

# SURFACE TENSION OF ALKANES SATURATED WITH HELIUM OR HYDROGEN

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*The differential capillary method has been used to measure the capillary constant  $a^2$  and to determine the surface tension  $\sigma$  of alkanes (methane, ethane, propane) saturated with helium or hydrogen. Experiments have been made in temperature ranges from the triple-point temperature to temperatures close to the solvent critical point at pressures from that of saturated vapors of a pure liquid to 4 MPa. Equations have been suggested which describe the temperature, baric, and concentration dependences of  $a^2$  and  $\sigma$  of solutions. The data obtained have been used to calculate the relative adsorption. The results of measurements are discussed in the framework of thermodynamic models of the theory of surface phenomena.*

**KEY WORDS:** capillary constant, surface tension, gas solutions in liquid, adsorption, differential capillary method

## 1. INTRODUCTION

Surface phenomena play an important role at phase transitions. Surface tension determines to a large extent the work of formation of a new-phase nucleus (Skripov, 1974). Interfaces are responsible for such practically important processes as adsorption, catalysis, flotation, etc. (Jaycock and Parfitt, 1981). Besides, some processes that take place in bulk are initiated from the surface, and this first stage determines in many respects their further progress (Colinet et al., 2001).

A fundamental property of a liquid–vapor interface, most accessible for measurements, is surface tension. By now the surface tension of pure liquids has been investigated well enough (Baidakov, 1994). However, in nature and engineering, as a rule, one has to deal with solutions. Owing to adsorption, despite the small content of substances dissolved, surface tension in the dependence of the concentration may undergo considerable changes both decreasing and increasing. This paper presents the results of measuring the surface tension of a number of alkanes with small additions of helium or hydrogen. Both helium and hydrogen are in this case surface-active components, and their presence in a two-phase system leads to considerable changes in the properties of a liquid–vapor interface.

The paper has the following structure. The first section is an introduction. The second gives a short presentation of the procedure of measuring surface tension. The results of measurements and their discussion are given in the third section. The fourth section presents a thermodynamic analysis of the data obtained. The fifth is a conclusion.

## 2. PROCEDURE OF MEASUREMENTS

To measure surface tension, use was made of the differential variation of the method of capillary rise (Baidakov, 1994; Baidakov et al., 2012). The measuring cell contained an assembly of three glass capillaries. Two assemblies

### NOMENCLATURE

<p><math>a^2</math> capillary constant</p> <p><math>B</math> second virial coefficient</p> <p><math>b</math> radius of meniscus curvature in a capillary</p> <p><math>g</math> acceleration of gravity</p> <p><math>G</math> Gibbs molar free energy <math>g_{22} = \partial^2 G / \partial x^2</math></p> <p><math>h</math> height of liquid rise in a capillary</p> <p><math>k_B</math> Boltzmann constant</p> <p><math>p</math> pressure</p> <p><math>R</math> gas constant</p> <p><math>r</math> capillary radius</p> <p><math>S</math> area per mole of the surface layer</p> <p><math>T</math> temperature</p> <p><math>v</math> molar volume of vapor</p> <p><math>x</math> mole fraction in percent of the second component</p> <p><b>Greek Symbols</b></p> <p><math>\Gamma</math> relative adsorption</p>	<p><math>\Delta</math> difference</p> <p><math>\varepsilon</math> reduced value of temperature</p> <p><math>\mu_{sol}</math> molecular weight of the solution</p> <p><math>\pi</math> reduced value of pressure</p> <p><math>\rho</math> density</p> <p><math>\sigma</math> surface tension</p> <p><math>\tau</math> reduced value of temperature</p> <p><math>\varepsilon = 1 - \tau</math></p> <p><b>Subscripts</b></p> <p><math>c</math> critical state</p> <p><math>l</math> liquid</p> <p><math>v</math> vapor</p> <p><math>\sigma</math> surface layer</p> <p><math>*</math> pure substance</p> <p><math>sol</math> solution</p> <p><math>mix</math> mixture</p>
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of capillaries with different inner radii were used in the experiments. The capillary radii were determined by calibration with mercury and had the following values:  $r_1 = (0.2310 \pm 0.0003)$  mm,  $r_2 = (0.1253 \pm 0.0005)$  mm,  $r_3 = (0.06508 \pm 0.00009)$  mm (assembly I) and  $r_1 = (0.6393 \pm 0.0005)$  mm,  $r_2 = (0.2297 \pm 0.0003)$  mm,  $r_3 = (0.09607 \pm 0.00008)$  mm (assembly II). The cell was thermostatted ( $\pm 0.005$  K) in an aluminum block, which was located in the vacuum chamber of a cryostat. At temperatures lower than 185 K the cell with the capillaries was cooled with liquid nitrogen. Higher temperatures were achieved by pumping through the cryostat a cooling agent from a low-temperature thermostat FP88–MV (JULABO, Seelbach, Germany). The temperature in the block was measured by a platinum resistance thermometer with an uncertainty no more than  $\pm 0.02$  K.

An experiment began with filling the measuring cell with a solvent. After its condensation and the establishment of equilibrium, the pressure in the cell was determined with the help of a standard spring manometer. The uncertainty of pressure determination did not exceed  $\pm 0.007$  MPa. The height of the liquid rise in a capillary  $h_i$  was measured by a cathetometer with an uncertainty  $\pm 0.02$  mm. The capillary constant  $a^2$  was calculated in the approximation of complete wettability by liquid of the inner walls of the capillaries by the formula

$$a^2 = \Delta h_{ij} / (b_i^{-1} - b_j^{-1}), \quad (1)$$

where  $\Delta h_{ij} = h_i - h_j$ ,  $b_i$  and  $b_j$  are the radii of curvature of the meniscus vertices in the  $i$ th and  $j$ th capillaries, respectively, which were determined from data on the capillary radii  $r_i$  and  $r_j$  with the help of the Lane equation (Lane, 1973).

After the measurement of the capillary constant of a pure liquid the cell was filled with the substance being dissolved (helium or hydrogen). The solution was stirred, and the process of measurement was repeated with retention of the initial temperature. The use of a cell with three capillaries made it possible to obtain at given temperature and pressure two independent values of the capillary constant, which were subsequently averaged. The total uncertainty of the obtained data on the capillary constant was  $\pm 0.5\%$  at temperatures close to that of normal boiling of a pure solvent and increased to (1.5–2.0)% at temperatures of  $0.9 T_c$ , where  $T_c$  is the temperature of the solvent critical point.

The surface tension was calculated by the formula

$$\sigma = \frac{1}{2}ga^2(\rho_l - \rho_v). \quad (2)$$

Here  $g = 9.8162 \text{ m/s}^2$  is the local acceleration of gravity,  $\rho_l$  and  $\rho_v$  are the liquid and vapor densities on the line of phase equilibrium, respectively. The values of  $\rho_l$  and  $\rho_v$ , and also the compositions of the liquid  $x_l$  and the vapor  $x_v$  phase of the solution, were determined from literature data on the parameters of phase equilibrium. For this purpose use was made of both equations of state common to liquid and vapor (Kunz et al., 2007) and methods of simple interpolation of experimental data. In the absence of experimental data on  $\rho_l$  and  $\rho_v$  their values were calculated as follows. At a given pressure the molar volume of the liquid phase of the solution was taken equal to that of the pure solvent, and the molecular weight of the mixture  $\mu_{sol} = \mu_1(1 - x_l) + \mu_2 \cdot x_l$  was used to determine the density of the liquid phase  $\rho_l$ . Owing to the low solubility of helium and hydrogen in the liquid phase of hydrocarbons, such an approximation was justified. The density of the vapor phase of the mixture was calculated from the virial equation of state, in which the second virial coefficient of the mixture was determined as  $B_{mix} = B_{11}(1 - x_v)^2 + 2B_{12}(1 - x_v)x_v + B_{22}x_v^2$ , with  $B_{12} = \sqrt{B_{11}B_{22}}$ , where  $B_{11}$  and  $B_{22}$  are, respectively, the second virial coefficients of the first and second components of the solution.

### 3. RESULTS AND DISCUSSION

We have measured the capillary constant and calculated the surface tension of methane–helium (Baidakov and Grishina, 2013), ethane–helium (Baidakov and Sulla, 1995), and propane–helium, and also methane–hydrogen (Baidakov et al., 2017a) and ethane–hydrogen (Baidakov et al., 2017b) solutions. For methane–helium system, new values of surface tension were calculated with the use of recalculated density data. High-purity gases were used in experiments for preparing mixtures. The rated purity of helium is 99.995 mol%, hydrogen 99.999 mol%, methane 99.99 mol%, ethane 99.8 mol%, and propane 99.8 mol%. Measurements were conducted in a wide temperature range from the melting temperature of a pure solvent to temperatures close to that of its critical point at pressures from that of the solvent-saturated vapors to 4 MPa. The surface tension of the solvents investigated in this paper, i.e., hydrocarbons of methane series, was measured earlier (Baidakov, 1984; Baidakov et al., 1982; Baidakov and Sulla, 1985, 1987).

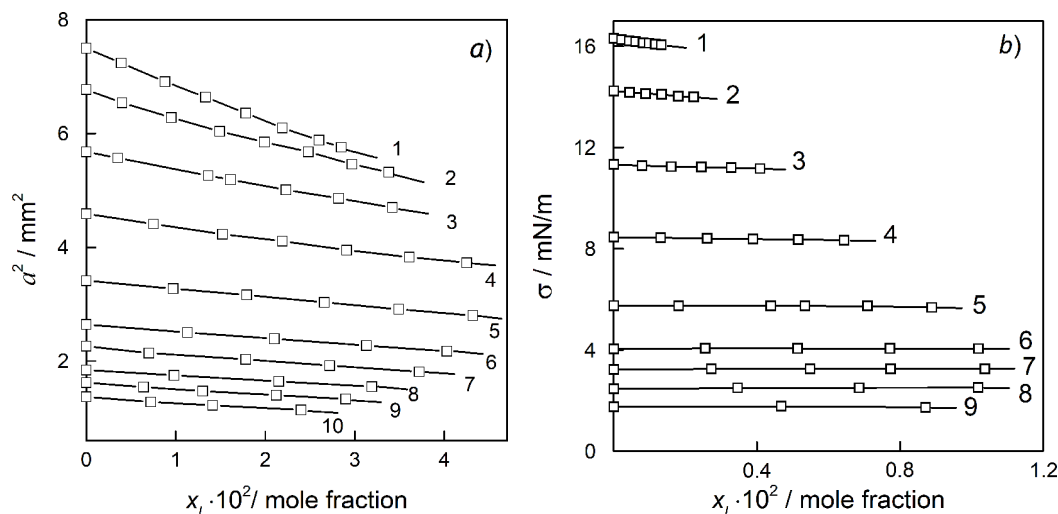
At a fixed pressure the solubility of the gas component in liquid hydrocarbons depends on its nature and temperature. The solubility of helium in hydrocarbons is lower than that of hydrogen. Thus, at  $p = 4 \text{ MPa}$  and the temperature of normal boiling of methane (111.7 K) the helium content in the liquid phase of a solution is about 0.3 mol%. The content of hydrogen is an order of magnitude larger and reaches 3.4 mol%. Approximately the same relationship between solubilities is observed for ethane–helium and ethane–hydrogen solutions.

Figures 1(a) and 1(b) show concentration dependencies of the capillary constant of the methane–hydrogen solution and the surface tension of methane–helium solution at fixed values of temperature. For high temperatures these dependences are close to straight lines and have a weakly expressed curvature in the vicinity of the temperature of the triple point of methane. In a methane–helium system at  $T > 120 \text{ K}$  the derivative  $(d\sigma/dx)_T$ , within the experimental error, is equal to zero. In a methane–hydrogen system the slope of capillary constant isotherms retains a nonzero value to the highest of the investigated temperatures,  $T = 176 \text{ K}$ . The surface tension of an ethane–helium solution at  $T < 220 \text{ K}$  is a decreasing and at  $T > 220$  a weakly increasing function of concentration.

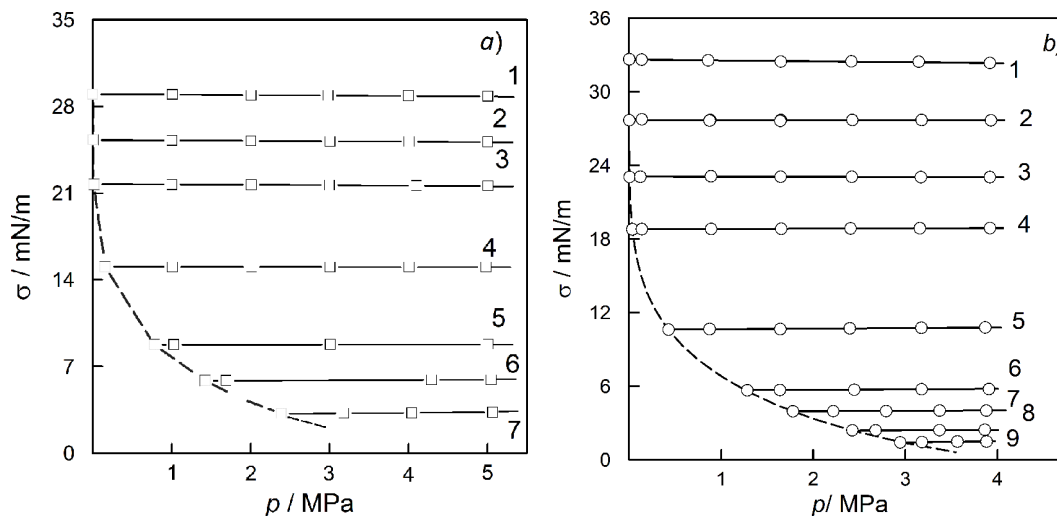
The solutions investigated have qualitatively different baric dependences of the capillary constant and surface tension. If in solutions of helium and hydrogen in methane in the whole temperature range under investigation  $a^2$  and  $\sigma$  are decreasing functions of pressure, in ethane–helium and propane–helium solutions the signs of the derivatives  $(da^2/dp)_T$  and  $d(\partial\sigma/dp)_T$  depend on the temperature [Figs. 2(a) and 2(b)]. The weak decrease of the surface tension of ethane–helium and propane–helium solutions with increasing pressure at low temperatures gives way to its increase at the approach to the critical point of the solvent.

The temperature, baric, and concentration dependences of  $a^2$  and  $\sigma$  of solutions are described by equations of the type

$$z(\varepsilon, \pi) = z_*(\varepsilon) + C(\pi - \pi_*) + D(\pi - \pi_*)^2, \quad (3)$$



**FIG. 1:** Capillary constant and surface tension as a function of the concentration of the volatile component of a solution along isotherms: 1 –  $T = 95$ ; 2 – 105; 3 – 120; 4 – 135; 5 – 150; 6 – 160; 7 – 165; 8 – 170; 9 – 173; 10 – 176 K [ $a^2$ , methane–hydrogen system, (a)] and 1 –  $T = 95$ ; 2 – 105; 3 – 120; 4 – 135; 5 – 150; 6 – 160; 7 – 165; 8 – 170; 9 – 175 K [ $\sigma$ , methane–helium system, (b)]



**FIG. 2:** Surface tension as a function of pressure along isotherms: 1 –  $T = 113.15$ ; 2 – 133.15; 3 – 153.15; 4 – 193.15; 5 – 233.15; 6 – 253.15; 7 – 273.15 K [ethane–helium system, (a)]; and 1 –  $T = 120$ ; 2 – 150; 3 – 180; 4 – 210; 5 – 270; 6 – 310; 7 – 325; 8 – 340; 9 – 350 K [propane–helium system, (b)]. Dashed lines show the surface tension of pure ethane (a) and propane (b)

$$z(\varepsilon, x_l) = z_*(\varepsilon) + C'x_l + D'x_l^2, \quad (4)$$

where  $z$  stands for the capillary constant  $a^2$  or surface tension  $\sigma$ ,  $\pi = p/p_c$ ,  $\varepsilon = 1 - \tau$ , and  $\tau = T/T_c$ .  $\pi$  and  $\tau$  are the reduced values of pressure and temperature,  $p_c$  and  $T_c$  are the pressure and temperature at the critical point of a pure solvent, respectively. The sign  $*$  points to the belonging of the present quantity to a pure solvent. In particular,  $\pi_* = p_*/p_c$  is the reduced pressure of a pure solvent on the saturation line. The approximation error does not exceed the uncertainty of the experimental data.

In the temperature range from the triple to the critical point, dependences of the capillary constant and the surface tension of pure solvents are presented as follows:

$$a_*^2(\varepsilon) = a_0^2 \varepsilon^n, \quad (5)$$

$$\sigma_*(\varepsilon) = \sigma_0 \varepsilon^\mu (1 + \sigma_1 \varepsilon + \sigma_2 \varepsilon^6). \quad (6)$$

The parameters of Eqs. (5) and (6), along with the values of the critical temperature of the substances under investigation, are given in Table 1.

Equations for  $\pi_*(\varepsilon)$  of the substances investigated by us have been obtained in papers by Wagner et al. (Setzmann and Wagner, 1991; Bücker and Wagner, 2006; Lemmon et al., 2009). The functions with  $Y(\varepsilon) = C(\varepsilon)$  or  $D(\varepsilon)$  or  $C'(\varepsilon)$  or  $D'(\varepsilon)$  are presented by us as

$$Y(\varepsilon) = \sum_{i=0}^3 \alpha_i \varepsilon^i. \quad (7)$$

The values of the free parameters  $\alpha_i$  that appear in them have been determined by the method of regression analysis and are presented in Table 2.

#### 4. THERMODYNAMIC ANALYSIS

Let us consider the effect of pressure on the surface tension of a two-component, two-phase system at a constancy of its temperature. An increase in pressure increases the solubility of gases in liquid and is accompanied by the redistribution of a substance being dissolved between the phases and the surface layer. This mechanism is most pronounced in the critical region, which leads to a qualitative difference in the baric dependence of surface tension close to and far from the critical point.

For a liquid solution–vapor system, according to the finite-thickness layer method (Rusanov, 1978)

$$S \left( \frac{d\sigma}{dp} \right)_T \approx - \frac{x_\sigma - x_l}{x_v - x_l} \nu_v, \quad (8)$$

where  $S$  is the area of surface per mole of the surface layer,  $x_\sigma$  is the mole fraction of the second component in the surface layer, and  $\nu_v$  is the molar volume of vapor.

According to Eq. (8), the sign of the derivative  $(d\sigma/\partial p)_T$  is determined by the sign of the concentration factor. Since  $x_v \gg x_l$ , when the composition of the surface layer is intermediate between the compositions of coexistent phases, the surface tension decreases with increasing pressure.

If the values of  $x_\sigma$  and  $x_v$  are very small, as is the case with the least soluble of helium gases, then (Rusanov, 1978)

$$S \left( \frac{d\sigma}{dp} \right)_T \approx \nu_\sigma - \nu_l. \quad (9)$$

At temperatures close to that of the solvent critical point the molar volume of the surface layer differs little from the molar volume of the liquid phase, and the derivative  $(d\sigma/dp)_T$  will be close to zero, which is observed in experiments for systems that contain helium.

**TABLE 1:** Values of the critical temperature and parameters of Eqs. (5) and (6) of alkanes

Substances	$T_c, ^\dagger \text{K}$	$p_c, ^\ddagger \text{MPa}$	$a_0^2 \cdot 10^6, \text{m}^2$	$n$	$\sigma_0, \text{mN/m}$	$\sigma_1$	$\sigma_2$	$\mu$
Methane	190.54	4.598	13.94	0.906	40.92	-0.095	0	1.258
Ethane	305.33	4.872	14.59	0.929	60.57	-0.245	0.309	1.300
Propane	370.4	4.251	13.60	0.909	57.82	-0.144	0.225	1.270

<sup>†</sup>The critical temperatures of the solvents were measured earlier (Baidakov et al., 1982; Baidakov and Sulla, 1985; Baidakov and Sulla, 1987).

<sup>‡</sup>The critical pressures are obtained from Setzmann and Wagner (1991), Bücker and Wagner (2006), and Baidakov (1994).

TABLE 2: Values of parameters  $\alpha_i$  of Eq. (7)

Solvent	Parameter	Function	$\alpha_0$	$\alpha_1$	$\alpha_2$	$\alpha_3$
<b>The substance being dissolved is helium</b>						
Methane	$a^2$	$C$	-0.08167818	0.1855435	0.9073746	0
		$D$	0.1737160	-0.4464645	0	0
	$\sigma$	$C$	0	2.063152	-12.55075	12.33307
		$D$	0	0	0.9777496	0
	$a^2$	$C'$	0	-1.635015	7.755634	0
		$D'$	0	1.778392	-8.056199	0
	$\sigma$	$C'$	0	4.177398	-17.82928	0
		$D'$	0	-4.206421	18.13633	0
Ethane	$a^2$	$C$	0.1035285	0.2277720	-0.6790452	0
		$D$	-0.1389875	0.4198178	0	0
	$\sigma$	$C$	0	1.974825	-7.765527	10.48576
		$D$	0	0	-1.645301	0
	$a^2$	$C'$	0	0.1103650	1.976810	0
		$D'$	0	0.02889107	-0.926979	0
	$\sigma$	$C'$	0	-0.6635866	4.924715	0
		$D'$	0	3.556006	-17.72042	0
Propane	$a^2$	$C$	-0.1172531	0.4236491	-0.1116347	0
		$D$	0.1573713	-0.2387991	0	0
	$\sigma$	$C$	0	5.601409	-21.68199	21.12060
		$D$	0	0	-0.7665293	0
	$a^2$	$C'$	0	-0.8945738	3.797913	0
		$D'$	0	0.4991944	-1.706275	0
	$\sigma$	$C'$	0	3.264703	-8.325053	0
		$D'$	0	-1.901308	6.705639	0
<b>The substance being dissolved is hydrogen</b>						
Methane	$a^2$	$C$	-0.869712	0	-6.415870	0
		$D$	0	0	9.504567	-15.33083
	$\sigma$	$C$	-1.557154	0	-19.16517	0
		$D$	0	0	19.79123	-28.14732
	$a^2$	$C'$	0	-1.405571	5.264825	-10.15951
		$D'$	0	0	0	0.1937314
	$\sigma$	$C'$	0	-2.609085	9.603917	-22.09500
		$D'$	0	0	0	0.5938643
Ethane	$a^2$	$C$	-0.633650	0	0	-4.880710
		$D$	0	0.804410	0	0
	$\sigma$	$C$	-1.771040	0	0	-13.38872
		$D$	0	1.069890	0	0
	$a^2$	$C'$	0	-0.8058310	1.197660	-7.338390
		$D'$	0	0	0	2.454320
	$\sigma$	$C'$	0	-4.825930	21.78280	-42.75250
		$D'$	0	0	0	2.687560

Similar results may be obtained in considering the composition dependence of surface tension. For a liquid solution–vapor system far from the critical state we have (Rusanov, 1978)

$$S \left( \frac{d\sigma}{dx_l} \right)_T \approx - (x_\sigma - x_l) g_{22}, \quad (10)$$

where  $g_{22} = \partial^2 G / \partial x^2$  is the second-order derivative of the Gibbs molar free energy. According to stability conditions, the value of  $g_{22}$  is always positive. From Eq. (10) it follows that if the surface layer of a liquid solution which is in equilibrium with vapor is enriched with one of the components as compared with the liquid phase, then the addition of this component to the solution will cause a decrease in the surface tension.

When the content of the dissolved gas in the liquid phase of the solution and the surface layer is small, the composition of the surface layer differs little from that of the liquid phase. Then Eq. (10) does not hold, and instead of it we have

$$S \left( \frac{d\sigma}{dx_l} \right)_T \approx (1 - x_l) \frac{v_\sigma - v_l}{v_v - v_l} g_{22}. \quad (11)$$

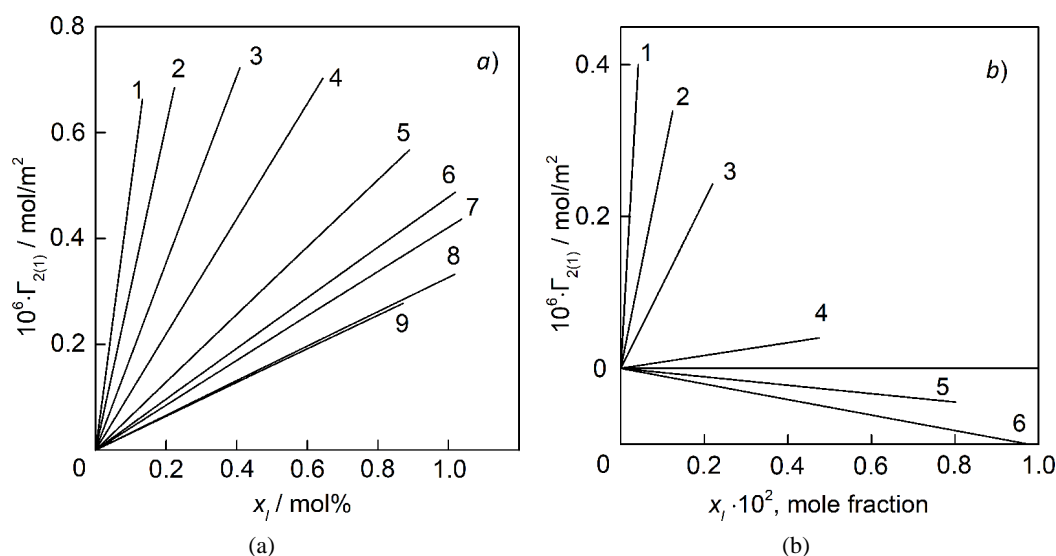
Since  $v_v - v_l > 0$ , the sign of the derivative  $(d\sigma/dx_l)_T$ , as well as of  $(d\sigma/dp)_T$ , will be determined by the sign of  $v_\sigma - v_l$  [see Eq. (9)]. Because the difference between the densities of liquid in bulk and in the surface layer is not usually large, and close to the critical point the molar volume of the surface layer may exceed that of the liquid phase, and the more it does, the larger is the molecular weight of the solvent, the surface tension will increase with an increase in the content of the poorly soluble component in the solution.

The relation between the surface tension and the relative adsorption  $\Gamma_{2(1)}$  is established by the Gibbs equation of adsorption (Gibbs, 1928), which for the case of a diluted solution may be written as

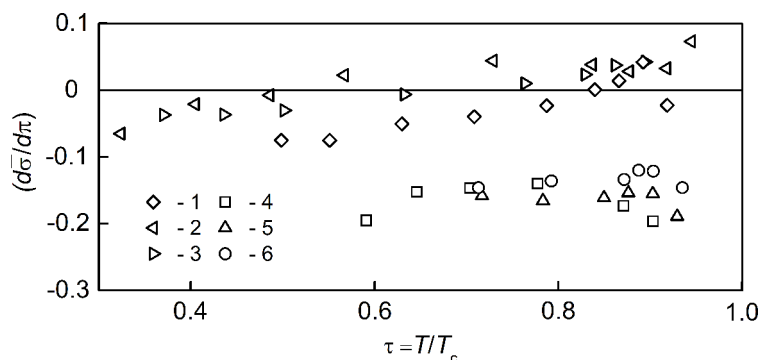
$$\Gamma_{2(1)} = \frac{x_l(1 - x_l)}{RT} \left( \frac{d\sigma}{dx_l} \right)_T. \quad (12)$$

The results of calculating by Eq. (12) isotherms of adsorption in methane–helium and ethane–helium systems are presented in Figs. 3(a) and 3(b). As follows from the figures, close to the ethane critical point helium is an inactive admixture ( $\Gamma_{2(1)} < 0$ ).

The values of the derivative  $(d\bar{\sigma}/d\pi)_\tau$  for hydrocarbons of methane series and cryogenic liquids saturated with helium are compared in reduced thermodynamic units in Fig. 4. Here  $\bar{\sigma} = \sigma / [(k_B T_c)^{1/3} p_c^{2/3}]$  is the reduced surface



**FIG. 3:** Isotherms of relative adsorption in methane–helium system (a): 1 –  $T = 95$ ; 2 – 105; 3 – 120; 4 – 135; 5 – 150; 6 – 160; 7 – 165; 8 – 170; 9 – 175 K and ethane–helium system (b): 1 –  $T = 113.15$ ; 2 – 133.15; 3 – 153.15; 4 – 193.15; 5 – 233.15; 6 – 273.15 K



**FIG. 4:** Temperature dependence of the derivative  $(d\bar{\sigma}/d\pi)_\tau$  of solutions: 1 – methane–helium; 2 – ethane–helium; 3 – propane–helium; 4 – oxygen–helium; 5 – argon–helium (Kaverin et al., 2006); 6 – nitrogen–helium (Baidakov and Kaverin, 2004)

tension, and  $k_B$  is the Boltzmann constant. According to Fig. 4, in the whole temperature range from the triple to the critical point the surface tension of solutions of cryogenic liquids, as distinct from solutions of hydrocarbons, decreases with increasing pressure.

## 5. CONCLUSION

The differential capillary method has been used to measure the capillary constant and to calculate the surface tension of three hydrocarbons of methane series saturated with helium or hydrogen. Experiments were conducted in a wide temperature range at pressures up to 4 MPa. The data obtained for  $a^2$  and  $\sigma$  are presented as analytical functions of temperature, pressure, and composition. In the range of state parameters under investigation one can observe a considerable difference between the solubilities of helium and hydrogen in liquid alkanes. In all the systems under investigation hydrogen is a surface active admixture: The surface tension decreases with an increase of its content in the solution. Since the solubility of helium in liquid hydrocarbons is very weak, an increase of its content in the solution acts on the surface tension qualitatively differently at low and high temperatures. The decrease in the solution surface tension at temperatures close to the triple point with increasing helium concentration gives way to a practically total absence of the dependence  $\sigma(x_l)$  close to the critical point of a low-molecular (methane) and an increase in  $\sigma$  with increasing  $x_l$  for a more high-molecular (propane) hydrocarbon.

## ACKNOWLEDGMENTS

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