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Implementation of Ammonia Reagent Gas for Chemical Ionization on 5973 MSDs

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Introduction

Ammonia is a particularly useful reagent gas for positive and negative chemical ionization. As a reagent gas for positive chemical ionization (PCI), ammonia has a very high proton affinity (204 kcal/mole) and produces comparatively little fragmentation for most molecules. As a buffer gas in electron capture negative ion chemical ionization (ECNI or NCI), ammonia has approximately seven times the thermalizing power of methane. This technical note describes the operating procedures for using ammonia as a CI gas on the 5973 platform. Both performance and hardware will suffer if these procedures are not implemented. This note does not replace information in the hardware manual which should be thoroughly read and understood before operating the MSD.

Reagent Gas Purity

No suitable gas filters are available for use with ammonia, as there are for methane and isobutane. It is therefore imperative to use ammonia of the highest purity available, certainly no less than 99.998% pure. Water and oxygen are the most common and critical contaminants and should be present at no more than 5 ppm. Water present in the methane reagent gas used for tuning will result in split and broadened peaks; it can be detected as a high 19/29 ratio in the tune report. The presence of water in ammonia will result in very short filament lifetimes. The rate of ammonia use by the Agilent 5973N MSD is very low, approximately 1 ml/min, and investing in the more expensive gas will be more cost-effective in the long run.

Ammonia Gas Connection

Ammonia requires a special corrosion-resistant regulator and a dual-stage regulator is recommended. These regulators have relatively limited lifetimes, greatly shortened by exposure to water, but improved by using high-purity ammonia. The other primary exposure to water occurs during cylinder switching. Some users purge the regulator with dry nitrogen to exclude contact with moist air and help extend the regulator's lifetime.

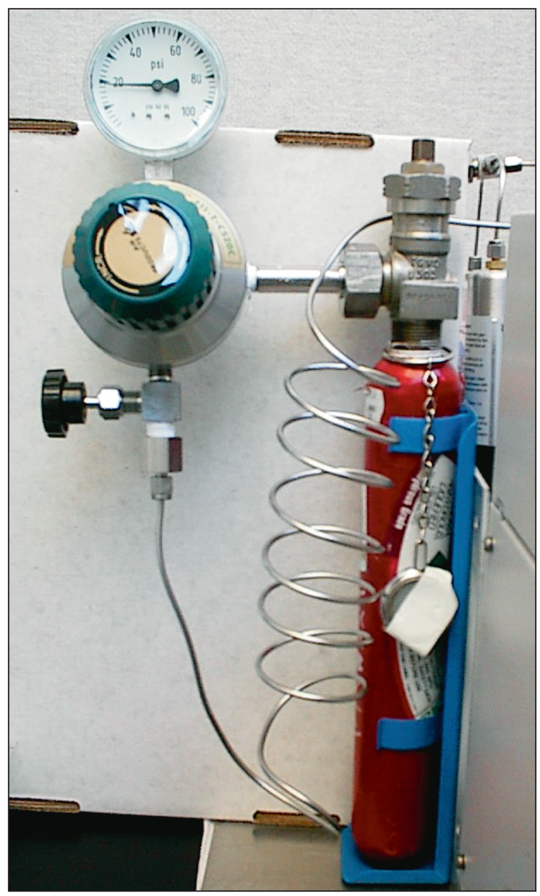

Figure 1. Example of coil for vaporization of ammonia reagent gas. Note that, typically, ammonia in lecture bottles is of poor quality and should be avoided.

[1] Do not use copper tubing or brass fittings for ammonia connections.

Because ammonia is a liquid in the cylinder, the regulator can actually deliver liquid ammonia to the system. The cylinder should be below the reagent gas Mass Flow Controller (MFC) to avoid siphoning liquid ammonia into the manifold. A coil of stainless-steel tubing[1] that runs vertically to the fitting on the back of the MFC enhances the opportunity for ammonia vaporization before introduction to the manifold (see Figure 1). The pressure of the regulator should be set to less than 20 psi.

If vaporization of the ammonia is incomplete, "bubbles" of liquid ammonia will produce "negative peaks" in the data baseline (Figure 2). These discontinuities in reagent flow will compromise the data.

Operation with Ammonia

To minimize usage of ammonia and its throughput to the mechanical pump, the reagent should be shut off at the end of a sequence. A macro[2] allows the user to turn off the CI gas automatically at the end of a sequence or method by invoking the command *cigasoff* as a post-run macro (Figure 3). Although the user can shut off the reagent gas automatically, the converse of automatically turning on the CI gas is not allowed for obvious reasons. (To obtain a copy of the necessary files, please visit to the Agilent web site under "Contributed Software": www.chem.agilent.com/cag/servsup/usersoft/main.html.) The G1701CA MSD Chemstation Software includes the *cigasoff* command that can be invoked at the end of a sequence or method.

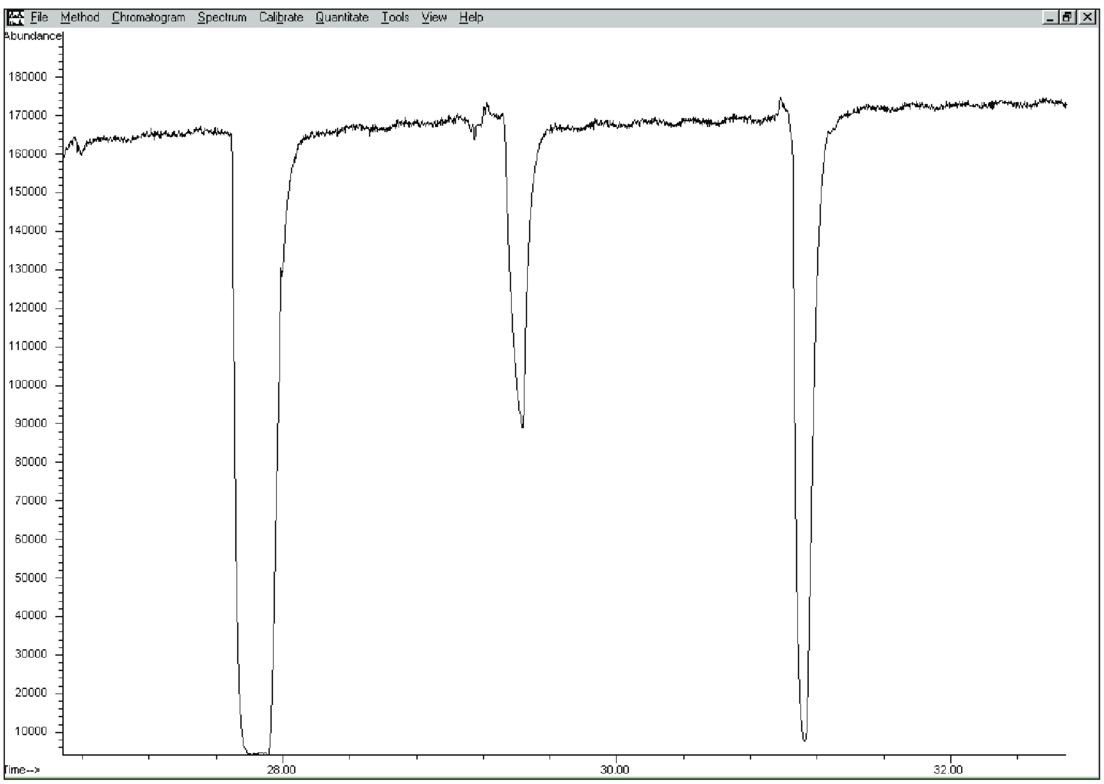

Figure 2. The effect of incomplete vaporization of the liquid ammonia prior to the ion source. The "negative peaks" result from bubbles of liquid ammonia and indicate that either siphoning is taking place or that an insufficient length of vertical coiled tubing has been used to connect the reagent, or both.

[2] This macro is available as a patch for the G1701BA software which must be version B.01.00 with the latest update present.

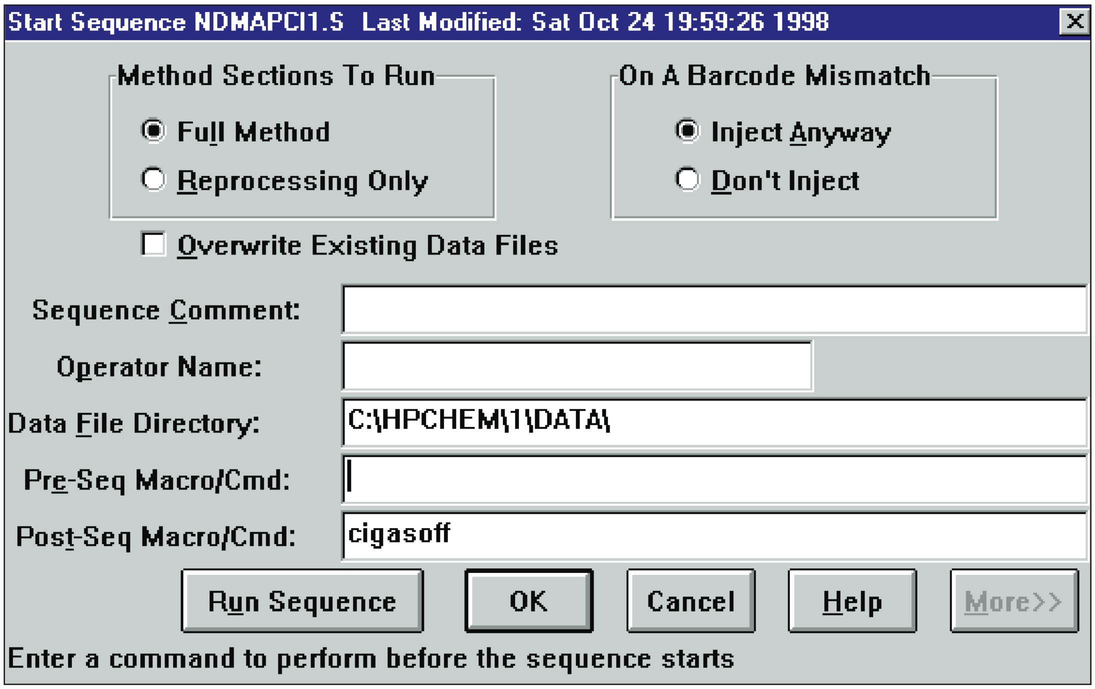

Figure 3. Implementing the *cigasoff* command at the end of a sequence in the BA version of software. The letters *cigasoff* are simply inserted on the Post-Seq Macro/Cmd line, which is displayed by clicking on the MORE button in the sequence panel.

Mechanical Pump Operation and Maintenance.

Ammonia gas reacts with water in the oil to raise the pH and attack the pump seals. To remove ammonia from the pump oil, it is necessary to ballast the pump for 30 minutes every day after using ammonia gas. The ballasting procedure, accomplished by rotating the knob on the top of the pump counter-clockwise at least two turns, increases the amount of air bubbling through the pump oil to flush out volatile components such as water, ammonia and solvents. This procedure should be executed only when the system is not operating in CI or during downtime, because the overall system pressure will rise slightly. (The 30-minute ballast time is an approximation. To optimize the ballasting time required for your application, the user can test the pH of the pump oil by dipping a strip of pH paper in the oil. A neutral range is normal and indicates when the dissolved ammonia and water is removed.) All necessary safety precautions and considerations need to be followed.

Automatic Ballasting of Edwards E2M1.5 Pumps

If there is a period of the day that the instrument is consistently inactive, then implementing automatic ballasting may be convenient. Edwards supplies an electrically operated gas ballast valve (Edwards Part Number EBV20) that can be connected to a common household timer. The part may be found by searching for the Part Number at the Edwards Web site at www.boc.com/evt/index.htm. However, it remains imperative that the ballasting does not take place during CI operation.

Pump Oil Change Frequency

The pump oil must be changed at least every three months, preferably more frequently. Whenever there is a significant change in oil color, the oil must be changed. Frequent oil changes are a relatively inexpensive insurance for good pump operation and lifetime. The CA version of the ChemStation software provides maintenance period reminders to notify the user automatically.

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Summary

Key aspects of successful operation with ammonia reagent gas:

- High-purity gas (99.998% or greater)
- a vaporizing connection to the CI reagent manifold
- daily ballasting of the mechanical pump for 30 minutes after CI operation
- frequent mechanical-pump oil changes
- utilizing the software shutoff of the ammonia following unattended operation

Attending to each of these issues will ensure long-term success with ammonia reagent gas.

Acknowledgments

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