MultiVap8: Automated concentration system

Introduction

Solvent evaporation is an essential step of sample preparation before chromatographic analysis and is commonly used in a variety of applications including drug discovery, clinical analysis, environmental testing and food chemistry.
The primary purpose is to increase the analytes concentration (especially in the range of ppb (µg/L) or below) and to improve the detection limit of the compounds of interest. In addition, solvent evaporation is an important step in many chemical synthesis: as the solvent used to generate an intermediate may not be compatible with the next reaction step and has to be removed.

Nitrogen stream evaporation is one of the most common and mature evaporation technique, typically used to evaporate solvents by heating, through a liquid bath, stable analytes and using a stream of Nitrogen gas which is directly blown through needles onto sample vials. Nitrogen blow down accelerates evaporation rate by decreasing the partial vapour pressure of the solvent just above the surface of the liquid. Pure Nitrogen gas is typically used as it is a relatively non reactive gas in contrast to air that could oxidize the compounds of interest.

LabTech MultiVap8 automated evaporation system is a modern and compact Nitrogen blow-down system that allows automatic parallel evaporation granting high repeatability and reproducibility. With this system users can evaporate to solid state or concentrate into a fixed end-point volume. Meanwhile, MultiVap8 liquid bath temperature and N_2 gas flow rate can be adjusted depending on solvent boiling point and volatility.
The experimental results presented below show MultiVap8 excellent analytical results during evaporation with significant reduction of time required to complete the process.

Product Overview

MultiVap8 is the perfect choice to complete organic sample preparation process after SPE with LabTech SepathsUP and SeplineS systems and is the ideal partner in a variety of evaporation applications including drug discovery, food testing and environmental analysis.

The unit has 8 sample positions and may be configured to accommodate either 50 mL or 200 mL concentration cups with 1 mL, 0.5 mL and 0 mL endpoints. The instrument is equipped with infrared sensor to automatically stop gas flow when evaporation has reached its end point, thereby eliminating the risk of drying and loss of volatile compounds.

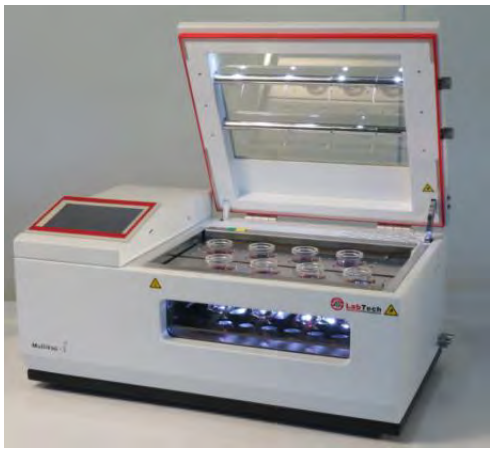

Figure 1. MultiVap8

The location and direction of Nitrogen purge can be adjusted according the concentration cup size and the sample volume to optimize the solvent surface area in contact with Nitrogen and achieve the highest concentration efficiency with Vortex Nitrogen purge. The Nitrogen needles angle direction affect the concentration efficiency depending on the solvents involved.

The vortex created by the moving gas flows down the tube to the solvent surface, where it increases the gas/solvent interface providing faster evaporation in comparison to conventional methods. The combination of gas flow and temperature control allows the optimization of sample drying. Nitrogen is recommended as the best choice of gas because it is inert and minimizes the risk of oxidation.

Figure 2. MultiVap8

As we can see in Figure 3, the instrument is provided with a touch screen control interface through which the customer can easily adjust the liquid bath temperature, the Nitrogen gas flow, and select the nitrogen concentration mode.

Figure 3. System main interface

To increase safety and simplify routine maintenance, the liquid inside the bath can be easily removed by the drain valve on the side of the unit and the instrument is provided with an efficient vapour exhausting system to grant maximum safety operations during the whole process.

Experimental Section

Reagents

Solvents: Water; Methanol (HPLC); Acetonitrile (HPLC)

Active Ingredients: Metronidazole; Sulfadiazine; Sulfamonomethoxine; Sulfamethazine; Sulfamethoxazole; Sulfaquinoxalinum

Pesticides/Insecticides: Imidacloprid; Bensulfuron methyl; Methamidophos; Fenpropathrin; Cyhalothrin; Deltamethrin; Fenvalerate; Permethrin.

Instruments:

MultiVap8 concentrator, HPLC

Experiments:

A series of experiments were conducted with pure solvents to test the evaporation efficiency of MultiVap8 among different sample tubes. N°5 test tubes (50 mL concentration cups) for each analyte were placed to test the evaporation repeatability.

15 psi Nitrogen gas pressure, 40°C liquid bath temperature and optimized Nitrogen needles angle were set to study the main important factors affecting evaporation efficiency.

In the tables below are compared different recoveries for the analytes of interest:

Analyte	Concentration (mg/L)	Solvent	Recovery	Note	Evaporation time from 50mL to 1 mL
Metronidazole	2	Methanol	88%	Melting point 160°C	19 min
Sulfadiazine	0.4	Acetonitrile	101%	Sensitive to light	23 min
Sulfamonomethoxine	0.4	Acetonitrile	89%	Sensitive to light	23 min
Sulfamethazine	0.4	Acetonitrile	100%	Sensitive to light	23 min

Sulfamethoxazole	0.4	Acetonitrile	100%	Sensitive to light	23 min
Sulfaquinoxalinum	0.4	Acetonitrile	108%	Sensitive to light	23 min

Figure 4. Recovery data for active ingredients

Analyte	Concentration (mg/L)	Solvent	Recovery	Note	Evaporation time from 50 mL to 1 mL
Imidacloprid	4	Acetonitrile	104%	Stable in pH 5-11	23 min
Bensulfuron methyl	2	Acetonitrile	96%	Degrades slowly in weak basic (pH=8) and acid solution	23 min
Methamidophos	2	Methanol	89%	Hydrolyzed in strong basic solution	19 min
Fenpropathrin	2	Methanol	102%	Hydrolyzed in strong basic solution	19 min
Cyhalothrin	2	Methanol	104%	Hydrolyzed in strong basic solution	19 min
Deltamethrin	2	Methanol	104%	Hydrolyzed in strong basic solution	19 min
Fenvalerate	2	Methanol	102%	Hydrolyzed in strong basic solution	19 min
Permethrin	2	Methanol	103%	Hydrolyzed in strong basic solution	19 min

Fig 5. Recovery data for pesticides and insecticides

For each analyte were performed 5 different analysis to assure the repeatability of the scientific data collected; here below we can compare and check the results:

Analyte	1	2	3	4	5	Average	RSD%
Metronidazole	88%	85%	87%	91%	89%	88%	2,5
Sulfadiazine	100%	101%	103%	101%	98%	101%	1,8
Sulfamonomethoxine	87%	88%	90%	90%	89%	89%	1,5
Sulfamethazine	99%	98%	103%	100%	100%	100%	1,9
Sulfamethoxazole	99%	99%	100%	102%	100%	100%	1,2
Sulfaquinoxalinum	108	107	107	110	109	108%	1,2

Fig 6. Repeatability of MultiVap8 for active ingredients

Analyte	1	2	3	4	5	Average	RSD%
Imidacloprid	100%	109%	102	100	108	104%	4,2
Bensulfuron methyl	97	95	95	99	94	96%	2,1
Methamidophos	90	89	90	89	88	89%	0,9
Fenpropathrin	100	105	101	101	101	102%	1,9
Cyhalothrin	106	102	103	101	106	104%	2,2
Deltamethrin	108	103	102	105	104	104%	2,2
Fenvalerate	108	100	101	102	101	102%	3,1
Permethrin	109	99	105	95	107	103%	5,6

Fig 7. Repeatability of MultiVap8 for pesticides and insecticides

Conclusion

MultiVap8 automated evaporation system significantly speeds up the workflow and reduces the labor time during evaporation. Compared to the traditional solvent concentrators, LabTech MultiVap8 evaporation system reduces the evaporation time up to 20% granting excellent recoveries of a wide range of analytes. Pesticides and active substances were efficiently concentrated before chromatographic analysis showing MultiVap8 excellent results in term of efficiency and reproducibility.

With its large-volume concentration vials, MultiVap8 can be used with sample extracts from a wide range of extraction procedure like Solid-Phase Extraction, Liquid-Liquid Extraction, Pressurize Fluid Extraction and Ultrasonic Extraction.