SUBSTITUTION OF THE CALDENE® SMT AS THE CLEANING SOLVENT PRIOR TO COATINGS APPLICATION

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ABSTRACT

To substitute the Caldene® SMT cleaning solvent used until now, acetone and isopropyl alcohol were evaluated.

A complete qualification process has been applied to the main industrial systems (substrate – primer – coating) used for the thermal control of the satellites parts.

This paper summarizes the validation tests which have been done so far to evaluate the new cleaning solvents prior to coatings application.

1. INTRODUCTION

Since its creation in 1986, MAP has used several cleaning solvents prior to the coating's application. Over 1986 to 2013 period, Forane® 141-B, one commercial reference of 1,1-Dichloro-1-fluoroethane was used. It belongs to the hydrochlorofluorocarbon family (HCFC) and is also known as R-141b or HCFC-141b. Due to the detrimental effect of the HCFC molecules towards the Ozone layer, its use was forbidden by the international protocol of Montréal [1]. The use of Forane® 141B for our applications was then substituted with Caldene® SMT in 2013. Caldene® SMT is a non-flammable azeotropic blend formulated based on: Hydrofluorocarbons (HFC 365mfc) and Trans-dichlorethylene (TDCE) [2]. This cleaning solvent has been especially developed for industrial uses.
As the Caldene® SMT solvent used since 2013 will soon become unavailable, alternative cleaning solvents such as acetone and isopropyl alcohol were evaluated in this work. The goal of this study was double, (1) to define alternative to Caldene® SMT and (2) to evaluate a non-commercial formulation to avoid any commercial obsolescence in the next years.
In order to check the properties of the systems obtained using the new cleaning solvents, we used the standard evaluation/qualification process used to qualify the systems for space uses. We have defined the following qualification plan:

1. Control of the coatings at initial stage. The adhesion was defined as the success criterion. Indeed, as the primers and coatings remain the same, only the adhesion could be impacted by the cleaning solvent change;
2. Ageing tests.

Based on our experience and the previous qualification plan made for Caldene® SMT validation [2], we only qualified the most used systems. Additional qualification could be done case by case for specific systems.
This paper presents the results of the qualification of the new cleaning solvents acetone and isopropyl alcohol.

2. MATERIALS, PROCESSES AND TECHNIQUES

2.1. Materials

The coatings and the substrates were selected to cover systems representing the largest application volumes.

Compared to the qualification carried out in 2013, Phosmap 11 has been substituted with MAP® HpO11 [3].

Table 1. Substrates, primers and coatings evaluated

Substrates	Primers	Coatings
2024-T3 alloy 2024-T3 alloy - Surtec 650 6061-T6 alloy - Alodine 6061-T6 alloy - Au 6061-T6 - Ag INVAR Fe36Ni TA6V alloy 304 L Stainless steel CFRP M18/M55J Kapton® HN 50 µm Al 1 face	E' PSX MAP®HpO11	PUK PU1 PNC SG121FD

Industrial grades of acetone (purity > 99.6 %) and isopropyl alcohol (purity > 99.8 %) were used.

2.2. Processes

The new cleaning procedure is as follows:

Depending on the substrates, surface treatment or surface finish, the generic procedure is carried out as follows:

1) Correct cleaning of the surfaces with a lint-free pad soaked in solvent.

2) Light dust removal on surfaces with a Scotch-Brite Multi-Flex 07521A-VFN pad or equivalent. Scuffing depends on the type of substrates. More information is available from MAP concerning the special cleaning procedures.

3) Correct cleaning of the surfaces with a lint-free pad soaked in acetone or isopropanol.

For the following materials and/or cases, acetone was used for the first and third steps:

- General case;
- Materials without surface treatment: Untreated Kapton®, untreated Al, Stainless steel, Invar, all substrate with a polished surface appearance;
- Alodine and Surtec;
- 6061-T6 with silver surface treatment;
- CFRP.

For the following materials isopropyl alcohol was used for the first and third steps:

- 6061-T6 alloy with gold surface treatment
- Bare titanium and titanium alloys.

Isopropyl alcohol was used as acetone cannot be used for gold treated surface nor for titanium. Moreover, halogenated solvent cannot be used for bare titanium [4, 5].

Primers and coatings were applied further to the technical data sheet of the products [6-12].

During the application of these test specimens, the temperature and humidity readings were within these temperature and humidity ranges: 18°C ≤ T≤ 25°C and 40% ≤ RH ≤ 60%

The polymerization cycle used is the fast curing which is composed of two steps:

1. 1h minimum in ambient temperature (18°C – 25°C) and humidity (40 – 60%) conditions;
2. 15 hours at 70°C.

Two specimens were tested, one as the control, and one for the tests.

2.3. Techniques

Adhesion was carried out at initial test and after ageing test by CNES further to ISO 2409 standard [13].

3. QUALIFICATION PLAN

In order to qualify the new cleaning solvents, adhesion must be in 0 to 1 class at initial state and after ageing test. This requirement come from the characteristics of the coatings and from the ECSS-Q-ST-70-17C which defines the durability testing of coatings [14].

Ageing tests consist in the following cumulative steps:

1. Damp heat test (7 days at 50°C and 95% RH);
2. Thermal cycling under vacuum (10 cycles between -170°C and 130°C under N_2 atmosphere);
3. Thermal cycling at atmospheric pressure (90 cycles between -170°C and 130°C under N_2 atmosphere).

4. RESULTS

4.1. INITIAL STATE

Adhesion was measured following the ISO 2409 standard [13]. The values are 0 and 1 class corresponding to the best rankings of adhesion. All the adhesions were compliant.

Table 2. Adhesion at initial state for all the systems tested

Coating	Primer	Substrate	No.	Thickness (µm)	Adhesion	Observation
PUK	**E'**	6061-T6 Alodine	1	58	0/5	NTR[1]
		6061-T6 Au	2	48	0/5	NTR
		2024-T3	3	67	0/5	NTR
		2024-T3 Surtec 650	4	54	0/5	NTR
	MAP® HpO11	6061-T6 Au	5	55	0/5	NTR
		6061-T6 Ag	6	57	0/5	NTR
		2024-T3 Surtec 650	7	71	0/5	NTR
	PSX	6061-T6 Alodine	8	61	0/5	NTR
		6061-T6 Ag	9	61	1/5	NTR
		2024-T3	10	59	0/5	NTR
		INVAR Fe-36Ni	11	NR	0/5	NTR
		TA6V alloy	12	50	0/5	NTR
		2024-T3 Surtec 650	13	66	0/5	NTR
PU1	**E'**	6061-T6 Alodine	14	61	0/5	NTR
		2024-T3	15	68	0/5	NTR
		Stainless steel (304L)	16	60	0/5	NTR
		2024-T3 Surtec 650	17	62	0/5	NTR
	MAP® HpO11	INVAR Fe-36Ni	18	NR	0/5	NTR
		TA6V alloy	19	66	0/5	NTR
		2024-T3	20	64	0/5	NTR
		2024-T3 Surtec 650	21	73	0/5	NTR
	PSX	6061-T6 Alodine	22	73	0/5	NTR
		6061-Ag	23	53	0/5	NTR
		2024-T3	24	70	0/5	NTR
		INVAR Fe-36Ni	25	NR	0/5	NTR
		TA6V alloy	26	49	0/5	NTR
		Stainless steel (304L)	27	65	0/5	NTR
		2024-T3 Surtec 650	28	63	0/5	NTR
PNC	**E'**	6061-T6 Alodine	29	38	1/5	NTR
		2024-T3	30	45	1/5	NTR
		2024-T3 Surtec 650	31	45	1/5	NTR
	PSX	6061-T6 Alodine	32	42	1/5	NTR
		2024-T3	33	40	1/5	NTR
		CFRP (M18/32%/116/55J)	34	NR	1/5	NTR
		INVAR Fe-36Ni	35	NR	1/5	NTR
		TA6V alloy	36	30	1/5	NTR
		Kapton® HN Al 50 µm[2]	37	42	NTR	NTR
		2024-T3 Surtec 650	38	39	1/5	NTR
SG121FD	**E'**	2024-T3 Surtec 650	40	108	0/5	NTR
	PSX	6061-T6 Ag	42	95	0/5	NTR
		2024-T3	43	105	0/5	NTR
		TA6V alloy	44	90	0/5	NTR
		2024-T3 Surtec 650	46	90	0/5	NTR

[1] NTR: Nothing to report
[2] Application on Kapton® face

4.2. AFTER AGEING TESTS

Ageing tests were carried out at the CNES facility.

The conditions of the damp heat (DH) tests were the following:

- Pressure: atmospheric (air) pressure
- Temperature: 50°C
- Relative humidity: 90% RH
- Duration: 7 days

The conditions of the vacuum thermal cycling tests (VTC) were the following:

- Pressure: 5.10^{-6} hPa
- Temperature: -173.7°C / +131.9°C ± 5°C
- Number of cycles: 10

The duration of a cycle was 93 min with a cold stage of 19 min and a hot stage of 19 min. The average increase slope was 11.5°C/min and the average decrease slope was 10.6°C/min.

The conditions of the atmospheric pressure thermal cycling tests (ATC) were the following:

- Atmosphere: nitrogen
- Temperature: -171.1°C / +137°C ± 5°C
- Number of cycles: 90

The duration of a cycle was 101 min with a cold stage of 21.5 min and a hot stage of 20 min. The average increase slope was 10.2°C/min and the average decrease slope was 10.4°C/min.

The values of adhesion measured after ageing tests were 0 and 1 class corresponding to the best rankings of adhesion. All the adhesions were compliant.

Table 3. Adhesion of all the systems tested after cumulative damp heat tests, thermal cycling under vacuum and thermal cycling at atmospheric pressure

					Initial state		DH		DH + VTC	DH + VTC + ATC	
Coating	**Primer**	**Substrate**	**No.**	**Thickness (µm)**	**Adh**	**Obs**	**Adh**	**Obs**	**Obs**	**Adh**	**Obs**
PUK	**E'**	6061-T6 Alodine	1	58	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		6061-T6 Au	2	48	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3	3	67	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3 Surtec 650	4	54	0/5	NTR	0/5	NTR	NTR	0/5	NTR
	MAP® HpO11	6061-T6 Au	5	55	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		6061-T6 Ag	6	57	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3 Surtec 650	7	71	0/5	NTR	0/5	NTR	NTR	0/5	NTR
	PSX	6061-T6 Alodine	8	61	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		6061-T6 Ag	9	61	1/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3	10	59	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		INVAR Fe-36Ni	11	NR	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		TA6V alloy	12	50	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3 Surtec 650	13	66	0/5	NTR	0/5	NTR	NTR	0/5	NTR
PU1	**E'**	6061-T6 Alodine	14	61	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3	15	68	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		Stainless steel (304L)	16	60	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3 Surtec 650	17	62	0/5	NTR	0/5	NTR	NTR	0/5	NTR
	MAP® HpO11	INVAR Fe-36Ni	18	NR	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		TA6V alloy	19	66	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3	20	64	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3 Surtec 650	21	73	0/5	NTR	0/5	NTR	NTR	0/5	NTR
	PSX	6061-T6 Alodine	22	73	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		6061-Ag	23	53	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3	24	70	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		INVAR Fe-36Ni	25	NR	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		TA6V alloy	26	49	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		Stainless steel (304L)	27	65	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3 Surtec 650	28	63	0/5	NTR	0/5	NTR	NTR	0/5	NTR
PNC	**E'**	6061-T6 Alodine	29	38	1/5	NTR	1/5	NTR	NTR	1/5	NTR
		2024-T3	30	45	1/5	NTR	1/5	NTR	NTR	1/5	NTR
		2024-T3 Surtec 650	31	45	1/5	NTR	1/5	NTR	NTR	1/5	NTR
	PSX	6061-T6 Alodine	32	42	1/5	NTR	1/5	NTR	NTR	1/5	NTR
		2024-T3	33	40	1/5	NTR	1/5	NTR	NTR	1/5	NTR
		CFRP (M18/32%/116/55J)	34	NR	1/5	NTR	1/5	NTR	NTR	1/5	NTR
		INVAR Fe-36Ni	35	NR	1/5	NTR	1/5	NTR	NTR	1/5	NTR
		TA6V alloy	36	30	1/5	NTR	1/5	NTR	NTR	1/5	NTR
		Kapton® HN[3]	37	42	NTR	NTR	NTR	NTR	NTR	NTR	NTR
		2024-T3 Surtec 650	38	39	1/5	NTR	1/5	NTR	NTR	1/5	NTR
SG121FD	**E'**	2024-T3 Surtec 650	40	108	0/5	NTR	0/5	NTR	NTR	0/5	NTR
	PSX	Al 6061-T6 Ag	42	95	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		Al 2024-T3	43	105	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		TA6V alloy	44	90	0/5	NTR	0/5	NTR	NTR	0/5	NTR
		2024-T3 Surtec 650	46	90	0/5	NTR	0/5	NTR	NTR	0/5	NTR

[3] Kapton® HN Al 50 µm - Application on Kapton® face

5. CONCLUSION

The use of two new cleaning solvents prior to coatings application has been successfully validated.

The cleaning procedure is based on a three steps process which consists in:

1) Correct cleaning of the surfaces with a lint-free pad soaked in solvent.
2) Light dust removal on surfaces with a Scotch-Brite Multi-Flex 07521A-VFN pad or equivalent. Scuffing depends on the type of substrates.
3) Correct cleaning of the surfaces with a lint-free pad soaked in acetone or isopropanol.

For Kapton® HN, 2024-T3, 6061-T6 Ag, 6061-T6 Alodine, 2024-T3 Surtec 650, Stainless steel (304L), CFRP and Invar, acetone was used.

For 6061-T6 Au and TA6V alloy, isopropyl alcohol was used.

Adhesion of all the systems tested (primers: PSX, E' or MAP® HpO11 and coatings: PU1, PUK, PNC and SG121FD) were compliant at initial state and after cumulative ageing test (Damp heat test, thermal cycling under vacuum and thermal cycling at atmospheric pressure).
The use of the new cleaning solvents acetone and isopropyl alcohol is now validated as the substitution of Caldene® SMT.

6. ACKNOWLEDGEMENTS

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7. REFERENCES

1. https://treaties.un.org/pages/ViewDetails.aspx?src=TREATY&mtdsg_no=XXVII-2-a&chapter=27&clang=_fr
2. CNES qualification report – Caldene® SMT qualification DCT-TV-TH-2013-02868, 2013
3. CNES qualification report – MAP® HpO11 qualification DSO-TB-TH-2018.21999, 2018
4. MAP, cleaning procedure of titanium, pn003_rev7
5. MAP, cleaning procedure of silver or gold surface treated parts, pn007_rev9
6. MAP, Technical data sheet – PSX
7. MAP, Technical data sheet – E'
8. MAP, Technical data sheet – MAP® HpO11
9. MAP, Technical data sheet – PU1
10. MAP, Technical data sheet – PUK
11. MAP, Technical data sheet – SG121FD
12. MAP, Technical data sheet – PNC
13. ISO 2409 standard, Paints and varnishes - cross-cut tests, 2007
14. ECSS-Q-ST-70-17C Durability testing of coatings, 2018