

**EUR 3287.e**

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

**THE RADIOLYTIC AND PYROLYTIC  
DECOMPOSITION OF ORGANIC COOLANTS**

**II. THE POSTPYROLYSIS OF PREIRRADIATED OM2**

by

**G. JUPPE, G. WEDEKIND, H. RAU and G. SCHÜTZ**

1967



**ORGEL Program  
Joint Nuclear Research Center  
Ispra Establishment - Italy  
Chemistry Department  
Organic Chemistry**

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The kinetics of the decompositions are described. The possible mechanisms responsible for the observed differences of reaction rates and product compositions are discussed.

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Published in the same series :

THE RADIOLYTIC AND PYROLYTIC DECOMPOSITION OF ORGANIC COOLANTS

- I. G. Juppe, M. Alvarenga and H. Hannaert ; The Pyrolytic Decomposition of Terphenyls ; EURATOM Report EUR 1647.e (1964).
- III. G. Juppe, H. Rau and F. Dorpema ; The Radiolysis and Pyrolysis of m-Terphenyl and m-Terphenyl-d<sub>11</sub> ; EURATOM Report EUR 3172.e (1967).
- V. G. Juppe and H. Rau ; The Thermal Decomposition of o-, m- and p-Terphenyl in the Presence of Attapulgius Clay ; EURATOM Report EUR 3281.e (1967).
- VII. G. Juppe and H. Rau ; The Pyrolysis of High Boiling Residue ; EURATOM Report EUR 3158.e (1966).

## SUMMARY

The postpyrolysis of in pile irradiated terphenyls (OM2) causes a higher rate of decomposition than the pyrolysis of unirradiated material. A higher formation of gaseous hydrocarbons, benzene, diphenyl and higher boiling aromatic compounds are also observed.

The kinetics of the decompositions are described. The possible mechanisms responsible for the observed differences of reaction rates and product compositions are discussed.

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## 1 - INTRODUCTION (\*)

In an ORGEL type reactor the organic coolant will be exposed to the simultaneous decomposing action of both irradiation and heat. For a thorough understanding of this overall decomposition process information about the quantitative effects of each of the two individual parameters - pyrolysis and radiolysis - has preferentially to be gained. This knowledge must, nevertheless, be supplemented by knowing how these two decomposing effects interact when simultaneously or consecutively applied.

In the present kinetic investigation the effect of postpyrolysis on preirradiated OM2 was studied.

## 2 - MATERIALS AND METHODS

### 2.1. MATERIALS

The virginal OM2 was a sample synthesized by Progil, France. The initial composition was 0,8% biphenyl, 23,2% o-terphenyl, 72,8% m-terphenyl and 3,6% p-terphenyl.

The samples of preirradiated OM2 had previously undergone a radiolysis in a loop experiment at the Mélusine Reactor at Grenoble. The sample had received as total dose 1,85 Wh/g, the temperature was 200°C. The composition of the preirradiated sample was 0,4% low boilers (benzene, toluene, xylene), 0,4% biphenyl, 19,4% o-terphenyl, 59,6% m-terphenyl and 3,8% p-terphenyl.

### 2.2. PYROLYSIS

Preparation and pyrolysis of the samples were carried out as previously described<sup>1)</sup>.

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<sup>1)</sup> G. Juppe, M. Alvarenga and H. Hannaert, EURATOM-Report EUR 1647.e  
(\*) Manuscript received on December 23, 1966.

Two runs of experiments were made for both preirradiated and virginal OM2. The chosen temperatures of 460°C and 480°C measured by thermoresistance were actually 1,3° lower due to the resistance of the connecting wires between the oven thermometer and the measuring device. The recorded temperature did not fluctuate more than  $\pm 1^\circ\text{C}$  around the set point for the majority of the runs. It proved not to be necessary to correct for a few short-time-fluctuations of up to 3°C.

### 2.3. ANALYSIS

#### 2.3.1. GASEOUS PRODUCTS

The determination of the total gas was performed in a conventional gas line as previously described<sup>1)</sup>. Benzene and low boilers had been separated before condensing them in a carbon dioxide / acetone mixture. The mass spectroscopic analysis of hydrogen, methane, ethane, ethylene, acetylene, propene, propylene, n-butane, i-butane, isobutylene, n-pentane and i-pentane were performed on an Atlas CH<sub>4</sub> spectrometer.

#### 2.3.2. LIQUID PRODUCTS

The gas chromatographic analysis of low boilers (benzene, toluene, xylene etc.), diphenyl, the three terphenyl isomers, the quaterphenyl isomers and the high boiling products formed were carried out by injecting the trichlorobenzene solutions containing an inner standard. A 2,50 m CsCl-column was used. The raise in temperature over a 150°C range was maintained at 20°C per second.

### 3 - RESULTS

#### 3.1. FORMATION OF GASES

A comparison of the total gas formation during the pyrolysis of virginal and of preirradiated OM2 is given in fig. 1. The rates of the formation of the respective individual gaseous components hydrogen, methane and ethane are listed in fig. 2.

The changes in total gas production during the pyrolysis of virginal and preirradiated OM2 are listed in the following table 1.

TABLE 1 - TOTAL GAS FORMATION AT 480°C (ml g<sup>-1</sup>h<sup>-1</sup>)

HOURS	OM2 VIRGINAL	OM2 PREIRRADIATED
0 - 1	0,13	4,2
1 - 2	0,13	1,75
2 - 3	0,13	1,4
3 - 4	0,13	0,8
4 - 5	0,13	0,5

The composition of the gas during the pyrolysis of virginal and preirradiated OM2 is listed in the following table 2.

TABLE 2 - COMPOSITION OF GAS (% corr.)

	OM2 VIRGINAL				OM2 PREIRRADIATED			
	1		5		1		5	
PYROLYSIS TIME (h)	1		5		1		5	
TEMPERATURE (°C)	460	480	460	480	460	480	460	480
HYDROGEN	88	91	89	90	23	26	26	43
METHANE	9	7	8	7	50	47	48	42
ETHANE	2	1	2	1,5	18	18	16	12
HIGHER HYDROCARBONS	1	1	1	1,5	9	8	10	3

3.2. FORMATION OF LIQUID PRODUCTS

A comparison of the experimental data about the formation of low boilers (benzene, toluene, xylene etc.) and diphenyl is graphically illustrated in fig. 3 and 4. The rates of the observed o- and m-decrease during the pyrolysis of virginal and preirradiated OM2 are listed in fig. 5 and 6. The kinetics of the formation of m, m-quaterphenyl, the total of quaterphenyls, quinquaphenyls and higher boiling compounds and the sum of intermediate and high boilers are compared in fig. 7 to 10.

On the basis of a zero order reaction for the formation of low boilers (benzene, toluene, xylene etc.) and diphenyl during the pyrolysis of virginal and preirradiated OM2 the rate constants listed in table 3 have been calculated.

TABLE 3 - RATE CONSTANTS ( $10^{-4}$  mole l<sup>-1</sup>h<sup>-1</sup>)

	VIRGINAL OM2		PREIRRADIATED OM2	
	460°	480°	460°	480°
LOW BOILERS (benzene, toluene, xylene etc.)	69	179	155	407
DIPHENYL	86	248	122	348

On the basis of a first order reaction for the change in o-, m-, p-terphenyl, m, m-quaterphenyl, quinquaphenyls and higher boiling compounds and the sum of intermediate and high boilers during the pyrolysis of virginal and preirradiated OM2 the rate constants listed in table 4 have been calculated. For the decomposition of the preirradiated OM2 a k-value for the initial pyrolysis representing only the first part of the reaction up to 18% decrease of the original terphenyl and a k-value representing the following course of the reaction have been calculated.

TABLE 4 - RATE CONSTANTS OF FIRST ORDER REACTIONS ( $10^{-3}h^{-1}$ )

	VIRGINAL OM2		PREIRRADIATED OM2 <sup>a)</sup>		PREIRRADIATED OM2 <sup>b)</sup>	
	460°	480°	460°	480°	460°	480°
o-TERPHENYL	16	46	62	134	32	96
m-TERPHENYL	15	51	92	260	20	61
p-TERPHENYL	-	-	102	321	22	62
m, m-QUATERPHENYL	-	-	1,0	3,25	-	-
QU INQUAPHENYL PLUS HIGHER BOILING COM- POUNDS	51	87	-	-	18	62
INTERMEDIATE PLUS HIGH BOILERS	17	86	220	603	26	75

a) pyrolysis up to 18% decrease in original o-, m-, plus p-terphenyl

b) pyrolysis after 18% decrease in original o-, m-, plus p-terphenyl

#### 4 - DISCUSSION

##### 4.1. FORMATION OF GASES

A comparison of the formation of gases originating from virginal and from preirradiated OM2 shows the following:

#### 4.1.1. TOTAL GAS

Compared with the pyrolysis of virginal OM2 a 32 fold gas production is observed when pyrolysing preirradiated OM2 at 480°C for 1 hour, corresponding to the simultaneous disappearance of 18% of the original o-, m-, p-terphenyls. Parallel results are obtained at 460°C.

The excess in total gas production decreases with pyrolysis time: At 480°C and after 5 hours, corresponding to the disappearance of 32% of the original o-, m-, p-terphenyls, only four times more gas is produced by preirradiated OM2. Equivalent results are obtained at 460°C.

#### 4.1.2. HYDROGEN

After 1 hour (18% decrease in original terphenyls) and at 460°C (480°C) pyrolysis temperature only 23% (26%) of the total gas formed from preirradiated OM2 consists of hydrogen. 88% resp. 91% of hydrogen is formed when virginal OM2 is pyrolyzed on the same conditions.

The amount of hydrogen is increasing with pyrolysis time: After 5 hours (32% decrease in original terphenyls) and at 460°C (480°C) pyrolysis temperature 25% (43%) of the total gas formed from preirradiated OM2 consists of hydrogen.

#### 4.1.3. HYDROCARBONS

After 1 hour (18% decrease in original o-, m-, p-terphenyl) and at 460°C (480°C) pyrolysis temperature 50% (47%) of the total gas formed from preirradiated OM2 consists of methane. Only 9% (7%) of methane is formed when virginal OM2 is pyrolyzed 1 hour at 460°C (480°C).

The amount of methane from preirradiated OM2 is decreasing with pyrolysis time: After 5 hours (32% decrease in original terphenyls) and at 460°C (480°C) pyrolysis temperature only 48% (42%) of the total gas formed from preirradiated OM2 consists of methane.

After 1 hour and at 460°C (480°C) pyrolysis temperature 18% (18%) of the total gas formed from preirradiated OM2 consists of ethane. Only 2% (1%) of ethane is formed when virginal OM2 is pyrolyzed on identical reaction conditions.

The amount of ethane from preirradiated OM2 is decreasing with pyrolysis time: After 5 hours and at 460°C (480°C) pyrolysis temperature only 16% (12%) of the total gas formed from preirradiated OM2 consists of ethane.

After 1 hour and at 460°C (480°C) pyrolysis temperature 9% (8%) of the total gas formed from preirradiated OM2 consists of C<sub>3</sub>- to C<sub>5</sub>-hydrocarbons, either saturated or unsaturated. Only 1% (1%) of these compounds are formed when virginal OM2 is pyrolyzed.

#### 4.2. FORMATION OF LIQUID PRODUCTS

A comparison of the formation of liquid products originating from virginal and from preirradiated OM2 shows the following:

##### 4.2.1. LOW BOILERS<sup>2)</sup> AND DIPHENYL

The formation of low boilers and of diphenyl from virginal and from preirradiated OM2 can in both cases be described as a zero order reaction. Even at high conversion rates corresponding to the simultaneous disappearance of 58% (32%) of the original o-, m-, p-terphenyls of virginal (preirradiated) OM2 the reaction remains reasonably well concentration independent.

Temperature increase from 460°C to 480°C results in a 2,8 fold increased rate for the production of low boilers and a 2,6 fold increased rate for the production of diphenyl. This is found for both virginal and preirradiated OM2.

A molar ratio of 1,6 (1,5) for the formation of low boilers/diphenyl is observed when virginal OM2 is pyrolyzed at 460°C (480°C). The relative amount of low boilers increases when not

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<sup>2)</sup> The low boiling fraction consists mainly of benzene with a small participation of its higher homologues as toluene and the xylenes. There exists mass spectroscopic evidence that the content of the latter compounds is somewhat higher after pyrolysis of preirradiated OM2.

virginal but preirradiated OM2 is thermally decomposed: 2,5 (2,3) times more low boiling compounds are formed at 460°C (480°C) than diphenyl molecules<sup>3)</sup>

Compared with the pyrolysis of virginal OM2 a 2,6 fold increase in the formation of low boilers is observed when pyrolyzing preirradiated OM2 at both 460°C and 480°C. 1,4 times more diphenyl is formed from the preirradiated starting material and this is also valid for both temperatures.

#### 4.2.2. o-, m-, p-TERPHENYLS

The disappearance of the initial o-, m-, and p-terphenyls originating from virginal OM2 can be reasonably well described as a first order reaction.

The disappearance of the initial o-, m-, and p-terphenyls originating from preirradiated OM2 has a considerably augmented rate of disappearance at the beginning of the reaction, approaching the rate of decrease of o-, m-, and p-terphenyl from virginal OM2 only at higher conversion rates: At 460°C (480°C) and during the first 3 (1) hours 5,8 (5,6) times more m-terphenyl disappears when the preirradiated starting mixture is thermally decomposed. After 15 (5) hours only 1,2 (1,3) more m-terphenyl of the virginal preirradiated OM2 is decomposed when the pyrolysis of preirradiated and virginal OM2 is compared at 460°C and 480°C (see fig. II). A similar relationship is observed for the disappearance of o-terphenyl.

Temperature increase from 460°C to 480°C results in a 2,9-3,0 fold decrease in o- and m-terphenyl regardless whether virginal or preirradiated OM2 is pyrolyzed. The same value is found for both the beginning and the advanced thermal decomposition of preirradiated material indicating that very similar values for

<sup>3)</sup>

As molecular weight for the low boiling fraction a value of 78 (C<sub>6</sub>H<sub>6</sub>) was chosen. The above mentioned factor of 2,5 (2,3) would decrease if corrected for the higher molecular weight benzene homologues (toluene, xylene etc.) contained in the low boiling fraction.



activation energies are to be expected. The only two chosen temperatures did not allow an exact determination of the activation energies involved.

#### 4.2.3. QUATERPHENYLS

A major portion of the pyrolysis products of terphenyl consists of quaterphenyls.

The formation of the sum of quaterphenyls originating from the pyrolysis of virginal and preirradiated OM2 can in both cases be described as a first order reaction. During the thermal decomposition of preirradiated OM2 higher rate constants are observed at the beginning of the reaction which become smaller with advanced pyrolysis.

After 5 hours and at 460°C 35% (6%) of the total high boiler fraction<sup>4)</sup> formed during the pyrolysis of virginal OM2 consists of quaterphenyls (m, m-quaterphenyl). After 5 hours and at 460°C 36% (3%) of the additional high boilers formed consist of quaterphenyls (m, m-quaterphenyl). A similar relation is observed at 480°C.

#### 4.2.4. QUINQUAPHENYLS AND HIGHER BOILING COMPOUNDS

Also the formation of the sum of quinquaphenyls and higher boiling compounds originating from the pyrolytic decomposition of virginal OM2 can be reasonably well described as a first order reaction.

The formation of the sum of quinquaphenyls and higher boiling compounds from preirradiated OM2 follows a similar rule as the disappearance of original o-, m-, p-terphenyl:

The considerably increased rate of formation at the beginning of the reaction is decreasing with advanced pyrolysis time. At 460°C (480°C) and during the first 3 (1) hours 13 (7) times more quinquaphenyls and higher boiling compounds are formed from preirradiated OM2 than from virginal OM2. After 15 hours only 1,5 times

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<sup>4)</sup> compounds with a molecular weight higher than terphenyl.

more quinquaphenyls and higher boiling compounds from the original preirradiated OM2 are formed at 460°C when compared to the pyrolysis of virginal OM2 at identical reaction conditions (see fig. 12).

Temperature increase from 460°C to 480°C results in a 5 fold increase in k-values calculated for high boiler production during the pyrolysis of virginal OM2. Only a 3 fold increase is observed for both the initial and the advanced pyrolysis of preirradiated OM2. This indicates that a lower activation energy is expected for the high boiler formation from preirradiated OM2 than from virginal OM2. The only two chosen temperatures did not allow an exact determination of the activation energies involved.

## 5 - CONCLUSIONS

The above mentioned differences in reaction rates, kind and composition of reaction products observed during the thermal decomposition of virginal and preirradiated OM2 can be explained on the following basis:

The intermediate and high boiler fraction of the preirradiated OM2 may contain hydrogenated and alkylated aromatic systems originating from previous radiolysis. The bond dissociation energies of the CC- and CH-bonds of aliphatic molecule arrangements are 10-20 kcal/mole lower than the CC- and CH-bond strengths appearing in the aromatic molecules, e.g. in bi- and terphenyls of virginal OM2 (table 5). Two competing reactions differing in their activation energies by only 10 kcal/mole will show a rate ratio of appr. 1:500, i.e. only the reaction with the lower dissociation energy for the rupture of the bond will appear to an appreciable extent.

TABLE 5 - BOND DISSOCIATION ENERGIES

COMPOUND	C <sub>6</sub> H <sub>5</sub> -C <sub>6</sub> H <sub>5</sub>	C <sub>6</sub> H <sub>5</sub> -H	CH <sub>3</sub> -CH <sub>3</sub>	C <sub>6</sub> H <sub>5</sub> -CH <sub>3</sub>	C <sub>6</sub> H <sub>5</sub> CH <sub>2</sub> -H
BOND STRENGTH	98 <sup>5)</sup>	102 <sup>6)</sup>	83 <sup>7)</sup>	89 <sup>8)</sup>	77 <sup>9)</sup>

The decomposition of terphenyls of virginial OM2 leads to the formation of phenyl-, diphenyl- and hydrogen radicals only<sup>1)</sup>. The pyrolysis of hydrogenated and alkylated aromatic systems would result in additional production of hydrogen and alkyl radicals. The rate of formation of the latter radicals would be considerably higher than the radical production from aromatic systems due to the lower bond dissociation energies involved for bond ruptures.

This increased rate of total radical production is only to be expected at the beginning of the thermal decomposition: The reactive species being initially produced from hydrogenated and alkylated aromatic systems undergo the same consecutive reaction steps with aromatic solvent molecules as previously described<sup>1)</sup>, leading finally by termination processes to the increased formation of molecular hydrogen, hydrocarbons and aromatic di- and terphenyl decomposition products. After the completion of decomposition of hydrogenated and alkylated systems contained in preirradiated OM2 the normal and slower pyrolysis of the pure aromatic compounds (di- and terphenyls) takes over.

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The presence of resonance stabilized high molecular weight radicals originating from previous radiolysis of virgin OM2 has to be discussed as in addition being responsible for the higher initial decomposition of preirradiated OM2 on consecutive pyrolysis.

Informative measurements done at this laboratory showed that radicals are present in the high boiler fraction of preirradiated OM2. These species being stable at the temperature of their formation by radiolysis could decompose to highly reactive radicals at a higher temperature. These highly reactive radicals could alone or together with the hydrogen, phenyl- and diphenyl radicals, formed by normal pyrolysis of aromatic compounds, count for an increased decomposition of preirradiated OM2 at the time of their formation, i.e. the beginning of pyrolysis following the reaction mechanism previously explained. The high formation of hydrocarbons from preirradiated OM2 can, nevertheless, not satisfactorily be explained in this way.

Experiments in progress may differentiate which of the two above mentioned reasons are to which extent responsible for the different thermal decomposition of preirradiated and original OM2.

#### ACKNOWLEDGEMENT

The authors take pleasure in acknowledging the work of Dr. S. Facchetti, Dr. B. Versino, Mr. S. Marell and Mr. H. Vissers for determining the analytic results. We also thank Dr. H. Hannaert for valuable discussions.

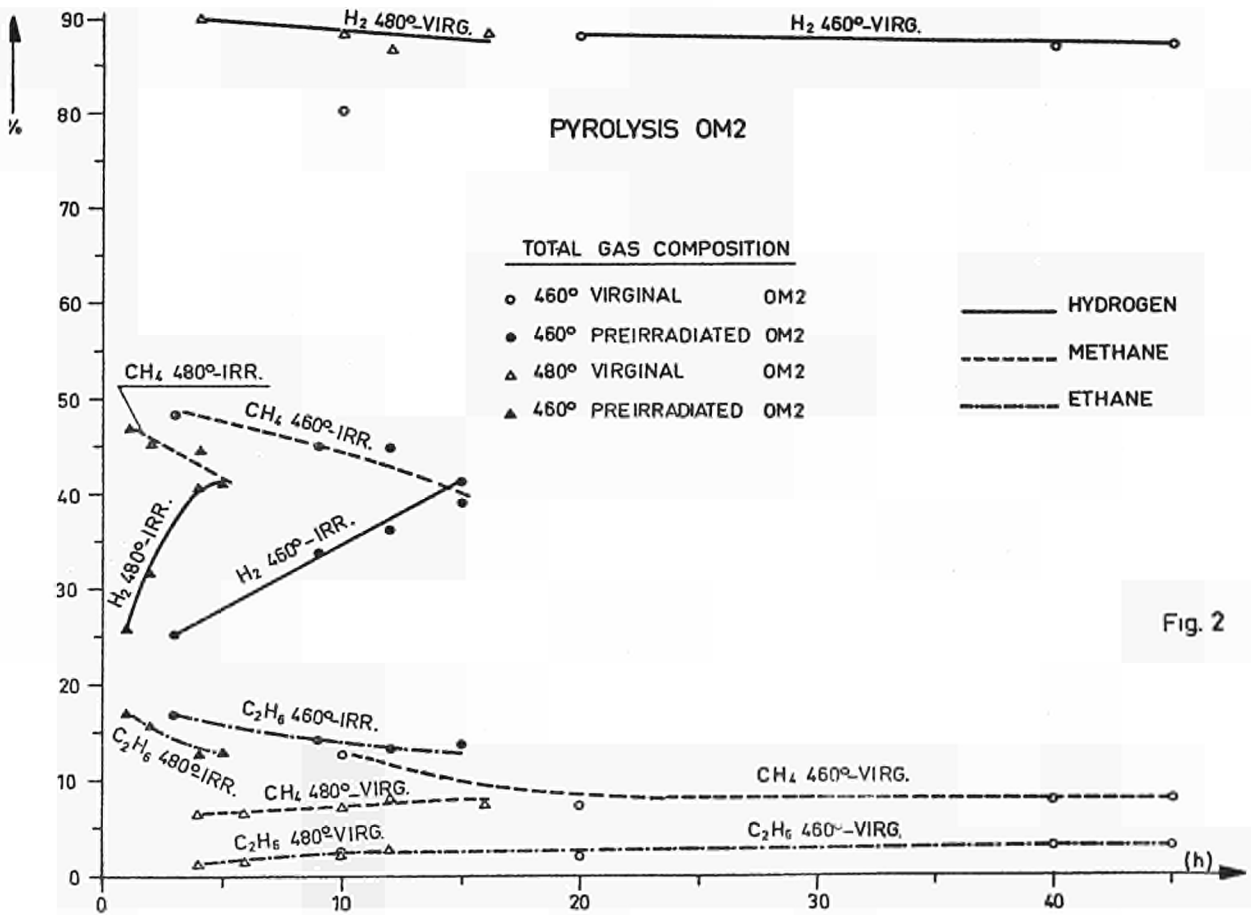
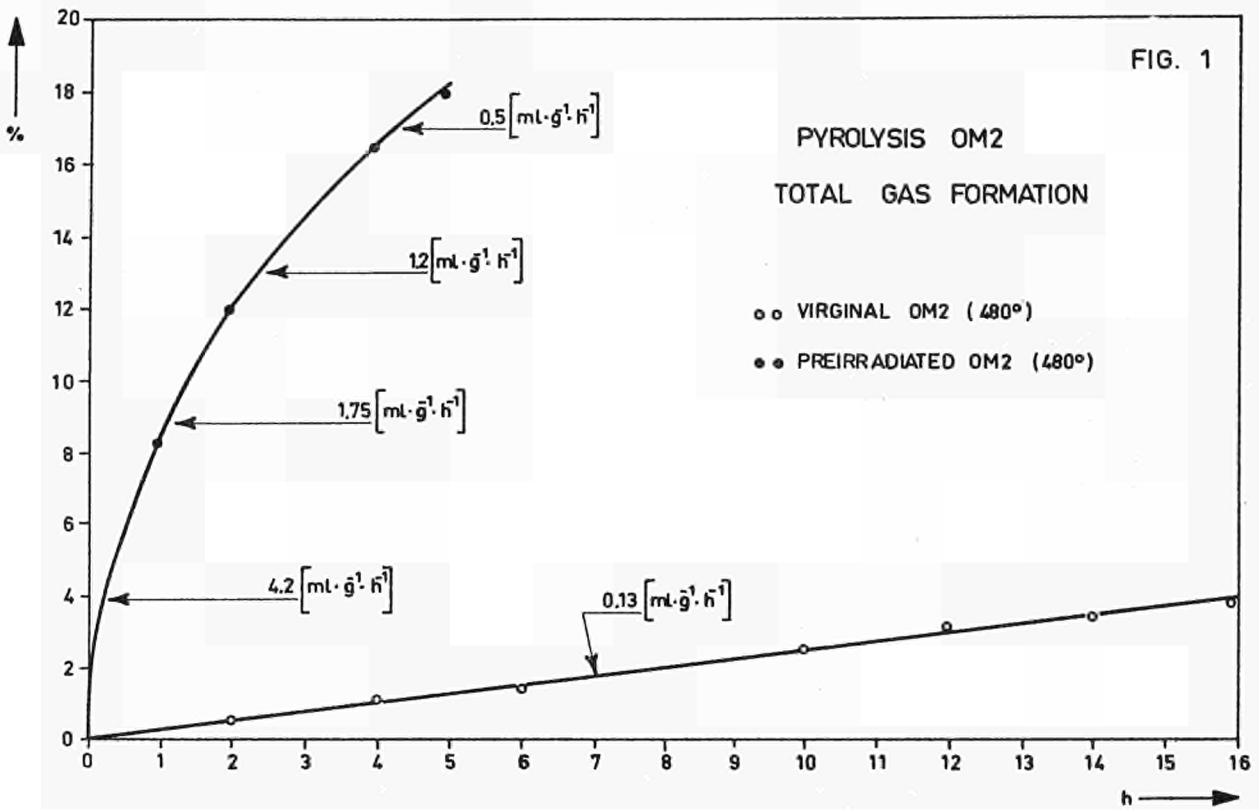
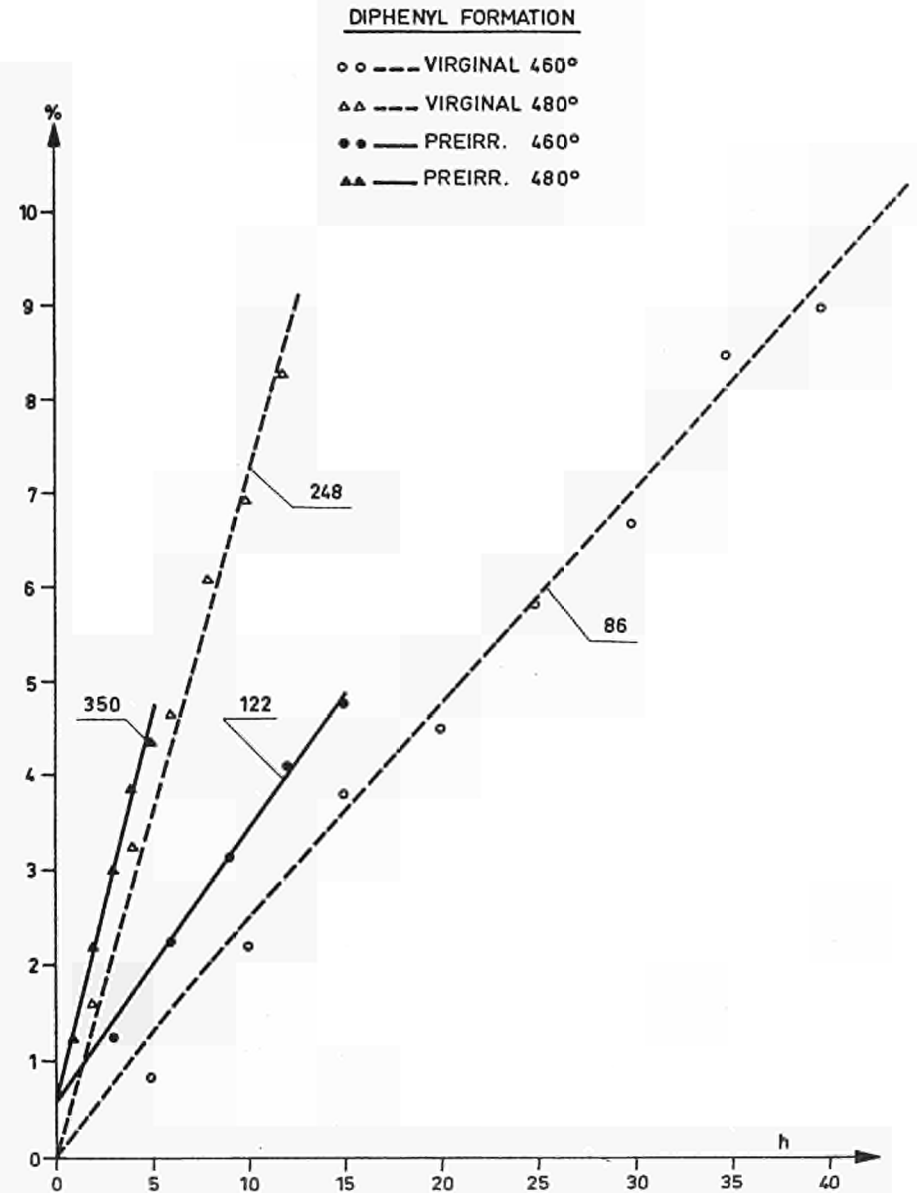
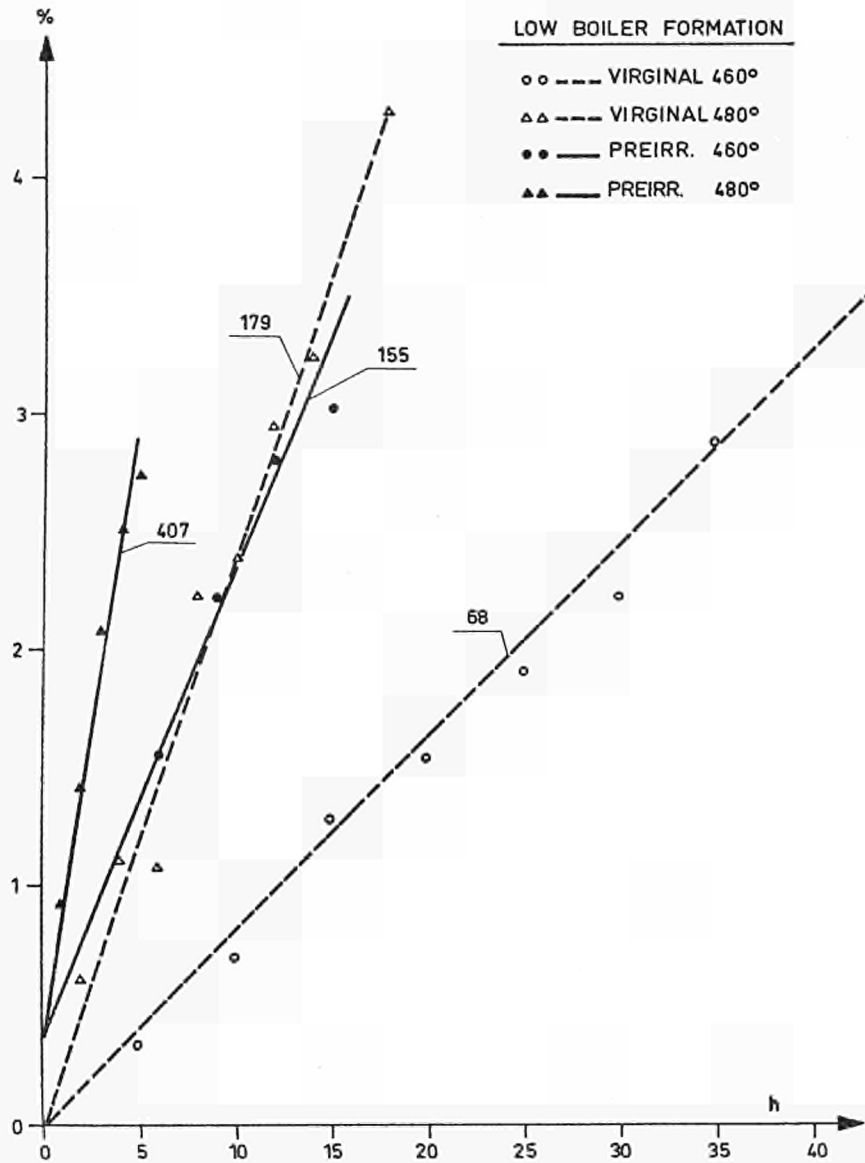
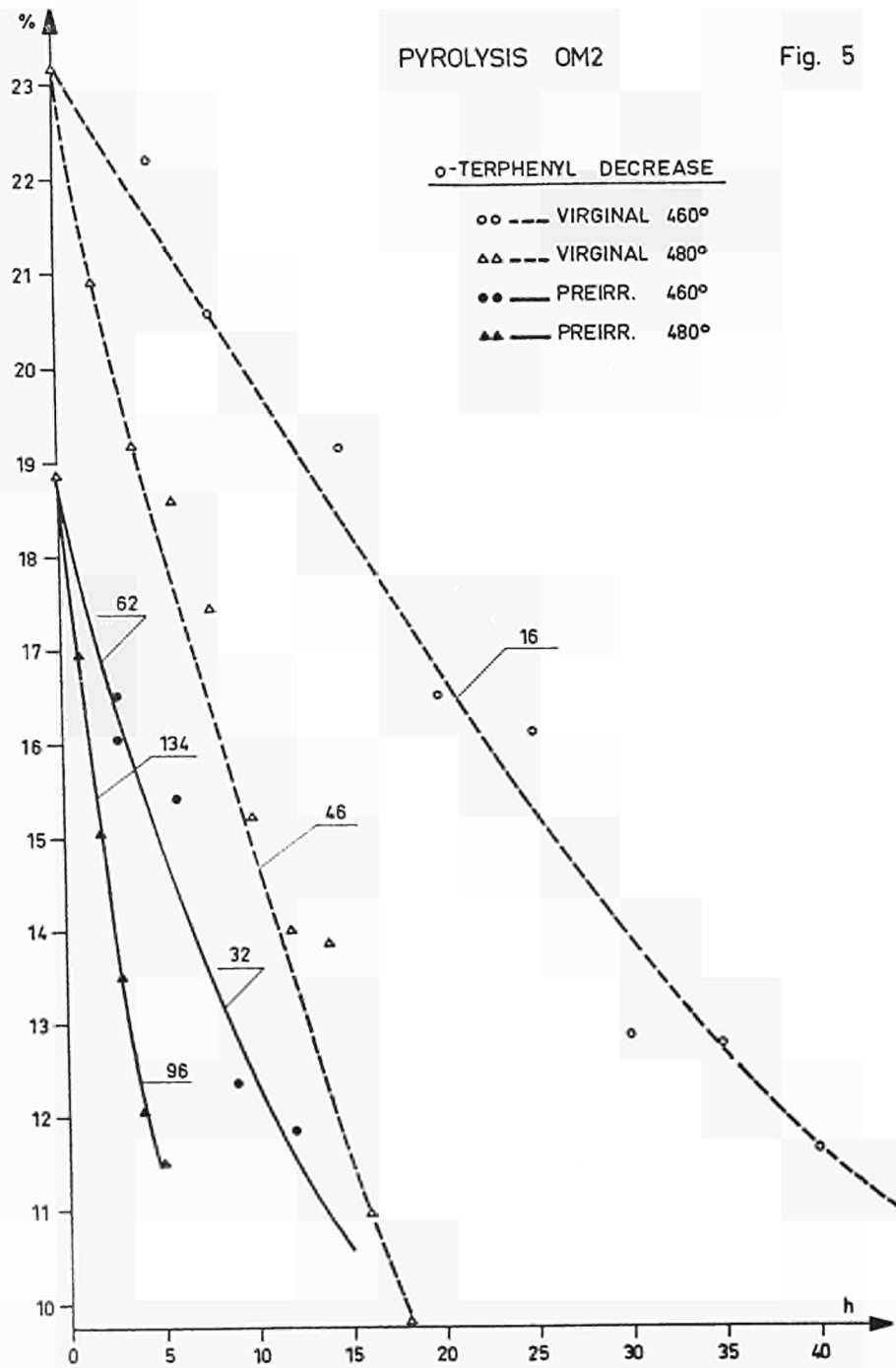


Fig. 2



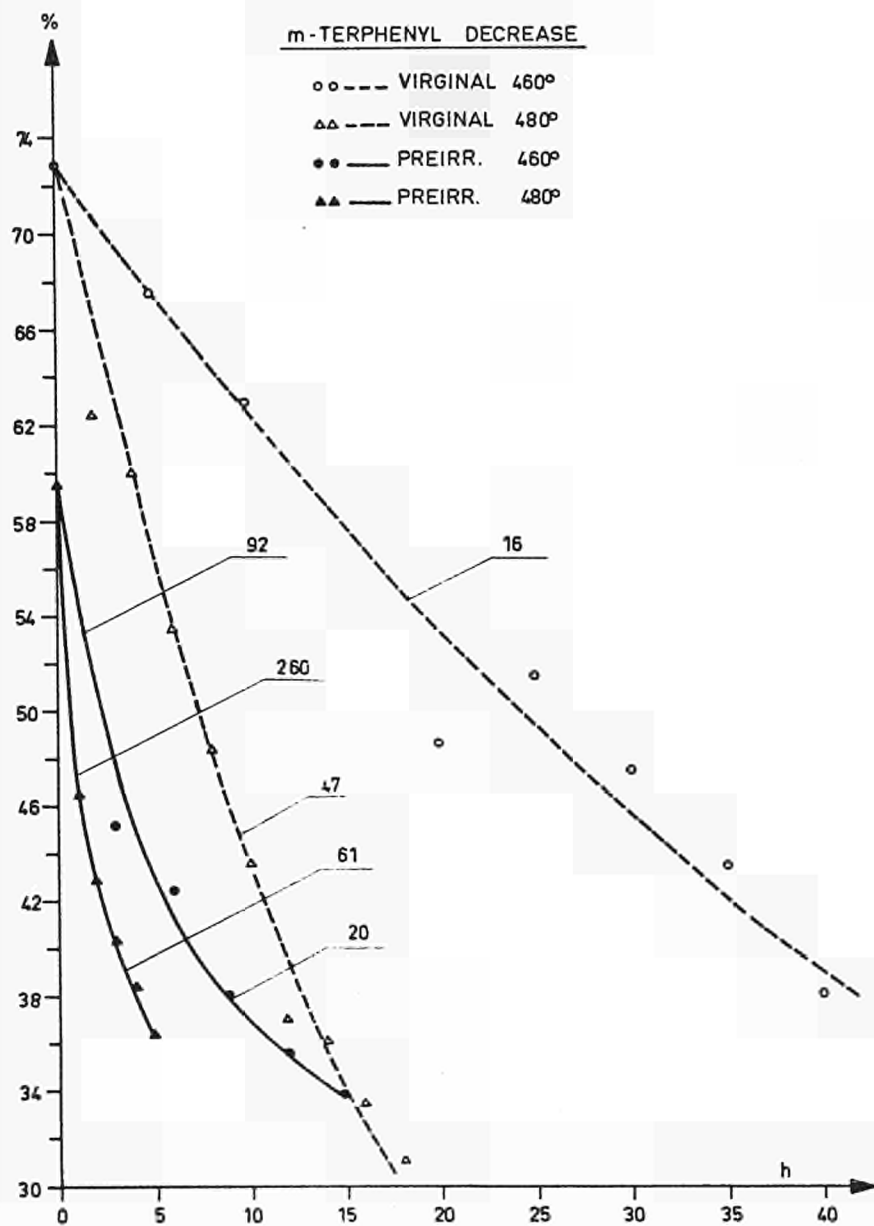
PYROLYSIS OM2

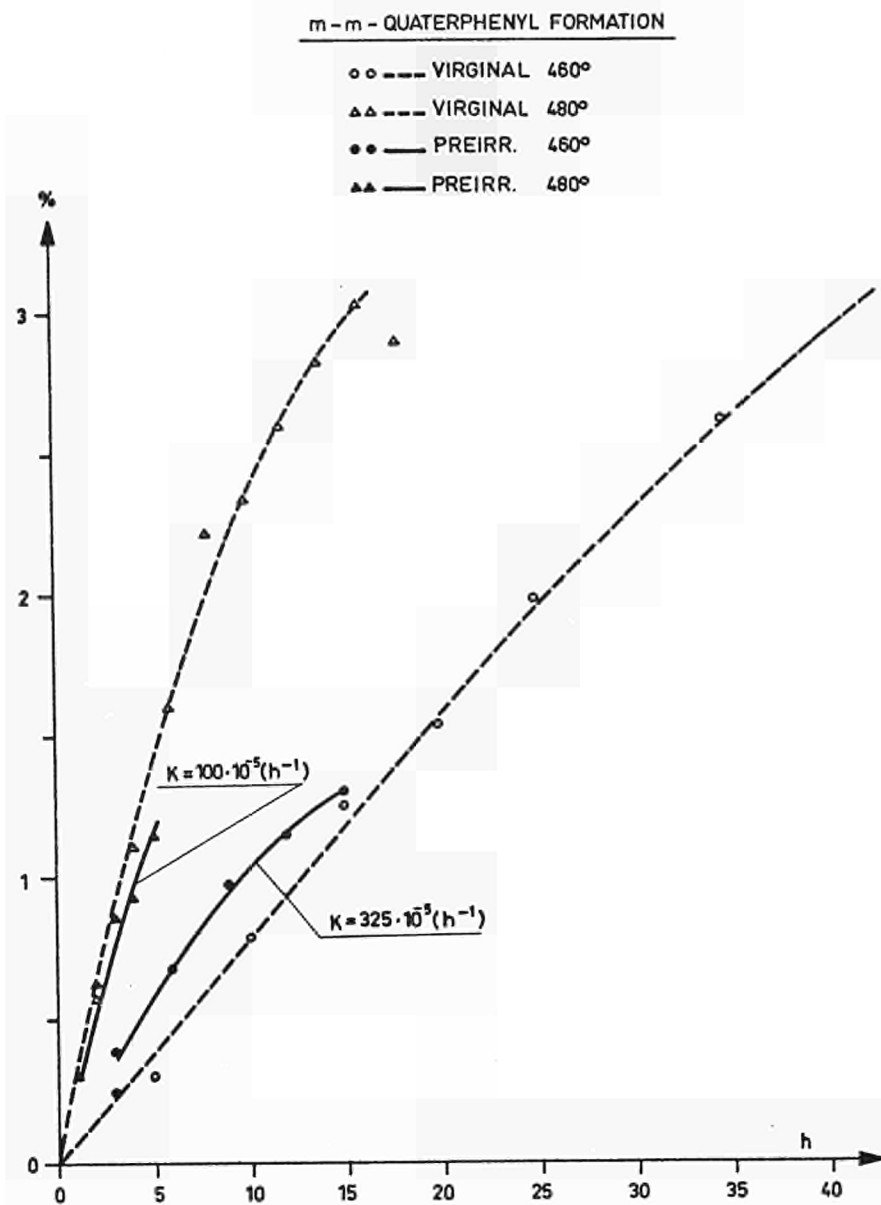
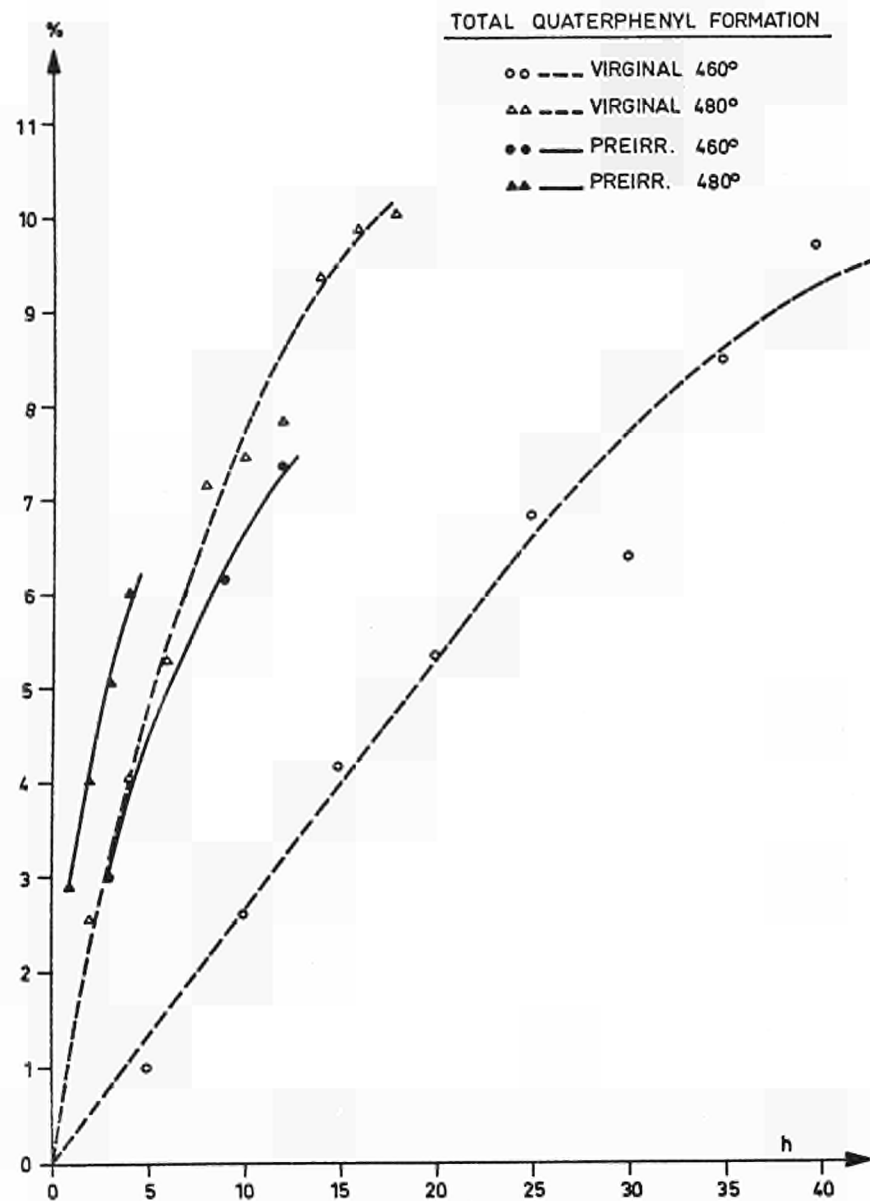
Fig. 5



PYROLYSIS OM2

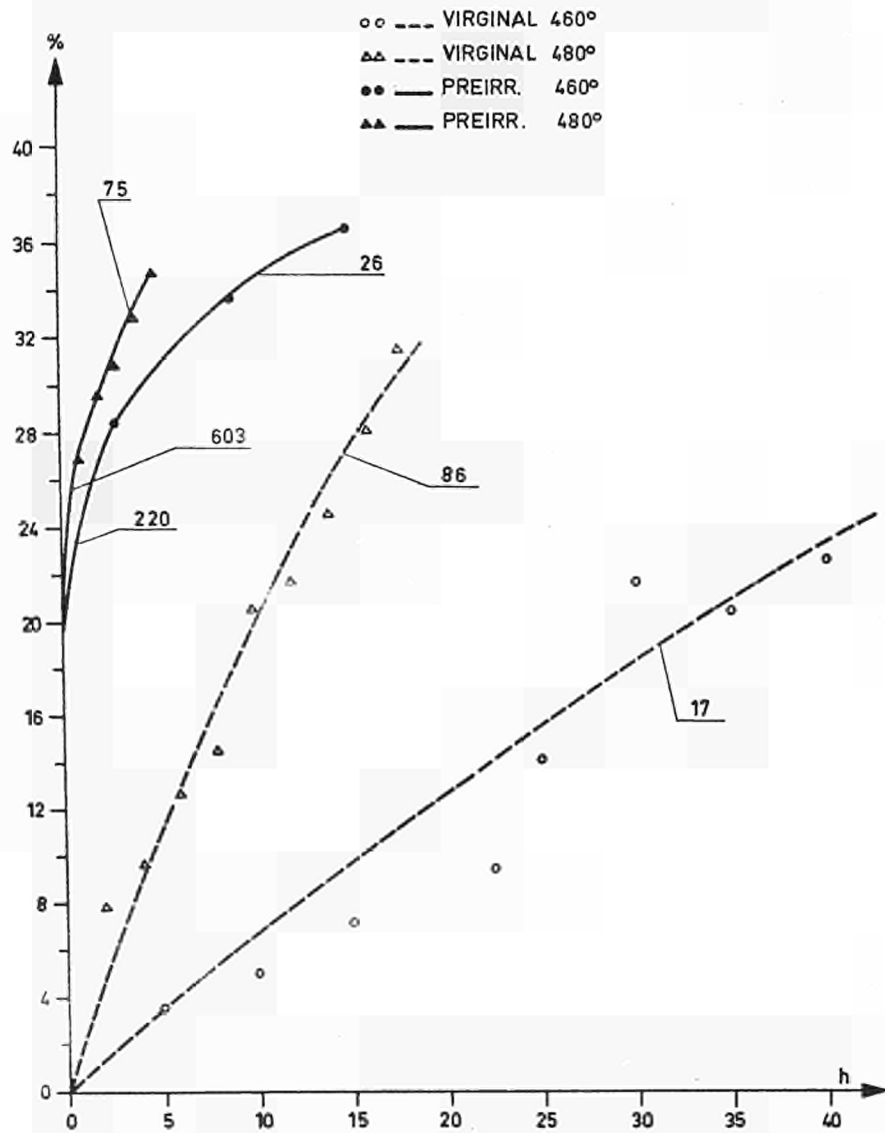
Fig. 6



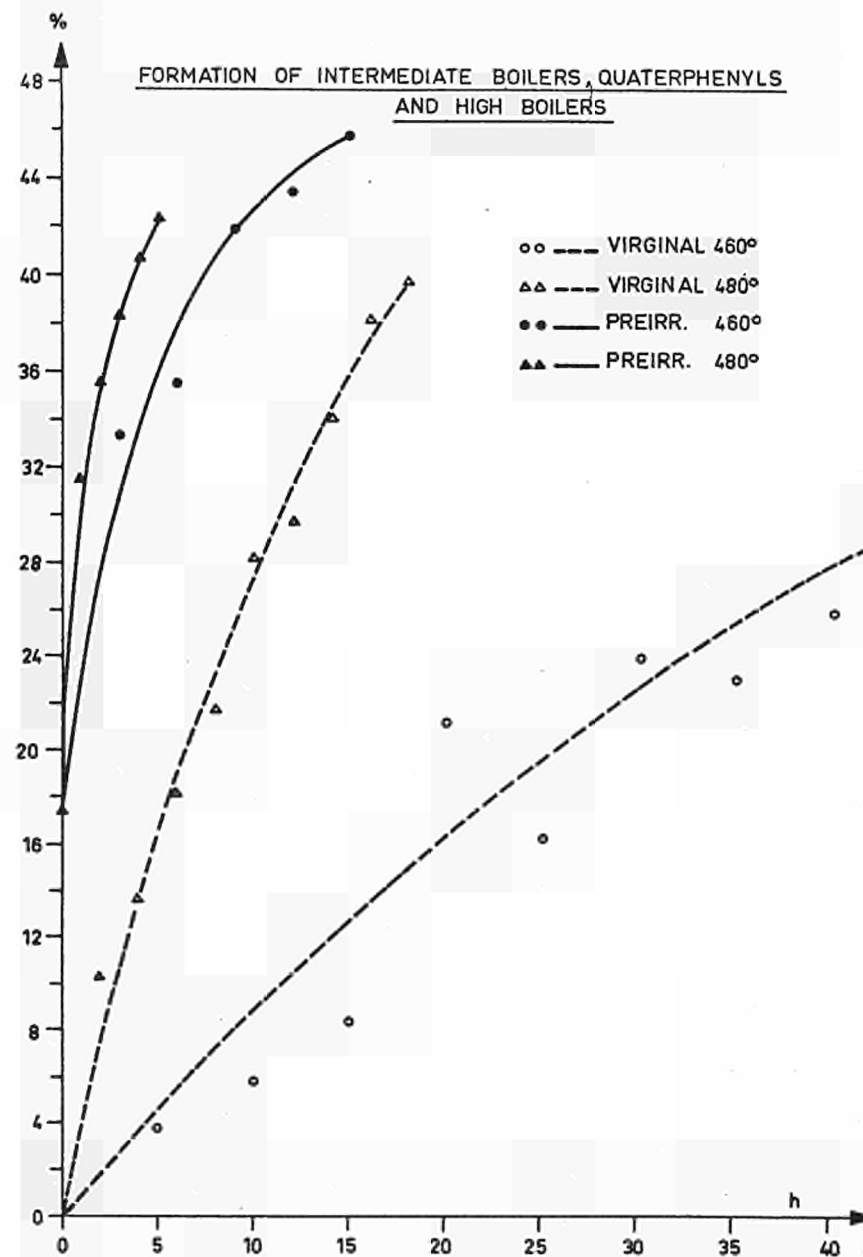


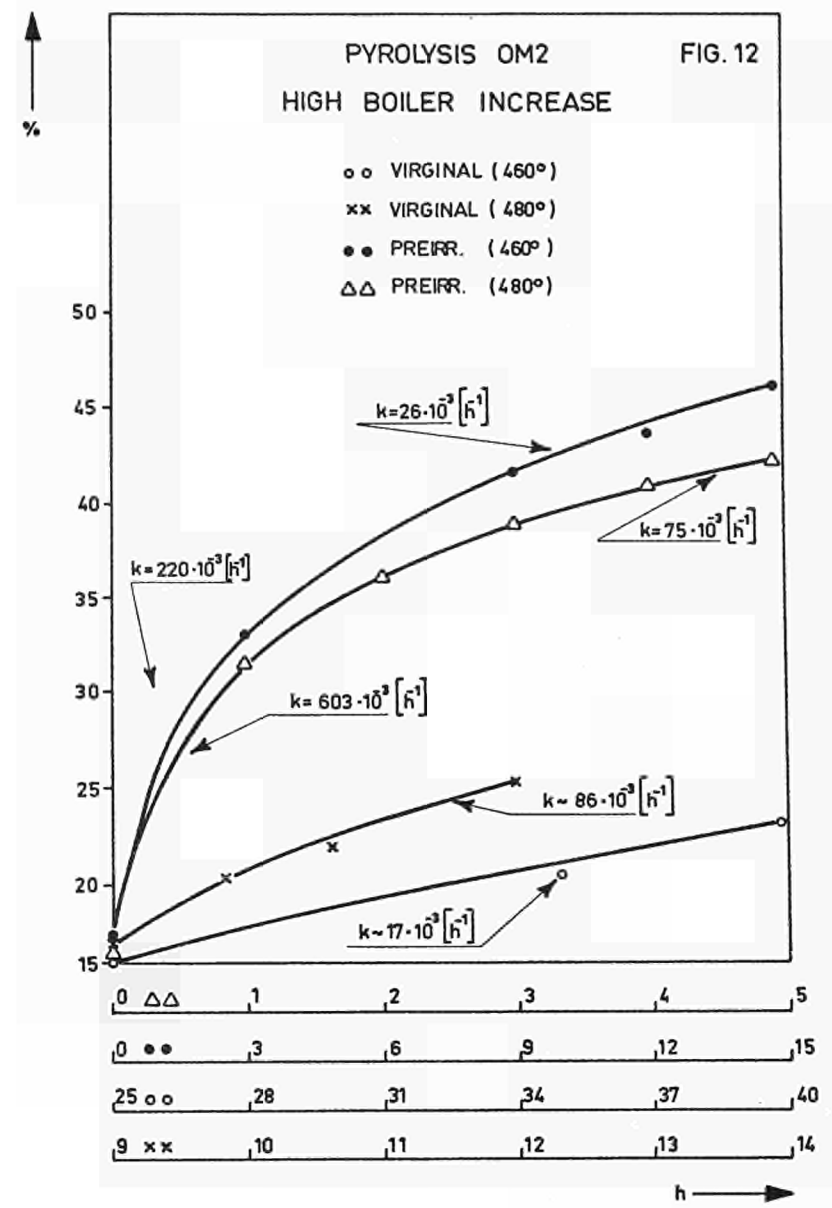
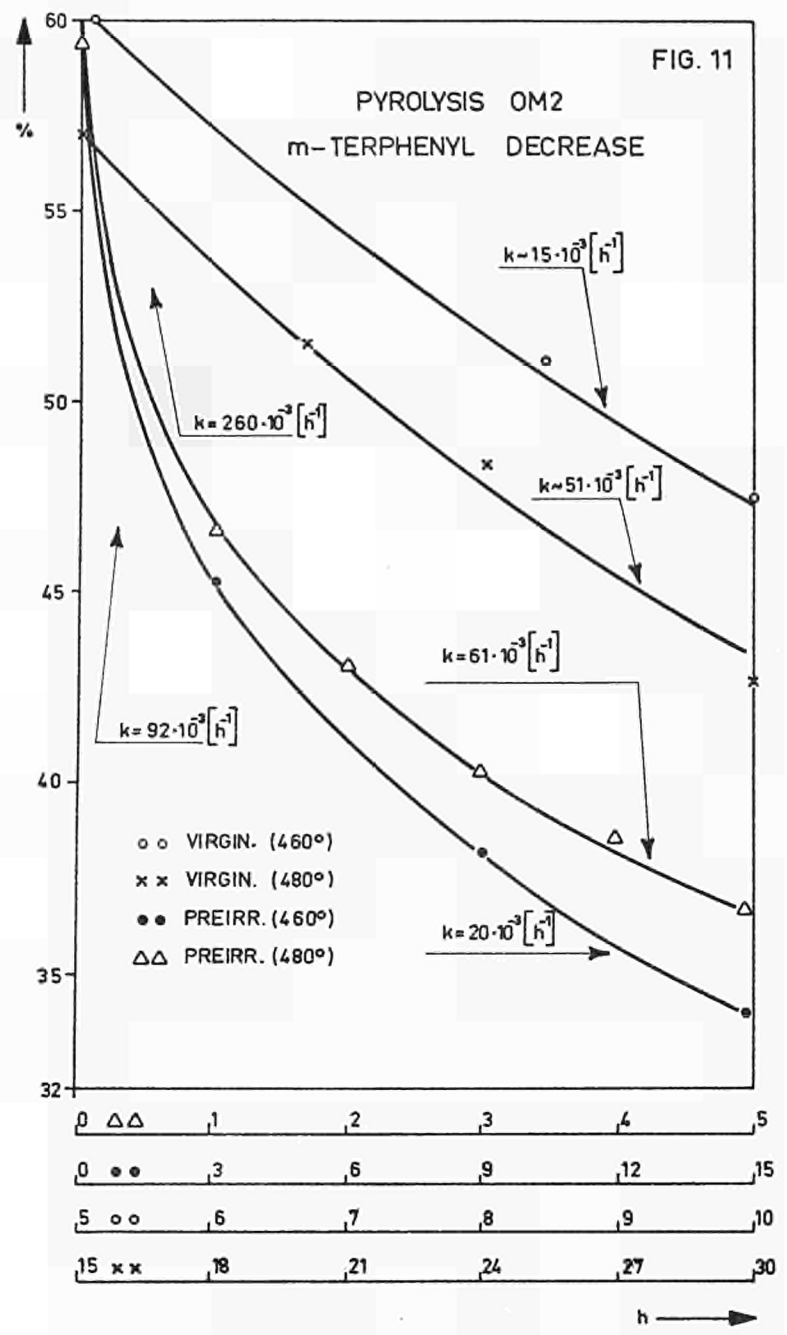


FORMATION OF QUINQUAPHENYLS AND HIGHER BOILING COMPOUNDS



FORMATION OF INTERMEDIATE BOILERS, QUATERPHENYLS AND HIGH BOILERS





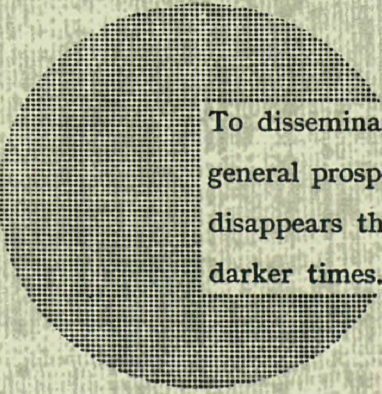
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**Alfred Nobel**

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