MultiVap54: Automated concentration system

Abstract

LabTech MultiVap54 is the latest automatic nitrogen evaporator developed by our R&D Team able to process up to 54 samples simultaneously. This advanced instrument allows automatic control of both the nitrogen flow rate and the vertical lift of nitrogen needles. These unique features can greatly speed-up the vapour removal and maximize the solution surface area in contact with nitrogen gas, thus significantly saving nitrogen gas consumption and improving evaporation efficiency. Meanwhile, MultiVap54 water bath temperature and N_2 gas flow rate can be adjusted during the evaporation process depending on solvent boiling point and volatility.

The experimental results presented in this article show that MultiVap54 is able to simultaneously evaporate most of the extraction solvents in optimal experimental conditions, making it a unique solution for all analytical laboratories.

Introduction

Solvent evaporation, as an essential step of sample preparation before chromatographic analysis, is commonly used in a broad range of fields, such as pharmaceutical, environmental, petrochemical and other industrial analytical laboratories.

The primary purpose of evaporation is to increase the analytes concentration (especially in the range of ppb (μg/L) or below) to improve their detection limit before analysis.

Nitrogen stream, rotary vacuum and centrifugal evaporator are the most commonly used techniques in analytical laboratories to evaporate samples. Generally major factors affecting the concentration rate are the heating temperature, the vapour removal and the solvent surface area.

Nitrogen stream evaporation is a very common and mature technique, typically used to evaporate solvents by heating stable analytes and using a stream of nitrogen gas which is directly blown in the solution. The critical point using nitrogen stream evaporation is the setting of liquid bath temperature to grant the highest evaporation speed without the risk of analytes evaporation or degradation.

Rotary vacuum is an evaporation technique where samples are placed into a round-bottom rotating flask immersed in an heated liquid bath. A vacuum pump is connected to reduce the pressure inside the flask and vacuum is applied to reduce the boiling point of solvents. Main disadvantage of rotary evaporator is the limited number of sample evaporating simultaneously.

Centrifugal evaporation systems typically involve a heated centrifuge with a vacuum pump to concentrate the samples; heating and vacuum functions facilitate the solvent evaporation at lower temperature respect the standard boiling point under atmosphere pressure. Main disadvantages of this technique are the temperature stability, the cross contamination and the inability to monitor the sample concentration during the process.

LabTech MultiVap54 Overview

MultiVap54 is the perfect choice to complete organic sample preparation process after SPE with LabTech Sepaths-UP and Sepline-S systems. MultiVap54 can concentrate up to 54 samples simultaneously from 1,5 mL to 200 mL volume.

Sample tube size can be customized according customer requests and nitrogen needle positions can be adjusted automatically or manually according to sample volume reduction during the evaporation process. This unique feature of MultiVap54 reduces the evaporation time and nitrogen gas consumption compared to traditional nitrogen stream evaporators. Once the distance between nitrogen blowing needle and solvent surface is optimized, the vortex solution flow in the sample tube increases the surface area in contact with nitrogen and speed up the evaporation rate.

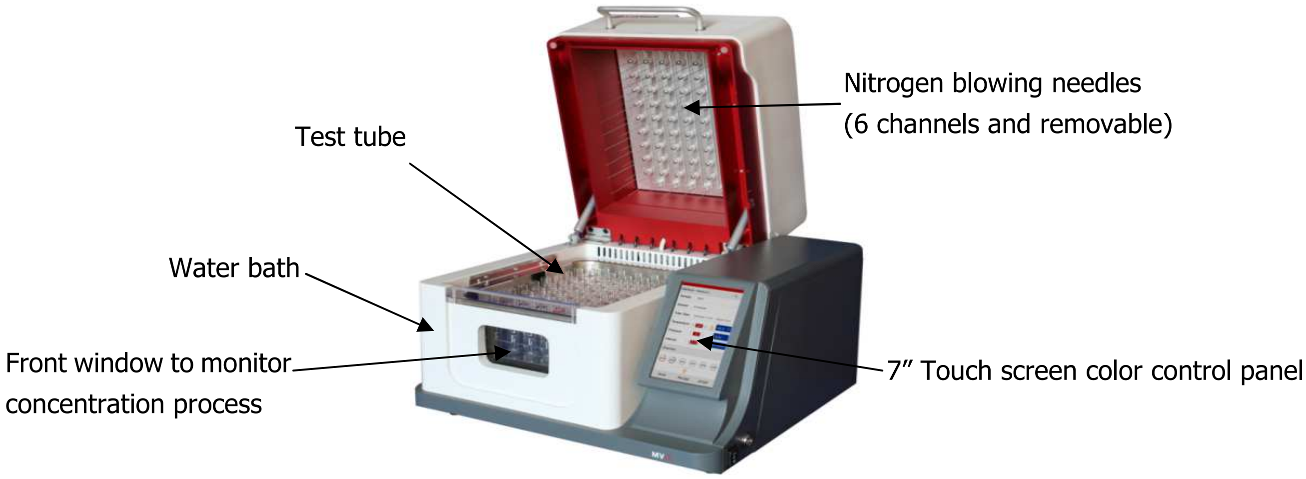

Figure 1. MultiVap54 evaporator

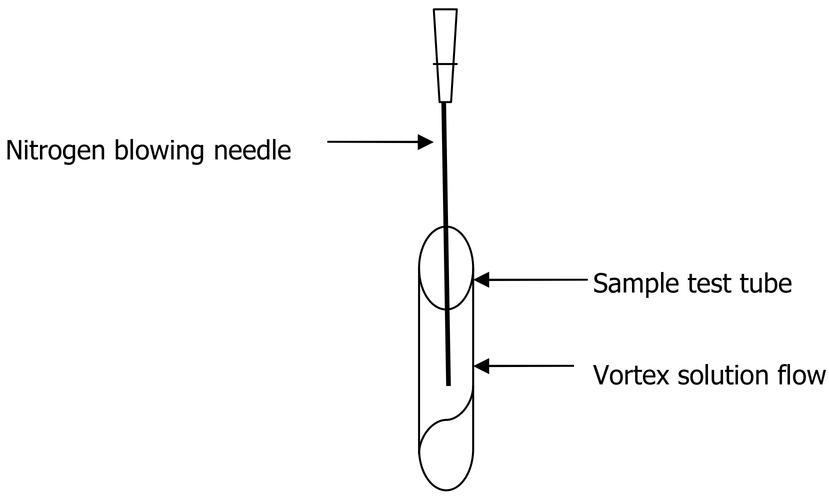

Figure 2. Vortex solution with nitrogen blowing needles

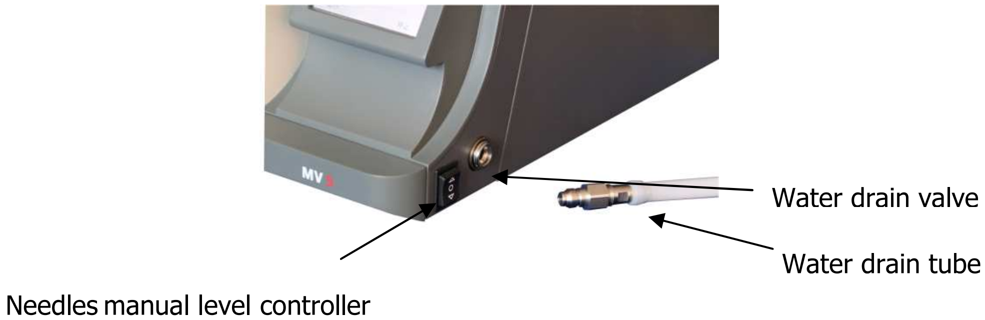

Figure 3. MultiVap54 evaporator side view

To increase safety and facilitate routine maintenance, the liquid inside the bath can be easily removed by the drain valve on the lateral side of the unit (see fig.3).

MultiVap54 is specifically designed as a fume hood free system. The exhaust hose on the backside of the instrument can be directly connected to any air vent system or fume hood to remove solvent vapours and grant user's safety (see fig. 4).

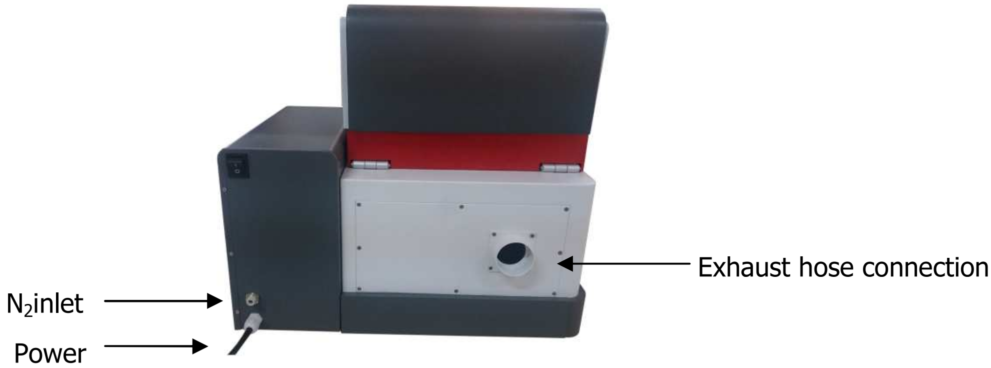

Figure 4. MultiVap54 evaporator back view

Experimental section

A series of experiments were conducted with pure solvents to test the evaporation efficiency of MultiVap54 among different sample tubes. N°30 test tubes for each solvents were placed to represent the whole sample tube rack area (26 around the perimeter and 4 in the middle section). 5 mL volume of six commonly used extraction solvents (methanol, dichloromethane, ethyl acetate, cyclohexane, hexane and acetonitrile), 0,5 mm/min nitrogen needle descend speed, nitrogen gas pressure from 2 psi up to 12 psi and different liquid bath temperatures were set to study the main important factors affecting evaporation efficiency.

Table 1. Experimental evaporation results

Solvent	Initial volume (mL)	H_2O Bath temp (°C)	N_2 pressure (psi)	Solution final volume (mL)	Evaporation time for final volume (min)	Time to dryness (min)
MeOH	5	44	10	1.1±0.3	25	28
		56	10			13
DCM	5	40	3			12
			7			11
			12	0.8±0.3	10	11
	5	35	12	1.0±0.3	10	13
EtAC: Cy (1:1)	5	76	12	0.8±0.4	6	9
EtAC: HEX (1:1)	5	65	12	0.9±0.4	6	9
ACN	5	55	12			24
		65	3			18
			7			16
			12	0.8±0.4	11	14
		75	12			12

---MeOH: methanol DCM: dichloromethane EtAC: ethyl acetate Cy: cyclohexane HEX: hexane ACN: acetonitrile

Looking at the results in table 1, it is evident that solvents were evaporated completely within 15 min (for 9 test conditions out of 13). N°4 tests requiring more than 15 min of evaporation time (methanol with liquid bath at 44°C, acetonitrile at 55°C, and acetonitrile at 65°C with N_2 pressure set both at 3 and 7 psi) show that liquid bath temperature and N_2 gas pressure have great impact on the evaporation rate.

Considering boiling points of methanol and acetonitrile (65 °C and 82 °C respectively), liquid bath temperatures set in MV5 (44°C and 55°C respectively) are certainly too low for these two solvents, which manifest their slow evaporation rates under these conditions. Once the water bath temperatures were raised close to the solvent boiling points, the evaporation rates were dramatically increased. Fig. 5 shows the ACN behavior: at increasing water bath temperature by 20 °C (from 55 °C to 75 °C), evaporation time decreases dramatically from 24 min to 12 min. Therefore, if the analytes are thermally stable, water bath temperature should be set as close as possible to the solvent boiling point to maximize the evaporation rate.

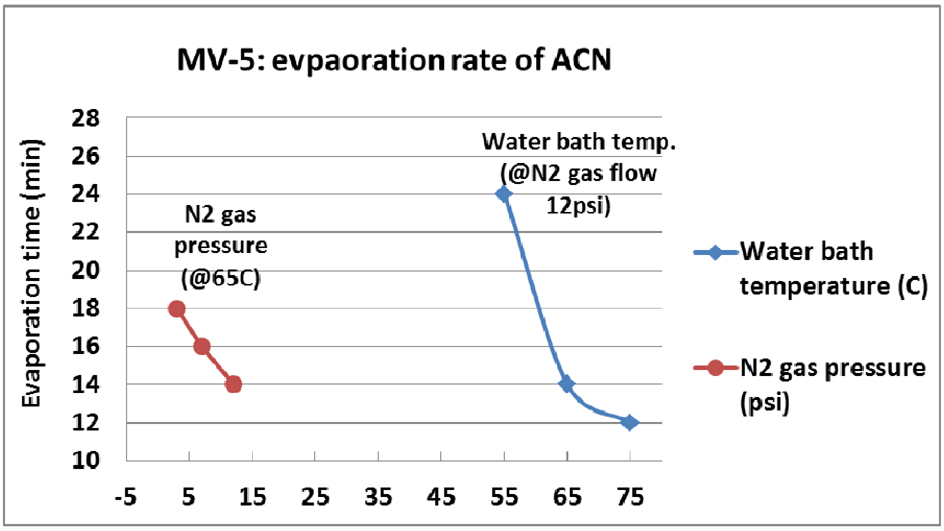

Figure 5. Evaporation time of ACN as function of N_2 gas pressure and water bath temperature

The effect of nitrogen gas pressure on evaporation efficiency was further analyzed. Three different N_2 pressure (3, 7 and 12 psi) were applied at both dichloromethane (water bath temperature 40°C) and acetonitrile (water bath temp 65°C) systems. For a volatile solvent such as dichloromethane (boiling point 40 °C), gas pressure appears not to greatly affect the evaporation rate, suggesting that water bath temperature is a dominant factor. Therefore, in the lab evaporation practice with volatile solvents, lower N_2 gas flow rate can still enable fast evaporation, with the positive effect of keeping low nitrogen gas consumption. By contrast, for less volatile solvents, as acetonitrile, which has relatively high boiling point (82°C), higher N_2 gas pressure leads to higher evaporate rate, as shown in fig. 5.

Final sample volumes, also listed in table 1, were measured to be ~1 mL for 6 test conditions, indicating that MultiVap54 is able of simultaneously evaporate sample solutions in different positions thanks to the particular design of nitrogen blowing needles (the small diameter of N_2 blowing needle allows N_2 gas to flow equally in all 6 different channels).

Conclusions

MultiVap54 automated evaporation system can significantly speed up the workflow and reduce the labor time during evaporation. All the features of MultiVap54, such as programmable method setup and automated vertical lift of nitrogen needles, promote the evaporation performance and reduce N_2 gas consumption. Preliminary studies demonstrate both water bath temperature and N_2 flow rate improve evaporation efficiency for different solvents. In the laboratory practice, water bath temperature should be appropriately set close to the solvent boiling points, not underestimating the risk of losing analytes in order to obtain good sample recovery.