PD 008

The determination of Thiram Residues in Fruit by UPLC-MS/MS

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Thiram is a non-systemic dimethyl dithiocarbamate fungicide. It is mainly used in the field as well as to protect harvested crops during transport and storage. It is considered to be a general use pesticide, licensed for use on a range of crops including fruit, vegetables and ornamentals to prevent fungal diseases. It is also used as an animal repellent to prevent damage caused by rabbits, rodents and deer.

The analysis of dithiocarbamates is generally performed by the measurement of liberated CS2, following decomposition in the presence of SnCl2/HCl, although this technique does not distinguish between the other related compounds that also produce CS2 in this way. As a result, there was a requirement to develop and validate a modern analytical method that was specific to thiram, and could be routinely used for the analysis of samples generated in crop residue trials.

There are a number of challenges to analysing thiram directly. The analyte can undergo decomposition when exposed to acidic plant juices, and it is also suggested that thiram stability is adversely affected by the presence of copper ions1. The analytical method that was developed involves the extraction of frozen homogenised samples with cold acetonitrile following the addition of anhydrous sodium sulphate. An aliquot of the extract is diluted with an aqueous EDTA solution, which stabilises the analyte, enabling routine analysis by UPLC-MS/MS. No further sample clean-up stage is required as there are no observable matrix effects using this approach. This method allows a range of fruit sample types to be analysed, using this quick, efficient, robust and reliable technique.

The method was fully validated to a limit of quantitation (LOQ) of 0.01 mg/kg in strawberry and apple, adhering to the appropriate regulatory guidelines relating to this type of work. The application of this method for the determination of thiram in fruit generated from numerous field trials confirms the suitability and reliability of this technique when applied to routine sample analysis. In addition, the same samples were further analysed using the CS2 technique, and the data generated for samples where residues were detected were in good agreement, demonstrating the acceptable performance of the LC-MS/MS method for residue analysis studies.

References

1. Filipe O.M. et al, (Aug 2008), J Agric Food Chem., 56(16):7347-7354.