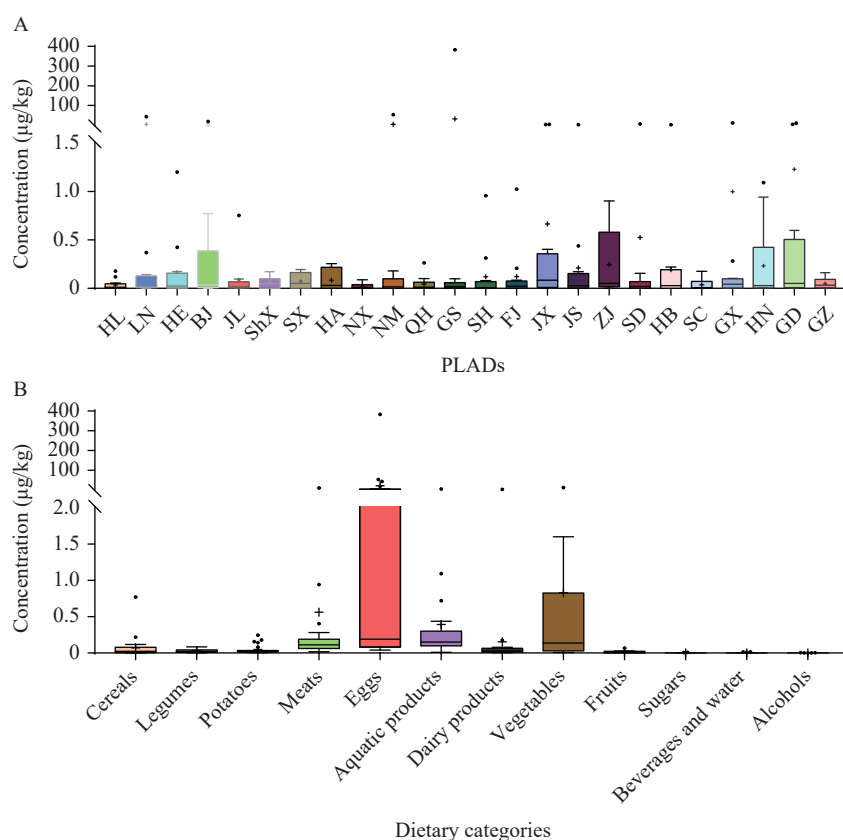


Supplementary Data

SUPPLEMENTARY TABLE S1. Mass spectrometric parameters of fipronil and its metabolites.

Analytes	Parent ion [M-H] ⁻ (m/z)	Fragment ions (m/z)
Fipronil	434.931	329.960, 249.958
Fipronil desulfinyl	386.969	350.987, 281.992
Fipronil sulfone	450.926	414.950, 281.992
Fipronil sulfide	418.936	382.960, 313.964
Fipronil- ¹³ C ₄ ¹⁵ N ₂	440.938	/

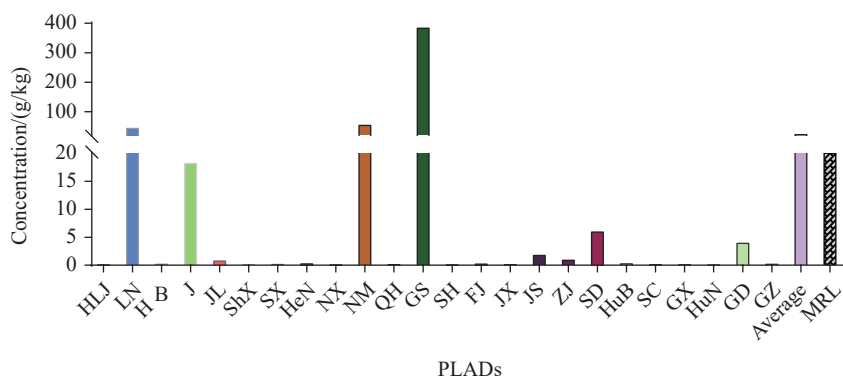
Note: “/” indicated that fragment ion information was not given. For chromatographic separation, the analytes were separated on an ACQUITY BEH C18 (100 mm×2.1 mm, 1.7 μm) column at 40 °C. A gradient elution was performed using water containing 0.1% formic acid (A) and methanol (B). The elution program was performed as follows: 0–6 min, 60%–80%B; 6–7 min, 80%–100%B; 7–8 min, 100%B; 8–8.1 min, 100%–60%B; 8.1–11 min, 60%B. The flow rate was 0.3 mL/min, and the injection volume was 5 μL.



SUPPLEMENTARY FIGURE S1. Total fipronil (FIPs) residue concentration. (A) Total FIPs residue concentration in 24 provincial-level administrative divisions from the Sixth China Total Diet Study (TDS); (B) Total FIPs residue concentration in different food categories from the Sixth China TDS, respectively. Concentrations are plotted on a box-and-whisker plot with 12 quartiles. Box limits were for the first and third quartiles, and the band inside the box was the second quartile (the median). Boxplot whiskers extended to 1.5 times the interquartile range. Outliers were represented by solid circles; “+” symbols indicate the mean value of residue data.

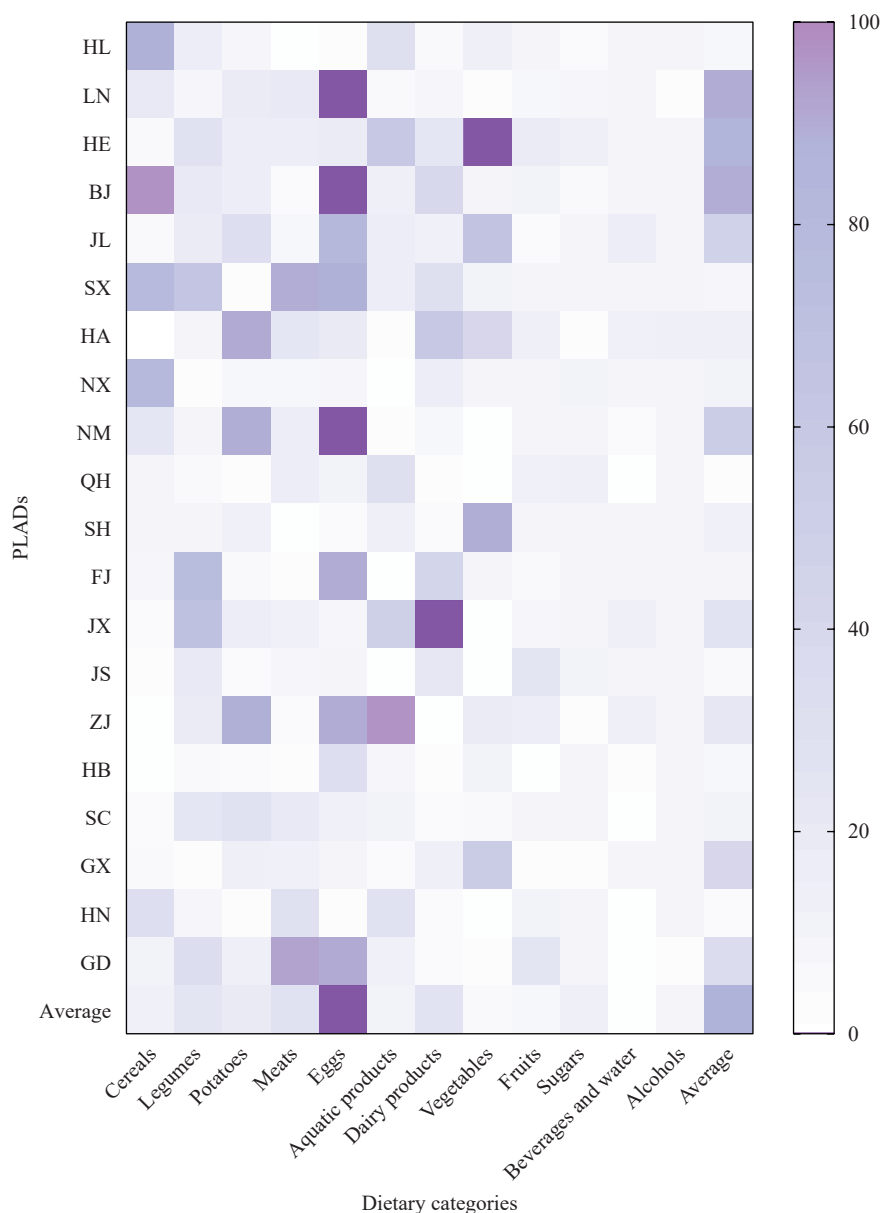
Abbreviations: TDS= total diet study; HLJ=Heilongjiang; LN=Liaoning; HE=Hebei; BJ=Beijing; JL=Jilin; SX=Shanxi; SN=Shaanxi; HA=Henan; NX=Ningxia; NM=Inner Mongolia; QH=Qinghai; GS=Gansu; SH=Shanghai; FJ=Fujian; JX=Jiangxi; JS=Jiangsu; ZJ=Zhejiang; SD=Shandong; HB=Hubei; SC=Sichuan; GX=Guangxi; HN=Hunan; GD=Guangdong; GZ=Guizhou.

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SUPPLEMENTARY FIGURE S2. Detection levels of total fipronils in egg samples from the 24 PLADs and the maximum residue limit value.

Abbreviations: HL=Heilongjiang; LN=Liaoning; HE=Hebei; BJ=Beijing; JL=Jilin; SX=Shanxi; SN=Shaanxi; HA=Henan; NX=Ningxia; NM=Inner Mongolia; QH=Qinghai; GS=Gansu; SH=Shanghai; FJ=Fujian; JX=Jiangxi; JS=Jiangsu; ZJ=Zhejiang; SD=Shandong; HB=Hubei; SC=Sichuan; GX=Guangxi; HN=Hunan; GD=Guangdong; GZ=Guizhou; PLADs=provincial-level administrative divisions.



SUPPLEMENTARY FIGURE S3. Relative changes in ratio value of total fipronils (FIPs) concentration from 20 PLADs between the Fifth and Sixth China TDS.

Notes: The white and purple squares in the heatmap indicate decreased and increased residue levels of total FIPs, respectively.

Abbreviations: HL= Heilongjiang; LN=Liaoning; HE=Hebei; BJ=Beijing; JL=Jilin; SX=Shanxi; HA=Henan; NX=Ningxia; NM=Inner Mongolia; QH=Qinghai; SH=Shanghai; FJ=Fujian; JX=Jiangxi; JS=Jiangsu; ZJ=Zhejiang; HB=Hubei; SC=Sichuan; GX=Guangxi; HN=Hunan; GD=Guangdong; PLADs=provincial-level administrative divisions; TDS=Total Diet Study.