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## NUCLEATION BEHAVIOUR OF THE GROWTH SOLUTIONS OF FLUORANTHENE IN 1,2-DICHLOROETHANE

**Abstract:** The nucleation kinetics of fluoranthene in 1,2-dichloroethane solution from which good quality crystals of that hydrocarbon were previously grown have been studied. Measurements of the metastable zone width (MSZW), represented by the maximum undercooling ( $\Delta\Theta_{max}$ ), which were carried out by means of the density, ultrasonic velocity and transparency techniques, have shown the great influence of the detection manner of the crystallisation onset on the determined values of the first precipitation temperatures ( $T_p$ ) as well as on the MSZW values. Moreover, from the assessment of this parameter as a function of cooling rate ( $b$ ), the linear dependence between  $\log b$  and  $\log \Delta\Theta_{max}$  was established. Basing on the nucleation orders ( $m$ ) and the nucleation rate constants ( $k_N$ ) resulting from this assessment, the nucleation rate ( $R_N$ ) and the interfacial tension ( $\gamma_{sl}$ ) values for fluoranthene in the solution at the growth temperature of its crystals have been determined. A good agreement obtained between the  $\gamma_{sl}$  values derived from the MSZW determined by these three techniques may be treated as one of the most interesting results of this study.

### 1. Introduction

The growth and nucleation of large size crystals of polycyclic aromatic hydrocarbons (PAHs) from chlorine derivative solutions are the subject of our previous papers [1-3]. Due to the extended  $\pi$  electron system of constituent molecules, charge carriers and excitons in this class of crystals can be excited

by light in the visible or near UV spectral range and this fact favours their potential applicability as low cost photoconductors [4]. The hydrocarbon fluoranthene ( $C_{16}H_{10}$ ) is usually used as a dopant of molecular crystals, prepared for laser spectroscopy [5].

This study deals with the nucleation studies of fluoranthene in the not yet investigated solution of this hydrocarbon in 1,2-dichloroethane ( $1,2-C_2H_4Cl_2$ ) from which its crystals with typical dimensions of  $32 \times 5 \times 2 \text{ mm}^3$  were grown on spontaneously formed seeds under conditions of constant temperature growth, as described in the previous paper [2]. The experimental investigations concerning the metastability zone width (MSZW) were carried out with the help of the three microprocessor instruments constructed by us for measurement of the density, ultrasonic velocity and transparency of solutions. The data of metastable zone width were then processed in order to determine the expression for the nucleation rate, according to Nyvlt [6], and to calculate the values of the interfacial tension of fluoranthene at the growth temperature of its crystals as well as the radius of the critical nucleus.

## 2. Experimental procedure

### 2.1. Purification and sample preparation

The analytically pure fluoranthene purchased from International Enzymes Limited, England, was the starting material. This material was initially recrystallised from distilled benzene, extensively chromatographed on columns filled with  $SiO_2$  and  $Al_2O_3$ , and sublimed under vacuum. Such prepurified material was then zone refined with the help of a multistage zone refiner with stirring [7] (two-fold passage of 100 molten zones with the rate of 1 cm/h in the first, and 0.5 cm/h in the second stage) and the material collected from the central and upper parts of the zone-melted ingots was used in the further studies. Gas chromatographic analyses (GC-FID and GC-MS) performed under conditions given in the previous paper [2] have shown that the total impurity content in such purified material was  $\leq 10^{-3}$  mass %.

The 1,2-dichloroethane ( $1,2-C_2H_4Cl_2$ ) solvent, used without further purification was supplied by Fluka, and had a guaranteed purity of  $\geq 99.5$  mass %. In all experiments, solutions of five different concentrations, from 0.1709 to 0.1820 mole fract., were used. The solutions were prepared in a tightly closed flask using an electronic balance with an accuracy of  $\pm 5 \cdot 10^{-8}$  kg. To ensure the complete dissolution of fluoranthene, they were agitated at the temperature of a few degrees above the saturation point.

## 2.2. Measurements

The nucleation studies were carried out with specially designed microprocessor instruments, described in detail in our previous paper [8]. According to the vibrating tube method [9] used by us, the density measurements involved determining the vibration periods of the empty ( $t_a$ ), filled with the investigated solution ( $t_x$ ) and filled with three-fold distilled water (conductivity lower than  $1 \mu\text{S}$ ) ( $t_w$ ) small diameter glass tube bent in the shape of the letter U. Then, the density of the investigated solution ( $d_x$ ) was calculated from the equation

$$d_x = \frac{t_x^2 - t_w^2}{K} + d_w, \quad (1)$$

in which the calibration constant  $K$  is expressed as  $K = \frac{t_w^2 - t_a^2}{d_w - d_a}$ , where  $d_w$  and

$d_a$  are the density of water ( $998.2 \text{ kg/m}^3$  at  $293\text{K}$ ) and the air ( $1.2 \text{ kg/m}^3$  at  $293\text{K}$ ), respectively.

The measurements of ultrasonic velocity involved determining the averaging travel time ( $t$ ) of the acoustic signal of  $1\text{-}10\text{MHz}$  through the solution sample placed in a vessel of  $5 \text{ cm}^3$  between the two  $\text{PbZrO}_3$  piezoelectric transducers. Knowing the acoustic path ( $l$ ) of the vessel measured as the travel time for three-fold distilled water used as a pattern, the velocity of ultrasonic waves propagation ( $u$ ) in the solution was determined as a quotient  $l/t$ .

The data processing sets installed in these two microprocessor instruments enabled measurements of temperature, density and ultrasonic velocity of the investigated solution with accuracy  $\pm 10^{-2} \text{ K}$ ,  $\pm 10^{-2} \text{ kg/m}^3$  and  $\pm 2 \cdot 10^{-2} \text{ m/s}$ , respectively.

The operation principle of the apparatus used in the transparency investigations involved the measurement of the photodiode potential which was generated by the light beam of  $680\text{-}800\text{nm}$  incident on it after passing through the sample of solution, placed in a glass cuvette. A magnetic stirrer was used for the agitation of the sample. In the apparatus used the potential was measured with accuracy of  $\pm 10^{-2} \text{ mV}$ , while the temperature with the precision of  $\pm 10^{-2} \text{ K}$ .

A Peltier thermostat used in all three instruments enabled the achievement of continuous linear cooling at a few different rates in the temperature range between 288 and 313 K. Therefore, it was possible to perform nucleation experiments under conditions of linear cooling of solutions with the five different rates equal to 0.072, 0.090, 0.114, 0.132 and 0.150 K/min. Under these conditions, monitoring the onset of crystallisation by changes in the solution density, ultrasonic velocity and transparency, the temperatures corresponding to the maximum undercooling  $\Delta\Theta_{max}$  achievable before precipitation occurred in them, have been determined. The values of  $\Delta\Theta_{max}$ , which is usually taken as a measure of the MSZW, were assessed as the differences between the solute solubility ( $T_s$ ) and first precipitation ( $T_p$ ) temperatures. Each experiment was performed 5-10 times to ensure reproducibility of the measured values of the first precipitation temperature. The solubility temperature of fluoranthene was measured using the synthetic-dynamic method described in a previous paper [10]. Reproducibility of the measurements was  $\pm 3$  K corresponding to a relative error in compositions  $< 1\%$ .

### 3. Results and discussion

As mentioned in the experimental section, in all three methods used in this study, the nucleation experiments were carried out for five different concentrations in the range between 0.1709 and 0.1820 mole fract. under conditions of the linear cooling with the five different rates equal to 0.072, 0.090, 0.114, 0.132 and 0.150 K/min. The first precipitation temperatures ( $T_p$ ) and the values of the MSZW (expressed as the difference between the solubility ( $T_s$ ) and  $T_p$  temperatures) determined for the five above cooling rates are listed in Table 1.

Table 1

Values of the saturation ( $T_s$ ) and first precipitation ( $T_p$ ) temperatures and the metastable zone width for solutions of fluoranthene in 1,2-dichloroethane

$b$ [K·min <sup>-1</sup> ]	Density		Ultrasonic velocity			Transparency	
	$T_s$ [K]	$T_p$ [K]	$\Delta\Theta_{\max(d)}$ [K]	$T_p$ [K]	$\Delta\Theta_{\max(u)}$ [K]	$T_p$ [K]	$\Delta\Theta_{\max(t)}$ [K]
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
$x_s = 0.1709$							
0.072	309.76	294.20	15.56	295.57	14.19	298.96	10.80
0.090	309.76	293.49	16.27	294.91	14.85	298.48	11.28
0.114	309.76	292.70	17.06	294.15	15.61	297.89	11.87
0.132	309.76	292.14	17.62	293.66	16.10	297.47	12.29
0.150	309.76	291.68	18.08	293.23	16.53	297.10	12.66
$x_s = 0.1737$							
0.072	310.31	295.21	15.10	296.37	13.94	299.62	10.69
0.090	310.31	294.50	15.81	295.72	14.59	299.14	11.17
0.114	310.31	293.69	16.62	294.96	15.35	298.53	11.78
0.132	310.31	293.17	17.14	294.46	15.85	298.12	12.19
0.150	310.31	292.69	17.62	294.01	16.30	297.74	12.57
$x = 0.1765$							
0.072	310.85	296.21	14.64	297.18	13.67	300.28	10.57
0.090	310.85	295.50	15.35	296.48	14.37	299.79	11.06
0.114	310.85	294.70	16.15	295.71	15.14	299.19	11.66
0.132	310.85	294.17	16.68	295.23	15.62	298.76	12.09
0.150	310.85	293.69	17.16	294.78	16.07	298.36	12.49
$x = 0.1793$							
0.072	311.38	297.21	14.17	298.06	13.32	300.92	10.46
0.090	311.38	296.49	14.89	297.35	14.03	300.43	10.95
0.114	311.38	295.71	15.67	296.58	14.80	299.81	11.57
0.132	311.38	295.16	16.22	296.08	15.30	299.40	11.98
0.150	311.38	294.68	16.70	295.65	15.73	298.99	12.39
$x = 0.1820$							
0.072	311.90	298.17	13.73	298.73	13.17	301.56	10.34
0.090	311.90	297.46	14.44	298.05	13.85	301.05	10.85
0.114	311.90	296.65	15.25	297.26	14.64	300.42	11.48
0.132	311.90	296.10	15.80	296.75	15.15	299.98	11.92
0.150	311.90	295.64	16.26	296.29	15.61	299.60	12.30

This table shows that the first precipitation temperatures determined for each cooling rate used increase with the solution concentration while the width of the metastable zone ( $\Delta\Theta_{\max}$ ) decreases.

It can also be seen from this table that the values of  $\Delta\Theta_{\max}$  assessed from the  $T_p$  temperatures for all the five solution concentrations differ significantly between each other. Depending on the used cooling rate, the mean values of  $\Delta\Theta_{\max}$  changed in the investigated range of solution concentration from 14.64 to 17.16, 13.66 to 16.05 and from 10.57 to 12.48 for the density, ultrasonic velocity and transparency methods, respectively.

The differences in the above values of  $\Delta\Theta_{\max}$  indicate that the detection manner of the onset of crystallisation strongly influences the assessment of the MSZW. It was proved that the most sensitive method is the one based on the detection of this onset of crystallisation by the change in the transparency of the solution observed as a rapid decrease of the photodiode potential ( $U$ ).

According to the empirical relation proposed by Nyvlt [6], nucleation kinetics is characterised by relationship between the maximum allowable supersaturation  $\Delta x_{\max}$  (*i.e.* MSZW) and the mass nucleation rate  $R_N$  in the form:

$$R_N = k_N \Delta x_{\max}^m, \quad (2)$$

where the exponent  $m$  is referred to as the apparent order of nucleation and  $k_N$  is the nucleation rate constant.

The relationship between the maximum allowable supersaturation  $\Delta x_{\max}$  and maximum allowable undercooling is expressed as:

$$\Delta x_{\max} = (dx^*/dT)\Delta\Theta_{\max}, \quad (3)$$

where  $dx^*/dT$  is the rate of change of solubility with temperature. With respect to nucleation in a solution cooled down at the constant rate, the following relation between the maximum allowable undercooling  $\Delta\Theta_{\max}$  and the cooling rate  $b$  is expressed

$$\log b = (m-1)\log(dx^*/dT) + \log k_N + m\log(\Delta\Theta_{\max}) \quad (4)$$

Thus a plot of  $\log b$  against  $\log(\Delta\Theta_{\max})$  should yield a straight line of a gradient  $m$ . The values of  $\Delta\Theta_{\max}$  obtained for the investigated solutions from the density, ultrasonic velocity and transparency measurements (Table 1) fulfil well the linear relation between  $\log b$  and  $\log(\Delta\Theta_{\max})$  as it is shown in Fig. 1.

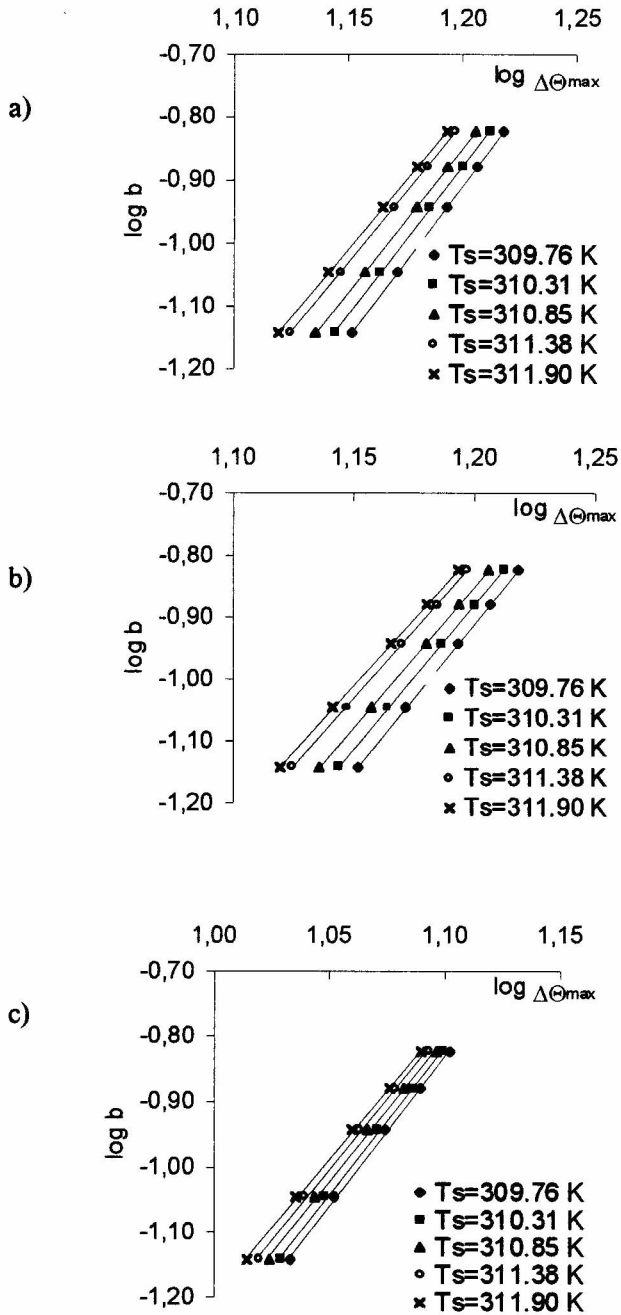


Fig. 1. Plots of  $\log b$  versus  $\log \Delta\Theta_{\max}$  from the density (a), ultrasonic velocity (b) and transparency (c) measurements

Analysing the data listed in Table 1 and looking at Figs. 1a-c, it can be noticed that the MSZW of the solutions increases with the cooling rate for all the experimental conditions explored. The values of the order of nucleation rate ( $m$ ) and the constant of nucleation rate ( $k_N$ ) of Eq. 2, based on empirical nucleation rate were evaluated by regression analysis from the slopes and intercepts found in Fig. 1 and shown in Table 2.

Table 2.  
Values of the nucleation parameters of eq. 2.

$T_s$ [K]	Density		Ultrasonic velocity		Transparency	
	$m_{(d)}$	$k_{N(d)}$	$m_{(u)}$	$k_{N(u)}$	$m_{(t)}$	$k_{N(t)}$
309.76	4.878	80.46	4.795	101.42	4.600	216.50
310.31	4.756	68.21	4.684	82.84	4.511	175.36
310.85	4.624	56.22	4.546	62.46	4.393	131.00
311.38	4.492	44.46	4.409	48.75	4.332	114.93
311.90	4.324	34.25	4.310	39.52	4.205	83.01

The values of  $m$  and  $k_N$  are in the range from 4.324 to 4.878, 4.310 to 4.795 and 4.205 to 4.600, and from 34.25 to 80.46, 39.52 to 101.42 and 83.01 to 216.50, respectively, in the case of the density, ultrasonic velocity and transparency measurements. The data obtained also indicate that the values of both these parameters appear to be dependent on the saturation temperature.

Allowing that the exponential decreasing of the induction time with the increase in supersaturation of fluoranthene-1,2- $C_2H_4Cl_2$  solutions was previously observed [2], as could be expected from the classical nucleation theory, values of the solid-liquid interfacial tension ( $\gamma_{sl}$ ) for fluoranthene in this solution were determined from plots  $\ln R_N$ , defined by Eq.2 versus  $(\ln S)^{-2}$ . The supersaturation  $S$  used by us was expressed in the form of the ratio of the equilibrium mole fraction  $x_s$  of fluoranthene at a given temperature to that ( $x_G$ ) at the temperature of 307.5 K, *i.e.* at the temperature at which the crystals of this hydrocarbon were previously grown [2]. The data of  $\ln R_N$  versus  $(\ln S)^{-2}$ , plotted in Fig. 2a-c can be well fitted by means of the straight lines expressed as :

$$\ln R_N = -11.79 - 0.0280 (\ln S)^{-2}, \quad (5)$$

$$\ln R_N = -11.56 - 0.0258 (\ln S)^{-2}, \quad (6)$$

$$\ln R_N = -10.58 - 0.0226 (\ln S)^{-2}, \quad (7)$$

respectively, in the case of the density, ultrasonic velocity and transparency measurements. The values of the solid-liquid interfacial tension ( $\gamma_{sl}$ ) estimated from the slopes ( $n$ ) of the above lines, which according to the expression for the homogeneous nucleation rate  $J$  [11-14] (Eq. 8)



$$J = A \exp\left(-\frac{4}{27} \cdot \frac{\beta^3 v^2 \gamma_{sl}^3}{\alpha^2 k^3 T^3 (\ln S)^2}\right) \quad (8)$$

may be expressed as

$$n = -\frac{4}{27} \cdot \frac{\beta^3 v^2 \gamma_{sl}^3}{\alpha^2 k^3 T^3}, \quad (9)$$

are listed in Table 3. Here  $v$  is the molecular volume,  $k$  is the Boltzmann constant, and  $\beta$  and  $\alpha$  denote, respectively, the surface and volume shape factors of spherical clusters of the new stable phase, which can form as thermal fluctuations in the metastable phase.

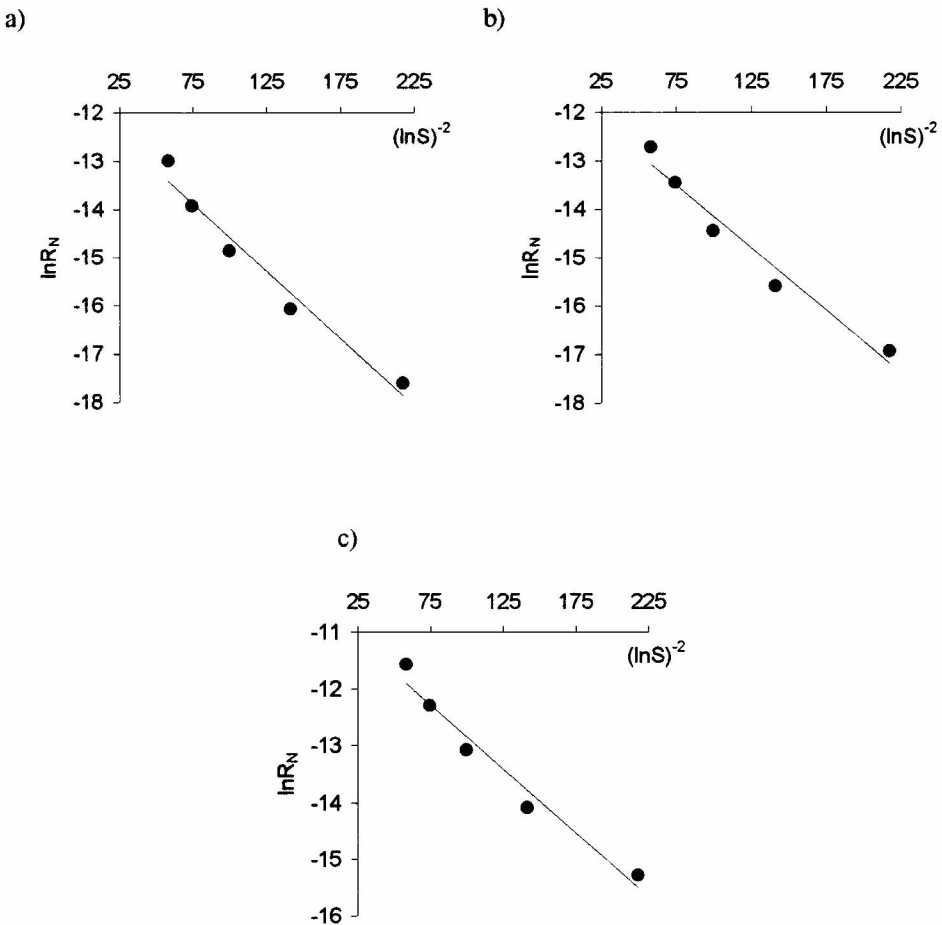


Fig. 2. Plots of  $\ln R_N$  versus  $(\ln S)^{-2}$  from the density (a), ultrasonic velocity (b) and transparency (c) measurements

Table 3

Interfacial tension ( $\gamma_{sl}$ ) and critical radius ( $r$ ) of nuclei for the nucleation of fluoranthene from solutions in 1,2-dichloroethane evaluated at the growth temperature of its crystals

$S$	$\gamma_{sl}$ [ $\text{mJ}\cdot\text{m}^{-2}$ ]			$r$ [ $\text{\AA}$ ]
	Density	Ultrasonic velocity	Transparency	
1.070				20.9
1.088				16.8
1.105	1.22	1.18	1.13	14.2
1.123				12.2
1.140				10.8

In spite of the fact that the values of  $\Delta\Theta_{\max}$  obtained by us from the density, ultrasonic velocity and transparency measurements differ significantly from each other (Table 1), the values of the interfacial tension estimated on the basis of these measurements are practically the same (Table 3). It can also be noticed from the Table 3 that the interfacial tension values are reasonably compatible with those obtained from the measurements of the induction times, the latter being equal to  $1.69 \text{ mJ/m}^2$  [2].

It is well known that the critical radius ( $r$ ) of spherical particles of non-electrolytes depends on the interfacial tension according to the Gibbs-Thompson equation:

$$r = \frac{2v\gamma_{sl}}{kT \ln S} \quad (10)$$

By inserting into Eq. 10 the values of supersaturation  $S$  (defined as above) and the average value of the interfacial tension between those estimated from the density, ultrasonic velocity and transparency measurements, the values of the critical radius of fluoranthene nuclei were estimated (Table 3). The radius of the critical nucleus and the number of molecules in this nucleus, both versus supersaturation ratio  $S$ , are shown in Figs 3a and b, respectively.

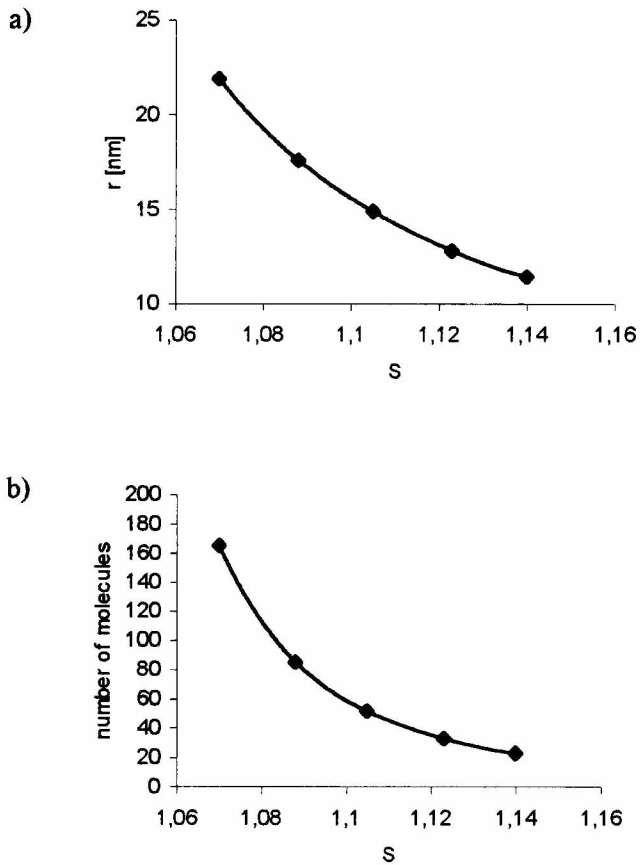


Fig. 3. Critical radius of a nucleus (a) and number of molecules in a nucleus (b) versus supersaturation ratio

The number of molecules in the nucleus was calculated dividing its volume by the molecular volume of fluoranthene; this latter value determined from the crystallographic data [15] is  $266.7 \text{ \AA}^3$ . The table and the figure show that the radius of the critical nucleus ranges from 11.0 to 24.2  $\text{\AA}$  while the number of molecules in these nuclei ranges from 220 at the lowest supersaturation down to just tens at high supersaturations.

#### 4. Conclusions

The nucleation of fluoranthene in 1,2-dichloroethane solutions from which good quality crystals of this hydrocarbon were previously grown [2], has been investigated by the metastability zone width measurements performed with the help of the density, ultrasonic velocity and transparency methods. It was found that the first precipitation temperatures ( $T_p$ ) determined by all these three methods increase with the increase in the solution concentration, while the assessed values of the MSZW represented by the maximum undercooling ( $\Delta\mathcal{O}_{\max}$ ), decrease.

It was also found that the values of both these parameters are strongly dependent on the detection manner of the "nucleation" onset (Table 1). Here, the most sensitive method turned out to be the one based on its detection by the changes in the transparency of solutions. In results of these experiments it was also proved that there is a linear dependence between  $\Delta\mathcal{O}_{\max}$  values and the cooling rate ( $b$ ) (Figs. 1a-c), irrespective of a possibility that the size of the particles detected by all the three methods used in this study could be greater than those of the newly formed nuclei.

The values of the nucleation order ( $m$ ) and the nucleation rate constant ( $k_N$ ) resulting from the linear dependencies obtained from the density, ultrasonic velocity and transparency measurements appear to be dependent on the saturation temperature ( $T_s$ ) (Table 2). From the slopes of the linear relation between the natural logarithmic nucleation rate ( $R_N$ ) determined on the basis of the above  $m$  and  $k_N$  values, and an inverse squared natural logarithm of the supersaturation ratio ( $S$ ) (Fig. 2a-c), the values of the solid-liquid interfacial tension ( $\gamma_{sl}$ ) for fluoranthene in 1,2-dichloroethane solution were estimated at the growth temperature of this hydrocarbon crystals. Such assessed  $\gamma_{sl}$  values are 1.22, 1.18 and 1.13 mJ/m<sup>2</sup> in the case of the density, ultrasonic velocity and transparency measurements, respectively. These values moderately agree with the value of this parameter (i.e. 1.69 mJ/m<sup>2</sup>) derived from the induction time measurements [2].

Due to the relatively low interfacial tension values, the radius of critical nucleus calculated from the Gibbs-Thompson equation (Eq. 10) is also quite small; its range is from 10.80 to 20.9 Å (Table 3, Fig. 3a). This in turn results in the fact that the number of molecules in the critical nucleus estimated as a quotient of its volume and the molecular volume of fluoranthene ranges from 220 at the lowest supersaturation down to just tens at high supersaturations (Fig. 3b).

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### Zachowania nukleacyjne roztworów wzrostowych fluorantenu w 1,2-dichloroetanie

**Streszczenie:** Badano kinetykę zarodkowania fluorantenu w roztworze 1,2-dichloroetanu, z którego otrzymano wcześniej dobrej jakości kryształy tego węglowodoru. Pomiar szerokości obszaru metastabilnego, wyrażonego w postaci maksymalnego przechłodzenia ( $\Delta\Theta_{\max}$ ), które przeprowadzono przy użyciu trzech różnych technik opartych na badaniu gęstości, prędkości rozchodzenia się fal ultradźwiękowych i przezroczystości próbek badanego roztworu, wykazały duży wpływ sposobu detekcji początku krystalizacji fluorantenu, zarówno na wyznaczone w ten sposób wartości temperatur odpowiadających temu początkowi ( $T_p$ ), jak i wartości szerokości obszaru metastabilnego.

Ponadto na podstawie oszacowania wartości  $\Delta\Theta_{\max}$  w funkcji szybkości ochładzania ( $b$ ) roztworu ustalono, że spełniona jest dla niego liniowa zależność pomiędzy  $\log b$  i  $\log \Delta\Theta_{\max}$ . Następnie, opierając się na wynikających z tego oszacowania wartościach rzędu zarodkowania ( $m$ ) oraz stałej szybkości zarodkowania ( $k_N$ ), wyznaczono wartości szybkości zarodkowania ( $R_N$ ) oraz oszacowano na ich podstawie wartości międzyfazowego napięcia powierzchniowego ( $\gamma_{sl}$ ) fluorantenu w temperaturze wzrostu jego kryształów. Dobra zgodność, którą uzyskano pomiędzy wartościami  $\gamma_{sl}$  wyznaczonymi na podstawie szerokości obszaru metastabilnego, zmierzonego przy użyciu trzech różnych technik pomiarowych, może być traktowana jako jeden z najbardziej interesujących wyników przeprowadzonych przez nas badań.