#### SUPPLEMENTAL MATERIAL

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# Stratigraphy of the SW UK

The lithological similarity between the St. Audrie's Bay and Lilstock sections allows correlation independent of changes in the organic carbon isotope record (Fig. DR1). Lithological details for the Westbury Formation and Lilstock Formation of the Penarth Group and the Blue Lias Formation of the Lias Group are provided in Fox et al., (2020). Because of the difference in thickness between the sections, the depth profile of St. Audrie's Bay is expanded such that lithological transitions between the two sections correlate and appropriate comparisons can be made, hence a 'St. Audrie's Bay original depth' is given in figures.

# Methods

#### **Sample Preparation**

Samples were collected from the St. Audrie's Bay (51.182833°, -32.286000°) and Lilstock (51.200757°, -3.176389°) outcrops in the southwest of the UK. Weathered surface edges were removed and samples were pre-cleaned with 9:1 DCM:MeOH (Dichloromethane:Methanol) 3 times (15 min) before being crushed to a fine powder using a Rocklabs SRM C+PB rock grinder. All glassware was annealed at 500 °C overnight and all apparatuses were thoroughly rinsed with 9:1 dichloromethane:methanol (DCM:MeOH) solution.

#### Bulk organic carbon isotopes and total organic carbon

Sedimentary carbonates were removed from powdered rock samples (<0.5 g) via 2M HCl acid digestion (12 hr). Samples were checked to ensure all inorganic carbon had been removed by adding additional 2M HCl. After digestion, samples were brought to a neutral pH using Milli-Q water, and freeze-dried (minimum 12 hr) to remove excess water. Total organic carbon (TOC, %) contents and carbon isotope measurements on the composition of bulk organic matter ( $\delta^{13}C_{org}$ ) were conducted at the West Australian Biogeochemistry Centre, University of Western Australia using a CNHS elemental analyzer and Delta V Plus MS connected to a Thermo Flash 1112 via a Conflo IV, respectively. Full details for elemental analysis can be found in Skrzypek and Debajyoti, 2006 and Skrzypek, 2013.

#### Extraction and separation of biomarkers

Powdered rock samples (between ca 30 - 150 g) were extracted for bitumen compounds using a Milestone Start-E microwave extraction system (21 °C to 80 °C over 10 mins, held at 80 °C for 15 mins) in 9:1 DCM:MeOH (50 mL). Samples were extracted twice, where necessary, to ensure complete extraction of organic material. Activated copper turnings (sonicated for 30 mins in 2M HCl, brought to a neutral pH using Milli-Q water, cleaned using 9:1 DCM:MeOH) were used to remove elemental sulfur. Total bitumen extracts were then fractionated by activated silica gel (heated to 80 °C overnight) chromatography into saturate (*n*-hexane), aromatic (3:1 *n*hexane:DCM) and polar (9:1 DCM:MeOH) fractions. St. Audrie's Bay saturated fractions were analyzed using an Agilent 6890N gas chromatograph (GC) fitted with a DB-1 (60 m length, 0.25 mm diameter, 0.25 µm film thickness) capillary column (temperature program of 4 °C/min from 50 °C to 325 °C, held at 325 °C for 24 mins), connected to a Micromass Autospec Ultima multiple reaction monitoring mass spectrometer (MRM-MS). Helium was used as a carrier gas (constant flow 1.1 mL/min). St. Audrie's Bay aromatic and Lilstock combined saturate and aromatic fractions were analyzed using an Agilent 7890B GC fitted with a DB-5 (60 m length, 0.25 mm diameter, 0.25 µm film thickness) capillary column (temperature program of 4 °C/min from 40 °C to 325 °C, held at 325 °C for 20.27 mins) connected to an Agilent 7010B triple quadrupole MS. Helium was used as a carrier gas (constant flow 1.1 mL/min). Compounds were identified using the GEOMARK standard and comparing peak retention times and elution patterns, and quantified using internal standards (D4 stigmastane for saturate compounds and D10 phenanthrene for aromatic compounds). Sample preparation was conducted at Curtin University, Australia and biomarker analysis was conducted at the Summons Lab, Massachusetts Institute of Technology.

Compound Specific Isotope Analysis (CSIA) was conducted at Curtin University, Australia using a Thermo Trace GC Ultra fitted with a DB-1MS column (60 m length, 0.25 mm diameter, 0.25 µm film thickness), coupled to a Thermo Delta V Advantage isotope ratio MS via a GC Isolink and Conflo IV. Helium was used as a carrier gas (constant flow 1.1 mL/min). Carbon isotope composition of compounds was achieved using the 44, 45, and 46 masses, which were run in triplicate and integrated manually. Prior to CSIA, samples were run on an Agilent 6890 GC (temperature program of 3 °C/min from 40 °C to 325 °C, held at 325 °C for 30 mins) fitted with a DB-1 capillary column (60 m length, 0.25 mm diameter, 0.25 µm film thickness) interfaced to an Agilent 5973 MS. Helium was used as a carrier gas (constant flow 1.1 mL/min).

Rock Eval analysis was undertaken at Oxford University using a Vinci Technologies Rock-Eval 6 standard analyzer unit 43, with pyrolysis and oxidation ovens, a flame ionization detector, and 44 infra-red cell. Temperature profiles were 300 to 650 °C and 300 to 850 °C for pyrolysis and oxidation ovens, respectively. Laboratory methods followed those of Behar et al., (2001), and samples were run with the IFP 160000 and in-house (SAB134; organic rich marl) standards.

### **Biomarkers**

The biomarkers used in this study are presented as either a ratio or a percentage/index. Table DR1 gives the MRM reactions (where applicable) and equations used to generate the biomarker data and the typical precursor for each biomarker compounds. Note that C<sub>40</sub> carotenoid compounds are compared to triaromatic steroids (TAS). Normalization of aromatic compounds using TAS was undertaken because 1) St. Audrie's Bay saturate fractions were measured by a Waters Autospec in GC-MRM mode whereas aromatic fractions were measured by an Agilent GC-QQQ (due to the former instrument ceasing to function correctly); and 2) total extracted lipids were fractionated into saturated, aromatic, and polar fractions. Thus, normalization of C<sub>40</sub> carotenoid compounds against another group of aromatic compounds was required to reduce stratigraphic bias, hence C<sub>26-28</sub> TAS (compounds originating from algae but have different diagenetic pathways compared to the saturated sterane compounds) were chosen. For further quality control, AGSO and GeoMark II oil standards were measured in the sample sequences on both instruments, and the Agilent GC-QQQ instrument is setup to attain the same chromatographic patterns as the Waters Autospec. Biomarkers indicating stratification, redox, and photic zone euxinia are given in the main text, whereas full biomarker profiles indicating changes to eukaryotes and select bacteria are given in Fig. DR2 an those of redox conditions given in Fig. DR3.

# **Rock Eval pyrolysis**

Rock-Eval pyrolysis is used to determine the H/C and O/C measurements on bulk organic matter, and typically used to evaluate the petroleum-generative potential, thermal maturity and type of source rocks. Organic matter can be broadly divided into four categories (Types I through IV). Type I kerogen, characterized by a very high hydrogen index (HI) and low oxygen index (OI) values, is generally ascribed to well preserved organic matter from algal origin, although it can also include the liptinitic components of vascular plants from lacustrine settings. Type II is generally comprised of amorphous or structureless organic matter and is the result of bacterial reworking of primary algal material in addition to the preserved bacterial organic matter, or a combination of all types of kerogen that plot as type II in the van Krevelen diagram. Type III kerogen is sourced from either vitrinite (a primary component of coals), high plant material and/or poorly preserved/partially oxidized algal or amorphous organic matter. Type IV generally

implies heavily oxidized and/or poorly preserved organic matter. Kerogen type is dependent on the mix, end members and preservation state of the various organic components and limited to immature to low maturity samples. Our samples are in this range, with maximum temperatures  $(T_{max})$  between 418°C and 435°C and production indexes (PI) between 0.00 and 0.14 (Table DR2). Kerogen type of these samples range between type II and type III-IV (Fig. DR4).

### **Episodic vs. Persistent PZE**

A cross plot of the aryl isoprenoid ratio (AIR) and pristane to phytane ratio (Pr/Ph) determines whether intervals of PZE were persistent or episodic (Fig. DR5). Pristane and phytane are generally formed from the phytyl side chain of chlorophyll *a* under oxic and anoxic conditions, respectively (e.g., Grice *et al.*, 1996), thus low ratios indicate anoxia. The  $C_{13-31}$  aryl isoprenoids are formed from carotenoid pigments produced by Chromatiaceae and Chlorobi that utilize H<sub>2</sub>S in the photic zone of the water column. The AIR is calculated from the ratio of  $C_{13-17}$  to  $C_{18-22}$ aryl isoprenoids. Because the concentrations of aryl isoprenoids in euxinic conditions correlate negatively to the AIR, consistent with degradation and diagenesis, the AIR is a good indication of persistent PZE when values are low, ~0.5 (Schwark and Frimmel, 2004; Jaraula et al., 2013). For the SW UK, samples during the calcification crisis plot close towards zero (Fig. DR5) indicating persistent PZE. Aryl isoprenoids can have multiple sources, therefore, the AIR is only calculated when total PZE biomarkers and total  $C_{13-22}$  aryl isoprenoids abundances are high. Note that those samples from within the Bristol Channel Basin carbon isotope excursion are not included because of their microbial-mat related origin (see Fox et al., 2020).

#### **Bivalves**

The bivalves recorded by Mander et al., (2008) and Atkinson and Wignall, (2019) in the St. Audrie's Bay section represent different groups that diverge in their mobility (stationary, mobile) and habitat (infaunal and epifaunal). A full list of the bivalves observed at St. Audrie's Bay and their ecology is given in Table DR4. A combination of proxies for PZfE from this study and others are compared to the SW UK bivalve assemblages amongst other fossil taxa (Fig. DR6). Note that the largest expansion in calcifying fossil assemblages occurs after the paper shales of the Blue Lias Formation and in close proximity to the return of ammonites.



Figure S1. Lithology of and correlation between St. Audrie's Bay (left) and Lilstock (right).



**Figure S2.** Biomarker inferred ecological changes at St. Audrie's Bay (circles and solid lines) and Lilstock (squares and dashed lined) relative to changes in the  $\delta^{13}C_{org}$  and total organic carbon (TOC) records. The two depositional environments are indicated by the shaded green (PZE persisting through much of the photic zone) and shaded pink (PZE limited to lower depths in the photic zone) areas. Initial CIE and precursor CIE given in yellow and pink, respectively. Biomarker ratios and indices are given below and their ecological indicators given above each respective profile. Although this paper primarily focuses on the paper shales, we include full data here to show changes across the latest Rhaetian for completeness. For full details of biomarkers see Table DR1. Formation abbreviations: W – Westbury Formation; LC – Lower Cotham Member (Lilstock Formation); UC – Upper Cotham Member (Lilstock Formation); L – Langport Member (Lilstock Formation); BL – Blue Lias Formation; W – Westbury Formation. SS – Sandstone.



**Figure S3.** Biomarker inferred redox changes at St. Audrie's Bay (circles and solid lines) and Lilstock (squares and dashed lined) relative to changes in the  $\delta^{13}C_{org}$  and total organic carbon (TOC) records. The two depositional environments are indicated by the shaded green (PZE persisting through much of the photic zone) and shaded pink (PZE limited to lower depths in the photic zone) areas. Initial CIE and precursor CIE given in yellow and pink, respectively. Biomarker ratios and indices are given below and their ecological indicators given above each respective profile. Although this paper primarily focuses on the paper shales, we include full data here to show changes across the latest Rhaetian for completeness. For full details of biomarkers see Table DR1. Formation abbreviations: W – Westbury Formation; LC – Lower Cotham Member (Lilstock Formation); UC – Upper Cotham Member (Lilstock Formation); L – Langport Member (Lilstock Formation); BL – Blue Lias Formation; W – Westbury Formation. SS – Sandstone



**Figure S4.** Left: St. Audrie's Bay and Lilstock total organic carbon (TOC) records and  $\delta^{13}C_{org}$  changes with  $\delta^{13}C_{org}$  symbols matching the Rock-Eval data. Blue shaded area indicates the paper shales. Although this paper primarily focuses on the paper shales, we include full data here to show changes across the latest Rhaetian. Formation abbreviations: W – Westbury Formation; LC – Lower Cotham Member; UC – Upper Cotham Member (Lilstock Formation); L – Langport Member (Lilstock Formation); BL – Blue Lias Formation. Right: Rock-Eval data with corresponding highlighted area as in left figure. A) Kerogen type by van Krevelen diagram. HC – hydrocarbon. B) kerogen type by relationship between remaining hydrocarbon potential and TOC. Dashed box indicates TOC lean samples.



**Figure S5.** Left: Cross plot of the aryl isoprenoid ratio (AIR) and pristane to phytane ratio (Pr/Ph) through the different lithologies of the St. Audrie's Bay and Lilstock sections. Fm – Formation, Mb – Member. Right: Changes in the sum of aryl isoprenoids and sum of  $C_{40}$  carotenoids (okenane, chlorobactane,  $\beta$ -isorenieratane, and isorenieratane) to triaromatic steroids (TAS) relative to the  $\delta^{13}C_{org}$  record. The two depositional environments are indicated by the shaded green (PZE persisting through much of the photic zone) and shaded pink (PZE limited to lower depths in the photic zone) areas. BL – Blue Lias Formation, L - Langport Member, UC – upper Cotham Member.



Index/Biomarker	Calculation	MRM	Typical precursor
Steranes			
C <sub>27</sub> Sterane Index (%) (all)	$\frac{\sum C_{27} \text{Steranes}}{\sum C_{27-30} \text{Steranes}} \times 100$	$372 \rightarrow 217$	Red Algae (Patterson, 1971; Kodner et al., 2008)
C <sub>28</sub> Sterane Index (%) (all)	$\frac{\sum C_{28} \text{Steranes}}{\sum C_{27-30} \text{ Steranes}} \times 100$	386 → 217	Chlorophyll <i>c</i> algae and prasinophytes (Volkman et al., 1994, 1998)
C <sub>29</sub> Sterane Index (%) (all)	$\frac{\sum C_{29} \text{ Steranes}}{\sum C_{27-30} \text{ Steranes}} \times 100$	$400 \rightarrow 217$	Green algae and/or terrestrial plants (Volkman et al., 1994; Kodner et al., 2008)
C <sub>30</sub> Sterane Index (%) (diasteranes)	$\frac{\sum C_{30} \text{ Diasteranes}}{\sum C_{27-30} \text{ Diateranes}} \times 100$	414 → 217	Marine algae (Peters et al., 2004)
Hopanes			
C31 2α-methylhopane Index (%)	$\frac{C_{31}2\alpha \text{ Methylhopane}}{C_{30}\alpha\beta \text{ Hopane}} \times 100$	426 → 205	Aerobic cyanobacteria (Summons et al., 1999), α-proteobacteria (Rashby et al., 2007; Ricci et al., 2015), shifts in environment (Ricci et al., 2014 and references therein), ecological stress (Ricci et al., 2017), nitrite-oxidizing bacteria (Elling et al., 2020)
C <sub>31</sub> 3β-Methylhopane Index (%)	$\frac{C_{31}3\beta \text{ Methylhopane}}{C_{30}\alpha\beta \text{ Hopane}} \times 100$	$426 \rightarrow 205$	Aerobic methanotrophs (Rohmer et al., 1984; Summons and Jahnke, 1990)
Gammacerane Index (%)	$\frac{C_{30} \text{ Gamma cerane}}{C_{30} \alpha \beta \text{ Hopane}} \times 100$	412 → 191	Stratification (Sinninghe Damsté et al., 1995)
C <sub>40</sub> Carotenoids			
Okenane Index	$\frac{Okenane}{\sum C_{26-28}TAS} \times 1000$	554 → 134	Chromatiaceae (Schaeffer et al., 1997; Brocks and Schaeffer, 2008)
Chlorobactane Index	$\frac{Chlorobactane}{\sum C_{26-28}TAS} \times 1000$	554 → 134	Green-pigmented Chlorobiaceae (Grice et al., 1998)
β-Isorenieratane Index	$\frac{\beta - isorenieratane}{\sum C_{26-28}TAS} \times 1000$	552 → 134	Brown-pigmented Chlorobiaceae (Grice et al., 1998)
Isorenieratane Index	$\frac{Isorenieratane}{\sum C_{26-28}TAS} \times 1000$	546 → 134	Brown-pigmented Chlorobiaceae (Jensen, 1965; Summons and Powell, 1986; Grice et al., 1996)
<i>n</i> -alkanes			·
C <sub>17</sub> <i>n</i> -alkane C <sub>18</sub> <i>n</i> -alkane C <sub>19</sub> <i>n</i> -alkane	For CSIA	(run on GC-MS and GC-IRMS)	Photosynthetic bacteria and algae (Cranwell et al., 1987)
C <sub>29</sub> <i>n</i> -alkane	For CSIA	(run on GC-MS and GC-IRMS)	Land plants (Eglinton et al., 1962)
Others			
Pristane to Phytane Ratio	Pristane Phytane	Identified using GC-MS	Redox conditions (Powell and McKirdy, 1973; Didyk et al., 1978)
Hopanes Vs. Steranes Index (%)	$\frac{C_{27-30} Steranes (reg)}{C_{27-35} Hopanes} + C_{27-30} Steranes (reg)$	See above	Eukaryotic vs. bacterial input (Peters et al., 2004)

# Table S1. Biomarker information including equations, MRM reactions, and typical precursors.

# Table S2. Rock-Eval pyrolysis data from St. Audrie's Bay and Lilstock.

St. A	udrie'	's Bay													
TOC	(%)	S1		S2		S3		PI		HI		OI		Tmax	к (°С)
Min	0.06	Min	0	Min	0.01	Min	0	Min	0	Min	11	Min	0	Min	421
Max	2.39	Max	0.12	Max	8.79	Max	0.7	Max	0.12	Max	367	Max	256	Max	435
Lilst	ock									1		1			
TOC	(%)	S1		S2		S3		PI		HI		OI		Tmax	(°C)
Min	0.05	Min	0	Min	0.02	Min	0.07	Min	0.01	Min	7	Min	8	Min	418
Max	4.25	Max	1.17	Max	27.18	Max	0.86	Max	0.14	Max	627	Max	360	Max	433

TOC - Total Organic Carbon (wt%)

S1 – Volatile hydrocarbon (HC) content (mg HC/mg rock)

S2 - Remaining HC generative potential (mg HC/mg rock)

 $S3 - CO_2$  content (mg CO<sub>2</sub>/mg rock)

Production Index (PI) – S1/(S1+S2)

Hydrogen Index (HI) – (S2\*100)/TOC

Oxygen Index (OI) – (S3\*100)/TOC, mg CO<sub>2</sub>/g TOC

**Table S3.** Bivalves of the lower Blue Lias Formation in St. Audrie's Bay and their ecology.Those that are facultative in their mobility are indicated by asterisks.

Mobile		
<b>Epifaunal Suspension Feede</b>	r	
Bivalve	Reported by	Reference
Chlamys Valoneinsis*	Mander et al., (2008)	(Johnson, 1984)
Pseudomytiloides dubius*	Mander et al., (2008)	(Ros-Franch et al., 2015)
Infaunal Suspension Feeder		
Bivalve	Reported by	Reference
Cardinia regularis*	Mander et al., (2008)	(Ros-Franch et al., 2014)
Isocyprina sp.*	Mander et al., (2008)	(Ros-Franch et al., 2014)
Isocyprina concentricum*	Mander et al., (2008)	(Ros-Franch et al., 2014)
Isocyprina ewaldi*	Mander et al., (2008)	(Márquez-Aliaga et al., 2010; Ros- Franch et al., 2014)
Lyriomyophoria postera*	Mander et al., (2008)	(Friesenbichler et al.; Ros-Franch et al., 2014)
Permophorus elongtus*	Mander et al., (2008)	(Mendes, 1952; Foster et al., 2019)
Pleuromya sp.*	Mander et al., (2008)	(Aberhan, 2004; Ros-Franch et al., 2014)
Prorocardia philippianum	Atkinson and Wignal (2019)	(Ros-Franch et al., 2014)
Protocardia rhaetica*	Mander et al., (2008)	(Ros-Franch et al., 2014)
Pteromya crowcombia*	Mander et al., (2008)	(Márquez-Aliaga et al., 2010; Ros- Franch et al., 2014)
Tutcheria cloacina*	Mander et al., (2008)	(Márquez-Aliaga et al., 2010; Ros- Franch et al., 2014)
Infaunal Deposit & Suspensi	ion Feeder	
Bivalve	Reported by	Reference
Palaeonucula navis*	Mander et al., (2008)	(Chen, 1982; Hodges, 2000)
Infaunal Deposit Feeder		
Bivalve	Reported by	Reference
Ryderia sp.*	Mander et al., (2008)	(Yin and McRoberts, 2006)
Rollieria bronni*	Mander et al., (2008)	(Ros-Franch et al., 2014)
Stationary		
<b>Epifaunal Suspension Feede</b>	r	
Bivalve	Reported by	Reference
Anningella cf. faberi	Atkinson and Wignal (2019)	(Ros-Franch et al., 2014)
Cassianella sp.	Mander et al., (2008)	(Nützel and Kaim, 2014; Ros-Franch et al., 2014)
Chlamys textoria	Atkinson and Wignal (2019)	(Johnson, 1984; Damborenea, 2002)
Liostrea spp.	Mander et al., (2008) and Atkinson and Wignal (2019)	(Ros-Franch et al., 2014)
Liostrea bristovi	Mander et al., (2008)	(Ros-Franch et al., 2014)
Liostrea hisingeri	Mander et al., (2008)	(Ros-Franch et al., 2014)
Mytilus cloacinus	Mander et al., (2008)	(Tutcher, 1908)
Oxytoma fallax	Mander et al., (2008)	(Ros-Franch et al., 2014)
Placunopsis alpina	Mander et al., (2008)	(Márquez-Aliaga et al., 2010)

Pseudolimea pectinoids	Atkinson and Wignal (2019)	(Szente, 1992; Damborenea and Mancenido, 2005)
Rhaetavicula contorta	Mander et al., (2008)	(MacFadyen, 1970)
Low-Level Epifaunal Suspensio	n Feeder	
Bivalve	Reported by	Reference
Plagiostoma giganteum	Mander et al., (2008) and Atkinson and Wignal (2019)	(Yin and McRoberts, 2006)
Plagiostoma punctatum	Mander et al., (2008)	(Paredes et al., 2013)
Semi-Infaunal Suspension Feed	er	
Bivalve	Reported by	Reference
Bivalve Gervillella ornata	Reported byMander et al., (2008)	Reference(Ros-Franch et al., 2014; Foster et al., 2019)
Bivalve Gervillella ornata Modiolus spp.	Reported byMander et al., (2008)Mander et al., (2008)	Reference(Ros-Franch et al., 2014; Foster et al., 2019)(Hodges, 2000; Márquez-Aliaga et al., 2010; Paredes et al., 2013)
Bivalve Gervillella ornata Modiolus spp. Modiolus hillanus	Reported byMander et al., (2008)Mander et al., (2008)Mander et al., (2008)	Reference(Ros-Franch et al., 2014; Foster et al., 2019)(Hodges, 2000; Márquez-Aliaga et al., 2010; Paredes et al., 2013)(Hodges, 2000; Paredes et al., 2013)
BivalveGervillella ornataModiolus spp.Modiolus hillanusModiolus minimus	Reported byMander et al., (2008)Mander et al., (2008)Mander et al., (2008)Mander et al., (2008) and Atkinson and Wignal (2019)	Reference(Ros-Franch et al., 2014; Foster et al., 2019)(Hodges, 2000; Márquez-Aliaga et al., 2010; Paredes et al., 2013)(Hodges, 2000; Paredes et al., 2013)(Hodges, 2000; Márquez-Aliaga et al., 2010)

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![](_page_22_Figure_1.jpeg)

Oxygen Index (mg  $CO_2/g$  TOC)

![](_page_22_Figure_3.jpeg)

![](_page_23_Figure_0.jpeg)

Fig. S5

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![](_page_24_Figure_0.jpeg)