

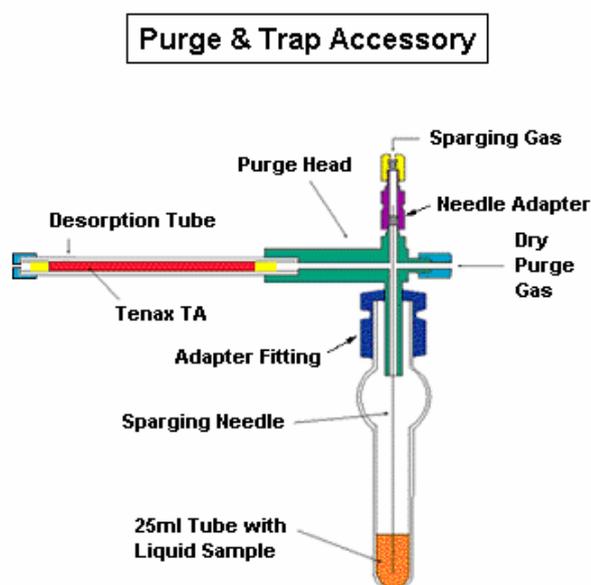
Measurement of Trace Sulfur Compounds in Isopropyl Alcohol by Purge & Trap / Gas Chromatography / Mass Spectrometry

Introduction

Many consumer products containing isopropyl alcohol must avoid having an objectionable odor. Producers of isopropyl alcohol refine their product to remove odor causing contaminants such as volatile organic sulfur compounds that may be present in raw material feed stocks. Purge & Trap / Gas Chromatography / Mass Spectrometry is an excellent technique to identify and measure trace sulfur compounds in isopropyl alcohol.

Purge & Trap Sample Preparation

A 2.5ml portion of isopropyl alcohol sample is diluted to 25ml with ultrapure water and acidified with 1:1 hydrochloric acid. This solution is transferred to the liquid sample tube of a Purge & Trap Accessory (pictured below) and sparged with dry nitrogen gas to remove volatile sulfur compounds. These compounds are carried by the nitrogen gas stream through a desorption tube containing Tenax TA adsorbent media which traps and holds the sulfur compounds.



The green trace is a typical isopropyl alcohol sample and the purple trace is isopropyl alcohol spiked with sulfur compounds in the 80-100ppb range.

Figure 1

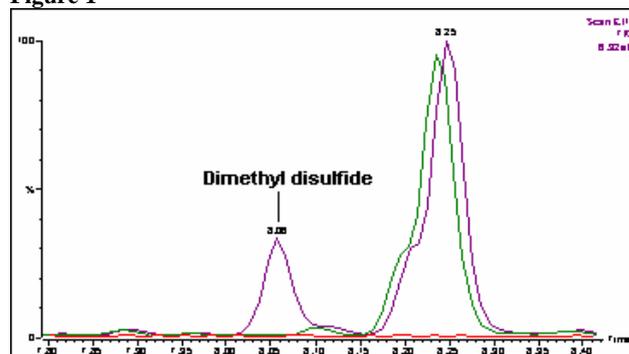
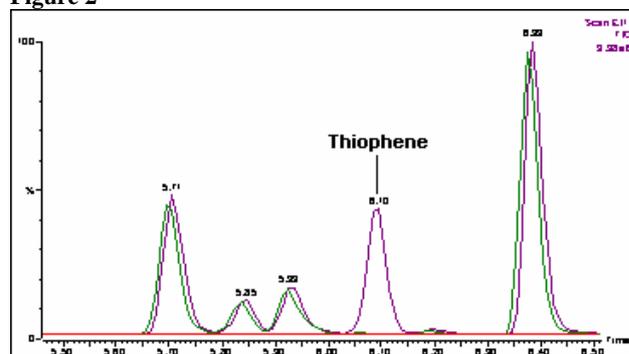


Figure 2



GC/MS Analysis

Sulfur compounds are desorbed from the Tenax TA media by heating the tube to 250C while flowing ultrapure helium gas through the tube. The sulfur compounds are concentrated in a cold trap and then injected into a gas chromatograph where they are separated into individual compounds. As each compound exits the chromatographic column it enters a quadrupole mass spectrometer where its mass spectrum is obtained. The mass spectrum provides compound identification while the area under the chromatographic peak allows measurement of concentration.

Example Results

Total ion chromatograms showing separation of example sulfur compounds from other volatile organics present in a typical isopropyl alcohol sample are presented in Figures 1 and 2. The red trace represents an unused Tenax TA control.

Detection limits, in parts-per-billion (ppb), for several sulfur compounds are presented in Table 1. Dimethyl sulfide is obscured by isopropyl alcohol using this method, and can not be measured accurately at low levels.

Table 1

Component	Boiling Point (degC)	Retention Time (min)	Detection Limit (ppb)
Dimethyl sulfide	38	2.69	n/a
Ethyl methyl sulfide	66	4.71	5.0
Thiophene	84	6.10	1.5
Dimethyl disulfide	109	8.06	1.5
Diisopropyl sulfide	120	9.53	1.5
Di-n-propyl sulfide	142	11.83	1.0
Diethyl disulfide	151	12.49	1.0