Density and Viscosity of Carbon Tetrachloride Solutions Containing Chlorine

Nguyen Q. Hien, Anthony B. Ponter,* and Walter Peier
Chemical Engineering Institute, Swiss Federal Institute of Technology of Lausanne, Switzerland

Densities and viscosities at temperatures of 12, 15, 20, 25, 30, and 35 °C have been measured for carbon tetrachloride and solutions of carbon tetrachloride containing different concentrations of chlorine. A comparison has been made between these data and those reported in the literature.

The literature on physical constants for gas-containing liquids is relatively scarce. For the carbon tetrachloride–chlorine system, the density and viscosity data of Lewis (*1*) and Clegg (*2*) are not in agreement. To determine the absorption mechanism for this system where a large heat release occurs, a knowledge of accurate physical data is necessary. This investigation was carried out to assess the reliability of the published data.

Experimental Section

Chlorine of 99.9% purity was dissolved in carbon tetrachloride in a constant-temperature reactor for at least 1 h to obtain a near-saturated solution. This solution in turn was split into approximately ten portions in gas-tight conical flasks containing different carbon tetrachloride volumes to provide a range of chlorine-containing mixtures. The flasks were maintained at the desired temperature in a water bath and the solutions were transferred directly to the densimeter or viscometer through a Teflon tube leading from the flask. Two series of titrations on samples of liquid entering and leaving the measuring instruments revealed no detectable loss of chlorine for any of the concentrations investigated. The chlorine concentrations were determined by automatic titration with potassium iodide and sodium thiosulfate solutions.

Liquid densities were measured using a Mettler DMA 50 precision digital densiometer which compared the period of oscillation of a U-shaped glass cell filled with the specimen liquid with that of the cell filled with calibrated fluids and which was accurate to $\pm 1 \times 10^{-4}$ g cm^{-3}. Temperature control using a calibrated thermometer was better than ±0.05 °C. Viscosities were measured using a Schott-Mainz Ubbelohde-type viscometer equipped with a digital timer accurate to 0.1 s which gave an error in the measurement of kinetic viscosities of ±0.1%. Temperature control was better than ±0.01 °C.

Results and Discussion

The density and viscosity data are presented in Table I and correlated in Table II.

The data are compared with those of Lewis (*1*) and Clegg and Mann (*2*) in Figures 1 and 2.

For the densities studied it is observed that at 25 °C Lewis data measured with a simple pycnometer gave slightly lower values (approximately 0.2%) than those reported in this investigation. The viscosity data, however, were significantly different. The values reported by Clegg and Mann are ca. 8% higher at approximately 0.16 mole fraction chlorine. No mention is made by these authors regarding the apparatus used for measuring viscosities, but the results are consistent with a loss of chlorine

Table I. Density and Viscosity Data for Chlorine-Containing Solutions of Carbon Tetrachloride at 12–35 °C

Temp, °C	Mole fraction	Density, g/cm^3	Kinematic viscosity, cSt	Viscosity, cP
12	0	1.6095	0.679	1.093
	0.0290	1.6081	0.670	1.077
	0.0301	1.6080	0.668	1.074
	0.0615	1.6067	0.657	1.055
	0.0746	1.6061	0.648	1.042
	0.0891	1.6055	0.645	1.035
	0.1340	1.6036	0.625	1.003
	0.1709	1.6021	0.607	0.972
	0.1823	1.6017	0.602	0.964
	0.1878	1.6015	0.599	0.960
15	0	1.6037	0.652	1.045
	0.0291	1.6023	0.642	1.028
	0.0532	1.6014	0.633	1.013
	0.0534	1.6013	0.630	1.009
	0.0888	1.5998	0.617	0.987
	0.1107	1.5988	0.605	0.968
	0.1333	1.5980	0.604	0.965
	0.1704	1.5963	0.583	0.931
	0.1804	1.5959	0.579	0.924
20	0	1.5940	0.610	0.972
	0.0299	1.5928	0.599	0.955
	0.0302	1.5927	0.598	0.953
	0.0377	1.5923	0.596	0.949
	0.0388	1.5922	0.595	0.948
	0.0435	1.5921	0.595	0.947
	0.0454	1.5920	0.594	0.946
	0.0468	1.5919	0.594	0.946
	0.0672	1.5910	0.587	0.934
	0.0874	1.5901	0.578	0.920
	0.0917	1.5900	0.576	0.916
	0.1326	1.5882	0.564	0.896
	0.1704	1.5865	0.550	0.872
25	0	1.5843	0.572	0.906
	0.0318	1.5832	0.561	0.889
	0.0367	1.5828	0.559	0.885
	0.0409	1.5827	0.558	0.883
	0.0565	1.5820	0.552	0.874
	0.0882	1.5806	0.544	0.860
	0.1032	1.5798	0.539	0.852
	0.1074	1.5796	0.538	0.850
	0.1097	1.5795	0.537	0.848
	0.1309	1.5786	0.531	0.839
	0.1448	1.5779	0.525	0.829
30	0	1.5746	0.537	0.846
	0.0309	1.5734	0.529	0.832
	0.0461	1.5727	0.524	0.824
	0.0534	1.5723	0.521	0.819
	0.0560	1.5722	0.519	0.815
	0.0867	1.5708	0.511	0.803
	0.1300	1.5688	0.501	0.786
	0.1350	1.5684	0.499	0.783
35	0	1.5648	0.506	0.792
	0.0299	1.5636	0.498	0.778
	0.0451	1.5629	0.493	0.771
	0.0468	1.5627	0.492	0.770
	0.0553	1.5624	0.492	0.768
	0.0838	1.5610	0.484	0.756
	0.1114	1.5596	0.476	0.743

---

* To whom correspondence should be addressed care of the Chemical Engineering Department, University of Aston, Birmingham, United Kingdom.

0021-9568/78/1723-0054$01.00/0

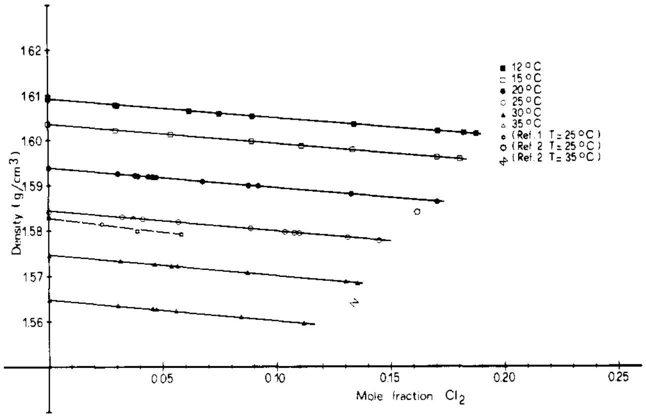

Figure 1. Comparison of density data with those of Lewis.

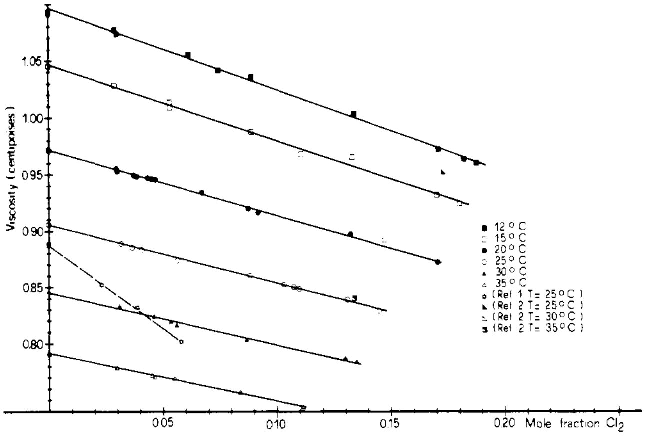

Figure 2. Comparison of viscosity data with those of Lewis and Clegg and Mann

Table II. Empirical Equations from Linear Regression Model: $y = B_0 + B_1x$

T, °C	y	B_0	B_1	N	Student's t^2 value at 97.5%	ΔB_1
12	ρ	1,6093	−0,0422	10	2.306	±0.0010
	ν	0.6816	−0.4333			±0.0178
	μ	1.0968	−0.7221			±0.0274
15	ρ	1.6036	−0.0429	9	2.365	±0.0009
	ν	0.6529	−0.4029			±0.0316
	μ	1.0469	−0.6710			±0.0507
20	ρ	1.5940	−0.0440	13	2.201	±0.0008
	ν	0.6097	−0.3501			±0.0112
	μ	0.9718	−0.5824			±0.0177
25	ρ	1.5845	−0.0451	11	2.262	±0.0015
	ν	0.5711	−0.3111			±0.0112
	μ	0.9049	−0.5164			±0.0176
30	ρ	1.5747	−0.0461	8	2.447	±0.0019
	ν	0.5364	−0.2805			±0.0238
	μ	0.8446	−0.4643			±0.0399
35	ρ	1.5649	−0.0471	7	2.571	±0.0028
	ν	0.5056	−0.2603			±0.0193
	μ	0.7911	−0.4300			±0.0299

[a] Key: x = molar fraction, N = number of experimental points, ρ = density (g/cm^3), ν = kinematic viscosity (cSt), μ = viscosity (cP), ΔB_1 = estimated error at 97.5% confidence interval.

from the liquid samples. The low values reported by Lewis are expected since in his experiments an atmosphere of chlorine was used above the liquid samples tested so that mass transfer would be taking place continuously during measurement.

Literature Cited

(1) Lewis, J. R., *J. Am. Chem. Soc.*, **47**, 626 (1925).
(2) Clegg G. T., Mann R., *Chem. Eng. J.*, **4**, 243 (1972).

Received for review April 7, 1977. Accepted September 7, 1977.

Solubility of Thallium(III) Sulfate in Aqueous Sulfuric Acid Solutions

Sanjay I. Amin
The Upjohn Company, Kalamazoo, Michigan 49001

The solubility of thallium(III) sulfate ($HTl(SO_4)_2 \cdot 4H_2O$) in aqueous sulfuric acid solutions (30–50% sulfuric acid by weight) was determined over the temperature range 0–25 °C. The data were correlated to show that the logarithm of solubility was a linear function of the absolute temperature and acid concentration. The correlation coefficient was found to be 0.9.

As a part of an ongoing study, it was necessary to know the solubility of thallium(III) sulfate in aqueous sulfuric acid solutions. There is no mention of this information in the literature, except for an article by Meyer (*1*). Meyer studied the solubility of $HTl(SO_4)_2 \cdot 4H_2O$ at 25 °C and in 50–70 wt % sulfuric acid solutions, which was beyond our range of use. Meyer found that $HTl(SO_4)_2 \cdot 4H_2O$ is hydrolyzed to Tl_2O_3 in sulfuric acid solutions having a concentration less than 10 wt %.

Table I. Solubility of Thallium(III) Sulfate [$HTl(SO_4)_2 \cdot 4H_2O$] in Aqueous Sulfuric Acid Solutions

Temp, °C	Wt % acid in water			
	30	40	50	70
0	12.37	0.76	0.288	
25	18.8	1.8	0.414 [0.1][a]	0.34[a]

[a] Reference 1.

Experimental Section

Thallium(III) sulfate was made by reacting freshly prepared Tl_2O_3 (reaction between $Tl(I)SO_4$ and H_2O_2 in basic media) with 50% sulfuric acid at 60–65 °C and cooling the solution to 5 °C on an ice bath. The precipitated flaky crystals of $HTl(SO_4)_2 \cdot 4H_2O$ were filtered and stored wet in an airtight bottle, to prevent de-

0021-9568/78/1723-0055$01.00/0