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Project Summary

Development of Analytical Methods for Specific Lawn-Applied Pesticides in House Dust

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The methods developed in this program were designed for detection of specific lawn-applied pesticides in house dust and in polyurethane foam (PUF) sleeves. PUF sleeves are used in a dislodgeable surface residue collection device, the PUF Roller. The method developed for the herbicide acids was applicable, as well, to the analysis of pentachlorophenol and 2,4-dichlorophenol. The method developed for the dinitro-aniline herbicides was also applied successfully to the analysis of several thiophosphate insecticides and a chlorinated fungicide.

Methods developed here provided recoveries better than 80% at spike levels that approximate house dust levels. The methods incorporated surrogate recovery standards and internal standards (for quantification) that are tailored to the specific analyte classes. This use of structurally similar surrogate recovery standards and internal standards provides a greater degree of confidence in the methods when they are used for field samples: the recovery of surrogates gives a clear picture of the expected analyte recovery. Structurally similar internal standards compensate for the minor chromatographic differences between samples and standards and these more accurately reflect the chromatography of the analytes.

The methods that were developed are rigorous and rugged, and performed well when applied in field studies of simulated track-in of lawn-applied pesticides into the home. Recoveries of surrogate recovery standards were generally greater than 90% in these studies.

This Project Summary was developed by EPA's National Exposure Research Laboratory, Research Triangle Park, NC, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

House dust appears to be a long-term sink within the home for pollutants, in general, and pesticides, in particular. Scientists, and now homeowners, are interested in the concentrations of hazardous pollutants adsorbed on house dust. Young children appear to ingest significant quantities of dust while crawling and playing on carpeted surfaces. With their less sophisticated immune systems and high degree of developmental change, younger children may be at greater risk to ingestion of contaminated dust than older children or adults.

House dust is a more complex matrix than soil or plant material. Debris from human skin and human activities (e.g. food, household cleaners) combined with combustion source-derived pollutants will be encountered in the house dust. Potential chemical interferences in the analysis of pesticides in dust include, but are not limited to, fatty acids, lipids, phenols, polycyclic aromatic hydrocarbons, and carpet additives. The ease in applying standard soil analysis methods to house dust samples for outdoor-applied pesticides will be compromised further by pesticide lev-

els that are expected to be significantly lower due to the dilution that occurs during any migration process. Therefore, methods suitable for outdoor levels are unlikely to be suitable for lower indoor levels in a specific matrix like house dust.

Results and Discussion

Two different analyte derivatization methods were developed and used on this program, methylation with diazomethane and pentafluorobenzyl bromide (PFBBr) derivatization. Methylation of analytes with at least two chlorines provides sufficient sensitivity for detection by GC/ECD (gas chromatography with electron capture detection) of mid to low ppb concentration levels. Detection limits for the mono-chloro phenoxy herbicide acids can be as much as a factor of 1000-fold higher, compared to the di-chloro phenoxy herbicide acids, with methylation and GC/ECD analysis. PFBBr derivatization offers the possibility of detection with enhanced sensitivity for these mono-chloro herbicide acids.

The analyte spike levels for studies involving methylation spanned a concentration range of 1000. Despite this wide concentration range, very similar recoveries were achieved for all analytes; recoveries were generally >85%. As seen from the study of recovery from PUF, the recovery of the 2,4-D sodium salt was only slightly less than the recovery of the 2,4-D free acid. We conclude that these methods are useful for recovery of herbicide salts that are typically applied to turf. For analyses using the PFBBr derivatization, the spike levels of the analytes were identical and recoveries were similar (>85%).

Conclusions

Methods developed here provided recoveries better than 80% at spike levels that approximate house dust levels. The methods incorporated surrogate recovery standards and internal standards (for quantification) that are tailored to the specific analyte classes.

The methods that were developed were rigorous and rugged, and performed well when applied in field studies of simulated track-in of lawn-applied pesticides into the home. Recoveries of surrogate recovery standards were generally greater than 90% in these studies.

Sonification was shown to be useful for extraction of both acidic herbicides and polar dinitro-aniline herbicides. This approach was rapid and eliminated the need for solvent-intensive Soxhlet extraction. Extraction of PUF sleeves by manual squeezing of the PUF in a polyethylene zip-lock bag was shown to be an efficient method for obtaining an extract of trace dislodgeable surface residues.

Due to the complexity of the house dust matrix, cleanup procedures had to be developed with each method. With sample extract cleanup, analyses could be performed using relatively low-cost, automated GC/specific detector analyses, rather than higher-cost, GC/MS analyses. The cleanup procedures also reduced significantly the organic burden placed on GC columns, thus prolonging their lifetime and enhancing their performance. Solid phase extraction with disposable prepacked cartridges was shown to be an effective and efficient method of cleanup. Additional liquid partition steps were used as necessary.

Analyses of nine residential house dust samples for herbicide acids and pentachlorophenol were carried out using the designated extraction and cleanup methods and both GC/ECD and NCI GC/MS analyses. These analyses showed good agreement in quantification between the two techniques, and no detectable interferences to the analytes of interest.

Recommendations

We recommend a further method development effort for the analyses of glyphosate in house dust. Degradation in GC column performance and/or detector performance was found with the method developed, indicating the need for development of an additional sample cleanup technique.

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The complete report, entitled "Development of Analytical Methods for Specific Lawn-Applied Pesticides in House Dust," (Order No. PB9; Cost: $25.00, subject to change) will be available only from:
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