p-V-T PROPERTIES OF NATURAL GAS AT TEMPERATURES BETWEEN 270 K AND 300 K AND PRESSURES OF UP TO 6 MPa

by

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National Engineering Laboratory East Kilbride, Glasgow p-V-T Properties of Natural Gas at Temperatures between 270 K and 300 K and Pressures of up to 6 MPa

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Introduction

There are two reasons why the knowledge of reliable values of the thermal variables of state of natural gas, i.e. pressure, density and temperature, is of significance. On the one hand, these data are required to judge the quality of methods applied for the computation of variables of state as they are, for instance, used in the charging of thermal gases. On the other hand, they are necessary to check methods for the measurement of the gas density which are often based on the calibration with pure gases of known density. Owing to the different thermodynamic properties of the gas to be measured and the gas used for calibration, the density values read may be affected by errors.

A thermodynamic state in the single-phase region of a fluid is defined by the related values of pressure p, temperature T and density ρ . In experiments, two variables of state are therefore given and the third variable, which is established in the state of equilibrium, is measured. Various experimental methods result from this. A given gas quantity is filled into a measuring vessel and its temperature or volume is changed so that different states of the gas are obtained. The method applied at the Physikalisch-Technische Bundesanstalt for measuring the thermal variables of state of gas mixtures combines the gravimetric density determination with the isothermal determination of the compressibility factor by the Burnett method. States in the vicinity of the two-phase region are obtained by quasi-isothermal cooling, starting from a temperature of 50 °C. During the test series, the density of the gas remains almost constant, the temperature is given and the resulting pressure measured.

Experimental procedure

The experimental apparatus is schematically shown in Fig.1. It essentially consists of three pressure vessels connected in series, the thermostat, and devices for measuring pressure and temperature. The pressure is measured by means of a piston gauge. The diaphragm of a differential manometer, which is located in an agitated water bath, serves as null indicator of the constant volume pressure tap. As pressure

transmission medium between diaphragm and piston gauge, nitrogen is used whose pressure can be controlled with the aid of an oil injector and acts on an oil column connected with the piston gauge. The sensitivity of the differential manometer is higher than of the piston gauge so that the uncertainty of the pressure measurement is not influenced by the pressure being tapped via the diaphragm. From the force due to gravity, the effective cross section of the piston and the barometer reading – taking the difference of altitude into account – the pressure is determined with a relative uncertainty of less than $1\cdot 10^{-4}$.

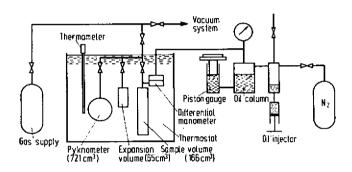


Fig.1: Apparatus for p-V-T measurements

The temperature of the controlled thermostat, which houses the pressure vessels and the diaphragm, is measured with two platinum resistance thermometers (R = 25 ohms) and a resistance bridge. Prior to each measurements, the otemperature is kept constant to 10 mK for at least one hour so that thermal equilibrium is established between the fluid under study and the thermostat bath.

The sample volume and the volume of the pyknometer were determined several times using distilled water which had been carefully degassed. At zero pressure and at t = 20 °C, the pyknometer volume V $_{0,k}(20 \text{ °C}) = 720,72 \text{ cm}^{3}$ and the cylindrical sample volume $_{0,k}(20 \text{ °C}) = 166,44 \text{ cm}^{3}$. The pressure and temperature dependence of the volumes is allowed for by the linear equation

$$V(t,p) = V_{0}(t_{0}) \cdot (1 + \alpha(t - t_{0}) + \beta(p - p_{0}))$$
 (1)

where $V_0(t_0)$ is the volume at zero pressure at the temperature t_0 and p_0 the atmospheric pressure. For the pyknometer, the coefficient of thermal expansion α and the pressure coefficient β have the values $\alpha_k = 47 \cdot 10^{-6} \text{ K}^{-1}$ and $\beta_k = 2.31 \cdot 10^{-4} \text{ MPa}^{-1}$, respectively, whereas the values are $\alpha_z = 49.5 \cdot 10^{-6} \text{ K}^{-1}$ and $\beta_z = 1.6 \cdot 10^{-5} \text{ MPa}^{-1}$, respectively,

for the sample $_{\rm -4}{\rm volume}$. The relative uncertainty of the volume values amounts to 5 $\,10^{-4}$ at most.

For isothermal expansion measurements, the gas from the sample volume is expanded into the expansion volume. The zero pressure cell constant V° + V°

 $N_{\infty} = \frac{V_1^{\circ} + V_2^{\circ}}{V_1^{\circ}}$ was determined at temperatures of 20 °C, 30 °C and 50 °C

from expansion runs with helium, whose volumetric behaviour is very exactly known. The mean value $N_{\omega} = 1.39000$.

Prior to filling the pressure vessels, the apparatus is evacuated down to a final pressure $p < 10^{-4}$ Pa using the turbomolecular pump connected with them. When gas mixtures are investigated, the temperature of the pressure vessels and of the gas supply are regulated to about 50 °C in order to achieve sufficient homogeneity. Heating of the connecting line is to prevent individual mixture components from accumulating at the surface of the filling line. After the pressure vessels have been rinsed several times, the main valve is closed and the thermostat temperature decreased to 20 °C.

During pressure measurement, the valves between pyknometer and sample volume are almost closed so that the pressure change due to the final closing of the connecting valves is negligibly small.

The mass of the gas confined in the sample volume results from the difference in mass of the filled and the evacuated pyknometer divided by the volume ratio of the pressure vessels at the temperature at which the valves were closed.

First a quasi-isochoric run is carried out. For this purpose, the thermostat temperature is usually varied in steps of 5 $^{\circ}$ C and the respective values of temperature and pressure are measured.

The uncertainty of the variables of state found results from the uncertainties by which the measurements of the temperature, the pressure, the volumes and the mass of the gas contained in them in affected. The relative uncertainty of the temperature measurement is $3 \cdot 10^{-5}$, that of the pressure measurement $1 \cdot 10^{-4}$ at most. The volume values and the values of the masses of confined gas are affected by an uncertainty of $5 \cdot 10^{-4}$ and less than $1 \cdot 10^{-3}$, respectively.

Following the quasi-isochoric test run, an expansion run can be carried out. The compressibility factor Z_j , after the j-th expansion, results from eq.(2).

$$Z_{j} = \frac{p_{j}}{p_{0} R T} \cdot N_{\infty}^{j} \prod_{j=1}^{j} \frac{1 + a_{1}p_{j} + a_{2}p_{j}^{2}}{1 + b_{1}p_{j-1} + b_{2}p_{j-1}^{2}}$$
(2)

 p_j is the pressure prevailing after the j-th expansion. The constants a_1 and b_1 result from the pressure distortion coefficients of the sample volumes and the pyknometer; the constants a_2 and b_2 were obtained from the non-linear regression of the measurements executed with helium to determine the constant of the apparatus. With this method, the so-called "run constant" of the expansion run $Z_0/p = \frac{1}{\rho_0 RT}$ can be obtained

by regression. When the density is measured with the pyknometer, the constant results directly from the gravimetrically determined density. On the one hand, the combination of the measurement methods makes use of the advantage offered by the method of constant volume which lies in the fact that the sensitivity is the same over the whole pressure range. The quantity of gas required becomes the greater, the higher the pressure becomes. A disadvantage of this method is the high expenditure of time which is due to the fact that for each filling, the mass of the filled-in gas must be determined.

By the expansion method according to Burnett, a larger number of p-V-T data are obtained in the same period of time, as this method basically requires neither the determination of the volume nor that of the mass, but only pressure measurements along an isotherm. This method is, however, only suitable for measurements in the gaseous region.

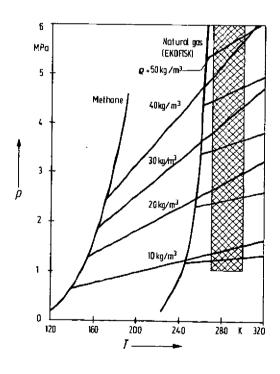


Fig.2: p,T diagram for methane and natural gas with isochores in the gaseous region

Attention must be paid to the fact that the distance between the isotherm and the two-phase region is sufficiently large so that condensation phenomena during expansion are definitely excluded. Whereas in the case of methane – the main component of natural gases – the region which is of interest here is situated at a clear distance from the two-phase region, with Ekofisk natural gas samples the condensation limit is almost reached for densities of more than 30 kg/m³ and at temperatures below 270 K. Fig.2 shows a comparison of the p,T diagrams for methane and for a natural gas sample from the Ekofisk field situated in the North Sea. The isochores in the gaseous region for densities from 10 kg/m³ to 50 kg/m³ have been entered. The plotted dew line of natural gas was experimentally determined by means of a dew point measuring instrument.

When the results of the expansion measurements are evaluated it should be borne in mind that for the computation of the compressibility factors Z, the uncertainty of the compressibility factors increases with rising pressures because the uncertainties of all compressibility factors at the lower pressures enter into the computation.

Experimental results

In order to check the experimental apparatus, initial measurements were carried out with a pure gas whose variables of state in the envisaged pressure and temperature range were well known. In view of the fact that tests with natural gas were to be carried out, methane, the main component of natural gases, was chosen for these preliminary tests. The mole fraction of the impurities of the methane used for the experiments was less than $5 \cdot 10^{-6}$.

In Fig.3, the variables of state measured are compared with values computed with the aid of three equations of state for methane / 1, 2, 3 /. In the figure, the relative deviations between measured and computed pressures have been plotted against the temperature for densities from 10 kg/m³ to 40 kg/m³. With the exception of the IUPAC values at the highest density measured, the equations of state represent the measured values within the uncertainty of measurement. Here the deviations of the IUPAC tables from the measured values are by about 0.1 % higher than those of the equations of state of Sievers et al. and Kleinrahm et al.

The two natural gas samples investigated stem from the Ekofisk field in the North Sea. They were taken from the natural gas pipeline at an interval of three years and differ above all in the mole fractions of methane and ethane. The results of the analyses are given in Table 1.

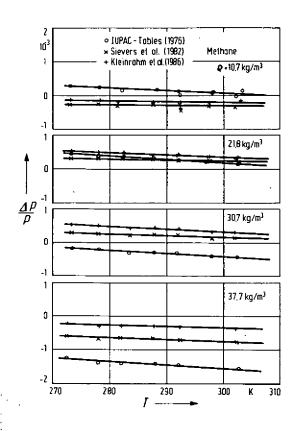


Fig.3: Comparison of 3 equations of state with experimental results of methane

 $\begin{tabular}{lll} \hline \textbf{Table 1:} & \textbf{Composition of the investigated natural gas samples from the Ekofisk field} \\ \hline \end{tabular}$

Component	Mole fraction in %		
· 	Sample 1	Sample 2	
CH ₄	84.47	83.48	
C2H6	8.86	9.47	
C3H8	3.18	3.62	
co2	1.85	1.80	
N ₂	0.43	0.53	
C ₄	1.21	1.10	

The volumetric behaviour of the Ekofisk natural gas sample 1 was also investigated by Achtermann et al. / 4, 5 / at the Institute of Thermodynamics of the Hannover University. Two different methods were applied – on the one hand the refractive index measurement and on the other hand, the measurement of the expansion behaviour (Burnett method). The variables of state determined by the two methods agree to 0.5 /oo. The authors state the relative uncertainty of the measurements to be 1 /oo.

In order to allow the p-p-T values measured with the isochore apparatus to be compared with the variables of state determined by Achtermann et al., the measured values must be represented by a correlation equation.

Representation of the measured values by a virial equation

A virial equation terminated after the third coefficient has proved to be suitable for representing our measurement results / 6 /:

$$\rho = \frac{\varrho \cdot R \cdot T}{M} \left(1 + \frac{B \cdot \varrho}{M} + \frac{C \cdot \varrho^2}{M^2} \right) \tag{3}$$

$$B = \sum_{i=1}^{6} \sum_{j=1}^{6} B_{ij}(T) \cdot x_{i} x_{j}$$
 (4)

$$C = \sum_{k=1}^{6} C_k(T) \cdot x_k \tag{5}$$

with R = general gas constant, M = molar mass, B = second and C = third virial coefficient of the mixture, x_i = mole fraction of the i-th component.

To limit the number of constants in the equation, only six components of the mixture were allowed for, i.e. methane, ethane, propane, carbon dioxide and nitrogen; the higher hydrocarbons were combined and treated like n-butane. The virial coefficients of methane were computed according to the equations stated by Sievers and Schulz / 2 /, those of ethane which, with a mole fraction of almost 10 %, is the most important sub-component of the Ekofisk natural gas, according to the equations given by Goodwin et al. / 7 /. The remaining virial coefficients of the other components and the mixed coefficients were taken from the book by Dymond and Smith / 8 / and approximated by a parabolic fit in the temperature range from 270 K to 300 K if sufficient data had been indicated.

If on the basis of two given variables of state the third variable is to be computed by means of the virial equation, in addition to the virial coefficients of the mixture, the molar mass M must be known. M can, for example, be computed from the complete analysis or from the density and the compressibility factor in standard state.

In order to achieve optimum adaptation to the measured values, M was varied for both natural gas samples until the standard deviation between measured and computed pressure values was minimum. For sample 1 with the lower ethane content, the relative deviation between the measured and computed values is on an average $-5 \cdot 10^{-6}$ with an empiric standard deviation of 6.7 \cdot 10^{-4} . As can seen in Fig.4, all values are within the 1 $^{\circ}$ /oo limit, i.e. the measured values are represented by the set-up equation of state within the scope of the uncertainty of measurement.

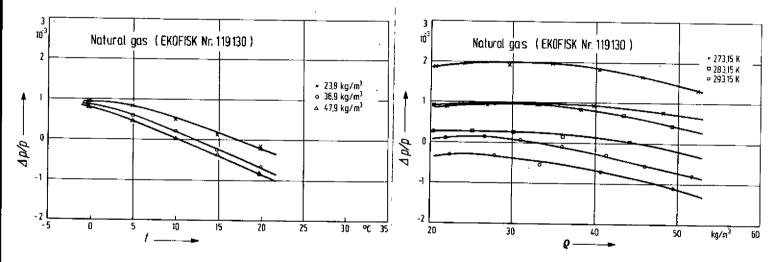


Fig.4: Comparison of the virial equation of state with experimental results on natural gas

Fig.5: Comparison of the virial equation of state with experimental results from Achtermann et al. / 4, 5 /

After a suitable and simple equation of state had been formed which represents the measured values within the scope of the uncertainty of measurement, a direct comparison with the results obtained by the Burnett method and the optical method could be made. In Fig.5, the relative deviations of the pressures measured by Achtermann et al. / 4, 5 /, from the pressures computed by means of the virial equation have been plotted against the density. For 36 values from the density region between 20 kg/m³ and 50 kg/m³ and at the temperatures 0_{\circ} °C, 10°C and 20°C, the mean relative deviation is $5.2 \cdot 10^{-4}$ with an empiric standard deviation of $8.5 \cdot 10^{-4}$. The deviations are greatest with respect to the variables of state determined at 0°C by the optical

method; however, these values, too, do not exceed 2 $^{\rm O}$ /oo. The other values agree to 1 $^{\rm O}$ /oo, i.e. within the scope of their relative uncertainty of measurement. The results prove that with the experimental method applied by us, reliable variables of state can also be determined for multi-component natural gases in a region which almost extends up to the dew line.

Application of the virial equation to other results

The simple equation of state set up by us in the form of a virial statement terminated after the third coefficient has proved its worth for the description of the volumetric behaviour of the Ekofisk natural gas samples investigated. It therefore seemed appropriate to check, whether this equation also furnishes comparably good results for other gas mixtures of similar composition.

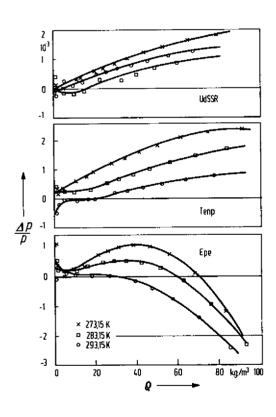


Fig.6: Comparison of the virial equation of state with experimental results on natural gas

In Fig.6, the relative deviations of the pressures computed with the virial equation, from measurement values taken from the literature / 4, 5 / are plotted against the density for the temperatures 0 °C, 10 °C and 20 °C. The composition of the natural gas samples are given in Table 2.

Table 2: Composition of natural gas samples

	Mole fraction in %		
Component	USSR	Tenp	Epe
сн ₄	93.53	85.27	85.50
с ₂ н ₆	1.88	5.40	8.06
С ₃ H ₈	0.49	1.59	2.86
CO ₂	0.23	1.46	1.86
. –	1.61	5.68	0.61
N ₂ C ₄	0.26	0.70	1.11
Standard density in kg/m³	0.7541	0.8346	0.8531
Gross calorific value in MJ/m³	40.249	40.287	44.100

For the USSR natural gas, the representation quality is very good over the whole density range; the relative deviations become greater with increasing density, however, they do not exceed 2 $^{\circ}$ /oo. The same trend can be observed with the Tenp natural gas; for this natural gas, too, the virial equation gives a good description of the volumetric behaviour. The relative deviations remain smaller than 2 $^{\circ}$ /oo up to a density of 60 kg/m³. In the range of low pressures, the Epe natural gas shows a behaviour similar to that of the USSR natural gas, i.e. the relative deviations slightly increase with density. At a density of about 40 kg/m³, this trend is reversed, the curves now drop with increasing density; at 80 kg/m³ the relative deviations amount to up to 2 $^{\circ}$ /oo.

In future, methods as the one described here for computing the gas density from analytical data and measured pressure and temperature values will gain in importance to the extent as reliable, automatic analyzers will be available.

Investigations into the adsorption behaviour of natural gas

The adsorption of the fluid to be measured on the walls of the pressure vessels may falsify the results of p-V-T measurements. When the gas density is determined by weighing a known volume, the adsorption of the

gas molecules at the walls leads to a decrease in pressure. When the measurement is carried out by the method of constant volume, the mass of the adsorbed gas may change as a function of temperature and pressure, i.e. the prerequisite of constant density is not guaranteed. The isothermal determination by the Burnett method can only by applied if the adsorption effects of the fluid to be measured can be neglected. As the expansion volume is evacuated after each expansion, only a small portion of the adsorbed gas molecules is left on the walls. After expansion, part of the gas "disappears" due to new adsorption. This falsification of the measured values becomes greater when the number of expansions increases. As so far no details of the adsorption behaviour of natural gas on metal surfaces are to be found in the literature, we carried out some orientating measurements which allow the influence of the adsorption effects on our p-V-T measurements to be estimated.

Experimental apparatus and carrying-out of the adsorption measurements

The experimental apparatus is schematically shown in Fig.7. The mass of the adsorbed gas is determined gravimetrically using a microbalance with a sensitivity of $1 \cdot 10^{-6}$ g for a maximum mass of 20 g.

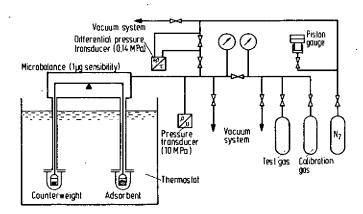


Fig.7: Apparatus for adsorption measurements up to 10 MPa

As the adsorption effects are small, it must be ensured that the sample has a highly specific surface. For this reason, only powdered samples or foils can be used for the measurements. The counterweight should consist of a sample of small surface, if possible of the same material. For reasons of expediency, the buoyancy correction should be determined with a gas whose adsorption is negligible in the range, which is of interest here, i.e. between 270 K and 300 K and at pressures up to 6 MPa, e.g. argon. For powdered samples, the surface effective during adsorption can be estimated if the distribution of the grain diameters

is known. The surface can be determined experimentally by applying the BET theory to adsorption measurements with pure gases. According to the literature, the uncertainty of the surface determined in this way is 10 % to 20 % / 9 /. The value of the surface on which our measurements are based is the mean value of the BET surfaces determined with carbon dioxide, propane and ethane. Depending on the size fraction, the surface values of the powders investigated lay between 1 $\rm m^2$ and 3.5 $\rm m^2$. When foil was investigated, only 1/10 of this surface could be placed into the weighing crucible.

Results of the adsorption measurements

To date, three metal powders and one foil have been investigated, i.e. titanium, iron and aluminium powder and ALFOL aluminium foil. With a few exceptions the measurements were carried out at 0 °C and at pressures of up to 3 MPa. Adsorbates were the natural gas components methane, ethane, propane and carbon dioxide and two natural gas samples whose composition is stated in Table 3.

Table 3: Composition of natural gas samples

Component	Mole fraction in %		
	EK0F1SK	NAM + USSR	
CH ₄	84.40	94.10	
C ₂ H ₆	8.87	1.49	
C3H8	3.17	0.39	
co2	1.92	0.41	
N ₂	0.42	3.42	
c₄	1.22	0.19	
•		<u> </u>	

Fig.8 shows typical adsorption isotherms by the example of the propane/titanium adsorption system at the temperatures $t=0\,^{\circ}\text{C}$ and $t=20\,^{\circ}\text{C}$. Initially, the mass of adsorbed gas increases strongly, then rises steadily, and finally increases strongly again when the saturation pressure is approached. The desorption branch of the isotherms sligthly offset in relation to the adsorption branch, is represented by broken lines. This hysteresis phenomenon is frequently observed with adsorption effects. Dependent on the adsorption system it is more or less pronounced. This is also reflected in Fig.9, where the adsorption of methane is compared with the adsorption of the two natural gas samples investigated, titanium powder being again used as adsorbent.

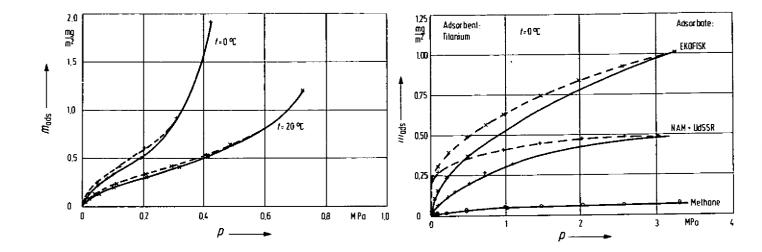


Fig.8: Adsorption isotherms of propane on titanium

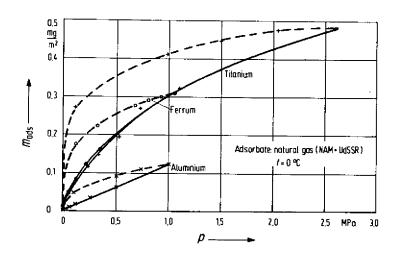
Fig.9: Adsorption isotherms of natural gas on titanium at 0 °C

While the adsorption of methane on the surface of the titanium powder is very low and practically no hysteresis phenomenon can be recognized at the scale chosen, the natural gas samples are adsorbed to a much greater extent. Although with 94.10 % of CH₄ and 3.42 % of N₂, the natural gas sample contains as much as 95.52 4 % of gases, which being pure gases - show only very low adsorption phenomena, 0.5 mg/m² of natural gas are adsorbed at 3 MPa. For the Ekofisk natural gas sample, even twice the amount of adsorbed gas is measured.

Fig. 10 shows the influence of the adsorbent. While the adsorption of natural gas on titanium and iron powder is almost the same, the amount adsorbed on aluminium is only approximately 1/3 of the value measured for iron and titanium. This effect is less pronounced with Ekofisk natural gas, as this gas contains larger quantities of higher hydrocarbons. The adsorption behaviour of ethane and propane on iron and titanium is approximately the same, and on aluminium, adsorption is slightly lower. With methane and carbon dioxide, the differences are clearly more pronounced; for carbon dioxide, the mass of the gas adsorbed on aluminium is only about half the amount adsorbed on iron and titanium.

In order to estimate the influence which the adsorption effects exert on the p-V-T measurements, we formed the ratio of the mass of adsorbed gas to the mass confined in the vessel. The volume of the pressure vessel of $V = 60~\rm cm^3$ corresponds to the volume of the expansion vessel used in the experiments. The surface of 85 cm² follows from the cylindrical geometry of the vessel. The surface, which becomes effective

during adsorption, depends on the surface treatment of the material and may be substantially larger. Fig.11 shows the ratio for the adsorption of Ekofisk natural gas on aluminium, iron and titanium. As can be seen, in the range of lower pressures of up to 1 MPa, the mass of adsorbed gas decreases rapidly from about $5 \cdot 10^{-5}$ to values below $1 \cdot 10^{-5}$ of the mass of gas contained in the vessel. Provided that the behaviour of stainless steel surfaces does not differ substantially from that of the metal surfaces investigated so far, it can be assumed on the basis of the results on hand that the influence of the adsorption effects remains by a factor of 10 smaller than the uncertainty of the p-V-T measurements.



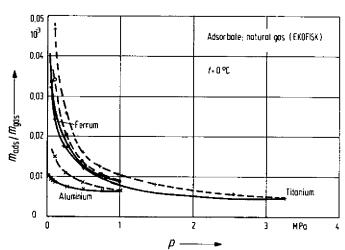


Fig.10: Adsorption isotherms of natural gas on different adsorbents

Fig.11: Effect of gas adsorption on p-V-T measurements in cylindrical pressure vessels (V = 60 cm³, S = 85 cm²)

Summary

The composition and thermal behaviour of natural gas may differ substantially depending on the sources from which it has been lifted. Frequently a mixing with other gases takes place. The method applied for the measurement of the thermal variables of state combines the gravimetric determination of the density with the isothermal determination of the compressibility factor by the Burnett method. States in the vicinity of the two-phase region are obtained by quasi-isochoric cooling, starting from a temperature of 50 °C. The measured p-V-T data can be represented within the uncertainty of measurement by a simple virial equation of state whose coefficients have not been adapted to the measured values. There is also a good representation of the thermal variables of state

of various natural gas samples taken from the literature. In order to allow the influence of the adsorption of the fluid to be measured on the walls of the pressure vessels to be estimated, the adsorption on metal surfaces of natural gas and its main components was gravimetrically investigated. The results obtained so far suggest that, in small vessels, the portion of the mass of adsorbed gas does not exceed 5 10^{-5} of the mass of gas contained in the vessel.

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