3M Medical Department
Medicine
Health Physics
Industrial Hygiene
Toxicology

3M Center Bldg. 220-2E-02
St. Paul, MN 55144-1000
612/733 1110

AR226-0241

3M

Analytical and Research Properties - 3M Industrial Hygiene Laboratory

January 1993

Scope: This is a method for the determination of N-Ethyl Perfluorooctanesulfonamide in air.

Summary of Method: Air is drawn through silica gel sampling tubes. The N-Ethyl Perfluorooctanesulfonamide is adsorbed on the silica gel. The primary and backup sections of the tubes are extracted separately with methanol. The liquid is then analyzed in a gas chromatograph with flame ionization or electron capture detector.

Apparatus:

1. Silica gel sampling tubes (such as SKC #226-10 or equivalent)
   Available from: SKC, Inc.
   334 Valley View Road
   Eight Four, PA 15330

2. Sampling pumps: Battery operated pumps capable of drawing air through the sample tubes at a rate of 50-500 ml/min.

3. Gas Chromatograph (such as Hewlett-Packard 5880A or equivalent) with capillary column (Restec Stabilwax 0.53 X 30 m, 1 micrometer film capillary column) and flame ionization detector (FID) or electron capture detector (ECD).

Sampling Procedure:

Sample in the employee's breathing zone at a rate of 50-500 ml/min, depending on desired length of sampling time. The maximum volume of air sampled per tube should be 30 liters. Local conditions such as very high humidity may require a smaller volume.

The air flow through the sample tube should be as indicated by the directional arrow on the tube.

At the end of the sampling period, the tubes should be capped with the supplied caps.

Analysis:

Extraction: After sampling, the primary and backup sections of the sampling tubes can be extracted separately with 1 ml of reagent grade methanol in an autosampling vial and shaken for 30 minutes.

Analysis: The samples can be injected into the gas chromatograph (2 µl injection) and analyzed by FID or ECD using a Restec Stabilwax 0.53 mm ID X 30 m, 1 micrometer film capillary column. The GC conditions we used were:

125°C for 2 minutes
10°C/minute to 225°C
Hold for 10 minutes

Injector Temperature: 225°C
FID Temperature: 225°C

Under these conditions the retention time was 6.85 minutes.

The lower quantitation limit was ~10 micrograms with an FID. A lower quantitation limit of ~0.1 micrograms was seen with an ECD.

Recovery Study:

Fifty to 1000 micrograms of N-Ethyl Perfluorooctanesulfonamide was spiked onto the primary sections of silica gel sample tubes. Thirty liters of precleaned air was then passed through the sample tubes at 1.0 liter/minute. The samples were analyzed as described above.

The regression equation for the standard material was:

µgs analyte* = 0.0138 analyte area - 15.3 for
concentrations from 25-1000 µgs (r^2 - 0.99).

* N-ethyl Perfluorooctanesulfonamide

Based on this calibration curve, the average recovery 95 ± 11%. There was no breakthrough from the primary sections to the backup sections under the conditions described above.

(SDS127-A&RPROP)