TELEDYNE TEKMAR
A Teledyne Technologies Company

Evaluating Food Products for Furan and Other Volatile Organic Compounds

Application Note

Abstract

The US Food and Drug Administration (FDA) published the results of a survey of furan in canned and jarred foods that undergo heat treatment in 2004. In the same year, the European Food Safety Authority agreed with the findings of the FDA report and determined that more research was needed on the study of furan in food.

One of the techniques used by the FDA for furan in the foods that were evaluated was static headspace. This poster presents a comparison of the static method to a dynamic headspace method in different foods such as coffee and meat.

Introduction

Furan is an aromatic heterocyclic compound of four carbon atoms and one oxygen atom. It has been found not only in heat processed foods, but also one of numerous compounds, including dioxins, produced during incineration of waste. Because of its association with dioxins, the National Toxicology Program has been evaluating furan for potential carcinogenetic properties and found it to have cytotoxic and carcinogenetic effects in laboratory animals[1].

In 2004 the FDA published the results of a survey of furan in canned and jarred foods that undergo heat treatment, which has also been updated in 2005, 2006 and 2008[2]. The European Food Safety Authority, agreeing with the FDA, determined that more research was needed on furan in food[3]. The FDA developed a method using static headspace and the standard addition method requiring 7 samples analysis for each food product sample[4].

This study presents the data for performing the current FDA static headspace standard addition method for the detection of furan in food along with a dynamic headspace method. Other volatile organic compounds can also be determined simultaneously with the furan assay.

A Teledyne Tekmar HT3 Headspace Analyzer was used for this poster in the both the static and the dynamic mode along with a Thermo Focus GC/DSQ II MS for the quantitation of furan in food. An environmental column was used for the quantitation of furan, and to assist in the detection and preliminary identification of additional volatile organic compounds (VOCs) released from the samples. These VOCs, including benzene and toluene can be quickly identified with the environmental column and confirmed when an USEPA Method standard is included in the analysis.

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Experimental-Instrument Conditions

HT3™ Headspace Instrument Parameters			
Static		Dynamic	
Variable	Value	Variable	Value
Valve Oven Temp	110°C	Valve Oven Temp	130°C
Transfer Line Temp	130°C	Transfer Line Temp	150°C
Platen/Sample Temp	60°	Platen/Sample Temp	60°
Sample Equil Time	5.00 min	Sweep Flow Rate	50 mL/min
Mixing Time	25.00 min	Sweep Flow Time	10.00 min
Mixing Level	Level 9	Dry Purge Time	2.00 min
Pressurize	13 psig	Dry Purge Flow	100 mL/min
Loop Fill Pressure	10psig	Desorb Temp	250°C
Inject Time	2.00 min	Desorb Time	1.00 min
		Trap Material	#9

Table 1: Static and Dynamic HT3™ Parameters

Thermo Focus GC/DSQ™ II MS Parameters	
Column	Restek Rtx® VMS, 20m, 0.18mm ID, 1µm; Constant Flow 0.90 mL/min
Oven Program	35°C for 4 min; 16°C/min to 160°C, 25°C/min to 250°C hold for 4 min, run time 19.1 min
Inlet:	Split Flow 45 mL/min, Temperature 220°C, Helium Carrier Gas
MS	Source and Transfer Line Temp 230°C, Full Scan 35.0 *m/z* to 270.0 *m/z* Scan Rate 1492.11

Table 2: Thermo Focus GC/DSQ II MS Parameters

Sample Preparation

For this study three types of ground coffee, a decaffeinated blend, a regular blend and a specialty house blend were obtained. The ground coffees were tested for furan by weighing the ground coffee beans into 22mL headspace vials and adding water. The coffee was also tested after brewing by following the package directions. The brewed coffees were tested for furan by placing 10mL of the brewed coffee into headspace vials for the static method. 2mL of the brewed coffee was placed into headspace vials for the dynamic headspace method.

Along with the coffee samples, chicken and beef Stage 1 baby food meats, and a Stage 2 baby food mix of sweet potatoes and turkey were obtained. These were weighed into 22mL headspace vials and the appropriate amount of water or saturated sodium chloride solution added.

Separate stock standard solutions of furan and furan-d4, the internal standard (IS), were prepared by pipeting 50µL of the standards into separate 22mL headspace vials containing 20.0mL of purge and trap methanol and weighing the amount of standard added. The stock standard solutions were further diluted in water to produce working standards close to the expected furan concentrations in the food samples.

A furan/furan-d4 calibration curve from 1 to 250 times the expected food furan concentration was prepared to determine the approximate concentration for the standard addition method. The furan mass 68 *m/z*/furan-d4 mass 72 *m/z* ratio was plotted versus the furan concentration in ng. The food samples were then analyzed singularly to estimate the ng furan in the food. This estimated ng concentration was then defined as x.

Seven preparations of each food sample were prepared by weighing or measuring equivalent sample amounts into seven vials. These vials were spiked with both the furan working standard and the furan–d4 IS working standard as follows. The furan-d4 working IS was added to all seven vials at approximately 2x.

The furan working standard was added to only 4 of the vials. Three vials were not spiked and are 0x. Two vials were spiked at 0.5x, one vial was spiked at 1x and the remaining vial was spiked at 2x. The vials were sealed and analyzed with the static headspace method parameters. One set of food was analyzed with the static headspace method following the FDA method. A second set of food was analyzed with the dynamic method to compare the furan results to the static method.

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Results

The peak area of the furan mass 68 *m/z* and the furan-d4 mass 72 *m/z* for vials were calculated with the Thermo EnviroLab Forms 3.0 software. The ratio of the furan mass 68 m/z peak area to the furan-d4 mass 72 m/z peak area for each of the 7 vials per sample was calculated. This ratio was then plotted versus the concentration of furan added to each of the 7 vials per sample.

The linear regression of the 7 data points for each sample was calculated. The ng furan for the sample was calculated by solving the linear regression equation for y=0, the y intercept. Figure 1 is a typical plot with the linear equation for the standard addition plot.

The ppb furan was calculated by dividing the ng furan found in the food sample by the weight or volume of sample used for analysis. The ppb furan for each food type is presented in Table 3 for both the static and the dynamic concentration.

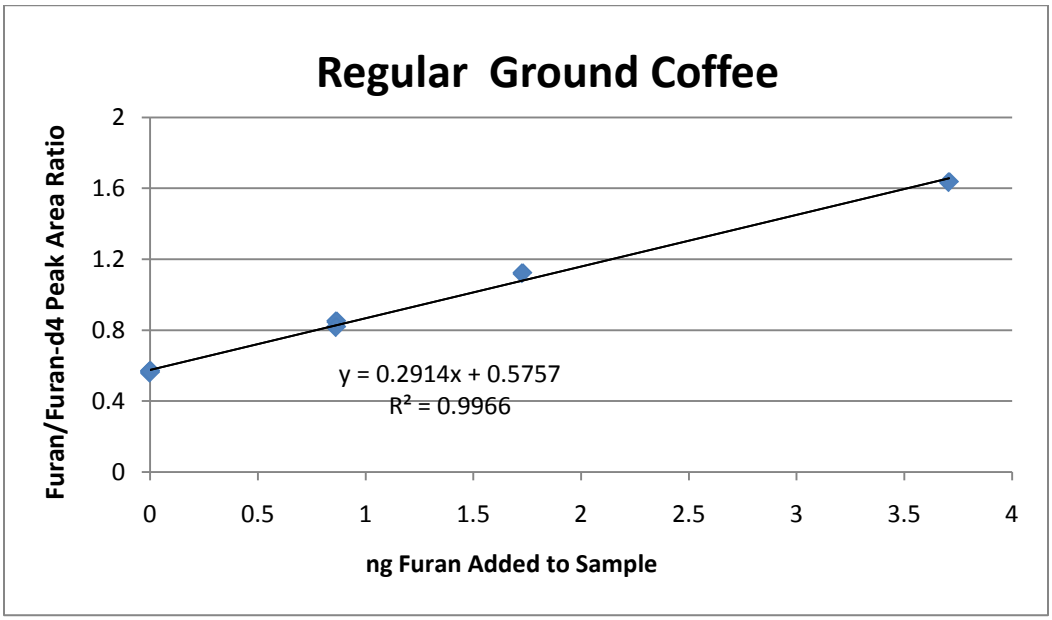

Figure 1: Standard Addition Curve Data for Regular Ground Coffee of the Furan/Furan-d4 Peak Area Ratio versus the Concentration in ng

		Static		Dynamic	
Coffee		Sample Amt	ppb Furan	Sample Amt	ppb Furan
Ground	Decaffeinated	1.5 g	1475.6	0.15 g	950.4
	Regular	1.5 g	1304.8	0.15 g	581.7
	Specialty	1.5 g	3309.7	0.15 g	2513.1
Brewed	Decaffeinated	10 mL	4.5	2 mL	4.9
	Regular	10 mL	10.2	2 mL	7.1
	Specialty	10 mL	22.5	2 mL	20.1
Baby Food					
Stage 1	Chicken and Chicken Broth	5 g	3.9	2 g	0.7
	Beef and Beef Broth	5 g	2.0	2 g	1.5
Stage 2	Sweet Potato and Turkey	1.5 g	25.1	0.5 g	7.8

Table 3: Calculated ppb Furan Concentrations for the Various Food Products Evaluated

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Conclusions

Various foods were tested with the current FDA static headspace for furan utilizing a Teledyne Tekmar HT3. The dynamic headspace option of the HT3, where the volatile compounds are trapped and concentrated to be rapidly desorbed to the GC/MS was also used for the quantitation of furan in food.

The FDA method lists a HP-Plot Q column while this poster was performed using a Restek RTx-VMS environmental application column. The FDA method uses a pressure balance headspace system while this poster used the HT3 which is a loop filled headspace instrument. A Thermo Focus GC with DSQII mass spectrometer was used in place of the GC/MS system listed in the FDA method.

The HT3 loop filled headspace instrument in the static mode with the environmental column and the Thermo GC/MS system performed exceptionally well for the detection and quantification of furan following the FDA standard addition method. The environmental column provided excellent separation and detection of not only the furan but other volatile food and beverage compounds of concern.

The various foods were also analyzed with the dynamic option of the HT3 instrument. The target compound, furan was well trapped with the #9 trap for quantitative desorption to the GC/MS. The dynamic headspace procedure allowed smaller quantities of food products to be used for the analysis. The dynamic method provided similar results to the current static headspace FDA standard addition method.

References

1. Furan in Food, Thermal Treatment: Request for Data and Information, Federal Register, 69 FR 25911 May 10, 2004

2. Exploratory Data on Furan in Food: Individual Food Products, US Food and Drug Administration, updated September 21,

3. Report of the Scientific Panel on Contaminants in the Food Chain on Provisional Findings on Furan in Food, Adopted on 7 December 2004, The EFSA Journal (2004) 137, 1-20, http://www.efsa.eu.int

4. 2009 Determination of Furan in Foods, US Food and Drug Administration, October 27, 2006

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