

Detection and Identification of Gulf Oil Dispersants (COREXIT® 9527 and 9500) by GC/MS and LC/MS/MS

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In an effort to contain the largest oil spill in United States history, nearly a million gallons of dispersant have been pumped into the Gulf of Mexico. Unfortunately, very little is known about the impact of these dispersants on humans, wildlife, and the environment. In this technical note we show how GC/MS with Zebron™ ZB-WAXPLUS™ GC columns can be used to detect COREXIT 9527 directly from seawater. To improve GC/MS stability, a cleanup procedure using the Strata™-X-A sorbent is used to virtually eliminate matrix interferences. The high efficiency Kinetex® core-shell columns simultaneously detect both COREXIT products at the low part per billion levels in seawater using LC/MS/MS. All solutions are optimized for high speed analysis and overall lab productivity.

Introduction

Revised estimates published by the National Incident Command's Flow Rate Technical Group (FRTG) report that 4.9 million barrels of oil were released into the Gulf of Mexico¹. The oil posed a significant health risk to the plant and animal life within the Gulf region. To help reduce the amount of oil that reached the coastline, dispersants were used to break-up the oil and sink it to the bottom of the ocean.²

Dispersants are composed of a mixture of solvents and detergents that break-up the heavy oil into droplets and allow it to be more readily decomposed in the environment. The primary dispersant used in the Gulf was COREXIT® 9500, although limited quantities of COREXIT® 9527 may have been used as well. To better understand the residual level of dispersants in the Gulf, robust analytical procedures are required. In this work we present solutions for the identification and quantitation of both COREXIT 9527 and 9500.

Materials and Methods

Methanol, acetonitrile, and other solvents used in LC/MS method development were obtained from Sigma-Aldrich (St. Louis, MO). Butoxyethanol (Fluka), Propylene Glycol, and Ethylene Glycol were also obtained from Sigma-Aldrich (St. Louis, MO). Sodium bis(2-ethylhexyl)sulfosuccinate, and its 13C4 enriched form were obtained from Cambridge Isotope Laboratories (Andover, MA). Other surfactants used in the analysis, n-heptanesulfonate, n-octanesulfonate, and n-dodecylsulfate, were obtained from Sigma-Aldrich (St. Louis, MO).

The GC method development was carried out on an Agilent® 6890 GC coupled with an Agilent 5973 MS detector (Agilent Technologies, Santa Clara, CA). The LC tandem MS data were collected on an AB SCIEX API 4000™ LC/MS/MS system (Foster City, CA) equipped with an Agilent UHPLC system containing 1200 SL binary pump, autosampler and column oven compartment (Agilent Technologies, Santa Clara, CA).

Results and Discussion

Nalco® has disclosed the exact composition of COREXIT 9527 and 9500 on their website (Table 1)³. The chemical composition of the COREXIT products was fairly similar, with the exception of 2-butoxyethanol, which was only found in COREXIT 9527. Both products contain a proprietary surfactant, which was disclosed to EPA to assist with the monitoring process.

Table 1.
Composition of COREXIT 9500 and 9527³

| Cas No. | Name |
|------------|----------------------------------------------------------------------------|
| 1338-43-8 | Sorbitan, mono-(9Z)-9-Octadecenoate |
| 9005-65-6 | Sorbitan, mono-(9Z)-9-Octadecenoate, poly(oxy-1, 2-ethanediyl) derivatives |
| 9005-70-3 | Sorbitan, tri-(9Z)-9-Octadecenoate, poly(oxy-1, 2-ethanediyl) derivatives |
| 577-11-7 | Bis(2-ethylhexyl) Sulfosuccinate* |
| 29911-28-2 | Propanol, 1-(2-butoxy-1-methylethoxy) |
| 64742-47-8 | Distillates (petroleum), hydrotreated light |
| 111-76-2 | 2-Butoxyethanol** |

* Contains propylene glycol

** Only included in COREXIT 9527

COREXIT 9527

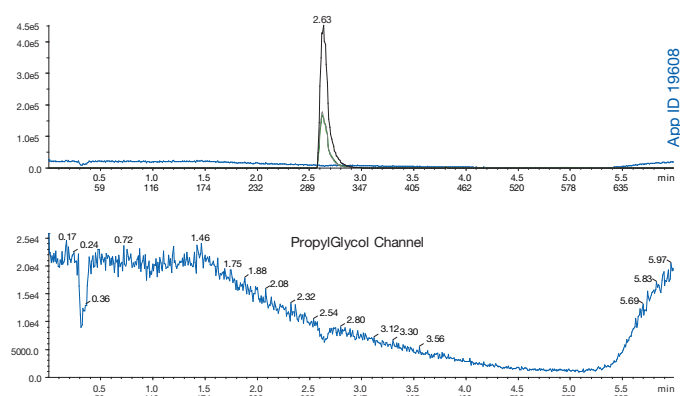
The presence of 2-butoxyethanol in a water sample would indicate that COREXIT 9527 has been used in that area. The analysis of 2-butoxyethanol was investigated using both GC/MS and LC/MS/MS. Since both COREXIT products also contained significant levels of propylene glycol, this component was also included in the analytical procedure.

An initial separation for all three compounds was done using a Kinetex® PFP 2.6 µm 50 x 2.1 mm column with the MS in positive ion mode (Figure 1). The mass channel for propylene glycol was extremely noisy and made detection impossible even at 50 ppb (µg/L). AB SCIEX systems use a glycol solution to tune the MS and correct the mass accuracy. Due to the sticky nature of glycols, the residual level of this solution in the instrument is too high to allow for quantitation. Considering these limitations, LC/MS/MS still appeared to be a viable option for the analysis of 2-butoxyethanol. The improved detection limits at the low ppb (µg/L) range, fast analysis time, and high specificity would be an asset for future studies.

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Figure 1.
Glycols by LC/MS/MS Using Kinetex® 2.6 µm PFP 50 x 2.1 mm



Column: Kinetex 2.6 µm PFP
Dimensions: 50 x 2.1 mm
Part No.: 00B-4477-AN
Mobile Phase: A: 5 mM Ammonium acetate
 B: Methanol
Flow Rate: 0.4 mL/min
Gradient:

| Time (min) | % B |
|------------|-----|
| 0 | 15 |
| 1.5 | 85 |
| 4 | 85 |
| 4.1 | 95 |
| 5.01 | 95 |
| 5.1 | 15 |
| 7 | 15 |

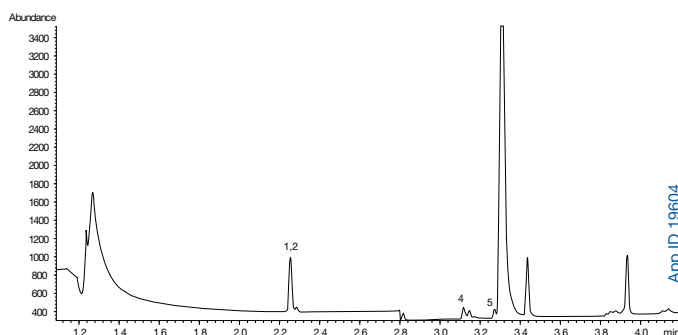
Temperature: 35 °C
Detection: Mass Spectrometer (MS)
Sample: 1. 2-Butoxyethanol (ESI+ Mode)
 2. Propylene glycol

In an effort to develop a methodology that would simultaneously detect both 2-butoxyethanol and propylene glycol, we investigated GC/MS as an alternative. The current reporting limit for 2-butoxyethanol is around 10 ppm (mg/L), which was well within the detection limits of the GC/MS.

We found that the Zebtron™ ZB-WAXPLUS™ phase provided the best resolution of all the glycols. In our experience, most labs avoid injecting water, especially when using WAX (polyethylene glycol) columns because many GC phases are not compatible with water. The Zebtron ZB-WAXPLUS was specially designed to be used with aqueous samples and gave very consistent performance for this study.

We first evaluated a method for direct analysis from seawater without any sample preparation other than a 0.45 µm Phenex™ syringe filter (Figure 2). The seawater sample contained impurities that did cause some problems with quantitation. The high salt content in the samples also necessitated more frequent system maintenance. Salt would accumulate in the liner and eventually cause problems with the calibration (Figure 3). If direct seawater injection is to be used, we would suggest doing full inlet maintenance at least daily, if not after every sample set. This should include replacing the liner and clipping 1-2 inches from the head of the GC column.

Figure 2.
Glycols from Seawater by GC/MS before SPE Cleanup



Column: Zebtron ZB-WAXPLUS
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G013-11
Injection: Split 10:1 @ 250 °C, 1 µL
Carrier Gas: Helium @ 1.5 mL/min (constant flow)
Oven Program: 100 °C to 200 °C @ 20 °C/min, post run 5 min @ 250 °C
Detection: MSD, SIM from 1-2.8 min mass 87 and 89, 2.8-5 min mass 31
Sample: Analytes are 1.7 ppm in seawater
 1. D4-2-Butoxyethanol (IS)
 2. 2-Butoxyethanol
 3. Formic acid (from elution)
 4. Propylene glycol
 5. Ethylene glycol (IS)

Figure 3.
Salt Contamination in GC Liner



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In order to eliminate the need for such frequent maintenance and reduce the impurities affecting quantitation, we explored ways to clean up the samples. Sample cleanup using traditional techniques was challenging because both compounds were extremely water soluble with Log P values <1.

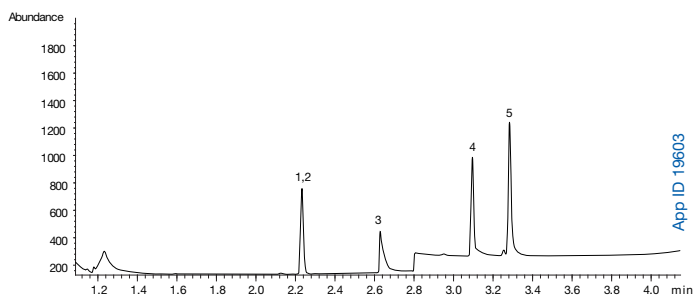
We evaluated several possibilities, but ultimately settled on a solid phase extraction (SPE) protocol using Strata™-X-A using the conditions listed in **Table 2**. To help improve method ruggedness, an internal standard was added prior to extraction and used to calculate relative recoveries. When choosing an internal standard, it is always best to use a deuterated analog, which was available for the 2-butoxyethanol, but not for propylene glycol. We were able to achieve acceptable results using ethylene glycol as an internal standard for propylene glycol. However, we have some concerns about potential interferences in Gulf Oil samples and would suggest evaluating alternatives for propylene glycol.

Table 2.
SPE Conditions using Strata X-A

| Strata-X-A 500 mg / 6 mL (Part Number: 8B-S123-HCH) | |
|-----------------------------------------------------|----------------------------------------------------------------------------|
| 1 | Condition: 3 mL Methanol, 3 mL Water |
| 2 | Load: 1 mL seawater (basified with ~25 µL of NH ₄ OH) |
| 3 | Dry: Full vacuum 5-10 minutes |
| 4 | Elute: 3 mL or 2 % Formic acid in Methanol |

Sample cleanup using the Strata-X-A sorbent significantly improves quantitation due to the elimination of interferences (**Figure 4**). Analysis of four replicate seawater samples spiked at 5 ppm (mg/L) showed relative recoveries greater than 96 % with less than 2 % RSD (**Table 3**). The calculated detection limits from 1 mL of seawater were about 1 ppm (mg/L). If lower detection limits were desired, a larger volume of seawater could be used.

Figure 4.
Glycols from Seawater by GC/MS after SPE Cleanup



Column: Zebtron ZB-WAXPLUS™
Dimensions: 30 meter x 0.25 mm x 0.25 µm
Part No.: 7HG-G013-11
Injection: Split 10:1 @ 250 °C, 1 µL
Carrier Gas: Helium @ 1.5 mL/min (constant flow)
Oven Program: 100 °C to 200 °C @ 20 °C/min, post run 5 min @ 250 °C
Detection: MSD, SIM from 1-2.8 min mass 87 and 89, 2.8-5 min mass 31
Sample: Analytes were extracted from seawater (1 mL load at 5 ppm)
 1. D4-2-Butoxyethanol (IS)
 2. 2-Butoxyethanol
 3. Formic acid (from elution)
 4. Propylene glycol
 5. Ethylene glycol (IS)

Table 3.
Recoveries for Glycols Using Strata X-A

| Compound | Criteria | Sample 1 | Sample 2 | Sample 3 | Sample 4 | Average | RSD |
|------------------|-----------------|----------|----------|----------|----------|---------|--------|
| 2-Butoxyethanol | Response Factor | 0.96 | 0.97 | 0.98 | 0.98 | 0.97 | 0.98 % |
| | Recovery | 99 % | 99 % | 101 % | 100 % | 100 % | 96 % |
| Propylene glycol | Response Factor | 0.72 | 0.74 | 0.74 | 0.75 | 0.74 | 1.71 % |
| | Recovery | 96 % | 99 % | 98 % | 100 % | 100 % | 1.71 % |

Butoxyethanol RF based on d4-2-butoxyethanol and propylene glycol based on ethylene glycol.

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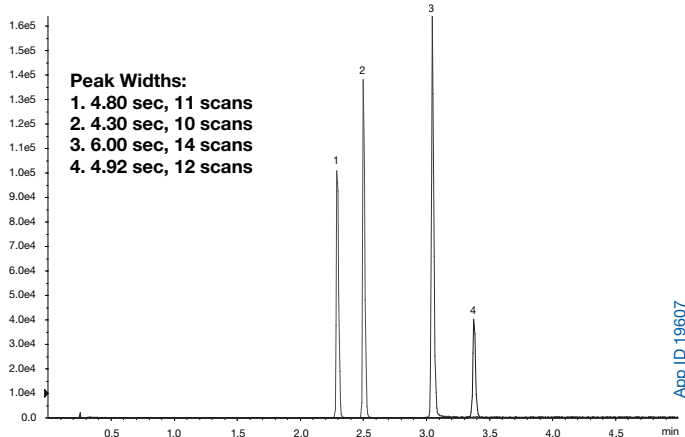
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COREXIT® 9500

The surfactants used in COREXIT are also used in a variety of other soaps and personal care products. Many surfactants are multi-component mixtures that are not amenable to GC/MS or LC/MS/MS analysis. The EPA has suggested that bis(2-ethylhexyl) sulfosuccinate (AOT) be used as a marker for COREXIT products.

A chromatographic separation was developed using the Kinetex® C8 2.6 µm 50 x 2.1 mm columns for a mixture of anionic surfactants that were likely to be present in seawater samples (Figure 5). The Kinetex core-shell material provided extremely high efficiencies allowing us to achieve an average peak width of only 5 seconds. This resulted in not only improved separation, but allowed us to achieve lower detection limits. The fast scan rate possible with the ABI 4000™ LC/MS/MS allowed for accurate quantitation enabling >10 scans/peak.

Figure 5. Chromatographic Resolution of Bis(2-ethylhexyl) Sulfosuccinate from Other Surfactant Interferences



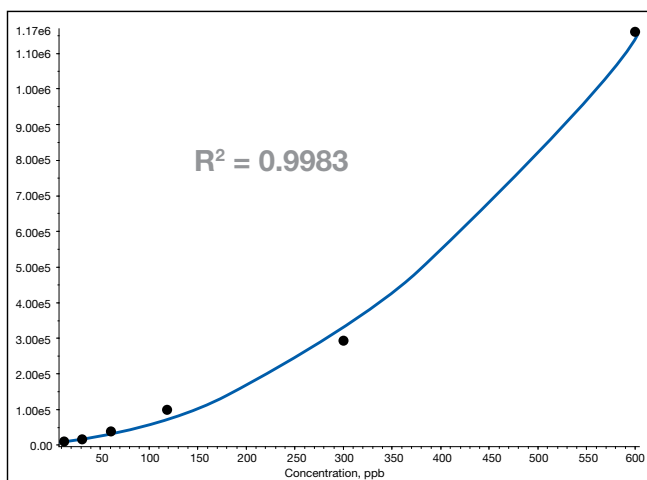
Column: Kinetex® 2.6 µm PFP
Dimensions: 50 x 2.1 mm
Part No.: 00B-4477-AN
Mobile Phase: A: 5 mM Ammonium acetate
 B: Methanol
Flow Rate: 0.4 mL/min
Gradient:

| Time (min) | % B |
|------------|-----|
| 0 | 15 |
| 1.5 | 85 |
| 4 | 85 |
| 4.1 | 95 |
| 5.01 | 95 |
| 5.1 | 15 |
| 7 | 15 |

Temperature: 35 °C
Detection: Mass Spectrometer (MS)
Instrument: API 4000
Sample: 1. Heptanesulfonate MRM 178.0⇒79.7
 2. Octanesulfonate MRM 192.9⇒79.9
 3. Dodecylsulfate MRM 265.0⇒96.8
 4. Bis(2-ethylhexyl) Sulfosuccinate MRM 421.1⇒80.9

As we suspected, our blank seawater matrix obtained from Redondo Beach, CA contained high levels of sodium lauryl sulfate (dodecylsulfate), which is commonly used in many personal care products. A calibration curve was run using the Kinetex C8 for seawater samples spiked at 10-600 ppb (µg/L). The resulting calibration curve plotted without the use of an internal standard showed a good correlation when using a quadratic 1/X weighting (Figure 6).

Figure 6. Calibration Curve for AOT without Internal Standard from 10-600 ppb with 1/X Weighting

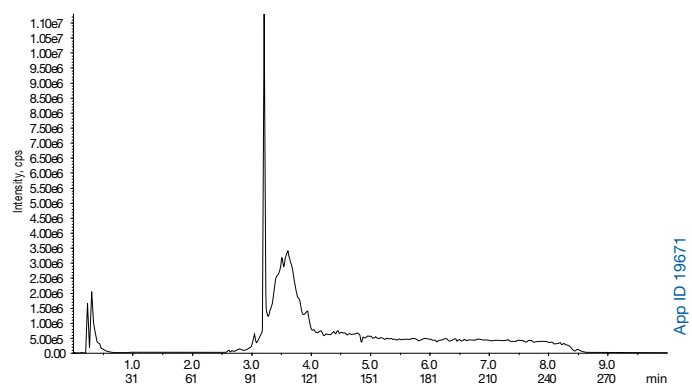


The shape of the curve indicated that there might be ion suppression or enhancement present at one end of the calibration range. Monitoring the 420-422 mass window (in single quad mode) in a sample fortified with AOT at 100 ppb revealed a partially co-eluting peak between 2.5-4 minutes (Figure 7). An isotopically labeled standard for AOT was available from Cambridge Isotope Labs (Andover, MA). Recalibration using the internal standard improved the linearity of the curve significantly (Figure 8). A larger mass scan range would have been performed to confirm the presence of additional co-eluting peaks had the narrow range scan been unsatisfactory.

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Figure 7.
Mass 420-422 Interference in Fortified Seawater Blank

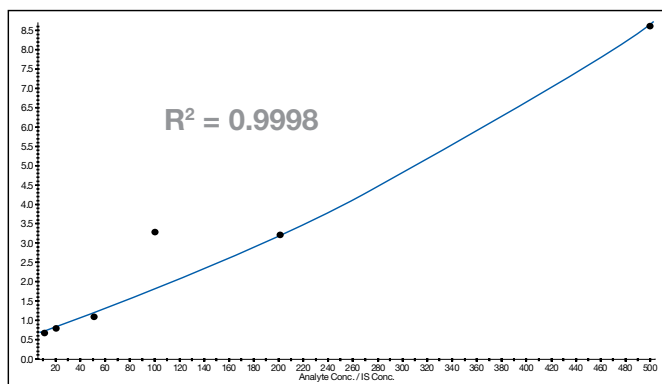


Column: Kinetex® 2.6 µm PFP
Dimensions: 50 x 2.1 mm
Part No.: 00B-4477-AN
Mobile Phase: A: 5 mM Ammonium acetate
 B: Methanol
Flow Rate: 0.4 mL/min
Gradient:

| Time (min) | % B |
|------------|-----|
| 0 | 15 |
| 1.5 | 85 |
| 4 | 85 |
| 4.1 | 95 |
| 5.01 | 95 |
| 5.1 | 15 |
| 7 | 15 |

Temperature: 35 °C
Detection: Mass Spectrometer (MS)
Sample: Bis(2-ethylhexyl) Sulfosuccinate (AOT)

Figure 8.
Calibration Curve using Deuterated Internal Standard from 10-500 ppb for AOT with 1/X weighting



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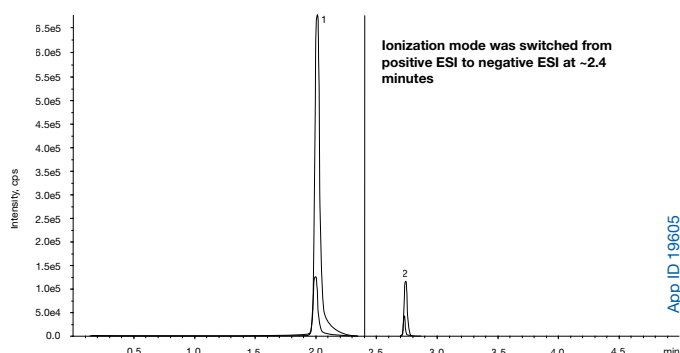
COREXIT® 9500 and 9527: Simultaneous Analysis

We have described analytical procedures for the analysis of both COREXIT products individually. To streamline workflow in an analytical testing laboratory, it would be beneficial to have a methodology to simultaneously analyze the two primary COREXIT markers. The acidic nature of AOT makes analysis by GC/MS impossible without some sort of derivatization, which indicated that LC/MS/MS would be the most appropriate technique.

AOT was best detected by LC/MS/MS using negative ion mode, while 2-butoxyethanol showed the best signal when using positive ion mode. In order to simultaneously analyze both compounds, it was necessary to switch polarity in the source during the elution window of each compound. The API 4000 LC/MS/MS instrument needs a 0.700 second settling time to switch polarity, requiring that the peaks be sufficiently separated.

The polar nature of these two compounds makes them difficult to separate using traditional reversed phase columns. The Kinetex® PFP phase is a pentafluorophenyl phase that has been shown to give unique polar selectivity. Using the Kinetex PFP 2.6 µm 50 x 2.1 mm column we were able to separate both peaks with almost one minute of separation (**Figure 9**). Peak shapes were again extremely sharp due to the high efficiency Kinetex core-shell particle.

Figure 9.
Simultaneous Analysis of Bis(2-ethylhexyl) Sulfosuccinate and 2-Butoxyethanol by LC/MS/MS



Column: Kinetex 2.6 µm PFP
Dimensions: 50 x 2.1 mm
Part No.: 00B-4477-AN
Mobile Phase: A: 5 mM Ammonium acetate
B: Methanol
Flow Rate: 0.4 mL/min
Gradient:

| Time (min) | % B |
|------------|-----|
| 0 | 15 |
| 1.5 | 85 |
| 4 | 85 |
| 4.1 | 95 |
| 5.01 | 95 |
| 5.1 | 15 |
| 7 | 15 |

Temperature: 35 °C

Detection: Mass Spectrometer (MS)

Instrument: API 4000™

Sample: 1. 2-Butoxyethanol (ESI+) MRM 119.9⇒45.2, 119.9⇒57.2 and 119.9⇒57.2
2. Bis(2-ethylhexyl) Sulfosuccinate (-ESI) MRM 421.1⇒80.9

Conclusions

Cleanup efforts following the Gulf Oil Spill are ongoing and there is a lot of work to be done. Due to the magnitude of this spill, there will be a lot of different types of samples that need to be tested and there is not one specific technique that can be applied to all. In this technical note we have discussed several different analytical solutions using LC/MS/MS and GC/MS for the detection of COREXIT markers.

The Kinetex core-shell columns provide ultra-high column efficiency, which improves resolution and detection limits. The Zebron ZB-WAXPLUS™ GC columns are 100 % aqueous stable and are a good solution when analyzing glycol products found in the COREXIT materials. The use of cleanup techniques such as SPE with Strata™-X-A can help improve the stability of the system reducing the amount of routine maintenance that must be performed.

The use of the procedures described in this technical note can help us better understand the impact of the dispersant chemicals on the Gulf ecosystem. Only through ongoing research can we hope to understand the long term effects of the oil spill. For more information on these or other Phenomenex solutions, please contact your local Technical Consultant or visit www.phenomenex.com.

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Ordering Information

Kinetex® 2.6 µm Analytical Columns (mm)

| | 30 x 4.6 | 50 x 4.6 | 75 x 4.6 | 100 x 4.6 | 150 x 4.6 |
|---------------|-------------|-------------|-------------|-------------|-------------|
| XB-C18 | — | 00B-4496-E0 | 00C-4496-E0 | 00D-4496-E0 | 00F-4496-E0 |
| C18 | 00A-4462-E0 | 00B-4462-E0 | 00C-4462-E0 | 00D-4462-E0 | 00F-4462-E0 |
| C8 | — | 00B-4497-E0 | 00C-4497-E0 | 00D-4497-E0 | 00F-4497-E0 |
| PFP | 00A-4477-E0 | 00B-4477-E0 | 00C-4477-E0 | 00D-4477-E0 | 00F-4477-E0 |
| HILIC | — | 00B-4461-E0 | 00C-4461-E0 | 00D-4461-E0 | 00F-4461-E0 |

Kinetex 2.6 µm MidBore™ Columns (mm)

| | 30 x 3.0 | 50 x 3.0 | 75 x 3.0 | 100 x 3.0 | 150 x 3.0 |
|---------------|-------------|-------------|-------------|-------------|-------------|
| XB-C18 | 00A-4496-Y0 | 00B-4496-Y0 | 00C-4496-Y0 | 00D-4496-Y0 | — |
| C18 | 00A-4462-Y0 | 00B-4462-Y0 | 00C-4462-Y0 | 00D-4462-Y0 | 00F-4462-Y0 |
| C8 | 00A-4497-Y0 | 00B-4497-Y0 | 00C-4497-Y0 | 00D-4497-Y0 | — |
| PFP | 00A-4477-Y0 | 00B-4477-Y0 | 00C-4477-Y0 | 00D-4477-Y0 | 00F-4477-Y0 |
| HILIC | 00A-4461-Y0 | — | — | — | 00F-4461-Y0 |

Kinetex 2.6 µm Minibore Columns (mm)

| | 30 x 2.1 | 50 x 2.1 | 100 x 2.1 | 150 x 2.1 |
|---------------|-------------|-------------|-------------|-------------|
| XB-C18 | 00A-4496-AN | 00B-4496-AN | 00D-4496-AN | 00F-4496-AN |
| C18 | 00A-4462-AN | 00B-4462-AN | 00D-4462-AN | 00F-4462-AN |
| C8 | 00A-4497-AN | 00B-4497-AN | 00D-4497-AN | 00F-4497-AN |
| PFP | 00A-4477-AN | 00B-4477-AN | 00D-4477-AN | 00F-4477-AN |
| HILIC | — | 00B-4461-AN | 00D-4461-AN | 00F-4461-AN |



Strata™-X-A

| Sorbent Mass | Part No. | Unit |
|----------------------|-------------|----------------|
| Tube | | |
| 30 mg | 8B-S123-TAK | 1 mL (100/box) |
| 30 mg | 8B-S123-TBJ | 3 mL (50/box) |
| 60 mg | 8B-S123-UBJ | 3 mL (50/box) |
| 100 mg | 8B-S123-EBJ | 3 mL (50/box) |
| 100 mg | 8B-S123-ECH | 6 mL (30/box) |
| 200 mg | 8B-S123-FBJ | 3 mL (50/box) |
| 200 mg | 8B-S123-FCH | 6 mL (30/box) |
| 500 mg | 8B-S123-HBJ | 3 mL (50/box) |
| 500 mg | 8B-S123-HCH | 6 mL (30/box) |
| Giga™ Tube | | |
| 500 mg | 8B-S123-HDG | 12 mL (20/box) |
| 1 g | 8B-S123-JDG | 12 mL (20/box) |
| 1 g | 8B-S123-JEG | 20 mL (20/box) |
| 2 g | 8B-S123-KEG | 20 mL (20/box) |
| 5 g | 8B-S123-LFF | 60 mL (16/box) |
| 96-Well Plate | | |
| 10 mg | 8E-S123-AGB | 2 Plates/Box |
| 30 mg | 8E-S123-TGB | 2 Plates/Box |
| 60 mg | 8E-S123-UGB | 2 Plates/Box |



Zebtron™ ZB-WAXPLUS™ GC Columns

| ID(mm) | df(µm) | Temp. Limits °C | Part No. |
|-----------------|--------|-----------------|-------------|
| 10-Meter | | | |
| 0.10 | 0.10 | 20 to 250/260 | 7CB-G013-02 |
| 15-Meter | | | |
| 0.25 | 0.25 | 20 to 250/260 | 7EG-G013-11 |
| 20-Meter | | | |
| 0.18 | 0.18 | 20 to 250/260 | 7FD-G013-08 |
| 30-Meter | | | |
| 0.25 | 0.25 | 20 to 250/260 | 7HG-G013-11 |
| 0.32 | 0.25 | 20 to 250/260 | 7HM-G013-11 |
| 0.32 | 0.50 | 20 to 250/260 | 7HM-G013-17 |
| 0.53 | 1.00 | 20 to 230/240 | 7HK-G013-22 |
| 60-Meter | | | |
| 0.25 | 0.25 | 20 to 250/260 | 7KG-G013-11 |
| 0.32 | 0.50 | 20 to 250/260 | 7KM-G013-17 |
| 0.53 | 1.00 | 20 to 230/240 | 7KK-G013-22 |

Note: If you need a 5 in. cage, simply add a (-B) after the part number, e.g., 7HG-G013-11-B. Some exceptions may apply. Agilent 6850 and some SRI and process GC systems use only 5 in. cages.



If Phenomenex products in this technical note do not provide at least an equivalent separation as compared to other products of the same phase and dimensions, return the product with comparative data within 45 days for a FULL REFUND.

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Strata-X is patented by Phenomenex, Inc. U.S. Patent No. 7,119,145

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