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Automating the Accurate Transfer of Highly Volatile to Highly Viscous Liquids using a Bench-top Workstation

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ABSTRACT

The manual transfer of liquid standards and solutions is usually part of the daily activities throughout the analytical laboratory. For example, liquids must be transferred when creating calibration standard samples, pipetting solvents, and combining liquids. The accurate and precise transfer of liquids can be critical to the analytical results. Liquids with low boiling points or high viscosities pose several challenges to achieving accurate and precise delivery of desired volumes. Verification of the volumes of liquids transferred would help verify the quality of the analytical procedure and ensure the high quality of the resulting data.

A single X-Y-Z coordinate autosampler and sample preparation robot commonly used for sample introduction in GC or HPLC can be used to perform a wide variety of sample preparation techniques using a single instrument and controlling software. The sampler can be configured as part of a GC or LC system, or as a bench-top workstation independent of the analytical instruments used, and can also include an analytical balance to provide weight verification of liquid transfers.

In this report, the automated transfer by the autosampler of a variety of liquid standards having a wide range of volatility and viscosity properties is discussed. A new vial venting tool that allows liquid samples to be transferred to a sealed vial while venting it is described and evaluated. Resulting weight verification, precision, and accuracy data from the assessment of example compounds transferred by the autosampler are provided that demonstrate the dramatic improvement in accuracy and precision for transferring these types of liquids.

INTRODUCTION

In addition to providing unattended routine operation as an autosampler for GC/MS or LC/MS, the GERSTEL MultiPurpose Sampler (MPS) in combination with MAESTRO software can completely automate a wide range of sample preparation steps, which would normally be performed manually. The system provides the flexibility to combine a multitude of different tasks by mouse-click while serving to improve sample throughput in the lab.

This study focuses on the automated transfer of highly volatile and highly viscous liquid standards including verification of the weight of the transferred liquid. For this purpose, the MPS has been fitted with an analytical balance. Using the GERSTEL MPS autosampler, all liquid transfers were performed with a liquid syringe. The use of the GERSTEL Purge Tool Option allowed the receiving vial to be vented during liquid transfer, resulting in improved accuracy and precision for the highly volatile liquids being transferred. Data obtained during the optimization of the automated transfer of liquids was used to assess the accuracy and precision of the transfers.

EXPERIMENTAL

Materials. Table 1 contains the liquid standards examined during this study along with their corresponding physicochemical properties. All liquid standards were of reagent grade or better.

Table 1. Liquid standard compounds information.

Compound	CAS	bp [°C]	Density at 25°C [g/mL]	VP at 20°C [kPa]	VP at 20°C [mm Hg]
Acetonitrile	75-05-8	81.3	0.786	9.71	72.83
Pentane	109-66-0	35.9	0.626	75.9	469.3
Freon 113	76-13-1	47.6	1.56	37.997	285
1,1-Dichloroethylene	75-35-4	30.0	1.213	66.661	500

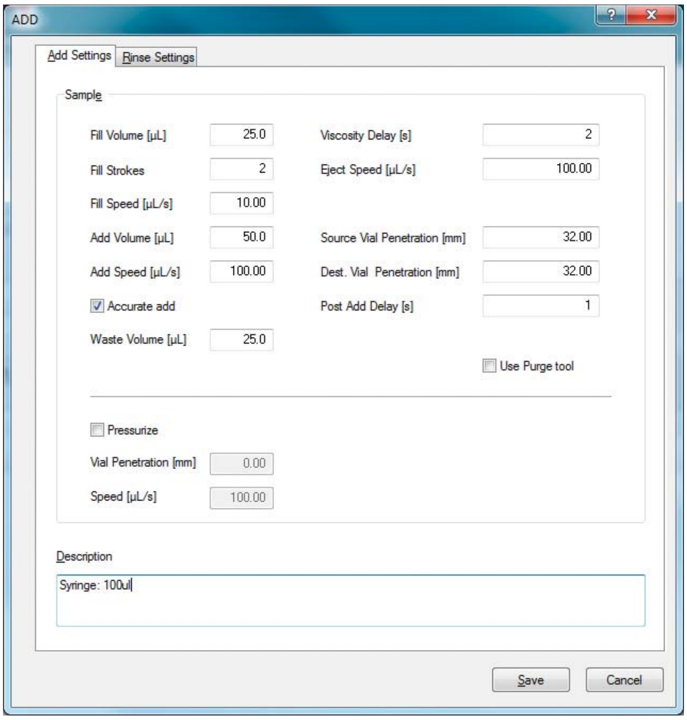
Instrumentation. All automated liquid transfers and weighings were performed using a GERSTEL MultiPurpose Sampler (MPS) configured with the GERSTEL Balance Option and Purge Tool Option as shown in Figure 1 and Figure 2. Vials containing the liquid standard to be used were placed in a thermostated tray holder on the MPS. Empty vials were capped using magnetically transportable caps and also placed onto the autosampler. The GERSTEL Balance Option incorporated a Sartorius Cubis Balance (model# MSU524S-000-DA) for collection of all weight data.

Figure 1. MultiPurpose Sampler (MPS) with the GERSTEL Purge Tool and Balance Options.

Figure 2. MultiPurpose Sampler (MPS) with the GERSTEL Balance Option.

RESULTS AND DISCUSSION

As shown in Figure 2, several features are available within the MAESTRO software to assist the user in the proper aspiration of a liquid sample, avoiding the formation of air bubbles and thereby, ensuring the proper transfer of liquids.



Fill Volume [µL] 25.0
Viscosity Delay [s] 2
Fill Strokes 2
Eject Speed [µL/s] 100.00
Fill Speed [µL/s] 10.00
Add Volume [µL] 50.0
Source Vial Penetration [mm] 32.00
Add Speed [µL/s] 100.00
Dest. Vial Penetration [mm] 32.00
☑ Accurate add
Post Add Delay [s] 1
Waste Volume [µL] 25.0
☐ Use Purge tool
☐ Pressurize
Vial Penetration [mm] 0.00
Speed [µL/s] 100.00
Description
Syringe: 100ul

Figure 3. Example of the MAESTRO ADD Prep Action.

The aspiration of a high viscosity liquid into a syringe may result in cavitation due to the pressure drop required to transport the viscous liquid through the narrow syringe needle at the specified rate. In such a case, the syringe barrel is raised to the proper level to aspirate the viscous liquid, but the liquid cannot follow at the rate, at which the plunger is raised. In addition to selecting an adequate aspiration rate, an additional delay time must be incorporated into the method in order to ensure an accurate transfer of the desired volume of viscous liquid. Adding a delay time following aspiration of highly viscous liquids allows the liquids to "catch up" with the syringe plunger prior to the syringe being moved to the receiving vial.

If an air bubble still happens to be present inside the syringe, the result would obviously be a reduction in the transferred amount. If the use of a viscosity delay time does not completely eliminate the presence of air bubbles, the use of the "Accurate ADD" feature allows the user to avoid the transfer of air bubbles by aspiration of an additional "Waste" volume of the liquid to be transferred. The following actions are automatically performed when selecting the "Accurate ADD" feature in the MAESTRO software:

- The MPS moves to source location and aspirates the user defined "Add volume".
- The MPS then aspirates the user defined "Waste volume" plus a tiny additional amount (2% of the syringe volume).
- While still at the source location, the MPS dispenses the tiny additional volume back into the vial in order to ensure the syringe plunger is in the proper position to dispense the aspirated volume both accurately and reproducibly.
- The MPS then moves to the destination and dispenses the user defined "Add volume".
- The MPS finally moves to the waste location and dispenses the user defined "Waste volume" including potential air bubbles underneath the plunger tip before rinsing the syringe for the next task.

The use of this strategy ensures that the liquid is delivered to the receiving vial without any air bubbles that could negatively influence the accuracy.

In order to avoid the evaporation of volatile chemicals (for example, liquid solvents), transfer of the liquids into vials sealed with septa is required. The act of dispensing a liquid into a sealed vial will increase the pressure within the vial. Keeping the pressure constant during liquid transfer steps can be crucial, since an increase in pressure will cause a change in the ability to accurately transfer some compounds.

As can be seen from the data in Table 2, using the Purge Tool to keep the pressure of the receiving vial constant during liquid transfer helped improve the accuracy when transferring volatile liquids.

Table 2. Improved accuracy for liquid transfers using a Purge Tool.

Compound	without Purge Tool		with Purge Tool	
	Amount transferred [g]	Difference from theor. [%]	Amount transferred [g]	Difference from theor. [%]
Pentane (500 µL) theoretical weight of 0.313 g	0.257	- 17.89	0.285	- 8.95
	0.253	- 19.17	0.287	- 8.31
	0.259	- 17.25	0.286	- 8.63
	0.263	- 15.97	0.290	- 7.35
	0.264	- 15.65	0.285	- 8.95
	0.258	- 17.57	0.285	- 8.95
mean	0.259	- 17.3	0.286	- 8.5
SD	0.00405	1.29	0.00197	0.628
% CV	1.56	- 7.50	0.687	- 7.37
1,1-Dichloroethylene (500 µL) theoretical weight of 0.609 g	0.457	- 24.96	0.546	- 10.34
	0.434	- 28.74	0.553	- 9.20
	0.398	- 34.65	0.554	- 9.03
	0.412	- 32.35	0.547	- 10.18
	0.410	- 32.68	0.551	- 9.52
	0.409	- 32.84	0.546	- 10.34
mean	0.420	- 31.0	0.550	- 9.8
SD	0.0216	3.55	0.003627	0.59432
% CV	5.14	- 11.43	0.659	- 6.08
Freon 113 (500 µL) theoretical weight of 0.785 g	0.732	- 6.75	0.748	- 4.71
	0.720	- 8.28	0.746	- 4.97
	0.718	- 8.54	0.746	- 4.97
	0.720	- 8.28	0.747	- 4.84
	0.718	- 8.54	0.745	- 5.10
	0.719	- 8.41	0.745	- 5.10
mean	0.721	- 8.1	0.746	- 4.9
SD	0.00538	0.686	0.00117	0.149
% CV	0.746	- 8.43	0.157	- 3.01

Since different liquids have different physicochemical properties, the ability to successfully transfer the liquid by a syringe can be affected by manipulating the environment of the liquid. Care must be taken, however, since this effect could be either beneficial or detrimental. Increasing the temperature of a highly viscous liquid will reduce its viscosity allowing it to flow better, minimize the chance of potential cavitation, and thereby allow the liquid to be reproducibly transferred by a syringe. Temperature control of viscous liquids can therefore be a crucial factor in ensuring their accurate transfer.

The data listed in Table 3 show that an increase in the temperature of 1,1-Dichloroethylene resulted in improved accuracy of the liquid transfer.

Table 3. Improved accuracy for 1, 1-Dichloroethylene using temperature control.

1,1-Dichloroethylene	at room temp.		at 20°C	
	Amount transferred [g]	Difference from theor. [%]	Amount transferred [g]	Difference from theor. [%]
500 µL theoretical weight of 0.609 g	0.546	- 10.34	0.506	- 16.91
	0.553	- 9.20	0.515	- 15.44
	0.554	- 9.03	0.469	- 22.99
	0.547	- 10.18	0.461	- 24.30
	0.551	- 9.52	0.443	- 27.26
	0.546	- 10.34	0.460	- 24.47
mean	0.550	- 9.77	0.476	- 21.89
SD	0.00362	0.594	0.0284	4.67
% CV	0.659	6.08	5.98	21.3
500 µL theoretical weight of 0.609 g	at 25°C		at 30°C	
	0.547	- 10.18	0.592	- 2.79
	0.554	- 9.03	0.568	- 6.73
	0.553	- 9.20	0.571	- 6.24
	0.549	- 9.85	0.573	- 5.91
	0.542	- 11.00	0.571	- 6.24
	0.549	- 9.85	0.573	- 5.91
mean	0.549	- 9.85	0.575	- 5.64
SD	0.00434	0.712	0.0087	1.43
% CV	0.790	7.23	1.51	25.3

Aspiration of highly volatile liquids can also be affected by temperature or pressure differences. Just as transferring cold samples is known to result in a larger amount of the solvent being transferred, transferring samples that are held at an increased temperature could result in a smaller amount of the solvent being transferred. Temperature and pressure can also affect the volume of an air bubble, which could in turn, affect the accuracy of the liquid transfer. Volatile solvents can evaporate into the air bubble, which would lead to an inaccurate, lower dispensed volume than intended. Also, when an air bubble is formed behind the solvent, the air could then act as a piston, increasing the pressure within the syringe resulting in the solvent dripping out of the syringe before it can be transferred.

The use of the GERSTEL Balance Option allows the user to deliver exactly the required amount of sample to a vial. Since the precision of liquid transfers performed using the MPS is observed to be extremely reproducible, a correction factor can be applied to the initially specified volume to be transferred resulting in the transfer of exactly the required amount. For example, if a user wanted to transfer exactly 1.00 gram of sample to a vial, and it was found after optimizing the transfer parameters, that by transferring 1.00 mL of sample, exactly 0.95 grams were being delivered, the volume being transferred could be adjusted using a correction value of 1.05, resulting in the transfer of 1.05 mL or exactly 1.00 grams.

Table 4 shows the accuracy data obtained for the volatile liquids examined following optimization of the liquid transfer by performing the following changes:

- Controlling the temperature of the liquid standards at 30°C
- Using the Purge Tool to control the pressure of the receiving vial
- Employing the software strategies available within the MAESTRO software including the "Accurate ADD" feature to ensure only the liquid standard was being transferred
- Transferring the liquid standards in two aliquots, and using a correction value of 1.05.

When compared to results obtained without optimization, the outlined strategies brought significant improvements in the % difference from the theoretical value: For 1,1-Dichloroethylene, the percent difference from theoretical was improved from -41.38 % to -0.19 %; for Acetonitrile, it was improved from -4.10 % to 2.46 %; for Pentane from -19.55 % to -0.96 %; and for Freon 113 from -10.64 % to 2.74 %.

Finally, for day to day reproducibility of transfers of highly volatile and highly viscous liquids, temperature control of the liquids being transferred can be critical especially when the temperature of the lab fluctuates. An independent laboratory used the same software and hardware strategies to create a custom blended gas standard of the volatile organic compounds shown in Table 5. After performing an automated accurate transfer of each liquid standard into a single, combined solution, the solution was transferred to a pressurized gas cylinder and mixed. As shown in Table 6, controlling the temperature of the liquid standards used, as well as implementing both the listed software (Accurate ADD, viscosity delays) and hardware (Purge Tool) strategies, enabled the accurate and precise transfer of liquids, resulting in relative % differences of less than 3.8 % from theoretical for all compounds, determined by analyzing the custom gas standard by GC-FID.

Table 4. Accuracy of volatile liquid transfers following optimization.

Compound	Amount transferred [g]	Difference from theor. [%]
1,1-Dichloroethylene original % difference from theoretical = - 41.38 %	0.604	- 0.82
	0.609	0.00
	0.610	0.16
	0.604	- 0.82
	0.610	0.16
	0.610	0.16
mean	0.608	- 0.19
SD	0.0030	
% CV	0.493	
Acetonitrile original % difference from theoretical= - 4.10 %	0.403	2.54
	0.404	2.80
	0.402	2.29
	0.404	2.80
	0.402	2.29
	0.401	2.04
mean	0.403	2.46
SD	0.0012	
% CV	0.301	
Pentane original % difference from theoretical= - 19.55 %	0.309	- 1.28
	0.309	- 1.28
	0.311	- 0.64
	0.309	- 1.28
	0.311	- 0.64
	0.311	- 0.64
mean	0.310	- 0.96
SD	0.0011	
% CV	0.353	
Freon 113 original % difference from theoretical= - 10.64 %	0.796	2.05
	0.805	3.21
	0.808	3.59
	0.796	2.05
	0.801	2.69
	0.802	2.82
mean	0.801	2.74
SD	0.0048	
% CV	0.599	

Table 5. Liquid standard compounds used in custom gas standard.

Compound	CAS	bp at 25°C [°C]	Density at 20°C [g/mL]	VP [kPa]
Acetone	67-64-1	56	0.8	46.4
Pentane	109-66-0	36	0.6	70.4
1,1-Dichloroethylene	75-35-4	31	1.2	80.1
Allyl chloride (3-Chloro-1-propene)	107-05-1	46	0.9	55.6
Freon 113	76-13-1	48	1.7	39.5
tert-Butyl methyl ether	1634-04-4	56	0.8	33.5
2-Methylpentane	107-83-5	60	0.7	28.4
Hexane	110-54-3	69	0.7	20.1

Table 6. Resulting % difference of custom gas standard from theoretical concentration.

Compound	Theoretical conc. [ppm]	Calculated conc. [ppm]	Difference [%]
Acetone	4.99	5.07	1.51
Pentane	5.01	4.89	- 2.31
1,1-Dichloroethylene	4.77	4.95	3.74
Allyl chloride (3-Chloro-1-propene)	5.01	4.98	- 0.6
Freon 113	4.98	4.94	- 0.88
tert-Butyl methyl ether	5.1	4.94	- 3.1
2-Methylpentane	5.03	4.93	- 2.06
Hexane	5.09	5.03	- 1.27

CONCLUSIONS

As a result of this study, we were able to show:

- When transferring volatile liquids, accuracy is improved when using a Purge Tool to control the pressure of the recieving vial.
- Optimizing and controlling the temperature of the liquid standard being transferred can have a positive effect on the accuracy of transfer.
- Complete optimization of automated liquid transfer parameters is possible using the GERSTEL MPS hardware and MAESTRO software.
- These software and hardware strategies can help ensure method robustness when transferring highly volatile and highly viscous liquids.

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AppNote

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