

US EPA ARCHIVE DOCUMENT



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UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
WASHINGTON, D.C. 20460

JAN 29 1985

OFFICE OF  
PESTICIDES AND TOXIC SUBSTANCES

MEMORANDUM

SUBJECT: PP#0F2413/FAP#0H5275/PP#3F2793/FAP#3H5378. Thiodicarb on Cotton and Soybeans. Evaluation of Method Trial Report Dated January 18, 1985 Regarding Analysis of Acetamide in Liver and Acetonitrile in Milk and Eggs.

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and

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EPA's Analytical Chemistry Section (COB, BUD) has completed method trials on acetonitrile in milk and eggs, and acetamide in poultry liver (see R. F. Thomas memo of 1/18/85). (Note - acetonitrile is being considered a marker compound for acetamide under FDA's SOM Policy (FR Vol. 44, No. 55 - March 20, 1979 - p. 17071)).

Because of the lack of an on column capillary injection system (a piece of equipment not available at most analytical laboratories), samples extracted and cleaned up at EPA's Beltsville, MD laboratory were taken to Union Carbide's laboratory at Research Triangle Park, NC for final analysis. The lack of available instrumentation also prohibited EPA's chemists from screening methanol (used for extraction of acetonitrile from milk and egg) for acetonitrile contamination.

### Part I - Acetonitrile in Milk and Egg Whites

Acetonitrile is extracted from milk and egg whites with methanol. Reagent blank values varied from 0.7 to 2.3 ppm. Control values for milk were 2.6 and 1.6 ppm (average = 2.0 ppm), while control values for egg whites were 1.4 and 0.5 ppm.

At fortifications of 0.1 ppm and 1.0 ppm acetonitrile in milk, recoveries ranged from 330-640% (average of 6 samples = 467%).

At fortifications of 0.3 ppm and 0.6 ppm acetonitrile in egg whites, recoveries ranged from 150-733% (average of 7 samples = 328%).

The high reagent blank and control values, as well as the extremely high recoveries indicate that the submitted methodology is not suitable for regulatory purposes.

The recoveries reported were not corrected for reagent blanks or control values.

### Part II - Acetamide in Liver

Acetamide is extracted from poultry liver with acetone. Reagent blank values were reported as 0.07 and 0.07 ppm acetamide. Control (liver) values were 0.16 and 0.13 ppm.

At fortifications of 0.4 ppm in liver, recoveries were 85% and 70%, while at fortifications of 1.0 ppm, recoveries were 46% and 62%.

The petitioner also reported high control values for acetamide in chicken liver ranging from 0.09 to 0.16 ppm (average of 5 samples = 0.11 ppm) (see M. Firestone review of 9/24/84). The petitioner has determined that the Lowest Limit of Reliable Measurement (LLRM) for analysis of acetamide in chicken liver is 0.4 ppm (note - the petitioner reports a LLRM of 0.77 ppm for analysis of acetamide in beef liver).

### Conclusions

- 1) Because of the very low recoveries at the 1.0 ppm fortification, RCB considers the method questionable for the determination of acetamide in liver.
- 2) Due to the determination of acetamide in reagent blanks by EPA's chemists, RCB is unable to determine the validity of the data on endogenous or ubiquitous acetamide in animal commodities. The petitioner should submit all raw data and calculation as requested in RCB's review of 12/6/84 (M. Firestone). These raw data should include analysis of solvents (i.e., acetone) used for residue extraction.

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3) The method for analysis of acetonitrile in milk and egg whites is inadequate due to extremely high reagent blank and control values. Thus, the determination of acetonitrile as a marker compound for acetamide in milk and eggs is not acceptable under FDA's SOM Policy.

cc:R.F., Circu, Reviewer, TOX, EAB, EEB, PP#0F2413/FAP#0H5275/  
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