

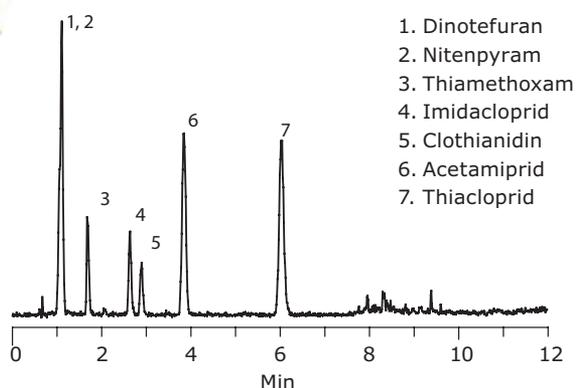
Analysis of Banned and Other Neonicotinoids from Plant Material

Back in 2018, the representatives of the Member States of the European Union decided to ban three products (clothianidin, imidacloprid, thiamethoxam) of the neonicotinoid family of insecticides deemed dangerous for bees on all outdoor crops permitting only greenhouse use.

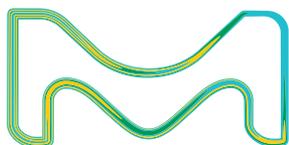
On Monday, January 15, 2020, the European Commission announced that they would not renew the license for a fourth neonicotinoid, thiacloprid, based on a scientific opinion from the European Food Safety Authority (EFSA). One of the major concerns was the impact on groundwater.

To help you ensure accurate testing of these pesticides, we offer a comprehensive portfolio of analytical standards and certified reference materials for the whole range of neonicotinoids, their main metabolites and isotope-labeled products.

The below application demonstrates the extraction and cleanup of these banned compounds and others of the neonicotinoid class from dandelion blossoms using the QuEChERS methodology.



For the QuEChERS approach, Supel[™] QuE tubes were used for extraction (Acetate) and cleanup (PSA/C18) prior to analysis of seven neonicotinoid pesticides. For the LC-MS analysis, an Ascentis[®] Express C18 Fused-Core[®] particle column was chosen to achieve high efficiency at relatively low backpressure compared to sub-2 μm UHPLC columns. This allowed the analysis to be performed on a standard pressure HPLC system with sufficient efficiency.



Conditions

sample/matrix	3 g pulverized dandelion blossoms, homogenized in 10 mL water
extraction process	add 25 mL of acetonitrile; add contents of Supel™ QuE Acetate extraction tube; shake immediately for 1 minute; centrifuge at 3400 rpm for 5 min
clean-up process	transfer 1 mL of the acetonitrile layer into a Supel™ QuE PSA/C18 cleanup tube; shake for 1 minute; centrifuge at 3500 rpm for 3 minutes; (draw off 700 µL of supernatant, evaporate to dryness at 50 °C under nitrogen; reconstitute in 200 µL of 50:50 0.1% formic acid:0.1% formic acid in methanol)
column	Ascentis® Express C18, 10 cm x 3.0 mm I.D., 2.7 µm particles
column temp.	25 °C
mobile phase	[A] 0.1% formic acid in water; [B] 0.1% formic acid in methanol
gradient	30% B from 0 to 5 min; to 100% B in 0.2 min; held at 100% B for 5.3 min; to 30% B in 0.5 min; held at 30% B for 5 min
flow rate	500 µL/min
pressure	3800 psi (262 bar)
injection	3 µL
detector	MS, ESI(+), MRM, m/z: 203.2/129.2, 271.2/225.0, 292.1/211.0, 256.0/175.2, 250.0/132.0, 223.2/126.0, 253.0/125.8

Materials

Product	Description
55234-U	Supel™ QuE Acetate extraction tube, AOAC 2007.01, pk of 50
55288-U	Supel™ QuE PSA/C18 cleanup tube, 2mL, AOAC 2007.01, pk of 100
52248-U	Supel™ QuE Empty 50 mL Centrifuge Tube with Lid, pk of 50
53814-U	Ascentis® Express C18, 10 cm x 3.0 mm I.D., 2.7 µm particles
1.00029	Acetonitrile hypergrade for LC-MS LiChrosolv®
1.06035	Methanol hypergrade for LC-MS LiChrosolv®.
1.15333	Water for chromatography (LC-MS Grade) LiChrosolv®
5.33002	Formic acid 98% - 100% for LC-MS LiChropur®

For more information about neonicotinoids visit us at

[SigmaAldrich.com/neonicotinoids](https://www.sigmaaldrich.com/neonicotinoids)

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Lit. No. MS_FL2076EN Ver. 1.0 31158 04/2020

Standards

Product	Description	Package Size
Acetamiprid		
33674	N-(6-Chloro-3-pyridylmethyl)-N-cyano-N-methylacetamidine Pestanal® analytical standard	100 mg
51625	N-(6-Chloro-3-pyridylmethyl)-N-cyano-N-methylacetamidine TraceCERT® certified reference material	50 mg
39246	d ₃ -Acetamiprid, analytical standard	50 mg
32979	Acetamiprid-N-desmethyl PESTANAL® analytical standard	10 mg
Clothianidin		
33589	Clothianidin PESTANAL® analytical standard	100 mg
68965	Clothianidin TraceCERT® certified reference material	50 mg
56816	d ₃ -Clothianidin PESTANAL® analytical standard	50 mg
Imidacloprid		
37894	Imidacloprid PESTANAL® analytical standard	100 mg
68694	TraceCERT® certified reference material	50 mg
46341	Imidacloprid solution, 100µg/mL, PESTANAL®, analytical standard	2 mL
34170	d ₄ -Imidacloprid PESTANAL® analytical standard	10 mg
34534	Imidacloprid-olefin PESTANAL® analytical standard	10 mg
37052	Desnitro-imidacloprid hydrochloride PESTANAL® analytical standard	25 mg
Thiacloprid		
37905	Thiacloprid PESTANAL® analytical standard	100 mg
14783	Thiacloprid TraceCERT®, certified reference material	50 mg
33897	Thiacloprid-amide PESTANAL® analytical standard	100 mg
30673	Thiacloprid-(thiazolidin ring-d ₄) analytical standard	10 mg
Thiamethoxam		
37924	Thiamethoxam PESTANAL® analytical standard	100 mg
68901	Thiamethoxam TraceCERT®, certified reference material	50 mg
38176	d ₃ -Thiamethoxam PESTANAL® analytical standard	25 mg
73348	N-Desmethylthiamethoxam Pestanal® analytical standard	50 mg
Metabolites		
19386	6-Hydroxypyridine-3-carboxylic acid analytical standard	100 mg
68678	6-Chloropyridine-3-carboxylic acid analytical standard	100 mg
31534	2-Imidazolidone PESTANAL® analytical standard	250 mg
Others		
46077	Nitenpyram PESTANAL® analytical standard	100 mg
32499	Dinotefuran PESTANAL® analytical standard	50 mg
69091	Dinotefuran reference material	50 mg

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