

CHROMATOGRAPHY

Think about **food**
Care about **safety**

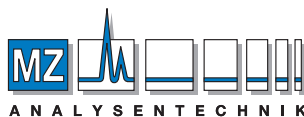


Vol. 7
Pesticides



MACHEREY-NAGEL

www.mn-net.com



AUTHORIZED DISTRIBUTOR

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Since 1911

Focus on pesticides

The global production and trading of food and feed products makes monitoring and control difficult.

Only analysis of food and raw materials for the production of food can ensure safe and edible food. Food safety is an essential part of our quality of life.

MN shows examples for food analysis that will help you with your work and make our food safer!

Vol. 7: Pesticides in food

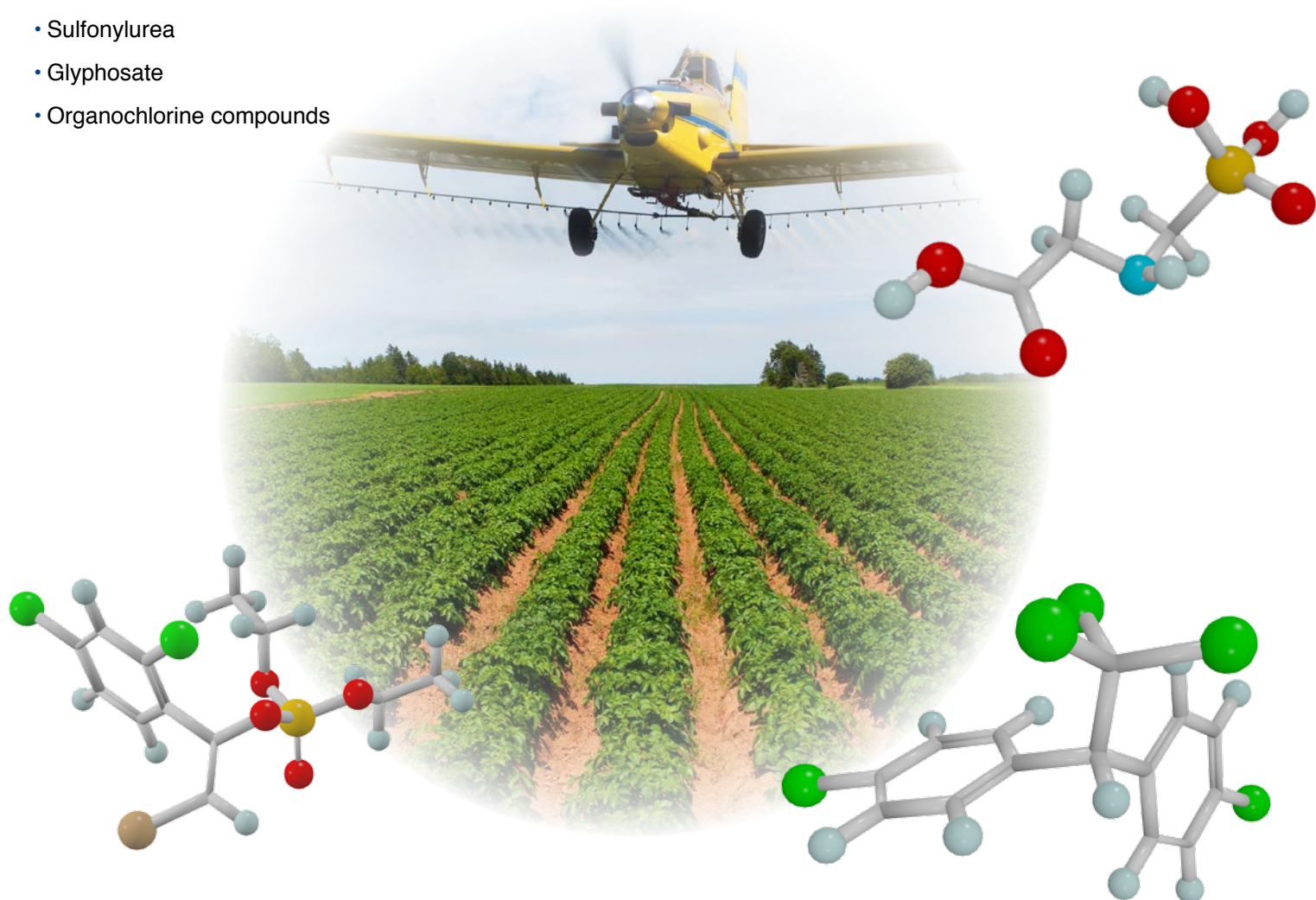
Pesticides are chemical agents used for prevention and control of various pests. Pesticides can be classified in herbicides (to control weeds), insecticides (to control insects), fungicides (to control molds and fungi) and rodenticides (to control rodents) [EPA – US Environmental Protection Agency – <http://www.epa.gov/pesticides/food/>].

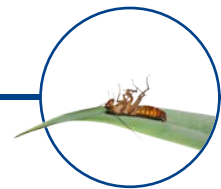
Most commonly used are

- Organophosphates
- Carbamates
- Pyrethroids
- Sulfonylurea
- Glyphosate
- Organochlorine compounds

Since pesticides can be harmful to health of humans, animals or the environment, they need to be monitored and very responsibly used [EPA – US Environmental Protection Agency – <http://www.epa.gov/pesticides/health/human.htm>].

Among other authorities, the US Environmental Protection Agency (EPA) sets limits on how much of a pesticide can be used on food during the growing and processing, and how much is allowed to remain in the food [EPA – US Environmental Protection Agency – <http://www.epa.gov/pesticides/safety/applicators/restrict.htm>].





Chemical structures of selected pesticides

Dioxathion 	Dimethoate 	Glyphosate 	Chlorpyrifos 	Chlorfenvinphos
Propamocarb 	Carbaryl 	Carbofuran 	Aldicarb 	Pirimicarb
Diuron 	Linuron 	Metoxuron 	Chlortoluron 	Pencycuron
Endosulfan 	Heptachlor 	Nonachlor 	Aldrin 	Endrin
Actochlor 	Metholachlor 	4,4'-DDD 	4,4'-DDE 	4,4'-DDT
Difenoconazol 	Cyproconazol 	Fenbuconazole 	Paclobutrazol 	Metazachlor
Triazines 	Compound Atrazine Simazine Terbutylazine Terbutryn	R₁ Cl Cl Cl S-CH₃	R₂ NH-C₂H₅ NH-C₂H₅ NH-C₂H₅ NH-C₂H₅	R₃ NH-CH(CH₃)₂ NH-C₂H₅ NH-C(CH₃)₃ NH-C(CH₃)₃

Typical samples, preparation

Pesticides from natural water

MN Appl. No. 305520

Column type:
CHROMABOND® HR-X, 3 mL, 60 mg
REF 730936

Column conditioning:

5 mL methanol, then 5 mL ultrapure water

Sample aspiration:

50 mL water sample is passed through the column by vacuum

Washing: 5 mL ultrapure water with 5 % methanol

Drying:

with gentle nitrogen stream, 15 min
(storage before elution step at 4 °C is possible for 1 month)

Elution:

2 x 3 mL methanol; then 1 x 3 mL methanol – ethyl acetate
(75:25, v/v)

Evaporation:

to dryness after addition of 10 µL internal standard

Reconstitution:

in 1 mL of the initial HPLC eluent

Compounds investigated:

acetochlor; alachlor; atrazine; azoxystrobin; carbaryl; carbofuran; 3-hydroxycarbofuran; chlorfenvinphos; chlorpyrifos; chlortoluron; 1-(3,4-dichlorophenyl)-3-methyl urea (DCPMU, metabolite of diuron); desethylatrazine (DEA, metabolite of atrazine); desethylterbutylazine (DET, metabolite of terbutylazine); desisopropylatrazine (DIA, metabolite of atrazine); dimethoate; dimetomorph; diuron; hexazinone; 1-(4-isopropylphenyl)-3-methyl urea (IPPMU, metabolite of isoproturon); 1-(4-isopropyl phenyl) urea (IPPU, metabolite of isoproturon); isoproturon; linuron; metazachlor; methomyl; metolachlor; metoxuron; pyrimicarb; simazine; terbutylazine; thiodicarb

We recommend using the **CHROMABOND® vacuum manifold for 12 columns** REF 730150

Pesticide residues from fruit and vegetables using the QuEChERS procedure

MN Appl. No. 305190

Column type:
CHROMABOND® QuEChERS Mix I, REF 730970
CHROMABOND® QuEChERS Mix III, REF 730972

Sample pretreatment:

The food sample is homogenized in a 50 mL centrifuge tube (REF 730223) with 8 mL acetonitrile and then cleaned up with pre-weighed mixes of salts and adsorbents in a 15 mL centrifuge tube.

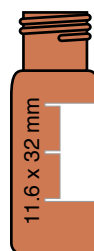
Procedure for complex food samples:

The sample is extracted with Mix I, then 1 mL supernatant is transferred to the centrifuge tube with Mix III (samples with low fat content). It is shaken, centrifuged and analyzed by HPLC.

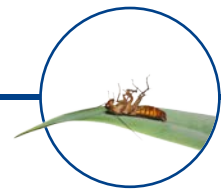
Compounds investigated:

aldicarb sulfone; aldicarb; desmethyl-pirimicarb; desmethyl-formamido-pirimicarb; carbofuran; paclobutrazol; terbutryn; pencycuron; dioxathion; fenpyroximate; oxamyl; pymetrozin; propamocarb; dimethoate; carbendazim; metalaxyl; methoxy-fenozide; tebufenozide; fenoxycarb; pyraclostrobin; difenconazole; propargite; spinosad; methiocarb sulfoxide; thiophanate-m; dimetomorph I; fludioxonil; fenbuconazole; famoxadone; prosulfocarb; dimetomorph II; cyproconazole I; cyproconazole II; hydroxycarbofuran

Transfer into a sample vial, e.g., 1.5 mL screw neck vials N 9, amber, flat bottom, label and scale, wide opening, REF 702284 and ready assembled screw closures N 9, blue, center hole with septum silicone / PTFE, REF 702287.1



For other applications see www.mn-net.com/apps



Fungicides carbendazime and thiabendazole from fruit juice

Solid phase extraction

MN Appl. No. 304740

Sample pretreatment:

see www.mn-net.com/apps

Column type:

CHROMABOND® HR-XC, 3 mL, 200 mg
REF 730952

Column conditioning: 5 mL methanol, then 5 mL 2% NH₃
(Do not let run the column dry!)

Sample aspiration: The prepared sample is passed through the column by vacuum.

Washing:

impurities are removed with the following washing mixtures:

- 2 mL 2% NH₃
- 2 mL 30% methanol – 5% NH₃
- 2 mL 0.1 mol/L HCl
- 2 mL methanol

Drying:

with nitrogen or air

Elution:

4 mL methanol / 5% NH₃

It is recommended to filter the prepared sample through disposable filters **CHROMAFIL® Xtra PET-45/25**
REF 729220



Detailed conditions are available online at
www.mn-net.com/apps

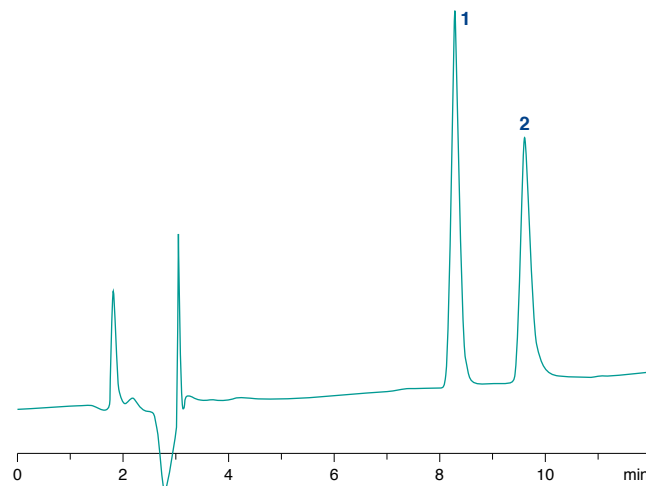
MN suggests using guard columns to protect your main column and significantly increase column life.

The **Column Protection System** (REF 718966)
is suitable for all analytical HPLC columns with 1/16" fittings!

HPLC analysis

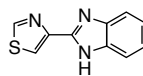
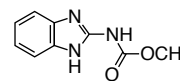
MN Appl. No. 122200

Column: EC 125/4 NUCLEODUR® C₁₈ Gravity, 5 μm
REF 760100.40



Peaks

1. Carbendazime
2. Thiabendazole



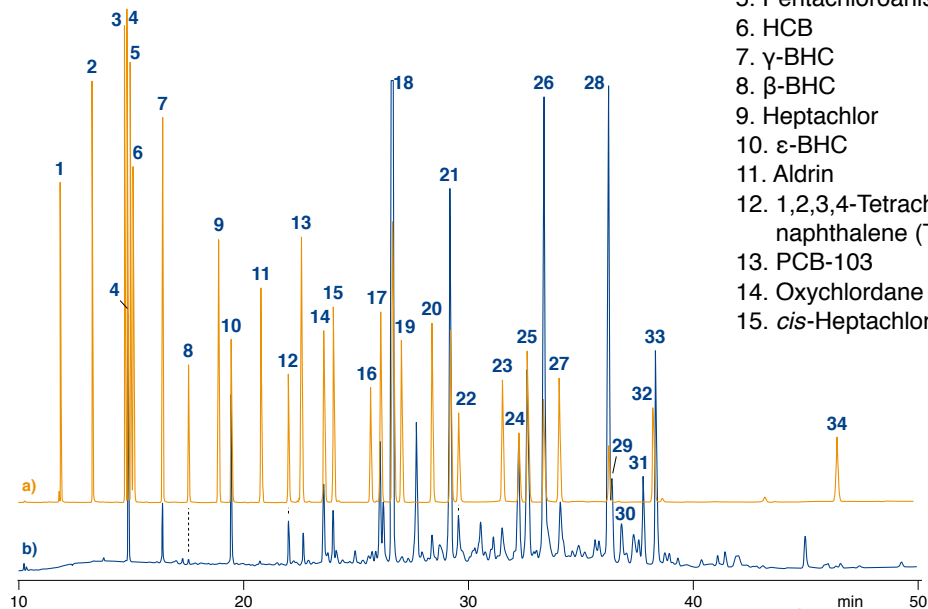
Subsequent analysis: GC

Pesticides from cod liver oil

MN Appl. No. 213270

R. Looser, K. Ballschmiter, J. Chromatogr. A 836, 271–284 (1999)

Column: OPTIMA® δ -3, 60 m x 0.25 mm ID, 0.25 μ m film, REF 726420.60



Peaks:

- | | |
|--|---|
| 1. Pentachlorobenzene | 16. 2,4'-DDE |
| 2. 2,4,6-Tribromoanisole | 17. <i>trans</i> -Chlordane |
| 3. Tetrachloro-1,4-dimethoxybenzene | 18. <i>cis</i> -Chlordane / <i>trans</i> -Nonachlor |
| 4. α -BHC | 19. Endosulfan I |
| 5. Pentachloroanisole | 20. 4,4'-DDE |
| 6. HCB | 21. Dieldrin |
| 7. γ -BHC | 22. 2,4'-DDD |
| 8. β -BHC | 23. Endrin |
| 9. Heptachlor | 24. 2,4'-DDT |
| 10. ϵ -BHC | 25. <i>cis</i> -Nonachlor |
| 11. Aldrin | 26. 4,4'-DDD |
| 12. 1,2,3,4-Tetrachloronaphthalene (TCN) | 27. Endosulfan II |
| 13. PCB-103 | 28. 4,4'-DDT |
| 14. Oxychlordane | 29. Parlar 41 |
| 15. <i>cis</i> -Heptachlor epoxide | 30. Parlar 40 |
| | 31. Parlar 44 |
| | 32. Endosulfan sulphate |
| | 33. Parlar 50 |
| | 34. Mirex |

It is recommended to filter the prepared sample through disposable filters CHROMAFIL® PTFE-45/25 REF 729205

Organochlorine pesticides (EPA 8081)

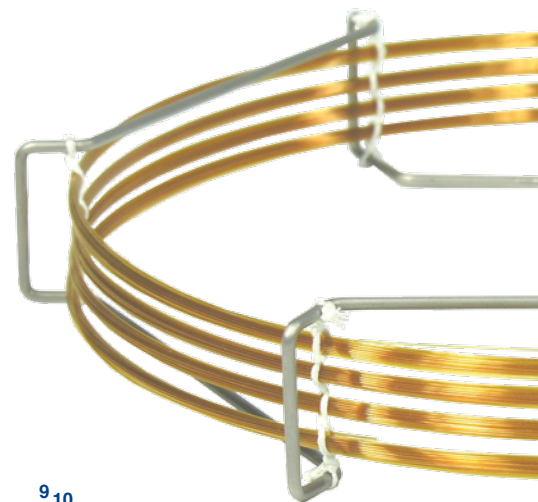
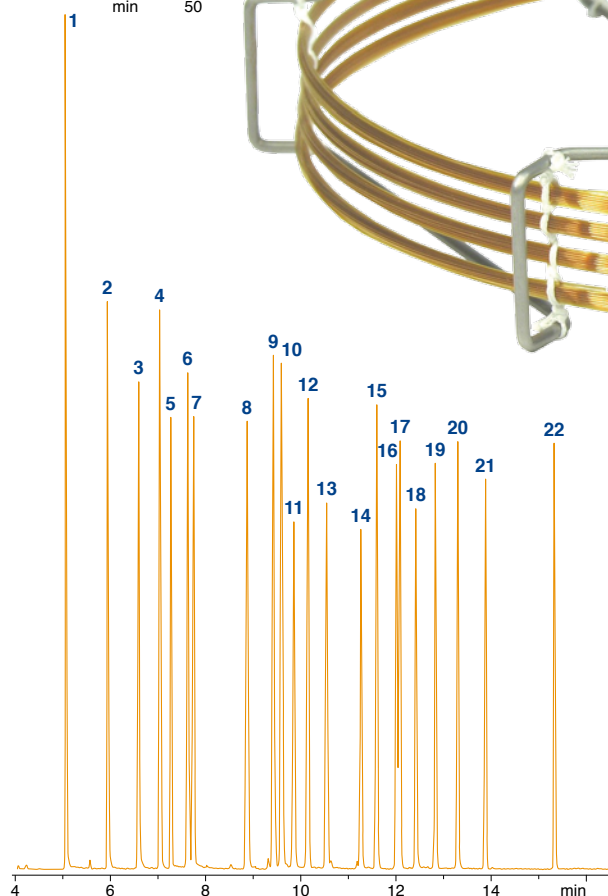
MN Appl. No. 213630

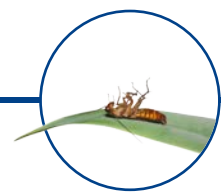
Column: OPTIMA® 17 MS, 30 m x 0.25 mm ID, 0.25 μ m film, REF 726162.30

Peaks:

- | | |
|--|------------------------|
| 1. 2,4,5,6-Tetrachloro- <i>m</i> -xylene | 12. 4,4'-DDE |
| 2. α -BHC | 13. Dieldrin |
| 3. γ -BHC (Lindane) | 14. Endrin |
| 4. Heptachlor | 15. 4,4'-DDD |
| 5. β -BHC | 16. Endosulfan II |
| 6. Aldrin | 17. 4,4'-DDT |
| 7. δ -BHC | 18. Endrin aldehyde |
| 8. Heptachlor epoxide | 19. Endosulfan sulfate |
| 9. γ -Chlordane | 20. Methoxychlor |
| 10. α -Chlordane | 21. Endrin ketone |
| 11. Endosulfan I | 22. Decachlorobiphenyl |

Detailed conditions are available online at www.mn-net.com/apps





Ordering information

Step	Product	Pack of	REF
SPE 1	CHROMABOND® HR-X, 3 mL, 60 mg	30	730936
	CHROMABOND® vacuum manifold for 12 columns	1	730150
SPE 2 (QuEChERS)	CHROMABOND® QuEChERS Mix I (citrate extraction mix)	50	730970
	CHROMABOND® QuEChERS Mix III (Diamino clean-up mix)	50	730972
SPE 3	CHROMABOND® HR-XC, 3 mL, 200 mg	30	730952
	CHROMABOND® vacuum manifold for 12 columns	1	730150
Filtration	Disposable filters CHROMAFIL® Xtra PET-45/25	100	729220
	Disposable filters CHROMAFIL® Xtra PTFE-45/25	100	729205
Vials and caps	1.5 mL screw neck vials N 9, amber, label and scale	100	702284
	Ready assembled screw closures N 9, blue, center hole with septum Silicone white / PTFE red	100	702287.1
HPLC			
Column	EC 125/4 NUCLEODUR® C ₁₈ Gravity, 5 µm	1	760100.40
Guard column	EC 4/3 NUCLEODUR® C ₁₈ Gravity, 5 µm	3	761903.30
Guard column holder	Column Protection System	1	718966
GC 1			
Column	OPTIMA® δ-3, 60 m x 0.25 mm ID, 0.25 µm film	1	726420.60
GC 2			
Column	OPTIMA® 17 MS, 30 m x 0.25 mm ID, 0.25 µm film	1	726162.30

Visit us at www.mn-net.com/chroma to get more helpful information

Selection tools for

Vials and caps

Syringe filters

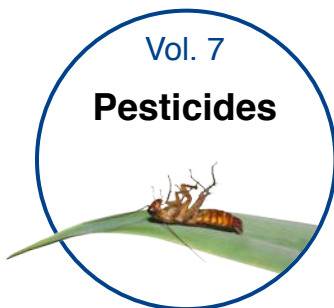
Troubleshooting guides

GC and HPLC

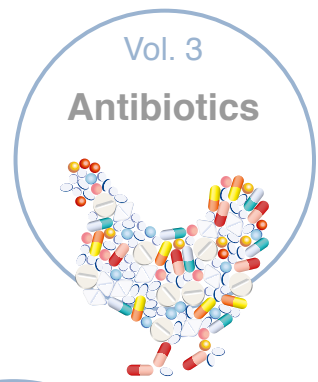
Detailed product information

...and much more

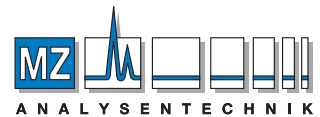




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Aeriatobok, Iovito, niccady, rrr145, anborne77, SkDEQ - foodia.com

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