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Short Communication

Time- and cost-saving approach for liquid chromatography–mass spectrometry vacuum systems

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ABSTRACT

A refrigerated trap system is described for recovering the mobile phase utilized with popular liquid chromatography–mass spectrometry interfaces. By efficiently freezing the excess LC effluent, vacuum system components are protected from rapid wear and scheduled maintenance intervals can be substantially extended. The modest cost of the system is easily justified based on the increased lifetime afforded to expensive vacuum pumps and the added convenience, time savings and the safety gained by the elimination of dry ice–solvent or liquid nitrogen bath systems.

INTRODUCTION

In LC systems, the analytes of interest typically comprise only a very minor proportion of the flowing mobile phase. When a mass spectrometer operating under high vacuum is utilized for LC detection, provisions must be made to accommodate the high mass transfer rates associated with removal of the mobile phase from the vacuum chambers. For some early LC–MS interfaces such as direct liquid injection or moving belt devices, simply increasing the effective pumping speed of the mass spectrometer's vacuum system was usually adequate because mass transfer was reduced prior to reaching the vacuum system. In the more modern LC–MS interfaces where the vacuum system must accept the full flow of the LC effluent, such as thermospray [1] and particle beam systems [2], the load on the vacuum mechanical pumps is much greater. The situation is often exacerbated by the use of solvents ill-suited for pumping by standard vacuum pumps. Water, for example, is a very common mobile phase component that is exceedingly corrosive to the internal parts of mechanical vacuum pumps.

The typical method for accommodating the LC mobile phase flows associated with modern LC–MS interfaces employs one or more additional mechanical pumps, which are usually protected by an in-line dry ice solution cold trap. The pumps either remove most of the solvent mixture before it arrives at the ion source in a differential pumping configuration, or pumps away the entire solvent stream from the ion source after a small fraction has been sampled by the mass spectrometer. These two configurations are commonly used in particle beam and thermospray interfaces, respectively [2]. The weak link in using these systems is usually the cold trap used

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TABLE I

TEMPERATURES OF DRY ICE BATHS

Values from ref. 3. Note: isopropanol value approximated based on melting point.

Solvent	Temperature (°C)
Ethylene glycol	−15
Carbon tetrachloride	−23
3-Heptanone	−38
Acetonitrile	−42
Cyclohexanone	−46
Chloroform	−61
Ethanol	−72
Acetone	−77
Isopropanol	−90
Diethyl ether	−100
Savant RTV4104 Refrigerated Trap	−104[a]

[a] Included for comparison.

to recover the mobile phase. As shown in Table I, the dry ice slurries prepared for these traps provide cooling to temperatures that are only marginally lower than the freezing point of the mobile phase components. (Acetonitrile, for example, has a freezing point of approximately −46°C.) These slurries only maintain their temperature for a few hours before requiring the addition of dry ice. After a few days, the entire slurry must be discarded because contamination by water condensing from the air causes the temperature of the slurry to rise substantially. Dry ice baths tend to bubble and froth when the coolant is first admitted to the dewar. Furthermore, the solvents commonly used in these cold traps are often flammable, and may also pose other safety concerns, such as exposure hazards. Liquid nitrogen traps offer a lower temperature alternative but share the remainder of the disadvantages of dry ice traps. In practice, it is rare that dry ice or liquid nitrogen traps are adequately maintained. As a consequence, a considerable fraction of the mobile phase is pumped all the way into the mechanical vacuum pump, where it condenses, collects, and corrodes the pump's internal parts. Unless the pump oil is drained and replaced each time it is contaminated, which may be on a daily basis, it is likely that the mechanical vacuum pump will need rebuilding or replacement every three to six months.

In this note, we describe a refrigerated solvent recovery system which replaces the dry ice–solvent cold trap. All the components of this system are commercially available at a cost low enough that the system quickly pays for itself in savings for dry ice and solvents. The system also affords superior protection against mobile phase induced damage to the mechanical vacuum pumps, which leads to the greatest cost savings, since rebuilding or replacing these pumps can be quite expensive. Most importantly, the system can result in considerable time savings for the personnel involved by minimizing routine start-up and shutdown time and by reducing instrument down time.

EQUIPMENT

The refrigerated solvent recovery system is illustrated in Fig. 1. The refrigerated trap and mechanical vacuum pump are shown in the configuration we have used extensively, with a thermospray type LC–MS interface (TSP1) on a triple stage quadrupole mass spectrometer (Finnigan MAT TSQ-46).

The refrigerated solvent recovery trap is a model number RVT4104 trap supplied by Savant Instruments (Farmingdale, NY, USA) at a cost of US $2500. A silicone heat transfer fluid is

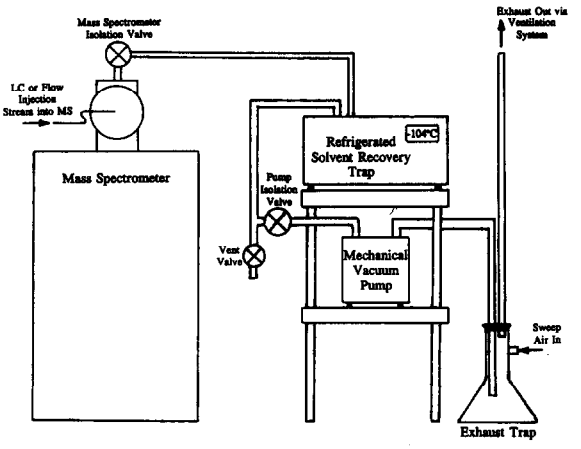

Fig. 1. The refrigerated trap mobile phase recovery system for LC–MS.

cooled to −103°C to −112°C by a low-temperature refrigeration system. This fluid remains a liquid, insuring good thermal contact with a 4-l glass vessel that collects the condensed solvent. Both the refrigeration system and the glass solvent collection vessel are enclosed in a square metal cover. Temperature monitoring is provided via a digital display. The glass collection vessel can be easily removed when it is filled by disconnecting the vacuum hoses (equipped with quick-disconnect fittings) and raising it through a large hole on the top of the unit. The three vacuum valves shown in the figure make it possible to perform this operation without breaking vacuum for the mechanical vacuum pump or the mass spectrometer. The mechanical vacuum pump is kept running and at full operating temperature at all times in order to prevent the condensation of corrosive solvents in the lubricating oil.

The mechanical vacuum pump is a Balzers UNO-016B single stage direct drive pump with a pumping speed of 360 l/min, designed expressly for LC–MS applications. During operation, the ballast is opened slightly. The pump is raised off the ground on the shelving shown in Fig. 1 in order to achieve the following two advantages: First, the exhaust line descends to the exhaust trap, allowing any condensable vapors expelled by the pump to collect in the ambient temperature exhaust trap. This prevents any contaminated liquids from refluxing back into the pump oil and minimizes their build up in the ventilation system. Second, routine maintenance for the mechanical vacuum pump is facilitated by locating it above the lab floor. One oil drain is located on the front of the pump, which projects out slightly from the shelf. A 3-in. (7.6 cm) diameter hole cut in the shelf, allows easy access to the other oil drain plug under the base of the pump.

RESULTS AND CONCLUSIONS

In a recent paper, we described techniques developed to carry out automated routine sample analyses using flow injection thermospray mass spectrometry [4] with the system shown in Fig. 1. As a result of our successful efforts in automation, this instrument is used quite heavily (over 4000 samples analyzed per year). Before installation of the refrigerated solvent recovery trap, a 1-l glass trap cooled in a dry ice–isopropanol bath was used in-line, just before the same mechanical vacuum pump. During operation, the pressure measured at the intake to the pump would typically rise to 1 Torr (133 Pa) or more, for a mobile phase flow-rate of 1.4 ml/min. The pressure in the mass spectrometer was approximately $2 \cdot 10^{-5}$ Torr (2.7 mPa). For a recently rebuilt or new mechanical pump, the pump oil would typically require replacement every 2–4 weeks. After a few months of service, even more frequent oil changes were required. On average, a full rebuilding of the pump was necessary every 6 months.

After installation of the new refrigerated trap system, the pressure rise at the mechanical pump intake became barely discernable when the same mobile phase flow was initiated. With the pump ballast open, the typical pressure during operation is less than 100 mTorr (13 Pa). The pressure in the mass spectrometer remains at $2 \cdot 10^{-5}$ Torr (2.7 mPa). The maintenance interval between pump oil changes was extended to once every 3 months, and the need to rebuild pumps should be reduced to a frequency approaching that of pumps operating in far less demanding applications (once every 3 to 5 years). In addition to these benefits, the new system is much easier to use than the messy

TABLE II

KEY FEATURES OF REFRIGERATED AND DRY ICE SOLVENT RECOVERY TRAPS

Feature	Dry ice	Refrigerated
Operating pressure	>1000 mTorr (>130 Pa)	<100 mTorr (<13 Pa)
Oil change frequency	2 weeks	3 months
Pump rebuild frequency	6 months	3–5 years[a]
Required attention frequency	4 h	1 week

[a] Estimated lifetime.

solvent bath trap. Instead of the daily preparation and hourly attention required by a solvent–dry ice bath, the only requirement is replacing the glass vessel with a clean one, which takes *ca.* 1 min. At our usage rate this is required only once a week. The comparative features of the refrigerated and dry ice types of vapor traps are given in Table II. Although, the refrigerated system has not been tested with particle beam LC–MS, we expect similar performance improvements for systems where the mechanical pumping system is required to accept liquid mobile phase flow-rates on the order of 1 ml/min.

In summary, the refrigerated trap system represents a significant improvement that can benefit many laboratories using LC–MS on a routine basis. The main benefits include better protection and less frequent maintenance intervals for mechanical vacuum pumps, no daily preparation and periodic attention to cold baths, and the elimination of flammable and potentially hazardous solvents. The net result is considerable savings in both time and money.

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