

## Supplementary Information

### Development of a Method Based on DLLME and UFLC-DAD for the Determination of Antibiotics in Honey Samples and the Study of Their Degradation Kinetics

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**Table S1.** Characteristics of the honey samples: place of acquisition, city and state of origin, blooming type and dates of production, validity and analysis

Sample	Place of acquisition	City and state of origin	Blooming type	Date of production	Validity	Date of analysis
1	trade	Palmas-TO	wildflowers	08/2012	08/2014	10/2012
2	trade	Serrinha-BA	wildflowers	09/2013	09/2015	05/2014
3	trade	São Felipe-BA	wildflowers	06/2013	06/2015	08/2014
4	trade	Feira de Santana-BA	wildflowers	12/2013	12/2015	08/2014
5	trade	Camaçari-BA	wildflowers	01/2014	01/2016	08/2014
6	trade	Aquiraz-CE	wildflowers	06/2014	06/2016	01/2015
7	trade	São Felipe-BA	wildflowers	12/2014	12/2016	01/2015
8	trade	Araripina-PE	wildflowers	08/2014	08/2016	01/2015
9	trade	Limoeiro do Norte-CE	wildflowers	01/2014	01/2016	01/2015
10	trade	Goiânia-GO	eucalyptus	06/2014	06/2016	01/2015
11	trade	Maceió-AL	wildflowers	05/2014	05/2016	01/2015
12	trade	Aquiraz-CE	wildflowers	09/2014	09/2016	01/2015
13	trade	Aquiraz-CE	wildflowers	02/2014	02/2016	01/2015
14	producer	Feira de Santana-BA	orange	11/2014	ND	01/2015
15	producer	Feira de Santana-BA	cashew	11/2014	ND	01/2015
16	producer	Beira Rio-BA	wildflowers	11/2014	ND	01/2015
17	producer	Alagoinhas-BA	wildflowers	11/2014	ND	01/2015
18	producer	Feira de Santana-BA	wildflowers	11/2014	ND	01/2015
19	producer	Feira de Santana-BA	wildflowers	11/2014	ND	01/2015
20	producer	Macaubas-BA	wildflowers	11/2014	ND	01/2015
21	producer	Riachão das Neves-BA	wildflowers	11/2014	ND	01/2015
22	producer	Itaberaba-BA	wildflowers	11/2014	ND	01/2015

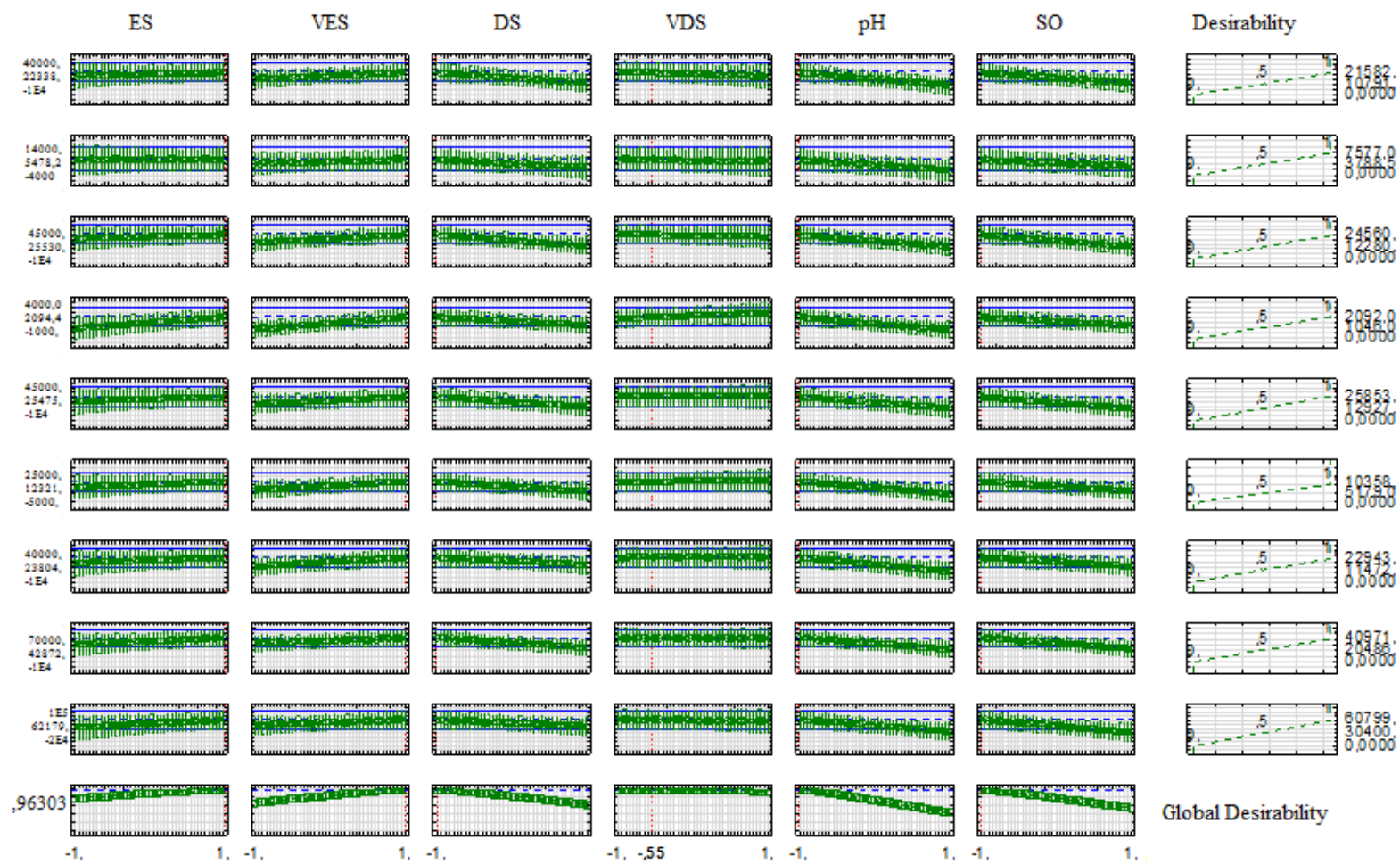
23	producer	Barreiras-BA	wildflowers	11/2014	ND	01/2015
24	producer	Muquem do São Francisco-BA	wildflowers	12/2014	ND	01/2015
25	producer	Angical-BA	wildflowers	12/2014	ND	01/2015
26	producer	Ibotirama-BA	wildflowers	12/2014	ND	01/2015
27	producer	Jupaguá-BA	wildflowers	12/2014	ND	01/2015
28	producer	Formosa do Rio Preto-BA	wildflowers	12/2014	ND	01/2015
29	producer	Cotegipe-BA	wildflowers	12/2014	ND	01/2015
30	producer	Buracica-BA	wildflowers	12/2014	ND	01/2015
31	producer	São Desidério-BA	wildflowers	12/2014	ND	01/2015
32	trade	Aquiraz-CE	wildflowers	08/2014	08/2016	01/2015
33	trade	Feira de Santana-BA	wildflowers	01/2014	01/2016	01/2015

ND: not declared in the label.

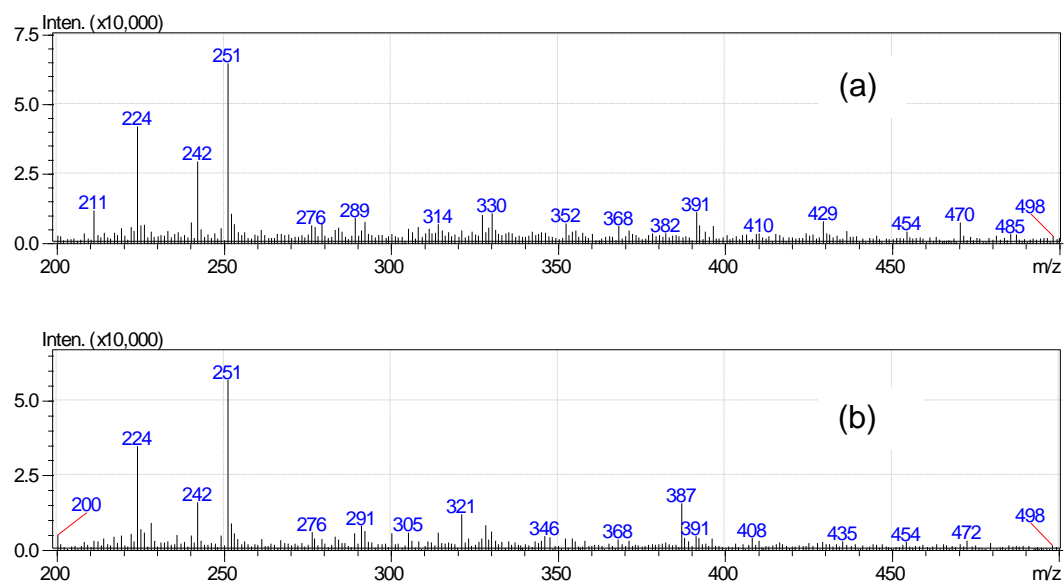
**Table S2.** Factors and levels of the fractional factorial design

Factor	Low (-1)	High (+1)
ES	dichloromethane	chloroform
DS	acetonitrile	methanol
ESV / $\mu\text{L}$	100	200
DSV / $\mu\text{L}$	500	1000
SO / g	0.3	0.8
pH	3.0	5.0

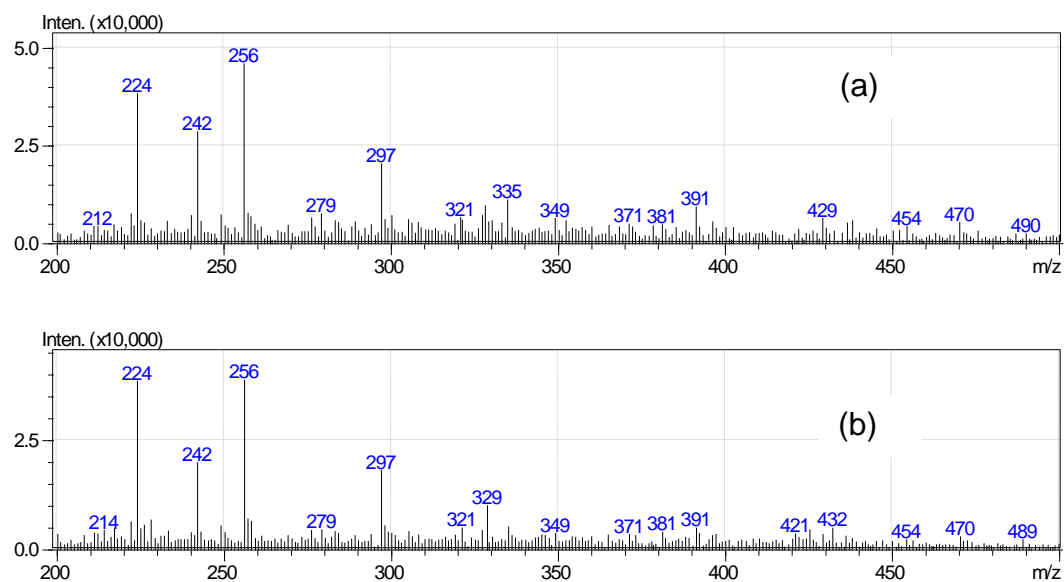
ES: extractant solvent; DS: dispersant solvent; ESV: volume of the extractant solvent; DSV: volume of the dispersant solvent; SO: mass of NaCl.



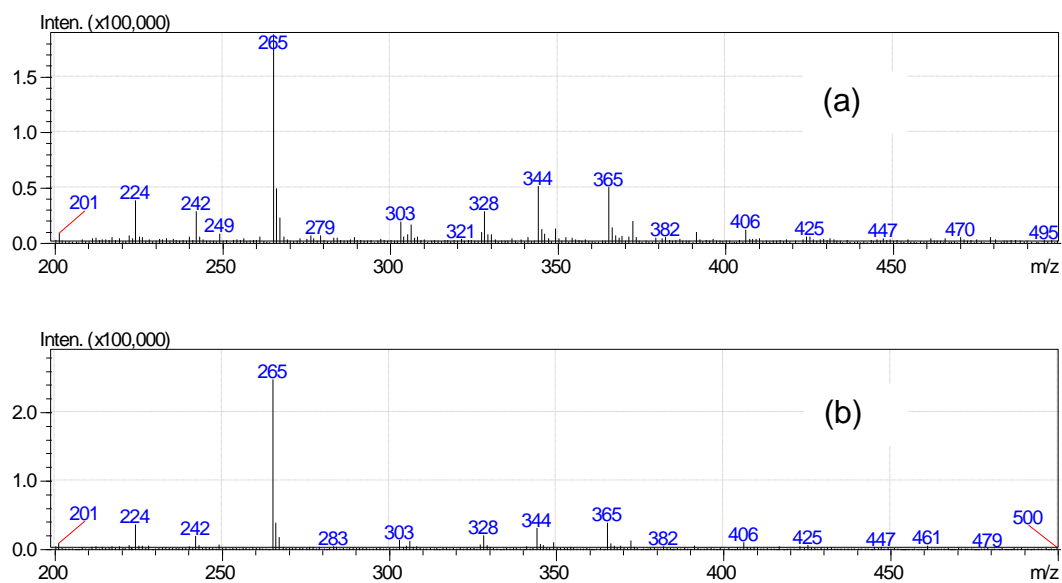
**Figure S1.** Application of the Derringer and Suich methodology for the fractional factorial planning proposed for optimization of the DLLME method. ES: extractant solvent; VES: volume of the extractant solvent; DS: dispersant solvent; VDS: volume of the dispersant solvent; SO: salting out effect.



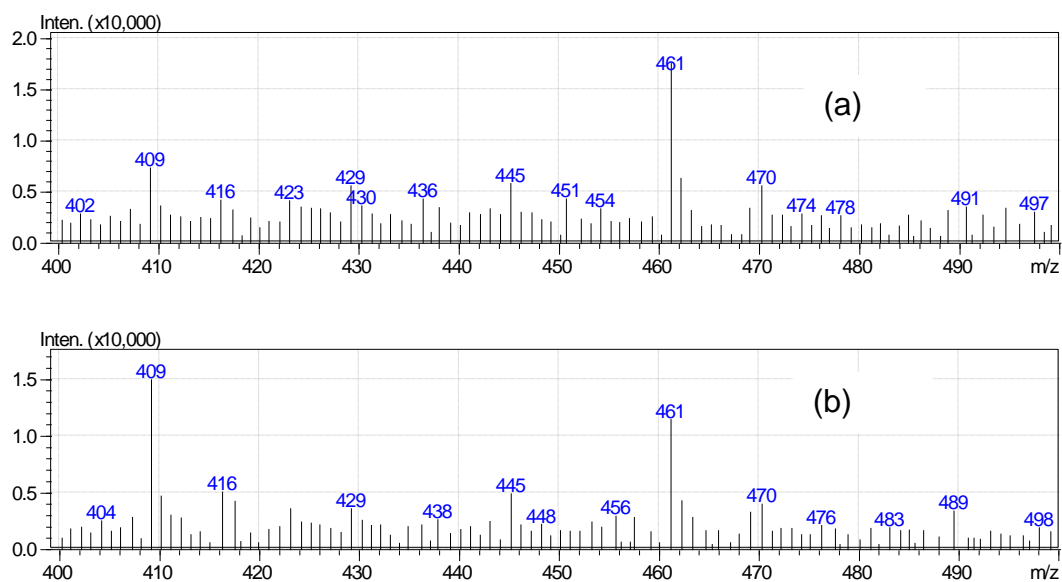
**Figure S2.** LC-MS spectra of (a) direct injection of the sulfadiazine standard; (b) sulfadiazine extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.



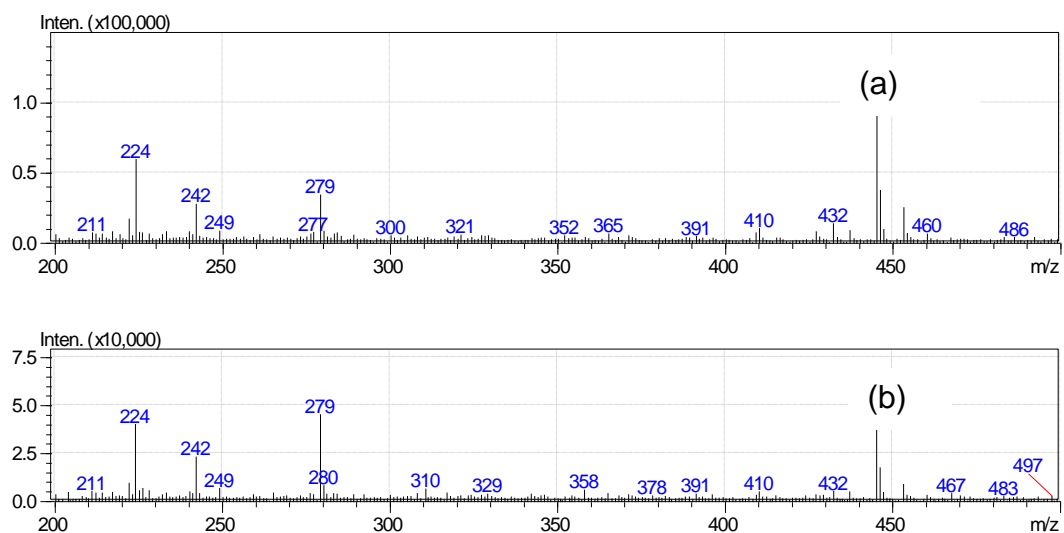
**Figure S3.** LC-MS spectra of (a) direct injection of the sulfathiazole standard; (b) sulfathiazole extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.



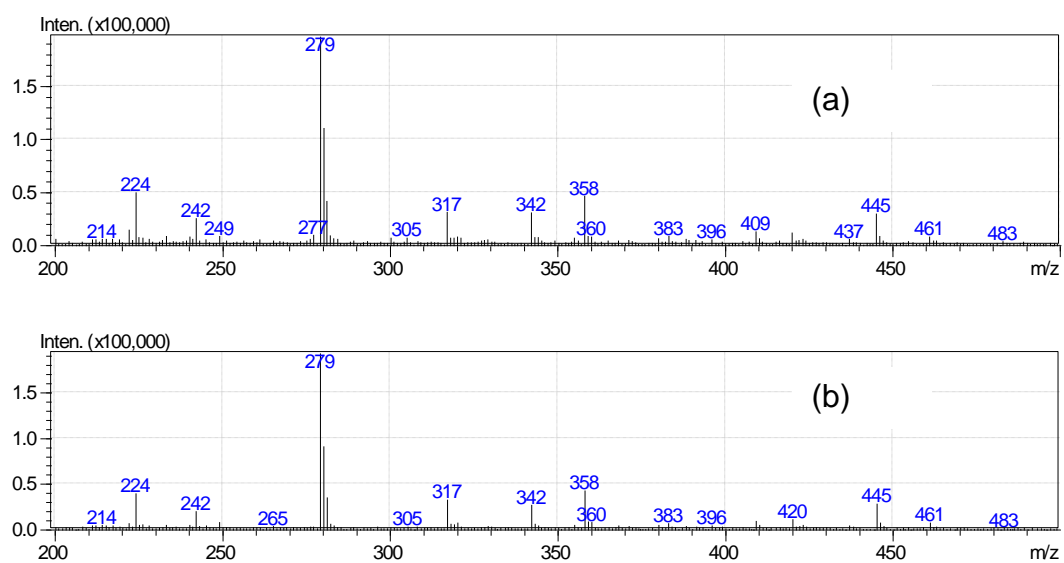
**Figure S4.** LC-MS spectra of (a) direct injection of the sulfamerazine standard; (b) sulfamerazine extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.



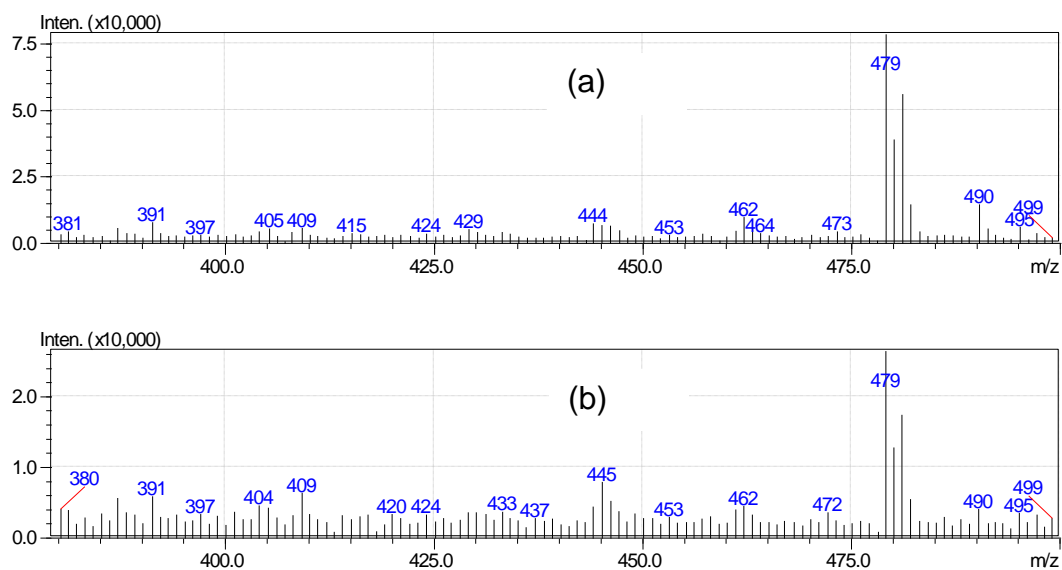
**Figure S5.** LC-MS spectra of (a) direct injection of the oxytetracycline standard; (b) oxytetracycline extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.



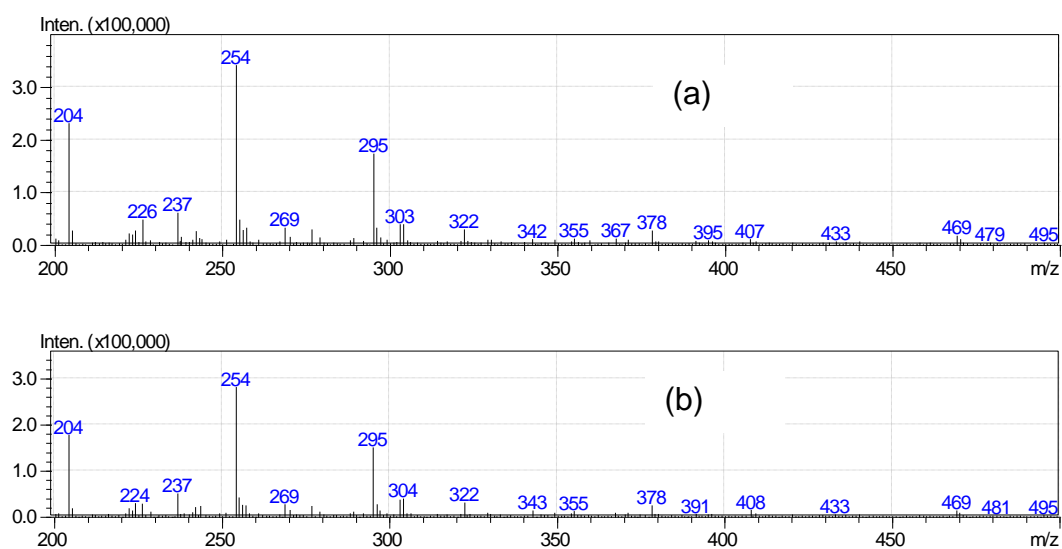
**Figure S6.** LC-MS spectra of (a) direct injection of the tetracycline standard; (b) tetracycline extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.



**Figure S7.** LC-MS spectra of (a) direct injection of the sulfamethazine standard; (b) sulfamethazine extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.

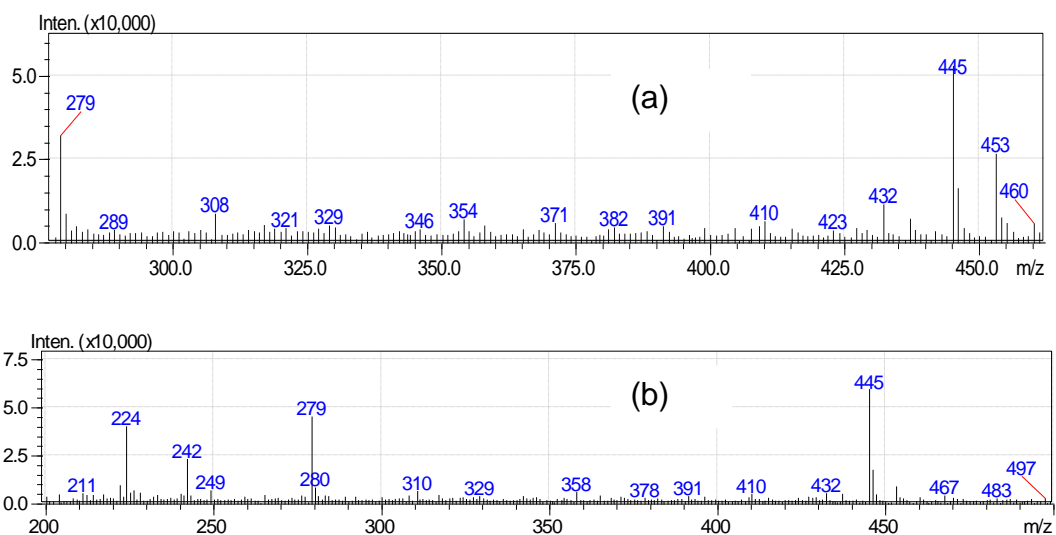


**Figure S8.** LC-MS spectra of (a) direct injection of the chlortetracycline standard; (b) chlortetracycline extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.

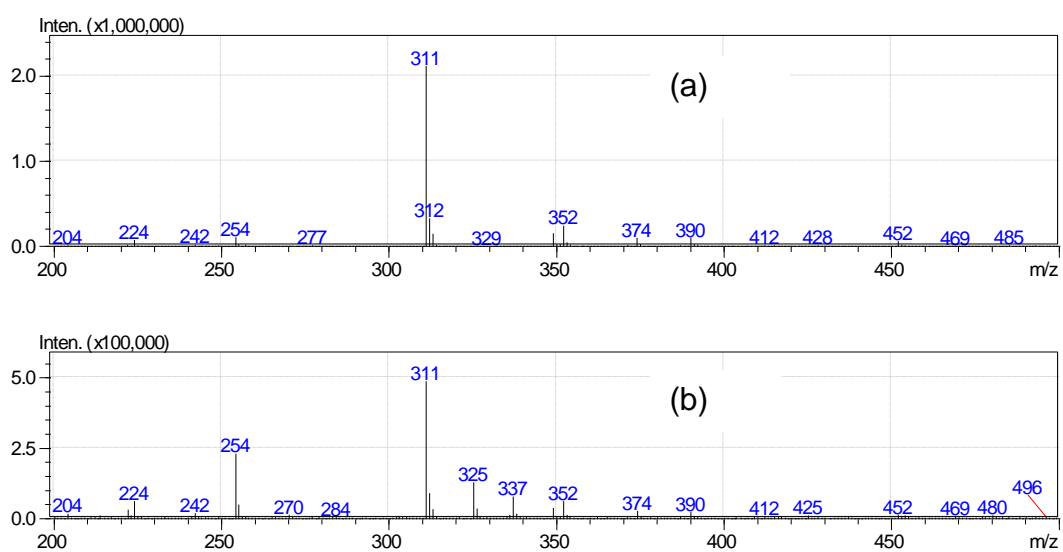


**Figure S9.** LC-MS spectra of (a) direct injection of the sulfamethoxazole standard; (b) sulfamethoxazole extracted from the honey sample, fortified at 100 µg kg<sup>-1</sup>.





**Figure S10.** LC-MS spectra of (a) direct injection of the doxycycline standard; (b) doxycycline extracted from the honey sample, fortified at  $100 \mu\text{g kg}^{-1}$ .



**Figure S11.** LC-MS spectra of (a) direct injection of the sulfadimethoxine standard; (b) sulfadimethoxine extracted from the honey sample, fortified at  $100 \mu\text{g kg}^{-1}$ .