

LITERATURE CORNER

μ BONDAPAK™ C₁₈ COLUMN AIDS IN FINDING EVIDENCE OF N-NITROSO COMPOUNDS IN AIRBORN PARTICLES (1)

Chemical, infrared, and thermal energy analyses of chromatographic fractions have provided evidence for the presence of N-nitroso compounds in extracts of airborne particles. The total N-nitroso concentrations in New York City air have been shown to be equivalent to the total concentrations of polycyclic aromatic hydrocarbons. Since 90 percent of the N-nitroso compounds that have been tested are carcinogens, the newly discovered but untested materials may represent a significant environmental hazard.

Samples of breathable, particulate matter were collected from air in New York City and extracted with dichloromethane. Potential interferences were removed by sequential extractions with 0.2N NaOH (removal of acids, phenols, nitrates, and nitrites) and 0.2N H₂SO₄ (removal of amines and bases). The samples were then subjected to a fractional distillation and a change of solvent to methanol.

Both thin-layer chromatography (TLC) and liquid chromatography (LC) (Figure 1) were used to separate the compounds present in the methanolic extract.

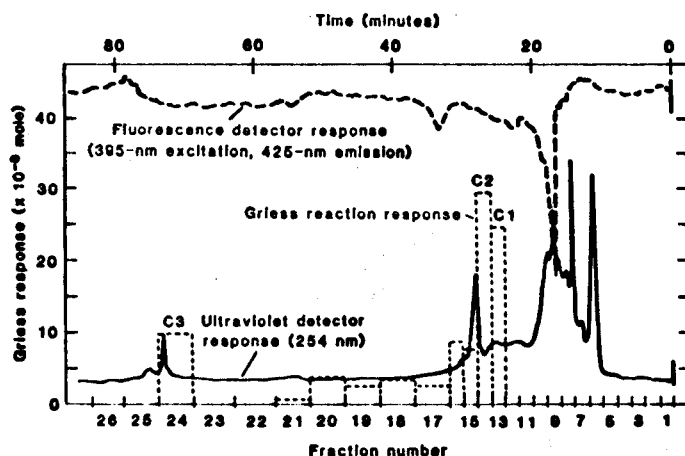


FIGURE 1: High-performance liquid chromatographic separation of unknown N-nitroso compounds. The colorimetric, photometric, and fluorometric detection responses are shown. A μ BONDAPAK™ C₁₈ column was used (30 cm by 3.9 mm). Solvent composition was programmed from 20 to 80 percent acetonitrile in water (pH = 4, phosphoric acid) over 20 minutes, was held isocratic (constant solvent composition) for 22 minutes, and then was programmed from 80 to 100 percent acetonitrile in 1 minute, and was held at 100 percent acetonitrile for 20 minute. The flow rate was 1.0 ml/min.

TLC was used to separate the N-nitroso compounds in the methanol solution from aliphatic and PAH classes by development of the silica-gel TLC plates with cyclohexane. Separations with LC then gave three fractions that were consistently positive by the Griess reaction (Figure 1).

The authors state that if the compounds found in this study are as potent as the other nitroso compounds evaluated in animal studies, then they may be more hazardous than benzo[a]pyrene. They continue, "This estimate that the hazard from this class of compounds may equal that for total PAH indicates the need to determine the structures of the N-nitroso materials and to test their biological activity." Clearly, LC has a significant role to play in scale-up and isolation in this type of research.

1. T. J. Kneip, J. M. Daisey, J. J. Solomon, R. J. Hershman, (Institute of Environmental Medicine, New York University Medical Center, New York 10016) Science, 221, 1045 (1983).