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Pressure-Density-Temperature Measurements of Ethylene

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As a contribution to current projects on the thermophysical properties of ethylene for the preparation of reference tables, measured PVT data are presented. For the measurements in the single-phase region, two types of equipment have been employed: an expansion apparatus to determine isotherms and a constant volume apparatus to determine isochores. Both the design and calibration of each type of equipment and the experimental procedures are described. The accuracy of the 251 measured PVT data on 10 isotherms and 16 isochores, which cover the temperature range from −30 to 80°C, with pressures up to 250 bar, is about 0.1% in the compressibility factors. The critical temperature was determined with the result $t_c = 9.22 \pm 0.02$°C by visual observation of the disappearance of the meniscus.

KEY WORDS: critical temperature; density; ethylene; PVT measurements; thermodynamic properties.

1. INTRODUCTION

For many applications of ethylene, which is one of the most important feedstocks of the chemical industry, it is desirable to know reliable properties. Effective process control, equipment design, safe storage, and equitable custody transfer are only a few examples. The large-scale production of ethylene by oil refineries has prompted pipeline distribution in order to reduce transportation costs. The big chemical companies in Europe are connected by the continental pipeline system. The delivered ethylene quantities are measured by positive displacement meters combined with temperature and pressure readings. For the calculation of the delivered mass, accurate density values are required.

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Stimulated by the Hoechst AG, Frankfurt(M), Federal Republic of Germany, who had difficulties in calculating the prices due to different density tables used, the compressibility factor of ethylene at temperatures from 0 to 50°C and pressures up to 21 bar was measured in our laboratory [1]. In 1972, when transport needs necessitated higher operating pressures, the investigation was extended to 100 bar at temperatures from −30 to 75°C. In cooperation with Hoechst AG, an extended Benedict-Webb-Rubin equation of state was developed and tables for custody transfer purposes were published [2].

A further increase of the pipeline capacity was available due to a change from the gaseous state to the single-phase liquid state in the lines. Our experimental studies were therefore extended to the liquid region at temperatures from −30 to 80°C and pressures up to 250 bar. Density tables up to 100 bar were produced from the results and were published by Hoechst AG in a small number of issues in their own publishing house [3]. This publication is known only to a few chemical companies engaged in custody transfer. We have therefore decided to present our accurate and precise PVT measurements as a contribution to current projects for the preparation of standard reference tables for ethylene.

The sample of ethylene used in the experimental work originated from commercially available ethylene, which is transported by the Ethylene Pipeline Company (Äthylen-Rohrleitungs-Gesellschaft), Marl, Federal Republic of Germany. The purity of the ethylene was shown to be 99.96% (volume) by gas-chromatographic analysis. Noncondensibles were removed from the sample by a purification procedure, which consisted of repeated cycles of freezing, pumping, and melting. For the PVT studies, two types of equipment have been employed in our laboratory: an expansion apparatus to determine isotherms in the gaseous region, and a constant volume apparatus to determine isochores mainly in the single-phase liquid region.

2. EXPANSION APPARATUS

The expansion method introduced by Burnett [4] for PVT determinations in the gaseous region requires the measurement of only temperature and pressure on successive isothermal expansions. The expansion apparatus consists of two thermostated pressure cells connected by an expansion valve (Fig. 1). Initially, the first cell is filled with the test gas, and its pressure and temperature are measured. The gas is then expanded into the evacuated second cell, and the pressure is remeasured. In a series of steps, the gas is expanded to a low pressure, with the second cell evacuated each time. The information generated by this experimental procedure is a series of isothermally measured pressures, p_0, p_1 ... p_{n-1}, p_n, called a single run. The

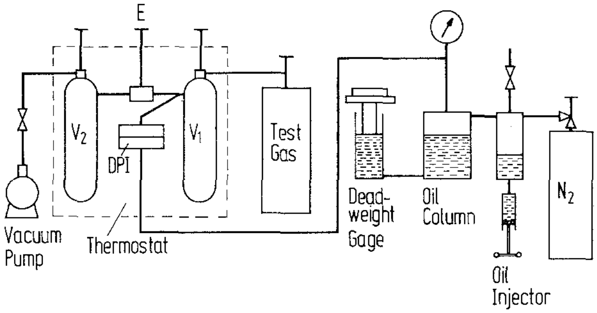

Fig. 1. Expansion apparatus with pressure measurement system.

following data treatment requires the knowledge of the cell constant $N = (V_1 + V_2)/V_1$, which is defined as the ratio of the volume of the gas after an expansion to that before. The cell constant has been determined for each isotherm with helium calibration runs with an accuracy of 0.005%. All observed pressures have been corrected for the elastic deformation of the cell volumes. Therefore, the following equations are developed by assuming that N does not change with pressure.

The volumetric behavior of a gas may be described in terms of the dependence of the compressibility factor Z upon the pressure p at the constant temperature T:

$$Z = \frac{p}{\rho \cdot R \cdot T} = \frac{p \cdot V}{m \cdot R \cdot T} = 1 + B \cdot p + C \cdot p^2 + D \cdot p^3 + \cdots \qquad (1)$$

where B, C, D . . . are called the second, third, fourth, . . . virial coefficients, respectively, and are functions of temperature only (density $\rho = 1/v$, specific volume v, gas constant R, volume V, and mass m). The low pressure region of each isotherm can be accurately fitted by the virial coefficients B and C. On this assumption, the following expression describes the gas in the first cell before the nth expansion in a series:

$$Z_{n-1} = \frac{p_{n-1} \cdot V_1}{m \cdot R \cdot T} = 1 + B \cdot p_{n-1} + C \cdot p_{n-1}^2 \qquad (2)$$

After expansion, if temperature and mass remain unchanged, the equation will be

$$Z_n = \frac{p_n (V_1 + V_2)}{m \cdot R \cdot T} = 1 + B \cdot p_n + C \cdot p_n^2 \qquad (3)$$

Division of Eq. (3) by (2) and rearrangement yields

$$\frac{(p_{n-1}/p_n) - N}{p_{n-1}} = B\,(N - 1) + C\,(p_{n-1} \cdot N - p_n) \tag{4}$$

By plotting the left-hand side of Eq. (4) against $(p_{n-1} \cdot N - p_n)$, the best straight line, fitted by least squares, is drawn through the points, and B and C are evaluated from the intercept and the slope.

Beyond the low pressure region of the isotherm, the compressibility factor for the decreasing pressures $p_0, p_1, \ldots, p_r$ of a run is computed from the run pressure p_r within the low pressure region for which Z_r is already known. Equations (2) and (3) may be combined to yield the relation

$$\frac{p_n \cdot N}{Z_n} = \frac{p_{n-1}}{Z_{n-1}} \tag{5}$$

The values of p_{n-1}/Z_{n-1} from Eq. (5) for $n = 2, 3, \ldots$ are successively substituted into Eq. (5) for $n = 1$ with the result

$$\frac{p_n \cdot N^n}{Z_n} = \frac{p_0}{Z_0} \tag{6}$$

Dividing Eq. (6) for $n = r$ by Eq. (6) gives

$$Z_n = Z_r \frac{N^n \cdot p_n}{N^r \cdot p_r} \tag{7}$$

From Eq. (7), the compressibility factor for each pressure point of the run can be calculated if Z_r at a run pressure p_r in the low pressure region of the isotherm is known.

The expansion apparatus is shown schematically in Fig. 1. The essential part is the expansion section, containing the two cells with the volumes V_1 and V_2 connected by an expansion valve E. With the volume of $V_1 \approx 1$ dm^3 and that of $V_2 \approx 0.5$ dm^3, the resulting value for the cell constant was $N = 1.45440$ at 24.99°C. A Ruska differential pressure null indicator (DPI) was used to separate the test gas in the cell V_1 from the pressure transmission fluid. Nitrogen was used instead of oil in order to avoid disturbing deflections of the metal diaphragm resulting from the periodically engaged drive necessary for the rotation of the dead-weight gage. The nitrogen pressure, which is adjustable by means of the oil injector, acts on an oil column, which is

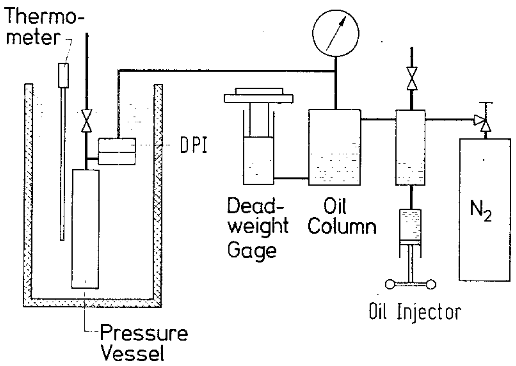

Fig. 2. Constant volume apparatus with pressure measurement system.

connected to the dead-weight gage. The pressure was measured with a Ruska Type 5000.10 dead-weight gauge by balancing the test gas with nitrogen by means of the DPI. The accuracy of the measured pressure is 0.01%.

The temperature was controlled with a thermostat surrounding the expansion section. The temperature level of the well-agitated bath was maintained with a thermistor-actuated temperature controller. Temperatures here and in all other experiments have been determined with a platinum resistance thermometer, calibrated on the 1968 IPTS scale. The accuracy of the measured temperatures is 0.01 K. Summarizing the error sources (pressure, temperature, cell constant, evacuation), we estimated the error in the compressibility factors to be about 0.1%.

3. CONSTANT VOLUME APPARATUS

The experimental method involved charging a known mass of the fluid into a vessel of known volume and measuring the equilibrium pressure of the fluid at various temperatures. The apparatus employed (Fig. 2) consisted essentially of a calibrated pressure vessel, a differential pressure null indicator, a thermostat, and devices for temperature and pressure measurement. The heavy-walled pressure vessel was made of A 286 stainless steel; its volume plus that of the associated tubing was calibrated for the temperature and pressure range of interest by the use of the PVT data for liquid carbon dioxide of Michels et al. [5]. Several tests produced the mean value $V_0 = 166.28\ \text{cm}^3$ at $T_0 = 273.15$ K and $p_0 = 1$ bar. Additionally, the volume was obtained by filling the system with distilled and degassed water and weighing the contents. The results of several water fillings confirmed the volume value obtained with carbon dioxide.

Table I. Experimental Compressibility Data for Gaseous Ethylene From Ref. [1][a]

t (IPTS-68) (°C)	p (bar)	$Z = (p/\rho \cdot R \cdot T)$
49.99	29.899	0.85816
	22.636	0.89468
	16.142	0.92624
	14.201	0.93542
	11.969	0.94582
	8.389	0.96241
	7.345	0.96727
	6.157	0.97263
	4.278	0.98107
	3.735	0.98348
39.99	23.742	0.87619
	16.723	0.91494
	12.690	0.93629
	8.747	0.95673
	6.561	0.96782
	4.473	0.97815
	3.336	0.98386
29.99	24.604	0.85523
	17.878	0.89782
	13.315	0.95234
	9.442	0.94801
	6.927	0.96244
	3.532	0.98110
19.99	24.788	0.83455
	18.601	0.87978
	13.588	0.91457
	9.928	0.93877
	7.111	0.95685
	3.636	0.97828
10	24.917	0.81050
	19.929	0.85369
	13.874	0.90228
	10.805	0.92533
	7.313	0.95081
	5.622	0.96263
	3.753	0.97548
0	20.537	0.82611
	14.132	0.88662
	11.329	0.91106
	7.516	0.94272
	5.943	0.95542
	3.874	0.97134
	3.042	0.97793

[a] Isotherms determined with the expansion method.

The effect of temperature and pressure on the volume can be represented as

$$V = V_0 [1 + \alpha(T - T_0) + \beta(p - p_0)] \tag{8}$$

where $\alpha = 49.5 \times 10^{-6}\ \mathrm{K}^{-1}$ is the cubic thermal expansion coefficient for the vessel material and $\beta = 1.6 \times 10^{-6}\ \mathrm{bar}^{-1}$ is the pressure expansion coefficient calculated according to the usual theory of elasticity. The accuracy in the actual volume thus obtained is 0.05%.

The pressure measurement system has already been described in Sec. 2. The vessel and DPI were completely immersed in a stirred bath. Samples were transferred from a weighing bottle to the vessel by condensation at liquid-nitrogen temperature. The mass of the charge was determined by differential weighing with an accuracy of 0.05%. Sixteen different masses of ethylene were charged so that the PVT surface of interest for custody transfer was covered.

4. CRITICAL TEMPERATURE

The critical temperature was determined visually as the temperature at which the liquid meniscus disappeared in the middle of permanently sealed glass tubes. Those tubes were made in our glass shop from Duran 50 glass with a 10 mm outside diameter, having a wall thickness of 4 mm and a length of 150 mm. Several tubes were filled to known densities close to the critical density (0.218 g/cm^3) taken from the literature [6]. Depending on the filling density, the meniscus will disappear at different heights in the tube. The tube in which the meniscus disappeared close to the middle of the tube height was taken for the observations. The temperature was then alternately lowered and raised until the meniscus appeared and disappeared. The critical point was passed very slowly during the experiments at the very small rate of temperature change of about 0.03 K per hour. The true critical temperature of ethylene was taken as the average of the temperatures at which the meniscus would just appear and disappear, with the numerical result $t_c = 9.22 \pm 0.02$°C. This value is in good agreement with that of Moldover [7], who found for the critical temperature the experimental value $t_c = 9.194$°C.

5. RESULTS

Because in our first PVT study on ethylene [1] we gave only the virial coefficients B and C for temperatures from 0 to 50°C, we present these experimental results now in the form $Z = Z(p, t)$, given in Table I and graphically shown in Fig. 3. The compressibility data measured with the

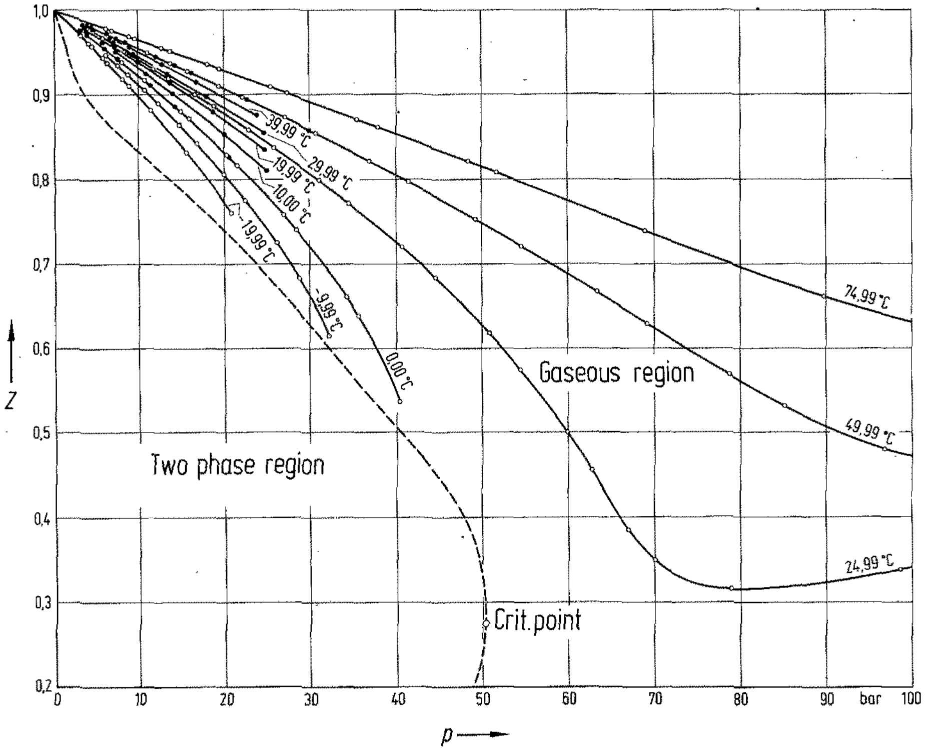

Fig. 3. Experimental compressibility data for ethylene. ●, data from Ref. [1], reported in Table I.

Table II. Experimental Compressibility Data for Gaseous Ethylene[a]

t (IPTS-68) (°C)	p (bar)	$Z = (p/\rho \cdot R \cdot T)$	t (IPTS-68) (°C)	p (bar)	$Z = (p/\rho \cdot R \cdot T)$
74.99	89.698	0.66268		25.762	0.83786
	68.921	0.74052		22.898	0.85795
	51.726	0.80827		18.735	0.88618
	48.448	0.82112		16.527	0.90062
	37.919	0.86171		13.381	0.92058
	35.364	0.87167		11.746	0.93096
	27.298	0.90219		8.260	0.95218
	25.367	0.90931		5.768	0.96706
	19.378	0.93138	0	40.333	0.53729
	17.963	0.93647		35.553	0.63870
	13.621	0.95215		34.139	0.66140
	12.606	0.95577		28.346	0.74059
	9.510	0.96677		26.896	0.75781
	8.792	0.96943		21.491	0.81661
	6.609	0.97702		20.236	0.82924
	6.104	0.97882		15.773	0.87168
49.99	96.843	0.48172		14.779	0.88074
	85.068	0.53205		11.335	0.91095
	78.758	0.56976		10.583	0.91728
	69.179	0.62926		8.030	0.93852
	63.403	0.66707		7.481	0.94303
	54.518	0.72121	−9.99	32.148	0.61546
	49.215	0.75305		28.717	0.68278
	41.421	0.79691		26.043	0.72509
	36.924	0.82168		22.418	0.77517
	30.557	0.85497		19.910	0.80623
	26.984	0.87332		16.751	0.84237
	22.059	0.89765		14.683	0.86466
	19.352	0.91089		12.173	0.89027
	15.688	0.92840		10.580	0.90615
	13.698	0.93764		8.688	0.92408
	11.038	0.95007		7.508	0.93514
24.99	98.643	0.33896		6.126	0.94771
	79.906	0.31633	−19.99	20.752	0.75973
	69.969	0.34968		15.614	0.83131
	66.961	0.38554		11.404	0.88307
	62.794	0.45642		8.829	0.91189
	59.880	0.50144		8.161	0.91909
	54.498	0.57613		6.252	0.93909
	50.842	0.61922		5.764	0.94411
	44.523	0.68455		4.386	0.95807
	40.652	0.72010		4.036	0.96151
	34.516	0.77185		3.057	0.97113
	31.015	0.79903			

[a] Isotherms determined with the expansion method.

Table III. Experimental p, ρ, t Data for Ethylene[a]

ρ (kg/dm^3)	t (IPTS-68) (°C)	p (bar)	ρ (kg/dm^3)	t (IPTS-68) (°C)	p (bar)
0.0910	5.43	42.344	0.3280	5.84	51.416
0.0909	10.02	44.235	0.3280	9.47	60.068
0.0909	19.64	48.112	0.3279	14.77	72.940
0.0909	19.93	48.228	0.3278	19.95	85.720
0.0908	39.67	55.916	0.3278	19.96	85.744
0.0906	80.00	71.012	0.3278	19.97	85.762
			0.3274	40.02	136.116
0.1217	9.78	48.642	0.3271	59.86	186.579
0.1216	15.35	51.944	0.3267	80.23	238.422
0.1216	19.65	54.450			
0.1216	24.67	57.335	0.3345	3.88	48.668
0.1215	29.73	60.208	0.3345	7.71	58.237
0.1215	40.20	66.062	0.3344	10.55	65.429
0.1214	49.68	71.284	0.3344	15.48	78.053
0.1214	49.74	71.313	0.3344	17.32	82.805
0.1212	80.27	87.787	0.3344	19.36	88.101
			0.3344	19.38	88.184
0.1700	10.02	51.077	0.3344	19.90	89.534
0.1700	14.22	54.874	0.3344	20.09	90.009
0.1700	20.19	60.153	0.3344	23.07	97.788
0.1699	25.04	64.389			
0.1699	29.25	68.041	0.3503	1.64	48.835
0.1698	40.06	77.344	0.3502	5.05	58.425
0.1697	49.71	85.584	0.3501	9.62	71.427
0.1697	49.84	85.694	0.3500	14.79	86.331
0.1694	79.85	110.977	0.3499	19.83	100.946
			0.3499	19.97	101.339
			0.3496	39.48	158.606
0.2262	12.39	54.254	0.3492	59.39	217.360
0.2262	18.75	62.110			
0.2262	19.60	63.169	0.3675	−3.59	42.798
0.2259	39.82	88.745	0.3675	−0.96	51.154
0.2254	80.49	140.782	0.3675	3.22	64.546
			0.3674	6.02	73.601
0.2907	20.00	72.000	0.3674	8.82	82.727
0.2907	20.54	73.033	0.3674	12.05	93.272
0.2904	39.94	110.666	0.3674	14.67	101.806
0.2901	59.88	150.154	0.3674	14.92	102.684
0.2896	89.31	208.735	0.3673	19.28	116.967
0.3022	8.44	51.821	0.3694	−3.21	45.163
0.3022	10.87	56.564	0.3693	0.21	56.202
0.3022	15.15	65.175	0.3692	5.76	74.348
0.3021	19.55	74.114	0.3691	12.16	95.472
0.3021	19.97	75.008	0.3689	19.95	121.385
0.3021	24.00	83.351	0.3689	20.07	121.775
0.3021	27.41	90.464	0.3685	40.11	188.838
0.3021	30.15	96.207	0.3681	59.71	254.397

Table III. Continued

ρ (kg/dm^3)	t (IPTS-68) (°C)	p (bar)	ρ (kg/dm^3)	t (IPTS-68) (°C)	p (bar)
0.3809	−5.21	47.514	0.4094	0.55	109.852
0.3809	−2.52	57.033	0.4090	19.56	193.415
0.3809	−2.04	58.766	0.4090	19.88	194.829
0.3809	1.07	69.791	0.4087	29.39	236.470
0.3809	3.41	78.126			
0.3809	4.97	83.709	0.4257	−19.93	45.029
0.3808	8.47	96.266	0.4256	−16.62	61.245
0.3808	8.61	96.800	0.4255	−10.65	90.484
0.3958	−9.51	46.527	0.4254	−6.33	111.681
0.3956	−1.70	77.385	0.4252	−0.25	141.522
0.3953	9.11	120.474	0.4247	20.35	242.210
0.3951	20.08	164.384	0.4247	20.39	242.381
0.3951	20.14	164.620			
0.3951	20.41	165.678	0.4451	−29.42	36.876
0.3947	39.94	243.633	0.4450	−24.09	66.953
			0.4449	−19.43	93.221
0.4098	−14.61	43.312	0.4447	−14.66	120.034
0.4097	−10.10	63.100	0.4445	−4.96	174.474
0.4095	−4.03	89.700	0.4442	5.18	231.001

[a] Isochores determined with constant volume apparatus.

expansion apparatus described in Section 2 are reported in Table II and are also shown in Fig. 3. Several series of expansions were made on each isotherm, starting at different initial pressures selected to give nearly evenly spaced data points. The p, ρ, t data determined with the constant volume apparatus are reported in Table III and shown in Fig. 4.

6. EQUATION OF STATE

For custody transfer purposes, density tables for ethylene were produced in 1976 [3]. An extended Benedict-Webb-Rubin equation of state (BWR) was developed to represent the data in the liquid and gaseous region:

$$p = RT\rho + \left(A_1 + A_2 T + \frac{A_3}{T^2} + \frac{A_4}{T^4}\right)\rho^2 + \left(A_5 + A_6 T + \frac{A_7}{T^2}\right)\rho^3$$
$$+ \left(\frac{A_8}{T^2} + \frac{A_9}{T^4}\right) e^{-\beta\rho^2}(1 + \beta\rho^2)\,\rho^3 + A_{10}\,\rho^6 + A_{11}\frac{\rho^4}{T^6} + A_{12}\frac{\rho^5}{T^6} \qquad (9)$$

The coefficients of this equation were computed from the PVT data of this work (Tables I–III) and the 67 measured PVT values of Michels and

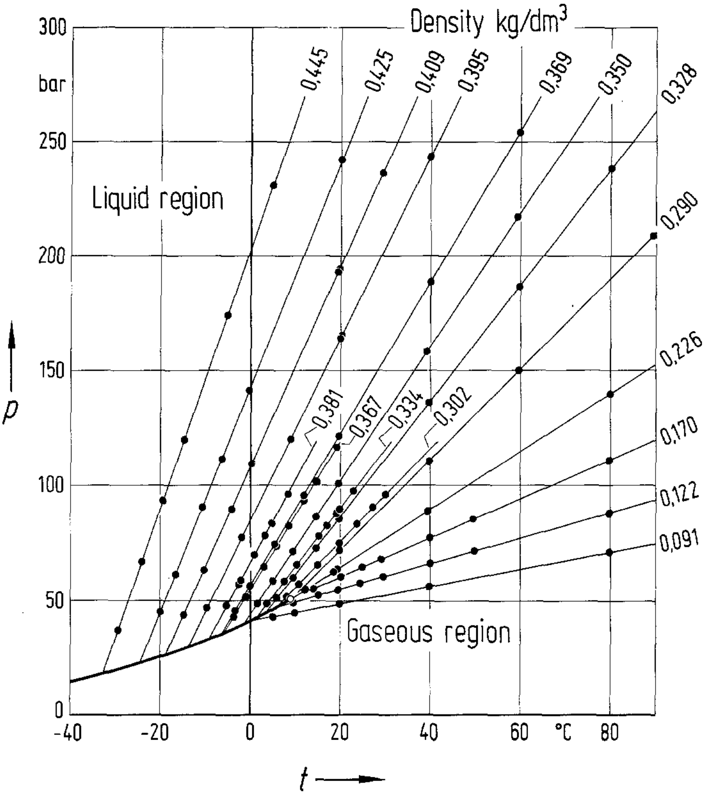

Fig. 4. Experimental p, ρ, t data for ethylene (isochores determined with constant volume apparatus).

Table IV. BWR Coefficients for Ethylene[a]

A	Subregion I	Subregion II	Subregion III
1	$-0.99440284 \times 10^{-2}$	0.13487387	$-0.10999985 \times 10^{-2}$
2	$0.15538290 \times 10^{-4}$	$-0.22284642 \times 10^{-3}$	$0.88948646 \times 10^{-6}$
3	$0.17517303 \times 10^{+3}$	$-0.95475961 \times 10^{+4}$	$-0.41031389 \times 10^{+3}$
4	$-0.81264911 \times 10^{+7}$	$0.26697125 \times 10^{+9}$	$0.83195243 \times 10^{+7}$
5	$0.14945133 \times 10^{-4}$	$-0.24313919 \times 10^{-4}$	$-0.24922766 \times 10^{-5}$
6	$-0.17142165 \times 10^{-7}$	$-0.46898016 \times 10^{-8}$	$0.19623448 \times 10^{-7}$
7	-0.47193713	$0.41394847 \times 10^{+1}$	0.15632716
8	0.29932596	-0.30684211	-0.16546068
9	$-0.64351593 \times 10^{+4}$	$0.21585671 \times 10^{+5}$	$0.46402462 \times 10^{+5}$
10	$0.43850930 \times 10^{-13}$	$0.15309629 \times 10^{-13}$	$0.46656437 \times 10^{-13}$
11	$0.89877298 \times 10^{+7}$	$-0.38226661 \times 10^{+8}$	$-0.20314817 \times 10^{+7}$
12	$-0.19440328 \times 10^{+5}$	$0.44958369 \times 10^{+5}$	$0.30823383 \times 10^{+4}$
β	$+0.26 \times 10^{-4}$	$+0.4 \times 10^{-4}$	$+0.18 \times 10^{-4}$

[a] Units: pressure p in bar, temperature T in K, density ρ in kg/m^3, $R = 0.00296367$ m^3 bar/kg K.

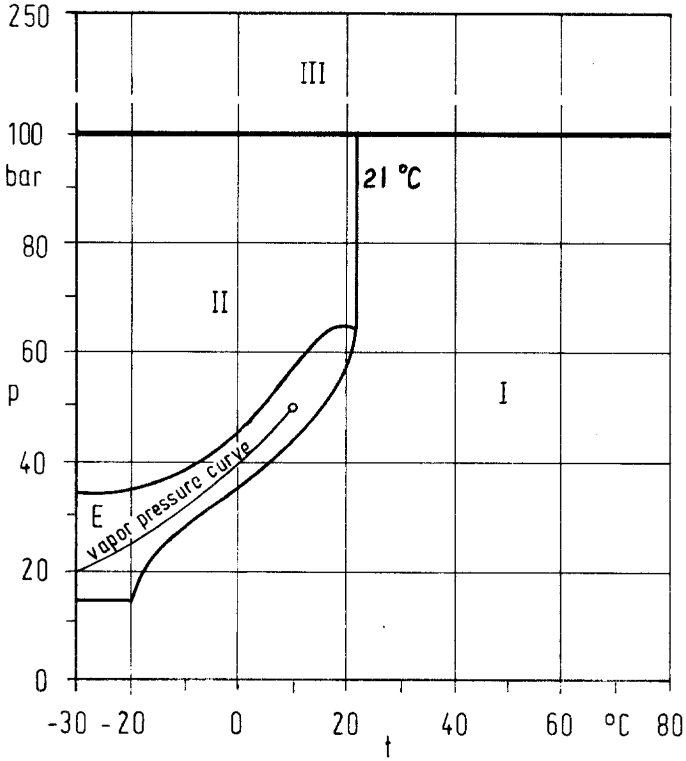

Fig. 5. Subregions of the BWR equation of state (E: excluded region).

Geldermans [8], corrected slightly according to their reference in a later paper [9] and also to the 1968 IPTS scale. It was found that one equation of state was not sufficient to represent all the input data to better than 0.1%, as demanded for the tables. Therefore, the three subregions I, II, and III were chosen, which are identified by Fig. 5. Table IV gives the BWR coefficients A_1 to A_{12} and β to be used in each subregion. The region E in Fig. 5 was not taken into consideration because a state near the vapor pressure curve and especially in the neighborhood of the critical point cannot be used as a delivery state in the meter stations of pipeline systems.

7. COMPARISON WITH OTHER PVT WORK

After our density tables [3] had been published, three major papers have been issued. Trappeniers et al. [10] repeated the measurements of Michels and Geldermans [8], and on the whole confirmed the results published in 1942, which we also have taken as input data for the calculation of the BWR coefficients. The PVT measurements on ethylene made by Douslin and

Harrison [11] are in good agreement with our results. The smoothed densities in the single-phase region correspond with our values to within 0.2%.

Recently Waxman and Davis [12] presented values of the second virial coefficient of ethylene between 0 and 175°C, determined from low-pressure Burnett PVT measurements. Their values are in good agreement with those obtained by Douslin and Harrison [11], which we just have discussed.

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