J. Chem. Eng. Data **2001,** *46,* 1089−1094

PVT Measurements for Toluene in the Near-Critical and Supercritical Regions[†]

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The *PVT* relationships of toluene were measured in the near-critical and supercritical regions. Measurements were made using a constant-volume piezometer immersed in a precision thermostat. The volume of the piezometer V_{PT} was corrected for both temperature and pressure expansions. The temperature was measured with a 10 Ω platinum resistance thermometer (PRT-10) with an uncertainty of 10 mK. Pressure was measured by means of a dead-weight gauge with an uncertainty of (0.0015 to 0.002) MPa. Uncertainties of the density measurements are estimated to be (0.05 to 0.10)%, depending on the experimental pressure and temperature. The volume of the piezometer V_{PT} was calibrated using a reference fluid (water) with well-known *PVT* properties at various temperatures and pressures. Measurements were made along various near-critical and supercritical isotherms between (591 and 673) K at pressures from (4 to 36) MPa. The measured *PVT* data for toluene were compared with values calculated from equations of state and previous measurements of other authors. The accuracy of the method was confirmed by *PVT* measurements for pure water in the critical and supercritical regions.

1. Introduction

Previously, the *PVT* properties of toluene have been studied by a number of authors.[1−14] Most of the previous *PVT* measurements for toluene were performed at temperatures up to 450 K except for the measurements of Straty et al.,[10] Akhundov and Abdullaev,[11,12] and Franck et al.[14] for which measurements were made at temperatures up to 673 K and at pressures up to 300 MPa. Akhundov and Abdullaev[11] presented very extensive experimental *PVT* data for toluene in the temperature range from (573 to 673) K at pressures up to 50 MPa. The uncertainty of these data is 0.05% for densities about 1000 $kg·m^{-3}$ and about 0.1% for densities lower than 1000 $kg·m^{-3}$. Measurements have been made using two piezometers with different volumes of 125 cm^3 and 890 cm^3. The differences between the measurements are (0.01 to 0.02)%. The pressure was measured with dead-weight pressure gauges MP-600 (at high pressures) and MP-60 (at low pressures) with an uncertainty of 0.01%. The sample had a purity of 99.98 mass %. Akhundov and Abdullaev[12] reported *PVT* measurements for toluene in the critical region along 13 near-critical isotherms between (591.15 and 603.15) K, densities from (275.00 to 359.51) $kg·m^{-3}$, and pressures between (4.2 and 4.9) MPa. The five isotherms (592.15, 593.15, 593.95, 598.15, and 603.15) K were measured two times to test the reproducibility of the data. The difference between the test measurements are (0.02 to 0.03)%. The pressure was measured using the MP-60 with a uncertainty of ±0.02%. The uncertainty of the density measurements is about (±0.02 to ±0.03)%. From the *PVT* measurements, the following critical parameters were extracted: T_c = 593.85 K, P_c = 4.2358 MPa, and ρ_c = (289.77 ± 3) $kg·m^{-3}$. The data presented by Straty et al.[10] cover the temperature range between (348 and 673) K at pressures up to 35 MPa. They determined 27 quasi-isochores from (156.6 to 829.2) $kg·m^{-3}$. Except for the critical region, the estimated uncertainty of density measurements is 0.2% at the highest densities and 0.35% at the lowest densities. The agreement between the Akhundov and Abdullaev[11,12] data and the Straty et al.[10] data is about 0.4%, except in the critical region where the differences reached up to about 1.5%. The maximum deviations are about (1.2 to 1.9)% along the isotherm 623.15 K at densities between (285.6 and 534.4) $kg·m^{-3}$. In general, there is excellent consistency between the Akhundov and Abdullaev[11,12] data and the Straty et al.[10] data. These data can be used as an experimental basis for developing fundamental equations of state (EOS). The density of toluene has been measured from (323 to 673) K and between (5 and 300) MPa by Franck et al.[14] along 16 isochores with densities from (335 to 950) $kg·m^{-3}$. Their measurements were made using a cylindrical autoclave with a volume of 66.3 cm^3. These data show good agreement to within 0.2% with the data by Magee and Bruno[9] and Pöhler and Kiran[13] at temperatures up to 423 K and pressures up to 50 MPa. The differences between the Franck et al.[13] data and measurements by Akhundov and Abdullaev[11,12] and Straty et al.[10] at high temperatures and low pressures (P < 50 MPa) reached up to (1.2 to 1.5)%. Magee and Bruno[9] reported data in the temperature range from (180 to 400) K and at pressures up to 35 MPa. Measurements were carried out on 20 liquid isochores between (791 and 973) $kg·m^{-3}$. The pressure was measured with an uncertainty

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[†] This contribution will be part of a special print edition containing papers presented at the Fourteenth Symposium on Thermophysical Properties, Boulder, CO, June 25−30, 2000.

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10.1021/je0002686 CCC: $20.00

of 0.01% at $P > 3$ MPa and 0.05% at $P < 3$ MPa. The uncertainty of density measurements is 0.05%. Measurement were made using a nearly constant volume container with a volume of (28.5193 ± 0.003) cm^3. These data show good agreement to within 0.2% with data derived by Akhundov and Abdullaev,[11,12] Kashiwagi et al.,[4] and Muringer et al.[6] Excellent agreement within 0.05% was reported for the Magee and Bruno[9] and Straty et al.[10] data. A Kay-type apparatus was used to measure the *PVT* of gaseous toluene from about (423 to 498) K by Marcos et al.[5] The values of the second virial coefficients were calculated for the measured isotherms (423.26, 448.13, 473.19, and 498.29) K.

Goodwin[15] developed a nonanalytic EOS for toluene for temperatures from the triple point (178.15 K) to 800 K, with pressures to 100 MPa using the *PVT* measurements by Straty et al.[10] and Akhundov and Abdullaev.[11,12] Franck et al.[14] developed a Tait type EOS for toluene. A simplified EOS for toluene was developed by Mamedov et al.[16,17] Recently, Lemmon and Jacobsen[18] developed a preliminary fundamental EOS for toluene which was fit to the available high-accuracy experimental *PVT*, C_p, and speed of sound data.

Toluene is often used as a solvent, and reliable thermodynamic properties are of direct technological importance. Scientific and technological interest in toluene arises because of its unusually wide liquid temperature range, which makes it a suitable reference fluid for calibration purposes and apparatus validation. Standard reference data of the transport properties of toluene have already been established (Nieto de Castro et al.[19]). This paper is a contribution in a program to provide data for the formulation of a standard reference of the thermodynamic properties of toluene (Lemmon and Jacobsen[18]).

2. Experimental Procedure

The present measurements of the *PVT* properties for toluene have been performed with a constant-volume method of extracting sample from the piezometer under isothermal conditions. The method of measurements, experimental procedure, and construction of the piezometer have been discussed in detail elsewhere (Abdulagatov et al.[20−28]). A cylindrical piezometer horizontally mounted in the center of an air thermostat was used. A piezometer was made of heat- and corrosion-resistant high-strength alloy EI-437B4. The thermostat has double walls, and it has an inside volume of 65 dm^3. The heating elements were arranged between the walls. To minimize temperature gradients in the air thermostat, two electrically driven high-speed fans were used. The temperature inside the thermostat was maintained uniform within 5 mK with the aid of guard heaters located between the thermostat walls and regulating heater which was mounted inside the thermostat. The temperature inside the thermostat and fluid temperature were controlled automatically (Bazaev[29]). The fluid temperature was detected by a 10 Ω platinum resistance thermometer (PRT-10) with a precision of ±10 mK and was calibrated against the ITS-90 within an uncertainty of 1 mK. The 10 Ω resistance of the thermometer (PRT-10) at the ice melting point is $R_0 = 9.9358 \pm 0.0005$ Ω. The temperature inside the thermostat was found during an experimental run to be the same as that of the sample of toluene confined in the piezometer. The pressure exerted on the piezometer was transmitted to another liquid-octane system through a diaphragm-type null indicator which was mounted on the end of the piezometer and has a precision of 2 kPa. The transferred liquid-octane pressure is transmitted to a dead-weight pressure gauge MP-600 with a resolution of 5 kPa. The diaphragm had a diameter of 40 mm and is 0.08 mm thick. A stirring ball was placed in the piezometer to agitate the mixtures.

The sample of toluene was transferred from the supply vessels to the piezometer using hand-operated screw presses. The toluene in the piezometer was then heated in the thermostat until its temperature reached the prescribed value and the pressure reached a maximum value of about 40 MPa. After thermal equilibration, the *PVT* measurements along the selected isotherm were made, starting from the maximum pressure of about 40 MPa. Measurements continued by extracting an appropriate amount of sample from the piezometer through a needle valve. The extracted samples were collected in separate collectors and weighed using a high-precision chemical balance with an uncertainty of 0.05 mg. Several data points were taken along each isotherm until the system pressure became about 4 MPa.

The density of the sample at a given temperature T and pressure P is determined from the simple relation

$$\rho_i = M_i/V_{TP}, \quad M_i = m_t - m_i, \quad (i = 1, N), \qquad m_t = m_1 + m_2 + m_3 + ... + m_N \tag{1}$$

where V_{TP} is the temperature- and pressure-dependent volume of the piezometer, M_i is the current mass of the toluene in the piezometer after each extraction, m_i is the mass of toluene at each extraction from the piezometer during the runs, m_t is the total mass or initial mass of the toluene in the piezometer, and N is the number of extractions during the runs or number of experimental data points for each run. After the last extraction, the total mass m_t of the toluene in the piezometer was calculated. The density of the toluene for a given temperature T and pressure P was calculated using eq 1.

Since the inner volume V_{TP} of the piezometer is subjected to the effects of both temperature T and pressure P, it is essential to make appropriate corrections for these influences. The inner volume of the piezometer was calculated taking into consideration the corrections of the elastic pressure deformation and thermal expansion. The volume of the piezometer was previously calibrated from the known density of a standard fluid (pure water) with well-established *PVT* information (IAPWS-95 formulation, Wagner and Pruss[30]) at a temperature of $T_0 = 673.15$ K and a pressure of $P_0 = 38.54$ MPa. The volume at these conditions was $V_{P_0T_0} = (32.210 \pm 0.003)$ cm^3. The variations of the volume depending on the temperature T and the pressure P were calculated from the equation

$$V_{TP} = V_{T_0P_0}[1 + 3\alpha(T - T_0) + \beta(P - P_0)] \tag{2}$$

where $\alpha = 1.3 \times 10^{-5}$ K^{-1} is the thermal expansion coefficient of the alloy (EI-437B4) which is virtually temperature-independent over a broad temperature range from (500 to 700) K, and $\beta = 4.12 \times 10^{-5}$ MPa^{-1} is the pressure expansion coefficient of the piezometer. The volume of the piezometer was measured with an uncertainty of (0.01 to 0.03)%. The present experimental apparatus had no “dead volumes”. Taking into account the uncertainties of temperature and pressure measurements, the total experimental uncertainty of density was estimated to be within (0.05 and 0.1)%, depending on the temperature T and pressure P. A high-purity (HPLC grade, 99.8 mol %) toluene sample was used in this work and a companion[31] study.

Table 1. Experimental Values of the *PVT* Data of Toluene

	P/MPa at the following ρ/kg·m^{-3} (run 1) values						
T/K	565.70	546.89	523.97	491.52	463.39	403.64	305.04
595.15	14.146	11.542	9.215	6.953	5.883	4.768	4.453
596.15	14.376	11.735	9.396	7.095	6.016	4.865	4.511
598.15	14.803	12.125	9.755	7.392	6.262	5.045	4.624
603.15	15.875	13.106	10.627	8.113	6.888	5.500	4.907
613.15	18.037	15.081	12.353	9.577	8.124	6.407	5.471
623.15	20.204	17.046	14.078	11.023	9.366	7.314	6.035
633.15	22.359	19.029	15.825	12.488	10.605	8.224	6.602
643.15	24.515	21.001	17.562	13.948	11.843	9.129	7.166
645.15	24.953	21.404	17.895	14.241	12.096	9.310	7.281
647.096	25.377	21.773	18.249	14.533	12.336	9.488	7.389
649.15	25.817	22.176	18.588	14.827	12.590	9.674	7.506
651.15	26.247	22.573	18.937	15.110	12.834	9.854	7.619
653.15	26.685	22.940	19.284	15.409	13.082	10.032	7.727
663.15	28.873	24.875	21.041	16.857	14.312	10.970	8.255
673.15	31.105	26.875	22.783	18.322	15.577	11.897	8.870

	P/MPa at the following ρ/kg·m^{-3} (run 1) values					
T/K	582.84	537.14	497.28	465.37	374.69	162.53
591.15	16.355	9.690	6.635	5.516	4.328	4.017
592.15	16.603	9.870	6.788	5.633	4.402	4.049
593.15	16.835	10.070	6.942	5.768	4.478	4.071
594.15	17.065	10.250	7.101	5.884	4.558	4.102
595.15	17.295	10.440	7.254	6.023	4.618	4.127
596.15	17.525	10.621	7.408	6.152	4.714	4.156
623.15	23.865	15.650	11.490	9.585	6.855	4.847
647.096	29.504	20.047	15.151	12.603	8.714	5.448
673.15	35.547	24.931	19.157	15.956	10.812	6.132

3. Results and Discussion

Measurements of the *PVT* relationship of the toluene were performed along ninteen near-critical and supercritical isotherms, namely (591.15, 592.15, 593.15, 594.15, 595.15, 596.15, 598.15, 603.15, 613.15, 623.15, 633.15, 643.15, 645.15, 647.096, 649.15, 651.15, 653.15, 663.15, and 673.15) K. The density ranged between (162 and 583) kg·m^{-3}, and the pressure ranged from (4 to 36) MPa. The experimental temperatures, densities, and pressures for near-critical and supercritical toluene are presented in Table 1. To check the reproducibility, the measurements at selected isotherms (595.15, 596.15, 623.15, 647.096, and 673.15) K were repeated (runs 1 and 2, see Table 1). The differences between these measurements are within the experimental uncertainties. The experimental isochoric and isothermal data measured for toluene are plotted in Figures 1 and 2. Comparison of the present *PVT* measurements with data reported by Straty et al.,[10] by Akhundov and Abdullaev,[11,12] and by Mamedov et al.[16,17] together with values calculated from the preliminary Lemmon and Jacobsen[18] EOS and the Goodwin[15] EOS along various near- and supercritical isotherms is shown in Figure 3. Figure 4 depicts deviations of the experimental densities from the values calculated using the preliminary fundamental EOS by Lemmon and Jacobsen.[18] This figure does not show a few data points for which the deviations are larger than 2%. Deviation statistics for the 120 measured densities are as follows: AAD = 0.65%, bias = 0.64%, std dev = 0.59%, std err = 0.06%, and max. dev = 2.18%. The maximum deviation is observed in the immediate vicinity of the critical pressure and the critical temperature. All of the measured density data show good agreement to within ±0.5% with values calculated from the Lemmon and Jacobsen[18] EOS at pressures higher than 15 MPa. Most of the data at pressures higher than 5 MPa show deviations within ±1%. Only the data in the temperature and pressure ranges close to the critical point show deviations of about 2%. Figure 5 shows the deviations between *PVT* data reported in the literature and values calculated from the preliminary EOS by Lemmon and Jacobsen[18] for toluene together with values presented in this work at the temperature 663.15 K. Most of the data show similar deviations. Therefore, agreement between these data sets is good, except for a few points, where deviations reached up to 1.2%.

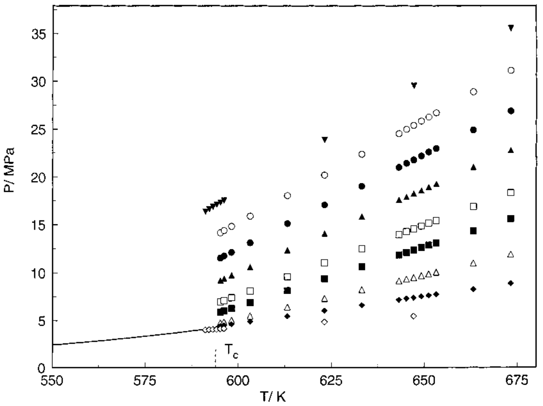

Figure 1. Measured pressures of toluene as a function of temperature along different densities in the near-critical and supercritical regions: ○, 566.29 kg·m^{-3}; ●, 547.45 kg·m^{-3}; △, 404.06 kg·m^{-3}; ▲, 524.51 kg·m^{-3}; ■, 463.87 kg·m^{-3}; ▼, 582.84 kg·m^{-3}; ◆, 305.35 kg·m^{-3}; □, 492.03 kg·m^{-3}; ◇, 162.53, kg·m^{-3}; —, vapor pressure curve from the Lemmon and Jacobsen[18] EOS.

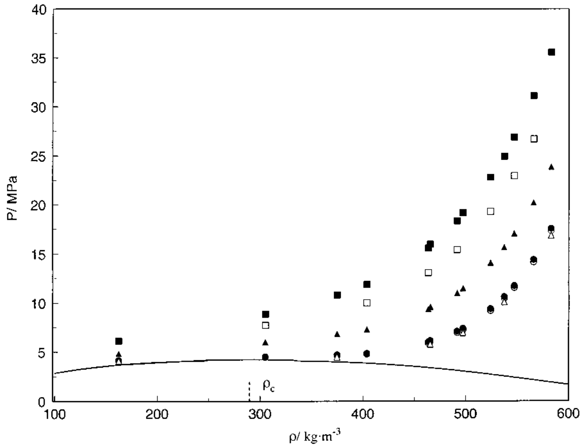

Figure 2. Measured pressures of toluene as a function of density along different near-critical and supercritical isotherms: ○, 595.15 K; ●, 596.15 K; △, 593.15 K; ▲, 623.15 K; ■, 673.15 K; □, 653.15 K; —, coexistence curve from the Lemmon and Jacobsen[18] EOS.

Table 2. Experimental Values of the *PVT* Data of Pure Water

T = 653.15 K		T = 673.15 K	
P/MPa	ρ/kg·m^{-3}	P/MPa	ρ/kg·m^{-3}
29.25	527.79	40.30	527.13
25.18	460.42	33.87	459.89
23.48	290.71	28.65	290.42
22.99	205.73	26.53	205.24
20.25	125.34	22.32	125.59
16.61	82.440	17.90	82.680
12.58	54.390	13.32	54.340

To check and confirm the accuracy of the method, the *PVT* measurements were made with pure water along one near-critical isotherm and one supercritical isotherm, (653.15 and 673.15) K. The measured values are presented in Table 2. The measured values of density and pressure on these isotherms are shown in Figure 6 together with values calculated from the IAPWS-95 formulation[30] and

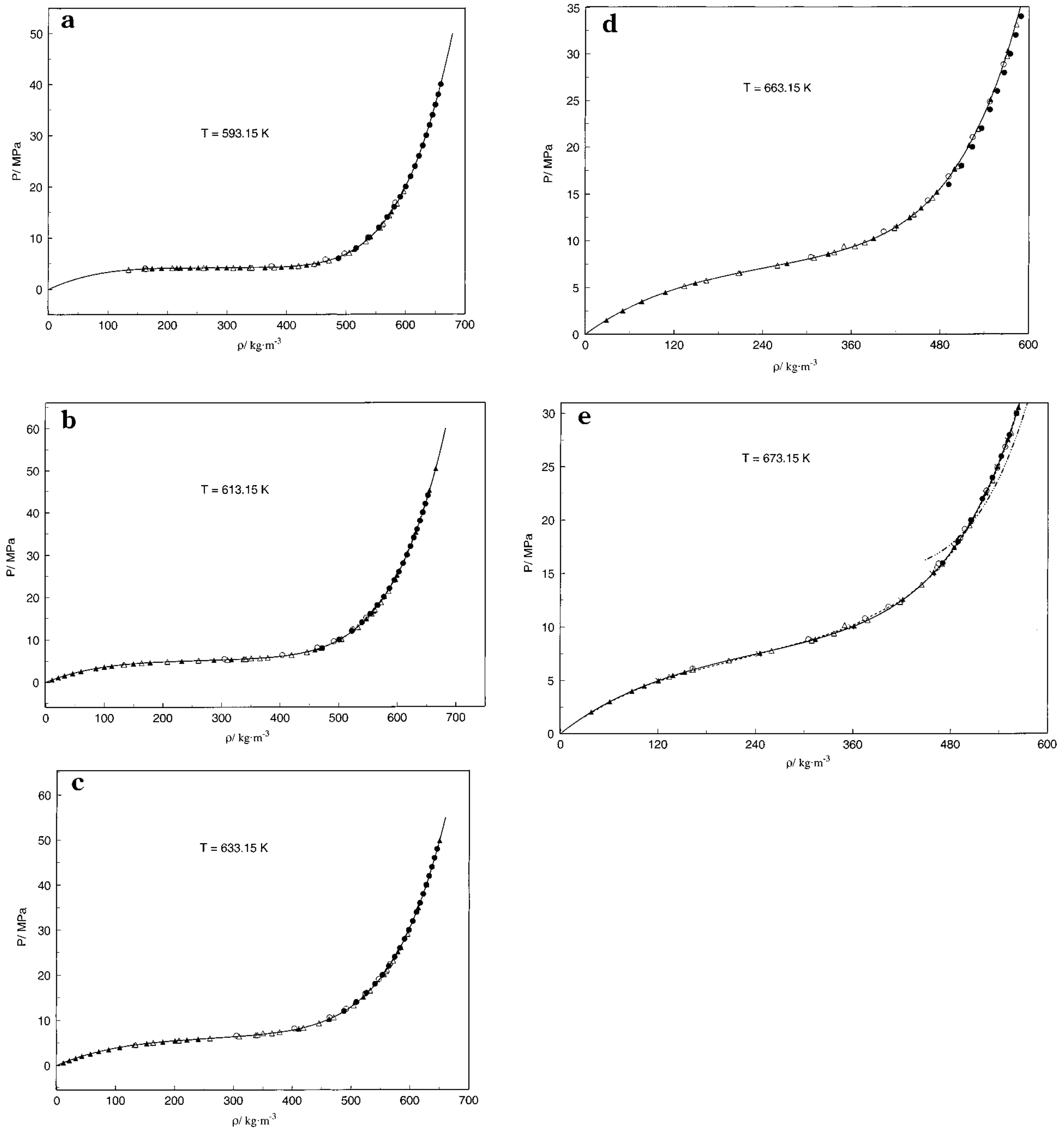

Figure 3. (a−e) Comparison of measured *PVT* data for toluene with values calculated from the Lemmon and Jacobsen[18] EOS: ○, this work; ▲, Akhundov and Abdullaev;[11] △, Straty et al.;[10] ●, Mamedov et al.;[16] ×, Mamedov and Akhundov;[17] —, Lemmon and Jacobsen[18] EOS; - - -, Goodwin[15] EOS; — • • •—, Franck et al.[14]

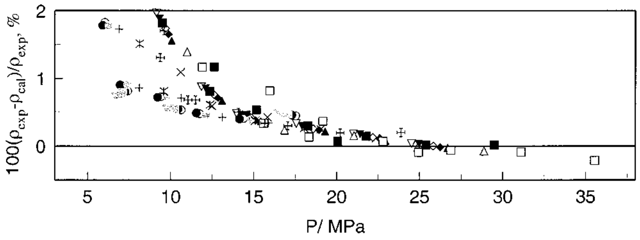

Figure 4. Percentage density deviations, $\delta\rho = 100((\rho_{exp} - \rho_{cal})/\rho_{exp})$, of the experimental densities for toluene from the values calculated with the preliminary Lemmon and Jacobsen[18] EOS: ×, 633.15 K; ∇,643.15; ▼, 645.15 K; ◆, 651.15; ◇, 649.15 K; ▲, 653.15; △, 663.15 K; ■, 647.15 K; □, 673.15 K; ●, 595.15 K; ○, 596.15 K; +, 603.15 K; Maltese cross, 623.15 K.

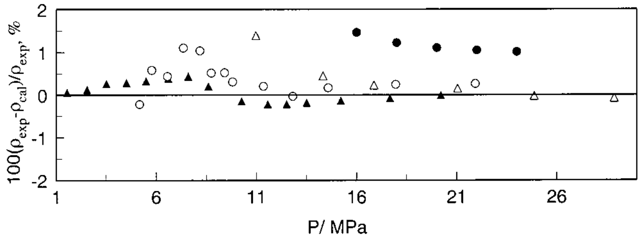

Figure 5. Percentage density deviations, $\delta\rho = 100((\rho_{exp} - \rho_{cal})/\rho_{exp})$, of the experimental densities for toluene from the values calculated with the preliminary Lemmon and Jacobsen[18] EOS along the isotherm 663.15 K: ▲, Akhundov and Abdullaev;[11] △, this work; ●, Mamedov and Akhundov;[16] ○, Straty et al.[10]

previously reported high-accuracy water *PVT* experimental data. Deviation statistics for the 14 measured densities are as follows: AAD = 0.44%, bias = 0.16%, std dev = 0.46%, std err = 0.12%, and max. dev = 0.7%. A deviation plot for

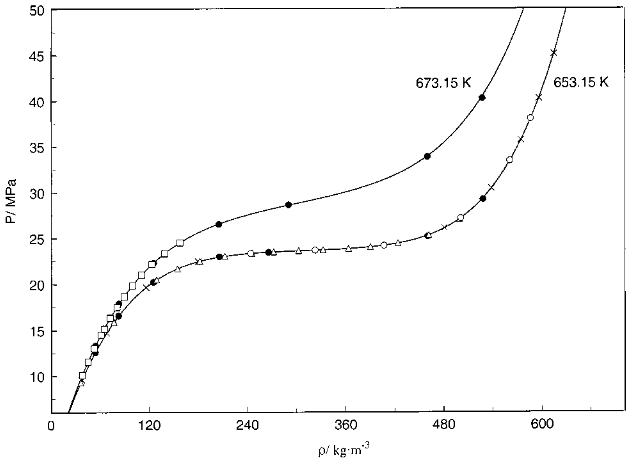

Figure 6. Comparison of measured *PVT* data for water with values calculated from the IAPWS-95 formulation:[30] ○, Hanafusa et al.;[32] △, Rivkin and Troyanovskaya;[35] ●, this work; ×, Alexandrov et al.;[33] □, Kell et al.;[34] —, IAPWS-95 formulation.[30]

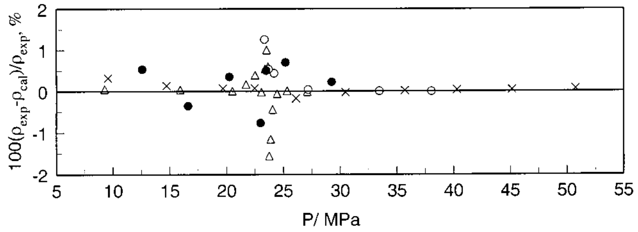

Figure 7. Percentage density deviations, $\delta\rho = 100((\rho_{exp} - \rho_{cal})/\rho_{exp})$, of the experimental densities for water from the values calculated with the IAPWS-95 formulation[30] along the isotherm 653.15 K: ×, Alexandrov et al.;[33] △, Rivkin and Troyanovskaya;[35] ●, this work; ○, Hanafusa et al.[32]

the present measurements, including data from the literature for pure water on the near-critical isotherm 653.15 K, is shown in Figure 7. One can see from Figures 6 and 7 that the published data sets show almost the same character of deviations as the present data. All of the data show that good agreement exists between the sets.

4. Conclusion

By means of a constant-volume piezometer, *PVT* properties for toluene have been measured in the range of temperatures from (591 to 673) K and pressures from 4 to 36 MPa with an estimated uncertainty of (0.05 to 0.1)%. Most of the measured data show good agreement within ±1% with other measurements and calculations by the preliminary fundamental EOS of Lemmon and Jacobsen.[18] The present *PVT* data together with isochoric heat capacity data reported in our companion paper by Polikhronidi et al.[31] can be recommended to develop a new crossover EOS for toluene in a broad range around the critical point. The accuracy of the method was confirmed by measurements of the *PVT* properties of pure water in the critical and supercritical regions.

Acknowledgment

The authors thank E. W. Lemmon for providing software for calculating thermodynamic properties of toluene from the preliminary fundamental EOS. One of us (I.M.A.) also thanks the Physical and Chemical Properties Division at the National Institute of Standards and Technology for the opportunity to work as a Guest Researcher at NIST during the course of this research.

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Received for review August 10, 2000. Accepted April 26, 2001. This work was supported by Grants RFBR 00-02-17856 and INTAS -96-1989.

JE0002686