The 5975C Series MSDs: Guidance in Implementing High Ion Source Temperatures

Technical Overview

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Introduction

The default ion source operating temperature for electron impact (EI) operation is 230 °C. This temperature has a wide range of applicability and has been the default for this reason. The 5973 and 5975A and B Series sources temperature maximums were raised to 300 °C under ChemStation G1701DA revisions[1]. By acquiring the proper source configuration, or acquiring and replacing the proper source parts, both EI and chemical ionization (CI) source temperatures as high as 350 °C are possible under ChemStation G1701EA (E.00.xx). The new 5975C MSD ion source is fully configured for use up to 350 °C.

Increasing the ion source temperature has advantages and disadvantages and should be carefully considered and tested in applications before implementation. It must be understood that the lifetime of the source *must and will be* lower at higher temperatures. Also understood is that operating at higher temperatures requires an even higher degree of care in every respect, especially in precluding leaks that can permanently damage the source material or components.

This overview will provide some guidance and recommendations as to how to implement and even whether to implement higher source temperatures. This will entail a discussion on how to choose an operating source temperature. Regardless, the section on ***analyzer baking*** will be of universal utility.

Source Configurations and Parts

Unless the source has been configured for higher temperature operation, it should not be allowed past the 300 °C limit. Table 1 gives part numbers for the sources and the upgrade parts for both EI and CI sources. An upgrade to the ChemStation software G1701EA (E.00.xx) is also required. This configuration is standard on the 5975C MSD ion source design.

High Ion Source Temperature Considerations

Tuning and Spectral Considerations

Tuning at higher source temperatures will result in a "tilt" of the EI calibrant, perfluorotributylamine (PFTBA), spectrum as shown in Figure 1. Notice that the relative amounts of 219 and 502 are lowered at the higher temperature versus the standard temperature. This should not be unexpected. Compound spectra may exhibit similar lowering of high-mass fragment ion intensities due to the higher thermal energy of the molecules. Many compounds that are known to be rather recalcitrant, like the polychlorinated biphenyls (PCBs), polynuclear aromatic hydrocarbons (PAHs), or other persistent organic contaminants/pollutants (POPs), should show little change in their mass spectra but many other compounds, such as some pesticides, should be expected to show a change. Because most libraries are acquired under lower source temperatures or by different sample introduction techniques than GC (for example, direct

Table 1. High Temperature Sources and Upgrade Parts

EI Ion source			
Standard EI Ion source complete, new	Ion Source High Cmpl, new EI Rep. Block	G3170-65750	
Standard EI Ion source complete, rebuilt	Ion Source High Cmpl, new EI Rep. Block	G3170-69750	
Inert EI Ion Source complete, new	Inert Ion Source High, new iEI Rep. Block	G3170-65760	
Inert EI Ion Source complete, rebuilt	Inert Ion Source High , rebuilt iEI Rep. Block	G3170-69760	
CI Ion Source			
CI Ion Source Complete, New	CI High Ion Source Complete	G3170-65403	
Source Upgrade Parts		**ROW**	**Japan**
Standard EI ion source	Anodized Standard EI Heater & Repeller Assembly	G3170-60172	G3170-60178
Inert EI ion source	Anodized Inert EI Heater & Repeller Assembly	G3170-60171	G3170-60179
CI ion source	CI Source Heater & Assembly	G3170-60415	G3170-60416

insertion probe), some loss in the spectral match quality to libraries may occur. These considerations should be kept in mind.

Choosing an Ion Source Temperature

A higher source temperature can offer advantages in analysis. If an optimal GC method has been achieved, examining it will reveal some logical limits to source temperature. For example, if the oven inlet is fairly cool (< 250 °C), a much higher source temperature may not provide much enhancement. In general, an ion source temperature about 25 °C or 50 °C below the elution temperature of the last component or the highest temperature of the GC oven program is worth exploring as a starting point.

To explore whether an increase in ion source temperature improves an analysis, a simple experiment can be conducted on the analytes of interest. Using a single GC method, run an analysis of a working standard at several source temperatures, such as 230 °C, 250 °C, 275 °C, 300 °C, 325 °C, and 350 °C and review the results from two perspectives. Examine the spectra to confirm that they remain useful (that is, there are no losses in signal for important higher mass confirming ions, no degradation in ratios, no increase in interfering ions, etc.) and then examine the signal intensities and their shape to see if the peak shapes have improved. As some examples, consider the following cases.

Organochlorine Pesticides

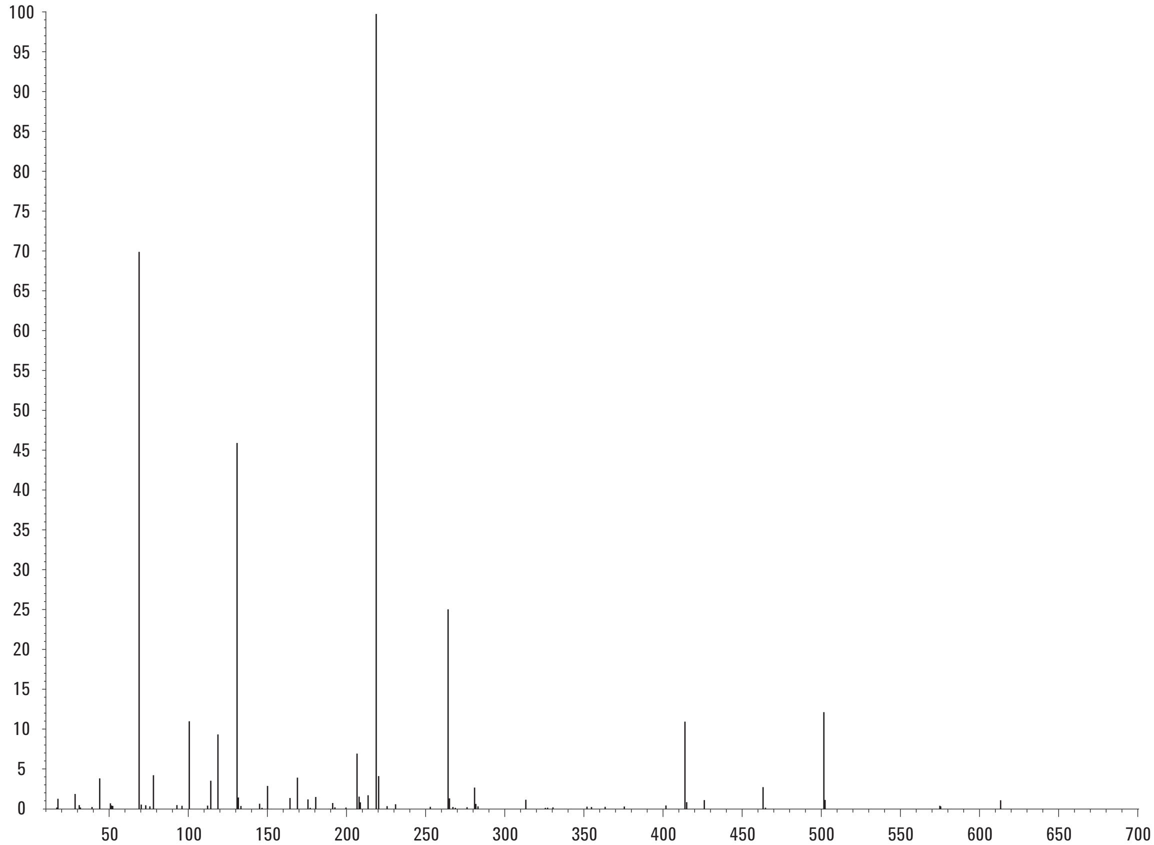
Figure 2 shows the overlaid reconstructed total ion chromatograms (RTICs) for scan acquisitions of several organochlorine pesticides at source temperatures from 230 °C to 350 °C. The upper panel shows the early eluting hexachlorocyclohexanes (HCCHs) show little effect in their total signal. However, the later eluting components of endosulfan sulfate, p,p'-DDT, and methoxychlor show an increase in their total response. This does not come for “free.” Even some of these environmentally recalcitrant and persistent compounds can show some relative loss of higher mass fragments in their spectra at higher temperatures. This means that comparisons must be made on the basis of target and qualifier ions. Also, the baseline is slightly higher due to the contribution of column bleed ions, which are enhanced at the higher source tempertures. Notice that source temperature does not affect all compounds to the same degree. The percent change in signal for p,p'-DDT versus endosulfan sulfate as a case in point.

Polynuclear Aromatic Hydrocarbons (PAHs)

Again, earlier eluting compounds show little positive effect at higher source temperatures but later eluting PAHs, such as those shown in Figure 3, show a marked enhancement. For these PAHs, there is little difference between 350 °C and 325 °C, which suggests that operating no higher than 325 °C will likely be optimal. Higher ring number PAHs like coronene (Figure 4) show a marked improvement in response and also in peak shape as less tailing is present at higher temperatures.

Analyzer Baking

Even if a higher source temperature is not applied in a method, it can be very useful for preparing an instrument for analysis. After source cleaning [2] and reinstallation, there will be a relatively high background of water, air, hydrocarbons, etc., which



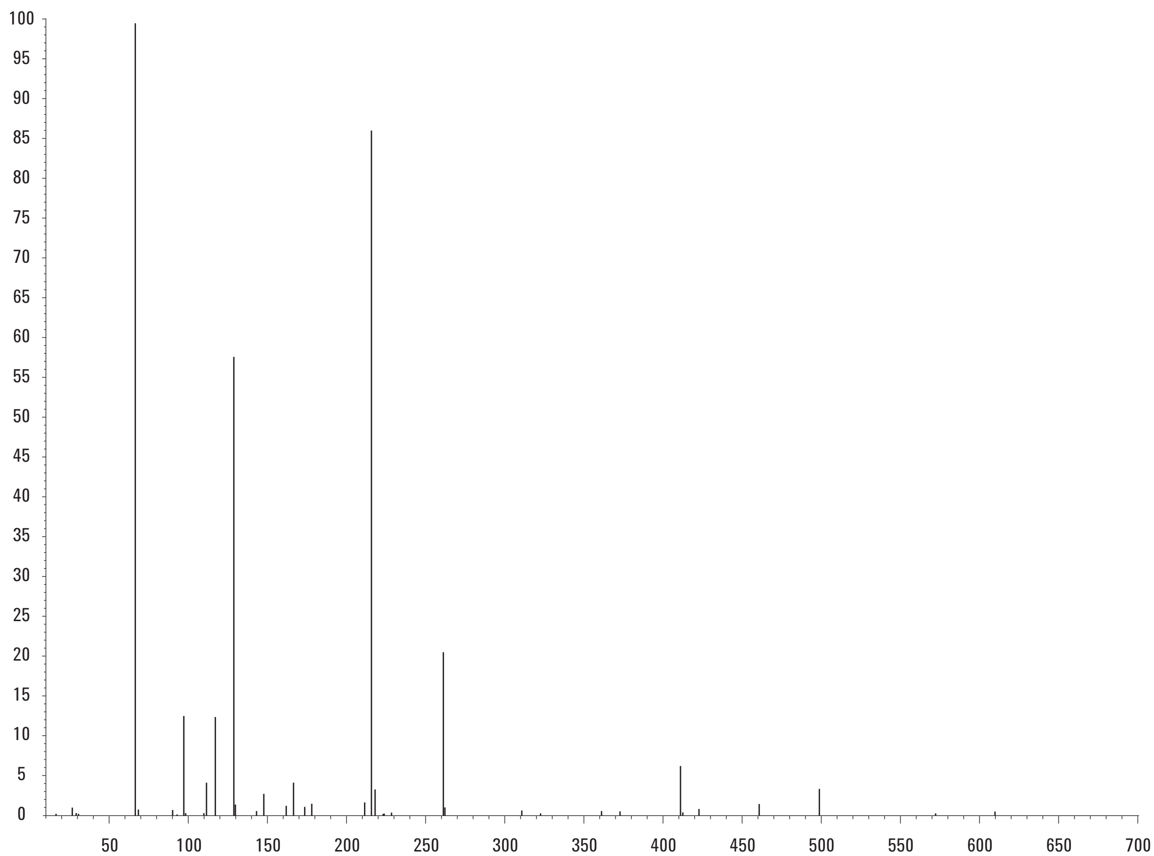


Figure 1. PFTBA spectra obtained in autotuning at 230 °C (upper) and 350 °C (lower) plotted as abundance vs *m/z*.

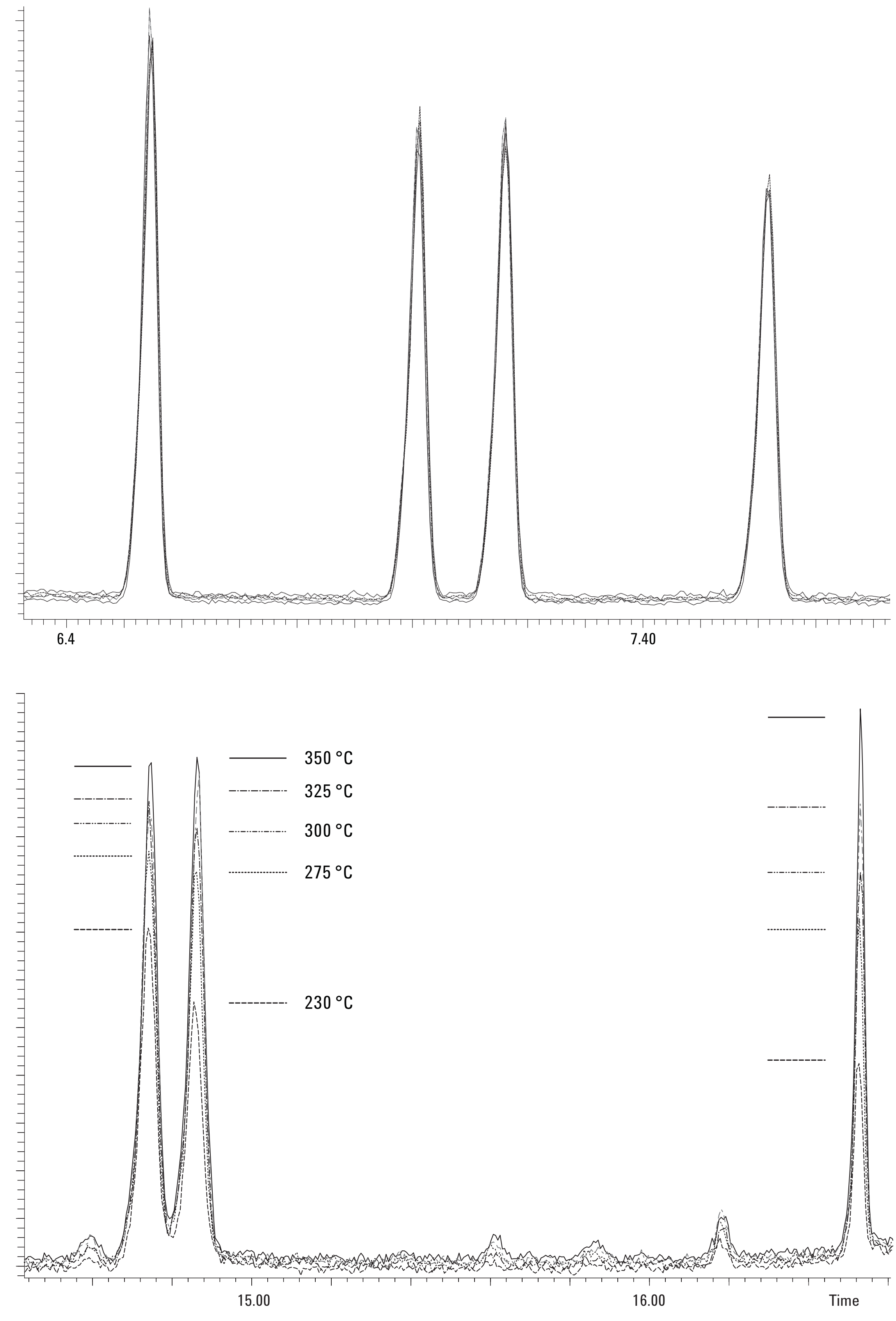


Figure 2. RTICs for selected OC pesticides at varying source temperatures: upper panel, the 4 HCCHSs; lower panel, Endosulfan Sulfate, p,p′-DDT, and Methoxychlor (as eluting from left to right).

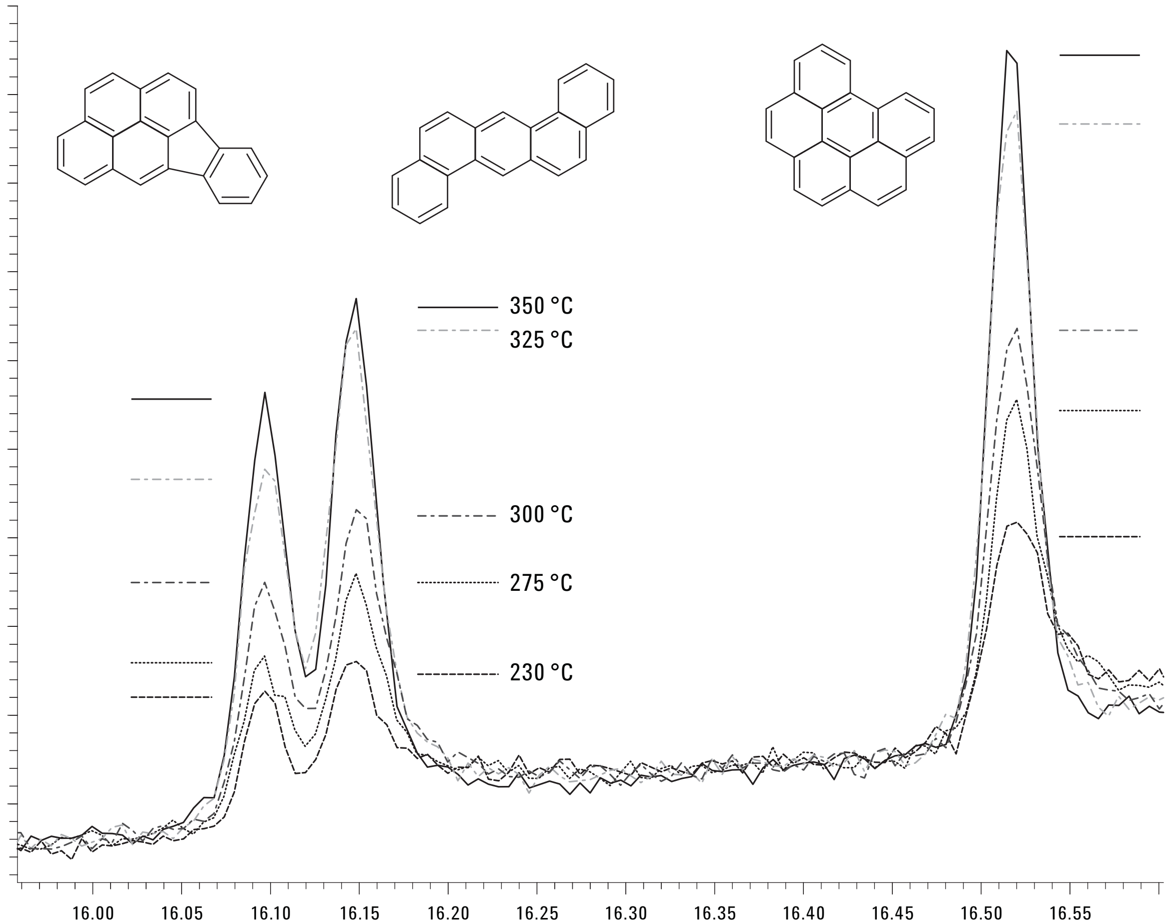


Figure 3. **RTICs for selected PAHs at varying source temperatures: indeno[1, 2, 3-cd] pyrene, dibenz[a,h]anthracene, and benzo[ghi]perylene.**

can be reduced by analyzer baking. However, it is critical that an analyzer should never be heated until it is established that there are *no* leaks. In other words, the power on temperatures should be set to 0 °C for both source and quadrupole. In this way if there is a leak, none of the analyzer components have been heated and, since the system zones are cool, it is easy to troubleshoot any leaks or rapidly vent the system if the need arises.

After it has been established that there are no leaks, there are two methods of baking the analyzer. The menu item *Bake out MSD* in the TUNE view under *Execute* allows the user to manually set the source and quad bake temperatures, the duration of the bake, and the final temperatures (Figure 5).

Another approach takes advantage of the utility to automatically bake the analyzer in a sequence. Figure 6 shows a sequence using the macro command BAKE. The command is called in the sequence as *BAKE SourceTemp, QuadTemp, Hours*. The working scenario is as follows:

After servicing and confirming that the MSD is leak-free, the CHECKOUT.Seq can be loaded and executed. This sequence automatically bakes the analyzer for a period, then tunes the MSD, then runs a series of standards to establish that the instrument is performing within the user's criteria. (A typical work practice is to service the GC and MSD at the end of a day and then perform this checkout for examination on arrival in the laboratory the next day).

The recommended baking temperatures are 200 °C for the quadrupole and 300 °C for the source, with an option being to heat to 350 °C. Baking times depend on the "history" of the MSD. If the system has been open to the air for quite some time, baking should be 6 hours or longer and at the higher 350 °C temperature.

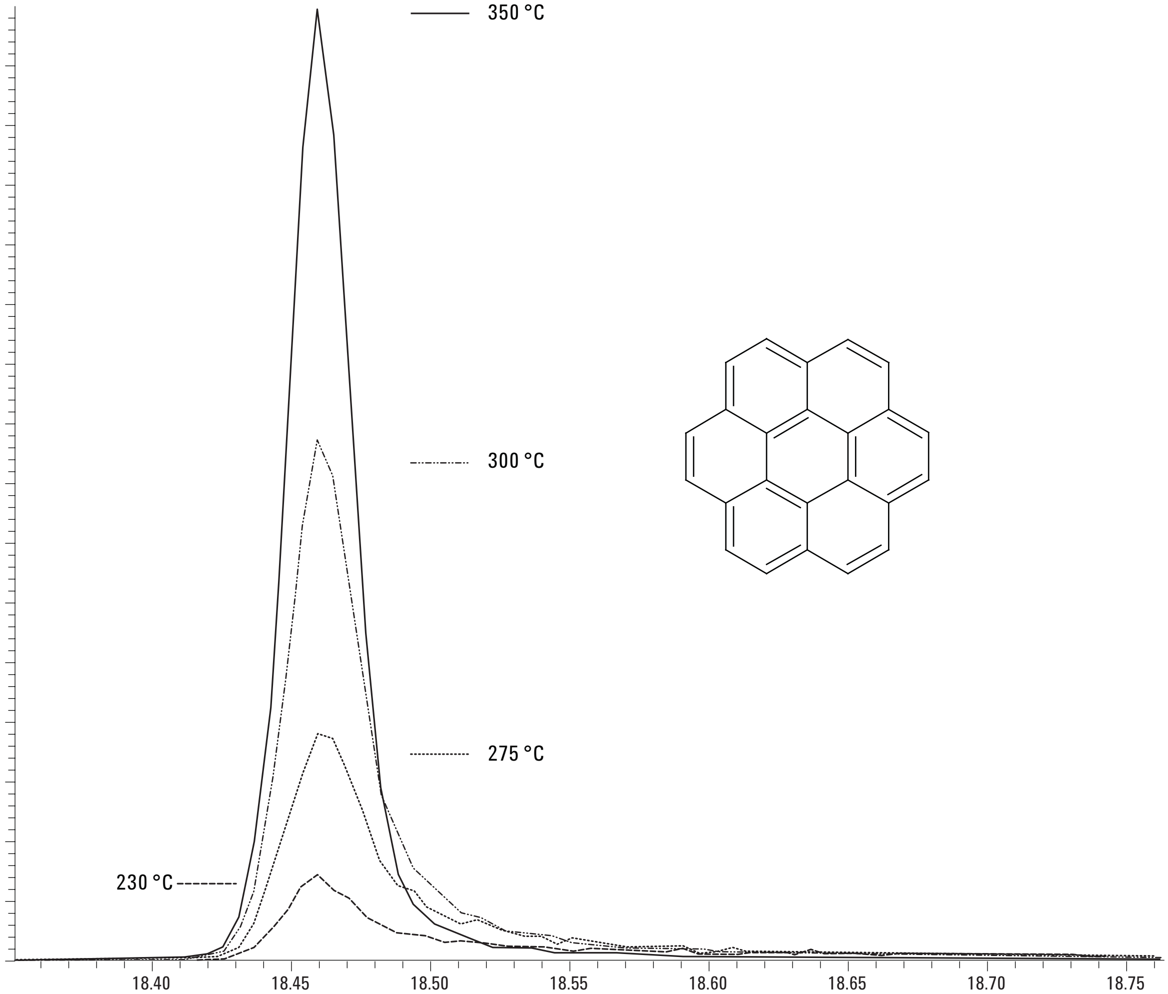


Figure 4. REICs for coronene at varying source temperatures.

Operational Guidance Summary

A few rules are necessary for optimal long-term performance.

1. Only properly configured sources can be brought to higher temperatures. The 5975C MSD ion source is already properly configured.

   Both EI and CI sources must consist of the specified components to allow operation beyond 300 °C.

2. Operate at the *lowest required* source temperature.

   "Required" means that temperature consistent with the demands of the application. Continual operation at extreme temperatures increases wear on the source components and leads to a shorter source life. This is an unavoidable fact.

3. Extreme care *must* be taken when source temperatures are raised to 250 °C or higher.

   This means that leaks *must* be carefully avoided as they will at the least "activate" the source or possibly irreversibly ruin a source or analyzer. The most common leak is encountered at the GC-MSD transfer line. The SilTite metal ferrules are slightly more difficult to use but have the advantage of not developing a leak as the transferline temperature is cycled or is simply raised. The SilTite ferrules should be considered mandatory for higher temperature operation of the MSD and GC.

4. Analyzer baking is *not* a method for cleaning a "dirty" source.

   When a source at its operating temperature has become "fouled," it should be removed and cleaned. Analyzer baking can then remove hydrocarbons, etc., and provide a lower background. The Agilent ion source design is very rugged and robust, and when it requires cleaning it is because a large amount of nonvolatile material has been entrained. At this stage the most cost- and time-effective procedure is to mechanically clean the source [2]; a rapid process of only about 30 minutes for all lens components. After reinstallation, baking can rapidly condition the instrument for use.

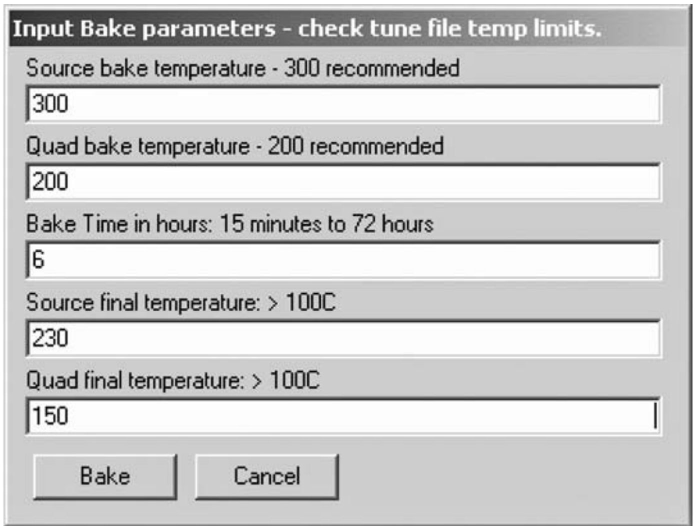
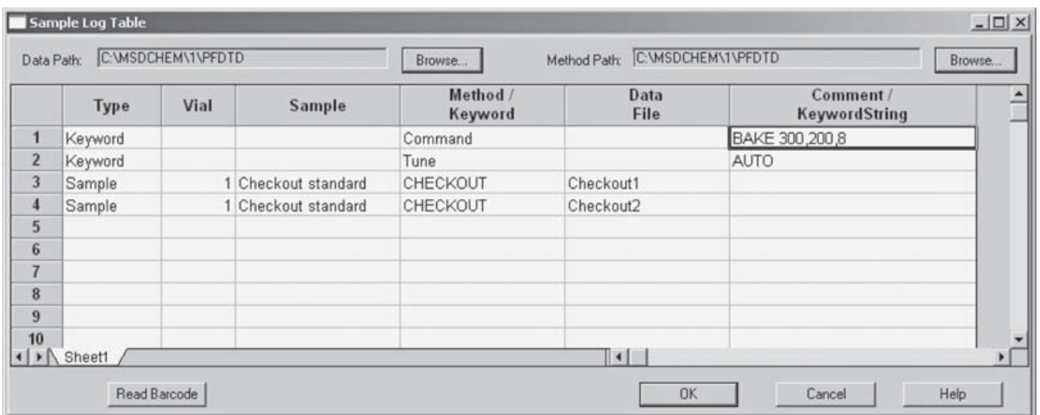


Figure 5. Manual command for bake of the MSD.



	Type	Vial	Sample	Method / Keyword	Data File	Comment / KeywordString
1	Keyword			Command		BAKE 300,200,8
2	Keyword			Tune		AUTO
3	Sample	1	Checkout standard	CHECKOUT	Checkout1	
4	Sample	1	Checkout standard	CHECKOUT	Checkout2	
5						
6						
7						
8						
9						
10						

Line	1.	Calls Bake Macro Bake *SourceTemp,QuadTemp,Hours* This example sets source to 300 and quad to 200 for 8 hours
	2.	Runs AUTOTune
	3. & 4.	Checks Standards using the method CHECKOUT.M

Figure 6. Automated analyzer baking in a sequence.

5. The recommended quadrupole operating temperature is 150 °C.

   During acquisition in electron impact (EI) or chemical ionization (CI) modes, the optimum and recommended quadrupole temperature is 150 °C. This temperature has been proven to provide the most robust operation and greatest mass axis and peak stability, even in the face of very “dirty” or complex samples with many high-boiling components. Certain customer analyses that encounter very dirty samples, or applications that use the high-temperature columns and have analytes eluting beyond 300 °C, will find the baking of the analyzer (quadrupole to 200 °C) helpful in maintaining the system.

The advantage of a heated and bakeable quadrupole can not be overemphasized. The monolithic quartz quadrupole has a prodigious lifetime and has proven to be rugged, robust, and stable in a tremendous range of difficult and demanding matrices. The heated quadrupole continually maintains its surface and rapidly lowers the background within the analyzer, which is critical in all MS modes of operation, but especially in NCI.

Frequently Asked Questions

Why not just run at the source maximum all the time?

The source temperature should be commensurate with the overall method and the nature of the compounds. If the compounds can survive higher injection port temperatures and if they are later eluting components, then higher source temperatures are worth exploring. But higher source temperatures come with the price of shorter source lifetimes.

I get transfer line leaks occasionally. How do I prevent this?

Cycling the transfer line between venting (cold) and operating (> 250 °C) temperatures, ramping the GC oven over large temperature ranges (especially to temperatures beyond 280 °C), and similar scenarios makes the polymeric ferrules shrink, begin to leak, and require retightening. If higher source temperatures are going to be explored, replacing the Vespel/graphite transfer line ferrule with the SilTite fittings is the best practice for avoiding leaks.

I am preparing to explore higher source temperatures. How should I configure my system?

Check that the injector temperature, oven temperature program, and column operating temperature range are all consistent with the higher temperature. Use the SilTite ferrules at the transfer line to minimize the opportunity for developing leaks.

Check that the source has been properly configured and set the new temperature limits in the *MS Temps* panel.

Before raising the source temperature, check the air and water background of the instrument. Check this frequently even if you do not retune the instrument.

References

Refer to the Agilent Web site: http://www.chem.agilent.com/, Literature Library, and select *Online Literature*.

1. C. Thomson and H. Prest, “The 5973N Inert MSD: Using Higher Ion Source Temperatures,” technical overview 5989-0678EN.
2. C. Thomson, M. Ruemler, M. Freed, and H. Prest, “MSD EI and CI Source Cleaning and Installation,” technical overview 5989-5974EN.

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