Implementation of Ammonia Reagent Gas for Chemical Ionization on the 5975 series MSDs

Application Note

Chemical Analysis

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Abstract

This note describes the operating procedures for using ammonia as a CI gas on 5975 inert MSD platforms.

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 5975 inert MSD platforms. 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 5975 inert 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, low-flow 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.

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Because ammonia is a liquid in the cylinder, the regulator can actually deliver liquid ammonia to the system. The cylinder and regulator MUST be located below the CI reagent gas Mass Flow Controller (MFC) to avoid siphoning liquid ammonia into the manifold. From the regulator to the rear of the CI manifold on the 5975 inert MSD, stainless steel tubing must be used with stainless steel fittings. A coil of stainless-steel tubing 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 <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. 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 was used to connect the reagent, or both.

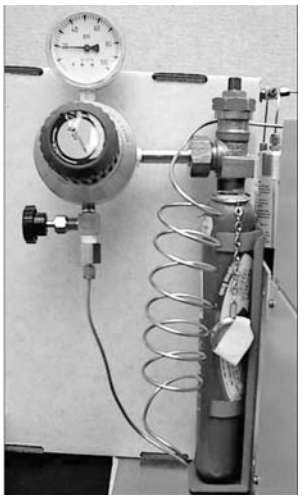

Figure 1. Example of coil for vaporization of ammonia reagent gas. Note that, typically, ammonia in lecture bottles is of poor quality and should be avoided.

Operation with Ammonia

When operating in CI mode, the gas setting is automatically implemented with the CI acquisition method which allows the user to alternate between positive and negative CI methods with either of two CI gases in a single sequence. We recommend the user consult Agilent publication 5989-4347 [1] describing new chemical ionization functionality.

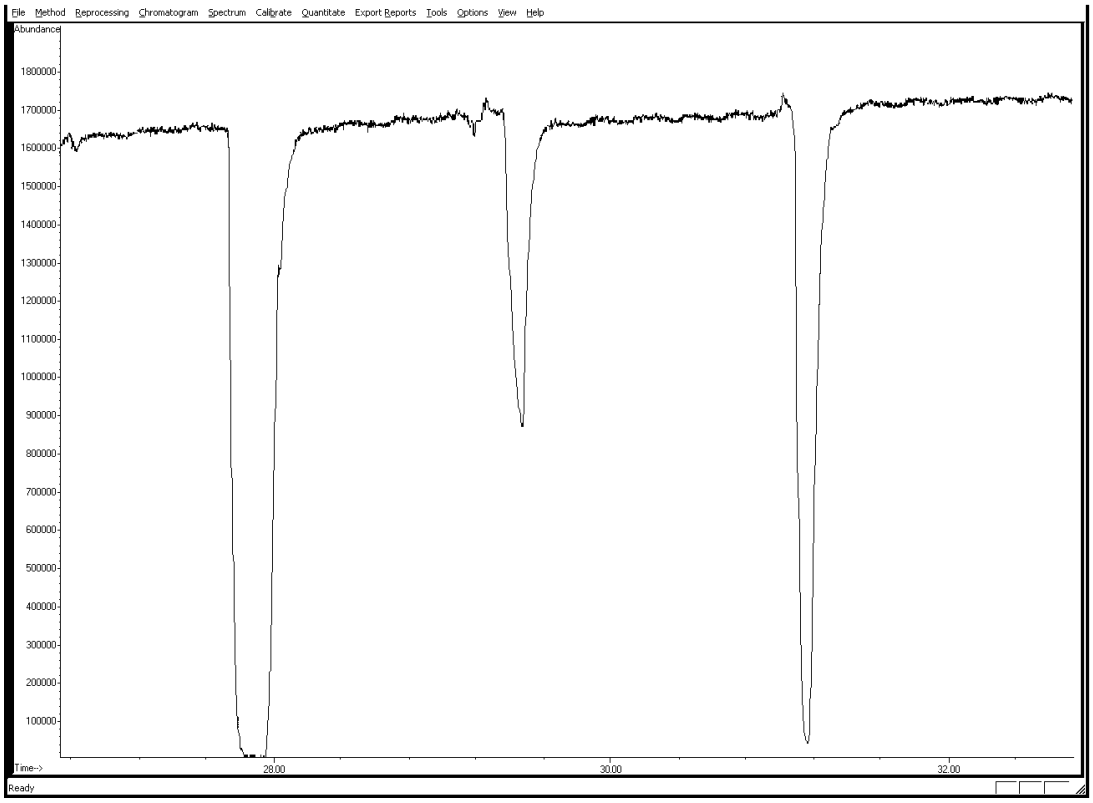

Figure 2. The effect of incomplete vaporization of the liquid ammonia prior to the ion source.

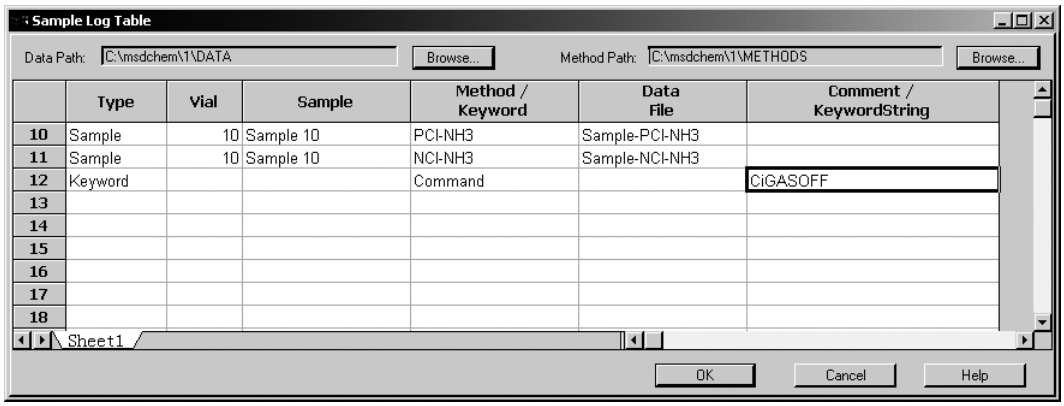

Figure 3. Implementing the cigasoff command at the end of a sequence.

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 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 macro command at the last line of the sequence (Figure 3).

Mechanical Pump Operation and Maintenance

Typically “condensables” that are introduced to the mechanical pump are removed by applying “gas ballasting” of the mechanical pump. This has always been a good approach when pumping a lot of water such as after a pumpdown of the MSD. However, ammonia gas reacts with water in the oil to raise the pH and attack the pump seals. Introducing more water by ballasting would only accelerate this destructive process. The new mechanical pumps handle condensables better by operating at higher temperatures and therefore no gas ballasting should be applied when the system is operating or has been operating with ammonia.

Pump Oil Change Frequency

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 ChemStation software provides maintenance period reminders to notify the user automatically.

Hi Reliability Ammonia Compatible Oil-Free Pump

Agilent part number G3170-80032, is the recommended oil-free (dry) pump alternative for MSD systems running routine ammonia applications. This pump has internal components designed and proven to be ammonia tolerant. Oil-free operation eliminates oil maintenance issues and the attendant risk of oil back streaming into the MSD analyzer. Ballasting is also NOT recommended with the G3170-80032. The recommended service/replacement interval is 12 months. The refurbished replacement pump part number is Agilent part number G3170-89032.

Summary

Key aspects of successful operation with ammonia reagent gas:

- High-purity gas (99.998% or greater)
- A connection to the CI reagent manifold that aids vaporizing of the ammonia reagent
- Frequent mechanical-pump oil changes
- No oil change is necessary with the oil-free, ammonia compatible G3170-80032
- Using the software for automatic shutoff of the ammonia following a completed sequence

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

Reference

1. “5975 inert MSD - Benefits of Enhancements in Chemical Ionization Operation”, Agilent Technologies, publication 5989-4347EN, www.Agilent.com/chem

For More Information

For more information on our products and services, visit our Web site at www.agilent.com/chem.

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