A new bacterial origin for the hydrocarbon-bearing zones in the Goldwyer Formation on the Broome Platform, Canning Basin, WA, from Nanoscale ToF-SIMS geochemical characterization.

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SUMMARY

The organic petrology and nano-scale geochemistry of Goldwyer Formation, Canning Basin were mapped to identify the organic macerals, their probable origin and their control by lithofacies. Samples were selected from Theia-1 and Solanum-1 guided by fine-scale lithology, logs and adjacent RockEval data. Most are from the Goldwyer III mudstone in Theia-1, where the highest TOC and gas readings occur, with the rest from Goldwyer I and Goldwyer II units in Theia-1 and a few from Goldwyer I in Solanum-1. The organic macerals were identified by incident and fluorescent light microscopy and inorganic and organic grains were analysed via Time of Flight – Secondary Ion Mass Spectrometry (ToF-SIMS) to obtain Mass-Spectrogram fingerprints for bituminite, graptolites, algae and possibly bacteria/archaea.

The shale minerals are dominated by illite with minor quartz or carbonate, the proportions of which vary correspondingly between the argillaceous, siliceous and calcareous shales. The organics in most samples are dominated by graptolites (vitrinite equivalent macerals), either Detrovitrinite from broken graptolite fragments or Telovitrinite (elongate graptolite stipes or branches). In contrast, a limited number of samples contain a high proportion of Liptinites, mainly fluorescing Bituminite groundmass and Lamalginite (thin algal lamellae or filaments). Discrete algal bodies of *G. prisca* are rare and seem to be restricted to very thin beds mainly within the Goldwyer I sequence. The maceral compositions control the potential gas zones found during drilling of Theia-1. The zones dominated by graptolites have low potential for hydrocarbons. In contrast, the zones with a pervasive Bituminite groundmass (probably derived from degradation of algae by bacteria/archaea) and filamentous Lamalginite bands are lipid-rich and correspond with the main hydrocarbon-bearing zones in Theia-1. This contrasts with the perception from previous studies that the discrete *G. prisca* algal bodies would be the main source of hydrocarbons.

Key words: Ordovician source-rock, Bacteria, *G. prisca,* Graptolite, ToF-SIMS, Hydrocarbon Generation

INTRODUCTION

This study reports the results of the first direct nano-scale analysis of the organic matter macerals within the Ordovician Goldwyer Formation by Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) which is able to discriminate the organic macerals and the associated inorganic matter. The results have a direct bearing on the generative potential of the Goldwyer shales and have found some unexpected results that challenge the current views.

Exploration in the Canning Basin has been episodic since the early 1920s. Just over 300 wells have been drilled, almost all onshore and mostly in or on the flanks of the deep troughs, with less than a dozen wells in the underexplored central areas. Until recently, this exploration focused on conventional plays, which has had varying success, while only since about 2010 has there been increasing focus on shale oil and gas.

The regional geology, exploration history, and development potential of the Broome Platform have been discussed previously including vanHattum *et al*. (2019); Johnson *et al*. (2020) and Iqbal *et al*. (2022). These publications detail the sequence stratigraphy, paleogeography, geochemistry, petrophysics and petroleum systems for this study area. An integrated study of the regional Canning Basin data by Theia Energy for shale gas exploration led to a focus on the Goldwyer III shale in the

Broome Platform, where the main hydrocarbon targets are in the Ordovician. This led to the successful drilling of Theia-1 within EP493, which intersected a 70m gross oil column between 1500 and 1570mMD.

The onshore Canning Basin is dominated by the NW-SE trending Munro Arch, comprising the Broome – Crossland Platforms, that separate the Fitzroy and Gregory Sub-basins in the north from the Willara and Kidson sub-basins in the south (Figure 1). The stratigraphic succession records over 18km of Ordovician to Quaternary sediments in the deep troughs including a complete Ordovician to Triassic section. The section is shallower and thinner in the Broome Platform where there is a mainly Ordovician and Permian section, with thin or missing Devonian and Carboniferous units, unconformably overlain by Jurassic-Cretaceous strata (vanHattum *et al*., 2019).

The Ordovician sediments were deposited in fault-controlled extensional sub-basins and recorded an overall large-scale transgressive succession. The section starts with marginal marine clastic and shallow marine sediments of the Nambeet Formation, overlain by the Willara Formation mainly comprising carbonate ramps and shallow tidal flats, and culminating in deposition of the Goldwyer Formation, which comprises marine shales, calcareous claystones and interbedded limestones. In detail, several transgressive-regressive cycles occur that develop the subtidal and restricted marine depositional environments behind barrier systems, where the organic-rich shales occur, some of which have good source potential.

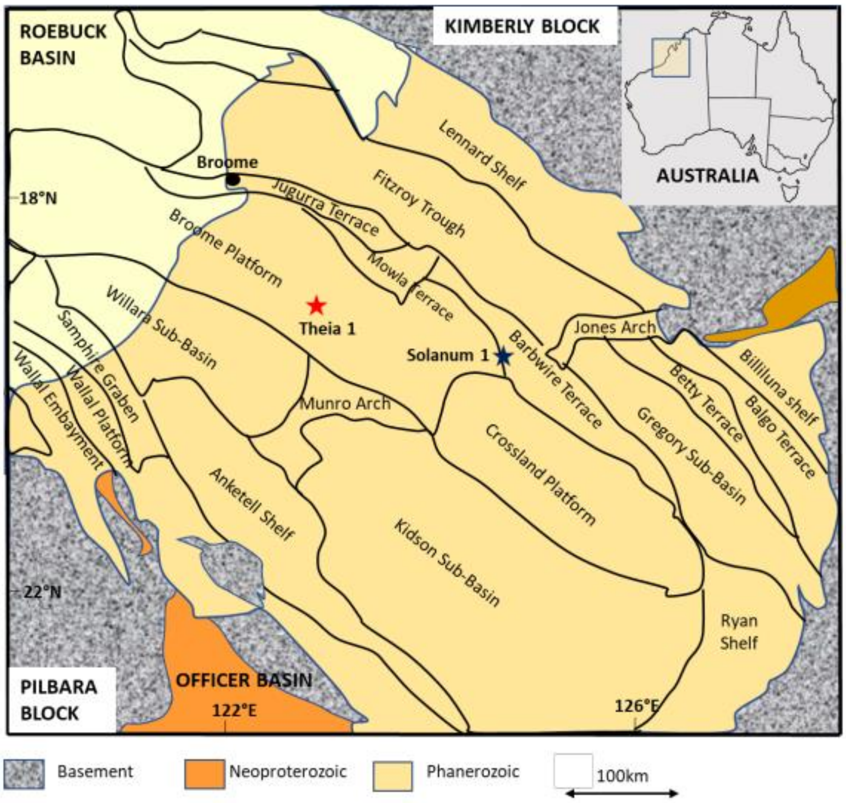

Figure 1. Structural elements of the Canning Basin showing the approximate locations of Theia-1 and Solanum-1 wells (Modified from Johnson *et al*., 2020).

The organic-rich sediments of the Goldwyer Formation contain a variety of organic macerals known to be dominated by graptolites which are mainly associated with open marine conditions and thought not to be oil-prone. The main the oil-prone macerals are considered to comprise algae (mainly *Gloeocapsomorpha prisca*), acritarchs and protospores that occur in localized areas and thin beds within the Goldwyer and other Formations. Most of the existing geochemical data for the Goldwyer shales is via destructive organic and inorganic geochemical analysis of whole samples (e.g. RockEval pyrolysis, GCMS, XRD) rather than direct, non-destructive in-situ analysis. These measurements are not able to discriminate the different organics and inorganics individually at the nano-scale.

In this study, organic-rich shales from the Goldwyer Formation were characterized with Time-of-Flight Secondary Ion Mass Spectrometry (ToF-SIMS) to discriminate the organic macerals individually and at the nano-scale and from the associated inorganic matter within the Goldwyer shales. The advantage of ToF-SIMS and allied techniques is that it is non-destructive and provides analyses at the nanoscale. In addition, fluorescence microscopy was used to screen the samples for organic richness. The results from this study were correlated with downhole logs and other analytical results to help highlight optimum zones for completion or geosteering in the study well.

METHODOLOGY

Approximately 50 gm of core was cut by the WA Geological Survey Core library from the Theia-1 and Solanum-1 core. A precision diamond micro-saw was used to cut the large sample into smaller pieces to fit into two 25 mm diameter molds. The cuts were made to provide a flat surface perpendicular to the bedding that was placed downwards into the molds. The molds were then filled with a quick cold setting resin (Epofix for the SEM samples and in some cases Technovit for the optical microscopy). The resin-mounted samples were then polished using diamond lapping machines down to approximately half microns for the optical microscopy and less for the SEM blocks.

Maceral Analysis

The maceral analysis was carried out using a Leitz Orthoplan microscope system equipped with white and fluorescent light, capable of both separate and simultaneous viewing of the samples. This allows confident identification of fluorescing liptinite and vitrinites from non-fluorescing vitrinites, inertinites, migrated bitumen, and mineral matter, which is critical in shales. The maceral occurrence was estimated using semi-quantitative visual analysis to identify the main maceral groups and screen them for the ToF-SIMS analysis and the proportions were recorded into the Hilgers DISKUS software.

IONTOF Analysis

The ToF-SIMS analysis of the organic matter in the shales was performed using the IONTOF 6 Scanning Electron Microscope (SEM) in the John de Laeter Centre at Curtin University (https://jdlc.curtin.edu.au/facilities/tof-sims-facility/). The instrument allows direct, non-destructive chemical mapping with high resolution and mass spectrometry of ion fragments sputtered from the surface of the shale samples. Samples for IONTOF analysis were evacuated down to <5e-7 mbar. The delayed extraction mode was used which gives high transmission with simultaneous high lateral and high mass resolution.

RESULTS

Maceral Analysis

The maceral analyses successfully identified the types of organic matter, their probable origin and composition. Importantly they immediately showed that the maceral composition controls the zones of high gas readings being targeted in the Theia-1 development well.

A summary of the results is given in Figure 3 which shows the maceral group %. TOC % is included to provide a guide to relative organic richness of each sample and it does not distort the percentages of the main macerals.

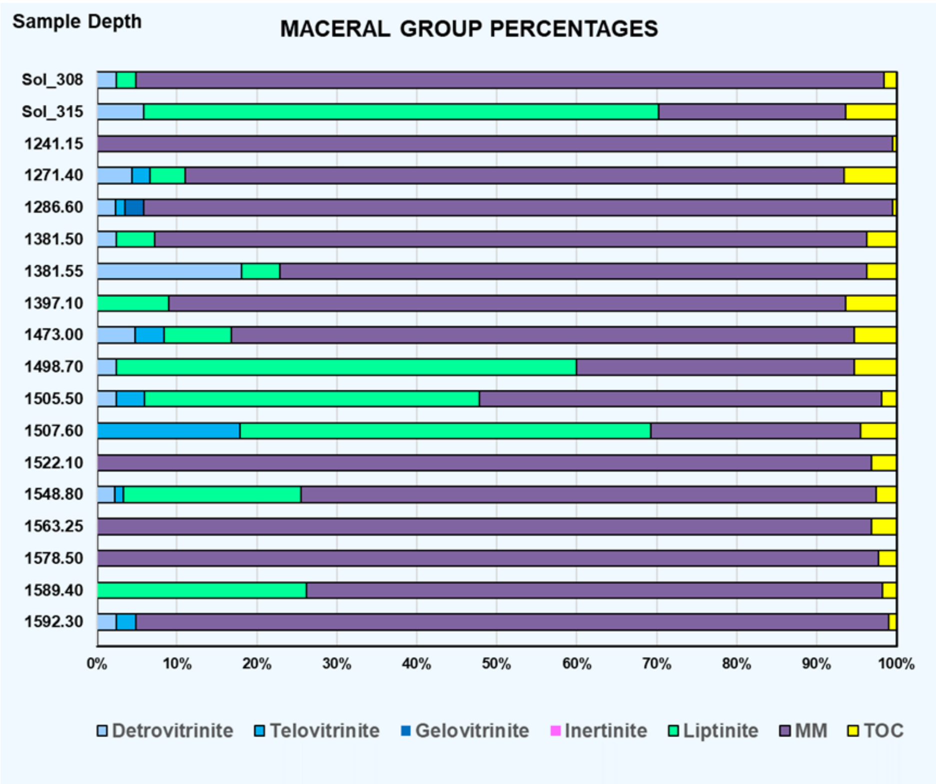

Figure 2. Summary of Maceral analyses plotted with TOC %

Graptolites

Graptolites are thought to be analogous in composition to vitrinite and are used to measure reflectance for rank determination (Luoa *et al*., 2020). They are taken to have Type II-III character and be gas prone since they are the dominant organic matter clast in Lower Palaeozoic source rocks. Hence, this makes it important to understand their composition. However, the compositional data for graptolites are sparse and most of the assumptions are based on inference from optical microscopy.

Most of the samples are dominated by mineral matter with minor proportions of vitrinite equivalent macerals, either detrovitrinite comprising broken graptolite fragments or telovitrinite comprising elongate graptolite fragments (Figure 3). Some samples with moderately high TOC% are mostly made up of graptolites, (usually graptolite fragments - detrovitrinite) with minor liptinites. Some samples have relatively high TOC readings that contain relatively high amounts of graptolites but notably these samples are not associated with the high gas readings.

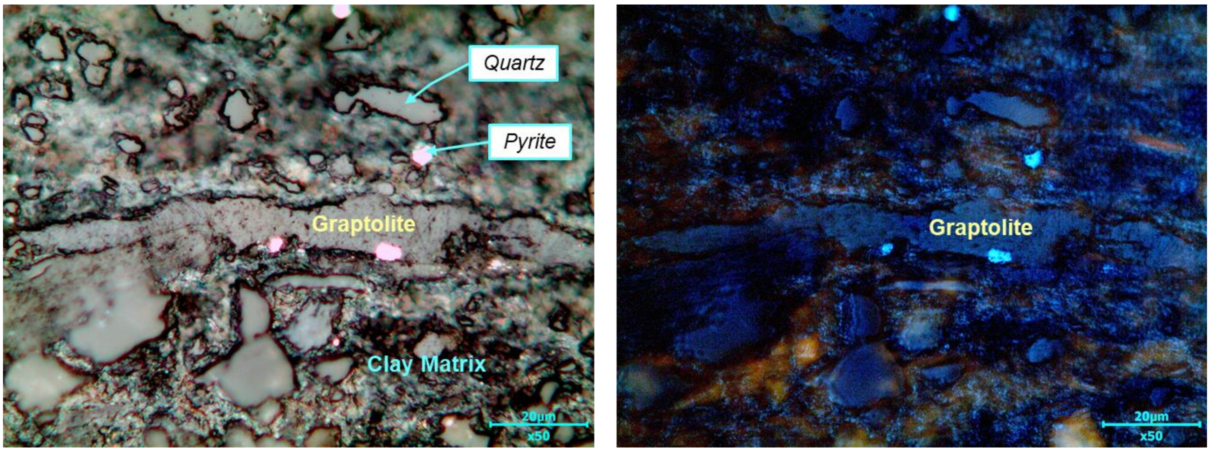

Figure 3. Photomicrograph 1505.7mMD: Elongate Graptolite phytoclast (showing pores) in a groundmass of quartz grains with a clay matrix. Incident white light (left), blue light (right), oil immersion, x50 objective.

Liptinites

A small number of samples contain a high proportion of Liptinites. These samples are closely associated with the high gas readings in Theia-1 (Figure 4).

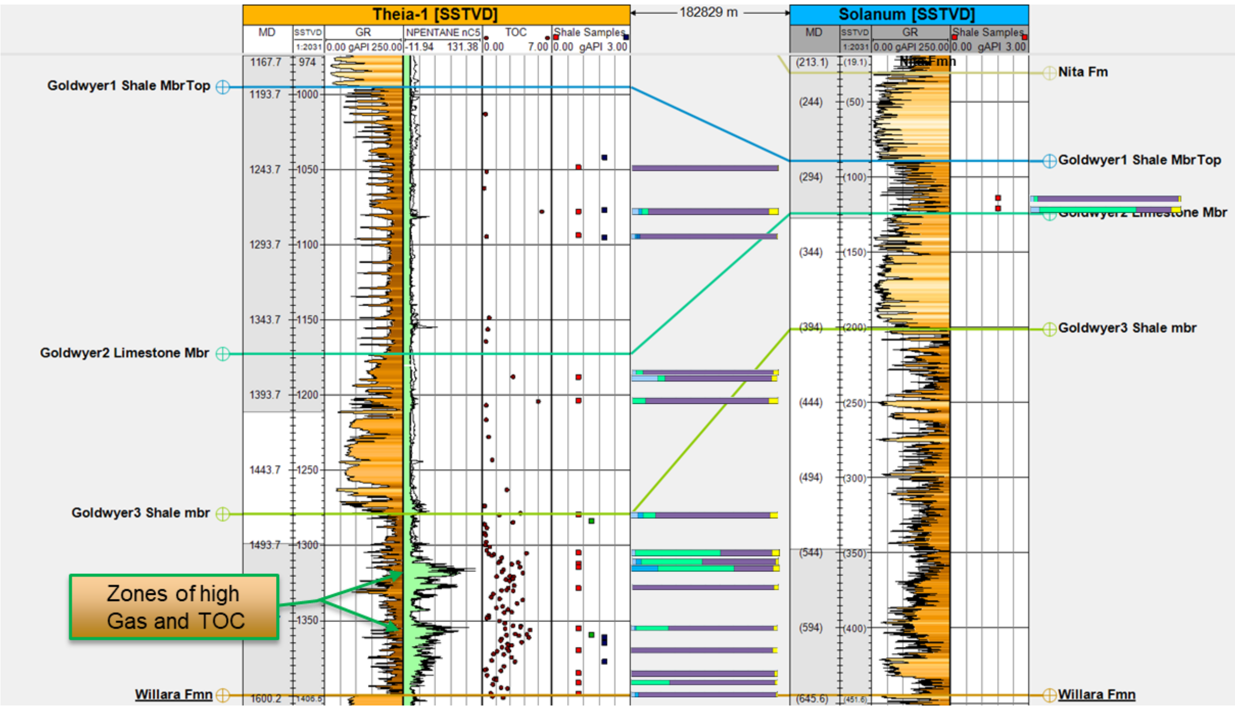

Figure 4. Goldwyer Formation sections in Theia-1 and Solanum-1 datumed on top Willara Formation. Logs include gamma ray and downhole gas readings and a summary of maceral analysis. Red and Blue squares indicate the sample depths used in the study.

The Liptinites are dominated by two main liptinite macerals: Alginite (Telalginites and Lamalginites) and Bituminite, which commonly occur in the same sample.

Telalginites

Telalginites are large algal clasts that include the characteristic Ordovician algae *G. prisca* which is known to be the main contributor to source rocks such as the Bakken Shale. It is not known for certain if *G. prisca* was a eukaryote (algae) or prokaryote (bacteria) although it is morphologically similar to the extant *Botryococcus baunii* which is a green algae.

The Theia-1 samples contain very few clasts of *G. prisca* and the preservation is poor. In contrast, some samples from Solanum-1 were analyzed for comparison because they were known to contain high proportions of *G. prisca* (Johnson *et al*., 2020) as shown in Figure 5.

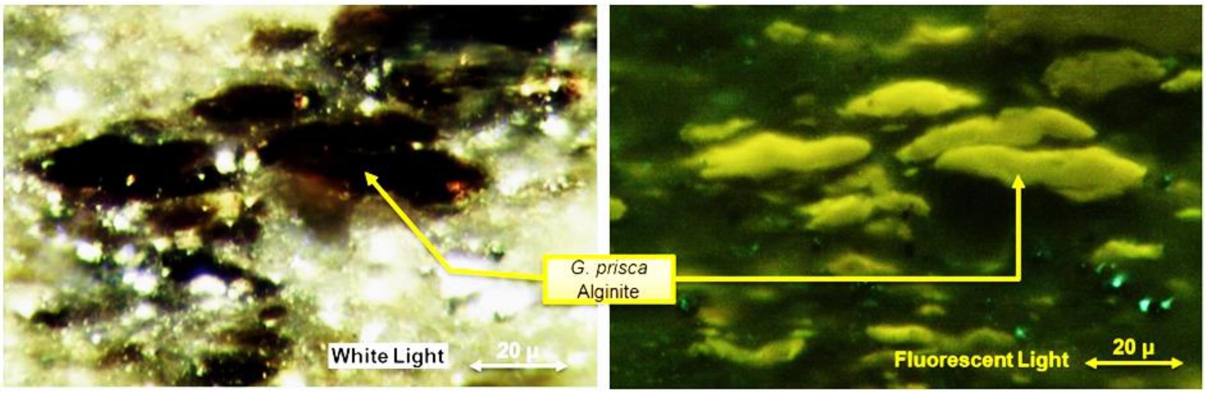

Figure 5. Photomicrograph of *G. prisca* algal bodies in a groundmass of silty clay. Solanum-1 315mMD. Incident white light (left), blue light excitation (right), oil immersion, x50 objective.

Solanum-1 sample at 315mMD also contains a high proportion of associated liptinite in some beds comprising a wide variety of liptinite macerals including protospores, acritarchs, and other phytoplankton cysts, all of which are highly fluorescent bright yellow. The yellow fluorescence indicates that the liptinite macerals are both potentially generative and still within the oil generative zone, as their fluorescence will diminish rapidly over the range (Ro) of ~1.3-1.5% as they go through a major chemical change associated with the end of the main oil window and into the main gas window (Smith & Cook, 1980).

Lamalginite

Some samples from Theia-1 contain diffuse laminae or bands of algal filaments or mats within the clay groundmass, interspersed with small globular algal bodies and are weakly fluorescent. Some enigmatic filamentous or laminae of liptinite phytoclasts occur for which the affinity is unclear but are probably algal (i.e. Lamalginite, Figure 6).

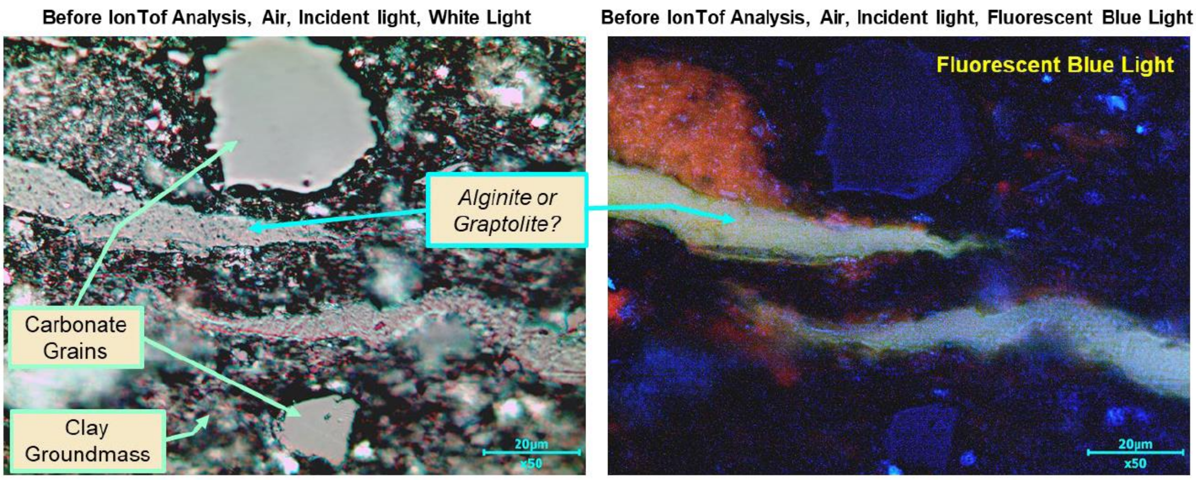

Figure 6. Photomicrograph 1507.6mMD from Theia-1 showing elongate phytoclast in a groundmass of carbonate grains with a clay matrix.

<u>*Bituminite*</u>

Some Theia-1 samples have a fine-granular groundmass of bituminite interbedded with clays (Figure 7). Bituminite is a primary in-situ liptinite whose botanical origin is poorly understood but is thought to be produced by bacterial metabolic and anaerobic degradation of algal remains (International Committee for Coal and Organic Petrology, 2017). The bituminite has weak brown fluorescence which is positive (+ve) with increasing exposure, in contrast to clays which exhibit negative (–ve) fluorescence with exposure.

In Theia-1, the samples that are dominated by high amounts of Bituminite directly correspond with the beds that recorded the high gas readings, which indicates that the bituminite is primarily responsible for the anomalous gas zone within the Goldwyer III (Figure 4).

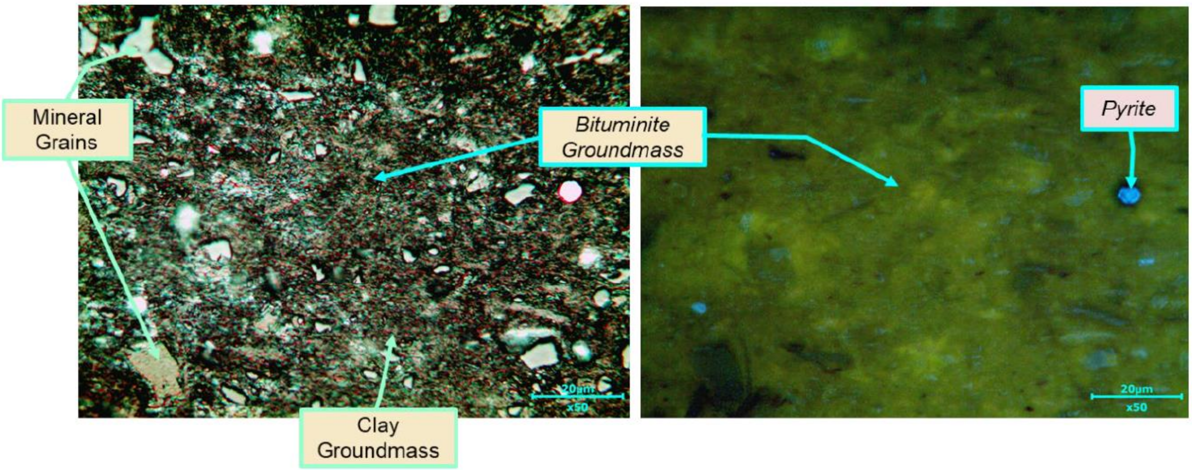

Figure 7: Photomicrograph of Theia-1 1498.5mMD sample with silt-sized quartz, carbonate, and pyrite grains in a Bituminite-Clay groundmass.

IONTOF Analysis

The main aims of the ToF-SIMS organic analysis were to map organic grains in the samples and obtain a Mass-Spectrogam of the organic grains (macerals) and fingerprint each different type of maceral, to distinguish between graptolites, algae, bituminite, and possibly bacteria/archaea.

Graptolites

A sample which contained graptolites in Theia-1 at 1505.7mMD was selected for IONTOF analysis and small 10x10µ areas were analysed (Figure 8). The results indicated that there are relatively few low Carbon mass fragments in the sample, and it is dominated by Oxygen and OH ion fragments, suggesting it is highly aromatic and may have been oxidized. The mass spectra are dominated by the O and OH ion peaks so to emphasise the minor peaks a log scale has been used for the plot (Figure 9).

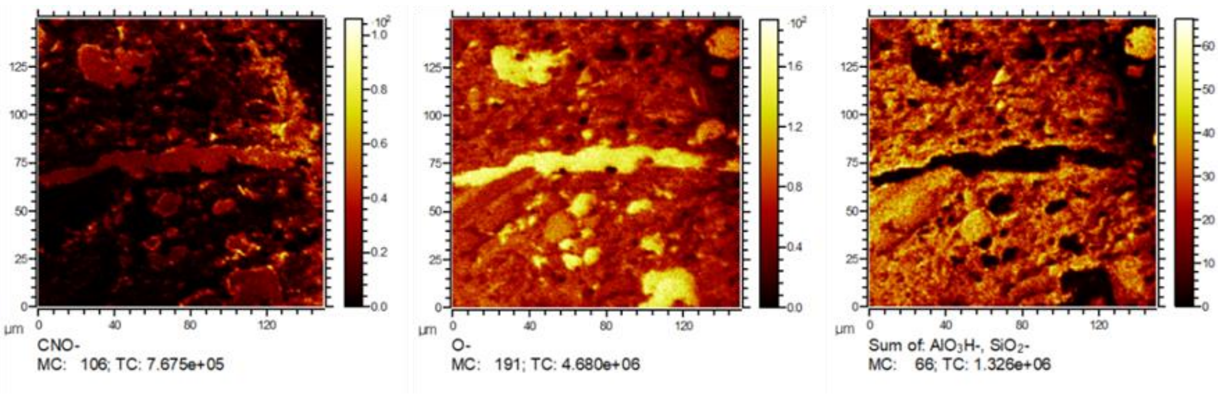

Figure 8. IONTOF Photomicrographs of elongate graptolite and graptolite fragments, with minor carbonate and quartz grains in a clay groundmass. Maps of negative ion fragments for CNO, O and sum of AlO3H and SiO2 ion fragments show the main composition character. Theia-1 1505.7mMD. Graptolite has low number CNO ion fragments (left), is highly oxidised (middle) and is not mineralised (right).

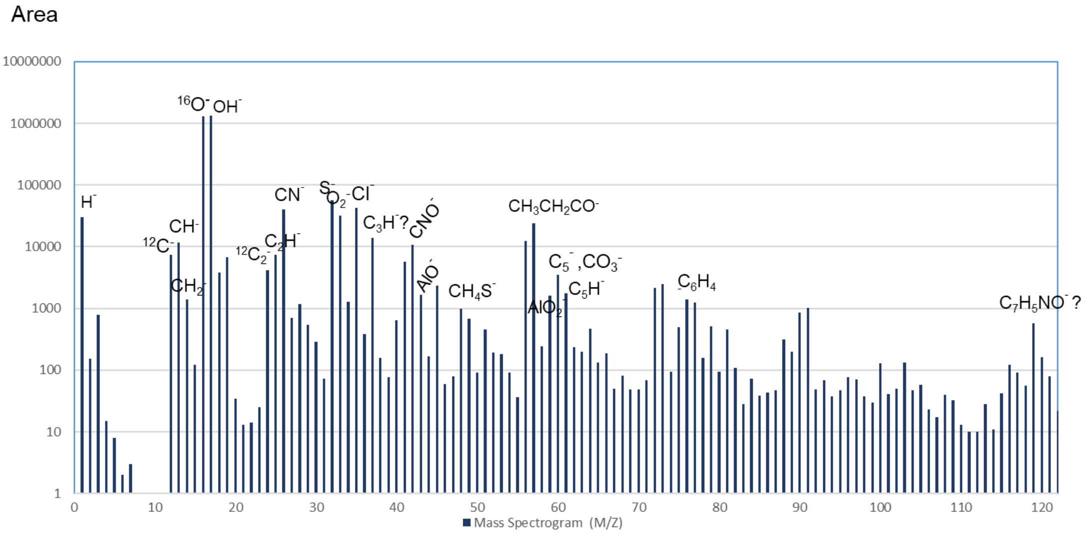

Figure 9. Theia-1 1505.7mMD IONTOF M/Z spectral plot for a Graptolite, –ve ion fragments. Note that a log scale is used because the Oxygen and OH ion peaks are dominant.

<u>Telalginites</u>

Gloeocapsomorpha prisca (*G. prisca*): Traditionally the source of the hydrocarbons in the Canning Basin has been credited to the Ordovician algal remains of *G. prisca*. This is based mainly on analogy with known source rocks such as the Bakken shale. Canning Basin, pyrolysis GCMS analyses of shales have detected traces of chemical biomarker fragments of *G. prisca* and a few studies that have found rare occurrences of these algae in some thin beds (Foster *et al*., 1990; Spaak *et al*., 2017; Johnson, 2019; Johnson, *et al*., 2020). However, these studies have not emphasized that they have only found traces of *G. prisca* in a few thin beds mainly in the Goldwyer I shales and that most beds do not contain *G. prisca* at all. The screening maceral analyses in this study similarly could not find well-preserved *G. prisca* in the Theia-1 samples, in either Goldwyer I, II or III beds. This brings into question the importance of *G. prisca* in the Ordovician source rocks within the Canning Basin. Hence, to obtain a fingerprint of *G. prisca*, some samples from Solanum-1 were used in which *G. prisca* had been reported in the Goldwyer I beds (Johnson *et al*., 2020). The sample from Solanum-1 at 315mMD contains a high proportion of well-preserved *G. prisca* as shown in (Figure 5). The IONTOF maps of this sample clearly show the algal bodies scattered parallel to the bedding (vertical direction) with high organic counts on the left (bright yellow) and very low oxygen counts on the right (black) of Figure 10.

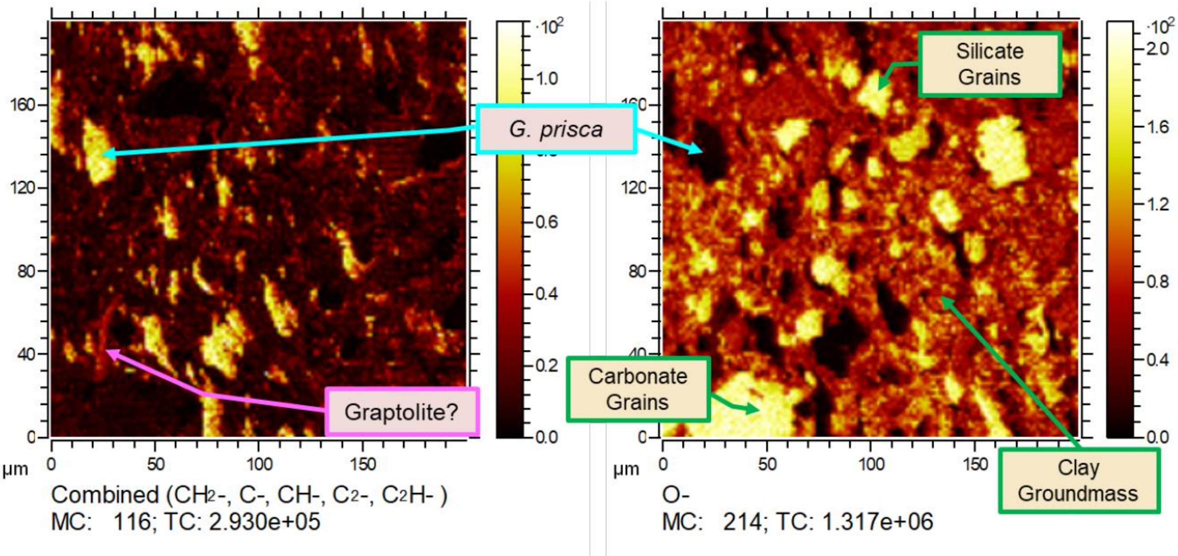

Figure 10. IONTOF Photomicrographs of shale with *G. prisca*, carbonate, and quartz grains in a clay groundmass. The combined map of the main organic ion fragments delineate the *G. prisca* (left) whereas the Oxygen ion fragment map (right) shows high counts for carbonate, quartz, and clay minerals and very low counts in the *G. prisca*. Solanum-1 315mMD, bedding subparallel to the vertical axis.

An area of clean *G. prisca* was analysed and the results indicate the area is almost entirely made up of organics, with a few pyrite spheroids and disseminated organic sulphur. A very clean area within that map was isolated as a region of interest (ROI) in the analysis software over which to produce the mass spectrogram (M/Z). The mass spectrogram of negative ion fragments for the *G. prisca* body is shown in Figure 11. It mainly contains Carbon (C) and Hydrogen (H) ion fragments with very few Oxygen (O) fragments and the C and H form a homologous series decreasing to higher carbon numbers. This is very similar to M/Z for hydrocarbons or lipids which is remarkable. C_2 dominates with minor C and C_4 and then decreasing amounts of higher C numbers, with essentially no inorganics observed.

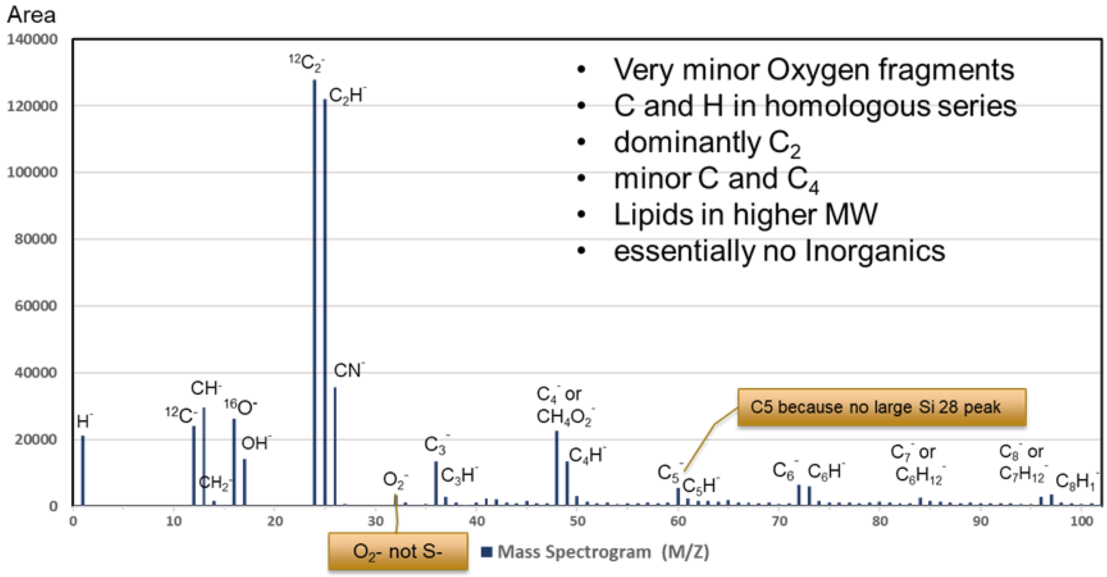

Figure 11. Solanum-1 315mMD sample - IONTOF mass spectra for *G. prisca*. Negative ions, M/Z range 0-100 on arithmetic scale.

Lamalginite

An area was selected for IONTOF analysis in the Theia-1 sample at 1507.96mMD which is a sample in the zone of highest gas readings (Figure 6). This sample and the adjacent sample contain very high proportions of alginite including telalginite and lamalginite in a groundmass dominated by bituminite and clays (Figure 12). The sample was taken into the IONTOF and a small 50x50μ area was analysed and shown to be entirely organic with only very low amounts of disseminated Sulphur that is probably combined with the organic molecules. The mass spectrogram M/Z plot is shown in Figure 13. This shows a complete dominance of Carbon and Hydrogen negative ion fragments indicating fragmentation from organic molecules of low aromaticity. There is an extensive homologous series of Carbon-Hydrogen ion fragments and it is very similar to the pyrolysis GCMS from the adjacent sample (Johnson, 2019). This is a truly remarkable M/Z plot and clearly indicates the current generative potential of this type of organic matter.

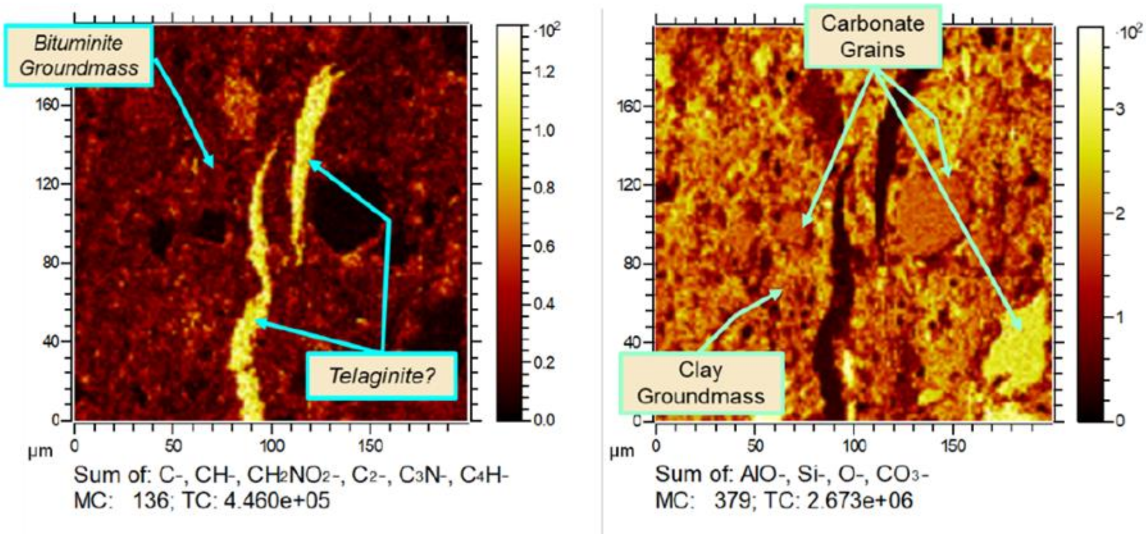

Figure 12: IONTOF Photomicrographs of Theia-1 1507.96mMD shale with filamentous or lamella Lamalginite?, carbonate and very fine quartz grains in a bituminite and clay groundmass. Combined map of organic ion fragments (left) clearly shows the elongate Lamalginite and nano-sized bituminite. Main inorganic ion fragments combined on the right showing high counts for carbonate, quartz and clay minerals. Note the bedding is subparallel to the vertical axis.

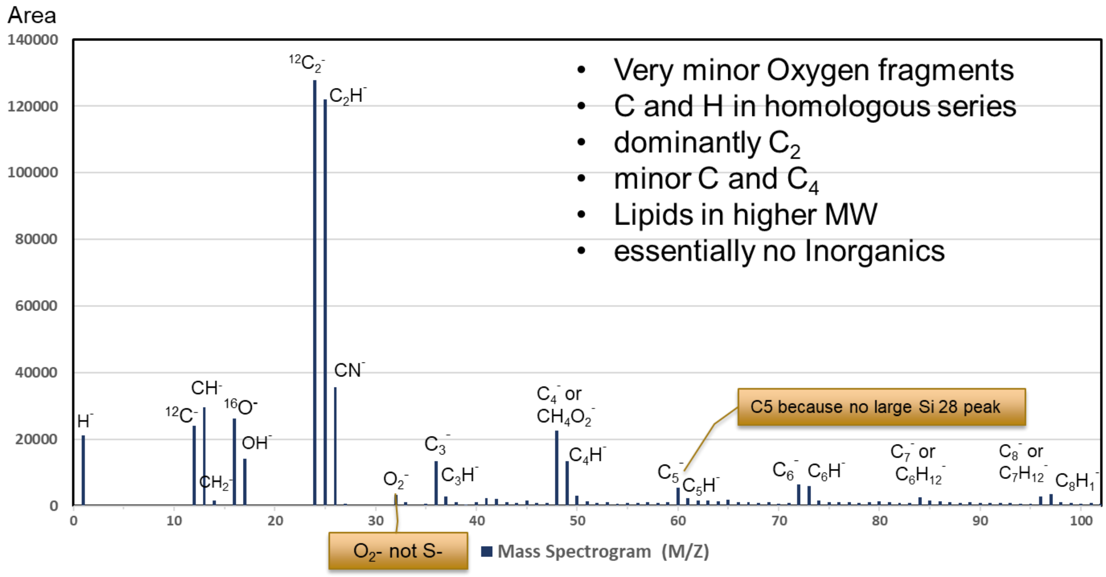

Figure 13. Solanum-1 315mMD sample - IONTOF mass spectra for *G. prisca*. Negative ions, M/Z range from 0-100 on an arithmetic scale.

Bituminite

An area was selected for IONTOF analysis in the Theia-1 1498.5mMD sample which contained a high proportion of bituminite with areas largely free from clastics (Figure 7). The sample was taken into the IONTOF and a small 50x50µ area was analysed and shown to be mainly bituminite and clays. Remarkably, the map of the organics showed very small elongate bodies typically 2x5µ that resemble the much larger *G. prisca* algal bodies (Figure 14). This suggests that bituminite is composed of smaller organic bodies of a similar size to Bacteria and the smaller forms may be Archaea. An ROI threshold analysis of these bodies was done in an attempt to obtain a pure M/Z for these bodies as with the corresponding M/Z plot given in Figure 15. The oxygen and OH peaks are presumably a combination of ion fragments from the organic matter and the clays, although the aluminum oxide ion fragments are low suggesting the clays are not the main component. Corresponding pyrolysis GCMS analysis is required to help distinguish an algal from bacterial signature. Spaak, *et al*. (2017) reported a bacterial signature for Goldwyer I samples in Solanum-1 but no data was obtained for Theia-1.

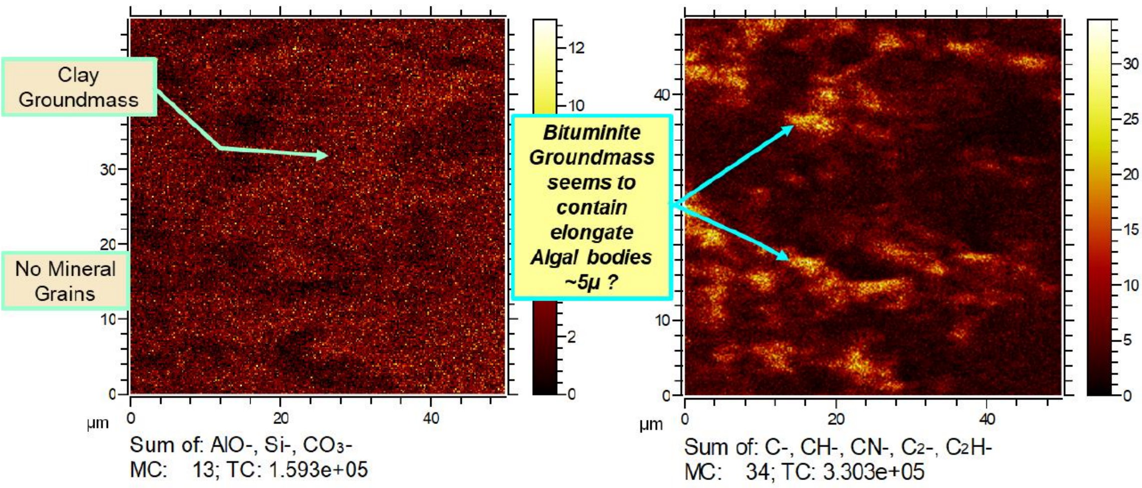

Figure 14: IONTOF Photomicrographs of an analysed small area of shale comprising bituminite-clay groundmass (blue square in Figure 22). Map of negative ion fragments for main inorganics on left demonstrating the area contains no mineral grains. Map of organics on right showing micron-sized elongate bodies resembling larger scale algal bodies. Theia-1 1498.5mMD, bedding subparallel to horizontal axis.

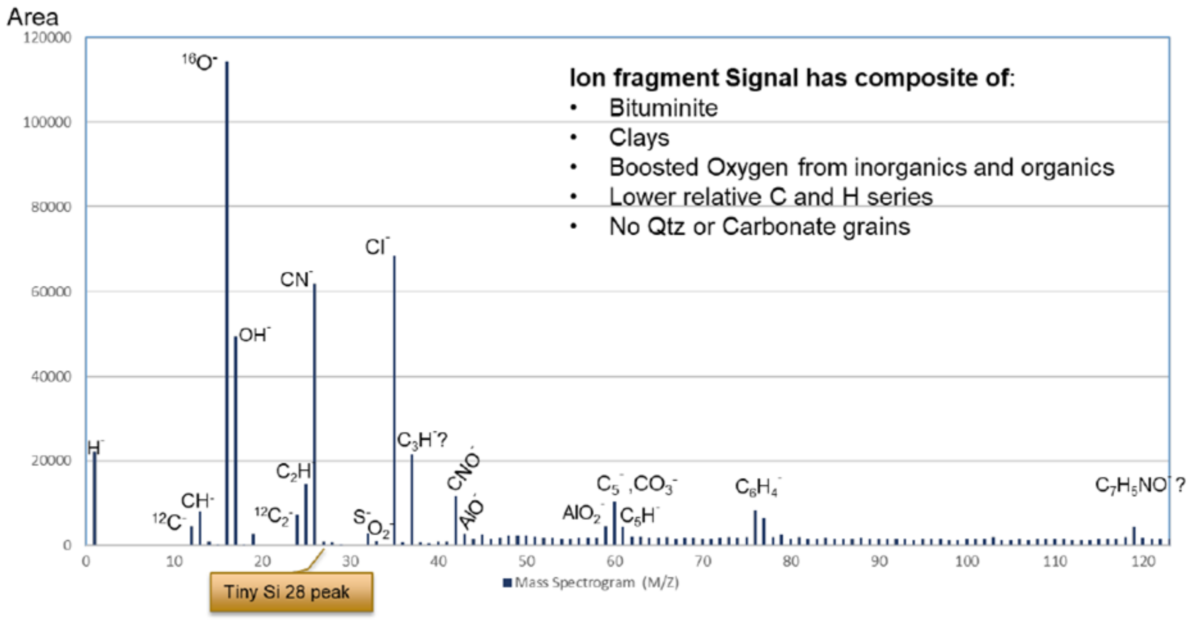

Figure 15: Theia-1 1498.5mMD sample - IONTOF mass spectra for Bituminite oval bodies segregated using threshold technique. Negative ions, M/Z range 0-120.

Given that the target anomalous gas zone in Theia-1 corresponds to the zone dominated by bituminite, it is probable the real source of these hydrocarbons is from sub-microscopic organisms. Bacteria and possibly Archaea are prime candidates although input from degraded algae cannot be discounted and requires further investigation. The generation of hydrocarbons from bacteria has long been suspected and these preliminary results appear to support the theory of Wang *et al*, (2020).

CONCLUSIONS

This study has obtained a better understanding of the organic matter types in the Ordovician Goldwyer Formation, Canning Basin. Direct non-destructive mass spectrograms have been produced for the first time using the IONTOF and the results are able to indicate the generative potential or otherwise of the specific different organic matter grains in the Ordovician source rocks. This technique should be extended to other known or potential source rocks throughout the Canning and other basins. One of the main practical results came from the routine screening of the samples using optical fluorescent microscopy prior to selection of the best sample for ToF-SIMS analysis. The fluorescent microscopy was in fact able to identify the main hydrocarbon zones in Theia-1 independent from the mudlogs. This was a very quick routine visual screening that surprisingly matched the main hydrocarbon zones with very little effort. Coupled with the new understanding of Bituminite in the groundmass this is a powerful technique.

The traditional source for the hydrocarbons in the Broome Platform has long been attributed to the Ordovician shales in the Goldwyer Formation and from the extinct alga *G. Prisca*. However, beds containing *G. prisca* are rare and no well-preserved *G. prisca* phytoclasts were found in Theia-1 samples nor have they been found by any other studies. Although *G. prisca* algal bodies from Solanum-1 well do have excellent generative potential as shown by the IONTOF mass spectrograms, this algae occurs in very thin beds within the Goldwyer I unit, for which there is only a limited understanding of its geographic distribution across the basin. Previous studies have only identified *G. prisca* in a few wells and have reported its occurrence in thin beds as well. This suggests that volumes of *G. prisca* present in the Canning Basin may not be large enough for the generation of economic hydrocarbons. Instead, this study has found that Bituminite in the groundmass together with Lamalginite-rich bands are primarily responsible for the main hydrocarbon zones in Theia-1. The IONTOF analyses show that bituminite is probably produced by micro-organisms and the most likely affinity is bacteria in the size range of 1-5µ.

The main hydrocarbon zones in Theia-1 are also characterised by filamentous or lamella alginite, (Telaginite or Lamalginite), of so far indeterminate affinity. Some beds contain diffuse laminae or bands of algal filaments or mats and these are interbedded with Bituminite. The mass spectrogram M/Z plots for both the Bituminite and the Lamalginite indicate these organic matter types are capable of and currently generative source rocks. In contrast, the mass spectrogram M/Z plots for Graptolites show a highly oxidized and very low Carbon-Hydrogen makeup. This presumably indicates they dominantly comprise aromatic molecules that are not fragmenting into the low atomic number ions which are very different from the alginite macerals.

Notwithstanding the excellent quality of the organic matter identified in this study, most of the Goldwyer Formation shales contain low proportions of organic matter. The beds that do contain organic matter are thin and need to be carefully identified and tracked if geosteering is to be utilized in their production.

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