



## **GEOLOGICAL SURVEY OF CANADA**

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M. G. Fowler, M. Obermajer, S. Achal, and M. Milovic

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## **Open File 5342 (CD-ROM)**

### **Results of geochemical analyses of an oil sample from Mizzen L-11 well, Flemish Pass, offshore Eastern Canada**

**Fowler, M.G., Obermajer, M., Achal, S. and Milovic, M.**

#### **INTRODUCTION**

A small amount of oil from the Mizzen L-11 well that was drilled in the Flemish Pass in 2003 was provided for geochemical analysis. The Mizzen L-11 well was classified as abandoned with an oil show. The sample came from a depth of 3350.6 m corresponding to Late Jurassic "Baccalieu Sandstone" (Canada Newfoundland Labrador Offshore Petroleum Board, 2005). This Open File contains the digital files of the data obtained from organic geochemical analyses of the Mizzen L-11 oil sample.

#### **EXPERIMENTAL**

Prior to fractionation, the sample was analysed by whole oil gas chromatography. Whole oil gas chromatography was carried out using a Varian 3400 GC equipped with a flame ionization detector (FID) and on-column injector. The column was a 30m x 0.25mm i.d x 0.25µm thickness DB1. The temperature program was 33°C for 6 minutes, ramped to 63°C at a rate of 3°C/min, then to 320°C at a rate of 6°C/min and held for 10 minutes. Gasoline range hydrocarbons (C5-C8) in the original oil sample were analyzed on a HP 5890 Gas Chromatograph with a flame ionization detector (FID) connected to an OI Analytical 4460 Sample Concentrator. The column was a 60 m DB-1 0.32 mm i.d. with a 1µm film thickness. The temperature program was 30 to 40°C at a rate of 1°C/min. The initial and final temperatures were held for 10 and 25 minutes respectively. Total analysis time was 45 minutes.

As only small amount of oil was available, it was not distilled prior to fractionation. Fractionation was carried out using liquid chromatography. A mixture of 28-200 mesh Silica Gel (MCB) and 80-200 mesh alumina (ALCOA) (1:3 by weight respectively) was used as a support for the column. The support is activated by heating at 120°-150°C for 12 hours. A glass wool plug is placed at a bottom of the column and covered with a 1 cm thick layer of sand. The support, weighed as 1 g of support/10 mg of sample, is slowly settled in pentane and any air trapped is released by gentle tapping on the column. The sample, is then added to the column. Saturates are recovered by eluting with pentane (3.5 ml/g support), aromatics with a 50:50 mixture of pentane and dichloromethane (4 ml/g support), resins with methanol (4 ml/g support) and any remaining asphaltene with chloroform. The solvents are rotary-evaporated, separate fractions transferred to tared 1 dram vials, dried in a slow stream of nitrogen and weighed to constant weight.

Saturated and aromatic hydrocarbon fractions were analyzed using gas chromatography (GC) and gas chromatography-mass spectrometry (GC-MS). Gas Chromatography used a Varian 3800 FID gas chromatograph with a 30 m DB-1 column with a temperature program from 60°C to 300°C at a rate of 6°C/min and then held for 30 minutes at 300°C. GC-MS analyses were performed on a HP 6890 Series GC coupled to a HP 5973 Series Mass Selective Detector operated in selective ion monitoring (SIM) mode. The GC was fitted with a 30 m DB-5ms column. During the saturate fraction analysis the temperature was initially held at 100°C for 2 min and then programmed at 40°C/min to 180°C and at 4°C/min to 320°C, then held for 7 min. The aromatic fraction analysis was initiated at 100°C, programmed at 3°C/min to 300°C, then held for 10 min. The column was an Agilent DB-5MS, 30 m x 0.32 mm i.d. and a film thickness of 0.25 microns. The addition of 100 µl of internal standard (D4 cholestane, 0.1 mg/ml) during the saturate fraction analysis allows absolute quantification of detected biomarker compounds.

## RESULTS

The gross composition of the Mizzen oil sample is as follows:

|                           |      |
|---------------------------|------|
| % hydrocarbons            | 81.9 |
| % saturated hydrocarbons  | 48.3 |
| % aromatic hydrocarbons   | 33.6 |
| % resins plus asphaltenes | 14.8 |

The predominance of n-alkanes and lack of a hump of unresolved compounds in the gas chromatograms indicate that the oil is not biodegraded. It generally shows saturate hydrocarbon and gasoline range characteristics similar to those observed for Jeanne d'Arc Basin oils (Fowler and Obermajer, 2001). The pristane/phytane ratio of 1.53 is on the high side of the range observed previously for Jeanne d'Arc Basin oils and is in the range of values observed for those oils believed to have a contribution from a source other than the Egret Member with a greater terrestrial contribution to its organic matter (Fowler et al., 1989; Fowler and McAlpine, 1995).

As indicated by the m/z 191 fragmentogram, the terpene distributions are very similar to those observed for Jeanne d'Arc Basin oils that are sourced from the Egret Member (e.g. Fowler and McAlpine, 1995); for example, the relatively high amounts of the rearranged hopanes including C<sub>29</sub> Ts and the C<sub>30</sub> diahopane and the low abundance of tricyclic and tetracyclic terpenes. The sterane carbon number (C<sub>27</sub>-C<sub>29</sub>) distribution is similar to Egret Member sourced oils. However, the sterane isomer distribution appears to indicate a very low maturity, lower than would be expected from the hopane isomers and closer to that expected for an immature source rock rather than a conventional oil. This is suggested by the predominance of the 5 $\alpha$ (H),14 $\alpha$ (H),17 $\alpha$ (H) 20R over the 5 $\alpha$ (H),14 $\alpha$ (H),17 $\alpha$ (H) 20S and 5 $\alpha$ (H),14 $\beta$ (H),17 $\beta$ (H) isomers and low abundance of diasteranes. Dinosteranes are abundant but not as high as generally found in Egret Member sourced oils.

Aromatic maturity parameters do not indicate a consistent maturity for the Mizzen oil. For example, the vitrinite reflectance calculated based the distribution of methylphenanthrenes ranges from 0.72% using the equation of Boreham et al. (1988) to 0.83% using the equation of Radke (1988). However, it is the experience of this laboratory that the Methylphenanthrene Index is not a reliable indicator of maturity for Jeanne d'Arc Basin oils. The distribution of methyl dibenzothiophenes has been found to work better for these oils. Using the equation of Radke (1988) to link the distribution of these compounds with a vitrinite reflectance gives a vitrinite reflectance equivalent of 0.67% indicating a relatively low maturity oil but not as low as suggested by the sterane distributions. Additional evidence for the low maturity of the Mizzen sample is provided by the very low triaromatic cracking ratio of 0.06.

## DISCUSSION

The variation in the maturity of the Mizzen L-11 oil sample indicated by different parameters suggests it likely has a complex origin. Some characteristics are those expected for an oil sourced from mature Egret Member or equivalent. Others, especially the steroidal compounds, suggest a very low maturity for an oil. A possible explanation is that this sample contains hydrocarbons from multiple sources. Previous reports have suggested the occurrence of multiple source rock intervals within the Upper Jurassic in Flemish Pass wells such as Panther P-52 (e.g. Fowler and McAlpine, 1995). Thus this sample could have a contribution from a deeper, more mature source interval such as those in the Rankin Formation (including the Egret Member) and from a lower maturity interval in close proximity to where the oil sample originated (perhaps the Tithonian source rock interval). A "Rankin Equivalent" was identified in this well at a depth of 3770 m (Canada Newfoundland Labrador Offshore Petroleum Board, 2005) and the sample would appear to come from the Fortune Bay-Jeanne d'Arc Formation interval where the Tithonian source interval is usually recognised (Fowler and McAlpine, 1995). It may also be significant that it is the sterane distributions that appear to be anomalous as these compounds are in significantly lower concentrations than the terpenes and therefore more likely to be affected by any mixing of hydrocarbons from a lower and higher maturity source. However, at present in the absence of additional data from this well, this must remain conjecture.

While the sample shows some characteristics similar to the Egret Member sourced oils from the Jeanne d'Arc Basin, there are also some differences. These could reflect changes in the Egret Member organic facies on moving into the Flemish Pass or the mixing of hydrocarbons from multiple sources as suggested above to explain the variations in thermal maturity indicated by different classes of compounds.

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