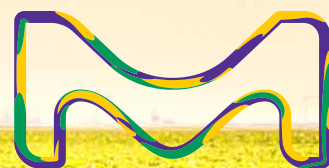


MILLIPORE
SIGMA

pesticide standards

For Food & Environmental Analysis



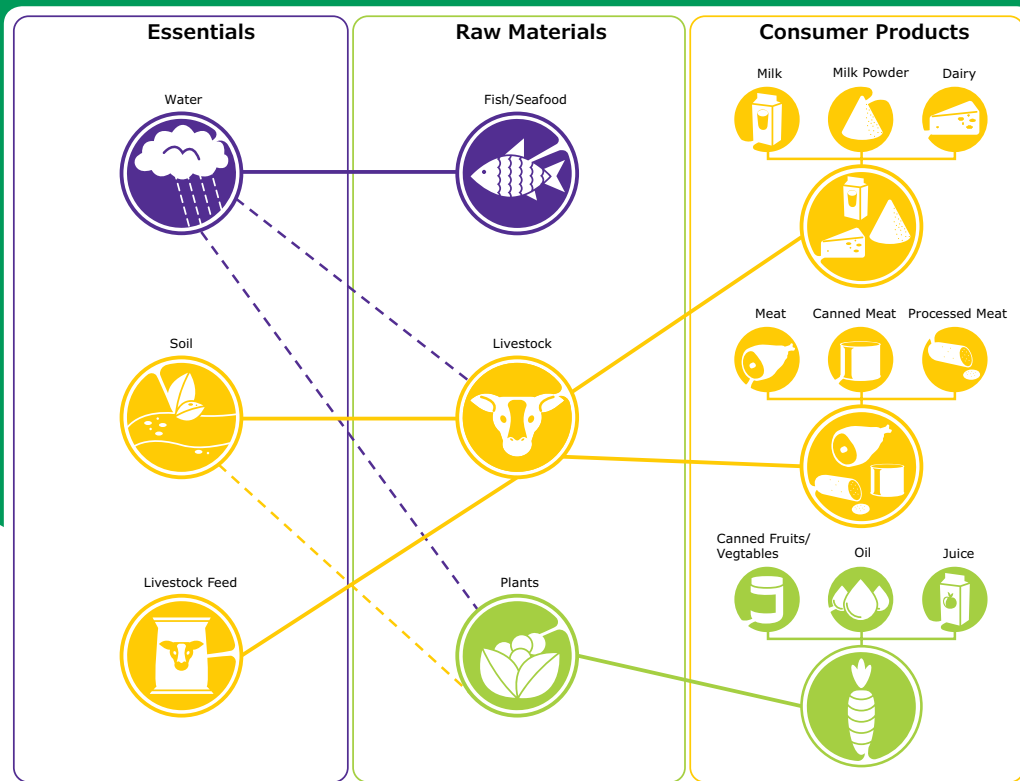
The life science business of Merck KGaA, Darmstadt, Germany operates as MilliporeSigma in the U.S. and Canada.

Analytical Products

Pesticides in Your Path

Pesticides are usually biological (such as viruses, bacteria, antimicrobials, disinfectants, etc.), chemical substances, or a mixture of substances intended to prevent, destroy, repel or mitigate any pests. Targeted pests include insects, rodents, weed, bacteria, plant pathogens, mollusks, nematodes (roundworms), mold, fungus and microbes that destroy property, cause nuisance, spread disease or are vectors for diseases.

Pesticides are released into the environment in order to eliminate these pests, but residues from these toxic chemicals also end up in the air, water, and even in food. Our pesticide standards help you to accurately monitor soil, water and food quality in compliance with national and international regulations.



Contents



Application Workflow

sample preparation and internal standards

Internal Standards: Isotope labeled pesticides

- Spike your sample with our isotopically-labeled pesticides for determination of material loss during workup
- We offer both neat material and solutions.
- Pesticides are available as ^{13}C -, ^{15}N - or D-labeled
- We are continuously adding new isotopically-labeled standards for pesticide and pesticide metabolite analysis to our already extensive portfolio of over 150 products

Learn more:

SigmaAldrich.com/pesticides



Reliable results

The right SPE products and accessories are essential for precise extraction and cleanup. Our method-specified SPE tubes and disks, like the ENVI SPE line, and the comprehensive portfolio of QuEChERS products for the AOAC 2007.01 and EN 15662:2008 methods, complemented by the zirconia coated silica material (Z-Sep), ensure reliable analyte recovery, and removal of matrix interferences. We also support you with vacuum manifolds, elution racks and shaker (QuEChERS).

For a complete listing, please visit

SigmaAldrich.com/spe

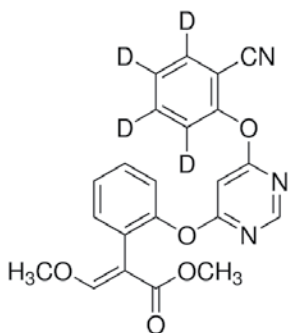
SigmaAldrich.com/quechers

Solid-phase microextraction (SPME) is a solvent-free extraction technique suitable for many environmental methods. As the market leader and key driver of this technique, we offer the most complete line of SPME fibers and accessories, to match your application, including new overcoated (OC) fibers for extraction from complex food samples, for example.

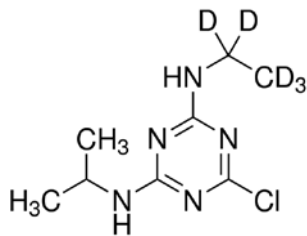
For the complete listing, please visit

SigmaAldrich.com/spme

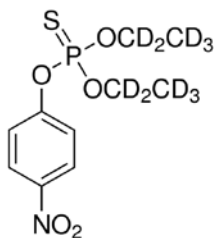
Our complete solution also includes purge traps, glassware, derivatization reagents, HPLC and GC columns, and accessories for extraction, cleanup and analysis of pesticides.



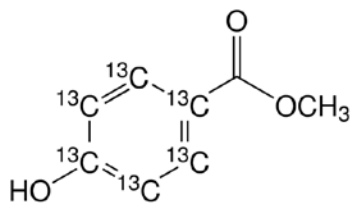
51949 Azoxystrobin-
(cyanophenoxy- d_4)



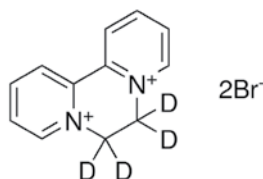
34053 Atrazine- d_5



33452 Parathion-ethyl- d_{10}



32135 Methyl
4-hydroxybenzoate-(ring- $^{13}\text{C}_6$)



03627 Diquat dibromide-(ethylene- d_4)
2Br⁻

sample analysis & calibration

TraceCERT® Certified Reference Materials (CRMs) for Pesticides—Neats and Solutions

For ISO/IEC 17025 accredited labs, it is mandatory to show traceability of all results, therefore certified reference materials (CRMs) must be used for calibration. Our rapidly expanding TraceCERT® product range includes an increasing number of CRMs for pesticides. Our ISO/IEC 17025 and ISO Guide 34 double accreditation is the highest achievable quality level for reference material producers, which ensures the quality of your reference materials.

Our neat CRMs are certified by quantitative NMR (qNMR) and are traceable to NIST SRM. The CRM solutions are produced gravimetrically using NIST traceable weights for balance calibration.

- Supplied with a comprehensive certificate according to ISO Guide 35 with properly calculated uncertainties, taking into account homogeneity and stability data
- Expiry date stated on the certificate of analysis (CoA)
- Convenient packages, either vials or amber-glass ampules, for better stability
- Use as calibration standards, internal standards, surrogate standards, spiking solutions or laboratory control samples (LCSs)
- Available as neat material, single-component solutions, or multi-component mixtures

Pesticide analytical standards, solutions and metabolites

For your routine analytics, choose your pesticide analytical standard from our broad portfolio:

- >1000 Neat standards and metabolites
- >110 single standard solutions
- Every CoA comes with an expiry date and a chromatogram

Learn more:
SigmaAldrich.com/pesticides

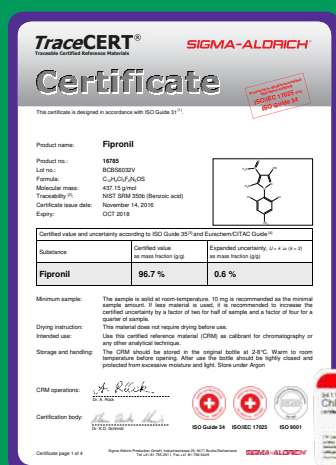
Pesticide metabolites

The active compound may be converted by target insects or plants, or may be degraded in the environment; monitoring of these transformation products is important to ensure more accurate measurements.

Choose from our broad metabolite portfolio, which we are continuously expanding.

- >120 metabolites available

For more information, and a list of our comprehensive offering of over 1700 pesticide standards and our certified reference standards, visit:
SigmaAldrich.com/pesticides



Looking for a GC or HPLC Column?

Visit us at SigmaAldrich.com/brighter



Certified Reference Material – Neat

Component	Unit Size	Cat. No.
λ-Cyhalothrin	50 mg	72765
2,4-D	100 mg	76514
2,4-DB	50 mg	73073
4,4'-DDT	100 mg	80076
Aldrine	50 mg	08573
Atrazine	50 mg	90935
Azoxystrobin	50 mg	43854
Bromobutide	50 mg	64148
Carbendazim	50 mg	79888
Chlorpyrifos	100 mg	94114
Cypermethrin	50 mg	51991
Deltamethrin	50 mg	05995
Diazinon	50 mg	68486
Diuron	50 mg	16902
Ethylparaben	50 mg	79577
Fipronil	50 mg	16785
Glufosinate-ammonium	100 mg	49677
Glyphosate	100 mg	89432
Malathion	50 mg	91481
Methylparaben	50 mg	79721
Paclobutrazol	50 mg	43900
Prochloraz	50 mg	64947
Propylparaben	100 mg	16897
Tebuconazole	50 mg	43897
Thiabendazole	50 mg	67554
Thiram	50 mg	43966
Triazophos	50 mg	68298
Trimethoprim	100 mg	16967

Certified Reference Material – Solutions/Mixes

Component	Description	Unit Size	Cat. No.
Aldrin solution	5000 µg/mL in methanol	1 mL	40220-U
Atrazine solution	1000 µg/mL in methyl tert-butyl ether	1 mL	48187
EPA505/525 Pesticide Mix A	500 µg/mL each component in acetone	1 mL	47725-U
EPA 608 Pesticide Mix	20 µg/mL each component in hexane:toluene (1:1)	1 mL	47915-U
EPA 8081 Pesticide Standard Mix	200 µg/mL each component in hexane:toluene (1:1)	1 mL	CRM46845

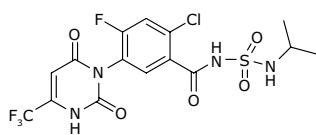
Metabolites – Analytical Standards

Component	Parent substance	Unit size	Cat. No.
Aldicarb-sulfoxide	Aldicarb	100 mg	31258
Boscalid Metabolite M510F01	Boscalid	10 mg	28001
Chlorbenside sulfone	Chlorbenside	25 mg	32231
Saflufenacil Metabolite M800H11	Saflufenacil	10 mg	18126
Saflufenacil Metabolite M800H02	Saflufenacil	10 mg	38586
Saflufenacil Metabolite M800H35	Saflufenacil	10 mg	76646

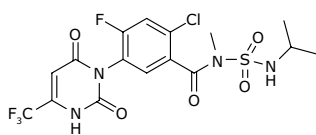
We are continuously expanding our *TraceCERT*® pesticide portfolio, search now: [SigmaAldrich.com/pesticides](https://www.sigmaaldrich.com/pesticides)

Examples of Our CRM Solutions/Mixes and Metabolites Program

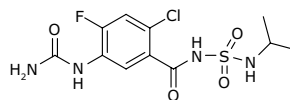
For more information, visit: [SigmaAldrich.com/pesticides](https://www.sigmaaldrich.com/pesticides)



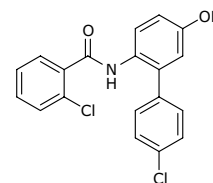
18126 Saflufenacil Metabolite M800H11



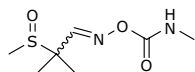
38586 Saflufenacil Metabolite M800H12



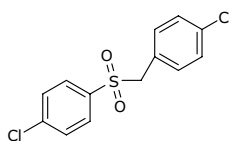
76646 Saflufenacil Metabolite M800H35



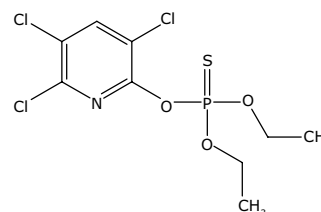
28001 Boscalid Metabolite M510F01



31258 Aldicarb-sulfoxide



32231 Chlorbenside sulfone



Chlorpyrifos

Quality Assurance

Matrix CRMs spiked with pesticides

Organic or inorganic compound matrix CRMs are most valuable in the validation of analytical methods. Their primary role is to calibrate the mass-transfer process, however they are also important for use as:

- Quality control tools for assuring the ongoing performance of test methods and the validity of associated test results
- A benchmark for comparing measurements between different laboratories

We are offering a variety of soil matrices, spiked with various pesticides:

- “Real world” matrices (non-fortified)
- Natural matrices, in which selected analytes have been fortified to give analytical profiles that meet the needs of analysts.

Most CRMs are method specific and all are supplied with certificates that show:

- Measured value plus uncertainty
- Expanded uncertainty confidence interval
- A prediction value, in addition to the mean value and standard deviation from the mean



Learn more:
[SigmaAldrich.com/ecrm](https://www.sigmaaldrich.com/ecrm)

Proficiency Testing

ISO 17025 accredited laboratories are required to take part in proficiency testing (PT) programs to demonstrate performance and method validation.

The Merck RTC brand represents over 20 years of experience and expertise in providing environmental PT studies worldwide. We produce and distribute over 20000 high-quality samples per annum for more than 2500 participants in our schemes. Our quality and services allow you to work smarter, enabling us to live in a safer and healthier world.

RTCs environmental PT programs cover:

- Water Pollution / Waste Water
- Water Supply / Drinking Water
- Solids / Soils Hazardous Waste

Laboratories can choose to participate in our quarterly or semi-annual PT programs, or simply purchase PT samples.

Find more information about PT registration and schedule at: [SigmaAldrich.com/pt](https://www.sigmaaldrich.com/pt)



Norms & Regulations

CONVENTIONS

Basel, Rotterdam and Stockholm

Persistent organic pollutants (POPs) are very stable hydrophobic molecules which are a threat to human health and to the environment. They can bio-accumulate through the food chain and pose significant health risks such as cancer, birth defects, reproductive disorders, and dysfunctional nervous and immune systems in humans and wildlife.

In 2001, the Stockholm Convention was organized under the guidance of the United Nations Environment Programme (UNEP) to eradicate the most dangerous of these chemicals. It entered into force in May 2004 and listed 12 of the most dangerous POPs (the so called "dirty dozen") with follow-up lists in May 2009, April 2011, May 2013 and May 2015. Since 2004 the list of POPs has been continuously extended.

Prior to Stockholm, the growth in chemical production and trade during the last three decades raised public and official concern due to the potential risks posed by hazardous chemicals and pesticides. Responding to these concerns, the UNEP and the Food and Agriculture Organization of the United Nations (FAO) started information-exchange programs in the mid 1980's to set up some guidelines on the Distribution and Use of Pesticides and for the Exchange of Information on Chemicals in International Trades.

In 1989, a Prior Informed Consent (PIC) procedure from the two organizations was introduced resulting in a joint convention text known as the Rotterdam convention for certain hazardous chemicals in international trade in March 1998. This entered into force in February 2004; since then, several amendments have been made, the latest in May 2015 resulted in a list of 16 additional POPs hazardous chemicals (including pesticides, PCBs, PCTs, and other organic pollutants). Three more substances are currently being evaluated and are proposed for listing under the convention.

- Dicofol
- Pentadecafluorooctanoic acid, its salts and PFOA-related compounds
- Perfluorohexanoic acid, its salts and PFHxS related compounds

Dealing with the control of transboundary movements of hazardous wastes and their disposal, the Basel convention entered into force in May 1992. Since then, various annexes have been included.

We offer certified reference materials (Neat and as a solution) as well as analytical standards for these hazardous chemicals targeted by the Stockholm and the Rotterdam Conventions.

Featured Products

Description	Grade	Cat. No.
Included in the initial 12th Stockholm Convention		
Aldrin	TraceCERT®	08573
	Pestanal®, analytical standard	36666
α-Chlordane	Analytical standard	442449
γ-Chlordane	Analytical standard	442599
4,4'-DDT	TraceCERT®	80076
	Pestanal®, analytical standard	31041
Dieldrin	Pestanal®, analytical standard	33491
Endrin	Pestanal®, analytical standard	32014
Heptachlor	Pestanal®, analytical standard	32349
Hexachlorobenzene	Pestanal®, analytical standard	45522
Mirex	Pestanal®, analytical standard	36170
Toxaphene	Pestanal®, analytical standard	N13586
Newly added pesticides since 2009–2017		
α-HCH	Pestanal®, analytical standard	33856
β-HCH	Pestanal®, analytical standard	33376
HCH (mixture of isomers)	Pestanal®, analytical standard	36756
Chlordecone	Pestanal®, analytical standard	45379
Lindane (γ-BHC)	Pestanal®, analytical standard	45548
Pentachlorobenzene	TraceCERT®	41231
	Pestanal®, analytical standard	35886
Pentachlorophenol	Analytical standard	48555-U
Endosulfan (α+β ~ 2+1)	Pestanal®, analytical standard	32015
α-Endosulfan	Pestanal®, analytical standard	45468
β-Endosulfan	TraceCERT®	40828
	Pestanal®, analytical standard	33385

Visit our website:

SigmaAldrich.com/standards

NORMS and Regulations

More than 1000 active ingredients are currently registered as pesticides worldwide. They are heavily regulated by both international (e.g., European regulation (EC) No. 1107/2009), and national regulatory bodies (e.g., the Environmental Protection Agency (EPA) in the USA, the Brazil Pesticide Law 9974, or the Pesticides Management Bill, 2008 in India).

Out of these regulations, quality test methods have been developed to control levels of pesticide residues in food and feed, drinking and waste water, and soil.

We offer a variety of multi-component solutions as certified reference materials for various EPA and EN (European Norm) methods.

Topics

Analysis of Fipronil in Eggs

Background

Fipronil is a broad spectrum insecticide that is normally used to treat dogs and cats for fleas.¹ It has also been used to combat red lice in poultry, although its use with food producing animals is not permitted in the European Union (E.U.).¹ It is a phenylpyrazole pesticide that is classified by the World Health Organization (WHO) as moderately hazardous.² In early June, this pesticide was detected in eggs and chicken meat originating from Belgium, and since that time, millions of eggs were removed from supermarket shelves throughout Europe.³

The maximum residue limit designated by the E.U. for fipronil in eggs is 5 ng/g (.005 mg/Kg). Analysis to this level can be achieved using the “Quick, Easy, Effective, Rugged and Safe” approach (QuEChERS) for extraction and cleanup. In the following application, QuEChERS extraction and cleanup followed by GC/MS/MS analysis was used for the determination of fipronil in egg samples spiked at 5 ng/g. Eggs have a relatively high fat content (approx. 10%), of which some will co-extract during the QuEChERS procedure. For a robust chromatographic analysis, a cleanup should be used which reduces these co-extracted fats. Z-Sep+, a silica sorbent functionalized with zirconia and C18, can be used to reduce a fatty matrix. The zirconia interacts via Lewis acid/base interactions to retain mono and diacylglycerols, phospholipids, and sterols. C18 retains by hydrophobic interaction, thus reducing triacylglycerols as well as other fats.

Application NOTES for pesticides

Pesticides are analyzed using multiple methods with gas, or liquid chromatography instrumentation, and in a wide variety of matrices (feed, food, water, soil) using different sample preparation methods. We have an extensive collection of applications specific to pesticide analysis.

SigmaAldrich.com/applications

Experimental

Eggs were obtained from a local grocery store and beaten until a homogeneous mixture was obtained. 10 g of sample was then weighed into a 50 mL centrifuge tube (extraction tube). The fipronil spike was added to a final concentration of 5 ng/g and allowed to equilibrate with the sample for 60 minutes. 10 mL of acetonitrile was then added, and samples were shaken for 10 minutes at 2250 rpm. The contents of a Supel™ QuE non-buffered extraction tube (55294-U) were then added to each sample. After shaking for 1 minute, the samples were then centrifuged at 5000 rpm for 5 minutes and the supernatant was removed for cleanup. Cleanup was done using Supel™ QuE Z-Sep+ by depositing 1 mL of extract in a 2 mL cleanup tube or 8 mL of extract into a 15 mL cleanup tube, and shaking for 1 minute. Samples were then centrifuged at 5000 rpm for 3 minutes, and the cleaned supernatant was removed for GC/MS/MS analysis. Spiked samples were quantitated against a 5-point matrix-matched calibration curve from 1–10 ng/mL prepared in blank egg extract. Analysis was carried out by GC/MS/MS following the conditions listed in **Table 1**.

Table 1. GC/MS/MS conditions

column	SLB®-PAHms, 30 m × 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, 254.9/228, 350.8/254.8, 366.8/212.8
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

Results and Discussion

Compared to no cleanup (Figure 1) Z-Sep+ sorbent reduced background (Figure 2), as seen by GC/MS scan analysis. Specifically, the cleanup reduced levels of fatty acids, fatty amide and cholesterol. The fatty acids eluted in the same retention range as fipronil, and thus have potential to interfere with low level detection. Z-Sep+ cleanup removed most of these fatty acids, resulting in a clean signal for fipronil at 5 ng/mL in the final extract (Figure 3). Average recovery for spiked replicates was 94%, with a relative standard deviation (RSD) of 8% (Table 1), falling well within the generally acceptable ranges of 70-120% recovery and RSD <20%.

Figure 1. GC/MS scan analysis of QuEChERS extract of egg, before cleanup.

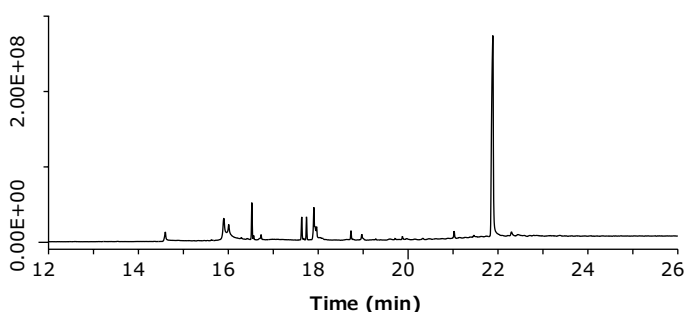


Figure 2. GC/MS scan analysis of QuEChERS extract of egg, after cleanup with Z-Sep+.

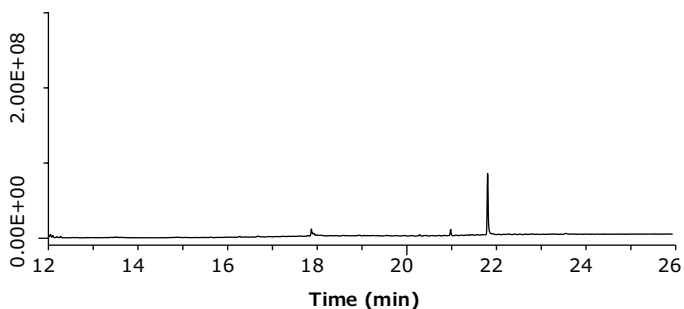


Figure 3. TIC of fipronil from QuEChERS extract of egg spiked at 5 ng/g, after cleanup with Z-Sep+.

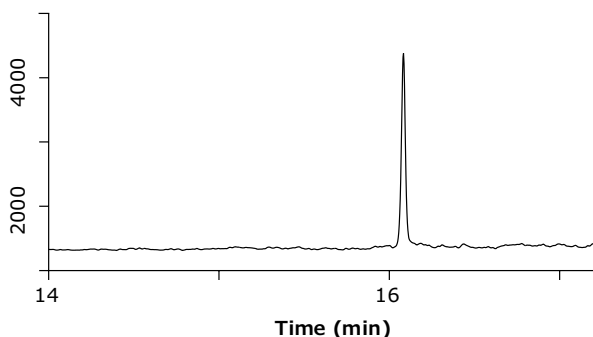


Table 1. Recovery and reproducibility for analysis of fipronil from eggs spiked at 5 ng/g.

	Avg. Recovery (ng/g)	Recovery	RSD (n=3)
Fipronil	4.7	94%	8%

Summary

QuEChERS extraction and cleanup can be used in the analysis of fipronil in eggs. Low level detection using GC/MS/MS was achieved with good recovery and reproducibility. Cleanup with Z-Sep+ reduced the levels of co-extracted fatty constituents, resulting in a clean analysis.

Featured Products

Description	Quality	Cat. No.
SLB®-PAHms, 30 m x 0.25 mm I.D., 0.25 µm		28340-U
Empty 50 mL tubes for extraction		55248-U
Supel™ QuE non-buffered extraction tube #1		55294-U
Supel™ QuE Z-Sep+ Tube, 2 mL		55408-U
Supel™ QuE Z-Sep+ Tube, 15 mL		55486-U
QuEChERS shaker and rack starter kit, 115V		55278-U
QuEChERS shaker and rack starter kit, 230V		55438-U
Fipronil	TraceCERT®, CRM	16785-50MG
Fipronil	Pestanal®, analytical standard	56451-100MG
Fipronil-(pyrazole- ¹³ C ₃ , cyano- ¹³ C)	analytical standard	79157-5MG
Fipronil sulfone	Pestanal®, analytical standard	32333-50MG
Fipronil sulfide	Pestanal®, analytical standard	34520-10MG
Fipronil-desulfinyl	Pestanal®, analytical standard	41865-25MG
Fipronil carboxamide	Pestanal®, analytical standard	34519-10MG

References

1. Fipronil Egg Scandal: What We Know. <http://www.bbc.com/news/world-europe-40878381> (accessed Aug 15, 2017).
2. *The WHO Recommended Classification of Pesticides by Hazard and Guidelines to Classification 2009*; International Program on Chemical Safety, World Health Organization, 2010.
3. 6 Countries Warned of Eggs Contaminated with Pesticide. <https://www.cbsnews.com/news/eggs-contaminated-pesticide-fipronil-europe-belgium-netherlands/> (accessed Aug 15, 2017).

We are continuously working on various application notes for Fipronil and relevant metabolites in eggs and egg containing products using GC-MS or LC-MS.

Please check for the latest news on SigmaAldrich.com/fipronil

Bee-toxic pesticides

In spring 2008, a mass death of bees in Germany's Baden-Württemberg state was reported, along with other reports of bee deaths. As bees are critically important in the environment for sustaining biodiversity by providing essential pollination for a wide range of crops and plants, in Europe, the European Food and Safety Agency (EFSA) is consistently monitoring bee health and has made a number of recommendations to improve surveillance on the multifactorial origins of the decline of bee numbers. Some contributing factors include intensive agriculture and pesticide use, starvation and poor bee nutrition, viruses, attacks by pathogens, and invasive species.

The EFSA assessments on the potential risks to bees for clothianidin, imidacloprid and thiamethoxam will be finalised following a final round of consultation with pesticide experts in the Member States.

We supply all relevant neonicotinoids, their main metabolites and diverse isotope-labeled ones in analytical standard quality or as certified reference materials (CRMs) for analysis and calibration.

Visit our website:
SigmaAldrich.com/pesticides

Substance	Product	Cat. No.	Description	Pack.size	Grade
Imidacloprid	Imidacloprid	68694		50 mg	TraceCERT®, CRM
		37894		100 mg	Pestanal®, analytical standard
	Imidacloprid-olefin	34534		10 mg	Pestanal®, analytical standard
	Desnitro-imidacloprid hydrochloride	37052		25 mg	Pestanal®, analytical standard
	Imidacloprid solution	46341	100 µg/mL in acetonitrile	2 mL	Pestanal®, analytical standard
	Imidacloprid-d ₄	34170		10 mg	Pestanal®, analytical standard
Clothianidin	Clothianidin	33589		100 mg	Pestanal®, analytical standard
	Clothianidin-d ₃	56816		50 mg	Pestanal®, analytical standard
Thiamethoxam	Thiamethoxam	37924		100 mg	Pestanal®, analytical standard
	N-Desmethylthiamethoxam	73348		50 mg	Pestanal®, analytical standard
	Thiamethoxam-d ₃	38176		25 mg	Pestanal®, analytical standard
Dinotefuran	Dinotefuran	32499		50 mg	Pestanal®, analytical standard
Acetamiprid	Acetamiprid	33674		100 mg	Pestanal®, analytical standard
	Acetamiprid-N-desmethyl	32979		10 mg	Pestanal®, analytical standard
	Acetamiprid-d ₃	39246		50 mg	Pestanal®, analytical standard
Thiacloprid	Thiacloprid	37905		100 mg	Pestanal®, analytical standard
	Thiacloprid-amide	33897		100 mg	Pestanal®, analytical standard
	Thiacloprid-(thiazolidin ring-d ₄)	30673		10 mg	Analytical standard
Nitenpyram	Nitenpyram	46077		100 mg	Pestanal®, analytical standard
Diverse metabolites of neonicotinoids	6-Chloropyridine-3-carboxylic acid	68678		100 mg	Pestanal®, analytical standard
	6-Hydroxypyridine-3-carboxylic acid	19386		100 mg	Pestanal®, analytical standard
	2-Imidazolidone	31534		250 mg	Pestanal®, analytical standard



pesticides in cannabis

Three United Nations conventions provide the international legal framework on drug control, instructing countries to limit drug supply and use to medical and scientific purposes. Yet debate is increasing on the legislation of drugs for non-medical purposes, particularly cannabis.

Cannabis is the drug most often mentioned in reports of drug law offences in Europe. It is also Europe's most commonly used illicit drug.

In the E.U., a system of limited distribution has evolved in the Netherlands since the 1970s, with further developments in the last few years. However, in Europe, no country has legally authorized cannabis smoking for recreational purposes.

In 2012, a U.S. state-wide system for the regulated distribution of cannabis for non-medical purposes was approved in the U.S. states of Colorado and Washington, becoming operational in 2014. Since then, more states have followed including Maine, Massachusetts, Alaska, California, Nevada and Oregon.

Uruguay introduced national laws to regulate the supply and use of cannabis for non-medical purposes in 2013.

For medical purposes, cannabis is already allowed in over 20 U.S. states, Uruguay, Canada, Australia, Croatia, Germany, Mexico, South Africa and Spain.

To be on the safe side for medical cannabis prescription, pesticide residue analysis should be performed.

We can offer a wide variety of pesticide standards either as CRMs or as analytical standards, in addition to the necessary SPE cartridges and GC columns to perform your analysis.

Learn more:
[SigmaAldrich.com/cannabis](https://www.sigmaaldrich.com/cannabis)





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technical assistance:**

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SigmaAldrich.com/techservice

To talk to a standards specialist,

standardsEU@sial.com

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Lit. No. MS_BR1103EN Ver. 1.0
2017-05273
02/2018