Application Note 86

Analyzing Stack Gas Semivolatiles, Using Supelpak-2 Adsorbent and Capillary GC (Modified Method 5, US EPA SW-846)

The demanding detection limits required for air sampling by Modified Method 5 (US EPA SW-846, Method 0010) necessitates extensive cleaning and meticulous handling of the adsorbent cartridge/trap during sampling and analysis. Supelpak-2 adsorbent is cleaned in accordance with and meets the purity criteria of EPA SW-846, Method 0010, for testing semivolatile emissions from incinerator stationary sources.

Key Words:

- air monitoring stack gases incinerator emissions
- polynuclear aromatic hydrocarbons semivolatiles

For decades, Amberlite® XAD®-2 adsorbent, a styrenedivinylbenzene copolymer material, has been used for adsorption of hydrophobic compounds. A dry, high-purity version of this adsorbent, Supelpak™-2 (Figure A), is extensively used for sampling airborne semivolatile compounds, particularly polynuclear aromatic hydrocarbons (PAHs). Source testing, as well as ambient and indoor air monitoring, can be performed with Supelpak-2 material. Physical characteristics and several applications for Supelpak-2 adsorbent are presented in Table 1. The demanding detection limits of these air sampling methods necessitate extensive cleaning and meticulous handling of the adsorbent cartridge/trap during sampling and analysis.

Modified Method 5 (US EPA SW-846, Method 0010)

Supelpak-2 adsorbent is cleaned in accordance with, and meets the purity criteria of, US Environmental Protection Agency SW-846, Method 0010, for testing semivolatile emissions from incinerator stationary sources. Supelpak-2 is packed into glass cartridges/traps. Gaseous and particulate pollutants are collected by pumping air through the sampling train at high flow rates (>20 liters/min). After the sample is collected, the cartridge is extracted with methylene chloride, the extract is concentrated, and the sample is analyzed by GC/FID, GC/MS, or HPLC/UV.

Method 0010, Appendix B calls for a blank adsorbent cartridge to contain no more than 10µg total semivolatiles per gram of adsorbent, and recommends the background be less than 4µg per gram. High purity Supelpak-2 adsorbent well exceeds these requirements. Figure B shows the total chromatographable organic (TCO) analysis for Supelpak-2 adsorbent, under GC conditions selected to encompass the elution range of PAHs. To monitor extraction efficiency a surrogate, such as 2-fluorobiphenyl, is spiked into the cartridge prior to extraction. Figure B also shows the TCO analysis of another "clean" XAD-2

material. Clearly, Supelpak-2 is the cleaner material. Figure C shows the analysis of a reference standard containing heptane and perylene-d₁₂ in addition to the surrogate. Although not part of the EPA method, the reference standard is used for quantification and as retention markers.

Supelpak-2 consistently exhibits extremely low background, well below the levels required in US EPA methods. This adsorbent allows immediate, reproducible, reliable use for airborne semivolatiles analysis. As leaders in resin cleaning technology, we

Table 1. Physical Characteristics and Uses for Supelpak-2 Adsorbent

Matrix: polyaromatic Mesh Size: 20-60 Surface Area: 300m²/a Mean Pore Diameter: 90Å

Applications

US EPA SW-846, 0010 (Modified Method 5 Sampling Train) US EPA SW-846, 0020 (Source Assessment Sampling System) TO13 (PAHs in ambient air) IP-7 (PAHs in indoor air)

Figure A. Trap Containing Supelpak-2 Adsorbent



995-0259





also are able and willing to fulfill your custom-adsorbent and custom purification needs - simply contact our highly qualified Technical Service chemists with your questions.

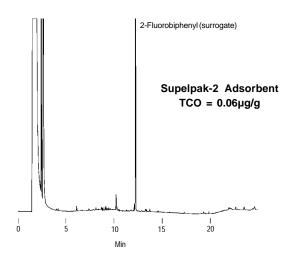
Figure B. Negligible Background in Supelpak-2 Adsorbent

Column: SPB™-5, 30m x 0.53mm ID, 0.50µm film

Cat. No.: 25317

40°C (4 min) to 300°C at 15°C/min, hold 4 min Oven: Carrier: helium, 6cc/min (nitrogen make-up gas, 40cc/min)

Det.: FID, 320°C 1µL, direct, 220°C Inj.:



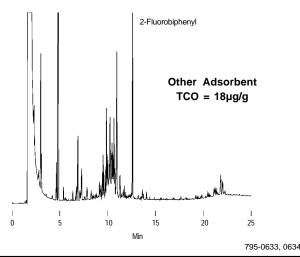


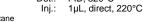
Figure C. Reference Standard

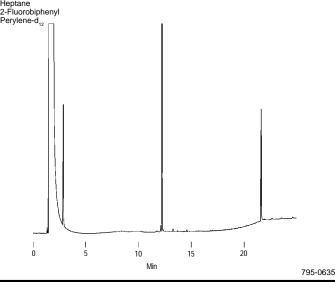
SPB-5, 30m x 0.53mm ID, 0.50µm film Column:

Cat. No.: 25317

40°C (4 min) to 300°C at 15°C/min, hold 4 min Oven: helium, 6cc/min (nitrogen make-up gas, 40cc/min) Carrier:

FID, 320°C Det.:





Ordering Information:

Supelpak-2 Adsorbent

100g 20279

Capillary GC Column

SPB-5, 30m x 0.53mm ID, 0.50µm film 25317

Trademarks

SPB, Supelpak - Sigma-Aldrich Co. Amberlite, XAD - Rohm and Haas Company

Contact our Technical Service Department (phone 800-359-3041 or 814-359-3041, Fax 800-359-3044 or 814-359-5468) for expert answers to your questions.

Note 86

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