

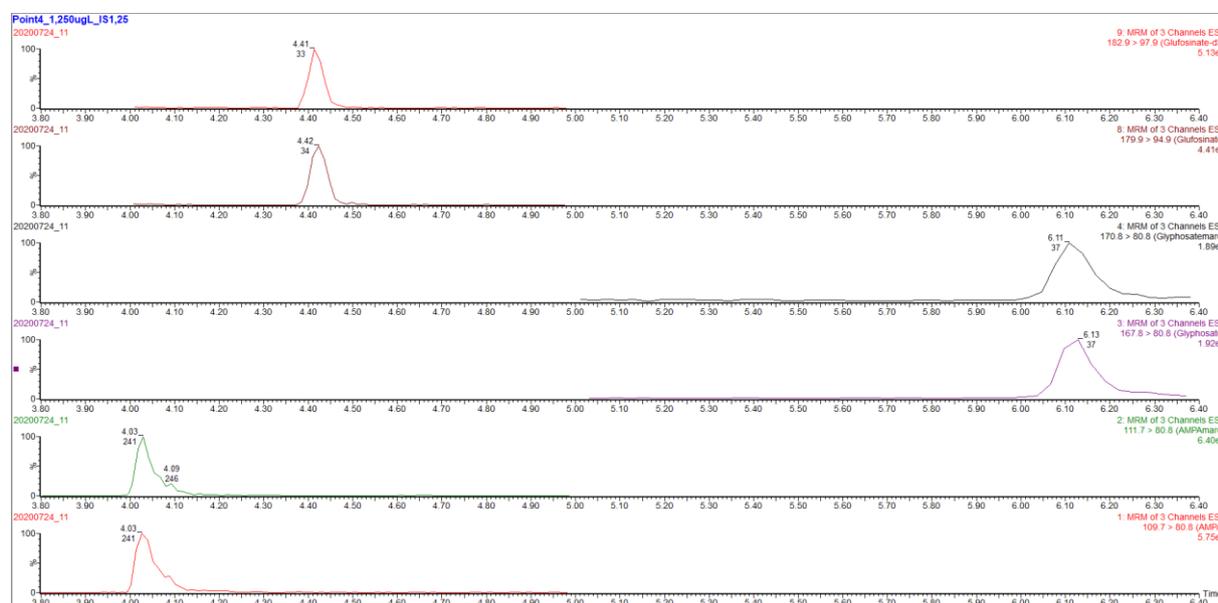
# Determination of glyphosate and AMPA in indoor settled dust by hydrophilic interaction liquid chromatography with tandem mass spectrometry and implications for human exposure

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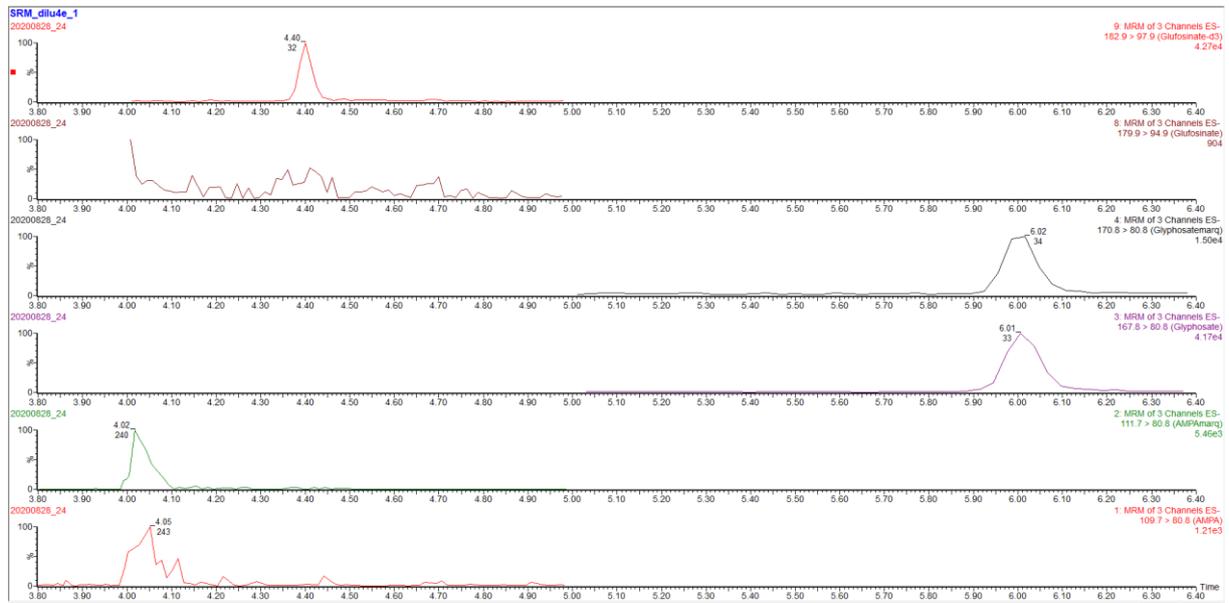
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## Supplementary material

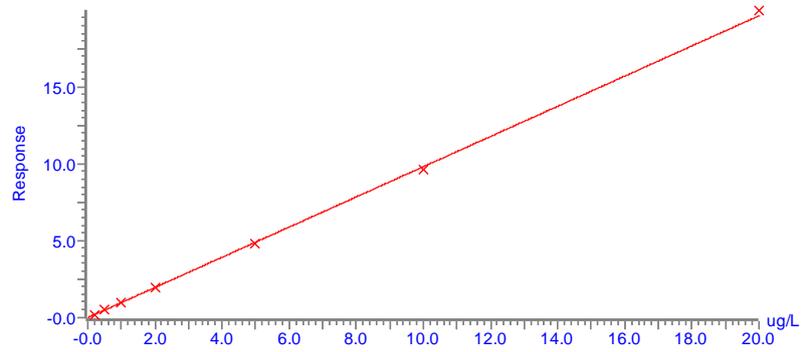


SI Figure 1: MRM chromatogram of AMPA, glufosinate, glyphosate, and their internal standards in a 1.25 µg/L calibration standard

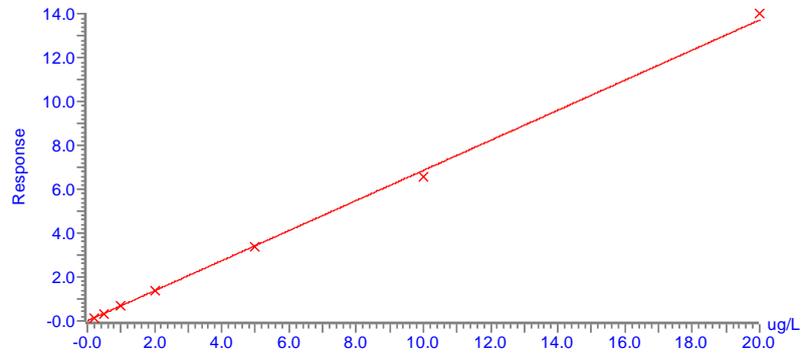


SI Figure 2: MRM chromatogram of AMPA, glufosinate, glyphosate, and their internal standards in the standard reference material SRM 2585.

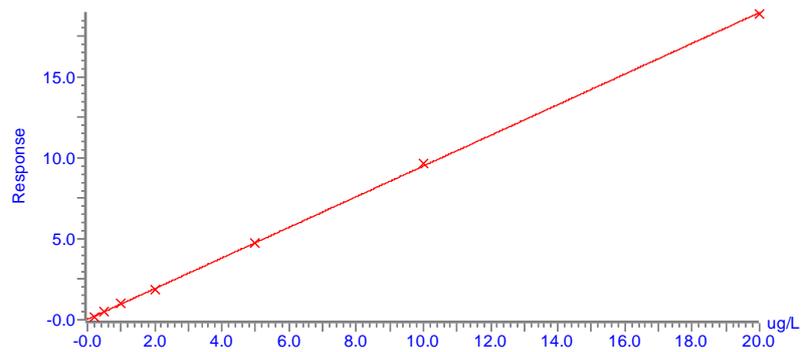
Compound name: AMPA 109.7 > 62.9  
Correlation coefficient:  $r = 0.999792$ ,  $r^2 = 0.999584$   
Calibration curve:  $0.983933 * x + -0.0300116$   
Response type: Internal Std ( Ref 4 ), Area \* ( IS Conc. / IS Area )  
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



Compound name: Glufosinate 179.9 > 118.9  
Correlation coefficient:  $r = 0.999578$ ,  $r^2 = 0.999157$   
Calibration curve:  $0.687236 * x + -0.00894083$   
Response type: Internal Std ( Ref 11 ), Area \* ( IS Conc. / IS Area )  
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



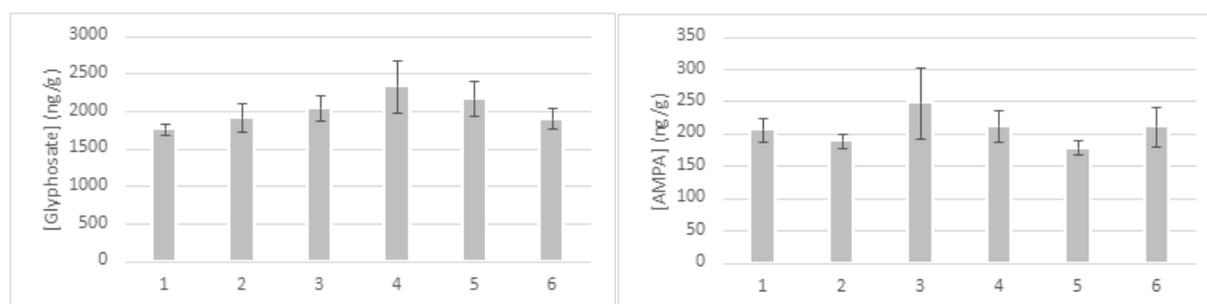
Compound name: Glyphosate 167.8 > 62.9  
Correlation coefficient:  $r = 0.999888$ ,  $r^2 = 0.999777$   
Calibration curve:  $0.947319 * x + 0.0270253$   
Response type: Internal Std ( Ref 19 ), Area \* ( IS Conc. / IS Area )  
Curve type: Linear, Origin: Exclude, Weighting: 1/x, Axis trans: None



SI Figure 3: Calibration curves for AMPA, glufosinate, and glyphosate

SI Table 1: Extraction yields of the internal standards AMPA-<sup>13</sup>C, <sup>15</sup>N, glufosinate-d<sub>3</sub>, and glyphosate-<sup>13</sup>C<sub>2</sub>, <sup>15</sup>N

Compound	Spiked (ng/g)	Recovery (%)				
		Dust C	SRM 2585	Dust D	Mean (%)	RSD (%)
AMPA- <sup>13</sup> C, <sup>15</sup> N	50	93%	88%	90%	91%	3%
	250	86%	79%	91%	85%	7%
	500	89%	82%	81%	84%	5%
Glufosinate-d <sub>3</sub>	50	93%	102%	117%	104%	12%
	250	100%	108%	92%	100%	8%
	500	98%	95%	90%	95%	4%
Glyphosate- <sup>13</sup> C <sub>2</sub> , <sup>15</sup> N	50	87%	101%	85%	91%	10%
	250	75%	71%	107%	84%	24%
	500	93%	79%	87%	86%	8%



SI Figure 4: Glyphosate and AMPA concentrations in Dust D for six series of three extractions

SI Table 2. Repeatability and reproducibility data for glyphosate

Series	Concentration (ng/g)		
	1	2	3
1	1763.00	1693.00	1838.00
2	2093.00	1711.00	1920.00
3	2225.00	1908.00	1984.00
4	2610.00	1936.00	2440.00
5	2440.00	2096.00	1984.00
6	2059.00	1804.00	1843.00

Number of series (I)	<b>6</b>
Number of measures (IJ)	<b>18</b>
Number of repetitions (J)	<b>3</b>
Sum of squares of residual deviations	<b>534880.00</b>
Sum of squares of total deviations	<b>1167301.61</b>
Sum of squares of inter-range differences	<b>632421.61</b>
Intermediate calculation of $s^2_B$	<b>27303.66</b>
Repeatability variance ( $s^2_r$ )	<b>44573.33</b>
Inter-series variance ( $s^2_B$ )	<b>27303.66</b>
Fidelity variance ( $s^2_{FI}$ )	<b>71877.00</b>

#### Fidelity

Average activity recovered	<b>2019.28</b>
Standard deviation of repeatability (sr)	<b>211.12</b>
Inter-series standard deviation (sB)	<b>165.24</b>
Standard deviation of fidelity (sFI)	<b>268.10</b>

%RSD

<b>10%</b>
<b>8%</b>
<b>13%</b>

Calculations according to NF V03-110

SI Table 3. Repeatability and reproducibility data for AMPA

Series	Concentration (ng/g)		
	1	2	3
1	207.00	223.00	188.00
2	183.00	202.00	183.00
3	272.00	287.00	184.00
4	185.00	226.00	226.00
5	165.00	184.00	185.00
6	241.00	179.00	213.00

Number of series (I)	<b>6</b>
Number of measures (IJ)	<b>18</b>
Number of repetitions (J)	<b>3</b>
Sum of squares of residual deviations	<b>10350.00</b>
Sum of squares of total deviations	<b>18904.28</b>
Sum of squares of inter-range differences	<b>8554.28</b>
Intermediate calculation of $s^2_B$	<b>282.79</b>
Repeatability variance ( $s^2_r$ )	<b>862.50</b>
Inter-series variance ( $s^2_B$ )	<b>282.79</b>
Fidelity variance ( $s^2_{FI}$ )	<b>1145.29</b>

#### Fidelity

Average activity recovered	<b>207.39</b>
Standard deviation of repeatability (sr)	<b>29.37</b>
Inter-series standard deviation (sB)	<b>16.82</b>
Standard deviation of fidelity (sFI)	<b>33.84</b>

%RSD

<b>14%</b>
<b>8%</b>
<b>16%</b>

Calculations according to NF V03-110

SI Table 4: Limits of quantification (LOQ) for glyphosate and AMPA according to analytical technique and sample mass

Reference	Analytical technique	Sample mass	LOQ in ng/g for Glyphosate	Equivalent LOQ for 25 mg for glyphosate	LOQ in ng/g for AMPA	Equivalent LOQ for 25 mg for AMPA
(Mendez et al., 2017)	FMOC/C18	2g (soil)	1,19	95,2	1,6	128
(Islas et al., 2014)	NQS/C18	0,1 g (soil)	180	720	270	1080
(Bento et al., 2016)	FMOC/C18	2 g (soil)	50	4000	50	4000
(Sun et al., 2017)	FMOC/C18	10 g (soil)	40	16000	-	-
(Delhomme et al., 2021)	FMOC/C18	15 g (soil)	30	18000	25	15000
This Study	HILIC	25 mg (indoor dust)	25	25	25	25

FMOC/C18: 9-fluorenylmethoxycarbonyl chloride (FMOC) derivatization / reverse phase (C18) UHPLC/MS-MS ; NQS/C18: 1,2-Napthoquinine-4-sulfonate (NQS) derivatizaion / reverse phase (C18) HPLC / UV ; HILIC : no derivatization / normal phase (HILIC) / UPLC/MS-MS

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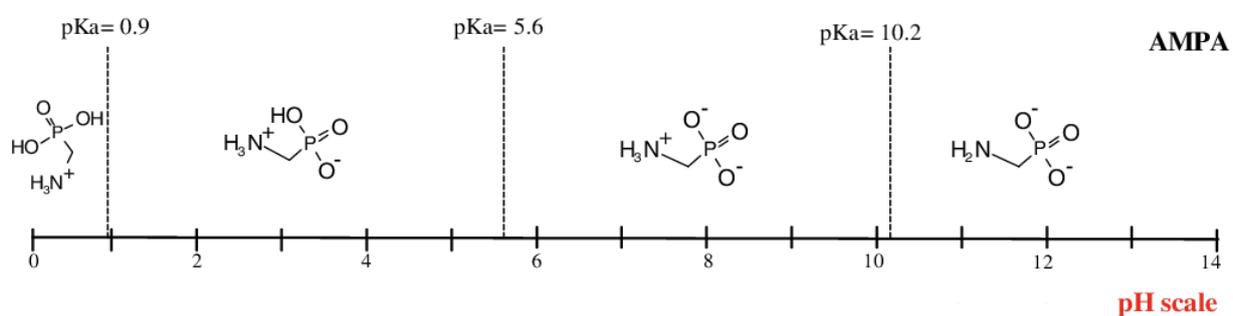
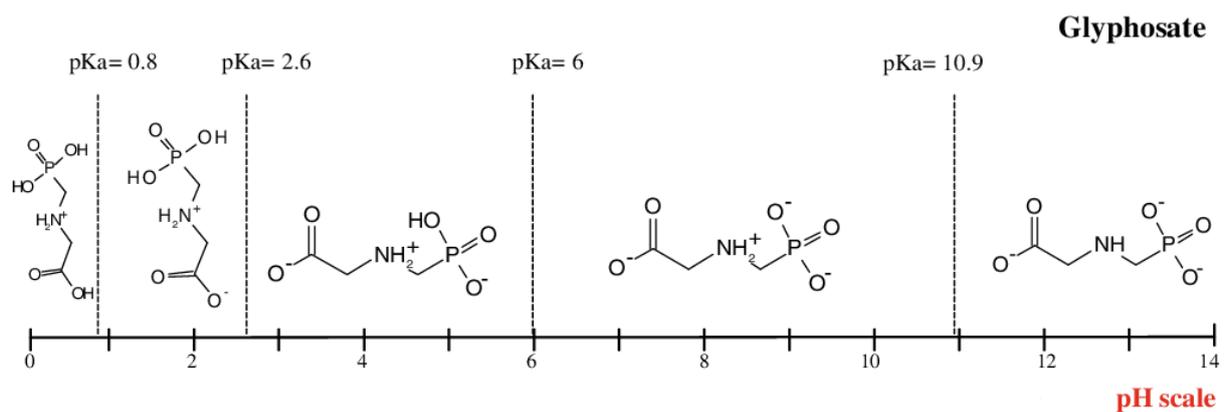
Delhomme, O., Rodrigues, A., Hernandez, A., Chimjarn, S., Bertrand, C., Bourdat-Deschamps, M., Fritsch, C., Pelosi, C., Néliu, S., Millet, M., 2021. A method to assess glyphosate, glufosinate and aminomethylphosphonic acid in soil and earthworms. *J. Chromatogr. A* 1651, 462339. <https://doi.org/https://doi.org/10.1016/j.chroma.2021.462339>

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Sun, L., Kong, D., Gu, W., Guo, X., Tao, W., Shan, Z., Wang, Y., Wang, N., 2017. Determination of glyphosate in soil/sludge by high performance liquid chromatography. *J. Chromatogr. A* 1502, 8–13. <https://doi.org/10.1016/j.chroma.2017.04.018>



SI Figure 5: Illustration of the zwitterionic forms and of glyphosate and AMPA (Mogusu et al., 2015)