Committee on Pesticide and Disinfectant Formulations

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Committee Actions

The committee has worked on 2 methods. The maleic hydrazide method (**2004.09**) is being proposed to the Official Methods Board for Final Action. The hydrazine in maleic hydrazide method is in the process of being voted on by the committee for move to First Action. Final voting will be recorded using the AOAC Website. The bifenthrin collaborative study is completed and manuscript is in draft. The committee agreed on the update of Chapter 7 and the prioritization of starting with the gas chromatography (GC) older column technology methods. The committee has agreed upon a procedure to data mine various sources including AAPCO Check Sample data, requests for data from laboratories through AgLabs ListServe. The committee agreed to continue meeting monthly by teleconference call with e-mail updates for members not able to attend.

At the 121st AOAC Annual Meeting, the committee voted and approved the following Terms of Reference for GC methods in OMA Chapter 7:

(1) Column technology update existing *Official Methods of Analysis*SM (OMA) method has referenced a GC or liquid chromatographic (LC) column with older column dimensions. Method will be updated with newer column dimensions without further validation required as long as the stationary phase (LC) or liquid phase (GC) has not changed. Method will be amended to include an example of available newer technology. This is not a method change.

(2) To change or remove an internal standard from an OMA method; this is a minor change and an AOAC single-laboratory validation (SLV) will be needed. A guidance of replacing an internal standard with another standard with similar retention time was noted.

(3) Change in solvent for the extraction/dilution solvent requires an SLV if the polarity of the new solvent is different, otherwise, no SLV required.

(4) The following changes are considered optimizing chromatography and not a method change: injection volume, column temperature/ramp, carrier gas, injector or detector temperatures, split ratio, GC column film thickness, and column length.

The following method was recommended for Final Action:

***2004.09**, Maleic Hydrazide (MH) in Technical and Pesticide Formulations.*

Disinfectants, Tom Phillips

Mixed Phenols and Phenates in Ready-To-Use and Concentrated Disinfectants, LC Method.—A method for the determination of *p*-chloro phenylphenol (PCP), 4-*t*-amylphenol (4TAP), and *o*-benzyl-*p*-chlorphenol (OBPCP) in disinfectants was developed and analyzed through an SLV. Concentration ranged from 0% 4TAP to 10% OBPCP in the products analyzed. These products represented ready-to-use (RTU) and concentrates.

The linearity of the method was tested using 7 phenolic disinfectants: *o*-phenyl phenol, Dowacides 2 and 4, 4-*t*-amyphenol, 2-chloro-6-phenylphenol, *o*-benzyl-*p*-chlorophenol, and *p*-chlorophenol. Concentrations ranged from 0.01 to 2.00 mg/mL. Standards were made on each of 6 different days, and analyzed on a Waters Alliance system (Waters Corp.,

Milford, MA) with a diode array detector (DAD). The analytical wavelength was 284 nm. Correlation coefficients were ≥0.999, with coefficient of variation (CV) of <5.0% for individual levels. The overall CV for the separation method was 1.1%.

The extraction/dilution method chosen was simple dilution in acidified methanol (2% acetic acid, v/v) to fit in the middle of the standard curve, filtering through a 0.22 m**[m ok or μm?]** nylon syringe filter, then subsequently analyzed by gradient HPLC using an ODS(2) column with UV detection. There were 9 samples chosen with concentrations ranging as follows: OPP: 0.4 to 10% active, 4TAP: 0 to 4% active, and OBPCP: 0.05 to 9% active. The samples were chosen to have single and multiple components, as well as the salts of the actives. Spike samples were analyzed using a disinfectant that contained pine oil, artificial color, and quaternary ammonium compounds. The range of spike concentrations was from 0.05 to 0.6% of OPP, 4TAP, and OBPCP. The separation method was composed of a DI water phase with 2% acetic acid and an organic phase composed of 2% acetic acid in acetonitrile. The gradient time is 15 min and separates all compounds with baseline resolution. The samples and spikes were analyzed on 6 different days.

Results were put into a spreadsheet designed by Mark Roman for the purpose of analyzing results from SLV studies. For OPP, the means showed no significant difference for the 9 samples. The HorRat values ranged from 0.68 to 1.3, and the total RSDs for the samples ranged from 1.6 to 4.6%. The 4 spike samples showed similar values. For 4TAP, the means were different, but the HorRat values ranged from 1.7 to 6.8, and the RSDs from 3 to 22%. The 4 spike samples showed similar trends. For OBPCP, the means were not different. The HorRat values were between 0.8 and 11.87, with RSDs ranging from 2 to 183%. The higher RSD was for values <0.1%. Overall, the method has potential for covering a wide range of active concentrations, as well as a variety of actives. Continue topic and recommend a round robin test for the method.

Pesticide Formulations: Fungicides and Rodenticides, Lynda Podhorniak
Podhorniak's term expired 2007.

No report filed.

Pesticide Formulations: Herbicides, James Daft
Daft retired 2007.

No report filed.

Pesticide Formulations: Insecticides, Synergists and Repellents, Khanh Nguyen
Nguyen no longer AOAC member.

Bifenthrin collaborative study completed and manuscript in draft. Continue topic.

CIPAC Studies, Warren Bontoyan

Warren R. Bontoyan, Acting CIPAC General Referee and U.S. CIPAC member, Chief-Maryland State Chemist Section, Maryland Department of Agriculture, 50 Harry Truman Dr, Annapolis, MD, 21401, Tel: 410-841-2765, Fax: 410-841-2765, E-mail: bontoywr@mda.state.md.us. The GR reported on the 51st Annual Meeting and Symposium of CIPAC held June 12–14, 2007, in Umhlanga Rocks, South Africa. The collaborative studies conducted during the year were presented and discussed. A review of methods resulted in the following actions: 6 analytical methods were accepted as *provisional* CIPAC methods; 11 analytical methods were accepted as *full* CIPAC methods; 3 analytical method extensions were accepted as *provisional* and 3 as *full*; 2 analytical methods remain *tentative*; one quantitative identity test was added to an analytical method; and 3 analytical methods relevant impurities in specific formulations were noticed and regarded to be suitable for such determinations. A list of decisions made at the meeting is included in the General Referee report and will be published separately in the Journal. No recommendations are made at this time.