



Certification Report

Certified Reference Material

BAM-K010g

Mixture of diesel oil and lubricating oil

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Summary

This report describes the preparation, characterization and certification of the reference material BAM-K010g.

The certified reference material (CRM) is available as liquid mixture of diesel oil and lubricating oil without solvents. BAM-K010g is intended to be used as calibration standard for the gas chromatographic determination of mineral oil hydrocarbons in water, soil and waste according to the analytical standard procedures ISO 9377-2, ISO 16703 and EN 14039.

The mass ratio of diesel oil to lubricating oil and the mass fraction of the boiling range between n-decane and n-tetracontane ($C_{10} - C_{40}$) have been certified:

Parameter	Value in g g ⁻¹	Uncertainty U¹⁾ in g g ⁻¹
Mass ratio of diesel oil / lubricating oil	1.000028	0.000014
Mass fraction of the boiling range $C_{10} - C_{40}$	0.969	0.015

¹⁾ Estimated expanded uncertainty U with a coverage factor of $k = 2$, corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the expression of uncertainty in measurement (ISO/IEC Guide 98-3:2008).

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List of abbreviations

(if not explained elsewhere)

ANOVA	Analysis of Variance
Bp	Boiling point
CoA	Certificate of Analysis
CRM	Certified reference material
DKD	Deutscher Kalibrierdienst
FAME	Fatty acid methyl esters
FID	Flame ionization detector / detection
GC	Gas chromatography
ID	Inner diameter
IRH	Individual reference hydrocarbon
ISO	International Organization of Standardization
<i>k</i>	coverage factor
KFT	Karl-Fischer titration
MSDS	Material Safety Data Sheet
RHM	Reference hydrocarbon mixture
SOP	Standard operation procedure
TPH	Total petroleum hydrocarbons

1. Introduction

Mineral oils / mineral oil hydrocarbons (synonymously used term: total petroleum hydrocarbons (TPH)) are worldwide produced from crude oil and widely used for several kinds of products including fuels, motor-, heating- and lubricating oils. Large-scale handling, accidents and spills during production, transport and use lead to environmental contaminations, particularly related to water and soil compartments. Due to ecologically harmful and adverse health effects caused by TPH, the surveillance, determination and reduction of mineral oils in environmental matrices is subject to the work of legislative bodies, industry and chemical laboratories.

Reference materials and especially certified reference materials (CRM) are suitable tools to verify the accuracy of analytical measurements. Certified matrix CRMs for TPH determination in soil, sediment and waste are commercially available. The international standard methods for the determination of TPH in water, soil and waste (ISO 9377-2 [1], ISO 16703 [2] and EN 14039 [3]) are based on gas chromatography coupled to flame ionization detection (GC-FID). These standard methods define 'Mineral oil hydrocarbons/TPH' as non-polar substances (not adsorbing on Florisil® after extraction with non-polar solvents) in a boiling range between n-decane (C₁₀H₂₂) and n-tetracontane (C₄₀H₈₂). A mixture of equal amounts of two different oil types (type A, e.g. diesel oil / type B, e.g. lubricating oil) without additives is prescribed as calibration standard according to the standard methods stated above. This calibration standard provides the basis for comparable measurements, and BAM is named in the documents as possible supplier of a calibration standard for TPH analysis.

The first lot of the BAM-K010 calibration standard was produced and certified at BAM in 2000. This report describes the preparation, characterization and certification of lot-no. VIII of the BAM-K010 calibration standard indicated by the extension "g" (BAM-K010g). Certification of reference material BAM-K010g was carried out based on ISO 17034 [4] and the relevant ISO-Guides [5,6].

2. Preparation of BAM-K010g

2.1 Candidate Materials

Diesel oil: Typical diesel fuel for vehicles but without additives and without FAME (fatty acid methyl esters), provided by Shell Global Solutions GmbH (Hamburg, Germany). It contains a complex mixture of hydrocarbons (paraffins, cycloparaffins, aromatic and olefinic hydrocarbons), number of carbon atoms are predominantly in the range C9 to C25. The density is 0.820-0.845 kg/L (15°C) according to the MSDS. The water content was determined by BAM to be 0.0045 % (coulometric KFT).

Lubricating oil (synonymously used terms: Lubricating base stock / lubricant): Additive free paraffinic base oil, group II/II+ (Chevron Neutral Oil 100R) provided by Shell Global Solutions GmbH (Hamburg, Germany). It is a highly refined hydrogen treated crude oil distillate containing light and heavy paraffinic components. The density is 0.8505 kg/L (15°C) according to MSDS and 0.8462 kg/L (25°C) determined by BAM. The water content was determined by BAM to be 0.0027 % (coulometric KFT).

2.2 Weighing process to prepare mineral oil mixture

All steps to prepare BAM-K010g were gravimetrically controlled using a calibrated balance (Sartorius LP6200S, weighing range: 6200 g). Additionally, calibrated weights were used for balance verification covering the relevant mass range. Furthermore, a precision hygro/thermo/barometer (Greisinger electronic GmbH) was used to record atmospheric pressure (1004.3 ± 1.5) mbar, relative humidity (57.3 ± 1.5) % and temperature (25.0 ± 0.1) °C at the beginning and at the end of the preparation process (mean values are given; uncertainties according to manufacturer's specifications). The values were used for buoyancy correction.

The following weighing procedure was applied to prepare the mineral oil mixture BAM-K010g:

- Step a) Balance verification using calibrated weights
- Step b) Weighing the empty can/container for mixing the mineral oils (tare)
- Step c) Weighing lubricating oil into the can
- Step d) Weighing diesel oil into the can
- Step e) Balance verification using calibrated weights

To step a) and e): Balance verification

Balance verification was done using five DKD calibrated weights (Kern & Sohn GmbH, Class E2, 500 g, 2 x 1.000 g and 2 x 2.000 g, Tab. 1) and combinations of them resulting in 12 different weights covering a mass range of 500 g to 6.000 g.

Tab. 1: Calibrated weights used for verification of the calibrated balance

DKD calibrated weights	Conventional mass* (g)	Uncertainty** (g)
1. mass piece 2000 g "1"	1999.9982	0.0060
2. mass piece 2000 g "2"	2000.0011	0.0060
3. mass piece 1000 g "1"	1000.0007	0.0032
4. mass piece 1000 g "2"	1000.0008	0.0032
5. mass piece "500 g"	500.00019	0.00160

*) Value from DKD certificate

*) Twofold max. permitted error limit according to the DKD certificate

Each calibrated weight (or combinations of them) was measured three times before (Tab. 2) and after the mineral oil mixing procedure (Tab. 3). After each removal of the weights, the displayed value of the unloaded balance was recorded.

Tab. 2: Balance verification before preparation of BAM-K010g

DKD calibrated weights	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"
			1 kg "1"	1 kg "2"	1 kg "1"	2 kg "2"	1 kg "1"	1 kg "2"	1 kg "1"	2 kg "2"	1 kg "1"	1 kg "2"
					1 kg "2"		2 kg "2"	2 kg "2"	1 kg "2"	2 kg "1"	2 kg "2"	2 kg "2"
									2 kg "2"		2 kg "1"	2 kg "1"
loaded 1	500.00	999.99	1499.99	1999.99	2499.99	3000.00	3500.00	4000.00	4500.02	5000.01	5500.02	6000.04
unloaded 1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
net 1	500.00	999.99	1499.99	1999.99	2499.99	3000.00	3500.00	4000.00	4500.02	5000.01	5500.02	6000.04
loaded 2	500.00	1000.00	1499.99	2000.00	2500.00	3000.00	3500.00	4000.01	4500.01	5000.01	5500.02	6000.05
unloaded 2	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
net 2	500.00	1000.00	1499.99	2000.00	2500.00	3000.00	3500.00	4000.01	4500.01	5000.01	5500.02	6000.05
loaded 3	500.00	1000.00	1499.99	2000.00	2500.00	2999.99	3500.01	4000.01	4500.01	5000.03	5500.03	6000.03
unloaded 3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
net 3	500.00	1000.00	1499.99	2000.00	2500.00	2999.99	3500.01	4000.01	4500.01	5000.03	5500.03	6000.03
average	500.000	999.997	1499.990	1999.997	2499.997	2999.997	3500.003	4000.007	4500.013	5000.017	5500.023	6000.040
theor. weight	500.0002	1000.0007	1500.0009	2000.0015	2500.0017	3000.0018	3500.0020	4000.0026	4500.0028	5000.0000	5500.0002	6000.0008
u (theor. weight)	0.0016	0.0032	0.0036	0.0045	0.0048	0.0068	0.0070	0.0075	0.0077	0.0091	0.0092	0.0096

Tab. 3: Balance verification after preparation of BAM-K010g

DKD calibrated weights	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"	"500 g"	1 kg "1"
			1 kg "1"	1 kg "2"	1 kg "1"	2 kg "2"	1 kg "1"	1 kg "2"	1 kg "1"	2 kg "2"	1 kg "1"	1 kg "2"
					1 kg "2"		2 kg "2"	2 kg "2"	1 kg "2"	2 kg "1"	2 kg "2"	2 kg "2"
									2 kg "2"		2 kg "1"	2 kg "1"
loaded 1	499.99	1000.00	1499.99	1999.99	2500.00	3000.00	3499.99	4000.00	4500.00	5000.01	5500.00	6000.00
unloaded 1	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.01	0.00	0.00	0.00
net 1	499.99	1000.00	1499.99	1999.99	2500.00	3000.00	3499.99	4000.00	4499.99	5000.01	5500.00	6000.00
loaded 2	500.00	1000.00	1500.00	2000.00	2500.00	2999.99	3500.00	4000.00	4500.01	5000.01	5500.00	5999.99
unloaded 2	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
net 2	500.00	1000.00	1500.00	2000.00	2500.00	2999.99	3500.00	4000.00	4500.01	5000.01	5500.00	5999.99
loaded 3	499.99	1000.00	1500.00	2000.00	2500.00	3000.00	3500.00	4000.01	4500.01	5000.01	5500.00	5999.99
unloaded 3	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
net 3	499.99	1000.00	1500.00	2000.00	2500.00	3000.00	3500.00	4000.01	4500.01	5000.01	5500.00	5999.99
average	499.993	1000.000	1499.997	1999.997	2500.000	2999.997	3499.997	4000.003	4500.003	5000.010	5500.000	5999.993
theor. weight	500.0002	1000.0007	1500.0009	2000.0015	2500.0017	3000.0018	3500.0020	4000.0026	4500.0028	5000.0000	5500.0002	6000.0008
u (theor. weight)	0.0016	0.0032	0.0036	0.0045	0.0048	0.0068	0.0070	0.0075	0.0077	0.0091	0.0092	0.0096

To step b), c) and d): Weighing the empty can (tare), diesel oil and lubricating oil

A 10 L tin can was used to mix both mineral oils because of its low tare mass. By this a maximum amount of BAM-K010g could be prepared only limited by the maximum capacity of the used balance of 6.200 g. To remove possible contaminations, the can was rinsed with toluene and n-heptane before use and dried in a stream of nitrogen. The can was put on the balance and weighed. After its removal the displayed value of the unloaded balance was recorded (Tab. 4). This procedure was repeated three times (Tab. 4). Afterwards, the amount of lubricating oil was calculated in this way, that the maximum limit of the balance will not be exceeded after addition of equal amounts of lubricating oil and diesel oil. Thus, nearly 2.700 g of lubricating oil were filled into the can and weighed three times following the same procedure as described for 'can'. Next, the same mass of diesel oil was added to the can and weighed three times according to the procedure above (Tab. 4). Finally, the can was closed with a screw cap and was shaken manually for 10 minutes to homogenize the mineral oil mixture.

Tab. 4: Weighing the mixing container/can and the mineral oil compounds lubricating oil and diesel oil

Can (Tare)	Unloaded balance	Difference (g)	Can + Lubricant	Unloaded balance	Difference (g)	Can + Lubricant + Diesel oil	Unloaded balance	Difference (g)
792.47	0.00	792.47	3490.73	0.00	3490.73	6189.00	0.00	6189.00
792.47	0.00	792.47	3490.73	0.00	3490.73	6188.99	0.00	6188.99
792.47	0.00	792.47	3490.72	0.00	3490.72	6188.98	0.00	6188.98
Mean		792.47			3490.73			6188.99

2.3 Correction of measured masses

Balance calibration:

Based on the measurement of the calibrated weights before and after the mineral oil mixing process (section 2.2), a balance verification/calibration was performed: measured masses (y) against the conventional (standard) masses (x) according to Tab. 5 using software program *B_LEAST* (v.1.11, BAM).

Tab. 5: Balance verification/calibration (500 – 6000 g) using DKD calibrated weights

Level	Standard (g)		Measurement (g)	
	x	u(x)	y	u(y)
1	500.0002	0.0016	499.9967	0.0054
2	1000.0007	0.0032	999.9983	0.0053
3	1500.0009	0.0036	1499.9933	0.0054
4	2000.0015	0.0045	1999.9967	0.0054
5	2500.0017	0.0048	2499.9983	0.0053
6	3000.0018	0.0068	2999.9967	0.0054
7	3500.0020	0.0070	3500.0000	0.0056
8	4000.0026	0.0075	4000.0050	0.0055
9	4500.0028	0.0077	4500.0083	0.0064
10	5000.0000	0.0091	5000.0133	0.0060
11	5500.0002	0.0092	5500.0117	0.0074
12	6000.0008	0.0096	6000.0167	0.0119

The obtained calibration parameter in Tab. 6 (analysis function), calculated with *B_LEAST* according to ISO 6143 [7], were used for subsequent correction of the measured masses of can, lubricating oil and diesel oil.

Tab. 6: Calibration parameter (analysis function) of the balance according to ISO 6143

intercept (g)	0.0084621825209
slope (g/g)	0.9999970332648
u(intercept)(g)	0.0041994947514
u(slope) (g/g)	0.0000014719473
cov (g)	-0.0000000051659

Correction of determined masses of can, lubricating oil and diesel oil

Based on the obtained calibration parameter (Tab. 6), the corrected masses were calculated according to Eq. 1, and the corrected uncertainties were calculated as stated in Eq. 2:

$$m_{corr} = m_{uncorr} * SL + INT \tag{Eq. 1}$$

- m_{corr} corrected mass
- m_{uncorr} uncorrected (measured) mass
- SL slope acc. to Tab. 6
- INT intercept acc. to Tab. 6

$$u_{corr} = \sqrt{SL^2 * u_{uncorr}^2 + u_{INT}^2 + u_{SL}^2 * m_{uncorr}^2 + 2 * COV * m_{uncorr}^2} \tag{Eq. 2}$$

- u_{corr} combined uncertainty of the corrected mass
- u_{uncorr} combined uncertainty of the uncorrected (measured) mass, see Eq. 3
- u_{INT} uncertainty of the intercept acc. to Tab. 6
- u_{LS} uncertainty of the slope acc. to Tab. 6
- COV covariance acc. to Tab. 6

The combined uncertainty of the uncorrected mass is determined by Eq. 3:

$$u_{uncorr} = \sqrt{SD^2 + u_{Scale}^2} \tag{Eq. 3}$$

- SD Standard deviation of the mean of three measured replicates acc. to Tab. 4
- u_{Scale} Resolution of the used balance (here: 0.005 g, i.e. half of the lowest visible digit 0.01 g)

The uncorrected (measured) and corrected masses are summarized in Tab. 7. The corrected values were used for the calculation of the diesel/lubricating oil mass ratio (Eq. 4).

Tab. 7: Overview of measured (uncorrected) masses and corrected masses after balance verification/calibration

Parameter		Uncorrected value (g)	Corrected value (g)
Tare (Can)	mass	792.4700	792.4761
	combined uncertainty	0.0050	0.0060
Tare + Lubricating oil	mass	3490.7267	3490.7248
	combined uncertainty	0.0060	0.0066
Tare + Lubricating oil + Diesel oil	mass	6188.9900	6188.9801
	combined uncertainty	0.0076	0.0097

2.4 Density determination

In order to perform a buoyancy correction for the mineral oil weighing procedure, the densities of lubricating oil and diesel/lubricating oil mixture are required according to Eq. 4.

Three 100 mL volumetric flasks were used for density determination. At first, the exact volumes of these flasks were determined by filling them with water at 25.0 °C (precision hygro/thermo/barometer; Greisinger electronic GmbH) and precise weighing (balance: Sartorius Cubis 320). Afterwards, the densities of lubricating oil and diesel/lubricating oil mixture were determined using the 'calibrated' flasks (Tab. 8).

Tab. 8: Determination of the densities of lubricating oil and diesel/lubricating oil

Parameter	Flask 1	Flask 2	Flask 3	Mean (kg/m ³)	SD (kg/m ³)
Water gross (g)	162.34700	162.46970	163.22890		
Flask tare (g)	62.87120	63.05060	63.57950		
Water net (g)	99.47580	99.41910	99.64940		
corr. Flask volume* (mL)	99.77032	99.71345	99.94444		
Lubricant gross (g)	147.27700	147.47940	148.12140		
Flask tare (g)	62.87150	63.05060	63.58060		
Lubricant net (g)	84.40550	84.42880	84.54080		
Lubricant density** (kg/m ³)	845.99807	846.71422	845.87800	846.197	0.261
K010g gross (g)	146.49650	146.68330	147.36560		
Flask tare (g)	62.87120	63.05050	63.58030		
K010g net (g)	83.62530	83.63280	83.78530		
K010g density** (kg/m ³)	838.17811	838.73135	838.31880	838.409	0.166

*) Corrected flask volume calculated using the density of water at 25.0°C: 0.997048 g/mL (<https://www.internetchemie.info/chemie-lexikon/daten/w/wasser-dichtetabelle.php>)

**) Density calculated by using the corrected flask volume

2.5 Calculation of the diesel/lubricating oil mass ratio

The ratio (D/S) of diesel oil mass (m_D) to lubricating oil mass (m_S) including buoyancy correction is based on Eq. 4 considering the determined values given in Tab. 9.

$$D/S = \frac{m_D}{m_S} = \frac{m_{DS} - m_S}{m_S} = \frac{m_{DS}}{m_S} - 1 = \frac{m_{DS}^w \cdot \left(\frac{1 - \frac{\rho_L}{8000}}{1 - \frac{\rho_L}{\rho_{DS}}} \right)}{m_S^w \cdot \left(\frac{1 - \frac{\rho_L}{8000}}{1 - \frac{\rho_L}{\rho_S}} \right)} - 1 = \frac{m_{DS}^w \cdot \left(1 - \frac{\rho_L}{\rho_S} \right)}{m_S^w \cdot \left(1 - \frac{\rho_L}{\rho_{DS}} \right)} - 1 = \frac{(m_{Brutto2}^w - m_{Tara}^w) \cdot \left(1 - \frac{0,348444 \cdot p - (0,00252 \cdot T - 0,020582) \cdot h}{(273,15 + T) \cdot \rho_S} \right)}{(m_{Brutto1}^w - m_{Tara}^w) \cdot \left(1 - \frac{0,348444 \cdot p - (0,00252 \cdot T - 0,020582) \cdot h}{(273,15 + T) \cdot \rho_{DS}} \right)} - 1$$

Eq. 4: Equation to calculate the mass ratio of diesel oil to lubricating oil of BAM-K010g

Tab. 9: Values used to calculate the mass ratio of diesel oil to lubricating oil of BAM-K010g

Symbol	Description of the parameter	Unit	Value	Uncertainty
m_{Tara}	mass tare (10 L can)	g	792.4761	0.0060
$m_{Brutto,1}$	mass (tare + lubricating oil)	g	3490.7248	0.0066
$m_{Brutto,2}$	mass (tare + lubricating oil + diesel)	g	6188.9801	0.0097
p	atmospheric pressure	mbar	1004.3	1.5
T	temperature	°C	25.0	0.1
h	relative humidity	%	57.3	1.5
ρ_S	density lubricating oil	kg/m ³	846.197	0.261
ρ_{ds}	density diesel/lubricating oil mixture	kg/m ³	838.409	0.166

The calculated and certified value of the diesel/lubricating oil mass ratio and its uncertainty are summarized in Tab. 10:

Tab. 10: Calculated and certified value of the diesel/lubricating oil mass ratio and its uncertainty

Parameter	Calculated values	Certified values
Mass ratio of diesel/lubricating oil (g g ⁻¹)	1.0000281	1.000028
Uncertainty ¹ of the mass ratio (g g ⁻¹)	0.0000131	0.000014

¹ Estimated expanded uncertainty U with a coverage factor of $k = 2$, corresponding to a level of confidence of approx. 95 %, as defined in the Guide to the expression of uncertainty in measurement (ISO/IEC Guide 98-3:2008, [8]).

According to DIN 1333 [9] the number of digits of the mass ratio is determined by the expanded uncertainty. Because the first digit $\neq 0$ of U is “1” (0.0000131), the relevant rounding position is 0.000013(1). Furthermore, the expanded uncertainty has to be rounded up to 0.000014 according to DIN 1333 (section 6.1). The mass ratio has the same number of decimal places as the uncertainty and was rounded to 1.000028.

2.6 Bottling and storage

BAM-K010g was bottled in 3 mL amber glass vials sealed with crimp caps and PTFE inlets. 3005 units of BAM-K010g were produced, each unit contains at least 2.0 mL (filled by using an automatic pipette, target volume: 2.1 mL). Each BAM-K010g vial was additionally packed into a cardboard box with a foam insert. Besides general information, vials and cardboard boxes were undoubtedly labelled with specific vial-numbers. The whole batch is stored at room temperature at a dark place.

3. Homogeneity testing

A satisfactory level of homogeneity was expected based on *i*) unlimited miscibility of diesel oil and lubricating oil and *ii*) 20 years of experience (7 lots of BAM-K010 already produced and characterized). It should be kept in mind that there is no analytical method available that is precise enough to detect minimal inhomogeneities of diesel/lubricant on the same high precision level of weighing. Therefore, GC-FID analysis was applied for two reasons, *i*) to investigate the 'between unit homogeneity' to detect serious inhomogeneities and *ii*) GC-FID is the mandatory standard method to analyze TPHs in environmental matrices. For reasons mentioned above no contribution from homogeneity testing was included in the overall uncertainty budget.

For quantitative demonstration, 25 units of the whole BAM-K010g lot (3005 units) were selected equally distributed (after each 120th vial). From each selected BAM-K010g unit three independent dilutions in n-heptane were prepared (37 mg/g) and directly analyzed by GC-FID (Tab. 11) under repeatability conditions in a random order.

Tab. 11: Gas chromatography (GC-FID) conditions used for homogeneity testing of BAM-K010g

GC conditions	
Instrument	Agilent 7890
Column	BPX-5 (15 m x 0.32 mm ID x 1 µm film) (SGE)
Pre-Column	Fused silica (5 m x 0.53 mm ID)
Carrier gas	Helium 5.0; flow rate: 3 mL min ⁻¹
Oven program	60 °C (5 min isotherm) → 360 °C (5 min isotherm)
Heating rate	20 K min ⁻¹
Injection volume	2 µL (on-column)
FID conditions	
FID temperature	370 °C
Flame gases	Synthetic air (400 mL min ⁻¹), Hydrogen 5.0 (40 mL min ⁻¹)
Make-up gas	Helium 5.0; flow rate: 45 mL min ⁻¹
Software	
	Agilent OpenLAB

For the assessment of homogeneity, retention time intervals were defined representing the diesel oil and the lubricating oil 'compound' (Fig. 1). The retention times were selected based on aspects of good recognition to facilitate the integration process (*Note:* An exact differentiation between diesel oil and lubricating oil is not possible because both products show partly overlapping signals in GC-FID. Thus, a peak area ratio Diesel/Lubricant of 1:1 cannot be expected).

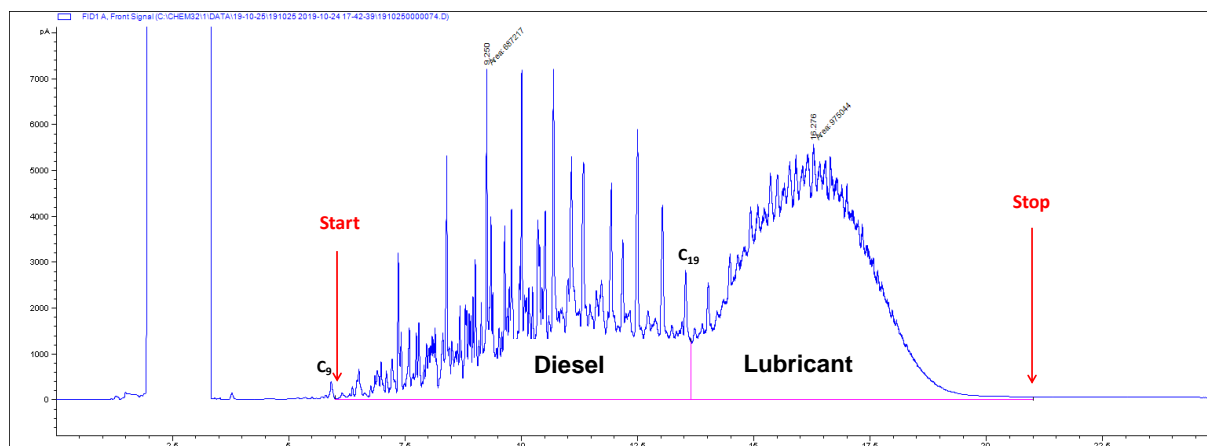


Fig. 1: GC-FID analysis of BAM-K010g (37 mg/g in n-heptane) for homogeneity testing

Results of the GC-FID measurements and the subsequent ANOVA evaluation are displayed in Fig. 2 and Tab. 12, respectively.

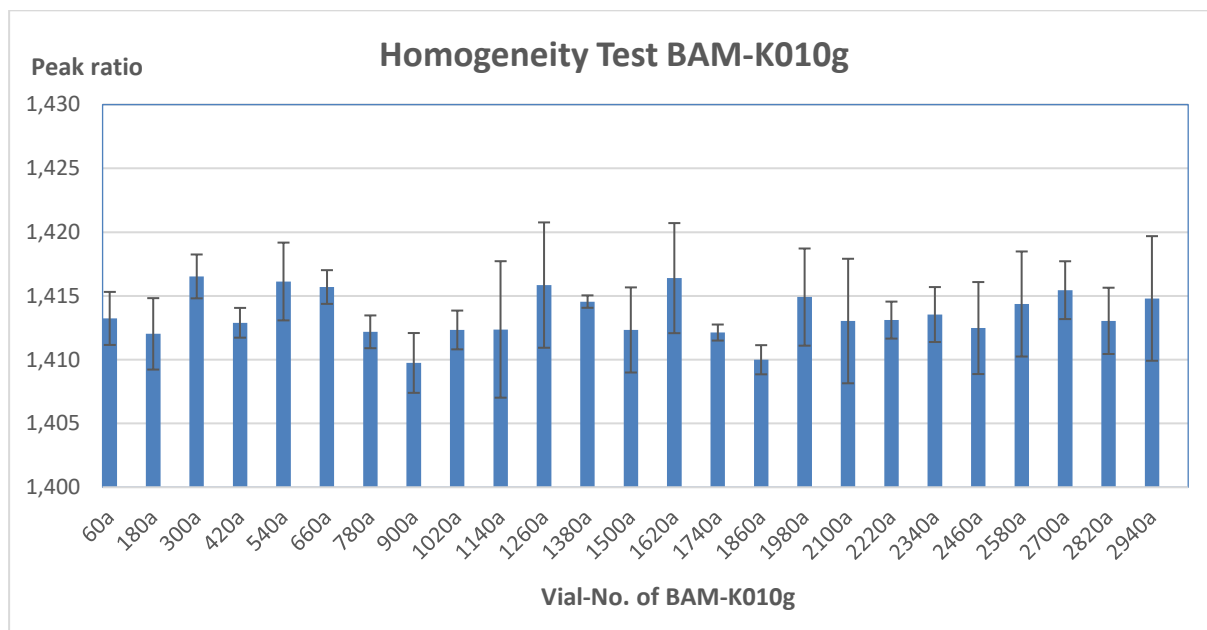


Fig. 2: GC-FID peak area ratio “Lubricating oil / Diesel oil” from BAM-K010g homogeneity testing; error bars represent the standard deviation (n=3)

Tab. 12: One-factorial ANOVA results from homogeneity measurements of BAM-K010g ($\alpha = 0.05$)

Source of variability	sum of squares (SS)	degrees of freedom (df)	mean squares (MS)	test statistic (F)	P value	critical value
differences between groups	0.00023395	24	9.74783E-06	1.0314918	0.4491099	1.7416199
differences within groups	0.00046306	49	9.45023E-06			
total	0.00069701	73				

The test criterion (F) is less than the critical value, indicating that there is no significant inhomogeneity of the batch (significance level α , $\alpha = 0.05$).

4. Stability monitoring

Results from the previous seven lots of BAM-K010 (about 20 years since 2000) have shown that the neat diesel/lubricating oil mixture (1:1, w/w) is stable stored at room temperature in a dark place. The following experiments have been conducted demonstrating satisfactory stability:

- a) Exact match of successive batches: All previous 7 lots of BAM-K010g were prepared in such a way that an exact match of the diesel/lubricant mass ratio was achieved. The large reserves of diesel and lubricating oil – not yet used for mixture preparation – were stored at room temperature in a dark place. That means, identical weights of diesel and lubricating oil (from stored reserves) led to identical BAM-K010 product properties over the last 20 years. No chemical or physical alteration effects (chemical: GC-chromatogram, FID-response; physical: color, clearness) could be observed.
- b) A short-term (6 weeks) and long-term (12 months) stability study at different temperatures (-20°C, 4°C, RT, 40°C and 60°C) was performed for BAM-K010b (3rd lot) in 2005. No degradation or alteration was observed.

- c) Tightness tests to detect evaporation losses: The previous batches of BAM-K010 were filled in different containers: Amber vials containing 6 mL with screw caps / PTFE inlets, amber glass ampoules containing 1.5 mL and, now for BAM-K010g, amber glass vials containing 2.0 mL with crimp caps and PTFE inlets. Tightness tests were done for the 6 mL vials with simple screw caps over a period of 3.5 months using originally sealed vials and vials after opening. Only minimal mass losses of < 0.01% were registered, which were in the range of the weighing precision. For the current lot BAM-K010g, tightness tests over 3, 6, 9 and 12 months at room temperature have been started in November 2019 (10 vials standing upright, 10 vials stored horizontally, 10 vials upside down). In addition to the weighing monitoring, these differently stored vials will be analyzed after 12 months by GC-FID to investigate chemical changes and regarding the lubricant/diesel peak area ratio (see section 4 "Homogeneity testing"). The latter test will be repeated regularly in the frame of a post-certification monitoring.

5. Mass fraction of the boiling range between n-decane (C₁₀) and n-tetracontane (C₄₀)

5.1 Principle and general remarks

The international standard methods for the determination of TPH in water, soil and waste (ISO 9377-2 [1], ISO 16703 [2] and EN 14039 [3]) are based on gas chromatography coupled to flame ionization detection (GC-FID). According to these standard methods, the TPHs are quantified in a defined boiling range between n-decane, C₁₀H₂₂ (C₁₀) and n-tetracontane, C₄₀H₈₂ (C₄₀). Therefore, the C₁₀-C₄₀ mass fraction is an important property value of the calibration standard BAM-K010g.

The determination of BAM-K010g's mass fraction of the boiling range between C₁₀ and C₄₀ is based on the BAM-internal SOP BAM-1.7-PV012 [10] describing the direct comparison of the FID-response of the mineral oil mixture with the FID-response of a reference hydrocarbon mixture (RHM). The RHM is consisting of several individual reference hydrocarbons (IRH) which should have the following properties:

- structurally/chemically defined hydrocarbons,
- different types of hydrocarbons (e.g., n-, cyclic- and branched aliphatic, mono-/di-cyclic aromatic compounds) which can also occur in natural mineral oils,
- high and known purities,
- boiling points within the range of C₁₀ to C₄₀,
- sufficient number of IRHs to simulate a representative mineral oil mixture

The aim is to combine different IRHs resulting in a final mixture (RHM) that has similar properties as the natural mineral oil mixture (BAM-K010g). One of these similar properties is the carbon (C) content. This criterion results from the fact that the FID response is directly proportional to the C-content of a hydrocarbon. However, because the actual C-content of a real-world mineral oil mixture is always unknown, the RHM model with an average C-content can only be a best-praxis approach.

In addition to achieve comparable FID responses of BAM-K010g and RHM, gas chromatographic discrimination effects should be avoided. Therefore, measurements should always be performed with a non-discriminatory GC-FID system (e.g. on-column injection), also prescribed in [1, 2, 3].

To separate compounds according to their boiling points, non-polar GC columns are commonly used. According to the international standard procedures mentioned above, GC columns consisting of polydimethylsiloxane (95% to 100%) are typically used. To avoid systematic errors/shifts, GC-FID measurements to determine BAM-K010g's mass fraction are performed on two non-polar GC columns with slightly different polarities. These (low bleeding) GC columns must be applicable up to high temperatures in order to analyse compounds until a boiling point of C₄₀ (525°C).

5.2 Reference hydrocarbon mixture (RHM)

Ten individual reference hydrocarbons (IRH), analytical standards with high purities supplied by Sigma-Aldrich, were selected for the preparation of the RHM based to the five criteria given above (5.1). Property values of the used IRH are summarised in Tab. 13.

Tab. 13: Individual reference hydrocarbons (IRH) for preparation of the reference hydrocarbon mixture (RHM)

IRH	Code	Sum formula	Bp (°C)	Provided purity (%)	Calculated Purity (%)	u_{pur} (%)
n-Pentylbenzene	PB	C ₁₁ H ₁₆	209.5	≥ 99.0	99.50	0.29
1,2,3,4-Tetrahydronaphthalene	THN	C ₁₀ H ₁₂	212.5	≥ 99.5	99.75	0.14
Tridecane	TD	C ₁₃ H ₂₈	234.0	≥ 99.5	99.75	0.14
Bicyclohexyl	BCH	C ₁₂ H ₂₂	239.7	≥ 99.0	99.50	0.29
Biphenyl	BP	C ₁₂ H ₁₀	254.8	≥ 99.5	99.75	0.14
2-Ethyl-naphthalene	EN	C ₁₂ H ₁₂	257.5	≥ 99.0	99.50	0.29
1-Phenyl-dodecane	PDD	C ₁₈ H ₃₀	331.0	≥ 99.5	99.75	0.14
1-Phenyl-tetradecane	PTD	C ₂₀ H ₃₄	357.3	≥ 99.5	99.75	0.14
Docosane	DC	C ₂₂ H ₄₆	369.0	≥ 99.5	99.75	0.14
Squalane	SQA	C ₃₀ H ₆₂	416.5	97.20*	97.20	0.25**

*) The 'provided purity' of squalane (97.2 %) is a certified value of the supplier and set to 'calculated purity'

***) The uncertainty of squalane's purity (0.25 %) was taken from supplier's certificate.

Tab. 13 displays the calculated purities (pur_{cal}) of the IRH determined using Eq. 5 based on a rectangular distribution. The pur_{cal} values were used to determine the concentration of the RHM solution (Tab. 14).

$$pur_{cal} = pur_{pro} + \left(\frac{100 - pur_{pro}}{2} \right) = pur_{pro} + HW_{rec} \quad \text{Eq. 5}$$

pur_{cal} calculated purity (%)
 pur_{pro} provided purity (%) by the suppliers
 HW_{rec} half width of the rectangular distribution

The corresponding uncertainty of the 'calculated purity' (u_{pur}) was determined using Eq. 6 based on the uncertainty calculation of a rectangular distribution:

$$u_{pur} = \frac{HW_{rec}}{\sqrt{3}} \quad \text{Eq. 6}$$

At first, separate solutions of the IRHs were gravimetrically prepared in n-heptane with concentrations of about 4.4 mg/g for each compound considering the purity (pur_{cal}) of each IRH according to Tab. 13. Before further mixing, the purities of the IRH provided by the suppliers (Tab. 13) were checked by GC-FID (BPX-5 column). Because these control measurements confirmed supplier's purities, the IRHs were taken for subsequent processing. Equal volumes of each IRH solution (10 mL) were combined in a 100 mL flask (Tab. 14) - all steps were gravimetrically controlled.

Tab. 14: Preparation of the reference hydrocarbon mixture (RHM) from 10 IRH solutions

Individual reference hydrocarbon (IRH)	IRH solution (g)	IRH absolute (mg)	Uncertainty IRH (mg)	Conc. IRH (mg/g)	Uncertainty IRH (mg/g)
PB	6.8181	30.8097	0.0889	0.4518	0.0013
THN	6.8145	30.3984	0.0439	0.4458	0.0006
TD	6.8129	30.2574	0.0437	0.4437	0.0006
BCH	6.8182	30.2660	0.0874	0.4439	0.0013
BP	6.8292	29.8941	0.0431	0.4384	0.0006
EN	6.8217	30.3611	0.0876	0.4452	0.0013
PDD	6.8105	30.0166	0.0433	0.4402	0.0006
PTD	6.8340	30.1202	0.0435	0.4417	0.0006
DC	6.8105	30.2764	0.0437	0.4440	0.0006
SQA	6.8198	29.1378	0.0728	0.4273	0.0011
Total (RHM):	68.1894	301.5377	0.1998	4.4221	0.0029

The concentrations of the 10 IRH in the RHM were between 0.42 to 0.45 mg/g (Tab. 14) resulting in a total of all compounds of 4.4221 mg/g ($\Rightarrow c_{RHM}$ acc. to Eq. 8).

5.3 Preparation of BAM-K010g test solutions

Five units of BAM-K010g were selected for the determination of the C₁₀-C₄₀ mass fraction (equally distributed over the whole batch of 3005 units: No. 840, 1440, 1920, 2520 and 3005). Five test solutions were gravimetrically prepared from each selected BAM-K010g unit in n-heptane (concentration about 24 mg/g) resulting in 5 x 5 = 25 measurement solutions ($\Rightarrow c_{MKW}^{total}$ acc. to Eq. 8). A BAM-K010g concentration of about 24 mg/g was defined based on GC-FID pre-tests including the RHM solution. The pre-tests aimed to find out comparable FID signals between BAM-K010g solution and RHM solution to ensure equal sensitivity/response.

5.4 GC-FID measurements and integration

All measurements were performed using an Agilent 7890 gas chromatographic system equipped with a flame ionization detector (FID). The Agilent OpenLab software was used for measurement and system control as well as for data evaluation. Two separate measurement series were recorded using column A and B (Tab. 15).

Tab. 15: GC-FID conditions for measurements to determine the C₁₀-C₄₀ mass fraction of BAM-K010g

	Column A	Column B
GC capillary column	BPX-5: 12 m x 0.32 mm x 1 μ m (SGE)	BP-1: 12 m x 0.32 mm x 1 μ m (SGE)
Internal column-no.	#84	#85
Guard column	Deactivated fused silica: 5 m x 0.53 mm (Agilent)	
On-column injection	2 μ L	
Carrier gas: N ₂ (5.0)	3 mL/min	
FID gases	Synthetic air: 400 mL/min; Hydrogen (5.0): 40 mL/min; Make-up gas N ₂ (5.0): 40 mL/min	
FID temperature	370°C	340°C
Oven program	60°C (5 min) \rightarrow 360°C (5min)	60°C (5 min) \rightarrow 330°C (5min)
Heating rate:	10 °C/min	15 °C/min

The following solutions were analysed in randomized order on both GC-FID systems (column A and B):

- 2 vials of n-alkanes mix in n-heptane for retention time setting of C₁₀ and C₄₀
- 25 BAM-K010g vials analysed in duplicate (50 injections)
- 6 RHM vials analysed in triplicate (18 injections)
- 10 vials of n-heptane (blanks) analysed in duplicate (20 injections)

At first, a n-alkanes mix containing 31 homologues between C₁₀ and C₄₀ was analysed to define the start/stop retention times for C₁₀ (start: at the end of the n-decane peak) and C₄₀ (stop: at the beginning of the n-tetracontane peak), see Fig. 3. These retention time marks were used to integrate all samples analysed with the same conditions.

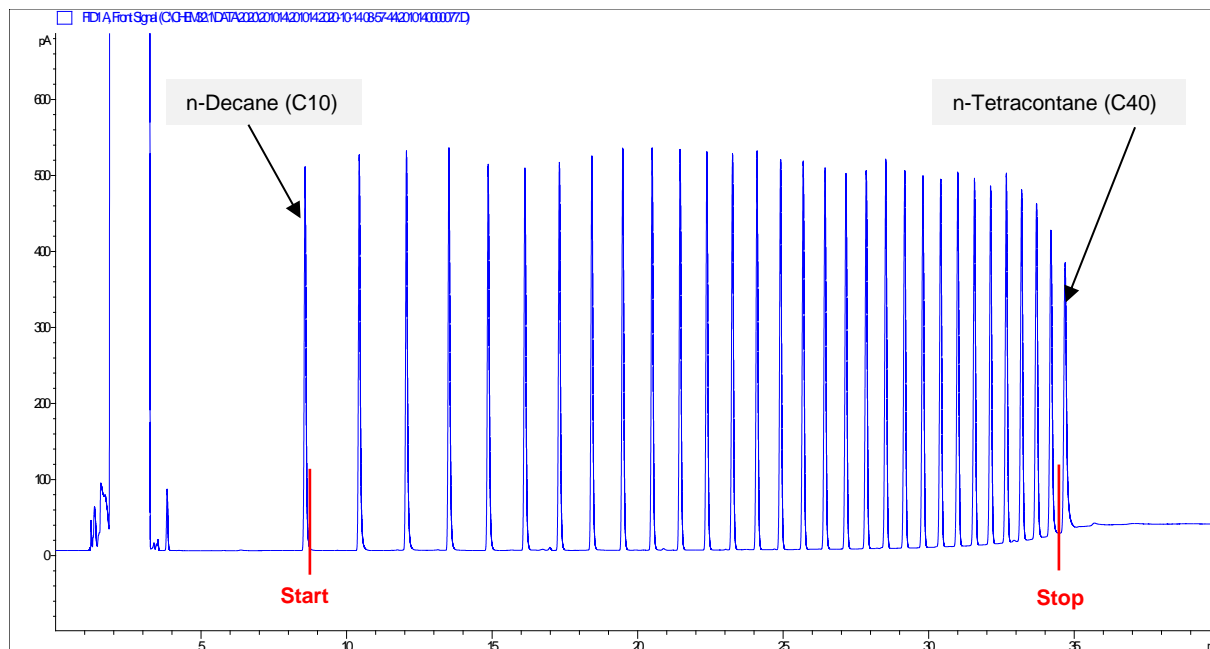


Fig. 3: GC-FID chromatogram of a n-alkanes mix containing 31 homologues between C₁₀ and C₄₀, analyzed on a BPX-5 column to define the start/stop retention times

Afterwards, the chromatograms of BAM-K010g and the pure solvent n-heptane (blank) were integrated between the start/stop marks determined before (Fig. 4, 5). The baseline was set at the level before the solvent peak acc. to the integration procedures of the standard methods [1, 2, 3].

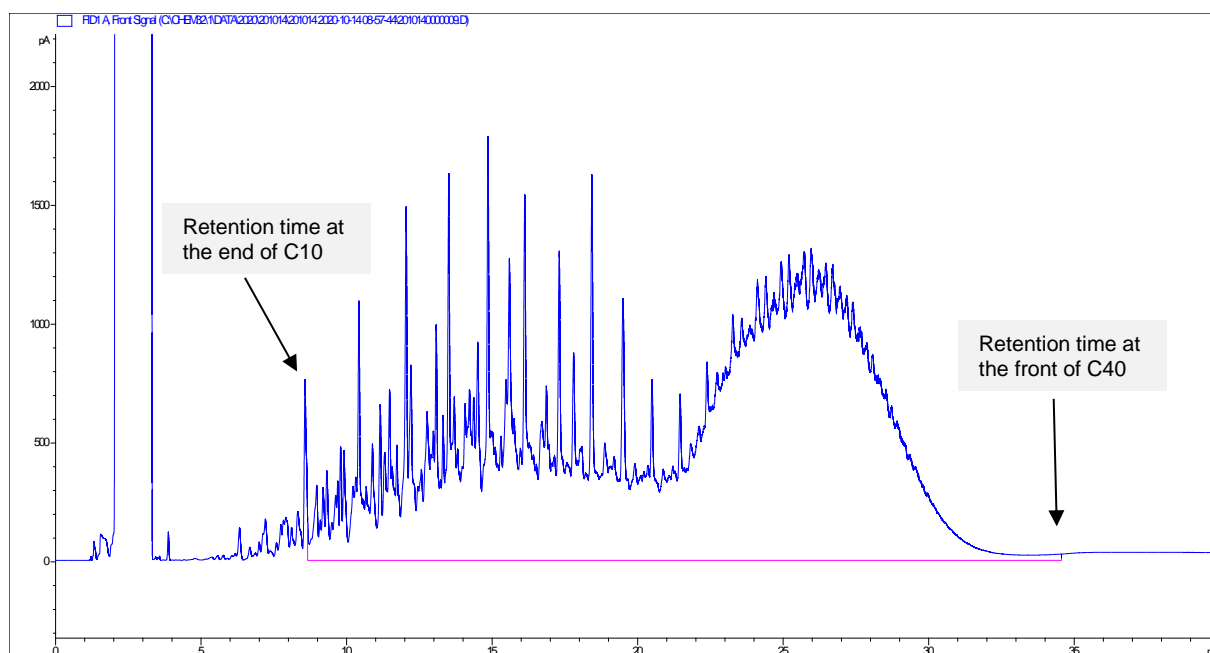


Fig. 4: Integrated GC-FID chromatogram of BAM-K010g (No. 1440) between the retention times of C₁₀ and C₄₀ (analyzed on a BPX-5 column)



Fig. 5: Integrated GC-FID chromatogram of n-heptane (blank, pure solvent used to prepare BAM-K010g dilutions) between the retention times of C₁₀ and C₄₀ (analyzed on a BPX-5 column)

The RHM chromatograms were not integrated over the broad range between C₁₀-C₄₀ to avoid the integration of substances' impurities. Instead, all 10 IRHs in the RHM-chromatogram were separately integrated (Fig. 6).

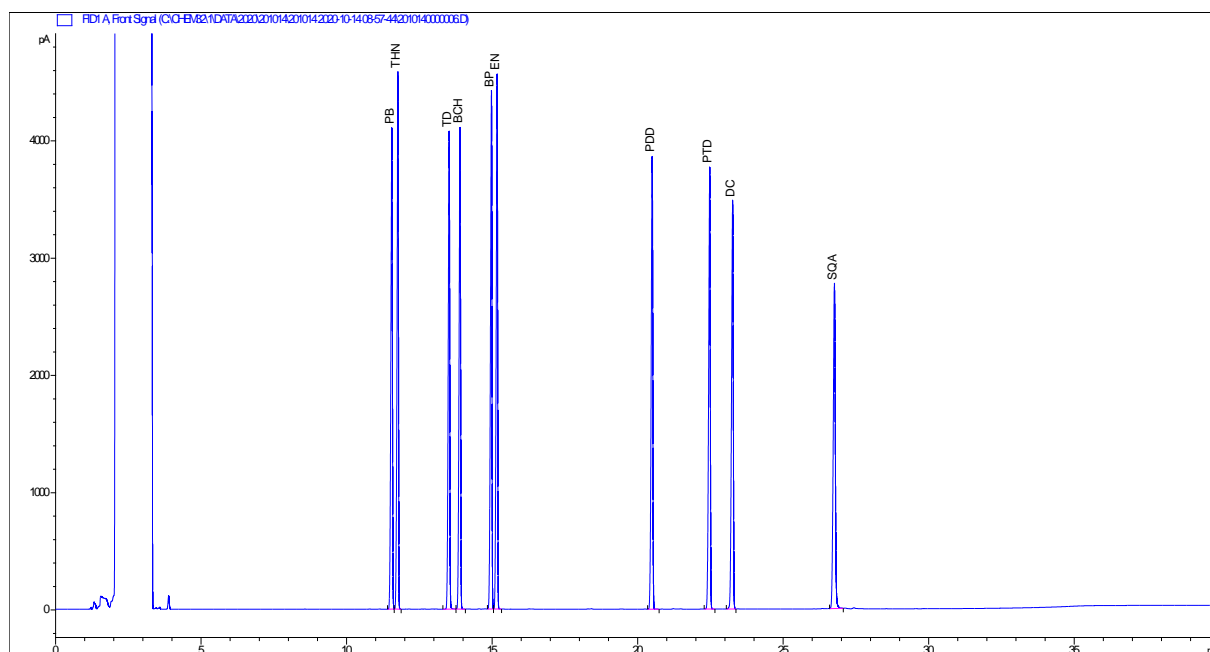


Fig. 6: GC-FID chromatogram of the reference hydrocarbon mixture (RHM) analyzed on a BPX-5 column; all 10 individual reference hydrocarbons (IRH) are integrated separately

5.5 Calculation of the C₁₀-C₄₀ mass fraction

As described above (5.1), the determination of the C₁₀-C₄₀ mass fraction is based on the general principal of equal FID-responses of BAM-K010g ($\Rightarrow R_{MKW}$) and reference hydrocarbon mixture ($\Rightarrow R_{RHM}$) acc. to Eq. 7:

$$R_{MKW} = R_{RHM} \quad \text{Eq. 7}$$

The FID response is defined as the ratio of the detector signal (peak area) to the injected mass of a substance or mixture. Because the injected mass (in pico- to nanogram range) is unusual to handle, the concentrations of RHM- and BAM-K010g solutions are used in the following equations (more precisely the mass fraction (mg/g) of the solutions), Eq. 8. This approach requires a constant injection volume (2 μ L) and the same type of solvent for all samples to be compared (n-heptane).

$$R_{MKW} = R_{RHM} = \frac{A_{MKW}^{total}}{C_{MKW}^{total}} = \frac{A_{RHM}}{C_{RHM}} \quad \text{Eq. 8}$$

A_{MKW}^{total}	total peak area of a BAM-K010g solution after blank correction (n-heptane), <i>not determined</i>
C_{MKW}^{total}	concentration of a BAM-K010g solution in n-heptane based on gravimetric preparation (representing a defined mass of BAM-K010g injected into the GC-FID system)
A_{RHM}	mean value of the peak areas of 18 RHM analyses (sum of 10 IRH for each analysis)
C_{RHM}	concentration of the RHM solution, i.e. sum of 10 IRH concentrations in RHM solution (representing a defined mass of 10 IRH injected into the GC-FID system)

Because BAM-K010g is a complex mixture of several hundred hydrocarbons (representing different types of hydrocarbons) it can be presumed that the FID response of the C₁₀-C₄₀ mass fraction is comparable to the FID response of all compounds of BAM-K010g (Eq. 9):

$$\frac{A_{MKW}^{total}}{C_{MKW}^{total}} = \frac{A_{MKW}^{C10-C40}}{C_{MKW}^{C10-C40}} \quad \text{Eq. 9}$$

$A_{MKW}^{C10-C40}$	peak area of BAM-K010g between C ₁₀ and C ₄₀ after blank correction (n-heptane)
$C_{MKW}^{C10-C40}$	concentration of a BAM-K010g solution for the C ₁₀ -C ₄₀ fraction (representing a defined mass of BAM-K010g's C ₁₀ -C ₄₀ fraction injected into the GC-FID system)

Based on Eq. 8 and Eq. 9 the concentration of the C₁₀-C₄₀ fraction of a BAM-K010g solution can be determined using Eq. 10:

$$C_{MKW}^{C10-C40} = \frac{A_{MKW}^{C10-C40}}{A_{RHM}} * C_{RHM} \quad \text{Eq. 10}$$

The final mass fraction of the C₁₀-C₄₀ boiling range is defined as the ratio of the calculated C₁₀-C₄₀ concentration of BAM-K010g to the total concentration of BAM-K010g based on gravimetric preparation (Eq. 11). As the concentrations represent the injected masses (see explanation above), $w_{MKW}^{C_{10}-C_{40}}$ is the C₁₀-C₄₀ mass fraction (g/g) of BAM-K010g.

$$w_{MKW}^{C_{10}-C_{40}} = \frac{c_{MKW}^{C_{10}-C_{40}}}{c_{MKW}^{total}} = \frac{m_{MKW}^{C_{10}-C_{40}}}{m_{MKW}^{total}} \tag{Eq. 11}$$

$m_{MKW}^{C_{10}-C_{40}}$ mass of BAM-K010g between C₁₀ and C₄₀ (experimentally determined by GC-FID)

m_{MKW}^{total} total mass of BAM-K010g (from gravimetric preparation)

Table 16 contains the C₁₀-C₄₀ mass fractions of BAM-K010g resulting from analysis of 5 units (25 sub-units) on two different GC-columns.

Tab. 16: C₁₀-C₄₀ mass fraction of BAM-K010g; each value represents the mean of 10 results (5 sub-units per unit, two results per sub-unit)

GC-column	C ₁₀ -C ₄₀ mass fraction (g/g)					GC-Column mean
	Unit 840	Unit 1440	Unit 1920	Unit 2520	Unit 3005	
A (BPX-5)	0.9662	0.9691	0.9666	0.9646	0.9651	0.9663
B (BP-1)	0.9829	0.9774	0.9646	0.9642	0.9664	0.9711
Mean of unit	0.9746	0.9733	0.9656	0.9644	0.9658	
Mean (exact)	0.9687					
Mean (rounded)	0.969					

The mean (exact) is the average of the two GC-column means. The rounded mean value from Tab. 16 being 0.969 g/g was taken as the assigned (= certified) C₁₀-C₄₀ mass fraction of BAM-K010g.

5.6 Uncertainty of the C₁₀-C₄₀ mass fraction

The terms contributing to the uncertainty of the C₁₀-C₄₀ mass fraction can be seen from Eq. 12 which results from inserting Eq. 10 into Eq. 11:

$$w_{MKW}^{C_{10}-C_{40}} = \frac{A_{MKW}^{C_{10}-C_{40}} * c_{RHM}}{A_{RHM} * c_{MKW}^{total}} \tag{Eq. 12}$$

Taking into account that the concentration of the RHM solution (c_{RHM}) is influenced by both, the weighing process itself and the IRH-purities (see Tab. 13/14 and Eq. 5/6), the combined uncertainty of the C₁₀-C₄₀ mass fraction determined by a specific GC-column (u_w^{col}) is calculated based on the data acc. to Eq. 13. The results for both GC-columns are displayed in Tab. 17 and 18.

$$u_w^{col} = w_{MKW_col}^{C_{10}-C_{40}} \sqrt{(u_{c_MKW}^{rel})^2 + (u_{c_RHM}^{rel})^2 + (u_{pur}^{rel})^2 + (u_{A_RHM}^{rel})^2 + (u_{A_MKW}^{rel})^2} \tag{Eq. 13}$$

$w_{MKW_col}^{C_{10}-C_{40}}$ C₁₀-C₄₀ mass fraction of BAM-K010g determined on GC-column A or B, respectively

Tab. 17: Uncertainty of the C₁₀-C₄₀ mass fraction of BAM-K010g (**column A: BPX-5**) based on Eq. 13

Uncertainty contribution		u_{rel}
Uncertainty of BAM-K010g concentration (gravimetric preparation) based on uncertainty estimations from weighing BAM-K010g and solvent (0.2 %)	$u_{c_MKW}^{rel}$	0.0020
Uncertainty of RHM concentration (gravimetric preparation) based on uncertainty estimations from weighing 10 IRHs and solvent (0.4 %)	$u_{c_RHM}^{rel}$	0.0040
Uncertainty of RHM purity (combined uncertainty of 10 IRH purity uncertainties acc. to Eq. 6)	u_{pur}^{rel}	0.0020
Uncertainty of RHM peak area (standard deviation of the mean of 18 RHM analyses); normalised to the RHM concentration	$u_{A_RHM}^{rel}$	0.0007
Uncertainty of BAM-K010g peak area (standard deviation of the mean of 50 BAM-K010g analyses); normalised to the concentration of specific BAM-K010g solution*	$u_{A_MKW}^{rel}$	0.0007
Uncertainty of the C₁₀-C₄₀ mass fraction (g/g)	$u_w^{col_A}$	0.0048

Tab. 18: Uncertainty of the C₁₀-C₄₀ mass fraction of BAM-K010g (**column B: BP-1**) based on Eq. 13

Uncertainty contribution		u_{rel}
Uncertainty of BAM-K010g concentration (gravimetric preparation) based on uncertainty estimations from weighing BAM-K010g and solvent (0.2 %)	$u_{c_MKW}^{rel}$	0.0020
Uncertainty of RHM concentration (gravimetric preparation) based on uncertainty estimations from weighing 10 IRHs and solvent (0.4 %)	$u_{c_RHM}^{rel}$	0.0040
Uncertainty of RHM purity (combined uncertainty of 10 IRH purity uncertainties acc. to Eq. 6)	u_{pur}^{rel}	0.0020
Uncertainty of RHM peak area (standard deviation of the mean of 18 RHM analyses); normalised to the RHM concentration	$u_{A_RHM}^{rel}$	0.0007
Uncertainty of BAM-K010g peak area (standard deviation of the mean of 50 BAM-K010g analyses); normalised to the concentration of specific BAM-K010g solution*	$u_{A_MKW}^{rel}$	0.0029
Uncertainty of the C₁₀-C₄₀ mass fraction (g/g)	$u_w^{col_B}$	0.0053

*) The standard deviation of the peak areas of the 50 BAM-K010g solutions alone would not be meaningful because these solutions have slightly different concentrations (resulting from weighing). Therefore, the peak areas were normalized by the specific concentration of BAM-K010g solution. In order to have a comparable basis, the same approach was necessary for the RHM, even if there was only one solution to be analyzed several times.

The overall uncertainty of the C₁₀-C₄₀ mass fraction of BAM-K010g (u_w) results from combining the outcomes of the investigations on two GC-columns (Tab. 17, 18). The expanded uncertainty (U_w) was calculated according to Eq. 14 applying a coverage factor of $k=2$:

$$U_w = k * u_w \quad \text{Eq. 14}$$

The expanded uncertainty of the C₁₀-C₄₀ mass fraction of $U_w = 0.0143$ g/g was rounded up to 0.015 g/g.

5.7 Certified C₁₀-C₄₀ mass fraction of BAM-K010g

The certified mass fraction of the boiling range between n-decane (C₁₀) and n-tetracontane (C₄₀) and its corresponding expanded uncertainty ($k=2$) is: **0.969 ± 0.015 g/g**.

6. Information on proper use of BAM-K010g

6.1 Transport, storage, handling and use

Intended use: The certified reference material BAM-K010g is intended to be used as calibration standard for the gas chromatographic determination of mineral oil hydrocarbons in water, soil and waste according to the analytical standard procedures ISO 9377-2, ISO 16703 and EN 14039.

Handling: Proper use of the reference material is essential for avoiding potential harm to the user. It is strongly recommended to handle and dispose of the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal. The content of the vial is preferably used completely at once. If not, the vial should be re-sealed with a new crimp cap or remaining standard should be filled in a tightly closed glass container and stored as specified.

Transport, Storage: Due to the proved stability a cooled dispatch of BAM-K010g is not necessary. On receiving, BAM-K010g is to be stored tightly closed at room temperature in a dark place. Under these storage conditions the material remains clear. If the material should become turbid by time, it should be replaced by a fresh unit and storage conditions should be checked and adjusted.

6.2 Shelf life

From stability monitoring of previous BAM-K010 batches, a material shelf life of at least 20 years is estimated for the storage at room temperature in a dark place. Since the dispatch to the end user may occur at any time the certificate of analysis (CoA) is valid for a period of two years beginning with the dispatch of the reference material from BAM. The validity of this information will be maintained by post-certification monitoring.

6.3 Legal notice

Neither BAM, its contractors nor any legal person acting on their behalf:

- (a) make any warranty or representation, express or implied, that the use of any information, material, apparatus, method or process disclosed in this document does not infringe any privately owned intellectual property rights; or
- (b) assume any liability with respect to, or for damages resulting from the use of any information, material, apparatus, method or process disclosed in this document save for loss or damage arising solely and directly from the negligence of BAM.

7. Metrological Traceability

Traceability of the certified value to the SI (Système International d'Unités) is ensured using a calibrated balance, additionally verified by DKD calibrated weights of class E2. The certification of the mass ratio is based on precise weighing of both mineral oil quantities to be mixed. A correction of buoyancy was applied considering the densities of the mineral oils.

The $C_{10} - C_{40}$ mass fraction (corresponding to the range between the boiling points of n-decane and n-tetracontane) is traceable to a hydrocarbon reference mixture consisting of ten hydrocarbon standards (Sigma-Aldrich) with defined and confirmed purities through direct comparisons using gas chromatography with flame ionisation detection (GC-FID) and gravimetric handling.

8. Information on and purchase of the CRM

The certified reference material BAM-K010g is supplied by

Bundesanstalt für Materialforschung und -prüfung (BAM)

Department 1 – Analytical Chemistry; Reference Materials

Division 1.7 - Organic Trace and Food Analysis

Richard-Willstätter-Str. 11, D-12489 Berlin, Germany

Phone: +49 30 8104 2061

Fax: +49 30 8104 72061

E-Mail: sales.crm@bam.de

Each unit of BAM-K010g will be distributed together with a detailed CoA containing the certified value and its uncertainty, a material description and information on recommend use, handling, storage and metrological traceability.

Information on certified reference materials can be obtained from BAM homepage <https://www.bam.de> and BAM-webshop www.webshop.bam.de.

9. References

- [1] ISO 9377-2 (2000), Water quality - Determination of hydrocarbon oil index - Part 2: Method using solvent extraction and gas chromatography.
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