

Determination of volatile compounds in silicone consumer products

Version 02
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1. Scope and field of application

This method describes a procedure for the determination of the release of volatile compounds from consumer products made of silicone.^{1,2}

CAUTION – The use of this procedure may involve the use of hazardous substances, operations and equipment. This procedure does not claim to address all security issues associated with its use. It is the responsibility of the user of this procedure to take appropriate health and safety precautions and to determine the applicability of any restrictive regulations prior to use.

2. Principle

The release of volatile compounds from silicone materials intended to come into contact with food and/or to come into contact with mucous membranes (e. g. soothers) must not exceed 0.5 weight percent [% (w/w)]. The cut sample is conditioned at 100 °C for 1 hour to remove sorbed water before treatment at 200 °C for 4 hours (high temperature treatment). The weight loss during the high temperature treatment is determined gravimetrically.

3. Apparatus and materials

3.1. Balance

Analytical balance with an accuracy of 0.1 mg or better.

3.2. Heating chamber

Heating chamber covering a temperature range from 100±5 °C to 200±5 °C with the possibility to disable forced convection.

3.3. Desiccator

Desiccator with suitable desiccant (e.g. anhydrous calcium chloride or silica gel with moisture indicator).

3.4. Weighing dishes

Weighing dishes made of electrically conductive material, e.g. stainless steel, platinum, aluminum.

NOTE – The use of electrically non-conductive or poorly conductive materials such as soda–lime glass, borosilicate glass or quartz glass is strongly discouraged because these materials do not or only slowly discharge static electricity. When using these materials, the measurement error for 10 g of sample material can exceed ± 50 mg.

3.5. Aluminum foil

Aluminum foil, heated at 380 °C for at least 48 hours.

4. Procedure

The experiments are performed in triplicate for each sample. Therefore, at least 30 g of material are required per sample.

In order to minimize the sorption of water, the exposure time of the samples to ambient air must be kept as short as possible. This applies both to the weighing itself as well as to the transport of the samples from the heating chamber (3.2) to the desiccator (3.3) and from the desiccator (3.3) to the balance (3.1). However, the storage time in the closed desiccator between weighing after conditioning and before high temperature treatment is not critical.

NOTE – Both, the conditioned and the high temperature treated samples can quickly sorb water from the air. A sample of 10 g can sorb more than 10 mg of water in a few minutes. This can negatively affect stable readings on the balance. Therefore, the weighing time should not exceed 30 seconds. If no stable reading is possible during this time, the lowest value at the beginning of the weighing process should be used. The transfer time from the desiccator to the balance should be kept as short as possible.

If the heating chamber (3.2) is equipped with setups enabling forced ventilation and/or fresh air supply, these must be disabled during conditioning (4.2) and high temperature treatment (4.3). Forced ventilation may only be used, if necessary, during the heating phases for faster and uniform adjustment of target temperatures in the unloaded heating chamber.

NOTE – Forced ventilation and/or fresh air supply during the high temperature treatment or conditioning can cause higher weight losses with higher standard deviations and diminished reproducibility.

4.1. Sample preparation

Before the test, the weighing dishes (3.4) are heated in a heating chamber (3.2) at 200 °C for 4 hours, then cooled in a desiccator (3.3) for at least 60 minutes and stored therein until the beginning of the experiment.

The sample is cut into pieces of approx. 1×2 cm. The empty weighing dish (3.4) is weighed with an accuracy of ±0.1 mg (M_{empty}). Subsequently, ca. 10 g of the sample is weighed on the weighing dish. The single pieces must overlap as little as possible.

NOTE – If necessary, e.g. for large sample numbers, the samples can be weighed in 10 g portions beforehand, stored in aluminum foil (3.5) and later transferred onto the weighing dishes (3.4) for accurate weighing.

4.2. Conditioning of the sample

To remove sorbed water from the sample pieces, the weighing dish (3.4) containing the sample is heated in a heating chamber (3.2) at 100±5 °C for 60±5 minutes without forced convection. The weighing dish (3.4) with the conditioned sample is cooled in a desiccator (3.3) for 30±5 minutes before the total weight (M_{cond}) of the weighing dish (3.3) and the conditioned sample is determined with an accuracy of ±0.1 mg.

4.3. High temperature treatment of the sample

The weighing dish (3.3) containing the conditioned sample is heated in a heating chamber (3.2) at 200±5 °C for 4 hours ±5 minutes without forced convection, then quickly transferred to the desiccator (3.3) and cooled therein for 60±5 minutes. The total weight (M_{HT}) of the weighing dish and the high temperature-treated sample is then determined with an accuracy of ±0.1 mg.

5. Calculation of the content of volatile compounds

The content of volatile compounds is calculated as follows:

$$\text{Volatile compounds } [\% (w/w)] = ((M_{cond} - M_{HT}) / (M_{cond} - M_{empty})) * 100$$

The result is to be determined as the mean value of three individual measurements.

6. Reporting of Results

The content of volatile compounds and the corresponding measurement uncertainties (including the coverage factor) are given in % (w/w) to 3 significant digits. An expanded relative measurement uncertainty of 25% (probability of 95%; $k = 2$) was determined from the data of a method evaluation study.²

e.g. 0.375 ± 0.094 % (w/w) ($k = 2$)

7. Literature

1. Towards a better comparability during GMP assessment – Identifying the main parameters that influence the loss of volatile organic compounds from silicone elastomers. Krüger, O.; Ebner, I.; Kappenstein, O.; Roloff, A.; Luch, A.; Bruhn, T.; *Food Packaging and Shelf Life* **2021**, 30, 100758. DOI 10.1016/j.fpsl.2021.100758.
2. Method evaluation study on the determination of volatile compounds in silicone materials; Krüger, O.; Ebner, I.; Roloff, A.; Kappenstein, O.; Bruhn, T.; *BfR-Wissenschaft* **2021**. DOI 10.17590/20210519-121342.

List of modifications

Version 01, 03/2022	First publication on the website of the NRL-DE-FCM
Version 02, 09/2023	An example of silicone materials intended to come into contact with mucous membranes was added under "2. Principle": The release of volatile compounds from silicone materials intended to come into contact with food and/or to come into contact with mucous membranes (e. g. soothers) must not exceed 0.5 weight percent [% (w/w)].