IN ESSENCE

Analysis of Fipronil and Fipronil Sulfone in Eggs, Chicken Meat and Mayonnaise

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Fipronil is a broad spectrum insecticide that is in the European Union (EU)[1] not permitted to be use with food producing animals. Fipronil sulfone is formed by degradation of fipronil, and is actually more toxic to birds and other organisms than the parent compound.[2] The maximum residue limit designated by the EU for fipronil in eggs 5 ng/g (.005 mg/Kg), reported as a sum of the parent compound and the sulfone degradant.

In this application, QuEChERS extraction and cleanup (see **Figure 1**) followed by GC/MS/MS analysis (conditions listed in **Table 1**) were used for spiked samples that were quantitated against a matrix-matched calibration curve. No internal standard was used, thus recoveries reported are absolute.

Supel™ Que Z-Sep+, used for extract cleanup, was found to significantly reduce levels of co-extracted fatty compounds, including cholesterol. **Figure 2** shows the reduced background after cleanup with Z-Sep+. The fatty acids, eluting in the same retention range as the fipronil and fipronil sulfone, were removed by the Z-Sep+ cleanup, resulting in a clean signal for both compounds at 5 ng/mL in the final extract (**Figure 3**). Recovery and reproducibility of the method was good (**Table 2**). The method was also applied to chicken meat and mayonnaise. The ruggedness test of the GC method was done by repeated injections (>70) of egg sample extracts, resulting in only a small change in signal throughout the run, with a variation of 12%.

Figure 1. Sample preparation procedure, QuEChERS extraction and cleanup with Z-Sep+.

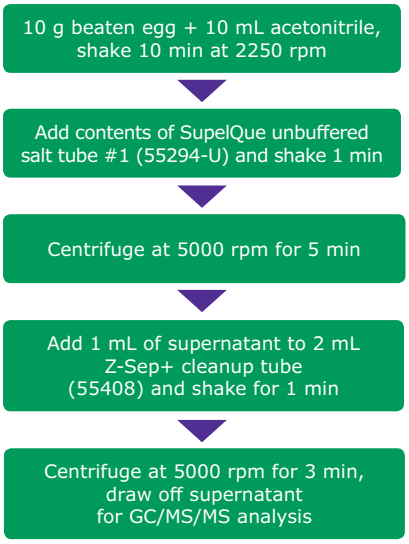

Table 1. GC/MS/MS conditions

column:	SLB®-PAHms, 30 m x 0.25 mm I.D., 0.25 µm (28340-U)
oven:	50 °C (2 min), 15 °C/min to 340 °C (10 min)
inj. temp:	250 °C
carrier gas:	helium, 1.2 mL/min, constant
detector:	MRM, Fipronil: 254.9/228, 350.8/254.8, 366.8/212.8 Fipronil sulfone: 382.8/254.9, 384.8/256.8, 254.9/227.9
injection:	1 µL, pulsed splitless (50 psi until 0.75 min, splitter on at 0.75 min)
liner:	4 mm I.D. FocusLiner™ with taper

Figure 2. GC/MS scan analysis of QuEChERS extract of egg.

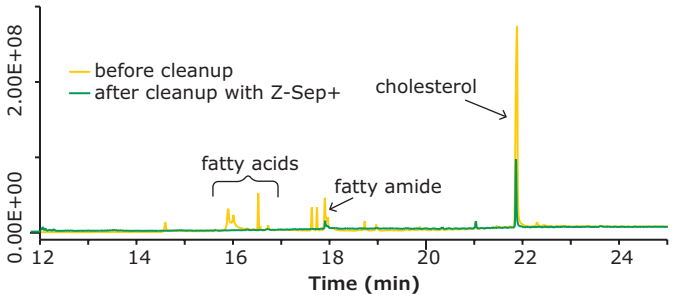

Figure 3. GC/MS/MS analysis of fipronil and fipronil sulfone in eggs at 5 ng/g.

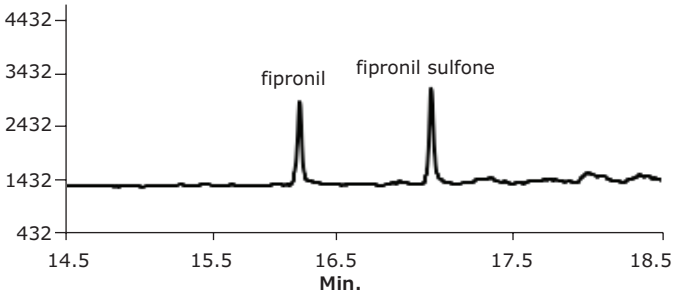

Table 2. Percent recovery and reproducibility (%RSD); spiking level of 5 ng/g.

n=3	Eggs	Chicken Meat	Mayonnaise
Fipronil	91 (0.5)	103 (3)	85 (3)
Fipronil Sulfone	91 (1.8)	116 (2)	87 (4)

For the full application data contact us or visit **SigmaAldrich.com/fipronil**

References

1. Fipronil Egg Scandal: What We Know. bbc.com, 8/11/2017 (accessed 8/15/17).

2 . Madsen, J.E.; Sandstrom, M.W.; Zaugg, Steven D., Methods of Analysis of the U.S. Geological Survey Nation Water Quality Laboratory-A Method Supplement for the Determination of Fipronil and Degradates in Water by Gas Chromatography/Mass Spectrometry; Open-file report 02-462; U.S.G.S.: Denver, CO, 2003.

Featured Products

Description	Cat. No.
SLB®-PAH ms, 30 m x 0.25 mm I.D., 0.25 µm	**28340-U**
Supel™ Que nonbuffered extraction tube #1	**55294-U**
Supel™ QuE Z-Sep + Tube, 2 mL	**55408-U**
Fipronil, Pestanal® analytical standard	**56451-100MG**
Fipronil Sulfone, Pestanal® analytical standard	**32333-50MG**