GS-OxyPLOT: A PLOT Column for the GC Analysis of Oxygenated Hydrocarbons

Technical Overview

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Introduction

GS-OxyPLOT is a porous layer open tubular (PLOT) column. The stationary phase is a proprietary, salt deactivated adsorbent with a high chromatographic selectivity for low molecular weight oxygenated hydrocarbons. It is designed for and ideally suited for application in the ASTM methods listed in Table 1. It is an appropriate replacement for Varian's CP-LowOx column, usually with little to no changes in analytical parameters. This column is particularly useful for the trace analysis of oxygenates such as those listed in Table 2. Other oxygenated hydrocarbons are also suitable for analysis with this column subject to limitations given below.

The column can be used as a single, primary analytical separation column for oxygenated compounds. In complex sample matrices that have high molecular weight species (ca. 300 mol. wt. and higher) and/or species with insufficiently high vapor pressure to migrate through the GS-OxyPLOT, this column can be used in multidimensional GC systems with other columns that have vastly different polarity and lower selectivity toward oxygenated hydrocarbons. For example, a nonpolar DB-1 column can be used as an injection precolumn to retain low volatility solutes, allowing the less retained, polar oxygenated solutes to move into the GS-OxyPLOT. Since the stationary phase of GS-OxyPLOT is an oxygenate adsorbent phase, the oxygenates that enter the column are trapped. As the GC oven temperature is increased, the oxygenates will begin to migrate and are separated in the column prior to detection.

When first installed, the GS-OxyPLOT should be conditioned at 300 °C for at least 3 hours. Experience has shown that this column has an infinite shelf life, but when the column has not been in use for extended periods of time, longer conditioning times of 8 hours or more may be required to obtain retention time stability. The column can be stored with septa placed over the ends of the column, returned to the original column box, and stored at normal ambient temperatures for future use.

GS-OxyPLOT has a minimum temperature limit of 0 °C, an isothermal maximum temperature limit of 300 °C, and an oven program maximum temperature of 350 °C. Because the stationary phase is a strong adsorbent for polar compounds, especially water, it is recommended that when the column is installed in a GC, but idle, that the GC oven be set to an isothermal temperature of 220 °C with normal carrier gas flow, so that the instrument can be brought back into operation quickly when samples are ready to be analyzed. Otherwise, if the column is left at low oven temperatures, it may require reconditioning at 300 °C for several hours to obtain stable retention times.

Saturated hydrocarbon solutes have virtually no interaction with the GS-OxyPLOT and elute from the column so long as the column temperature is hot enough to induce a high enough vapor pressure for the solute to move in the carrier gas. Normal alkanes up to C_{18} will elute from GS-OxyPLOT within the program temperature maximum limit of the column. Because of the highly polar character of the GS-OxyPLOT phase, as would be

expected for oxygenate-selective PLOT column, the column has a relatively low sample load capacity for these nonpolar solutes. The low sample loading capacity is manifested chromatographically as a tailing peaking, indicative of phase overload in GS-OxyPLOT columns. Unsaturated hydrocarbons and aromatic hydrocarbons have relatively high retention. Injection of these organic compound classes should be limited to organic compounds with 11 carbons or less to prevent the column from fouling. As with the normal alkanes, the alkyl benzenes will show phase overloading at relatively low concentrations.

While GS-OxyPLOT is an ideal analytical solution for low molecular weight, oxygenated hydrocarbons, like all other similar oxygenate-selective PLOT columns,it is not recommended for higher molecular weight alkenals (e.g., 1-hexenal and 1-ocetenal). The combined interaction of the unsaturated and carbonyl functional groups can instigate tailing due to strong interactions and in some cases reaction between the phase and solutes.

Table 1. ASTM Standardized Methods for Which GS-OxyPLOT Is Specifically Designed

ASTM Method D7059	Determination of Methanol in Crude Oils by Gas Chromatography with Flame Ionization Detection
Proposed ASTM Method	Determination of C_1 to C_5 Oxygenates at Trace Levels in High Ethanol Content Gasoline Streams by Multidimensional Chromatography with Flame Ionization Detection*
Proposed ASTM Method	Determination of Oxygenates in Ethene, Propene, and C_4 and C_5 Hydrocarbon Matrices by Gas Chromatography and Flame Ionization Detection*

*These are "proposed methods" (i.e., do not have method designation numbers) that are destined for approval by ASTM Committee D2. These methods have already been accepted by, and are being implemented in, petrochemical refineries around the world.

Table 2. Examples of Oxygenated Compounds Suitable for GC Analysis Using the GS-OxyPLOT Column

1. Dimethyl Ether	13. Acetone
2. Diethyl Ether	14. Isovaleraldehyde
3. Acetaldehyde	15. Valeraldehyde
4. Ethyl t-Butyl Ether	16. Methyl Ethyl Ketone
5. Methyl t-Butyl Ether	17. Ethanol
6. Diisopropyl Ether	18. 1-Propanol
7. Propionaldehyde	19. Isopropyl Alcohol
8. tert-Amyl Methyl Ether	20. Allyl Alcohol
9. Propyl Ether	21. Isobutyl Alcohol
10. Isobutraldehyde	22. tert-Butyl Alcohol
11. Butylaldehyde	23. sec-Butyl Alcohol
12. Methanol	24. n-Butyl Alcohol
	25. 2-Methyl-2-Pentanol

Ordering Information for the GS-OxyPLOT Column

ID (mm)	Length (m)	Film Thickness (µm)	Temperature Limit (°C)	Cage Size	Part Number
0.53	10	10	350	7″	115-4912
0.53	10	10	350	5″	115-4912E

References

1. A. K. Vickers, “A ‘Solid’ Alternative for Analyzing Oxygenated Hydrocarbons—Agilent’s New Capillary GC PLOT Column,” Agilent Technologies publication 5989-6323EN, Feb 2006.
2. A New Megabore GC Column for the Adsorption and Chromatographic Separation of Oxygenates in Hydrocarbon Matrices, poster, Pittcon07-27.
3. Analysis and Chromatographic Separation of Oxygenates in Hydrocarbon Matrices, Power Point presentation, Pittcon07-20.

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