

**USDA Pesticide Data Program
Water Monitoring Program
Proficiency Testing Set #120**

Pesticide Data Program (PDP) proficiency testing set #120 is scheduled for shipment **September 13, 2004**. Ultra Scientific, Inc. will provide custom solutions to the Colorado Department of Agriculture, Montana Department of Agriculture, and New York Department of Agriculture and Markets laboratories for use in preparing these check samples.

Custom solutions cannot be provided at working levels due to stability concerns; therefore, they will be prepared and ampouled at higher concentrations and shipped to laboratory Quality Assurance Officers for dilution and spiking. All documentation (Certificates of Analysis and Data Paks) shall be supplied to MPO by Ultra and kept confidential until results have been reported.

This set shall consist of one sealed ampoule containing all compounds under evaluation for this proficiency testing set. Analytical method and limit of detection/limit of quantitation data provided to the Monitoring Programs Office (MPO) were used to determine the analytical profile and concentrations. Due to differences in analytical profiles and methodologies between the laboratories, the solution may contain some compounds not analyzed by all laboratories. Laboratories will not be held accountable for compounds not routinely analyzed in their water testing program.

Compounds will be dissolved in methanol and may be stored at room temperature overnight, or in the refrigerator or freezer for longer periods of time. Each laboratory Quality Assurance Unit (QAU) will spike the diluted solution mix into an aliquot of matrix blank for each analytical method. **Note: for this set, ethane sulfonic and oxanilic acid metabolites of chloroacetanilide pesticides were not available so laboratories should NOT spike/analyze an aliquot for this method.** Dilution/preparation instructions are detailed on page 2 of this document. Each extraction set should be accompanied by a matrix blank.

One month will be allowed for sample analysis and reporting, with results due **October 15, 2004**. After all results for test samples and associated matrix blanks have been received, laboratories will be sent the target values and MPO will prepare and issue a final report.

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Dilution and Fortification Instructions**

1. **Glassware Cleaning:** To minimize possible contamination all glassware including amber sample bottles should be solvent washed and dried prior to use. Alternatively, the laboratory may choose to clean and bake all glassware at high temperature to remove contaminants, with the exception of volumetric ware.
2. **Preparation of Spiking Solutions:** Each vial contains approximately 1 mL of stock solution. Stocks shall be diluted by removing 100 ul of ampouled solution and diluting to a final volume of 10 mL in methanol.
3. **Collection and Dechlorination of Matrix (Tap Water):** For each method, at least one liter of unfiltered tap water shall be collected from the same source of tap water used by the analytical group. The water shall be collected into an amber glass bottle containing appropriate dechlorinating agents (e.g., 1 g¹ sodium thiosulfate, 1.0 ml of 1% hydroxylamine hydrochloride per liter).

Samples shall be held for at least one hour following dechlorination prior to spiking. If samples are to be held overnight before spiking, they shall be refrigerated at 4°C.

4. **Sample Spiking:** For each method, a 1.0 ml aliquot of the diluted standard from step 2 shall be diluted to a final volume of one liter with the previously collected tap water. The spiked samples shall be transferred to labeled amber glass bottles and presented to the analytical group with the accompanying worksheet/reporting forms (see pages 3 and 4 of this document).

¹ Gram.