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Agilent Technologies
Premier Solution Partner

H53 analytics

High throughput with Intuvo 9000

Application Note

Environment

ABSTRACT

This application note demonstrates the high throughput analytics of H53 samples for determination of the hydrocarbon index with Intuvo 9000 GC system by Agilent Technologies. As runtime of the H53 analysis is approx. 2 minutes, this system offers significant time savings compared to conventional GC systems with standard runtimes of 20-30 minutes.

INTRODUCTION

Measurements were carried out on basis of DIN EN ISO 9377-2:2000, which is called H53 analytics, according to its source (DEV53). This procedure is used for determination of the hydrocarbon index and carries the subtitle “Teil.2: Verfahren nach Lösemittelextraktion und Gaschromatographie (H53)” (part 2: procedure after solvent extraction of and gas chromatography (H53)).

The purpose of this application note is a demonstration of the new Agilent Intuvo 9000 for high throughput analytics of H53 samples.

The standard runtime of an H53 application is 20 - 30 minutes, depending on the GC system and method parameters. Utilizing the Intuvo 9000, the runtime can be shortened significantly through extremely high heating rates. The introduced method provides advantages with an analysis time of approx. 2 minutes. Combined with the subsequent cooling-down period the run-to-run time is approx. 8 minutes.

Intuvo 9000 presents a novel generation of GC systems, which defines gas chromatography in a completely new way through innovative technologies.

Including:

- **direct heating oven, shorter cycle duration** – planar column design
- **fast, reliable column change** – click-and-run connections
- **no cutting of columns** – Intuvo Guard Chip technology
- **immediately available system information** – intuitive touchscreen
- **more space in the lab** – compared to conventional benchtop GCs only half the floor space

EXPERIMENTAL

Chemicals and solutions

- hydrocarbon standards of 0.025 – 5.0 mg/mL
- Alkane20 (C_{10} to C_{40})
- various real samples

Instrument

- Intuvo 9000 S/SL FID
- ALS

Software

- OpenLab CDS 2.1

Method parameters

Parameter	
Agilent Intuvo 9000	
Column	Intuvo DB1-HT
Column flow (const.)	7 mL/min
Carrier gas	helium
Inlet	splitless, 350 °C
Oven temperature program	40 °C for 0.25 min, 250 °C/min to 370 °C, 1min
Detector FID	350 °C
Intuvo Guard Chip	350 °C
Intuvo Bus temp.	350 °C
ALS	
Syringe	10 µL
Inj. Vol.	1 µL

RESULTS AND DISCUSSION

According to paragraph 9.7.1 of the H53 norm, a discrimination-free injection and an optimized separation is shown by referring to an alkane standard in the range of C_{10} to C_{40} (see Fig. 1). For this purpose, the relative response (peak area) between n-tetracontane ($C_{40}H_{82}$) and n-icosane ($C_{20}H_{42}$) is determined. The target for the RF value of > 0.8 is being observed (see Tab. 1).

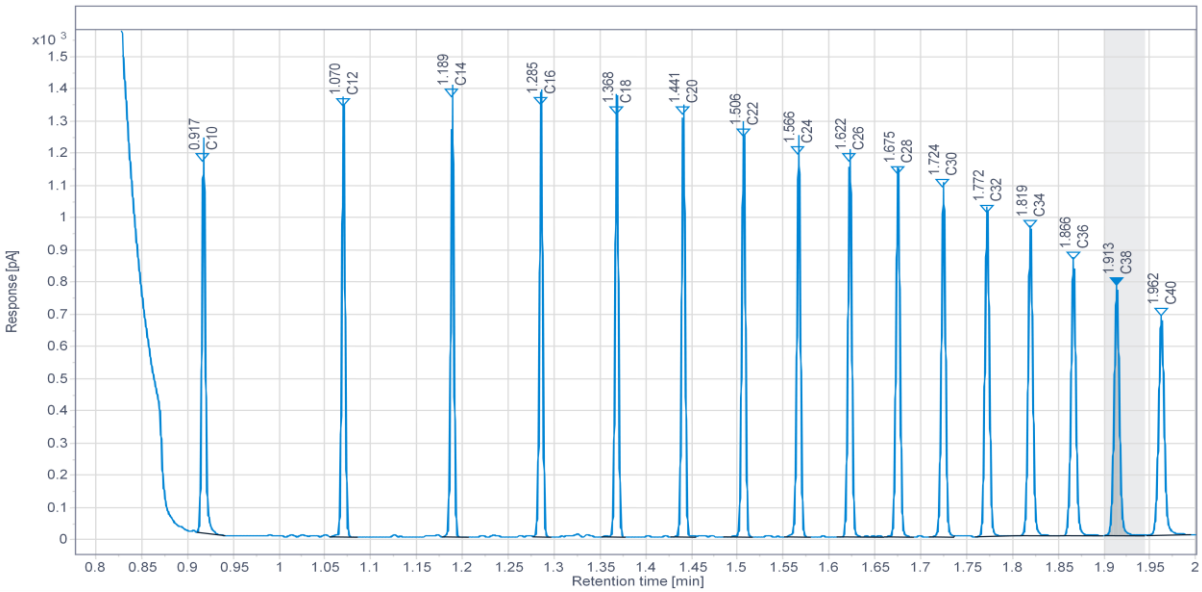

Fig. 1 Alkane mix C_{10}-C_{40}

Tab. 1 Evaluation alkane mix C_{10}-C_{40} with relative response factor (RF, C_{40}/C_{20})

#	Name	RT (min)	Height	Area
1	C10	0.917	1148.141	322.912
2	C12	1.07	1329.915	331.323
3	C14	1.189	1358.742	332.223
4	C16	1.285	1335.291	334.698
5	C18	1.368	1306.249	338.606
6	C20	1.441	1303.974	341.700
7	C22	1.506	1236.435	340.773
8	C24	1.566	1184.042	342.072
9	C26	1.622	1159.585	344.574
10	C28	1.675	1119.192	340.118
11	C30	1.724	1080.058	337.827
12	C32	1.772	998.037	333.64
13	C34	1.819	951.105	336.646
14	C36	1.866	852.478	319.051
15	C38	1.913	770.672	309.836
16	C40	1.962	673.808	290.047

RF= 0.85

For calibration, standards of a 1:1 diesel fuel/lube oil mix in the range between 0.025 and 5.0 mg/mL was used. The end of C_{10} and the start of the C_{40} peak were used as integration markers for the petroleum hydrocarbons. All chromatograms of the calibration standard between 0.025 – 5.0 mg/mL, including blank value, are illustrated in Fig. 2.
A magnification of the chromatograms in the range between 0.025 and 0.05 mg/mL, including blank value, is shown in Fig. 3.

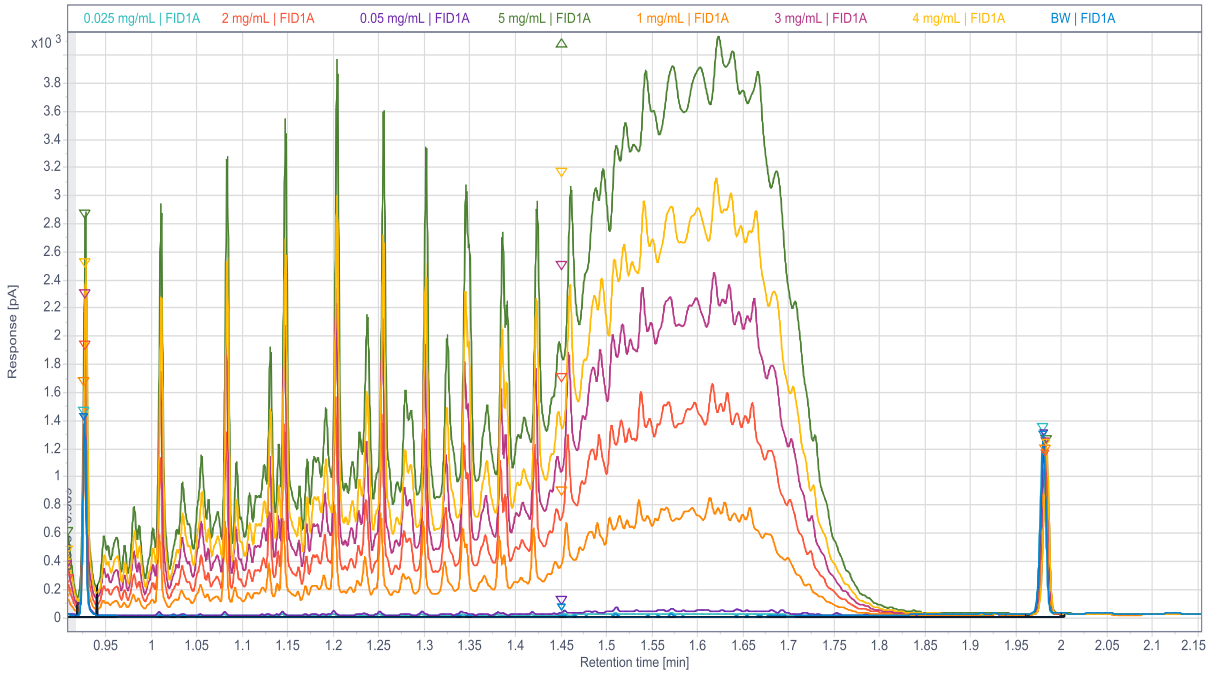

Fig. 2 Overlay calibration standards 0.025 – 5.0 mg/mL and blank

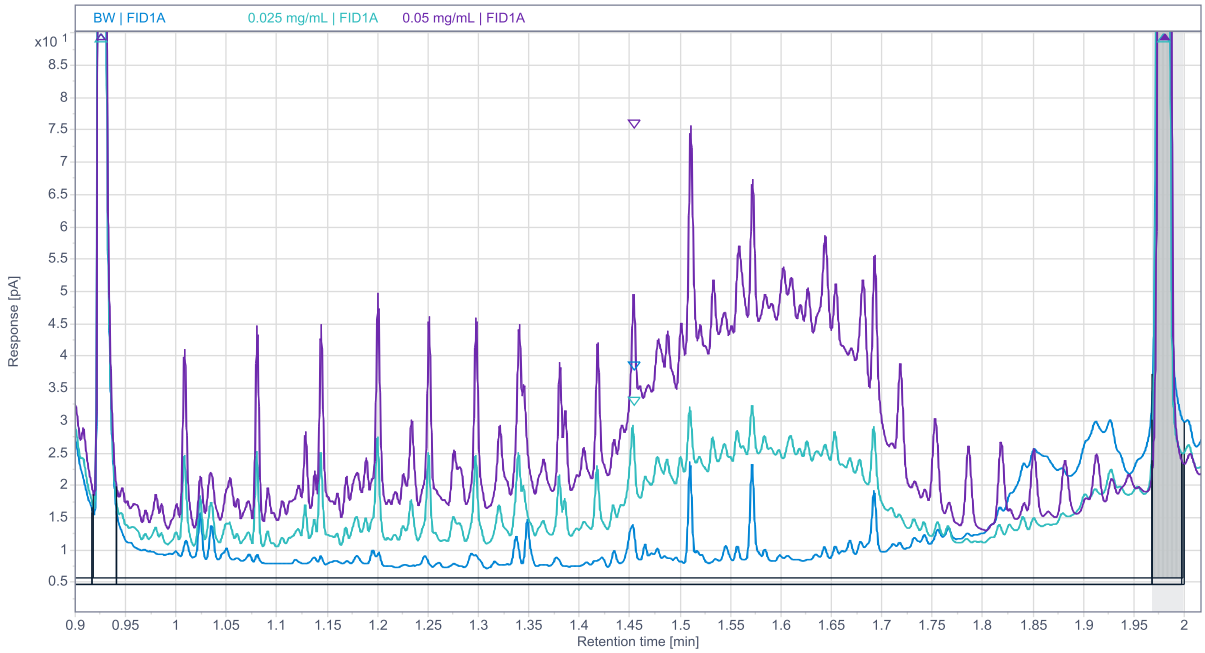

Fig. 3 Magnification: overlay blank value – 0.025 mg/mL and 0.05 mg/mL

The reference function with prognosis interval is illustrated in Fig. 4. Coefficient of determination (R^2) as well as the standard deviation for the method (SDx_o) can be found on the legend.

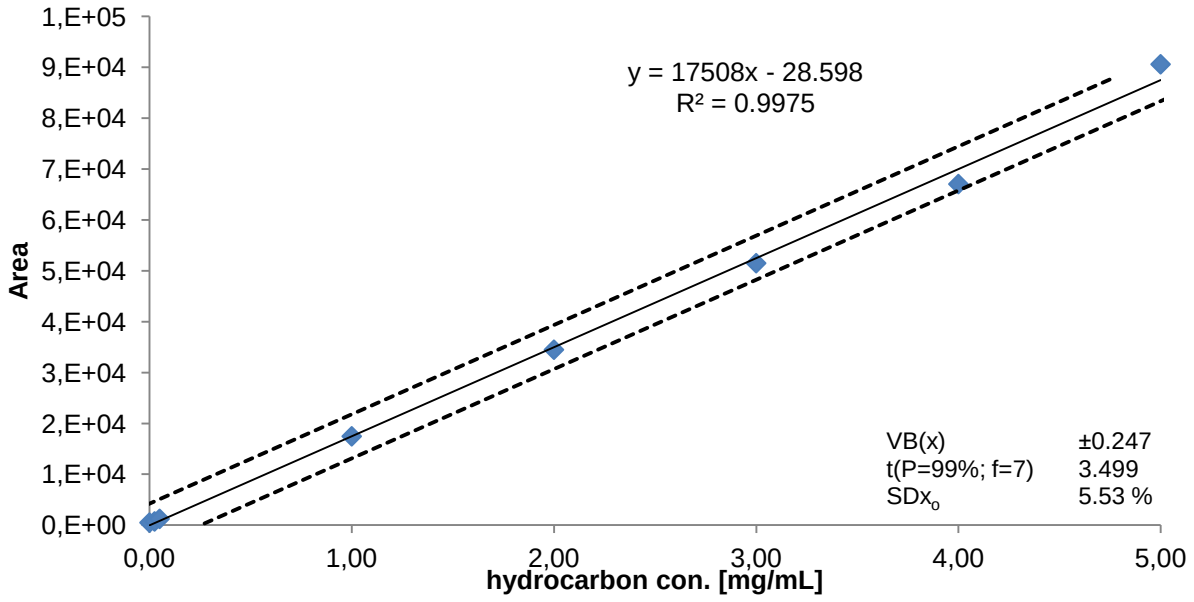

Fig. 4 Calibration of petroleum hydrocarbons from 0.025 mg/mL – 5.0 mg/mL

Samples of pure diesel fuel and lube oil standards with a concentration of 1.0 mg/mL (Fig. 5) as well as reference mix standards with 0.5 and 1.0 mg/mL and two real soil samples were used for testing. The results are listed in Tab. 2. A Trace GC Ultra UFM by Thermo Scientific was used as reference system. Concentration determined by the Trace GC was used as setpoint. Each sample was injected threefold.

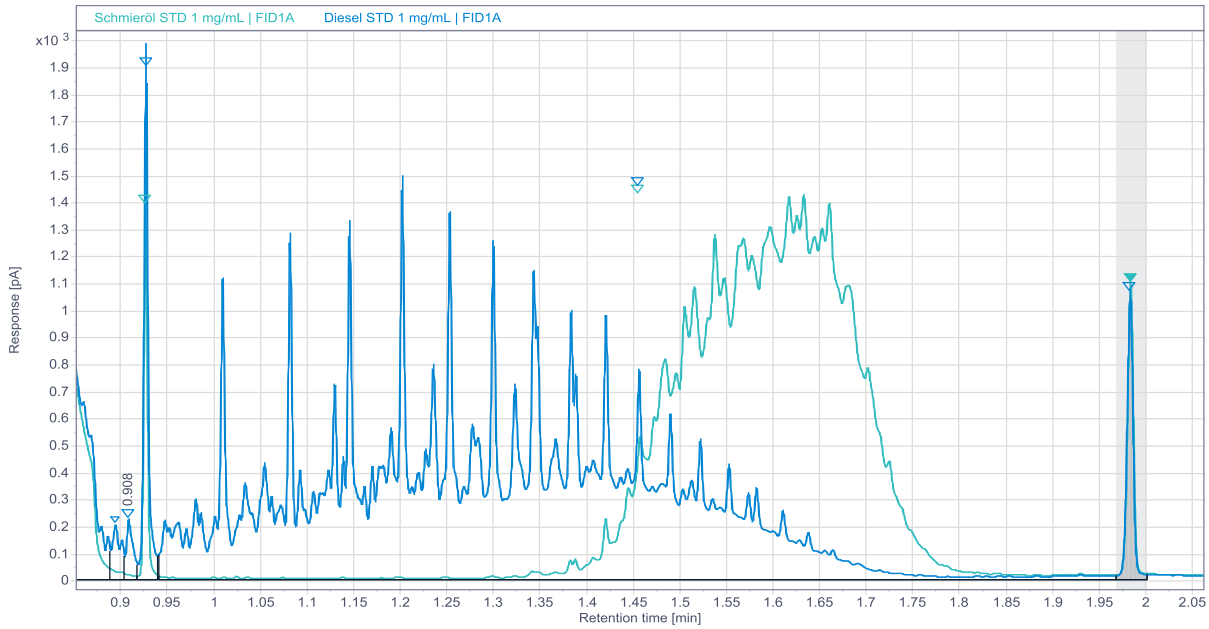

Fig. 5 Overlay of the samples lube oil STD 1 mg/mL (green) and diesel fuel STD 1 mg/mL (blue)

Tab. 2 Results of test measurements

Sample	Inj.	Area	Conc. [mg/mL]	Mean Conc. [mg/mL]	SD (Area)	RSD% (Area)	Setpoint [mg/mL]	Actual Val./Setpoint %
Lube oil-	1	17866.	1.022					
STD	2	17883.	1.023					
1 mg/mL	3	17770.	1.017	1.021	0.003	0.340	1.051	97.128
Diesel-	1	16091.	0.921					
STD	2	16176.	0.926					
1 mg/mL	3	16136.	0.923	0.923	0.002	0.264	0.977	94.521
Referenc	1	9087.9	0.521					
0.5	2	9077.1	0.520					
mg/mL	3	9053.2	0.519	0.520	0.001	0.195	0.529	98.242
Referenc	1	17561.	1.005					
1.0	2	17355.	0.993					
mg/mL	3	17560.	1.005	1.001	0.007	0.678	1.020	98.110
	1	6659.1	0.382					
S1	2	6616.2	0.380					
	3	6658.1	0.382	0.381	0.001	0.367	0.382	99.901
	1	Aberration						
S2	2	34557.	1.975					
	3	34807.	1.990	1.983	0.010	0.509	1.843	107.55

CONCLUSION

The clear time advantage of the method is apparent with consideration of the performed test measurements. Thus, compared to a 30-minute-method with a standard benchtop GC (which needs an additional cooling-down period of at least 5 minutes), four samples instead of one can be measured. A discrimination-free injection and an optimized separation are confirmed by reference to the alkane mix (Fig. 1). Linearity of calibration (Fig. 4) is verified through a concentration range from 0.025 – 5.0 mg/mL under the method parameters. Compared to setpoint concentration, the confirmed hydrocarbon concentration of the samples is within the range from 94 and 107%.

REFERENCES

1. DIN EN ISO 9377-2:2001-07 Water quality - Determination of hydrocarbon oil index - Part 2: Method using solvent extraction and gas chromatography (ISO 9377-2:2000); German version EN ISO 9377-2:2000