



5973Network Mass Selective Detector

This publication provides general technical information about the 5973Network MSDs to accompany the specifications and brochure. The MSD series was originally developed when Agilent Technologies was a part of the Hewlett-Packard Company.

General Description and Configurations

The Agilent Technologies 5973Network MSD is a transmission quadrupole mass spectrometer for qualitative and quantitative analysis by electron ionization (EI), positive chemical ionization (PCI), and negative chemical ionization (NCI). The 5973N is available in six configurations that differ in their capabilities (See Table 1). Use considerations will dictate which system is most appropriate.^{1, 2}

The 6890/5973N systems can be configured with additional accessories to adapt them for various applications. The MS Productivity ChemStation Data System can process data from both the MS and conventional GC detectors. Table 2 is a partial list of options that may be added to enhance the sample introduction, separation, and detection capabilities. With devices such as the programmable temperature vaporizer (PTV), overall sensitivity can be dramatically increased. Some of the accessories are provided by third-party channel partners who have been qualified by Agilent Technologies. Check with your local sales representative to discuss your exact needs and the availability of the accessories in your area.

Table 1. Agilent Technologies 5973Network MSDs.

GC/MS System	GC		Modes			System Control	
	6850	6890	EI	PCI	NCI	LCP ^a	ChemStation
Diffusion pump EI	✓		✓			✓	
Diffusion pump EI		✓	✓			✓	✓
Standard turbo EI		✓	✓			✓	✓
Performance turbo EI		✓	✓			✓	✓
Standard turbo PCI		✓	✓	✓		✓	✓
Performance turbo CI		✓	✓	✓	✓	✓	✓

^a Local Control Panel

Table 2. Partial list of available 6890 accessories.

Sample Introduction	Inlet	Detectors
Automatic liquid sampler	Split/splitless injector	Electron capture
Headspace sampler	Cool on-column	Flame ionization
Purge and trap	Cool on-column with solvent vapor exit	Thermal conductivity
Gas and liquid sampling valves	Programmable temperature vaporizer	Flame photometric
Air canister sampler ^a	Volatiles inlet	Nitrogen-phosphorus
Thermal desorber ^a	Pre-column separation ^a	
Pyrolyzer ^a		
Solid probe ^a		
Solid phase micro extraction ^a		

^a Option available through channel partner

Instrument Technology

Autosampler

The optional 7683 autosampler provides great flexibility for a variety of applications. It can be set for fast or slow plunger speeds with variable pre- and post-injection dwell times. It can perform automated cool on-column injections into 0.32 mm and 0.25 mm i.d. columns. Variable needle depth allows ambient headspace, multi-phase, and small-volume sampling. Up to eight samples can be automatically injected with just the tower. For more samples, up to two injectors can share a 100-sample tray.

Advanced Electronic Pneumatics Control

The Agilent Technologies gas chromatographs now incorporate a fourth-generation electronic pneumatics control (EPC) that is the result of 10 years experience and research. The EPC can maintain optimum column flow as the oven temperature changes and can even compensate for fluctuations in laboratory temperature and pressure. This means that you will get reproducible retention times even if you are running samples over several days. Ramped flow and pulsed split/splitless as well as standard injection modes are available.

Independently Heated, Short GC/MS Interface

The 5973N GC/MS interface is designed to preserve the chromatographic separation. The independently heated 17-cm interface is one of the shortest on any mass spectrometer (see Figure 1). In poorly heated or longer interfaces, uneven temperatures may lead to chromatographic peak tailing. An interface that is too hot may thermally degrade some compounds or give rise to high column bleed. The independently heated 5973N interface allows you to select the temperature that is best suited for your application.

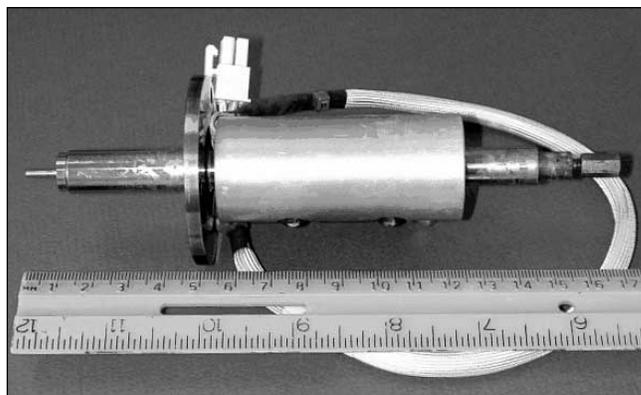


Figure 1. Short GC/MS interface minimizes thermal problems.

External Ionization Sources

The 5973N can perform electron ionization (EI), positive chemical ionization (PCI), and negative chemical ionization (NCI). The 5973N provides classic library-searchable EI spectra. Chemical ionization is used for more selective and sensitive analysis or where molecular weight information is desired. NCI is particularly suited for compounds that are generally analyzed by electron-capture detectors (ECDs) such as halogenated compounds; polynuclear aromatics; those containing oxygen, nitrogen, or phosphorus; and organometallics. The sensitivity of NCI in selected ion monitoring is comparable to that achieved with the ECD, but spectral information is obtained as well as chromatographic retention-time data.

The 5973N ion sources are optimized to provide the highest quality data and the greatest sensitivity for each technique. Electron ionization typically requires source pressures of 0.001–0.005 Torr. Chemical ionization uses source pressures ranging from a fraction of a Torr to several Torr. Rather than compromise spectral quality or sensitivity, dedicated sources are used on the 5973N. The 5973N provides direct temperature control of the ion source to ensure repeatable, reliable operation.

Metallized Quadrupole

Ions generated in the MSD source are separated in a metallized quartz quadrupole. Earlier generations of quadrupoles were composed of machined molybdenum rods held precisely in position by ceramic spacers. But the rods can become misaligned if the assembly is dropped or otherwise subjected to excessive shock. Because the 5973N quadrupole is made of a single piece of quartz, the critical distances remain constant with use, affording many years of high-precision analyses. The gold coating on the quadrupole is a very smooth surface that provides uniform fields. The state-of-the-art manufacturing processes used in the production of this quadrupole yield greater sensitivity, long-life cleanliness, and greater repeatability from instrument to instrument.

The source, lens, and quad assembly is designed for easy maintenance. All parts that need cleaning are easily accessible. The variable-voltage entrance lens on the 5973N allows ions to pass through the fringing fields at the main entrance to the quadrupoles without using pre-filters. The entrance lens is cleaned along with the all the other lenses. Among the tens of thousands of MSDs in use, very few quadrupoles have had to be cleaned, or even more seldom, replaced. (See Figure 2.)

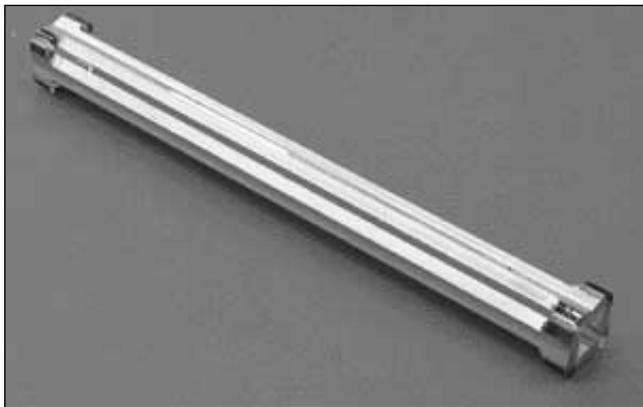


Figure 2. Gold quadrupole provides long-term performance stability.

High Energy Dynode-Electron Multiplier Detector

The 5973N uses an off-axis high-energy-dynode electron multiplier (HED-EM) detector to reduce noise from neutrals. The HED-EM offers better sensitivity to higher-mass ions than do photomultiplier systems or EM detectors without the HED. This additional sensitivity to high-mass ions is valuable for qualitative and quantitative analyses of molecules that are easily fragmented.

LAN and Local Control Panel

The 5973N was designed to allow you to place the instrument and data system wherever you want. Each MS ChemStation Data System can control two 5973N GC/MSD systems using a Local Area Network (LAN). You can monitor an analysis, review data, and process reports from the comfort of an office. At the same time, you reduce expensive laboratory space requirements by half. Even though the instrument and data system are separated, you still have control over the instrument through the Local Control Panel (LCP). You can tune, check, and vent the MSD, run a method or sequence, or check the network. A quick glance gives the operator a wealth of information such as whether the instrument is ready, what test it is performing, or whether it is pumping down. You can access data in the office without losing the ability to control the GC/MSD in the laboratory. (See Figure 3.)



Figure 3. Local Control Panel provides at-instrument control when the ChemStation is located remotely.

Macro Language

Most users will be satisfied with the convenient menu systems and forms of the MS ChemStation software. For advanced applications, the data system offers a full-capability macro language to manipulate chromatographic and spectral data. Many customers have used this macro language to customize their menus, data review, and reporting. A single mouse click can substitute for many steps. In addition to the GC/MS data files, the macro language can process GC data files.

Instrument Performance

Autotuning

Mass spectrometers must be tuned to ensure that peak shapes, isotope ratios, and mass assignments are correct. In the past, tuning was a tedious, manual process that required an expert user. Hewlett-Packard developed automatic MS tuning to make the technique available to even occasional users. Autotuning is available for PCI and NCI as well as EI operation. Additionally, there are specialized tunes for maximum sensitivity, for user-set target ratios, and for meeting the DF/TPP/BFB tuning criteria for U. S. Environmental Protection Agency (EPA) methods in EI.

Classic Spectra

It is critical that spectra be comparable between instruments and laboratories, not just that they be library searchable. Sophisticated library-search algorithms can perform matches even when there are extraneous or missing peaks. The 5973N produces classic spectra that have isotope ratios representative of the natural abundances. The spectral integrity is not compromised by artifacts from ion-molecule reactions or spectral changes due to concentration changes. The MSD has been the most popular GC/MS used in environmental, drugs-of-abuse, and sports doping testing where results must stand up to the most rigorous scientific and legal scrutiny. It has been the predominant instrument used to obtain and confirm spectra in spectral libraries.

Sensitivity

Sensitivity is typically specified as the signal-to-noise ratio for an injection of a pure standard in a solvent. Many factors determine the sensitivity for your specific application, including system cleanliness, the compound that is being measured, and the matrix. In a survey of 5973 users representing various applications and expertise level, only 16 responses out of 497 indicated that EI sensitivity is inadequate for some samples. The same survey showed high degrees of satisfaction for both PCI and NCI sensitivity.

System Inertness and Temperature Control

A GC/MS should analyze the sample without degrading analytes because of active or hot surfaces. The 368/386 ion ratio in the cholesterol spectrum has been the classic test. In a good system, the ratio of 368/386 should be less than 50%. Figure 4 shows the spectra of cholesterol at 200°C and 250°C. At the lower temperature, the ratio is 45%, thus passing the test. At the higher temperature, the ratio is 56%, thus failing the test. Without the ability to independently control the temperature of the column oven, the GC/MS interface, and the ion source, you may not be able to optimize conditions for your analytes. The MSD allows you to set these temperatures to ensure the best performance.

Retention Time Locking

The advanced electronic pneumatics control on the 6890 and 6850 GCs has led to the development of retention-time locking (RTL). RTL allows the matching of retention times from instrument to instrument and from laboratory to laboratory. Table 3 shows retention times for three compounds collected on different instruments, with different injectors and detectors, operated by different people.

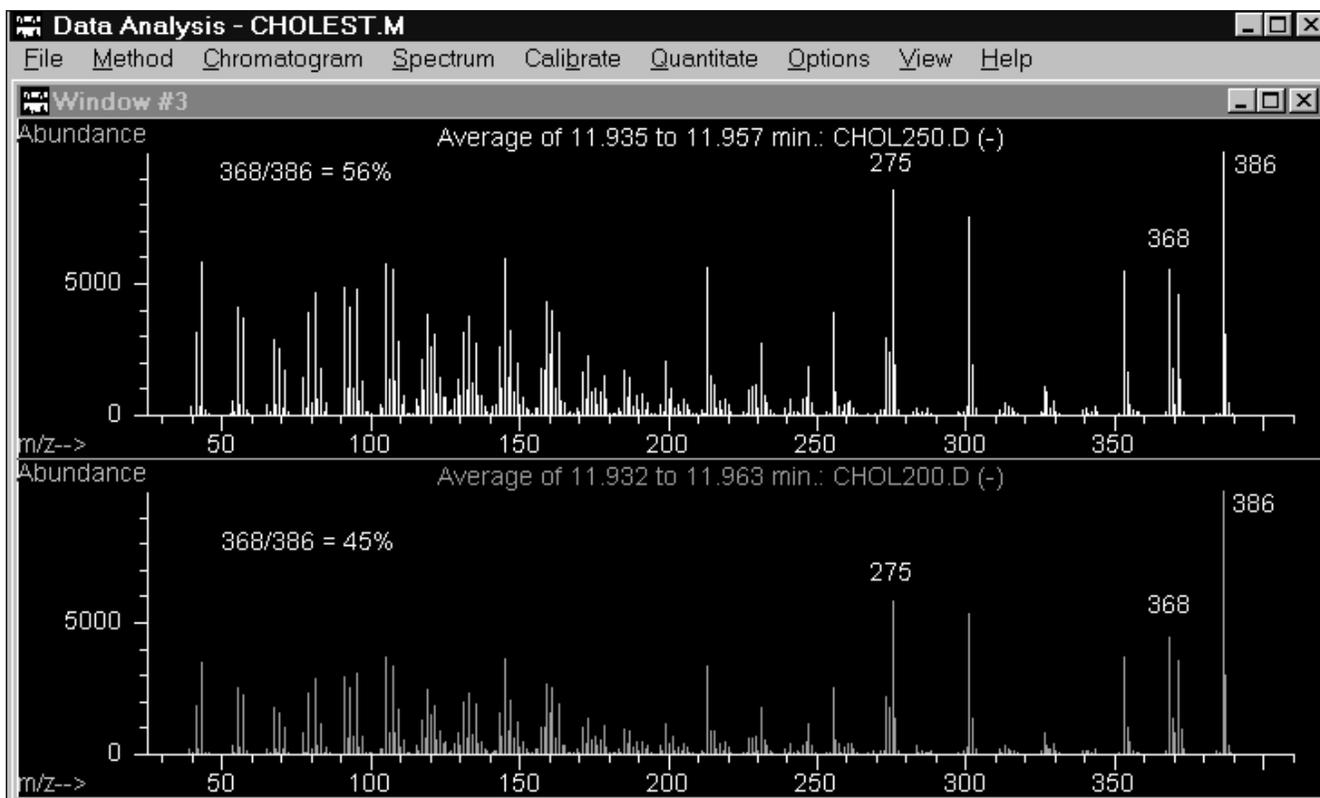


Figure 4. Source temperature has a major effect on the spectrum of cholesterol.

Constant retention times benefit the laboratory in several ways. For a single instrument, it means that the retention times can be kept within narrow windows. Retention times in quantitation databases can be maintained even after the front part of the GC column is clipped in normal maintenance. For laboratories with multiple GC/MSDs, a master method can be developed and used for all instruments, simplifying initial setup and subsequent data review.

Retention time locking allows easy matching of compounds detected on non-MS detectors. Peaks found by a conventional GC detector can be confidently matched with those on a MS (See Table 3).

Fast Chromatography

Faster chromatography requires makes more stringent demands on the GC/MS system. In order to effectively use smaller bore columns, the GC injection system must be able to inject small volumes, rapidly and reproducibly. The 7683 autosampler can inject 0.1 μ l of extract and can support a large split ratio of 7500:1. Although the standard 6890 performs fast and reproducible temperature ramps, even greater capabilities are available on the high-voltage model. To perform fast chromatography, the mass spectrometer must scan faster but still provide good quality data. It is possible to specify a fast scan rate and still collect data that display changes in the ion ratios or losses in isotope peaks. The 6890/5973 system performance is shown in reducing a 17 minute analysis to less than 4 minutes while maintaining spectral integrity.³

Table 3. Reproducibility of retention times across different instruments and detectors operated by different people using RTL.

Inlet	Detector	Injection Technique	Dichlorvos	Chlorpyrifos Methyl	Mirex
COC	MSD	On-column	5.862	16.607	29.836
PTV	MSD	Splitless	5.897	16.593	29.800
S/SL	FID	Splitless	5.797	16.587	29.856
S/SL	AED	Splitless	5.829	16.600	29.839
S/SL	AED	Splitless	5.837	16.604	29.851
PTV	Micro-ECD	Split	5.798	16.576	29.876
PTV	Micro-ECD	Split	5.860	16.597	29.864
PTV	Micro-ECD	Cold splitless	5.862	16.589	29.867
S/SL	Dual FPD	Splitless	5.814	16.596	Undetected
S/SL	NPD	Splitless	5.814	16.596	Undetected
High-low			0.100	0.028	0.076
Average			5.837	16.595	29.849
Standard deviation			0.033	0.009	0.024
Relative standard deviation			0.560 %	0.054 %	0.080 %

Instrument Ease-of-Use and Maintenance

Learning Products

The 5973N comes standard with a variety of learning products to make it easy to understand mass spectrometry and use the instrument. The Reference Collection CD includes a computer-based training program to help you learn MS theory. There is a short *Getting Started* manual for the basic instrument operations, as well as the Windows online help. When it is time to perform operations like changing the septum or cleaning a source, online video files show you the exact procedure. These tools help you to operate and maintain the instrument.

Easy Venting and Column Changes

The 5973N was designed for easy maintenance. Instrument venting can be accomplished through the MSD ChemStation Data System or the Local Control Panel. You remove one nut from inside the GC oven to remove the old column and install the new one. You can change a column without moving the instrument or removing the ion source.

Swing-Out Design for Easy Access to the Source, Lenses, and Detector

Experience with earlier MSDs has allowed us to improve accessibility to various components. On the 5973N, the parts that are typically serviced are mounted on a metal plate that swings out from the left side. There is no need to remove components from the

back or top of the instrument or to separate the GC and MS. The 19-part source and lens assembly is removed from the mounting plate by unscrewing two thumbscrews. Detector service is just as easy. You only need to squeeze a spring clamp to remove or reinstall the electron multiplier horn. No extra alignment is necessary. The procedure is easy and rapid. System venting is minimized with the dual filament design. (See Figures 5 and 6.)

Complete Instrument Control and Data Storage

The MSD Productivity ChemStation Data System provides integrated control of the 7683 autosampler, 6890 gas chromatograph, and 5973N mass spectrometer. You get the full capabilities of all components, not just the basic functions. System methods store parameters for instrument control, data acquisition, data processing, and custom reporting. You can set up special displays to monitor such values as temperatures at various points and foreline pressure. Tune values are automatically saved so that long-term trends may be evaluated. This complete information storage helps with the GLP and ISO compliance.

Application Based on Standard Windows NT Environment

The ChemStation software is a 32-bit Windows NT application that runs on a personal computer. Since most laboratory personnel are familiar with the graphical user interface, the application is easy to learn. The application software provides instrument control, data processing, and reporting capabilities.

Flexible Software for Processing, Reviewing, and Reporting Data

The MS ChemStation Data System allows you to define complex methods that are easy to run. Data can be collected in either the scan or selected ion monitoring (SIM) mode. Method developers can set 50 groups of 30 ions each in SIM acquisition with independent dwell times, adjust ionization energies or currents, and time program events. Menus and prompts guide the user through the quantitation setup. The software comes complete with common general reports, as well as industry-specific ones.

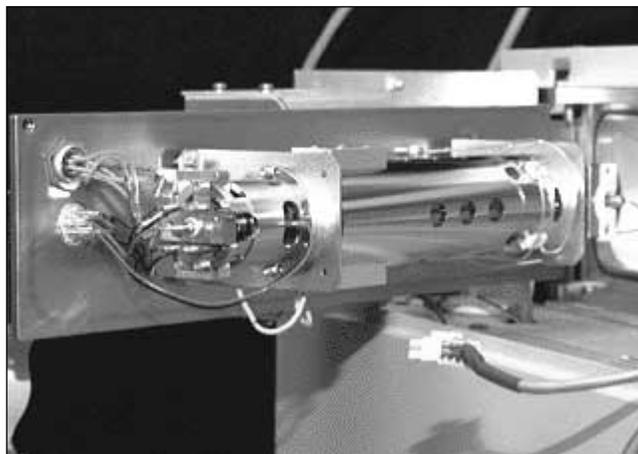


Figure 5. Serviceable components of the MSD are easily accessible through a swing-out panel.

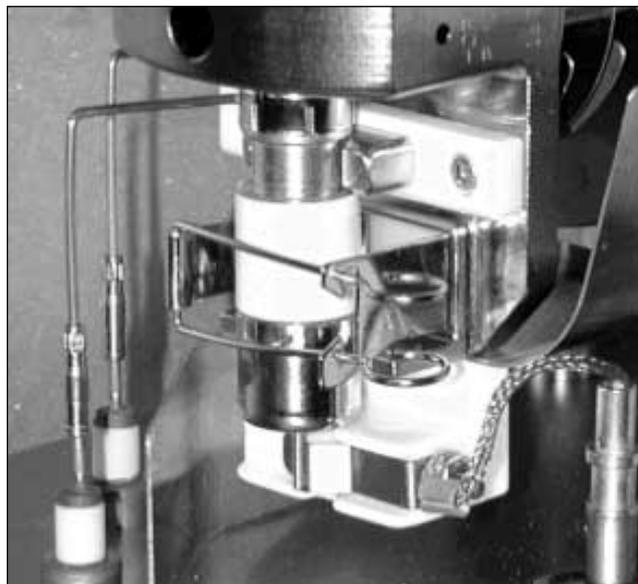


Figure 6. The MS multiplier is easily replaceable without tools.

Should further custom reports be required, the software includes a report writer that can incorporate graphical and textual elements. Once the report template is set up, the reports can be automatically generated when a method is run. Additional flexibility is provided by sending the results to a database or to other Windows applications.

Reference Libraries

To assist in spectral interpretation, there are two library search algorithms available: Probability Based Matching and the optional NIST algorithm. The spectral libraries available are the following:

- NIST 98 (129K spectra)
- Wiley 6th edition (275K)
- Pflieger drug (4300)
- Stan pesticide (340)

There is also a chemical structures library that can be used to incorporate publication-quality graphics.

The 5973N can also use a unique 560 pesticide and endocrine disruptor library based on the retention-time locking capabilities of the gas chromatograph. This collection includes prominent pesticides used in the United States, Europe, Japan and elsewhere. With the retention-time locking capabilities of the 6890 and 6850, you and your colleagues around the world can reproduce these results and add other compounds. The laboratory can also build its own RTL libraries.

Applications-Oriented Operating Modes

The operating mode of the ChemStation Data System can be selected for a particular laboratory application. The standard operating mode provides the tools needed for general applications. In addition, there are modes tailored for specific applications: environmental, drugs-of-abuse, and aromatics in gasoline analyses. These applications provide calculations and reports that are widely accepted in their particular industries.

Common Software for GC and GC/MS

The MSD Productivity ChemStation Data System can collect and process data from standard GC detectors as well as the MS. Four detectors can be monitored on up to two gas chromatographs. This may be two 5973Ns or a 5973N along with a conventional detector, or 6890s with conventional detectors. The data system will display and align signals from both the MS and the other GC detector during the run. Signals from the MS and conventional detectors from the same or different data files may be overlaid for comparison. Since the software supports the file formats of the Analytical Instrument Association (AIA), it can process data from other instruments, reducing the number of software systems that have to be learned. Having the same software for GC and GC/MS makes it easy for new users to operate more of the equipment in the laboratory, thereby reducing operating costs and avoiding errors.

Agilent Technology's Commitment to Quality

Quality Standards

Agilent Technologies is the largest manufacturer of GC/MS systems in the world. Products are developed under ISO 9001 standards and comply with common safety standards. We have a certificate of validation for our process of creating, testing, and validating our software. As part of our audited processes, we track the service calls for each model in order to improve reliability during the model lifetime and for any successive-generation instrument. In the first year, the 5973 MSD quality exceeded that of the 5972 at maturity.

Rugged Instrumentation

The GC/MSD is designed for less-than-optimal transportation and operating conditions. The instrument operates between 15°C and 35°C with the relative humidity between 25% and 50%. MSDs have been successfully used in mobile laboratories for critical tasks such as environmental and chemical-warfare-agent analyses. A broad environmental operating range means that a GC/MSD can operate reliably in many facilities without modifications.



5973Network MSD

Although the instrument has been designed to run under adverse environmental conditions, it has no requirements for extraordinary support utilities. The electronic autosampler does not require air for actuation; the vacuum pumps do not require water-cooling; and the GC oven can operate at 5°C above ambient temperature without requiring cryogenic cooling. Both GCs offer rapid heating and cooling rates, as well as precise temperature control. The EPC even compensates for temperature and pressure changes in the laboratory.

Agilent Technologies designs instruments that provide great flexibility. The linear dynamic range of an analysis is dependent on a large number of factors including the analyte chemistry, system inertness, and other factors. However, the MSD electronics support a total dynamic range of six orders of magnitude. To accommodate other factors that influence the linear dynamic range, the software provides various curve-fitting routines. The 5973 may well operate beyond the limits of your analyses.

Services

We have developed support services and partnerships to help customers meet their analytical challenges in the coming decades. Consult the Agilent Technologies web pages or your local sales representative for additional information about products, services, applications, and seminars.

References

1. Prest, H. F., "Ionization Methods in Gas Phase Mass Spectrometry; Operating Modes of the 5973Network MSDs, Agilent Technologies. Application Note 5968-7957, Oct. 1999.
2. Prest, H. F., "GC Column Selection and Pumping Considerations for Electron and Chemical Ionization MSD Operation," Agilent Technologies. Application Note 5968-7958, Oct. 1999.
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