ANALYTICAL METHODOLOGY FOR THE DETECTION OF FUMIGANTS AT THE SJVASC

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Abstract.

Recent work to improve analytical methodology for the detection of fumigants in the gas, or solid / liquid phase will be summarized. A barrier ion discharge detector (BID) is shown to quantitatively detect fumigant gasses in concentrations ranging over several orders of magnitude (i.e., 822 to 0.036 mg/L for propylene oxide). Headspace analysis using either solid-phase microextraction or a headspace auto sampler equipped with an adsorbent trap, is shown to accurately quantify fumigant residues in foodstuffs without lengthy sample preparation or the use of solvents.

Presentation Summary.

This presentation will summarize the most recent efforts to improve analytical methodology for the detection of fumigant gasses in either the gas phase (i.e., for monitoring the concentration during a postharvest fumigation) or the solid phase (i.e., for measuring fumigant gas residue following treatment). The focuses of this research is to:

- Determine optimal instrumentation / detectors for fumigant gas quantitation
- Develop faster methods of sample preparation for analysis
- Develop sample preparation methods that require fewer resources, in terms of man hours and materials.

A barrier ion discharge detector (BID) is an accurate and universal detector for fumigant gasses. Coupled with a gas chromatograph (GC, Shimazdu Tracera), it only requires one gas (helium) for operation (as opposed to other GC detectors commonly used for fumigant analysis) and shows linear response to fumigant concentrations ranging over several orders of magnitude. Fumigants tested, with limits of quantitation, include: methyl bromide (MB) 15916 - 5.24 ppm, phosphine (PH_3) 1002 - 10.12 ppm, propylene oxide (PPO) 822 - 0.036 mg/L, sulfuryl fluoride (SF) 122 - 1.47 mg/L, and ethyl formate (EF) 109 - 0.003 mg/L.

Headspace sampling (collecting analyte from the air above a solid or liquid sample) is a commonly used method to detect fumigant gas residues in foodstuffs due to their relatively high vapor pressures. To ensure a sufficiently high gas phase concentration, the commodity is often homogenized in the presence of solvent before letting the fumigant reach equilibrium between the solid, liquid, and gas phases and sampling the headspace with a gas-tight syringe. While this can be an effective method of analysis, it is also time-consuming, wasteful, and requires carefully controlled extraction conditions to ensure precision. By using an adsorbent material to preconcetrate the analyte prior to injection these lengthy sample preparation steps can be eliminated. We present alternate methods for the determination of PPO and MB residues in

multiple commodities using a headspace trap autosampler (Perkin Elmer, Turbomatrix) or solid phase microextraction (SPME). Using the Turbomatrix allows for the detection of PPO, as well as its toxic degradation products propylene chloro/bromo hydrin, at levels ~3 orders of magnitude lower than those observed with liquid extractions (60ng/g vs. 30µg/g). Analysis of MB residues using either the Turbomatrix or SPME sampling allows for sub-ppm levels of detection with a quick one-step, solvent-free preparation method, rather than extraction with a buffered solution, followed by several hours of equilibration.