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EUROPEAN ATOMIC ENERGY COMMUNITY — EURATOM

PHYSICAL PROPERTIES OF ORGANIC NUCLEAR REACTOR COOLANTS

by

S. ELBERG (C.E.A.) and G. FRITZ (EURATOM)

1963

ORGEL Program

Euratom contract No 007-60-12 ORGF with C.E.A. (CEN Grenoble, France)

Paper presented at the 144th National ACS Meeting Division of Industrial and Engineering Chemistry
Symposium on Organic Nuclear Reactor Coolant Technology,
Los Angeles (Calif., USA) March 31st to April 5th, 1963

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PHYSICAL PROPERTIES OF NUCLEAR REACTOR COOLANTS

SUMMARY

Apparatus for the following properties were developed : density, viscosity, thermal conductivity, specific heat, vapour pressure, surface tension, latent heat of vaporisation and critical temperature.

The influence of high-boilers content by pyrolitic and radiolytic decomposition was studied. An important result : Besides the percentage of high boiler (% HBR) the nature of formation of that percentage has an influence on the physical properties.

Some other results with pure high-boilers are presented and the data of the latent heat of vaporisation.

INTRODUCTION

At C. E. N.-Grenoble — connected with EURATOM by a contract within the ORGEL program — apparatus for the following physical properties were developed : density, viscosity, thermal conductivity, specific heat and vapour pressure. All equipments are working up to 500 °C and 20 kg/cm^2.

Besides the measurements of the properties of diphenyl and terphenyls, the influence of high-boiler content formed by radiolytic and pyrolytic decomposition was studied. About this the following question is important : Is there a change in physical properties if the HBR is obtained at different temperatures and different irradiation dose?

On the other hand the data of pure high-boilers are of interest. Moreover some properties are not yet wellknown (like latent heat of vaporisation and surface tension). These data — important for boiling and burnout — should also be measured. This program was started in Ispra.

1 — DESCRIPTION OF THE DIFFERENT APPARATUS

1.1 — The apparatus developed in Grenoble

The equipments are all provided for a temperature range up to 500 °C and a pressure up to 20 kg/cm^2. The most important points of view for the construction have been the following : simplicity, easy to handle, elimination of glands, small number of joints.

1.1.1 — The densimeter (1) (2)

It is based on the principle of Mohr's balance : the weight of a quartz plummet immerged in the liquid is determined with an electromagnetic balance which regulates automatically the current of equilibrium. Between current and weight we have a linear dependence. The current is measured with a potentiometric method. The error in temperature measurement — done with platinum resistance wire — is in the range of 1/4 °C. The accuracy of density determination is about 0.1 %. Fig. 1-1 shows the apparatus.

1.1.2 — The viscometer

The principle of the method is Lawaczek's falling cylinder viscometer. The velocity of a cylinder (diameter d) in vertical tube (diameter d') filled with a liquid of dynamic viscosity μ

is approximately :

$$v = \frac{(d' - d)^3 \cdot (\varrho_1 - \varrho_2) \cdot g}{24\mu \cdot d}$$

where ϱ_1 and ϱ_2 are the densities of cylinder and liquid, and g is the gravity acceleration.

The velocity v is measured by two magnetic signals corresponding to a certain falling distance. The reset of the plummet to the starting position is also done magnetically.

A series of cylinders is provided to cover a range of viscosity from 0.15 to 10 centipoises. In the optimum zone of each plummet the error on viscosity determination is within -0.7% at room temperature and about 1.5% at 500°C. The global error on temperature — measured with a platinum resistance thermometer — is about 1/2°C. Fig. 1-2 shows the apparatus.

1.1.3 — The equipment for vapour pressure measurements (1) (3)

The vapour-pressure is determined with a zero-method. The sample is placed in a container with uniform temperature, which can be evacuated. It is closed by a metallic bellows. The vapour-pressure is counter-balanced by a nitrogen pressure, which is measured with manometers of 0.5% accuracy. The temperature error is less than 1/3°C. Fig. 1-4 gives a view of the apparatus

1.1.4 — Measurement of thermal conductivity (1)

A non-stationary wire method was chosen. The equipment is working nearly full automatically to minimize reading errors during the short measuring period.

A very thin resistance wire is immerged in the liquid. The temperature rise caused by a heating electrical current step in the wire multiplied by time is inversely proportional to the thermal conductivity of the liquid. The law is valid in the few seconds which follow the heating step.

The main parts of the equipment are : the wire-support, measuring bridge, direct current amplifier, an electronic multiplying and differentiating system and a recorder.

Toluene was taken for calibrating. The accuracy on thermal conductivity determination relative to that of toluene is estimated to be in the range of 1.5% (included 0.5% systematic error)

Fig. 1-4 shows the wire-support, which was provided to put into the furnace of the densimeter (Fig. 1-1).

1.1.5 — The calorimeter

It is an adiabatic type with a small heat capacity of the sample container. The container is tightly closed and heated electrically. The temperature rise is measured by a platinum resistance wire.

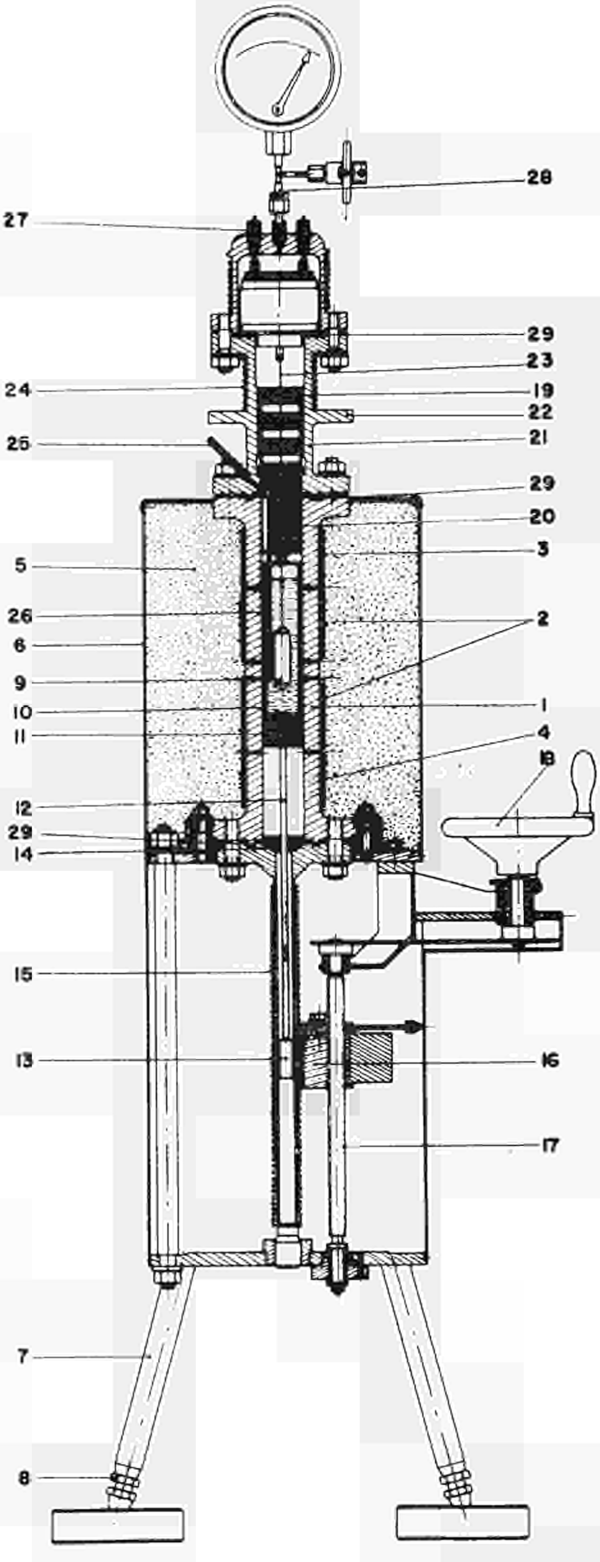
The vapour pressure in the container is counter-balanced by an outer-pressure to avoid destruction of the thin walls. The container is surrounded by a copper-jacket which is following the container-temperature to obtain adiabatic conditions.

The container and the heated jacket are placed in a vessel kept on uniform temperature which holds the counter-pressure for the sample container.

The regulation is done with differential thermocouples.

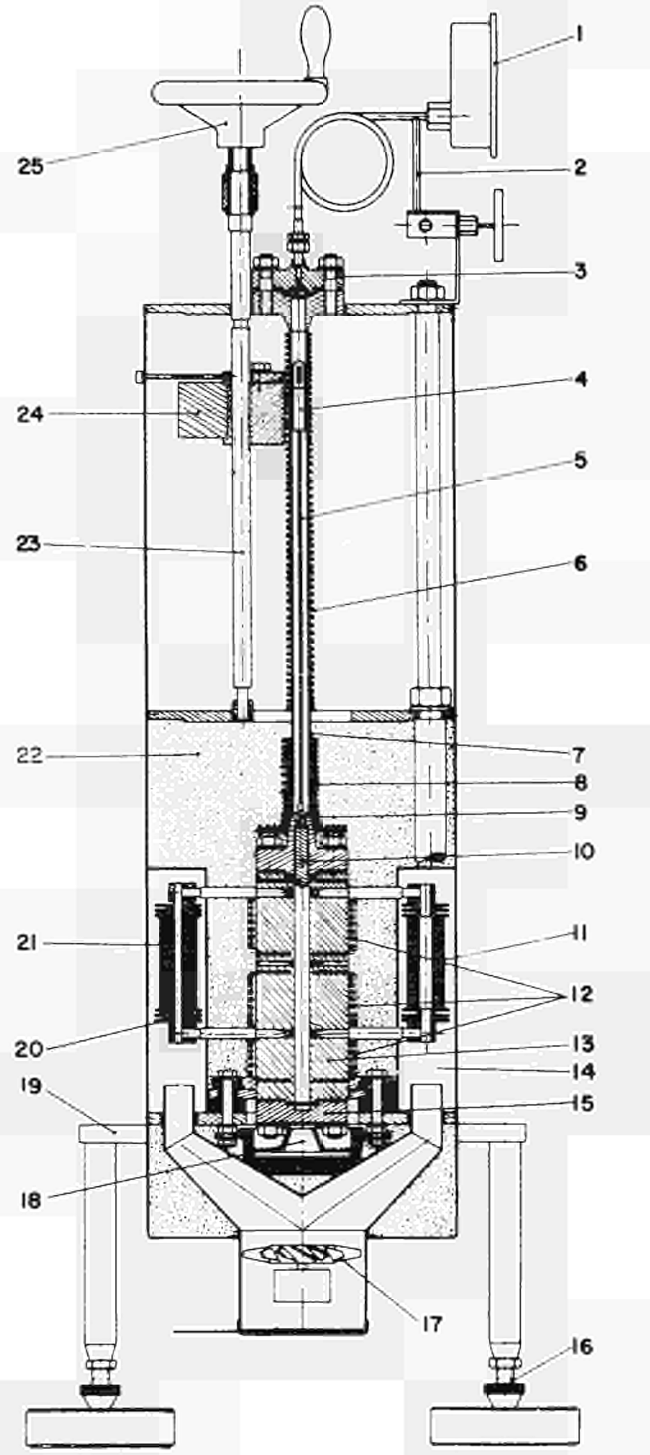
The scattering of the results was within ± 0.5%. The systematic error in the upper temperature range is estimated to be in the same range.

The photographs on Fig. 1-5 give an impression of the calorimeter.



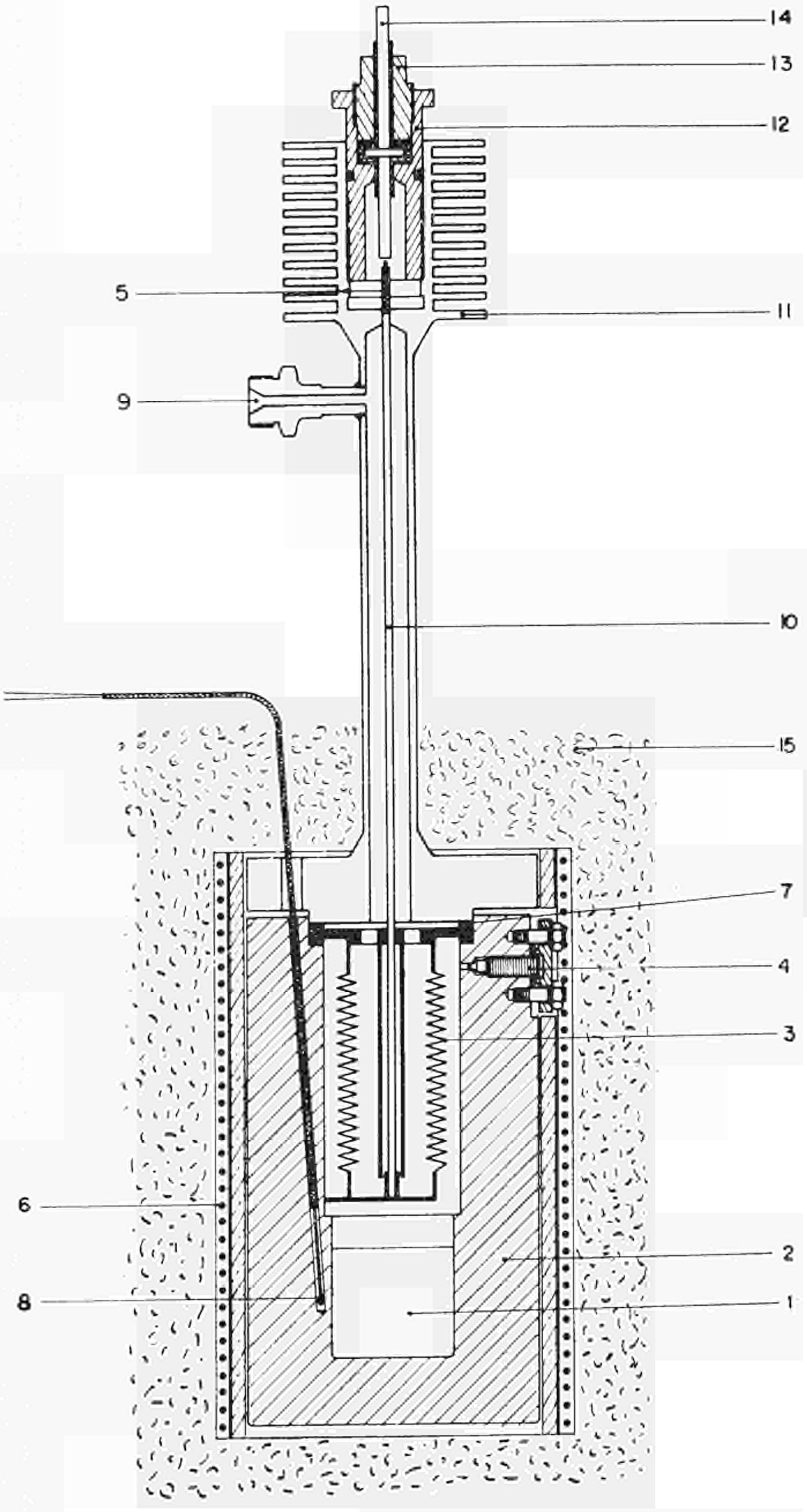
1 — Cylindrical furnace of stainless steel
2 — Mean heating collar
3-4 — Compensation heating collars
5 — Insulating material
6 — Iron hood
7-8 — Adjustable supports
9 — Quartz plummet
10 — Container
11 — Support of container 10
12 — Rod connecting 11 to the iron core 13
13 — Iron core
14 — Plug of "Asbestex"
15 — Sleeve
16 — Magnet
17 — Worm screw
18 — Hand wheel
19 — Condensation blocking
20 — Plug of "Asbestex"
21 — Muff coupling supporting the condensation plugs
22 — Machined flange for verticality adjustment
23 — Suspension wire of the quartz plummet
24 — Cooling warm
25 — Tight output
26 — Platinum resistance thermometer
27 — Insulated electric contacts
28 — Vacuum and inert gas input
29 — Gaskets

Fig. 1-1 — The densimeter



1 — Manometer
2 — Vacuum and inert gas input
3 — Upper flange
4 — Iron cylinder
5 — Jointed rod
6 — Heating element
7 — Leading tube — Expansion vessel
8 — Compensation heating
9 — Magnet
10 — Cylindrical piston
11 — Inductor coil
12 — Heating collars
13 — Measuring cylinder
14 — Coils housing
15 — Lower flange
16 — Adjustable supports
17 — Fan
18 — Compensation heating
19 — Horizontal base
20 — Cooling fins
21 — Receiving coil
22 — Insulating material
23 — Ordering screw of magnet 24
24 — Ticonal magnet
25 — Hand wheel

Fig. 1-2 — The viscometer



1 — Sample
2 — Stainless steel container
3 — Bellows
4 — Cone-pointed screw
5 — Spring
6 — Heating element
7 — Copper gaskets
8 — Platinum resistance thermometer
9 — Vacuum and inert gas inlet
10 — Minitube
11 — Cooling fins
12 — Movable electric contact holder
13 — Tight output
14 — Insulated electric contact
15 — Insulating material

Fig. 1-3 — The vapour pressure apparatus

1.2 — Apparatus developed at C.C.R. Ispra

In Ispra up to now are finished apparatus for measuring the following properties : density, viscosity, surface-tension, thermal conductivity, latent heat of vaporisation and critical temperature. Mostly (except for heat of vaporisation) they are micro-methods with sample quantities from 50 to 500 mg, to be able to measure also pure high-boiler which are difficult to obtain in larger quantities. The chosen methods for density, viscosity and thermal conductivity are different from those taken in Grenoble for better detection of probable systematic errors.

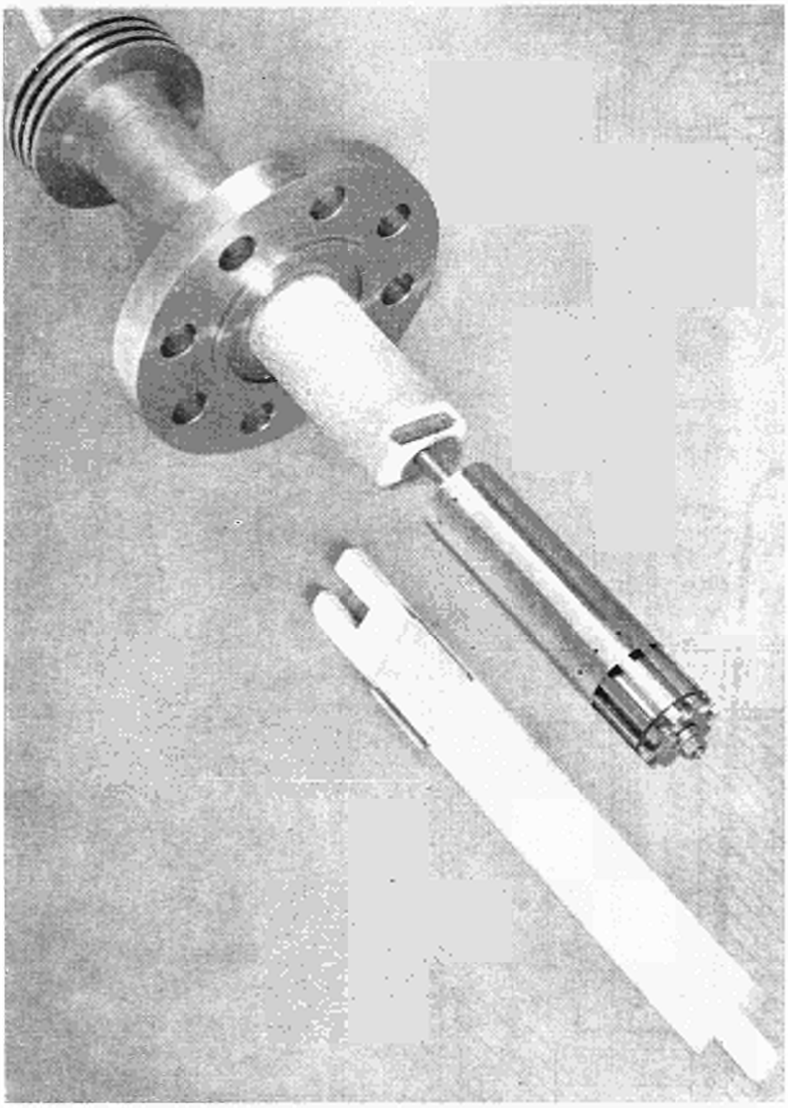
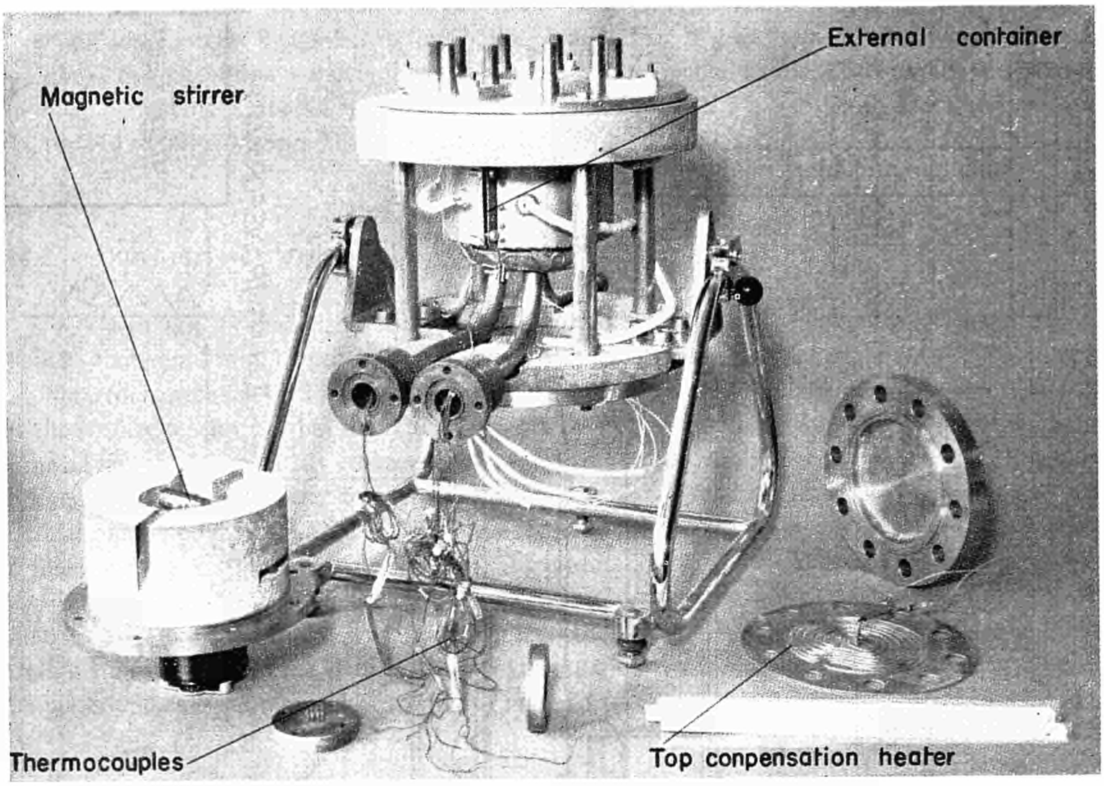


Fig. 1-4 — The thermal conductivity probe

1.2.1 — Measurement of density (4)

Fig. 1-6 shows the principle of the method. The length of a liquid thread in a capillary fused at one end is measured with a cathetometer while heated up in a glass furnace with slitted copper bloc. Afterwards the capillary is weighed on a μ-balance. The relation between length



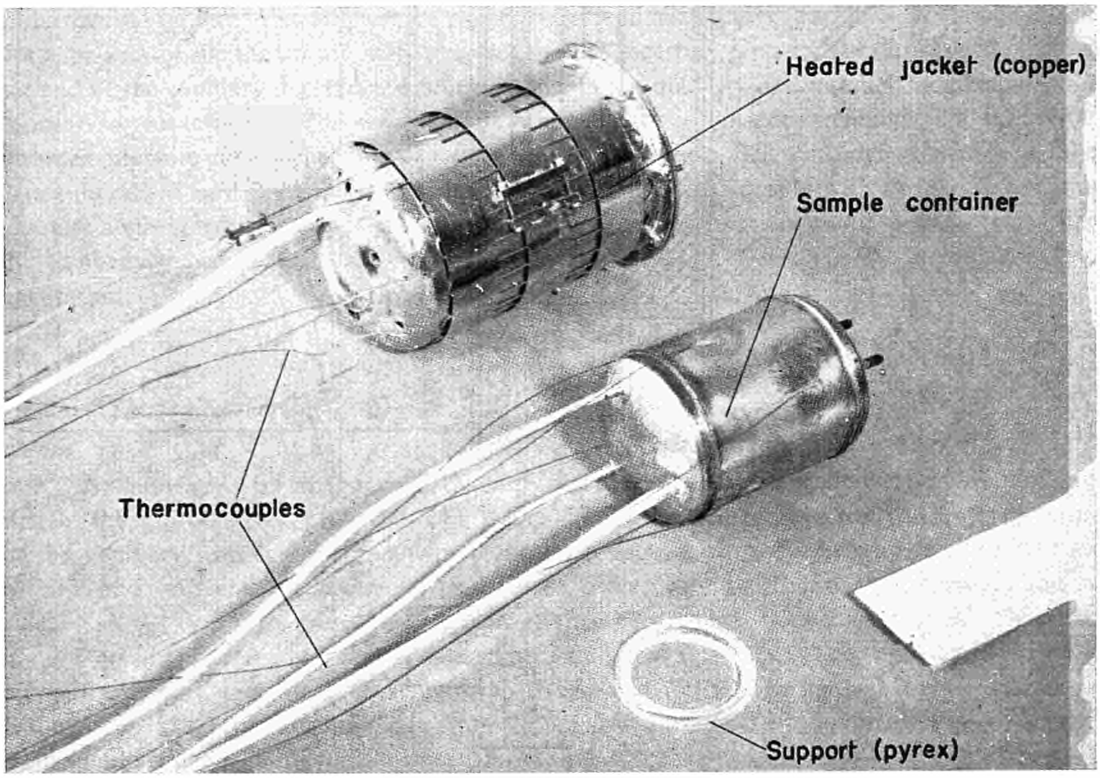
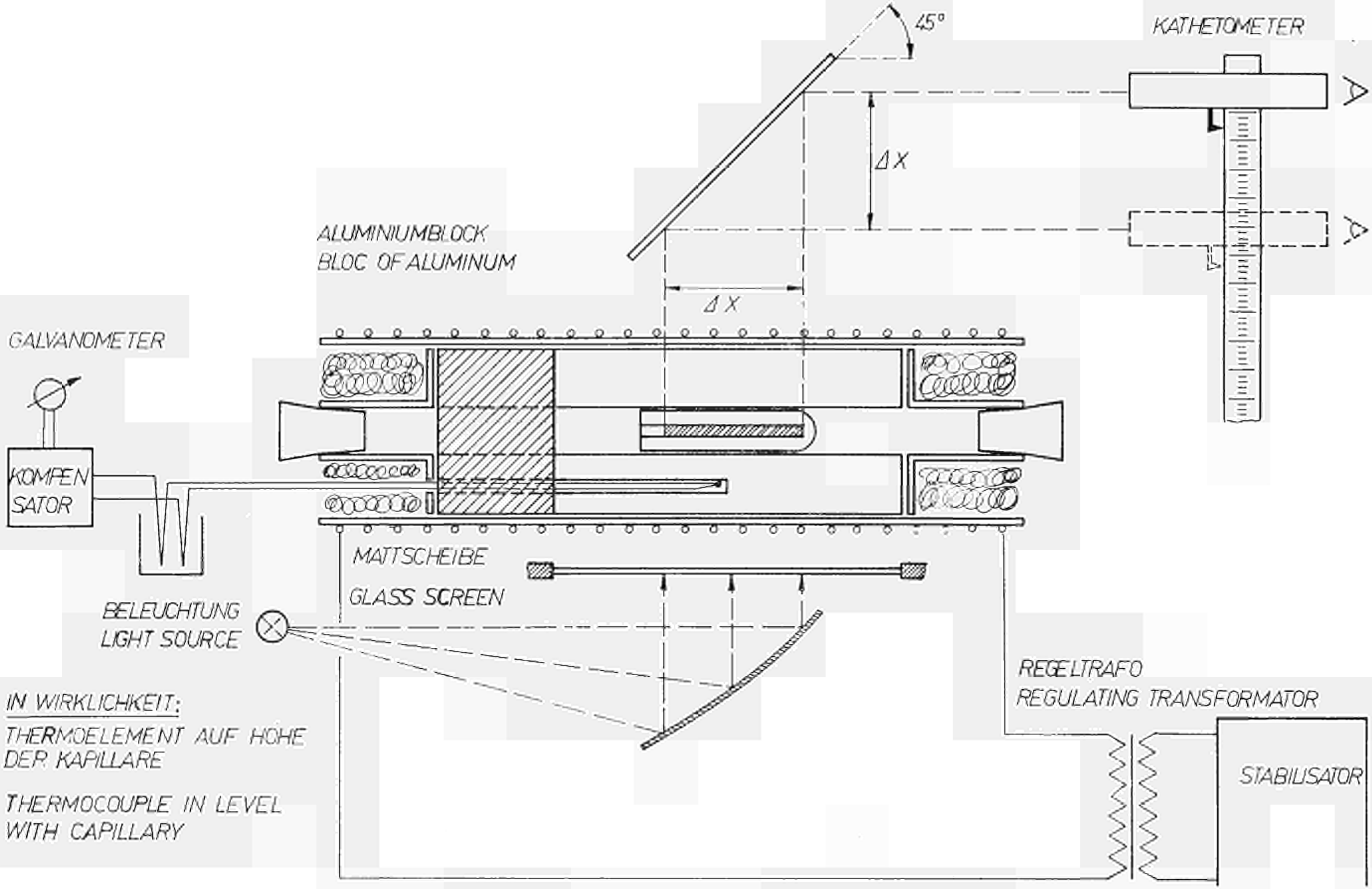


Fig. 1-5 — The calorimeter



IN WIRKLICHKEIT:

THERMOELEMENT AUF HOHE
DER KAPILLARE

THERMOCOUPLE IN LEVEL
WITH CAPILLARY

of thread and volume was tested before by water calibration. The open capillary works up to a certain temperature range below the boiling point. For higher temperatures the capillary is fused and the method is working like a dilatometer. For injecting system and capillary a sample quantity of 100 mg is necessary. Accuracy is estimated to be 0.5% for the absolute method and 1% for the relative method (fused capillary). Ref. (^{4}) gives details.

1.2.2 — Viscosity measurement

A capillary is placed horizontally in a furnace with slit for observing. A small liquid-thread (diameter 2 mm, length 30 mm) is pushed by a differential pressure through the measuring capillary (diameter ca. 0.12 mm) and observed with a cathetometer. The thread is kept together by the surface tension so that the liquid does not flow out. Horizontal capillary and furnace have some advantages : no density correction on measuring values, the liquid flow stops when pressure difference is zero and better temperature distribution of the horizontal furnace instead of the vertical one. A counter, started and stopped by hand, was taken for a stop watch. The capillary is filled with a special injecting system. The minimum need of substance for injecting and measuring is in the range of 200 mg. The temperature is measured with thermocouples in the bloc. In a special experiment it was verified that the liquid thread takes the temperature of the bloc. Calibrating was done with distilled water at different temperatures. The error relative to the water values is estimated to be in the range of 1%. (See fig. 1-7).

1.2.3 — Surface-tension measurements (^{4}) (^{5})

A liquid thread is placed within a capillary with 0.3 mm diameter which is heated up in an horizontal furnace. A small pressure pushes the thread to the end of capillary. Without pressure both surfaces of the thread are half spheres, but with a certain pressure the liquid surface at the cut end of the capillary is plane and reflects a maximum quantity of a parallel light beam which is observed in a telescope. The pressure is determined with a miniscope (reading 1/100 mm H_2O). Between pressure and surface-tension exists a linear relation. The calibration was done with 7 test liquids at well known surface tension. The method works only with full wetting liquids (contact angle zero). With another method — the bubble pressure method — was verified that the terphenyls and their mixtures fulfil this condition. Fig. 1-8 shows the principle of the apparatus.

1.2.4 — Thermal conductivity

A stationary wire method was taken to determine the thermal conductivity. A platinum wire of 0.1 mm diameter in the axis of a 2 mm capillary is heated electrically. The temperature of the capillary wall is measured with platinum resistance wire. With temperature difference of wire and wall and the produced heat in the center wire the thermal conductivity can be calculated. An end correction must be applied and the convection must be avoided. The latter is done by heating with different energies and extrapolating to zero. The capillary is placed in a pressure system wich allows working up to 20 atm and 450°C. The method is an absolute one. Comparison with other absolute data from Riedel (obtained with another method) for Benzene, Toluene and Carbon tetrachloride gave an agreement within ± 1%.

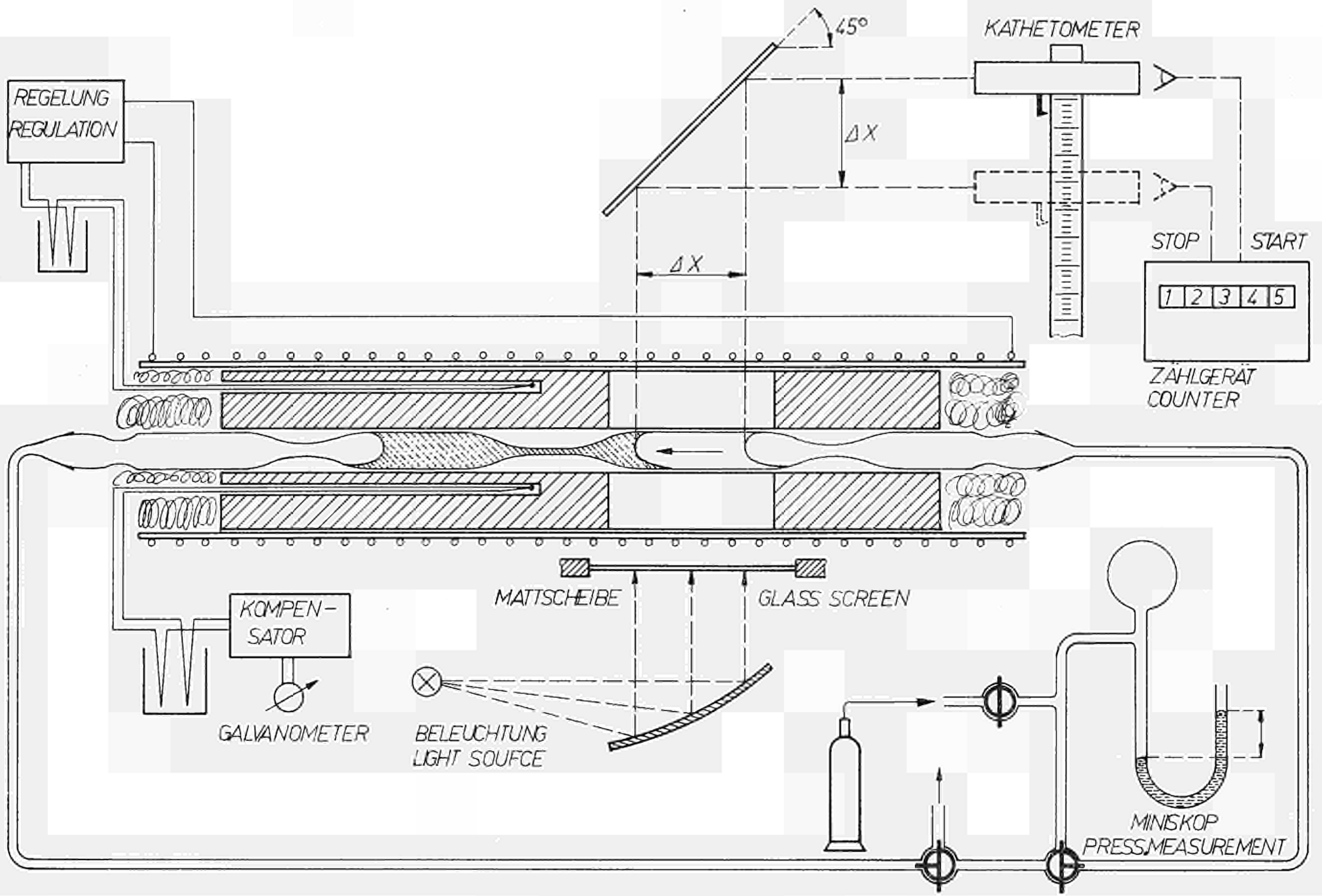


Fig. 1-7 — Viscosity-measurement

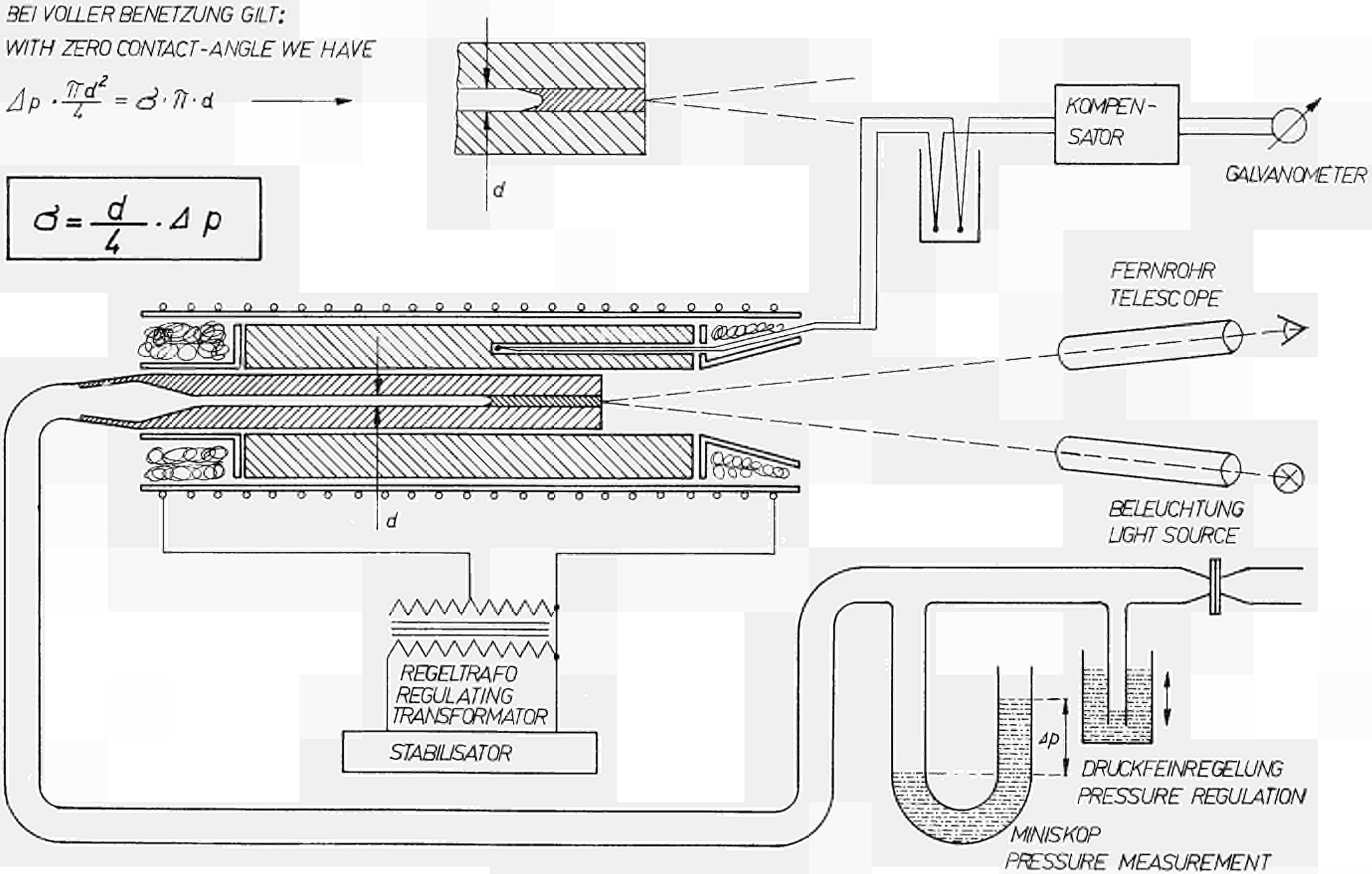


Fig. 1-8 — Surface-tension measurement

1.2.5 — Latent heat of vaporisation

Up to now there existed only a few direct measurements of the latent heat of vaporisat of polyphenyls. A certain property, evaporated isothermally and adiabatically, flows thro heated tubes and valves to the condenser. Heat loss in the container is reduced to a minim by a high vacuum container, which surrounds the sample container. An oil flow system he the two containers and the tubes. The condensed portion is weighed and the electrical ene is measured. The quotient gives heat of vaporisation. By graphical extrapolation to infi heating energy the influence of the heat losses is eliminated. The temperature range is limi by the thermostate oil near 250°C. The temperature dependence can be taken from a generali curve, following the law of corresponding states which is very well fulfilled in the case of heat of vaporisation. The critical temperature must be known.

1.2·6 — Critical temperature

Direct measurements of the critical temperature were done by observing the vanish of liquid surface in ampoules. The temperature measurements must be made very quickly, ot wise a strong pyrolitic decomposition occurs. After the run the samples were analysed and change in critical temperature by HBR was corrected by a calculation. The heating and cool periods were in the range of 20 seconds respectively, but even during this short time we fo HBR production up to 30%.

2. RESULTS

2.1 — Results obtained at C.E.N.Grenoble

2.1.1 — Density, viscosity and vapour-pressure of irradiated polyphenyls and mixtures

Figures 2-1, 2-2, 2-3, 2-4 give results of measurements of the density and viscosity diphenyl, the terphenyls OMP and OM_2 with HBR obtained by inpile radiolysis. OMP a OM_2, from PROGIL (France), had the following composition :

OMP	*o*-terphenyl	12.5%
	m-terphenyl	63 %
	p-terphenyl	24.5%
OM_2	*o*-terphenyl	20.5%
	m-terphenyl	76 %
	p-terphenyl	3.5%

OM_2 with 35.6 HBR was produced by radiolysis at a dose of 26.4 W h/g and a temperat of 380°C.

The products of 10, 20 and 30% HBR were obtained by dilution of pure OM_2 in 35.6% product.

The curves of the vapour pressure are presented in fig. 2-5·

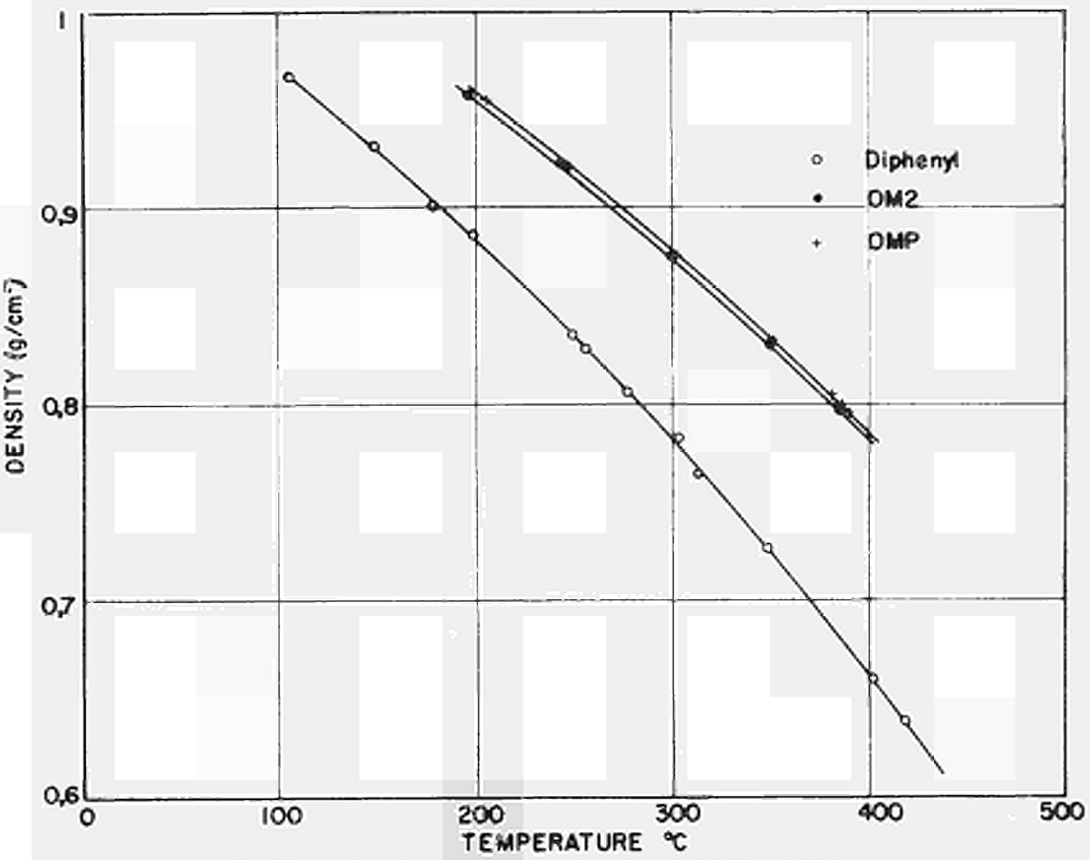


Fig. 2-1 — Density of diphenyl and terphenyls OMP and OM_2

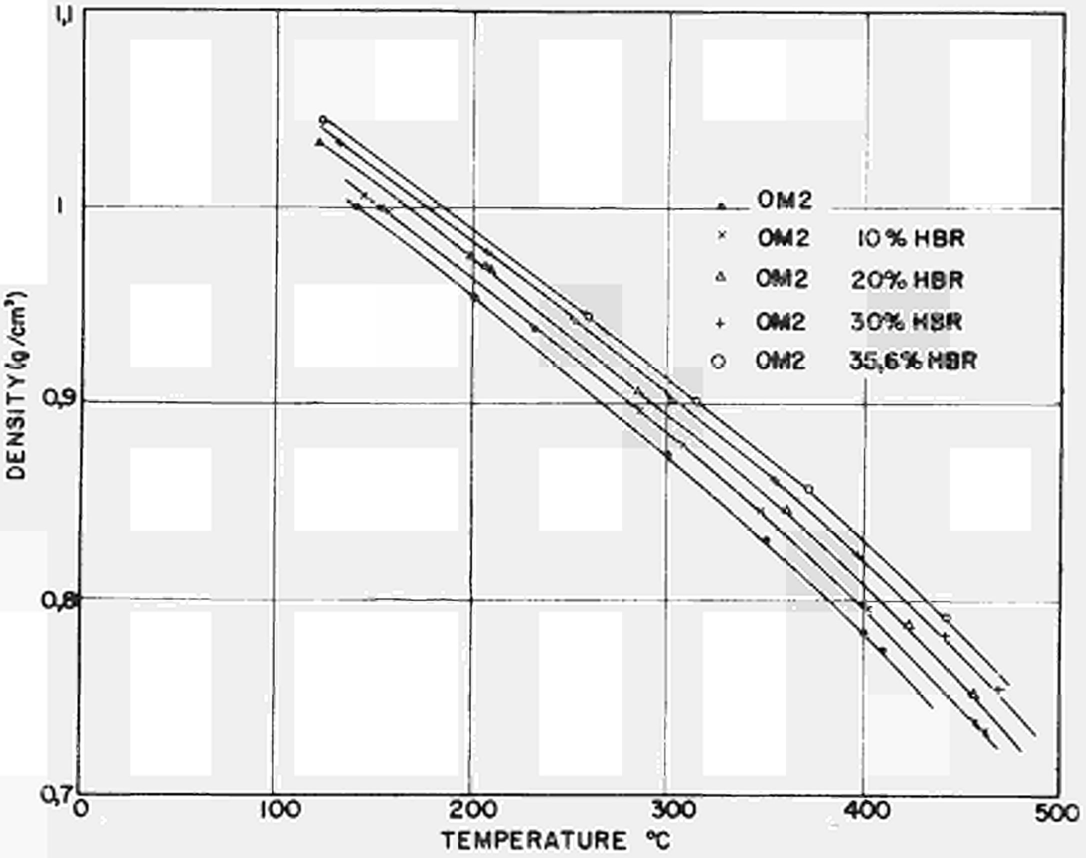


Fig. 2-2 — Density of terphenyl OM_2 with HBR

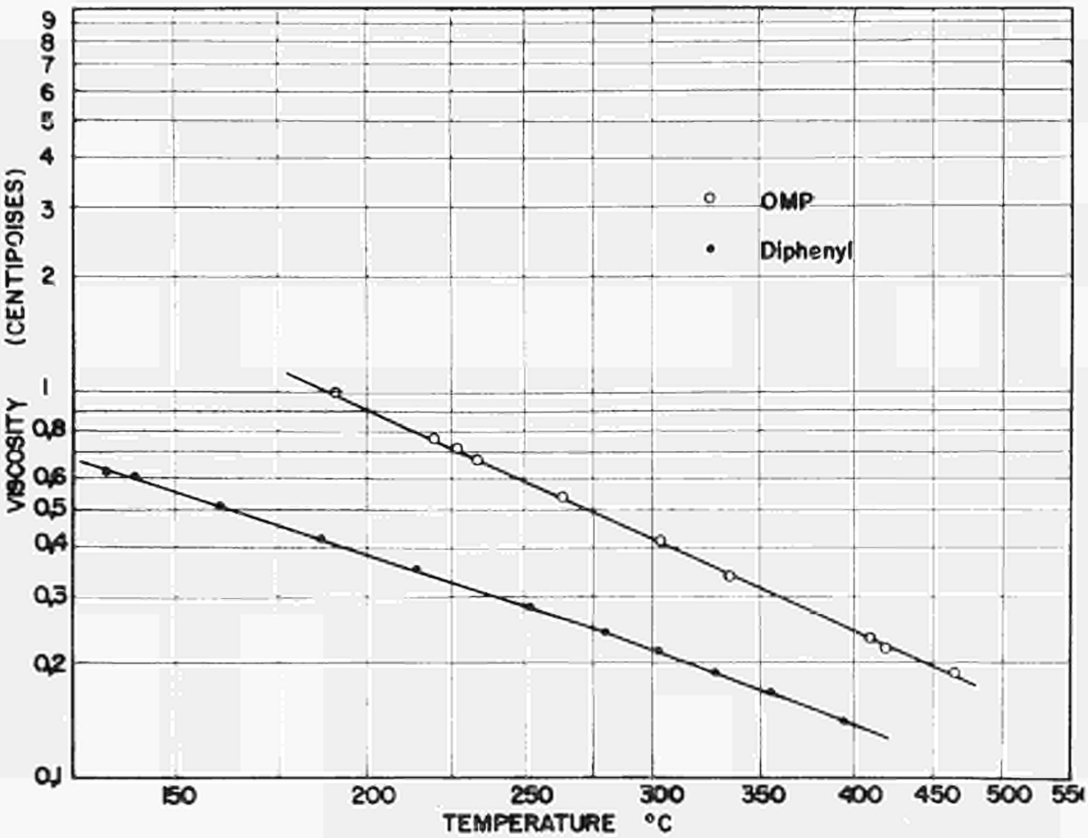


Fig. 2-3 — Viscosity of diphenyl and terphenyl OMP

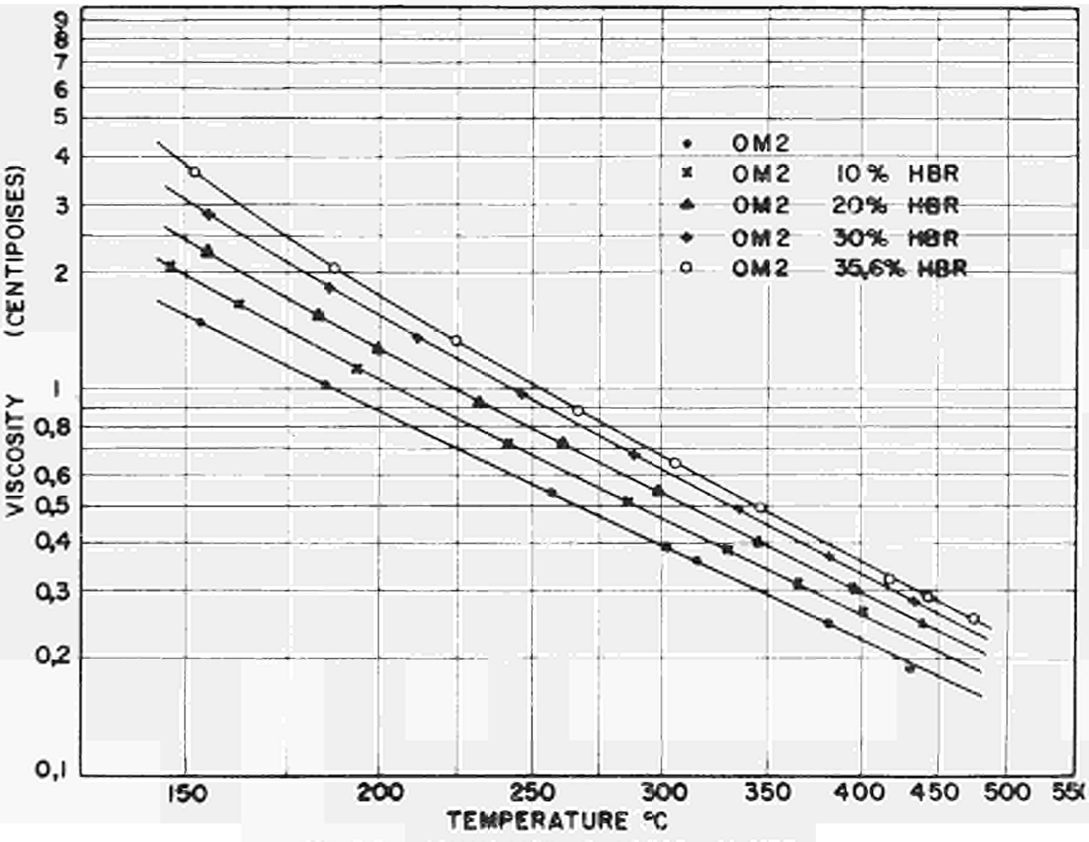


Fig. 2-4 — Viscosity of terphenyl OM_2 with HBR

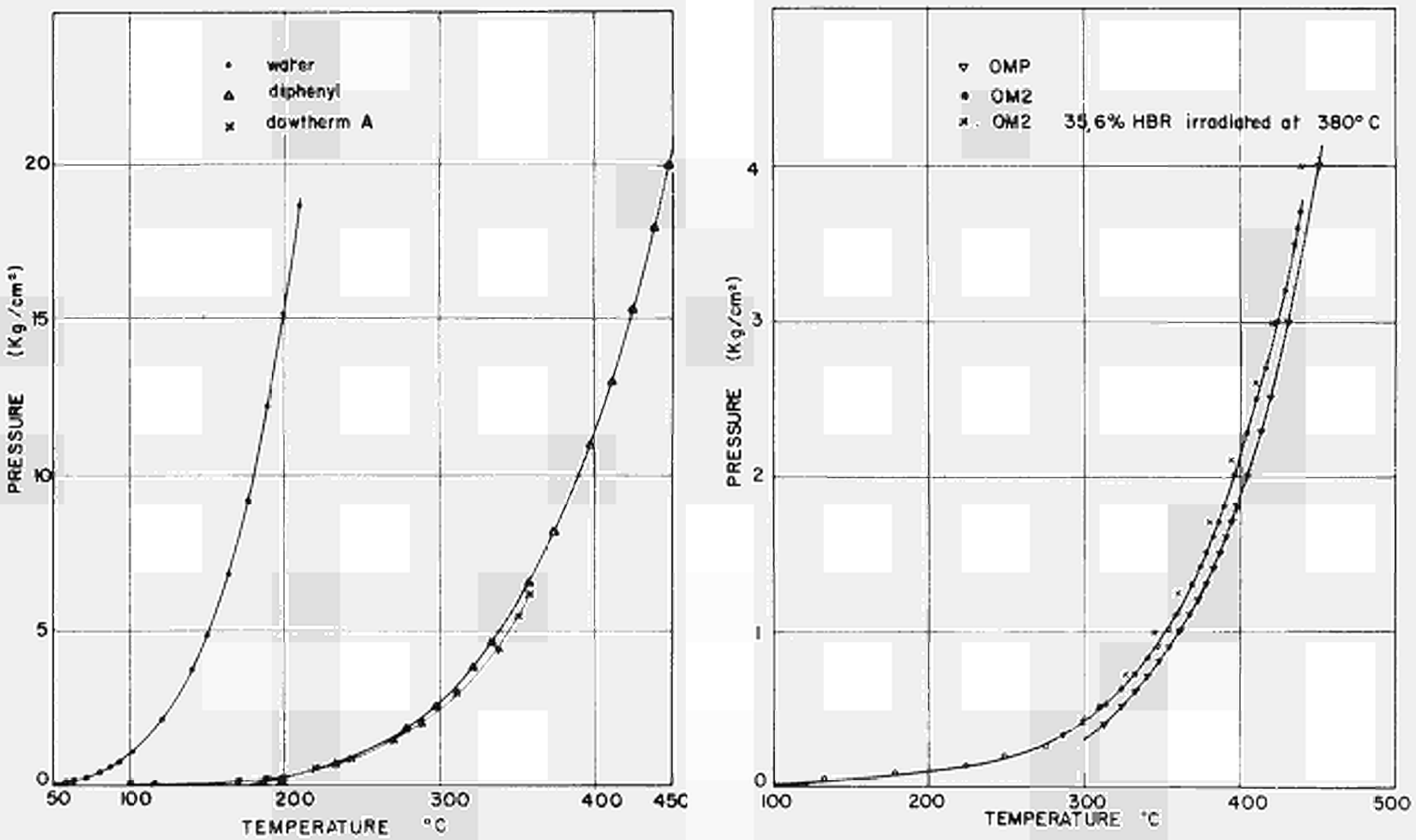


Fig. 2-5 — Vapour pressure measurements

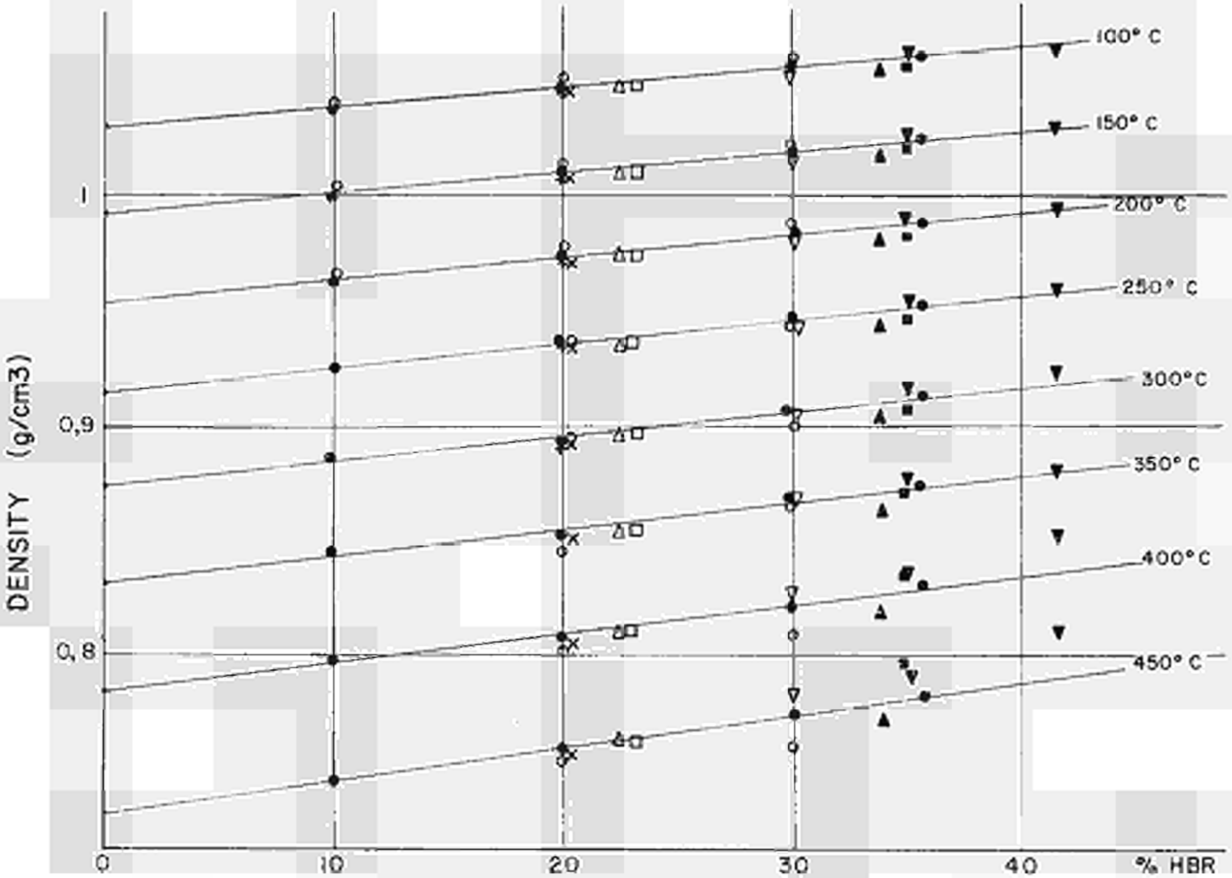


Fig. 2-6 — Density of irradiated terphenyl OM_2

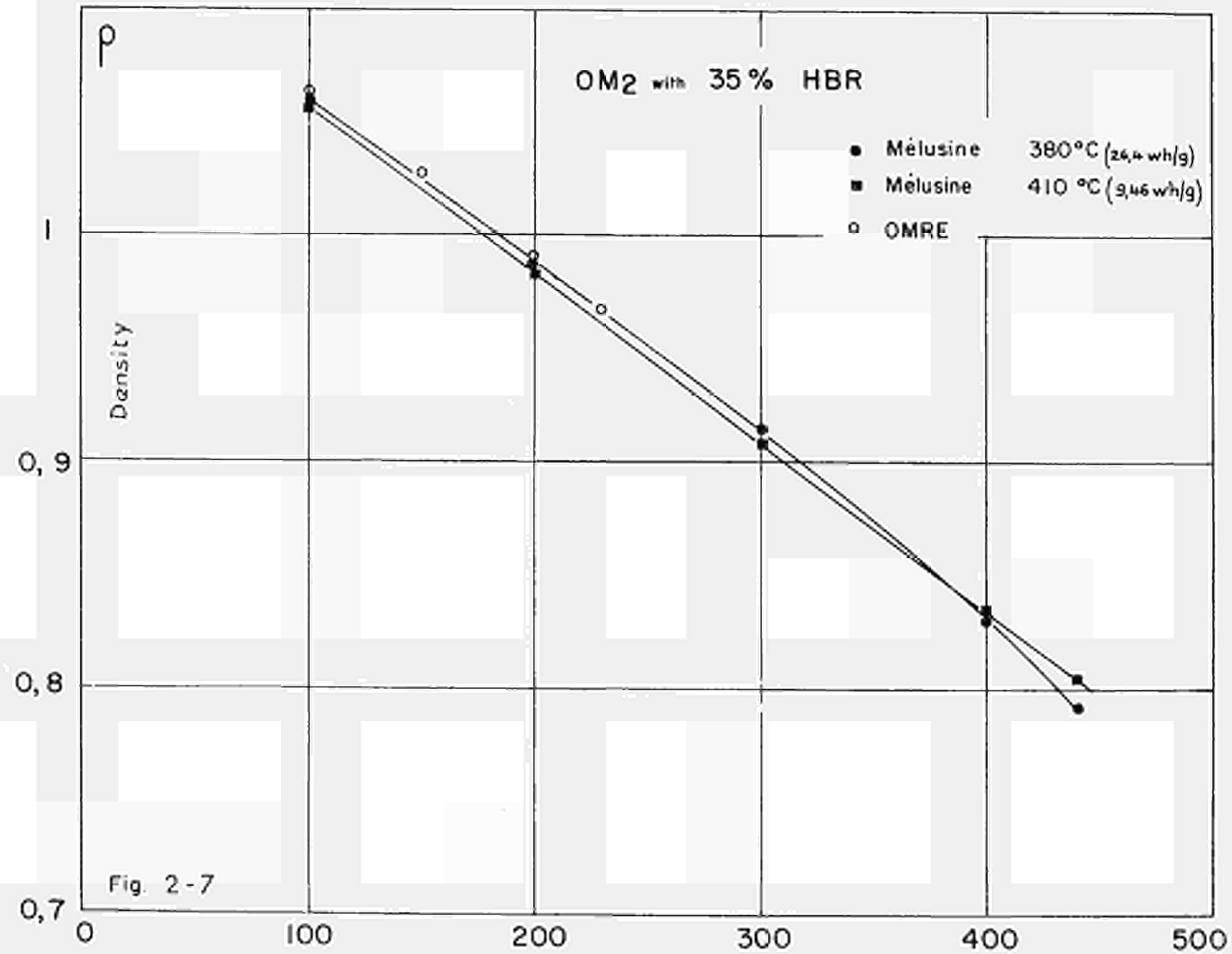


Fig. 2-7 — Density of irradiated terphenyl OM_2 with 35% HBR

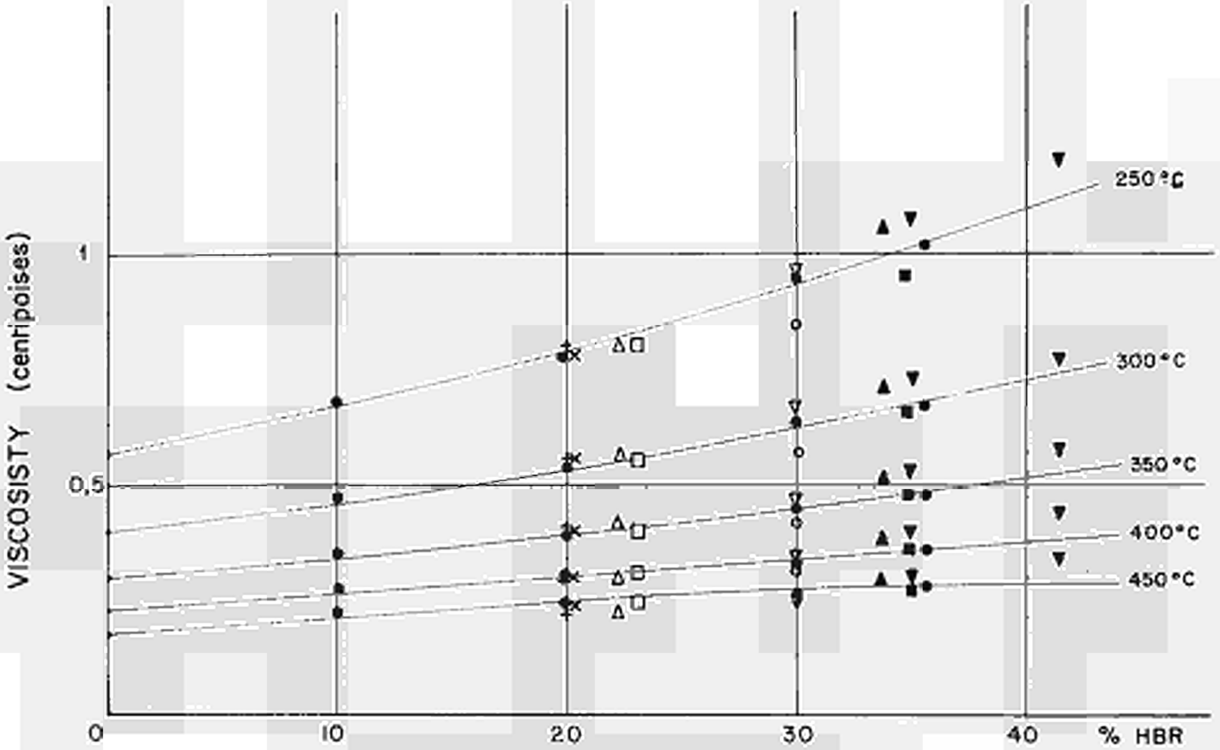


Fig. 2-8 — Viscosity of irradiated terphenyl OM$_2$

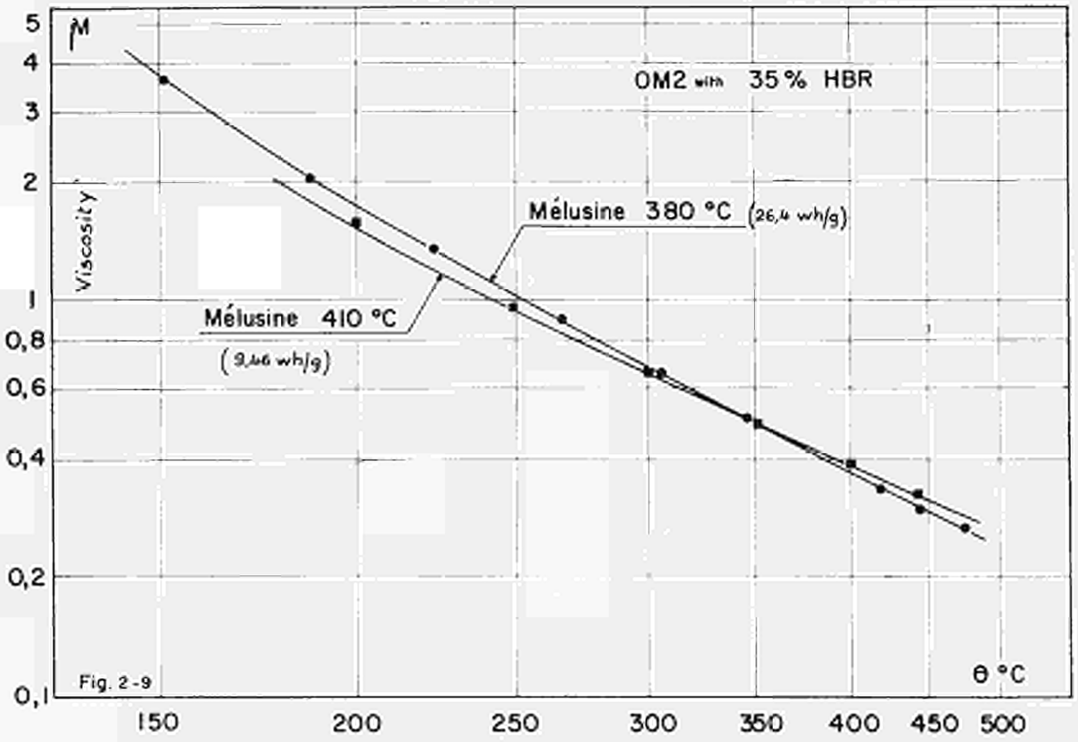


Fig. 2-9 — Viscosity of irradiated terphenyl OM$_2$ with 35% HBR

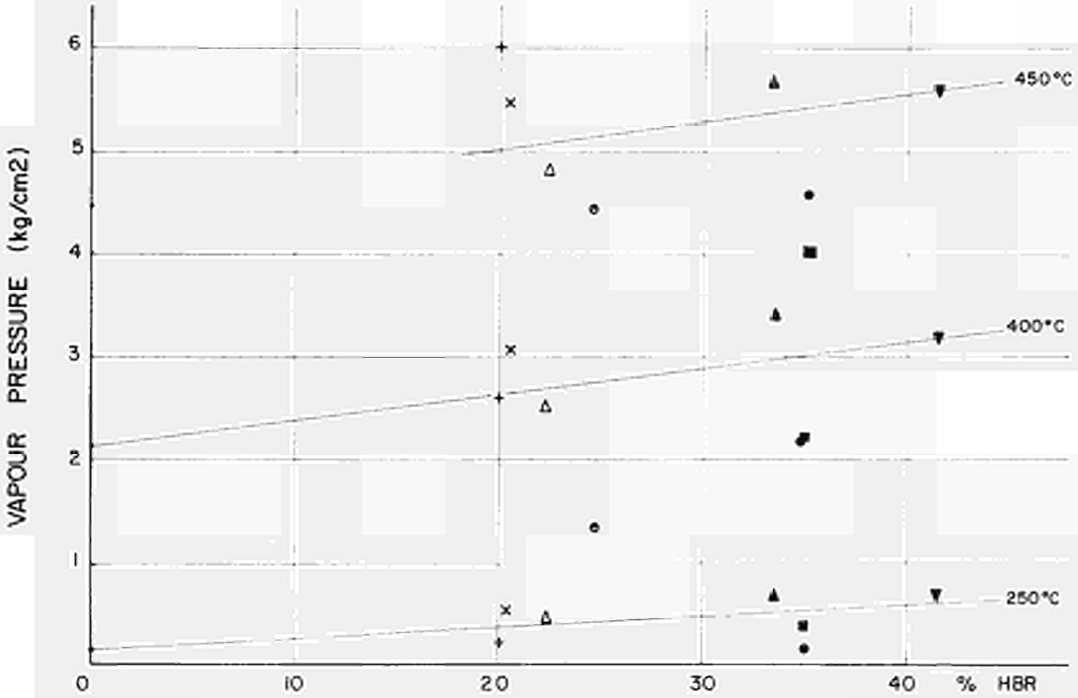


Fig. 2-10 — Vapour pressure of irradiated terphenyl OM$_2$

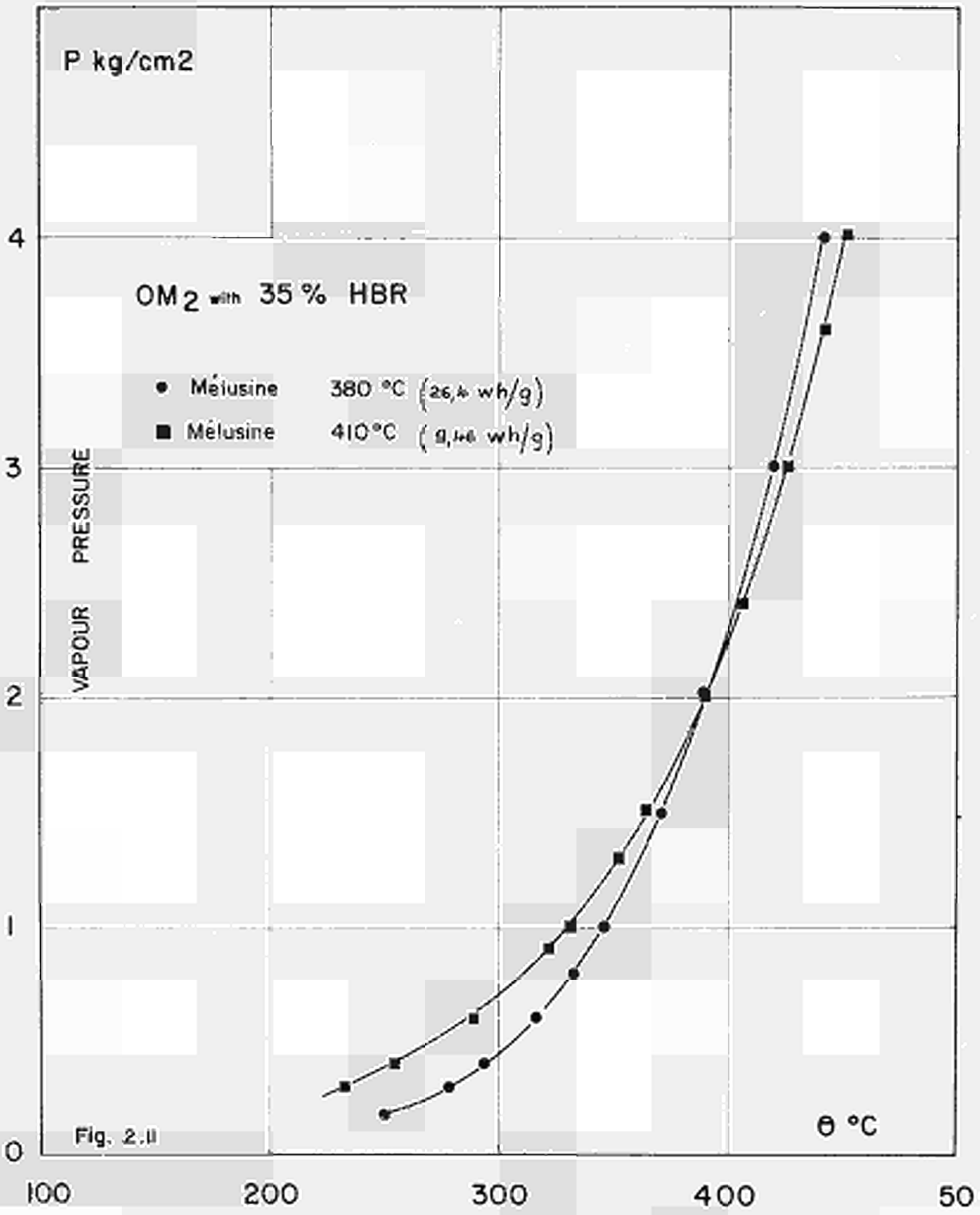


Fig. 2-11 — Vapour pressure of irradiated terphenyl OM_2 with 35% HBR

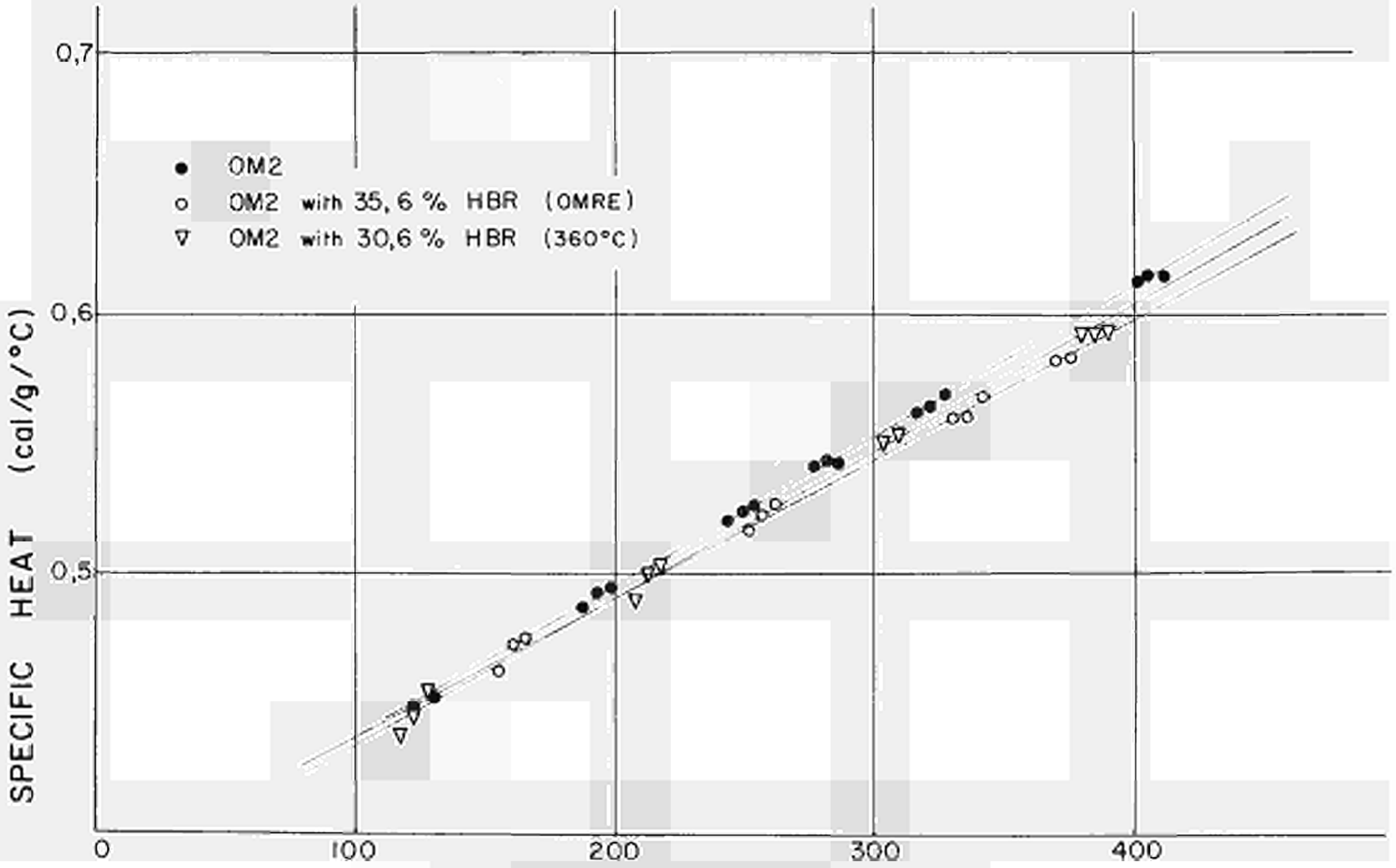


Fig. 2-12 — Specific heat of irradiated terphenyl OM$_2$

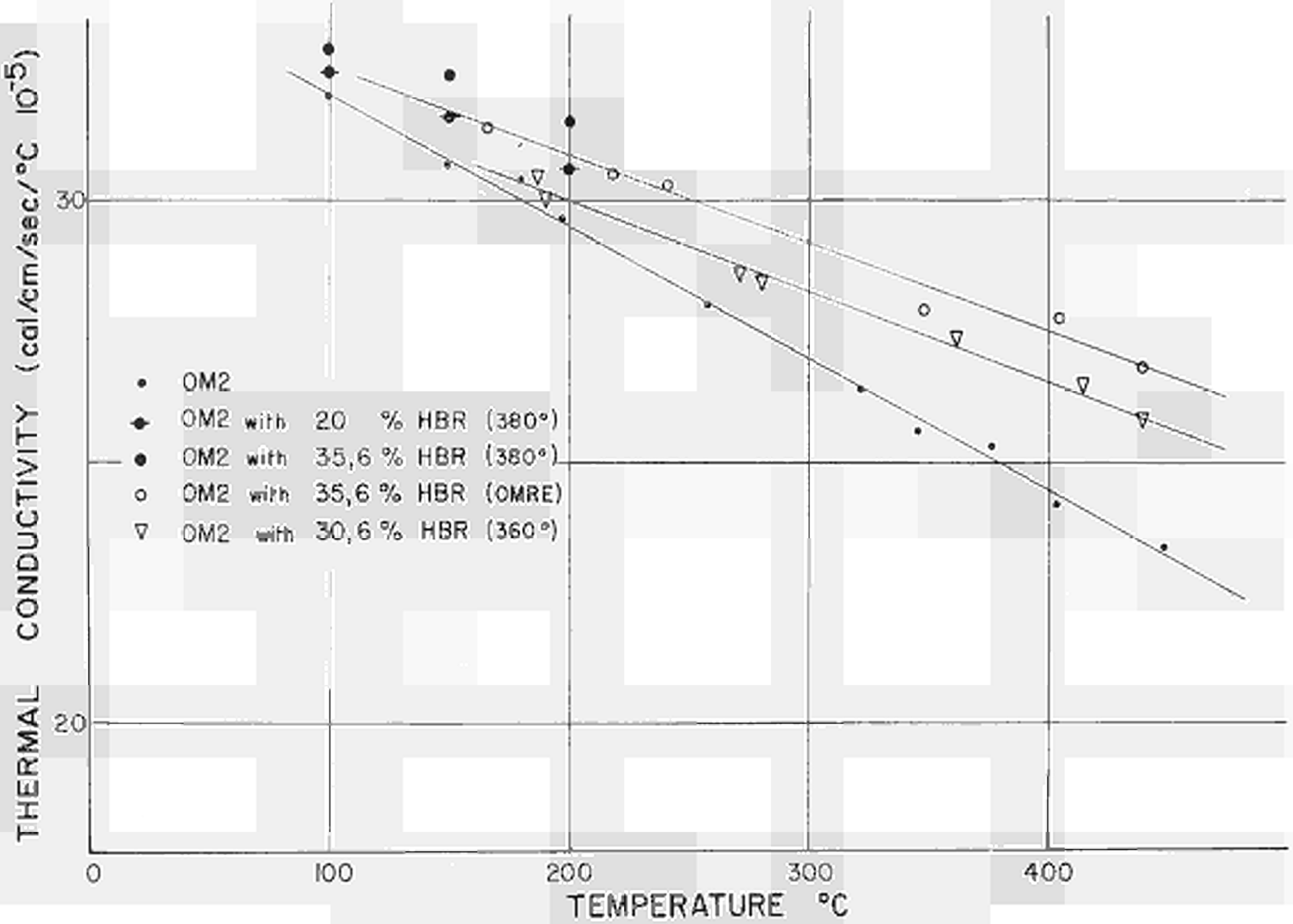


Fig. 2-13 — Thermal conductivity of irradiated terphenyl OM_2

The numerous measurements of viscosity, density and vapour pressure with irradiated terphenyl OM_3 at different temperatures demonstrate that although the samples have the same HBR content the properties could differ remarkably. It can be concluded that, besides the HBR-influence, the properties depend also on the "history" of producing the HBR (HBR product at different temperatures and different radiation doses). The vapour pressure is much more influenced than density and viscosity.

Table 2-14 presents a list of the characteristics of the samples and the curves 2-6 to 2-11 show the results.

The products were irradiated in an inpile loop placed in the reactor Melusine (The laboratories of the "Institut Français du Pétrole" were in charge of this loop).

2.1.2 — Specific heat and thermal conductivity

Measurements with the calorimeter and the apparatus for the thermal conductivity started some months ago.

The specific heat seems to be the least sensitive property concerning HBR. Fig. 2-12 presents the results obtained with pure OM_2 and (at different temperatures) irradiated OM_2.

Fig. 2-13 presents the variation of the thermal conductivity curves with the HBR.

2.2 — Results obtained at C.C.R. Ipsra

2.2.1 — Density

The densities of the pure diphenyl and terphenyls and some mixtures were determined up to 420°C.

A program for high-boilers started with two quinquaphenyls and an hexaphenyl. The results of these measurements are presented in diagram (2-14). A certain error can be caused by the vapour correction because vapour density is unknown. It will however be not too big because boiling point lies in the range of 400°C.

TABLE 2-14

Tested samples of irradiated OM_2 terphenyl

% H.B.R.	Irradiation Temperature °C	Dose Wh/g	Composition				Remarks	Marks on curves
			diphenyl	ortho-terph.	meta-terph.	para-terph.		
0				20,4	76	3,6		-
10-20-30	380	26,4	0,9	11,5	48,5	3,5	10-20 and 30% obtained by dilution	●
35,6								
10-20-30			OM + OMRE H.B.R.				«	○
35,6								
20,27	200	11,74	0,33	19	56,94	3,46		+
20,3	410	5	2,2	13,6	59	4,3		×
22,25	380	8,2	0,4	14	59	4,35		△
23,03	380	9,11	0,9	15,12	57,15	3,8		□
30,62	360	15,35	0,71	14,07	50,93	3,67		▽
33,80	400	11,9	2,7	11,7	47,6	4,20		▲
34,90	410	9,5	2,8	9,7	48,80	3,80		■
35-41,45	380	21	1,29	10,3	43	3,96	35% obtained by dilution	▼
24,45	420	4,87	3,82	16,18	52,07	3,48		◎

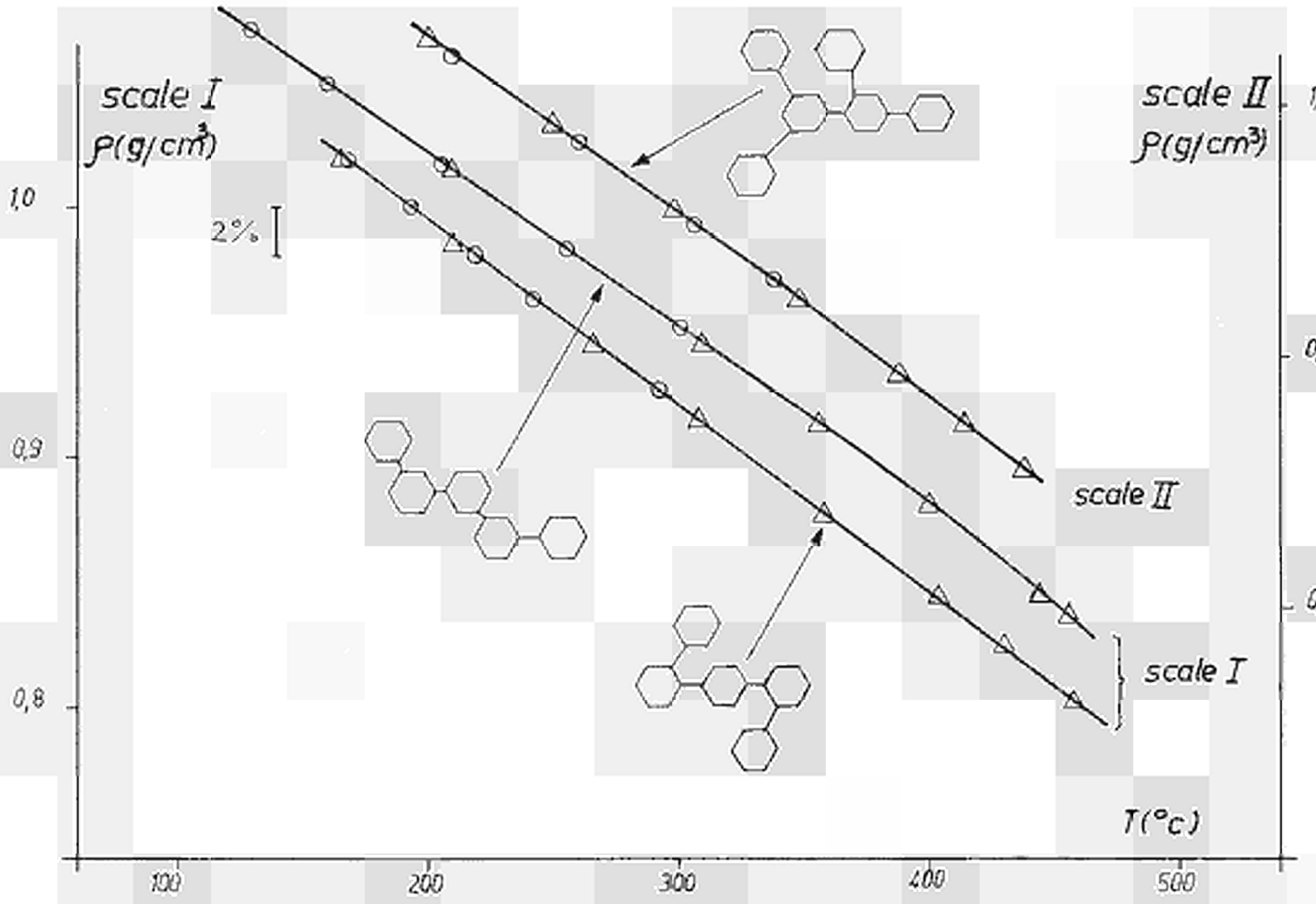


Fig. 2-14 — Density of three polyphenyls

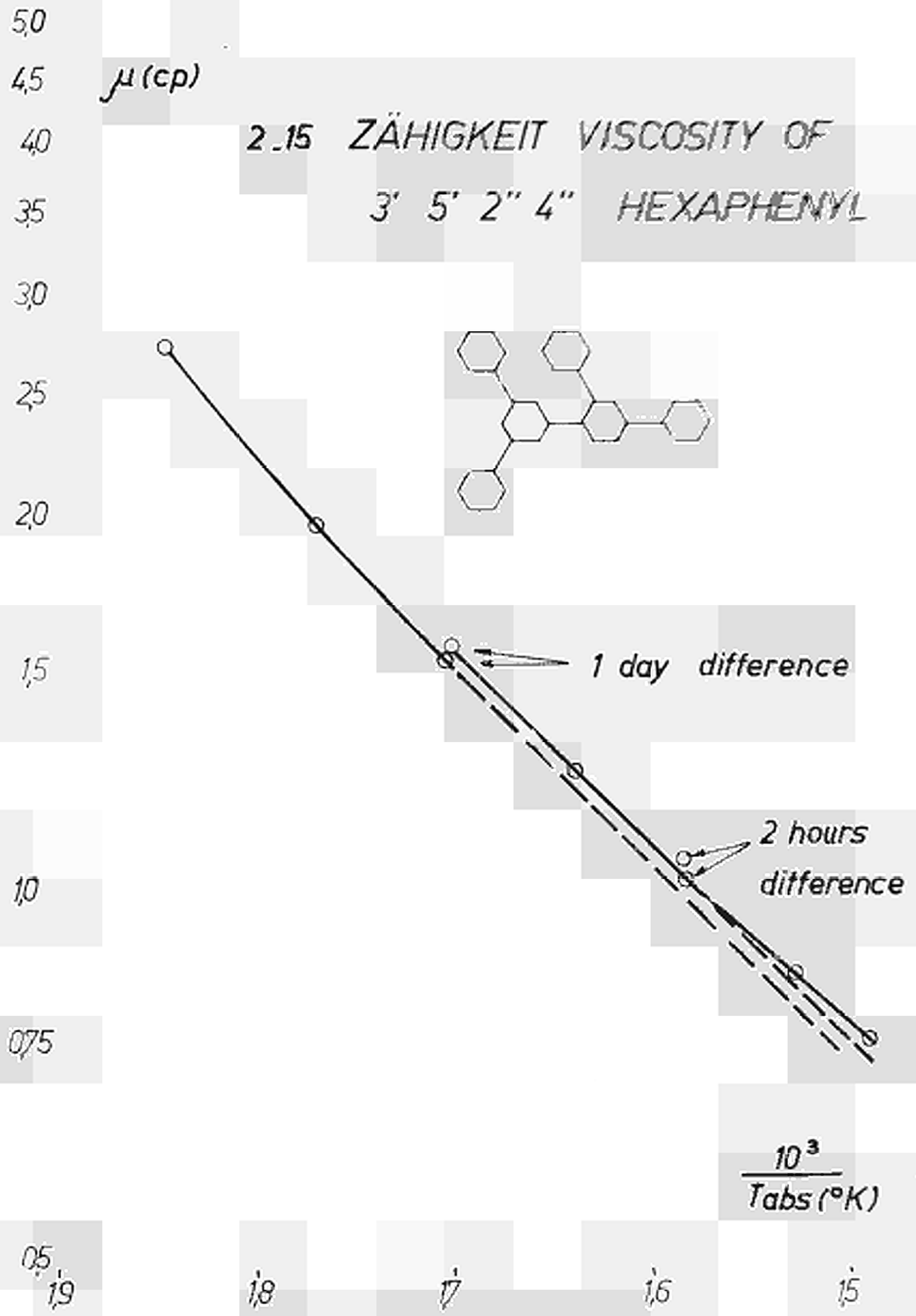


Fig. 2-15 — Viscosity of 3' 5' 2_0 4_0 hexaphenyl

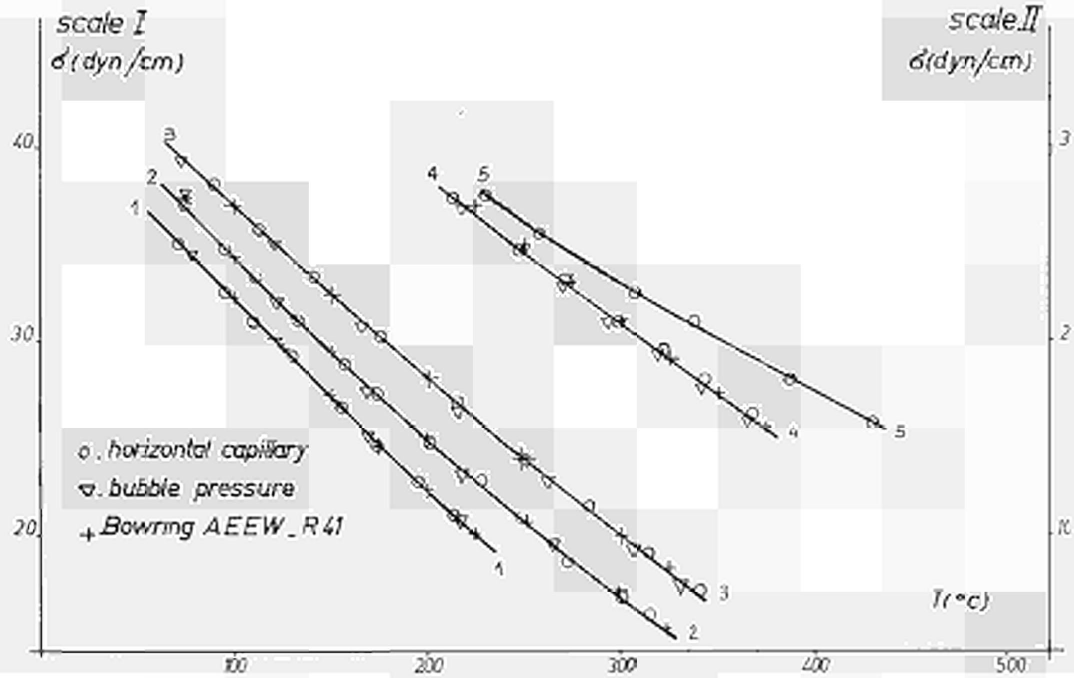


Fig. 2-16 — Surface tension

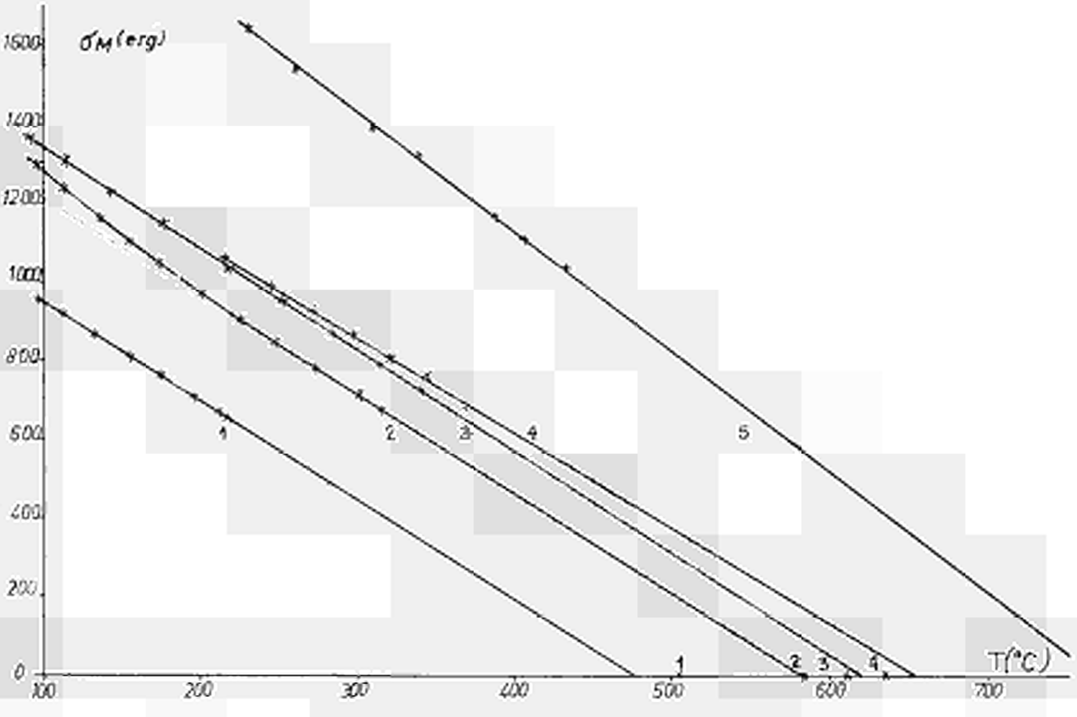


Fig. 2-17 — Molar surface tension

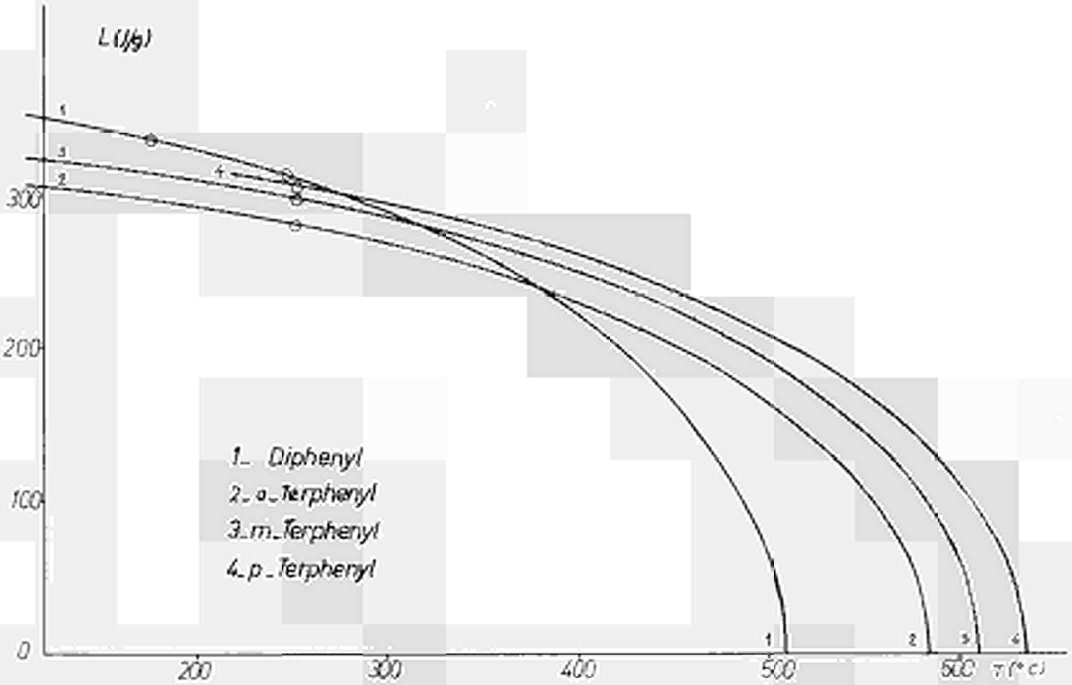
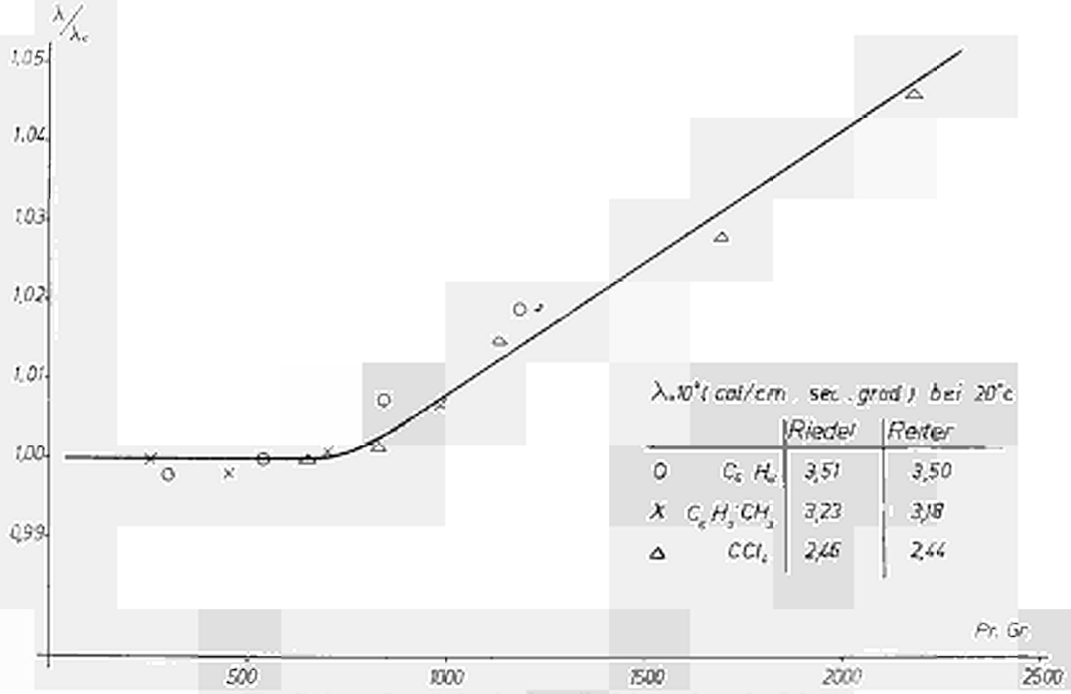


Fig. 2-18 — Latent heat of vaporisation



$\lambda . 10^4$ (cal/cm . sec . grad) bei 20°c

		Riedel	Reiter
O	C_6H_6	3,51	3,50
X	$C_6H_5 \cdot CH_3$	3,23	3,18
△	CCl_4	2,46	2,44

Fig. 2-19 — Convection in the cell of thermal conductivity

2.2.2 — *Viscosity*

Besides the pure substances of diphenyl, terphenyl and mixtures the $3'5'2_0 4_0$-hexaph was measured (see fig. 2-15). At present a program for the binary system of pure *m*-terph with a quaterphenyl was started to obtain the mixing rule. The results will be reported.

2.2.3 — *Surface-tension*

The results for the pure substances (diphenyl and terphenyl) agreed well with meas ments in Winfrith (Bowring et al.-AEEW-R41). With the bubble-pressure method was veri that the liquids are really full wetting. The molar surface energy has the theoretical linear de dence from the temperature (constant EÖTVÖS-coefficient) except the *o*-terphenyl which an Eötvös-coefficient at 100 °C of 3.1 erg/°C and 2.48 erg/°C at 300 °C. Due to the fact viscosity and melting behaviour of *o*-terphenyl show also irregularities, it may probably expected that there is some kind of anisotropy of the liquid state. The diagram of the m surface energy for a hexaphenyl allows to make a rough determination of the critical temperat See fig. 2-16 and 2-17.

2.2.4 — *Latent heat of vaporisation and critical data*

The measurements for diphenyl and the three terphenyls were done in the range of 25C The temperature dependence can be calculated with a generalized curve which is valid for a of liquids of very different classes (included polar liquids) if critical temperature is known. fig. 2-18 presents the results.

2.2.5 — *Thermal conductivity*

The measuring program started in this week with diphenyl so that only a few data v obtained. For Toluene, Benzene and Carbone tetrachloride the values are presented in func of the product Gr.Pr and the data are compared with those from Riedel obtained with flat p and concentric cylinders. See fig. 2-19.

CONCLUSION

Terphenyl OM_2 with different high-boiler content was studied up to temperature 450 °C.

The main results were :

1. Concerning to the HBR-influence we can make a series : viscosity (strong influen thermal conductivity (medium influence), density and specific heat (small influen
2. Besides the HBR percentage we have a dependence on the "history" of the liq Here the vapour pressure is in the most affected property (important influence low boilers). Also viscosity shows an effect.

Some data for pure high-boilers are presented. New results were obtained with di measurements of the latent heat of vaporisation.

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EUR 400.e

CORRIGENDUM

PHYSICAL PROPERTIES OF ORGANIC NUCLEAR REACTOR COOLANTS

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