# Flavor Threshold for Acetaldehyde in Milk, Chocolate Milk, and Spring Water Using Solid Phase Microextraction Gas Chromatography for Quantification

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The detection threshold of acetaldehyde was determined on whole, lowfat, and nonfat milks, chocolate-flavored milk, and spring water. Knowledge of the acetaldehyde threshold is important because acetaldehyde forms in milk during storage as a result of light oxidation. It is also a degradation product of poly(ethylene terephthalate) during melt processing, a relatively new packaging choice for milk and water. There was no significant difference in the acetaldehyde threshold in milk of various fat contents, with thresholds ranging from 3939 to 4040 ppb. Chocolate-flavored milk and spring water showed thresholds of 10048 and 167 ppb, respectively, which compares favorably with previous studies. Solid phase microextraction (SPME) was verified as an effective method for the recovery of acetaldehyde in all media with detection levels as low as 200 and 20 ppb in milk and water, respectively, when using a polydimethyl siloxane/Carboxen SPME fiber in static headspace at 45 °C for 15 min.

**Keywords:** *Acetaldehyde; milk; threshold; fat content; solid phase microextraction*

## INTRODUCTION

Storage and exposure to ultraviolet (UV) light are two factors that greatly influence off-flavor development in milk. UV light initiates a variety of chemical reactions that result in the increase in volatile compounds such as acetaldehyde, propanal, *n*-butanal, *n*-hexanal, dimethyl disulfide, and methional, often to levels above human threshold (*1*). These chemicals can lead to unwanted flavor changes in milk, previously described as sunlight, oxidized, activated, burnt, scorched, cabbage, and mushroom (*2*−*4*). The concentration of these chemicals is also increased by higher storage temperatures and with time (*3*, *4*).

Acetaldehyde is also a degradation product formed in the melt processing of poly(ethylene terephthalate) (PETE) (*5*, *6*). PETE is becoming an increasingly popular packaging choice for milk and beverage products. The migration of acetaldehyde from the container into the product is an issue to be explored in the use of PETE packaging in the food industry, particularly in the water industry, for which low acetaldehyde grades of PETE have been developed. Pure acetaldehyde possesses a pungent irritating odor, but at dilute concentrations it gives a pleasant fruity, green apple-like flavor. It occurs in a large number of natural food products such as yogurt and vinegar in concentrations up to 1000 mg/L (vinegar) (*7*). In the dairy industry, acetaldehyde is most often identified with fermented products, especially yogurt (5−40 mg/L) (*8*). Freshly pasteurized milk typically contains acetaldehyde in quantities around 10 ppb (*9*). Bills et al. determined the flavor threshold for acetaldehyde in low-fat (2%) milk as 800 ppb (*10*). The detection threshold can be defined as an energy level below which no sensation would be produced by a stimulus and above which a sensation would reach consciousness (*11*).

The objectives of this study were to determine (i) the flavor threshold for acetaldehyde in whole milk (3.25% milkfat), low-fat milk (2% milkfat), nonfat milk (0.5% milkfat), chocolate milk (3.25% milkfat), and spring water and (ii) if solid phase microextraction (SPME) is an effective method for acetaldehyde recovery.

## MATERIALS AND METHODS

Milk of various fat contents, chocolate-flavored milk, and spring water were analyzed. An untrained panel of 25 people and three-sample alternate forced choice test series were used for sensory analysis of all media (*11*). Quantification of acetaldehyde was done on all media using SPME coupled with gas chromatography (SPME-GC).

**Milk Processing.** Fresh raw milk was obtained from the Virginia Polytechnic Institute and State University (Blacksburg, VA) dairy farm. Milk was prewarmed to 55 °C and separated (C 292G) into cream and skim milk using a pilot plant separator (Elecrem separator, model 1G, Bonanza Industries, Inc., Calgary, AB). Milk was standardized at various fat contents (0.5, 2, and 3.25%) by adding cream to skim milk in appropriate proportions.

Milk was pasteurized at 63.3 °C for 30 min in a batch pasteurizer (Creamery Package Mfg. Co., Chicago, IL) and cooled to 25 °C. Milk (2 and 3.25% milkfat) was homogenized at 13.8 MPa (first stage) and 3.4 MPa (second stage) on a laboratory scale homogenizer (APV Gaulin, model 15 MR,

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Everett, MA), and stored at 4 °C in appropriate containers until needed. The fat content of milk was determined each time according to the Babcock method (*12*).

**Chocolate Milk.** Chocolate milk was formulated by adding 6.35% sucrose, 1.24% cocoa, and 0.112% stabilizer to whole milk (3.25% milkfat). Formulated milk was pasteurized at 81 °C for 30 min in a laboratory scale batch pasteurizer and cooled to 25 °C. Milk was homogenized at 13.8 MPa (first stage) and 3.4 MPa (second stage) on a laboratory scale homogenizer (APV Gaulin, model 15 MR) and stored at 4 °C in appropriate containers until needed. The fat content of milk was determined according to the Babcock method (*12*).

**Water.** Kroger spring water (Kroger Co., Cincinnati, OH) was used for all analyses.

**Preparation of Acetaldehyde-Spiked Samples.** Acetaldehyde (≥99.5%) was obtained from Fisher Scientific (Cincinnati, OH). Milk and water samples were used for acetaldehyde threshold testing within one week of processing. All samples were spiked volumetrically with 10 levels of acetaldehyde, in geometric progression of concentration steps. Concentration steps for milk were 0, 200, 400, 800, 1200, 1600, 3200, 6400, 9600, and 12800 ppb; for chocolate milk were 0, 400, 800, 1600, 3200, 6400, 9600, 12800, 19200, and 25600 ppb; and for water were 0, 2.5, 5, 10, 20, 40, 80, 120, 160, and 320 ppb. The acetaldehyde-spiked solutions were thoroughly mixed and stored in sealed amber glass containers at 4 °C until sensory testing.

**Quantification of Acetaldehyde in Milk and Water.** The concentration of acetaldehyde in spiked milk, chocolate milk, and water samples was determined. Eight milliliters of sample, 5 $\mu$L of internal standard solution (10000 $\mu$g/mL 4-methyl-2-pentanone; Fisher Scientific), and a micro stirring bar were placed in a 20 mL amber glass container capped with a black Viton septum (Supelco, Inc., Bellefonte, PA). Samples were held at 4 °C until analyzed the next day. The vial septum was prepierced in the center, if required, with a sharp thin probe just before analysis to facilitate insertion of the SPME needle. The SPME fiber (Supelco, Inc.) was exposed, with the end of the fiber ~1 cm above the surface of the sample. The SPME unit was clamped in this position, and magnetic stirring was begun. Acetaldehyde was adsorbed on a (PDMS/Carboxen) SPME fiber (Supelco, Inc.) in static headspace at 45 °C for 15 min (*13*).

After exposure was completed, the SPME unit was withdrawn from the septum and inserted into the injector port of the gas chromatograph. The injector temperature was 250 °C. The fiber was left in the injection port for 20 min before removal, minimizing the possibility of carry-over. Acetaldehyde was thermally desorbed in the injector port of a Hewlett-Packard gas chromatograph (model 5890A, Hewlett-Packard, Avondale, PA) equipped with an HP 5895A ChemStation and a flame ionization detector. Separation was completed on an HP-5 capillary column (25 m × 0.32 mm, 1.05 $\mu$m) (Supelco, Inc.) with helium flow rates of 1.0 mL/min. The temperature program was 35 °C for 1 min, raised at 8 °C/min to 180 °C, and then after 1 min raised to 250 °C at 14 °C/min, with final time of 3 min. All injections were made in the splitless mode. Acetaldehyde identification and quantification were based on retention time and peak area results for the standard solutions using the method of additions technique and an internal standard (*13*).

**Sensory Testing.** Sensory testing was done on all media to determine the acetaldehyde threshold. A three-sample alternate forced choice test series was used with a panel of 25 people. The study was repeated twice to verify that thresholds were within 20% of each other (*11*).

Each panelist was presented first with a warm-up sample at a suprathreshold level of acetaldehyde (12800 ppb for milk, 320 ppb for water, and 25600 ppb for chocolate milk) to familiarize the panelists with the expected taste of discrimination. The panelists were requested to complete the human subjects' consent form while resting from the warm-up sample.

Panelists were presented with 10 three-sample triangle sets (7 °C) in ascending concentration series. Triangle sets were presented on three trays, with three, three, and four sample sets, respectively. Panelists were informed to choose the sample that tasted "different" within each three-sample set. If subjects responded negatively at the highest level or showed correct choices at even the lowest levels, the individuals were retested to confirm the highest or lowest concentrations of detection (*11*).

Panelists were instructed to rinse with warm spring water between three-sample sets and were allowed to rest between trays to prevent fatigue. Panelists were not informed of the ascending concentration characteristics of the samples, although they might have acquired the knowledge by participating on multiple panels. Each three-sample set included two samples of unspiked medium (milk, chocolate milk, or water) and one acetaldehyde-spiked sample at the given concentration. Each sample was coded with a three-digit number to remove bias, and the position of the spiked sample within the three-sample set was randomized.

A panel of 25 people was randomly selected for testing of each milk product and water. Panelists were seated in individual sensory booths. Each panel was replicated twice.

**Data Evaluation.** The threshold for acetaldehyde was interpreted in two ways: geometric mean threshold and logistic regression. The threshold of individual panelists was determined by taking the geometric mean of the last incorrect concentration and the first correct concentration (when all subsequent choices were correct) for each product (*11*). Geometric mean is the antilog of the mean of the log values for the last incorrect concentration and the first correct concentration step. Group threshold was calculated by taking the geometric mean of the individual panelist's thresholds. Sensory analysis was duplicated to ensure that group thresholds were within 20% of each as specified by Lawless and Heymann (*11*).

Logistic regression is a technique for predicting the probability of "success" as a function of some predictor variable. In this context, the concentration of the acetaldehyde in the medium ($x$) is the predictor variable and a correct identification of a spiked sample is a success.

Let $x$ = the concentration of acetaldehyde in the medium and let $p(x)$ = the probability that a panelist correctly identified a sample that contained acetaldehyde. The logistic regression model is

$$p(x) = 1/[1 + \exp(-\alpha - \beta x)]$$

where $\alpha$ and $\beta$ are parameters that are estimated from the data. Data were analyzed using SAS (*14*).

## RESULTS AND DISCUSSION

Acetaldehyde detection, through sensory analysis, can be measured in two different ways. The traditional approach of geometric mean determination was used when the concentration of acetaldehyde, below which the subjects lack the sensitivity to detect the acetaldehyde in a sample, can be determined. An alternative approach is logistic regression, where the probability of "success"—the probability that acetaldehyde-spiked samples will be identified correctly—as a function of acetaldehyde concentration in the medium can be predicted (*13*).

The use of SPME as the headspace extraction method for acetaldehyde in all media was verified as effective.

**Geometric Mean Approach.** Table 1 reports the group threshold of acetaldehyde in the different media. Thresholds for acetaldehyde in milk of different fat contents were very similar, with a difference of only 100 ppb. Miyake and Shibamoto reported that fat is one of the major carriers of carbonyl compounds as well as acetaldehyde; therefore, milk with a higher fat content should contain more acetaldehyde. Although a higher threshold value for acetaldehyde was found in whole milk as compared to nonfat milk in our study, the

**Table 1. Thresholds for Acetaldehyde in Unflavored Milk (Nonfat, Low-Fat, Whole), Chocolate-Flavored Milk, and Spring Water As Determined by the Geometric Mean Approach**

| medium[a] | group threshold[b] (ppb) | min/max individual thresholds (ppb) | % variation between replications |
|---|---|---|---|
| nonfat milk | 3939 | 14/23406 | 12.8 |
| low-fat milk | 4020 | 283/23406 | 3.8 |
| whole milk | 4040 | 14/23406 | 2.5 |
| chocolate milk | 10048 | 566/46757 | 2.1 |
| spring water | 167 | 7/784 | 12.6 |

[a] Nonfat milk (0.5% milkfat), low-fat milk (2% milkfat), whole, and chocolate milk (3.25% milkfat). [b] Twenty-five panelists for each of two replications.

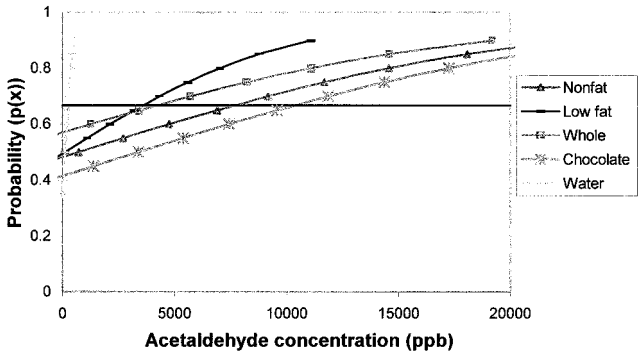


**Figure 1.** Probability of correct identification of acetaldehyde-spiked sample using logistic regression when $p(x) = 1/[1 + \exp(-\alpha - \beta x)]$.

threshold values were not significantly different ($p < 0.05$) (*15*). On the basis of these results, fat content does not play a major role in the threshold of acetaldehyde in milk. Bills et al. found flavor threshold levels for acetaldehyde in low-fat milk (2% milkfat) at 5 °C of 800 ppb. This threshold is substantially lower than what was observed in our study because they used trained panelists (*10*).

Chocolate milk shows a group threshold for acetaldehyde of 10048 ppb. These data compare well with studies done on strawberry-flavored milk with a threshold of 11700 ppb (*10*). Panelists were not able to identify acetaldehyde-spiked samples in chocolate milk at concentrations that were easily detected in unflavored milk. This could be ascribed to masking of acetaldehyde flavor by chocolate flavor (*10*).

Spring water showed a group flavor threshold of 167 ppb. This value compares well with previous studies done on flavor threshold of acetaldehyde in water. Over the past 30 years various research studies have reported flavor thresholds for acetaldehyde in water ranging from 22 to 1300 ppb (*16*−*18*). Nijssen et al. also reported an odor threshold used by the mineral water industry as ranging from 20 to 40 ppb (*19*).

Table 1 also shows great variability in individual thresholds of panelists. Lawless and Heymann confirm that individuals have very different abilities to detect flavor compounds, with some subjects "blind" (unable to detect) to certain flavors. It is important that such individuals are also included in group threshold tests because they are part of the general public and will also consume the product (*11*).

Because individual thresholds vary substantially, which can influence the group threshold significantly, valid threshold measurements require group threshold values with <20% variability between two replications

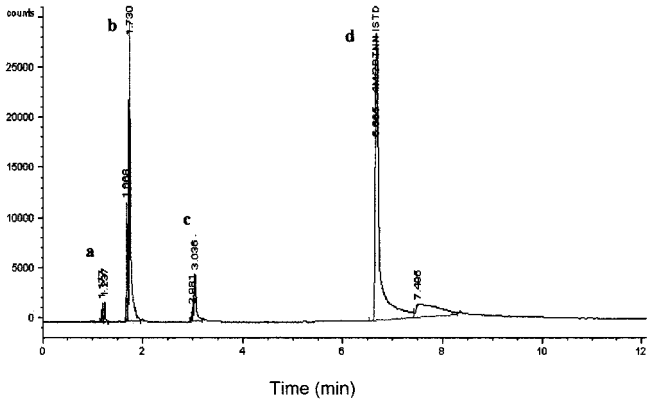


**Figure 2.** Acetaldehyde in milk medium (3.25% milkfat) at 200 ppb: (a) acetaldehyde; (b) acetone; (c) *n*-butanone; (d) 4-methyl-2-pentanone (internal standard).

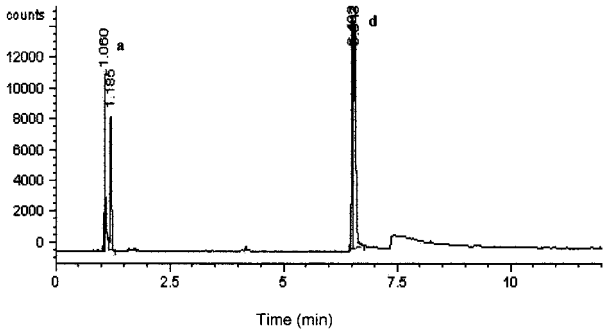


**Figure 3.** Acetaldehyde in water medium at 20 ppb: (a) acetaldehyde; (d) 4-methyl-2-pentanone (internal standard).

(*11*). In this study variability in thresholds between replications ranged from 2.1 to 12.8% (Table 1).

**Logistic Regression.** Logistic regression and the geometric mean approach measure detection in two different ways. The geometric mean is based on the information of where the subjects' detection abilities break down, and logistic regression predicts where a certain percentage of the panelists will correctly identify the acetaldehyde-spiked milk.

Figure 1 shows the probability of correct identification of acetaldehyde-spiked sample in all media. Lawless and Heymann suggest an arbitrary level of 50% above chance guessing for determining threshold when an alternative approach, such as logistic regression, is used (*11*). This level is calculated by making use of Abbotts's formula (*20*):

$$\text{adjusted proportion correct} = (\text{observed proportion} - \text{chance})/(1 - \text{chance})$$

$$0.5 = (0.667 - 0.33)/(1 - 0.33)$$

The 50% above chance guessing for the triangle test thus requires 66.7% correct identification. For example, in low-fat milk, the logistic predicts that at a concentration of 3570 ppb of acetaldehyde, 66.7% of the panelists should be able to identify the milk that is spiked with acetaldehyde.

The probability of correct identification of acetaldehyde-spiked sample in all media at the threshold levels found when using the geometric mean approach is shown in Table 2. This means that at the threshold level for acetaldehyde in low-fat milk (4020 ppb) 68.7% of the panelists used would be able to correctly identify an acetaldehyde-spiked sample. Thresholds calculated us-

**Table 2. Predicted Concentration of Acetaldehyde at Probability of 66.7% Using Logistic Regression As Compared to Group Thresholds Using Geometric Means**

| medium | acetaldehyde threshold[a] at $p(x) = 0.667$ (ppb) | geometric mean threshold[b] (ppb) | probability (%)[c] for group thresholds (ppb) |
|---|---|---|---|
| nonfat milk | 7656 | 3939 | 58.0 |
| low-fat milk | 3570 | 4020 | 68.7 |
| whole milk | 4174 | 4040 | 66.4 |
| chocolate milk | 10334 | 10048 | 66.1 |
| spring water | 252 | 167 | 55.8 |

[a] Calculated at $p(x) = 0.667$ from logistic regression with $p(x) = 1/[1 + \exp(-\alpha - \beta x)]$. [b] Calculated using geometric mean. [c] Probabilities calculated with $p(x) = 1/[1 + \exp(-\alpha - \beta x)]$ when using group thresholds obtained from geometric mean approach.

ing logistic regression compared very well with thresholds from geometric mean calculation for low-fat, whole, and chocolate-flavored milk, whereas nonfat milk and spring water showed thresholds with fair comparisons.

The geometric mean approach is easily influenced by incorrect responses by panelists as a result of fatigue or sensory adaptation. Logistic regression might therefore be a valuable method in threshold determination because it does not rely as strongly on individual responses.

**SPME.** Various analytical methods have been used over the years for the detection of acetaldehyde and other volatile flavor compounds in foods and beverages (*1*, *13*, *21*). Dynamic headspace collection of volatiles coupled with gas chromatography is a sensitive technique; however, it is time-consuming, especially in cleaning equipment and glassware, and it involves relatively expensive equipment. SPME-GC is a solventless extraction technique that is simple, relatively cheap, and effective for isolating and detecting low levels of flavor compounds in foods and beverages (*21*). Marsili used SPME to isolate various aldehydes in nonfat and low-fat milk (*13*).

In our study, SPME was effective for the isolation and concentration of acetaldehyde from the headspace of the milk or water. Acetaldehyde was detected at concentrations as low as 200 and 20 ppb in milk and water media, respectively, when using a 75 $\mu$m PDMS/Carboxen fiber and an HP-5 capillary column (25 m $\times$ 0.32 mm, 1.05 $\mu$m).

Acetaldehyde showed a double peak in both media. This is most likely due to the volatility of acetaldehyde (bp 21 °C), which might prevent it from being optimally focused on the 1.05 $\mu$m column used (R. Shirey, Supelco, Bellefonte, PA, personal communication, 1999). Cryogenic trapping at the capillary column inlet or the use of a thicker film stationary phase capillary column might have eliminated the double peaks. Internal standard quantification was based on summing peak areas of double peaks. In a milk medium the retention times of the peaks are 1.175 and 1.236 min, whereas they shift to 1.060 and 1.185 in a water medium. This shift is unexpected but was not researched further in this study.

Tables 3−5 show recovery amounts of acetaldehyde in water, milk of all fat content, and chocolate milk, respectively. Recovered amounts in whole milk samples are higher than the spiked concentrations in almost all cases. This could be due to the fact that acetaldehyde also is present naturally in milk at low concentrations (*9*). It was not detected in control milk samples, with a limit of detection estimated at 100 ppb. Recovered and spiked amounts for water compared very well considering the low concentrations at which acetaldehyde was spiked and analyzed.

**Table 3. Acetaldehyde Recovery from Spring Water Using SPME Concentration Technique**

| spiked concn (ppb) | recovered concn (ppb $\pm$ $\sigma$)[a] |
|---|---|
| 20 | 18 $\pm$ 9 |
| 40 | 35 $\pm$ 11 |
| 80 | 84 $\pm$ 9 |
| 120 | 112 $\pm$ 13 |
| 160 | 173 $\pm$ 21 |
| 320 | 314 $\pm$ 18 |

[a] Mean ($N = 3$) $\pm$ standard deviation.

**Table 4. Acetaldehyde Recovery from Whole Milk (3.25% Milkfat), Low-Fat Milk (2% Milkfat), and Nonfat Milk (0.5% Milkfat) Using SPME Concentration Technique**

| spiked concn (ppb) | recovered concn (ppb $\pm$ $\sigma$)[a] | | |
|---|---|---|---|
| | whole milk | low-fat milk | nonfat milk |
| 200 | 280 $\pm$ 70 | 171 $\pm$ 91 | 220 $\pm$ 60 |
| 400 | 511 $\pm$ 89 | 430 $\pm$ 85 | 347 $\pm$ 86 |
| 800 | 904 $\pm$ 106 | 857 $\pm$ 69 | 876 $\pm$ 94 |
| 1200 | 1169 $\pm$ 94 | 1296 $\pm$ 108 | 1310 $\pm$ 130 |
| 1600 | 1492 $\pm$ 110 | 1677 $\pm$ 86 | 1517 $\pm$ 124 |
| 3200 | 3331 $\pm$ 118 | 3101 $\pm$ 111 | 3307 $\pm$ 114 |
| 6400 | 6351 $\pm$ 102 | 6371 $\pm$ 108 | 6358 $\pm$ 90 |
| 9600 | 9792 $\pm$ 120 | 9688 $\pm$ 90 | 9564 $\pm$ 110 |

[a] Mean ($N = 3$) $\pm$ standard deviation.

**Table 5. Acetaldehyde Recovery from Chocolate Milk (3.25% Milkfat) Using SPME Concentration Technique**

| spiked concn (ppb) | recovered concn (ppb $\pm$ $\sigma$)[a] |
|---|---|
| 400 | 490 $\pm$ 89 |
| 800 | 730 $\pm$ 146 |
| 1600 | 1749 $\pm$ 94 |
| 3200 | 3337 $\pm$ 110 |
| 6400 | 6250 $\pm$ 178 |
| 9600 | 9430 $\pm$ 150 |
| 12800 | 13600 $\pm$ 230 |
| 19200 | 19380 $\pm$ 190 |
| 25600 | 25560 $\pm$ 104 |

[a] Mean ($N = 3$) $\pm$ standard deviation.

Calibration was based on peak area results for the standard solutions using the method of additions technique and an internal standard (4-methyl-2-pentanone) (*13*). The calibration curves for acetaldehyde in all products showed a linear relationship between acetaldehyde concentration and area ratio of peaks with correlation coefficients of 0.996, 0.999, 0.997, 0.999, and 0.998 for water and whole, nonfat, low-fat, and chocolate-flavored milk, respectively. Linear regression coefficients (intercept and slope) for whole milk, low-fat milk, and nonfat milk were $y = 0.985x + 64$, $y = 0.986x + 43$, and $y = 0.995x + 29$, respectively. Linear regression coefficients for water and chocolate-flavored milk were $y = 1.00x - 67$ and $y = 0.996x - 0.2$, respectively.

To conclude, the acetaldehyde thresholds in spiked milk of various fat contents were not significantly different from one another, with thresholds ranging from 3939 to 4040 ppb. These levels were significantly higher than those obtained in milk packaged in PETE containers and exposed to fluorescent lights for 18 days, indicating subthreshold development of acetaldehyde (due to light oxidation and migration from PETE) (*22*). Chocolate-flavored milk and spring water showed thresholds of 10048 and 167 ppb, respectively, which compare favorably with previous studies. SPME proved to be an effective method for the recovery of acetaldehyde in

milk, chocolate milk, and water. Levels as low as 200 and 20 ppb for milk and water media, respectively, have been quantified.

## ABBREVIATIONS USED

SPME-GC, solid phase microextraction gas chromatography; PDMS, polydimethyl siloxane; UV, ultraviolet; PETE, poly(ethylene terephthalate).

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