

Binary Diffusion Coefficients of Liquid Vapors in Gases

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Preface

Molecular mass transfer in rarefied and compressed gases is widely used in engineering. All main processes of chemical, petroleum, and gas industries are partly or completely diffusion processes: absorption, adsorption, chemisorption, drying, distillation, rectification, condensation, evaporation, mixing, chemical transformations in diffusion and intermediate regions, etc.

Equations to describe mass transport in a gas phase under isothermal conditions always include some coefficients, which characterize molecular mass transport in binary mixtures and evaluate the kinetics of diffusion process.

The properties are necessary for both justified calculations in developing new technological processes and apparatus and improvement of available technologies and equipment. They are also used in traditional design work and automated designing system.

Moreover, the properties of molecular mass transfer in the gas phase are used to determine the parameters of molecular interaction, in the theory and practice of gas chromatography, to solve the problems of environmental control, in aeronomy, and to teach students.

Therefore, diffusion mass transport in dilute and dense binary mixtures in the gas phase was studied in many works.

However, existing theoretical and empirical methods to calculate the properties of molecular mass transport in gases have some restrictions and often give inexact results. This problem cannot also be solved by a purely experimental way because there are a great deal of systems, which are important for engineering and science, and experimental methods are complex, labour-consuming, con-

nected with the use of expensive equipment, and restricted by ranges of parameters and the number of substances that are within reach of experiment.

Therefore, currently, science has no way of determining the properties of molecular mass transport and no necessary experimental data in this field.

Nevertheless, modern scientific and technical progress requires wide and reliable data on the kinetic properties of rarefied and, especially, compressed binary systems in the gas phase.

Then, it is clear that, on the one hand, it is necessary to supplement available experimental results by new results.

On the other hand, it is useful to derive equations, which correctly reflect the dependence of the transport coefficients on the parameters of state. One of the possible solutions of this problem is direct approximation of many high-precision experimental results, using the statistical theory of treating measurement results.

Binary diffusion coefficients are the basic properties in the problem of molecular isothermal diffusion. They evaluate the rate of molecular mass conductivity, by determining the propagation rate of concentration or chemical potential fields; that is, these are coefficients of molecular potential conductivity under isothermal conditions. On the other hand, binary diffusion coefficients are defined as the root-mean-square distances, at which molecules move per unit time during molecular transport.

Then, it is clear that theoretical and experimental methods to determine binary diffusion coefficients in gaseous mixtures are the key problem of the problem of diffusion.

The kinetic theory of gases allows us, in principle, to calculate transport properties and to establish a numerical relationship between them.

Theoretical discussions and the formalism of derivation of the relationship between transport properties were repeatedly and comprehensively presented in fundamental monographs such as [5, 25, 26].

Theoretical methods to calculate the properties of molecular mass transport in gases are complex and inconvenient for practical implementation.

Methods to calculate binary diffusion coefficients of dilute mixtures in the gas phase can only partly satisfy the requirements of modern engineering and science in terms of the accuracy of results, the number of substances, and temperature range within reach of calculation [3, 5, 19, 20, 146]. The calculation errors far exceed the accuracy of experimental determination of binary diffusion coefficients.

Methods to calculate binary diffusion coefficients in dense gases lead to significant errors increasing with pressure [40, 99, 20]. Hence, the values of binary diffusion coefficients calculated under compression conditions cannot be recommended for use.

Empirical methods to calculate binary diffusion coefficients in the gas phase were developed for a limited number of mixtures and can only be used in narrow

ranges of the parameters of state [3, 12].

Therefore, in the whole, available methods to calculate the properties of molecular mass transport in binary systems in gases cannot be accepted as satisfactory methods.

Experimental ways to determine the properties of molecular mass transport in binary mixtures in the gas phase are the most reliable origin of knowledge.

Experimental studies of diffusion in gases were first carried out by Graham [124] in 1829, and those of liquid vapor diffusion to gases, by Stefan [168, 169] in 1871.

The basic experimental methods of investigating diffusion in the gas phase were comprehensively reviewed in the works [1, 146]. The available handbooks [4, 27] give experimental data on binary diffusion coefficients obtained only till 1970. Liquid vapor diffusion to compressed gases, however, was not comprehensively studied till 1970.

In this book, we describe experimental methods of measuring binary diffusion coefficients of liquid vapors in gases. Experimental data on binary diffusion coefficients of liquid vapors in gases are given in wide temperature and pressure ranges (studies were performed by various investigators and by us).

When writing this book, we used many scientific works and, hence, we would like to thank the authors of all used papers.

1

Experimental Measurements of Binary Diffusion Coefficients for Liquid Vapors in Gases

Experiment is the most reliable way to find binary diffusion coefficients. Measurements of binary diffusion coefficients are based on the first and second diffusion laws, established by Fick for steady- and unsteady-state processes [117, 118]

$$J_1 = -D\nabla c_1$$

$$\frac{\partial c_1}{\partial \tau} = D\nabla^2 c_1$$

Therefore, steady- and unsteady-state methods exist to determine binary diffusion coefficients. Experimental devices are made with taking into account side effects which cause additional streams.

Molecular diffusion is known to be accompanied by temperature gradient (Dufour effect) [115]. It was shown in [15] that the diffusion thermoeffect is absent in ideal systems. This effect is considered as a local exhibition of heat of mixture in the course of diffusion of nonideal components. Moreover, diffusion in any system is always accompanied by viscous mass transfer, which results in

pressure gradient (Kirkendall effect). In so doing, heavier molecules move to a region with higher pressure. It would appear reasonable that this effect is more pronounced in closed systems than in open ones, all things being equal.

The thermo- and baroeffects most often hinder concentration mixing. The intensity of the thermoeffect is evaluated by the thermal-diffusion coefficient, and that of the baroeffect, by the barodiffusion coefficient.

Being a function of concentration gradient, parameters of processes, and the physical nature of components of a mixture, the thermoeffect can be significant. As pressure increases, the thermoeffect substantially decreases; it is often experimentally undetected in sufficiently dense media. Usually, the thermodiffusion coefficient is smaller than the binary diffusion coefficient by three to five orders of magnitude in studies into diffusion in dilute and dense gases. When carrying out experiments, investigators tried to eliminate the possible thermoeffect by ensuring reliable thermostatic control of a diffusion cell.

The baroeffect depends on the parameters of processes, the ratio of the masses of mixture components, the compressibility of components, and the geometry of a system. The contribution of the baroeffect to the total transfer is usually significantly smaller than that of the thermoeffect [5]. However, as pressure increases and temperature decreases, the baroeffect increases [33] and can be substantial [32]. The higher the discrepancy between the compressibilities of gases studied, the higher the baroeffect. If, however, the parameters of experiment are far from the critical region, the baroeffect is small [57]. The baroeffect, which occurs in each diffusion cell, is taken into account either during the derivation of a formula to calculate binary diffusion coefficients or directly in experimental technique.

If experiments to determine binary diffusion coefficients are correctly performed, the contributions of the thermo- and baroeffects to the total transfer are virtually zero or fall within the limits of experimental error.

Diffusion in gases was first studied by Graham [124] in 1829, who found the Maxwell diffusion coefficients. Diffusion equation, including the Maxwell and Fick diffusion coefficients, and the relationship between these coefficients were given in [152]. The Maxwell diffusion coefficient did not gain acceptance in further experimental practice.

1.1 Method of Steady-state Evaporation From an Open Tube

The method of steady-state evaporation from an open tube was developed by Stefan [165–171] and bears his name. This method was extensively used by Winkelmann [178–182] and other investigators. The method consists in that a diffusion cell, represented as a tube sealed from one end and partially filled with liquid, is blown by a gas, to which vapors of an evaporating liquid diffuse (Fig. 1.1.1). The gas velocity and flow rate are chosen so that the concentration of liquid vapors to

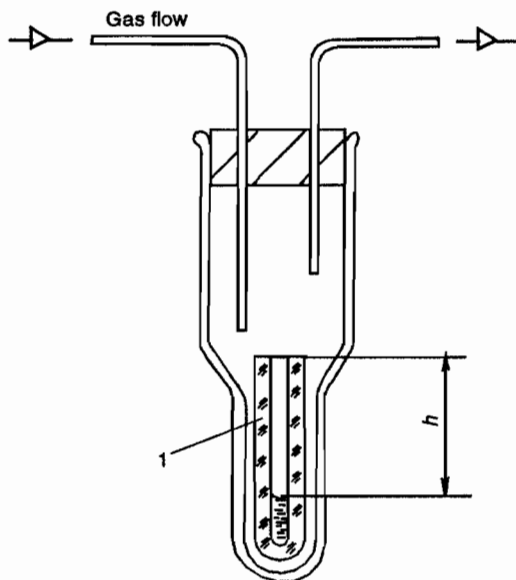


Fig.1.1.1 Diffusion Stefan device for the method of steady-state evaporation from an open tube.
1 – diffusion cell (tube); h – diffusion length.

be zero at the open end of the diffusion cell. The evaporation kinetics of the liquid depends on the rate of molecular transfer of the vapors in the diffusion cell.

A formula to calculate binary diffusion coefficients D is conveniently derived from the first Fick law, written in the coordinate system moving with mean mole velocity u :

$$J_1 = -D \frac{\partial c_1}{\partial z} + c_1 u \quad (1.1.1)$$

where $c_1 u$ is the convective flow of the mixture (Stefan's flow); J_1 is the molar flow of the vapors relatively to the stationary coordinate system; and c_1 is the volume molar concentration of the vapors.

Integration of Equation 1.1.1, under the boundary conditions

$$c_1 = c_s \text{ at } z = 0 \text{ and } c_1 = 0 \text{ at } z = h,$$

gives

$$D = \frac{h \Delta h c_l \rho_l}{\tau c \mu} \frac{1}{\ln \frac{c}{c - c_s}} \quad (1.1.2)$$

where μ is the molar weight of the liquid vapors; ρ_l is the density of liquid; τ is the time for changing level Δh of the liquid in the diffusion cell; h is the arithmetic mean diffusion length measured from the beginning to the end of experiment; c is the molar density of the vapor-gas mixture; and c_l is the volume molar concentration of the saturated vapor directly over the surface of the evaporating liquid. A change in the level of the liquid is usually measured by cathetometer.

Formula (1.1.2) was derived, assuming that the gas is stationary and does not dissolve in the liquid and that the molar density of the vapor-gas mixture is constant within the diffusion length. In the general case, however, the molar density of the vapor-gas mixture is constant only for gases, which are governed by the equation of state of the ideal gas. Therefore, formula (1.1.2) cannot be used for a compressed gas, which significantly differs from the ideal gas and dissolves in the liquid to a great extent.

Semenov and Berezhnoi [77] obtained the formula

$$D = -\frac{h\Delta h c_l x_1}{\tau} \frac{\bar{v}_1 \left[1 + \frac{\bar{v}_2 x_2}{\bar{v}_1 x_1} \right]}{\ln \left[1 - c_s \bar{v}_1 \left(1 + \frac{\bar{v}_2 x_2}{\bar{v}_1 x_1} \right) \right]} \quad (1.1.3)$$

which is applicable to calculate D by the Stefan method in compressed gases. Here, c_l is the molar density of the liquid phase; x_1 and x_2 are the molar fractions of liquid and gas in the liquid phase, respectively; and \bar{v}_1 and \bar{v}_2 are the partial molar volumes of the vapors of liquids and gas in the gas phase, respectively.

The main difference between the derivations of formulas (1.1.3) and (1.1.2) consists in the fact that the former was derived, assuming that the partial molar volumes rather than the molar density are constant within the diffusion length. When the gas obeys the equation of state of the ideal gas and does not dissolve in the liquid, formula (1.1.3) transforms to formula (1.1.2).

When the properties of the components of the vapor-gas mixture are close to those of the ideal gases, the formula

$$D = \frac{h\Delta h}{\tau} \frac{\rho_l RT}{p\mu} \frac{1}{\ln \frac{p}{p-p_s}} \quad (1.1.4)$$

where p is the mixture pressure; p_s is the pressure of the saturated vapors of the liquid at the experimental temperature; and R is the universal gas constant, can be used.

The Stefan flow arises owing to the fact that the diffusion gas flow moving down cannot permeate through the surface of liquid. The gas content over the

surface of liquid becomes higher, thus increasing the density of the vapor-gas mixture at a constant concentration of the saturated vapors. As a result, an excess pressure arises over the surface of liquid, and a hydrodynamic stream appears.

The error in determining D by the Stefan method is 1–2% at atmospheric pressure [12], whereas it can achieve 2–4% [148]. Winkelmann [178–182] showed that the values of D depends on the height h of the working part of the cell; however, they become constant starting from a certain height. This height is experimentally found. Thus, the true values of D are achieved, beginning from a certain ratio of the height h of the working part to the diameter d of the cell for a system analyzed at a given parameters. The values are independent of the geometric sized of the cell and the gas flow rate or velocity (within continuous motion) and are determined only by the properties of a vapor–gas system studied, pressure, and temperature. Under these conditions, the evaporation rate is small, and the temperature of the liquid surface is close to the temperature of experiment. According to [140], a decrease in the temperature of the surface layer of easily volatile liquids is 0.1–0.2 K at pressures close to normal pressure, whereas the decrease is even smaller in other cases. Nevertheless, investigators always tend to compensate the heat expended in evaporation in precision measurements. To this end, the diffusion cell has as small diameter as possible, and its material should well conduct heat. Other precautions are taken to improve the thermostatic control.

The existence of a meniscus of the liquid in the cell leads to the fact that the diffusion length differs from the length measured along the axis of the tube. Moreover, the molecular mode deteriorates inside the cell at its open end, when the cell is blown at very high gas velocities. As a result, the diffusion length decreases. The features of the method were discussed in the works [88, 178–182, 140, 141]. In most cases, the effects of these factors are eliminated by performing experiments with various lengths of the working part h of the cell and by correctly chosen gas flow velocities. Note that the meniscus of the liquid significantly decreases with increasing pressure.

In some works, metallic rather than glass tubes were used to improve thermostatic control. Special guides were used to ensure laminar gas flow and to avoid break-off at the open end of the cell. This modification was described elsewhere [61, 140, 141]. The copper cell [61] was hung from the beam of an analytical balance. Apart from thermostatic control, this modification allowed the authors to continuously measure the amount of the evaporating liquid. All measurements were carried out at atmospheric pressure.

When the vapor density of the evaporating liquid is smaller than the gas density, free convection may appear. Then, the diffusion cell is positioned bottom upwards. The liquid is held from flowing out from the cell of a small size by surface tension force. Moreover, any porous material may be used to hold the liquid from flowing out. Provision should be made for the surface of the porous material

to be horizontal and smooth. The porous material fills a part of the cell. The rest volume of the cell serves to ensure a necessary diffusion length. The amount of the evaporating liquid is measured by weighing [140]. The cell, however, may be retained in its unturned position, and then control experiments should be carried out, using the cells with various diameters. The smaller the cell diameter, the smaller the probability of convection. The value of diameter, at which convection is absent, was found in [140].

After the beginning of experiment, the time it takes for the steady state to be maintained is required. This time was determined in [38]; the steady state was found to appear when the Fourier diffusion criterion $Dt/yl^2 > 0.5$.

The disadvantage of the experiment is considered to be its long duration. In order the error of measurements by cathetometer to be smaller than 1%, a decrease in the level of the liquid evaporating in the cell should be greater than 1 mm. The time it takes for this decrease to occur depends on the physical properties of a system studied and the parameters of the process; it can be irrationally long. Moreover, the method is appropriate only at temperatures and pressures corresponding to equilibrium in the liquid-vapor system, namely, in the range between boiling and freezing points of liquids.

The baroeffect in the diffusion cell was taken into account in the derivation of a formula for calculating D . The thermoeffect is virtually zero because the cell has a small diameter and good thermostatic control, and the content of one of the components is low. Therefore, the method allows one to obtain the true values of D .

Measurements by the Stefan method under compression were carried out in [53, 77, 78]. The experimental setup consists of a diffusion device, thermostat, and a system to create and maintain pressure.

In the diffusion device (Fig. 1.1.2), diffusion cell 1 is set in a brass holder. The diffusion cell is a glass tube, sealed from the lower end, 5.5 mm in inside diameter, 7.5 mm in outside diameter, and 73.5 mm in height. The inside and outside diameters were constant everywhere along the length of the tube. The upper polished end of the tube, which is perpendicular to its axis, is in one plane with the horizontal surface of the holder. The boundary condition of the Stefan method, namely, zero concentration of liquid vapors at the open end of the tube, is ensured by blowing off the tube with a gas supplied onto the horizontal surface of the holder. The casing of the diffusion device has the inside diameter 100 mm and the height 270 mm. The liquid is put in the diffusion cell through a hole in the cover of the diffusion device, which is closed by a plug. The evaporation of the liquid is observed through two sight holes 2 in the casing of the diffusion device. One of the holes provides access of light. Quartz glasses of the holes are packed according to the model of uncompensated area. The diffusion device, all its parts, and all supplying tubes are made of stainless steel.

Diffusion device 1 (Fig. 1.1.3) is positioned in aqueous thermostat 2 with a capacity of 180 l. Heaters 3 are uniformly arranged in the thermostat. Water is

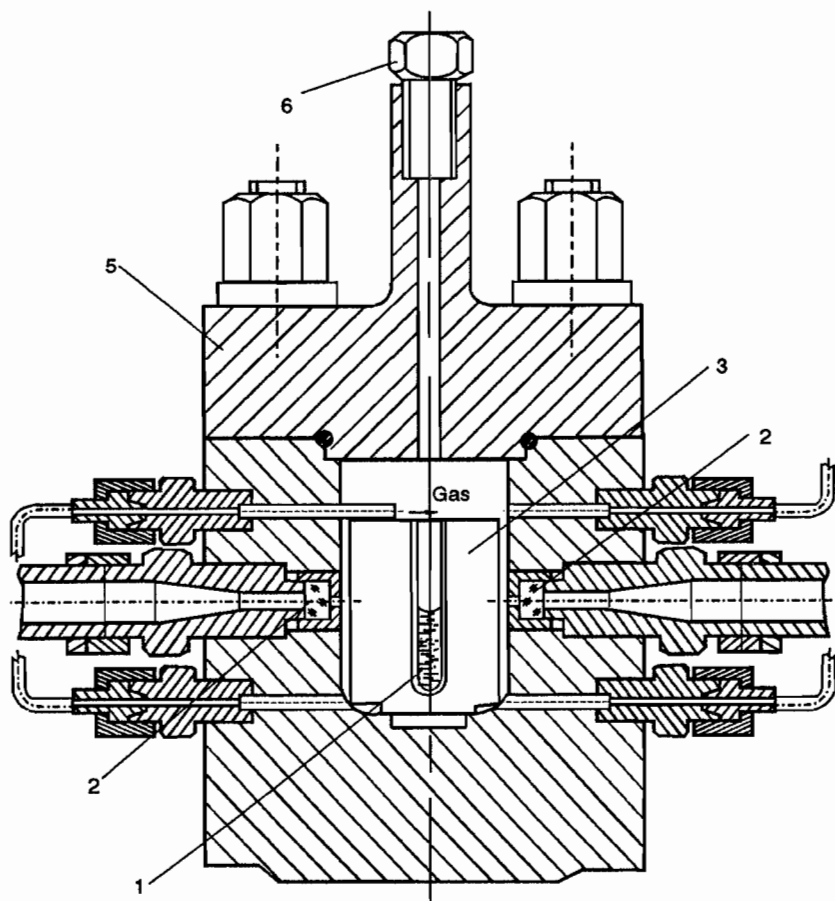


Fig. 1.1.2 Diffusion device for the method of steady-state evaporation from an open tube.
 1 – diffusion cell; 2 – sight holes; 3 – holder; 4 – casing of high-pressure vessel; 5 – cap; 6 – plug.

mixed by mixers 4 with circulating pumps. A gas studied goes from cylinder 5 through drier 6 and enters the diffusion device. Before entering the diffusion device, the gas is heated to the temperature of experiment, passing through a 18-m-long coil located in the thermostat. A given pressure of the gas in the device is maintained with pressure regulator 7. The second pressure regulator 8 reducing pressure to atmospheric pressure is placed at the outlet of the diffusion device. High-pressure needle valves 9 are used to valve off the inlet and outlet of the diffusion device. Vacuum pump 10 is used to evacuate air. Changes in the level of

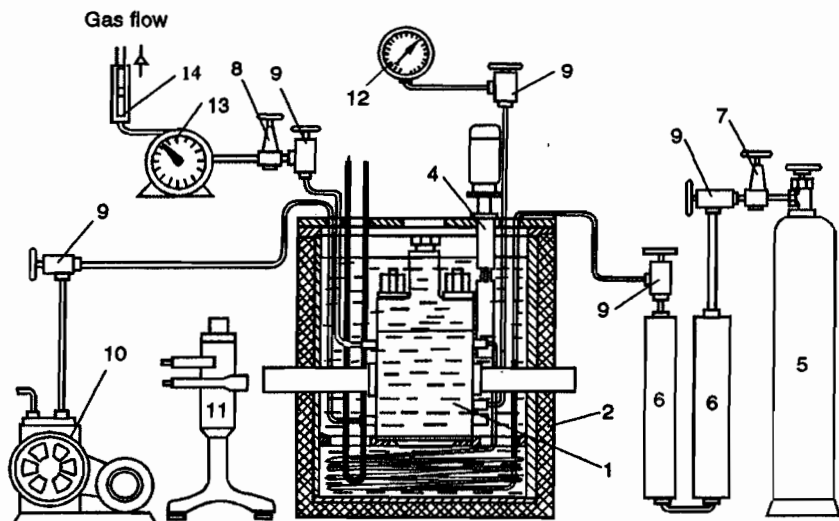


Fig. 1.1.3 A schematic diagram of the experimental device for the method of steady-state evaporation from an open tube. 1 – diffusion device; 2 – aqueous thermostat; 3 – heater; 4 – mixer; 5 – cylinder with gas; 6 – dryer; 7 and 8 – gas pressure regulators; 9 – valve; 10 – vacuum pump; 11 – cathetometer; 12 – sample pressure gauge; 13 – gas counter; 14 – rotameter.

the liquid in the diffusion cell are measured by cathetometer 11. Pressure is measured by Bourdon-tube-based reference manometers 12. The gas flowrate is measured by gas counter 13 and ball rotameter 14. Temperature in the diffusion device is measured by chromel–copel thermocouple.

Air is evacuated from the device prior to experiment. Then, the setup is blown down by the gas studied, and the liquid is put in the diffusion cell. The diffusion setup is sealed, and required pressure and temperature are set. The experiment begins after the complete dissolution of the gas in the liquid.

Measurements by the Stefan method under compression are also known from the works [29, 35]. In these studies, the diffusion cell was made as an open from above cylindrical vessel 25 mm in diameter and 30 mm in height, made of a 0.05-mm-thick copper foil. A certain amount of liquid is put in the diffusion cell to fill about its half.

The diffusion cell is placed in the diffusion setup, which is a high-pressure vessel. Part of the vessel is filled with a packing of adsorbent. The vapors of liquids passing from the diffusion cell are absorbed by the adsorbent. The concentration of the vapors at the outlet from the diffusion cell is determined from the vapor pressure of this liquid over the adsorbent.

In order to decrease the probability of convection flows in the diffusion cell,

a folded insert of aluminum foil 0.04 mm thick is placed in it. Thus, the diffusion cell is separated into narrow slots about 3 mm thick. The liquid put in the diffusion cell touches the insert and forms a meniscus in the gaps. Under these conditions, the surface of the liquid deteriorates (evaporation area increases), and the diffusion length significantly decreases. These features lead to significantly overestimated values of binary diffusion coefficients [29].

The amount of the evaporating liquid is determined by hydrostatic weighing; that is, the density of the medium, in which the diffusion cell is placed, is taken into account. A diffusion device used in hydrostatic weighing will be described in the next section.

1.2 Method of Evaporation in a Closed Volume

A method to determine binary diffusion coefficients D of vapor-gas systems from the rate of evaporation of liquids in a closed vessel was developed by Irisov [11]. The diffusion cell is a closed cylinder with a volume of 250–300 ml, 5–6 mm in diameter, and partially filled with liquid. The device is placed in a thermostat. A branch pipe to a pressure gauge exists in the upper part of the cylinder. First, the pressure of the vapors of the liquid evaporating in the vessel increases. In due course, however, the partial pressure becomes constant and equal to the pressure of the saturated liquid vapors. Because the density of flow is proportional to the rate of increase of the partial vapor pressures, it follows from Dalton's equation [114] for the evaporation of liquid that

$$\frac{dp}{dt} = AD(p_s - p) \quad (1.2.1)$$

Here, A is the device constant depending on the shape and size of the diffusion cell, and p is the actual value of the partial vapor pressure of liquid.

Integration of (1.2.1) and some transformations give [12]

$$D = \frac{1}{A(\tau_2 - \tau_1)} \ln \frac{p_s - p_1}{p_s - p_2} \quad (1.2.2)$$

where p_1 and p_2 are the partial vapor pressures at times τ_1 and τ_2 , respectively.

One of the disadvantages of the method is the volume technique of measuring the flowrate of the evaporating liquid in the vessel with a relatively large diameter, which has a low accuracy. Nevertheless, according to [11], D can be determined with an accuracy of up to 1% at pressures close to normal pressure.

In the Irisov device [11], the pressure of mixture increases in the course of experiment; therefore, D gradually decreases. The higher the pressure of the saturated vapors of the evaporating liquid, the more pronounced this effect. Hence,

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