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Determination of aromatic hydrocarbon types according to DIN EN 12916:2016

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SUMMARY

In this work aromatic hydrocarbons were determined under normal phase conditions using an AZURA® analytical HPLC plus system with RI detection according to the DIN EN 12916:2016. The instrumental setup and method can be used to determine the content of hydrocarbons in motor diesel fules, which is important for protecting the environment and public health. The standards used for system suitability according to DIN EN 12916:2016 are also part of the calibration used in the IP391(2000)/ASTM D6591 methods but the method settings are slightly divergent.

INTRODUCTION

The content of hydrocarbons in motor diesel fuels affects exhaust emissions and fuel combustion characteristics. These emissions are measured by the cetane number which is an indicator of the combustion speed of diesel fuel and compression needed for ignition [1]. It is important to measure these values due to an incomplete burning, for protecting the environment and public health. The DIN EN 12916:2016 is suitable for the determination of monoaromatic (MAH), diaromatic (DAH) and tri+ – aromatic (T+AH) hydrocarbons in diesel fuels containing up to 30 % (v/v) fatty acid methyl esters (FAME) and petroleum distillates with a boiling range of 150 °C up to 400 °C. The amount of polycyclic aromatic (Poly-AH) hydrocarbons will be calculated as the sum of diaromatic and tri+- aromatic hydrocarbons. [2] Working according to this regulatory also requires a system suitability test to make sure that chosen HPLC hardware as well as the selected column are suitable for the application.

Additional Information

Determination of aromatic hydrocarbon types according to DIN EN 12916:2016

RESULTS

The detailed requirements and calculations for performing the system suitability are described in DIN EN 12916:2016. After achieving all necessary system specifications, a calibration was made. **Fig 1** exemplary shows the separation of system calibration standard 1 (SCS 1) containing cyclohexane, 1-phenyldodecane, o-xylene, hexamethyl benzene, naphthalene, dibenzothiophene and 9-methylanthracene. The calibration standard consists of three different compounds: 1,2-dimethyl benzene, fluorene and phenanthrene. **Tab 1** shows the concentrations for each compound at four different levels. **Fig 2** shows the chromatogram of calibration standard A. The calculation of the number of hydrocarbons in real samples corresponds to retention times of MAH, DAH and T+AH determined in the calibration.

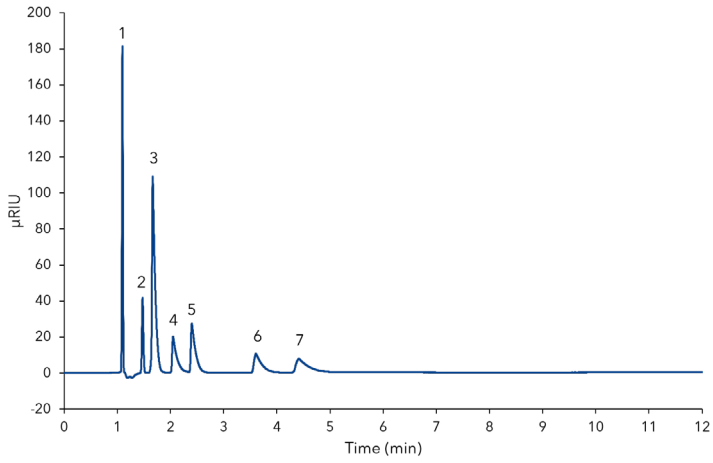


Fig. 1 System calibration standard 1 (SCS 1), 1) cyclohexane, 2) 1-phenyldodecane, 3) 1,2-dimethyl benzene, 4) hexamethyl benzene, 5) naphthalene, 6) dibenzothiophene,7) 9-methylanthracene

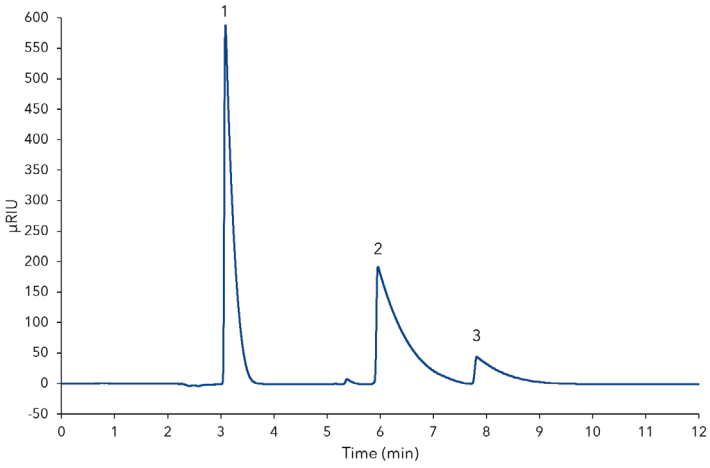


Fig. 2 Calibration standard level A, 1) 1,2-dimethyl benzene, 2) fluorene, 3) phenanthrene

Tab. 1 Calibration concentrations at four different levels

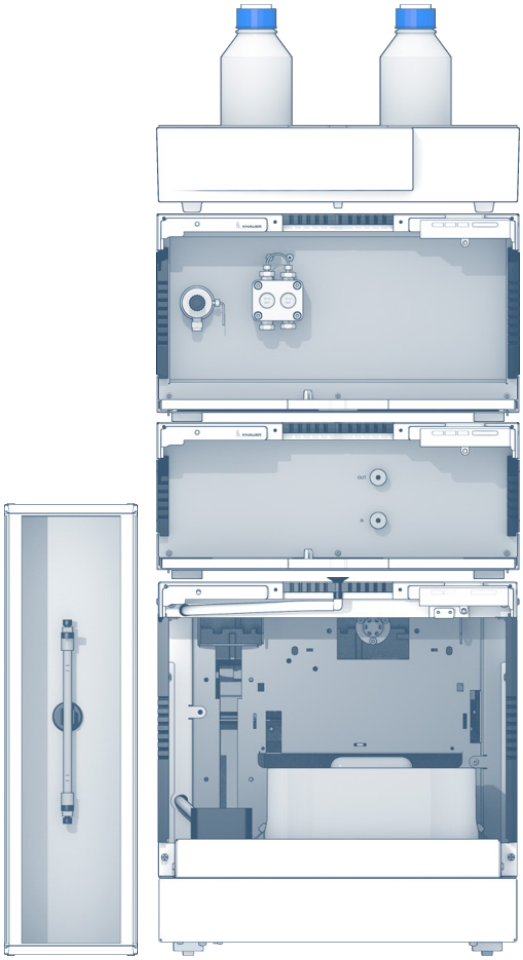
Calibration standard	1,2-Dimethylbenzene (g/100 mL)	Fluorene (g/100 mL)	Phenanthrene (g/100 mL)
A	4.00	2.00	0.40
B	2.00	1.00	0.20
C	0.25	0.25	0.05
D	0.05	0.05	0.01

MATERIALS AND METHODS

An analytical AZURA HPLC system was used for this application. It consisted of an isocratic AZURA P 6.1L pump, suitable for normal phase application. Furthermore, an AZURA RID 2.1L detector, an AZURA AS 6.1L autosampler and an AZURA CT 2.1 column thermostat. The eluent was n-heptane at a flow rate of 1.2 mL/min. The column temperature was set to 25 °C. Detector settings were set to 20 Hz with a time constant of 0.05 s. The column in a dimension 250 x 4 mm ID was filled with Nucleodur 100-5 NH_2 silica.

CONCLUSION

Using this instrumental setup, it is possible to determine mono and di-aromatic hydrocarbons according to the DIN EN 12916:2016.



REFERENCES

[1] http://www.astm.org/Standards/D6591.htm

[2] DIN EN 12916:2016 Petroleum products – Determination of aromatic hydrocarbon types in middle distillates – High performance liquid chromatography method with refractive index detection, German version

ADDITIONAL MATERIALS AND METHODS

Tab. A1 Method parameters

Eluent A	n-heptane		
Gradient	isocratic		
Flow rate	1.2 mL/min		
Run temperature	25°C	Run time	30 min
Injection volume	10 µL	Injection mode	Full loop
Detection wavelength	RI	Data rate	20 Hz
		Time constant	0.05 s

Tab. A2 System configuration & data

Instrument	Description	Article No.
Pump	AZURA P6.1L, isocratic, normal phase	APH30ED
Autosampler	AZURA AS 6.1L	AAA00AA
Detector	AZURA RID 2.1L	ADD31
Column thermostat	AZURA CT 2.1	A05852
Column	Nucleodur 100-5 NH2 for normal phase, 250 x4 mm ID	25DE190NDJ
Software	ClarityChrom 7.4.2 – Workstation, autosampler control included	A1670

RELATED KNAUER APPLICATIONS

VEV0080 – Determination of mono- and polyaromatic hydrocarbons in petrol with AZURA® Analytical HPLC system using RI detection

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