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# CONTINENTAL SHELF INSTITUTE

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A total of twenty three samples were subjected to Total Organic Carbon (TOC), Rock-Eval and Pyrolysis-gas chromatography (Py-GC) analysis.

The TOC values vary from extremely low (0.01%) to extremely high (52.07%).

The T  $_{\rm max}$  values from Rock-Eval pyrolysis indicate immaturity for most of the samples.

The kerogen analysis by Py-GC is in good agreement with the Rock-Eval results, suggesting that the samples contain the following kerogen types: M-9089 (1876m) and M-9090 (1885.5m): III/IV or IV; M-9091 (1892m): II or mixed II/III; M-9092 (1900m) to M-9095 (1907m): mixed II/III and III/II; M-9096 (1909.5m) to M-9100 (2349.9m): III/IV or IV; M-9101 (2391m) and M-9102 (2451.5m): III or III/IV; M-9103 (2484.7m): III; M-9104 (2531m) and M-9087 (2709.1m): III; M-9105 (2738.5m): III/IV or IV; M-9106 (2750.5m): III;M-9107 (2759.5m): III or III/IV; M-9108 (2761.6m) III and M-9109 (2811m): III/IV or IV.

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Pvrolvsis

Analysis

KEY WORDS/ STIKKORD

SUMMARY/ SAMMENDRAG

#### EXPERIMENTAL

## Total Organic Carbon (TOC)

The samples were crushed and aliquots were weighed into Leco crucibles, treated with hot 2N HCl to remove carbonate, and washed twice with destilled water to remove traces of HCl. The crucibles were then placed in a vacuum oven at  $50^{\circ}$ C and evacuated to 20mm Hg for 12 hrs. The total organic carbon (TOC) content of the dried samples was determined using a Leco EC12 carbon analyser. Results are given in Table 1.

## Rock-Eval Pyrolysis

100mg crushed sample was weighed into a platinum crucible the base and cover of which are made of sintered steel, and analysed on a Rock-Eval pyrolyser. Results are given in Table 1.

## Pyrolysis-Gas Chromatography (Py-GC)

Two Py-GC methods were used in this study. A temperature programmed pyrolysis technique, Py-GC (Progr.), was applied for samples with TOC values of ca. 1% and lower. Flash pyrolysis at  $600^{\circ}$ C, Py-GC ( $600^{\circ}$ C), was used for samples with TOC values higher than 1%.

### Thermal Extraction

20-30mg of fine ground whole rock sample was placed in a boat shaped sample probe and heated in a stream of helium at 300<sup>0</sup>C for five minutes.

## $Py-GC (600^{\circ}C)$

A chemical Data Systems (CDS) Pyroprobe 120 interfaced to a Varian 3700 gas chromatograph was used for flash pyrolysis. Fine ground thermally extracted whole rock sample was suspended in methanol and added to the platinum ribbon sample probe with a disposable Pasteur pipette. The sample was flash pyrolysed and flushed directly onto a capillary column via a laboratory built interface/splitter and analysed under the GC conditions given below.



Instrumental conditions:

Pyrolysis: 600<sup>°</sup>C for 5 sec. in nitrogen Column: 25m OV-1 fused silica capillary; ID 0.3mm Carrier gas: Nitrogen with inlet pressure 6 psi; Flow ca. 1.5ml/min. Split ratio: 1:30. Oven programme: 40<sup>°</sup>C/1 min. to 270<sup>°</sup>C at 4<sup>°</sup>C/min.

## Py-GC (Progr.)

20-30mg of thermally extracted whole rock sample was pyrolysed in a stream helium  $(300^{\circ}C \text{ to } 550^{\circ}C \text{ at } 35^{\circ}C/\text{min.})$  using a tube furnace type pyrolyzer interfaced to a Varian 3700 gas chromatograph. The outlet of the pyrolyzer was connected to a fused silica capillary column via an interface/splitter (sample/split ratio; 1:30). The pyrolysis product was trapped in a cooled (liquid nitrogen) U-shaped part at the front of the column.

The outlet of the splitter was connected to a flame ionisation detector (FID) and the course of the pyrolysis could be followed by the detector response of the bulk pyrolysis product which was recorded as a broad peak. At the end of the pyrolysis, the trapped pyrolysis product was injected by removing the liquid nitrogen bath at ambient temperature and analysed under the GC conditions given below.

Instrumental conditions: Pyrolysis: 300°C to 550°C at 35°C/min. Column: 25m OV-1 fused silica capillary; ID 0.3mm. Carrier gas: Helium with inlet pressure 10 psi; Flow ca. 1.5 ml/min. Split ratio: 1:30. Oven program: 40°C/1 min. to 270°C at 4°C/min.

## **RESULTS AND DISCUSSION**

A total of twenty three samples were subjected to TOC, Rock-Eval and Py-GC analysis.

## Total Organic Carbon (TOC)

The TOC values are listed in Table 1 varying from extremely low (0.01%) to very high (52.07%).

## Rock-Eval Pyrolysis

M-9088 (1868m) The results are misleading due to very low TOC value.

<u>M-9089 (1876m) and M-9090 (1885.5m)</u> The low T<sub>max</sub> values indicate immaturity. The low hydrogen indices are typical for type III/IV and IV kerogens. Type IV kerogen is used to describe the inertinite group of macerals or reworked material with a poor potential for hydrocarbons (mainly gas).

<u>M-9091 (1892.5m)</u> The hydrogen index (455) is characteristic for a type II kerogen.  $T_{max}$  indicate immaturity.

<u>M-9092 (1900m), M-9093 (1903m), M-9094 (1906m) and M-9095 (1907m).</u> The moderate hydrogen indices (227 to 378) and low  $T_{max}$  values suggest immature mixed type II/III and III/II kerogens for these samples.

<u>M-9096 (1909.5m)</u>, <u>M-9097 (1916m)</u>, <u>M-9098 (1935m)</u> and <u>M9099 (2295m)</u>. The low hydrogen indices are typical for type III/IV and IV kerogens.  $T_{max}$  indicate immaturity.

<u>M-9100 (2349.9m)</u> The low hydrogen index suggests a type III/IV or IV kerogen. The oxygen index is too high.  $T_{max}$  seems very high and is probably misleading.

<u>M-9101 (2391m)</u> The low hydrogen index and  $T_{max}$  value suggest an immature type III or III/IV kerogen.

<u>M-9102 (2451.5m)</u> The low hydrogen index indicates a type III/IV or IV kerogen.  $T_{max}$  seems too high and is probably misleading.

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<u>M-9103 (2484.7m)</u> The hydrogen index and T<sub>max</sub> value suggest an immature type III kerogen.

<u>M-9104 (2531m)</u> The low hydrogen index and  $T_{max}$  value suggest an immature type III or III/IV kerogen.

<u>M-9087 (2709.1m)</u> The hydrogen index and  $T_{max}$  value suggest an immature type III kerogen.

<u>M-9105 (2738.5m)</u> The low hydrogen index indicates a type III/IV or IV kerogen. T<sub>max</sub> (547<sup>0</sup>C) is far too high.

<u>M-9106 (2750.5m)</u> The hydrogen index and  $T_{max}$  value suggest an immature type III kerogen.

<u>M-9107 (2759.5m)</u> The hydrogen index suggests a type III or III/IV kerogen.  $T_{max}$  indicates immaturity or marginal maturity.

<u>M-9108 (2761.6m)</u> The hydrogen index and  $T_{max}$  value suggest an immature type III kerogen.

<u>M-9109 (2811m)</u> The low hydrogen index is typical for type III/IV and IV kerogens. T<sub>max</sub> seems very high and is probably misleading.

## Pyrolysis-Gas Chromatography (Py-GC)

The instrumental conditions are described in the experimental section. Based on retention and mass spectrometric data from other kerogens, the peaks in the pyrograms are tentatively identified: The numbered peaks are n-alkene/n-alkane doublets of the corresponding carbon number. The alkenes have the shorter retention time. T = toluene; X = m/p-xylenes and Pr = pristenes.

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<u>M-9088 (1868m)</u> No pyrogram was obtained for this sample due to very low TOC value.

<u>M-9089 (1876m) and M-9090 (1885.5m)</u> The pyrograms of these two samples are very similar showing a high abundance of aromatics and a short range  $(C_7 \text{ to } C_{15})$  aliphatic homology. The abundance of the alkanes is much higher than of the alkenes. The pyrograms show a type III or mixed type III/IV kerogen fingerprint.

<u>M-9091 (1892.5m), M-9092 (1900m), M-9093 (1903m), M-9094 (1906m) and</u> M-9095 (1907m)

The pyrograms of these five samples are very similar showing a high abundance of aromatic compounds suggesting a type III kerogen derived from higher plants. However, the pyrograms also show an n-alkene/n-alkane homology ranging from  $C_7$  to  $C_{25}$  indicating an input of lipid material. The pyrograms show a mixed type III/II kerogen fingerprint.

## <u>M-9096 (1909.5m), M-9097 (1916m), M-9098 (1935m), M-9099 (2295m) and</u> M-9100 (2349.9m)

The pyrogram of these five samples are overall very similar to M-9089 and M-9090, i.e. the pyrograms show a type III or mixed type III/IV kerogen fingerprint.

<u>M-9101 (2391m)</u> The pyrogram shows an n-alkene/n-alkane homology ranging from  $C_7$  to  $C_{21}$ . The abundance of aromatics is high relative to the aliphatics. Generally the pyrogram shows a type III kerogen fingerprint.

<u>M-9102 (2451.5m)</u> The pyrogram is very similar to M-9096, i.e. a type III or mixed type III/IV kerogen fingerprint.

<u>M-9103 (2484.7m)</u> The pyrogram shows an aliphatic homology ranging from  $C_7$  to  $C_{27}$  and a high abundance of aromatics. Generally the pyrogram shows a type III kerogen fingerprint.

<u>M-9104 (2531m) and M-9087 (2709.1m)</u> The pyrograms of these two samples are overall very similar to M-9101, i.e. a type III kerogen fingerprint.

<u>M-9105 (2738.5m)</u> The pyrogram is very similar to M-9089, i.e. the pyrogram shows a type III or III/IV kerogen fingerprint.

<u>M-9106 (2750.5m)</u> The pyrogram shows a high abundance of aromatic compounds indicating a type III kerogen. However, the pyrogram also shows an n-alkene/n-alkane homology with a high abundance in the  $C_{15}$  to  $C_{27}$  region indicating an input of lipid rich material (spores, cuticles, resins etc.). The pyrogram shows a mixed type III/II kerogen finger-print.

<u>M-9107 (2759.5m)</u> The pyrogram shows some similarity to M-9089, i.e. a type III or mixed type III/IV kerogen fingerprint.

<u>M-9108 (2761.6m)</u> The pyrogram shows a high abundance of aromatics and an n-alkene/n-alkane homology ranging from  $C_7$  to  $C_{27}$  indicating a type III kerogen with some input of lipid material.

<u>M-9109 (2811m)</u> The pyrogram is very similar to M-9105, i.e. a type III or III/IV kerogen fingerprint.



#### CONCLUSION

A total of twenty three samples were subjected to TOC, Rock-Eval and Pyrolysis-gas chromatography (Py-GC) analysis.

The TOC values vary from extremely low (0.01%) to extremely high (52.07%).

There is an overall good correlation between the Py-GC and Rock-Eval results. However, until more Py-GC results for type III/IV and IV kerogens are available, it is in some cases difficult to discriminate between a type III and a type III/IV or IV kerogen on the basis of Py-GC results alone.

Due to the complexity of kerogens, kerogen studies should therefore ideally include both microscopic and physico chemical methods in order to more completely understand the nature and interrelationships of the contributing material.

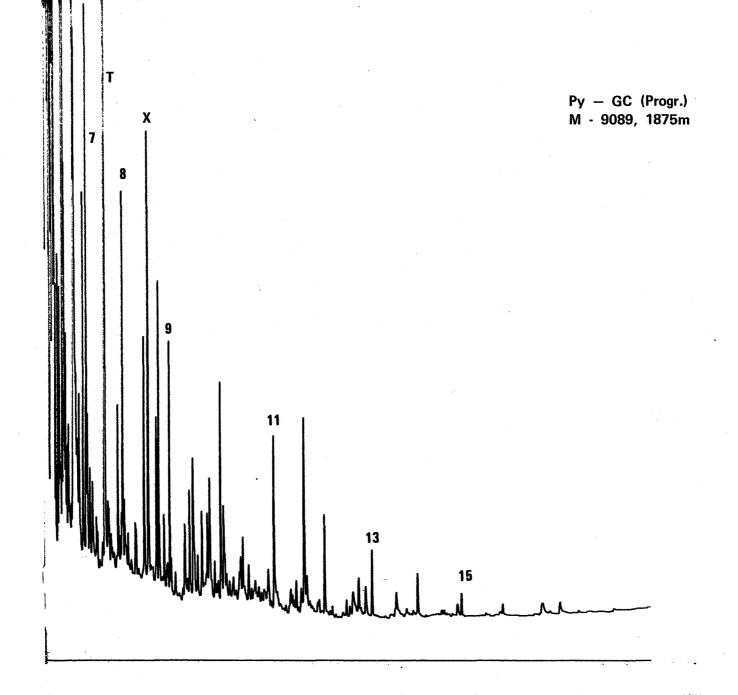
The Rock-Eval and Py-GC results suggest that samples M- 9089 (1876m) and M-9090 (1885.5m) contain immature type III/IV or IV kerogens; M-9091 (1892.5m) and immature type II or mixed type II/III kerogen; M-9092 (1900m) to M-9095 (1907m) immature mixed type II/III and III/II kerogens; M-9096 (1909.5m) to M-9100 (2349.9m) immature type III/IV or IV kerogens; M-9101 (2391m) and M-9102 (2451.5m) immature type III or III/IV kerogens; M-9103 (2484.7m) an immature type III kerogen; M-9104 (2531m) and M-9087 (2709.1m) immature type III kerogens; M-9105 (2738.5m) a type III/IV or IV kerogen; M-9106 (2750.5m) an immature type III kerogen; M-9107 (2759.5m) an immature or marginal mature type III or III/IV kerogen; M-9108 (2761.6m) an immature type III kerogen and M-9109 (2811m) a type III/IV or IV kerogen. TABLE 1.

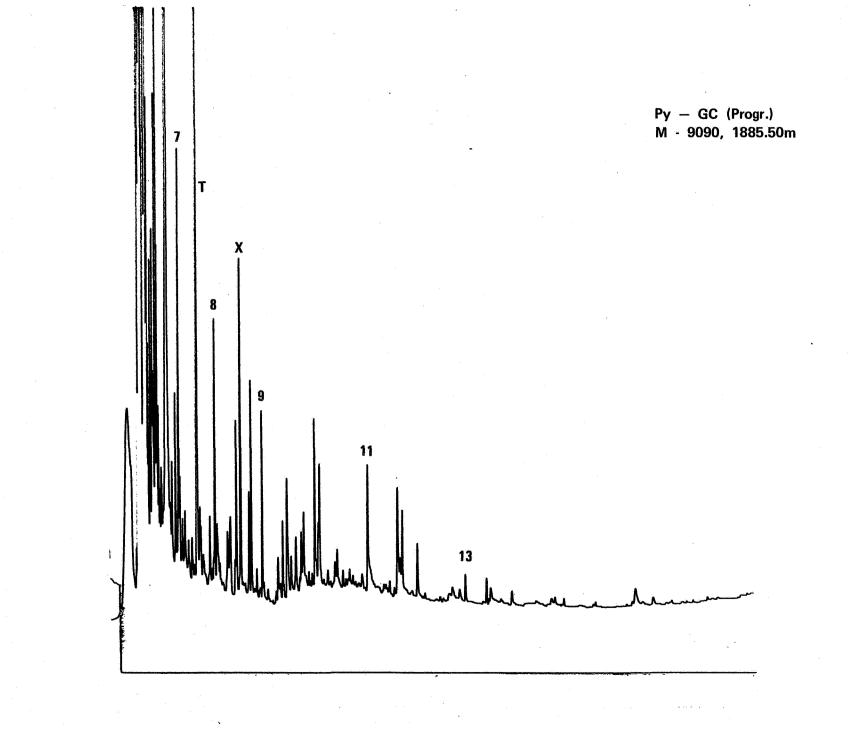
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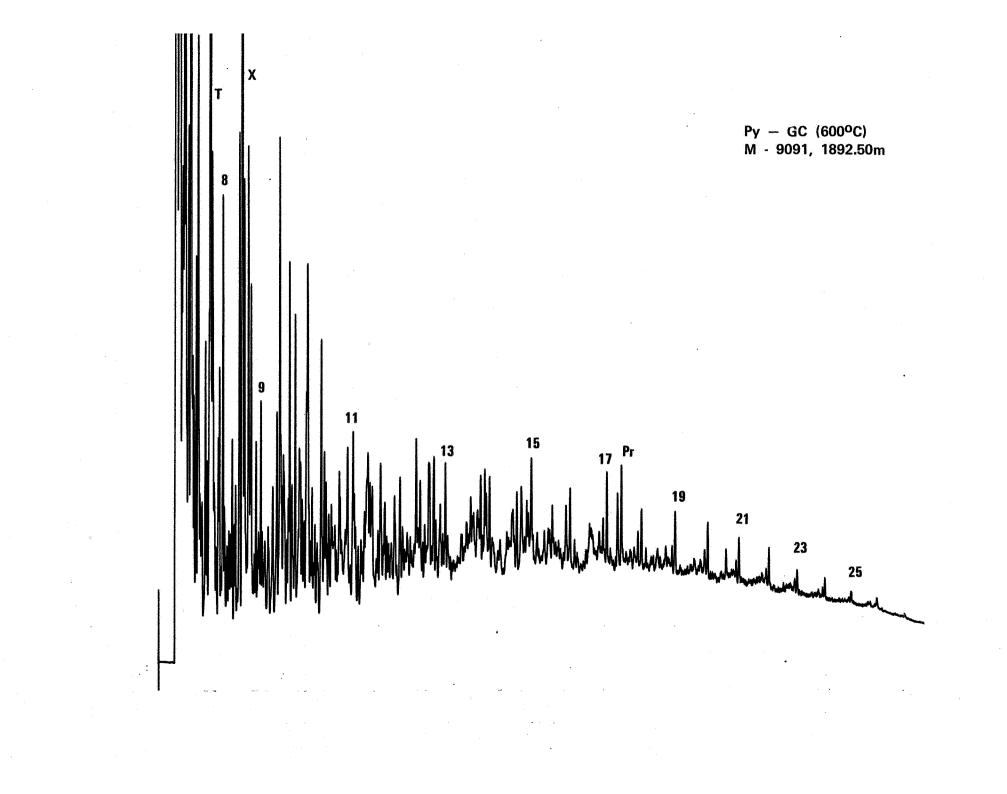
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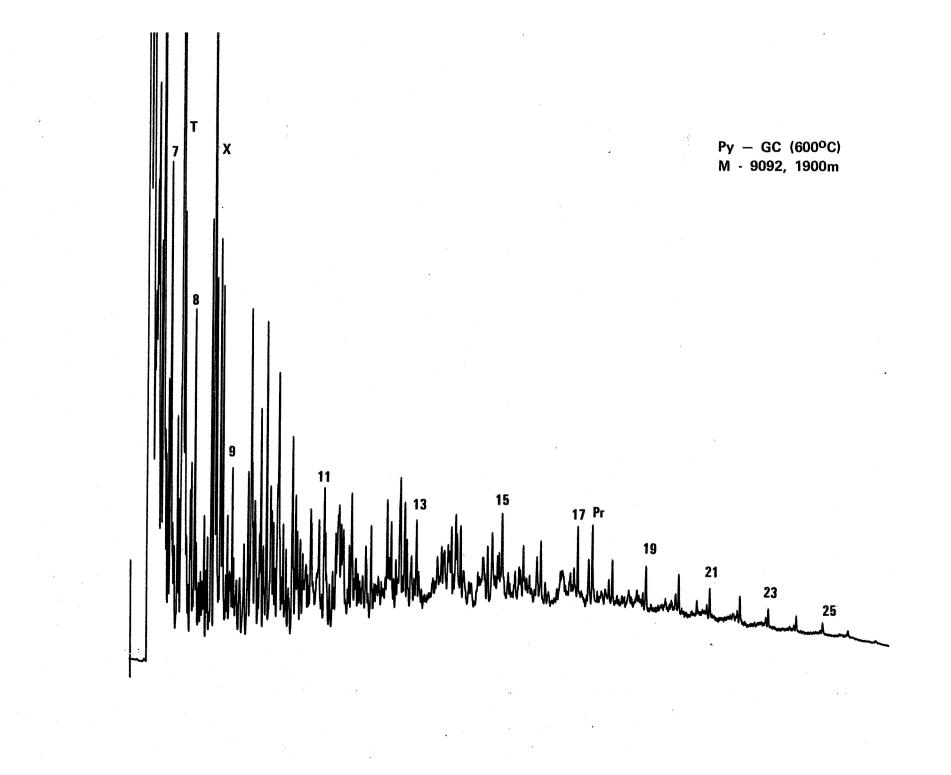
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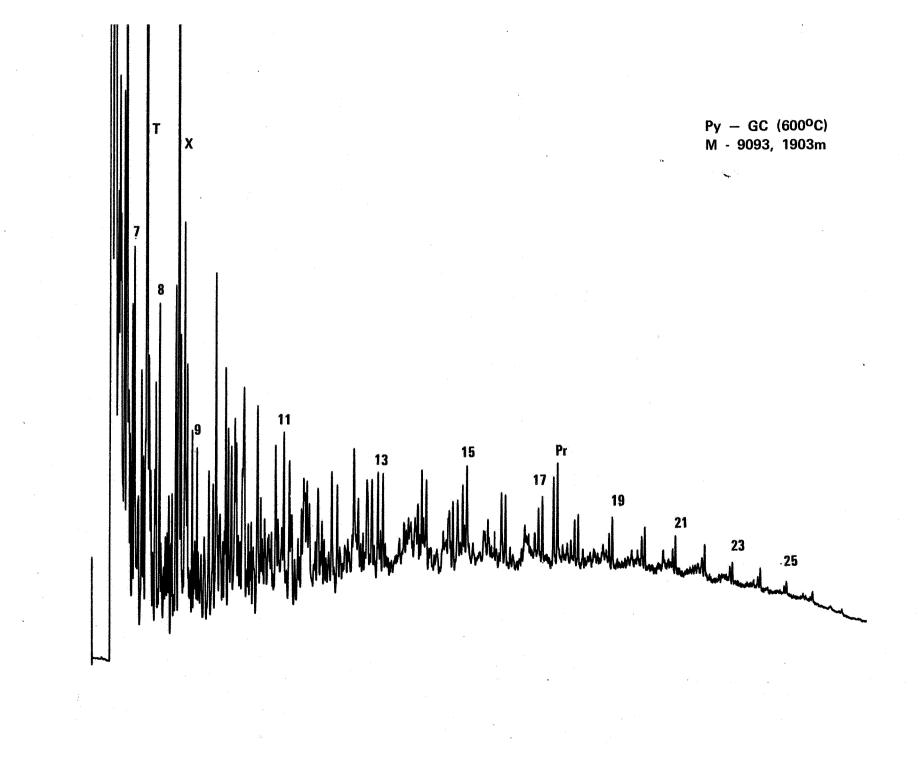
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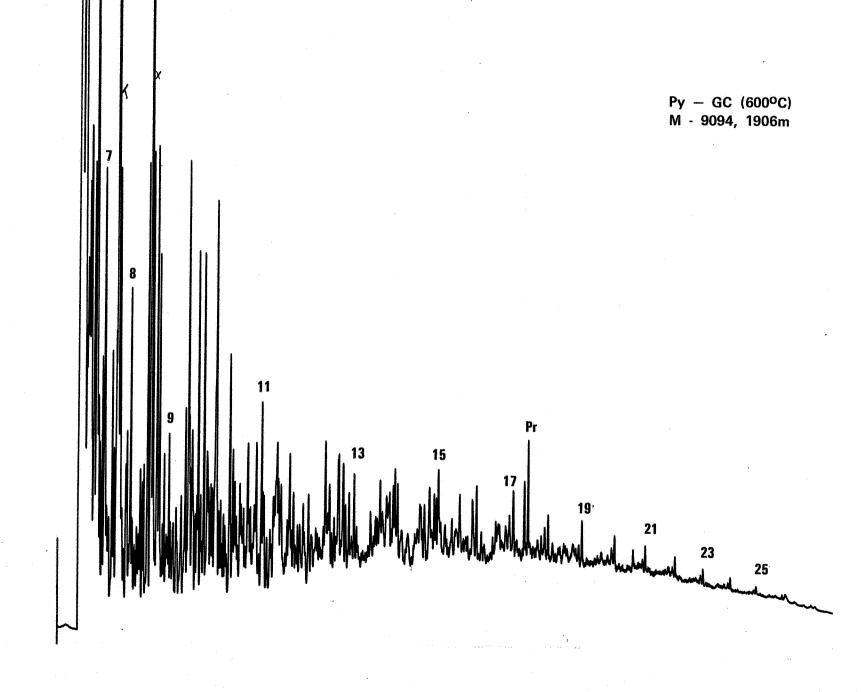


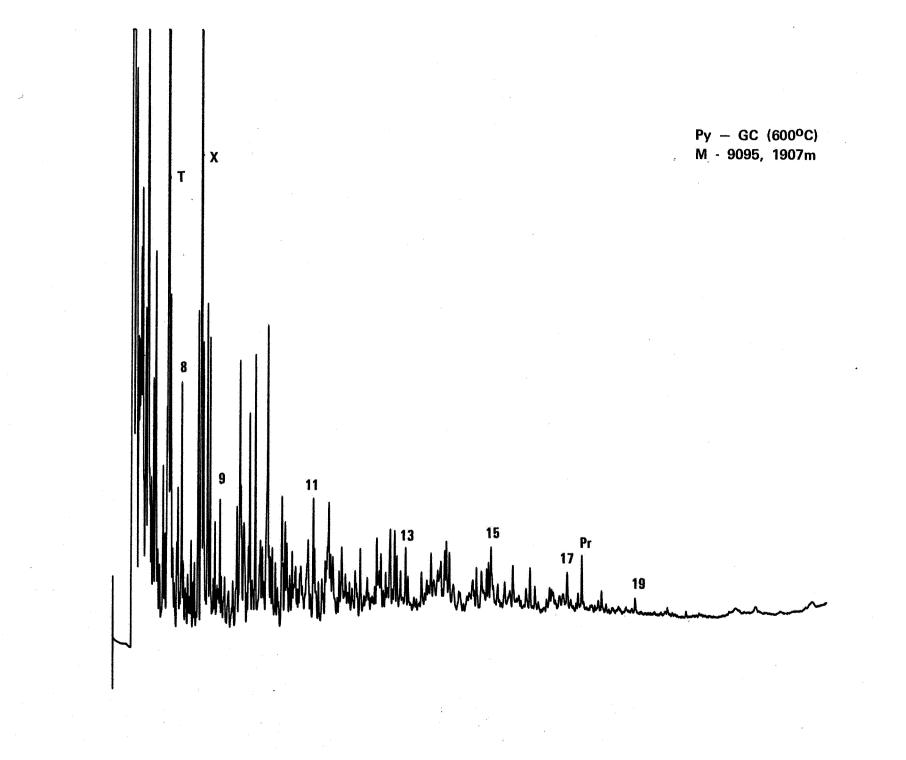


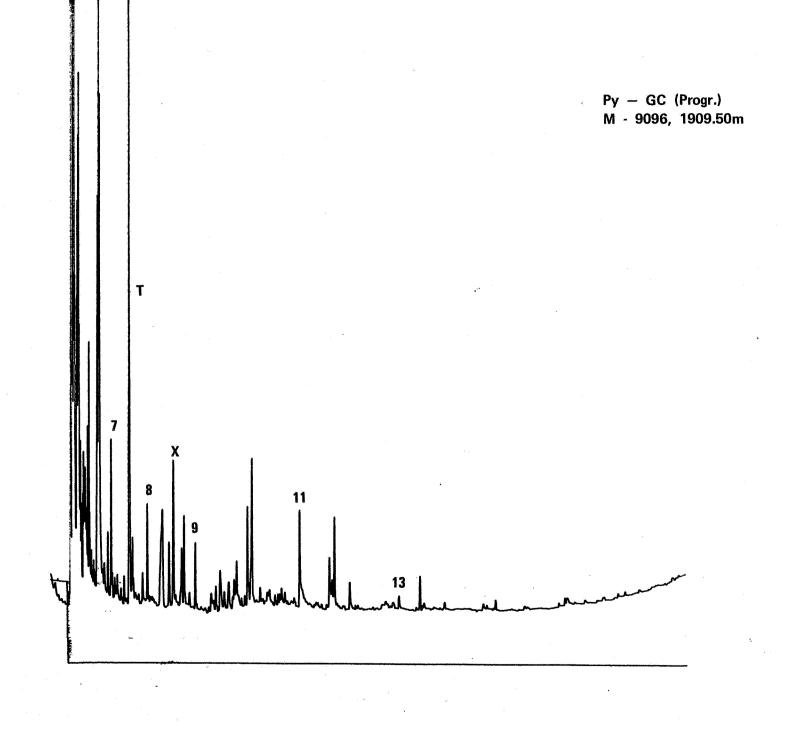


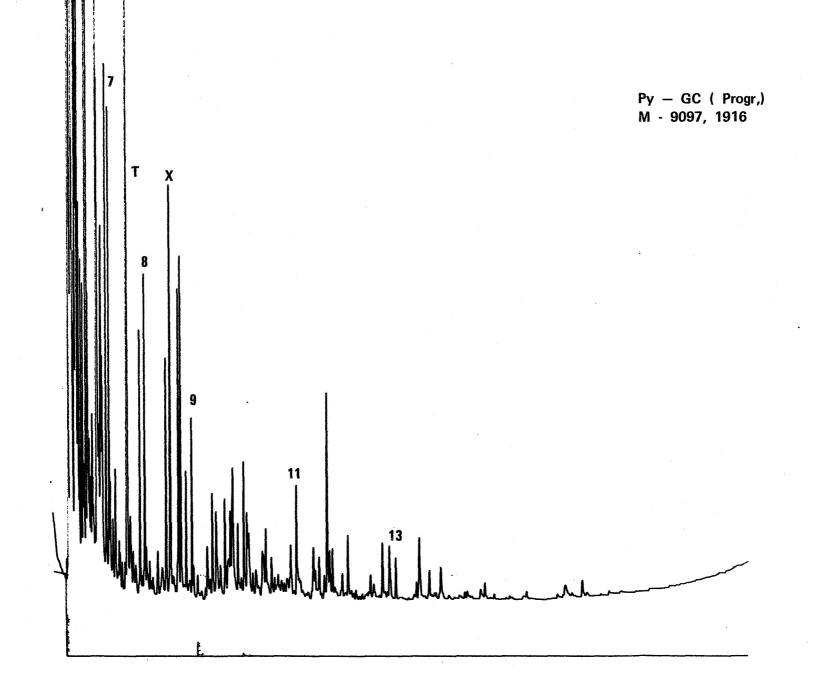












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