	0.062	0.03	2.13	3	187	40
		INTA	1.11	0.03	1.06	0.65	0.01	0.83	0.46	0.08	1.28	0.029	<0.01	1.16	3	202	40
		CNES	0.90	0.10	0.86	0.83	0.10	1.06	0.07	0.01	0.19	0.001	0.001	0.06	3	2660	166
		MBB	1.52		1.46	[illegible]		1.72	0.18		0.50	0.000		0.00	1		
		DFVLR	0.50	0.02	0.48										2	177	177
		ESTEC 2	1.07		1.03	0.70		0.89	0.37		1.02				1	1765	165
		DERTS	1.12	0.12	1.07	0.55	0.02	0.70	0.57	0.15	1.58				2	281	
		SNIAS	0.97		0.93										1	711	133
	Average values		1.04			0.79			0.36			0.019					
5	Chemglaze Z 306 black polyurethane paint	ESTEC 1	1.37	0.06	0.97	0.43	0.03	0.86	0.94	0.02	0.90	0.054	0.02	0.53	3	148	35
		INTA	1.15	0.14	0.82	0.58	0.12	1.16	0.57	0.06	0.54	0.113	0.06	1.11	3	114	35
		CNES	1.41	0.25	1.00	[illegible]	0.21	2.48	0.17	0.04	0.16	0.121	0.06	1.19	2	306	35
		MBB	1.62		1.15	[illegible]		0.96	1.14		1.09	0.120		1.18	1		35
		DFVLR	0.90		0.64										1	10	10
		ESTEC 2	1.62		1.15	0.23		0.46	1.30		1.37				1	258	35
		DERTS	2.27	0.47	1.61	0.03	0.13	0.06	2.08	0.33	1.98				2	134	35
		SNIAS	0.92		0.65										1	710	35
	Average values		1.41			0.50			1.05			0.102					
6	Araldite AV134B HY994 epoxy	ESTEC 1	2.58	0.03	1.13	1.93	0.05	0.85	0.66	0.01	1.57	0.064	0.01	0.61	3	225	40
		INTA	2.55	0.06	1.12	2.13	0.07	0.94	0.43	0.02	1.02	0.072	0.02	0.69	3	201	40
		CNES	2.44	0.29	1.07	2.36	0.30	1.04	0.08	0.01	0.19	0.182	0.03	1.74	[illegible]	1890	299
		MBB	3.23		1.42	2.98		1.32	0.25		0.60	0.100		0.96	1		
		DFVLR	0.51	0.03	0.22										2	154	154
		ESTEC 2	2.10		0.92	1.96		0.87	0.14		0.33				1	1686	157
		DERTS	3.21		1.41	2.23		0.99	0.98		2.33				1	305	
		SNIAS	1.64		0.72										1	710	91
	Average values		2.28			2.26			0.42			0.105					
7	Rilsan BMNO polyamide grains	ESTEC 1	0.93	0.01	0.89	0.66	0.01	0.83	0.27	<0.01	1.01	0.052	0.01	0.91	3	195	40
		INTA	0.88	0.06	0.84	0.69	0.03	0.86	0.20	0.01	0.76	0.017	0.01	0.30	3	196	40
		CNES	1.06	0.06	1.01	0.94	0.07	1.18	0.12	0.01	0.46	0.110	0.02	1.92	[illegible]	1990	40
		MBB	0.95		0.91	0.59		0.74	0.37		1.41	0.05[illegible]		0.87	1		40
		DFVLR	0.78	0.01	0.74										2	305	40
		ESTEC 2	1.43		1.36	1.15		1.44	0.28		1.07				1	609	40
		DERTS	1.09		1.04	0.76		0.94	0.33		1.26				1	302	40
		SNIAS	1.27		1.21										1	710	40
	Average values		1.05			0.80			0.26			0.057					

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A metal disc of 10 mm diameter, on which the material was prepared was heated by means of an HF coil outside the vacuum system; the sample was surrounded by liquid nitrogen-cooled walls, the temperature of the metal disc was controlled by a thermocouple on a dummy metal disc situated close to the sample disc. Corrections for the forces, caused by the HF heating on the metal disc hanging from the vacuum microbalance were taken into account.

Between the metal disc and the front of the material sample there is a temperature difference which depends on the nature of the thermal contact with the metal disc, on the thermal conductivity and thickness of the material, and on its emissivity. An approximate calculation by DFVLR showed a 10^{o} temperature difference for 3 mm-thick samples.

Typically, for samples of 140 to 180 mg, the three materials SIL-105, BSL-203 and AV-134 with thicknesses of 2 -3 mm gave the lowest $TML/TML_{average}$ values in Table 5 (respectively 0.55, 0,48 and 0.22). The three paint samples PSG-120, PSE-109 and Z-306 (resp. 24,8 and 10 mg) gave very similar (to each other) but still low values (resp. 0.65, 0.72 and 0.64). Best correlation with other data was obtained for Rilsan, which might be explained by the cavity sample holder which was used instead of the metal disc because of the granular structure of Rilsan. The reproducibility of the DFVLR tests is quite good.

The ESTEC-2 (vacuum-balance system) values do not differ significantly from other values, which is interesting because samples 3, 4 and 6 were 20 x 20 x 4 mm^3 blocks; hence the size of these three samples did not seem to be critical.

The DERTS average value ($TML/TML_{average}$) of 1.23 is high mainly because of high water sorption by samples 2, 5 and 6, with corresponding ratios, 1.48, 1.61 and 1.41. The water sorption correlation can easily be seen by comparing the $TML/TML_{average}$ values with WVR_a in the last column of Table 5.

Recovered Mass Loss (RML)

Table 6 gives the $RML/RML_{average}$ values from column 8 in Table 4 in an alternative form.

The RML values should be independent of conditioning parameters as long as such pre- and post-conditioning parameters as humidity, temperature and time are the same. In other words, the influence of sorbed water (=WVR) should be cancelled out in the RML data. From the fact that the RML data are not as selfconsistent as the TML data, we can conclude that either conditioning or other parameters (e.g. test temperature) caused the poorer correlation.

As the post-conditioning differed widely for all participants (which fact will be explained in Section 6.3 from the widely

TABLE 6

$RML/RML_{average}$

Institute / Material	ESTEC 1	INTA	CNES	MBB	DFVLR	ESTEC 2	DERTS	SNIAS	WVR_a %
1 PSG-120	0.84	1.08	1.20	0.86	-	-	1.02	-	0.04
2 PSE-109	0.85	0.86	0.99	0.90	-	-	1.40	-	0.83
3 SIL-105	1.11	1.08	1.04	1.09	-	0.89	0.79	-	0.05
4 BSL-203	0.82	0.83	1.06	1.71	-	0.89	0.70	-	0.36
5 Z-306	0.86	1.16	2.48	0.96	-	0.46	0.06	-	1.05
6 AV-134	0.85	0.94	1.04	1.32	-	0.87	0.99	-	0.42
7 Rilsan	0.83	0.86	1.18	0.74	-	1.44	0.94	-	0.26
Average value	0.88	0.97	1.28	1.08	-	0.91	0.84	-	
1 σ ±	0.10	0.13	0.53	0.33	-	0.35	0.41	-	

TABLE 5 $TML/TML_{average}$

Institute / Material	ESTEC-1	INTA	CNES	MBB	DFVLR	ESTEC-2	DERTS	SNIAS	WVR_a %
1 PSG-120	0.88	1.16	1.23	0.94	0.65	-	1.14	1.04	0.04
2 PSE-109	0.94	0.91	0.95	0.99	0.72	-	1.48	1.00	0.83
3 SIL-105	1.16	1.13	1.07	1.14	0.55	0.94	0.88	1.14	0.05
4 BSL-203	1.10	1.06	0.86	1.46	0.48	1.03	1.07	0.93	0.36
5 Z-306	0.97	0.82	1.00	1.15	0.64	1.15	1.61	0.65	1.05
6 AV-134	1.13	1.12	1.07	1.42	0.22	0.92	1.41	0.72	0.42
7 Rilsan	0.89	0.84	1.01	0.91	0.74	1.36	1.04	1.21	0.26
Average value	1.01	1.01	1.03	1.14	0.57	1.08	1.23	0.96	
1 σ ±	0.12	0.15	0.12	0.22	0.18	0.18	0.27	0.21	

differing WVR data), we cannot assume that the pre-conditioning was the same for all participants, and hence no further conclusions can be withdrawn from the data.

Ignoring the Chemglaze Z-306 paint (≈ 74% of the total mass loss is due to water) in the CNES data, we obtain an average value of 1.09 ± 0.08 instead of 1.28 ± 0.53 and for DERTS 0.97 ± 0.24 instead of 0.84 ± 0.41.

As with the TML data, the RML data for BSL-203 and AV-134 are high in the MBB results.

Ignoring SIL-105, the ESTEC-1 average value becomes 0.84 ± 0.02, indicating a very low 1 σ value.

Water Vapour Regain (WVR)

Table 7 gives the $WVR/WVR_{average}$ values in an alternative form to Table 4 (column 11), except for the non-water-sensitive silicones. The water-vapour regain (WVR) measured by CNES is only 27% of the average water-vapour regain, and the DERTS values are 71% higher. These high DERTS values also caused a high total mass loss (Table 5), which means that the pre-conditioning caused the high water sorption. The low WVR data recorded by CNES cannot be traced in the TML data (Table 5), which means that the pre-conditioning was probably much better than the post-conditioning.

Comparison of ESTEC-1 TML data with data from other institutes

The two micro-VCM systems (ESTEC-1 and INTA) gave a good correlation for five of the seven materials (1.03 ± 0.02); the value of 1.19 for the Z-306 paint could be explained by a 30% higher water sorption in the ESTEC sample. The low value of 0.76 for PSG-120 could not be explained, as already mentioned before.

Collected Volatile Condensible Materials (CVCM)

Table 9 shows the $CVCM/CVCM_{average}$ ratio from column 14 of Table 4.

The average value is only taken for six materials. The results for BSL-203 have not been included because of their lowness (0.018%).

The correlation in the CVCM data is very poor, especially for the materials PSE-109, AV-134 and Rilsan. The low 1 σ values for the CVCM measurements (see column 13 of Table 4) might indicate a better correlation. The ESTEC micro-VCM condensors are two chromium-plated aluminium discs (33 mm ø) and one sodium-chloride disc (25 mm ø). No noticeable difference in CVCM was observed with the two different condensor types.

The INTA system had only three sodium-chloride discs, while the CNES macro-VCM system had a quartz disc of 30 mm diameter as condensors. The MBB macro-VCM system with a vacuum balance had an

TABLE 7 $WVR/WVR_{average}$

Institute / Material	ESTEC-1	INTA	CNES	MBB	DFVLR	ESTEC-2	DERTS	SNIAS	WVR_a%
2 PSE-109	1.16	0.91	0.33	1.21	-	-	1.39	-	0.83
4 BSL-203	1.42	1.23	0.19	0.50	-	1.02	1.58	-	0.36
5 Z-306	0.90	0.54	0.16	1.09	-	1.32	1.98	-	1.05
6 AV-134	1.57	1.02	0.19	0.60	-	0.33	2.33	-	0.42
7 Rilsan	1.03	0.76	0.46	1.41	-	1.07	1.26	-	0.26
Average value	1.22	0.90	0.27	0.96	-	0.94	1.71	-	
1 σ ±	0.28	0.28	0.19	0.39	-	0.43	0.44	-	

TABLE 8 Comparison of ESTEC-1 TML results with those from other institutes.

Institute / Material	ESTEC/ INTA	ESTEC/ CNES	ESTEC/ MBB	ESTEC/ DFVLR	ESTEC/ ESTEC-2	ESTEC/ DERTS	SNIAS
1 PSG-120	0.76	0.72	0.94	1.35	-	0.77	0.85
2 PSE-109	1.03	0.99	0.94	1.30	-	0.63	0.94
3 SIL-105	1.03	1.08	0.91	2.11	1.23	1.32	1.02
4 BSL-203	1.04	1.28	0.76	2.30	1.07	1.03	1.19
5 Z-306	1.19	0.97	0.85	1.52	0.85	0.60	1.49
6 AV-134	1.01	1.06	0.80	5.05	1.23	0.80	1.57
7 Rilsan	1.06	0.88	0.98	1.19	0.65	0.85	0.73
Average value	1.02	1.00	0.88	2.11	1.01	0.86	1.11
1 σ ±	0.13	0.17	0.08	1.36	0.25	0.25	0.32

TABLE 9 $CVCM/CVCM_{average}$

Institute / Material	ESTEC	INTA	CNES	MBB	$CVCM_{average}$%
1 PSG-120	0.75	1.16	0.88	1.21	0.173
2 PSE-109	0.22	0.35	1.35	2.08	0.298
3 SIL-105	0.99	0.66	1.15	1.21	0.397
4 BSL-203	2.33	1.61	0.06	0.00	0.018
5 Z-306	0.53	1.11	1.19	1.18	0.102
6 AV-134	0.61	0.69	1.74	0.96	0.105
7 Rilsan	0.91	0.30	1.92	0.87	0.057
Average (BSL-203 not included)	0.67	0.71	1.37	1.25	
$1\sigma \pm$	0.28	0.36	0.39	0.43	

TABLE 11 1σ values for TML in percent of TML

Institute / Material	ESTEC	INTA	CNES	DFVLR
1 PSG-120	4.1	2.1	1.0	1.9
2 PSE-109	0.4	2.4	3.6	6.2
3 SIL-105	0.0	2.3	6.6	0.8
4 BSL-203	4.3	2.7	11.1	4.0
5 Z-306	2.9	12.2	17.7	-
6 AV-134	1.2	2.3	11.9	5.9
7 Rilsan	1.1	4.5	5.7	1.3
Average	2.0	4.1	8.2	3.4

aluminium plate of 70 mm diameter as condensor. The interpretation of the CVCM data cannot lead to any form of conclusion as to which parameter or parameters caused the discrepancies.

The most critical parameters in this case are:

(i) Sample temperature: thermal degradation increases by more than 15% per degree increase in sample temperatures.
(ii) Sticking coefficient of condensor material: perhaps not such a critical parameter as several hundred monolayers of condensed material shield the original material.
(iii) Geometry of sample compartment and condensor arrangement.
(iv) Condensor temperature.

In the case of poor thermal conductivity condensors like NaCl and quartz, one can expect a temperature difference across the condensor. Re-evaporation of condensed materials, with a vapour pressure of some 2×10^{-7} torr at 25°C, might show enormous differences in CVCM values when the condensor temperature varied by only 1°C.

Accuracy of Measurements

Table 10 gives the sum of the 1 σ values for TML, RML and CVCM (from columns 4, 7 and 13 of table 4)

TABLE 10

Sum of 1 σ values

	TML	RML	CVCM	
ESTEC-1	0,18	0.18	0.14	7 materials and 3 samples
INTA	0.48	0.45	0.17	7 materials and 3 samples
CNES	1.07	1.07	0.20	7 materials and 2 samples
DFVLR	0.34	-	-	6 materials and 2 samples

In practice there is no difference between the 1 σ values for TML and RML, which is surprising in that the influence of sorbed water is, in principle, cancelled out in the RML, so that more accurate RML data were expected.

Table 11 gives the 1 σ values for TML in percent TML for the four participants who tested two or three samples of the same material. The largest errors in the INTA and CNES results are due to the Z-306 paint, for which about 74% of the total mass loss is caused by water.
From Table 12 can be concluded that:-
The 1σ values for the TML data seem to be more or less independent of the height of the TML, which implies that 1 σ errors of about 0.13% can normally be expected. The bold figures differ from the average TML values by more than 0.20%.

TABLE 12

Total Mass Loss (%TML) figures from the micro- and macro-VCM tests at INTA, ESTEC and CNES.

Material / Institute	PSG-120	PSE-109	SIL-105	BSL-203	Z-306	AV-134	Rilsan
ESTEC	0.72	5.49	2.64	1.10	1.37	2.60	0.93
	0.72	5.50	2.64	1.16	1.34	2.54	0.92
	0.76	5.47	2.65	1.19	1.41	2.60	0.93
INTA	0.94	5.42	2.57	1.07	1.30	2.48	0.91
	0.98	5.18	2.64	1.13	1.03	2.59	0.85
	0.95	5.38	2.51	1.13	1.11	2.58	0.89
CNES	1.01	5.52	2.33	0.90	1.23	2.75	1.01
	1.03	5.81	2.55	1.00	1.58	2.17	1.10
	-	5.42	-	0.81	-	2.41	-
$TML_{average}$	0.89	5.47	2.57	1.05	1.30	2.52	0.94
$1\ \sigma$	0.13	0.16	0.11	0.13	0.17	0.16	0.08

The acceptable 1 σ values on outgassing results (test are carried out in triplicate) from the micro-VCM test as being fixed in specification ESA/PSS-09/QRM-02T issue 2 are:

(a) $\pm$ 0.05 for TML and RML data up to 0,50% and $\pm \frac{1}{10}$ of the data is excess of 0.50%.
(b) $\pm$ 0.03 for CVCM data up to 0.15% and $\pm$ 1/5 of the data in excess of 0.15%.

The ESTEC and INTA micro-VCM data are within above 1 σ values except for the TML figure on Z-306 from INTA. Also the TML figures on the materials BSL-203, Z-306 and AV-134 from CNES are not within the above limits.

The overall accuracy on TML data around 1.0% is within 15% for the micro-VCM systems.

The overall accuracy on CVCM data around 0.10% is poor, approximately a factor of 2, which figure is high as the present acceptance criteria for spacecraft materials selection are micro-CVCM outgassing figures of TML $\leq$ 1.0% and CVCM $\leq$ 0.10%.

CONCLUSION

Total Mass Loss data of reasonable accuracy can be obtained on organic materials with the micro-VCM, macro-VCM and vacuum-balance systems as long as sample conditioning and test temperature are within the limits as specified in ESA/PSS-09/QRM-02T.

Also special attention should be paid to the test procedures as low water sorption time constants of materials (e.g. down to about 5 minutes for polyurethane paints) may influence the outgassing figures significantly.

The low accuracy of the CVCM data obtained with the micro-VCM and macro-VCM systems, of which systems most of the critical parameters have been fixed within narrow limits, indicate that prediction of spacecraft contamination based on measurements of outgassing rates and condensation rates will be of a much lower accuracy as most of the "contamination critical" spacecraft parameters cannot be predicted very accurately.

Further investigation on outgassing and condensation phenomena as well as on the absolute calibration of outgassing systems seems to be worthwhile in order to find out what the critical parameters are and how critical they are.

PROPOSED IMPROVEMENTS

(i) Better pre- and post-conditioning of samples
(ii) Closer working to specification ESA/PSS-09/QRM-02T, issue 2
(iii) Use of blank condensors to verify the cleanliness of the system
(iv) If possible, closer temperature tolerances; 125 $\pm$ 0.2^{o}C as sample temperature and 25 $\pm$ 0.2^{o}C as condensor temperature
(v) Investigation of the use of a pure material as a standard for equipment calibration.

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