Storing and Handling Volatile Residual Solvent Gases in Secondary Containers

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The presence of highly flammable hydrocarbons, such as propane and butane, within cannabis concentrates pose a significant safety concern to the end consumer. Laboratory testing of highly volatile gases is complicated by many factors, with the stability and storage of analytical reference standards posing a significant issue. Improper storage of these highly volatile compounds can lead to significant loss of analyte and difficulty in acquiring accurate quantification.

The general guidelines below can help extend the shelf life of volatile standards. The exact shelf life of the standards will vary based on usage and should be verified by a lab prior to use.

Related Products

- *Micro-Vials with Graduated Marking Spot (cat.# 21050 & 21051)*
- *Mininert Precision Sampling Valves (cat.# 24900 & 24903)*
- *Residual Solvent Gases Mix (cat.# 36024)*
- *Residual Solvent Gases Singles (cat.# 36020, 36021, 36022 & 36023)*

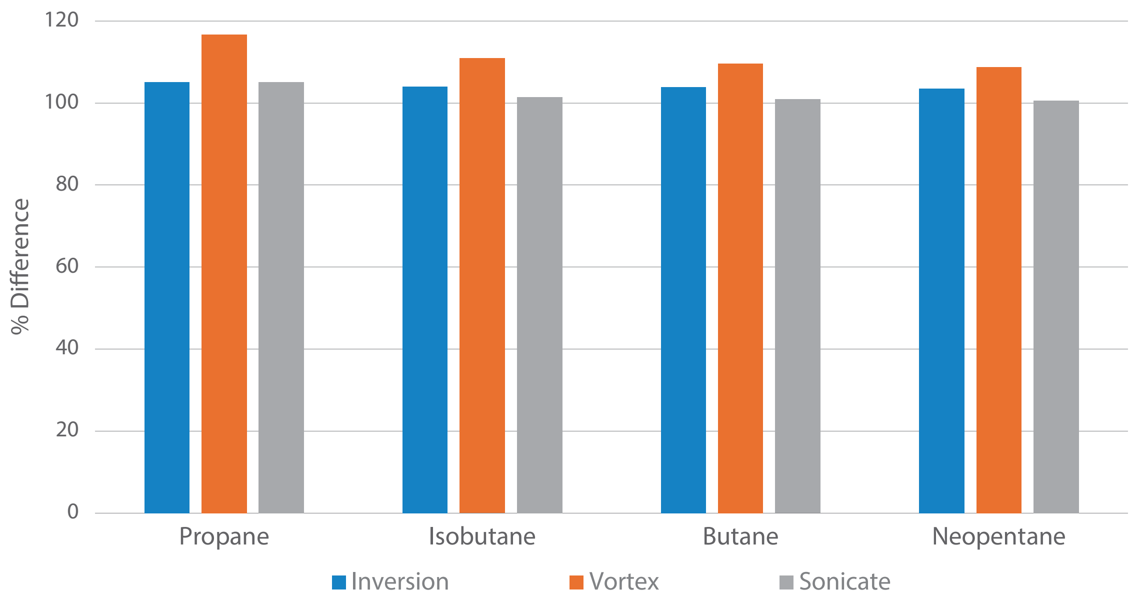
General Guidelines

1. Use the analytical reference standard cold (0 °C or colder) and do not allow it to warm to room temperature prior to use. The ampul should be gently inverted to ensure the liquid rests at the bottom of the ampul.
2. Transfer the largest volume possible to a secondary container and do not retransfer into other containers.
3. Store in micro-vials with Mininert Precision sampling valves at 0 °C or colder to minimize analyte loss, (3 mL, cat.#s: 21051, 24903).

Why formulate in trimethylbenzene?

The most critical aspect when trying to maximize the stability of volatile gases standards is the solvent used. For Residual's Residual Solvents Gases Standard Mix (cat. # 36024), 1,2,4-trimethylbenzene (TMB) was selected for several reasons: (1) its nonpolar nature increases the solubility of aliphatic analytes, such as propane and butane; (2) its low freezing point (-44 °C) allows it to be stored in typical laboratory freezers without freezing (unlike DMSO or benzene); (3) its relatively low viscosity allows easier manipulation even while cold; and (4) its high boiling point (169 °C) allows for separation with other analytes of interest. To further increase the standard's stability during shipping, ampuls are filled to minimize headspace. Stressed agitation studies of the sealed ampul demonstrate robust retention of even the most volatile analytes ensuring the integrity of the analyte is preserved in shipping (Figure 1).

Figure 1: Using trimethylbenzene as the solvent helps ensure robust retention of all residual solvent gases during stressed agitation studies. Results have been normalized against testing without agitation.



Handling Guidelines

General

In all studies, the standard was stored at -20 °C. Each day a new unopened ampul was tested as a control to ensure instrument stability throughout the study. Ampuls were gently inverted twice prior to being opened. For testing, 10 µL of standard and 5 µL of internal standard (10,000 ppm trifluorotoluene in methanol) were added to a 20 mL headspace vial. Each datapoint was obtained in triplicate unless otherwise stated.

Temperature

The standard should be handled cold (0 °C or colder) and not allowed to warm to room temperature prior to use. A loss of 22-30% relative to recovery from a cold ampul was observed for ampuls warmed to 25 °C, and a loss of 28-32% was observed for ampuls warmed to 50 °C (Table I).

Table I: An increase in temperature results in a higher loss of recoveries compared to the control.

	% Recovery vs. Control	
	25 °C	50 °C
Propane	70.75	68.12
Isobutane	75.60	70.53
Butane	77.04	71.54
Neopentane	78.24	72.11

Liquid Handling and Secondary Transfers

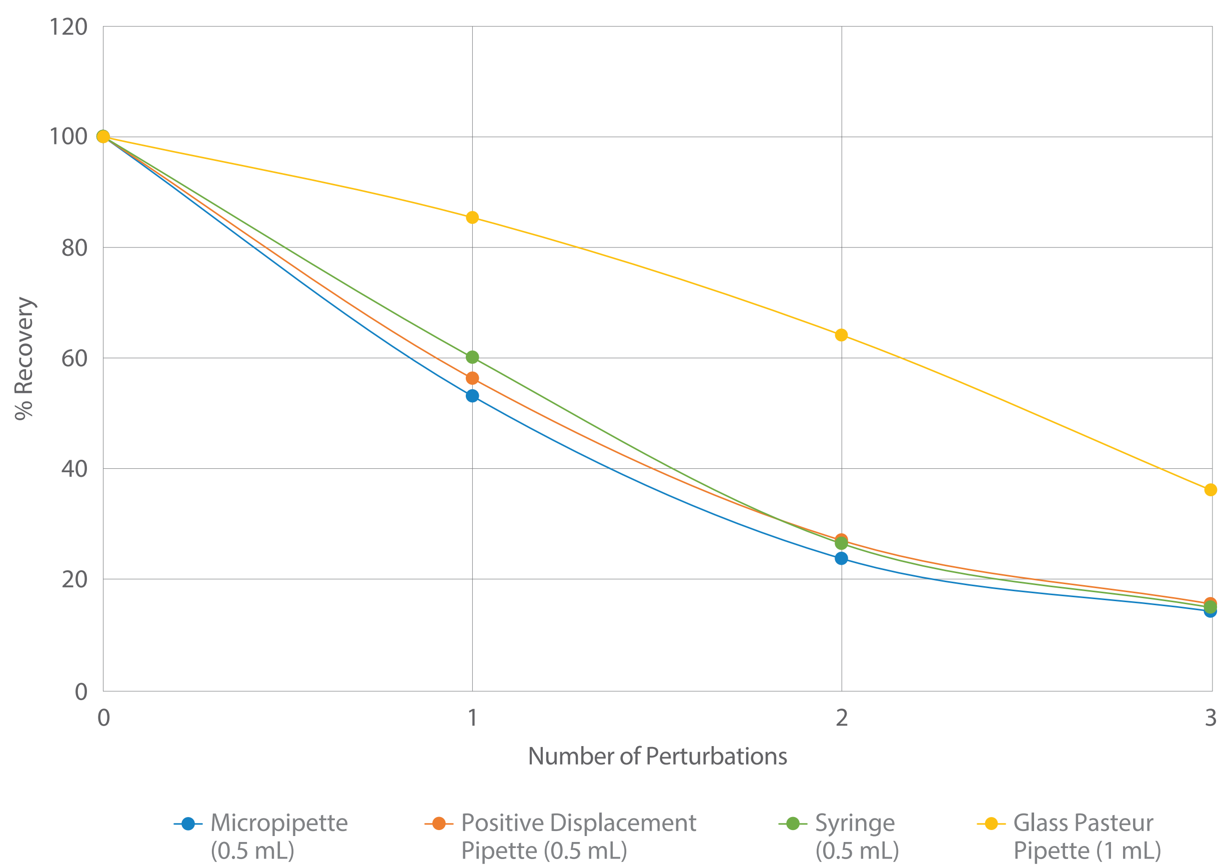
When handling the standard, there was a notable loss of analyte upon transferring from the ampul to a secondary container. To demonstrate differences in liquid handling, a new ampul was tested using the following liquid handling techniques:

1. air-aspiration micropipettes
2. positive displacement micropipettes
3. air-tight syringes

Furthermore, to test the effect of transferring to a secondary container, 500 µL of the standard from each liquid handling test was transferred to a secondary container and retested (perturbation 1). Then, 200 µL of the standard in a secondary container was transferred again to another secondary container and retested (perturbation 2). Finally, 100 µL of the standard was transferred to another secondary container and retested (perturbation 3).

For an additional comparison point, a glass Pasteur pipette was also used to transfer to each secondary container. The initial aliquot was roughly 1 mL using graduates on the transfer volume to approximate volume transferred. Test aliquots were removed using a gas-tight syringe (Figure 2).

Figure 2: A comparison between the different liquid handling techniques, with a glass Pasteur pipette showing the best results across all three perturbations.



All liquid handling techniques had similar area for each analyte without any perturbation. Each technique demonstrated a loss of analyte even after just a single transfer with each giving remarkably similar results. The best results were from the glass Pasteur pipette using a larger initial transfer volume. Given the similarity in response from each technique, it could be inferred that the volume used to transfer, not the technique, is the most important factor.

Figure 3: 3 mL and 1 mL micro-vials with Mininert Precision sampling valves.

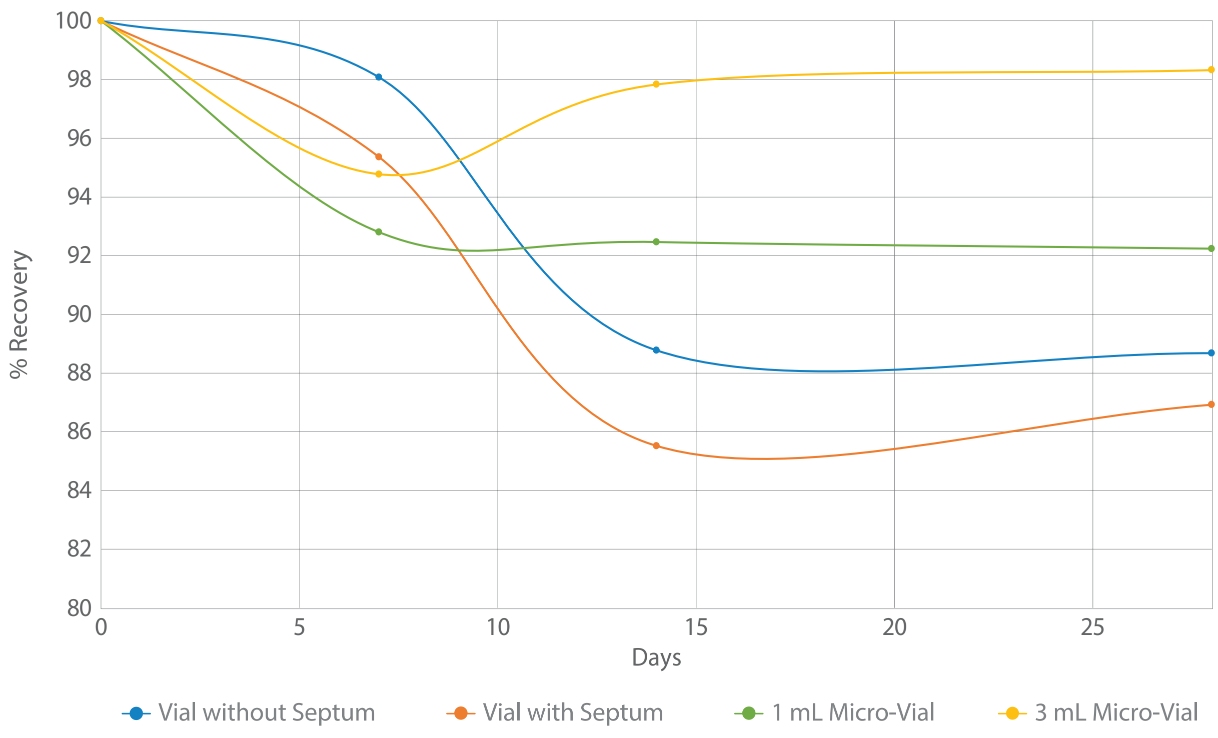
Storage Guidelines

Finally, we looked at storage in various secondary containers (Figure 3). In all cases, the secondary container was filled with as much liquid as possible to minimize headspace and stored at -20 °C. The following secondary containers were tested:

1. 2 mL vial with septum
2. 2 mL vial without septum
3. 1 mL micro-vial with Mininert Precision sampling valve (cat. #: 21050, 24900)
4. 3 mL micro-vial with Mininert Precision sampling valve (cat. #: 21051, 24903)

For the 2 mL vials, testing aliquots were removed by removing the cap and drawing with a positive displacement pipette. For the micro-vials, an air-tight syringe was used. Samples were tested in triplicate at 0, 7, 14, and 28 days. Results have been normalized to the results obtained on day 0 (Figure 4).

Figure 4: Propane recovery data showing a comparison between the different storage conditions, with a 3 mL micro-vial providing the highest recoveries after 14 days.



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For all storage conditions, there was a loss of analyte between day 0 and day 14, with the loss rate plateauing between day 14 and day 28. For propane, both 2 mL vials demonstrated a loss of 12-15%. For the micro-vials, there was a loss of only 2-8% within the same timeframe. The loss of analyte was most pronounced for propane, but the other gases had a similar effect. The loss of the less volatile gases (neopentane, isobutane, and butane) was not observed while using the micro-vials (Table II).

Table II: Micro-vials provide higher recoveries compared to 2 mL autosampler vials.

Vial without Septum (Days)	Propane	Isobutane	Butane	Neopentane	1 mL Micro-Vial (Days)	Propane	Isobutane	Butane	Neopentane
0	100	100	100	100	0	100	100	100	100
7	98.08	100.56	103.09	102.06	7	92.80	105.06	108.73	109.65
14	88.78	93.86	95.27	95.71	14	92.47	112.52	117.66	120.12
28	88.68	94.17	95.63	95.96	28	92.24	111.52	117.46	120.86
Vial with Septum (Days)	**Propane**	**Isobutane**	**Butane**	**Neopentane**	**3 mL Micro-Vial (Days)**	**Propane**	**Isobutane**	**Butane**	**Neopentane**
0	100	100	100	100	0	100	100	100	100
7	95.36	92.75	92.51	91.24	7	94.78	103.98	105.90	105.90
14	85.52	89.03	90.19	90.67	14	97.84	111.74	116.16	117.97
28	86.92	90.45	92.61	93.19	28	98.33	112.05	117.45	119.22

It should be noted that the 2 mL vials needed to have their caps removed for subsampling. If one were to remove the cap for more frequent subsampling (such as for daily QC), the storage shelf life would likely be much shorter. The micro-vials never needed to have their caps removed and may hold up to repeated subsampling for much longer.

Micro-Vials with Graduated Marking Spot

Cap Size	Volume	Material	Septa	Thread Type	Color	qty.	cat.#
13-425	1 mL	Borosilicate Glass	PTFE/Silicone (attached)	Screw-Thread	Clear with black cap	12-pk.	21050
20-400	3 mL	Borosilicate Glass	PTFE/Silicone (attached)	Screw-Thread	Clear with black cap	12-pk.	21051

Mininert Precision Sampling Valves

Cap Size	Thread Type	qty.	cat.#
13-425	Screw-Thread	12-pk.	24900
20-400	Screw-Thread	12-pk.	24903

Residual Solvent Gases Standards

- Designed for cannabis labs testing residual solvents gases at different threshold limits by headspace GC.
- Our proprietary manufacturing process was optimized to ensure standards are accurate, reproducible, and stable.
- High analyte concentrations allow for flexible calibration curves.
- Second independent lots available to ensure you can meet your requirements without needing to source another supplier.
- Verified composition and stability.

Residual Solvent Gases Mix

Contains each following compounds at a concentration of 5000 µg/mL:

n-Butane (C4) (106-97-8)
2,2-Dimethylpropoane (Neopentane) (463-82-1)
2-Methylpropane (Isobutane) ()75-28-5)
n-Propane (C3) (74-98-6)

Concentration in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp	Volume	Cat.#
5000 µg/mL in 1,2,4-Trimethylbenzene	Yes	6 months	60 months	Ambient	0 °C or colder	2.0 mL	36024

Residual Solvent Gases Singles

Product	Concentration in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp	Volume	Cat.#
Propane Standard	5000 µg/mL in 1,2,4-Trimethylbenzene	Yes	6 months	60 months	Ambient	0 °C or colder	2.0 mL	36020
2-Methylpropane (Isobutane) Standard	5000 µg/mL in 1,2,4-Trimethylbenzene	Yes	6 months	60 months	Ambient	0 °C or colder	2.0 mL	36021
n-Butane Standard	5000 µg/mL in 1,2,4-Trimethylbenzene	Yes	6 months	60 months	Ambient	0 °C or colder	2.0 mL	36022
2,2-Dimethylpropane (Neopentane) Standard	5000 µg/mL in 1,2,4-Trimethylbenzene	Yes	6 months	60 months	Ambient	0 °C or colder	2.0 mL	36023

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