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5973N*etwork* Series MSDs

GC Column Selection and Pumping Considerations for Electron and Chemical Ionization MSD operation

Introduction

This brief describes the helium carrier-gas flow recommendations for the various pumping configurations available for the 5973N family of MSDs. The intention is to provide information that will allow customers to choose the systems that best fit their chromatography needs. The operating recommendations for electron ionization (EI) and chemical ionization (CI) modes are summarized in Table 1.

Considerations of Capillary-Column Dimensions for GC/MS-EI Analysis

Diffusion-Pumped Systems versus Turbo Molecular Pumps

Turbomolecular pumps offer several advantages over diffusion pumps. Turbomolecular pumps are more rugged with respect to fluctuations in vacuum integrity than diffusion pumps; that is, these systems have a better chance of enduring "accidental excursions" in pressure or catastrophic vacuum failure. Also, turbomolecular pumps tolerate oxygen, water, and other moderately reactive contaminants far better than diffusion pumps. (Consistent trace levels of oxygen not only rapidly destroy GC capillary-column phases, but quickly damage diffusion pump oil and degrade pumping speed and performance.) However, for clean systems, diffusion pumps can be simpler and less expensive to maintain, and have longer lifetimes if properly maintained, serviced, and operated.

Standard versus Performance Turbomolecular Pumped Systems

The additional gas throughput tolerated by the larger performance turbomolecular pump system has advantages for applications that require larger-bore columns. The performance turbomolecular pump system offers the user a wider range of operating carrier-gas flows, which provides some flexibility in method development. However, many users are moving toward more rapid analysis which has been achieved by utilizing the narrow and microbore

Table 1. MSD System pumping configurations, specifications, and recommendations.

System Identifier	G2577A	G2578A	G2579A	G2588A	G2589A
System Pumping Configuration	Diffusion Pump	Standard Turbomolecular Pump	Performance Turbomolecular Pump	Standard Turbomolecular Pump	Performance Turbomolecular Pump
Ionization Modes	EI only	EI only	EI only	EI and PCI	EI, PCI and NCI
Optimal helium gas flow	1 ml/min	1 ml/min	1 to 2 ml/min	1 ml/min	1 to 2 ml/min
Maximum recommended helium gas flow for EI operation	1.5 ml/min	2 ml/min	4 ml/min	2 ml/min	4 ml/min
Recommended column dimensions for EI operation (maximum i.d. × minimum length)	0.25 mm × 30 m	0.32 mm × 30 m	0.53 mm × 30 m	0.32 mm × 30 m	0.53 mm × 30 m
Maximum recommended helium gas flow for CI operation	–	–	–	1.2 ml/min	2.4 ml/min
Recommended column dimensions for CI operation (maximum i.d. × minimum length)	–	–	–	0.25 mm × 30 m	0.25 mm × 15 m

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capillary columns (0.200-mm i.d. and smaller). These narrower columns offer several advantages, one of which is lower carrier-gas flows than with the larger columns and consequently reduced pumping requirements. To aid users in scaling their existing GC methods toward the more rapid methods, an Application Note has been written describing the scaling of GC methods,[1] and a user-contributed software module[2] is available. The user-contributed software module can also be used to calculate the expected volumetric carrier-gas flow introduced by a particular column configuration which will aid in choosing the appropriate pumping configuration.

Considerations of Capillary-Column Dimensions for GC/MS-CI Analysis

In CI operation, the reagent gas is maintained at high pressure in the source; as a result, a significant portion of the pumping capacity is dedicated to evacuating the reagent gas. The sum of the reagent gas flow and helium carrier-gas flow should not exceed the maximum recommended helium-gas flow for EI operation cited in Table 1. CI techniques focus primarily on the detection of trace amounts of analytes, and consequently the quantities introduced into the analytical column are typically much less than 1 ng. Therefore, column capacity considerations suggest that capillary column diameters equal to or less than 0.25 mm i.d. will be the most appropriate. This inherently places a flow restriction on columns such that the optimum practical helium carrier-gas flow will seldom exceed ~1-ml/min (~40 cm/sec linear velocity). An exceptional case is the chromatography of highly active compounds (e.g., underivatized drugs) which can be improved by very high carrier-gas flows. For these unusual cases in which column flows that routinely exceed 1.2 ml/min for PCI analysis are required, the larger performance turbomolecular pumping system is recommended.

As a precautionary note, users should not expect equivalent performance in PCI on the standard and performance turbomolecular pumped systems nor that methods developed on one platform will directly translate to the other. Although both systems demonstrate outstanding sensitivity, reproducibility, and spectral quality, it is noted that adduct formation and other features of PCI are dependent on absolute source pressures, which can differ between the two platforms.

Electron-capture negative ion mass spectrometry (also known as negative chemical ionization MS) requires that the performance turbomolecular pumping system be used (since buffer gas loads exceed those of reagent gas flows in PCI); therefore pumping capacity is quite large and carrier flows could be chosen to be large. Again, in view of the requirements for trace analysis with capillary columns, helium carrier–gas flows in excess of 2 ml/min are unlikely and the user can be assured of excellent performance with 0.25-mm i.d. columns in NCI.

The 5973N series of MSDs offers a variety of configurations that allow users to tailor their choice of system to their gas chromatographic requirements based on their particular methods and future analytical intentions.

References

1. Quimby, B. D., Blumberg, L. M., et al., *Precise scaling of gas chromatographic methods using method translation and retention time locking,* Hewlett-Packard Company, Application Note 5967-5820E, *www.agilent.com/chem,* select PUBLICATIONS, then search using note number as the keyword.
2. GC Capillary Column Method Translating Software 2.0.a.c. available at *www.agilent.com/chem,* select TECHNICAL SUPPORT, then USER CONTRIBUTED SOFTWARE.

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