



UNITED STATES ENVIRONMENTAL PROTECTION AGENCY

WASHINGTON, D.C. 20460

Analytical Chemistry Section
Building 402, ARC-East
Beltsville, Maryland 20705

OFFICE OF
PESTICIDES AND TOXIC SUBSTANCES

August 20, 1985

MEMORANDUM

SUBJECT: Analysis of Milk Samples for Acetamide

FROM: Dallas Wright, Jr., Chemist DPW.
Analytical Chemistry Laboratory

TO: Warren R. Bontoyan, Head
Analytical Chemistry Section

Samples of pasteurized and unpasteurized milk and distilled water were analyzed for acetamide using the Union Carbide method entitled, "Method of Analysis for Acetamide in Milk and Eggs (ACETAMIDE-NPD-Milk and Eggs)". The milk samples were spiked at 0; 0.1; and 0.5ppm, and the distilled water was spiked at 0 and 0.5ppm. The results of the analyses are summarized in Table 1.

In addition several samples were analyzed at ACL and ECL by low resolution GC/MS. Our conclusion, based on these analyses, is that there appears to be acetamide in the pasteurized and unpasteurized milk samples we tested. We base our conclusion on the following observations:

- 1) When scanning at the molecular ion for acetamide, the retention time of peaks in the samples matched that of the standard.
- 2) The peak shape of the samples and standard are similar.
- 3) Mass spectra taken from the peaks in the milk samples matched that of the standard.

- 4) An injection of a sample and standard made simultaneously showed only a single peak in the area of interest.
- 5) Samples of distilled water analyzed were free of acetamide indicating that there is no contamination in the laboratory.

The results of the GC/MS analysis are summarized in Table 2. A more precise identification of the peak in the samples could be made using high resolution GC/MS.

Because of all these observations we feel that, within the limitations of the techniques used, the peaks observed in the milk samples analyzed are probably acetamide.

Comments:

- 1) Pasteurized milk was purchased locally
- 2) Unpasteurized milk was obtained from USDA Beltsville, MD
- 3) Acetamide standard was purchased from Fisher Scientific Co. and recrystallized. The purity was determined by DSC.
- 4) The recoveries were very low in most of the samples. Since the purpose of this project was the qualitative identification of acetamide, there was no attempt made to determine why recoveries were low.

Table 1

Results of Recoveries of
Acetamide in Milk and Water

<u>Matrix</u>	<u>PPM Spiked</u>	<u>PPM Found</u>	<u>After Control adjustment</u>	<u>Net Recovery (%)</u>
Pasteurized Milk	Control	0.16	-	-
	Control	0.11	-	-
	0.1	0.17	0.03	30
	0.1	0.15	0.01	10
	0.5	.09	(-.05)	-
	0.5	.04	(-.10)	-
Raw Milk	Control	0.12	-	-
	Control	0.10	-	-
	0.1	0.19	0.08	80
	0.1	0.14	0.03	30
	0.5	0.24	0.13	26
	0.5	0.24	0.13	26
Distilled H ₂ O	Control	ND	-	-
	Control	ND	-	-
	0.5	0.04	.04	8
	0.5	0.06	.06	12

Table 2

<u>Sample</u>	<u>GC/MS Confirmation By SIM</u>	<u>GC/MS Confirmation Full Spectra</u>
Pasteurized Milk 0.5ppm Spike	Yes	Yes
Pasteurized Milk Control	Yes	Yes
Unpasteurized Milk 0.5ppm Spike	Yes	Yes
Unpasteurized Milk Control	Yes	Yes