

fraction lost during the test regime where such losses cannot be easily measured. Additional calculations demonstrate our ability to use the loss of the preimpregnant from the sampler to gain an improved estimate of the time-weighted average concentration of airborne contaminants. A patent application has been filed for this procedure.

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**OVERCOMING THE DILEMMA OF MULTIPLE CONFLICTING BLANKS IN BLANK-CORRECTING AIR SAMPLE RESULTS.** D.E. Shirey, Supelco, Inc., Bellefonte, PA

For years the author has marveled at how, despite the abundant existence of guidelines, individuals uniquely interpret and apply data regarding the blank-correction of air sample results used to determine compliance with industrial hygiene standards. Most confusion stems from the necessity of incorporating multiple blank results, such as are associated with large batches of samples, which differ sometimes substantially from one another. Rarely do industrial hygienists anticipate the challenge presented by multiple conflicting blanks (MCB), yet when faced with this dilemma, rarely can IHS do anything except search for the least potentially objectionable method of applying this decision to data that realistically cannot be regenerated. Handling of this issue by individuals can be categorized as either "aggressive" or "defensive" based on their approach. Sometimes the interpretation of MCB tied to sample results already near the compliance threshold can sway the outcome. Recommendations are offered that will prepare the IH to meet this inevitable challenge with objectivity and confidence.

## 263

**DEVELOPMENT OF A NEW APPROACH FOR TOTAL ISOCYANATE DETERMINATION USING THE REAGENT 9-ANTHRACENYL-METHYL 1-PIPERAZINECARBOXYLATE.** Y.-M. Roh, NIOSH, Cincinnati, OH

Isocyanate compounds to which workers are exposed may include monomers, prepolymers, and/or other polymeric species generated during polyurethane production or thermal breakdown. Methods for the determination of isocyanate compounds in air typically involve derivatization of the isocyanate species with an amine reagent and analysis of the derivatized isocyanates by high-performance liquid chromatography (HPLC). These methods rely on a number of assumptions with regard to determining nonmonomeric isocyanate species.

It must be assumed that peaks derived from isocyanate species can be recognized in the chromatogram and that the detector response factor of the unknown isocyanate species is the same as that of the derivatized monomer. It also must be assumed that all isocyanate species have eluted from the analytical column as observable peaks. Even when all these assumptions are reasonable, the task of identifying and quantifying numerous isocyanate peaks in a chromatogram requires much judgment on the part of the analyst and is labor intensive, making it difficult to process numerous samples in a routine manner.

9-Anthracenylmethyl 1-piperazine carboxylate (PAC) is a newly developed derivatizing reagent designed to enable much simpler total

isocyanate analysis. Like other amine reagents, it reacts rapidly with isocyanates to form stable derivatives, enabling HPLC analysis of monomers or other specific isocyanate species. However, PAC derivatives also undergo a cleavage reaction under mild, but selective, conditions to generate the same single analyte for all species. This has several advantages over conventional total isocyanate analysis: (1) Identification can be based on chromatographic retention time; (2) there is no compound-to-compound variability in response factor; (3) there is no concern about failure to elute all isocyanate species; and (4) quantification involves measurement of a single peak.

Moreover, this technique may enable measurement of isocyanate groups chemically bound to particle surfaces, which cannot be measured by conventional chromatographic techniques.

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**DEVELOPMENT OF A THERMAL DESORPTION METHOD FOR 1,3-BUTADIENE, ACRYLONITRILE, AND STYRENE.** L. Wu, S. Uang, T. Shih, Institute of Occupational Safety and Health, Council of Labour Affairs, Taipei, Taiwan

1,3-Butadiene, acrylonitrile, and styrene are widely used in resin synthesis. However, there is no simultaneous air sampling tube for these three compounds. The aim of this study was to screen suitable thermal desorption solid sorbents for 1,3-butadiene, acrylonitrile, and styrene. Several sorbents such as Tenax-TA, Porapak N, Carbosieve-S-III, Molecular Sieve 13 X, Carboxen B, and Carboxen 569 were screened. Analytes were directly spiked onto sorbent tubes for static standing and clean air drawn through dynamic tests. From screening tests, Tenax-TA and Carboxen B were found capable of absorbing and desorbing styrene. Porapak N and Carbosieve-S-III were able to absorb acrylonitrile; however, Porapak N was superior. Carbosieve-S-III and Carboxen 569 were equally capable of absorbing 1,3-butadiene. Single-bed sorbent tube was recommended for single compound air sampling. Tenax-TA sorbent tube is suitable for styrene sampling, Porapak N for acrylonitrile, and Carbosieve-S-III for 1,3 butadiene. For simultaneous sampling of 1,3 butadiene, acrylonitrile, and styrene, a multibed sampler packed with 100 mg Tenax-TA and 200 mg Carbosieve-S-III should be used.

## 265

**VALIDATION OF A DYNAMIC PERSONAL SAMPLER FOR CHLOROACETONE.** Y.-W. Lin, S. Que Hee, University of California, Los Angeles, CA

Dynamic air sampling for aldehydes using derivatization with O-(2,3,4,5,6-pentafluorobenzyl)hydroxylamine hydrochloride (PFBHA) has been developed with more than 90% recovery for several aldehydes. The other important carbonyl compounds, ketones, can also form the O-oxime derivatives with PFBHA. They may be positive interferences in the aldehyde sampling test. The aim of this study was to validate the dynamic personal air sampling method for chloroacetone vapors and to assess possible application to other ketones. Tenax TA (80/100 mesh) was coated with 20% (w/w) PFBHA by rotary evaporation. Coated 200-mg solid sor-

bent was packed into 7-cm long Pyrex glass tubes (7-mm OD and 5-mm ID) with Pyrex glass wool layers at both ends. Desorption efficiency was tested by spiking specific amounts of chloroacetone in a 4-mL vial containing 200-mg coated sorbents then left at room temperature (21°C) for more than 12 hours. The sampling tube was connected to a 10-L gas bag with 1 ppm and 5 ppm of chloroacetone vapor. A calibrated personal sampling pump was connected to the other side of the packed sampler at 50 mL/min sampling rate. Different sampling durations were tested to investigate the maximum capacity of the samplers. Each test was performed at least in triplicate. The PFBHA chloroacetone oxime was desorbed by hexane with agitation at least 2 minutes, and analyzed by gas chromatography/electron capture detection (GC/ECD). The lowest quantitative level (LQL) was 500 pg. The desorption efficiency was  $96.1 \pm 5.8\%$ . The recovery percentages were  $105 \pm 2\%$  for 15 ppm-min,  $93.9 \pm 5.6\%$  for 45 ppm-min,  $97.5 \pm 4.5\%$  for 60 ppm-min,  $90.9 \pm 8.9\%$  for 150 ppm-min,  $89.3 \pm 6.1\%$  for 180 ppm-min,  $96.8 \pm 5.5\%$  for 210 ppm-min,  $99.5 \pm 2.9\%$  for 240 ppm-min, and  $97.8 \pm 3.6\%$  for 270 ppm-min. Each sampling tube provided 0.16 mmole PFBHA for reaction and the validated capacity of chloroacetone was  $5.48 \times 10^{-4}$  mmole or a molar ratio of PFBHA to chloroacetone of 290:1, whereas a ratio of 10:1 sufficed for aldehydes. This may be due to the steric factors of ketones. More ketones need to be investigated.

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**MEASUREMENT OF STYRENE OXIDE IN AIR.** R. Tornero-Velez, S. Rappaport, University of North Carolina, Chapel Hill, NC; L. Denovan, University of Washington, Seattle, WA

Styrene-7,8-oxide (SO) is generated at low concentrations from the oxidation of styrene during the processing of reinforced plastics. Since evidence indicates that exposure to SO has important health implications, we developed air sampling and analytical methods to measure airborne SO in the presence of styrene as well as other oxidation products of styrene, namely phenylacetaldehyde (PAA) and acetophenone (AP). Both active and passive air monitors are used. The active sampling method employed 45 mg of Tenax as the absorbent. After sampling, the Tenax was desorbed with ethyl acetate and the analytes measured by gas chromatography with FID. By injecting at 70°C, the thermal rearrangement of SO to PAA was minimized. The limit of detection (LD) for SO, PAA, and AP was 1 ppb with a volume of 36 L. The passive sampler (3M 3500) measured styrene and SO only. The efficiency of desorption of SO from the 3M monitor with ethyl acetate depended on the loading as described by the regression equation:  $C = (3.55) C_0^{1.29}$ , where C represents the observed concentration and  $C_0$  represents the true extract concentration. The LD for this method was 5 ppb assuming 8 h of sampling at a rate of 19.4 mL/min, as determined in laboratory experiments. Measurements at various U.S. facilities where boats, bathtubs, pipes, and tanks were manufactured, showed mean SO levels at 22.5 ppb for the passive method (n=44) and 26.9 ppb for the active method (n=35) with an  $r^2$  of 0.82. Levels of PAA were below the LD except for three boat laminators where the average expo-

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