

Characterisation of bituminite and testing reproducibility of vitrinite and bituminite reflectance measurements in organic-carbon-rich intervals of the Kimmeridge Clay Formation: ICCP Identification of Dispersed Organic Matter Working Group round robin exercises

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ABSTRACT

Three round robin exercises (RRE) aimed at characterizing bituminite and testing the reproducibility of random vitrinite (VR_o %) and bituminite ($BitR_o$ %) reflectance measurements on samples of dispersed organic matter from organic-carbon rich shale intervals of Kimmeridge Clay Formation were conducted in 2016, 2018, and 2020. This work was conducted by the Identification of Dispersed Organic Matter Working Group (IDOM WG) of the International Committee for Coal and Organic Petrology (ICCP). The Kimmeridge Clay Formation represents the major marine source rock for the North Sea hydrocarbon province. Samples collected from Kimmeridge Bay, Dorset, England, United Kingdom and Filey Bay, North Yorkshire, England, United Kingdom outcrops and the 16/17–14 well in the Tiffany oil field, Viking Graben, North Sea, were analyzed by 19 to 23 participants in 15 to 17 laboratories from 14 to 15 countries. Samples contain moderate to high organic matter content [5.32 to 12.10 wt% total organic carbon (TOC) content] with thermal maturity ranging from immature to thermally mature (T_{max} 418 to 445 °C).

Participants used a standard description sheet to record bituminite morphological characteristics, including its form, size, mode of appearance, and impurities, for use in the revision of the bituminite definition. The usage of previously published classifications of bituminite types (e.g., bituminite I *sensu* Teichmüller and Ottenjann, 1977) in the RRE was not found to be helpful. The indistinct, amorphous character of boundaries of bituminite, and the poor preparation of whole rock pellets for the 2018 and 2020 RRE hampered identification of bituminite. Mean random $BitR_o$ %, including outlying values, varied in all three RRE from 0.19 to 0.35% and is reported here for the first time. Application of the criterion standard deviation (SD) $< 0.15 \times BitR_o$ % to exclude outlying $BitR_o$ % values showed virtually no change in modified $BitR_o$ % in immature samples (2016 and 2018 RRE) and an unexpected increase to 0.43% $BitR_o$ in the thermally mature sample (2020 RRE).

Mean VR_o values, including outliers in the three RRE, range from 0.47 to 0.71%, confirming the immature to thermally mature nature of samples of the Kimmeridge Clay Formation used in this study. Application of the criterion SD $< 0.15 \times VR_o$ to exclude outlying values results in a comparable range of modified VR_o values and a decrease in group standard deviation. Precision of VR_o values (including outlying values) evaluated via the averaged unsigned multiple of the standard deviation rule (< 1.5) showed moderate to high precision comparable to historical values from similar RRE. An average reproducibility limit (RL) of 0.29% (including outlying values) showed an improvement in reproducibility relative to similar historical RRE (0.35%), which was attributed to the application of a common methodology (ASTM D7708, 2014b, 2023) to enhance the quality of data from the reflectance test method.

1. Introduction

Bituminite is a liptinite maceral *sensu* Pickel et al. (2017), which has been well-studied in the past decade (e.g., Hackley et al., 2018, 2022a; Kus et al., 2016, 2017; Teng et al., 2021; Synnott et al., 2016; Damoulianou et al., 2022; Holland et al., 2024). Although bituminite was initially identified in subbituminous coals (Hamrla, 1959), Teichmüller (1971) was the first to describe its presence in petroleum source rocks, and this maceral is now recognized in many oil shales worldwide, e.g., the Devonian to Mississippian Bakken Formation, Lower Cretaceous Toolebuc Formation, the Lower Jurassic Posidonia Shale, and the Upper Jurassic Kimmeridge Clay Formation (ICCP, 1993; Hackley and Cardott, 2016; Pickel et al., 2017; Hackley et al., 2022b). In combination with sub-microscopic lipid substances, bituminite serves as the main source material for crude oil (ICCP, 1993), and its identification is therefore important in the evaluation of kerogen type in source rocks. Further, as an oil-prone maceral, bituminite converts to a polar-rich residue of solid bitumen upon thermal maturation (Hackley et al., 2018, 2022a, 2025). This is in the line of thinking of Potonié (1920), who stated that “bituminite” (a general term in the early 20th century for organic rich rocks) is especially rich in bitumen. Despite its importance, the petrographic distinction between bituminite and similar macerals such as lamalginite, solid bitumen, and with bituminous mineral-rich groundmass remains challenging, requiring further refinement of the definition of bituminite (e.g., Kus et al., 2017; Hackley et al., 2018; Sanei, 2020; Teng et al., 2021).

Research on bituminite and alginite in sedimentary rocks has been pursued by the International Committee for Coal and Organic Petrology (ICCP), leading to the establishment of the Identification of Organic Matter Working Group (IDOM WG) in 2006, within ICCP Commission II, Geological Applications of Coal and Organic Petrology (<https://www.iccop.org/commissions/commission-ii/>). The main goals of the IDOM WG were to (1) determine the applicability of existing ICCP maceral nomenclature [e.g., bituminite and alginite as described in textbooks for

organic petrology (Stach et al., 1982; Taylor et al., 1998)] and recently outlined in The Society for Organic Petrology-ICCP classification of dispersed organic matter (Gonçalves et al., 2024), and (2) to further constrain definitions of bituminite as necessary. Kus et al. (2017) published the first IDOM WG results for three round robin exercises (RRE) conducted on photomicrographs prepared from kerogen isolates (KI) (2006) and from whole rock pellets (2009, 2011). Kus et al. (2017) noted that (1) the bituminite definition required clarification of its description of morphological characteristics, and (2) that differentiation of bituminite from other alginite macerals or from bituminous mineral-rich groundmass was necessary. Therefore, the round robin exercise in 2016, 2018, and 2020 by the IDOM WG focused mainly on bituminite with the aim of improving usage of bituminite terminology in samples from the Kimmeridge Clay Formation. In addition, the reproducibility of vitrinite and bituminite reflectance measurements in samples from the Kimmeridge Clay Formation were tested in the 2016, 2018, and 2020 RRE.

2. Samples and methods

2.1. Sample selection

All samples were obtained from outcrops and subcrops of the Kimmeridge Clay Formation, the major marine source rock for the North Sea hydrocarbon province, located in United Kingdom or the North Sea (Figs. 1, 2). Rock samples were selected from thermally immature and mature organic matter containing bituminite maceral.

The 2016 RRE sample was collected by Dr. Alan Cook³ (Keiraville Konsultants Pty. Ltd., Australia) from the organic-rich shales, of the coastal cliff sections at Kimmeridge Bay, Dorset, England, United Kingdom (Table 1). The 2018 RRE sample, an organic-rich shale, was

³ Deceased November 17, 2011.

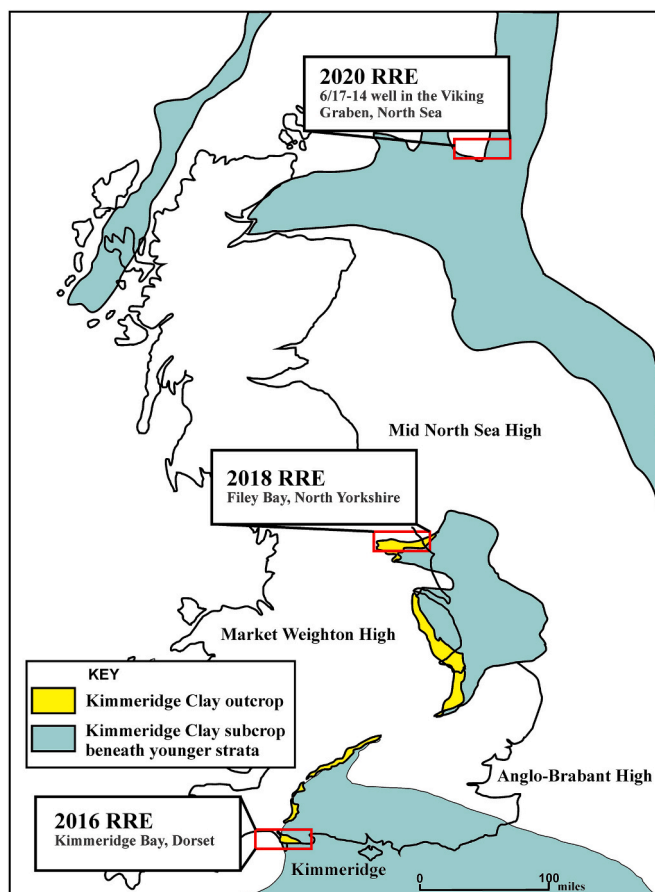


Fig. 1. Sketch map of the outcrop and subcrop of the Kimmeridge Clay Formation showing locations of the samples for the 2016 round robin exercise (RRE) (Kimmeridge Bay, Dorset, England, United Kingdom), 2018 RRE (Filey Bay, North Yorkshire, England, United Kingdom), and 2020 RRE (16/17–14 well in the Viking Graben, North Sea). Modified from Gallois (2004).

provided by Prof. Paul Wignall (University of Leeds, UK) from the foreshore outcrop at Filey Bay, North Yorkshire, United Kingdom (Table 1). The 2020 RRE sample was obtained from Dr. Mike Howe (National Geological Repository, NGR Keyworth Core Store of the British Geological Survey, UK) and collected from a Kimmeridge Clay Formation core plug of the 16/17–14 well in the Viking Graben, North Sea (Table 1).

2.2. Sample preparation

Sample preparation was conducted at Federal Institute for Geosciences and Natural Resources (BGR, Hannover, Germany) and involved cleaning (washing in water), air-drying at ambient conditions (2016 RRE) according to ISO 18283 (2022), or drying at 25 °C in a nitrogen-flushed drying oven (2018 RRE). The 2016 and 2018 RRE involved jaw crushing to a grain size of about 1–2 mm, and homogenizing (quartered and mixed) with no further sieving. The 2020 RRE sample was crushed in a jaw crusher to a grain size of about 1–5 mm, sieved to 1–2 mm mesh size, and homogenized. Crushing, sieving, and homogenizing were according to ISO 7404-2 (2009).

2.3. Polished whole rock pellets and kerogen isolate preparation

The 2016 RRE sample was used for preparation of both polished whole rock pellets and kerogen isolates (KI). Preparation of polished whole rock pellets in the 2016 RRE was undertaken by each of the participants in accordance with ISO 7404-2 (2009). Kerogen isolates

(KI) were prepared and kindly provided by Prof. João Graciano Mendonça Filho (Universidade Federal do Rio de Janeiro, Brazil). Kerogen was isolated from the rock matrix using standard non-oxidative procedures (Tyson, 1995; Mendonça Filho et al., 2002, 2010, 2023). The samples were crushed to a size of about 2–5 mm and acid washed successively to remove carbonates, silicates, and neoformed fluorides (HCl 37% for 18 h, HF 40% for 24 h, and HCl 37% for 3 h, respectively). Between each step, samples were washed with distilled water until the rinse was neutral. Thereafter, ZnCl₂ (density = 1.9 to 2 g/cm³) was added to the samples to separate the kerogen from sulfides using a centrifuge. The floated organic material was then washed with HCl (10%) and distilled water to eliminate ZnCl₂. Isolated kerogen was sieved at 10 µm diameter and mounted as strew slides.

Aliquots of the 2018 and 2020 RRE samples were sent to participants for preparation of pellets in accordance with ISO 7404-2 (2009). Difficulties were reported from conventional water-based preparation including excessive polishing relief, presence of undulating or inclined surfaces of rock grains, and the lack of coherence of organic matter with bituminous mineral-rich groundmass causing a porous examination surface and scalloping. Swelling of phyllosilicates was invoked as a cause of preparation difficulties.

The 2016 RRE samples and KI were made available to 24 participants. Twenty-two participants provided bituminite descriptions, 22 provided random vitrinite reflectance (VR_o %) values, and 21 provided random bituminite reflectance (BitR_o %) values. The 2018 RRE samples were provided to 23 participants. All 23 participants provided bituminite descriptions and VR_o % values, and 22 provided BitR_o % values. For the 2020 RRE, samples were sent to 19 participants and all participants provided bituminite descriptions, VR_o %, and BitR_o % values.

2.4. Incident light microscopy methods

Identification and reflectance measurements were performed by each participant under incident bright-field and fluorescence mode (UV/blue light excitation) using reflected light microscopes of different brands and types.

Incident light bright field microscopy employs a white light source (12 V 100 W tungsten halogen bulb or LED), oculars with 10× magnification, and an oil immersion objective lens, generally featuring magnifications of 40–60× with brand-related numerical aperture and free working distance. Photometers included photomultiplier tubes, diode arrays, and digital cameras (e.g., ASTM D7708, 2023; Gonçalves et al., 2024). The calibration standards of individual participants, based on their own laboratory standards, included synthetic glass (e.g., N-LASF), spinel, and leuco-sapphire, covering a reflectance range from about 0.4% to about 1.0%.

Incident light fluorescence microscopy was conducted using stabilized HBO 100 W/2 high pressure mercury arc lamps or similar illumination sources, and different filter sets generally including excitation band pass (e.g., 365 nm), beam splitter (e.g., 395 nm), and emission long pass (e.g., 397 nm, 420 nm).

Identification of vitrinite in in organic carbon-rich intervals of the Kimmeridge Clay Formation in the 2016, 2018, and 2020 RRE adhered to ASTM D7708 (2014b) using the general definition of vitrinite (vitrodetrinite) reported in International Committee for Coal and Organic Petrology (ICCP) (1998). Identification of bituminite in the Kimmeridge Clay Formation in the 2016 RRE followed the ICCP terminology of bituminite approved at the 67th ICCP Meeting in Potsdam in 2015 and supplied to participants. Participants used a bituminite description sheet (Appendix A) to provide a description and characterisation of bituminite in the whole rock sample and KI. Identification of bituminite in the 2018 and 2020 RRE was performed according to the ICCP terminology for bituminite (Pickel et al., 2017). Participants performed random reflectance measurements of vitrinite and bituminite in polished whole rock pellets and KI in accordance with ASTM D7708 (2014b). VR_o % and BitR_o % were reported to 3 decimal places,

followed by the number of measurements, and standard deviation (SD). SD was reported to 4 decimal places.

2.5. Organic geochemical screening

Total organic carbon (TOC) content of the RRE samples was determined at the Organic-geochemical and Petrological Laboratory of the Federal Institute for Geosciences and Natural Resources, Hannover, Germany with a LECO CS-230 elemental analyser (LECO Instruments, Germany) using decalcified (10% hydrochloric acid; HCl at 80 °C) and dried samples. About 180 mg of each sample was burned in a high frequency induction furnace in an oxygen atmosphere and quantification was achieved by use of the absorption signal at the infrared detector. The instrument was calibrated using a suite of commercially available standards (LECO). Reproducibility was $\pm 0.02\%$.

All RRE samples were analyzed using a Rock-Eval 6™ analyser (Vinci Technologies) using a standard programme (according to Espitalié et al., 1977; Lafargue et al., 1998). The samples were treated as follows: isothermal phase at 300 °C (for 3 min), then heating at a rate of 25 °C/min to 650 °C. Hydrocarbons (thermal distillate S1 and pyrolyzate S2)

and CO₂ (S3) evolved were determined using a flame ionization detector (FID) and a thermal conductivity detector (TCD), respectively. Hydrogen and oxygen indices (HI and OI, respectively) were calculated from the S2 and S3 peaks in the pyrograms [$HI = S2 \times 100/TOC$ (mg hydrocarbons (HC)/g TOC) and $OI = S3 \times 100/TOC$ (mg CO₂/g TOC)]. The T_{max} value, a measure of the thermal maturity of the sample, was determined as the temperature at which the maximum rate of pyrolytic hydrocarbon generation occurred.

2.6. Round robin exercises

All three RRE were performed within the ICCP IDOM WG. The RRE focused on both (1) microscopic characterisation of bituminite, and (2) random reflectance measurements of vitrinite and bituminite. Investigations of organic matter in KI were addressed only in the 2016 RRE.

The convenor required RRE participants to identify bituminite according to its published definition and description (Pickel et al., 2017). The bituminite description sheet (Supplementary material) was circulated to help standardise the responses of RRE participants of the optical

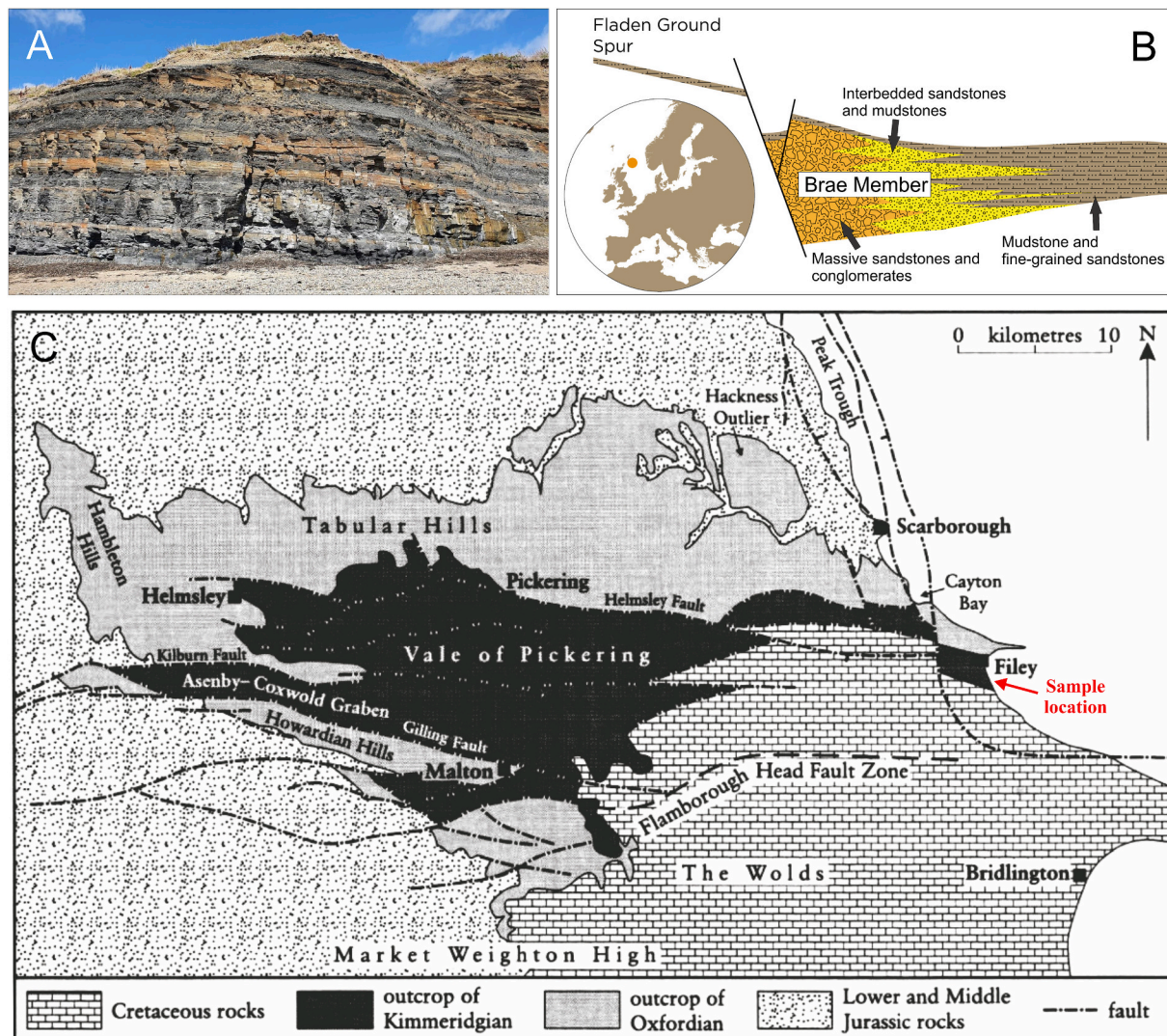


Fig. 2. Outcrops of the Kimmeridge Clay Formation in the United Kingdom and schematic cross-section in the North Sea. A – Kimmeridge Clay Formation outcrop in Kimmeridge Bay, Dorset, England, United Kingdom. <https://www.kimmeridgebay.org/>. B – Generalised cross-section through a submarine fan system of the Kimmeridge Clay Formation Brae member in the South Viking Graben, North Sea. Modified after Raji et al. (2015). <https://geoexpro.com/the-best-of-brae/>. C – Map showing the Oxfordian and Kimmeridgian beds as well as sample location in the area around Filey Bay, North Yorkshire, England, United Kingdom after Wright and Cox (2001).

appearance of bituminite. A synthesized scheme for bituminite description also was provided to help participants to complete a useful record of its optical characteristics. Likewise, the convenor provided guidance text for each RRE, specifying the reporting style for VR_o % and BitR_o %. For determination of BitR_o %, participants were asked to select measurement sites free of granular internal structure per recommendations of the IDOM WG from the 2017 69th ICCP Meeting. Finally, the convenor provided a compilation and summary of the main outcomes and conclusions related to bituminite identified in source rocks (Kus et al., 2017).

Difficulties with instrument calibration or sample mislabelling were not reported, although preparation-related difficulties were reported for the 2018 and 2020 RRE. In this case, the reported VR_o % and BitR_o % measurements may be vulnerable to biases, which impacted participant outcomes. Although not compulsory, some participants provided feedback related to more detailed bituminite description or to VR_o % and BitR_o % measurements.

3. Results and discussion

For clarity, the applied microscopic characterisation of bituminite is provided in Table 2 and displayed in Fig. 3 to elucidate the descriptions of bituminite forms and mode of appearance.

3.1. 2016 round robin exercise

Geochemical data for the 2016 RRE Kimmeridge Clay Formation sample are provided in Table 3.

3.1.1. General comments

Bituminite was mostly described by participants as forming irregular streaks and laminae with bands and elongated lenses, and minor wisps analogous to Fig. 3 and Table 2. Participants also reported lenses of bituminite and amorphous organic matter (OM) with remnants of various organisms (not further differentiated). One participant provided a differentiation into bituminite A (translucent and darker colour in incident white light with dark orange to brownish fluorescence) and bituminite B (translucent and lighter colour in incident white light with yellow fluorescence). In contrast to the KI, margins between dark and light parts of bituminite in incident white light in whole rock pellets were more evident.

The size of bituminite laminae and streaks varied in length from c.2–4 µm to c.70–200 µm (Fig. 4A–F). Bituminite thickness of the longer (c.70–200 µm) laminae and streaks was wide-ranging and variable from c.2–40 µm (Fig. 4C, D–F). In comparison, the thickness of bituminite lenses was < c.10 µm with lengths ranging from c.5–150 µm (Fig. 4A). Flaser length varied from c.10–20 µm. The appearance of bituminite varied from mostly vein-like (Fig. 4D) (discontinuous to elongated and irregular), followed by minor streaks and laminae, to rare diffuse and equidimensional (i.e., plate-like) bituminite of various shapes. Vein-like bituminite was reported to show diffuse margins, with smaller laminae appearing more diffuse than larger bituminite of more homogenous appearance. Rare particles with translucent brown borders and dark centers were also reported. Participants were not unanimous in their description of bituminite impurities, reporting both bituminite

Table 2

Description of bituminite forms and modes of appearance reported in the 2016, 2018, and 2020 round robin exercises.

Bituminite form or mode of appearance	Definition
Laminae	Form – Thin (< c.5–10 µm), parallel, elongated, and usually uniform, parallel to sub-parallel layers of bituminite deposited parallel to sub-parallel to micro-layering. Even and usually characterised by alternating layers of bituminite, particularly common in fine-grained marine sediments.
Irregular streaks	Form – Elongated, unevenly shaped, non-uniform, and lacking in steadiness or regularity of occurrence marks or patterns of bituminite of discontinuous lateral extent.
Wisps	Form – Thin (< c.5–10 µm), short, and discontinuous bodies.
Flaser	Form – Layers of aggregates composed of lenticular and wavy bodies spreading out laterally. Individual bodies are frequently neighbouring each other.
Pods	Form – Isolated, elongated, lenticular pod-like bodies of bituminite, occurring rarely in large continuous units.
Threads	Form – Thin (< c.5–10 µm) laminae of discontinuous character.
Bands	Form – Thick (> c.10 µm), parallel, and usually non-uniform, horizontal layers of bituminite deposited parallel to sub-parallel to micro-layering. Individual bands can occur on top of each other and can be arranged in alternating bands; particularly common in fine-grained marine sediments.
Elongated lenses	Form – Elongated flat-shaped convex-lens-like bodies, gradually thinning out in a lateral direction; individual lenses may form lenticular or lentiform aggregates occurring along parallel to sub-parallel micro-layering.
Clast or plate-like	Mode of appearance – Thick (> c.10 µm) clast-like, homogenous, diffuse bodies of bituminite with sizes of c.10 × 10 to c.15 × 15 µm.
Vein-like	Mode of appearance – Branching networks of thin (< c.5–10 µm) laminae of bituminite resembling intricate, web-like, or cord-like laminae patterns, discontinuous, elongated, irregular.
Finely disseminated	Mode of appearance – Uniformly scattered, dispersed throughout the mineral-rich bituminous groundmass rather than concentrated or amalgamated.

characterised by impurities and bituminite lacking impurities. Reported impurities include minerals, corroded (degraded) algal remnants, and liptodetrinite (Fig. 4B). Minerals were predominantly represented by framboidal pyrite (Fig. 4D), followed by quartz, and carbonate. Clay mineral impurities were also reported, occurring at sizes of < c.1–2 µm either interlaminated with bituminite or disseminated in the form of small lenses. In addition, inertinite (micrinite in particular) was reported as a rare impurity in bituminite. In terms of internal structure as it relates to mineral inclusions, the dominant internal arrangement was fine granular and streaky to flow-like (aligned, internal structure). Participants also occasionally described bituminite internal structure as homogenous, spongy, and corroded, or massive. The colour of bituminite in incident white light was pale to dark gray and translucent brown to dark brown with reddish tint or reflections. A black appearance in white

Table 1

Sample information used in the 2016, 2018, and 2020 round robin exercises (RRE) of the Identification of Dispersed Organic Matter Working Group.

RRE	Location	District	Depth (m)	Sampling Bed
RRE 2016	Kimmeridge Bay, England, United Kingdom	Purbeck	0.0 (outcrop at sea level)	Eudoxus Zone (lower), bed group 26 (Morgans-Bell et al., 2001)
RRE 2018	Filey Bay, England, United Kingdom	North Yorkshire	0.0 (outcrop at sea level)	Pectinatus Zone, bed groups 46–49 (Wignall, 1993)
RRE 2020	16/17–14 well, Viking Graben, North Sea	UK Quadrant 16	(4200.81 m; 13,782.2 ft. – 4200.91 m; 13,782.5 (sub-Kelly Bushing)	NA

RRE – round robin exercise; NA – not available.

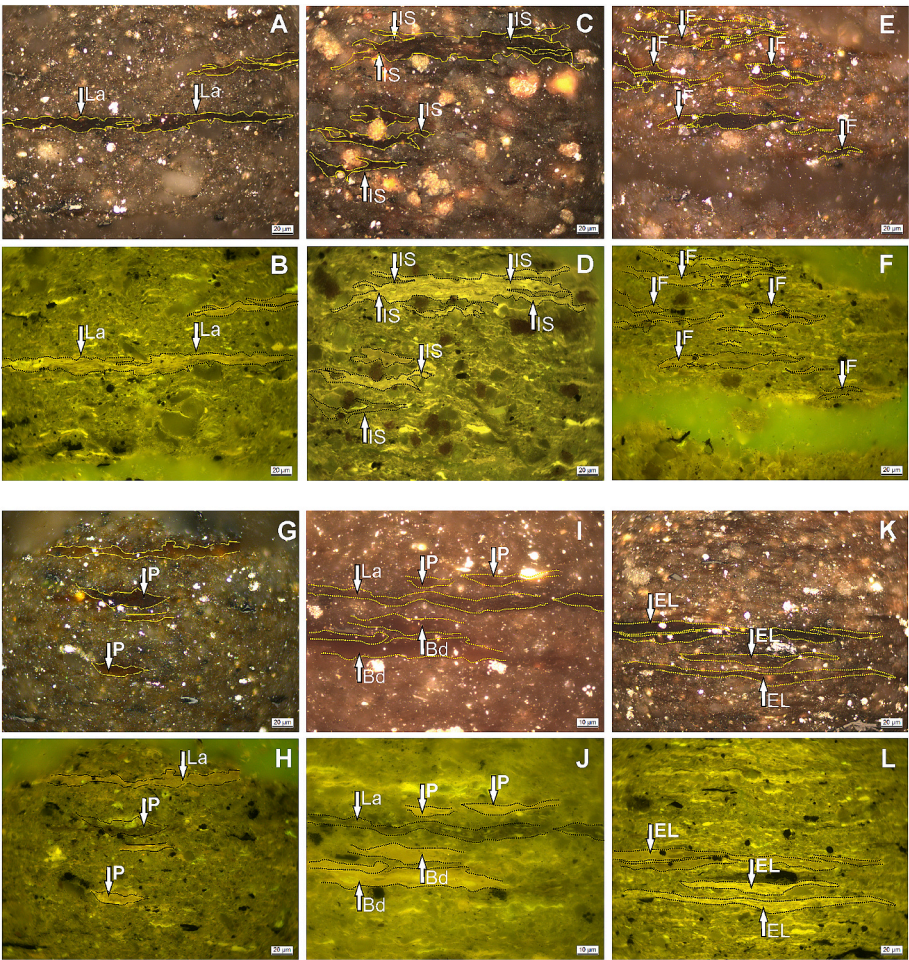


Fig. 3. Photomicrographs of bituminite forms in Kimmeridge Clay Formation, Kimmeridge Bay, Dorset, England, United Kingdom, investigated during the 2016 round robin exercise in reflected white light (A, C, E, G, I, K) and the same fields under blue-light excitation (B, D, F, H, J, L) using oil immersion. A – B Laminae of bituminite; C – D Irregular streaks of bituminite; E – F Flaser of bituminite; G – H Laminae and pods of bituminite; I – J Laminae, pods, and bands of bituminite; K – L Elongated lenses of bituminite; La – Laminae of bituminite; IS – Irregular streaks of bituminite; F – Flaser of bituminite; P – Pods of bituminite; Bd – Bands of bituminite; EL – Elongate lenses of bituminite. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

Table 3

Summary organic carbon and programmed pyrolysis data of samples from Upper Jurassic Kimmeridge Clay Formation from the 2016, 2018, and 2020 round robin exercises (RRE).

Code	TOC %	S _{total} %	S1 mg/g	S2 mg/g	S3 mg/g	T _{max} °C	HI mg HC/g TOC	OI
RRE 2016	12.10	1.64	1.08	68.60	1.01	419	666	10
RRE 2018	8.62	2.37	0.70	46.44	1.44	418	539	17
RRE 2020–1	5.32	4.95	3.97	6.01	0.36	439	113	7
RRE 2020–2	5.38	2.80	4.03	11.62	0.17	445	214	3

TOC – total organic carbon in weight %; S_{total} – total sulfur in weight %; S1 – free hydrocarbons present in the sample before the analysis in mg hydrocarbon/g rock; S2 – volume of hydrocarbons that formed during thermal pyrolysis of the sample in mg hydrocarbon/g rock; S3 – CO₂ yield during thermal breakdown of kerogen in mg CO₂/g rock; T_{max} in °C is the program temperature at peak S2 emission; HI – hydrogen index in mg hydrocarbon/g TOC (S2*100/TOC); OI – oxygen index in mg CO₂/g TOC (S3*100/TOC).

light was rarely reported. Colour under UV or blue light ranged from yellowish to brownish with rarely reports of greenish to reddish fluorescence. Only minor bituminite was reported as non-fluorescent.

Participants identified bituminite primarily from the description of bituminite I from [Teichmüller and Ottenjann \(1977\)](#). Other definitions of bituminite were very rarely selected such as that of bituminite I of Beaufort-Mackenzie Basin ([Creaney, 1980](#)), amorphinite A, B, and C ([van Gijssel, 1982](#)), or sapropelinite I ([Mukhopadhyay et al., 1985a, 1985b](#)). Some prior bituminite descriptions were not used by any

participant, such as bituminite II from the Mackenzie-Basin ([Creaney, 1980](#)) and sapropelinite II ([Mukhopadhyay et al., 1985a, 1985b](#)). Participants acknowledged that selecting a certain previously defined bituminite description as an appropriate description of bituminite in the 2016 RRE sample was challenging due to the amorphous outline of bituminite itself, even at high magnifications, or due to difficulties in accessing the respective literature. Finally, a clear differentiation of bituminite and solid bitumen ([Fig. 4A–F](#) and [Fig. 4H, J, L](#), respectively) was questionable when both were embayed by euhedral carbonate

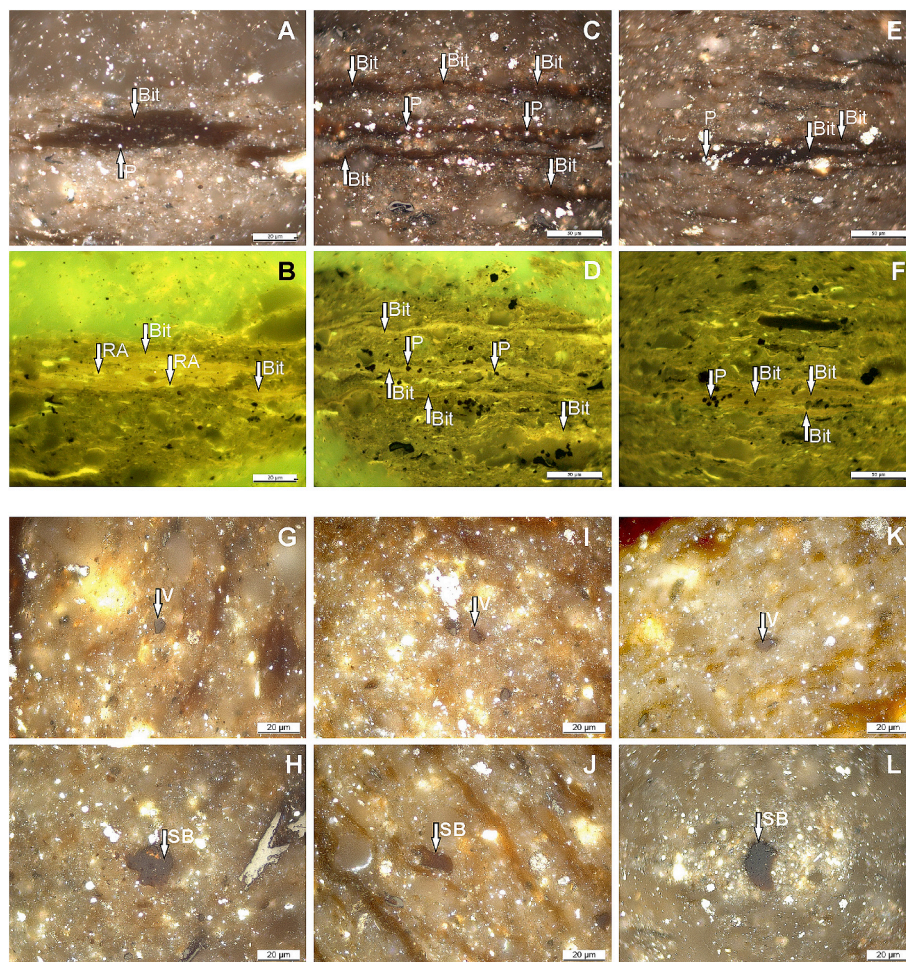


Fig. 4. Kimmeridge Clay Formation in the 2016 round robin exercise with the sample from Kimmeridge Bay, Dorset, England, United Kingdom. A – F Photomicrographs of laminae, irregular streaks, and bands of dark brown to black bituminite (Bit) in incident white light (panels A, C, E) and pale to dark orange in blue light illumination (Bit; panels B, D, F), oil immersion. Note remains of corroded (degraded) alginite macerals (RA) and association of pyrite microcrystals (P). G, I, K – Photomicrographs of vitrinite (V), in incident white light, oil immersion. H, J, L – Photomicrographs of solid bitumen (SB) finely dispersed in mineral-bituminous groundmass, in incident white light, oil immersion. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

crystals. For the reference to description of solid bitumen the reader is referred to Jacob (1989), Mastalerz et al. (2018), and Sanei (2020). Corresponding lack of clear differentiation of bituminite and solid bitumen can be noted in Fig. 4A and C, where morphology of bituminite (pods) appears to be similar to the morphology of solid bitumen in Fig. 4H, J, L.

3.1.2. Vitrinite reflectance in whole rock pellet

Examples of vitrinite from the 2016 RRE sample are displayed in Fig. 4G, I, K. Results (including outliers) from VR_o % measurements in the whole rock pellet are compiled in Fig. 5A, C, and Table 4, and reported in Kus et al. (2026). The number of individual VR_o % measurements per participant ranged from 4 to 100 with an average of 35. Mean VR_o % values ranged from 0.34 to 0.74% with a group mean (GM) of 0.47% (Fig. 5A, Table 4). The group standard deviation (GSD) of 0.10 from 22 participants ($N = 22$) indicated a high scatter of measured reflectance values. The evident outliers are illustrated by four data points showing anomalous SD (0.17; 0.14; 0.14; 0.14) and one high reflectance value (0.74%; Fig. 5C). The VR_o % histogram for the group results indicated a bimodal distribution of vitrinite data (Fig. 5A). Bimodality is reflected by 2 peaks at c.0.40% and 0.57% VR_o . Application of the criterion $SD < 0.15 \times VR_o$ % for acceptable mean random vitrinite reflectance values used in the ICCP Dispersed Organic Matter Vitrinite Reflectance (DOMVR) Accreditation Program (<https://www.>

[iccop.org/accreditation/general-information/](https://www.iccop.org/accreditation/general-information/)) and applied by Hackley et al. (2020), resulted in elimination of 9 mean VR_o % values from the group results and a modified GM result of 0.46% VR_o with $N = 13$ and GSD of 0.08. The range of mean VR_o % values in the modified GM are from 0.34 to 0.58%.

3.1.3. Bituminite reflectance in whole rock pellet

$BitR_o$ % measurements in the 2016 RRE whole rock pellet are compiled in Fig. 5A and Table 4. The number of $BitR_o$ % measurements per participant varied from 4 to 113 with an average of 51. The mean $BitR_o$ % varied between 0.11 and 0.29% with GM of 0.19%. The GSD of 0.05 was obtained by 21 participants and indicated a low scatter of measured reflectance values. One noticeable outlier has an anomalous SD (0.17%; Fig. 5D). The $BitR_o$ % histogram illustrated a unimodal distribution (Fig. 5A) with a peak at 0.19% $BitR_o$. As noted by some participants, reliable $BitR_o$ % measurements were obtained from dark gray bituminite lacking micrinite inclusions. Using the criterion $SD < 0.15 \times BitR_o$ % for acceptable mean random $BitR_o$ % from the ICCP DOMVR Accreditation Program, the modified GM of $BitR_o$ % is 0.20% for $N = 11$ results ranging from 0.12 to 0.29%, with discard of the results from 10 participants. The modified GSD is 0.05.

3.1.4. Vitrinite reflectance in kerogen isolate

VR_o % data from the KI are shown in Fig. 5B and Table 4. VR_o %

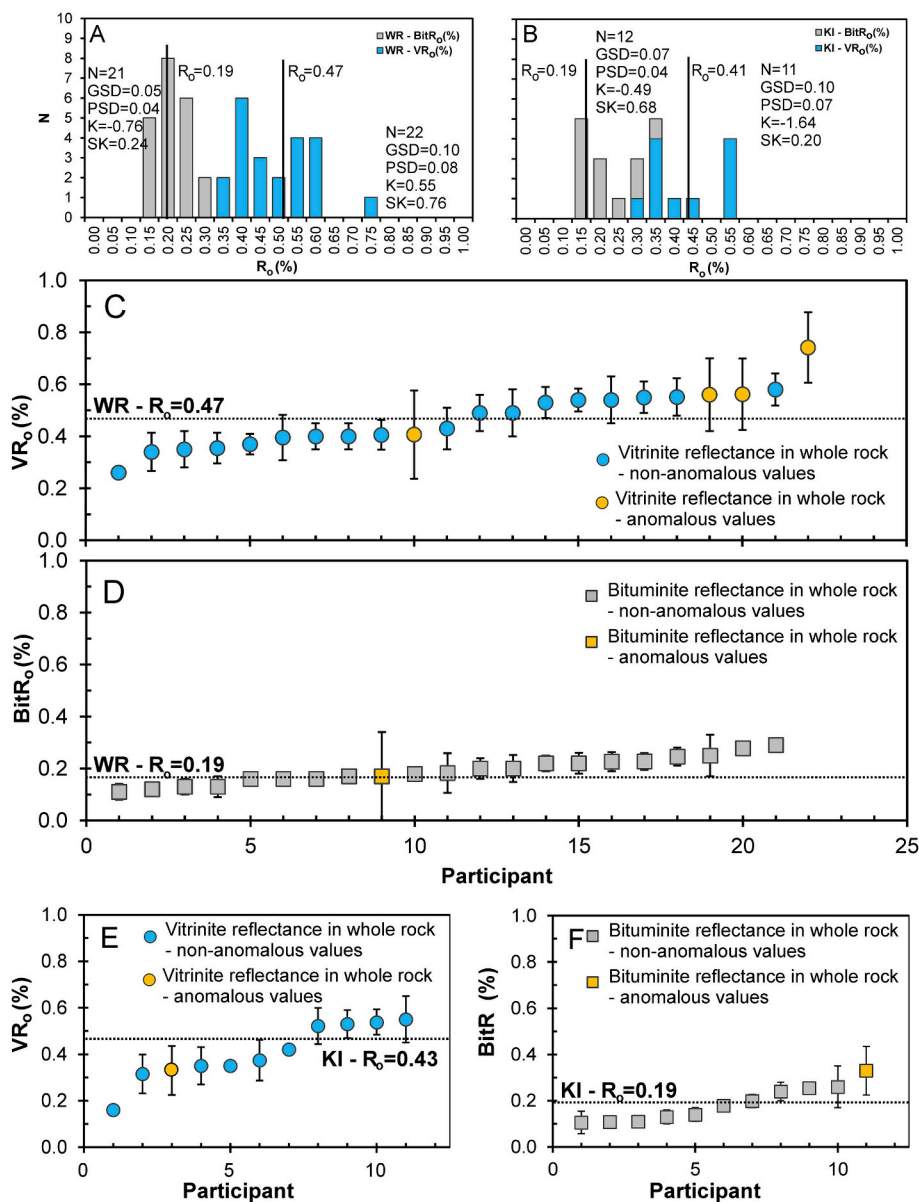


Fig. 5. Data from organic carbon-rich intervals of the Kimmeridge Clay Formation in the 2016 round robin exercise in Kimmeridge Bay. A – Reflectance histogram of bituminite reflectance (BitR_o in %, gray) and vitrinite reflectance (VR_o in %, blue) in whole rock (WR) pellet. B – Reflectance histogram of BitR_o and VR_o in kerogen isolate (KI) pellet. C – Distribution of VR_o in WR pellet with four anomalous high standard deviation (SD, 0.17; 0.14; 0.14; 0.14) values and one anomalously high VR_o value (0.74%) marked in orange. D – Distribution of BitR_o in WR pellet with one anomalous high SD (0.17) marked in orange. E – Distribution of VR_o in KI with one anomalous high SD (0.11) marked in orange. F – Distribution of BitR_o in KI with one anomalous high SD (0.17) marked in orange. N – number of measurements; GSD – group standard deviation; PSD – personal standard deviation (average standard deviation reported by all participants); K – kurtosis; SK – skewness; R_o – random reflectance. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article).

values range from 0.26 to 0.55% in the KI and are comparable with the modified range (using only $SD < 0.15 \times VR_o$) from the whole rock pellet (0.34 to 0.58%). The average VR_o % in the KI is 0.41%, lower than the VR_o % value for the whole rock pellet (0.47%). While the number of measurements ($N = 11$) is lower in comparison to whole rock pellet ($N = 22$), the GSD of 0.10 in KI is equal to GSD (0.10) for whole rock pellets. We speculate that fewer participants reported results from the KI than from the whole rock pellet because of difficulties with maceral identification due to the finely ground nature of the KI and the loss of the rock texture. One apparent outlier has an anomalous SD (0.11; Fig. 5E). Only six VR_o % values from the KI are within the criterion $SD < 0.15 \times VR_o$ %, which indicates difficulties for identification of representative (first-cycle) vitrinite once the rock texture has been eliminated by acid

digestion. The VR_o % histogram (Fig. 5B) indicates a bimodal distribution with two peaks at 0.34 and 0.54%.

3.1.5. Bituminite reflectance in kerogen isolate

BitR_o % values obtained from the KI are displayed in Fig. 5B, F, and Table 4. As shown, BitR_o ranges from 0.11 to 0.33% and is comparable to that for the modified range of BitR_o % in the whole rock pellet (using $SD < 0.15 \times BitR_o$ %, the modified range is 0.11 to 0.29% in the whole rock pellet). Average BitR_o % is 0.19%, comparable to the value from the whole rock pellet (0.19%). Although the number of measurements ($N = 12$) is lower than the whole rock pellet ($N = 21$), the GSD of 0.07 is higher in comparison (0.05). This is associated with one noticeable outlier with anomalous SD (0.11; Fig. 5F). The BitR_o % histogram

Table 4

Summary of vitrinite (VR₀) and bituminite reflectance (BitR₀) measurements in Upper Jurassic Kimmeridge Clay samples from the 2016 round robin exercise (RRE). SD – standard deviation. N – number of measurements. UMSD – unsigned multiple of the standard deviation. NA – analyst not available in this RRE (analysts 1–29 are the same in all 3 RRE). UMSD values >1.5 are displayed in bold font.

Analyst	Vitrinite in 2016 RRE				Bituminite in 2016 RRE			
	VR ₀ %	SD	N	UMSD	BitR ₀ %	SD	N	UMSD
1	0.40	0.09	9	0.754	0.12	0.02	53	1.379
2	0.49	0.09	80	0.181	0.13	0.03	100	1.184
3	0.35	0.07	31	1.201	0.20	0.04	59	0.178
4	NA	NA	NA	NA	NA	NA	NA	NA
5	0.40	0.05	21	0.707	0.16	0.02	21	0.600
6	NA	NA	NA	NA	NA	NA	NA	NA
7	0.40	0.05	21	0.707	0.16	0.02	21	0.600
8	0.53	0.06	28	0.576	0.22	0.04	30	0.567
9	0.43	0.08	4	0.411	0.22	0.03	4	0.567
10	0.54	0.04	100	0.674	0.18	0.02	100	0.231
11	0.41	0.17	14	0.644	0.18	0.08	19	0.163
12	0.58	0.06	30	1.069	0.20	0.05	100	0.178
13	0.56	0.14	68	0.872	0.13	0.04	41	1.184
14	0.56	0.14	16	0.887	NA	NA	NA	NA
15	0.41	0.06	9	0.653	0.28	0.01	13	1.693
16	0.37	0.04	11	1.003	0.29	0.01	28	1.928
17	0.74	0.14	50	2.665	0.23	0.04	50	0.689
18	NA	NA	NA	NA	NA	NA	NA	NA
19	0.55	0.06	28	0.773	0.25	0.08	28	1.150
20	0.55	0.07	13	0.785	0.25	0.04	11	1.063
21	0.34	0.07	30	1.299	0.17	0.03	30	0.406
22	0.36	0.06	70	1.151	0.23	0.03	113	0.703
23	NA	NA	NA	NA	NA	NA	NA	NA
24	NA	NA	NA	NA	NA	NA	NA	NA
25	0.39	NA	NA	NA	0.14	NA	NA	NA
26	0.49	0.07	7	0.181	0.11	0.03	100	1.573
27	NA	NA	NA	NA	NA	NA	NA	NA
28	NA	NA	NA	NA	NA	NA	NA	NA
29	0.54	0.09	100	0.674	0.17	0.17	100	0.406

(Fig. 5B) indicates a bimodal distribution marked by two peaks of 0.15% and 0.35% BitR₀. Similar to the whole rock pellet, when the acceptability criterion is applied ($SD < 0.15 \times \text{BitR}_0$ %), the reported BitR₀ % decreases to 0.17% with $N = 6$ and GSD of 0.06. GSD incorporates an acceptable scattering of BitR₀ % data ranging from 0.11 to 0.25%.

3.2. 2018 round robin exercise

The Kimmeridge Clay Formation sample from the outcrop near Filey Bay, North Yorkshire (Table 3) used for the 2018 RRE shows high TOC of 8.62 wt% and low T_{max} of 418 °C, suggesting thermally immature OM. S2 is 46.44 mg/g and total sulfur is 2.37%.

3.2.1. General comments

Participant comments from the bituminite description sheets for the 2018 RRE are summarized in the following paragraph. The main forms of bituminite included streaks, lenses, and laminae, whereas wisps, threads, bands, and pods were rarely described (Fig. 6A–F), with bands the least commonly reported form. Streaks were described as irregular, laminae as filamentous, and lenses as elongated (Fig. 6A–B). Bituminite A (a translucent and darker colour in incident white light with dark orange to brownish fluorescence) was reported to form irregular streaks and short lenses, whereas bituminite B (a translucent and lighter colour in incident white light with yellow fluorescence) was characterised by threads and elongated lenses (Fig. 6C–F) or irregular streaks. Laminae, flaser, “Schlieren” lenses, and threads were mostly present in a clayey laminated lithotype, whereas irregular forms and wisps were encountered in a coarser-grained lithotype. Difficulty of recognition of bituminite due to its indistinguishable boundaries also was reported. Finally, darker and more compact bituminite showed a continuous transition to

bituminous mineral-rich groundmass.

The sizes of bituminite laminae, streaks, and threads varied in length from c.2 to <200 µm and in thickness from <2–30 µm, whereas lenses were generally reported to be up to <100 µm in length, although 6 analysts reported lenses up to >200 µm in length, and typically <2–10 µm in thickness. One participant reported large clast-like particles with larger diameters of various shape (e.g., 35 × 70 µm).

Bituminite in the 2018 RRE had either a vein-like, finely disseminated appearance or occurred as clast-like particles with larger diameters. Bituminite of “laminated” form and lenses occurred parallel or sub-parallel to sedimentary microlamination. Occasionally, a vein-like or finely disseminated appearance of bituminite was reported. It was suggested that the word “particle” should be avoided as a bituminite descriptor because sometimes its boundaries are not as clear as discrete particles embedded in a matrix. Finally, bituminite A and B were mainly described as homogenous, diffuse, clast-like bodies.

Impurities in bituminite included minor minerals, corroded algal remnants, and liptodetrinite. Observed minerals included pyrite microcrystals and framboidal pyrite, quartz, and clays, in order of abundance. Bituminite A was mainly associated with pyrite with included algal remnants from dinoflagellate cysts and acritarchs, whereas liptodetrinite inclusions were thought to be fine-grained *Prasinophytes* alginite. Bituminite B was mainly associated with inclusions of liptodetrinite, sporinite, and alginite. Bituminite was also reported to include micrinite, other inertinite, and, to a much lesser extent, vitrinite.

The internal structure of bituminite was described as fine granular and streaky as opposed to dominantly homogenous and flow-like (aligned, internal structure). In incident white light, bituminite was mostly dark gray to brown, rarely black or pale gray. Upon UV or blue light examination, bituminite predominantly displayed yellowish to brownish translucent fluorescence. The lack of bituminite fluorescence was reported by one participant, whereas another reported bituminite B with intense yellow to yellowish fluorescence, and bituminite A with brownish fluorescence. Participants associated bituminite mostly with bituminite I of Teichmüller and Ottenjann (1977), whereas bituminite II, III of Teichmüller and Ottenjann (1977) and bituminite I of Creaney (1980) were less frequently reported. Other definitions of bituminite were rarely reported, e.g., bituminite II of Creaney (1980), amorphinite A, B, and C (van Gijssel, 1982), sapropelinite I and II (Mukhopadhyay et al., 1985a, 1985b), or fluoramorphinite (Senftle et al., 1987). One participant suggested that bituminous mineral-rich groundmass should be described from the 2018 RRE sample instead of bituminite, i.e., the sample did not contain representative bituminite.

One participant drew a comparison between the optical appearance of bituminite in the 2016 and 2018 RRE samples, concluding that (1) the outline of bituminite was more visible and (2) the contrast between bituminite and bituminous mineral-rich groundmass was greater in the 2018 RRE sample, giving rise to more confident measurements of BitR₀ %. Bituminite inclusions of framboidal pyrite and micrinite were more evident when inserting the field diaphragm, with the bituminite surface described as ‘rough’, reflecting, and finely granular. Finally, it was suggested that the historical division of bituminite and equivalents (amorphinite, sapropelinite) into multiple sub-macerals (Creaney, 1980; Mukhopadhyay et al., 1985b; Teichmüller and Ottenjann, 1977; van Gijssel, 1982) was unhelpful, as these terms cannot be applied in a reproducible way. Further, this participant recommended to: (1) use the term “bituminite” as a general statement to refer to a broad category of organic matter in a continuum with other macerals, including solid bitumen, mineral-bituminous groundmass, and lamalginite (e.g., Hackley et al., 2018) and (2) to describe in more detail, if preferred or required, its optical appearance and properties.

3.2.2. Vitrinite reflectance

As noted above, participants reported preparation difficulties from swelling clays for the 2018 RRE sample, leading to a limited number of suitable particles for reflectance measurement and ultimately

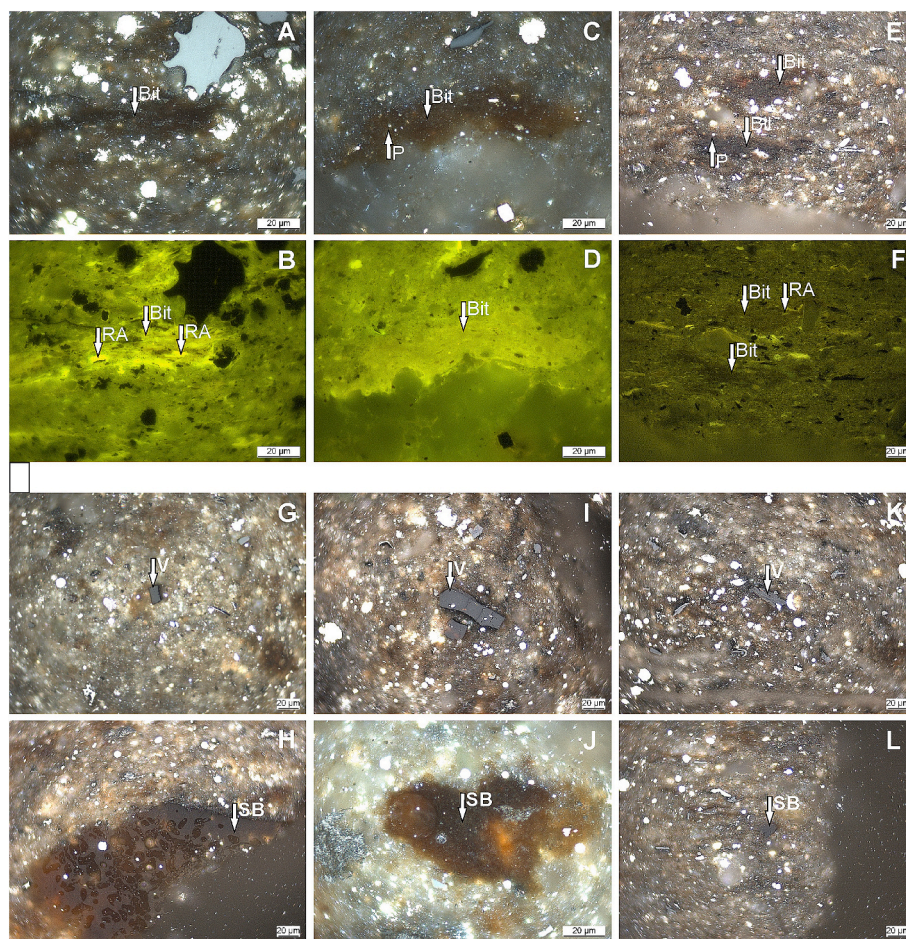


Fig. 6. Kimmeridge Clay Formation used in the 2018 round robin exercise with the sample from Filey Bay, North Yorkshire. A – F Photomicrographs of streaks and stringers of dark brown to dark gray bituminite (Bit) in incident white light (panels A, C, E) and pale to dark orange in blue light illumination (Bit; panels B, D, F), oil immersion. Note remnants of alginite macerals (RA) and association of pyrite microcrystals (P). G, I, K – Photomicrographs of vitrinite (V), in incident white light, oil immersion. H, J, L – Photomicrographs of solid bitumen (SB) finely dispersed in bituminous mineral-rich groundmass, in incident white light, oil immersion. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

complicating maceral identification. Examples of vitrinite from the 2018 RRE sample are displayed in Fig. 6G, I, K. VR_o % data is shown in Fig. 7A, 6B, and Table 5. Overall, mean values from 0.23 to 0.70% were obtained, with a GM of 0.46%, comparable to the 2016 RRE sample (range of 0.34–0.74 VR_o %; GM = 0.47 VR_o %). One participant provided two data sets (lower and higher reflectance values) which were included separately in the data pool considered for this sample. The high GSD = 0.11 obtained from the 23 values was similar to the GSD of 0.10 from the 2016 RRE sample and indicated a high dispersion of VR_o % data. Average SD was 0.06 with 3 noticeable outliers at SD of 0.10–0.13 and two anomalously low VR_o % values (both 0.23). Three of five analysts reporting outlying values did not hold ICCP DOMVR accreditation. The number of VR_o % measurements ranged from 9 to 200 with an average of 35, again comparable to the 2016 RRE (range of 4–100; avg. of 35). The group VR_o % histogram indicated a unimodal distribution (Fig. 7A). Using the criterion $<0.15 \times VR_o$ % to eliminate unacceptable results, GM increased to 0.49 VR_o % and GSD decreased to 0.07 with $n = 15$. This represents an acceptable scattering of VR_o % data with a modified range of 0.37 to 0.59%. The modified GSD and GM are comparable to the modified 2016 RRE results (modified GSD = 0.08; modified range of 0.34 to 0.58%).

3.2.3. Bituminite reflectance

Bit R_o % measurements are shown in Fig. 7A and Table 5. The number of individual measurements per participant ranged from 14 to 200 with

an average of 40. Bit R_o % values varied from 0.09 to 0.28% with GM of 0.19%, also similar to Bit R_o % reported for the 2016 RRE sample (range of 0.11–0.29%; GM = 0.19%). Low GSD of 0.04 ($N = 22$) indicated a low scatter of measured values, also in line with Bit R_o % reported for the 2016 RRE sample (GSD = 0.05; $N = 21$). The group Bit R_o % histogram showed a unimodal distribution (Fig. 7A, C). Application of the criterion $SD < 0.15 \times BitR_o$ % resulted in GM decrease to 0.17% for $N = 6$, essentially equivalent to the modified GM = 0.20 from $N = 11$ in the 2016 RRE. Likewise, GSD decreases to 0.03 in comparison to 0.05 in the modified 2016 RRE with a range of 0.15–0.24% in close comparison to the modified range (0.12–0.29%) from 2016.

3.3. 2020 round robin exercise

The sample of the Kimmeridge Clay Formation used in the 2020 RRE from the 16/17–14 well in the Viking Graben, North Sea, is characterised by high TOC with an average of 5.35% from 2 measurements. The oil source rock is thermally mature with T_{max} of 439 and 445 °C and average S2 of 8.82 mg HC/g rock from 2 measurements. These data, and the lower average HI of 164 mg/g indicate higher maturity in the 2020 RRE sample compared to the 2016 and 2018 RRE samples (Table 3).

3.3.1. General comments

Bituminite in the 2020 RRE sample was reported to occur in a gradational presentation spectrum, from bituminous mineral-rich

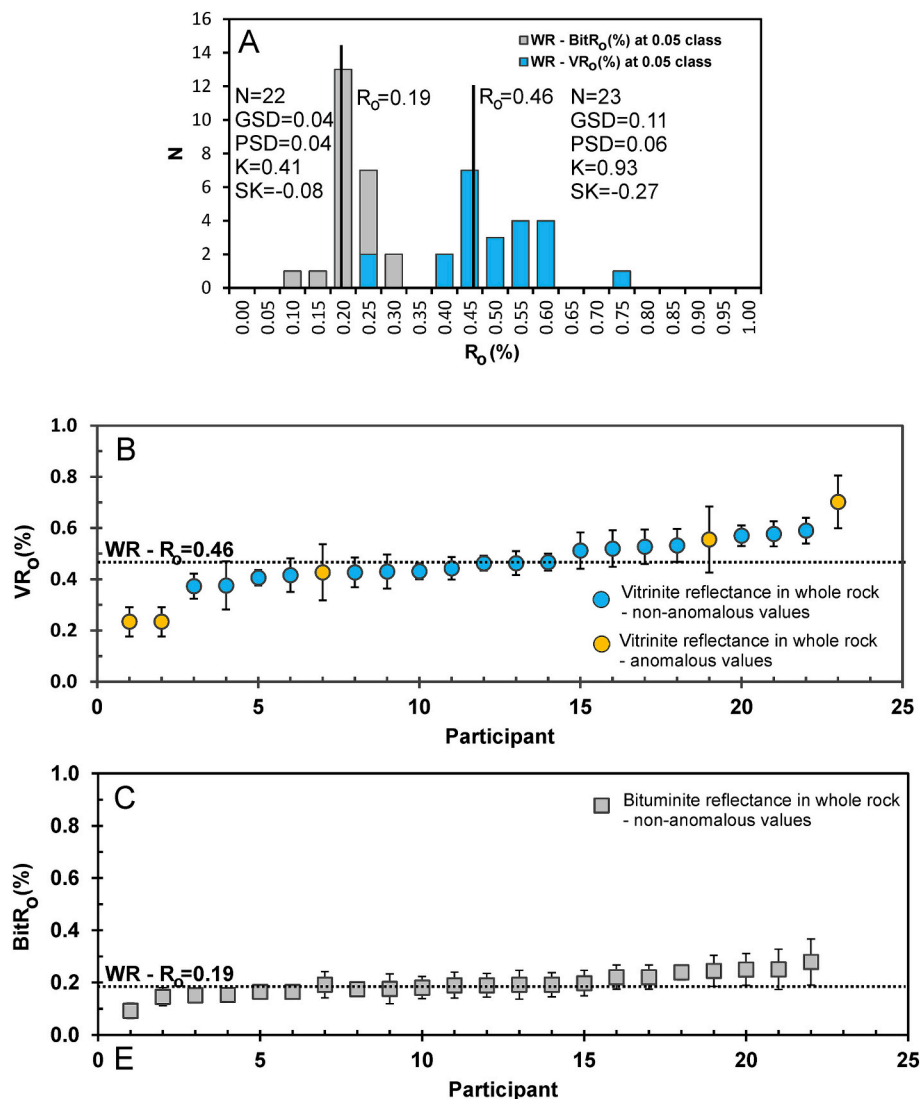


Fig. 7. Data from organic-carbon rich intervals of the Kimmeridge Clay Formation from Filey Bay, North Yorkshire, used in the 2018 RRE. A – Reflectance histogram of bituminite reflectance (BitR_o in %, gray) and vitrinite reflectance (VR_o in %, blue) in whole rock (WR) pellet. B – Distribution of VR_o in WR pellet with three anomalous SD values (0.10; 0.11; 0.13) and two anomalously low VR_o values (0.23%; 0.23%) marked in orange. C – Distribution of BitR_o in WR pellet. N – number of measurements; GDS – group standard deviation; PSD – personal standard deviation (average standard deviation reported by all participants); K – kurtosis; SK – skewness; R_o – random reflectance. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article).

groundmass, where bituminite is intimately associated with admixed minerals, to stringers of homogeneous fluorescent lamalginite, to *in-situ* development of solid bitumen with a gray reflecting surface (e.g., Hackley et al., 2018).

The dominant forms of bituminite ranged from laminae, streaks, lenses or pods, and bands (Fig. 8A–F). The laminae and streak forms were described as irregular and filamentous (Fig. 8F) with small- to point-sized laminae finely dispersed in the clay-rich groundmass. Elongate lenses and pods were less common, occurring in association with accumulations of terrigenous macerals. Bands were the least commonly occurring bituminite form.

Laminae (Fig. 8A) and band (Fig. 8C) forms varied in length from c.2–>200 μm and in thickness from <c.2–20 μm, similar to lenses and pods. Forms of laminae to bands were rarely >200 μm in length. Bituminite appearance was predominantly described as fine disseminations and equidimensional particles as opposed to vein-like, which was reported as secondary or a minor occurrence. Equidimensional (i.e., plate-like) bituminite forms with sizes of 10 × 10 to 15 × 15 μm were reported. Fine bituminite disseminations occurred in association with mineral inclusions (e.g., pyrite, feldspar, quartz). Similarly, corroded

(degraded) algal remnants and liptodetrinite were reported as bituminite inclusions. Corroded (degraded) algal remnants were interpreted as dinoflagellate cysts, whereas liptodetrinite was thought to represent faunal remains. Bituminite also was reported to enclose micrinite, solid bitumen, fusinite, cutinite, and rare vitrinite. The internal structure of bituminite was mainly fine granular (Fig. 8A), flow-like (aligned, internal structure), and streaky as opposed to be homogenous. In incident white light, bituminite was mostly dark gray with transition to brown (Fig. 8A, C, E), rarely black or pale gray. Upon UV or blue light irradiation, bituminite autofluorescence was dominantly brownish-reddish to orange fluorescence (Fig. 8B, D, F).

Participants ascribed bituminite in the 2020 RRE sample to bituminite I of Teichmüller and Ottenjann (1977), whereas bituminite III (Teichmüller and Ottenjann, 1977) was of secondary occurrence. Other definitions of bituminite were rarely reported, e.g., bituminite II (Teichmüller and Ottenjann, 1977), bituminite I and II of Creaney (1980), amorphinite A, B, and C (van Gijssel, 1982) or sapropelinite I and II (Mukhopadhyay et al., 1985a, 1985b). As with the 2018 RRE, one participant suggested to use the universal term of bituminite for all forms of bituminite and to add its brief description without selection of a

Table 5

Summary of vitrinite and bituminite reflectance measurements (VR_o % and BitR_o %, respectively) in samples from the Upper Jurassic Kimmeridge Clay Formation used in the 2018 round robin exercise (RRE). SD – standard deviation. N – number of measurements. UMSD – unsigned multiple of the standard deviation. NA – analyst not available in this RRE (analysts 1–29 are the same in all 3 RRE). UMSD values >1.5 are displayed in bold font.

Analyst	Vitrinite in 2018 RRE				Bituminite in 2018 RRE			
	VR _o %	SD	N	UMSD	BitR _o %	SD	N	UMSD
1	0.37	0.05	9	0.852	0.09	0.03	23	2.380
2	NA	NA	NA	NA	NA	NA	NA	NA
2	NA	NA	NA	NA	NA	NA	NA	NA
3	0.41	0.03	17	0.543	0.15	0.04	20	1.118
4	0.57	0.04	20	0.990	NA	NA	NA	NA
5	0.53	0.06	21	0.634	0.19	0.05	24	0.090
6	0.53	0.07	21	0.588	0.20	0.05	22	0.097
7	0.52	0.07	21	0.522	0.19	0.05	23	0.043
8	0.51	0.07	50	0.448	0.18	0.02	30	0.441
9	0.43	0.11	16	0.347	0.15	0.02	14	0.955
10	NA	NA	NA	NA	NA	NA	NA	NA
11	0.56	0.13	22	0.849	0.28	0.09	21	1.990
12	0.38	0.09	25	0.826	0.18	0.06	60	0.422
13	0.44	0.04	30	0.197	0.18	0.04	43	0.300
14	NA	NA	NA	NA	NA	NA	NA	NA
15	0.47	0.03	41	0.027	0.17	0.02	29	0.674
16	0.46	0.03	47	0.010	0.17	0.02	29	0.674
17	0.43	0.03	10	0.310	0.24	0.03	40	1.055
18	NA	NA	NA	NA	NA	NA	NA	NA
19	NA	NA	NA	NA	NA	NA	NA	NA
20	NA	NA	NA	NA	NA	NA	NA	NA
21	0.43	0.07	35	0.319	0.25	0.06	45	1.312
22	0.46	0.05	70	0.010	0.19	0.05	44	0.043
23	0.58	0.05	22	1.055	0.18	0.04	43	0.300
24	0.23	0.06	10	2.151	0.22	0.05	32	0.634
25	0.23	0.06	10	2.151	0.22	0.05	32	0.634
26	NA	NA	NA	NA	NA	NA	NA	NA
27	0.42	0.07	40	0.450	0.19	0.05	51	0.043
28	NA	NA	NA	NA	NA	NA	NA	NA
29	0.70	0.10	200	2.223	0.25	0.08	200	1.336

particular sub-macerals identification. Optical fading (reduction of fluorescence intensity upon UV/blue light excitation) of bituminite was also observed.

3.3.2. Vitrinite reflectance

Examples of vitrinite from the 2020 RRE sample are displayed in Fig. 8G, I, K. Fig. 9A–B and Table 5 display VR_o % data from the 2020 RRE sample.

A range of VR_o % values from 0.54 to 0.98% was documented, with a GM of 0.71%, indicating higher maturity than the 2016 and 2018 RRE samples, and consistent with the geochemical screening results (Table 3). Outliers with anomalous SD (0.15) and low reflectance value (0.54%) are included in the GM value. High GSD (0.10, $N = 19$) is comparable to the prior RRE (0.10–0.11) and indicated a high dispersion of VR_o % data. The number of individual measurements varied from 8 to 100 with an average of 44, again comparable to prior RRE (range of 4–100; avg. of 35 in 2016 and range of 9–200; avg. of 35 in 2018). The group VR_o % histogram showed a unimodal distribution (Fig. 9A). Application of the $SD < 0.15 \times VR_o$ criterion resulted in a decrease in GM to 0.70 VR_o % ($n = 17$) with a modified range from 0.58 to 0.81% and GSD decrease to 0.06.

3.3.3. Bituminite reflectance

The sample from the Kimmeridge Clay Formation used in the 2020 RRE hosts abundant framboidal pyrite in bituminite, potentially interfering with BitR_o % measurement. Moreover, the inclusion of all bituminite variants in the bituminite spectrum (see general comments) consequently resulted in high SD. BitR_o % measurements ranged from 0.09% to 0.59%.

BitR_o % values are displayed in Fig. 9A and Table 6. The number of BitR_o % measurements from each participant varied from 3 to 100 with an average of 33. BitR_o % ranged from 0.09 to 0.59% with GM of 0.35%. High GSD of 0.17 ($N = 19$) indicated a high scatter of BitR_o % values, in clear contrast to the 2016 RRE (GSD = 0.05) and the 2018 RRE (GSD = 0.04). A bimodal distribution of BitR_o % values (Fig. 9A) is constituted by peaks at c.0.26% and 0.43%. Two participants reported high SD values (0.12; 0.28), indicating measurement of a mixed population. Application of the criterion $SD < 0.15 \times \text{BitR}_o$ % resulted in GM increase to 0.43% for $N = 9$. Likewise, GSD increased unexpectedly to 0.18.

3.4. Summary of bituminite identification and characterisation

Bituminite is regarded as a Type I–II kerogen (e.g., Boussafir et al., 1995), a major component in many petroleum source rocks of marine origin and the most oil-prone maceral, indicating the importance of its role in prediction of HC generation and for the evaluation of petroleum systems in sedimentary basins. Its identification and microscopic characterisation remain a key issue when considering hydrocarbon generative potential of source rocks (e.g., Sherwood and Cook, 1986; Taylor et al., 1991). We note that the qualitative maceral description of bituminite can also be relevant in assessment earlier oil generative potential (Alpern, 1980; Cook, 1982) and maturity evaluation (e.g., Gorbanenko and Ligouis, 2014).

The microscopic identification of bituminite performed in the RRE is based on the established concept of its “unfigured” (after Marlies Teichmüller lacking a distinct shape) or amorphous boundaries (Hamrla, 1959; Teichmüller, 1971, 1977). This concept relates to its indiscernible and indistinguishable margins adjacent to bituminous mineral-rich groundmass (Pickel et al., 2017). Consequently, the margins of bituminite cannot be easily distinguished or defined as evidenced from the RRE. Therefore, a primary property of bituminite is the lack of clarity or distinctness in its maceral boundaries in contrast to the distinct boundaries of other liptinite macerals such as *Tasmanites*. Participants in the RRE used the term “amorphous” to refer to the overall appearance of bituminite, which is in opposition to the description of bituminite as containing internal structure described as “homogenous, streaky, sometimes flow-like (aligned, internal structure), and finely granular” (Pickel et al., 2017). Although the margins of bituminite lack clarity or distinctness, its overall form and internal structure can be characterised in a Transmission Electron Microscopy examination of bituminite (Taylor et al., 1991). The bituminite forms reported in the RRE (laminae, irregular streaks, wisps, flaser, pods, threads, bands and elongated lenses) provide an extension to the definition of bituminite reported by Pickel et al. (2017).

The same is true for bituminite size delineation (streak and thread forms are < c.2–200 µm long and < c.2–4 µm thick; lenses, flaser and pods are < c.2–10 µm long and < c.2–4 µm thick) and appearance (vein-like appearance, fine disseminations, and equidimensional particles of various shapes).

The usage of bituminite types other than those established by Teichmüller and Ottenjann (1977) were not widely implemented in the RRE. The previous division of bituminite and its equivalents into multiple bituminite types was unhelpful to the analysts, as these terms may have not been applied in a consistent way during the RRE. Further, the presence of both bituminite and solid bitumen generated from bituminite in thermally immature to early mature source rocks (e.g., Hackley et al., 2018, 2022a; Petersen et al., 2020; Sanei, 2020), and the interlamination of bituminite with lamalginite, may pose additional challenges in adequately ascribing multiple bituminite types. Therefore, we conclude that differentiation of certain different bituminite types as described in historical literature was not found to be helpful, and that bituminite should instead be described as presented.

The transitional nature of bituminite macerals with solid bitumen, lamalginite, and bituminous mineral-rich groundmass explains its overall gradational character. While the reported forms of bituminite

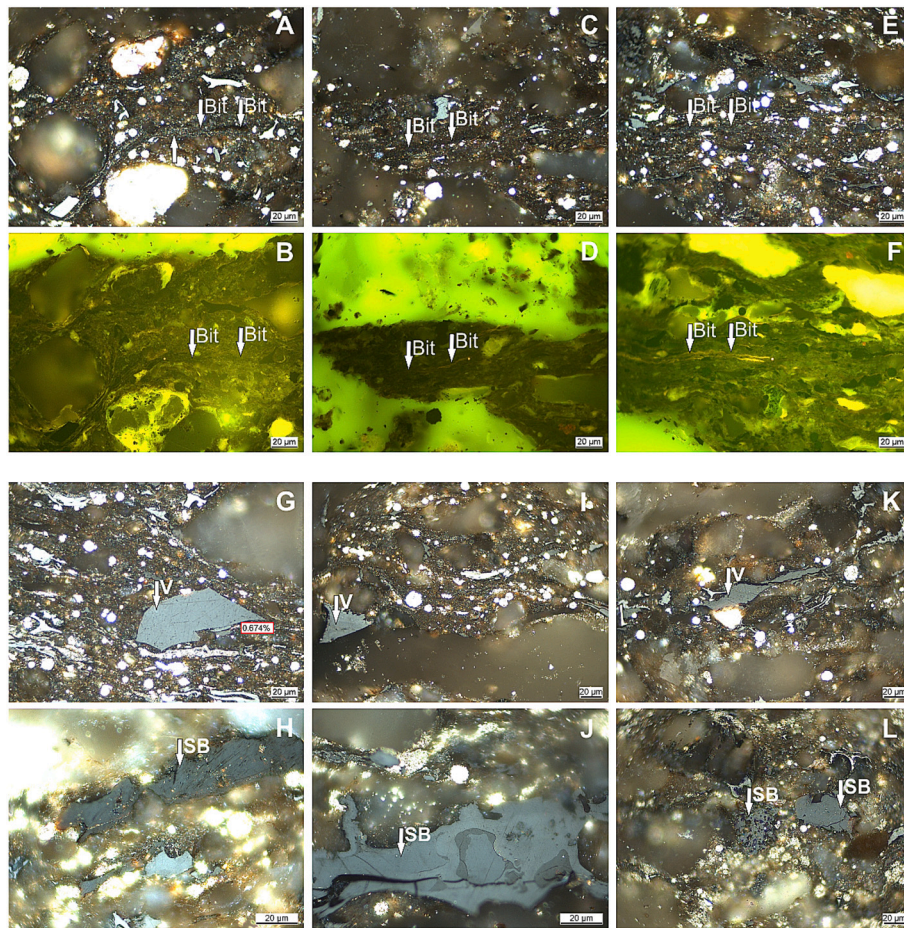


Fig. 8. Kimmeridge Clay Formation in the 2020 RRE in 16/17–14 well in the Viking Graben, North Sea. A – F Photomicrographs of laminae and streaks of dark gray to black bituminite (Bit) in incident white light dark orange to dark brown in blue light illumination, oil immersion. G, I, K – Photomicrographs of vitrinite laths (V), in incident white light, oil immersion. H, J, L – Photomicrographs of thick lamellae of solid bitumen (SB), multiphase-solid bitumen, and globules of solitary multiphased solid bitumen finely dispersed in bituminous mineral-rich groundmass, in incident white light, oil immersion. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article).

fall under the definition of macerals according to the International Handbook of Coal Petrography (ICCP, 1975), the interstitial form of bituminite associated with bituminous mineral-rich groundmass clearly does not, and therefore remains a challenge in terms of application of an appropriate terminology and community adherence to a specific definition. This outcome suggests that the current ICCP-approved definition of bituminite may require revision.

3.5. Evaluation of precision of vitrinite reflectance measurements in whole rock pellets

Evaluation of precision of VR_o % in sedimentary rocks other than coal in eight RRE conducted by the ICCP on 23 samples between 1981 and 2009 was investigated by Borrego (2009) using the unsigned multiple of the standard deviation (UMSD). If the dispersion around mean values (UMSD) for the n samples included in the RRE of a given participant was <1.5 , the results were considered acceptable and precise enough to pass the RRE. The UMSD value of 1.5 corresponds to 86.6% of the data being within $\pm 1.5 \times \text{GSD}$ in a normal Gaussian distribution and it is the pass/fail value used in ICCP accreditation programs since 1994 (<https://www.iccop.org/>). The same UMSD criterion was applied to the data in the current study (including outliers) to evaluate precision. In all three RRE, most participants had individual values of UMSD between 0 and 1.0, indicating moderate to high precision (Fig. 10). In the 2016

RRE, only one participant did not satisfy the UMSD criterion. In the 2018 RRE, three participants did not satisfy the UMSD criterion, while in the 2020 RRE, two participants did not meet the criterion. The moderate to high precision obtained in the RRE is comparable with precision observed in similar RRE conducted by Borrego et al. (2006) and Hackley et al. (2015). The average UMSD (AUMSD) for all participants in the three RRE (including outliers) were 0.85, 0.74, and 0.70, respectively, comparable with historical values of 0.76 reported by Borrego (2009) and 0.80 documented by Hackley et al. (2020).

Four of the six participants who did not satisfy the AUMSD rule in the 2016, 2018, and 2020 RRE did not hold an ICCP DOMVR accreditation for dispersed vitrinite reflectance measurement. This confirms that ICCP DOMVR accreditation is a positive indicator of quality standards and that it ensures the best practice of quality enforcement procedures.

Borrego (2009) applied an ILS reproducibility limit (RL) from ISO 7404-5 (2009), calculated as $RL = 2\sqrt{2} \times \text{GSD}$. For samples with thermal maturity of $VR_o < 1.0\%$, the RL ranged between 0.09 and 0.58 with mean RL of 0.35 (Borrego, 2009). In comparison to the mean RL of 0.35 from Borrego (2009), the RL from the 2016, 2018, and 2020 RRE ranges between 0.28 and 0.31 with a mean RL of 0.29. As such, the current study shows an improvement in precision, with a lower mean RL (0.29) compared to the mean RL (0.35) reported by Borrego (2009). The improved RL is attributed here to use of a standardized test method, the ASTM D7708 (2014b; 2023) standard.

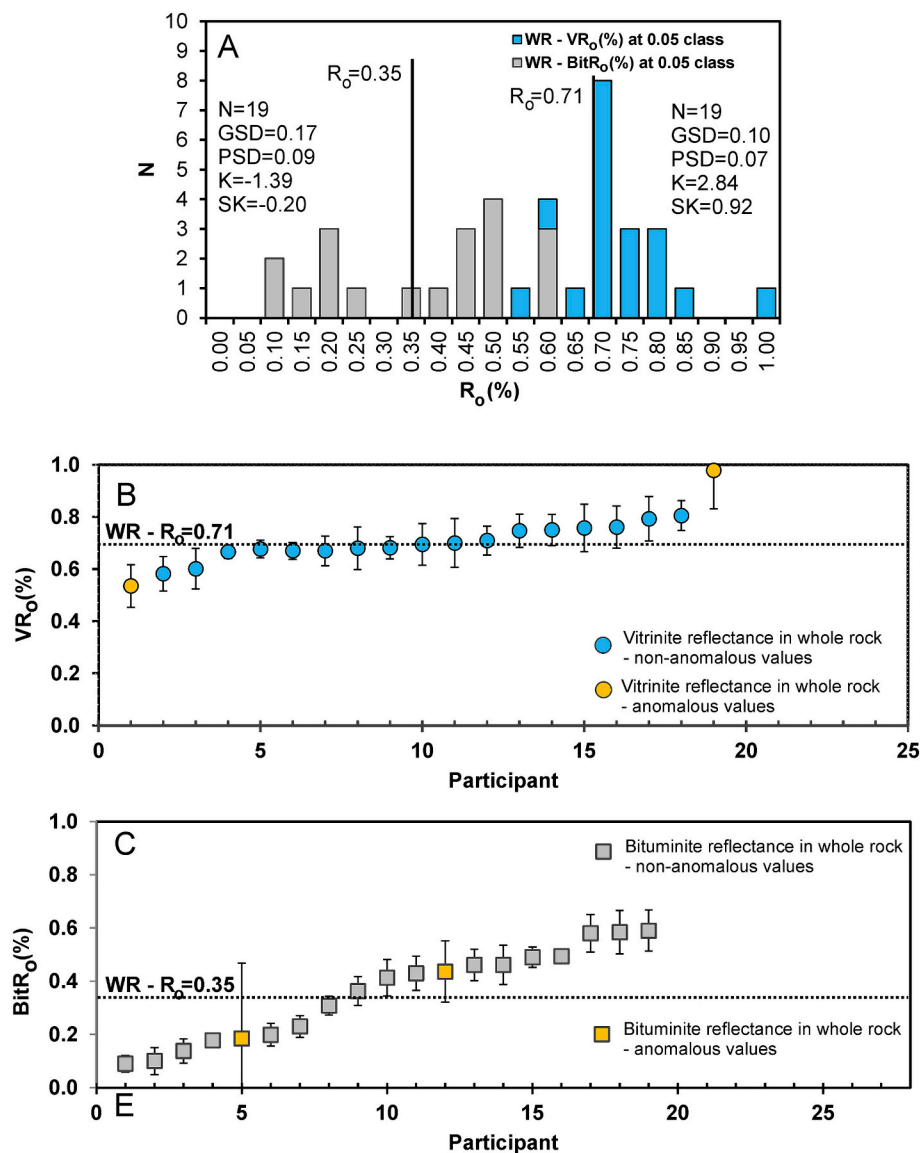


Fig. 9. Data from organic carbon-rich intervals of the Kimmeridge Clay Formation in the 2020 RRE 16/17–14 well in the Viking Graben, North Sea. A – Reflectance histogram of bituminite reflectance (BitR_o in %, gray) and vitrinite reflectance (VR_o in %, blue) in whole rock (WR) pellet. B – Distribution of VR_o in WR pellet with two data points delineating one anomalous standard deviation (SD) value (0.15) and one anomalously low VR_o value (0.54%) marked in orange. C – Distribution of BitR_o in WR pellet with two anomalous SD (0.12, 0.28). N – number of measurements; GSD – group standard deviation; PSD – personal standard deviation (average standard deviation reported by all participants); K – kurtosis; SK – skewness; R_o – random reflectance. (For interpretation of the references to colour in this figure, the reader is referred to the web version of this article.) (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

3.6. Vitrinite reflectance measurements in kerogen isolate

Petrographic measurements in KI can be influenced by the preparation method [Mendonça Filho et al. \(2010\)](#). Mean VR_o (VR_o = 0.43%) reported for the KI was 0.05% lower in comparison to mean VR_o (VR_o = 0.47%) for WR in the 2016 RRE. This is in line with [Mendonça Filho et al. \(2010\)](#), who reported VR_o (VR_o = 0.45%) to be 0.04% lower in KI than in WR. This is also in line with [Borrego \(2009\)](#) based on data from [Castaño \(1994, 1995\)](#) for KI. [Mendonça Filho et al. \(2010\)](#) explained the lower VR_o reported in KI to be due to easier discrimination of recycled vitrinite or inertinite resulting in a lower VR_o value. The lower VR_o values recorded in KI in the current study can be attributed to the poorer polishing of the KI. A contradictory result was documented by [Barker \(1996\)](#) and [Mendonça Filho et al. \(2010\)](#) for KI where VR_o was 0.03% higher than in WR. As a plausible reason for the higher VR_o reported in KI, [Mendonça Filho et al. \(2010\)](#) suggested better polishing of

whole rock pellets. Incorporating the results of this study, we attribute poorer polishing of KI to lower values than measured from WR samples.

3.7. Bituminite reflectance in whole rock pellets

To our knowledge, this is the first RRE study quantifying the optical properties of bituminite. The BitR_o data indicated a comparable reflectance range and acceptable measurement precision in the 2016 and 2018 RRE (2016 RRE BitR_o 0.11–0.29%; 2018 RRE BitR_o = 0.09–0.28%, 2016 RRE GM = 0.19%; 2018 RRE GM = 0.19%, and 2016 RRE GSD = 0.05; 2018 RRE GSD = 0.04). However, with increasing thermal maturity in the 2020 RRE sample, there was also an increase in BitR_o GSD to 0.17. The higher GSD could be explained by both the higher thermal maturity of bituminite and potentially dissimilar redox conditions at the sediment surface.

In general, buried OM undergoes heat- and pressure-related changes

Table 6

Summary of vitrinite (VR_o %) and bituminite reflectance (BitR_o %) measurements in the Upper Jurassic Kimmeridge Clay sample from the 2020 round robin exercise (RRE). SD – standard deviation. N – number of measurements. UMSD – unsigned multiple of standard deviation. NA – analyst not available in this RRE (analysts 1–29 are the same in all 3 RRE). UMSD values >1.5 are displayed in bold font.

Analyst	Vitrinite in 2020 RRE				Bituminite in 2020 RRE			
	VR _o %	SD	N	UMSD	BitR _o %	SD	N	UMSD
1	0.70	0.09	30	0.088	0.10	0.05	10	1.497
2	NA	NA	NA	NA	NA	NA	NA	NA
3	NA	NA	NA	NA	NA	NA	NA	NA
4	NA	NA	NA	NA	NA	NA	NA	NA
5	0.67	0.03	8	0.446	0.58	0.07	19	1.354
6	0.68	0.03	8	0.330	0.58	0.08	19	1.330
7	0.67	0.03	10	0.404	0.59	0.08	21	1.389
8	0.79	0.09	35	0.890	0.44	0.12	30	0.482
9	0.76	0.09	15	0.522	0.09	0.03	18	1.556
10	NA	NA	NA	NA	NA	NA	NA	NA
11	0.98	0.15	87	2.836	0.46	0.07	42	0.629
12	0.71	0.06	17	0.011	0.14	0.05	55	1.275
13	0.75	0.06	70	0.438	0.23	0.04	31	0.732
14	NA	NA	NA	NA	NA	NA	NA	NA
15	0.68	0.04	22	0.278	0.49	0.01	5	0.824
16	0.67	0.06	26	0.404	0.49	0.04	3	0.800
17	0.60	0.08	38	1.127	0.31	0.04	25	0.273
18	NA	NA	NA	NA	NA	NA	NA	NA
19	0.75	0.06	56	0.406	0.19	0.28	25	0.997
20	NA	NA	NA	NA	NA	NA	NA	NA
21	0.68	0.08	50	0.299	0.43	0.06	41	0.447
22	0.70	0.08	77	0.141	0.18	0.02	43	1.038
23	0.81	0.06	77	1.016	0.36	0.05	18	0.052
24	NA	NA	NA	NA	NA	NA	NA	NA
25	NA	NA	NA	NA	NA	NA	NA	NA
26	NA	NA	NA	NA	NA	NA	NA	NA
27	0.76	0.08	51	0.553	0.46	0.06	50	0.629
28	0.54	0.08	55	1.824	0.20	0.04	72	0.914
29	0.58	0.07	100	1.330	0.41	0.07	100	0.346

in chemical composition, and thus in its optical properties. However, variations in the chemical composition of OM, e.g., differences in aromaticity and hydrogen to carbon ratio, can arise independently of thermal maturity of OM. One of the plausible causes for such variation is related to differences in the precursor of bituminite or to differences in bituminite type. Both could be influenced by variations in the depositional environment or dissimilar diagenesis and early catagenesis processes (Stasiuk and Goodarzi, 1988; Bruns and Littke, 2015; Synnott et al., 2016, 2017). Variation in depositional environment is evident in the current study as samples from the Kimmeridge Clay Formation from Kimmeridge Bay, Dorset, and Filey Bay, North Yorkshire were deposited in an epicontinental basin setting, which formed highly fossiliferous mudstones and shales with minor beds of cementstone (diagenetic carbonate) and coccolith-rich limestone. In contrast, the Brae Member of the Kimmeridge Clay Formation in the North Sea accumulated in a fault-bounded clastic submarine-fan deposit composed of gray-brown, medium- to coarse-grained sandstone and conglomerate interbedded with fine-grained sandstone, mudstone and mud-matrix supported breccia (Richards et al., 1993). As such, the Kimmeridge Clay Formation in the North Sea contains autochthonous algae intermixed with varying amounts of terrigenous constituents sourced by the surrounding highs (Macquaker and Gawthorpe, 1993). It can be postulated that precursors of bituminite in the 3 RRE samples were similar but subjected to different redox conditions at the sediment surface. In particular, the marine algal bituminite precursor in the Brae Member is hypothesised to be sourced from eroded and reworked highstand sediments during regressive episodes, and subsequently redeposited in clastic submarine fans (e.g.: Stow et al., 1982; Turner and Connell, 2018), potentially experiencing greater oxidative degradation than bituminite precursors in epicontinental settings.

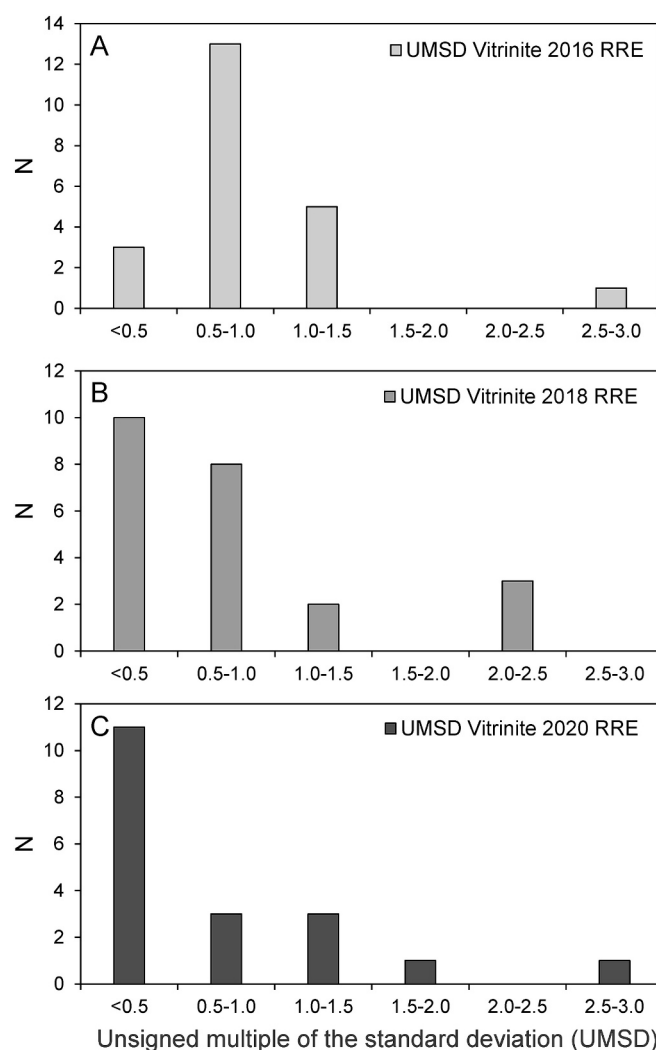


Fig. 10. Unsigned multiple of the standard deviation (UMSD) histograms for vitrinite reflectance. A – 2016 RRE. B – 2018 RRE. C – 2020 RRE. RRE – round robin exercise.

In addition to differences in depositional environment, dissimilar diagenesis and early catagenesis processes could also lead to a difference in bituminite type, independent of chemical changes caused by increasing thermal maturity. Stasiuk and Goodarzi (1988) reported that dissimilar forms of bituminite Type III existed, i.e., low-reflecting (< 0.40% BitR_o), intermediate reflecting (0.40–0.60% BitR_o) and high-reflecting (0.60–1.20% BitR_o). These authors argued that the variations in reflectance and bituminite forms could not be attributed to catagenesis processes associated with hydrocarbon generation and instead must result from a depositional or early diagenesis process. Stasiuk and Goodarzi (1988) suggested that the low reflecting bituminite III variant may be the precursor of a more severely biodegraded, higher reflecting bituminite III. Synnott et al. (2016) similarly hypothesised that micrometer-scale bacterial sulfate reduction processes could elevate reflectance in bituminite macerals. Overall, the high GSD of bituminite reflectance in the 2020 RRE could result from: (1) higher thermal maturity of OM and (2) dissimilar redox conditions at the sediment surface.

Within the ICCP DOMVR Accreditation Program, the evaluation criterion was established as $SD < 0.15 \times VR_o \%$. This threshold is derived from historical standard deviations compiled across years of analytical data (Borrego, 2024). This empirical approach is effective across most of the thermal maturity scale. It offers valuable advantages,

as analysts can easily adopt it during reflectance measurements and evaluation to actively monitor and guide its acquired data. However, this criterion faces limitations when evaluating low reflectance such as those of low-reflecting vitrinite values ($< 0.4 \text{ VR}_0 \%$). Because the baseline reflectance values are predominantly low, the allowable standard deviation becomes exceptionally narrow, leading to a disproportionately low criterion. Previously, Hackley et al. (2020) applied this criterion to vitrinite reflectances (VR_0 0.90–1.83%) and solid bitumen reflectance (BR_0 0.85–2.04%) in thermally mature shales, demonstrating a consistent reduction in the reproducibility limit (R) in accordance to ASTM E691-13 (2014a). Consequently, Hackley et al. (2020) recommended that reported mean reflectance measurements of dispersed organic matter should strictly adhere to this threshold, maintaining a standard deviation (SD) below $0.15 \times \text{VR}_0 \%$ or below $0.15 \times \text{BR}_0 \%$.

This criterion $\text{SD} < 0.15 \times \text{VR}_0 \%$ was intentionally adopted as $\text{SD} < 0.15 \times \text{BitR}_0 \%$ to evaluate its validity for the reflectance of bituminite across both thermally immature and mature samples from the RRE carried out in 2016, 2018, and 2020. The results of the analyses demonstrate that the effect of the applied criterion $\text{SD} < 0.15 \times \text{BitR}_0 \%$ on low-reflectance bituminite in 2016 and 2018 RRE is disproportionately limited due to the narrow standard deviation threshold. Therefore, the criterion showed minimal impact on GM and GSD in the thermally immature samples. In addition, its application led to an unexpected increase in the GM and GSD of $\text{BitR}_0 \%$ for the thermally mature 2020 RRE sample. This finding for thermally mature sample highlights the fact that the inherent scatter and unique optical properties of bituminite introduce a significant statistical uncertainty, which cannot be satisfied consistently. Consequently, this might necessitate a more flexible statistical approach for bituminite reflectance measurements in thermally mature samples, a phenomenon that can be associated among others with the *in-situ* formation of lower reflecting solid bitumen and transformation of bituminite into higher reflecting bituminite.

3.8. Recommendation to amend the ICCP-approved definition of bituminite

The description of bituminite applied in the 2016, 2018, and 2020 RRE contained detailed and categorised morphological characteristics related to its form, size, impurities, and internal structure. These criteria surpassed the descriptive terms ascribed to bituminite from Pickel et al. (2017), e.g., these criteria were extended to morphological characteristic such as the appearance and impurities of bituminite. It is evident from the current study that impurities hosted by bituminite (e.g., minerals, micrinite, alginite, liptodetrinite, corroded algal remnants, solid bitumen) are an important observation and shall be further tested whether or not they have an impact on the bituminite definition and/or classification. The co-occurring impurities may suggest a transitional state of bituminite and a preservation state of former alginite precursors. Further, bituminite-related impurities can also document the HC generation stage of the bituminite by recognition of secondary products (e.g., solid bitumen) of hydrocarbon generation.

The well-known and widely reported “amorphous” or “unfigured” (after Marlies Teichmüller lacking a distinct outline) boundaries of bituminite (e.g., Teichmüller, 1971) remain the fundamental morphological characteristic of bituminite in incident white light microscopy. However, it is the “amorphous” or “unfigured” boundaries of bituminite causing difficulty to analysts in the 2016, 2018, and 2020 RRE, which cannot be adequately resolved by conventional incident light microscopy. Further, in some cases the “amorphous” or “unfigured” boundaries of bituminite prohibited its comparison and attribution to the published bituminite types (Supplementary material) in a reproducible manner. In conclusion, this study has demonstrated that the use of formerly published bituminite types was not widely implemented and therefore remained largely unhelpful. In addition, the formerly published bituminite-type definitions as reported by Pickel et al. (2017) do

not cover adequately the herein reported observations. Therefore, a revisit of the established descriptive term of bituminite maceral reported by Pickel et al. (2017) with detailed and categorised morphological criteria as reported herein seems necessary, by also extending these criteria to cover the appearance and impurities of bituminite.

4. Conclusions

Three round robin exercises (RRE) were conducted in 2016, 2018, and 2020 to microscopically characterise bituminite and to test the reproducibility of vitrinite and bituminite reflectance measurements in the organic carbon-rich intervals of Kimmeridge Clay Formation. Twenty-four laboratories in North America, Europe, Asia, and Australia received the samples and provided results. The results can be summarized as follows:

1. The descriptive approach of bituminite focused on detailing its form (laminae, irregular streaks, wisps, flaser, pods, threads, bands, and elongated lenses), its mode of appearance (vein-like, finely disseminated), and allowed comparability between the RRE. Further, the RRE results confirmed the gradational character of bituminite from its particular form (laminae; irregular streaks; wisps; flaser; pods; threads; bands; elongated lenses) to its interstitial form in mineral-bituminous groundmass. These results present an advancement in the description of bituminite form provided by Pickel et al. (2017) and therefore should be included in a renovated definition of bituminite maceral.
2. The appearance [vein-like, finely disseminated, homogenous, diffuse, roughly equidimensional (e.g., plate-like) of various forms], size (e.g., streak and thread forms are $< 0.2\text{--}200 \mu\text{m}$ long and $< 0.2\text{--}4 \mu\text{m}$ thick; lenses, flaser and pods are $< 0.2\text{--}10 \mu\text{m}$ long and $< 0.2\text{--}4 \mu\text{m}$ thick), and impurities (lack of impurities; minerals; corroded algal remnants; liptodetrinite; solid bitumen) provided additional microscopic characterisation of bituminite, also signifying that that further work could help to identify the forms and mode of occurrence and that renovation and modification of the ICCP bituminite definition may be required.
3. Identification of and implementation of the definitions of bituminite types (e.g., bituminite I *sensu* Teichmüller and Ottenjann, 1977) were unhelpful for the purpose of interlaboratory comparison.
4. Organic-rich (5.32 to 12.10 wt% TOC) samples from the Kimmeridge Clay Formation used for the three RRE displayed T_{max} from 418 to 445 °C and HI between 113 and 666 mg HC/g TOC, indicating immature to thermally mature conditions. Dispersed organic matter was mostly composed of bituminite with secondary solid bitumen, vitrinite, and other liptinite macerals (mainly lamalginite).
5. Mean vitrinite reflectance values in WR in 2016, 2018, and 2020 RRE (Figs. 5, 7, 9) confirmed the thermal maturity obtained by T_{max} and ranged from 0.47 to 0.71% ($0.10 \geq \text{GSD} \leq 0.11$; $19 \geq n \leq 23$) including outlying values. When applying the criterion $\text{SD} < 0.15 \times \text{VR}_0$ to exclude outlying values, the modified $\text{VR}_0 \%$ ranged from 0.46 to 0.70% ($0.06 \geq \text{GSD} \leq 0.08$; $13 \geq n \leq 17$). Application of the criterion $\text{SD} < 0.15 \times \text{VR}_0$ led to a decrease in GSD in all three RRE from 0.10 to 0.11% to 0.06–0.08%.
6. Mean bituminite reflectance values in whole rock samples (WR) in 2016, 2018, and 2020 RRE (Figs. 5, 7, 9) were lower than the corresponding vitrinite reflectance results and varied from 0.19 to 0.35% ($0.04 \geq \text{GSD} \leq 0.17$; $19 \geq n \leq 22$) including outlying values. When applying the criterion $\text{SD} < 0.15 \times \text{BitR}_0$ to exclude outlying values, the modified $\text{BitR}_0 \%$ ranged from 0.19 to 0.43% ($0.03 \geq \text{GSD} \leq 0.18$; $6 \geq n \leq 11$), indicating that the mean bituminite reflectance values were too low to show sensitivity to this criterion. Application of the criterion $\text{SD} < 0.15 \times \text{BitR}_0$ in the 2020 RRE led to an increase of $\text{BitR}_0 \%$ in the thermally mature sample. No significant change in GSD was realized by application of the criterion.

7. Mean vitrinite reflectance value of kerogen isolate was 0.05% lower in comparison to whole rock pellet and attributed to poorer polishing of the kerogen isolate (KI).
8. Precision of vitrinite reflectance via unsigned multiple of the standard deviation (UMSD) rule (< 1.5) was satisfied with the UMSD of most participants between 0 and 1.0 indicating moderate to high precision. Average UMSD values were 0.84, 0.74, and 0.70, respectively, and were comparable to equivalent historical values of 0.76 to 0.80 from similar exercises.
9. A reproducibility limit (RL) for VR_o computed via $RL = 2\sqrt{2} \cdot GSD$ ranged between 0.28 and 0.31% with a mean value of 0.29% and showed an improvement over the equivalent historical value of 0.35% from similar exercises. We suggest the application of a common methodology (ASTM D7708) improved RL over the historical interlaboratory studies.

CRediT authorship contribution statement

J. Kus: Writing – review & editing, Writing – original draft, Validation, Supervision, Software, Resources, Project administration, Methodology, Investigation, Formal analysis, Data curation, Conceptualization. **A.G. Borrego:** Writing – review & editing, Investigation. **B. Bielowicz:** Writing – review & editing, Investigation. **P. Crosdale:** Writing – review & editing, Investigation. **M.-E. Damoulianou:** Writing – review & editing, Investigation. **D. Flores:** Writing – review & editing, Investigation. **C. Fonseca:** Writing – review & editing, Investigation. **P. A. Gonçalves:** Writing – review & editing, Investigation. **P.C. Hackley:** Writing – review & editing, Investigation. **M. Hámor-Vidó:** Writing – review & editing, Investigation. **S. Henne:** Writing – review & editing, Investigation. **B. Holstein:** Writing – review & editing, Investigation. **I. Jelonek:** Writing – review & editing, Investigation. **S. Kalaitzidis:** Writing – review & editing, Investigation. **M. Luzhbina:** Writing – review & editing, Investigation. **J.G. Mendonça Filho:** Writing – review & editing, Investigation. **T.R. Menezes:** Writing – review & editing, Investigation. **M. Misz-Kennan:** Writing – review & editing, Investigation. **R. Orban:** Writing – review & editing, Investigation. **W. Pickel:** Writing – review & editing, Investigation. **N.V. Pronina:** Writing – review & editing, Investigation. **P. Ranasinghe:** Writing – review & editing, Investigation. **J. Reyes:** Writing – review & editing, Investigation. **G. Siavalas:** Writing – review & editing, Investigation. **A.K. Singh:** Writing – review & editing, Investigation. **P.K. Singh:** Writing – review & editing, Investigation. **I. Suárez-Ruiz:** Writing – review & editing, Investigation. **I. Šýkorová:** Writing – review & editing, Investigation. **Q. Luo:** Writing – review & editing, Investigation. **D. Životić:** Writing – review & editing, Investigation. **P. Wignall:** Writing – review & editing, Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at <https://doi.org/10.1016/j.coal.2026.105072>.

Data availability

Data will be made available on request.

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