

BPX1

| ID (mm) | Film Thickness (µm) | Length (m) | Temperature Limits (°C) | Part No. |
|-----------------------|---------------------|------------|-------------------------|----------|
| Polyimide Clad | | | | |
| 0.1 | 0.1 | 10 | -30 to 400/400 | 054777 |
| 0.53 | 2.65 | 6 | -30 to 370/370 | 0548025 |
| 0.53 | 0.1 | 10 | -30 to 400/400 | 054803 |
| 0.53 | 0.9 | 10 | -30 to 400/400 | 054801 |
| 0.53 | 2.65 | 10 | -30 to 370/370 | 054802 |
| Aluminum Clad | | | | |
| 0.53 | 0.1 | 5 | -30 to 430/430 | 054800 |
| 0.53 | 0.17 | 5 | -30 to 430/430 | 054782 |
| 0.53 | 0.1 | 10 | -30 to 430/430 | 054779 |

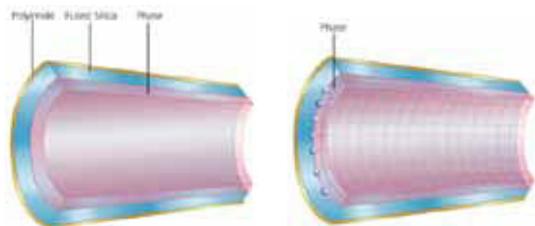
GC Columns and Applications

GC Capillary Columns | 100% Dimethyl Polysiloxane in a Sol-Gel Matrix

SolGel-1ms™

What is Sol-Gel?

Sol-Gel is essentially a synthetic glass with ceramic-like properties. These modified Sol-Gels offer the best of both worlds – ceramic-like properties with the film-forming properties of the associated polymer. The Sol-Gel process involves hydrolysis and condensation of alkoxides that lead to the formation of a glassy material at ambient temperatures. This method has been used to produce high quality ceramics and mono- and multi-component glasses of high homogeneity and purity. The further modification of this ceramic material with polymeric material (with appropriate functionality) leads to the formation of organic-inorganic nanomaterials.



Conventional Phase
The phase is coated onto the surface of the fused silica resulting in weak intermolecular bonding, ie no covalent bonding, ie no anchoring.

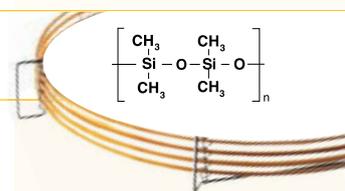
Sol-Gel Phase
Anchored to the surface of the fused silica through covalent bonding.

Where can Sol-Gel materials be used?

The further organic-modified Sol-Gels have been incorporated in a variety of high-end technology products including membrane chemical and pH sensors, films for protection of optical lenses, cosmetic and electronic products.

SGE and Sol-Gel materials?

At SGE, Sol-Gel processes are used to manufacture stationary phases for gas chromatography capillary columns. SGE is the first company to offer Sol-Gel technology capillary columns. The organic component in our case is a GC stationary phase. The final Sol-Gel product has all the properties of the GC phase as well as the additional properties of the Sol-Gel part. The Sol-Gel material is able to covalently bond to the surface of the fused silica. The 'heavy-duty' bonding imparts better thermal stability of the phase leading to ultra-low bleed capillary columns. To date, two Sol-Gel phases have been developed by SGE, namely SolGel-1ms™ and SolGel-WAX™. The SolGel-1ms™ stationary phase is a non-polar phase derived from 100% dimethyl polysiloxane. SolGel-WAX™ is a polar phase which incorporates polyethylene glycol in the matrix.



Expert Tip :

Always use SilTite™ or SilTite™ Finger-Tite ferrules when connecting a column to a GC/MS interface.



SolGel-1ms™ has a robust, inert, high temperature, non-polar phase for use with mass spectrometers.

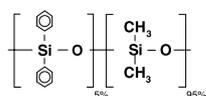
- Highly inert.
- Less bleed means:
 - Better MS library identification.
 - Less ion source maintenance.
 - Better sensitivity.
- Can also be used for all non-MS detectors.
- Same selectivity as BP1.
- 340 /360 °C upper temperature limit.

GC Columns and Applications

| | |
|---|---|
| Especially Suitable for these Industries: |    |
| Application Areas: | Recommended for highly active compounds. Applications ARO14, ENV51. |
| Operating Temperature: | 0.25 µm film thickness 0 °C to 340/360 °C. |
| Suitable Replacement for: | DB-1, DB-Petro, HP-1, HP-1MS, Rtx-1, Ultra-1, SPB-1, SPB-1 Sulfur, Petrocol DH, CP-Sil 5CB, VB-1, ZB-1, VF-1ms. |

| ID (mm) | Film Thickness (µm) | Length (m) | Temperature Limits (°C) | Part No. |
|---------|---------------------|------------|-------------------------|----------|
| 0.25 | 0.25 | 30 | 0 to 340/360 | 054795 |
| 0.25 | 0.25 | 60 | 0 to 340/360 | 054793 |
| 0.32 | 0.25 | 30 | 0 to 340/360 | 054798 |
| 0.32 | 0.25 | 60 | 0 to 340/360 | 054794 |

GC Capillary Columns | 5% Phenyl / 95% Dimethyl Polysiloxane



BP5

- Excellent general purpose GC column.
- Low bleed.
- Non-polar.
- High temperature.
- 320/340 °C upper temperature limit - dependent on film thickness.

| | |
|---|--|
| Especially Suitable for these Industries: |  All Industries |
| Application Areas: | General purpose, aromatics, pesticides, herbicides, drugs of abuse, hydrocarbons, solvent impurities, PCB congeners or Aroclor mixes, essential oils, semivolatiles. Applications FOO02, AMI03, PHA08, PHA 10. |
| Suitable Replacement for: | DB-5, Rtx-5, HP-5, Ultra-2, PTE-5, SPB-5, MDN-5, CP-Sil 8CB, VB-5, ZB-5. |

| ID (mm) | Film Thickness (µm) | Length (m) | Temperature Limits (°C) | Part No. |
|---------|---------------------|------------|-------------------------|----------|
| 0.22 | 0.25 | 12 | -60 to 320/340 | 054167 |
| 0.22 | 0.25 | 25 | -60 to 320/340 | 054168 |
| 0.22 | 0.25 | 30 | -60 to 320/340 | 054171 |
| 0.22 | 0.25 | 50 | -60 to 320/340 | 054169 |
| 0.22 | 1 | 50 | -60 to 320/340 | 054175 |
| 0.25 | 0.25 | 15 | -60 to 320/340 | 054182 |
| 0.25 | 0.25 | 30 | -60 to 320/340 | 054183 |
| 0.25 | 0.5 | 30 | -60 to 320/340 | 054202 |
| 0.25 | 1 | 30 | -60 to 320/340 | 054203 |

For your instrument specific septa see the Instrument Quick Pick Guide on pages 167-180.

Sol-Gel Technology for Gas Chromatographic Columns

INTRODUCTION

Capillary gas chromatography (GC) is a separation technique that has existed for many years. Over time, a variety of materials have been used as capillary column stationary phases, most of which have been based on polyorganosiloxanes.^{1,2} Superior stationary phase technology has led to enhanced thermal stability and improved inertness of many GC columns, allowing for a greater range of GC applications.²

The use of Sol-Gel materials as stationary phases for GC capillary columns is an attractive option due to the materials having good retentive characteristics, being inherently inert, easy to prepare and thermally stable at relatively high temperatures.

Sol-Gel processing consists of hydrolysis and condensation of a metal alkoxide (for example, tetraethoxysilane or TEOS) to form a glassy material at room temperature.^{3,5,6} During this process, a colloidal suspension of particulates (a 'Sol') is converted into a 'Gel' via polymerization (polycondensation). In essence, the hydrolyzed monomers of the metal alkoxide undergo polycondensation reactions promoting crosslinking to form a three dimensional network. Upon drying the material is transformed into a dried gel. The hydrolysis can be either acid or base catalyzed, while the condensation has been shown to proceed faster at higher pH.^{3,5,6}

This paper reviews the use of Sol-Gel capillary columns demonstrating inertness, partitioning capabilities and robustness.

EXPERIMENTAL CONDITIONS

Chemicals

All general chemicals were purchased from Sigma-Aldrich (Australia). Polymeric and sol-gel based stationary phases used in this study were synthesized in-house.

Column Preparation

Polyimide coated fused silica capillary columns were manufactured in-house. The coating procedure followed the general method outlined previously,^{6,8} the exact details of which are proprietary.

Chromatography Instrumentation

General gas chromatographic analyses were carried out using a Hewlett Packard 5890 series II gas chromatograph with a flame ionization detector. Bleed tests were carried out using a Hewlett Packard 6890 series GC system with a 5973 mass selective detector. Data acquisition was controlled by HP Chemstation (Revision A) software.

RESULTS

The chemistry involved in the preparation of the sol-gel phases is outlined in Figure 1. The SolGel-1ms™ material is non-polar, since commercially available poly(dimethylsiloxane) (PDMS) is part of the matrix. The SolGel-WAX™ material is more polar, since standard poly(ethylene glycol) (PEG) is part of the matrix, and is designed to be used in a similar manner to conventional wax-based stationary phases.

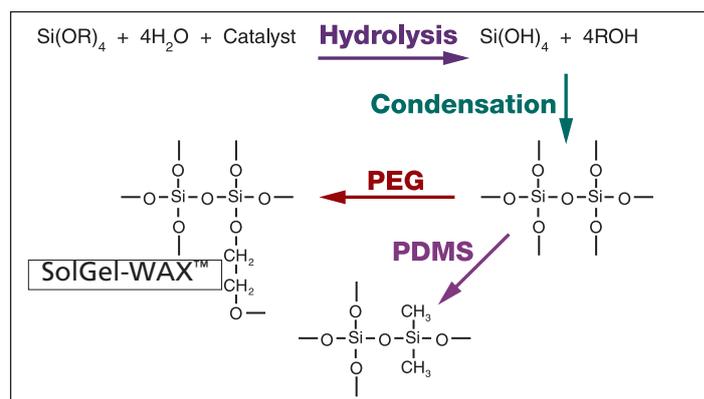


Figure 1. General chemistry involved in making SolGel-1ms and SolGel-WAX™ phases.

The non-polar SolGel-1ms™ column was tested using a stringent test mix containing a primary amine, an alcohol and an acidic phenol (Figure 2). The lack of activity towards the amine or phenol demonstrates the inertness of the sol-gel phase on the column. It also indicates that complete coverage of the active sites on the sol-gel is achieved. In order to test the partitioning properties of the SolGel-1ms™ column a Kovats Index comparison between the SolGel-1ms™ capillary column, the SGE BP1 100 % poly(dimethylsiloxane) column and a non-SGE 100 % poly(dimethylsiloxane) column was undertaken (Table 1). It is evident from Table 1 that all three columns are virtually identical in terms of partitioning capability. This result means that SolGel-1ms™ capillary columns can be used with existing chromatographic methods for analyses using standard 100 % poly(dimethylsiloxane) columns, with almost identical chromatographic results.

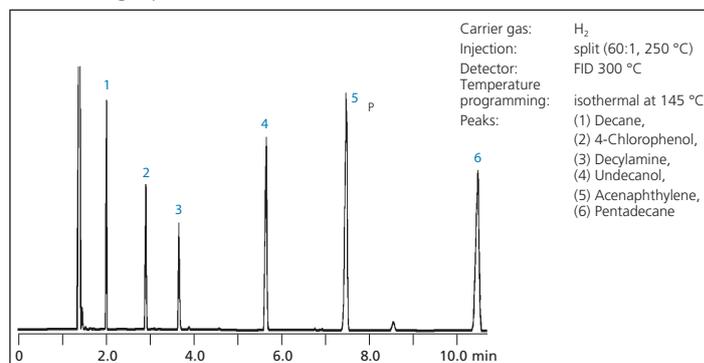


Figure 2. Standard test mix run on a SolGel-1ms™ column (30 m x 0.25 mm ID x 0.25 µm film thickness).

| No. | Component | Kovats | | |
|-----|----------------|-------------|------|--------------------------------------|
| | | SOLGEL-1ms™ | BP1 | Non-SGE 100 % Poly(dimethylsiloxane) |
| 1 | Decane | 1000 | 1000 | 1000 |
| 2 | 4-Chlorophenol | 1165 | 1167 | 1164 |
| 3 | Decylamine | 1240 | 1241 | 1239 |
| 4 | Undecanol | 1357 | 1358 | 1356 |
| 5 | Acenaphthylene | 1433 | 1434 | 1434 |
| 6 | Pentadecane | 1500 | 1500 | 1500 |

All columns 30 m, 0.25 mm ID, 0.25 µm film thickness; run conditions listed in Figure 2.

Table 1. Kovats Index comparison of a SolGel-1ms column with BP1 and a non-SGE 100 % polydimethylsiloxane columns.

The chromatograms in Figure 3 show the separation of a selection of compounds taken from the US EPA 8270 semi-volatiles method using a SolGel-1ms™ column. Excellent response was achieved for all compounds, including 2,4-dinitrophenol (peak 5) and 4-nitrophenol (peak 7), for which it is often difficult to obtain a satisfactory peak response.

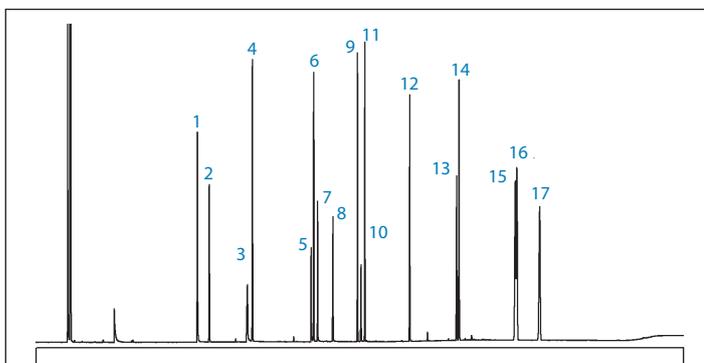


Figure 3a. The chromatogram shows separation of a selection of compounds from the US EPA 8270 semi-volatiles method using a SolGel-1ms™ column (30 m x 0.25 mm ID x 0.25 µm film thickness).

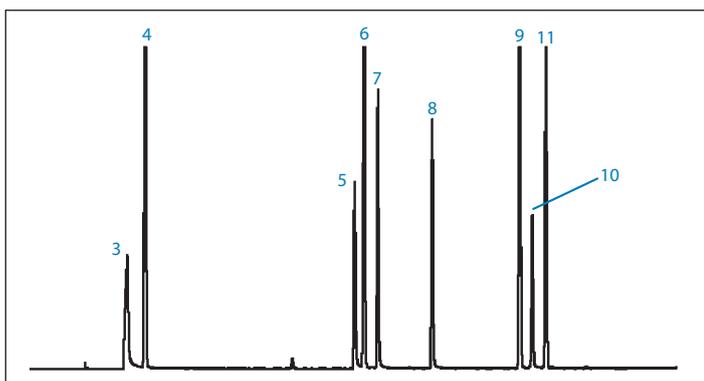


Figure 3b. Expansion of peaks 3-11.

Carrier gas: He
 Injection: Split (80:1, 250 °C)
 Detector: FID 300 °C
 Temperature programming: 40 °C to 270 °C at 10 °C min⁻¹, hold 12 min, 270 °C to 300 °C at 30 °C min⁻¹.

Peaks:
 (1) Aniline, (2) 1,4-Dichlorobenzene-d4,
 (3) Benzoic acid, (4) Naphthalene-d8,
 (5) 2,4-Dinitrophenol,
 (6) Acenaphthylene-d10, (7) 4-Nitrophenol,
 (8) 2-Methyl-4,6-dinitrophenol,
 (9) 4-Aminobiphenyl, (10) Pentachlorophenol,
 (11) Phenanthrene-d10, (12) Benzidine,
 (13) 3,3'-Dichlorobenzidine, (14) Chrysene-d12,
 (15) Benzo[b]fluoranthene,
 (16) Benzo[k]fluoranthene, (17) Perylene-d12

Bleed comparisons were made between a commercially available, low bleed 100 % poly(dimethylsiloxane) column and a SolGel-1ms™ column using a mass selective detector (Figure 4). Conditioning of the commercially available column was performed according to the manufacturer's instructions. While the magnitude of the bleed for the non-polar SolGel-1ms™ column was lower at all temperatures tested, the bleed chromatogram also exhibited significantly lower noise, which is important for detection of compounds at very low concentrations.

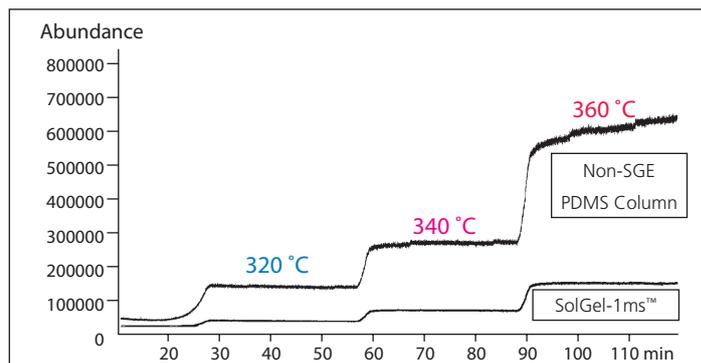


Figure 4. Bleed chromatograms generated from a commercially available poly(dimethylsiloxane) (PDMS) column (top) and a SolGel-1ms™ column (bottom) using a mass selective detector. Both columns were 30 m x 0.25 mm ID x 0.25 µm film thickness.

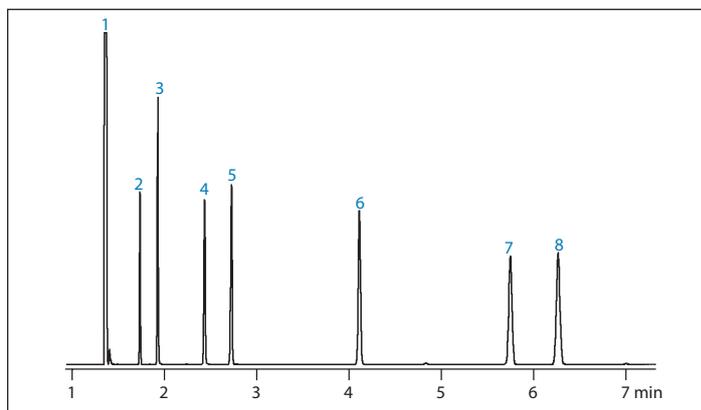


Figure 5a. Separation of a selection of compounds using a SolGel-WAX™ column (30 m x 0.25 mm ID x 0.25 µm film thickness), before heating at 250 °C for 1 hour with the carrier gas turned off.

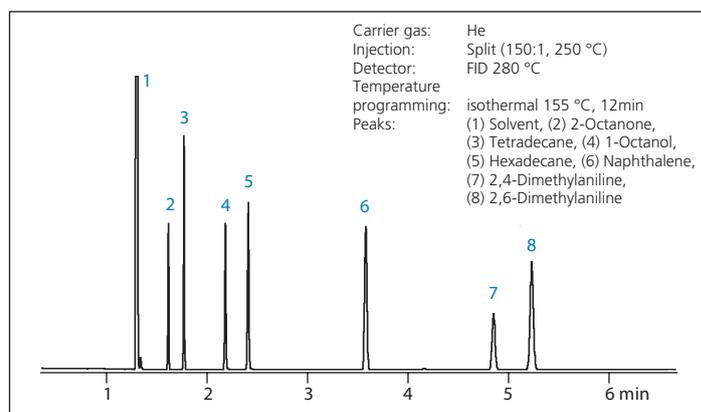


Figure 5b. SolGel-WAX™ column (30 m x 0.25 mm ID x 0.25 µm film thickness) after heating at 250 °C for 1 hour with the carrier gas turned off.

The chromatogram in Figure 5a shows the separation of various compounds using a SolGel-WAX™ column. This column was subsequently heated to 250°C and held at this temperature for one hour with the carrier gas turned off. The chromatogram in Figure 5b shows the re-testing of this column with identical conditions. It is evident from shortened retention times in the bottom chromatogram that there has been some phase loss. There was no evidence of peak broadening of the early eluting peaks which indicates that the phase has not been extensively degraded. The harsh conditions used to test the robustness of the SolGel-WAX™ column would begin to destroy most PEG (wax) phases, so the sol-gel matrix has significantly aided thermal stability of the stationary phase without having a detrimental effect on the partitioning capability.

The impact of water on a SolGel-WAX™ capillary column was tested by examining the retention time of acetone in water from repeated injections (Figure 6). Examination of the retention times for acetone from 300 injections showed that the water in the testing sample had no effect on the performance of the column. The advantage of using the sol-gel phase in this case was clearly demonstrated by maintenance of the inertness of the column to water. One useful application of this water resistance would involve low level detection of industrial solvents in waste water. To this end, the chromatogram in Figure 7 shows the separation of a range common industrial solvents using a SolGel-WAX™ column.

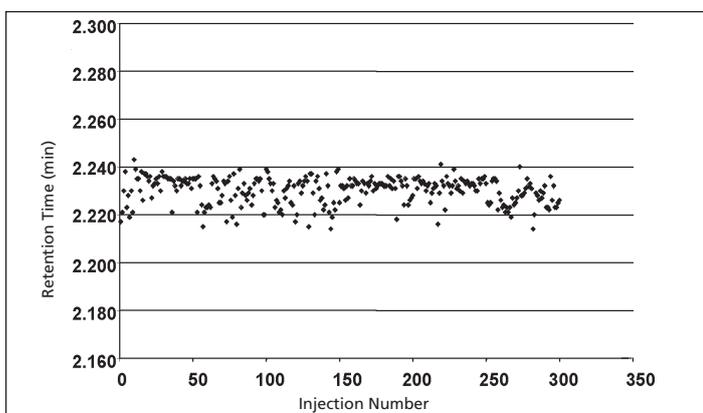


Figure 6. Graph showing the retention times of acetone in water over 300 injections using a SolGel-WAX™ column (30 m x 0.25 mm ID x 0.25 μm film thickness).

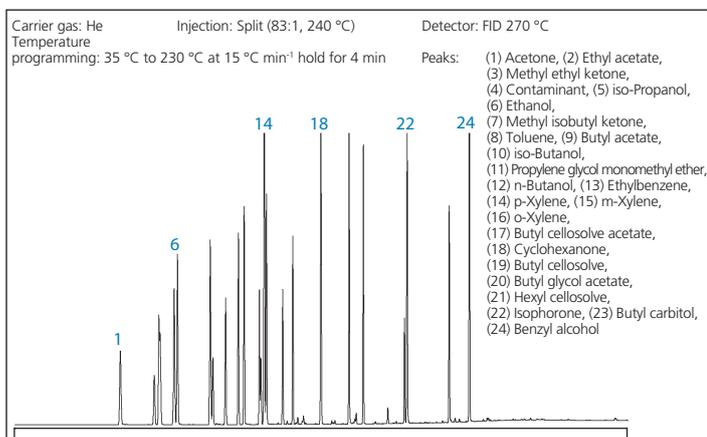


Figure 7. SolGel-WAX™ column (30 m x 0.25 mm ID x 0.25 μm film thickness) after heating at 250 °C for 1 hour with the carrier gas turned off.

Commercially available PEG or wax-type columns are commonly used to separate isomers of xylene, in particular m- and p-xylene. The selectivity of a SolGel-WAX™ column was examined using a range of aromatic compounds with similar chemical structures (BTEX) (Figure 8). From the chromatogram in Figure 8 it is evident that all compounds could be successfully partitioned with good peak shape and response being obtained.

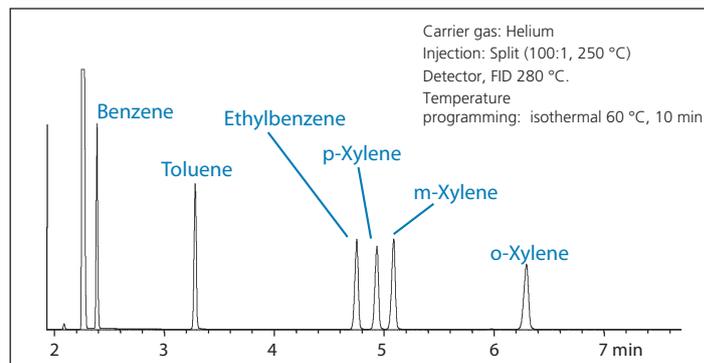


Figure 8. Chromatogram showing separation of a BTEX mixture using a SolGel-WAX™ column (30 m x 0.25 mm ID x 0.25 μm film thickness).

CONCLUSION

GC capillary columns with stationary phases based on Sol-Gel materials are inherently inert, thermally stable and can be successfully used to separate a range of compounds as the coating technology uses standard stationary phase material.

ACKNOWLEDGEMENTS

We wish to thank Dr. J. Luong (Dow Chemical Corporation, Canada) for his assistance with the testing of these columns, especially in relation to the data obtained for Figure 6.

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