Measuring Hydrocarbon Oil Index according to ISO 9377-2 (DIN H53)

Environmental Application

Gas Chromatography

Bernhard Wüst

Abstract

Agilent Technologies developed this analytical method to comply with ISO 9377-2 and DIN H53 Standards. The method employs a simple instrument configuration consisting of a Gas Chromatograph (GC) equipped with a Split/Splitless (S/SL) inlet and Flame Ionisation Detector (FID), making the method both easy to implement and robust to operate. The combination of an Agilent GC system (see configuration above), selected capillary GC column, proprietary injection port liner, appropriate chemical standards as well as method installation and support by an Agilent Applications Specialist, provide a *complete solution* for the measurement of the Hydrocarbon Oil Index.

Introduction

The Hydrocarbon Oil Index (HOI) is defined as the total amount of compounds which can be extracted from the sample (potable water, surface water and waste water) with a non-polar solvent having a boiling point between 39°C and 69°C (replacing the use of halogenated solvents). In addition, the compounds must not absorb on Florisil and must elute between n-decane ($C_{10}H_{24}$) and n-tetracontane ($C_{40}H_{82}$) when analysed by GC using an apolar analytical column. Restrictions on the use of halogenated hydrocarbons as solvents for analytical applications combined with the increasing cost of and reduced availability of technical grade halogenated solvents as well as documented environmental considerations resulted in the change to a non-polar solvent for HOI determinations.

This analytical method complies with the ISO Standard 9377-2 for the determination of the Hydrocarbon Oil Index by Gas Chromatography. The method is suitable for HOI determinations in concentrations above 0.1 mg/L in drinking waters, surface waters, waste waters and waters from sewage treatment plants.

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Experimental

The Agilent 6890 and Agilent 6850 Gas Chromatographs are well suited to the HOI application. Both instruments use the same Split/Splitless inlet system and identical Flame Ionisation Detector. Discrimination effects on higher boiling compounds are the most critical aspect of the analysis and are prevented by the use of a special injection port liner which, in combination with the Agilent S/SL inlet system, produces robust and reliable data. Additional benefits such as reduced maintenance time and faster analytical cycle times are also available by using a S/SL inlet as compared to a Cool-on-Column inlet.

The Agilent GC ChemStation standard software is able to subtract background signals to produce a chromatogram with any column bleed removed. In addition, both the Agilent 6890 and 6850 are able to save column compensation signals in memory and subsequently subtract the column background from the sample data and provide a background-subtracted signal to the ChemStation or data integration system. Standard ChemStation software tools are used to control the GC and perform the data analysis required of the sample data. A custom report template is included with the HOI method and generates a report conforming to the ISO Standard (see figure 3).

Instrument Parameters

Oven temperature profile	Isothermal 35°C (1.5 min), ramp 5°C/min to 60°C hold 0 min, ramp 15°C/min to 350°C hold 5 min.
Inlet (S/SL)	375°C Splitless Mode
Detector (FID)	375°C
Analytical Column	Agilent Part Number 19095Z-221E (HP1, 15m x 0.53mm x 0,15 µm)
Inlet Liner	Agilent Part Number 5183-4647
Carrier Gas	Helium
Carrier Gas Flow Rate	7.4 ml/min Constant Flow Mode
Injection Mode	Automated (Agilent 7683) Fast Injection
Injection Volume	1.0 µl

Calibration Curve

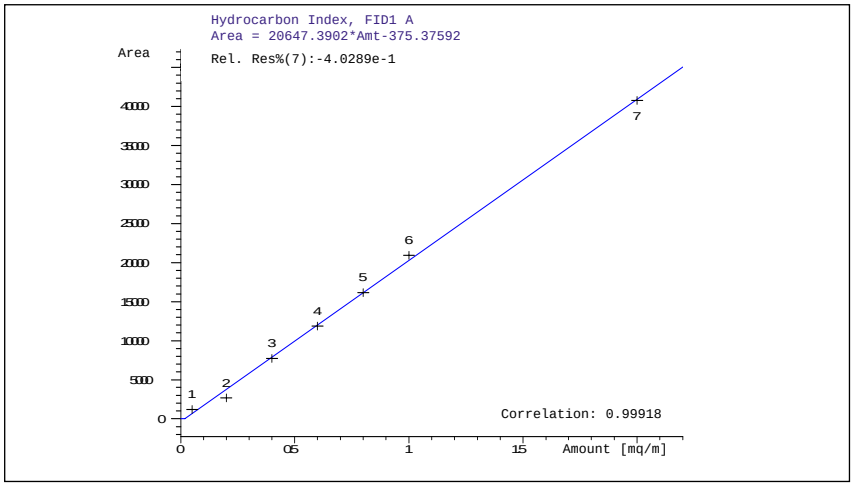


Figure 1: Oil Standards – Calibration Curve

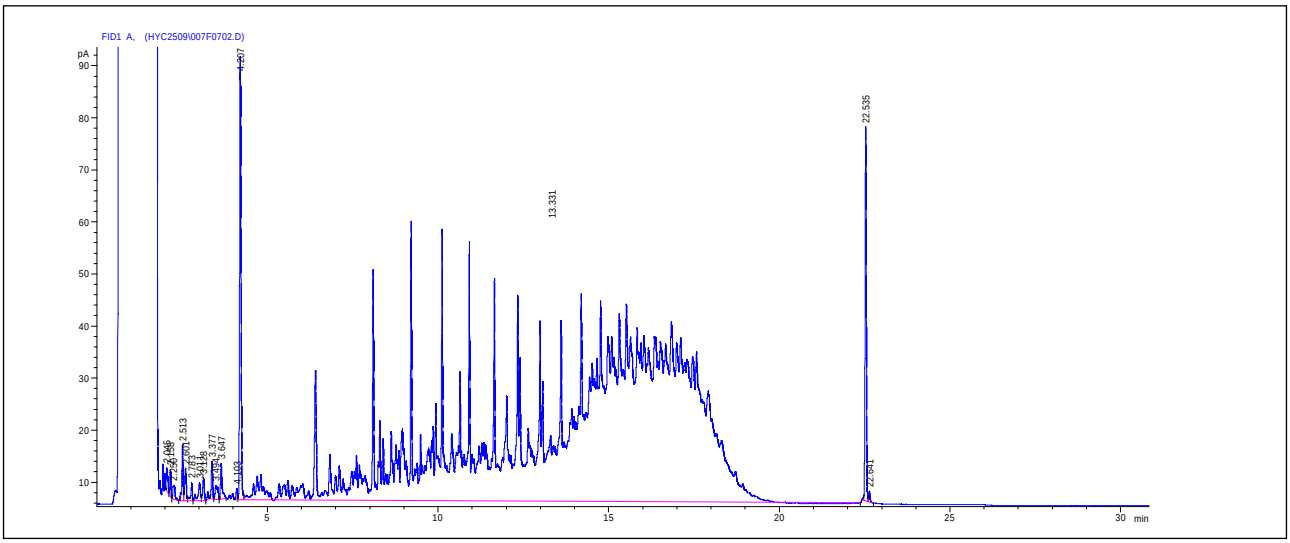


Figure 2: 0.6 mg/ml Oil Standard

Discrimination Test:

25 sequential injections of a standard containing tetracontane C-40 and decane C-10 were run to test for discrimination effects within the analytical system. The data obtained is presented in Figure 3 and demonstrates the robustness of the method and that the analytical system clearly passes the discrimination test specified in the ISO Standard.

Results and Dicussion

The Agilent 6890 and 6850 Gas Chromatographs equipped with a Split/Splitless inlet and Flame Ionisation Detector can be used for Hydrocarbon Oil Index determinations in accordance with ISO 9377-2 (DIN H53) standard. In addition, the analytical system is easy to operate and requires minimal maintenance. Together, the Agilent GC, analytical capillary column, special inlet liner, custom report template, selected chemical standards and Agilent applications expertise provide a complete analytical solution for Hydrocarbon Oil Index determinations.

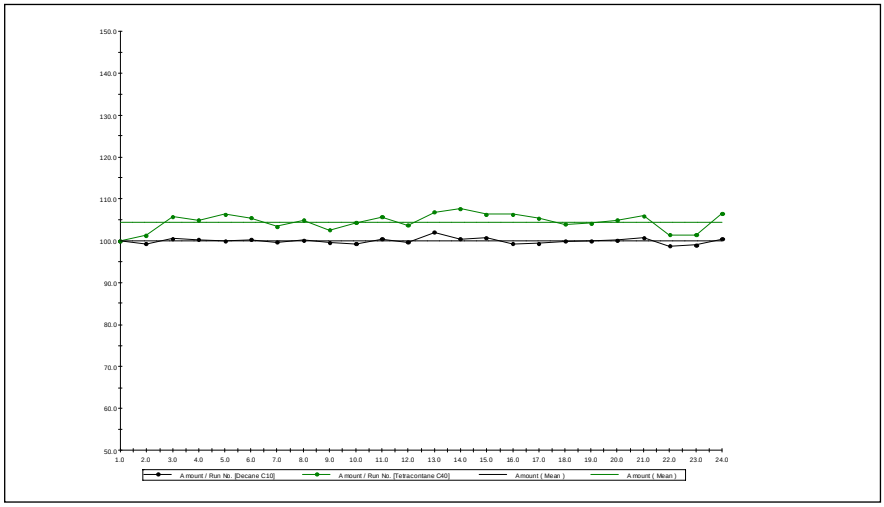
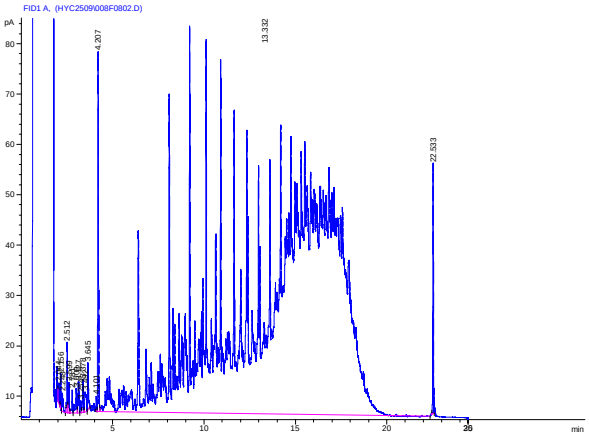


Figure 3: Discrimination Test

```
Data file   : C:\HPCHEM\1\DATA\HYC2509\008F0802.D
Sample Name : 1000 mg/l                                          1
================================================================
Injection Date    : 9/26/2000 3:23:50 AM       Seq Line : 8
Sample Name       : 1.0 mg/ml                  Vial No. : 8
Acq Operator      : berwuest                   Inj. No. : 2

                                               Inj. Vol. : -
Acq.Method        : HYCA04.M
Analysis Method   : C:\HPCHEM\1\METHODS\ISO93774.M
Last Changed      : Fri, 29. Sep. 2000, 10:04:10 am

Analysis of Hydrocarbon Index according to Iso 9377-4 and
DIN H53
```




```
================================================================================
                              Hydrocarbon Index Report
================================================================================
Calib. Data Modified      : Fri, 29. Sep. 2000,08:25:56 am
Multiplier                : 1.000000

        Hydrocarbon Index    1.052 mg/ml
        Low Boilers          9.061 mg/l
        High Boilers         not detectable  mg/l

================================================================================
                              Detailed Report
================================================================================
Name              StartTime EndTime Ret Time Total Area  Res.F    Amount
--------------------------------------------------------------------------------
Low Boilers          2.00    4.00     -        239.48    0.03784  9.06
Decane C10            -       -       4.21    213.73    0.03784  8.09
Hydrocarbon Index    4.31   22.37    13.33  15408.08    0.00006  1.05
Tetracontane C40      -       -      22.53    205.67    0.03247  7.94
High Boilers          -       -       -        -         -       n.d.
================================================================================
```

Figure 4: Report Printout

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