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Furan determination in jarred baby food purchased from the Spanish market by headspace gas chromatography-mass spectrometry

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Abstract

The aim of this paper is to offer a method based on headspace gas chromatography-mass spectrometry technique (US FDA 2006) in-house validated and use to estimate furan concentrations in jarred baby food samples purchased from the Spanish market. The validation was performed according to ISO 17025 and EFSA requirements and the results obtained (LOD 0.05 µg.kg^{-1}; LOQ 4 µg.kg^{-1}, lowest validated level; RSD% 3.1-10.5; recoveries 85.4-101.5%) confirm that this method is fit for the routine analysis of furan in jarred baby food control. Furan was analysed in 39 different baby-food samples and the mean levels varied between 64.6 µg.kg^{-1} (rice and chicken samples) and ≤ LOQ (fruit-based samples). The mean concentration values found for the different matrices were 5.0, 37.8, 25.2, 33.8 and 30.5 µg.kg^{-1} for fruit, vegetables, meat/vegetables, fish/vegetables and dairy-containing baby foods, respectively. According to the statistical analyses, fruit-based baby food samples had significantly lower concentrations of furan. Mean values for the other matrices were at least five times higher, and this is in accordance with the levels reported in other studies.

Keywords: furan; baby-food; headspace-gas chromatography-mass spectrometry.

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Introduction

Furan is a lipophilic contaminant which is formed during the heating processes used for the manufacture of foods. It is an aromatic heterocyclic compound, highly volatile; which was already detected, although at low concentration levels in the 1960s in a great variety of heat-processed foods (canned meat, bread, coffee, hydrolyzed proteins…) (Stoffelsma et al., 1968; Persson et al., 1973).

Experimental models multiple precursors and alternative routes for the furan formation have been proposed: thermal oxidation of ascorbic acid, non-enzimatic browning involving reducing sugars only (Strecker degradation) or together with amino acids [Maillard reactions (Maga 1979), polyunsaturated fat acids oxidation (Locas et al., 2004; Becalski et al., 2005; Märk et al., 2006)].

In 2004 US Food and Drug Administration (US FDA 2004b) reported the presence of this compound in a great variety of thermally treated food, such as meat, vegetables, and fruits, canned sauces in tins or jars especially including baby-food. The presence of furan, classified as possibly carcinogenic to human (Group 2B) (IARC 1995) in food has been confirmed by different laboratories (Reinhard et al., 2004; Becalski et al., 2005; Heppner et al., 2007)

The scientific panel on contaminants in the food chain from the European Food Safety Authority (EFSA 2004) considered furan in foodstuff as an urgent matter and made a scientific report about its presence that was presented in Brussels. The report concluded that it was necessary to generate reliable data of the furan content in products commercialized within the European Community to create a good database and study the risk of furan toxicity. In 2007, the European Commission recommended to the members states to follow the presence of furan in thermally treated food throughout 2007-2008 (EC 2007).

As an answer to the Commission and EFSA recommendation, our laboratory taking into account the lack of data available in Spain, decided to carry out a market surveillance targeted on foods intended to be consumed by infants, since they are the most susceptible consumer group (Suk et al., 2003) and because relatively high amounts of furan were previously detected in commercial baby-food (Crews et al., 2007; Morehouse et al., 2008).

The determination of furan is very challenging due to its high volatility, low molecular weight, and its presence in low concentrations in foodstuff. It is very important to have a suitable method to analyze this compound. The most used analytical technique is

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headspace extraction combined with gas chromatography-mass spectrometric determination (Nyman et al., 2006; Nyman et al., 2008; Bianchi et al., 2006; Zoller et al., 2007; Vranová et al., 2007). Our laboratory used the method proposed by FDA (US FDA 2006) as reference, with slight modifications.

The sample (purification was not necessary) was weighted in a headspace vial and placed in the static headspace autosampler, heated at 80º C for 30 min and determined by CG-MS in SIM mode, using furan-d4 as an internal standard.

Experimental

Reagents and apparatus

Furan (purity ≥ 99%) and d_4 -Furan (isotopic purity ≥ 98%) were provided by Sigma-Aldrich. Methanol (HPLC grade) from Merck. Ultrapure water was prepared using a Direct-Q system (Millipore).

The following equipment was used: Gas chromatograph (6890 Agilent) equipped with a mass spectrometer (5973 Agilent) and a Headspace injector (7694 Agilent). The chromatographic capillary column was a HP-PLOT Q by J&W Scientific (15 m x 0, 32 mm x 20 µm).

20 ml headspace vials with PTFE septa (Agilent Technologies) were used. To prevent the presence of contaminants in the vials, they were heated in a muffle oven (Heraeus) at 400º C for 1 hour before being used.

The stock solutions were prepared and transferred using microsyringes (Hamilton Gastight®).

Standards preparation

Stock furan and d_4-furan solutions were prepared in methanol with a minimum concentration of 3000 $mg.l^{-1}$ and stored in a freezer for no longer than one week.

From the stock solutions, intermediate solutions of furan and d_4-furan were prepared (50 µl to 50 ml methanol) and from these, working solutions in water in the in the range 4 - 400 $\mu g.l^{-1}$ for furan and at 10 $\mu g.l^{-1}$ for d_4-furan, approximately. The precise concentrations were calculated from the stock solutions. All these processes were performed in an ice bath to avoid losses due to the volatilization of furan.

Commercial samples for the study

Thirty nine jarred baby food samples, corresponding to three different commercial brands, and including a wide range of different matrices were purchased in retail food

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stores of the Spanish market. The samples were stored unopened and refrigerated, until they were analyzed. The main ingredients of the samples analyzed are shown in Table 2.

Sample preparation

A 5 g aliquot of the homogenised sample (homogenisation was performed under cold conditions) was weighted in a headspace vial, diluted with 5 ml of purified water and the internal standard was added at the same concentration as used in the calibration curve. All these processes were performed in an ice bath. The vials were hermetically sealed as fast as possible to avoid analyte losses and were shaken for 1 min until a homogeneous aspect was obtained. All the samples were twice analyzed, following the procedure already described. Additionally, a triplicate for each sample was prepared spiked with furan at a concentration of 100 $\mu g.l^{-1}$ in order to be used as a quality control sample.

HS-GC-MS analysis

Gas chromatographic conditions as well as (split ratio of 1:10) MS conditions were the same as those described in the US FDA 2006 method. The headspace operating conditions were as follows: oven temperature at 80º C with an equilibrium time of 30 min (stirring), loop at 95º C, transfer line at 110º C and an injection time of 1 min.

Selected ion monitoring (SIM), ions m/z 68, 39 (furan), 72 (furan d-4). Chromatograms for furan and d_4- furan standards and for a baby food sample are presented in figure 1.

(Figure 1)

Validation

In-house validation was carried out following the standard protocol from our laboratory, which is based on ISO 17025 (2005). The following parameters were assesed: specificity, linearity, detection and quantification limits, precision and accuracy (expressed as recovery).

Specificity was tested verifying the lack of response at the retention time of the analyte by analysing in triplicate a blank sample. A response was considered to be present if it was ≥1/5 the value of the quantification limit.

Linear regression calibration was performed by plotting the relative response of furan (m/z 68) and d_4-furan (m/z 72) against the furan concentration in $\mu g.l^{-1}$. An average calibration curve was calculated from three curves obtained in different days, each one at six concentration levels in the range 4 to 400 $\mu g.l^{-1}$. Linearity was established by

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estimating the relative standard deviation of the mean slope, according to the following equation:

$$Cm\% = 100 \times \frac{Sm}{m}$$

Where *Sm* is the standard deviation of the mean slope and *m* is the slope of the mean calibration curve. Values of *Cm* ≤ 5 % are considered satisfactory. The correlation coefficient of the linear regresión was also calculated (≥ 0.996).

Limits of quantification (*LOQ*) and detection *(LOD)* were calculated from the mean calibration curve using the following equations:

$$LOQ = \frac{10 \times S_b}{m\sqrt{n}}$$

$$LOD = \frac{3 \times S_b}{m\sqrt{n}}$$

Where S_b is the intercept standard deviation, *m* is the slope of the mean calibration curve and *n* is the number of calibration curves.

Precision and accuracy were assessed spiking a blank sample with furan at three different levels of concentration (Table 1) Each level was analyzed three times on three different days (*n* = 9). Precision was expressed in terms of relative standard deviation (RSD% ≤ 20) and the accuracy as percentage of recovery for each concentration level (80 -120 %).

Results and discussion

Previous to method validation, some experiments were performed using different headspace incubation temperatures, in order to evaluate the influence on the furan response. These temperatures were: 60º C, as recommended in the FDA method, and 80ºC which is the temperature we have previously used in our laboratory for other volatile compounds, and also used by other authors (Nyman et al., 2006; Zoller et al., 2007; Morehouse et al., 2008).

To check the effect of the temperature, we obtained calibration curves prepared on different matrices: water, fruit juice and vegetable purée, at both temperatures. The slopes

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of the regression lines, including all the matrices as well as both temperatures were compared. No significant difference was found between the mean slope obtained at 60ºC (n=3) and at 80ºC (n=4), (t test, α=0.05). Therefore, we concluded that setting the headspace temperature at 80ºC would not lead to a significant increase of the furan concentration in the samples within the scope of our study.

The in-house validation was performed as it has been described using a fruit based baby-food sample, where the absence of furan had been previously checked (<1/5 LOQ), which was taken as the blank sample.

The mean calibration curve (of three curves obtained on three days) with a furan concentration range 4 to 400 $\mu g.l^{-1}$ (equivalent to $\mu g.kg^{-1}$ sample) showed a good linearity (Cm% = 0.25) and the correlation coefficient was 0.9999.

Detection and quantification limits were calculated from the intercept value: 0.05 $\mu g.kg^{-1}$ and 0.17 $\mu g.kg^{-1}$, respectively. The lowest validated concentration level was 4 $\mu g.kg^{-1}$, and this was considered as the quantification limit for the report of sample results.

Precision (RSD %) and accuracy (% recovery) results are presented in Table 1. Both parameters were satisfactory according to our internal requirements.

(Table 1)

Therefore, method performance parameters assesed in the method validation showed that the analytical procedure was well applicable for the reliable determination of furan in baby food with a limit of quantification suitable for the purpose of our study. Furthermore, the validation results fulfil EFSA requirements (EFSA 2007): quantification limit less than 5 $\mu g.kg^{-1}$, precision (as RSD %) less than 20% and a minimum recovery of 80%. Also, the suitability of the method was confirmed by participating in an inter-laboratory trial (FAPAS®) on a vegetable based baby-food. The z-score obtained was 0.4, which was in the range of ≤ ±2, considered as satisfactory.

The method was applied to the analysis of the jarred baby food samples described in Table 2. All the samples were analyzed twice and a parallel recovery analysis was done for all of them. The results are shown in Table 2.

(Table 2)

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When the baby-food data were processed, precision data of duplicate analysis satisfied the Thompson modified Horwitz equation (Thompson 2000). This is equivalent to a $HorRat_r \leq 1$.

The average recovery value was 101.5% with a relative standard deviation RSD% = 5.6, which is in complete agreement with the results obtained in the method validation.

The thirty-nine baby-food samples analyzed by the method previously described were classified depending on their composition (most common ingredients) in five different groups. Table 3 summarizes the mean value per group as well as the maximum and minimum found values.

(Table 3)

From Table 3 it is evident that fruit-based baby food showed the lowest furan concentrations (5 $\mu g.kg^{-1}$) and the highest values were found in the meat (chicken, turkey and seabass) and vegetables group (62 to 65 $\mu g\ kg^{-1}$). Due to the limited number of analyzed samples based only on vegetables and dairy products (n=4), the results for these groups might not be representative and have to be carefully considered.

Furan concentrations in baby food samples from the Spanish market included in our study are in agreement with the levels determined in other studies (Bekalsi et al., 2005; Bianchi et al., 2006; Morehouse et al., 2008; Zoller et al., 2007); neither are they in disagreement with those included in the EFSA database where concentration levels ranged from not detectable to 112 $\mu g\ kg^{-1}$ (EFSA 2004). In our study the levels found were from <LOQ up to 65 $\mu g\ kg^{-1}$.

All samples analysed were commercial baby foods prepared by autoclaving in glass jars, technology which has been suggested to favor furan formation over other procedures used for commercial baby food preparation and domestic practices (Roberts et al., 2008; US FDA, 2004a; Zoller et al., 2007).

It must be noticed that fruit based products with added ascorbic acid contain also organic acids. It has been reported that the formation of furan from sugars or ascorbic acid is less likely at low pH (Becalski et al., 2005; Limacher et al., 2007). Apparently, the preparation of autoclaved baby foods does not include processing conditions of low water activity at high temperatures typical for non-enzymatic browning reactions to occur; thus furan is more likely formed through other mechanisms (Maillard reaction). It is possible to speculate that most potential precursors for furan in such baby food are polyunsaturated

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lipids such as polyunsaturated fatty acids and carotenoids (Becalski et al., 2005; Locas et al., 2004).However; this was not the purpose of our study

The composition of baby food seems to be a key factor to be taken into account in furan formation (presence of ascorbic acid, polyunsaturated fatty acids, aminoacids and carotenoids), as has already been described in previous studies regarding this analyte (Jestoi et al., 2009; Lachenmeier et al., 2009).

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Table 1: Mean recoveries and relative standard deviations of furan determinations at three different spiking levels used. Each spiking level is represented by 9 replicates.

n = 9	Level 1 (4 µg.kg^{-1})	Level 2 (115 µg.kg^{-1})	Level 3 (384 µg.kg^{-1})
Mean recovery (%)	85.4	101.5	101.1
RSD (%)	10.5	3.1	4.0

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Table 2: The different jarred baby food samples analysed with the ingredients reported by the manufactures and furan mean concentration found.

Number	Brand	Product	Ingredients	Furan µg.kg^{-1}
1	A	Vegetables and Spanish veal	Water, potatoes, Spanish veal, beans, carrots, peas, celery and rice flour.	15.1
2	A	Multifruits	Peach, apple, banana, orange, water, sugar, rice flour and vitamin C.	NQ.*
3	A	Mixed vegetables	Potatoes, water, green beans, carrots, peas, onion, celery, tomato, sunflower oil, rice flour and salt.	53.4
4	A	Rice and chicken	Water, chicken, carrots, rice flour, corn starch, sunflower oil, onion, celery, tomato and salt.	NQ*
5	B	Carrots and veal	Water, veal, potatoes, carrots, rice, tomatoes, starch, sunflower oil, onion, celery and salt.	11.9
6	B	Turkey and vegetables	Water, turkey meat, potatoes, peas, tomato, rice, onion, starch, sunflower oil and salt.	12.8
7	B	Ham, veal and vegetables	Water, potatoes, ham, tomato, veal, carrots, rice, onion, starch, sunflower oil, salt and garlic.	10.4
8	B	Vegetable soup and lamb	Water, potatoes, lamb carrots, peas onion, starch, sunflower oil, salt, parsley and garlic.	21.3
9	B	Fruit cocktail	Pears, apples, bananas, oranges, water, sugar, rice flour and vitamin C.	NQ*
10	B	Vegetables soup and lamb	Water, potatoes, lamb, carrots, peas, onions, starch, sunflower oil, salt, parsley and garlic.	29.3
11	B	Chicken and rice	Water, chicken, rice, carrots, starch, tomato, sunflower oil, onion, celery y salt.	17.4
12	B	Chicken, veal and vegetables	Water, potatoes, green beans, chicken, tomato, carrots, peas, veal, starch, celery, sunflower oil, onion, sugar and salt.	28.8
13	B	Ham, veal and vegetables	Water potatoes, ham, tomato, veal, carrots, rice, onion, starch, sunflower oil, salt and garlic.	20.6
14	B	Veal and carrots	Water, veal, potatoes, carrots, rice, tomato, starch, sunflower oil, onion, celery and salt.	19.9
15	B	Rice and carrots in chicken soup	Chicken soup, carrots, chicken, rice, onion, corn starch, olive oil, vegetables oils, parsley, salt and bay leaves.	11.3
16	B	Rice and hake	Water, potatoes, hake, rice, onion, sunflower oil, celery and salt.	13.9
17	B	Sole and béchamel	Water, potatoes, skimmed milk, sole, tomato, butter, starch, onion, celery and salt.	34.2
18	B	Peas and ham	Water, peas, potatoes, milk, ham, onion, corn starch, olive oil, vegetables oils, salt and aroma.	15.8
19	B	Chicken and vegetables	Water, potatoes, carrots, milk, chicken meat, onions, corn starch, tomato, celery, olive oil, vegetable oil and salt.	22.8
20	B	Vegetables with hake in a cream	Water, milk, hake, rice, onion, sunflower oil, celery, starch and salt.	14.2
21	B	Veil with carrots with olive oil	Water, veil meat, potatoes, carrots, rice, tomato, corn starch, olive oil, vegetable oil, onion, celery, salt.	12.6
22	B	Apple compote rich in vitamin C	Apple, sugar, starch, lemon juice, cinnamon and vitamin C.	5.1
23	B	Banana, tangerine and pear rich in vitamin C	Banana, tangerine, pear, apple, carrots, sugar, starch, lemon juice and vitamin C.	5.2
24	B	Varied fruit	Peach, apple, banana, apricot, orange, grape, corn starch, rice and vitamin C.	4.4
25	B	Peach and banana	Peach, banana, grape, corn starch and rice and vitamin C.	4,8
26	B	Milk dessert with biscuits	Milk, sugar, water, biscuits and rice flour.	39.8
27	B	Lentil stew	Water, lentils, potatoes, veil meat, carrots, tomato, onion, sunflower oil, starch, salt and spices.	22.2
28	B	Crème Caramel	Milk, sugar, water, rice flour, starch and vanilla.	21.2

Number	Brand	Product	Ingredients	Furan µg.kg^{-1}
29	C	Cream of béchamel and vegetables with chicken	Milk, water, chicken, potato, carrot, corn starch, butter, leak and green beans.	43.9
30	C	Rice and veal	Water, potato, carrot, veil, rice, onion, tomato, olive oil, leak and corn starch.	37.2
31	C	Banana, apple, orange and cereals.	Banana, apple, flour, honey, orange, lemon and vitamin C.	NQ*
32	C	Béchamel sauce and hake	Water, milk, hake, rice flour, corn starch, butter, salt and nutmeg.	34.2
33	C	Chicken with vegetables	Potato, water, carrot, chicken meat, leak, peas, onion, olive oil, corn starch and salt.	42.8
34	C	Gluten and additive free varied fruit.	Orange, apple, pear, apricot, water, sugar, rice flour and vitamin C.	7.4
35	C	Gluten and additive free Six fruit dessert.	Apple, peach, apricot, banana, orange, pineapple, sugar, water, rice flour and vitamin C.	7.8
36	C	Banana, orange and biscuit.	Banana, orange, water, sugar, gluten free biscuits, rice flour, corn, lemon and vitamin C.	9.0
37	C	Gluten and additives free chicken and rice.	Water, milk, chicken, potato, carrot, rice, corn starch, olive oil and salt.	64.6
38	C	Vegetable puree and turkey. Dinner for little ones.	Water, carrots, potatoes, milk, turkey, onion, green beans, olive oil, corn starch and salt.	62.8
39	C	Vegetables and seabass.	Water, carrots, potato, seabass, milk, turkey, onion, peas, green beans, olive oil, leak, corn starch and salt.	62.2

* NQ = Not quantifying, LOQ = 4 µg.kg^{-1}

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Table 3: Mean and maximum-minimum values per group depending on their composition.

Baby food based on:	**n**	**Mean value (µg.kg^{-1})** [(1)]	**Maximum value-Minimum value (µg.kg^{-1})**
Fruits	10	5.0	NQ – 9.0
Vegetables	2	37.8	22.2 – 53.4
Meat and vegetables	20	25.2	NQ – 64.6
Fish and vegetables	6	33.8	13.9 – 62.2
Dairy products	2	30.5	21.2 – 39.8

[(1)] To calculate the mean value we have taken value 2 (LOQ/2) for the Non Quantifying samples.

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Equation (1):

$$Cm\% = 100 \times \frac{Sm}{m}$$

Equation (2):

$$LOQ = \frac{10 \times S_b}{m\sqrt{n}}$$

Equation (3):

$$LOD = \frac{3 \times S_b}{m\sqrt{n}}$$

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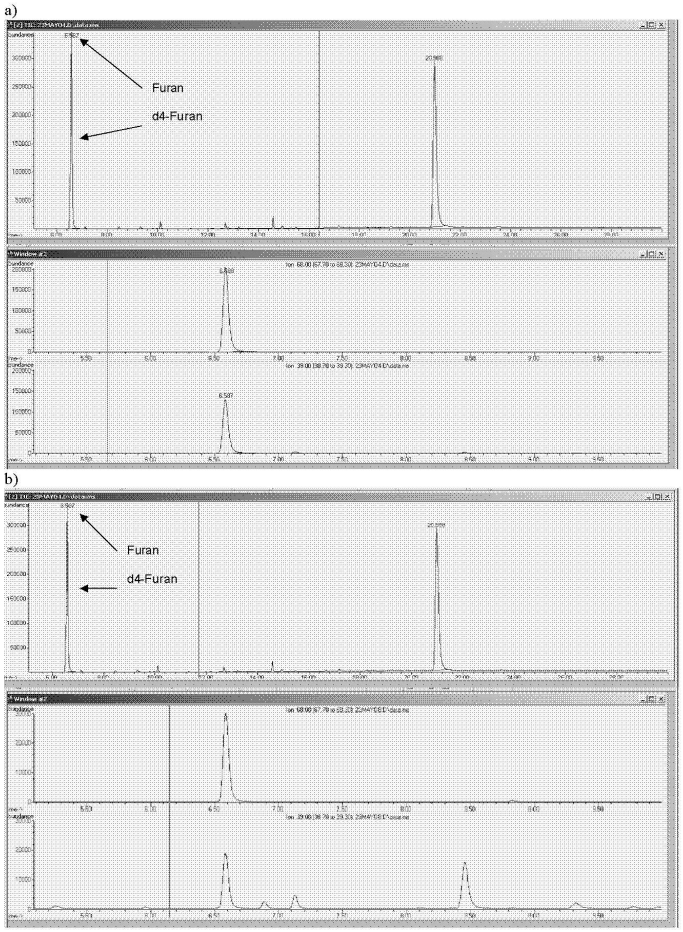

Figure 1. Typical HS-GC-MS SIM chromatogram and extracted single ions m/z 68 and m/z 39 of: a) 98.6 µg.kg^{-1} furan standard and b) a meat and vegetables baby food sample (29.3 µg.kg^{-1}).

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