

Certificate of Analysis

Analytical reference material RMPMO P50Plus-Mix

Product identifier

Product No.: RMPMO P50Plus-Mix

Lot No.: 2604

Expiry: 04/2028

Storage

Tightly closed at -18°C in the dark. The reference material is shipped at room temperature.

Intended use

RMPMO P50Plus-Mix is an analytical reference material for the qualitative analysis of MOSH and MOAH by GC×GC. The material is intended for use as a performance standard, and/or in method development. It can be used to identify the different substance classes of MOAH (1-2 ring MOAH, 2.5 ring MOAH, and 3 and more ring MOAH). The material is not suitable for quantification purposes.

Material description

RMPMO P50Plus-Mix is bottled in an amber glass vial with a PTFE seal. The vial contains at least 1.4 ml of the mix solution, solved in hexane. The mix solution consists of mineral oil products and further spiked components and is solved in hexane (see details below).

Composition

The composition of the analytical reference material is based on the work of Maurus Biedermann et al., Official Food Control Authority of the Canton of Zürich (1) and Martin Lommatzsch et al., Laboratory Lommatzsch & Säger GmbH (2).

Different types of mineral oil products are solved in toluene to prepare a stock solution of mineral oil products (see table 1).

Table 1. Ratio of the mineral oil products in the stock solution

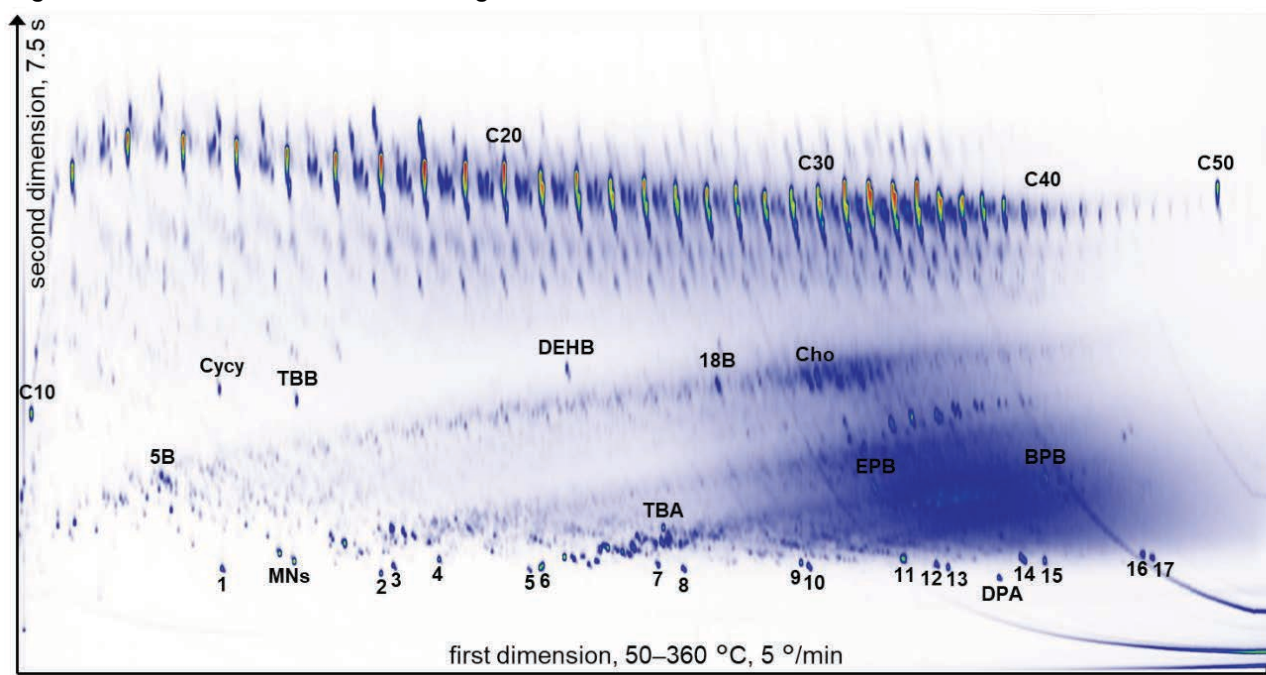
Mineral oil products	Ratio [w/w %]
Heating oil	13
Batching oil	26
Mid-range crude distillate	3
Heavy crude distillate	29
Distillate aromatic extract (DAE)	22
Paraffin wax, mp ≥ 65°C (CAS 8002-74-2)	7

The stock solution is spiked with marker substances and diluted with hexane to prepare the analytical reference material RMPMO P50Plus-Mix. The marker substances as well as the approximate concentration levels in RMPMO P50Plus-Mix are summarised in table 2. The provided concentration levels are approximate concentrations only and are not suitable for calibration purposes. The mineral oil products might contain unspecified amounts of the marker substances. RMPMO P50Plus-Mix contains about 7.5 % of toluene due to the stock solution of the mineral oil products, which is prepared in toluene.

Table 2. RMPMO P50Plus-Mix - composition and approximate concentration levels

Component	Abbreviation	CAS No.	Approximate concentration level [mg/l]
Mineral oil products (see table 1)		-	7500
n-Pentylbenzene	5B	538-68-1	2.1
n-Octadecylbenzene	18B	4445-07-2	2.1
Cyclohexylcyclohexane	Cycy	92-51-3	2.2
Cholestane	Cho	481-21-0	2.1
Tri-tert-butylbenzene	TBB	1460-02-2	2.1
1-Methylnaphthalene	1-MN	90-12-0	2.2
2-Methylnaphthalene	2-MN	91-57-6	2.1
Perylene	Per	198-55-0	2.1
1,4-Di(2-ethylhexyl)benzene	DEHB	87117-22-4	2.2
Benzo[rs]pentaphene	BPP	189-55-9	2.1
Coronene	Cor	191-07-1	2.1
2-(Tert-butyl)anthracene	TBA	18801-00-8	2.1
4-Ethyl-4'-(trans-4-pentylcyclohexyl)biphenyl	EPB	79709-85-6	2.1
4,4'-Bis(trans-4-propylcyclohexyl)biphenyl	BPB	85600-56-2	2.1
9,10-Diphenylanthracene	DPA	1499-10-1	2.1
n-Pentacontane	C50	6596-40-3	11
Acenaphthene		83-32-9	2.0
Acenaphthylene		208-96-8	2.0
Anthracene		120-12-7	2.0
Benz(a)anthracene		56-55-3	2.0
Benz[a]pyrene		50-32-8	2.0
Benzo(b)fluoranthene		205-99-2	2.0
Benzo(g,h,i)perylene		191-24-2	2.0
Benzo(k)fluoranthene		207-08-9	2.0
Chrysene		218-01-9	2.0
Dibenz(a,h)anthracene		53-70-3	2.0
Fluoranthene		206-44-0	2.0
Fluorene		86-73-7	2.0
Indeno(1,2,3-cd)pyrene		193-39-5	2.0
Naphthalene		91-20-3	2.0
Phenanthrene		85-01-8	2.0
Pyrene		129-00-0	2.0

Figure 1. GC×GC-FID chromatogram of the reference material RMPMO P50Plus-Mix



Cycy	Cyclohexylcyclohexane
C50	n-Pentacontane
5B	n-Pentylbenzene
TBB	Tri-tert-butylbenzene
MNs	1-Methylnaphthalene, 2-methylnaphthalene
DEHB	1,4-Di(2-ethylhexyl)benzene
18B	n-Octadecylbenzene
Cho	Cholestane
TBA	2-(Tert-butyl)anthracene
EPB	4-Ethyl-4'-(trans-4-pentylcyclohexyl)biphenyl
BPB	4,4'-Bis(trans-4-propylcyclohexyl)biphenyl
DPA	9,10-Diphenylanthracene
1	Naphthalene
2	Acenaphthylene
3	Acenaphthene
4	Fluorene
5	Dibenzothiophene
6	Anthracene, phenanthrene
7	Fluoranthene
8	Pyrene
9	Benz(a)anthracene
10	Chrysene
11	Benzo(b)-fluoranthene, benzo(k)fluoranthene
12	Benzo(a)pyrene
13	Perylene
14	Indeno(1,2,3-cd)pyrene, dibenz(a,h)anthracene
15	Benzo(g,h,i)-perylene
16	Coronene
17	Benzo[<i>rst</i>]pentaphene

Measurement method and conditions related to figure 1:

GC conditions

Instrument:	Thermo Scientific Trace 1610 GC
Pre-column:	0.2 m x 0.53 mm, deactivated in-house with 1,3-diphenyltetramethyldisiloxane
Separation column 1:	15 m x 0.25 mm i.d., OV-17ht, 0.15 µm film thickness
Separation column 2:	3.0 m x 0.15 mm i.d., PS-255 (dimethyl polysiloxane with 0.3 % methylvinyl siloxane), 0.04 µm film thickness, coated in-house
Injector:	PTV equipped with on-column liner for on-column injection
Injection volume:	0.5 µL
PTV temperature program:	analog to GC oven temperature program
Carrier gas:	hydrogen
Inlet pressure:	80 kPa, constant pressure
GC temperature program:	50 °C (4.5 min), 5 °/min to 360 °C (5 min); no secondary GC oven
FID:	380 °C, H ₂ 35 mL/min, air 350 mL/min, N ₂ 20 mL/min

Modulator settings

Instrument:	SepSolve INSIGHT-Thermal GC×GC modulator
Loop:	1.2 m of GC separation column 2
Cold jet:	air, -80 ±5 °C, 20 L/min (5 min), -0,2 L/min, 10 L/min (17 min)
Hot jet:	air, ~2 bar, 250 °C (5 min), 3 °/min, 400 °C (17 min), pulsed at 350 ms
Modulation time:	7.5 s

The analysis of RMPMO P50Plus-Mix as shown in figure 1 is performed by Maurus Biedermann, Official Food Control Authority of the Canton of Zürich.

Recommendations for use

The following recommendations are not binding and are provided as a helping hand and starting point for adjustment and improvement of internal procedures in the labs.

Recommendations for optimal column loads for direct injection of RMPMO P50Plus-Mix are:

Injection volume	Concentration P50Plus-Mix
0.5 µl	7500 mg/l
10-20 µl	125 mg/l (60x diluted, e.g. 167 µl/10 ml hexane)
80 µl	25 mg/l (300x diluted, e.g. 33 µl/10 ml hexane)

Example for the MOAH fraction of RMPMO P50Plus-Mix:

Inject 50 µl of RMPMO P50Plus-Mix (7500 mg/l) into the LC-GC system for fractionation. Dilute the MOAH fraction (approx. 450 µl) with hexane to a final volume of 1.5 ml. The resulting solution corresponds to a concentration level of 250 mg/l of the mineral oil products. Inject 10-20 µl of the solution into the GC×GC-FID system.

For further information, please refer to the publications of Maurus Biedermann et al. (1) and Martin Lommatzsch et. al. (2). An official method for the determination of MOAH by number

of aromatic ring systems by GC×GC is currently under development by DGF (draft of method C-VI 23 (26)). Further work is in progress within the European Screenfood project (3).

Homogeneity testing

Homogeneity testing is subcontracted to a lab, which holds an accreditation according to DIN EN ISO/IEC 17025:2018 (4).

The homogeneity of the most critical solution, the mineral oil mix solution is checked by analysing three sub-samples of the solution by LC-GC-FID for MOSH and MOAH. Relative standard deviations are 0.91 % related to total MOSH and 0.96 % related to total MOAH respectively. Homogeneity is confirmed. No further homogeneity tests are performed related to RMPMO P50Plus-Mix. It is a diluted solution in solvent. Homogeneity can be assumed. The analytical standard is for quantitative purposes only.

Metrological Traceability

The traceability of the certified value to the SI is ensured using calibrated balances and calibrated pipettes.

Handling

The usual laboratory safety precautions apply. See Safety Data Sheet.

Legal Notice

The producer certifies that this reference material meets the specification stated in this certificate until the expiry date, provided it is stored unopened at the recommended temperature herein. The reference material is produced and verified in accordance with DIN EN ISO 17034:2017 (5).

Supplier

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PROOF-ACS GmbH is accredited by the German accreditation authority DAkkS according to DIN EN ISO 17043:2023 (D-EP-22211-01-00) (6). PROOF-ACS GmbH does not have any analytical laboratory facilities of its own. Homogeneity testing and stability testing are subcontracted to laboratories, accredited according to DIN EN ISO/IEC 17025:2018 (4).

Customers with any concerns related to the reference material are invited to contact PROOF-ACS GmbH as mentioned before.

The certificate is authorised on behalf of PROOF-ACS GmbH by

References

1. Biedermann M, Eicher A, Altherr T, McCombie G. Quantification of mineral oil aromatic hydrocarbons by number of aromatic rings via comprehensive two-dimensional gas chromatography: First results in food. *Journal of Chromatography Open*. 2022 Nov;2:100072. doi:10.1016/j.jcoa.2022.100072
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3. Screenfood - Mineral oils [Internet]. [cited 2026 May 7]. Available from: <https://screenfood.eu/mineral-oil/>
4. DIN-Normenausschuss Qualitätsmanagement Statistik und Zertifizierungsgrundlagen (NQSZ). General requirements for the competence of testing and calibration laboratories. DIN EN ISO/IEC 17025:2018-03. 2018.
5. DIN-Normenausschuss Qualitätsmanagement S und Z (NQSZ) DNM (NMP). General requirements of the competence of reference material producers (ISO 17034:2016). DIN EN ISO 17034:2017 [Internet]. 2016. Available from: www.din.de
6. DIN EN ISO/IEC 17043:2010. Conformity assessment - General requirements for proficiency testing. DIN EN ISO/IEC 17043:2010-05. 2011. doi:10.1007/s00738-009-0685-2