

Analyzing Chlorinated Pesticides by Packed Column GC

Chlorinated pesticides are difficult to analyze by GC. Some of these compounds elute with very similar retention times, others break down during analysis. Two specially prepared and tested GC packings, 1.5% SP-2250/1.95% SP-2401 on 100/120 SUPELCOPORT and 4% SE-30/6% SP-2401 on 100/120 SUPELCOPORT, ensure uniformly high deactivation and alternative retention patterns for chlorinated pesticide analyses.

Key Words:

- organochlorine pesticides ● pesticides
- environmental analyses ● DDT

Chlorinated pesticides are difficult to analyze by GC because some of these compounds elute with very similar retention times, and others break down during analysis. To minimize breakdown, the packing must be thoroughly deactivated. Even small lot to lot differences in packing deactivation can drastically affect sample stability. Because thorough deactivation is difficult to achieve, consistent column to column performance does not come easily.

Thompson, et al. (1) developed two packings, 1.5% OV[®]-17/1.95% QF-1 and 4% SE-30/6% QF-1 on Chromosorb[®] W AW, for analyzing chlorinated pesticides. Based on their work, we developed two superior packings, GP[•] 1.5% SP[™]-2250/1.95% SP-2401 and GP 4% SE-30/6% SP-2401, on SUPELCOPORT[™] support, specifically for analyses of chlorinated pesticides. SP-2250 and SP-2401 phases are similar to OV-17 and QF-1, respectively, but the SP phases have lower viscosity, for higher efficiency columns, and better thermal stability. Our packings can resolve picogram quantities of 16 priority pollutant chlorinated pesticides in wastewater samples, according to US EPA Method 608 (2,3). Either packing also can be used to monitor these pesticides in agricultural products or other samples.

In the United States, a GC column used to analyze priority pollutant chlorinated pesticides in environmental samples must meet the following criteria from the US Environmental Protection Agency's *Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples* (4):

1. 4,4'-DDT (p,p'-DDT) loss or decomposition to 4,4'-DDD and 4,4'-DDE in the column must be less than 3% at a concentration of 60pg/μL.
2. Endrin breakdown must be less than 10% at a concentration of 100pg/μL.
3. A six foot packed column must have an efficiency of at least 2700 theoretical plates.
4. At 200°C, the retention time for 4,4'-DDT should be 4.18 times the retention time for aldrin, ±2 minutes.

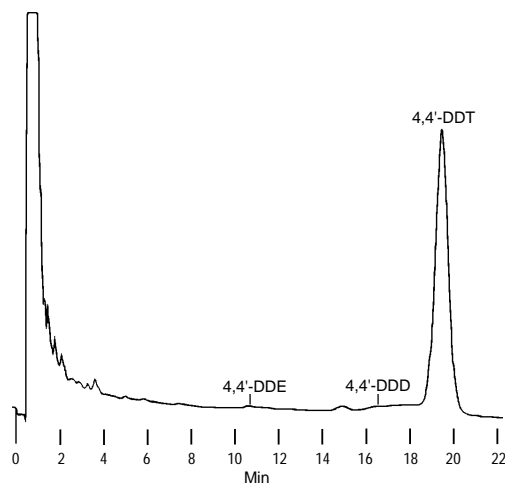
5. The column must permit detection of 100-500pg of each of 13 specified pesticides. These are normally encountered on-column quantities.

We use these criteria to evaluate each lot of GP 1.5% SP-2250/1.95% SP-2401 on 100/120 SUPELCOPORT. This packing meets or exceeds all EPA criteria for analyzing picogram quantities of chlorinated pesticides, and is cited for chlorinated pesticides analyses in EPA Method 608 (2,3).

Analysts following the EPA Manual of Analytical Methods (4) sometimes use columns containing 1.5% OV-17/1.95% QF-1 packing. However, the EPA recommends treating these columns with a silylating reagent and high concentrations of pesticides prior to use (3). This preparation takes two days or longer. Furthermore, the column may require periodic retreatment with the silylating reagent. In contrast, 1.5% SP-2250/1.95% SP-2401 on 100/120 SUPELCOPORT meets all EPA criteria for pesticide analyses with no preparative treatment. (In fact, this packing should not be treated with reagents or pesticides.) This saves considerable time. Figure A shows a typical evaluation for 4,4'-DDT loss in a 2.0m TightSpec[™] column packed with the

Figure A. Near-Complete Recovery of 4,4'-DDT from an Inert Packed Column

Packing: GP 1.5% SP-2250/1.95% SP-2401 on 100/120 SUPELCOPORT
 Cat. No.: 11947
 Column: 2.0m x 4mm ID glass
 Oven Temp.: 200°C
 Carrier: nitrogen, 60mL/min
 Det.: ECD⁶³Ni
 Inj.: 5μL iso-octane containing 300pg 4,4'-DDT

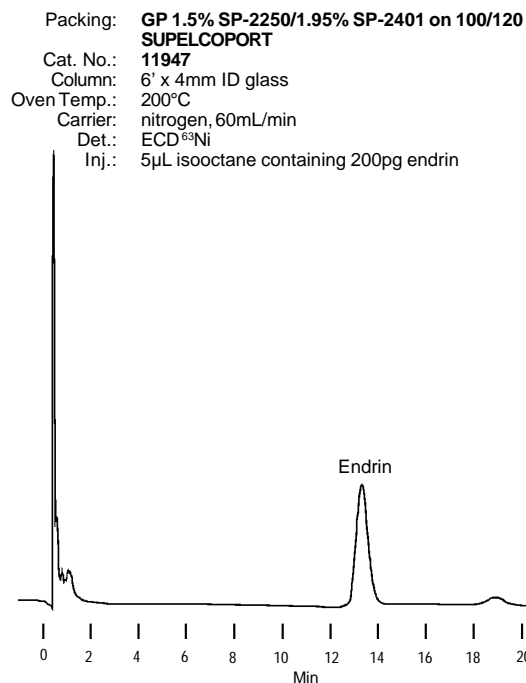


Note: It is particularly important to use oxygen-free carrier gas with an electron capture detector, because oxygen will elicit a response from an ECD. For details on carrier gas purification, request Bulletin 848. A halogenated gas such as chloromethane or chlorotrifluoromethane is used to determine linear velocity by ECD.

796-0585

SP-2250/SP-2401 packing. After routine overnight conditioning at 250°C, 4,4'-DDT loss/breakdown to 4,4'-DDD and 4,4'-DDE was only 1.5%. This loss is well within the limit prescribed by the EPA, as is the 6.9% loss of endrin (Figure B). Figure C shows an analysis of picogram quantities of 13 pesticides listed in EPA Method 608 (2,3). The retention time for 4,4'-DDT was well within the EPA limit of $4.18 \times t_{R \text{ aldrin}} \pm 2$ minutes. The efficiency of this particular column was 4316 theoretical plates.

Figure B. High Recovery of Endrin from an SP-2250/SP-2401 Column

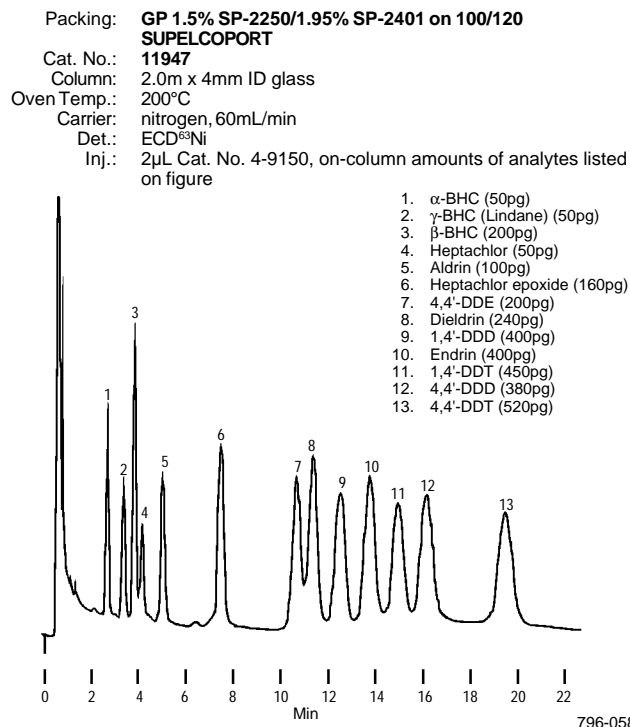


We also have tested 1.5% SP-2250/1.95% SP-2401 against equivalent packings from other sources, and have concluded that pesticide resolution on this packing is the best available on a packed column. For one such comparison, we ordered 1.5% OV-17/1.95% QF-1 on 100/120 Chromosorb W HP from three other suppliers, packed the four packings into silanized 6' x 4mm ID glass columns, and blind tested the columns in our QA laboratories, without treatment with silylating reagent or pesticides. As Figure D shows, only the SP-2250/SP-2401 column met the EPA criteria for resolution and 4,4'-DDT breakdown. The Supelco packing provided the best separation of 4,4'-DDE and dieldrin (peaks 7 & 8), and was the only column to elicit a quantifiable response for endrin (peak 10). It provided the best response for 1,4-DDT (o,p-DDT) and 4,4'-DDT, and the most symmetrical peak for the latter. Subsequent silanization of the OV-17/QF-1 columns according to the EPA procedure (4) did not improve analyses. The results, in fact, were worse.

Additional Packings for Monitoring Chlorinated Pesticides

In some situations, a packing other than 1% SP-2250/1.95% SP-2401 on SUPELCOPORT may be useful for providing an alternative or confirming separation. A second packing we test according to EPA criteria for chlorinated pesticide analyses, GP 4% SE-30/6% SP-2401 on 100/120 SUPELCOPORT, provides different retention times, but the same elution order, for these pesticides

Figure C. Thirteen Pesticides Monitored at Picogram Levels



(Figure E). When confirmation is necessary, the sample can be analyzed on both packings. If a component is retained for the appropriate time on each column, and the peaks represent approximately the same amount of material on each column, you usually can be confident the pesticide is properly identified.

Two other packings, 3% SP-2100 on 100/120 SUPELCOPORT and 5% SP-2401 on 100/120 SUPELCOPORT, also can be used to separate chlorinated pesticides. We prepare these packings with the same care as the GP packings, and evaluate them for efficiency and retention times with the 13 pesticides shown in Figures C and E. The latter two packings often are used to monitor pesticides in agricultural or other samples. Differences in the analyses of chlorinated pesticides in these samples are primarily in sample preparation. Detailed sample preparations are described in references 5-7.

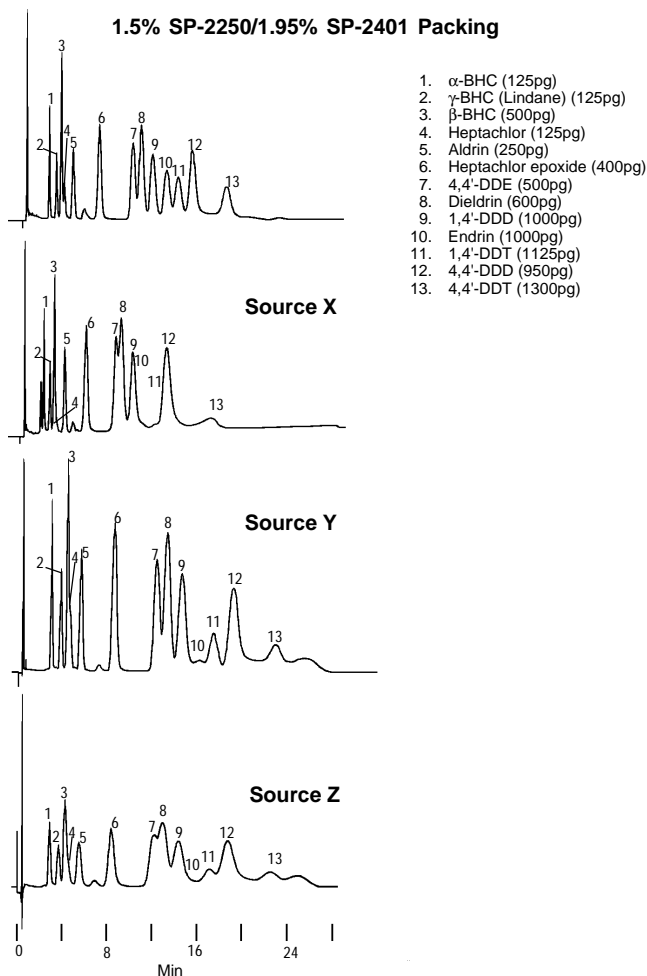
Preparing and Conditioning Packed Columns for Pesticides Analyses

Because metal tubing can be adsorptive, glass columns usually are preferred for analyzing pesticides at picogram levels. To ensure inertness, we recommend you either purchase silanized columns or silanize the inside of the column with a 5% solution of dimethyldichlorosilane (DMCS) in toluene (see our general catalog). We treat all Supelco glass columns with DMCS. Other silanization reagents, such as hexamethyldisilazane (HMDS), trimethylchlorosilane (TMCS), bis(trimethylsilyl)acetamide (BSA), and mixtures of reagents (i.e., REJUV-8™), are less effective for this purpose.

Use *small* silanized glass wool plugs at each end of the column. Excess wool can adsorb sample components. Use forceps to handle these plugs – skin oils can contribute irrelevant peaks to the chromatogram. Condition the column according to the procedures included with the packing. It may be necessary to periodically change the glass wool plug at the column inlet, especially if samples have not been well cleaned (8).

Figure D. Specially Tested Pesticide Packing Compared to Analogous Packings

Packing: **GP 1.5% SP-2250/1.95% SP-2401 on 100/120 SUPELCOPORT**
 Cat. No.: **11947**
 Column: 6' x 4mm ID glass
 Oven Temp.: 200°C
 Carrier: nitrogen, 60mL/min
 Det.: ECD⁶³Ni
 Inj.: 5µL Cat. No. 4-9150, on-column amounts of analytes listed on figure



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If you want consistent pesticide analyses, but are unfamiliar with column filling procedures or can't spare the time to fill your own columns, you can order columns filled with any of our pesticide packings.

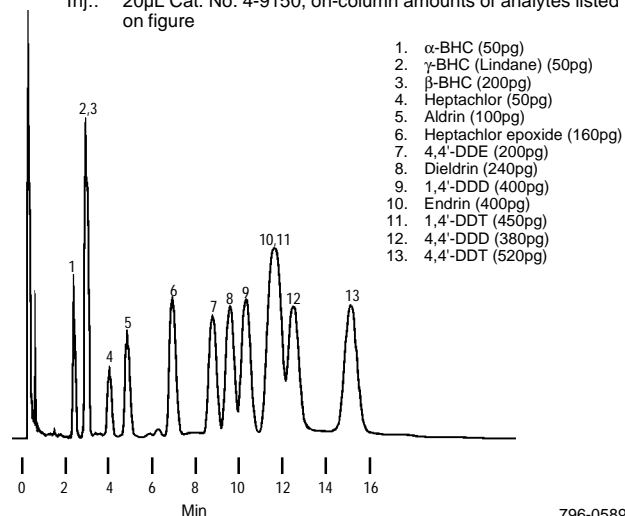
Capillary Chromatography from a Packed Column Chromatograph: Using Wide Bore Capillary Columns

If you would like to use capillary columns for pesticides analyses, but don't want to incur the expense of purchasing capillary-specific equipment (e.g., a capillary chromatograph), you will find 0.75mm ID SPB-5 capillary columns ideal. These borosilicate glass columns combine a large sample capacity (up to 15,000ng/analyte) with the column efficiency needed for resolving pesticides (Figure F). Because 0.75mm ID columns do not require a splitter or a detector specifically designed for capillary analyses, they can be used in packed column systems.*For simple connections, our 0.75mm ID columns have flexible fused silica tubing attached to both ends. We also offer kits that quickly adapt the

injection and detector ports in a packed column instrument to accept 0.75mm ID capillary columns. These adapters can be easily removed to reconvert the instrument to packed column use. For information about the conversion kits, refer to the Supelco catalog.

Figure E. Selectivity for Pesticides on an Alternative Packing

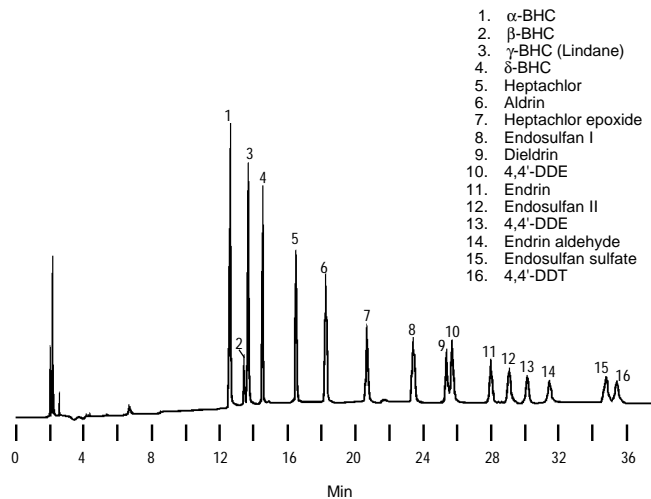
Packing: **GP 4% SE-30/6% SP-2401 on 100/120 SUPELCOPORT**
 Cat. No.: **11948**
 Column: 6' x 4mm ID glass
 Oven Temp.: 200°C
 Carrier: nitrogen, 80mL/min
 Det.: ECD⁶³Ni
 Inj.: 20µL Cat. No. 4-9150, on-column amounts of analytes listed on figure



796-0589

Figure F. Chlorinated Pesticides on a 0.75mm ID Capillary Column

Column: **SPB-5, 60m x 0.75mm ID, 1.0µm film**
 Cat. No.: **23721**
 Oven Temp.: 120°C to 215°C at 8°C/min, hold
 Carrier: helium, 15mL/min (set at 120°C)
 Det.: ECD
 Inj.: 1.0µL n-decane containing 200pg each analyte, direct injection



796-0590

References

- Thompson, J.F., A.C. Walker, and R.F. Moseman, J. Assoc. Offic. Anal. Chemists, **52**: 1263-1277 (1969).
- US EPA Test Methods: Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA Publication No. 600/4-82-057, July 1982). Obtain from Environmental Monitoring and Support Laboratory, US Environmental Protection Agency, Cincinnati, OH 45268.
- Federal Register, Vol. 49, No. 209, 40 CFR, Part 136: 43321-43336 (Nov. 26, 1984).
- Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples, Section 4A (EPA-600/880-038) (1980). Obtain from US Environmental Protection Agency, Health Effects Research Laboratory, Research Triangle Park, NC 27711.
- Horwitz, W. (Ed.), Official Methods of Analysis of the Association of Official Analytical Chemists (13th ed., 1980). Obtain from Association of Official Analytical Chemists, Washington, D.C.
This manual contains individual and multicomponent methods for complete analyses of pesticide residues (and many other compounds). The methods have been subjected to exhaustive validation studies at several laboratories to ensure that any analyst following the instructions will achieve the specified results. Because of these validation studies, the methods are conservative and describe analyses for compounds that have been in use for some time.
- McMahan, B.M. (Ed.), Pesticide Analytical Manual, U.S. Department of Health, Education, and Welfare, Food and Drug Administration, Washington, D.C. (updated regularly).
This manual contains individual and multicomponent methods for complete analyses of pesticide residues from plant and animal tissues. The methods include extraction, cleanup, and qualitative and quantitative determinations.
- Mills, P.A., B.A. Bong, L.R. Kamps, and J.A. Burke, J. Assoc. Offic. Anal. Chemists, **55**: 39-43 (1972).
The original description of the micromethod for Florisil® cleanup, using methylene chloride-containing solvents as eluants.
- Thompson, J.F., A.C. Walker, and R.F. Moseman, J. Assoc. Offic. Anal. Chemists, **52**: 1251-1262 (1969).

References not available from Supelco.

Additional Sources of Information on Pesticides Analyses

US Government Manuals

The US Food and Drug Administration (FDA) has prepared two volumes on analyzing pesticide residues in foods:

- Pesticide Analytical Manual, Volume I (Methods for Detecting Multiple Residues)
- Pesticide Analytical Manual, Volume II (Methods for Detecting Individual Residues)

Ordering Information

1/4 inch Stock Packed Glass Columns

Packed with GP 1.5% SP-2250/1.95% SP-2401 on 100/120 SUPELCOPORT (Cat. No. 1-1947), for US EPA Method 608 and other analyses of chlorinated pesticides, NIOSH Method 5503 analysis of Aroclor® PCB mixtures. All columns silane treated. We can prepare columns with this or our other pesticide packings on request. For ordering information, call our Sales Department.

Instrument Manufacturer and Model									
HP 5700 GC/MS	HP 5880 5890	PE 900	PE Sigma	PE 8300	Shimadzu®	Tracor 560*	Varian 3300	Varian 3700	
<i>2 meter (6.6') x 4mm ID TightSpec column</i>									
23075-U	23077	23079	23080-U	25961	25962¹	23081	25963	23089	
<i>6 ft. x 4mm ID nominal glass column</i>									
25964	25965	25966	25967	25968	25969²	25970-U	25971-U	25972	

*Circular configuration.

¹2 meter x 5mm OD x 3mm ID ²1.7 meter x 5mm OD x 3mm ID

Packings for Pesticides/PCBs Analyses

Description	Cat. No.
GP 1.5% SP-2250/1.95% SP-2401 on 100/120 SUPELCOPORT	11947
GP 4% SE-30/6% SP-2401 on 100/120 SUPELCOPORT	11948
3% SP-2100 on 100/120 SUPELCOPORT	11738
5% SP-2401 on 100/120 SUPELCOPORT	11764-U
10% SP-2401 on 100/120 SUPELCOPORT	11979

GP indicates packing has been tested for specific analysis shown in this bulletin.

Order from The National Technical Information Service, 5285 Port Royal Road, Springfield, VA 22161.

Volumes I & II: order NTISUB/C/117

Volume I only: order NTISUB/C/118

Volume II only: order NTISUB/C/119

The Environmental Protection Agency has prepared the following manual of pesticide residue analyses, dealing with human tissue, blood, and urine, as well as water, air, soil, and dust samples:

●Manual of Analytical Methods (J.F. Thompson, Ed.), December 1974 revision
Obtain from Quality Assurance Section, Chemistry Branch, US Environmental Protection Agency, Environmental Toxicology Division - Pesticides, Health Effects Research Laboratory, Research Triangle Park, NC 27711.

Reviews

Analytical Chemistry – Application Reviews published in alternate years (1979, 1981, etc.). One section deals with pesticide residues.

Analysis of Pesticides (M.C. Bowman, Ed.) – a special review in the May, June, and July 1975 issues of the Journal of Chromatographic Science.

Residue Reviews (F.A. Gunther, Ed.) – a total of 54 volumes (Springer-Verlag, New York, NY).

Chromatographic and Biological Aspects of DDT and Its Metabolites (L. Fishbein) – Chromatographic Reviews, J. Chromatogr., **98**: 175-251 (1974).

Gas Chromatographic Analysis of Pesticides (K. Hammerstrand) – Varian Instrument Company, Palo Alto, CA (1975).

Miscellaneous

Extraction and Cleanup of Organochloride, Organophosphate, Organonitrogen, and Hydrocarbon Pesticides in Produce for Determination by Gas-Liquid Chromatography M.A. Luke, J.E. Froberg, and H.T. Masumoto, JAOAC **58**: 1020-1026 (1975).

IUPAC Commission of the Development, Improvement and Standardization of Methods of Pesticide Residue Analysis P. Slade, J. Assoc. Offic. Anal. Chemists **58**: 1244-1255 (1975).

This report, by the Commission of the Pesticide Section of the Applied Chemistry Division of the IUPAC, is a summary of the proceedings. The commission concluded that it is scientifically unsound, in most cases, to specify a single method for analyzing a pesticide residue that can be applied to all substrates, under all conditions. It is possible to recommend methods that have been used successfully in a number of laboratories, but these recommendations must be considered only guidelines. The chemist is free to modify the methods according to the nature of the samples and the equipment and chemicals available.

Relative Retention Ratios of Ninety-Five Pesticides and Metabolites on Nine Gas-Liquid Chromatographic Columns Over a Temperature Range of 170 to 240°C in Two Detection Modes J.F. Thompson, J.B. Mann, A.O. Apodaca, and E.J. Kantor, J. Assoc. Offic. Anal. Chemists **58**: 1037-1050 (1975).

Use this compilation of relative retention values to estimate when a pesticide will elute from any of these columns.

Wide Bore Capillary Column

Description	Cat. No.
SPB™-5, 60m x 0.75mm ID borosilicate glass, 1.0µm film	23721

Liquid Chromatography (Drying) Columns



912-0333

Recommended in many US EPA procedures for pesticide and other priority pollutant analyses. Fill with appropriate adsorbents when cleaning or drying pesticide-containing samples.

Description (length x OD)

200mm x 7mm
200mm x 9mm

with coarse frit

300mm x 10mm
400mm x 19mm
400mm x 22mm

with Teflon® stopcock
300mm x 22mm

with coarse frit & Teflon® stopcock

300mm x 10.5mm
400mm x 19mm
300mm x 22mm
400mm x 22mm

with coarse frit, Teflon® stopcock & 24/40 inlet joint

300mm x 10.5mm
400mm x 19mm
300mm x 22mm
400mm x 22mm

Cat. No.

64748
64747

64749
64750
64751

64760-U

64752
64753-U
64754
64755

64756
64757
64758-U
64759-U

Reusable Sample Cleanup Columns



912-0130

These glass "champagne" columns can't contaminate your sample with plasticizers. The large columns (~6" long) have a 30mL reservoir; mini-columns (~3" long) have a 4mL reservoir.

Champagne columns, box of 6

58099

Miniature champagne columns, box of 10

58098

Florisil® PR

This specially processed 60/100 mesh Florisil provides consistent results for column cleanup and separation of chlorinated pesticide residues prior to gas or thin-layer chromatography. Each batch meets the performance characteristics described in *Changes of Official Methods of Analysis*, J. Assoc. Official Anal. Chemists, Chapter 24, 208(h), p. 233, vol. 49, No. 1, 1966. Packaged in glass container.

Florisil PR, 900cc

20280-U

Nuchar® – Attaclay®

Removes chlorophyll and other plant materials without retaining pesticides.

Nuchar – Attaclay, 100g

20246-U

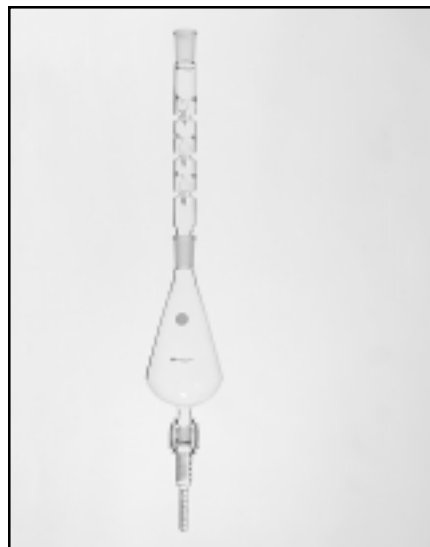
Celite® 545 AW Reagent Grade Filter Aid

Acid washed under rigid quality control, assuring batch-to-batch uniformity.

Celite 545 AW, Reagent Grade, 454g

20199-U

Macro Kuderna-Danish Concentrator



912-0173

The Kuderna-Danish apparatus was developed for concentrating material dissolved in volatile solvents, and has been widely used in pesticide analyses. Heavier fractions reflux until the final concentrate is collected in the receiving vessel. Includes 310mm, 3-ball Snyder column, 500mL flask, graduated 15mL receiving vessel. Refer to our catalog for replacement parts, other components, and other Kuderna-Danish apparatus.

Macro Kuderna-Danish Concentrator

64685-U

We offer solid phase extraction products for rapid, near-complete recovery of many pesticides from a range of matrices. For more information about these products, refer to our current catalog, or call our Technical Service chemists.

Liquid-Liquid Extractor



910-0100

Designed for continuous extraction of extractable organic compounds from one liter water samples, this extractor makes labor-intensive separatory funnel-type extractions obsolete. It also eliminates emulsion formation, a common feature of separatory funnel extractions.

Using a heating mantle (not included), a volatile, heavier-than-water solvent (e.g., methylene chloride) is boiled in the round bottom flask (A). Vapors are channeled to the water-cooled condenser (B). Recondensed solvent drips through a frit in the drip spout union (C) and disperses into small droplets. These fall through the aqueous sample in the extraction chamber (D), extracting organic material. The solvent reenters the flask from the bottom of the extraction chamber through a Teflon tube (E). Cycling continues until most of the extractable material is removed from the sample.

Glass and Teflon construction ensures contamination-free extraction. The apparatus can be dismantled for cleaning. Parts most likely to be dirtied are constructed of Teflon and can be economically replaced when necessary. The extraction chamber has a flat bottom, so the apparatus can stand on a lab bench. The condenser (T45/50 joint) is compatible with many Soxhlets and other extraction apparatus.

Includes extraction chamber, drip spout union, side arm elbow, condenser, ball and socket clamp, two Teflon elbow unions, two 13" lengths of Teflon tubing, 500mL round bottom flask. Order tubing for the condenser, etc. separately.

Liquid-Liquid Extractor Kit

64769

Pesticide Mixes for Evaluating GC Columns

Chlorinated pesticides in ng/ μ L and μ g/ μ L concentrations, specially prepared for evaluating the ability of a chromatographic column to separate pesticides and their degradation products in environmental samples. Data sheet and chromatogram included.

Component	Concentration (in isoctane)
Aldrin	0.050
α -BHC	0.025
β -BHC	0.100
2,4'-DDD	0.200
4,4'-DDD	0.190
4,4'-DDE	0.100
2,4'-DDT	0.225
4,4'-DDT	0.260
Dieldrin	0.120
Endrin	0.200
Heptachlor	0.025
Heptachlor epoxide	0.080
γ -BHC (Lindane)	0.025

CP Mix, components at ng/ μ L, 5mL

49150-U

CPM Mix, components at μ g/ μ L, 1mL

49151

We offer single-analyte solutions and small, neat quantities of more than 700 chlorinated and other pesticides and pesticide metabolites. We also offer an extensive line of pesticide mixes and Aroclor PCB standards for many methodologies. If you need an unusual standard, or unusual concentrations of analytes, we can quickly custom-prepare standards to meet your specific needs. For our complete selection of pesticide and PCB standards, please see our current catalog.

Trademarks

REJUV-8, SP, SPB, SUPELCOPORT, Sylon, TightSpec – Supelco, Inc.

Aroclor – Monsanto Co.

Attaclay – Analabs Co.

Celite, Chromosorb – Manville Corp.

Florisil – U.S. Silica Co.

Nuchar – West Vaco

OV – Ohio Valley Specialty Chemical Co.

Shimadzu – Shimadzu Corp.

Teflon – E.I. du Pont de Nemours & Co., Inc.

• GP indicates packing is tested for this application.

• Tested lots of OV-17/QF-1 packing are assumed to be representative of the three suppliers' packings (extent of lot to lot variability is unknown). Every lot of 1.5% SP-2250/1.95% SP-2401 packing meets all EPA criteria for chlorinated pesticide analyses.

• Before using these columns, we recommend you read about using an electron capture detector with capillary columns.

For more information, or current prices, contact your nearest Supelco subsidiary listed below. To obtain further contact information, visit our website (www.sigma-aldrich.com), see the Supelco catalog, or contact Supelco, Bellefonte, PA 16823-0048 USA.

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