UPDATING THE MAP® ATOX 41-B PRODUCT WITH TOLUENE-FREE THINNER

G. Sierra [(1)], S. Gomes [(1)], O. Guillaumon [(1)]

[(1)] MAP, ZI - 2 Rue Clément Ader, 09100 Pamiers, France, g.sierra@map-coatings.com

ABSTRACT

MAP® ATOX 41-B is a low outgassing silicone varnish developed in the 1980s. Until now, a toluene-based thinner was used to allow spray gun pulverization.

In order to propose safer products for people and less-environmental impact, the thinner composition has been updated by eliminating the toluene.

This paper summarizes the validation tests which have been done so far to characterize the new version of MAP® ATOX 41-B. All the properties were compared to the current version of MAP® ATOX 41-B.

1. INTRODUCTION

Since its creation in 1986, MAP has developed numerous products for the space industry. Most of these products are silicone-based adhesives, varnishes or coatings.
MAP® ATOX 41-B is a low outgassing silicone varnish obtained via a solvent-free purification process (patented by CNES) that makes it possible to obtain degassing values compatible with space applications [1]. This product has been developed to be sprayed on printed circuit boards (PCB) and electronic components. The sprayable property is obtained thanks to the use of a thinner.
Until now, the thinner is toluene-based. In order to remove toluene from its composition, we have developed a new thinner with less environment and human impact.
In order to check the properties of the new version of MAP® ATOX 41-B, we have defined the following qualification plan:

1. Control of the product at initial stage and comparison of the properties of the new version of MAP® ATOX 41-B with the current one;
2. Ageing tests.

As the composition of the MAP® ATOX 41-B was not changed, only those of the thinner was updated, it was decided to carry out a partial qualification to validate this change.
This paper first presents the properties of the new version of MAP® ATOX 41-B at initial state. These properties are compared to those of the current version. Secondly, the results after ageing tests are presented.

2. MATERIALS, PROCESSES AND TECHNIQUES

2.1. Materials

MAP® ATOX 41-B is a two-component RTV-2 silicone elastomer. The base is composed of silicone polymers and catalyst. The hardener is composed of a mix silicone polymers and cross-linker. Base and hardener are 100% solids content products. In order to reach the low outgassing rates as defined by the ECSS [1], a solvent-free purification process is used.

To obtain the final material, it is necessary to mix the base and the hardener in the respective weight proportions of 100 to 10. The mix is then diluted with the thinner in a weight ratio varying from 60% to 80%. The standard curing process corresponds to (1) 7 days at 23°C and 55% relative hygrometry (RH) whereas an alternative one is (2) 4h of pre-curing @ 25°C + 12h at 65°C. The chemical reaction gives a final elastomer. The main characteristics of the current elastomer [2, 3] are listed in Tab.1 and Tab.2.

Table 1. General properties of current MAP® ATOX 41-B diluted with toluene-based thinner with a weight ratio of 60% to 80%

Viscosity – ISO cup 4 (s)	55 – 70
Viscosity – Afnor cup 4 (s)	23 - 33
Pot-life (min) at 20°C	120

Table 2. General properties of current MAP® ATOX 41-B silicone varnish cured at 23°C and 55% RH for at least 24 hours

TML (%)	0.45
RML (%)	0.45
CVCM (%)	0.05
Electrical surface resistance (Ω/□)	$>10^{14}$

PSX primer is used on aluminium and PCB substrates in order to improve the adhesion and to get a compliant adhesion (0 class) further to the ISO 2409 standard [4]. PSX primer was applied further to the indications

mentioned on the TDS [5] using a spray gun pulverization.
The PCB samples were prepared further to the steps hereunder:

1. PSX primer application using a spray gun pulverization with the parameters defined on the TDS [5];
2. MAP® ATOX 41-B application with a spray gun pulverization further to the TDS [3].

Arlon 35N PCB from Systronic – Cimulec were used [6]. The thickness of the coatings was around 20 µm.

2.2. Techniques

Outgassing rates are measured further to ECSS-Q-ST-70-02C standard [1]. The measurements were taken at Airbus Toulouse.

All the other characteristics were measured in-house by MAP further to the following ISO standards which are included in the reference section:

- Viscosity and pot-life using ISO cups [7] and AFNOR cups [8];
- Surface electrical resistance further to the ASTM D257-99 standard [9];
- Adhesion further to ISO 2409 standard [4].

3. QUALIFICATION PLAN

In order to qualify the new version of MAP® ATOX 41-B, its characteristics must meet the requirements listed in Tab.3. These requirements come from the characteristics of the current MAP® ATOX 41-B and from the ECSS-Q-ST-70-02C outgassing standard [1].
The viscosity is measured on the MAP® ATOX 41-B silicone resin thinned with 60 to 80 % MAP®ATOX 41-B thinner.

Table 3. Requirements for MAP® ATOX 41-B silicone resin

Properties	Requirements
RML (%)	≤ 1
CVCM (%)	< 0.1
Adhesion	0 class

The characterization of the products was performed at the initial state for all the characteristics: rheological, outgassing, and electrical properties.

Some of the characteristics were characterized after a damp heat test (7 days at 50°C and 95% RH) and after a cumulative damp heat test + thermal cycling under vacuum (10 cycles between -170°C and 130°C under N_2 atmosphere) + thermal cycling at atmospheric pressure (90 cycles between -170°C and 130°C under N_2 atmosphere).

4. RESULTS

4.1. INITIAL STATE

4.1.1. GENERAL PROPERTIES

The outgassing properties were measured at the Airbus Toulouse facility on a product after 7 days curing at 23°C and 55% relative hygrometry. The results are listed in Tab.4 [10, 11].

Table 4. Outgassing results for MAP® ATOX 41-B cured during 7 days at 23°C and 55% RH

MAP® ATOX 41-B	TML (%)	RML (%)	CVCM (%)
Current version MAP®ATOX 41-B Thinner	0.45	0.45	0.05
New version MAPSIL® QS Thinner	0.54	0.52	0.02

4.1.2. RHEOLOGICAL PROPERTIES

The values of the viscosity measurements are listed in Tab.5. The viscosity is measured on the MAP® ATOX 41-B silicone resin thinned with 75 to 85 % MAPSIL® QS thinner. A slight increase is observed using MAPSIL® QS thinner versus MAP® ATOX 41-B thinner. Nevertheless, the spraying process parameters are kept the same.

Table 5. Viscosity measurements for MAP® ATOX 41-B silicon resin thinned with MAPSIL® QS thinner

Properties	Value
Viscosity – ISO cup 4 at 23°C (s)	75 -97
Viscosity – Afnor cup 4 at 23°C (s)	31 - 41

The pot-life was kept at an identical value of 120 minutes at 20°C for the new version of MAP® ATOX 41-B.

4.1.3. ADHESION

For the new version of MAP® ATOX 41-B, adhesion was measured following the ISO 2409 standard. The measurements were carried out on aluminium and PCB samples after PSX primer application. The values are 0 class corresponding to the best ranking of adhesion.

4.1.4. ELECTRICAL PROPERTIES

MAP® ATOX 41-B is an electrical insulating material. The electrical surface resistance was greater than 2.0 x 10^{12} Ω/□.
These values are the same as those measured using the current version (Table 1).

4.2. AFTER AGEING TESTS

Ageing tests were carried out at the CNES facility for the current and the new versions of MAP® ATOX 41-B. A damp heat test was done at 50°C and 95% RH for 7 days. Additional thermal cycling tests were performed following two steps:

1. Thermal cycling tests under vacuum. 10 cycles were performed between -170°C and 130°C with a 10-minute plateau at high and low temperatures (gradient = 5°C/min);
2. Thermal cycling tests were performed under N_2 atmosphere. 90 cycles were performed between -170°C and 130°C with a 10-minute plateau at high and low temperatures (gradient = 5°C/min).

The results are shown in table 6. The adhesion of the coatings is 0 class on 5 and does not evolve during the ageing test.
The recorded temperatures under the thermal vacuum cycles were the following: -172°C/+130°C. For the thermal cycling at atmospheric pressure the temperatures were the following: -176°C/+135°C.

Table 6. Adhesion characteristic of MAP® ATOX 41-B at initial state and after ageing tests – New version of MAP® ATOX 41-B cured during 7 days at 23°C and 55% RH

MAP® ATOX 41-B New version	Initial state	After damp heat test	After damp heat test + Thermal cycling
Adhesion	0 / 5	0 / 5	0 / 5

5. CONCLUSION

The current version of MAP® ATOX 41-B (MAP® ATOX 41-B Thinner) is proposed with a toluene-based thinner to allow spray gun pulverization. In order to propose safer products for people and less-environmental impact, the thinner composition has been updated by eliminating the toluene.
The new version of MAP® ATOX 41-B (MAPSIL® QS Thinner) was characterized at initial state and after ageing tests (damp heat test and cumulative thermal cycling). The characteristics were compared to those of the current version. A slight increase in the viscosity of the mix is observed using MAPSIL® QS thinner versus MAP® ATOX 41-B thinner which does not change the spraying process parameters. The outgassing properties and adhesion characteristics remained the same for the new version of the MAP® ATOX 41-B.

6. ACKNOWLEDGEMENTS

The authors wish to thank P. Nabarra and S. Perraud from CNES for their assistance in carrying out the ageing tests.

7. REFERENCES

1. ECSS-Q-ST-70-02C, Thermal vacuum outgassing test for the screening of space materials, 2008
2. Qualification of adhesives and potting resins produced by MAP, CNES report, 89/CT/DRT/TVE/TH n°074, February 7, 1989;
3. MAP, Technical data sheet – MAP® ATOX 41-B
4. ISO 2409 standard, Paints and varnishes - cross-cut tests, 2007
5. MAP, Technical data sheet – PSX
6. Arlon 35N technical data sheet
7. NF EN ISO 2431 standard, Paints and varnishes — Determination of flow time by use of flow cups, 2011
8. NF T 30-014 standard, Determination of the flow time of paints, varnishes and similar preparations using French cups, 1983
9. ASTM D257-99 standard, Standard Test Methods for DC Resistance or Conductance of Insulating Materials, 2007
10. Outgassing report, Airbus M19943.8, 2020
11. CNES qualification report – MAPSIL 213 DCT-TV-TH-2012-13180, 2012