Heterocyclic Letters
Vol. 15| No.1|217-225|Nov-Jan|2025
ISSN : (print) 2231–3087 / (online) 2230-9632
CODEN: HLEEAI
http://heteroletters.org

A REVIEW ON FURAN: DETECTION AND ANALYSIS METHODS

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ABSTRACT: Furan is a volatile organic compound with a five-member aromatic ring made up of four carbon atoms and one oxygen atom. It is volatile cyclic ether found in cigarette smoke, and is used in the production of resins and lacquers, agrochemicals, and pharmaceuticals. It is formed during combustion processes such as waste incineration, power generation, metal production, and fuel burning. These compounds are found in small amounts in the air, water and soil. Furan is a colorless, flammable, highly volatile liquid with a boiling point close to room temperature. It is soluble in common organic solvents, including alcohol, ether, and acetone, and is slightly soluble in water. Its odor is "strong, ethereal; chloroform-like". It is toxic and may be carcinogenic in humans. The furan ring system is the basic skeleton of numerous compounds possessing cardiovascular activities. These compounds are widely employed as antibacterial, antiviral, anti-inflammatory, anti-fungal, anti-tumor, anti-hyperglycemic, analgesic, and anti-convulsant. High-resolution mass spectrometry is an analytical technique used for quantifying furans.

KEYWORDS: Furan, Furan derivatives, occurrence, detection, analysis methods.

INTRODUCTION:

Furan (C_4H_4O) is an organic volatile (B.P. 32 ^{0}C) compound with a five-member aromatic ring made up of four carbon atoms and one oxygen atom (Fig. 1).

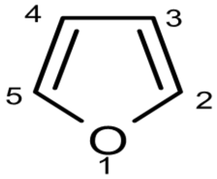


Figure 1. Structure of Furan.

It is a colorless cyclic ether and lipophilic organic compound, with a molecular weight of 68.

It is a found in cigarette smoke, and is used in the production of lacquers and resins, agrochemicals, and pharmaceuticals [i]. Furans are commonly produced by combustion of fossil fuels and incineration of municipal waste, as a byproduct of pulp and paper bleaching, and in the production of other chemicals. 2,3,7,8-Tetrachlorodibenzo-*p*-dioxin is the most toxic member of this family. It is an endocrine disrupter as well as a potent animal carcinogen and teratogen that persist in both the environment and biological tissues [ii-iii].

In Meda, Italy, on July 10, 1976, a valve broke at the Industry Chimiche Meda Societa Azionaria chemical plant releasing about 3,000 kg of dioxin-containing chemicals into the atmosphere. Approximately 4% of local farm animals died, and roughly 80,000 animals were killed to prevent the contamination from moving up the food chain. It is believed that this exposure affected the sex ratio in future progeny [iv].

Furan is a volatile chemical formed in food when it is heated. It is found particularly in food sold in cans and jars where it cannot escape through evaporation. The highest levels are found in roasted coffee, starchy canned foods, baby foods, sauces, and soups. These findings are of concern as furan is anticipated to be a human carcinogen and genotoxic. Reduction of furan formation during food manufacture is difficult because foods must be heated to ensure the microbiological safety [v].

Furan is naturally occurring at low levels in many foods and drinks. Furan and its derivatives contribute to the sensory properties of food [vi]. The presence of furan in a variety of heated processed foods (soups, coffee, juices, and canned and jarred fruits and vegetables, including baby foods) by the FDA following the posting on its website in 2004 of data on the occurrence of the contaminant in food. The concerns over furan is due to its classification as a ‘possible carcinogen to humans’ (Group 2B) (IARC, 1995), and the finding that the compound causes cancer in rodents (National Toxicology Program, 1993).

The scientific and regulatory community was interested to determine exposure to furan, explore the mechanisms of formation of furan and determine occurrence of the contaminant in different food categories, which helps to access methods for reducing furan levels in foods.

However, domestic cooking procedures reduce the furan content of foods like brewing coffee causes a dramatic decrease, and stirring food or leaving it to stand is also effective [vii]. Furan can be determined by headspace-sampling gas chromatography with mass spectrometric detection [viii].

International Agency for Research on Cancer (IARC) classifies furan as possibly carcinogenic to humans, based on studies with laboratory animals. The U.S. FDA has recently published a report on the occurrence of furan in a large number of thermally processed foods, especially canned and jarred foods, including baby foods and infant formulas. Thermal degradation of carbohydrates, such as glucose, lactose and fructose are considered as the primary source of furan in food. Coffee contained the largest amount of furans among all the foods tested in various papers [ix].

The detection of furan in food products is difficult due to its high volatility and low molecular weight but it can be determined, with high accuracy, by headspace methods. Headspace solid-phase micro extraction coupled with gas chromatography/mass spectrometer (GC/MS) is generally used for analysis of furan in food samples [x-xi]. This will demonstrate a rapid method for the identification and quantification of furan in food samples, using gas chromatography with headspace sampling and mass spectrometry.

The risk assessment of furan can be characterized using margin of exposure approach (MOE). Conventional strategies including reheating of commercially processed foods with stirring, cooking in open vessels, and physical removal using vacuum treatment have remained unsuccessful for the removal of furan due to the complex production mechanisms and possible precursors of furan. The innovative food-processing technologies such as high-pressure processing (HPP), high-pressure thermal sterilization (HPTS), and Ohmic heating have been adapted for the reduction of furan levels in baby foods. But in recent years, only HPP has gained interest due to successful reduction of furan because of its non-thermal mechanism. HPP-treated baby food products are commercially available from different food companies [xii].

In addition to method optimization and standard analysis, a number of food samples analyzed for furan. Coffee containing drinks, sauces, and canned foods, as previous studies demonstrated high levels of furan in these foods [xiii]. Most of the fermenting microorganisms are able to reduce furans to their corresponding less toxic alcohols. 2-furaldehyde (furfural) and 5-hydroxymethyl furfural (HMF) are the main degradation compounds among furan derivatives, generated from pentoses and hexoses degradation, respectively [xiv].

HMF is redu-ced to 2,5-*bis*-hydroxymethylfuran and furfural to furfuryl alcohol, and both could be also oxidized to formic acid under anaerobic conditions [xv]. If furans are present at high concentration, they exert an inhibitory effect interfering with glycolytic enzymes and synthesis of macromolecules provoking an enlarge of the lag phase and reducing the ethanol productivity [xvi]. These effects depend on furan concentration but are highly related with the yeast strain. Based on mixed aromatic-dienic properties, furan displays a peculiar chemical behavior [xvii]. Furan is the least aromatic in character and the most dienic member as compared with the thiophene and pyrrole homologues. This property is demonstrated by the readiness with which furan and some of its derivatives undergo the Diels-Alder cycloaddition.

Due to excellent properties of furan-based polyesters for industrial applications, they have attracting extensive attention. Du’s group reported a series of degradable poly (silylethers) based on biobased furan derivatives. The furan-containing polyesters can be cross-linked by using bis-maleimide cross-linkers to give polymer networks via Diels-Alder reactions and can be further thermally reversed back to the linear polyester via Retro-Diels-Alder reactions [xviii-xix].

Furans, a well-known environmental pollutant, are extremely toxic to humans and many other species. Dibenzofurans are listed as pollutant of concern. It pollutes into the air from combustion sources and persist in the environment, potential to bioaccumulate, and toxicity to humans and the environment. When these compounds are adsorbed on soils or other substrates, are highly persistent under normal environmental conditions [xx]. Based on US Environmental Protection Agency's (EPA) concept of toxic equivalent factors (TEFs) for assessing cancer risk associated with these chemicals. The cancer potency of all other isomers chlorinated in the 2,3,7, and 8 positions is related to tetrachlorodibenzofuran (TCDF).

It is necessary to know the proportion of total furans, when calculating toxic equivalents. Depending upon the dose and the length of exposure to these compounds, various health conditions may develop, such as, abdominal pain, muscle pain, acne rash, coughing, severe respiratory infections, chronic bronchitis, changes in skin color, headache, increased blood triglycerides, vomiting, diarrhea, leukocytosis, limb neuralgia, peripheral neuropathy, paresthesia, weight loss, and liver disease. They are also responsible for causing ‘Yusho’ disease; common symptoms included irregular menstrual cycles, dermal and ocular lesions, and a lowered immune response. In children, there were reports of poor cognitive development [xxi].

EXPERIMENITAL:

2. Detection and Analysis methods:

2.1 Gas chromatography-mass spectrometry (SPME-GC-MS):

Furan detection carried out by using number of analytical methods. Solid-phase microextraction-gas chromatography-mass spectrometry (SPME-GC-MS) was invented and verified as a quick and the easiest method to find out the proportion of furan in fruits and vegetable-based foods. Quality parameters including LOD (limit of detection), LOQ (limit of quantification), correlation coefficient (r2), and relative standard deviation (RSD). This method showed detection of furan at ultra-trace level, and excellent results were achieved at LOD (25.70 ng/kg) and LOQ (41.7 ng/kg). This technique was used for the calculation of furan in baby-food samples. Many commercial samples as well as homemade vegetables and fruits were given for analysis. The furan concentration in baby foods samples was determined using solid-phase micro extraction followed by GC-MS. The method was accurate for the analysis of furan. Twenty-one different baby food samples were analyzed for furan concentrations that were found in the range of 4.7 and 90.3 µg/kg. The mean levels of furan concentration for vegetables-based, fruit-based and meat-based baby foods were 37.0, 9.2, and 49.6 µg/kg, respectively. The fruit-based baby food samples had significantly less concentration of furan as compared to other baby food samples [xxii].

In Taiwan the Headspace solid-phase micro-extraction (HSSPME) coupled with GC/MS was used to analyze furan formation in different food samples to find out the risks of exposure of furan. This procedure provided acceptable linearity at r2 = 0.9990 with LOD (0.02 ng/g) and LOQ (0.06 ng/g) [xxiii]. Sarafraz-Yazdi et al. (2012) [xxiv] developed a method for the determination of furan in baby food and fruit juices. They used the HS-SPME method with two different coatings synthesized by sol-gel technology coupled with gas chromatography-flame ionization detector (GC-FID). This method was able to determine furan levels in the range of ng/mL in the food samples. In China, an optimized automated headspace gas chromatography-mass spectrometry method (HS-GC-MS) was used to evaluate the levels of furan in thermal-processed commercial foods. This method was validated in terms of LOD (0.3 ng/g) and LOQ (1.0 ng/g). This showed good linearity (r2 = 0.9993) and precision of analysis was done in terms of RSD (3.0%). The headspace with an oven temperature 70 ◦C and equilibration time 30 min in combination with GC column was used.

The quantization of furan in 11 categories of foods (a total of 133 kinds of food samples) was done by the suggested HS-GC-MS method. The mean level of furan was 3.2 to 27.0 ng/25 foods including infants and baby foods were analyzed for the determination of furan using SPME-GC-MS in Korea. The range (2.74 to 33.9 ng/g) of the furan concentration in jarred baby foods was determined [xxv]. Recently, Condurso et al. (2018) [xxvi] also used HS-SPME-GC-MS for the accurate detection and quantification of furan. The LOD, LOQ, r2, and RSD determined for this method were 0.029 ng/g, 0.097 ng/g, 0.9996, 1.2 to 3.5%, respectively. This is solvent-free, rapid technique, very specific, and precise in terms of repeatability and precision and easily detected furan in commercially available baby food products.

A study explained an optimized and verified process to quantify furan in foods ingested by infants and toddlers by using GC-MS. The precision of the method was RSD (10 to 15%) and the process was applicable at LOQ (1 to 2 µg/kg), LOD (0.3 to 0.6 µg/kg). This method was applied for the analysis of 134 food samples. Furan was found in 84% and quantified in 61% of the samples, at average concentrations ranging from 0 to 44 µg/kg [xxvii-xxviii].

Besides this, quantitative assessment of furan, it is necessary for the risk management of furan. There is no official standard method for furan detection in concerned foods which is important in case in which risk management measures of certain foods are suggested or applied. For proper laboratory analysis, certified reference materials would be necessary. There is more research work is necessary on the development of various furan-free baby foods using HPTS, HPP, and Ohmic heating and other novel thermal technologies such as radiofrequency heating. In the baby food industry, this type of novel processing will be of high interest as it can help to reduce both heat-induced chemical changes and exposure of infants and babies to hazardous furan [xxix].

2.2 Surface Acoustic Wave (SAW) based electronic nose:

Due to high concentrations of dioxins and furans in the environment resulting from widespread incineration of waste, there is rise in birth defects in Japan and other countries throughout the world have been traced. To test for the presence of these toxic compounds in air, water, and soil, there is a need for real time instruments. A new Surface Acoustic Wave (SAW) based electronic nose was evaluated to meet this, need.

Unlike previous electronic nose technology utilizing arrays of polymer coated sensors, the GC/SAW electronic nose utilizes a single temperature controlled 500 MHz SAW resonator crystal (Figure 1) to measure the adsorption and desorption characteristics of analytes as they exit a temperature programmed GC capillary column [xxxiv-xxxviii], The sensing crystal and oscillator circuitry are enclosed within a 2-inch diameter enclosure.

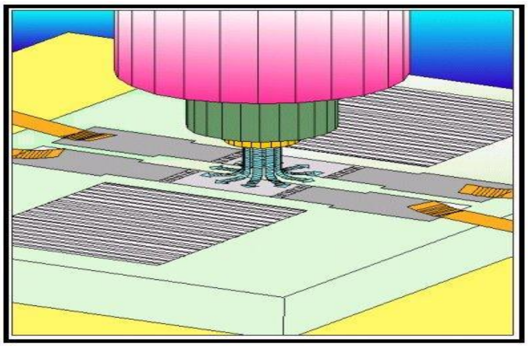


Figure 1. Design details of surface acoustic wave (SAW) detector.

The SAW crystal is used to provide closed loop frequency control of an oscillator circuit and the oscillator frequency is a direct measure of the amount of material deposited. A heated nozzle is used to focus the He carrier gas of the GC (Gas Chromatography) such that it impinges the acoustic standing wave pattern in an optimal manner. Focusing column effluent and the inherent high Q of an uncoated crystal results in a 10,000-fold increase in sensitivity over polymer coated delay line SAW sensors. The detection limit of the SAW sensor was determined by the oscillator phase noise and crystal temperature (60 ^{0}C). Precise control of the crystal temperature over the range 0-150°C was by means of a thermoelectric element. Using a counter sample time of 20 milliseconds resulted in an Allan variance of 1-2 Hz. Sensitivity to dioxin and furan isomers was approximately 10 Hzf picogram, hence detection levels for the SAW sensor were at the part per trillion (picogram) level [xxx-xxxiii].

2.3 Proton Magnetic Resonance (PMR):

Whether furan is a component of complex organoleptic structures has long been a challenge due to the lack of a generally effective diagnostic method. Chemical and color tests can only provide a limited amount of information, as they are not very specific and the chemical properties of the ring system are variable. Ultraviolet spectroscopy is not very useful as furans not absorb in that range. Correlations of certain bands in infrared spectra have been made for some furans, but the nature of absorption and the assumption that there are no interfering bands renders the infrared data useless when more than just confirmation is required. Another important issue with simple as well as complex structures that contain the furan system is where the substituents are located on the ring. This problem has also been resistant to solution by physical methods in the context of investigations on the structures of some natural products that are currently being conducted in these labs and also due to the widespread presence of furan. We have been interested in these problems for some time and have attempted to use other methods to solve them.

The structural analysis for furans using proton magnetic resonance, a technique that has been shown to be very effective in the cases we have studied so far. Our nuclear magnetic resonance measurement with furan derivatives shows that in general, the protons connected to furan nuclei are less shielded than those connected to double bonds, and in this way, are similar to the protons associated with benzenoid nuclei. A comparison of furans and vinyl ethers shows that proton resonance is at a lower field for the former than the latter, which is expected from the presence of diamagnetic N-electron. This difference between furanoid, ethylenic, and paraffinyl proton resonance is not unexpected, and is a useful and desirable one for structural analysis [xxxix]. The PMR spectrum and chemical shift value of F are as follows. There are two sets of protons H_a and H_b with chemical shift value 7.38 δ and 6.30 δ. H_a protons appears at higher δ value as these protons are adjacent to electronegative oxygen atom, so deshielded protons as compared to H_a protons.

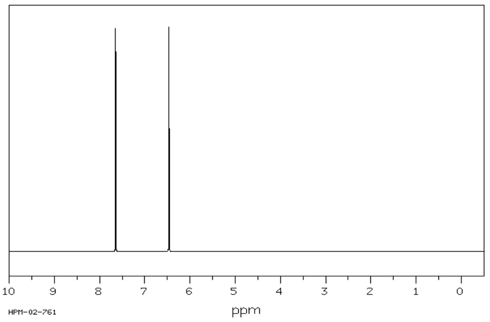
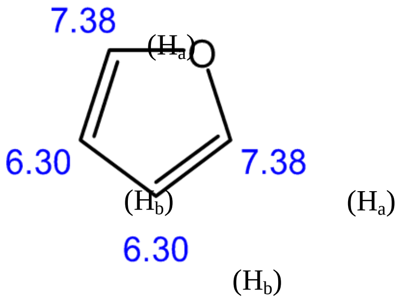


Figure 2 PMR of Furan



Acronyms and Abbreviations:

EPA- Environmental Protection Agency's, TEFs- concept of toxic equivalent factors, IARC- International Agency for Research on Cancer, HMF - 5-hydroxymethyl furfural, TCDF- tetrachlorodibenzofuran, LOD- limit of detection; LOQ- limit of quantification; r2-correlation coefficient; SD-relative standard deviation (Lambert et al. (2018) Abbreviations), SAW- Surface Acoustic Wave; GC-FID- Gas chromatography-flame ionization detector; HS-GC-MS- Headspace gas chromatography-mass spectrometry, HSSPME- Headspace solid-phase micro-extraction.

CONCLUSION:

Furan formation particularly in baby foods is a serious food safety concern that needs to be addressed. To reduce the formation of furan in these types of products alternative strategies should be developed at the industrial level. It is also necessary to identify the factors which affect the level of furan formation in the different infant and baby food samples. To describe the occurrence and exposure assessment of furan, the identification of a biomarker for exposure of furan is a good approach.

The limited use of precursors for furan formation should be encouraged. This involves the levels of unsaturated fatty acids and the oxidation of oil. Besides this, quantitative assessment is necessary for the risk management of furan. There is no official standard method for detection of furan in concerned foods that is important in case in which risk management measures of certain foods are suggested or applied.

For proper laboratory performances, certified reference materials would be necessary.

There is a need for more research work to be conducted on the development of various furan-free baby foods using HPTS, HPP, and Ohmic heating as well as other novel thermal technologies such as radiofrequency heating. This type of novel processing can help to reduce both heat-induced chemical changes and exposure of infants and babies to hazardous furan.

It is possible to test for the presence of dioxins and furans at the picogram level by using SAW sensors. Testing for dioxin/furan in soil and liquid matrices surrounding the source of dioxins/furan emissions (e.g. incinerators) is also possible. In these cases the concentrations are higher (nanograms) and more dangerous to humans because they have accumulated over time. In addition, the SAW sensor output frequency forms a natural VaporPrint^{TM} image of any fragrance without the need for artificial intelligence software. The detection limit of the SAW detector is determined by the spectrum of SAW oscillator phase noise. Sensitivity to dioxin and furan isomers is nominally 10-20 Hz/picogram, hence detection levels are at the part per trillion (picogram) level.

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Received on November 20, 2024