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ELIMINATION OF "MEMORY" FROM SAMPLE HANDLING AND INLET SYSTEM OF A MASS SPECTROMETER

BACKGROUND OF THE INVENTION

1. Field of the Invention:

The present invention relates to the use of mass spectrometers. In particular, the present invention relates to the phenomenon of "memory", the effect on subsequent sample analysis caused by the lingering of a previous sample in sample handling and inlet system of a mass spectrometer. The United States Government has rights in this invention pursuant to Contract No. DE-AC09-89SR18035 between the U.S. Department of Energy and Westinghouse Savannah River Company.

2. Discussion of Background:

Most isotopic analysis is currently performed using mass spectrometry. A sample is processed in a sample handling and inlet system and is then injected into a vacuum chamber, where its atoms are ionized and accelerated in one direction, forming a beam. After moving through either a magnetic field or a double-focusing combination electric and magnetic field, the ions in the beam are sorted out according to their mass, charge and velocity, with some of them being steered into a detector. By varying field strengths, it is possible to steer first one and then another

part of the beam into the detector according to mass, and measure the relative number of ions of each mass. After analysis, all traces of the sample should be removed so that there is no residual matter to interact with later samples and cause inaccuracies in the test results.

Mass spectrometers typically cost several hundreds of thousands of dollars. Furthermore, a single mass spectrometer must often be shared among several different experiments or sampling points. Either with a shared mass spectrometer or with one used to analyze a series of samples, there is a problem associated with mass spectrometer use called "memory." Memory is a phenomenon in which there is an effect of a previous sample on subsequent ones as a result of lingering traces of the previous sample (or samples) analyzed after it has supposedly been purged from the sample inlet system and the spectrometer. This problem is especially great following analysis of samples of krypton and xenon gas because of the tendency of these gases to adhere tenaciously to the walls of the sample handling system and the spectrometer.

It is known that gases can be used for flushing gas chromatographs. See U.S. 4,873,058 for a description of this method by Arnold, et al. However, they do not identify a particular gas. In a quite different type of process, supercritical CO_2 is known as a solvent for extraction of derivatives, as described in U.S. 4,597,943 issued to Sugiyama, et al.

However, there remains a need for a technique for eliminating or substantially reducing, the problem of memory in mass spectrometers.

SUMMARY OF THE INVENTION

According to its major aspects and broadly stated, the present invention is a method for eliminating mass spectrometer "memory" by the flushing of the spectrometer's sample handling and inlet system with a supercritical fluid, in particular supercritical carbon dioxide. By flushing with a supercritical fluid, which is typically an energetic solvent, traces of samples will be removed from the interior of the sample handling system as a result of molecular collisions and can be vented from the system.

Use of supercritical CO_2 is especially preferred for flushing. Not only is supercritical CO_2 especially energetic and an effective solvent, but it does not itself readily cling to the walls and other interior surfaces of the mass sample handling and inlet system.

In particular in the case of analysis of krypton and xenon, which have a more pronounced lingering effect than other materials, backflushing with supercritical fluids such as supercritical CO_2 is important to prevent errors in subsequent analyses. Also, to the extent that any traces of the CO_2 remain in the sample handling and inlet system, they will not affect subsequent analyses of these two gases because their masses

and that of CO_2 are sufficiently different and the mass spectrometer can easily distinguish them.

Other features and advantages of the present invention will be apparent to those skilled in the art from a careful reading of the Detailed Description of a Preferred Embodiment presented below.

DETAILED DESCRIPTION OF A PREFERRED EMBODIMENT

The memory effect of samples, especially of those samples that tend to persist and to adhere to the interior surfaces of the sample handling and inlet system, can be removed by flushing the interior of the entire sample preparation and inlet system of a mass spectrometer with a supercritical fluid and venting the system. Supercritical fluids are composed of highly energetic molecules which collide with the atoms of the sample to remove them from the walls of the spectrometer.

Supercritical CO_2 is especially effective as a backflushing supercritical fluid. Its molecules are energetic, do not damage the system, and do not themselves adhere to the surfaces of the sample handling and inlet system interior. It is also effective in removing gases such as krypton and xenon which adhere tenaciously to such surfaces. Furthermore, to the extent that some CO_2 remains after venting, it will not interfere or compromise the analysis of subsequent samples of these gases because their masses are much different and the mass

spectrometer can easily distinguish them. It will be apparent to those skilled in the art that many changes and substitutions can be made to the preferred embodiment herein described without departing from the spirit and scope of the present invention as defined by the appended claims.

ABSTRACT OF THE DISCLOSURE

A method for preparing the sample handling and inlet system of a mass spectrometer for analysis of a subsequent sample following analysis of a previous sample comprising the flushing of the system interior with supercritical CO_2 and venting the interior. The method eliminates the effect of system "memory" on the subsequent analysis, especially following persistent samples such as xenon and krypton.

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