## Introduction 5972 and 5973N

1(a) Quantitative analysis and characterization has been done using a mass spectrometric procedure: ATD-GC-MSD

1(b) Quantitative analysis and characterization has been done using a mass spectrometric procedure: $CS_2$-GC-MSD.

1(c) Quantitative analysis and characterization has been done using a mass spectrometric procedure: ATD-GC-MSD with cryogenic oven control.

1(d) Quantitative analysis and characterization has been done using a mass spectrometric procedure: $CS_2$-GC-MSD with cryogenic oven control.

## Multisorbent Tubes

2(a) The VOC MB glass sorbent tube dimensions are 10 X 140 mm, and the tube is packed with Carbotrap C, Carbopack B, Carboxen 569, Carbosieve S-III sorbents, or Carbotrap Y, Carbotrap X, Carboxen 569, Carbosieve S-III.

2(b) The VOC SS stainless steel sorbent tube dimensions are 6.3 X 140 mm, and the tube is packed with Carbotrap Y, Carbotrap X, Carboxen 569, Carbosieve S-III.

## ATD Preparation

3(a) Gas bags were sub-sampled onto Envirochem analytical C-tubes and/or injected directly through an empty tube onto the primary trap. The analytical tubes are 6mm outside diameter Envirochem multisorbent tubes packed with Carbotrap C, Carbopack B and Carbosieve S-III; or Carbotrap Y, Carbotrap X and Carobsieve S-III.

3(b) Exposed SS <u>field tubes</u> were returned to OSB Lab where they were processed for analysis. The MB tubes were desorbed and transferred at 280°C for 10 minutes onto C <u>analytical tubes</u> using the TDU system. The analytical tubes are 6 mm outside diameter Envirochem multisorbent tubes packed with Carbotrap C, Carbopack B and Carbosieve S-III; or Carbotrap Y, Carbotrap X and Carobsieve S-III.

3(c) Exposed MB <u>field tubes</u> were returned to OSB Lab where they were processed for analysis. The MB tubes were desorbed and transferred at 280°C for 10 minutes onto C <u>analytical tubes</u> using the TDU system. The analytical tubes are 6 mm outside diameter Envirochem multisorbent tubes packed with Carbotrap C, Carbopack B and Carbosieve S-III; or

Carbotrap Y, Carbotrap X and Carbosieve S-III.

4 Sample splitting was carried out at this stage using critical capillary orifices.

5(a) Each sample was spiked with Toluene-$d_8$ the primary internal standard. Currently four surrogates are employed: 1,1,1,2-Tetrafluoroethane, 1,4-Difluorobenzene, Toluene-$d_8$, and Dodecane-$d_{26}$ which cover the entire chromatogram period.

5(b) Each sample was spiked with Toluene-$d_8$ the primary internal standard. Currently four surrogates are employed: 1,1,1,2-Tetrafluoroethane, 1,4-Difluorobenzene, Toluene-$d_8$, and Hexachloro-1,3-butadiene which cover the entire chromatogram period.

**ATD Analytical Method 5972 or 5973N**

6(a) Analysis was carried out on the <u>Analytical VOC System</u> (AVS) which consists of an Envirochem 810A Concentrating Capillary Inletting System coupled to a Hewlett Packard HP 5890E Series II Plus Gas Chromatograph (GC) and HP 5972A Mass Selective Detector (MSD) via a 260ºC heated nickel steel transfer tube (line). The C <u>analytical tubes</u> were desorbed at 260ºC on the Envirochem Unit sequentially onto a primary and then secondary internal analytical trap. The secondary smaller focusing trap then automatically injected, by means of thermal desorption, any VOCs into the GC which is equipped with a 60m HP-624 capillary column via the transfer line. VOCs were detected by the MSD in SCAN mode. The MSD was interfaced to an HP ChemStation Data System G1701BA.B.02.00 with a National Institute for Science and Technology (NIST/EPA/NIH) MS compound library, with NIST02/HP-Agilent combination mass spectral search programs obtained from ChemSW. The above procedure is similar to EPA SW846 Method 8260B, NIOSH Method 2549 and equivalent to EPA compendium Method TO-17. Calibration is achieved using individual chemical response factors (TRFs) normalized against Toluene-$d_8$.

6(b) Analysis was carried out on the <u>Analytical VOC System</u> (AVS) which consists of an Envirochem 810A Concentrating Capillary Inletting System coupled to an Agilent Technologies 6890 Series Plus Gas Chromatograph (GC) and HP 5973N Mass Selective Detector (MSD) model G2578A Turbo via a 260ºC heated nickel steel transfer tube (line). The C <u>analytical tubes</u> were desorbed at 260ºC on the Envirochem Unit sequentially onto a primary and then secondary internal analytical trap. The secondary smaller focusing trap then automatically injected, by means of thermal desorption, any VOCs into the GC which is equipped with a 60m DB-624 capillary column via the transfer line. VOCs were detected by the MSD in SCAN mode. The MSD was interfaced to a ChemStation Data System G1701CA-C.00.00 with a National Institute for Science and Technology (NIST/EPA/NIH) MS compound library, with NIST02/HP-Agilent combination mass spectral search programs obtained from ChemSW. The above procedure is similar to EPA SW846 Method 8260B, NIOSH Method 2549 and equivalent to EPA compendium Method TO-17. Calibration is achieved using individual chemical response factors (TRFs) normalized against Toluene-$d_8$.

**CS2 Preparation**

3(d) Tubes containing 600mg charcoal were transferred to 4mL vials and extracted over 24 hours with 2mL Carbon Disulphide spiked with a known concentration of a mixture of 1,4-Difluorobenzene, Toluene-$d_8$ and 1,2,4-Trichlorobenzene referred to as surrogates.

3(e) Tubes containing 600mg charcoal were transferred to 7mL vials and extracted over 24 hours with 4mL Carbon Disulphide spiked with a known concentration of a mixture of 1,4-Difluorobenzene, Toluene-$d_8$ and 1,2,4-Trichlorobenzene referred to as surrogates.

3(f) Tubes containing 600mg charcoal were transferred to 7mL vials and extracted over 24 hours with 4mL Carbon Disulphide spiked with a known concentration of Toluene-$d_8$ or a mixture of 1,4-Difluorobenzene, Toluene-$d_8$ and 1,2,4-Trichlorobenzene referred to as surrogates.

3(g) Tubes containing 160mg charcoal were transferred to 2mL vials and extracted over 24 hours with 1mL Carbon Disulphide spiked with known concentration of 1,4- Difluorobenzene, Toluene-$d_8$ and 1,2,4-Trichlorobenzene referred to as surrogates.

**CS2 Analytical Method 5972 or 5973N**

6(c) Analysis was carried out on the Analytical VOC System (AVS) which consists of a Hewlett Packard HP5890E Series II Plus Gas Chromatograph (GC) and HP 5972A Mass Selective Detector (MSD). Analysis was carried out by direct injection chromatography onto a 60m SupelcoWax – 10 capillary column. VOCs were detected by the MSD in SCAN mode. The

MSD was interfaced to an HP ChemStation Data System G1701BA-B.02.00 with a National Institute for Science and Technology (NIST/EPA/NIH) MS compound library, with NIST02/HP-Agilent combination mass spectral search programs obtained from ChemSW. The above procedure is similar to NIOSH Methods 1450, 1500, 1501 and 1003. Calibration is achieved using individual chemical response factors (TRFs) normalized against Toluene-$d_8$ and determined from extraction experiments using neat chemicals (not dissolved in $CS_2$) spiked on charcoal under the same protocol conditions.

6(d) Analysis was carried out on the <u>Analytical VOC System</u> (AVS) which consists of an Agilent Technologies 6890 Series Plus Gas Chromatograph (GC) and 5973N Mass Selective Detector (MSD) model G2578A Turbo. Analysis was carried out by direct injection chromatography onto a 60m SupelcoWax – 10 capillary column. VOCs were detected by the MSD in SCAN mode. The MSD was interfaced to an Agilent ChemStation Data System G1701CA-C.00.00 with a National Institute for Science and Technology (NIST/EPA/NIH) MS compound library, with NIST02/HP-Agilent combination mass spectral search programs obtained from ChemSW. The above procedure is similar to NIOSH Methods 1450, 1500, 1501 and 1003. Calibration is achieved using individual chemical response factors (TRFs) normalized against Toluene-$d_8$ and determined from extraction experiments using neat chemicals (not dissolved in $CS_2$) spiked on charcoal under the same protocol conditions.