

Quantification of 3-chloro-7-hydroxy-4-methylcoumarin (CHMC) in urine as a biomarker of coumaphos exposure by high-performance liquid chromatography-fluorescence detection (HPLC-FLD)_Dataset

Introductory Information

The organophosphate pesticide coumaphos is used to control Cattle Tick Fever carried by multiple species of ticks and is a known hazard for workers treating livestock. The USDA Cattle Fever Tick Eradication Program (CFTEP) requires regular blood draws to measure depressed cholinesterase levels as biomarkers of effect of long-term coumaphos exposure, however, the gap between blood draws may miss intermittent high exposures. Urine biomonitoring can supplement blood draws, offering personnel a simple and cost-effective method to monitor short-term exposures. Our objective was to improve and validate a previously published method to analyze the coumaphos metabolite 3-chloro-7-hydroxy-4-methylcoumarin (CHMC). Urine samples were hydrolyzed with glucuronidase and then extracted prior to analysis with high-performance liquid chromatography-fluorescence detection. Method validation tests followed the NIOSH Manual of Analytical Methods evaluation criterion.

General Description of Collection Methods

Three urine samples were collected by USDA APHIS from 19 CFTEP study participants potentially exposed to the pesticide coumaphos. A previously developed preparatory and analytical method to determine a biomarker of coumaphos exposure in urine was updated and evaluated following NIOSH NMAM guidelines. Samples were hydrolyzed with glucuronidase, isolated with solid-phase extraction, and analyzed by HPLC-FLD. Method evaluation included calibration curves prepared in pooled control urine and aqueous solution, quality control standards, storage stability, detection limit determination, accuracy and precision of the sample preparation, and recovery from spiked urine.

List of Publications Based on the Dataset

Robbins, Z. G., Striley, C. A., & Wugofski, L. (2025). Quantification of 3-chloro-7-hydroxy-4-methylcoumarin (CHMC) in urine as a biomarker of coumaphos exposure by high-performance liquid chromatography-fluorescence detection (HPLC-FLD). *MethodsX*, **14**, 103171.

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Contact Information

Chemical and Biomonitoring Branch (CBMB), Health Effects Laboratory Division (HELD),
National Institute for Occupational Safety and Health (NIOSH), Cincinnati, OH