

**EUR 2646 e**

**Part III**

EUROPEAN ATOMIC ENERGY COMMUNITY - EURATOM

**THE RETENTION OF ORGANIC AND  
INORGANIC COMPOUNDS ON CLAY MINERALS**

**Part III : The Interaction of Attapulugus Clay  
and Hydrogenated Polyphenylic Nuclear Reactor Coolant**

by

R. LOPES CARDOZO and D. VAN VELZEN

1969



**Joint Nuclear Research Center  
Ispra Establishment - Italy**

**Chemistry Department  
Organic Chemistry**

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## **ABSTRACT**

Following a study of the interaction of Attapulugus Clay and polyphenylic reactor coolants, an investigation of the interaction of this clay material and hydrogenated polyphenylic reactor coolants has been carried out.

It is shown that the retention mechanism and the sorbate compounds are essentially the same in both cases.

The reaction rate constant and the retention capacity of the clay with hydrogenated polyphenylic coolants are superior to the corresponding values for polyphenylic coolants.

It is proved that Attapulugus Clay catalyses cracking, polymerisation and isomerisation reactions in non-irradiated, polyphenylic as well as hydrogenated polyphenylic coolants. In irradiated coolants, containing substantial amounts of High Boilers and other decomposition products, these catalytic effects are greatly reduced, in some cases they are even negligible.

## **KEYWORDS**

ADSORPTION

CLAYS

POLYPHENYLS

ORGANIC COOLANT

HYDROGENATION

REACTION KINETICS

CATALYSIS

CRACKING

POLYMERIZATION

ISOMERS

CHEMICAL REACTIONS

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## 1. INTRODUCTION (\*)

One of the major problems in the development of an organic cooled reactor is the control of fouling on fuel elements. A process for the elimination of fouling inducing constituents is sorption on Attapulugus Clay.

Initially some promising results have been obtained in batch experiments in the United States and Canada [1,2]. Afterwards Attapulugus Clay sorption systems were installed and operated in a by-pass loop at the OMRE during Core III-B and III-C [2], in the U-3 and X-7 loop at Chalk River and finally in the WR-1 reactor at Whiteshell (A.E.C.L.).

In a preceding study it has been proved that the purifying action of Attapulugus Clay consists of irreversible chemisorption of a very high molecular weight part of the coolant [3]. These products often contain oxygenated compounds, so that Attapulugus Clay treatment also causes a certain deoxygenation of the coolant.

The studies upon which the above statements are based, have been performed with blends of OM-2\*) and High Boilers from OMRE Core III-A. The OMRE facility (Organic Moderated Reactor Experiment) consisted of a 5 to 6 Mwt reactor, cooled and moderated by a mixture of terphenyls, generally at a temperature of 310°C. During Core III-A the organic liquid used consisted of Santowax OMP, a terphenyl blend of about 12 %w o-, 60 %w m- and 28 %w p-terphenyl. During this period the reactor operated at an average HB concentration of approximately 5 %w.

The only organic cooled reactor which is actually in operation, is the WR-1 reactor in Whiteshell (Canada). This reactor is an organic cooled, heavy water moderated experimental reactor with a thermal power of approximately 35 MW. The coolant used in this reactor consists of a mixture of partly hydrogenated terphenyls (40-60 % of saturation) which is manufactured by Monsanto under the name of HB-40. This coolant has a much more naphthenic character than the purely aromatic coolants, OM-2 and Santowax OMP. This fact may have its influence on the chemical nature of the very high molecular weight products sorbed by the Attapulugus Clay.

It is therefore of interest to investigate whether the mechanism proposed for OM-2 based coolants is also valid for HB-40 based coolants.

This complementary study was made feasible by the availability of about 400 kg of actual WR-1 coolant, dated 20/3/68, at the C.C.R. Ispra. The coolant is an equilibrium coolant obtained at a reactor temperature of 325-350°C. The HB content (determined according to the microdistillation method in use at Whiteshell [7]) has been maintained between 30 and 35 %w for nearly 8 months. The average molecular weight of the HB is 690, the autoignition temperature 420°C and the kinematic viscosity of the coolant is 0.80 cS at 300°C.

\* \*) A terphenyl mixture consisting of approximately 25 %w o-, 70 %w m- and 5 %w p-terphenyl.

\*) Manuscript received on 24 April 1969

## 2. NATURE OF THE REACTING IMPURITIES

In analogy with the preceding study [3] the Heavy High Boiler (HHB) and the oxygen content of the coolant were used as a yardstick for the detection of the nature of the reacting impurities.

The HHB content is defined as the percentage of the product which is insoluble in dodecane at room temperature. The method of determination is essentially the same as before, except that presently dodecane is used as a solvent instead of decane. The analytical procedure has been described in [3].

The oxygen content has been determined by activation analysis [4]. The experimental studies were carried out in a 4 cm diameter stainless steel column. The HHB and the oxygen content of the feed and of each percolation fraction has been determined. The clay used was the sieve fraction between 50 and 70 mesh from Attapulugus AA/LVM 30/60 obtained from the Minerals and Chemicals Philipp Corporation. It has been pretreated at 150°C, 10 cm Hg for 2 hours. Data, results and conditions of these experiments are given in Table I.

It appears that also in the present case an appreciable amount of HHB is retained on the Attapulugus Clay column. The effect rapidly decreases with increasing contact time.

Contrary to the results obtained with OM-2 based coolants, no sorption of oxygenated products can be observed.

Practically all deviations of the oxygen content are within the limit of error of the determination, which is approximately  $\pm 50$  ppm.

The conclusion is that the very high molecular weight compounds present in the WR-1 coolant are sorbed on Attapulugus Clay, but that these products are apparently free from oxygenated compounds.

## 3. THE SORPTION PROCESS

### 3.1. Retention Capacity

The retention capacity of Attapulugus Clay in contact with the WR-1 coolant has been determined by means of the same technique as has been used formerly.

It involves drying of the clay to constant weight before use under standard conditions (10 cm Hg and 150°C), thorough elution and washing after a percolation run and subsequent drying of the spent clay to constant weight under the same standard conditions. The resulting increase in weight is considered as the quantity of sorbate retained.

Elution is performed at working temperature by passing virgin HB-40 through the bed until the brown colour of the eluate has completely disappeared. This usually requires a quantity of 5 to 10 times of the bed weight. Subsequent washing of the bed is done with xylene at 130°C to 140°C until the solvent passes colourless and finally with 5 times the bed weight of benzene or toluene in order to remove all xylene.

TABLE I

Percolation experiments on Attapulugus Clay.

Flow direction		Downflow				Downflow			
Column diameter	(cm)	4.0				4.0			
Bed weight	(g)	320				320			
Temperature	(°C)	185				315			
Feed rate	(g.min <sup>-1</sup> )	18				18			
Feed		WR-1 coolant				WR-1 coolant			
HHB content of feed	(%w)	13.7				13.7			
Oxygen content of feed	(ppm)	480				480			
Contact Time (min)	Weight (g)	E/A	HHB Content (%w)	Oxygen Content (ppm)	Weight (g)	E/A	HHB Content (%w)	Oxygen Content (ppm)	
16*) - 20	70	0 -0.2	7.9	480	68	0 -0.2	3.5	540	
20 - 35	281	0.2-1.1	8.9	370	230	0.2-0.9	7.3	450	
35 - 50	268	1.1-1.9	10.5	480	265	0.9-1.8	10.5	490	
50 - 65	264	1.9-2.7	11.2	490	282	1.8-2.6	11.6	560	
65 - 80	272	2.7-3.6	12.0	440	280	2.6-3.5	12.0	580	
80 - 95	273	3.6-4.4	12.6	460	279	3.5-4.4	12.9	500	

\*) First drop appears after 16 minutes from start.



The percolation runs have been carried out in a glass column, internal diameter 12 mm, length 530 mm. The feed was introduced by means of a calibrated metering pump. Contact time was varied from 10 to 120 minutes. The results and conditions of these experiments are given in Table II and Fig. 1.

Fig. 1 shows that the sorption curve for the WR-1 coolant has the same nature as the curves for the reaction controlled chemisorption for OM-2 based coolants. These are given as a reference in the same figure. It is noted that the saturation capacity of Attapulugus Clay is substantially higher for the WR-1 coolant than for HB obtained from Santowax OMP (Core IIIA HB). This effect may be due to a higher average molecular weight and/or to a possible difference in chemical nature of the active compounds.

### 3.2. Estimation of the Reaction Rate Constant

In the preceding study [3] it has been shown that the sorption rate is either reaction or diffusion controlled.

The process is reaction controlled at relatively high impurity concentrations and diffusion controlled at low concentrations.

Evidently the impurity concentration in the WR-1 coolant is rather high, so that the process in this case is reaction controlled. The shape of the sorption curve (Fig. 1) and the high HHB content of the coolant (13.7 %w) bring this statement into evidence.

It follows that the available data only permit the evaluation of the reaction rate constant. The law governing the process can be expressed as:

$$-\ln (1 - W/Q) = \frac{K_R t}{Q}, \text{ or}$$

$$K_R = -\frac{Q}{t} \ln (1 - W/Q) \text{ in which}$$

- W = sorbate concentration, integrated over the bed height
- Q = saturation capacity
- t = contact time
- K<sub>R</sub> = reaction rate constant.

The saturation capacity has been estimated as 15 %w (Figure 1). The calculated value for the reaction rate constant becomes  $5.0 \times 10^{-3} \text{ min}^{-1}$ , all values being comprised between  $6.9 \times 10^{-3}$  and  $3.0 \times 10^{-3} \text{ min}^{-1}$ . This value is higher than the one found for OM-2 based coolants ( $2.0 \times 10^{-3} \text{ min}^{-1}$ ), but is still of the same order of magnitude.

The expression for the exit impurity concentration for the reaction controlled case is:

$$C_x = C_o - \frac{AK_R}{L} \exp \left( -\frac{K_R t}{Q} \right) \text{ in which}$$

$C_x$  = exit impurity concentration  
 $C_o$  = initial impurity concentration  
 $A$  = total weight of clay  
 $L$  = feed flow rate.

With this expression it is possible to calculate the HHB concentration of the percolated coolant as a function of contact time. The exit HHB concentration is equal to:

$$\begin{aligned}
 (\text{HHB})_x &= (\text{HHB})_o - (C_o - C_x) \\
 &= (\text{HHB})_o - \frac{AK_R}{L} \exp\left(-\frac{K_R t}{Q}\right)
 \end{aligned}$$

The evolution of the HHB content with contact time has been calculated for the two percolation runs in the 4 cm diameter stainless steel column (Table I).

The calculated lines together with the experimental points are given in Fig. 2.

It is concluded that the experimental data agree quite satisfactorily with the calculated lines and that the increase in temperature from 185°C to 315°C shows little effect. Similar conclusions have been drawn in the preceding study.

TABLE II

Percolation experiments in glass columns.

Column diameter : 12 mm Temperature : 175°C Feed rate : 13 g.min <sup>-1</sup> Feed : WR-1 coolant			
Contact Time (min)	Bed Weight		W*)
	In (g)	Out (g)	(%w)
15	26.90	28.92	7.5
25	28.42	31.03	9.2
45	26.70	29.69	11.2
85	30.00	33.66	12.2
125	28.32	32.41	14.4

\*) W = the percentage weight increase of the clay bed, i.e. the weight of the sorbate retained.

#### 4. ISOMERISATION AND CRACKING

It has precedently been proved that Attapulugus Clay has not only a sorptive, but also a catalytic action, which strongly increases polymerisation, cracking and isomerisation reactions. It has been shown that at approximately 350°C pure o-terphenyl has been decomposed for more than 60 % in 5 hours, mainly to m-terphenyl. It was also reported that the o-terphenyl content of OM-2 decreased from 23.3 to 19.1 %w, during the same contact time [5].

Pyrolysis experiments with o- and m-terphenyl in quartz capsules at 451°C showed an approximately two fold increase of the formation of benzene, diphenyl and HB, a strong isomerisation and a 50 times increased gas formation rate in presence of Attapulugus Clay [6].

In the present study the stability of HB-40 and the Canadian reactor coolant in the presence of Attapulugus Clay has been investigated. The liquids under investigation were boiled under reflux with and without addition of the clay. The resulting products were submitted to a fractional distillation analysis, during which a sample of 50 g has been divided into 3 fractions:

- One with a boiling point lower than 150°C at 20 mm Hg.
- One with a boiling point between 150 and 240°C at 20 mm Hg.
- One with a boiling point higher than 240°C (the residue).

In some cases the top temperature of 240°C at 20 mm Hg has not been attained; in these cases the distillation was stopped when the bottom temperature reached 360°C. The reflux ratio is 3, the distillation bottom has constantly been heated at a rate of 135 W and the column is a Vigreux column of 40 cm effective length.

The results of these analyses are given in Table III. This table also contains the fractional distillation analyses of the percolation fractions from the experiments summarised in Table I.

It appears that non-irradiated and irradiated HB-40 behave quite differently in the presence of Attapulugus Clay. The original product shows a poor stability: about 20 %w are converted into lighter products (in the range of diphenyl) and at the same time about 25 %w are polymerised. Besides, a strong isomerisation occurs, which qualitatively appears from the gaschromatographic analyses illustrated in Fig. 3.

Apparently the reaction is practically terminated after 2 hours. No sensible difference between the 2 and 5 hours experiments could be shown with the actual method of analysis.

Contrary to the behaviour of the original product, the irradiated coolant does not show a distinct change in composition upon Attapulugus Clay contact. Only the very first fraction of the percolation at 315°C apparently shows a formation of light products. However, this effect may be due to a chromatographic effect which gives rise to an enrichment in light products of this fraction during percolation.

All other samples shows a light decrease of 3 to 6 %w in the diphenyl-fraction, which points to a moderate polymerisation of

the light products. Generally, also a light decrease of the residue content is observed. The fact that part of the HB are chemisorbed by the Attapulugus Clay, accounts for this decrease. Besides, it should be kept in mind that the precision of the fractional distillation analysis is very limited and permits a very general evaluation only.

By gaschromatographic means no isomerisation could be shown, see Fig. 4.

Subsequently it has been investigated, if also in the case of OM-2 based coolants a difference in the behaviour of the original and irradiated coolants upon Attapulugus Clay treatment could be observed.

Virginal OM-2 and two irradiated products from irradiation experiments in the BLO-4 loop in the SILOE-reactor (Grenoble, France) were included in this investigation. The two irradiated products were near-equilibrium coolants from feed-and-bleed experiments of the following main characteristics:

	<u>Sample I</u>	<u>Sample II</u>
Run	C5-41-420	C2-41-320
HB-content	13.5 %w	43.1 %w
Temperature of irradiation	427°C	328°C
Dose intensity in irradiating vessel	1.4 W.g <sup>-1</sup>	2.8 W.g <sup>-1</sup>
Fast neutron fraction of energy deposited in the coolant	0.195	0.28

The liquids were boiled under reflux for 2 and 5 hours, with and without Attapulugus Clay addition.

The resulting products were analysed by gaschromatography. The results are given in Table IV.

Also in this case a substantial difference between unirradiated and irradiated coolants is observed. Under the actual experimental conditions (360°C) no formation of low boiling or polymerised products can be shown, neither with irradiated nor with original coolants. The isomerisation of o-terphenyl into m-terphenyl is very important in the case of the original coolants, as has been shown precedently [5]. The o-terphenyl content decreased from 23.7 to 17.0 %w in 2 hours and to 14.7 %w in 5 hours. A slight increase in p-terphenyl content could be observed, but the major part of the o-terphenyl changes into m-terphenyl.

These isomerisation effects are much less important for the two irradiated coolants. The experiments with the coolant containing 13.5 %w HB showed a decrease from 17.2 to 16.4 %w of o-terphenyl in 2 hours and to 15.2 %w in 5 hours, whereas in the experiments with the C2-42-320 sample containing 43.1 %w HB the decrease in o-terphenyl content is even smaller: from 11.2 to 10.5 %w in 5 hours

TABLE III

Polymerisation and cracking of HB-40 in presence of Attapulugus Clay.

200 g of HB-40 have been boiled under reflux in presence of 50 g of Attapulugus Clay.

Boiling Time	Fraction $\phi_2^*$ (%w)	Fraction $\phi_3^*$ (%w)	Residue* (%w)
Virginal HB-40, ex Monsanto			
Original product	0.0	96.0	4.0
5 h without clay	0.0	87.3	12.8
2 h with clay	19.1	48.7	29.5
5 h with clay	22.3	50.9	26.6
Coolant ex A-loop WR-1 reactor, d.d. 20/3/68			
Original product	13.5	44.6	40.9
2 h with clay	7.7	50.3	40.2
5 h with clay	10.0	50.0	39.5
Fraction n°	Fractions of the percolation run (Table I) at 185°C		
1	8.6	45.4	44.9
2	7.1	56.8	33.6
3	8.9	58.7	32.4
4	10.3	50.5	34.6
5	9.2	53.2	36.5
6	9.2	Lost	Lost
Fraction n°	Fractions of the percolation run (Table I) at 315°C		
1	29.5	Lost	Lost
2	13.5	46.8	36.6
3	9.7	50.1	40.5
4	8.6	51.3	36.9
5 and 6 combined	10.3	51.7	36.4

\*<sup>1</sup>) Fraction  $\phi_2$ : Boiling point less than 150°C/20 mm Hg.  
 Fraction  $\phi_3$ : Boiling point comprised between 150 and 240°C/  
 20 mm Hg.  
 Residue : Boiling point superior to 240°C/20 mm Hg.

For the difference in behaviour of irradiated and unirradiated coolants in contact with Attapulugus Clay, two main causes can be responsible:

- 1) The high molecular weight products in the irradiated coolants are rapidly chemisorbed on the clay surface; the active spots are covered and the clay soon loses its catalytic activity.
- 2) In the case of HB-40 based coolants, the less stable compounds which were initially present, have precedly been decomposed and replaced by more stable products during the irradiation process.

TABLE IV

Polymerisation and cracking of OM-2 in presence of Attapulugus Clay.

200 g of coolant have been boiled under reflux in presence of 50 g of Attapulugus Clay.

Boiling Time	Diphenyl (%w)	Terphenyls			HB (%w)
		ortho (%w)	meta (%w)	para (%w)	
Virginal OM-2					
Original product	-	24.1	70.4	5.2	0.3
5 h without clay	-	23.3	69.8	4.9	2.0
2 h with clay	-	17.0	73.9	6.4	2.7
5 h with clay	-	14.7	75.9	7.3	2.1
Sample C5-41-420					
Original product	1.3	17.1	63.6	4.6	13.4
5 h without clay	1.2	17.2	63.3	4.6	13.7
2 h with clay	1.5	16.4	63.4	4.8	13.9
5 h with clay	1.3	15.2	64.6	5.3	13.6
Sample C2-42-320					
Original product	0.8	11.2	41.9	3.0	43.1
5 h without clay	0.7	11.2	41.6	3.1	43.4
2 h with clay	0.7	10.8	42.4	3.3	42.8
5 h with clay	1.0	10.5	42.4	3.5	42.6

## 5. CONCLUSIONS

The present complementary study has shown that the action of Attapulugus Clay in the presence of hydrogenated polyphenyl based coolants consists of irreversible chemisorption of impurities from the HHB range of the coolant. This is also the case with the action of this material in contact with non-hydrogenated polyphenylic coolants [3].

Contrary to the polyphenylic case, no decrease in the oxygen content upon Attapulugus Clay treatment could be observed for a hydrogenated coolant.

For an actual coolant from the WR-1 reactor the overall sorption rate is determined by the rate of reaction; diffusion and adsorption rates can be neglected.

The reaction rate constant has been determined as  $5 \times 10^{-3} \text{ min}^{-1}$ . This value is superior to the corresponding constant for the polyphenylic coolant, which is  $2 \times 10^{-3} \text{ min}^{-1}$ .

The saturation capacity of the clay is approximately 15 %w, which is also superior to the saturation capacity of Attapulugus Clay in contact with polyphenylic coolants, i.e. 5-10 %w.

It has been shown that Attapulugus Clay causes very important cracking, polymerisation and isomerisation reactions in virginal HB-40. The irradiated reactor coolant only undergoes minor changes in composition.

It has been proved that this last conclusion is also valid for OM-2 based coolants.

## NOTATION

All dimensions are given in terms of weight (w), time (t) and length (l).

A	: total weight of sorbent	(w)
C <sub>0</sub>	: initial sorbate concentration	(-)
C <sub>x</sub>	: final sorbate concentration	(-)
(HHB) <sub>0</sub>	: initial HHB concentration	(-)
(HHB) <sub>x</sub>	: final HHB concentration	(-)
K <sub>R</sub>	: reaction rate constant	(t <sup>-1</sup> )
L	: liquid flow rate	(w.t <sup>-1</sup> )
Q	: saturation capacity	(-)
W	: sorbate concentration on sorbent, integrated over bed height	(-)

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# ATTAPULGUS CLAY SORPTION

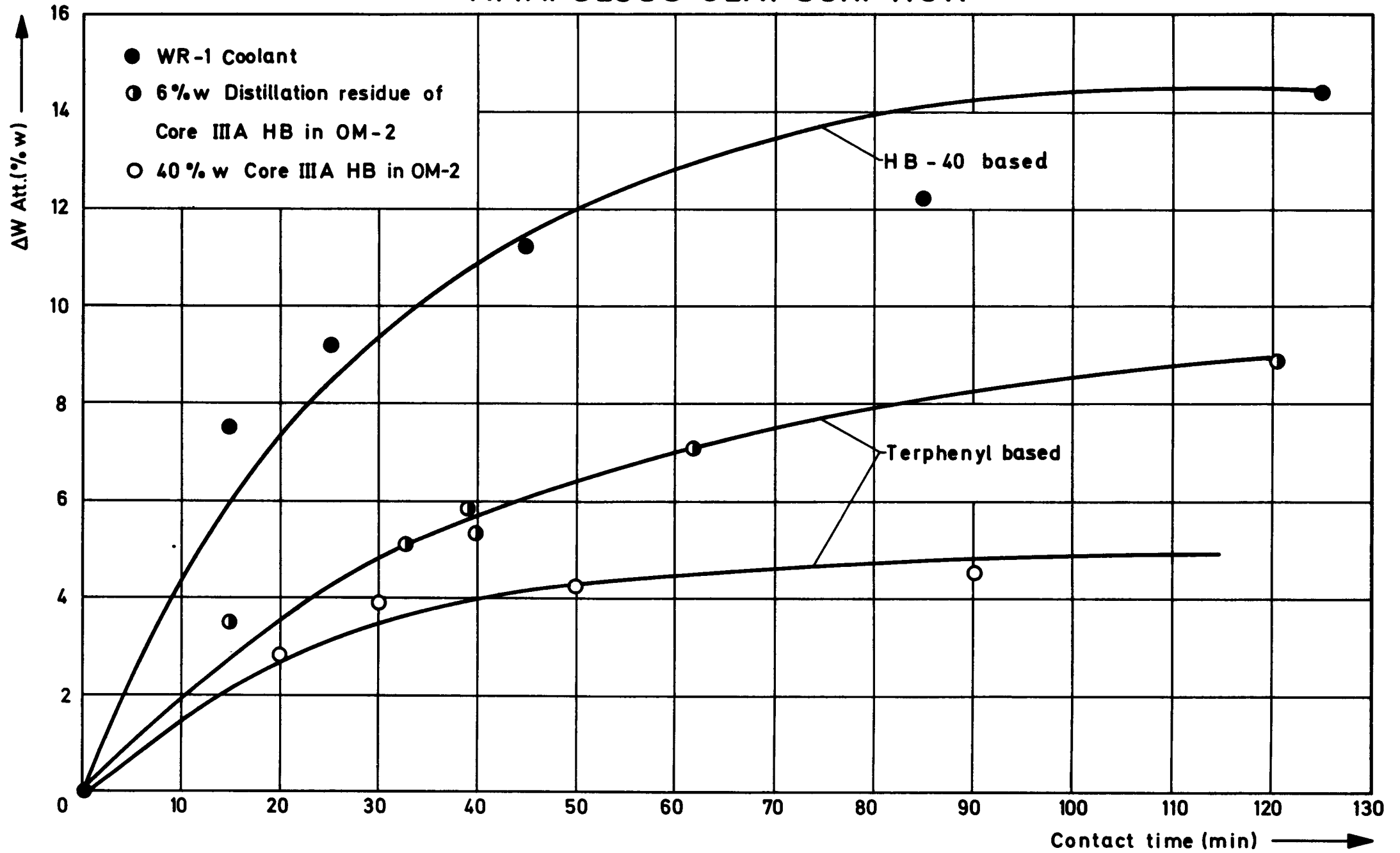
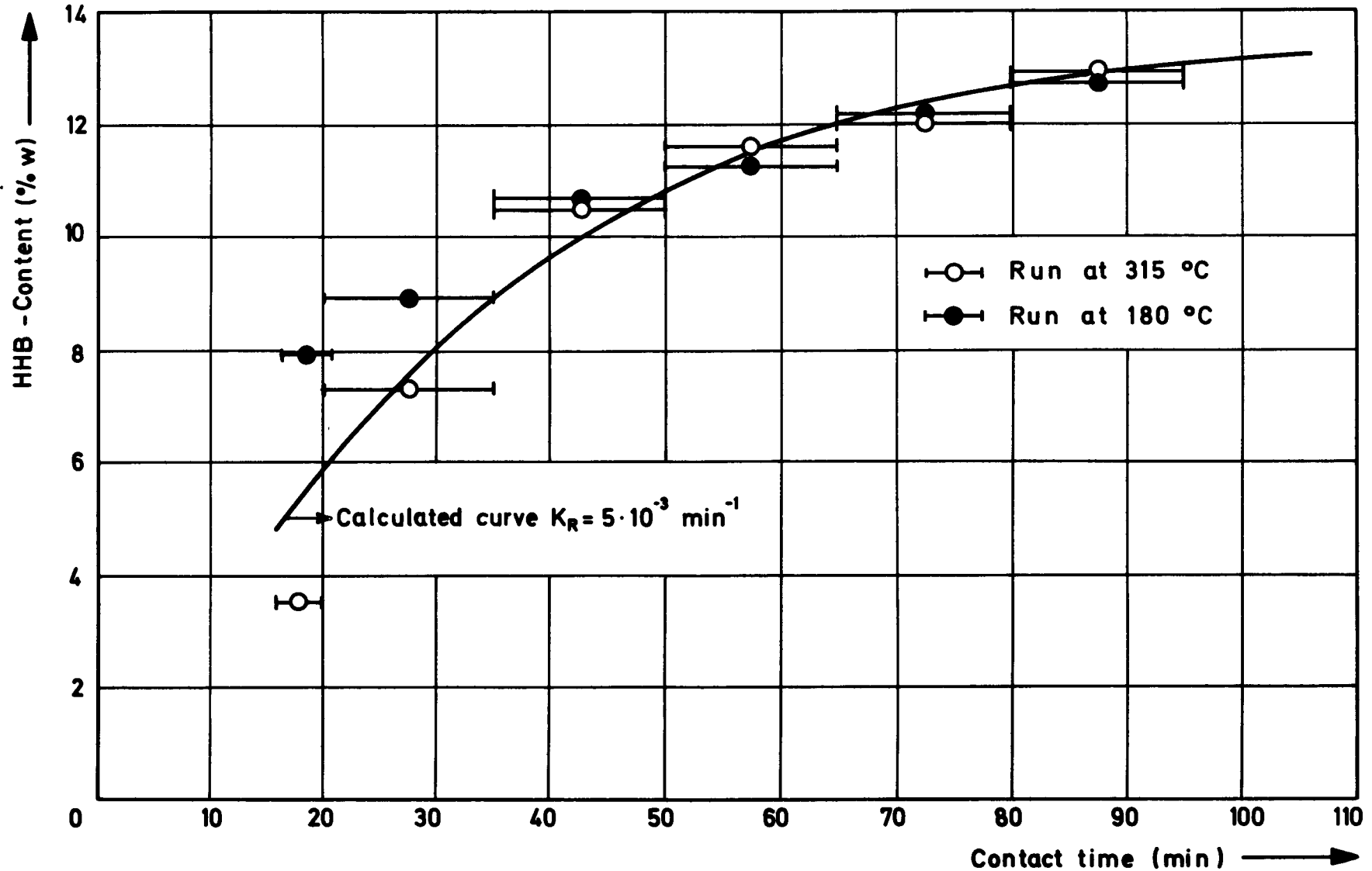


Figure 2

SORPTION ON ATTAPULGUS CLAY OF WR-1 COOLANT  
EXPERIMENTAL AND CALCULATED RESULTS



**Figure 3**  
**CHROMATOGRAM OF UNIRRADIATED**  
**HB-40 ON DIASPORT S+10% DOW 11**

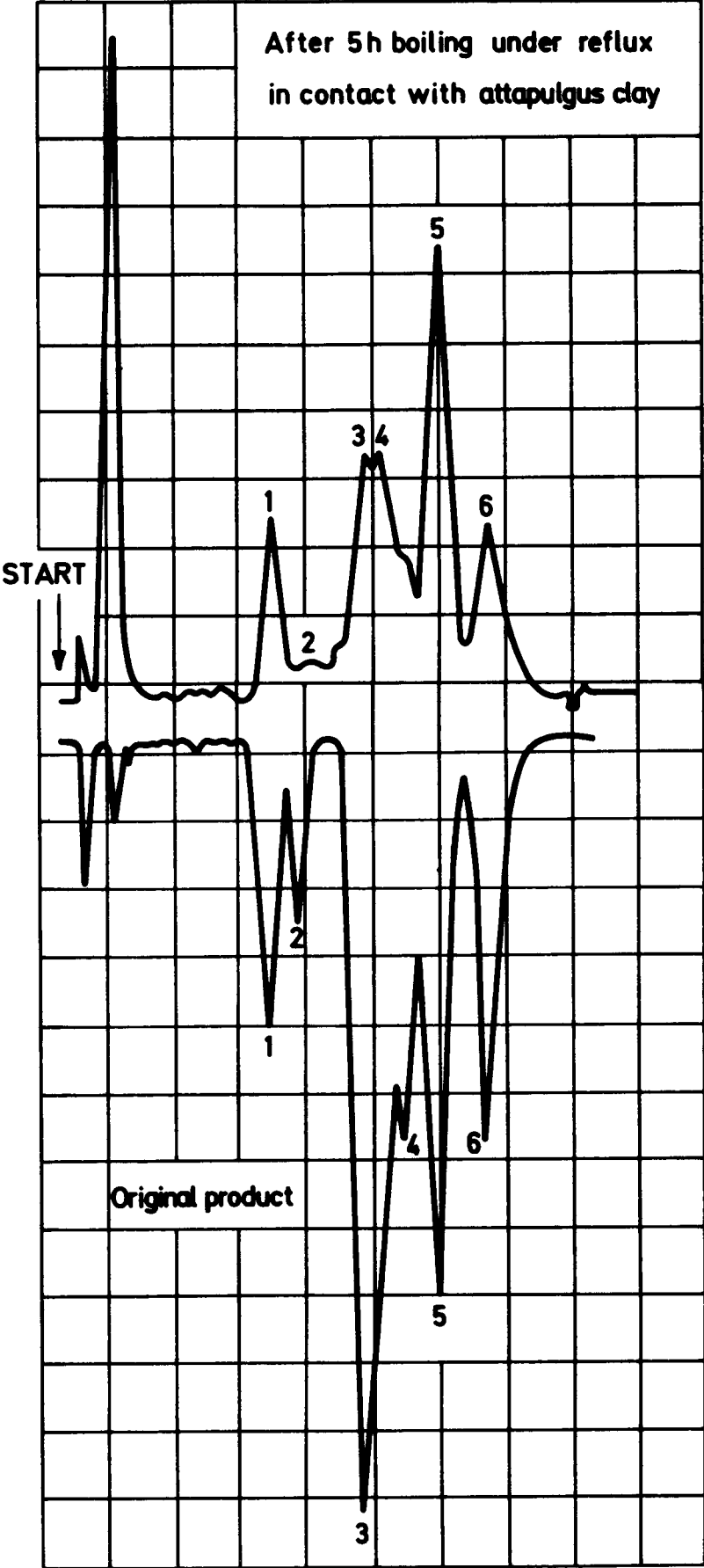
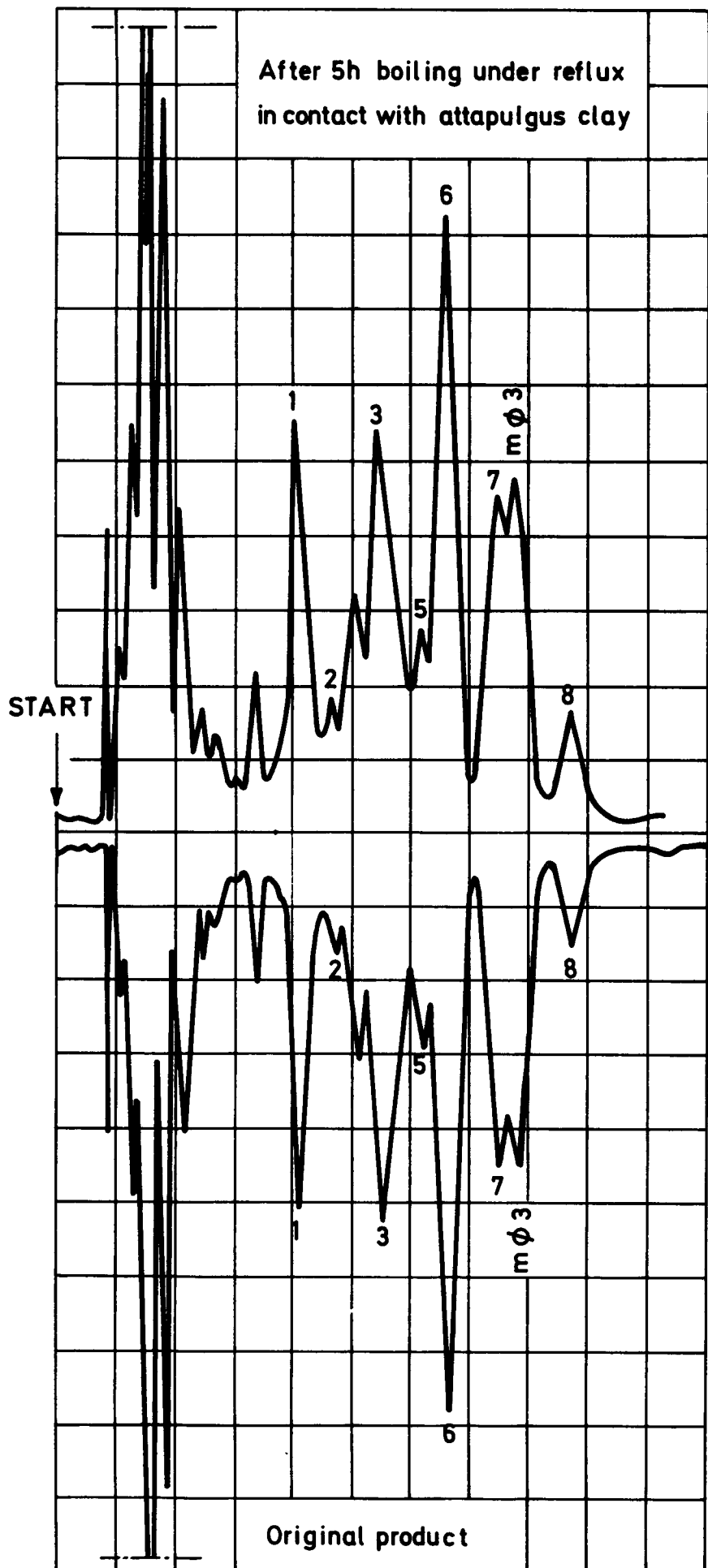


Figure 4  
CHROMATOGRAM OF IRRADIATED HB-40  
(WR-1-COOLANT) ON DIASPORT S+10% DOW 11



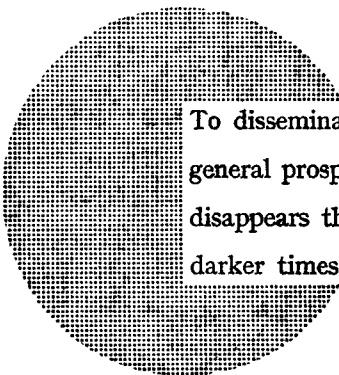
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Alfred Nobel

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