

TEST REPORT

TÜV SÜD Industrie Service GmbH

Department of Chemical Analysis
Ridlerstraße 65
80339 Munich, Germany



Industrie Service

Add value.
Inspire trust.

Report No: **19-V1904-01-en**
This is an unofficial translation. Only the German version dated 2019-04-09 is binding.

Client: VICI AG International
Dr. Andre Hütz
Parkstr. 2
CH-6214 Schenkon

Date of order: 2019-01-14

Date: 2019-05-20

Our reference:
IS-USL-MUC/qs

Order number: ---

This Document consists of
7 Pages.
Page 1 of 7

Sample arrival: 2019-01-18

Test sample: **VICI cap, 3 ports, GL45 thread (# JR-S-11002)
(VICI AG International)**

Excerpts from this document
may only be reproduced and
used for advertising purposes
with the express written approval
of
TÜV SÜD Industrie Service
GmbH.

Test period: 2019-02-18 – 2019-03-18

The test results refer exclusively
to the units under test.

Test specification: Customer-specific requirement –
**Comparison of emissions from solvents and solvent
mixtures from bottles with VICI Safety Caps and unsealed
bottles**


(Dipl.-Ing. Gabriele Glomsda)
Head of Department


(Dr. Quirin Sinz, State-certified food chemist)
Technical Expert



1. Subject of the order

A comparison of the emission of solvents and solvent mixtures from bottles with VICI Safety Caps and unsealed bottles was ordered. According to the customer request, this comparison was carried out by determination of the changes in density and volume of a solvent mixture, the change in volume of pure solvents, and the emission of solvents using emission chamber tests applying defined storage conditions.

2. Test samples

VICI caps, 3 ports, GL45 thread, with fittings (# JR-S-11002) were provided by VICI AG International for testing. Two ports were bridged by a PFA tube (1/8" × 1.59 mm ID / # JR-T-4003-M3), the third port was provided with an air inlet valve with 4 mm filter (# JR-S-20003).



Figure 1: VICI Safety Cap



Industrie Service

3. Experimental procedure

3.1 Determination of the changes in density and volume of a solvent mixture

To determine the changes in density and volume of a solvent mixture, 1000 ml laboratory glass bottles were each filled with 1000 ml of a methanol/water mixture (80:20, v/v). A bottle closed with a VICI Safety Cap and an unsealed reference bottle were prepared for each measurement day. The entire experiment was carried within a laboratory fume hood. The determination of the density and the residual volume of the solvent mixture in the bottle with a VICI Safety Cap and the reference bottle took place on Days 0, 2, 4, 7, and 8 using a pycnometer at 20 °C and differential weighing, respectively.

3.2 Determination of the change in volume of pure solvents

To determine the change in volume of pure solvents, 1000 ml laboratory glass bottles were filled with 1000 ml acetonitrile and methanol, respectively. A bottle closed with a VICI Safety Cap and an unsealed reference bottle were prepared for each solvent. The entire experiment was carried within a laboratory fume hood. The residual volume of solvent in the bottle with a VICI Safety Cap and the reference bottle was determined on Days 0, 7, and 28 using differential weighing.

3.3 Determination of the emission of pure solvents using emission chamber tests

The emission of methanol and acetonitrile, respectively, were determined using emission chamber tests. Laboratory glass bottles with a capacity of 1000 ml were filled with 1000 ml acetonitrile and methanol, respectively. A bottle closed with a VICI Safety Cap and an unsealed reference bottle were prepared for each solvent. One test chamber each was equipped with both the sealed or unsealed bottles. The following test conditions were used:

Temperature: 22.7 °C to 23.0 °C

Relative humidity: 50%

Chamber volume: 212 l

Air exchange rate: 0.5/h

Loading: 1 bottle each of acetonitrile and methanol per test chamber

Sample location: bottles centred on the chamber floor

The measurement of the solvent concentration in the room air was carried out using suction through silica gel (methanol) and activated carbon (acetonitrile), respectively, solvent extraction of the adsorbents, gas chromatographic separation, and mass-selective detection.



4. Test results

4.1 Determination of the changes in density and volume of a solvent mixture

The following changes were detected by determination of the density and the residual volume of the solvent mixture on Days 0, 2, 4, 7, and 8 using a pycnometer and differential weighing, respectively.

Table 1: Changes in density and volume of a solvent mixture

Day	Volume [%]		Density [g/cm ³]		Volume fraction of methanol* [%]	
	VICI Cap	Reference bottle	VICI Cap	Reference bottle	VICI Cap	Reference bottle
0	100	100	0.855	0.855	80.5	80.5
2	100	88.5	0.855	0.861	80.5	78.9
4	100	85.1	0.855	0.864	80.5	78.1
7	100	80.8	0.855	0.866	80.5	77.3
8	100	77.9	0.855	0.869	80.5	76.6

*Calculated from density value tables with linear interpolation

4.2 Determination of the change in volume of pure solvents

The following changes in volume could be detected by determining the residual volume of pure solvents on Days 0, 7, and 28 by differential weighing.

Table 2: Changes in volume of pure solvents

Day	Volume of methanol [%]		Volume of acetonitrile [%]	
	VICI Cap	Reference bottle	VICI Cap	Reference bottle
0	100	100	100	100
7	100	79.3	100	84.2
28	100	41.0	100	53.2

4.3 Determination of the emission of pure solvents using emission chamber tests

The determination of the emission of methanol and acetonitrile in the emission chambers after 24 h, 3 d, and 7 d using suction, solvent extraction, and GC/MS resulted in the following room air concentrations.

Table 3: Emission of pure solvents

Day	Room air concentration of methanol [mg/m ³]		Room air concentration of acetonitrile [mg/m ³]	
	VICI Cap	Reference bottle	VICI Cap	Reference bottle
1	0.51	1220	3.14	2310
3	0.72	1200	3.94	2010
7	0.76	1200	3.94	1900



Industrie Service

5. Summary and result

5.1 Changes in density and volume of a solvent mixture

Determination of the changes in density and volume of a solvent mixture facilitates the requested investigation of how much the solvent composition of a mixture in a laboratory glass bottle with VICI Safety Cap changes compared to an unsealed reference bottle over a holding time of up to eight days. It should be noted that the study was carried out in a laboratory fume hood with a high air exchange rate.

For the unsealed reference bottles, a volume decrease of 22.1% and a density increase of 0.014 g/cm³ could be measured over the 8-day test period. Using a density value table and linear interpolation, a 3.9% decrease in the methanol volume fraction could be determined from the density increase.

Accordingly, it was possible to detect a volatilisation of the solvent mixture from the unsealed reference bottles in which a higher proportion of the more volatile methanol is lost. This results in a change in the solvent composition (Figure 2).

The laboratory glass bottles sealed with a VICI Safety Cap showed a constant volume and a constant density throughout the 8-day test period. Accordingly, no evaporation of the solvent mixture, and no change in the methanol volume fraction and in the solvent composition, respectively, could be detected.

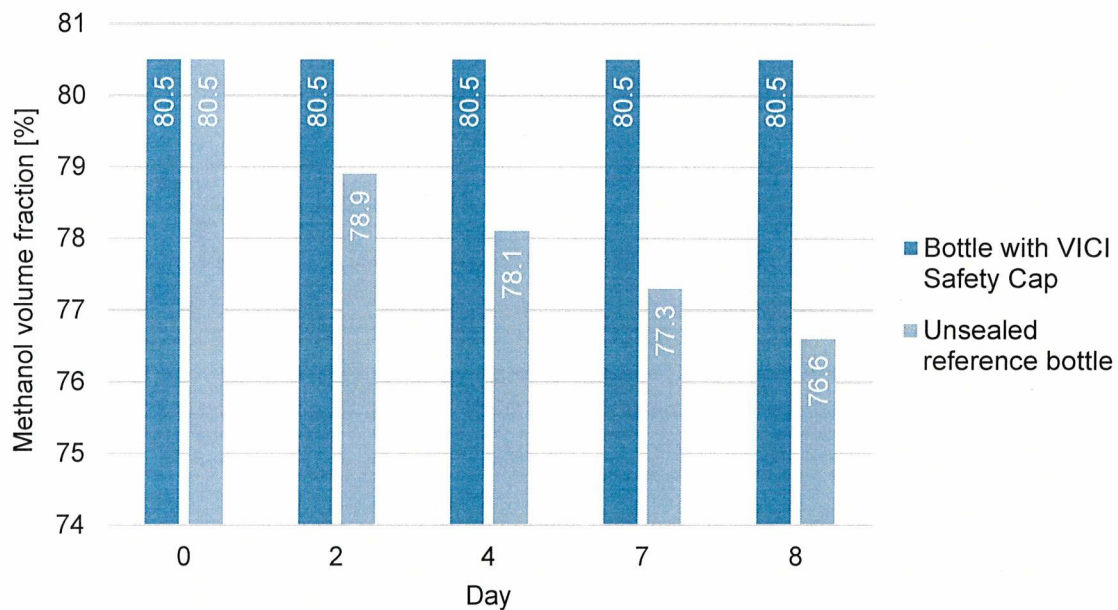


Figure 2: Methanol/water mixture composition



Industrie Service

5.2 Change in volume of pure solvents

Determination of the changes in volume of pure solvents facilitates the requested investigation of how much solvent is lost from a laboratory glass bottle with VICI Safety Cap compared to an unsealed reference bottle over a holding time of up to 28 days. It should be noted that the study was carried out in a laboratory fume hood with a high air exchange rate.

In the case of the unsealed reference bottle filled with 1000 ml methanol, a 20.7% weight loss or a 207 ml volume decrease due to volatilization could be measured by difference weighing within the first seven days. Over the subsequent 21 days, an additional 383 ml of methanol volatilised, corresponding to a 59% weight loss and a 590 ml volume decrease over the full 28-day period.

In the case of acetonitrile, a 15.8% weight loss and 158 ml volatilization, respectively, could be detected over the first seven days. Over the subsequent 21 days, the acetonitrile volume was reduced by a further 310 ml, corresponding to a weight loss of 46.8% and a volume decrease of 468 ml over the full 28-day period.

In the case of the laboratory glass bottles sealed with a VICI Safety Cap, a volatilisation of 0.21 ml methanol and 0.35 ml acetonitrile, respectively, could be detected by difference weighing over the full 28-day period.

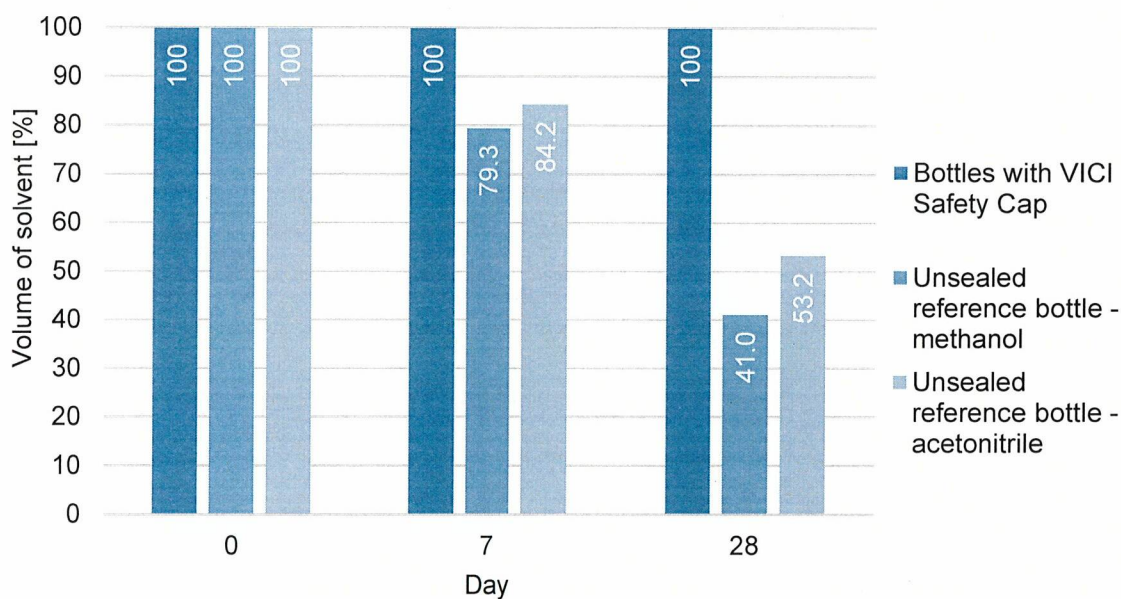


Figure 3: Changes in volume of pure solvents

5.3 Emission of pure solvents

The determination of the emission of methanol or acetonitrile in the emission chamber studies facilitates the requested investigation of what amounts of solvent can escape into the room air from a laboratory glass bottle with a VICI Safety Cap compared to an unsealed reference bottle through volatilisation during a holding time of up to 8 days.

When the test chamber was loaded with the unsealed reference bottles, a room air concentration of 1215 mg/m³ for methanol and 2306 mg/m³ for acetonitrile could be measured after 24 hours. These concentrations decreased slightly in the later measurements, after seven days room air concentrations of 1200 mg/m³ for methanol and 1900 mg/m³ for acetonitrile were measured.

The room air concentrations of methanol and acetonitrile in the test chamber containing the laboratory glass bottles sealed with a VICI Safety Cap were 0.51–0.76 mg/m³ for methanol and 3.14–3.94 mg/m³ for acetonitrile over the seven-day study period.

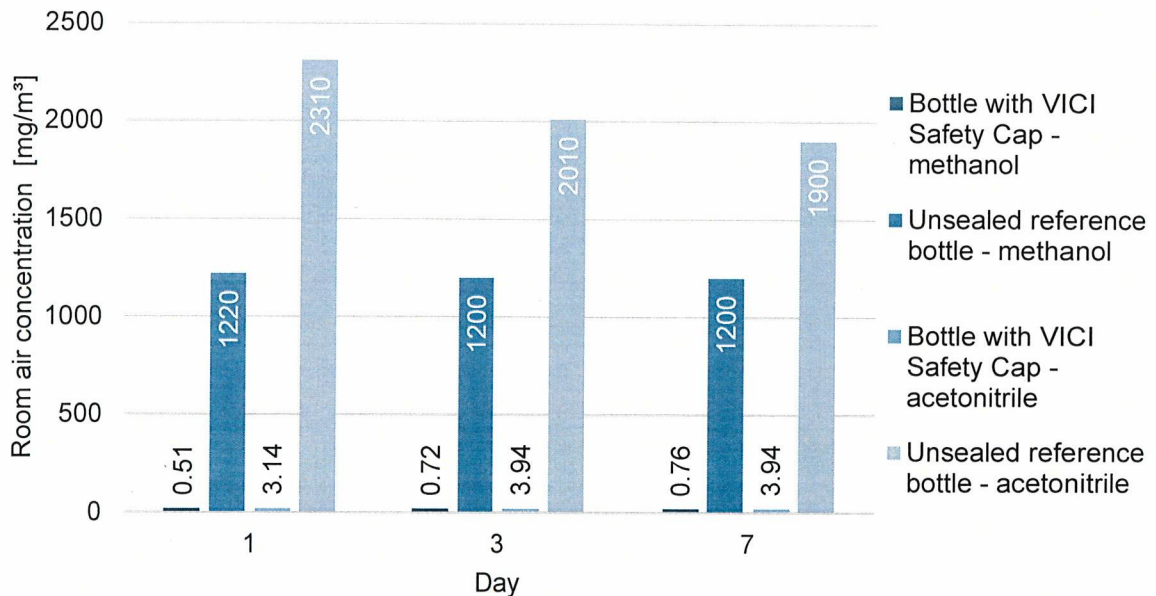


Figure 4: Emission of pure solvents