

SLB®-35ms

Intermediate Polar MS-Grade Capillary GC Columns

Our MS-Grade columns are designed for GC and GC/MS analysts who require low bleed, inert, durable, and consistently performing capillary GC columns. Their use can help achieve low detection limits, easy mass spectral identification, less instrument downtime, good resolution, short analysis times, and long column life. SLB-35ms is the newest addition to the line. It is an intermediate polar MS-Grade column suitable for environmental applications that require dual-column confirmatory analysis, such as pesticides, PCBs, and herbicides. It is also suitable for PAHs, triglycerides, GCxGC applications, and other uses. A complete list of specifications can be found in **Table 1**.

Dual-Column Analysis of Organochlorine Pesticides

Analysis for pesticides is routinely performed by analysts in environmental laboratories. Many methods specify the use of a GC equipped with an electron capture detector (ECD). An ECD is highly sensitive, capable of detecting analytes at picogram levels. Its highly sensitive nature, plus the low detection limit requirements of promulgated methods, dictates the use of GC columns with low bleed. In addition, the susceptibility of several pesticides to degradation and adsorption make inertness an extremely important characteristic when choosing analytical columns.

Many of these methods also require each sample extract to be analyzed on two columns with differing selectivity. The first column, often referred to as the "primary" column, is used to determine, by retention time comparison with a standard, if any of the target analytes could be present in the sample. If peaks are found within an analyte's retention time window on the primary column, the presence of the analyte must be "confirmed" on a secondary or "confirmation" column that has a different selectivity.

Therefore, each column must have different selectivity, a low bleed characteristic, and exhibit good inertness. It is also desirable for both columns to have similar maximum temperature limits, because both columns will be in the same GC oven. The SLB-5ms / SLB-35ms pair meets all of these requirements.

A low-level mixture of twenty organochlorine pesticides and two surrogate compounds, each analyte at 50 ppb, was prepared in hexane. This mixture was first injected on the non-polar SLB-5ms column using conditions which maximized resolution. The mixture was then injected on the intermediate polar SLB-35ms column using the same run conditions. The resulting chromatograms are displayed in **Figure 1**.

As shown, the SLB-5ms / SLB-35ms pair offers the selectivity necessary to successfully resolve all 22 analytes between both columns. The use of identical run conditions allows their use in the same GC oven. Peaks are well integrated and baselines are exceptionally stable considering the high final oven temperature (340 °C). Minimal baseline rise is detected, and the last analyte, decachlorobiphenyl, is eluted in less than 20 minutes.

Table 1. SLB®-35ms Column Specifications

Application: The 35% phenyl equivalent phase provides a higher polarity option compared to columns containing a lower phenyl content, resulting in a greater retention of polar analytes relative to non-polar compounds. Its selectivity is complementary when paired with SLB-5ms for applications that require dual-column confirmatory analysis, such as for environmental analytes (pesticides, PCBs, and herbicides). The low bleed characteristics, inertness, and durable nature make it a great column anywhere a low bleed intermediate polar column is required.

USP Code: This column meets USP G42 requirements.

Phase: Bonded and highly crosslinked; proprietary polymer virtually equivalent in polarity to poly (35% diphenyl/65% dimethyl siloxane)

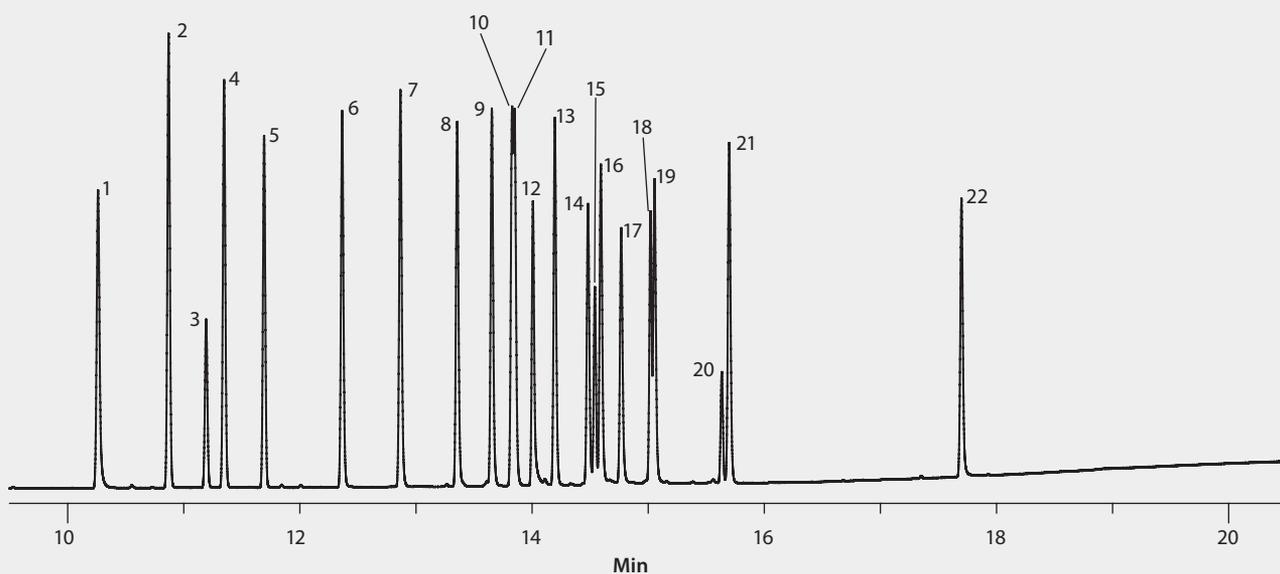
Temp. Limits: ≤0.32 mm I.D.: ambient to 350 °C (isothermal) or 360 °C (programmed)

Temp. Limits: ≥0.53 mm I.D.: ambient to 330 °C (isothermal) or 340 °C (programmed)

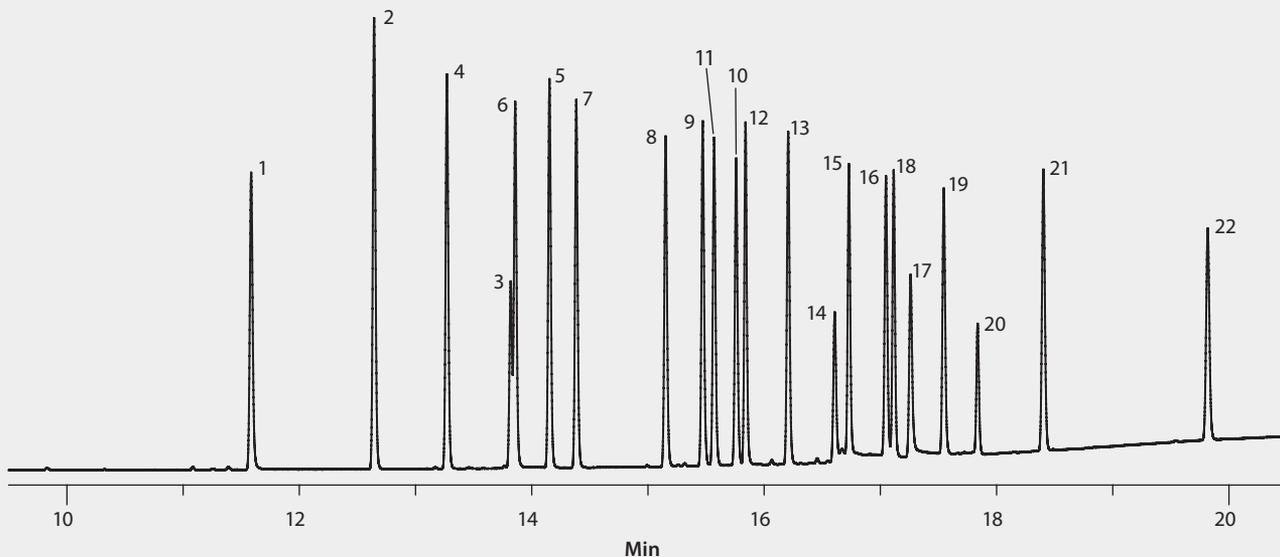
Figure 1. Dual-Column Analysis of Organochlorine Pesticides

column : SLB-5ms, 30 m × 0.25 mm I.D., 0.25 μm (28471-U)
 oven : 100 °C (2 min), 15 °C/min to 340 °C (5 min)
 inj. temp. : 250 °C
 carrier gas : helium, 0.9 mL/min
 detector : μECD, 340 °C
 injection : 1.0 μL, splitless (0.75 min)
 liner : 4 mm I.D., split/splitless type, wool
 packed single taper FocusLiner™ design
 sample : 200 ppm organochlorine pesticide standard mix
 (CRM46845) containing 22 analytes, diluted to
 50 ppb with hexane

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



column: SLB-35ms, 30 m × 0.25 mm I.D., 0.25 μm (29804-U)
 All other conditions and peak IDs are the same as above



PAHs in Soybean Oil

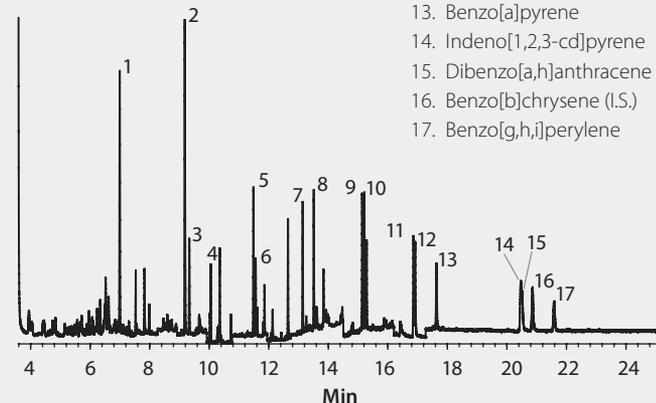
The sources of polycyclic aromatic hydrocarbons (PAHs) are both natural and man-made sources of combustion. They can contaminate food crops through environmental exposure to particulates, automobile exhaust, etc. In the case of edible oils, PAHs can also be introduced during actual production processes such as seed drying. Soybean oil is one of several popular edible oils. **Figure 2** shows the chromatogram of a soybean oil sample that was spiked with 16 PAHs and an internal standard. The sample was first processed using solid phase extraction (SPE) with a specialized sorbent material selective for PAHs. As shown, SLB-35ms provides:

- Desired selectivity (resolution of analytes)
- Necessary inertness (sharp peak shapes and great signal-to-noise)
- Low bleed characteristic (negligible baseline rise from 60 to 340 °C)

Figure 2. PAHs in Soybean Oil

sample/matrix: soybean oil spiked with PAHs at 10 ng/g
 SPE tube: Supelclean™ EZ-POP NP, 2.5 g/12 mL (54341-U)
 conditioning: 10 mL acetone, dry under 10-15" mercury for 10 minutes
 sample addition: 0.5 mL weighed accurately onto top frit
 elution: 15 mL acetonitrile
 eluate post-treatment: concentrate under nitrogen at 40 °C to a final volume of 0.5 mL
 column: SLB-35ms, 30 m x 0.25 mm I.D., 0.25 µm (29804-U)
 oven: 60 °C (1 min), 20 °C/min to 340 °C (10 min)
 inj. temp.: 300 °C
 detector: MSD, SIM, MS source at 250 °C, quadrupoles at 200 °C
 MSD interface: 330 °C
 carrier gas: helium, 1 mL/min constant flow
 injection: 0.5 µL, pulsed splitless (60 psi until 0.75 min), splitter open at 0.75 min
 liner: 2 mm I.D., split/splitless type, wool packed single taper FocusLiner™ design

- | | |
|-------------------|-----------------------------|
| 1. Naphthalene | 7. Fluoranthene |
| 2. Acenaphthylene | 8. Pyrene |
| 3. Acenaphthene | 9. Benzo[a]anthracene |
| 4. Fluorene | 10. Chrysene |
| 5. Phenanthrene | 11. Benzo[b]fluoranthene |
| 6. Anthracene | 12. Benzo[k]fluoranthene |
| | 13. Benzo[a]pyrene |
| | 14. Indeno[1,2,3-cd]pyrene |
| | 15. Dibenzo[a,h]anthracene |
| | 16. Benzo[b]chrysene (I.S.) |
| | 17. Benzo[g,h,i]perylene |



GCxGC: Allergens in Perfume

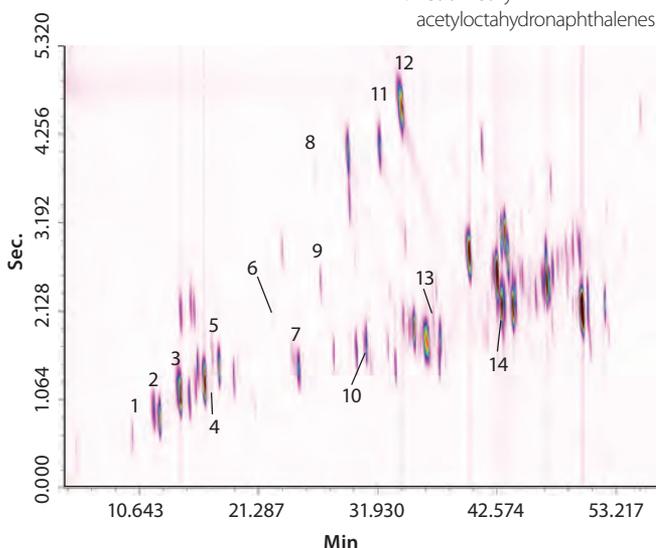
Perfumes are complex mixtures comprised of essential oils, aroma compounds, fixatives, and solvents. Some of these compounds can cause contact dermatitis, an allergic reaction following skin contact. Currently, 82 compounds are classified as contact allergens in humans, of which 54 are single chemicals and 28 are natural extracts.¹ A perfume sample was subjected to GCxGC analysis to determine if any of the 54 single chemicals could be detected. The calibration solutions and the sample were analyzed using the same conditions. The resulting chromatogram of the perfume sample is shown in **Figure 3**. Using SLB-5ms as the 1st dimension column and SLB-35ms as the 2nd dimension column, 14 single chemical allergens were detected. Mass spectral matching was carried out by using the FFNSC database.²

Figure 3. GCxGC Analysis of Allergens in Perfume

column: (1D) SLB-5ms, 20 m x 0.18 mm I.D., 0.18 µm (28564-U)
 column: (2D) SLB-35ms, 5 m x 0.25 mm I.D., 0.25 µm; cut from a 30 m x 0.25 mm I.D., 0.25 µm column (29804-U)
 oven: (1D) 45 °C, 3 °C/min to 230 °C
 oven: (2D) 50 °C, 3 °C/min to 230 °C
 oven: (flow modulator) stainless steel accumulation loop (20 cm x 0.71 mm O.D. x 0.52 mm I.D.), modulation period 5.4 sec (accumulation period 4.9 sec, injection period 0.5 sec)

inj. temp.: 310 °C
 detector: qMS, 200 °C, m/z = 40-360
 MSD interface: 250 °C
 carrier gas: (1D) helium, 0.46 mL/min
 carrier gas: (2D) helium, 7 mL/min
 injection: 1 µL, 10:1 split
 liner: 3.4 mm I.D., split/splitless type, wool packed straight FocusLiner™ design (2877601-U)
 sample: perfume

1. α-Pinene
2. β-Pinene
3. Limonene
4. Terpinolene
5. Linalool
6. α-Terpineol
7. Linalyl acetate
8. Cinnamal (Cinnamaldehyde)
9. Hydroxycitronellal
10. Eugenol
11. Vanillin
12. Coumarin
13. α-Isomethyl ionone
14. Tetramethyl acetyloctahydronaphthalenes



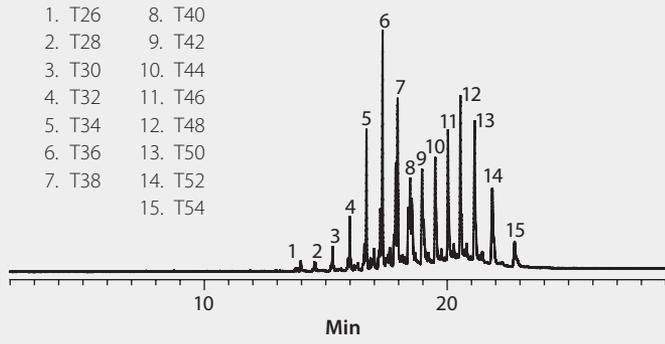
Triglycerides in Butter

The main constituent of animal fats and vegetable oil are a complex mixture of individual triglycerides. Efficient capillary GC columns are required for ample resolution and subsequent identification. Triglycerides are large compounds, so GC columns must also be capable of a relatively high final oven temperature. The chromatogram presented in **Figure 4** shows the triglycerides found in a butter sample using an SLB-35ms column. Analysis using a 10 m long column to a final oven temperature of 380 °C was found to provide good resolution and reasonable elution times.

Figure 4. Triglycerides in Butter

column: SLB-35ms, 10 m × 0.25 mm I.D., 0.10 µm; made by cutting down a 30 m × 0.25 mm I.D., 0.10 µm column (29802-U)
oven: 75 °C (0.1 min), 15 °C/min to 380 °C (10 min)
inj. temp.: track oven: 75 °C (0.1 min), 15 °C/min to 380 °C (10 min)
detector: FID, 380 °C
carrier gas: helium, 1.5 mL/min
injection: 1 µL, on-column
sample: butter, melted then diluted, 0.1% (v/v) in chloroform

- | | |
|--------|---------|
| 1. T26 | 8. T40 |
| 2. T28 | 9. T42 |
| 3. T30 | 10. T44 |
| 4. T32 | 11. T46 |
| 5. T34 | 12. T48 |
| 6. T36 | 13. T50 |
| 7. T38 | 14. T52 |
| | 15. T54 |



References

- European Union Scientific Committee on Consumer Safety (SCCS) Opinion on fragrance allergens in cosmetic products. http://ec.europa.eu/health/scientific_committees/consumer_safety/docs/sccs_o_102.pdf (accessed June 2, 2015).
- Mondello, Luigi. *Flavors and Fragrances of Natural and Synthetic Compounds 2, 2nd Edition*. John Wiley & Sons, Inc.: Hoboken, NJ, 2011; ISBN 978-1-118-14583-8.

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+ Ordering Information

Description	Cat. No.
SLB-35ms Capillary GC Columns	
15 m × 0.10 mm I.D., 0.10 µm	29808-U
20 m × 0.18 mm I.D., 0.18 µm	29809-U
30 m × 0.25 mm I.D., 0.10 µm	29802-U
15 m × 0.25 mm I.D., 0.25 µm	29803-U
30 m × 0.25 mm I.D., 0.25 µm	29804-U
60 m × 0.25 mm I.D., 0.25 µm	29805-U
30 m × 0.25 mm I.D., 0.50 µm	29806-U
60 m × 0.25 mm I.D., 0.50 µm	29807-U
30 m × 0.32 mm I.D., 0.25 µm	29810-U
60 m × 0.32 mm I.D., 0.25 µm	29811-U
30 m × 0.53 mm I.D., 0.50 µm	29812-U
30 m × 0.53 mm I.D., 1.00 µm	29814-U

+ Related Products

Description	Cat. No.
SLB-5ms Capillary GC Columns	
15 m × 0.10 mm I.D., 0.10 µm	28466-U
20 m × 0.18 mm I.D., 0.18 µm	28564-U
30 m × 0.25 mm I.D., 0.10 µm	28467-U
15 m × 0.25 mm I.D., 0.25 µm	28469-U
30 m × 0.25 mm I.D., 0.25 µm	28471-U
60 m × 0.25 mm I.D., 0.25 µm	28472-U
30 m × 0.25 mm I.D., 0.50 µm	28473-U
60 m × 0.25 mm I.D., 0.50 µm	28474-U
30 m × 0.32 mm I.D., 0.25 µm	28482-U
30 m × 0.53 mm I.D., 0.50 µm	28541-U
30 m × 0.53 mm I.D., 1.00 µm	28559-U
30 m × 0.53 mm I.D., 1.00 µm	29814-U

Related Information

Additional chromatograms, product information, real-time availability, and ordering information is available 24 hours a day at sigma-aldrich.com/slb