

Enhanced Results of Diesel Range Organics Analysis with High Temperature Zebron[™] Inferno[™] GC Columns

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Petroleum hydrocarbon analysis is widely performed on soil and water samples to determine the presence and quantified levels of contamination from petroleum fuel products and crude oils. Fuel hydrocarbons are segmented into gasoline and diesel range organic groups (GRO and DRO). DRO compounds have boiling points similar to diesel fuel (C₁₀-C₂₈), ranging from 170-430 °C. These heavy compounds are amenable to GC/FID analysis; however, important considerations should be taken to ensure reproducible results over repeated injections.

Introduction

Although DRO analytical methods may not reach excessively high temperatures, the nature of the samples being injected requires periodic GC oven ramps to fully remove contaminants from the column. Without ramping of the oven to sufficiently high temperatures, heavy contaminants can remain on the column, thereby decreasing reproducibility over repeated injections. Another reason for chemists to employ aggressive oven ramps is to produce rapid method run times. However, steep oven ramps may approach the temperature limits of typical GC columns, and repeated use of a column near its temperature limit can cause excessive phase bleed and column brittleness.

For these reasons, high temperature GC columns are preferred for DRO analysis. Zebron Inferno GC columns carry the highest temperature limits of any non-metal GC columns (up to 430 °C). This elevated temperature limit provides chemists the flexibility to increase method throughput with aggressive oven ramps, extend column lifetime with rugged temperature stability, and improve reproducibility by baking off contaminants. This study presents two methods for analyzing DROs using Zebron Inferno GC columns.

Materials and Methods

An Agilent[®] 6890 GC/FID system was used. Conditions for each application are shown in **Table 1**.

Table 1.
Operating conditions for DRO methods

| | ZB-5HT Inferno (Figure 1) | ZB-1HT Inferno (Figure 2) |
|-------------------|---------------------------------------|---|
| Column Dimensions | 15 m x 0.32 mm x 0.10 µm | 15 m x 0.32 mm x 0.25 µm |
| Injection | On-Column 0.1 µL @ 63 °C | Splitless 20 µL @ 300 °C |
| Oven Ramp | 60 °C to 380 °C @ 35 °C/min for 5 min | 50 °C for 2 min to 320 °C @ 30 °C/min for 5 min |
| Carrier Gas | Constant Flow Helium, 2.7 mL/min | Constant Flow Helium, 2 mL/min |
| Detector | FID @ 400 °C | FID @ 330 °C |

Recommended GC Accessories

- Easy Seals[™] Inlet Base Seals (Part No. AGO-8620)
- PhenoRed[™]-400 Injector Septa (Part No. AGO-4696)
- Split/Splitless, Single Taper Liner, 4 mm x 78.5 mm x 6.45 mm (Part No. AGO-7516)

Results and Discussion

Figure 1 shows a 10-minute diesel analysis using an on-column injection technique, which increases sensitivity of late-eluting compounds by reducing inlet discrimination. However, the on-column technique can be limited by the volume injected onto the column. To improve sensitivity, a large volume injection of 20 µL is shown in **Figure 2**.

Figure 1.

Diesel fuel on ZB-5HT. Sample was 0.1 mg/mL in dichloromethane. Analytes: 1) Decane, 2) Tetracontane

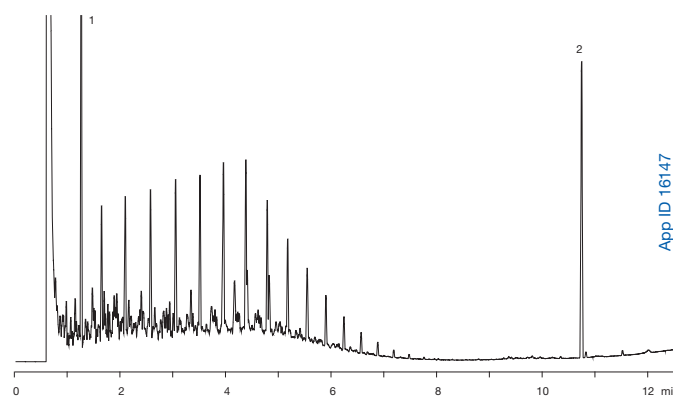
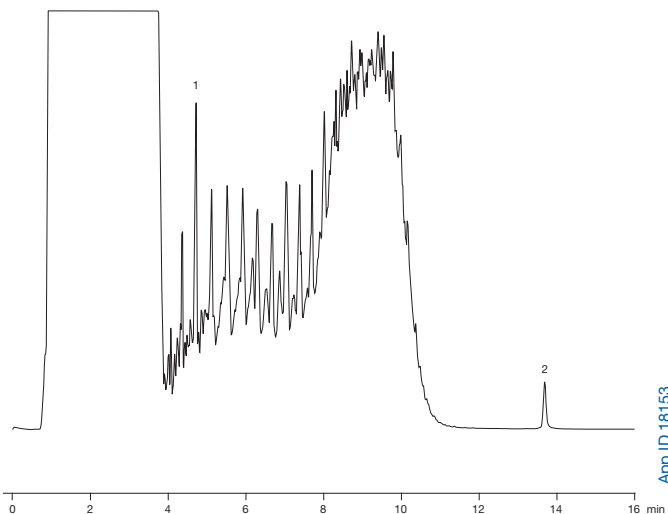


Figure 2.

Hydrocarbons from water DIN EN ISO 9377-2 (DEV H53) on ZB-1HT. Analytes: 1) Decane, 2) Tetracontane



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Conclusions

Two robust methods were presented for the DRO fraction analysis using on-column and large volume injection techniques. While both use high temperature columns with the ability to safely condition at extreme temperatures, the large volume injection provides improved sensitivity. This increased sensitivity can reduce volumes of extracted samples and provide labs savings on sampling, transport, and extraction solvent costs.

References

The large volume injection method (**Figure 2**) was provided by Dr. Alexander Ruderish from AGROLAB Labor GmbH in Bruckberg, Germany.

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Ordering Information

| Zebron ZB-1HT Inferno GC Columns | | | |
|----------------------------------|---------|-----------------|-------------|
| ID (mm) | df (µm) | Temp. Limits °C | Part No. |
| 5-Meter | | | |
| 0.53 | 0.10 | -60 to 400/430 | 7AK-G014-02 |
| 10-Meter | | | |
| 0.32 | 0.25 | -60 to 400/430 | 7CM-G014-11 |
| 15-Meter | | | |
| 0.25 | 0.10 | -60 to 400/430 | 7EG-G014-02 |
| 0.25 | 0.25 | -60 to 400/430 | 7EG-G014-11 |
| 0.32 | 0.10 | -60 to 400/430 | 7EM-G014-02 |
| 0.32 | 0.25 | -60 to 400/430 | 7EM-G014-11 |
| 0.53 | 0.15 | -60 to 400 | 7EK-G014-05 |
| 20-Meter | | | |
| 0.18 | 0.18 | -60 to 400/430 | 7FD-G014-08 |
| 30-Meter | | | |
| 0.25 | 0.10 | -60 to 400/430 | 7HG-G014-02 |
| 0.25 | 0.25 | -60 to 400/430 | 7HG-G014-11 |
| 0.32 | 0.10 | -60 to 400/430 | 7HM-G014-02 |
| 0.32 | 0.25 | -60 to 400/430 | 7HM-G014-11 |
| 0.53 | 0.15 | -60 to 400 | 7HK-G014-05 |

| Zebron ZB-5HT Inferno GC Columns | | | |
|---|---------|-----------------|-----------------|
| ID (mm) | df (µm) | Temp. Limits °C | Part No. |
| 10-Meter with 2-Meter Spliced Guard (0.53 mm ID) | | | |
| 0.32 | 0.10 | -60 to 400/430 | 7CM-G015-02-GST |
| 15-Meter | | | |
| 0.25 | 0.10 | -60 to 400/430 | 7EG-G015-02 |
| 0.25 | 0.25 | -60 to 400/430 | 7EG-G015-11 |
| 0.32 | 0.10 | -60 to 400/430 | 7EM-G015-02 |
| 0.32 | 0.25 | -60 to 400/430 | 7EM-G015-11 |
| 0.53 | 0.15 | -60 to 400 | 7EK-G015-05 |
| 15-Meter with 2-Meter Spliced Guard (0.53 mm ID) | | | |
| 0.32 | 0.10 | -60 to 400/430 | 7EM-G015-02-GST |
| 20-Meter | | | |
| 0.18 | 0.18 | -60 to 400/430 | 7FD-G015-08 |
| 30-Meter | | | |
| 0.25 | 0.10 | -60 to 400/430 | 7HG-G015-02 |
| 0.25 | 0.25 | -60 to 400/430 | 7HG-G015-11 |
| 0.32 | 0.10 | -60 to 400/430 | 7HM-G015-02 |
| 0.32 | 0.25 | -60 to 400/430 | 7HM-G015-11 |
| 0.53 | 0.15 | -60 to 400 | 7HK-G015-05 |
| 60-Meter | | | |
| 0.25 | 0.25 | -60 to 400/430 | 7KG-G015-11 |



If Zebron columns do not provide at least an equivalent separation as compared to any other GC column of the same phase and comparable dimensions, return the column with your comparative data within 45 days for a FULL REFUND.

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