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A Sensitive Gas Chromatographic-Mass Spectrometric (GC–MS) Method for the Determination of Bisphenol A in Rice-Prepared Dishes

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Abstract

Certain chemicals possess the potential to modulate endocrine systems, and thereby interfere with reproductive and developmental processes. Bisphenol A is suspected to be one of them. The compound is widely used as a plastic additive, lacquer, resin, or plastic and can usually be found in food samples. An accurate and reproducible gas chromatographic-mass spectrometric (GC–MS) method to detect and measure trace amounts of the compound in rice-prepared dishes samples is proposed. Solid-liquid extraction with acetonitrile was carried out in order to isolate and pre-concentrate the analyte. The solvent was removed and a silylation step using *N,O*-bis(trimethylsilyl)trifluoro acetamide/pyridine (BSTFA/PYR) was carried out. The silylated compound was identified and quantified by GC–MS using a DB-5 MS column. Bisphenol F was used as a surrogate internal standard. The detection limit was 2.0 ng g⁻¹ while inter- and intra-day variability was less than 6%. Due to the absence of reference materials, the method was validated using standard addition calibration and a recovery assay. Recoveries for spiked samples were between 90% and 105%

1 **Keywords:** Bisphenol A; Migration; Gas chromatography–mass spectrometry (GC–MS);
2 Rice-Prepared Dishes.

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7 8 **Introduction**

9 Various epidemiological and laboratory studies describing, in some cases, severe
10 disturbances in the endocrine system of humans and wildlife have been published in the
11 past few years (Rivas et al., 1997; Paris, et al., 2002). Today, it is well known that around
12 100 industrial chemicals show estrogenic activity in addition to their desired chemical
13 properties. Most of these endocrine disrupter chemicals (EDC's) are widely used organic
14 compounds, which are ubiquitous in the environment and in biological samples (Fromme
15 et al., 2002; Harris et al., 1997). At the same time, ECD's accumulate in certain tissues in
16 humans and their effects could be passed to future generations via placenta and/or milk
17 (Maffini et al., 2006; Hood, 2005; Lopez-Espinosa et al. 2008)

18 Most of these compounds, commonly called endocrine disrupter chemicals (EDCs),
19 are synthetic organic chemicals introduced into the environment by way of anthropogenic
20 inputs. Aware of the problem, both the European Union and the United States (EPA) have
21 authored a "priority" list of substances for further evaluation of their role in endocrine
22 disruption (Groshart and Okkerman, 2000; Multi-Year Plan [FY2000-2012] for Endocrine
23 Disruptor) and indicate the need to assess the levels and effects of EDCs.

24 One of the representative compounds of the group of EDC's is bisphenol A (BPA).
25 BPA is a compound with high reactivity and is the raw material for a large amount of
26 manufactured products, such as epoxy resin, or polysulfones. It is also used as an
27 antioxidant or stabilizer. However, one of the most important applications of the compound
28 is the production of polycarbonate plastics, which are mainly a condensed polymer of BPA
29 and carbonyl chloride or diphenyl carbonate. Since it is transparent, has excellent heat
30 resistance and impact resistance, and can be used for high-temperature uses and in
31 microwave ovens, it is used in items such as children's tableware, coffee makers and food
32 containers.

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4 1 Some studies have reported that BPA has an estrogen-like action of toxicity;
5 2 moreover, it was discovered that the compound was released from a flask made of
6 3 polycarbonate and showed binding to the estrogenic receptor. In fact, BPA induced
7 4 progesterone receptors in cultured breast cancer cells and its activity was about 1/5,000
8 5 that of estradiol (Krishnan et al., 1993). Different studies show statistically significant
9 6 reproductive and developmental toxicity in rats and mice at high doses of BPA (Morrissey
10 7 et al., 1987). Nevertheless, there still controversy over whether low doses of BPA may
11 8 cause reproductive and developmental effects in humans (Goodman et al. 2006).

12 9 Significant published data is available on BADGE or BPA migration from can coatings
13 10 into foods and food simulants (Biles et al., 1997; Kawamura et al., 1999; Yoshida et al.,
14 11 2001; Goodson et al., 2002; Kang and Kondo, 2003; Cabado et al., 2008) but little is
15 12 known about how the migration level would be influenced by damage to the can or storage
16 13 conditions. However, some workers have investigated the effects of the food processing
17 14 conditions on the migration of BPA from food or from simulating liquids (Munguia-Lopez
18 15 and Soto-Valdez, 2001, Sajiki et al., 2005; Le et al, 2008; Poças and Hogg, 2007; Mungia-
19 16 Lopez et al., 2002).

20 17 Sterilization conditions, normally applied by industry for the scenarios used in our
21 18 study are relatively extreme (high temperature and sterilization time). In view of the
22 19 potentially long shelf-life of most canned foods and the current interest surrounding the
23 20 exposure to BPA from food packaging, it was considered important to obtain information
24 21 on all of these factors.

25 22 The Scientific Committee for Food reviewed the Tolerable Daily Intake (TDI) for
26 23 BPA and the TDI reduced its value to a temporary one of 0.01mg kg^{-1} body weight day per
27 24 day. Therefore, it is of crucial importance to devise analytical methodology for detecting
28 25 and quantifying these compounds in food.

29 26 Gas chromatography-mass spectrometry has traditionally been used as the main
30 27 analytical methodology for analyzing BPA together with other EDCs (nonylphenol,
31 28 phthlates or bisphenol A diglycidyl ether) in water, milk or beverages (Mol et al., 2000;
32 29 Rodríguez et al., 2003; Helaleh et al., 2001; Ding and Chiang, 2003; 2000; Schoene et al.,
33 30 1994; Nakamura et al., 2001; Fine et al., 2003; Casajuana and Lacorte, 2004;). In contrast,
34 31 there are very few examples where GC-MS has been applied to powdered (Kuo and Ding,
35 32 2004) or solid foods (Thomson and Grounds, 2005). Goodson et al., (2004) investigated
36 33 the potential effects, on the migration of BPA from can coatings, of cooking or heating
37 34 foods in the can prior to consumption using GC-MS as their analytical technique.

1 Alternatively, liquid chromatography (Katayama et al., 2001; Shao et al., 2005 and 2007;
2 Petrovic and Barceló, 2001; Maragou N.C. et al., 2008) and capillary electrophoresis
3 (Tsukagoshi et al., 2002) have also been used.

4 In this paper, an accurate, simple and reproducible method to detect and quantify trace
5 amounts of BPA in rice-prepared dishes is reported. The main purpose of the study was the
6 application of the method as routine methodology in a food company for BPA
7 contamination control. Samples taken from different supermarkets were also analyzed as a
8 preliminary step and the method was checked by recovery assays in spiked samples.

10 **Materials and Methods**

11 *Reagents and standards*

12 Unless noted otherwise, all chemicals and solvents purchased were high-purity. Water
13 was purified with a Milli-Q plus system (Millipore, Bedford, USA).

14 Acetonitrile (HPLC-gradient, PAI-ACS), hexane (UV-IR-HPLC, PAI), absolute
15 ethanol (HPLC-gradiente, PAI), ethyl acetate (UV-IR-HPLC, PAI-ACS), sodium chloride
16 (PA-ACS-ISO) were supplied from Panreac (Barcelona, Spain). All solvents and reagents
17 were checked to ensure they were free of contamination from bisphenol A. Bisphenol F
18 (BPF) purum, $\geq 98.0\%$ (Fluka), bisphenol A (BPA) purum, $\geq 99\%$, silylation agent *N,O*-
19 bis(trimethylsilyl) trifluoroacetamide (BSTFA) puriss. p.a., for GC, $\geq 99.0\%$ and pyridine
20 anhydrous, 99.8% (Sigma-Aldrich) were supplied by Sigma-Aldrich-Fluka (Madrid,
21 Spain).

22 Stock solutions of analyte BPA and surrogate BPF (100 $\mu\text{g}/\text{ml}$) were prepared in
23 ethanol. BPF was selected as the surrogate due to the structure similarity to BPA and to the
24 fact that it has not been detected in a previous study in canned food (Goodson et al. 2002).
25 Mixtures of the analytes for working standard preparation and sample fortification were
26 also prepared in ethanol. All stock solutions and mixtures were stored at $-10\text{ }^{\circ}\text{C}$ in the dark
27 until use, remaining stable for at least three months.

29 *Sample preparation*

30 Rice-prepared dishes are a pre-cooked meal which main component is rice (50-65 %) and also contain a large variety of other minor components (vegetables such as carrots, green pepper, onion, peas; meat such as chicken, pig or beef and other ingredients such as oil, salt, garlic and spices). The rice-prepared dishes are presented in plastic containers.

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4 1 Samples were randomly selected from different supermarkets and also from a rice-
5 2 prepared dishes production plant situated in Seville (Spain). A total of 250 different rice-
6 3 prepared dishes were acquired and analyzed.

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9 4 As well, rice-prepared samples were collected previous to the packing procedure
10 5 directly in the production plant. These samples were homogenized and tested as negative
11 6 controls for BPA and, at the same time, were used for fortification and/or recovery studies.

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14 7 All samples were carefully mixed and homogenized using a food mixer. The
15 8 homogenized samples were stored in glass bottles, previously cleaned with nitric acid (1:1;
16 9 v/v), in the dark and at 4 °C until treatment. The analysis was performed with the minimum
17 10 possible delay and as described below in the *Extraction and derivatization* section in order
18 11 to avoid sample degradation.

12 13 *Extraction and derivatization*

14
15 14 Prior to extraction, sample was carefully homogenized with a mixer and sample (20 g)
16 15 were placed in 50 mL falcon tube. After that, the sample was spiked with bisphenol F as a
17 16 surrogate (100 ng·g⁻¹). Extraction was carried by adding 5 mL of acetonitrile and shaking
18 17 vigorously in a vortex-mixer for 1 min. The mixture was centrifuged 2 min at 6000 r.p.m.
19 18 at room temperature. Supernatant was transferred into a 25 mL separation funnel, and the
20 19 extraction was repeated twice. NaCl (5 g) were added to the combined organic phase in the
21 20 separation funnel and three phases, entailing a solid sodium chloride phase, saturated NaCl
22 21 aqueous solution phase and an organic phase, could be observed. After removing the solid
23 22 and the aqueous layer, the acetonitrile phase was washed with n-hexane (2 mL) to remove
24 23 fat traces and decanted into a 10 mL assay tube in order to evaporate to dryness in a speed-
25 24 vac system. The extract was re-dissolved using 1.0 mL of ethyl acetate and transferred into
26 25 a chromatographic vial.

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28 26 After evaporation to dryness under nitrogen, 50 µL of a mixture of ethyl
29 27 acetate/BSTFA/Pyridine (2:1:1; v/v/v) were added into the tube to resuspend the residue
30 28 and to carry out the derivatization. BPA and BPF are suitable for derivatization due to their
31 29 chemical structure. Once the derivatization process was completed (1 minute), 1 µL of the
32 30 reaction mixture was injected into the GC–MS system.

31 32 *Apparatus and software*

1 GC analysis was performed using an Agilent 6890 Series GS System GC fitted with a
2 splitless injector for a low background, a split/splitless deactivated glass liner (78.5 mm ×
3 6.3 mm × 4.0 mm, wool packed) for capillary injection port was used. A J&W Scientific
4 Inc capillary column DB-5MS fused-silica, 30 m × 0.25 mm I.D.; 0.25 µm film thickness;
5 5% phenyl-95% dimethyl arylene siloxane was used. Detection was carried out with a
6 5973 mass-selective single quadrupole detector (Agilent technologies). The GC-MS
7 operation control and the data process were carried out by ChemStation software.

8 The injector port of the GC was set at 250 °C. The silylated samples were
9 automatically injected using the splitless-injection mode. The transfer line of the GC to the
10 MS was set at 280 °C, and the electron ionisation (EI) ion source of the MS set at 250 °C.
11 The GC oven temperature program applied was as follows: the initial oven temperature
12 was set at 150 °C, held for 0.5 min, then the temperature was increased to 280 °C via ramp
13 of 20 °C min⁻¹ and held for 5.0 min, finally the temperature was raised to 300°C at 40 °C
14 min⁻¹, and maintained for 2.5 min. The total run time was 15.0 min. The carrier gas used
15 was helium (purity 99.999%) at a flow rate of 1.0 mL min⁻¹. Delay time was 3 min in order
16 protect the ion multiplier of the MS instrument from saturation and the sample volume in
17 the direct injection mode was 1 µL.

18 For qualitative analysis, the MSD was operated in full-scan mode. The conditions for
19 electron impact ionization (EI) were: ion energy of 70 eV, multiplier 1800 and the mass
20 range scanned was 50-400 *m/z*. The MS was tuned everyday to *m/z* 69, 219 and 502 with
21 perfluorotributylamine (PFTBA) as a calibration standard.

22 For quantitative analysis, single ion monitoring (SIM) acquisition mode (dwell time
23 100 ms/ion) was used. The retention times for BPF and BPA trimethylsilyl derivatives
24 were 8.9 min and 10.7 min, respectively. The mass spectrum of silylated BPF showed a
25 base peak at 344 *m/z*, corresponding to the molecular ion, and the mass spectrum of
26 silylated BPA showed the molecular ion peak at 372 *m/z*, whereas the base peak appears at
27 357 *m/z*, corresponding to the loss of a methyl group

28
29 Statgraphics Centurion XV, vs 15.1.02 software package (1982-2006 Statpoint Inc)
30 was used for the statistical analysis of data.

31 32 **Results and discussion**

1 *Isolation and pre-concentration procedure*

2 Rice-prepared dishes contain a complex mixture of a large number of components
3 (rice, meat, peas, oil, fat, etc.). Determinations at the trace level require isolation and a pre-
4 concentration step of the samples to reach nanogram levels of concentration. An extraction
5 procedure was selected as appropriate to obtain the analyte from the homogenized rice dish
6 samples. The extraction was optimized by adjusting parameters that influence in analyte
7 extraction, *e.g.*, nature of extraction solvent, extraction time or cleaning solvent.

8 A large number of mixtures of diethyl ether, methanol and acetonitrile were tested for
9 extraction of analytes from samples. Pure solvents and different mixtures of diethyl ether,
10 methanol and acetonitrile (ratios 3:1, 1:1 and 1:3) were tested for extraction of analytes
11 from samples. Extraction yield for analyte isolation (at 100 ng g⁻¹ spiking samples) was in
12 all cases between 60% and 90%, except for pure acetonitrile that showed the highest yield
13 (96%)

14 In addition, n-pentane, n-hexane and chlorinated solvents (dichloromethane,
15 chloroform or carbon tetrachloride) were tested to remove fat traces from acetonitrile
16 extracts in separating funnel. Although carbon tetrachloride and chloroform showed good
17 results (close to 90% of de-fating ratio, and less than 15 % of analyte losses), they were not
18 used due to their high toxicity for humans and the environment. n-Pentane showed a
19 relative defating ratio very close to n-hexane but the analyte loss was 40% for pentane and
20 only 12 % for n-hexane. Therefore, acetonitrile was selected for BPA extraction and n-
21 hexane was used for extract cleaning.

22 *Derivatization method*

23 Trimethylsilyl derivatives of BPA and BPF were obtained using a BSTFA/Pyridine
24 mixture as silylation reagent. This reagent was selected because of its fast reactivity with
25 compounds containing hydroxyl groups, its high volatility resulting in no co-elution of
26 early eluting peaks, and low thermal degradation and good solubility of the derivatized
27 compounds in common organic solvents. Derivatized samples had a better separation of
28 the analyte under GC-MS analysis, because of their higher volatility and lower interaction
29 with the stationary phase.

30 The optimisation of the derivatization procedure was carried out by applying the
31 experimental design methodology. The effect of varying the percentage of silylation agent
32 (BSTFA/PYRIDINE 1:1, v/v) in ethyl acetate, temperature of process and reaction time
33 were tested on the analytical response. The three factors were simultaneously optimised by
34

1 application of a 2³ central composite design plus face centred (with three centred points).
2 The BSTFA/Pyridine concentration was studied from 0 to 100%, temperature from 25
3 (room temperature) to 95 °C and the reaction time was varied between 0 and 60 min. A
4 50% of silylation agent in ethyl acetate (v:v), 1 min and room temperature were the
5 procedural conditions selected.

7 *Gas chromatographic-mass spectrometric analysis*

8 An increase in the signal-to-noise ratio for BPA was clearly observed in the
9 derivatized sample. Figure 1 show a characteristic chromatogram obtained in SCAN mode
10 (A) and SIM mode (B) for a silylated sample containing BPA and BPF as surrogate.

12 **Figure 1**

14 Another feature of the application of derivatization reactions is that trimethylsilyl
15 derivatives produce ions with higher m/z in the GC-MS system in contrast to those
16 obtained from underivatized compounds. The selection of high mass fragments as
17 quantification ions is of great interest, particularly when complex matrices, as rice-
18 prepared dishes, are to be analyzed, due to the decreased likelihood of interferences. In our
19 research a high increase in sensitivity and selectivity was reached. The mass spectra
20 obtained in scan mode are shown in Figure 2.

22 **Figure 2**

24 The figure displays the EI mass spectra and tentative fragmentation of the bis-*O*-TMS
25 derivative of bisphenol A and surrogate. Either the molecular ions $[M]^+$ or the $[M-CH_3]^+$
26 ions were the base peaks in the derivative. The molecular ion peak of silylated BPA
27 appears at 372 m/z , whereas the peak corresponding to loss of the methyl group is at 357
28 m/z . The mass spectra obtained for silylated BPF show the base peak at 344 m/z
29 corresponding to the molecular ion. Therefore, these ions were selected to be used as the
30 quantitation ions to obtain maximum detection sensitivity and specificity in the SIM mode.
31 All derivatives displayed an ion at m/z 73 $[(CH_3)_3Si]^+$ which was characteristic of the TMS
32 group and commonly observed in all TMS derivatives. The selected conditions for SIM
33 mode are shown in table 1.

Table 1*Analytical performance. Validation of the methodology*

Calibration graphs for samples treated according to the analytical procedure described above were made using SIM mode. BPF was used as surrogate.

The standard addition calibration was carried out by fortification of homogenized rice samples aliquots. 20 g of raw samples free of BPA were placed in a 50 mL falcon tubes collection. Then, samples were spiked with BPF as a surrogate ($100 \text{ ng}\cdot\text{g}^{-1}$) and with BPA as analyte at growing concentrations. The standard samples were strongly shaken in order to mix surrogate, analyte and sample and treated as described in the “*Extraction and derivatization*” section. Linearity of the calibration using standard addition graphs was tested according to the Analytical Methods Committee (Analytical Methods Committee, 1994). The *lack-of-fit* test was applied to the residuals of a calibration curve. Two experimental replicates and three injections of each standard were carried out. The results for the intercept (a), slope (b), correlation coefficient (R^2) and probability level of the *lack-of-fit* test, P_{lof} (%), are summarized in Table 2.

Table 2

Validation was performed according to the U.S. Food and Drug Administration (FDA) guideline for bioanalytical assay validation (U.S. Department of Health and Human Services, 2001). The analytical performance parameters assessed for the overall assay were linearity, precision, accuracy, sensitivity, and selectivity.

Linearity. A concentration range from the minimal amount detectable by this methodology up to two orders of magnitude higher ($0.X$ to 100 ng g^{-1}) was selected for method application.

The response of compound was checked in the range of application of the analytical method by linear regression analysis by the least-squares method of peak area ratio of analyte/surrogate against different analyte concentrations. The response was linear in the range of concentrations evaluated.

In the specific case of higher BPA concentration than the selected range, we should test the linearity of method beyond 100 ng g^{-1} or simply dilute the corresponding samples if necessary. In our application, no levels higher than 100 ng g^{-1} were found.

1 *Precision.* The precision expressed as relative standard deviation (RSD), at three
2 concentration levels, was obtained from thirty replicates of spiked samples obtained from
3 10 aliquots of a homogenate of 10 different rice dishes and analyzed during the same day
4 (repeatability) and in three different days (reproducibility). A total of 30 analyses were
5 carried out in order to calculate precision. The relative standard deviation is lower than
6 10% in all cases as is shown in Table 3. Data indicate that the analytical method is precise
7 (repeatable and reproducible). It is important to note that the precision varied with
8 concentration. Important factors such as sample weight, standard preparation, instrument
9 response or calibration uncertainty can limit precision.

10 *Accuracy.* A recovery assay was performed by comparing the analytical results for
11 extracted samples, free of BPA, spiked at three concentration levels. The concentration of
12 the compound was determined by interpolation in the standard addition calibration curve
13 within the linear dynamic range and compared with the added amount. Ten replicates, by
14 spiking 20g of rice samples with the analyte, at three concentration levels were analyzed.
15 The recoveries for BPA were between 90% and 105 % in all cases as is shown in Table 3.

Table 3

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19 The recoveries were quite good considering the amount of sample, and the low
20 concentration of the analyte and complexity of the sample. The values indicate that
21 compounds are quantitatively extracted.

22 *Sensitivity.* A fundamental aspect which needs to be examined in the validation of any
23 analytical method is its limit of detection in order to determine if an analyte is present in
24 the sample. In this work, a criterion for method performance has been used that includes
25 the decision limit, CC_{α} , and the detection capability, CC_{β} (COMMISSION DECISION,
26 2002). Decision limit (CC_{α}) is defined as the limit at and above which it can be concluded
27 with an error probability of α that a sample is non-compliant. Detection capability (CC_{β}) is
28 defined as the smallest content of the substance that may be detected, identified and/or
29 quantified in a sample with an error probability of β . They were calculated according to the
30 calibration curve procedure described in commission decision of 12 August 2002
31 implementing Council Directive 96/23/EC concerning the performance of analytical
32 methods and the interpretation of results. Decision limit and detection capacity which are

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1 better adjusted to a statistical evaluation are implemented. Thus, CC_{α} ($\alpha = 5\%$) and CC_{β} ($\beta = 5\%$) are also summarized in Table 2.

3 *Selectivity.* Compound was quantified using selected ion recording mode (SIM).
4 Analyte appear to be well resolved and free from interference peaks (Figure 1). The
5 identity of the chromatographic peak was confirmed not only by its retention time but also
6 by its mass spectrum (3 fragments were used).

8 *Application of the method*

9 The proposed method has been applied as a routine method for the determination of
10 BPA in prepared rice dishes packed in plastic containers from different brands
11 commercialized in Spain. These containers are manufactured using different plastic
12 materials which could include polycarbonate plastics. 250 samples of rice-prepared dishes,
13 picked up from different points were analyzed. An example of the chromatogram obtained
14 in SCAN mode and in SIM mode for a natural sample is included in Figure 1 (C and D).
15 BPA does not seem to be present above the reported lower detection limits proposed in this
16 work.

18 **Conclusions**

19 The present study shows that BPA can be detected and quantified reliably in rice-
20 prepared dishes combining GC-MS with a solid-liquid extraction procedure. The assay
21 involves a preconcentration and the removal of interferences step in conjunction with a
22 silyl-derivatization procedure previous to CG-MS analysis. The GC-MS analytical method
23 developed in this study has been shown to be reliable and had a low limit of detection for
24 BPA, and has been successfully applied to spiked and non-spiked rice samples. Proper
25 sample collection in conjunction with sound storage practice prior to analysis allows for
26 good recovery values in all cases as demonstrated by the validation procedure employed.
27 The method has been used routinely for monitoring the presence of BPA in the production
28 of rice-prepared dishes and no contamination has been found.

29 Our results indicate that the levels of BPA identified in rice-prepared dishes are
30 unlikely to be of concern to adult health, and there is no reason for consumers to change
31 their consumption patterns as a result of these findings.

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FIGURE CAPTIONS

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3 **Figure 1.** Chromatograms of a spiked sample in SCAN (A), SIM (B) mode ($100 \text{ ng}\cdot\text{g}^{-1}$)
4 and a non-spiked sample in SCAN mode (C) and SIM mode (D).

5
6 **Figure 2.** Mass spectra of bisphenol A and surrogate (bisphenol F) in a spiked sample.

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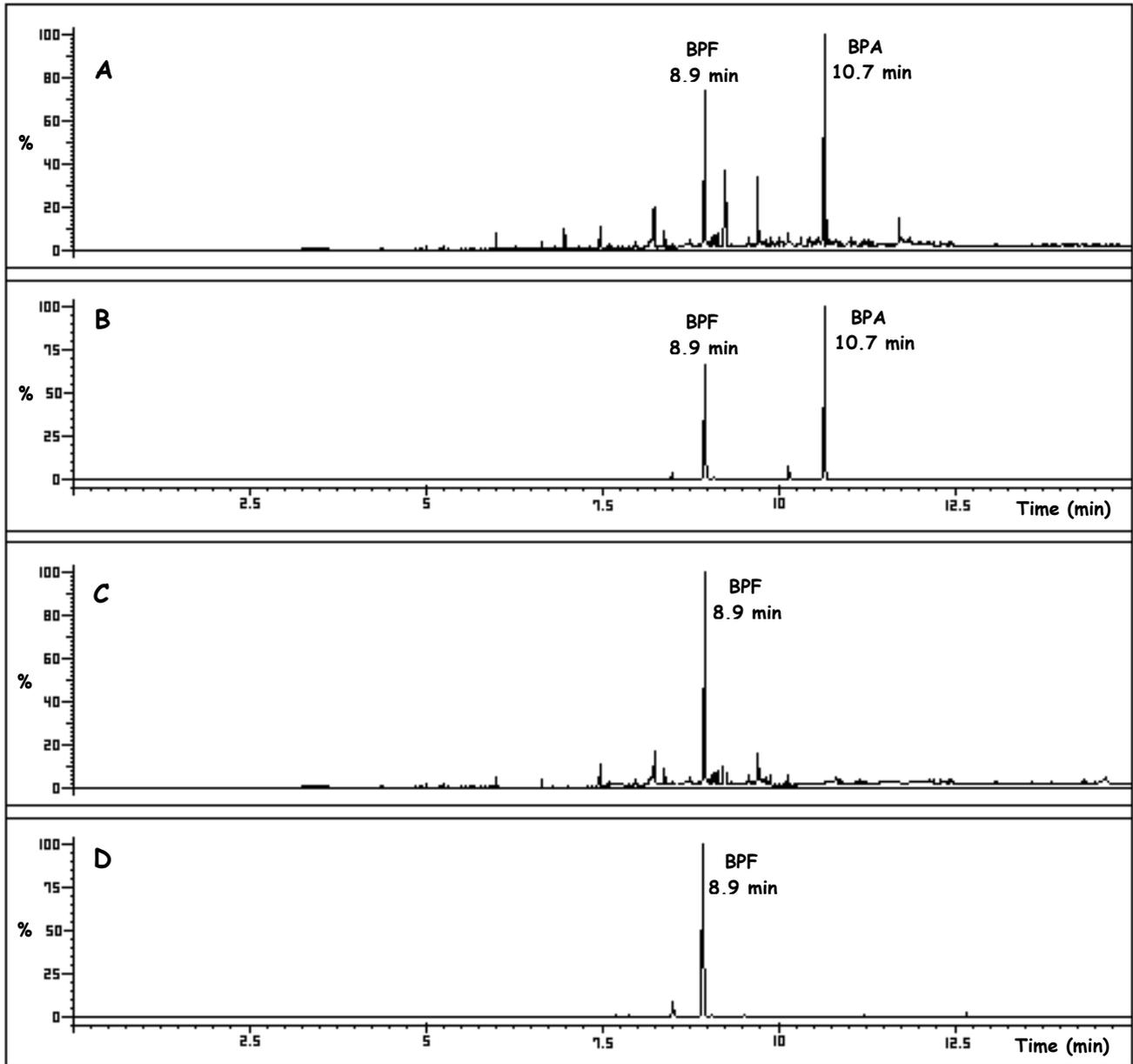


Figure 1

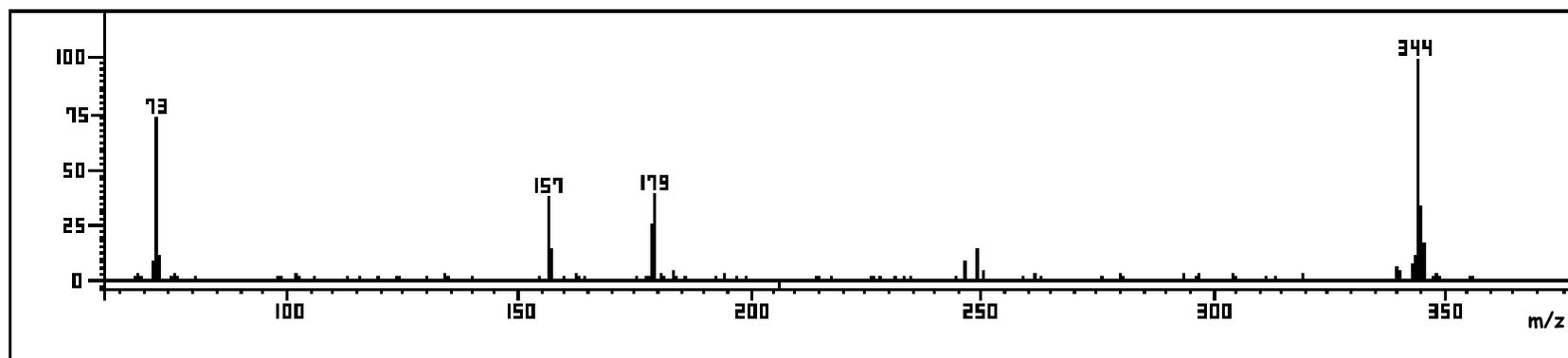
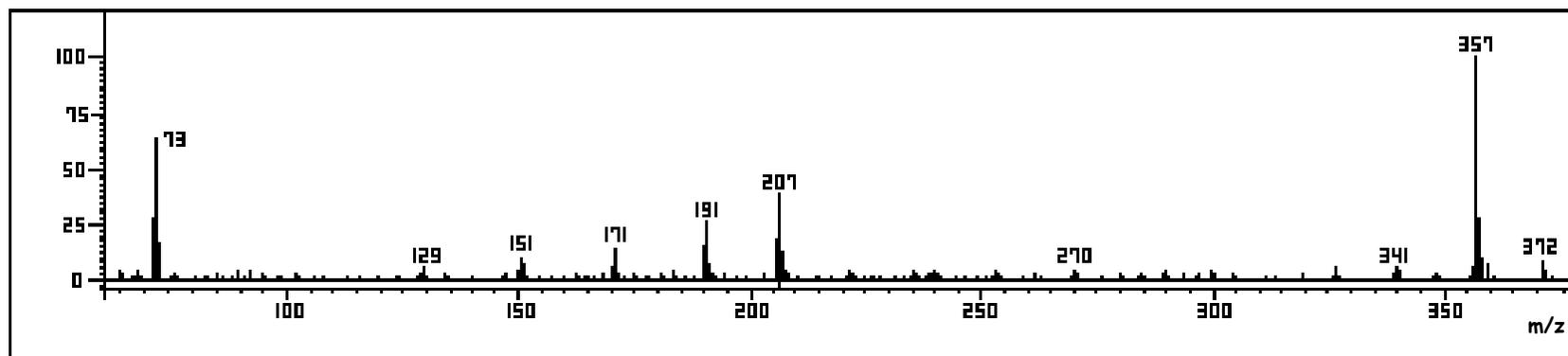


Figure 2

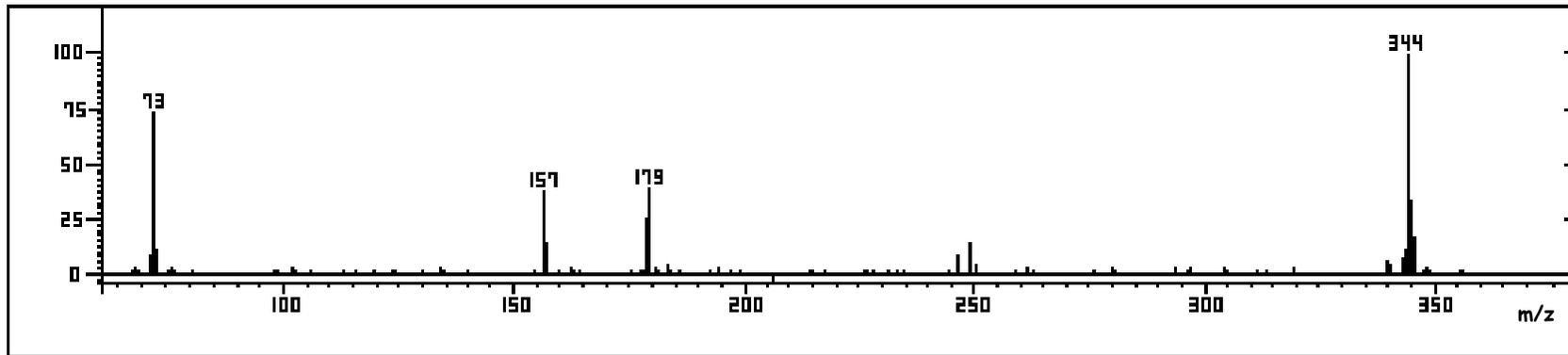
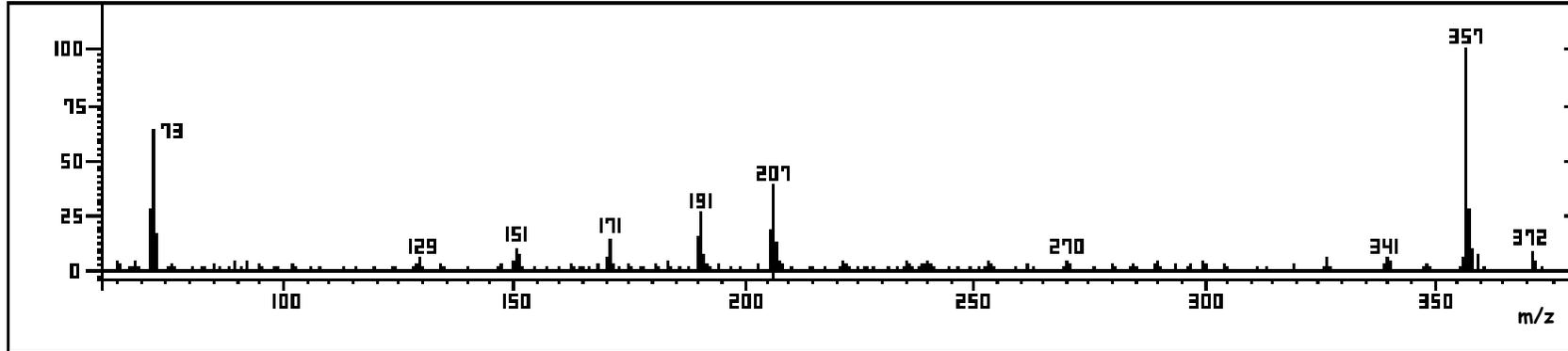


Figure 2

Table 1. SIM mode characterization and structural assignments of the fragments.

	t_r (min)	Dwell time (ms)	Range (min)	Fragments (m/z)
BPF	8.9	100	7.5 - 10.0	344 [M] ⁺ , 179, 157
BPA	10.7	100	10.0 - 15.0	357 [M – Met] ⁺ , 207, 179

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Table 2. Analytical and statistical parameters of the proposed method.

Parameter	Value
n	6
a	$3.0 \cdot 10^{-3}$
s_a	$2.8 \cdot 10^{-3}$
b (g ng⁻¹)	$1.20 \cdot 10^{-2}$
s_b (g·ng⁻¹)	$1.25 \cdot 10^{-3}$
R² (%)	99.7
LDR (ng g⁻¹)	0.8 - 100.0
S_{y/x}	$2.45 \cdot 10^{-3}$
CC_α (ng g⁻¹)	0.5
CC_β (ng g⁻¹)	0.8
P_{lof} (%)	53.1

n, calibration levels; **a**, intercept; **S_a**, intercept standard deviation; **b**, slope; **S_b**, slope standard deviation; **R²**, determination coefficient; **LDR**, linear dynamic range; **S_{y/x}**, regression standard deviation; **CC_{α,0.05}**, decision limit; **CC_{β,0.05}**, detection capability; **P_{lof}**, P-value for *lack-of-fit* test.

Table 3. Recovery assay, precision (repeatability and reproducibility) and accuracy of BPA ($n = 30$).

	Spiked (ng g ⁻¹)	Found* ± SD (% , RSD)	Recovery (%)
	10.0	9.3 ± 0.7 (6.4)	93.0
Bisphenol A	50.0	52.8 ± 2.5 (4.7)	105.6
	100.0	93.1 ± 4.1 (4.4)	93.1

* Mean of thirty determinations (ng·g⁻¹); **SD**: standard deviation; **RSD (%)**: relative standard deviation.

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