

The Norwegian Industry Guide to Organic Geochemical Analyses

**Edition 4.0
30 May 2000**

Published by

Norsk Hydro
Statoil
Geolab Nor

SINTEF Petroleum Research
Norwegian Petroleum Directorate

Reference to this online publication:

Weiss, H.M., Wilhelms, A., Mills, N., Scotchmer, J., Hall, P.B., Lind, K. and Brekke, T. (2000): NIGOGA - The Norwegian Industry Guide to Organic Geochemical Analyses [online]. Edition 4.0 Published by Norsk Hydro, Statoil, Geolab Nor, SINTEF Petroleum Research and the Norwegian Petroleum Directorate. 102 pp [cited *CITATION_DATE*]. Available from World Wide Web: <<http://www.npd.no/engelsk/nigoga/default.htm>>.

where

CITATION_DATE = date when the document was seen or downloaded, e.g. "2000-07-21" or "21 June 2000"

 **STATOIL**



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CONTENTS

PREFACE	3
PURPOSE OF THIS GUIDE AND GENERAL REMARKS	4
DISCLAIMER	5
ANALYSIS GUIDE	6
General analysis scheme	7
Screening analyses	8
Headspace and occluded gas analysis	8
Determination of the total organic carbon content of rocks	10
Bulk-flow pyrolysis-FID (/TCD) (Rock-Eval pyrolysis)	12
Fluid, extract and gas analyses	15
Oil density	15
Separation of b.p. >210°C fraction (topping).....	17
Solvent extraction of rocks	19
Asphaltene precipitation.....	22
Bulk composition of deasphalted oils or rock extracts by TLC-FID (Iatroscan)	24
Liquid chromatographic separation of deasphalted oils or rock extracts	27
Stable carbon isotope analysis of oil or EOM and kerogen.....	29
GC analysis of whole ("stabilised") fluid	31
GC analysis of the saturated hydrocarbon fraction	36
GC analysis of the aromatic hydrocarbon fraction	39
GC-MS analysis of oil, EOM or saturated hydrocarbon fraction.....	43
GC-MS analysis of oil, EOM or aromatic hydrocarbon fraction	56
Gas-chromatography isotope-ratio mass-spectrometry (GC-IRMS) analysis.....	66
GC analysis of natural gas	70
Stable isotope analysis of gas compounds	72
Kerogen analyses	74
Thermal extraction and temperature-programmed pyrolysis-GC	74
Kerogen isolation	80
Preparation of kerogen slides	82
Maturity evaluation using transmitted light	83
Visual kerogen description using kerogen concentrates	85
Preparation of samples for vitrinite reflectance measurement and incident light organic petrography.....	88
Vitrinite reflectance measurements.....	90
Maceral description in reflected light.....	93
REPORTING GUIDE	95
Principal rules and remarks	96
The individual parts of a standard report	96
1 Title page	96
2 List of contents.....	97
3 Summary	97
4 Introduction.....	97
5 Results and discussion	98
6 Conclusions	98
7 References	98
8 Experimental procedures.....	98
9 Tables.....	98
10 Figures.....	98
Digital data	100
Numeric (tabular) data	100
Graphic data	100
GENERAL REFERENCES	101

PREFACE

Difficulties in the compilation of organic geochemical data from different laboratories caused the Norwegian oil companies Norsk Hydro, Saga Petroleum and Statoil, and the Norwegian Petroleum Directorate (NPD) in 1986 to agree on a set of common analytical procedures and reporting formats. These were put down in a "Standard analytical procedure requirement and reporting guide" which was revised in 1988 and 1993 when it also was renamed into "The Norwegian Industry Guide to Organic Geochemical Analyses" (NIGOGA). The revisions concerned mainly the standardisation of the peak integration methods, of the terminologies used in optical kerogen description and (twice) of the labelling of biomarker compounds. New methods such as TLC-FID were included, and descriptions of alternative, not yet fully established methods were added.

The creation of organic geochemical reference samples by the Norwegian Petroleum Directorate (Dahlgren et al. 1998 a-c) and the recent advance in electronic communication made a further revision of the guide necessary, and provoked a change in its concept, format and distribution.

This fourth edition puts, where possible, more weight on the specification of the result (quality) than on the description of the analytical procedures. Where the results from the NPD intercalibration justified this, the Norwegian Geochemical Standard (NGS¹) samples were chosen as reference samples, and their use has been made compulsory for some analyses. At the same time, the guide allows a greater flexibility with respect to the analytical techniques (e.g. the choice of column types) in order to avoid "freezing" of the analytical development. Quantitative GC and GC-MS analysis of oil and rock extract fractions using internal standards has been made compulsory.

A consistent format was introduced for all analysis descriptions in order to make the guide easier to use. All information relating to a certain type of analysis (work procedure and reporting) is now collected at only one place, while the Reporting Guide now contains only the general rules for reporting. Requirements are distinguished from suggestions by different typography.

The choice of the World Wide Web as the distribution channel for the NIGOGA was guided by the wish for quick, world-wide access and easy update.

The revision was carried out by the committee specified below, and was financed by Norsk Hydro ASA, Saga Petroleum ASA, Statoil and SINTEF Petroleum Research.

30 May 2000

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¹ Not to be confused with the Natural Gas Standards (IAEA-NGS) available from NIST.

² formerly IKU

PURPOSE OF THIS GUIDE AND GENERAL REMARKS

The aim of this guide is to provide reasonably detailed and, where standard samples are applicable, result-oriented guidelines for the performance and reporting of geochemical analyses as applied in the Norwegian petroleum industry, in order to enhance standardisation and comparability of the analytical results and their presentation.

The methods described herein should be viewed as minimum standards. If any analyses are not carried out in accordance with NIGOGA, this must be noted at the relevant point in the geochemical report.

NIGOGA is divided into two main parts: The "[Analysis Guide](#)" includes technical procedures, quality criteria and reporting specifications for the individual analysis types, together with recommendations, notes and selected key references. The "[Reporting Guide](#)" contains general guidelines for reporting and digital data transfer.

A simplified representation of the alkanes, omitting the hydrogen atoms, is used throughout the guide (e.g. C₁ = methane, i-C₄ = iso-butane).

The procedures in NIGOGA assume that the wells were drilled with water-based mud. If oil-based or other new generation synthetic muds are used, the customer may specify a special, and possibly different, sample treatment.

Norwegian Geochemical Standard (NGS) samples are available through the Norwegian Petroleum Directorate, attn.: The sample release committee, P.O. Box 600, N-4003 Stavanger, Norway. This committee evaluates applications on April 1st, August 1st and December 1st each year. The NPD will demand that any laboratory that receives the standard samples should, on NPD's request, submit data for future refinement of the standards' calibration. More information on the NGS samples and the way to apply for them is found at NPD's web site

The present edition of the NIGOGA generally quotes "permissible ranges" and "most likely values" from the Norwegian Geochemical Standards Newsletters (Dahlgren 1998 a-c) where also a full definition of these terms is found. However, some of the values reported herein have been modified due to recent information or for practical considerations, and this is commented in the respective tables. The values may be updated or additional quality criteria may be introduced in future editions of the NIGOGA, independently from possible future intercalibration rounds organised by the NPD.

The variables to be used for quality control of the reference analyses were chosen from practical and technical viewpoints rather than geochemical relevance. They are hence not always identical to the variables that have to be reported for the analysed samples.

The institutions that initiated this guide wish to continually improve the procedures and to increase the precision and reliability of the analytical results. Any suggestions for improvement of the analytical procedures, or of the Guide itself, are therefore highly appreciated and should be sent to the Norwegian Petroleum Directorate, attn.: Trond Brekke, P.O. Box 600, N-4003 Stavanger, Norway; phone +47-51 87 60 00, fax +47-51 55 15 71, e-mail Geochem@npd.no.

DISCLAIMER

The use of the procedures recommended by NIGOGA does not guarantee that the analytical results are acceptable for the customer. Neither the committee nor any of its members nor any of the institutions represented in the committee can be made responsible or liable for any problems or conflicts which may arise from the use of these procedures.

Compliance with NIGOGA does not of itself confer immunity from legal obligations. The use of registered names, trademarks, etc. in NIGOGA does not imply, even in the absence of a specific statement, that such names are exempt from the relevant protective laws and regulations and therefore free for general use.

ANALYSIS GUIDE

General analysis scheme

The figure below gives an overview of the flow and fractionation of the sample material, the analyses described in this Guide and the use of internal standards.

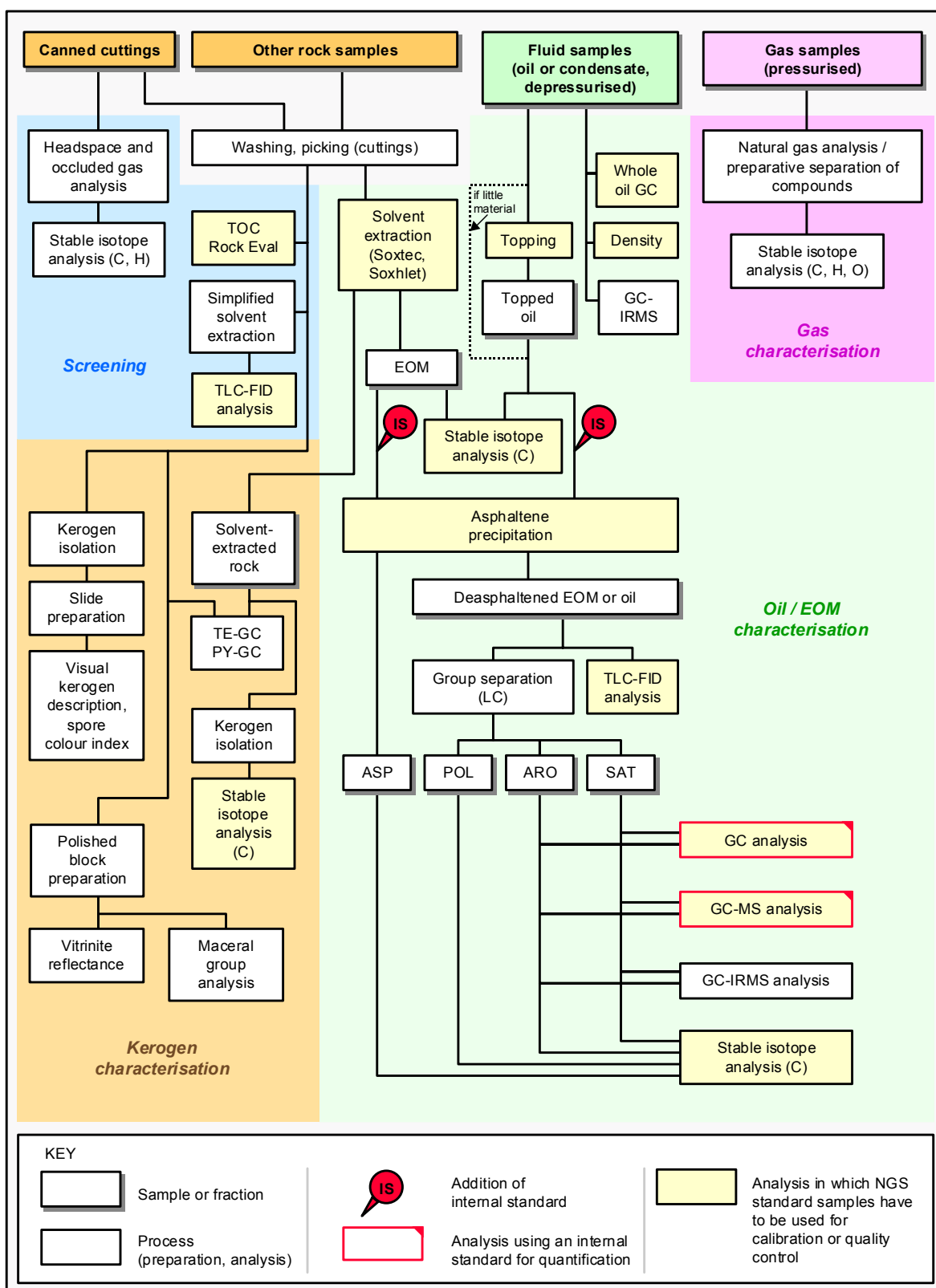


Figure ANA 1: General analysis scheme.

Screening analyses

Headspace and occluded gas analysis

No NGS standard applicable.

HSOC

Purpose, range of application, terminology

- Headspace gas = gas contained in the headspace (airspace) of a sealed can containing drill cuttings or core chips and mud or water (interpreted as the gas contained in open pores of the rock).
- Occluded gas (cuttings gas) = gas contained in the headspace of a sealed ball mill after crushing of a rock sample in water (interpreted as the gas contained in closed pores of the rock).
- Only the hydrocarbons are analysed and quantified relative to rock weight.

Samples to be analysed

- Headspace gas: Canned cuttings.
- Occluded gas: An aliquot (preferably 10-20 g) of washed but undried canned cuttings (preferably the 1-4 mm size fraction).

Procedural requirements

- The can must be checked for leakage of gas or liquid. Gas from leaking cans should normally not be analysed, but if this is done, it must be reported.
- An aliquot of the headspace gas must be extracted from the can and analysed by gas chromatography for the hydrocarbons C₁, C₂, C₃, i-C₄, n-C₄ and C₅₊. C₅₊ comprises the hydrocarbons eluted in backflush mode.
- After analysis, the headspace volume in the can must be determined by topping up with water.
- The sample must be washed, using cold water or an appropriate cleaning agent on 4 mm, 1 mm and 0.125 mm sieves to remove drilling mud. If occluded gas is to be analysed, an aliquot, preferably of the 1-4 mm fraction, must be taken and weighed into a ball mill. The remaining material must be weighed while wet, then dried at approximately 35-40°C and weighed again when dry.
- The aliquot of the wet sample has to be crushed in a gas-tight ball mill with water for 10 minutes.
- The gas from the headspace of the ball mill (= occluded gas) must be analysed in the same way as the headspace gas.
- After analysis, the gas volume in the ball mill must be determined by topping up with water.
- The gas chromatograph must be calibrated before the first analysis and at least once a day, using a calibration gas containing C₁, C₂, C₃, n-C₄ and some C₅₊ gases as external standard. Control analyses of the standard gas must be run at least three times a day. If the results (peak areas) from the control analyses deviate by more than 5% from the previous calibration, a new calibration must be carried out.
- The detected hydrocarbon compounds must be quantified on the basis of the peak areas (relative to those of the calibration gas). The concentrations of headspace and occluded gas components must be calculated in µl gas/kg dry rock, using the water content of the rock (in weight % of the wet rock) determined on an aliquot of the wet cuttings. Remember to include

the weight of the aliquot used for occluded gas analysis in the weight of the (total) sample used for headspace gas analysis.

Acceptance criteria and reference samples

(none, except for the acceptable variation of the gas standard control analyses specified under "Procedural requirements")

Reporting requirements

- The following variables must be reported for each analysed gas fraction (headspace, occluded):

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Concentrations of C ₁ , C ₂ , C ₃ , i-C ₄ , n-C ₄ , C ₅₊	µl/kg dry rock	x	x
Wetness = 100 * [Sum of C ₂ to n-C ₄] / [Sum of C ₁ to n-C ₄]	volume %	x	
i-C ₄ / n-C ₄	volume ratio	x	

- If both headspace and occluded gas were analysed, a table of the sum of headspace and occluded hydrocarbon concentrations is to be included in the printed report. This table shall contain the same variables as the tables for headspace and occluded gas.
- If not the 1-4 mm fraction was used, it must be reported which fraction was used.
- If alkenes are present (typically in the occluded gas), this must be stated in the report, and their approximate abundance relative to alkanes should be noted.
- If gas from a leaking sample can was analysed, this must be noted.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- The concentrations of the individual compounds in the calibration gas should be in the range of 100 - 4000 volume ppm.

Key references

(none)

Figures

(none)

Determination of the total organic carbon content of rocks

NGS standard applicable.

TOC

Purpose, range of application, terminology

- The purpose of this analysis is to determine the total organic carbon (TOC) content of a rock.

Samples to be analysed

- Crushed rock samples (washed and dried cores, side-wall cores (SWC) or picked cuttings of various lithologies).
- In the case of cuttings, the 1-4 mm size fraction should preferably be used.

Procedural requirements

- The total organic carbon (TOC) content must be determined using a carbon analyser (e.g. Leco) or a bulk-flow (Rock-Eval type) pyrolyser with oxidation unit.
- Sample preparation for carbon analyser: Aliquots of the samples must be weighed into crucibles, treated with 10% (vol) concentrated HCl (i.e. 1 part conc. HCl to 9 parts water) and heated to $60 \pm 5^\circ\text{C}$ to remove carbonate. Finally they must be washed with distilled water to remove all traces of HCl and water-soluble chlorides.
- Sample preparation for pyrolyser: Powdered rock samples are analysed without further treatment. (See [Bulk-flow pyrolysis-FID.](#))
- At least one NGS reference sample must be analysed as a control sample at the beginning and the end of each batch, and at least once every ten analyses.

Acceptance criteria and reference samples

- If the TOC content was determined on a carbon analyser (e.g. LECO) or Rock-Eval 2-5 pyrolyser, the results from control analyses of the reference samples must be within the following permissible ranges:

NGS sample SR-1

Variable (unit)	permissible range	most likely value	Comment
TOC (% of rock weight)	2.11 - 2.28	2.17	

NGS sample JR-1

Variable (unit)	permissible range	most likely value	Comment
TOC (% of rock weight)	11.3 - 12.5	11.9	Most likely value (median) differs from NGS Newsletter (modal value). Lower limit reduced to 11.3 (NGS Newsletter: 11.5) to allow for instrument uncertainty of $\pm 5\%$.

- If the TOC content was determined on a Rock-Eval 6 pyrolyser, the results should be within the following preliminary permissible ranges:

NGS sample SR-1

Variable (unit)	permissible range	most likely value	Comment
TOC (% of rock weight)	2.16 - 2.64	not defined	Preliminary values, based on results from 2 laboratories. Not in NGS Newsletter.

NGS sample JR-1

Variable (unit)	permissible range	most likely value	Comment
TOC (% of rock weight)	11.2 - 13.4	not defined	Preliminary values, based on results from 2 laboratories. Not in NGS Newsletter.

The JR-1 sample should not be used for calibration but only for control, as the most likely TOC value for this sample is still poorly defined.

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
TOC content	% of rock weight	x	x

- If Rock-Eval pyrolysis was carried out, the TOC data must be reported together with the Rock-Eval results.
- The method used (carbon analyser or pyrolyser) and the make/model/version of the instrument must be noted.
- If a pyrolyser is used, the maximum temperature of the oxidation step must also be noted.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- *The samples should be crushed in a centrifugal mill (if using a Siebtechnik mill, crushed for 1 min) or, when the sample is very small, crushed in a mortar. The resulting particle size should not exceed 63 µm.*
- *Pyrolysers like Rock-Eval 2-5, which use relatively low oxidation temperatures (600°C during the "normal cycle"), tend to underestimate the TOC content of highly mature rocks (particularly coals or organic rich shales). Rock-Eval 6 seems to eliminate this problem by using a maximum oxidation temperature of 850°C (Lafargue et al. 1998).*

Key references

- Lafargue, E., Marquis, F., Pillot, D. (1998): *Rock-Eval 6 applications in hydrocarbon exploration, production, and soil contamination studies. Revue de l'Institut Français du Pétrole* 53, 421-437.

Figures

(none)

Bulk-flow pyrolysis-FID (/TCD) (Rock-Eval pyrolysis)

NGS standard applicable.

RE

Purpose, range of application, terminology

- The purpose of Rock-Eval pyrolysis is to quickly obtain information on hydrocarbon generation potential, presence or absence of non-indigenous hydrocarbons, organic matter type and thermal maturity of a rock.
- S1, S2, S3 = quantified peak areas (mg HC or CO₂/g rock), PP = petroleum potential (mg HC/g rock) = S1 + S2, PI = production index (weight ratio) = S1 / (S1 + S2), Tmax (°C) = temperature at maximum of S2 peak at 25°C/min. heating rate, HI = hydrogen index (mg HC/g TOC) = 100 * S2 / TOC, OI = Oxygen index (mg CO₂/g TOC) = 100 * S3 / TOC. For further definitions applicable to Rock-Eval 6 see Lafargue et al. (1998).

Samples to be analysed

- Crushed rock samples (washed and dried cores, side-wall cores (SWC) or picked cuttings of selected lithologies).
- In the case of cuttings, the 1-4 mm size fraction should preferably be used.

Procedural requirements

- An aliquot of the crushed sample has to be weighed into a crucible and analysed in a bulk-flow pyrolysis-FID(/TCD) instrument of the Rock-Eval type.
- The sample weight must be adjusted to the expected pyrolysate yield of the sample in such a way that the obtained signal is within the linear part of the response curve of the detector. If overloading occurs, a smaller aliquot of the sample must be reanalysed.
- The instrument must be calibrated with a standard sample.
- At least one NGS reference sample must be analysed as a control sample at the beginning and the end of each batch, and at least once every ten analyses.

Acceptance criteria and reference samples

- If a Rock-Eval 2-5 was used, the results from control analyses of the reference samples must be within the following permissible ranges:

NGS sample SR-1

Variable (unit)	permissible range	most likely value	Comment
S1 (mg/g rock)	1.3 - 1.7	1.5	Lower limit changed to allow for ±8% instrument uncertainty (NGS Newsletter: 5.2).
S2 (mg/g rock)	5.0 - 6.1	5.4	
Tmax (°C)	433 - 440	435	

NGS sample JR-1

Variable (unit)	permissible range	most likely value	Comment
S1 (mg/g rock)	8 - 11	9	
S2 (mg/g rock)	67 - 79	73	
Tmax (°C)	429 - 435	430	

If a Rock-Eval 6 was used, the results should be within the following preliminary permissible ranges:

NGS sample SR-1

Variable (unit)	permissible range	most likely value	Comment
S1 (mg/g rock)	0.9 - 1.2		Preliminary values, based on results from 2 laboratories. Not in NGS Newsletter.
S2 (mg/g rock)	5.0 - 6.1	5.4	same as for Rock-Eval 2-5
Tmax (°C)	433 - 440	435	same as for Rock-Eval 2-5

NGS sample JR-1

Variable (unit)	permissible range	most likely value	Comment
S1 (mg/g rock)	6.7 - 7.5		Preliminary values, based on results from 2 laboratories. Not in NGS Newsletter.
S2 (mg/g rock)	67 - 79	73	same as for Rock-Eval 2-5
Tmax (°C)	429 - 435	430	same as for Rock-Eval 2-5

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
S1	mg/g rock	x	x
S2	mg/g rock	x	x
TOC	wt % of rock	x	x
HI	mg/g TOC	x	
PI	weight ratio	x	
PP	mg/g rock	x	
Tmax	°C	x	x

- The make/model/version of the instrument must be noted.
- If the used temperature program deviates from the "Cycle 1" of the Rock-Eval 2-5 pyrolyser (see Recommendations and notes), the program must be fully specified.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- The samples should be crushed in a centrifugal mill (if using a Siebtechnik mill, crushed for 1 min) or, when the sample is very small, crushed in a mortar. The resulting particle size should not exceed 63 µm.
- The standard temperature program (Cycle 1) on Rock-Eval 2-5 is as follows: 300°C (3 min.) - 25°C/min. linear temperature gradient - 600°C (1 min.). CO₂ trap shut off at 390°C (only relevant if S3 measured). The final temperature on older Rock-Eval instruments is 550°C. The programme may have to be modified on other pyrolysers (e.g. Rock-Eval 6) to obtain correct values for the control samples.

Key references

- Espitalié, J., Deroo, G., Marquis, F. (1985 a): La pyrolyse Rock-Eval et ses applications. Première Partie. Revue de l'Institut Français du Pétrole 40, 563-579.
- Espitalié, J., Deroo, G., Marquis, F. (1985 b): La pyrolyse Rock-Eval et ses applications. Deuxième Partie. Revue de l'Institut Français du Pétrole 40, 755-784.

- *Espitalié, J., Deroo, G., Marquis, F. (1986): La pyrolyse Rock-Eval et ses applications. Troisième Partie. Revue de l'Institut Français du Pétrole 41, 73-89.*
- *Peters, K.E. (1986): Guidelines for evaluating petroleum source rock using programmed pyrolysis. American Association of Petroleum Geologists Bulletin 70, 318-329.*
- *Lafargue, E., Marquis, F., Pilot, D. (1998): Rock-Eval 6 applications in hydrocarbon exploration, production, and soil contamination studies. Revue de l'Institut Français du Pétrole 53, 421-437.*

Figures

(none)

Fluid, extract and gas analyses

Oil density

No NGS standard applicable.

DENS

Purpose, range of application, terminology

- The purpose of this analysis is to determine the density of the whole (untopped) fluid.
- API Gravity ($^{\circ}$) = $141.5 / \text{Density (g/cm}^3) - 131.5$

Samples to be analysed

- Whole fluid (untopped)

Procedural requirements

- Density measurements must be performed according to one of the accepted standard methods (see "Key references").

Acceptance criteria and reference samples

- No quantitative acceptance criteria are set, but the most likely value and permissible range for the NSO-1 reference sample are noted for information:

NGS oil standard NSO-1

Variable (unit)	most likely value	permissible range	Comment
Density (g/cm ³)	0.861	0.858 - 0.862	
API gravity ($^{\circ}$)	32.9	32.6 - 33.3	

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Oil density at 15°C and atmospheric pressure	<u>both</u> g/cm ³ <u>and</u> °API	x	x

- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- *It is recommended to analyse one NSO-1 reference oil sample with each batch for control.*

Key references

- *IP 160/96: Density of Crude Oils and Petroleum Products. Hydrometer Method (PM-B-4). The Institute of Petroleum, London.*
 - *ASTM D1298-85(1990)e1 Standard Practice for Density, Relative Density (Specific Gravity), or API gravity of Crude Petroleum and Liquid Petroleum Products by Hydrometer Method.*
 - *ASTM D5002-94 Standard Test Method for Density and Relative Density of Crude Oils by Digital Density Analyzer.*
 - *ASTM D1250-80(1997)e1 Standard Guide for Petroleum Measurement Tables.*
- For ordering information and updated lists of ASTM and IP standards see <http://www.astm.org> and <http://www.petroleum.co.uk/pubindex.htm>

Figures

(none)

Separation of b.p. >210°C fraction (topping)

NGS standard applicable.

TOPPING

Purpose, range of application, terminology

- The purpose of topping is to remove the low molecular weight compounds and thus to obtain an oil which is similar to EOM in terms of carbon number distribution. Topping of oil samples for chromatographic separation, isotope analysis etc. is generally recommended, but may under certain circumstances (e.g. very small samples) be neither desirable nor practical. Customer and service company must therefore agree whether a sample shall be topped or not.

Samples to be prepared

- Whole fluid (oil)

Procedural requirements

- The fluid must be distilled under atmospheric pressure to a temperature of 210°C in the headspace.
- For small samples (< ~1 ml) a rotary evaporator can be used. In this case the oil should be evaporated for 10 minutes at about 90°C with the water pump turned to maximum. (A temperature of 89°C and a pressure of 10 mm Hg (13 mbar) are equivalent to 210°C at atmospheric pressure (at 95°C the required pressure is 22 mbar).
- If GC or GC-MS analyses are expected to be carried out on the oil or its hydrocarbon fractions, a known amount of an internal standard must be added to the topped oil. For recommendations see [Solvent extraction of rocks](#).
- Control analyses of at least one aliquot of the NSO-1 reference oil sample must be carried out once a week, although analysis prior to and after each contiguous topping series (batch) is preferred. Control analyses must also be carried out when experimental parameters are changed.

Acceptance criteria and reference samples

- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1

Variable (unit)	most likely value	permissible range	Comment
Topped oil (C ₁₅₊ , residue) (wt % of whole oil)	77	70 - 83	

Reporting requirements

The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Topped oil (C ₁₅₊ , residue)	wt % of whole oil	x	x

- If oils (e.g. small samples) are not topped, this must be reported.
- Results from the following control analyses must be reported: (1) last valid control analysis before the start of the batch, (2) all control analyses run during the batch and (3) first valid control analysis after the batch. See [Figure TOPPING 1](#) for examples.

- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

(none)

Key references

(none)

Figures

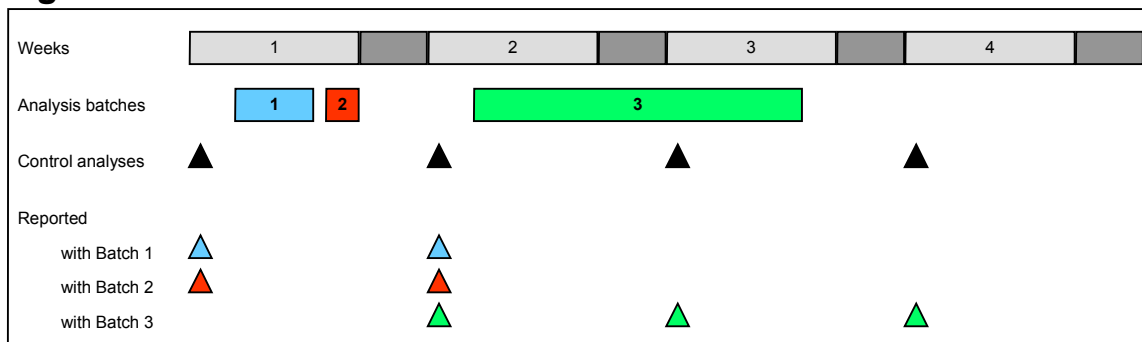


Figure TOPPING 1 Reporting of control analyses for topping.

Solvent extraction of rocks

NGS standard applicable.

EOM

Purpose, range of application, terminology

- EOM = Extractable Organic Matter = soluble bitumen = (total) solvent extract

Samples to be analysed

- Crushed rock samples (washed and dried cores, side-wall cores (SWC) or picked cuttings of selected lithologies).
- In the case of cuttings, the 1-4 mm size fraction should preferably be used.

Procedural requirements

- Extraction must be carried out using an extraction solvent consisting of dichloromethane (DCM) with 7 vol% methanol (i.e. volume ratio 93:7).
- Soxtec- or Soxhlet-type systems can be used, but the actual technique used must be noted.
- Each thimble must be pre-extracted (at least 10 min. boiling and 20 min. rinsing) before being used for sample extraction.
- Activated copper must be added to the extraction solvent for removal of elemental sulphur. The copper must be activated by washing in concentrated HCl and/or HNO₃ and then be washed with water, methanol and DCM/methanol.
- If GC or GC-MS analyses are expected to be carried out on the rock extract or its hydrocarbon fractions, a known amount of an internal standard must be added to the EOM. For recommendations see below.
- If Soxtec is used, the samples must be boiled for 1 h and rinsed for 2 h. If Soxhlet is used, the samples must be extracted for 24 hours.
- The solution must then be filtered or centrifuged and the solvent be reduced at a maximum temperature of 30°C and a minimum pressure of 200 mbar. Complete evaporation must be avoided, as it typically leads to a loss of low molecular weight hydrocarbons.
- Control analyses on at least one aliquot of the SR-1 or JR-1 NGS reference sample must be carried out once a week, although analysis prior to and after each contiguous extraction series (batch) is preferred. Control analyses must also be carried out when experimental parameters are changed.

Acceptance criteria and reference samples

- The hydrocarbon composition down to n-C₁₅ must not be affected by evaporation. This has to be checked at least once a week by gas chromatography of the saturated hydrocarbon fraction of an NGS reference sample. For acceptance limits see [GC analysis of the saturated hydrocarbon fraction](#), but also note the recommendations below.
- In addition to this, the results from control analyses of the reference samples must be within the following permissible ranges:

NGS rock standard SR-1

Variable (unit)	permissible range	most likely value	Comment
EOM (mg/kg rock)	4300 - 5800	4800	

NGS rock standard JR-1

Variable (unit)	permissible range	most likely value	Comment
EOM (mg/kg rock)	14000 - 18000	16000	

- If the control analyses bracketing a batch are outside the permissible range and the amount of rock sample is too small for re-extraction of all samples in the batch, the service company shall inform the customer and they together shall agree upon the further procedure. This has to be stated in the report.

Reporting requirements

- The method used (Soxhlet or Soxtec) must be noted.
- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Weight of extracted rock	g	x	x
Weight of EOM	mg	x	x
TOC content of the rock (if determined on the material [fraction] used for extraction)	wt % of rock	x	x

- If the bulk composition of the EOM was analysed, these variables shall be reported together with the bulk composition (see [TLC-FID](#)).
- Results from the following control analyses must be reported: (1) last valid control analysis before the start of the batch, (2) all control analyses run during the batch and (3) first valid control analysis after the batch. See [Figure EOM 1](#) for examples.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- *The use of the Soxtec technique (instead of Soxhlet) is recommended.*
- *As the solvent must not be completely removed from the extract solution, the weight of the total EOM should be determined by drying and weighing an aliquot of the solution, and by multiplying the obtained weight by the volume ratio between the whole solution and the aliquot.*
- *It is recommended to reduce the potential risk of photodegradation, in particular of the aromatic fraction, by using brown vials for separated fractions.*
- *Although values for the control of evaporation losses are currently defined only for the SAT fraction, it is strongly recommended to run control analyses also on the ARO fraction, as this is typically more sensitive to evaporation than the SAT fraction (particularly the methyl-naphthalenes).*
- *Below is a list of commercially available compounds that could be used in the internal standard. The choice of the compounds should be made in consultation with the customer.*

For saturated hydrocarbon GC analysis:

Squalane, deuterated n-alkanes (e.g. C₁₂D₂₆, C₂₀D₄₂, C₂₄D₅₀).

For aromatic hydrocarbon GC and GCMS analysis:

Deuterated PAH (e.g. D₈ Naphthalene, D₁₀ 1-Methylnaphthalene, D₁₂ 1,8-Dimethylnaphthalene, D₁₀ Phenanthrene, D₁₀ Pyrene, D₁₄ 2,2'-Binaphthyl, D₈ Dibenzothiophene), 2-Methyl-2-(4,8,12-trimethyltridecyl)chroman (for methyl-dibenzothiophenes).

For saturated biomarker analysis:

5β(H)-Cholane (C₂₄β_{αα}20R), deuterated cholestanes.

For aromatic biomarker analysis:

Deuterated steranes, e.g. D₃ C₂₁ Monoaromatic sterane, D₃ C₂₈/C₂₉ Monoaromatic sterane, D₂ C₂₀ Triaromatic sterane, D₂ C₂₇/C₂₈ Triaromatic sterane, D₂ C₂₈ Triaromatic sterane.

Key references

(none)

Figures

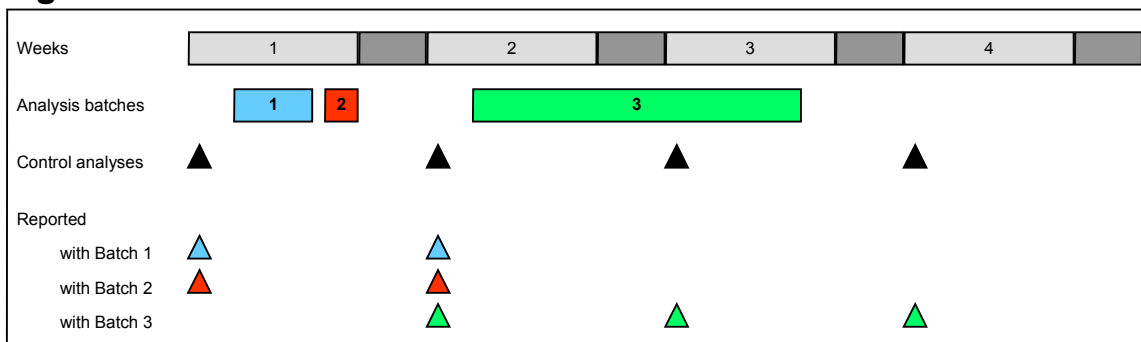


Figure EOM 1 Reporting of control analyses for extraction.

Asphaltene precipitation

NGS standard applicable.

ASP

Purpose, range of application, terminology

- The purpose of asphaltene precipitation is (1) to determine the asphaltene content of topped / untopped oil or EOM gravimetrically and (2) to obtain an asphaltene-free fraction (maltene fraction) for bulk-group analysis by TLC-FID (Iatroscan) and for preparative group separation by MPLC.
- Asphaltenes (ASP) are defined in this Guide as the fraction of an topped / untopped oil or rock extract which is insoluble in n-pentane at ambient temperature.

Samples to be analysed

- EOM from rock extraction or topped / untopped oil, containing an internal standard (as described under [Solvent extraction of rocks](#)).

Procedural requirements

- The EOM or topped / untopped oil must be dissolved/diluted in approximately 3 µl of dichloromethane / methanol (93:7 v/v) per mg of EOM, and n-pentane must be added in 40 fold excess of the volume of EOM + dichloromethane / methanol.
- The solution must be stored in the dark (to minimise coagulation of resins) for at least 8 h at ambient temperature and then be filtered or centrifuged.
- The asphaltenes from the filter must be combined with the asphaltenes that remained in the original flask, and quantitatively transferred to a pre-weighed vial. The remaining solvent must be evaporated, and the weight must be recorded when constant.
- Control analyses on at least one aliquot of the SR-1 or JR-1 NGS reference sample must be carried out once a week, although analysis prior to and after each contiguous asphaltene precipitation series (batch) is preferred. Control analyses must also be carried out when experimental parameters are changed.

Acceptance criteria and reference samples

- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1 (must be topped before asphaltene precipitation³):

Variable (unit)	permissible range	most likely value	Comment
ASP / topped OIL (wt %)	1 - 4	1.9	

NGS rock standard SR-1

Variable (unit)	permissible range	most likely value	Comment
ASP / EOM (wt %)	6 -21	16	

NGS rock standard JR-1

Variable (unit)	permissible range	most likely value	Comment
ASP / EOM (wt %)	6 -17	13	

- If the control analyses bracketing a batch are outside the permissible range and the amount of rock sample is too small for re-extraction of all samples in the batch, the service company

³ Most likely values and permissible ranges for untopped NSO-1 oil are not available.

shall inform the customer and they together shall agree upon the further procedure. This has to be stated in the report.

Reporting requirements

- The following extraction data must be reported for all analyses, including valid control analyses of NGS reference samples:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
ASP / EOM or ASP / OIL (topped or untopped)	wt % of EOM or topped / untopped oil	x	x

- If further group separation is carried out, the asphaltene content must be reported together with the results from TLC-FID.
- Results from the following control analyses must be reported: (1) last valid control analysis before the start of the batch, (2) all control analyses run during the batch and (3) first valid control analysis after the batch. See [Figure ASP 1](#) for examples.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

(none)

Key references

(none)

Figures

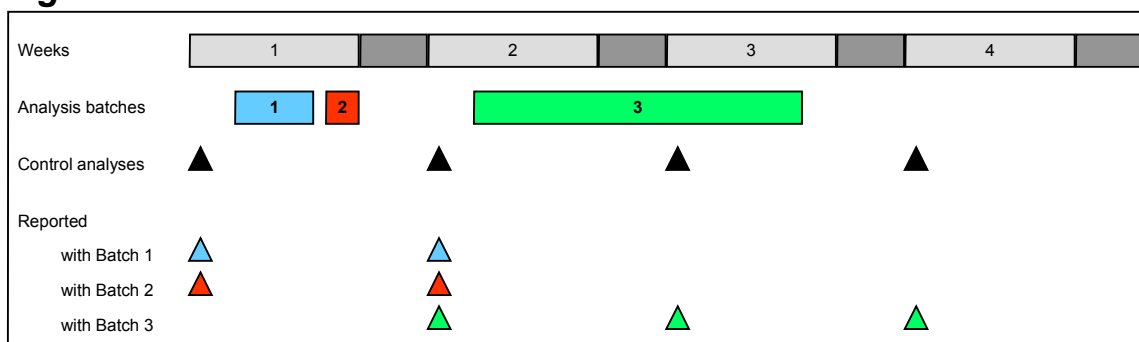


Figure ASP 1 Reporting of control analyses for asphaltene precipitation.

For a graphic scheme of the extraction, asphaltene precipitation and group separation procedures and terminology see [Figure TLCFID1](#) in the specification of TLC-FID analysis.

Bulk composition of deasphalted oils or rock extracts by TLC-FID (Iatroscan)

NGS standard applicable.

TLC-FID

Purpose, range of application, terminology

- The purpose of this analysis is, in combination with asphaltene precipitation, to obtain (semi-) quantitative information on the bulk group composition of EOM or topped / untopped oil ([Figure TLCFID1](#)).
- TLC-FID = Thin-Layer Chromatography - Flame Ionisation Detection; Trade name: Iatroscan.
- Saturated hydrocarbons (SAT) = aliphatic and alicyclic hydrocarbons, excluding monoaromatic compounds.
- Aromatic hydrocarbons (ARO) = aromatic hydrocarbons, including monoaromatic steroids and aromatic compounds with one heterocycle (e.g. thiophenes, benzo- and dibenzothiophenes, dibenzofurans).
- Polar fraction (POL) = non-asphaltenic polar compounds.

Samples to be analysed

- Deasphalted EOM or deasphalted topped / untopped oil (maltene fraction). The analysis must not be carried out on whole (i.e. not desphaltened) EOM or topped / untopped oil.

Procedural requirements

- The quantification of the saturated hydrocarbon, aromatic hydrocarbon and polar fractions must be performed using the Iatroscan liquid chromatography system with FID detection, using Chromarod S-III rods and the following solvents: hexane for elution of saturated hydrocarbons, toluene for elution of aromatic hydrocarbons and dichloromethane / methanol (93:7 v/v) for elution of the polar fraction.
- The three elution tanks must contain sufficient solvent such that the lower 0.5 cm of the silica layer on the rods are submerged. The tank atmosphere must be homogeneously saturated with solvent vapour.
- Before application, the rods must be activated by running through a "Blank Scan" on the Iatroscan instrument. The time between the activation and the elution must not exceed half an hour.
- The application spot must be as small as possible to ensure a good separation of the fractions.
- For calibration of the peak areas and control of the elution, at least one NGS reference sample (NSO-1 oil) must be included on each sample-rod holder.
- Response factors for quantification must be based on the peak area values (horizontal baseline) obtained from analysis of the NSO-1 oil reference sample. The nominal composition of this sample is SAT = 55 wt %, ARO = 37 wt %, POL = 8 wt % of the asphaltene-free, topped / untopped oil (see the table below and Recommendations and notes).
- Each sample and reference sample must be analysed at least in duplicate and the reported values (i.e. area percentages, not the areas themselves) must be averaged from the parallel analyses.

Acceptance criteria and reference samples

- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1

Variable (unit)	permissible range	most likely value	Comment
SAT / (SAT + ARO + POL) (wt %)	40 - 60	55	Median values, normalised to 100%; different from the modal values quoted in the NGS Newsletters.
ARO / (SAT + ARO + POL) (wt %)	25 - 42	37	
POL / (SAT + ARO + POL) (wt %)	5 - 13	8	
SAT / (SAT + ARO + POL) (area %)	40 - 50		No most likely values given due to poor value distributions.
ARO / (SAT + ARO + POL) (area %)	36 - 48		
POL / (SAT + ARO + POL) (area %)	9 - 17		

NGS rock standard SR-1

Variable (unit)	permissible range	most likely value	Comment
SAT / (SAT + ARO + POL) (wt %)	37 - 47		No most likely values given due to poor value distributions.
ARO / (SAT + ARO + POL) (wt %)	18 - 25		
POL / (SAT + ARO + POL) (wt %)	30 - 50		
SAT / (SAT + ARO + POL) (area %)	23 - 31		No most likely values given due to poor value distributions.
ARO / (SAT + ARO + POL) (area %)	15 - 22		
POL / (SAT + ARO + POL) (area %)	47 - 55		

NGS rock standard JR-1

Variable (unit)	permissible range	most likely value	Comment
SAT / (SAT + ARO + POL) (wt%)	24 - 30		No most likely values given due to poor value distributions.
ARO / (SAT + ARO + POL) (wt%)	32 - 47		
POL / (SAT + ARO + POL) (wt%)	23 - 32		
SAT / (SAT + ARO + POL) (area %)	14 - 19		No most likely values given due to poor value distributions.
ARO / (SAT + ARO + POL) (area %)	42 - 45		
POL / (SAT + ARO + POL) (area %)	37 - 42		

Reporting requirements

- The following variables must be reported for all analyses, including valid control analyses of NGS reference rock samples:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Weight of extracted rock	g	X	X
Weight of EOM	mg	X	X
EOM / rock	mg / kg rock (= wt ppm)	X	
SAT / EOM	wt % of EOM	X	X
ARO / EOM	wt % of EOM	X	X
POL / EOM	wt % of EOM	X	X
ASP / EOM	wt % of EOM	X	X
HC / EOM	wt % of EOM	X	
TOC / rock (fraction used for extraction)	wt % of rock	X	X

- In addition, the weight and area percentages specified under "Acceptance criteria and reference samples" must be tabulated for all valid control analyses of NGS reference samples.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- When not in use, the rods must be stored dry (desiccator with dry silica gel).
- A homogeneously vapour-saturated tank atmosphere is best obtained by coating the side walls and the rear wall of the tank with filter paper.
- The elution solvent should be changed every second day or after 50 samples, whichever is earlier, in order to prevent variations in the solvent polarity (e.g. water uptake by hexane) and poor chromatographic separation.

- For transfer of the samples to the rods, the samples should be dissolved with dichloro-methane / methanol (93:7 v/v) using as little solvent as possible, and a defined amount of the solution (1 - 3 μ l, ideally containing 10 - 15 μ g of sample) should be applied on the rods with a syringe. Quantitative transfer must be ensured, e.g. by taking up about 0.5 μ l of dichloro-methane / methanol before the solution is drawn into the syringe.
- The following elution distances are recommended to ensure separation of the fractions:

Fraction	Solvent	Elution distance	Approximate elution time
SAT	n-hexane	10 cm	25 - 32 min.
ARO	toluene	5 cm	12 - 14 min.
POL	DCM/MeOH (93:7 v/v)	2 cm	2 - 3 min.

- After elution of each hydrocarbon fraction, the rods should be air-dried (about 2 minutes in a fume cupboard) and it must be checked visually that the solvent has evaporated.
- After elution of the polar fraction, the rack of rods should be dried in an oven at 60°C for 90 seconds to ensure a similar "C₁₅₊" residue for all samples.
- NIGOGA adopts the NSO-1 oil reference sample as a (secondary) calibration standard, as only small quantities of the calibration standard described by Bharati et al. (1997) are available.

Key references

- Bharati, S., Patience, R. Mills, N., Hanesand, T. (1997): A new North Sea oil-based standard for Iatroscan analysis, *Organic Geochemistry* 26, 49-57.

Figures

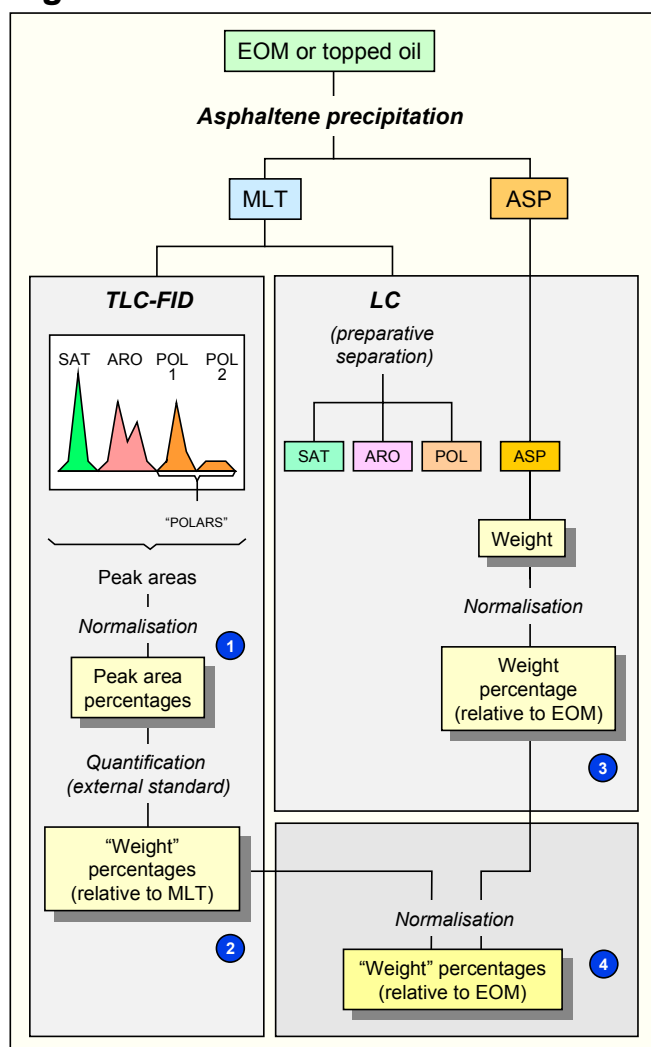


Figure TLCFID 1
Determination of the bulk composition of EOM or topped / untopped oil by a combination (4) of asphaltene precipitation with TLC-FID analysis of the maltene fraction (1), using an external, oil-based standard for quantification (2). Separation of the maltene (MLT) fraction (3) by LC is only for preparation of fractions for further analysis.

Liquid chromatographic separation of deasphalted oils or rock extracts

LC

NGS standard applicable.

Purpose, range of application, terminology

- The purpose of this preparation is to obtain saturated hydrocarbon, aromatic hydrocarbon and polar fractions of EOM or topped / untopped oil for further analyses (GC, GC-MS, isotope analysis).
- MPLC, HPLC = Medium (High) Performance Liquid Chromatograph(y).
- Saturated hydrocarbons (SAT) = aliphatic and alicyclic hydrocarbons, excluding monoaromatic compounds.
- Aromatic hydrocarbons (ARO) = aromatic hydrocarbons, including monoaromatic steroids and aromatic compounds with one heterocycle (e.g. thiophenes, benzo- and dibenzothiophenes, dibenzofurans).
- Polar fraction (POL) = non-asphaltenic polar compounds.

Samples to be analysed

- Deasphalted EOM or deasphalted topped / untopped oil (maltene fraction).

Procedural requirements

- A liquid chromatography technique must be used which can separate the deasphalted EOM or deasphalted topped / untopped oil into saturated hydrocarbons, aromatic hydrocarbons and the polar fraction.
- For control analyses see Recommendations and notes.

Acceptance criteria and reference samples

- Being a preparative tool, no quantitative acceptance criteria have been set for LC separation alone. However, if samples are analysed for biomarkers, the m/z 253 ion must also be monitored in the saturated hydrocarbon fraction in order to document that the monoaromatic steroids eluted in the aromatic fraction. If monoaromatic steroids are detected in the SAT fraction (as shown in Figure [SATGCMS 7](#) of the description of EOM / SAT GC-MS analysis), the hydrocarbon fractions must be recombined and the liquid chromatographic separation of the hydrocarbons be repeated. Furthermore, reanalysis of all other samples from the same batch should be considered in conjunction with the customer.

Reporting requirements

- The LC technique employed must be stated and the method should be shortly described in the chapter "[Experimental procedures](#)".
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- *A possible approach is the MPLC system described by Radke et al. (1980). This system basically contains two packed columns, a pre-column (deactivated silica) and a main column (silica gel), with n-hexane as the mobile phase.*

- The pre-column must be sufficiently active for retaining the polar fraction but not the aromatic fraction. It can be packed with deactivated silica of maximum particle size 45 µm. Packing the column with 40 µm particle size deactivated silica at both ends (1 cm each) prevents the small particles from flowing through the sinter.
- The main column must be able to separate the saturated hydrocarbons from the aromatic hydrocarbons which also include the monoaromatic steroids. It can be packed with silica gel 60 of a particle size between 40 and 63 µm. Special attention should be paid to loss of activity in the main column due to moisture in the hexane. This can easily be avoided by using an alumina moisture-capture column situated upstream from the injection port.
- After eluting the saturated hydrocarbons, the main column should be backflushed to elute the aromatics in order to save time.
- The polar fraction must be eluted by backflushing the precolumn with dichloromethane / methanol (volume ratio 93:7).
- A differential refractometer (for detection of all fractions) and a UV fluorescence detector (for detection of the aromatic fraction) should be used for control of the separation process.
- The solvent should never be completely removed from the collected fractions in order to avoid loss of low molecular weight compounds. Reduction of the solvent should be carried out at a pressure not less than 200 mbar and a temperature not greater than 30 °C.
- Although MPLC is in this Guide regarded as a preparative and not analytical method, it is strongly suggested to perform control analyses at least once a week, in accordance with the routines for [EOM](#) and [asphaltene precipitation](#) (see there), and to monitor the results. The most likely compositions of the NGS samples are listed for reference, although these are not very reliable as they are based on very few data.

NGS oil standard NSO-1

Variable (unit)	Permissible range	most likely value	Comment
SAT / (SAT + ARO + POL) (wt %)	56 - 58	57	n=3, unreliable values
ARO / (SAT + ARO + POL) (wt %)	26 - 30	27	n=3, unreliable values
POL / (SAT + ARO + POL) (wt %)	13 - 19	16	n=3, unreliable values

NGS rock standard SR-1

Variable (unit)	Permissible range	most likely value	Comment
SAT / (SAT + ARO + POL) (wt %)	40 - 43	41	n=3, unreliable values
ARO / (SAT + ARO + POL) (wt %)	17 - 22	20	n=3, unreliable values
POL / (SAT + ARO + POL) (wt %)	36 - 42	40	n=3, unreliable values

NGS rock standard JR-1

Variable (unit)	Permissible range	most likely value	Comment
SAT / (SAT + ARO + POL) (wt %)	29 - 33	31	n=2, unreliable values
ARO / (SAT + ARO + POL) (wt %)	30 - 40	35	n=2, unreliable values
POL / (SAT + ARO + POL) (wt %)	28 - 40	34	n=2, unreliable values

Key references

- Radke, M., Willsch, H., Welte, D.H. (1980): Preparative hydrocarbon group type determination by automated medium pressure liquid chromatography. *Analytical Chemistry* 52, 406-411.

Figures

(none)

Stable carbon isotope analysis of oil or EOM and kerogen

NGS standard applicable.

FRACISO

Purpose, range of application, terminology

The purpose of this analysis is to obtain stable carbon isotope ($\delta^{13}\text{C}$) ratios.

Samples to be analysed

- Solvent-free total rock extract (EOM) or topped / untopped oil (OIL), solvent-free saturated HC (SAT), aromatic HC (ARO), polar (POL) and asphaltene (ASP) fractions, and pre-extracted kerogen concentrate (KER) can be analysed. The customer has to specify the fractions to be analysed.

Procedural requirements

- The system must be calibrated using an international standard (e.g. the NBS 22 oil standard) and regularly controlled using a laboratory-internal standard.
- Control analyses of NGS reference samples must be included with each batch and after each tenth analysis within a batch.

Acceptance criteria and reference samples

- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1

Variable (unit)	permissible range	most likely value	Comment
$\delta^{13}\text{C}$ SAT (‰ PDB)	-29.4 to -28.9	-29.1	
$\delta^{13}\text{C}$ ARO (‰ PDB)	-28.3 to -27.8	-28.2	
$\delta^{13}\text{C}$ POL (‰ PDB)	-28.2 to -27.6	-28.1	
$\delta^{13}\text{C}$ ASP (‰ PDB)	-28.2 to -27.5	-27.9	
$\delta^{13}\text{C}$ topped OIL (‰ PDB)	-28.8 to -28.4	-28.6	
$\delta^{13}\text{C}$ whole OIL (‰ PDB)	-28.6 to -28.2	-28.4	n = 2, permissible range unreliable

NGS rock standard SR-1¹⁾

Variable (unit)	Permissible range	most likely value	Comment
$\delta^{13}\text{C}$ SAT (‰ PDB)	-33.5 to -32.8	-33.3	
$\delta^{13}\text{C}$ ARO (‰ PDB)	-32.5 to -31.9	-32.1	
$\delta^{13}\text{C}$ POL (‰ PDB)	-32.5 to -32.2	-32.3	
$\delta^{13}\text{C}$ ASP (‰ PDB)	-32.3 to -31.9	-32.0	
$\delta^{13}\text{C}$ EOM (‰ PDB)	-32.7 to -32.3	-32.6	

NGS rock standard JR-1¹⁾

Variable (unit)	Permissible range	most likely value	Comment
$\delta^{13}\text{C}$ SAT (‰ PDB)	-32.4 to -31.9	-32.0	
$\delta^{13}\text{C}$ ARO (‰ PDB)	-31.4 to -31.0	-31.1	
$\delta^{13}\text{C}$ POL (‰ PDB)	-31.2 to -30.9	-31.1	
$\delta^{13}\text{C}$ ASP (‰ PDB)	-31.2 to -30.7	-30.9	
$\delta^{13}\text{C}$ EOM (‰ PDB)	-31.6 to -31.3	-31.3	

¹⁾ No control data available for kerogen.

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Carbon isotope $\delta^{13}\text{C}$ values for SAT, ARO, POL, ASP, KER, EOM or OIL (whatever analysed)	‰ PDB	x	x

- If fluid or fluid fractions were analysed, it must be stated whether the fluid was topped or not.
- The isotopic value for the calibration standard used must be quoted, together with error limits.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- If the results from the control analyses deviate from the permissible ranges, the reason is likely to be found in the preparation of the analysed fractions. Check the following: Kerogen isolation (not carbonate- or solvent-free), rock extraction/asphaltene precipitation (correct solvent, complete evaporation of solvent from all fractions used for isotope analysis), oil topping (incomplete), group separation (wrong fraction cut).*
- Information on reference materials for stable isotope analysis (such as the NBS 22 oil) is available e.g. at the Isotope Hydrology Laboratory of the International Atomic Energy Agency (IAEA), http://www.iaea.org/programmes/rial/pci/isotopehydrology/reference_materials.htm, e-mail stabiso@refmat.iaea.org.*

Key references

(none)

Figures

(none)

GC analysis of whole ("stabilised") fluid

NGS standard applicable.

WOGC

Purpose, range of application, terminology

- The purpose of this analysis is to obtain the (mass) composition of the gasoline-range hydrocarbons for characterisation and correlation of fluids. The analysis may also provide a fingerprint of the total composition of the "stabilised" fluid (untopped, at ambient surface conditions, i.e. ~1 bar and ~15°C).

Samples to be analysed

- Fluid (stabilised oil or condensate) or sediment extract (EOM). Note that these samples typically have an undocumented p,T-history and that the results from this analysis cannot be compared with those from a PVT analysis.

Procedural requirements

- At least one NSO-1 reference oil sample must be analysed together with each contiguous series (batch) of analyses. If more than ten samples are analysed, one NSO-1 reference sample must be included per ten samples.
- The fluid must be analysed on a gas chromatograph fitted with a capillary column with a non-polar stationary phase and an FID.
- The peak areas of at least the following compounds must be determined:

Compound name	Label	Footnote
n-Butane	n-C4	
n-Pentane	n-C5	
2,2-Dimethylbutane	2,2-DMC4	
Cyclopentane	CyC5	
2,3-Dimethylbutane	2,3-DMC4	
2-Methylpentane	2-MC5	
3-Methylpentane	3-MC5	
n-Hexane	n-C6	
2,2-Dimethylpentane	2,2-DMC5	
Methylcyclopentane	MCyC5	
2,4-Dimethylpentane	2,4-DMC5	
Benzene	Benz	
3,3-Dimethylpentane	3,3-DMC5	
Cyclohexane	CyC6	
2-Methylhexane	2-MC6	
2,3-Dimethylpentane	2,3-DMC5	
1,1-Dimethylcyclopentane	1,1-DMCyC5	
3-Methylhexane	3-MC6	
<i>cis</i> -1,3-Dimethylcyclopentane	<i>c</i> -1,3-DMCyC5	
<i>trans</i> -1,3-Dimethylcyclopentane	<i>t</i> -1,3-DMCyC5	
3-Ethylpentane	3-EC5	1
<i>trans</i> -1,2-Dimethylcyclopentane	<i>t</i> -1,2-DMCyC5	1
n-heptane	n-C7	
<i>cis</i> -1,2-Dimethylcyclopentane	<i>c</i> -1,2-DMCyC5	2
Methylcyclohexane	MCyC6	2
Ethylcyclopentane	ECyC5	
Toluene	Tol	
n-Octane	n-C8	
(<i>m+p</i>)-Xylene	<i>mp</i> -Xyl	
n-Nonane	n-C9	
<i>i</i> -Decane (acyclic isoprenoid)	<i>i</i> -C10	3
n-Decane	n-C10	
<i>i</i> -Undecane (acyclic isoprenoid)	<i>i</i> -C11	3

Compound name	Label	Footnote
n-Undecane	n-C11	
n-Dodecane	n-C12	
i-Tridecane (acyclic isoprenoid)	i-C13	3
n-Tridecane	n-C13	
i-Tetradecane (acyclic isoprenoid)	i-C14	3
n-Tetradecane	n-C14	
i-Pentadecane (acyclic isoprenoid)	i-C15	3
n-Pentadecane	n-C15	
i-Hexadecane (acyclic isoprenoid)	i-C16	3
n-Hexadecane	n-C16	
n-Heptadecane	n-C17	
i-Octadecane = Norpristane (acyclic isoprenoid)	i-C18	3
n-Octadecane	n-C18	
i-Nonadecane = Pristane (acyclic isoprenoid)	i-C19	3
n-Nonadecane	n-C19	
i-Eicosane = Phytane (acyclic isoprenoid)	i-C20	3

Footnotes:

- 1,2 May coelute under certain conditions.
3 For identification see Figure WOGC 1.

- If n-alkanes and isoprenoids with higher C-numbers are detected, their peak areas should also be determined.

Acceptance criteria and reference samples

- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1

Variable (unit)	most likely value	permissible range	Comment
Pristane / n-C17 (peak area ratio)	0.60	0.55 - 0.64	
Benzene / Hexane (peak area ratio)	0.41	0.38 - 0.42	

- The following peaks must be separated to the baseline:
 - 2,2-DMC5 / MCyC5 / 2,4-DMC5
 - Benzene / 3,3-DMC5 / Cyclohexane
 - 2-MC6 / 2,3-DMC5 / 1,1-DMCyC5 / 3-MC6 / Dimethylcyclopentanes
 - Pristane / n-C17

Reporting requirements

- The following variables must be reported for at least all compounds listed under "Procedural requirements":

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Peak identity		x	x
Peak area	area units (usually μ Vs)	x	x

- The peak area ratios specified under "Acceptance criteria and reference samples" must be tabulated for valid control analyses of the NGS reference samples.
- All peak ratios referred to in the interpretation must be tabulated, and their calculation explained (formula). Peak ratios are to be based on peak areas.
- Gas chromatograms of all analyses, including valid control analyses of the reference sample, must be included.
- For further reporting requirements see the [Reporting Guide](#), in particular the sections on [Reference figures](#) and [Digital data](#).

Recommendations and notes

(none)

Key references

- *Thompson, K.F.M. (1983): Classification and thermal history of petroleum based on light hydrocarbons. Geochimica et Cosmochimica Acta 47, 303-316.*
- *Mango, F.D. (1997): The light hydrocarbons in petroleum: a critical review. Organic Geochemistry 26, 417-440.*
- *Walters, C.C., Hellyer, C.L. (1998): Multi-dimensional gas chromatographic separation of C₇ hydrocarbons. Organic Geochemistry 29, 1033-1041.*

Figures

see next page

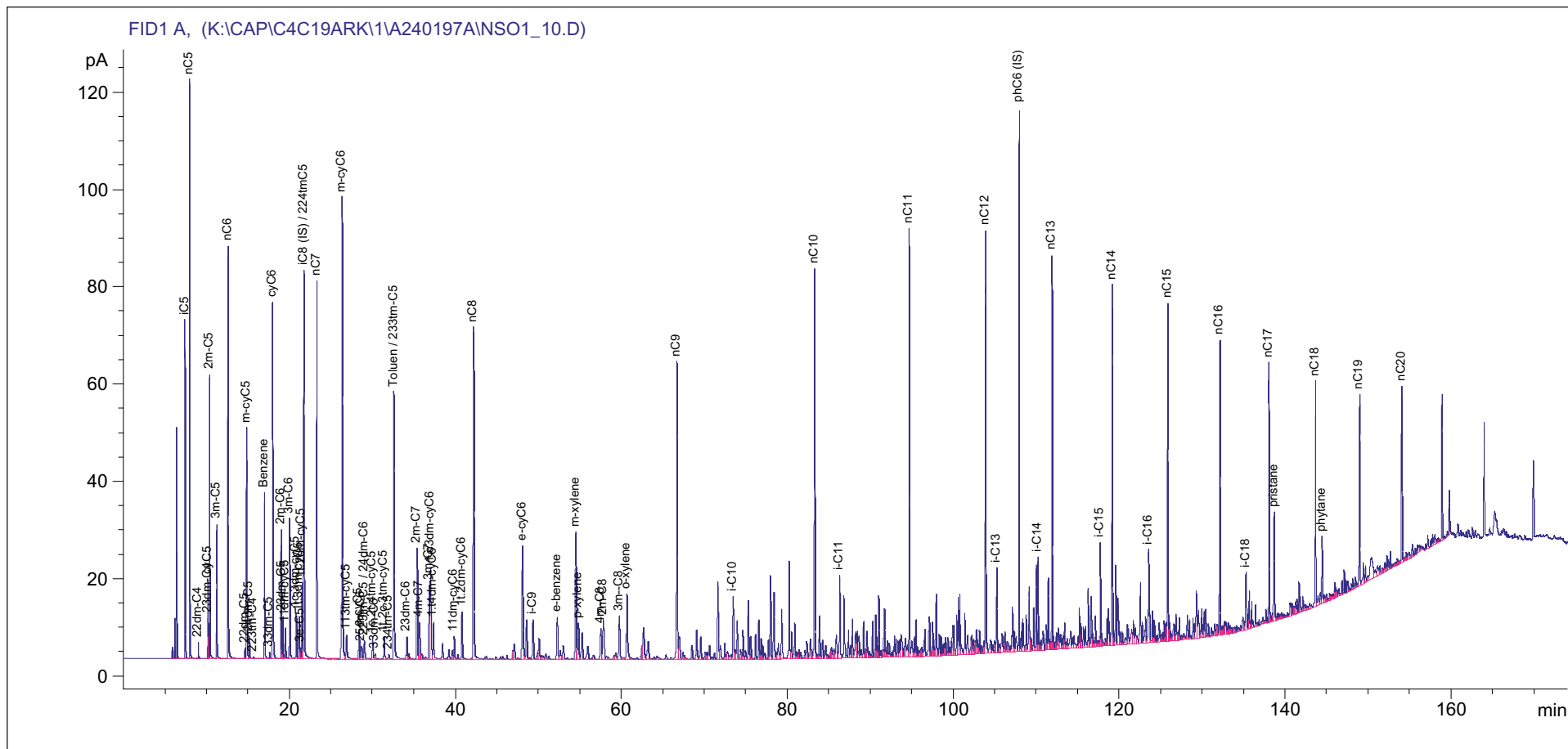


Figure WOGC 1 Whole oil gas chromatogram of NGS oil sample NSO-1 with annotated peaks and baseline. (Total chromatogram for overview only. For details see next figure.)

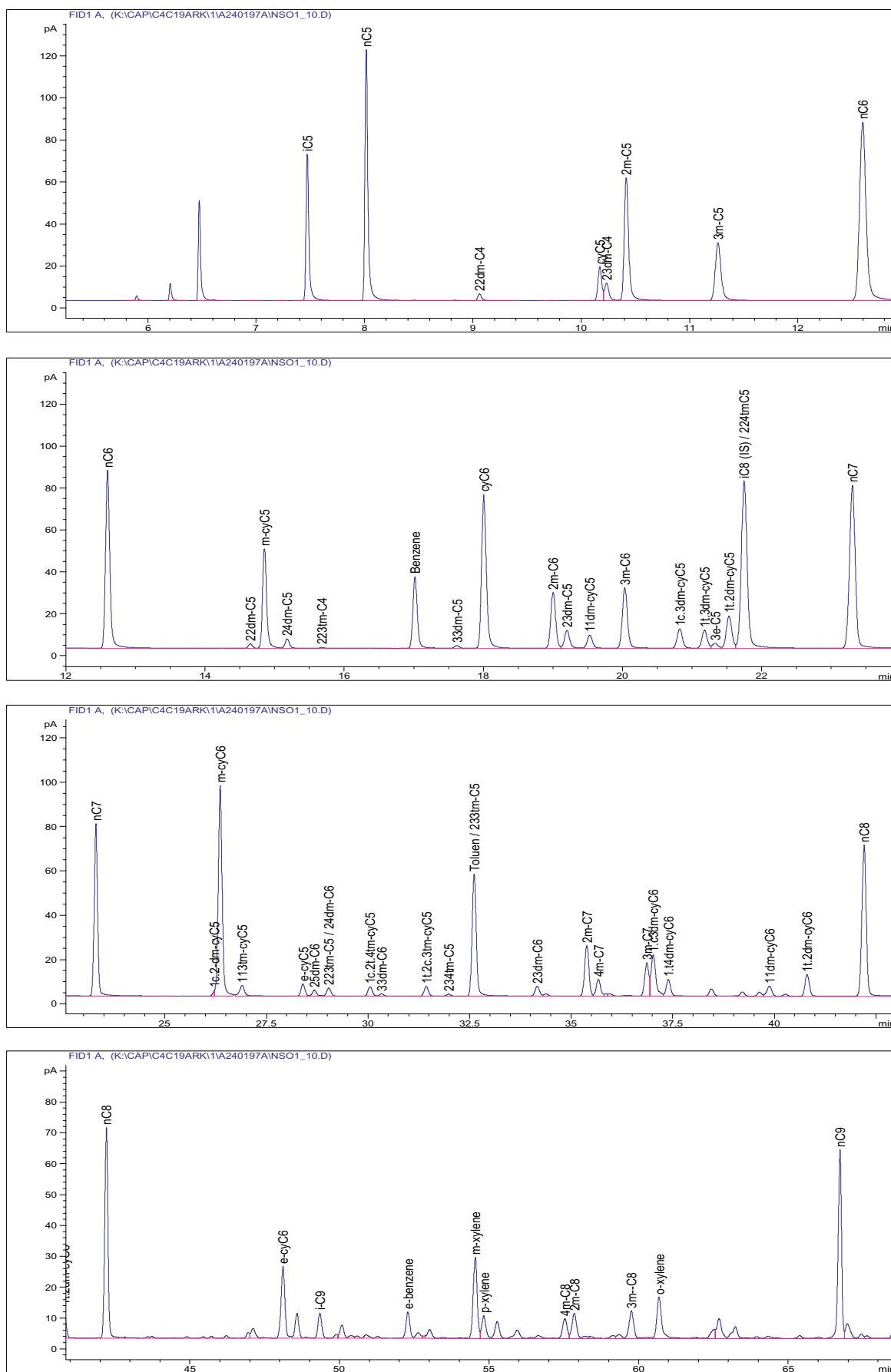


Figure WOGC 2 Whole oil gas chromatogram of NGS oil sample NSO-1 with annotated peaks and baseline: Expanded views of n-C3 to n-C6, n-C6 to n-C7, n-C7 to n-C8 and n-C8 to n-C9 regions.

GC analysis of the saturated hydrocarbon fraction

NGS standard applicable.

SAT GC

Purpose, range of application, terminology

- The purpose of this analysis is to obtain quantitative information on the molecular composition of the saturated hydrocarbon fraction. This may or may not include the high-molecular “waxy” compounds that can only be separated by high-temperature gas chromatography.

Samples to be analysed

- Saturated hydrocarbon fraction of extract or fluid, containing an internal standard that must be added to the EOM or topped / untopped oil before asphaltene precipitation.

Procedural requirements

- At least one NGS reference sample must be analysed together with each contiguous series (batch) of analyses. If more than ten samples are analysed, one NGS reference sample must be included per ten samples.
- The analysis should be performed on a gas chromatograph (GC) fitted with a capillary column of low polarity and a flame ionisation detector (FID).
- Peak areas of all identifiable n-alkanes and at least the acyclic isoprenoids pristane and phytane must be determined using a GC data system (i.e. no manual measurement on paper). The integration baseline must be set on top of the unresolved complex mixture (UCM) as illustrated in [Figure SATGC 1](#).
- The compound concentrations must be calculated from the peak areas, using the internal standard.

Acceptance criteria and reference samples

- Pristane and n-C₁₇ must be separated to the baseline.
- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1

Variable (unit)	permissible range	most likely value	Comment
Pr/n-C17 (peak area ratio)	0.55 – 0.66	0.60	
n-C15/n-C20 (peak area ratio)	1.4 – 2.0	1.8	
n-C30/n-C20 (peak area ratio)	0.20 – 0.32	0.29	
n-C17/(n-C17 + n-C27) (peak area ratio)	0.75 – 0.82	0.79	

NGS rock standard SR-1

Variable (unit)	permissible range	most likely value	Comment
Pr/n-C17 (peak area ratio)	2.0 - 2.3	2.15	
n-C15/n-C20 (peak area ratio)	1.1 - 1.5	1.3	
n-C30/n-C20 (peak area ratio)	0.1 - 0.2	0.18	
n-C17/ (n-C17 + n-C27) (peak area ratio)	0.72 - 0.80	0.77	

NGS rock standard JR-1

Variable (unit)	permissible range	most likely value	Comment
Pr/n-C17 (peak area ratio)	1.10 - 1.16	1.12	
n-C15/n-C20 (peak area ratio)	1.6 - 2.1	1.7	
n-C30/n-C20 (peak area ratio)	0.18 – 0.32	0.24	
n-C17/ (n-C17 + n-C27) (peak area ratio)	0.82 - 0.86	0.84	

Reporting requirements

- The following variables must be reported for all detected n-alkanes and acyclic isoprenoids:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Peak identity		x	x
Peak area	area units (usually $\mu\text{V}\cdot\text{s}$)	x	x
Concentration	ng / g EOM or oil	x	x

- The peak area ratios specified under "Acceptance criteria and reference samples" must be tabulated for valid control analyses of the NGS reference samples.
- All peak ratios referred to in the interpretation must be tabulated, and their calculation explained (formula). Peak ratios are to be based on peak areas or concentrations (specify!), not peak heights.
- Gas chromatograms of all analyses, including valid control analyses of the reference sample, must be included. The following peaks must be labelled in at least one gas chromatogram: n-C₁₅, n-C₁₇, pristane, n-C₁₈, phytane, n-C₂₀, n-C₂₅, n-C₃₀, n-C₃₅ etc.
- For further reporting requirements see the [Reporting Guide](#), in particular the sections on [Reference figures](#) and [Digital data](#).

Recommendations and notes

- Capillary columns with dimethyl polysiloxane phase (DB-1, OV-1, HP-1, BP-1, SE-30, CP-Sil 5CB etc.) are recommended. Length approx. 25 m, i.d. 0.25-0.32 mm, film thickness approx. 0.2 μm .
- Recommended temperature programme: 80°C (1 min.) - 4-6°C/min - 300°C (~20 min.).
- The GC performance (variation in response, discrimination by molecular weight etc.) should be tested regularly using a mixture of e.g. n-alkanes that spans a wide C-number interval.

Key references

- Bray, E.E., Evans, E.D. (1961): Distribution of n-paraffins as a clue to recognition of source beds. *Geochimica et Cosmochimica Acta* 22, 2-15.
- Marzi, R., Torkelson, B.E., Olson, R.K. (1993): A revised carbon preference index. *Organic Geochemistry* 20, 1303-1306.

Figures

see next page

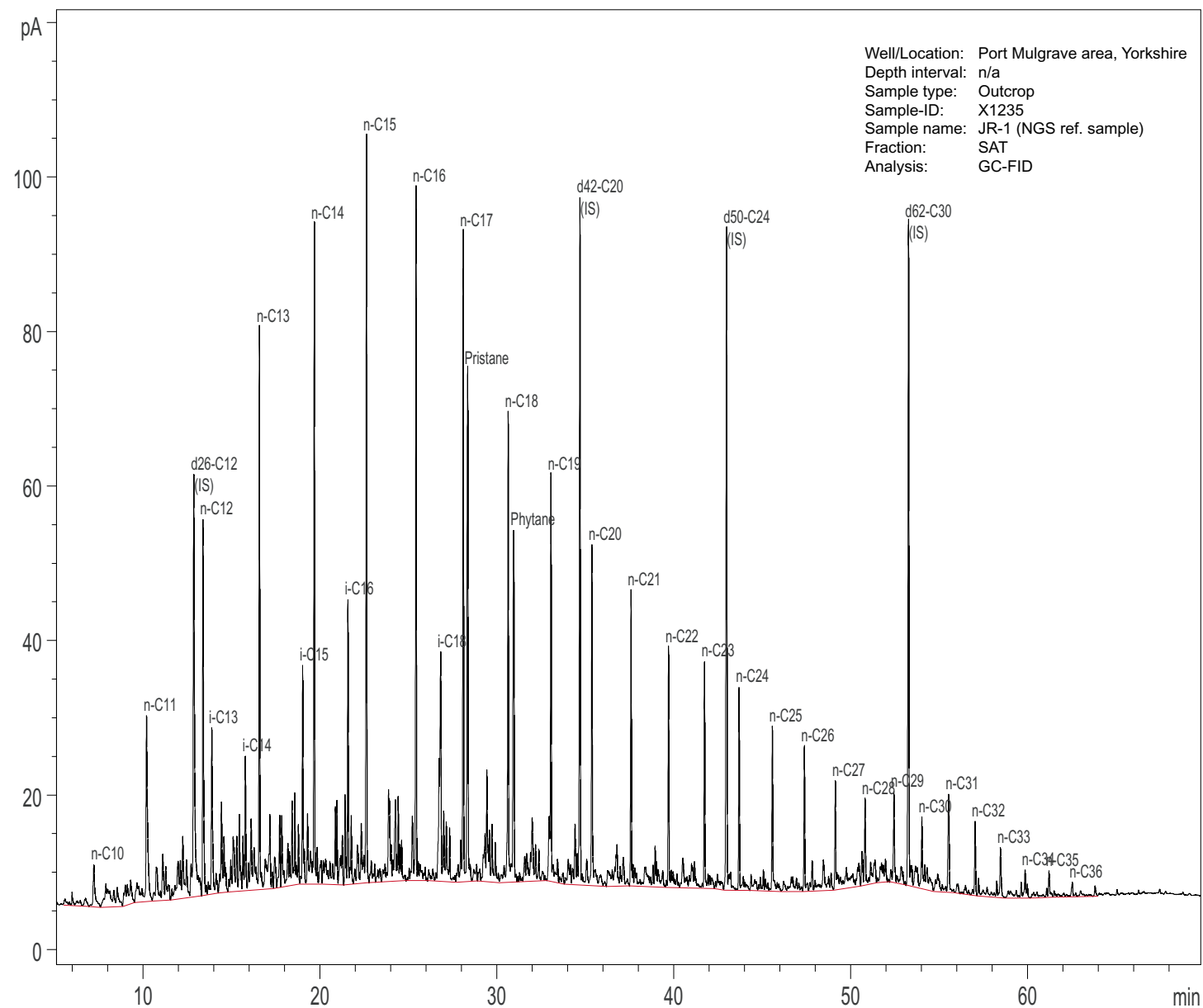


Figure SATGC 1 Gas chromatogram of the saturated hydrocarbon fraction from NPD oil standard NSO-1 with annotated peaks and baseline (IS = internal standard).

GC analysis of the aromatic hydrocarbon fraction

NGS standard applicable. Also see alternative: ARO GCMS

ARO GC

Purpose, range of application, terminology

- The purpose of this analysis is to obtain a fingerprint of the aromatic hydrocarbon fraction.
- The committee thinks that GC analysis of the aromatic fraction should only be performed to obtain a fingerprint and recommends that all molecular ratios previously reported from GC-FID/FPD data should from now on be derived from GC-MS data. However, if the customer specifically requests the determination of molecular ratios by GC-FID(/FPD) analysis, the procedural requirements stated below must be followed.

Samples to be analysed

- Aromatic hydrocarbon fraction of extract or fluid.

Procedural requirements

- The aromatic fraction must be analysed on a gas chromatograph fitted with a capillary column of low polarity and a flame ionisation detector.
- If specified by the customer, the gas chromatograph must be equipped with a glass-lined outlet splitter, a flame ionisation detector and a dual-flame photometric detector (FPD), to resolve coelution of methylphenanthrenes and aromatic sulphur compounds.
- If the determination of molecular ratios by GC-FID(/FPD) is specifically requested by the customer, at least the following peaks must be quantified, using an internal standard: Phenanthrene (P) and the methylphenanthrenes (MP) 3-MP, 2-MP, 9-MP and 1-MP.
- Quantification should preferably be based on peak areas, but peak heights may be used if the peaks are poorly resolved. It must be stated whether peak areas or heights were used.

Acceptance criteria and reference samples

(none)

Reporting requirements

- If requested by the customer, the following variables must be reported for P and the four MPs:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Peak identity		x	x
Peak height	height units (usually μV)	x	x
Peak area (if resolution is sufficient)	area units (usually $\mu\text{V}\cdot\text{s}$)	x	x
Concentration	ng / g EOM or oil	x	x

- Gas chromatograms of all analyses, including valid control analyses of the reference sample, must be included. The following peak groups must be labelled in at least one gas chromatogram (one label per group plus a bracket showing the elution time range of the respective group): C₂⁻, C₃⁻, C₄-naphthalenes, phenanthrene, C₁⁻, and C₂-phenanthrenes. If an FPD was used, dibenzothiophene, C₁⁻, C₂⁻ and C₃-dibenzothiophenes must also be labelled.
- For further reporting requirements see the [Reporting Guide](#), in particular the sections on [Reference figures](#) and [Digital data](#).

Recommendations and notes

- *Recommended column: length approx. 50 m, i.d. 0.25 - 0.32 mm, ~0.2 µm capillary column with 5% phenyl- and 95% methylpolysiloxane stationary phase (SE-54, SPB-5, BP-5, CP-Sil 8, DB-5 etc.). Recommended temperature programme: as for saturated hydrocarbons.*
- *It is recommended to analyse at least one NGS reference sample, preferably NSO-1, together with each contiguous series (batch) of analyses for control.*

Key references

(none)

Figures

see next page

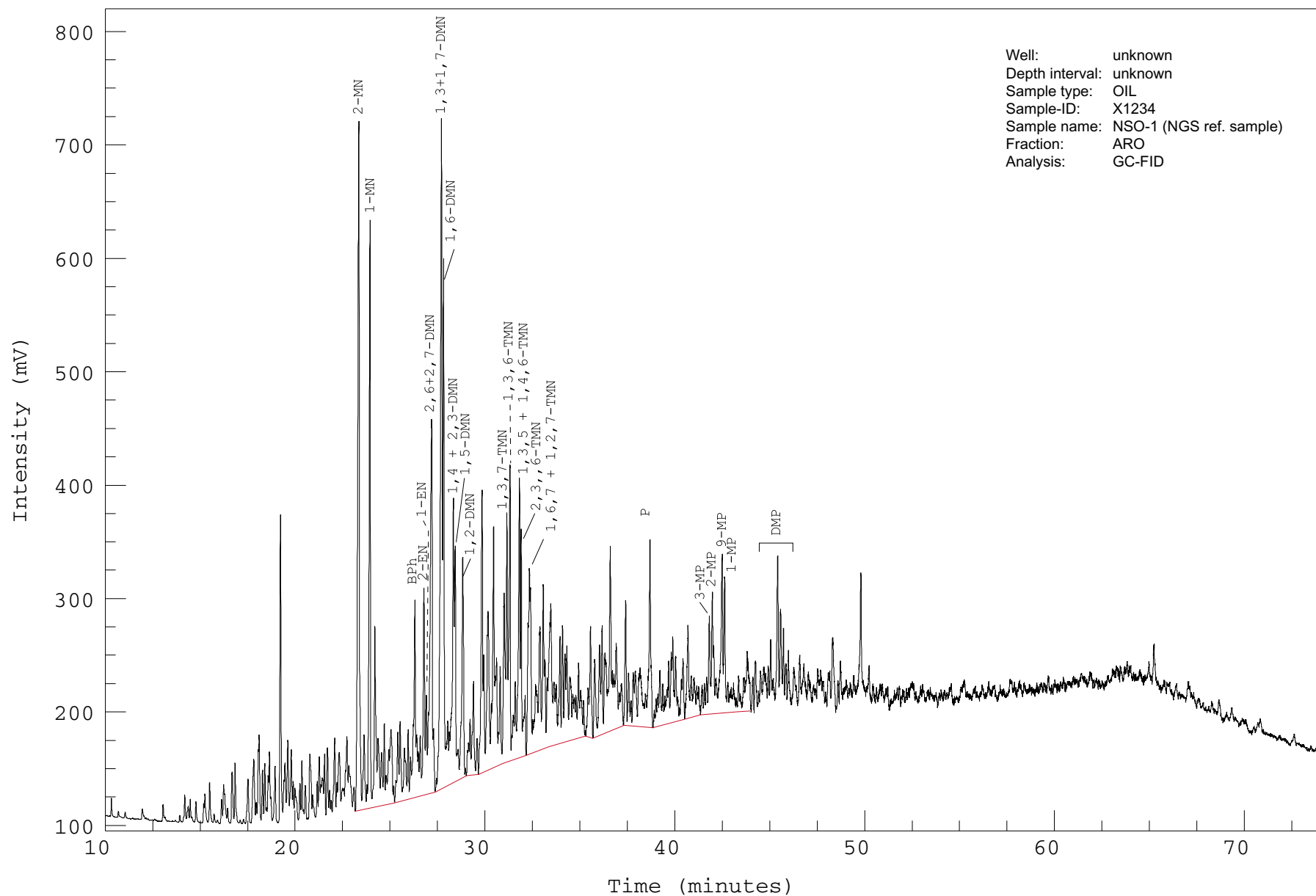


Figure AROGC 1 Gas chromatogram (FID) of the aromatic fraction from NGS oil sample NSO-1 with annotated peaks and baseline.

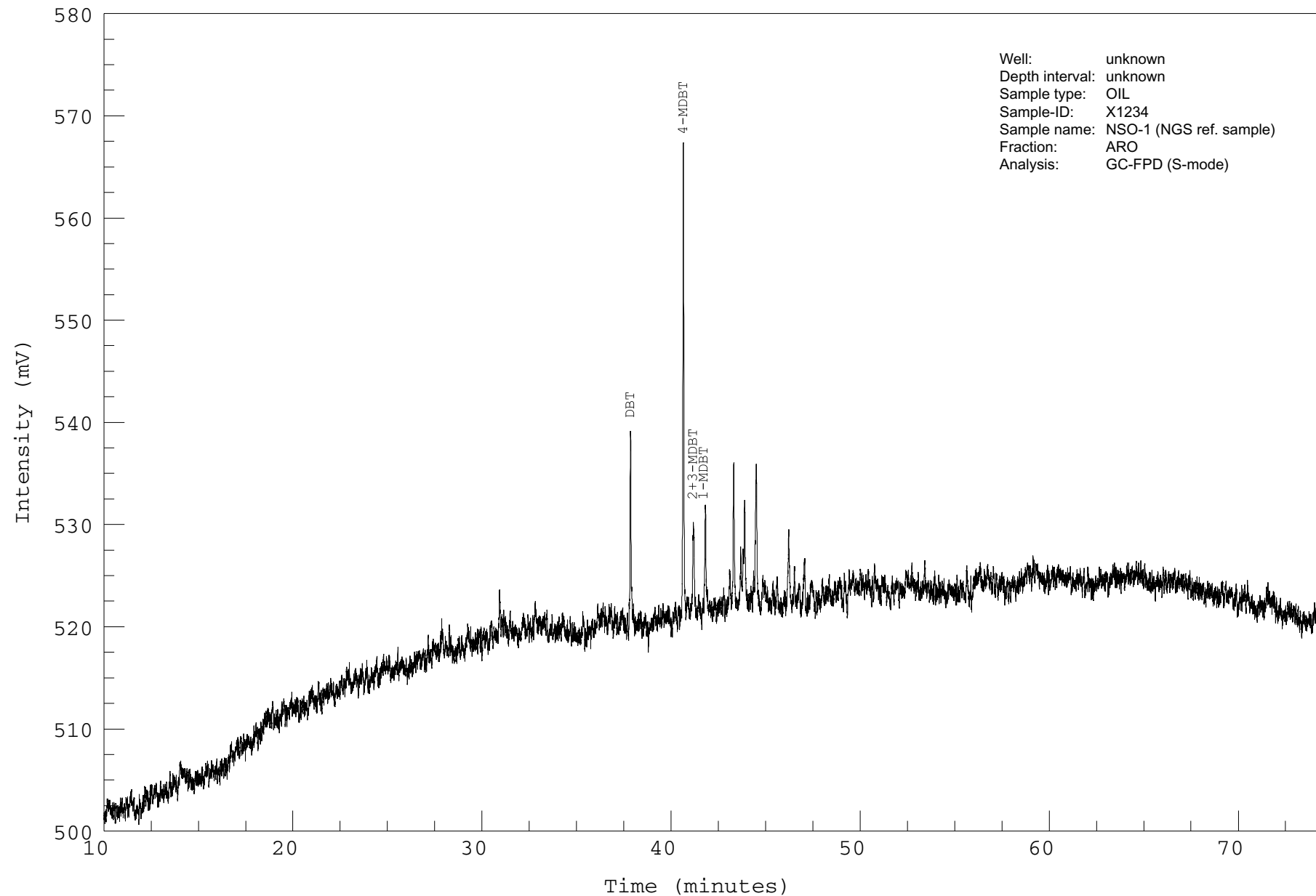


Figure AROGC 2 Gas chromatogram (FPD) of the aromatic fraction from NGS oil sample NSO-1 with annotated peaks.

GC-MS analysis of oil, EOM or saturated hydrocarbon fraction

NGS standard applicable.

SAT / EOM GCMS

Purpose, range of application, terminology

- The purpose of this analysis is to obtain quantitative data (i.e. concentrations relative to rock or oil weight) on biomarkers, including terpanes and steranes (“saturated biomarkers”).
- Codes (abbreviations) for biomarker compounds are listed under “Reporting requirements”.

Samples to be analysed

- Either the saturated hydrocarbon fractions of extracts or fluids using low or high resolution MS or the whole (stabilised) fluid or deasphalted extract using high resolution MS.
- The samples must contain an internal standard for quantification (see “[Topping](#)” and “[Solvent extraction of rocks](#)”).

Procedural requirements

- At least one NGS reference sample must be analysed together with each contiguous series (batch) of analyses. If more than ten samples are analysed, one NGS reference sample must be included per ten samples.
- The analysis should be performed on a gas chromatograph (GC) fitted with a capillary column of low polarity and coupled with a mass spectrometer (MS) or mass-sensitive detector (MSD).
- If nothing else is specified, the analyses shall be run in selective ion recording (SIR/MID) mode. The use of full-scan (FS), metastable ion monitoring (SMIM/MRM) or tandem mass spectrometry (MS-MS) must be specified or approved by the customer.
- As a minimum the following ion fragments must be monitored:

177.1643	terpanes (particularly demethylated hopanes)
191.1800	tri- and tetracyclic diterpanes, pentacyclic triterpanes
205.1956	pentacyclic, hopane-type triterpanes, particularly methylhopanes and C ₃₁
217.1956	regular and rearranged steranes
218.2035	regular and rearranged steranes (mainly ββ-steranes)
231.2113	4-methylsteranes
253.1956	monoaromatic steroids (which must <u>not</u> occur in the SAT fraction)
259.2426	rearranged steranes (diasteranes)
- For whole extract/fluid all ions listed for both the saturated and aromatic fraction should be monitored (for aromatic compounds see “[GC-MS analysis of oil, EOM or aromatic hydrocarbon fraction](#)”).
- Peak heights must be determined using a GC-MS data system (i.e. no manual measurement on paper). The peaks to be selected are specified under "Reporting requirements".
- The integration baseline should follow the top of the unresolved complex mixture (UCM) as shown in [Figure SATGCMS 4](#).
- The biomarker concentrations must be calculated from the peak heights, using the internal standard.

Acceptance criteria and reference samples

- The peak doublet $29\alpha\beta/29T$ s in m/z 191 must be separated at 40% height (of the smaller peak) or better. The peak doublet $29\beta\beta S/29\beta\beta R$ in m/z 217 must be separated at 90% height (of the smaller peak) or better.
- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1

Variable (peak height ratio)	most likely value	permissible range	Comment
$[23/3] / 30\alpha\beta$ in m/z 191	0.07	0.04 – 0.09	
$35\alpha\beta R / 30\alpha\beta$ in m/z 191	0.08	0.06 – 0.13	
$25nor30\alpha\beta / 25nor28\alpha\beta$ in m/z 177	0.5	0.3 – 0.8	
$29\alpha\alpha R / 27d\beta S$ in m/z 217	0.3	0.2 – 0.6	
$29\beta\beta S / 27\beta\beta R$ in m/z 218	0.9	0.7 – 1.2	

NGS rock standard SR-1

Variable (unit)	most likely value	permissible range	Comment
$[23/3] / 30\alpha\beta$ in m/z 191	0.28	0.1 – 0.4	
$35\alpha\beta R / 30\alpha\beta$ in m/z 191	0.04	0.02 – 0.08	
$29\alpha\alpha R / 27d\beta S$ in m/z 217	0.26	0.21 – 0.39	
$29\beta\beta S / 27\beta\beta R$ in m/z 218	0.8	0.6 – 1.3	

NGS rock standard JR-1

Variable (unit)	most likely value	permissible range	Comment
$[23/3] / 30\alpha\beta$ in m/z 191	0.16	0.13 – 0.28	
$35\alpha\beta R / 30\alpha\beta$ in m/z 191	0.05	0.01 – 0.07	
$29\alpha\alpha R / 27d\beta S$ in m/z 217	1.0	0.9 – 1.6	
$29\beta\beta S / 27\beta\beta R$ in m/z 218	0.7	0.5 – 0.8	

Reporting requirements

- The following technical information must be reported:
 - Method (SIR/MID, MSIM/SMIM/MRM, MS-MS),
 - Resolution (low or high) and
 - Analysed material (fraction or topped / untopped oil or EOM).
- The following variables must be tabulated for all peaks marked with “quantify” in the compound lists below (if present in the fragmentograms):

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Peak identity		x	x
Peak height	height units (usually μV or pA)	x	x
Concentration	ng / g EOM or oil	x	x

- The peak height ratios specified under “Acceptance criteria and reference samples” must be tabulated for valid control analyses of the NGS reference samples.
- All peak ratios referred to in the interpretation must be tabulated, and their calculation explained (formula). Peak ratios are to be based on peak heights.
- Mass fragmentograms for all specified masses and from all analyses must be presented, including those from valid control analyses of reference samples and also those that show no peaks.
- For further reporting requirements see the [Reporting Guide](#), in particular the sections on [Reference figures](#) and [Digital data](#).

- Compound list:

DI- and TRITERPANES (m/z 191)

Name	Label	Foot- notes	Quantify
C ₂₀ H ₃₆ tricyclic terpane	20/3		
C ₂₁ H ₃₈ tricyclic terpane	21/3		
C ₂₃ H ₄₂ tricyclic terpane	23/3		quantify
C ₂₄ H ₄₄ tricyclic terpane	24/3		quantify
C ₂₅ H ₄₆ tricyclic terpane	25/3R	1, 2	quantify
C ₂₅ H ₄₆ tricyclic terpane	25/3S	1, 2	quantify
C ₂₄ H ₄₂ tetracyclic terpane	24/4		quantify
C ₂₆ H ₄₈ tricyclic terpane	26/3R	1, 3	quantify
C ₂₆ H ₄₈ tricyclic terpane	26/3S	1, 3	quantify
C ₂₈ H ₅₂ tricyclic terpane	28/3R	1	
C ₂₈ H ₅₂ tricyclic terpane	28/3S	1	
C ₂₉ H ₅₄ tricyclic terpane	29/3R	1	
C ₂₉ H ₅₄ tricyclic terpane	29/3S	1	
C ₃₀ H ₅₆ tricyclic terpane	30/3R	1	
C ₃₀ H ₅₆ tricyclic terpane	30/3S	1	
18 α (H)-22,29,30-trisnorhopane	27Ts		quantify
17 α (H), 21 β (H)-25,28,30-trisnorhopane	25nor28 $\alpha\beta$		
17 α (H)-22,29,30-trisnorhopane	27Tm		quantify
17 β (H)-22,29,30-trisnorhopane	27 β		
17 α (H), 21 β (H)-28,30-bisnorhopane	28 $\alpha\beta$		quantify
17 α (H), 21 β (H)-25-norhopane	25nor30 $\alpha\beta$	4	quantify
17 α (H), 21 β (H)-30-norhopane	29 $\alpha\beta$		quantify
18 α (H)-30-norhopane	29Ts		quantify
15 α -methyl-17 α (H)-27-norhopane (diahopane)	30d		quantify
17 β (H), 21 α (H)-30-norhopane (normoretane)	29 $\beta\alpha$	5	quantify
18 α (H)-oleanane	30O		quantify
17 α (H), 21 β (H)-hopane	30 $\alpha\beta$		quantify
17 β (H), 21 α (H)-hopane (moretane)	30 $\beta\alpha$		quantify
Gammacerane	30G	6	quantify
17 β (H), 21 β (H)-hopane	30 $\beta\beta$		
17 β (H), 21 α (H)-homohopane	31 $\beta\alpha$		
17 α (H), 21 β (H), 22(S)-homohopane	31 $\alpha\beta$ S		quantify
17 α (H), 21 β (H), 22(R)-homohopane	31 $\alpha\beta$ R		quantify
17 α (H), 21 β (H), 22(S)-bishomohopane	32 $\alpha\beta$ S		quantify
17 α (H), 21 β (H), 22(R)-bishomohopane	32 $\alpha\beta$ R		quantify
17 α (H), 21 β (H), 22(S)-trishomohopane	33 $\alpha\beta$ S		quantify
17 α (H), 21 β (H), 22(R)-trishomohopane	33 $\alpha\beta$ R		quantify
17 α (H), 21 β (H), 22(S)-tetrakishomohopane	34 $\alpha\beta$ S		quantify
17 α (H), 21 β (H), 22(R)-tetrakishomohopane	34 $\alpha\beta$ R		quantify
17 α (H), 21 β (H), 22(S)-pentakishomohopane	35 $\alpha\beta$ S		quantify
17 α (H), 21 β (H), 22(R)-pentakishomohopane	35 $\alpha\beta$ R		quantify
C ₃₀ H ₅₄ $\Delta^{13(18)}$ -hopane	30D13		

TRITERPANES (m/z 177)

Name	Label	Foot- notes	Quantify
17 α (H), 21 β (H)-25,28,30-trisnorhopane	25nor28 $\alpha\beta$		quantify
17 α (H), 21 β (H)-25,30-bisnorhopane	25nor29 $\alpha\beta$		
17 α (H), 21 β (H)-25-norhopane	25nor30 $\alpha\beta$	4	quantify
17 α (H), 21 β (H), 22(R)-25-norhomohopane	25nor31 $\alpha\beta$ R		
17 α (H), 21 β (H)-30-norbishomohopane	30nor32 $\alpha\beta$		

Footnotes regarding di- and triterpanes:

- The elution order of the 22R- and 22S-isomers of the tricyclic diterpanes still appears to be uncertain. In this Guide it is assumed that 22R elutes before 22S on non-polar columns.
- C₂₅ tricyclic terpane should be broad peak or doublet.
- C₂₆ tricyclic terpane should be doublet peak.
- Peak height to be quantified in both m/z 191 and 177.
- 29 $\beta\alpha$ may co-elute with at least two other compounds, depending on column polarity.
- Where co-elution occurs with 31 $\alpha\beta$ R, the peak height of gammacerane can be calculated by difference:
30G = 31 $\alpha\beta$ R - (32 $\alpha\beta$ R / 32 $\alpha\beta$ S * 31 $\alpha\beta$ S).

STERANES (m/z 217)

Name	Label	Foot- notes	Quantify
13 β (H), 17 α (H), 20(S)-cholestane (diasterane)	27d β S		quantify
13 β (H), 17 α (H), 20(R)-cholestane (diasterane)	27d β R		quantify
13 α (H), 17 β (H), 20(R)-cholestane (diasterane)	27d α R		
13 α (H), 17 β (H), 20(S)-cholestane (diasterane)	27d α S	8	
5 α (H), 14 α (H), 17 α (H), 20(S)-cholestane	27 $\alpha\alpha$ S	6	quantify
5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	27 $\beta\beta$ R	7, 14	
5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	27 $\beta\beta$ S	8, 14	
5 α (H), 14 α (H), 17 α (H), 20(R)-cholestane	27 $\alpha\alpha$ R		quantify
24-methyl-13 β (H), 17 α (H), 20(S)-cholestane (diasterane)	28d β S		
24-methyl-13 β (H), 17 α (H), 20(R)-cholestane (diasterane)	28d β R	10	
24-methyl-13 α (H), 17 β (H), 20(R)-cholestane (diasterane)	28d α R	6	
24-methyl-13 α (H), 17 β (H), 20(S)-cholestane (diasterane)	28d α S		
24-methyl-5 α (H), 14 α (H), 17 α (H), 20(S)-cholestane	28 $\alpha\alpha$ S		
24-methyl-5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	28 $\beta\beta$ R	9, 14	
24-methyl-5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	28 $\beta\beta$ S	14	
24-methyl-5 α (H), 14 α (H), 17 α (H), 20(R)-cholestane	28 $\alpha\alpha$ R		quantify
24-ethyl-13 β (H), 17 α (H), 20(S)-cholestane (diasterane)	29d β S	7	quantify
24-ethyl-13 β (H), 17 α (H), 20(R)-cholestane (diasterane)	29d β R		quantify
24-ethyl-13 α (H), 17 β (H), 20(R)-cholestane (diasterane)	29d α R		
24-ethyl-13 α (H), 17 β (H), 20(S)-cholestane (diasterane)	29d α S	9	
24-ethyl-5 α (H), 14 α (H), 17 α (H), 20(S)-cholestane	29 $\alpha\alpha$ S		quantify
24-ethyl-5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	29 $\beta\beta$ R	14	quantify
24-ethyl-5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	29 $\beta\beta$ S	14	quantify
24-ethyl-5 α (H), 14 α (H), 17 α (H), 20(R)-cholestane	29 $\alpha\alpha$ R		quantify
24-propyl-5 α (H), 14 α (H), 17 α (H), 20(S)-cholestane	30 $\alpha\alpha$ S		
24-propyl-5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	30 $\beta\beta$ R		
24-propyl-5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	30 $\beta\beta$ S		
24-propyl-5 α (H), 14 α (H), 17 α (H), 20(R)-cholestane	30 $\alpha\alpha$ R		
4-methyl-14 α (H), 17 α (H)-cholestanes	M28 $\alpha\alpha$	11	
4,24-dimethyl-14 α (H), 17 α (H)-cholestanes	M29 $\alpha\alpha$	12	
4-methyl-24-ethyl-14 α (H), 17 α (H)-cholestanes	M30 $\alpha\alpha$	12	
4,23,24-trimethyl-14 α (H), 17 α (H)-cholestanes (dinosteranes)	M30D	13	

STERANES (m/z 218)

Name	Label	Foot- notes	Quantify
5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	27 $\beta\beta$ R	14	quantify
5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	27 $\beta\beta$ S	14	quantify
24-methyl-5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	28 $\beta\beta$ R	14	quantify
24-methyl-5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	28 $\beta\beta$ S	14	quantify
24-ethyl-5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	29 $\beta\beta$ R	14	quantify
24-ethyl-5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	29 $\beta\beta$ S	14	quantify
24-propyl-5 α (H), 14 β (H), 17 β (H), 20(R)-cholestane	30 $\beta\beta$ R		quantify
24-propyl-5 α (H), 14 β (H), 17 β (H), 20(S)-cholestane	30 $\beta\beta$ S		quantify

Footnotes regarding steranes:

- | | |
|------------|---|
| 6, 7, 8, 9 | Pairs of coeluting compounds. |
| 10 | Occurs as 24S/24R doublet. |
| 11 | Various possible isomers at 4,5,20 positions. |
| 12 | Various possible isomers at 4,5,20,24 positions. |
| 13 | Various possible isomers at 4,5,20,23,24 positions. |
| 14 | Peak height to be quantified in both m/z 217 and 218. |

Recommendations and notes

- Recommended column types are SE-54 (40 m), DB1 (50 m) or similar. A recommended temperature programme is as follows: Start at 50°C, 35°C/min to 150°C, 2°C/min to 310°C, isothermal at 310°C for 20 min. Higher start temperature may lead to significant peak tailing.

- *It is recommended to check the peak height ratios between early and late eluting compounds using a synthetic standard mixture that contains known concentrations of terpanes and steranes which cover the relevant range of elution times.*
- *If possible and appropriate, major and/or geochemically significant peaks in addition to those specified under "Reporting requirements" (including peaks in m/z 205, 231 and 259) should also be identified, and their heights and concentrations should be reported.*
- *If peak ratios are reported, they should preferably be of the type $100 \times a / (a + b)$ [%]. This saves space (as the redundant "0." of fractions is avoided) and avoids the problems connected with ratios of the type a/b (division by zero, very large or even infinite values etc.).*
- *In maturity-related peak ratios the numerator should contain the thermally more stable compound(s) and the denominator the unstable compound(s), which results in an increase of the ratio with thermal maturity. For example, the ratio $100 \times 27Ts / (27Ts + 27Tm)$ is preferred to the ratios $27Tm/27Ts$ or $27Tm/(27Ts + 27Tm)$.*
- *Peak ratios should not be based on peaks which under certain circumstances coelute (e.g. $29\beta\alpha$), as they may not be comparable depending on the experimental conditions. Ratios such as $30d/29\beta\alpha$ (= "TtX") should therefore be avoided.*

Key references

- *Peters, K.E., Moldowan, J.M. [eds.] (1993): The biomarker guide. Interpreting molecular fossils in petroleum and ancient sediments. 363 pp. Englewood Cliffs, N.J. (Prentice Hall). ISBN 0-13-086752-7.*
- *Philp, R.P. (1985): Fossil fuel biomarkers. Methods in Geochemistry and Geophysics 23. Elsevier, New York, 323 p.*

Figures

See following pages.

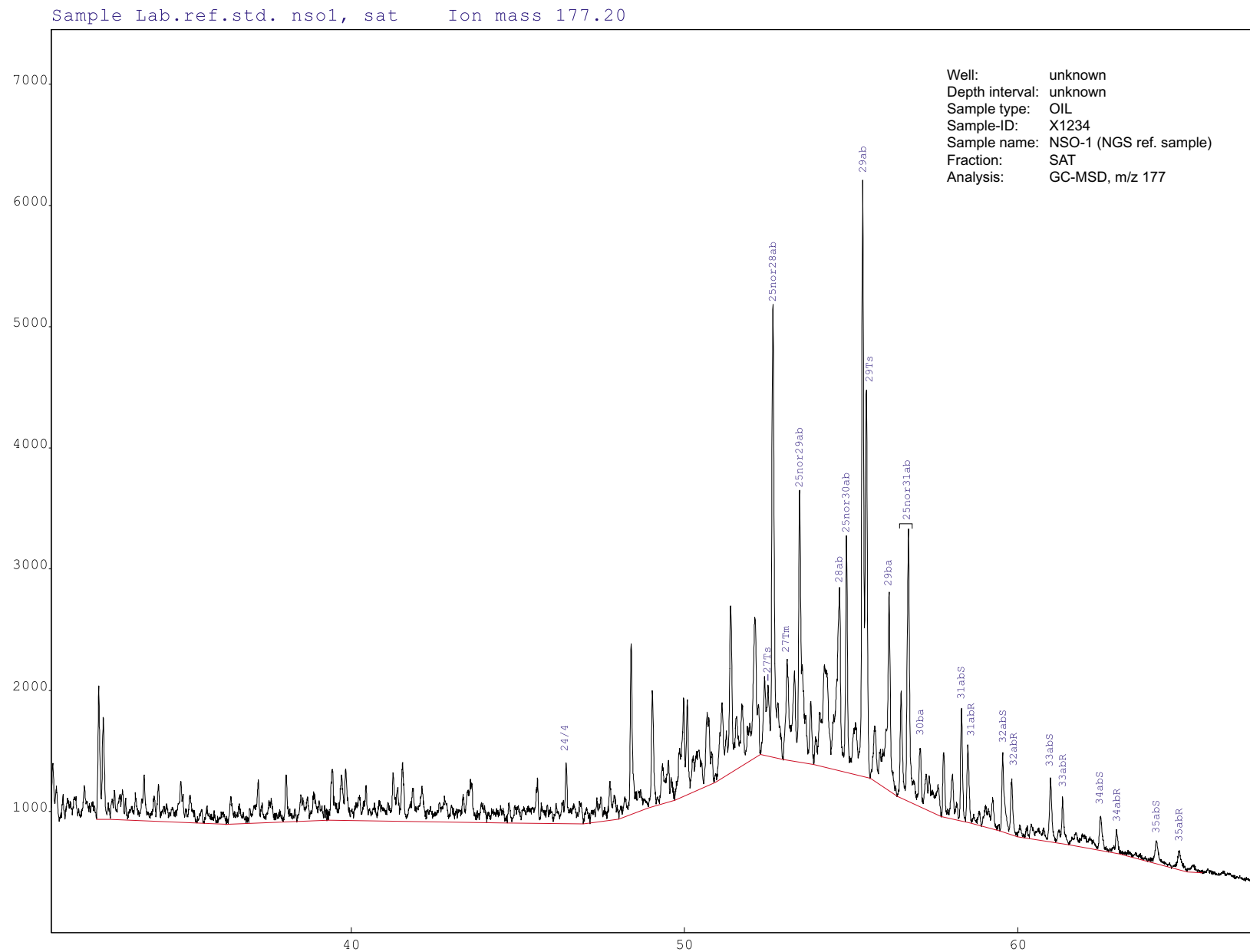


Figure SATGCMS 1 Mass fragmentogram m/z 177 of the saturated hydrocarbon fraction from NGS oil sample NSO-1 with annotated peaks and baseline.

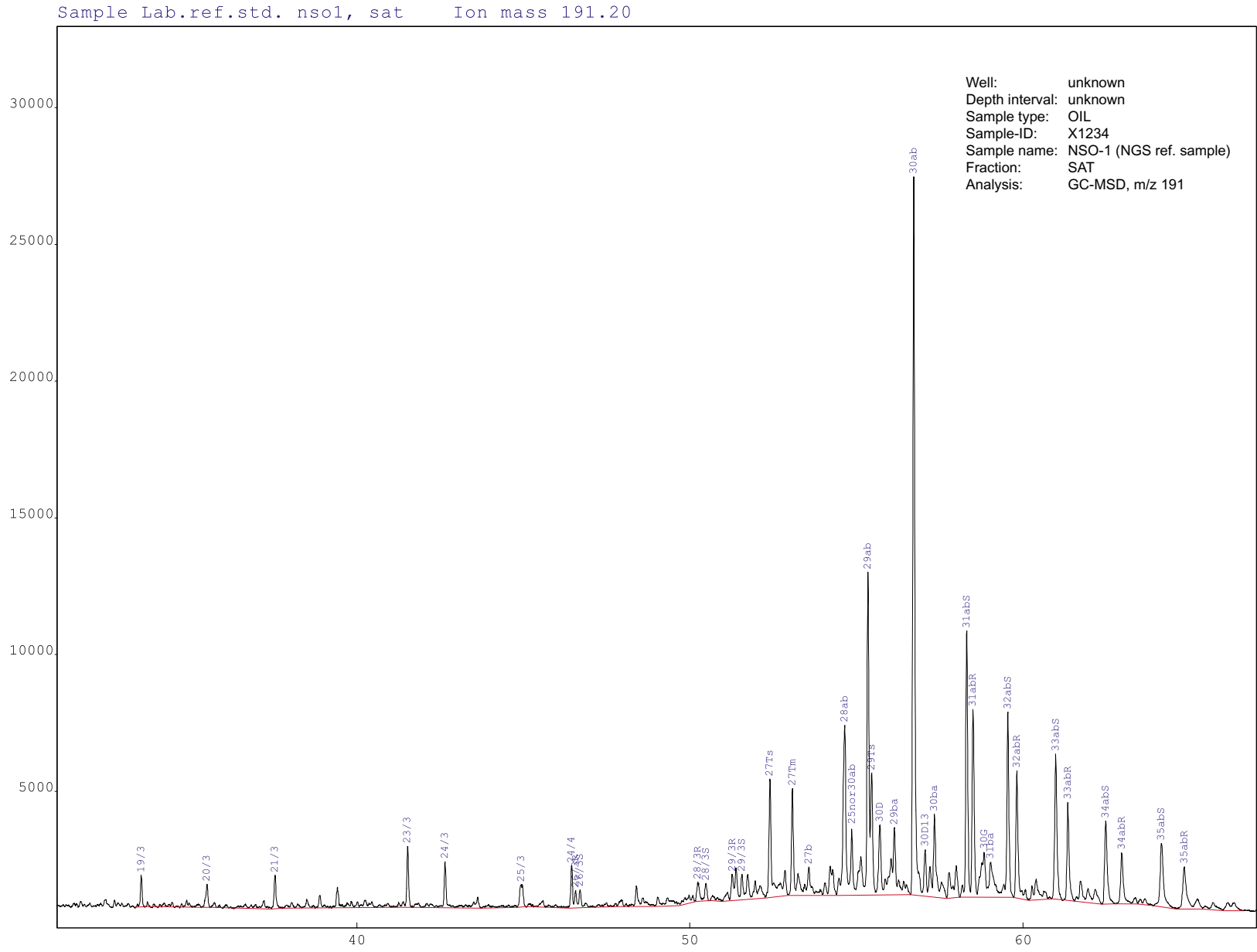


Figure SATGCMS 2 Mass fragmentogram m/z 191 of the saturated hydrocarbon fraction from NGS oil sample NSO-1 with annotated peaks and baseline.

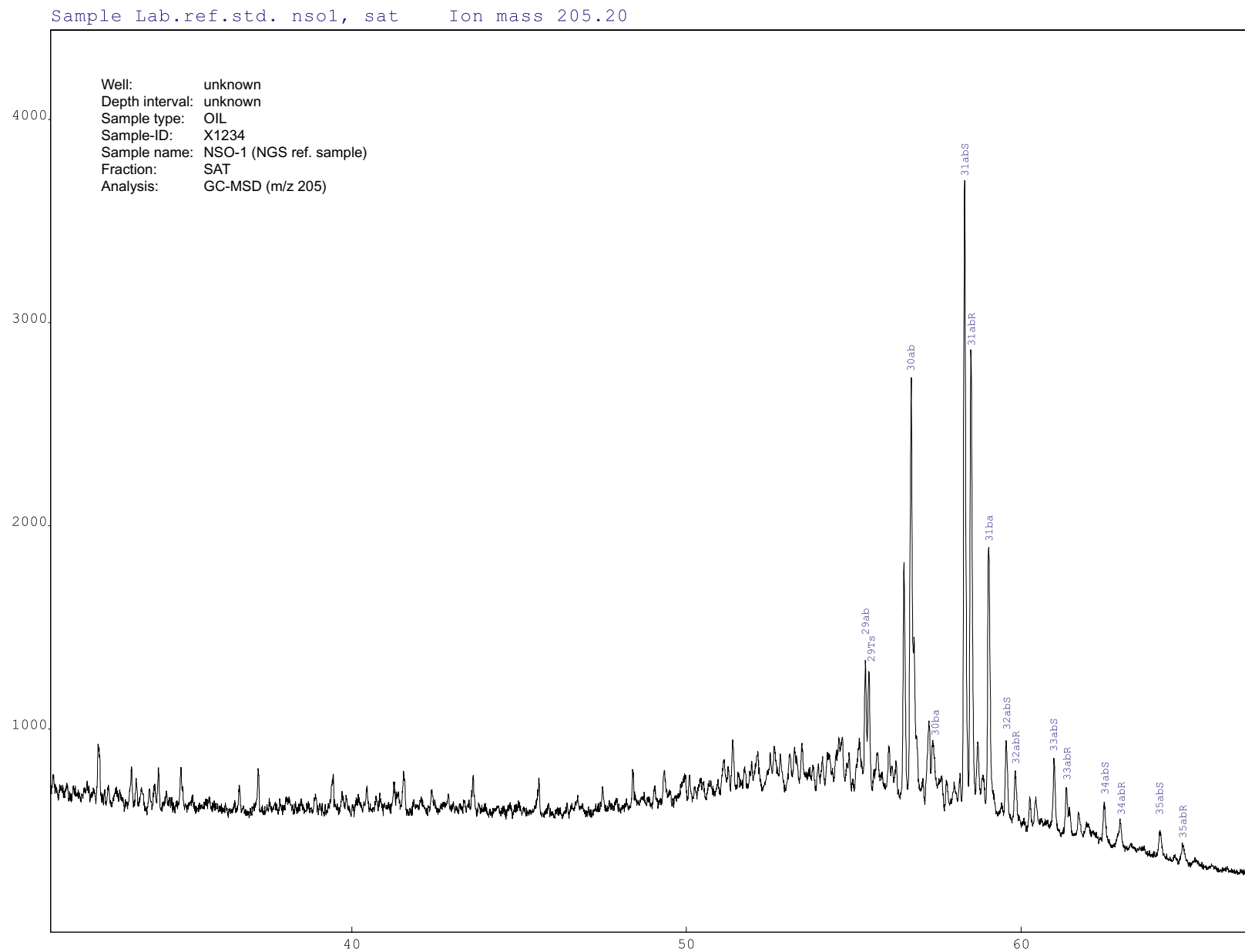


Figure SATGCMS 3 Mass fragmentogram m/z 205 of the saturated hydrocarbon fraction from NGS oil sample NSO-1 with annotated peaks.

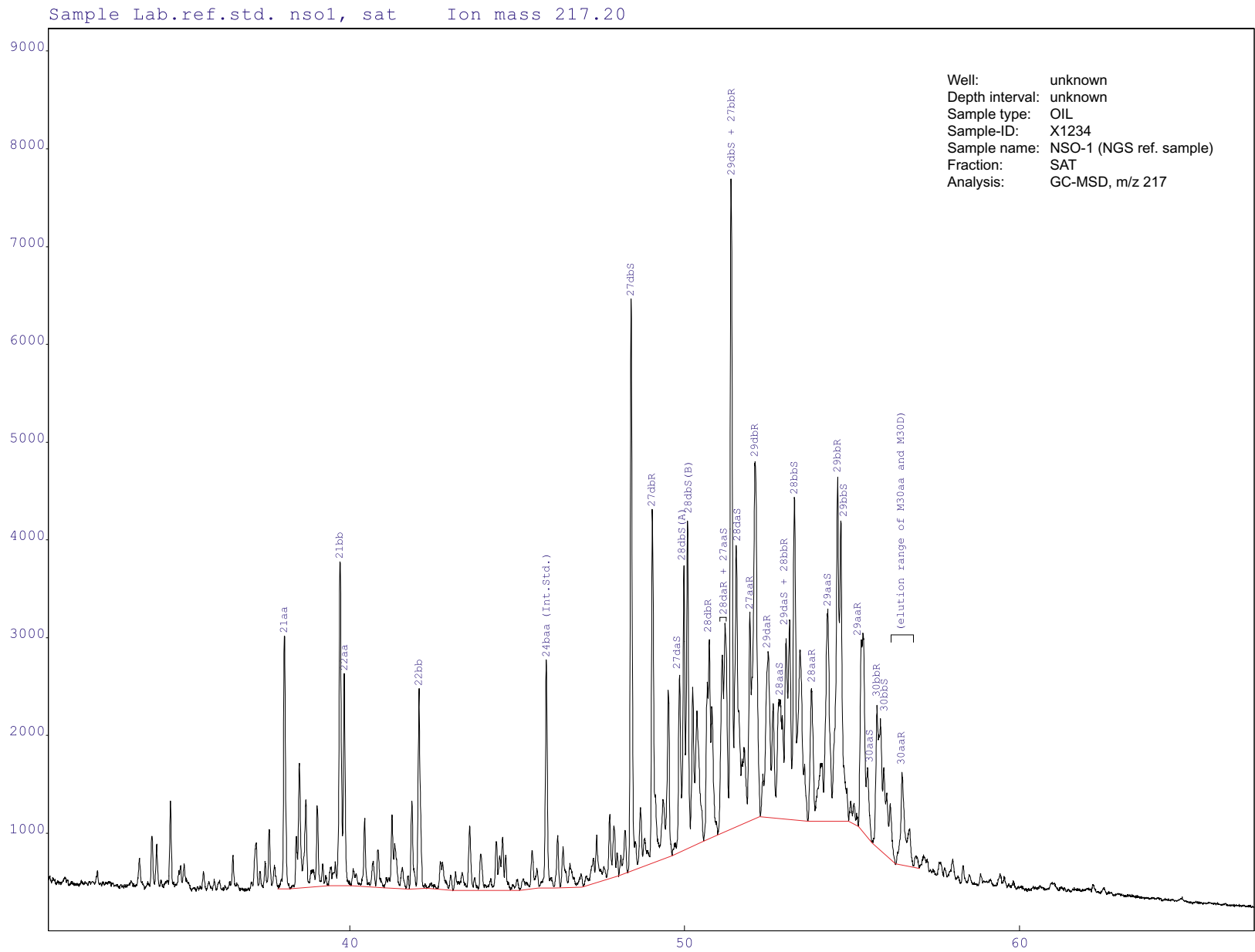


Figure SATGCMS 4 Mass fragmentogram m/z 217 of the saturated hydrocarbon fraction from NGS oil sample NSO-1 with annotated peaks and baseline.

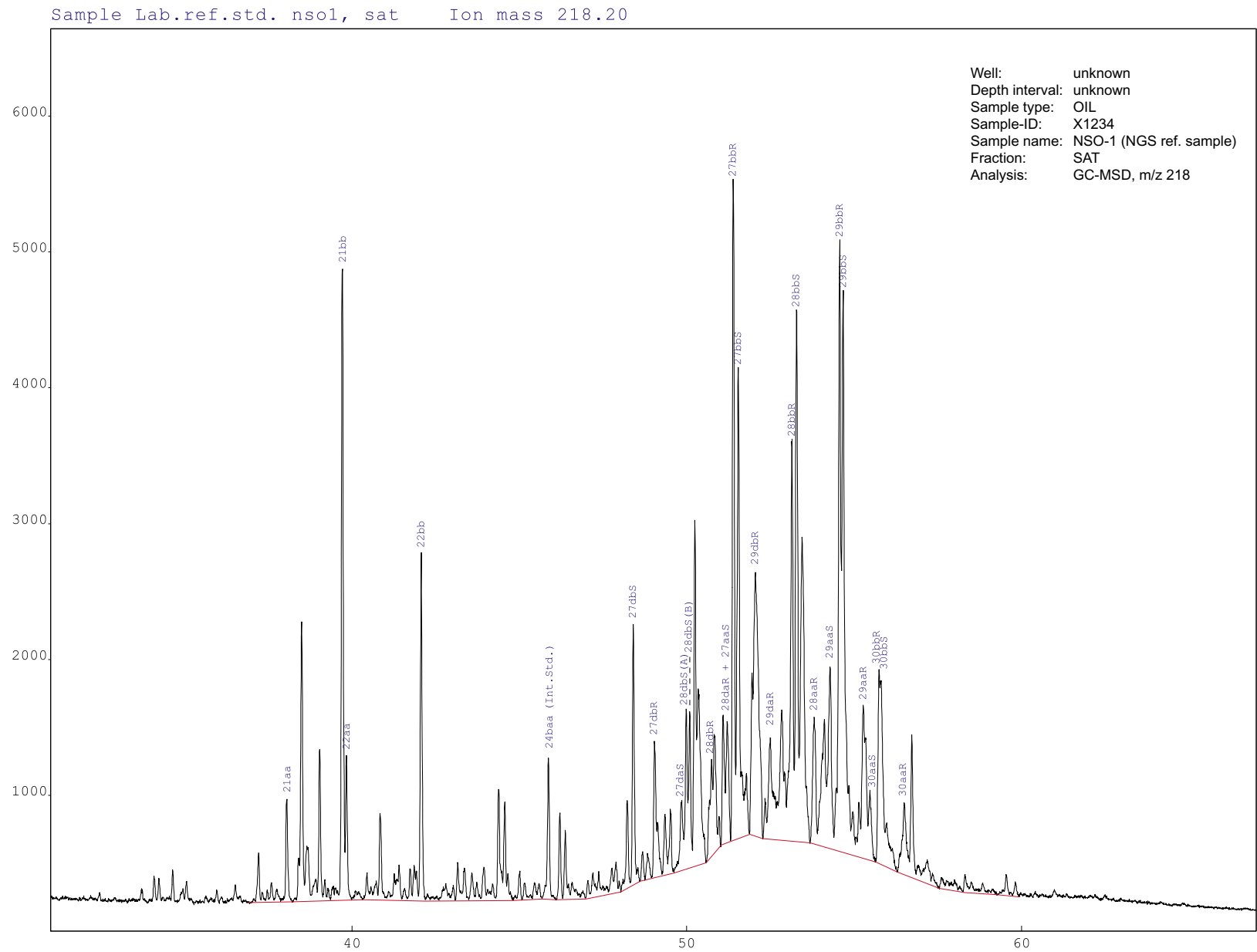


Figure SATGCMS 5 Mass fragmentogram m/z 218 of the saturated hydrocarbon fraction from NGS oil sample NSO-1 with annotated peaks and baseline.

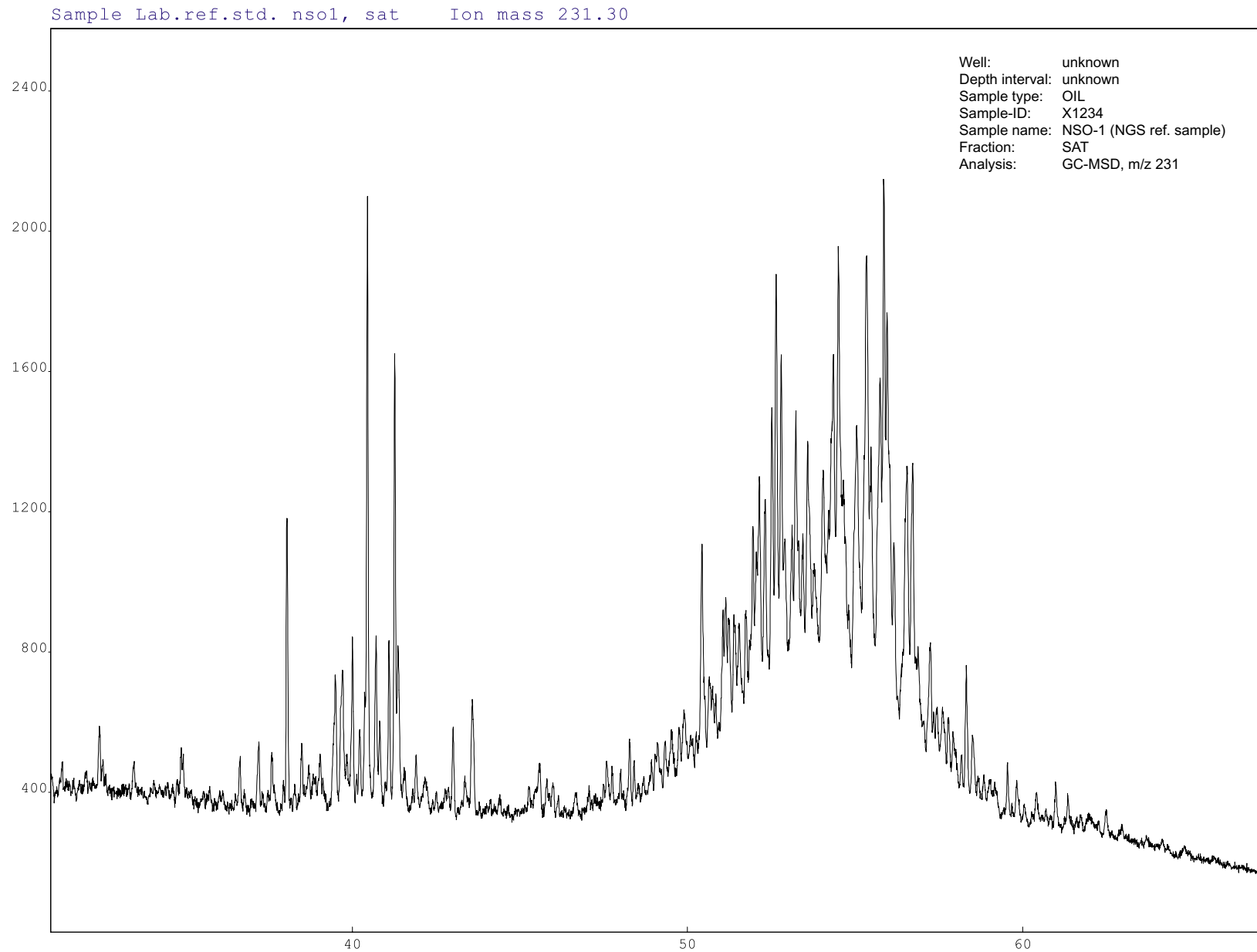


Figure SATGCMS 6 Mass fragmentogram m/z 231 of the saturated hydrocarbon fraction from NGS oil sample NSO-1.

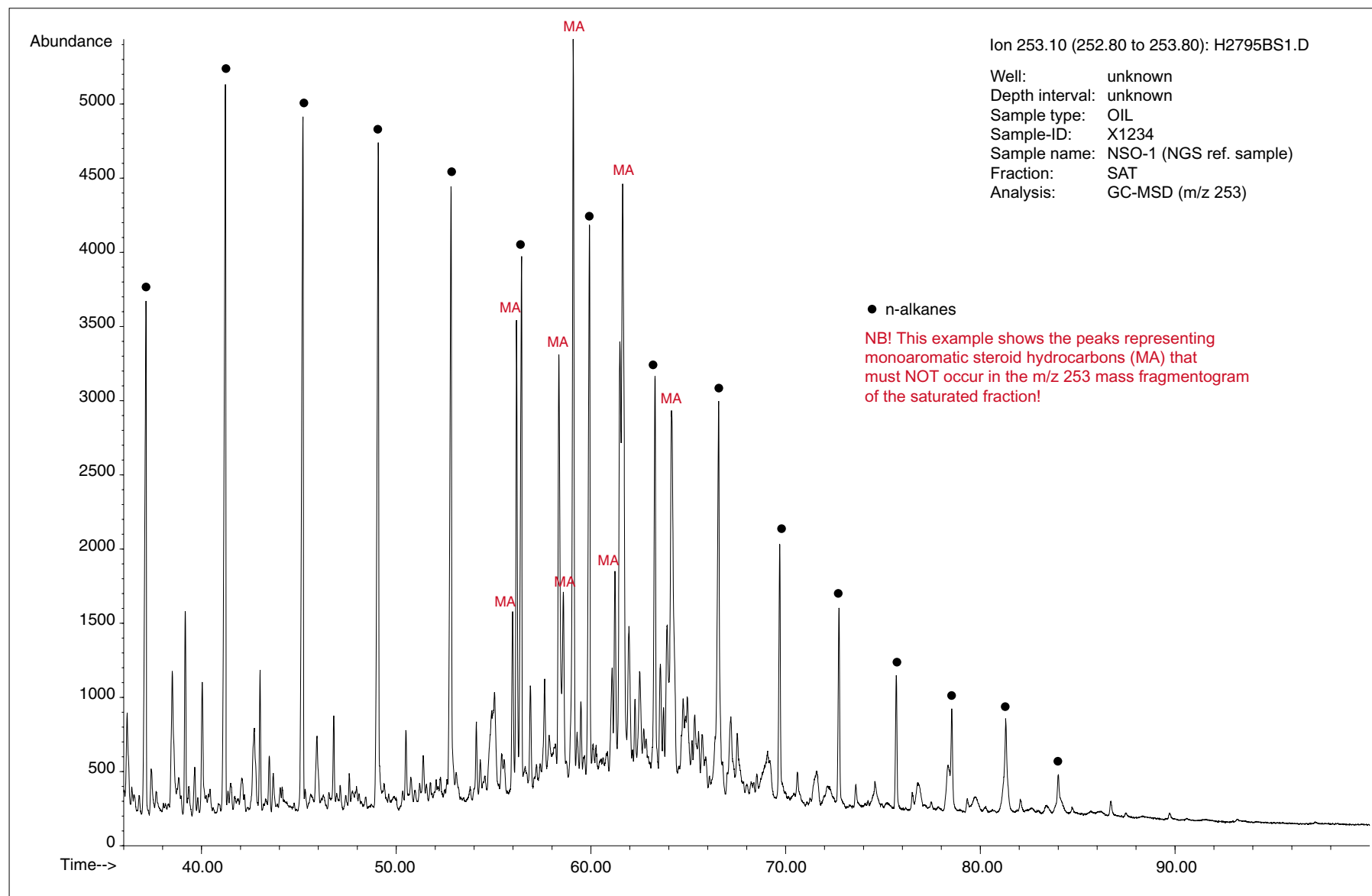


Figure SATGCMS 7 Mass fragmentogram m/z 253 of the saturated hydrocarbon fraction from NGS oil sample NSO-1, demonstrating poor group separation.

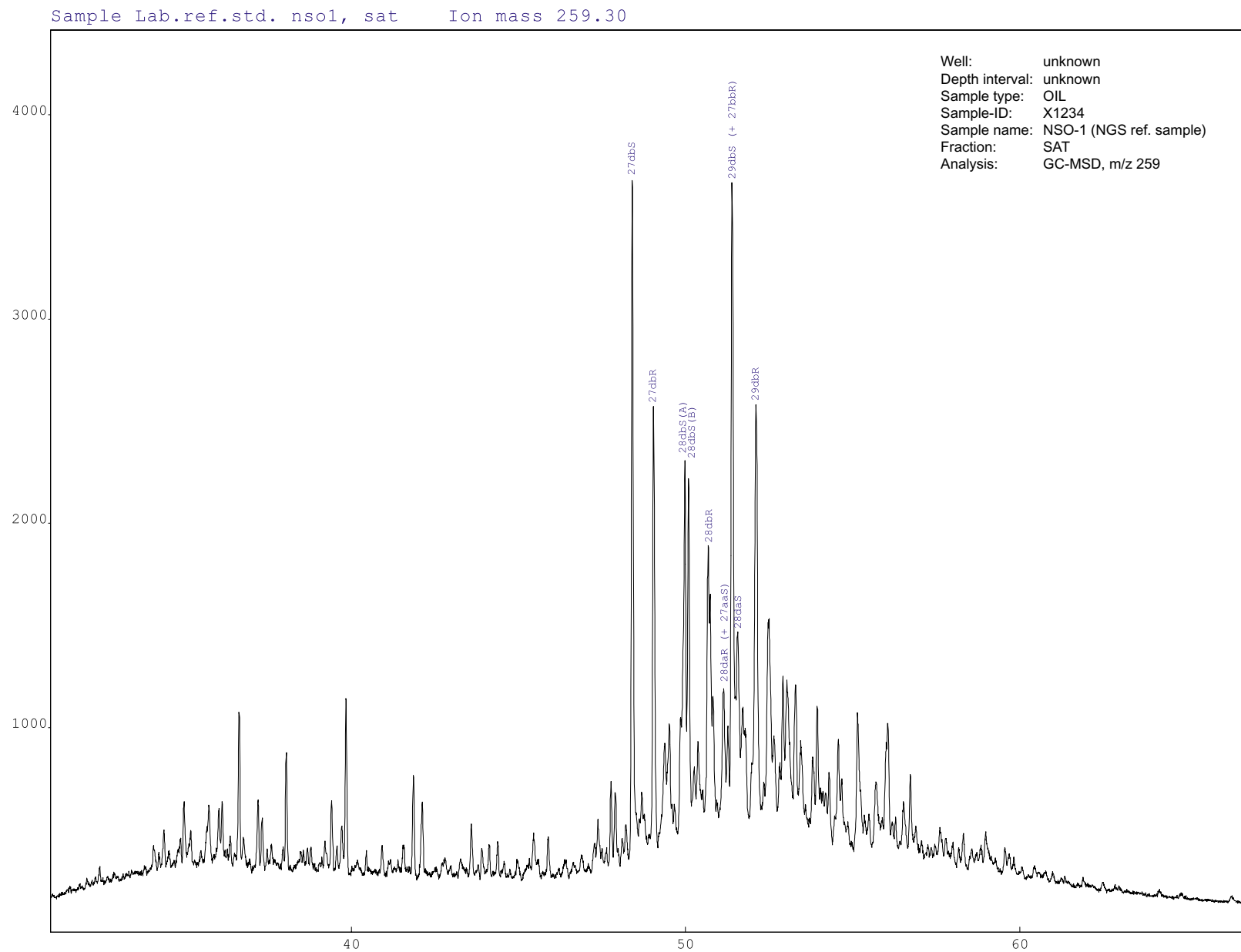


Figure SATGCMS 8 Mass fragmentogram m/z 259 of the saturated hydrocarbon fraction from NGS oil sample NSO-1 with annotated peaks.

GC-MS analysis of oil, EOM or aromatic hydrocarbon fraction

NGS standard applicable.

ARO / EOM GCMS

Purpose, range of application, terminology

- The purpose of this analysis is to obtain quantitative data (i.e. concentrations relative to rock or oil weight) on mono- and triaromatic steroid hydrocarbons ("aromatic biomarkers") and polycyclic aromatic hydrocarbons (PAH), including pure hydrocarbons (naphthalenes, phenanthrenes etc.) and aromatic sulphur compounds (dibenzothiophenes).
- Codes (abbreviations) for aromatic compounds are listed under "Reporting requirements".

Samples to be analysed

- Either the aromatic hydrocarbon fractions of extracts or fluids using low or high resolution MS or the whole (stabilised) fluid or deasphalted extract using high resolution MS.
- The samples must contain an internal standard for quantification (see "[Topping](#)" and "[Solvent extraction of rocks](#)").

Procedural requirements

- At least one NGS reference sample must be analysed together with each contiguous series (batch) of analyses. If more than ten samples are analysed, one NGS reference sample must be included per ten samples.
- The analysis should be performed on a gas chromatograph (GC) fitted with a capillary column of low polarity and coupled with a mass spectrometer (MS) or mass-sensitive detector (MSD).
- If nothing else is specified, the analyses shall be run in selective ion recording (SIR/MID) mode. The use of full-scan (FS), metastable ion monitoring (SMIM/MRM) or tandem mass spectrometry (MS-MS) must be specified or approved by the customer.
- As a minimum the following ion fragments must be monitored:

142.0783	methylnaphthalenes
156.0939	C ₂ -naphthalenes
170.1096	C ₃ -naphthalenes
178.0783	phenanthrene
184.0347	dibenzothiophene
192.0939	methylphenanthrenes
198.0503	methyldibenzothiophenes
206.1096	dimethylphenanthrenes
219.1174	retene (base peak)
231.1174	triaromatic steroids
253.1956	monoaromatic steroids

- In addition, the m/z 253 ion fragment must be monitored in the SAT fraction to ensure that all monoaromatic steroids are contained in the ARO fraction (see [Liquid chromatographic separation of deasphalted oils or rock extracts](#) and [GC-MS analysis of oil, EOM or saturated hydrocarbon fraction](#)).
- For whole extract/fluid all ions listed for both the saturated and aromatic fraction should be monitored (for saturated compounds see [GC-MS analysis of oil, EOM or saturated hydrocarbon fraction](#)).
- Peak heights must be determined using a GC-MS data system (i.e. no manual measurement on paper). The peaks to be selected are specified under "Reporting requirements".

- The integration baseline should follow the top of the unresolved complex mixture (UCM) as shown in [Figure AROGCMS 5](#).
- The biomarker concentrations must be calculated from the peak heights, using the internal standard.

Acceptance criteria and reference samples

- The results from control analyses of the reference samples must be within the following permissible ranges:

NGS oil standard NSO-1

Variable (peak height ratio)	permissible range	most likely value	Comment
1-MP / P	0.53 – 0.70	0.59	m/z 192, 178
A1/E1	0.3 - 0.7	0.5	m/z 253. Not in NGS Newsletters.
a1 / d1	0.2 – 0.4	0.31	m/z 231

NGS rock standard SR-1

Variable (peak height ratio)	permissible range	most likely value	Comment
1-MP / P	0.6 – 0.9	0.7	m/z 192, 178
A1/E1	0.2 - 1.3	1.0	m/z 253. Not in NGS Newsletters.
a1 / d1	0.3 – 1.3	0.8	m/z 231

NGS rock standard JR-1

Variable (peak height ratio)	permissible range	most likely value	Comment
1-MP / P	0.44 – 0.54	0.49	m/z 192, 178
A1/E1	0.3 - 0.9	0.6	m/z 253. Not in NGS Newsletters.
a1 / d1	0.1 – 0.4	0.2	m/z 231

Reporting requirements

- The following technical information must be reported:
 - Method (SIR/MID, MSIM/SMIM/MRM, MS-MS),
 - Resolution (low or high) and
 - Analysed material (fraction or topped / untopped oil or EOM).
- The following variables must be tabulated for all peaks marked with “quantify” in the compound lists below (if present in the fragmentograms):

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Peak identity		x	x
Peak height	height units (usually μV or pA)	x	x
Concentration	ng / g EOM or oil	x	x

- The peak height ratios specified under “Acceptance criteria and reference samples” must be tabulated for valid control analyses of the NGS reference samples.
- All peak ratios referred to in the interpretation must be tabulated, and their calculation explained (formula). Peak ratios are to be based on peak heights.
- Mass fragmentograms for all specified masses and from all analyses must be presented, including those from valid control analyses of reference samples and also those that show no peaks.
- For further reporting requirements see the [Reporting Guide](#), in particular the sections on [Reference figures](#) and [Digital data](#).

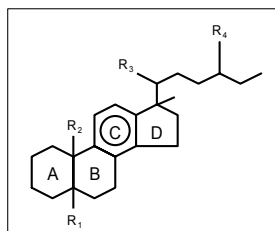
- Compound list:

C-RING MONOAROMATIC STEROID HYDROCARBONS (m/z 253)¹

Peak or peak group	Substituents (see figure below)				Label	Quantify
	R ₁	R ₂	R ₃	R ₄		
A1					C ₂₁ MA	quantify
B1					C ₂₂ MA	quantify
C1	β(H)	CH ₃	S(CH ₃)	H	βSC ₂₇ MA	quantify
	β(CH ₃)	H	S(CH ₃)	H	βSC ₂₇ DMA	quantify
D1	β(CH ₃)	H	R(CH ₃)	H	βRC ₂₇ DMA	quantify
	β(H)	CH ₃	R(CH ₃)	H	βRC ₂₇ MA	quantify
	α(H)	CH ₃	S(CH ₃)	H	αSC ₂₇ MA	quantify
E1	β(H)	CH ₃	S(CH ₃)	CH ₃	βSC ₂₈ MA	quantify
	α(CH ₃)	H	R(CH ₃)	H	αRC ₂₇ DMA	quantify
	β(CH ₃)	H	S(CH ₃)	CH ₃	βSC ₂₈ DMA	quantify
F1	α(CH ₃)	H	S(CH ₃)	CH ₃	αSC ₂₇ DMA	quantify
G1	α(H)	CH ₃	R(CH ₃)	H	αRC ₂₇ MA	quantify
	α(H)	CH ₃	S(CH ₃)	CH ₃	αSC ₂₈ MA	quantify
	β(H)	CH ₃	R(CH ₃)	CH ₃	βRC ₂₈ MA	quantify
	β(CH ₃)	H	R(CH ₃)	CH ₃	βRC ₂₈ DMA	quantify
	β(H)	CH ₃	S(CH ₃)	C ₂ H ₅	βSC ₂₉ MA	quantify
	bCH ₃	H	S(CH ₃)	C ₂ H ₅	βSC ₂₉ DMA	quantify
H1	α(H)	CH ₃	S(CH ₃)	C ₂ H ₅	αSC ₂₈ MA	quantify
	α(H)	CH ₃	R(CH ₃)	CH ₃	αRC ₂₈ MA	quantify
	β(H)	CH ₃	R(CH ₃)	C ₂ H ₅	βRC ₂₉ MA	quantify
	bCH ₃	H	R(CH ₃)	C ₂ H ₅	βRC ₂₉ DMA	quantify
I1	α(H)	CH ₃	R(CH ₃)	C ₂ H ₅	αRC ₂₉ MA	quantify

¹ Not all possible αDMA isomers are listed (rarely present in geological samples).

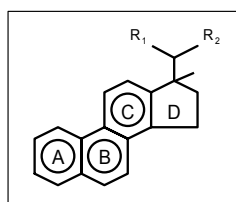
² The monoaromatic steroid groups C1, D1, E1, G1 and H1 can all be present as multiple peaks (see Figure AROGCMS5). These should be quantified individually wherever possible.



Positions of substituents in monoaromatic steroid hydrocarbons

ABC-RING TRIAROMATIC STEROID HYDROCARBONS (m/z 231)

Peak or peak group	Substituents (see figure below)		Label	Quantify
	R ₁	R ₂		
a1	CH ₃	H	C ₂₀ TA	quantify
b1	CH ₃	CH ₃	C ₂₁ TA	quantify
c1	S(CH ₃)	C ₆ H ₁₃	SC ₂₆ TA	quantify
d1	R(CH ₃)	C ₆ H ₁₃	RC ₂₆ TA	quantify
	S(CH ₃)	C ₇ H ₁₅	SC ₂₇ TA	quantify
e1	S(CH ₃)	C ₈ H ₁₇	SC ₂₈ TA	quantify
f1	R(CH ₃)	C ₇ H ₁₅	RC ₂₇ TA	quantify
g1	R(CH ₃)	C ₈ H ₁₇	RC ₂₈ TA	quantify



Positions of substituents in triaromatic steroid hydrocarbons

POLYCYCLIC AROMATIC HYDROCARBONS AND SULPHUR COMPOUNDS

m/z (ion)	Name	Label	Quantify
142	2-Methylnaphthalene	2-MN	quantify
142	1-Methylnaphthalene	1-MN	quantify
156	2-Ethylnaphthalene	2-EN	quantify
156	1-Ethylnaphthalene	1-EN	quantify
156	2,6+2,7-Dimethylnaphthalene	2,6- + 2,7-DMN	quantify
156	1,3+1,7-Dimethylnaphthalene	1,3- + 1,7-DMN	quantify
156	1,6-Dimethylnaphthalene	1,6-DMN	quantify
156	2,3+1,4-Dimethylnaphthalene	2,3- + 1,4-DMN	quantify
156	1,5-Dimethylnaphthalene	1,5-DMN	quantify
156	1,2-Dimethylnaphthalene	1,2-DMN	quantify
170	1,3,7-Trimethylnaphthalene	1,3,7-TMN	quantify
170	1,3,6-Trimethylnaphthalene	1,3,6-TMN	quantify
170	1,3,5+1,4,6-Trimethylnaphthalene	1,3,5- + 1,4,6-TMN	quantify
170	2,3,6-Trimethylnaphthalene	2,3,6-TMN	quantify
170	1,6,7+1,2,7-Trimethylnaphthalene ¹	1,6,7- + 1,2,7-TMN	quantify
170	1,2,6-Trimethylnaphthalene	1,2,6-TMN	quantify
170	1,2,4-Trimethylnaphthalene	1,2,4-TMN	quantify
170	1,2,5-Trimethylnaphthalene	1,2,5-TMN	quantify
178	Phenanthrene	P	quantify
192	3-Methylphenanthrene	3-MP	quantify
192	2-Methylphenanthrene	2-MP	quantify
192	9-Methylphenanthrene	9-MP	quantify
192	1-Methylphenanthrene	1-MP	quantify
206	2-Ethylphenanthrene +9-Ethylphenanthrene +3,6-Dimethylphenanthrene	2-EP+9-EP+3,6-DMP	
206	1-Ethylphenanthrene	1-EP	
206	2,6+2,7+3,5-Dimethylphenanthrene	2,6- + 2,7- + 3,5-DMP	
206	1,3+2,10+3,9+3,10-Dimethylphenanthrene	1,3- + 2,10- + 3,9- + 3,10-DMP	
206	1,6+2,5+2,9-Dimethylphenanthrene	1,6- + 2,5- + 2,9-DMP	
206	1,7-Dimethylphenanthrene	1,7-DMP	
206	2,3-Dimethylphenanthrene	2,3-DMP	
206	1,9+4,9+4,10-Dimethylphenanthrene	1,9- + 4,9- + 4,10-DMP	
206	1,8-Dimethylphenanthrene	1,8-DMP	
219	Retene (= 1-methyl-7-isopropylphenanthrene)	Retene	
184	Dibenzothiophene	DBT	quantify
198	4-Methyldibenzothiophene	4-MDBT	quantify
198	3+2-Methyldibenzothiophene	(3+2)-MDBT	quantify
198	1-Methyldibenzothiophene	1-MDBT	quantify

¹ 1,6,7- = 2,3,5-TMN

Recommendations and notes

- Recommended column types are SE-54 (40m), DB1 (50m) or similar. A recommended temperature programme is as follows: Start at 50°C, 35°C/min to 150°C, 2°C/min to 310°C, isothermal at 310°C for 20 min. Higher start temperature may lead to significant peak tailing.
- If possible and appropriate, major and/or geochemically significant peaks in addition to those specified under "Reporting requirements" should also be identified, and their heights and concentrations should be reported.
- Peak ratios should preferably be of the type $100 \times a / (a + b)$ [%]. This saves space (as the redundant "0." of fractions is avoided) and avoids the problems connected with ratios of the type a/b (division by zero, very large or even infinite values etc.).
- In maturity-related peak ratios the numerator should contain the thermally more stable compound(s) and the denominator the unstable compound(s), which results in an increase of the ratio with thermal maturity.

Key references

- Kvalheim, O.M., Christy, A.A., Telnæs, N., Bjørseth, A. (1987): *Maturity determination of organic matter in coals using the methylphenanthrene distribution. Geochimica et Cosmochimica Acta* 51, 1883-1888. [Definitions of F1 and F2].
- Peters, K.E., Moldowan, J.M. [eds.] (1993): *The biomarker guide. Interpreting molecular fossils in petroleum and ancient sediments. 363 pp. Englewood Cliffs, N.J. (Prentice Hall). ISBN 0-13-086752-7. [techniques, applications, practical hints, many references]*
- Radke, M. (1987): *Organic geochemistry of aromatic hydrocarbons. In: Brooks, J., Welte, D. (eds.): Advances in Petroleum Geochemistry, Vol. 2, 141-207, London etc. (Academic Press). [with references to relevant earlier work on aromatic hydrocarbon maturity parameters]*

Figures

see next page

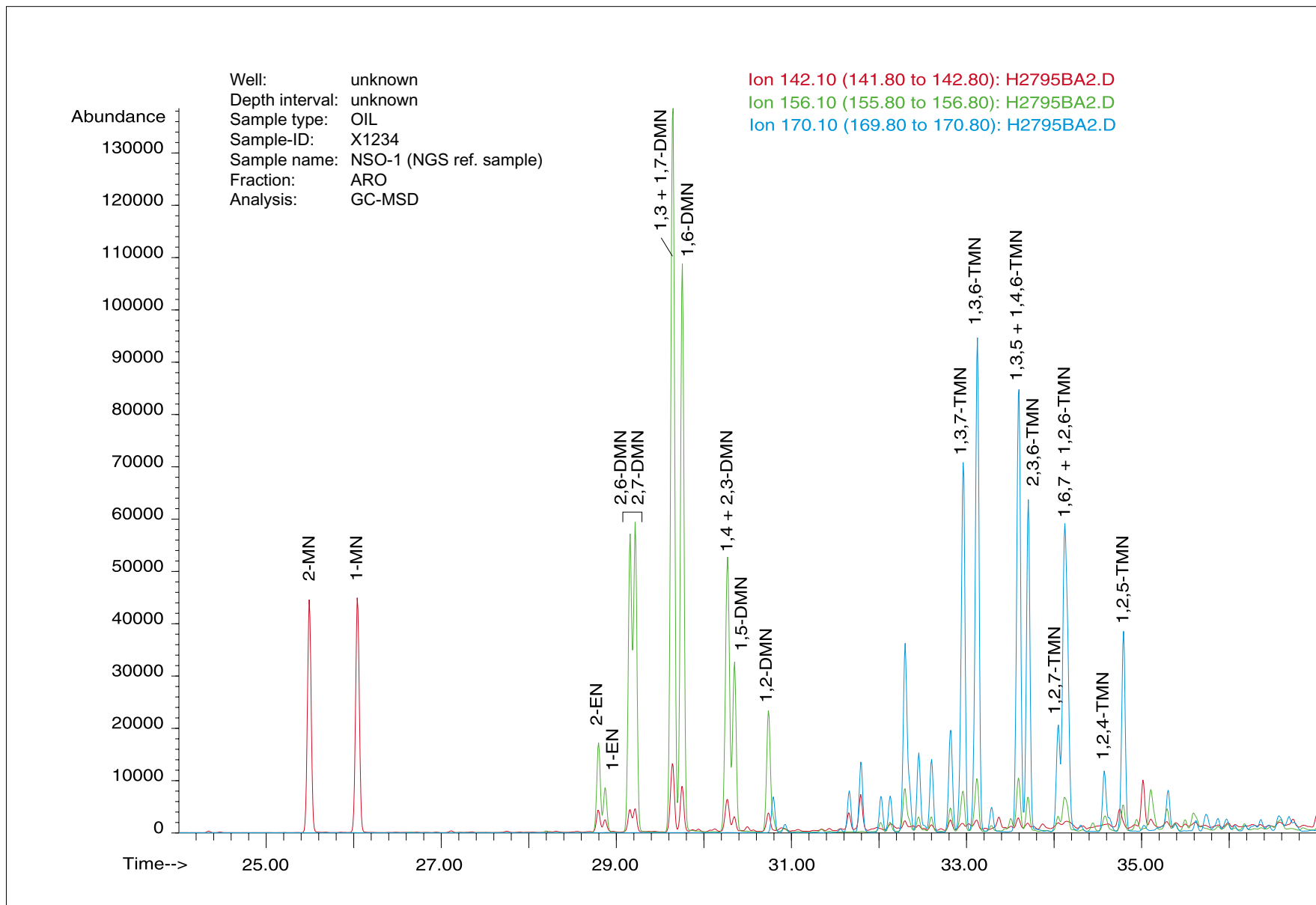


Figure AROGCMS 1 Mass fragmentograms m/z 142, 156 and 170 of the aromatic fraction from NGS oil sample NSO-1 with annotated peaks.

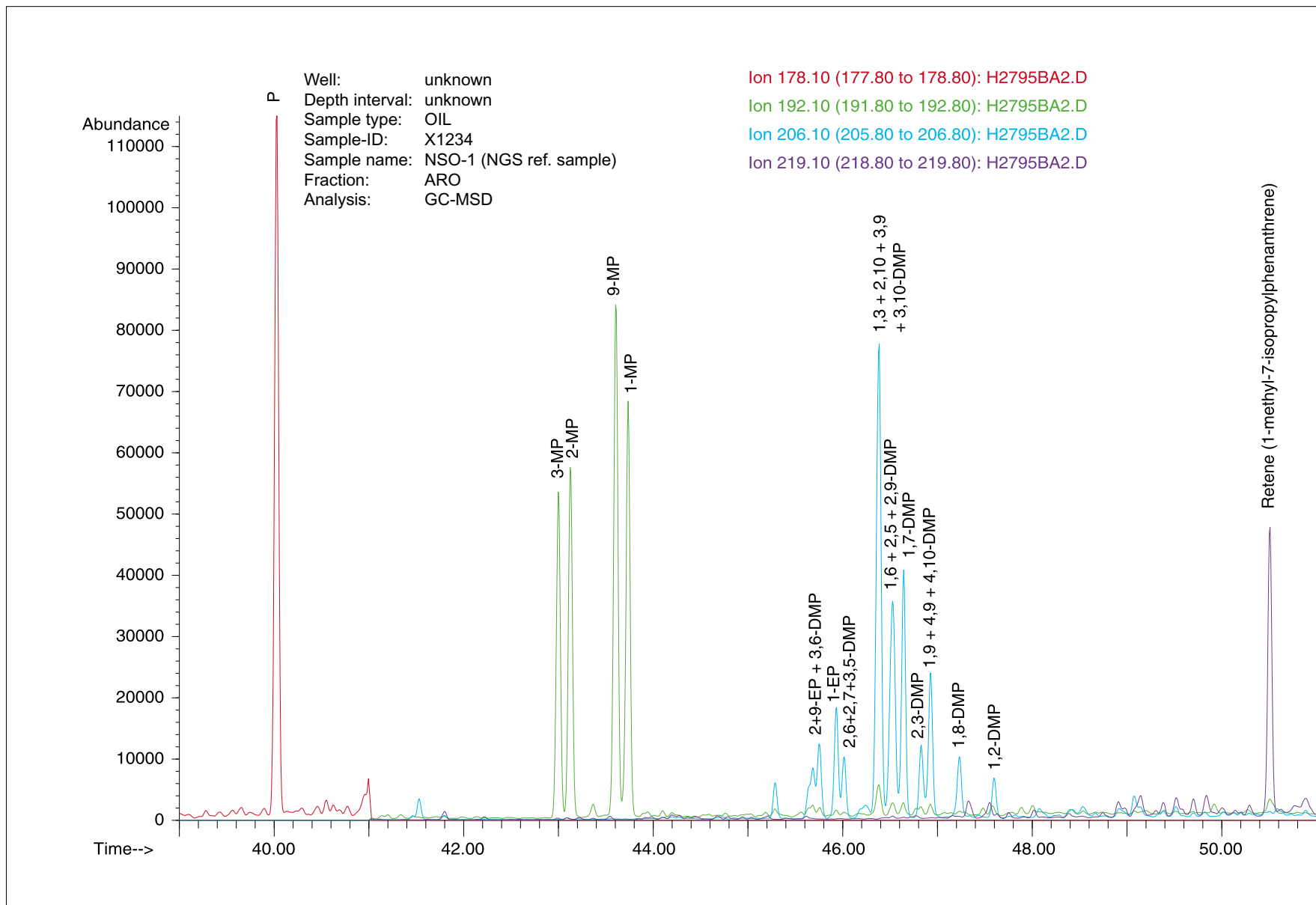


Figure AROGMS 2 Mass fragmentograms m/z 178 and 192 of the aromatic fraction from NGS oil sample NSO-1 with annotated peaks.

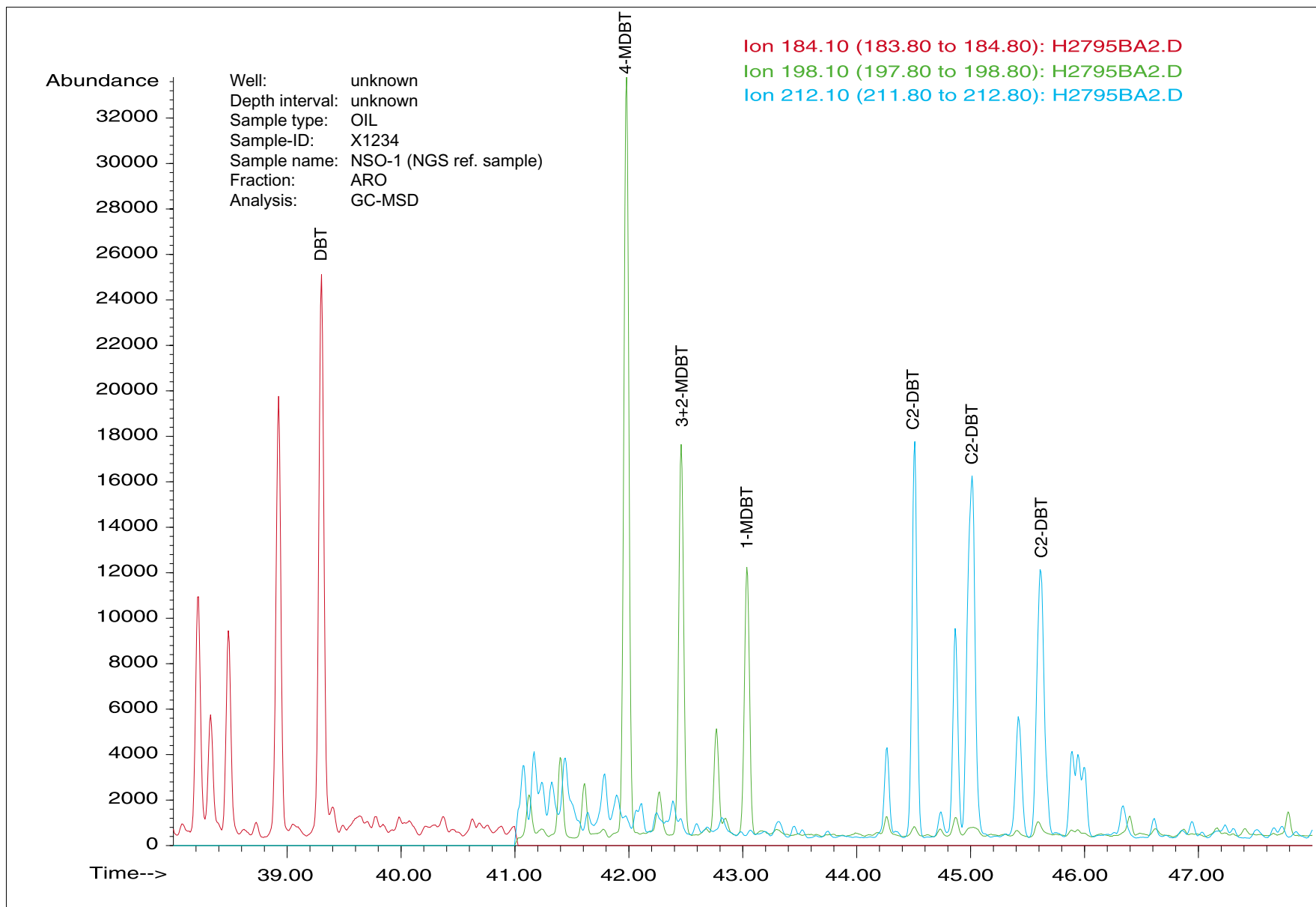


Figure AROGCMS 3 Mass fragmentograms m/z 184 and 198 of the aromatic fraction from NGS oil sample NSO-1 with annotated peaks.

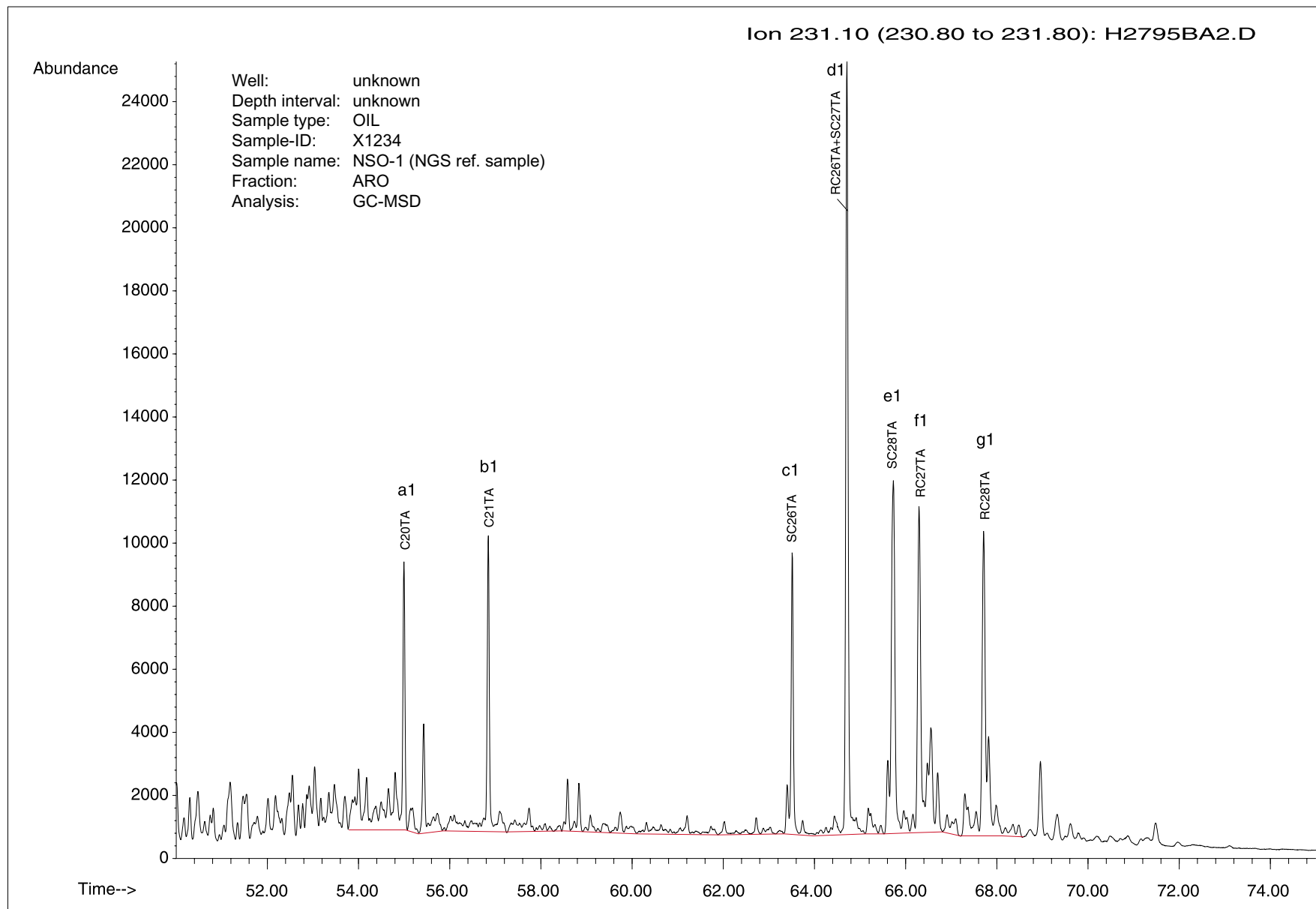


Figure AROGCMS 4 Mass fragmentogram m/z 231 of the aromatic fraction from NGS oil sample NSO-1 with annotated peaks.

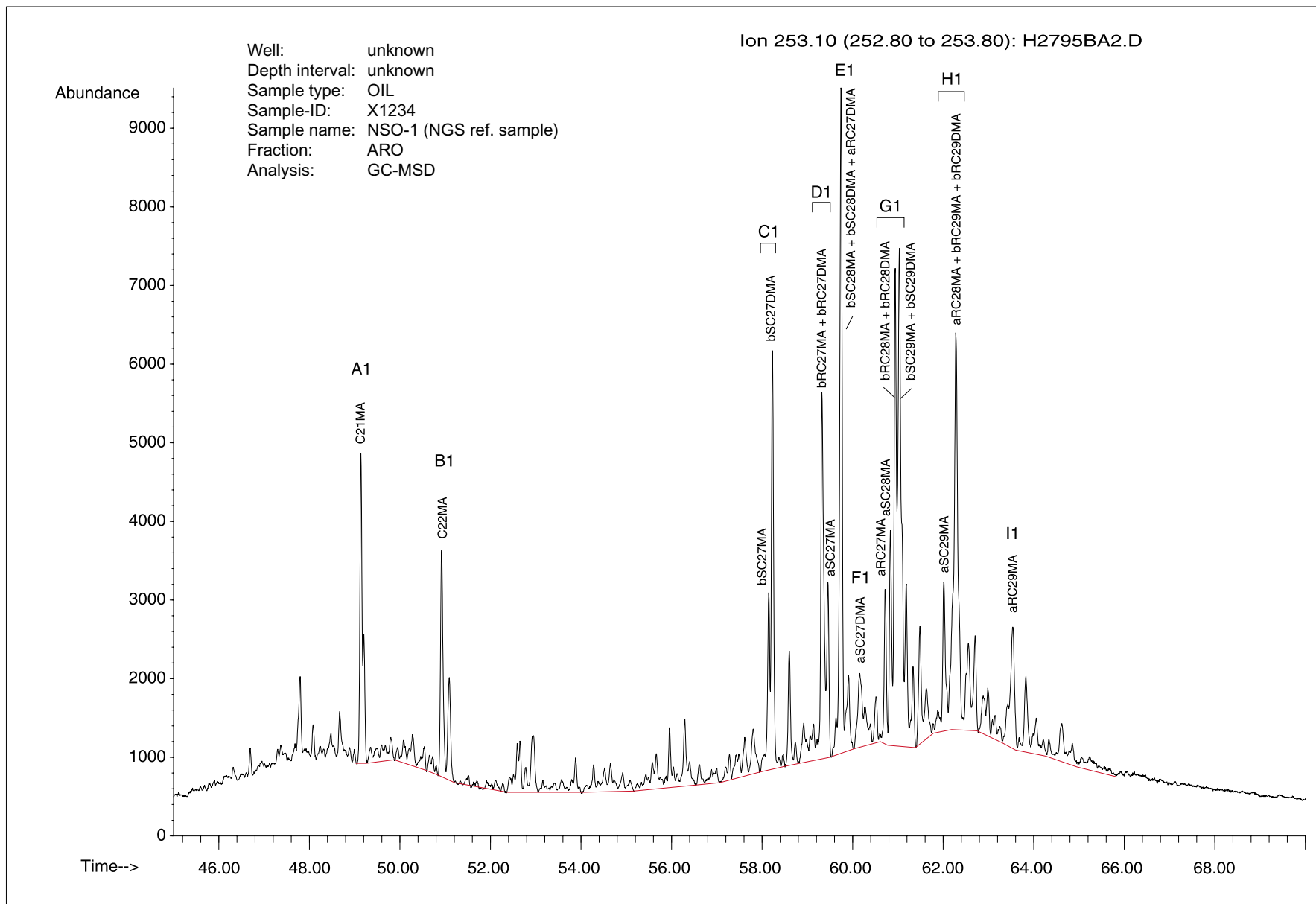


Figure AROGCMS 5 Mass fragmentogram m/z 253 of the aromatic fraction from NGS oil sample with annotated peaks.

Gas-chromatography isotope-ratio mass-spectrometry (GC-IRMS) analysis

No NGS standard applicable.

GC-IRMS

Purpose, range of application, terminology

- The purpose of GC-IRMS analysis is to obtain stable carbon isotope data for individual compounds in liquids or gases, mainly for correlation.

Samples to be analysed

- Liquids (whole topped / untopped oils, SAT HC, ARO HC, and branched/cyclic and n-alkane fractions) and gases. Internal standards such as deuterated n-alkanes of known isotopic composition can be employed, but normally the reference gas and calibration with the n-alkane standard is sufficient.

Procedural requirements

- Currently no official procedure is defined, but the recommendations below should be followed, if possible.

Acceptance criteria and reference samples

- Currently no official acceptance criteria are defined, but the recommendations below should be followed, if possible.

Reporting requirements

- Currently no particular reporting requirements are defined for this analysis. For general reporting requirements see the [Reporting Guide](#), in particular the sections on [Reference figures](#) and [Digital data](#).

Recommendations and notes

- *Equipment and procedure:*

The GC should be fitted with a column that leads directly into the combustion furnace using helium as carrier gas. For analysis of liquids, a fused silica column of 50 m or more should be used, with a non-polar phase such as OV1 or DB1. These analyses are carried out mainly in split mode. For analysis of gases, the GC should be fitted with a Poraplot Q column of 25 m or more (length depending on whether light or gasoline range hydrocarbons are being analysed). These analyses are carried out in split or splitless mode.

The major beam current should be $>10^{-8}$ A in order to avoid drifting of the results over time.

A CO₂ reference gas must be introduced in series of pulses before and after the array of chromatogram peaks of interest. This reference gas should be calibrated against the NBS 22 oil international standard. In addition, the instrument should be calibrated against an alkane standard each day. Duplicate analyses of the n-alkane standard should be performed at least each 10th sample. Duplicate values need not be reported.

Background subtraction must be used and care must be taken to select the correct baseline before this background subtraction is made. The background subtraction value should be fairly stable for any one sample (variation $< \pm 10\%$).

- *Acceptance criteria and reference samples:*

The reference CO₂ gas pulses in the zero run should have a reproducibility equal to or better than 9.0×10^{-7} . Results from the methane standard should have an accuracy of ± 0.5 ‰ PDB and for the n-alkane standard of better than ± 0.3 ‰ PDB.
- *Reporting:*

The carbon isotope data for each analysed compound should be tabulated. Due to the complex nature of the types of hydrocarbons analysed, a single standard table is not always practicable. However, compounds belonging to a common group (e.g. n-alkanes, branched and cyclic alkanes, etc.) should be tabulated together.
- *Information on reference materials for stable isotope analysis (such as the NBS 22 oil) is available e.g. at the Isotope Hydrology Laboratory of the International Atomic Energy Agency (IAEA), http://www.iaea.org/programmes/rial/pci/isotopehydrology/reference_materials.htm, e-mail stabiso@refmat.iaea.org.*

Key references

- Bjorøy, M., Hall, K., Jumeau, J. (1990): Stable carbon isotope ratio analysis on single components in crude oils by direct gas chromatography-isotope analysis. *Trends in Analytical Chemistry* 9, 331-337.
- Hayes, J.M, Freeman, K.H., Popp, B.N. Hoham, C.H. (1990): Compound-specific isotopic analyses: A novel tool for reconstruction of ancient biogeochemical processes. *Organic Geochemistry* 16, 1115-1128 [= *Advances in Organic Geochemistry* 1989].
- Matthews, D.E., Hayes, J.M. (1978): Isotope-ratio-monitoring gas chromatography mass spectrometry. *Analytical Chemistry* 50, 1465-1473.

Figures

See next page.

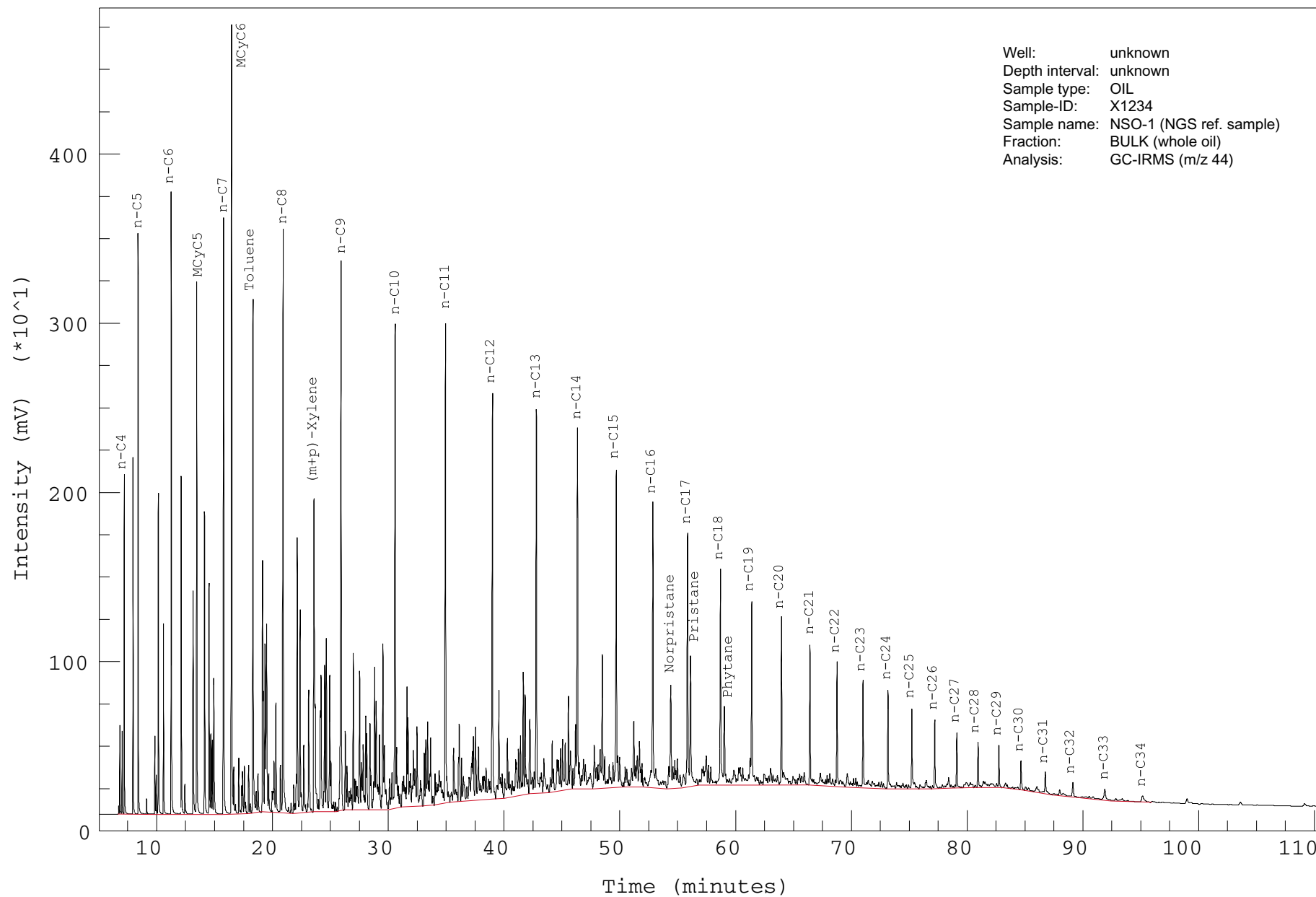


Figure GCIRMS 1 Mass fragmentogram m/z 44 of NGS oil sample NSO-1 with annotated peaks: Whole oil.

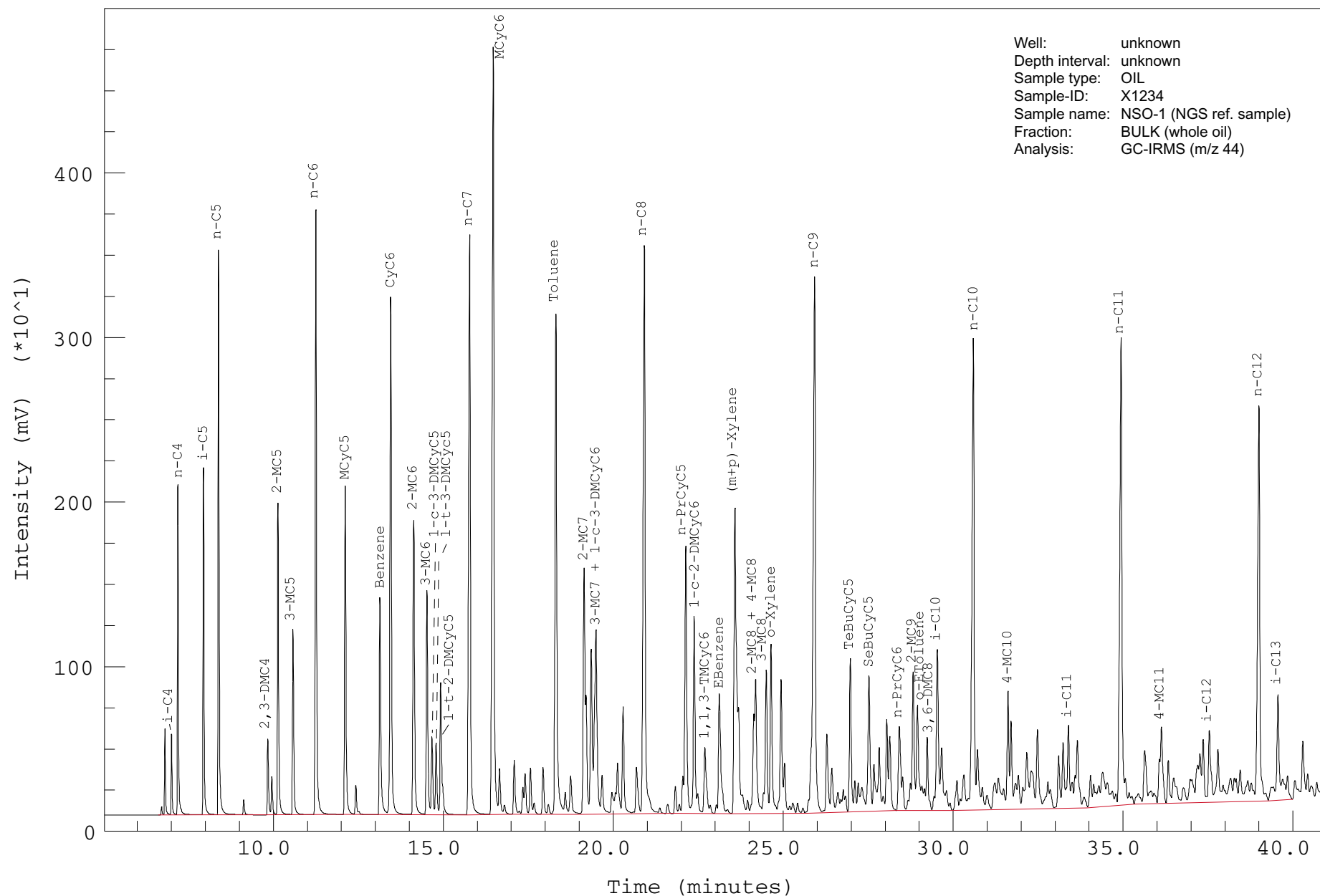


Figure GCIRMS 2 Mass fragmentogram m/z 44 of NGS oil sample NSO-1 with annotated peaks: Expanded gasoline range view.

GC analysis of natural gas

No NGS standard applicable.

GAS GC

Purpose, range of application, terminology

- The purpose is to determine the molecular (or volume) composition of natural gases.
- This analysis is typically combined with stable isotope analysis of gas compounds (see [Stable carbon isotope analysis of gas compounds](#)).

Samples to be analysed

- Natural gas in pressurised cylinder.

Procedural requirements

- The gas samples must be separated by gas chromatography and analysed using FID and/or TLC detection.
- The concentration (by volume of the analysed gas) of at least the following hydrocarbon and non-hydrocarbon components in the gas must be determined: C₁, C₂, C₃, i-C₄, n-C₄, C₅₊, CO₂.
- The concentrations must be determined based on peak areas, using a commercially available calibration gas with appropriate composition ("natural gas standard") as an external standard.
- Calibration must be carried out at least once a day. Control analyses of the standard gas must be run at least three times a day. If the results (peak areas) from the control analyses deviate by more than 5% from the previous calibration, a new calibration must be carried out.

Acceptance criteria and reference samples

(none, except for the acceptable variation of the gas standard control analyses specified under "Procedural requirements")

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Concentrations of C ₁ , C ₂ , C ₃ , i-C ₄ , n-C ₄ , C ₅₊ and CO ₂	volume % of reported compounds	x	x
Concentrations of Sum of C ₁ to n-C ₄ , Sum of C ₂ to n-C ₄	volume % of reported compounds	x	
Wetness = 100 * [Sum of C ₂ to n-C ₄] / [Sum of C ₁ to n-C ₄]	volume %	x	
i-C ₄ / n-C ₄	volume ratio	x	

- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

(none)

Key references

- Osjord, E.H., Malthe-Sørenssen, D. (1983): Quantitative analysis of a natural gas in a single run by the use of packed and capillary columns. *Journal of Chromatography* 297, 219-224.
- Walters, C.C., Hellyer, C.L. (1998): Multi-dimensional gas chromatographic separation of C₇ hydrocarbons. *Organic Geochemistry* 29, 1033-1041.

Figures

(none)

Stable isotope analysis of gas compounds

No NGS standard applicable.

GAS ISO

Purpose, range of application, terminology

- The purpose is to determine stable carbon (and optionally hydrogen and oxygen) isotope ratios of individual compounds in natural gases.
- This analysis is typically combined with the determination of the molecular composition of the gas (see [GC analysis of natural gas](#)).

Samples to be analysed

- Natural gas, headspace, occluded, adsorbed gas.

Procedural requirements

- The gas samples must first be separated by gas chromatography for (1) quantification of the hydrocarbon and non-hydrocarbon components (see [GC analysis of natural gas](#)) and (2) physical separation and collection of the individual gases prior to isotopic analysis.
- The individual gas compounds must be oxidised in separate CuO ovens in order to prevent cross-contamination.
- The combustion products, CO₂ and H₂O must be frozen into collection vessels and separated by fractional distillation. H₂O must be further reduced in sealed quartz tubes in order to generate hydrogen for analysis.
- The analytical system must be calibrated using an international standard and regularly controlled using a laboratory natural gas standard.
- GC-IRMS is suggested as an alternative method for gas isotope analysis of samples with low hydrocarbon concentrations. It is important to calibrate both analytical methods with the same natural gas standard in order to ensure that results are compatible. GC-IRMS does not permit determination of δD for methane and $\delta^{18}O$ for CO₂. The use of GC-IRMS requires approval from the customer.

Acceptance criteria and reference samples

- It is recommended to use an international natural gas standard (IAEA-NGS standards⁴) for calibration of the system.

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Carbon isotope ($\delta^{13}C$) values for C ₁ , C ₂ , C ₃ , i-C ₄ , n-C ₄ and, if requested, CO ₂	‰ PDB	x	x
Hydrogen isotope (δD) value for C ₁	‰ SMOW	x	x

- The isotopic values and standard deviation for replicate analyses of the laboratory gas standard must be quoted.
- For general reporting rules see the [Reporting Guide](#).

⁴ Information on these standards is available at <http://silicon.nist.gov/outputs/ngs.html>.

Recommendations and notes

- *A reference gas (CO₂, H₂) is always used for stable isotope determinations. The reference gas has to be calibrated with calibration standards, for instance NBS 22 oil and a water standard (GISP or SMOW) from IAEA. Information on these reference materials is available at the Isotope Hydrology Laboratory of the International Atomic Energy Agency (IAEA), http://www.iaea.org/programmes/rial/pci/isotopehydrology/reference_materials.htm e-mail stabiso@refmat.iaea.org.*
- *It is recommended to quote the isotopic values for the calibration standards in the report.*

Key references

(none)

Figures

(none)

Kerogen analyses

Thermal extraction and temperature-programmed pyrolysis-GC

No NGS standard applicable.

TEGC/PYGC

Purpose, range of application, terminology

- TEGC = Thermal extraction - gas chromatography, PYGC = temperature-programmed (open-system) pyrolysis-gas chromatography.
- The main purpose of this analysis is to obtain a fingerprint of the pyrolysate for characterisation of hydrocarbon potential and kerogen type. TEGC can be carried out as a preparative stage for non-solvent-extracted samples to be analysed by PYGC, or in its own right for characterisation of oil stain. The customer therefore must specify (1) whether or not the samples should be thermally extracted, (2) whether TE-GC data should be reported and (3) whether PY-GC shall be carried out.

Samples to be analysed

- Source rock pyrolysis can be performed on pre-extracted (i.e. solvent-extracted) or non-extracted material.

Procedural requirements

- **Thermal extraction (S1)(if requested):** Samples which have not been solvent-extracted, must be thermally extracted. The thermal extract, which corresponds to the S1 peak in Rock-Eval pyrolysis, is obtained by heating the pyrolysis chamber at 330 °C for 4 minutes. If requested, the thermal extract must be analysed on a gas chromatograph fitted with a capillary column of low polarity and a flame ionisation detector (FID).
- **Pyrolysis GC (S2)(if requested):** The pyrolysis temperature programme should be as follows: initial temperature 330 °C, 25 °C/min to 600 °C, isothermal for 3 minutes at 600 °C. The pyrolysate, which approximately corresponds to the S2 peak in Rock-Eval pyrolysis, must be analysed on a gas chromatograph fitted with a capillary column and a flame ionisation detector (FID).
- A blank analysis must be carried out prior to the sample analyses.
- The NGS rock standard JR-1 must be analysed at least once with every batch of samples and after each tenth sample. Although no most likely values or permissible ranges have been defined for this reference sample, the chromatograms are required as a qualitative basis for assessment of instrument stability and comparison of results obtained during different analysis series.
- Quantification of the pyrolysis-gas chromatogram must be carried out as follows:
 - (1) Subtract the blank run from the chromatogram, using the data system.
 - (2) Draw the baseline on the blank-subtracted chromatogram as a horizontal line from the beginning to the end of the chromatogram, in order to include the unresolved complex mixture (UCM) in the quantification of the groups. Determine the total peak areas of the following carbon number ranges: C₁, C₂-C₅, C₆-C₁₄, C₁₅₊, where e.g. C₆-C₁₄ means the retention time window between the trailing end of the n-C₅ alkane peak and the trailing end of the n-C₁₄ alkane peak, as shown in [Figure PYGC 1](#). Record these peak areas and calculate the

percentages.

(3) Draw the baseline on the blank-subtracted chromatogram on top of the UCM and determine the total peak areas (excluding the UCM) for the carbon number intervals mentioned above.

(4) Determine the peak areas (excluding the UCM) for the following individual peaks: n-heptene, toluene, n-octene, (m+p)-xylene.

Acceptance criteria and reference samples

- Methane and ethane must be separated to the baseline.
- O-Xylene and n-nonene must be separated.
- NGS reference samples are not strictly applicable due to the lack of quantitative quality criteria, but control analyses of the JR-1 reference rock must be documented for qualitative comparison. Also see "Recommendations and notes".

Reporting requirements

(1) TEGC data (if requested):

- The chromatogram must be included in the report.
- If present, at least the following peaks must be identified and quantified in at least one TEGC chromatogram: all identified n-alkanes, pristane, phytane.
- If possible, the following parameters must be tabulated for the peaks listed under "Procedural requirements":

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Peak identity		x	x
Peak area	area units (usually μVs)	x	x

(2) PYGC data (if requested):

- The chromatogram must be included in the report.
- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Peak area percentages ($100 \cdot \text{peak area} / \text{sum of peak areas}$) for the carbon number groups C ₁ , C ₂ -C ₅ , C ₆ -C ₁₄ , C ₁₅₊ , <u>including</u> the UCM	area percentage	x	x
as above, but <u>excluding</u> the UCM	area percentage	x	x
Peak areas of n-heptene, toluene, n-octene, (m+p)-xylene (<u>excluding</u> the UCM)	area units (usually μVs)	x	x

- For further reporting requirements see the [Reporting Guide](#), in particular the sections on [Reference figures](#) and [Digital data](#).

Recommendations and notes

- *It is recommended to use capillary columns with dimethyl polysiloxane stationary phase (DB-1, OV-1, SE-30, CP-Sil 5CB, BP-1 etc.), length ~25 m, i.d. 0.25-0.32 mm, film thickness ~0.2 μm . Recommended temperature programme: Start at -10°C, 4-6°C/min to 300°C, isothermally at 300°C for about 20 min.*
- *It is strongly recommended to report the variables listed under "Reporting requirements" also for the JR-1 reference sample in order to create a basis for future determination of quantitative quality criteria for TEGC and PYGC results from this sample.*

Key references

- Solli, H., Bjorøy, M., Leplat, P., Hall, K. (1984): *Analysis of organic matter in small rock samples using combined thermal extraction and pyrolysis-gas chromatography. Journal of Analytical and Applied Pyrolysis 7, 101-119.*

Figures

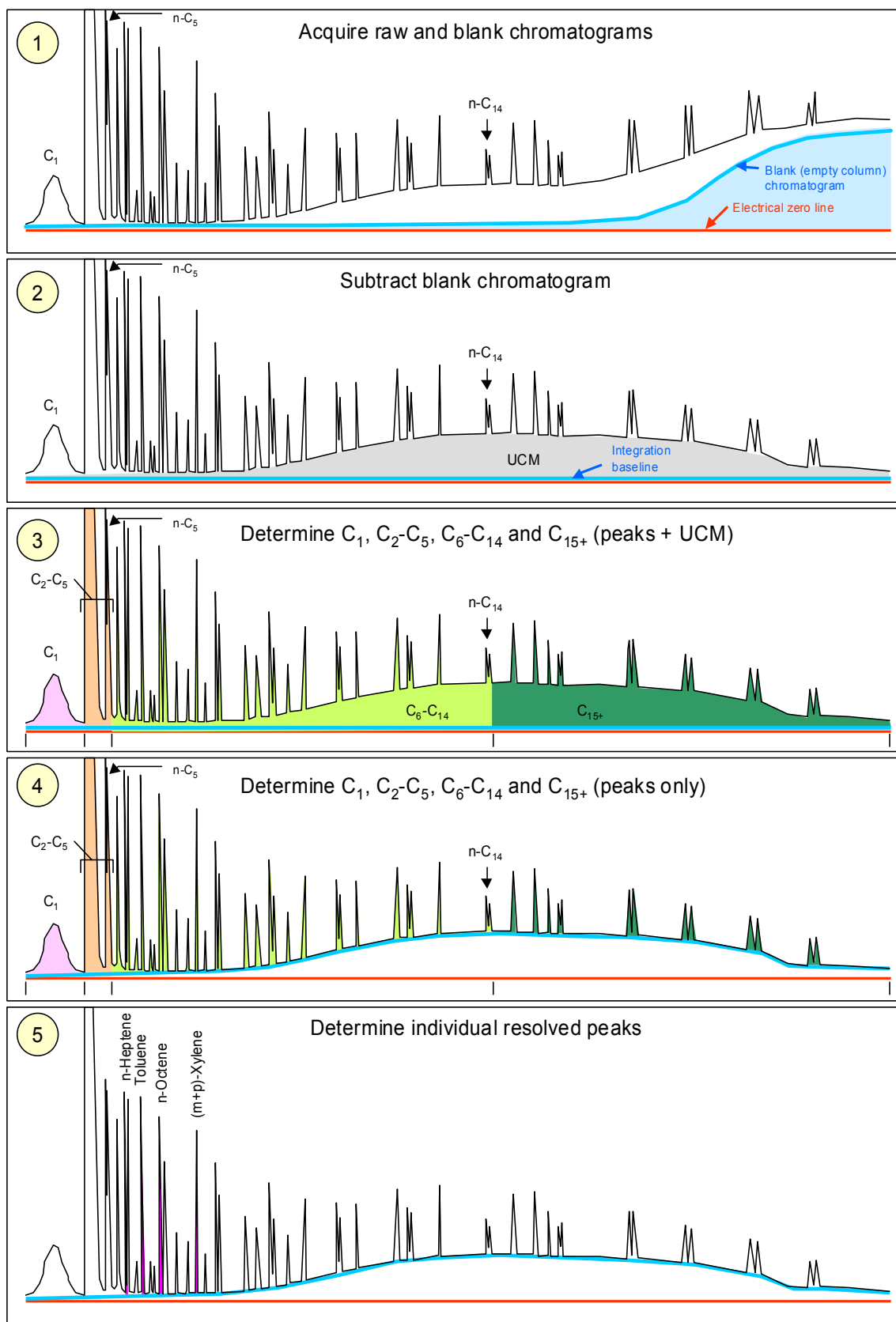


Figure PYGC 1

Principle of processing of pyrolysis gas chromatograms (schematic). Light blue = area subtracted from the sample chromatogram. Pink, orange, light green, dark green = areas corresponding to the groups C_1 , C_2-C_5 , C_6-C_{14} , C_{15+} .

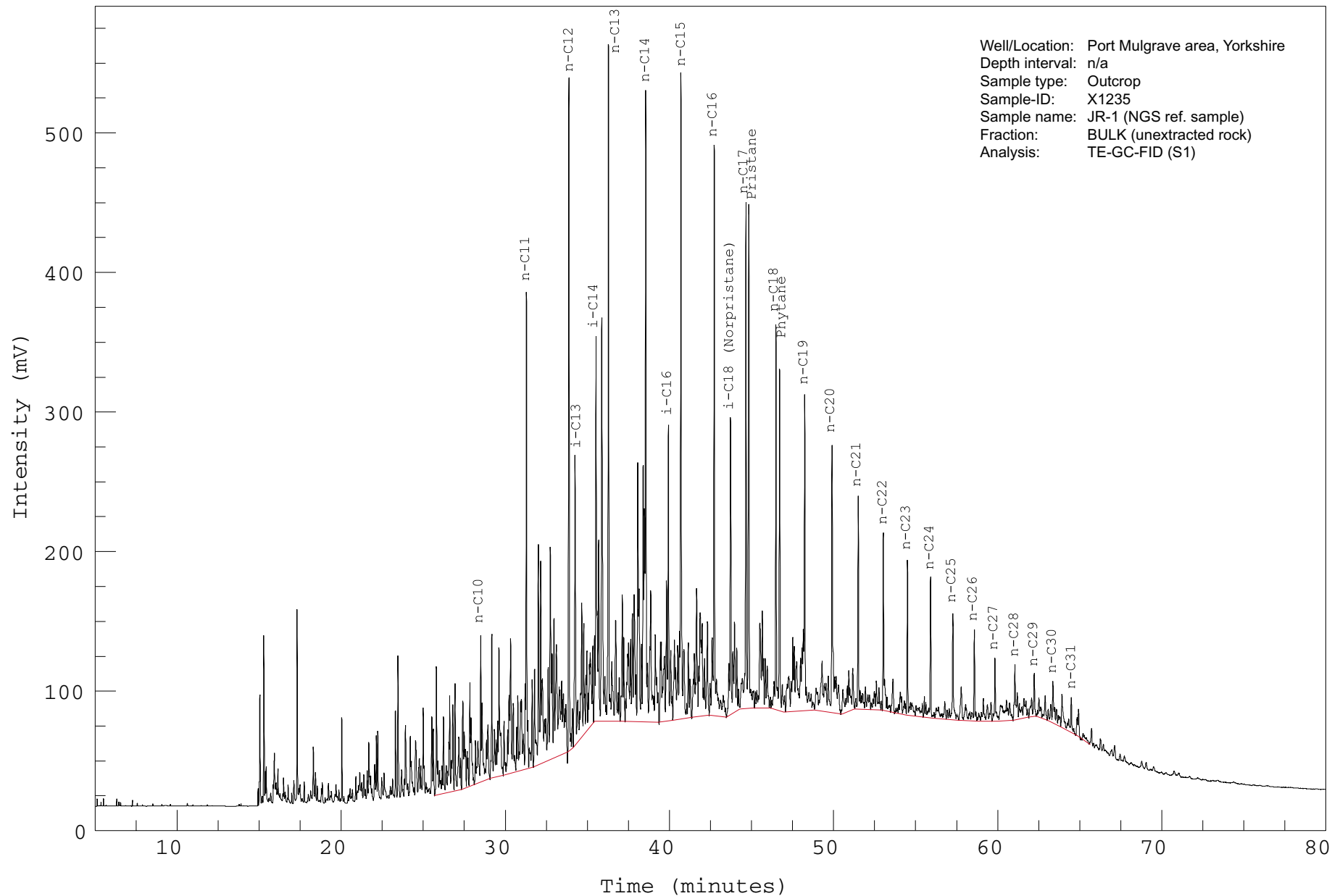


Figure PYGC 2 Thermal extract gas chromatogram of NGS rock sample JR-1 with annotated peaks and baseline.

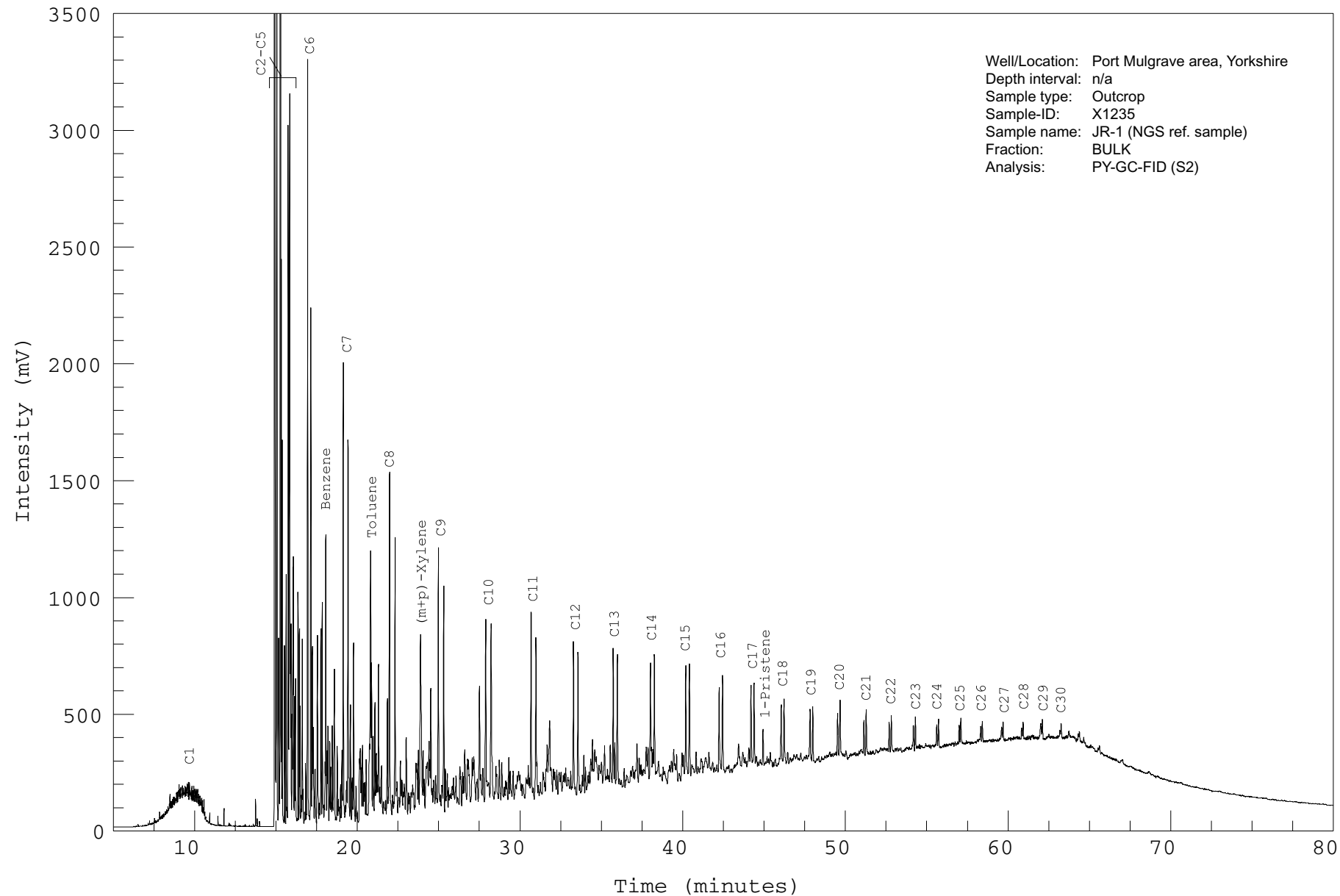


Figure PYGC 3 Pyrolysis gas chromatogram of thermally extracted NGS rock sample JR-1 with annotated peaks. For baseline setting see Figure PYGC1.

Kerogen isolation

No NGS standard applicable.

KER

Purpose, range of application, terminology

The purpose of this preparation is to obtain a mineral-free concentrate of insoluble organic matter (kerogen) for optical analysis, isotope analysis, pyrolysis (kinetics analysis) etc.

Samples to be prepared

- Washed and dried rock samples.

Procedural requirements

- For preparation of kerogen for optical analyses, the rock samples must be crushed to a particle size of approximately 1 mm. Kerogens to be submitted for isotope analyses must be prepared from (finely crushed) solvent-extracted rock. If kerogens from the same sample are to be analysed for isotopes and e.g. optical analyses, the kerogens should be prepared separately.
- Concentrated HCl must be added stepwise to remove carbonate from the sample.
- The beaker containing the sample and the HCl must be allowed to stand in a hot (approximately 60 - 70°C) fluid bath for at least 12 hours.
- Following this, the acid must be decanted from the beaker and the sample washed in distilled water repeatedly until slight acidity (pH = 6 - 6.5) is obtained.
- HF must then be added to the beaker containing the neutralised sample material, the beaker being allowed to stand in a hot (approximately 60 - 70°C) fluid bath for at least 24 hours, or until the silicate material has been dissolved. The sample must be continuously agitated at least during the first 4 hours.
- The HF must then be decanted from the beaker and the sample washed until neutrality (pH = 7) has been attained.
- Any carbonate released during HF treatment must be removed by a final treatment with HCl, followed by flushing to a neutral pH.

Acceptance criteria and reference samples

(none)

Reporting requirements

(none)

Recommendations and notes

- *Kerogen concentrates for isotope analysis should be prepared from solvent-extracted rocks in order to remove any soluble bitumen. Solvent extraction of isolated kerogen is not recommended, as the complete removal of the solvent from a kerogen concentrate may be difficult.*
- *It is important that the temperature is kept high during the whole HF treatment in order to prevent precipitation of complex fluorides that are very difficult to remove.*
- *Some new mud types which coat the minerals may cause problems in kerogen isolation. The customer should therefore in their own interest inform the service company if such muds were used. Likewise, the service company should inform the employer if particular problems occur during kerogen isolation.*

Key references

- *Durand, B., Niçaise, G. (1980): Procedures for kerogen isolation. In: Kerogen. Insoluble organic matter from sedimentary rocks. Edited by Bernard Durand. Paris (Éditions Technip), 35-53. ISBN 2-7108-0371-2.*
- *Taylor, G.H., Teichmueller, M., Davis, A. Diessel, C.F.K., Littke, R., Robert, P. et al. (1998): Organic Petrology. 1998. XVI, 704 p., Berlin, Stuttgart (Gebrüder Borntraeger), ISBN: 3-443-01036-9. [Whole rock mounts: Chapter 7 "Methods and procedures", p. 335 ff.; concentration of organic matter and preparation of polished sections: Sections 8.112 and 8.113, p. 453-454.]*

Figures

(none)

Preparation of kerogen slides

No NGS standard applicable.

KER SLIDE

Purpose, range of application, terminology

The purpose of this preparation is to obtain slides (strew mounts) containing a thin and statistically homogeneous layer of representative, finely dispersed kerogen for “visual kerogen” analysis in transmitted and fluorescent light.

Samples to be prepared

- Kerogen concentrate (unsieved, not oxidised) is the minimum. For other preparations see “Recommendations and notes”.

Procedural requirements

- A non- or low-fluorescent mounting medium must be used.
- Care must be taken to exclude air bubbles in the mounting medium and to ensure a flush fit between the mounting medium and the cover slip.
- Each slide must be labelled with a unique identifier that allows to trace the well number, depth, sample type and type of preparation (e.g. total, sieved, oxidised kerogen).

Acceptance criteria and reference samples

(none)

Reporting requirements

(none)

Recommendations and notes

- *Weakly fluorescent acrylic resins such as Elvacite 2044 have proven to be good mounting media.*
- *For better identification of kerogen constituents, it is recommended to prepare slides from sieved (approximately 15 - 20 μm), non-oxidised kerogen and sieved, weakly oxidised kerogen in addition to the total kerogen slide. Oxidation for 2 minutes with 60% HNO_3 at 20 $^\circ\text{C}$ removes pyrite without damaging structured kerogen, which greatly aids palynofacies description. The different kerogen preparations can be mounted under separate cover glasses on the same slide.*

Key references

(none)

Figures

(none)

Maturity evaluation using transmitted light

No NGS standard applicable.

SCI

Purpose, range of application, terminology

SCI = Spore coloration index as defined by Fisher et al. (1980).

Samples to be analysed

- Slides of kerogen concentrate (whole or preferably sieved, but not oxidised).

Procedural requirements

- The sieved kerogen fraction should be used for maturity evaluation.
- If available, the SCI set of slides provided by Robertson Research International shall be used as a standard for palynomorph colour determination.
- If several palynomorph populations with different SCI are present, the SCI of each population must be determined.

Acceptance criteria and reference samples

(none)

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
SCI for each palynomorph population	1 - 10 scale	x	x
Comments on relative abundances of the palynomorph populations selected for SCI determination	(code(s) or verbose description, e.g. stained, reworked, bleached)	x (as part of the table or in a text section that follows the table)	x
Comments on quality/reliability of SCI values for each population	(as above)	x (as above)	x

- Spore colour in transmitted white light should be reported on a scale from 1 to 10 equivalent to the Spore Colour Index (SCI) of Robertson Research International (Fisher et al. 1980).
- If no original slides from Robertson are available, a table must be provided which shows the correlation between the used scale and the SCI scale.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- *It is recommended to include histograms or a statistical table which shows the abundance and distribution of the palynomorph groups used in the assessment.*
- *Suggested terms for palynomorph quality characterisation are indigenous, stained, reworked, bleached, caved.*
- *Suggested terms for characterisation of abundance are absent, rare, occasional, common, abundant, dominant.*

Key references

- Fisher, M.J., Barnard, P.C., Cooper, B.S. (1980): *Organic maturation and hydrocarbon generation in the Mesozoic sediments of the Sverdrup Basin, Arctic Canada. IV International Palynological Conference, Lucknow (1976-77), 2, 581-588. [Definition of SCI]*
- Smith, P.M.R. (1983): *Spectral correlation of spore coloration standards. In: Petroleum Geochemistry and Exploration of Europe [J. Brooks, ed.], Geological Society Special Publication 12, Oxford (Blackwell), 289-294.*

Figures

(none)

Visual kerogen description using kerogen concentrates

No NGS standard applicable.

VK

Purpose, range of application, terminology

- The purpose of this analysis is to obtain an estimate of the physical composition of the kerogen isolated from a rock.

Samples to be analysed

- Slides made of kerogen concentrate.

Procedural requirements

- Kerogen description must be performed on total kerogen concentrates in both transmitted white light and incident ultra-violet light.
- Sieved or oxidised concentrates must not be used in the estimation of the composition, but only in the identification of kerogen constituents, where they may be extremely helpful.
- The following groups of organic matter must be distinguished and their relative abundance estimated:
 - Amorphous Organic Matter (AM): "structureless" organic debris.
AM must be sub-divided into the following groups using criteria defined by Senftle et al. (1987):
 - Fluoramorphinite (FA): fluorescent amorphous material.
 - Hebamorphinite (HA): non-fluorescent or weakly fluorescent amorphous material.
 - Algal Organic Matter/Phytoplankton (AL): identifiable algae and phytoplankton, including dinocysts.
 - Herbaceous Organic Matter (HE): identifiable species and fragments of spores, pollen and cuticles.
 - Woody Organic Matter (WO): translucent, structured woody tissues.
 - Coaly Organic Matter (CO): completely opaque, usually angular material.
- Coal maceral or maceral group names must NOT be used to define organic matter described in transmitted light, since these have been defined using criteria that are observable only in reflected light.

Acceptance criteria and reference samples

(none)

Reporting requirements

- The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Amorphous OM (fluoramorphinite and hebamorphinite separately), algal OM, herbaceous OM, woody OM and coaly OM	estimated volume % of total kerogen	x	x
SCI (if determined, see separate procedure)	1-10 scale	x	x
Remarks		x	x

- The kerogen composition should be reported to a precision of $\pm 10\%$ or better, and the values must be reported as integers.
- The remarks should include information on (1) any special preparative or measuring procedures (e.g. sieving), (2) sample/preparation quality and reliability of the kerogen composition estimate, (3) any observations which may be of interest in an assessment of the hydrocarbon generation potential of the rock, e.g. high abundance of cuticula, reworking, oxidation etc. If algal material occurs, the type of algae should be indicated, e.g. unicellular Tasmanitids, colonial *Botryococcus*.
- Photos (micrographs) must be provided if specially requested by the customer. In this case, both transmitted and fluorescent light views must be included. Each photo must either contain a scale bar or the length represented by the picture width must be indicated in the figure caption.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- *The problems in establishing a standardised procedure and nomenclature have been recognised by the TSOP Research Subcommittee and are discussed in Teerman et al. (1995), where also some general suggestions are made.*
- *When requested by the customer, a “visual kerogen description” can with little additional effort be extended into a “palynofacies description” which considers the shape and the degree of degradation of terrestrial plant tissues. In contrast to standard visual kerogen description, palynofacies description provides a more detailed characterisation of the main terrestrial and marine palynomorph groups and can provide additional valuable information about the depositional environment.*

The categories (palynomacerals) used for the classification of the organic particles are related to botanical/morphological categories. It is suggested to use the following system of twelve palynomacerals, slightly modified after van der Zwan (1990):

1 Amorphous material	7 Spores (including fungal spores)
2 Tasmanitids	8 Black woody debris (equidimensional)
3 Acritarchs	9 Black woody debris (blade-shaped)
4 Dinoflagellate cysts	10 Leaf cuticles and cuticular debris
5 Bisaccate pollen	11 Structured, well preserved wood
6 Freshwater algae	12 Degraded wood

The palynomacerals can be easily grouped into the organic matter categories used in conventional visual kerogen description (1 = AM, 2+3+4+6 = AL, 5+7+10 = HE, 8+9 = CO, 11+12 = WO). It should be noted, however, that the palynofacies estimates on material from coastal environments are normally carried out on sieved (screened) residues, while visual kerogen descriptions are made on total organic residues.

Key references

- Burgess, J.D. (1974): *Microscopic examination of kerogen (dispersed organic matter) in petroleum exploration. Geological Society of America Special Paper 153, 19-30.*
- Senftle, J.T., Brown, J.H., Larter, S.R. (1987): *Refinement of petrographic methods for kerogen characterisation. International Journal of Coal Geology 7, 105-107.*
- Teerman, S.C., Cardott, B.J., Harding, R.W., Lemos De Sousa, M.J., Logan, D.R., Pinheiro, H.J., Reinhardt, M., Thompson-Rizer, C.L., Woods, R.A. (1995): *Source rock/dispersed organic matter characterization - TSOP Research Subcommittee Results. Organic Geochemistry 22, 11-25.*
- Tyson, R.V. (1995): *Sedimentary organic matter. Organic facies and palynofacies. 615 pp, London (Chapman & Hall). [ISBN 0 412 36350 X]*

- *van der Zwan, C.J. (1990). Palynostratigraphy and palynofacies reconstruction of the Upper Jurassic to Lowermost Cretaceous of the Draugen Field, offshore Mid Norway. Review of Paleobotany and Palynology 62, 157-186.*

Figures

(none)

Preparation of samples for vitrinite reflectance measurement and incident light organic petrography

POL SEC

No NGS standard applicable.

Purpose, range of application, terminology

The purpose of this preparation is to obtain polished particulate mounts of sufficient representativity and surface quality for vitrinite reflectance measurement and incident light organic petrographic analysis.

Samples to be prepared

- Rock chips (preferred) or kerogen concentrate (if necessary because of poor organic richness). The type of material used for preparation has to be agreed on between customer and service company.

Procedural requirements

- Rock samples must be broken up to a particle size of 1-2 mm, with a minimum of fine material, and mounted in fast-setting synthetic resin. Alternatively, kerogen concentrate may be mounted.
- The temperature of the sample/resin must not exceed 50°C during setting in order to avoid maturation of thermally immature organic material.
- The top surface of the plug must be cleared of excess resin and carefully polished until a scratch-free, polished surface is obtained.
- The procedures recommended in the handbooks and standards listed under "Key references" should be followed as closely as possible and as far as applicable to organic rich rocks other than coal.

Acceptance criteria and reference samples

(none)

Reporting requirements

(none)

Recommendations and notes

(none)

Key references

- *International Committee for Coal Petrology (ICCP) [ed.] (1963): International Handbook of Coal Petrography, 2nd ed., Paris (Centre National de la Recherche Scientifique). [Part II contains methods of analysis, including a section named "Recommendations for the preparation of polished surfaces of lump and particulate samples"]*
- *International Organization for Standardization (ISO) (1985): Methods for the petrographic analysis of bituminous coal and anthracite -- Part 2: Preparation of coal samples. [ISO 7404-2:1985](#), Geneva (ISO).*
- *Taylor, G.H., Teichmueller, M., Davis, A. Diessel, C.F.K., Littke, R., Robert, P. et al. (1998): Organic Petrology. 1998. XVI, 704 p., Berlin, Stuttgart (Gebrüder Borntraeger), ISBN: 3-443-01036-9. [Whole rock mounts: Chapter 7 "Methods and procedures", p. 335 ff.; concentration of organic matter and preparation of polished sections: Sections 8.112 and 8.113, p. 453-454.]*

Figures

(none)

Vitrinite reflectance measurements

VR

No NGS standard applicable.

Purpose, range of application, terminology

Vitrinite (or huminite) reflectance measurements are made to obtain information on the thermal maturity of kerogen. The technique is adapted from coal petrology and applicable to the whole range of maturities relevant to petroleum exploration. For terminology see ICCP (1963, 1971), ISO (1994a) and Taylor et al. (1998).

Samples to be analysed

- Polished plugs made from rock chips (preferred) or kerogen concentrate (if necessary because of poor organic richness). The type of material used for preparation has to be agreed between customer and service company.

Procedural requirements

- The reflectance measurements must be made on vitrinite/huminite (preferably telocollinite) under immersion oil ($n_{e(546\text{ nm})} = 1.518$) at a wavelength of 546 nm (unpolarised light) according to the procedure defined for bituminous coal by the ICCP or by ISO (1994 b), with the necessary adjustments to dispersed organic matter in rocks (e.g. lower number of measurements, see Taylor et al. 1998).

Acceptance criteria and reference samples

- Presently no NGS sample is defined as control sample.
- The artificial standards, work routines and acceptance criteria for calibration and control of the analytical equipment described in ISO (1994 b) should be applied.

Reporting requirements

The following variables must be reported:

Variable	Unit of measure	Include in printed report tables	Include in digital data transfer
Type of sample preparation (whole rock or kerogen concentrate)		x	x (fraction)
Mean random reflectance ¹⁾	% R (546 nm, oil imm.) ²⁾	x	x
Number of readings ¹⁾		x	x
Standard deviation of the reflectance distribution ¹⁾	% R (546 nm, oil imm.) ²⁾	x	x
Remarks on data reliability (at least for the indigenous population)		x	x (analysis comment)
Fluorescence colour of liptinite	(descriptive text or code with explanation)	x	x
Remarks on nature and quality ¹⁾		x	x (analysis comment)

¹⁾ For each phytoclast population measured.

²⁾ Reflectance is often also abbreviated R_o (o for oil immersion), R_r (r for random) or R_m (m for mean).

- The following reliability classes are a minimum requirement: Good (G) - Fair (F) - Poor (P).
- Terms for characterisation of the nature / quality are e.g. indigenous, stained, reworked, caved, bitumen, low-reflective vitrinite.
- The results from each sample must be reported in the form of histograms of R.

- Photos (micrographs) must be provided if specially requested by the customer. In this case, both reflected and fluorescent light views must be included. Each photo must either contain a scale bar or the length represented by the picture width must be indicated in the figure caption.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- *In case of organic-rich rocks other than coal, a minimum of twenty (20) measurements should be made on a clean huminite/vitrinite (ideally collinite or telocollinite) surface, where possible.*
- *A more detailed characterisation of the quality of the sample and/or the reflectance determination might be desirable in cases when reflectance data from different sources have to be compiled, e.g. in regional studies. Such a characterisation should contain information about both the reasons for the total reliability rating and the probable influence of a poor sample quality on the mean reflectance value.*

An example of one such descriptive method (modified after T. Throndsen, Institute for Energy Technology, Kjeller, Norway) is given below. This type of presentation provides much information, but could probably be improved in a way that would make the voluminous legend unnecessary.

Vitrinite reflectance (mean random reflectance in oil) (%R)	Standard deviation (%R)	Number of measurements	Overall data quality (+ / / -)	Sample quality Items 12345
0.16	0.03	9		-0000
0.29	0.05	21		reworked
0.34	0.05	18	-	--+0--
0.38	0.04	3		--+000
0.61	0.12	2	-	
0.32	0.06	18		00-00
0.23	0.09	14	-	bituminite
0.45	0.05	25	+	00000

CODES FOR DATA QUALITY	
<u>Overall data quality:</u>	
+	high quality
(no code)	medium quality
-	low quality
The <u>sample quality</u> is characterised by five items as follows: 1 abundance of vitrinite, 2 identification of vitrinite, 3 type of vitrinite, 4 particle size, 5 particle surface quality	
Each item is characterised by one of the following codes:	
+	may give a too high vitrinite reflectance value
0	has no effect on the resulting vitrinite reflectance value
-	may give a too low vitrinite reflectance value
An ideal sample is characterised as follows: 00000	

Key references

- *International Committee for Coal Petrology (ICCP) [ed.] (1963): International Handbook of Coal Petrography, 2nd ed., Paris (Centre National de la Recherche Scientifique). [Part II contains methods of analysis, including a section named "Recommendations for the preparation of polished surfaces of lump and particulate samples"]*
- *International Committee for Coal Petrology (ICCP) [ed.] (1971, 1975): International Handbook of Coal Petrography, Supplements to the 2nd ed., Paris (Centre National de la Recherche Scientifique).*

- *International Organization for Standardization (ISO) (1994 a): Methods for the petrographic analysis of bituminous coal and anthracite -- Part 1: Vocabulary (available in English only). [ISO 7404-1:1994](#), Geneva (ISO).*
- *International Organization for Standardization (ISO) (1985): Methods for the petrographic analysis of bituminous coal and anthracite -- Part 2: Preparation of coal samples. [ISO 7404-2:1985](#), Geneva (ISO).*
- *International Organization for Standardization (ISO) (1994 b): Methods for the petrographic analysis of bituminous coal and anthracite. - Part 5: Method of determining microscopically the reflectance of vitrinite (available in English only). [ISO 7404-5:1994](#), Geneva (ISO).*
- *Senftle, J.T., Brown, J.H., Larter, S.R. (1987): Refinement of petrographic methods for kerogen characterisation. *International Journal of Coal Geology* 7, 105-107.*
- *Taylor, G.H., Teichmueller, M., Davis, A. Diessel, C.F.K., Littke, R., Robert, P. et al. (1998): *Organic Petrology*. 1998. XVI, 704 p., Berlin, Stuttgart (Gebrüder Borntraeger), ISBN: 3-443-01036-9. [Chapter 7 "Methods and procedures", p. 335 ff.]*

Figures

(none)

Maceral description in reflected light

No NGS standard applicable.

MGA

Purpose, range of application, terminology

Maceral description in reflected light (and fluorescent light) is normally done on coal samples (maceral/maceral group analysis), but may also be performed on organic rich rocks as a supplement to the “visual kerogen” analysis of kerogen concentrate in transmitted light. For terminology see ICCP (1963, 1971), ISO (1994 a) and Taylor et al. (1998).

Samples to be analysed

- Polished plugs made from rock chips (preferred) or kerogen concentrate (if necessary because of poor organic richness).

Procedural requirements

- Maceral descriptions shall follow the guidelines laid down by the ICCP (1963, 1971, 1975) and ISO (1994 c). These are also described in Taylor et al. (1998).
- Other procedures, such as microlithotype analysis, must also follow the guidelines specified by the ICCP unless otherwise agreed with the customer.

Acceptance criteria and reference samples

(none)

Reporting requirements

- Maceral descriptions shall be reported in terms of the three main maceral groups, (i.e. Vitrinite, Liptinite, Inertinite) and Mineral Matter. These main groups may be further subdivided as necessary.
- The level of precision for these descriptions should be $\pm 10\%$ or better, the percentages being reported as integers.
- Any deviation from the guidelines specified by ICCP or ISO must be detailed in the chapter [Experimental procedures](#) of the analytical report.
- For general reporting rules see the [Reporting Guide](#).

Recommendations and notes

- The use of the following abbreviations is suggested:

1	Liptinite	LI	11	Telocollinite	VE
2	Alginite	LA	12	Desmocollinite	VS
3	Cutinite	LC	13	Vitrodetrinite	VD
4	Sporinite	LS	14	Inertinite	IN
5	Resinite	LR	15	Fusinite	IF
6	Bituminite	LB	16	Semifusinite	IS
7	Liptodetrinite	LD	17	Sclerotinite	IL
8	Vitrinite	VI	18	Macrinite	IM
9	Telinite	VT	19	Micrinite	IK
10	Collinite	VC	20	Inertodetrinite	ID

Key references

- *International Committee for Coal Petrology (ICCP) [ed.] (1963): International Handbook of Coal Petrography, 2nd ed., Paris (Centre National de la Recherche Scientifique).*
- *International Committee for Coal Petrology (ICCP) [ed.] (1971, 1975): International Handbook of Coal Petrography, Supplements to the 2nd ed., Paris (Centre National de la Recherche Scientifique). [For ordering information see <http://www.iccop.org/public.htm>].*
- *International Organization for Standardization (ISO) (1994 a): Methods for the petrographic analysis of bituminous coal and anthracite -- Part 1: Vocabulary (available in English only). [ISO 7404-1:1994](#), Geneva (ISO). [For ordering information see <http://iso.ch/>].*
- *International Organization for Standardization (ISO) (1994 c): Methods for the petrographic analysis of bituminous coal and anthracite -- Part 3: Method of determining maceral group composition (available in English only). [ISO 7404-3:1994](#), Geneva (ISO). [For ordering information see <http://iso.ch/>].*
- *Senftle, J.T., Brown, J.H., Larter, S.R. (1987): Refinement of petrographic methods for kerogen characterisation. International Journal of Coal Geology 7, 105-107.*

Figures

(none)

REPORTING GUIDE

Principal rules and remarks

- The aim of a standard geochemical report is to present and describe the data obtained by the various analyses. The extent of detailed interpretation - in the form of both text and figures - should be agreed upon by customer and service company.
- As a general principle, all results must be provided also in digital form, in addition to the written report. Unless specified otherwise, the requirements stated below therefore apply to both the written report and the digital data transfer.
- If any analyses are not carried out in accordance with this Guide, this must be noted.
- If no results can be obtained from an analysis, or if the obtained results are unreliable or doubtful, this has to be noted and the reason for this should be mentioned.
- Wherever the Guide requires control analyses, the results from these must be reported, separately from the “normal” analyses. The tables must contain all variables used as quality criteria (which may differ from those to be reported for the “normal” analyses). They must also include the name(s) of the control sample(s) and should contain the most likely values and permissible ranges quoted in the NIGOGA.
- The sample type must be given. It must be clear if bulk or picked cuttings were used.
- Both top and bottom depth must be reported for cutting, drill stem test and production test samples. Measured depth relative to rotary kelly bushing (MD RKB) must always be reported, and it must be stated whether this is driller’s or logger’s depth. The customer has to make this information available to the service company.
- All tables and figures should be mentioned in the text.
- Any nomenclature (for peaks, ratios, kerogen constituents etc.) and units of measure stated in the NIGOGA must be followed, unless items (e.g. compounds, ratios) have to be reported which are not mentioned in this Guide.
- All terms (codes, abbreviations, compound names) and units of measure that are necessary for the understanding of the report text, the figures or tables and that are not defined or specified in this Guide must be explained. This information can conveniently be collected in a separate table (list of terms) which has to be included also in the electronic data transfer.
- The unit of measure must be given for each reported variable. “Implicit” or “self-explanatory” units of measure do not exist! Incomplete concentration units like “%”⁵, “ppm”, “ppb” etc. are not acceptable, as they neither tell which properties were determined (e.g. volume, weight, peak area, peak height) nor to which variable the values were normalised (e.g. sum of recorded peak areas, sample weight, sample volume). When concentrations are determined from GC or GC-MS peak data, it must be clearly stated whether these are based on peak areas or peak heights. When peak ratios are reported, the formula must be given, and it must always be stated whether the ratios are based on areas or heights or concentrations. If they are based on concentration it must be stated whether the concentrations ultimately are based on peak heights or areas.

The individual parts of a standard report

1 Title page

The number of volumes must be noted on the title page (e.g. “Volume 1 of 2”).

⁵ The term “normalised %” is a tautology, as a percentage *by definition* is normalised to 100.

2 List of contents

A list of contents should be inserted at the beginning of the report. It should present the content of all volumes.

Example for a list of contents (the content itself may of course vary):

Contents	
Volume 1 of 2: Main volume	
Summary	Page 2
Introduction	4
Results and discussion	5
Conclusions	25
References	27
Experimental procedures and comments (etc. ...)	28
Volume 2 of 2: Appendix volume	
Saturated hydrocarbon gas chromatograms	
Saturated hydrocarbon mass fragmentograms	
Vitrinite reflectance histograms (etc. ...)	

3 Summary

The summary should contain the most important information about sample types and analytical results from the chapters "Introduction" and "Conclusion". Guidelines for location maps and summary figures are found in section 10 below.

4 Introduction

This chapter should mention the following topics:

- Objective(s) of the project
 - Number and types of samples analysed
 - Analytical programme
-] This information can be presented in a summary table as shown in the example below.
- Information provided by the customer [e.g. depth unit (m or ft), depth type (MD or TVD, driller's or logger's) and reference level (RKB or MSL), formation tops, total depth (TD), casing points, turbo drilled intervals, mud systems, sampling intervals for cuttings, core shift information, perforation intervals and names for DST samples].
 - Any other information which may be necessary or useful for the interpretation of the data (e.g. sample quality, analytical problems) or for tracing of the results (e.g. laboratory contact person(s), subcontractor(s)).

Example of a tabular analysis summary:

Analysis	Number of analyses					Total
	Cuttings	SWC	Core	Oil or condensate	Gas	
Headspace and occluded gas	50					0
TOC	70	12	15			97
Rock-Eval pyrolysis	50	12	15			77
Vitrinite reflectance	17	8	5			30
Solvent extraction	7	5	3			15
Group separation (MPLC)	7	5	3	1		16
TLC-FID analysis	7	5	3	1		16
GC SAT	7	5	3	1		16
Gas composition (GC)					1	1
Isotope composition (etc...)				1	1	2

5 Results and discussion

The results should be discussed and the relevant geochemical information be interpreted in this chapter. The following sequence of topics is recommended:

1. Source type and potential (separately for each formation or group, where this information is available),
2. Thermal maturity (trend throughout the well, based on different types of analyses),
3. Presence and character of migrated hydrocarbons (separately for each formation or group, where this information is available).

6 Conclusions

The chapter "Conclusions" should include a brief but comprehensive summary of the essential information provided by the analyses. It is strongly recommended to relate the conclusions clearly to the objectives defined in the chapter "Introduction".

7 References

If references are listed, they should include author(s), year, title, journal, volume and range of pages.

8 Experimental procedures

- This chapter should contain a short description of the experimental procedures, the analytical conditions and the calibration or reference samples.
- If non-NIGOGA methods are used, they must be described here.
- Guidelines for interpretation of the results must be included if requested by the customer.

9 Tables

- The report has to contain one table which specifies the following for all samples analysed: Sample identification code⁶, top depth and bottom depth of sampled interval, sample name (if applicable, e.g. "DST 1"), sample type, sample lithology (if determined or otherwise known) and the types of analyses performed on the sample.
- All other tables have to contain two columns specifying sample bottom depth and sample identification code. Top depth (e.g. of cutting or DST samples), sample type and a shortened lithology description should be included, if space permits.
- The units of measure should preferably be included in the table headers, rather than in a separate legend. (Also see [Principal rules and remarks.](#))

10 Figures

10.1 Location map

- The purpose of a location map is to show the position(s) of the analysed well(s) in relation to neighbouring wells (e.g. wells within the same licence or block) and oil or gas fields, if present. Location maps in standard well reports should show a level of detail similar to that of the maps commonly included in NPD's press releases.

⁶ The laboratory's sample identification number or alphanumeric code.

10.2 Summary figures

- The purpose of summary figures is to give an overview of the main geochemical characteristics and trends in the analysed samples, such as organic richness, hydrocarbon generation potential, thermal maturity and occurrence of migrated hydrocarbons. If appropriate and possible, they should present the geochemical characteristics in relation to sample depth, lithology and stratigraphy.
- Summary figures have to be agreed between customer and service company.
- All summary figures should be included in the main (text) volume and may be placed in the text or in a separate section.

10.3 Reference figures

- Reference figures include chromatograms⁷, vitrinite reflectance histograms etc. They may be included in separate appendix volumes.
- Reference figures must be included for all analyses, including valid control analyses of reference samples. Chromatograms must also be included, even if they show no peaks.
- Each reference figure must be labelled with the well name, the sample depth interval, the sample type (cuttings, core chip, SWC, oil, gas etc.), the laboratory's sample reference code (sample-ID) and, where applicable, sample name (DST 1b, MDT, FIT etc.) and fraction type (SAT, thermal extract etc.).
- Chromatograms must be at least half an A4 page in size in order to allow a detailed visual assessment.
- The retention time interval of each chromatogram must be wide enough to display all peaks that shall be reported (see the respective "Reporting requirements" sections of the [Analysis Guide](#)). To increase readability, chromatograms may be divided into maximal two retention-time intervals, shown in separate frames but with the same time and intensity scales. In this case, a full chromatogram in a single frame should also be included to allow an overview of the whole trace. Related mass fragmentograms (e.g. m/z 177, 191, 205 for terpanes) must have identical time axes.
- The vertical expansion should be normalised to the highest peak of the chromatogram, except for large contaminant or standard peaks which should be truncated. PY-GC chromatograms should be normalised to the highest peak in the C₆₊ range.
- Mass fragmentograms of polycyclic aromatic compounds, where the peaks representing the various alkylated isomers do not overlap, may be plotted within the same frame (e.g. m/z 178, 192 and 206 for phenanthrenes). If the data system allows, each trace should be normalised to its (own) maximum intensity, and these intensity values must be quoted in the figure to allow an estimate of the relative intensities between different ions.
- For each type of chromatogram (whole fluid, SAT, ARO, TE-GC, PY-GC and each ion mass) run under the same conditions, a typical example with labelled peaks must be included. All peaks for which heights and concentrations are tabulated and all peaks referring to internal standards must be labelled. Additional identified peaks that are particularly prominent, geochemically significant and/or referred to in the text should be labelled in the respective chromatogram(s).
- The use of the peak labels (peak codes) listed in the peak tables of the Analysis Guide is compulsory. Greek letters (α , β) can be replaced by the corresponding Latin letters (a, b). Possible additional labels (e.g. for hopenes or sterenes) must be listed together with the full compound names. (See "[Principal rules and remarks](#)" and "[Experimental procedures](#)").

⁷ "Chromatogram" is here used as a collective term for all kinds of gas chromatograms and mass fragmentograms.

Digital data

Digital data shall be available together with the final report, and at the same time as the final report is dispatched from the service company.

Numeric (tabular) data

- The data files shall be in the format required by the customer and that by the Norwegian Petroleum Directorate (NPD 1995, regularly updated). On request, licence partners can also obtain a data tape in their required format, subject to approval by the customer/operator.
- Numeric fields must never contain any text data (e.g. *, -, n.d.p., 2000 – 2020 m, <0.1), but exclusively numeric data (e.g. 2020, -32.75). Comments regarding coelution, analytical problems etc. must be stated in a separate comment field. Values regarded by the laboratory as useless should be reported as missing, and the reason should be stated in the comment field.
- Derived variables which can be calculated from the variables reported digitally (e.g. Hydrogen index or peak ratios) do not have to be included in the electronic data transfer.
- It is, however, compulsory to include information on methods, instruments, units etc. and notes on possible deviations from the NIGOGA procedures as described in the “Principal rules and remarks”. This information should be inserted into the respective comment fields of the digital data files or, where this is unfeasible, in a separate file.

Graphic data

Graphic data (gas chromatograms, mass fragmentograms, reflectance histograms, micrographs) in digital form should be provided on request by the customer. Technical details such as size and resolution, storage medium (e.g. CD-ROM) and file format (e.g. TIFF, WMF, EPS, PDF) must be agreed between service company and customer.

GENERAL REFERENCES

The Norwegian Petroleum Directorate 1995: Provisions relating to digital transmission of geological and reservoir technical data in connection with the final report. (Drilling regulations, Section 12). The Norwegian Petroleum Directorate, November 1995. YA-061. ISBN 82-7257-476-4. [Therein: Appendix 6 - Specification of transfer format for geochemical data (GC-NPD-95 version 1.0, Dictionary GC-DIC-V1); NB! regularly updated].

Dahlgren, S., Hanesand, T., Mills, N., Patience, R., Brekke, T., Sinding-Larsen, R. 1998 a: Norwegian Geochemical Standards Newsletter vol. 1, Norwegian Geochemical Standard samples: Svalbard Rock – 1 (NGS SR-1). The Norwegian Petroleum Directorate, Stavanger, Norway.

Dahlgren, S., Hanesand, T., Mills, N., Patience, R., Brekke, T., Sinding-Larsen, R. 1998 b: Norwegian Geochemical Standards Newsletter vol. 2, Norwegian Geochemical Standard samples: Jet Rock – 1 (NGS JR-1). The Norwegian Petroleum Directorate, Stavanger, Norway.

Dahlgren, S., Hanesand, T., Mills, N., Patience, R., Brekke, T., Sinding-Larsen, R. 1998 c: Norwegian Geochemical Standards Newsletter vol. 3, Norwegian Geochemical Standard samples: North Sea Oil – 1 (NGS NSO-1). The Norwegian Petroleum Directorate, Stavanger, Norway.

The NGS Newsletters are also accessible on the World Wide Web under the following URL: <http://www.npd.no/engelsk/npetres.ngs.htm>.