# Evaluating low & ultra-low outgassing silicones for space applications

WHITE PAPER

## INTRODUCTION

For decades, silicone adhesives, coatings and elastomers have been used in space applications due to their broad operating temperatures, ability to maintain elasticity over these temperatures and low modulus to reduce stress. These properties make them ideal for bonding and protecting sensitive components and electronics from vibration, shock, dust, moisture, chemicals and other environmental factors.

However, not all silicones are created equal. Some may outgas volatile materials that can condense on and contaminate sensitive surfaces, such as windows, radiators, lenses, solar arrays, sensors or other optically sensitive surfaces. These outgassed materials can further degrade due to ultraviolet (UV) and atomic oxygen (AO) exposure, causing adverse effects and shortening the lifetime of hardware.[1]

To counteract these conditions, silicone manufacturers like NuSil® offer highly purified, low outgassing silicones that satellite and space vehicle manufacturers can use to avoid contamina-tion and prevent material degradation.

The value of low outgassing silicones for space-based applications is most commonly seen in the assembly of solar panels. Solar panels are assembled in a variety of ways, but they all have three things in common:

- A substrate as the foundation of the panel, often built from a lightweight honeycomb composite
- The solar cells themselves
- Protective covering glass

Low outgassing silicone adhesives are used to adhere the solar cells to the substrate; some panel manufacturers use silicone primers to prep the substrate for adhesive application. CV silicones are also widely used to adhere the protective covering glass to the solar cell.

## NASA LOW OUTGASSING AND ULTRA-LOW OUTGASSING TESTING AND EVALUATION STANDARDS

NASA uses ASTM E595 to evaluate and screen materials for space applications.[2] The acceptance limits for these tests are ≤ 1.0 % total mass loss (TML) of the material and ≤ 0.1 % collected volatile condensable materials (CVCM). When materials are used in more sensitive areas on spacecraft, more stringent and detailed testing may be required where CVCM acceptance limits may be ≤ 0.01 %, and NASA recommends ASTM E1559.[3]

The advantage of ASTM E1559 is that it not only measures TML and volatile condensable materials (VCM), but it also measures outgassing as a function of time, temperature and the deposition rate of outgassed materials on different temperature surfaces. Having this kinetic data helps to model the outgassing process to determine whether it is diffusion-limited, reaction-limited or a combination thereof.[1] One can calculate the total outgassing amount from the material based on the initial results and overall intended time used in the application to help guide proper material selection.[4]

## MATERIALS TESTED

In the past, NuSil analyzed a suite of products, via ASTM E595 and E1559, detailing the chemistry and processes used to make the controlled volatility (CV) and the Ultra-Low Outgassing product lines (SCV), and their effects on outgassing kinetics.[5,6,7] In these studies, only addition (platinum) cure materials were investigated. Since these are not the only type of materials used in space applications, it is important to understand how other cure chemistries and material forms, such as condensation cure and sheeting applications, compared to the past work on addition cure materials.

The goal is to develop a holistic understanding of how every space-grade material performs to help guide design engineers toward the best possible material solutions for their specific space applications.

**TABLE 1:** Summary of NuSil products tested by ASTM E 595 and E 1559. The shading groups products of similar chemistry and/or applications.

| Sample | Product Family | Materials Chemistry | ASTM E595 (TML/CVM) | ASTM E1559 TML (80 K QCM)/ VCM (298 K QCM) | Outgassing Rate (pg/cm²/s)/(pg/gm/s) |
|---|---|---|---|---|---|
| CV-2289-1 | Adhesive/potting/encapsulating | 2-Part pt cure, diphenyl-dimethyl | 0.52/0.05 | 0.43485/0.01159 | 207.5/2,756 |
| SCV-2585 | Adhesive/potting/encapsulating | 2-Part pt cure, diphenyl-dimethyl | 0.06/0.00 | 0.08498/0.00091 | 35.1/237 |
| CV10-2568 | Ablative, adhesive/potting | 2-Part pt cure, diphenyl-dimethyl | 0.44/0.03 | 0.29972/0.00591 | 109.0/2,685 |
| SCV-2586 | Adhesive/potting | 2-Part pt cure, diphenyl-dimethyl | 0.07/0.01 | 0.07145/0.00054 | 20.3/470 |
| CV16-2500 | Embedding/potting, clear | 2-Part pt cure, diphenyl-dimethyl | 0.05/0.01 | 0.06536/0.00088 | 108.5/977 |
| SCV1-2590 | Embedding/potting, optically clear | 2-Part pt cure, dimethyl | 0.04/0.00 | 0.04613/0.00121 | 36.3/526 |
| CV-2646 | Electrically conductive | 2-Part alkoxy cure, diphenyl-dimethyl | 0.09/0.01 | 0.07613/0.00323 | 31.5/173 |
| SCV1-2596 | Electrically conductive | 2-Part pt cure, dimethyl | 0.02/0.00 | 0.01920/0.00083 | 26.9/172 |
| CV-1142 | Adhesive, sealant | 1-Part oxime cure, diphenyl-dimethyl | 0.43/0.02 | 0.31453/0.01035 | 93.5/1,436 |
| CV-1144-0 | Coatings (AO, electronics) | 1-Part oxime cure, dispersion, diphenyl-dimethyl | 0.52/0.09 | 0.30517/0.03992 | 140.6/2,433 |
| CV-1152 | Conformal coating | 1-Part oxime cure, diphenyl-dimethyl | 0.28/0.03 | 0.18337/0.01948 | 71.0/1,216 |
| CV4-1161-5 | Tape, temporary bonding | Cured, dimethyl | 0.42/0.03 | 0.17138/0.03096 | 23.8/1,454 |
| CV-2680-12 | Film adhesive | 2-Part pt cure, dimethyl | 0.36/0.06 | 0.19118/0.02445 | 35.3/1,795 |

**TABLE 2:** Cumulative amounts of outgassed materials as percentage of total mass loss

| Sample | Materials Chemistry | Very High Volatility % | High Volatility % | Medium Volatility % | Low Volatility % |
|---|---|---|---|---|---|
| CV-2289-1 | 2-Part pt cure, diphenyl-dimethyl | 21.8 | 43.7 | 31.8 | 2.7 |
| SCV-2585 | 2-Part pt cure, diphenyl-dimethyl | 89.7 | 5.5 | 3.7 | 1.1 |
| CV10-2568 | 2-Part pt cure, diphenyl-dimethyl | 33.9 | 41.4 | 22.8 | 2.0 |
| SCV-2586 | 2-Part pt cure, diphenyl-dimethyl | 80.6 | 7.9 | 10.8 | 0.8 |
| CV16-2500 | 2-Part pt cure, diphenyl-dimethyl | 80.2 | 9.4 | 9.0 | 1.3 |
| SCV1-2590 | 2-Part pt cure, dimethyl | 71.5 | 15.5 | 10.4 | 2.6 |
| CV-2646 | 2-Part alkoxy cure, diphenyl-dimethyl | 16.6 | 10.5 | 68.6 | 4.2 |
| SCV1-2596 | 2-Part pt cure, dimethyl | 71.6 | 12.8 | 11.2 | 4.3 |
| CV-1142 | 1-Part oxime cure, diphenyl-dimethyl | 51.7 | 30.2 | 14.8 | 3.3 |
| CV-1144-0 | 1-Part oxime cure, dispersion, diphenyl-dimethyl | 25.4 | 26.1 | 35.4 | 13.1 |
| CV-1152 | 1-Part oxime cure, diphenyl-dimethyl | 23.3 | 38.6 | 27.5 | 10.6 |
| CV4-1161-5 | Cured, dimethyl | 15.6 | 6.8 | 59.5 | 18.1 |
| CV-2680-12 | 2-Part pt cure, dimethyl | 24.7 | 26.0 | 36.5 | 12.8 |

## TESTING METHODOLOGY

For complete details on how the ASTM E595 and ASTM E1559 tests were conducted, see the “Testing Methodology” section of the appendix.

### Results of ASTM testing

As space components become more advanced and sensitive to contamination, it will be important to make sure the materials with the best physical properties for the intended application also have the appropriate outgassing characteristics to ensure mission success.

Tables 1 and 2 and Figures 1 through 4 summarize the NuSil silicones tested by both ASTM E595 and E1559. Figure 1 demonstrates that the TML for space-grade condensation cure materials, tape and film adhesive have similar performance levels as the other addition cure low outgassing materials tested. This expands the possibilities for different cure chemistries and form factors for use in space applications, making it possible to then focus on the physical properties of the material for the intended application.

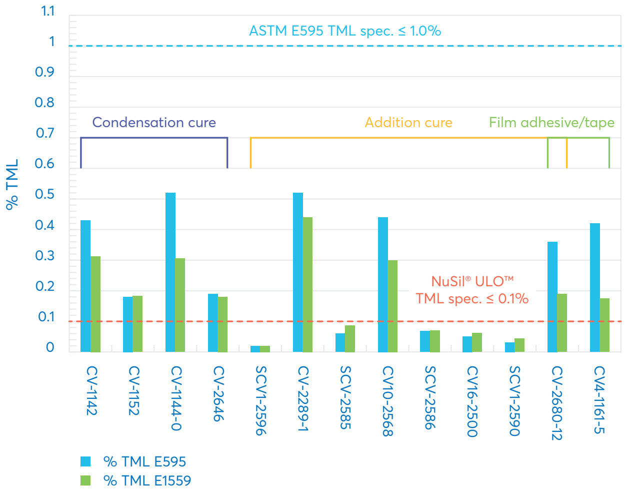


**FIGURE 1:** Comparison of the % TML at the end of testing by ASTM E 595 vs. E 1559

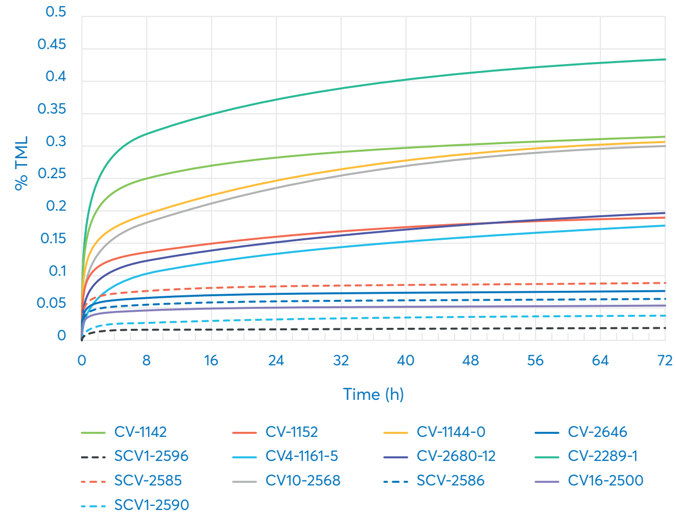


**FIGURE 2:** Comparison of the TML % (80 K QCM) over time for NuSil products tested by ASTM E 1559. Dashed lines are ultra-low outgassing materials and solid lines are low outgassing materials.

For the most stringent space applications, where even the low amounts of volatiles might outgas from traditional space-grade materials and cause potential problems, NuSil developed ultra-low outgassing materials. These materials are held to a much higher standard with a TML of ≤ 0.1 % and CVCM ≤ 0.01 % by ASTM E595. When comparing ultra-low outgassing to low outgassing materials, TML is reduced by approximately four to nine times, as tested by both ASTM E595 and E1559 (Table 1, Figures 1 and 2).

Evaluating TML as a function of time, ASTM E1559 (Figure 2) showed a sharp rise in TML during the first two to four hours of the testing for all samples. Then, the low outgassing materials started to level off between 24 to 48 hours, whereas the ultra-low outgassing materials leveled off much earlier, within four to eight hours.

The percentage of CVCM from ASTM E1559 (Figure 3) was just as important as TML since the materials that condensed on the 298 K QCM (quartz crystalline microbalance) were higher molecular weight materials with lower volatility. These materials were the likely candidates to cause issues with other sensitive equipment and further degraded upon UV and AO exposure, and therefore should be avoided. While all materials had CVCM of ≤ 0.04 % over 72 hours of testing, ultra-low outgassing silicones demonstrated their advantages by having CVCM values of ≤ 0.001 %, a full order of magnitude less compared to the low outgassing silicones.

When taking a closer look at the different types of outgassed materials (Table 2), understanding the percent total mass loss was very important for space applications because of what the volatiles indicated. Very high volatility corresponded to water and solvents while high, medium and low volatilities corresponded to molecular weights of approximately 50 to 200, 200 to 400 and ≥ ~400 atomic mass units (AMU), respectively.[8] All the ultra-low outgassing silicones had > 70 % of their cumulative volatiles of very high volatility. This meant there were minimal amounts of medium or high molecular weight materials that caused contamination issues. Therefore, these materials are preferred for applications in sensitive areas.

Outgassing rate data for all of the silicones tested can be seen in Figures 4. In the first few hours, the outgassing rates started higher. Then, they stabilized in the five- to ten-hour range and began reaching a steady state towards the end of testing.

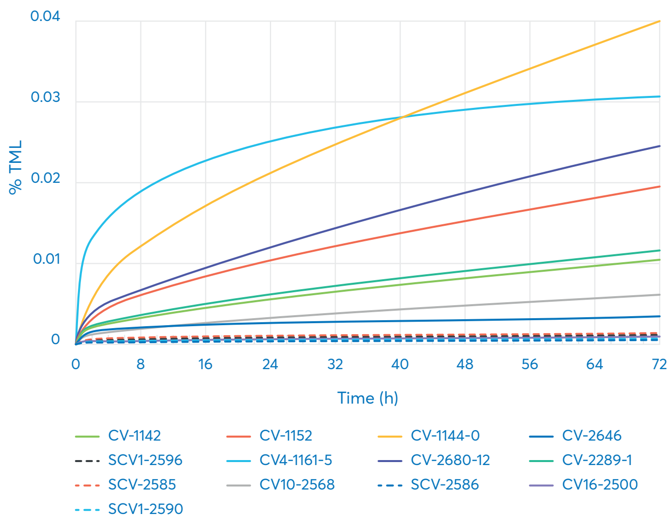


**FIGURE 3:** Comparison of the VCM % (298 K QCM) over time for NuSil products tested by ASTM E 1559. Dashed lines are ultra-low outgassing materials and solid lines are low outgassing materials.

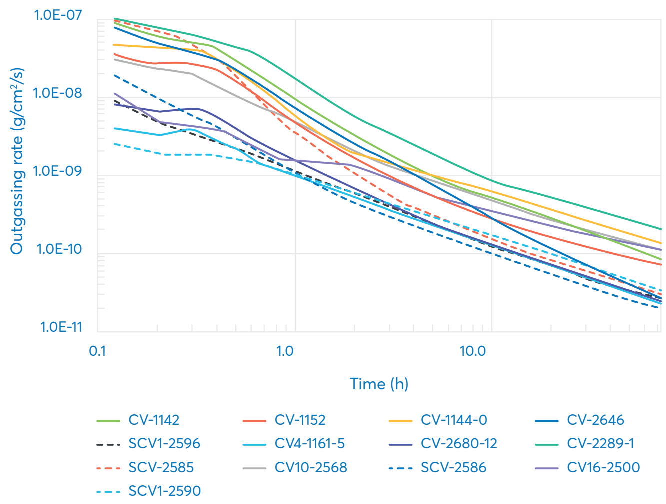


**FIGURE 4:** Comparison of outgassing rates (80 K QCM) for different NuSil products from ASTM E 1559 testing. Dashed lines are ultra-low outgassing silicones and solid lines are low outgassing silicones.

The different slopes of the outgassing curves at the beginning of testing compared to the end highlighted the different processes occurring in the samples. These processes can include reaction and/or diffusion-limited outgassing kinetics combined with possible nonrandom distribution of outgassing materials.[1] The initial high outgassing rates were likely due to outgassing from materials at or near the surface, transitioning into more of a diffusion-limited process.

Outgassing rates for the ultra-low outgassing silicones dropped off significantly faster at the beginning of testing compared to the low outgassing materials and, in most cases, ended up approximately three- to six-times lower by the end of the test (Table 1)

## CONCLUSIONS:

As spacecraft materials and components become more advanced, specialized and sensitive, a multitude of different low outgassing materials to suit each application are needed. In this study, all materials were measured by ASTM E595 and ASTM E1559 to better understand the outgassing and kinetics of the process to guide engineers when selecting the appropriate material for their application.

In some cases, an addition cure chemistry may be preferred over a condensation cure, while in other situations, a condensation cure chemistry may be preferred over an addition cure. However, in both cases, the materials should have low outgassing characteristics to reduce the contamination of sensitive equipment. Both addition cure and condensation cure low outgassing silicones offer similar performance in terms of TML and outgassing rates, so one does not have to compromise performance for the cure chemistry best suited for the application.

For the most stringent applications, NuSil ultra-low outgassing materials were developed, which had an order of magnitude lower TML (< 0.1 %) and CVCM (< 0.01 %) when tested according to ASTM E595, compared to their low outgassing counterparts. In addition, these materials had much lower (approximately three to six times) outgassing rates while offering the same physical property performance.

This demonstrated that they offered an ideal solution for sensitive areas where contamination must be kept to a minimum to extend hardware operation lifetimes. Having material options for numerous applications with either addition or condensation cure along with other form factors, such as tapes and film adhesives, expands the toolbox for engineers to design the most advanced performing spacecraft.

## TESTING METHODOLOGY

### ASTM E5959

This test method is used to measure the volatile content of materials when exposed to a vacuum environment. The parameters measured are total mass loss (TML) and collected volatile condensable materials. One additional parameter to be measured is water vapor regained (WVR).

Before testing, the sample is preconditioned at 23 C and 50% relative humidity for 24 hours. Next, the sample is weighed and then placed in the test stand and the chamber is evacuated to a pressure of ≤ 5 X 10-5 torr. The samples are heated to 125 C and then held under these conditions for 24 hours. As volatile materials come off the sample, they escape through an exit port where they can condense on a collector plate (preweighed) held at 25 C. After the test is over, the sample and collector plate are weighed. TML % and CVCM % are measured using the following equations:

01. TML % = $((M_{S,i} - M_{S,f})/M_{S,i}) \times 100$
02. CVCM % = $((C_i - C_f)/M_{S,i}) \times 100$

With: $M_{S,i}$ = initial sample mass

$M_{S,f}$ = final sample mass after testing

$C_i$ = initial collector plate mass

$C_f$ = mass of collector plate after testing

### ASTM E155910

This test method is used to measure the outgassing kinetics of a sample as a function of time, temperature and vacuum. The data produced can then be used in models to predict the amounts of outgassing materials and their potential deposition on spacecraft surfaces over time. Further details of the test method and apparatus have been published previously.[3,6] A brief overview is given below along with the testing parameters.

Testing parameters:

- Sample temperature: 125 C
- Sample mass range for tested materials: (~0.9 to 5.6 g)
  - Sample masses allowed, according to the ASTM, can range between 0.5 to 10.0 g (not including substrate)
- Material state: Cured
- Test duration: 72 h
- QCM temperatures: 80 K, 160 K, 220 K, 298 K
- Chamber pressures were 10-10 to 10-8 torr
- View factor from a QCM to the sample was 415.02 cm2
- Sensitivity of each of the four QCMs was 4.43 x 10-9 g/cm2/Hz

Before testing, the sample is preconditioned at 23 C and 50% relative humidity for 24 hours and then weighed. After preconditioning and weighing the sample, it is placed in a tempera-ture-controlled effusion cell in a vacuum chamber. During the test, the outgassing materials exit the effusion cell and will condense on one of four Quartz Crystal Microbalances (QCMs) that are temperature-controlled at 80, 160, 220 and 298 K.

The TML and outgassing rate data for the sample are determined based on the mass of materials that condense on the 80 K QCM throughout the test. The higher temperature QCMs are used to collect Volatile Condensable Material (VCM). In this paper, the VCM data discussed is based on the mass of outgassed materials that condensed on the 298 K QCM. The materials that outgas during the isothermal test are also monitored by a mass spectrometer to help aid in the identification of the actual outgassing materials.

Molecular weight ranges for the materials in the different volatility categories can be estimated based upon engineering experience related to materials condensability and mass spectrometer data. The "very high volatility" group (80 K QCM) of materials is primarily water and solvents. The "high volatility" (160 K QCM) materials most likely have molecular weights of 50 to 200 atomic mass units (amu,) the "medium volatility" (220 K QCM) materials fall in the 200 to 400 amu range, and the "low volatility" (298 K QCM) materials probably have molecular weights above 400 amu.

After the test is complete, QCM thermogravimetric analysis (QTGA) is performed by heating the QCMs at 1 K/min from their experimental temperature up to 398K. The mass loss of the QCM is plotted as a function of time and temperature, and this helps identify how many different types of materials are present and at what temperatures they will condense or evaporate.

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