

sure was 2.8 ppb. All measurements of AP were below the LD. The presence of SO and PAA was confirmed by GC-MS. Given the low levels of PAA and AP indicated by this sample of data, it is concluded that past assessments of SO within the reinforced plastics industry were probably not highly biased.

267

DEVELOPMENT OF NIOSH METHOD 5042: TOTAL PARTICULATES AND BENZENE SOLUBLE FRACTION (ASPHALT FUME). L. Olsen, B. Belinky, P. Eller, R. Glaser, R. Lunsford, C. Neumeister, S. Shulman, NIOSH, Cincinnati, OH

During asphalt fume exposure assessment surveys, the results obtained using NMAM 5023 (coal tar pitch volatiles) often exceeded the results obtained using NMAM 0500 (particulates not otherwise regulated, total). After reviewing the methods, NMAM 5023 was withdrawn and the development of a new method using a single sampler for both the total particulates and the benzene soluble fraction was initiated.

The resulting method (NMAM 5042) uses a tared, 37-mm, 2- μ m pore size, polytetrafluoroethylene membrane filter for sampling. After sampling, the tared filter is reweighed, and the difference is the amount of total particulate collected. The tared filter is then extracted with 3 mL of benzene, and the extract filtered. Next, 7.5 mL of the filtered extract is dried in a tared weighing cup in a vacuum oven. Once the benzene evaporates, the tared weighing cup is reweighed and the difference is the benzene soluble fraction of the total particulates. The pooled relative standard deviation was 4.8% for loadings equal to or greater than 0.10 mg per sample for total particulates. For the benzene soluble fraction, the pooled relative fraction was 6.1% for loadings equal to or greater than 0.20 mg per sample. Since no independent method for determining total particulates is available, no estimate of the bias was made. However, if the total particulate loading is equal to or greater than 0.10 mg per filter, there is at least 95% probability that the determination will be within 25% of the true value 95% of the time if the upper 97.5% confidence limit on the bias is less than 11.5%. For benzene solubles, for which the bias is constant when more than 0.20 mg is on the filter, there is at least 95% probability that the determination will be within 25% of the true value 95% of the time if the loading is equal to or greater than 0.20 mg per filter. The limit of detection (LOD) and the limit of quantitation (LOQ) for total particulates were 0.04 and 0.13 mg per filter, respectively. The LOD and LOQ for the benzene soluble fraction were 0.04 and 0.13 mg per filter, respectively. An independent chemist evaluated the method with pyrene (1.08, 0.392, 0.216 mg per filter). A mean total particulate recovery of 106% (SD=6.1%) was obtained, and the mean benzene soluble fraction recovery was 109% (SD=11.0%).

268

RECENT ADVANCES IN MEASURING SOLVENTS IN URINE. S. Ghittori, L. Maestri, Fondazione Salvatore Maugeri, IRCCS, Pavia, Italy; D. Cottica, E. Grignani, Fondazione Salvatore Maugeri; M. Imbriani, Occupazionale e di Comunit dell'Università di Pavia, Italy

In the analysis of solvents in urine, the sample preparation step is frequently the most time consuming and the primary cause of analyte loss from matrix. Currently, volatiles are analyzed using either head space (HS) or purge-and-trap (PT) methods. HS is largely confined to higher concentration samples. PT sampling, although very sensitive, is expensive and prone to leaks and contaminated traps. The ideal sample preparation method should be fast, inexpensive, solventless, portable, relatively independent of the instrument design, and amenable to automation. A simple method is described combining solid phase microextraction (SPME), thermal desorption (TD) and gas chromatography-mass spectrometry (GC-MS) for determining volatile organic compounds in urine. Volatile organic compounds (VOCs) found as contaminants in urine of subjects occupationally and nonoccupationally exposed include benzene, toluene, xylenes, and chlorinated hydrocarbons (such as chloroform and carbon tetrachloride). Because these organic compounds are toxic to humans, their sensitive and rapid determination is of critical importance. A sorbent cartridge built with a Gore-Tex membrane (GORE, Elkton, USA) was used as extraction media for this study. The sorbent cartridge was filled with 300 mg of Tenax (35/50 mesh) previously thermally desorbed to ensure maximum purity. The urine samples were collected in polycarbonate bottles and immediately transferred in glass vials containing a Gore-Tex/Tenax sorbent so that the solvents can diffuse on absorbent substrate when vials, containing the urine, are sealed with airtight plugs without silicone. The Tenax sorbent cartridge serves to collect solvents that are present in the urine and to keep in unstable the equilibrium between the urine and Gore-Tex/Tenax system. After 24 hours from the sample collection the sorbent cartridge is removed from vial and successively analyzed. The solvents are thermally desorbed from Tenax and injected in a column (Thermal Tube Desorber-Supelco; 250(C thermal flash; borosilicate capillary glass-column crosslinked dimethylsilicone 25 m length, 0.33 mm I.D., 0.02 mm film thickness; gas chromatograph-mass selective detector). Experiments show that detection limits are in the 25-50 ng/L range for spiked solutions, and the linear dynamic range extended up to 50 mg/L.

269

SYNTHESES OF NICOTINE N-OXIDE AND COTININE N-OXIDE AND THEIR ANALYSIS BY HIGH PERFORMANCE LIQUID CHROMATOGRAPHY. S.H. Yim, S.S. Que Hee, University of California, Los Angeles, CA

The N-oxides of nicotine and cotinine are major potential carcinogenic metabolites of tobacco because of the nitroso-moiety. Therefore, sensitive analytical methods are needed to separate these metabolites from other urinary and blood interferences. High performance liquid chromatography (HPLC) is a widely used analytical method for urine and blood samples. Both nicotine N-oxide and cotinine N-oxide have not been resolved by HPLC. Nicotine N-oxide and cotinine N-oxides were synthesized (purity >99%). Then a simple and reliable reversed-phased HPLC method with ultraviolet detection (254 nm) was developed for nicotine, cotinine, and their two N-oxide metabolites. Nicotine N-oxide and cotinine N-

oxide were synthesized by adding purified m-chloroperoxybenzoic acid (>95%). The crude N-oxides were dried under vacuo and column chromatographed on aluminum oxide (basic) with 7% methanol in chloroform (v/v) eluent followed by recrystallization.

A HP1090 liquid chromatograph, HP1050 UV detector and Supelcosil LC-ABZ column with gradient mobile phase (0.007M KH₂PO₄ in water and in 50% acetonitrile(v/v)) were used. The column temperature was 39 \pm 0.1C and flow rate was 0.8 mL/min until nicotine, cotinine N-oxide, and nicotine N-oxide eluted and then 1.2 mL/min to elute cotinine. Retention times were nicotine, 6.1 min; nicotine N-oxide, 13.3 min; cotinine, 19.9 min; cotinine N-oxide, 11.7 min, respectively. The linear range for nicotine was from 200 ng to 30 mg ($y=6E+0.6x-2E+06$, $R^2=0.9976$), nicotine N-oxide was from 500 ng to 30 mg ($y=5E+0.6x+704574$, $R^2=0.9980$), cotinine was 100 ng to 15 mg ($y=5E+0.6x+992212$, $R^2=0.9929$), and cotinine N-oxide was 50 ng to 15 mg ($y=2E+0.7x-1E+06$, $R^2=0.9992$), where y is the peak area in arbitrary units and x is mg in 10 mL injected.

LEAD

Papers 270 - 279

270

TASK SPECIFIC EXPOSURE TO AIRBORNE LEAD DURING RESIDENTIAL ABATEMENT. S.G. Brumis, G.J. Reames, L.L. Lance, California Department of Health Services, Emeryville, CA; M. Nicas, University of California, Berkeley, CA

Residential lead abatement frequently involves a series of short term tasks. Air sampling to obtain an 8-hour time-weighted average (TWA) result provides data for meeting OSHA requirements, but does not provide information on the exposure potential from discrete operations. The study objectives were to characterize worker exposure to airborne lead by performing short term, task specific air sampling during residential lead abatement and to use the sample results to test an equation for predicting airborne exposure levels from the amount of lead to be disturbed and the task to be performed. A total of 224 short term (mean of 32 minutes) personal air samples were collected from 16 workers employed by three contractors at 11 private San Francisco Bay Area residences. Tasks sampled included paint scraping, demolition, HEPA vacuuming or wet wiping, chemical stripping, and wet sanding. Air sample results ranged from 30 mg/m³ (10.27% of air samples), while eight workers had at least one result >50 mg/m³ (6.25% of air samples). The data demonstrate the effectiveness of short-term, task based air sampling. The predictor formula was evaluated by performing correlation analyses to determine the strength of the linear relationship between air sample results and lead in paint (mg/cm²), paint removed (%), and the task area (cm²). The low correlation of the variables to air sample results is related to the considerable variability in air sample results between contractors, workers, tasks, and within tasks.

271

FIELD DEMONSTRATION OF TECHNOLOGIES FOR REMOVAL OF LEAD-BASED PAINT. J.R. Kominsky, Environmental Quality Management, Inc. Cincinnati, OH; P. Clark, A.

Abstracts

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