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Study of Viscosity of Mono-, Di-, and Trialkylamines

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Viscosities of several mono-, di-, and trialkylamines have been measured in the temperature range 298 to 333 K. It is observed that viscosities are highly dependent on shape, size, and association through H-bond or through dipole. Following the transition state theory, energy, Gibbs free energy, and entropy of activation of viscous flow have been calculated. The values of expansion energy for these liquids have also been calculated using free volume theory, and subsequently amines have been classified as volume-restrained or energy-restrained liquids. The group contribution method of Van Velzen, Cardozo, and Langenkamp for estimating viscosity has been examined with the present and literature data, and the new group contribution increments ΔN_i and ΔB_i for amines have been evaluated.

KEY WORDS: activation parameters; alkylamines; energy-restrained liquids; free volume theory; group contribution method; transition state theory; viscosity; volume-restrained liquids.

1. INTRODUCTION

Alkylamines are important organic bases because of their strong electron donating capability. Primary and secondary amines are self-associated, while tertiary alkylamines are weakly polar, nonassociated, strong proton acceptors. The molecular properties of these liquids are not known extensively and the information available on acoustic, volumetric, compressibility, refractivity, viscosity data is limited. As we are engaged in the studies on the molecular interactions in binary liquid mixtures containing amines, the properties of pure components are required [1–4]. In this

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paper, we report viscosities of mono-, di-, and trialkylamines from 298.15 to 333.15 K and show how this bulk property depends on the molecular complexity, size, and shape of molecules. These data also permit us to the check group contribution method of Van Velzen et al. [5] to estimate viscosities of any alkylamine as a function of temperature.

2. EXPERIMENTAL

2.1. Method

The viscosity measurements were made with a modified Ubbelohde suspended level viscometer. The efflux time of a definite volume of liquid through the capillary was measured with an accurate stopwatch with a resolution of 0.1 s. In the experiments, the viscometers were suspended vertically for a sufficient time in a constant-temperature bath maintained within 0.02 K. Doubly distilled water and purified benzene were used for the calibration of the viscometers. The densities for pure components required for the measurements of viscosity were determined by the calibrated glass pycnometer. The details of measurements and procedures of viscosity and density have been described previously [1, 4, 6]. Values of densities and viscosities for water and benzene used for the calibration of pycnometer and viscometer are given in Table I. The estimated accuracy of the measured density ρ and viscosity η is 0.01 and 0.3%, respectively.

2.2. Source and Purity of Samples

n-Propylamine (Merck-Schuchardt), *n*-butylamine (Riedel DeHaen AG; >98%), *n*-hexylamine (Fluka AG; >99%), *n*-octylamine (Fluka AG; >98%), *n*-decylamine (Sisco Chem), *n*-dodecylamine (Sisco Chem),

Table I. Densities and Viscosities for the Calibration Liquids

Liquid	T (K)	ρ ($kg \cdot m^{-3}$)	η ($mPa \cdot s$)
Water	298.15	997.05 [7]	0.8903 [7]
	303.15	995.65 [7]	0.7973 [7]
	313.15	992.22 [7]	0.6526 [7]
Benzene	298.15	873.60 [7]	0.6028 [7]
	303.15	868.29 [7]	0.5621 [7]
	313.15	857.69 [8]	0.4923 [7]

di-*n*-propylamine (Fluka AG; >99%), diisopropylamine (Wilson Laboratories), di-*n*-butylamine (Fluka AG; >98%), di-*n*-hexylamine (Fluka AG; >95%), di-*n*-octylamine (Fluka AG; >95%), triethylamine (Fluka AG; >99%), tri-*n*-propylamine (Fluka AG; >98%), tri-*n*-butylamine (Fluka AG; >99%) were allowed to stand over sodium hydroxide pellets for several days [7]. They were then distilled using a fractionating column. In the case of higher amines distillation was carried out under reduced pressure. Tri-*n*-hexylamine (Fluka AG; >95%), tri-*n*-octylamine (Fluka AG; >95%), triisooctylamine (Riedel; >98%), and tri-*n*-dodecylamine (Sisco; >99%) were used without further purifica-

Table II. Density and Refractive Index of the Pure Liquids Used

Liquid	Temp. (K)	Density ($kg \cdot m^{-3}$)		Refractive index	
		Expt.	Lit.	Expt.	Lit.
Propylamine	298.15	712.4	712.1 [7]	1.3893	1.3851 [7]
Butylamine	298.15	733.2	733.08 [10] 734.52 [11]	1.3997	1.3987 [7]
Hexylamine	303.15	757.6	756.5 [12]	1.4160	1.4160 [13]
Octylamine	298.15	779.3	778.9 [13]	1.4271	1.4279 [13]
Decylamine	303.15	786.1		1.4356	1.4374 [13]
Dodecylamine	303.15	794.7	791.2 [12]	1.4397	1.4401 [13]
Diethylamine	298.15	700.3	699.48 [10] 701.6 [7]	1.3840	1.3825 [7]
Dipropylamine	298.15	735.6	735.25 [12] 733.68 [10]	1.4053	1.4018 [7]
Diisopropylamine	298.15	714.8	710.0 [7]		
Dibutylamine	298.15	755.9	755.72 [10] 757.7 [7]	1.4159	1.4152 [7]
Dihexylamine	303.15	782.9		1.4336	1.4319 [13]
Dioctylamine	298.15	797.3	796.8 [13]	1.4420	1.4415 [13]
Triethylamine	298.15	723.6	723.18 [10] 723.8 [12]	1.4015	1.3980 [7]
Tripropylamine	298.15	753.7	753.1 [14] 752.34 [10]	1.4159	1.4141 [12]
Tributylamine	298.15	774.2	774.3 [7] 774.6 [14]	1.4267	1.4265 [15] 1.4268 [14]
Trihexylamine	298.15	979.7	976.4 [14] 793.7 [15]	1.4419	1.4404 [14] 1.4398 [15]
Trioctylamine	298.15	808.3	808.6 [16] 807.4 [9]	1.4474	1.4478 [14] 1.4476 [12]
Triisooctylamine	298.15	814.1	814.7 [14]	1.4493	1.4495 [14]
Tridodecylamine	298.15	821.5	820.7 [15]	1.4567	1.4567 [13]

tion. The purity of liquid components was checked by boiling points, gas–liquid chromatography, density, and refractive index and it was observed that the purity was better than the labeled purity. Table II compares measured densities and refractive indices with literature values [7, 9–16].

3. RESULTS AND DISCUSSION

3.1. General

The viscosities of each alkylamine liquid have been measured at several temperatures ranging from 298 to 333 K or up to approximately 20 K below the normal boiling point of the liquid.

The values of viscosity η determined previously for n-prpNH$_2$ at 298.15 K [7] and 303.15 K [17], n-butNH$_2$ at 313.15 K [18], eth$_2$NH at 303.15 K [17, 19], n-prp$_2$NH at 298.15 K [17], n-but$_2$NH at 313.15 K [18], and eth$_3$N, n-prp$_3$N, n-but$_3$N, n-hex$_3$N, n-oct$_3$N, and n-dod$_3$N from 298.15 to 323.15 K [7, 9, 17–20] are close to our values. There is a confusion in literature about the viscosity of n-butNH$_2$ and n-but$_2$NH. Values of η for n-butNH$_2$ of 0.68 mPa · s [21] and 0.578 mPa · s [17, 22] at 298.15 K and 0.501 mPa · s [23] at 303.15 K are higher than interpolated values by Arrhenius Eq. (1) from the work of Kohler et al. [18], who have reported η as 0.523 and 0.401 mPa · s at 293.15 and 313.15 K, respectively. Similarly, η values for n-but$_2$NH of 0.95 mPa · s [24] at 293.15 K, 0.946 [17] at 298.15 K, and 0.83 mPa · s [22, 24] at 303.15 K are higher than those of Kohler et al. [18] obtained by interpolation from η data, 0.85 and 0.64 mPa · s at 293.15 and 303.15 K, respectively. Our results for n-butNH$_2$ and n-but$_2$NH agree closely with those reported by Kohler et al. [18] within 0.5 to 2.6%. The discrepancy between our data and that of Kohler et al. [18] is not much considering the variation of physical properties of amines when kept for some time [25]. We have always measured properties of freshly distilled amines.

It is interesting to note that the viscosities depend so much on molecular shapes, size, and association through H-bonds or through dipoles. The comparison of the viscosities (mPa · s) for isomer molecules with various molecular masses (M); illustrates this point: $M = 101.19$—n-hexNH$_2$ ($\eta = 0.715$), n-prp$_2$NH ($\eta = 0.479$), iso-prp$_2$NH ($\eta = 0.357$), and eth$_3$N ($\eta = 0.359$); $M = 129.25$—n-octNH$_2$ ($\eta = 1.165$) and n-but$_2$NH ($\eta = 0.759$); $M = 185.36$—n-dodNH$_2$ ($\eta = 2.664$), n-hex$_2$NH ($\eta = 1.893$), and n-but$_3$N ($\eta = 1.167$); and $M = 353.68$—n-oct$_3$N ($\eta = 6.950$) and iso-oct$_3$N ($\eta = 9.539$). Low viscosities are observed for tri-n-alkylamines, high viscosities for n-alkylamines, and intermediate viscosities for di-n-alkylamines; this difference in behavior is attributed to the role of strong

H-bonds in primary amines and weak H-bonds in secondary amines. Viscosities values for normal and branched-chain dipropyl and trioctyl amines clearly show the influence of the shape of the molecule on flow properties.

3.2. Transition State Theory

Following the transition state theory [26], the dependence of absolute viscosity η at constant pressure p on temperature T is given by

$$\ln \eta = \ln \eta^* + E_{\mathrm{P}}^{\ddagger}/RT \qquad (1)$$

where $\ln \eta^*$ is a constant, $E_{\mathrm{P}}^{\ddagger}$ is the energy of activation for viscous flow at constant pressure, and η is liquid viscosity in Pa · s. Plots of $\ln \eta$ for mono-, di-, and trialkylamines are linear functions of $1/T$ in the temperature range studied. The values of $\ln \eta^*$ and $E_{\mathrm{P}}^{\ddagger}$, together with the standard deviations obtained from the method of least squares, are given in Table III. In Fig. 1, the dependence of $E_{\mathrm{P}}^{\ddagger}$ as a function of carbon atoms (C_n) in the primary, secondary, and tertiary alkylamine is shown.

Table III. Constants of Eq. (1) and Standard Deviations σ Along with Activation Parameters of Viscous Flow for Alkylamines

Amine	$\ln \eta^*$	$E_{\mathrm{P}}^{\ddagger}$ (kJ · mol^{-1})	$\sigma \times 10^3$	$\Delta G^{\ddagger a}$ (kJ · mol^{-1})	$\Delta S^{\ddagger a}$ (kJ · mol^{-1} · K^{-1})
Propylamine	−11.636	9.250	1.2	10.757	−5.05
Butylamine	−11.740	10.097	4.1	11.778	−5.64
Hexylamine	−11.753	11.367	0.0	13.755	−8.01
Octylamine	−11.949	13.089	0.0	15.540	−8.22
Decylamine	−12.449	15.535	5.1	18.478	−9.71
Dodecylamine	−12.758	17.211	12.0	18.530	−4.65
Diethylamine	−11.296	7.702	1.0	10.800	−9.89
Dipropylamine	−11.530	9.794	4.0	12.790	−10.05
Diisopropylamine	−11.227	8.285	2.4	12.110	−12.84
Dibutylamine	−11.730	11.460	8.9	14.490	−10.16
Dihexylamine	−12.415	15.489	3.2	17.700	−7.31
Dioctylamine	−12.144	16.511	7.3	19.920	−11.47
Triethylamine	−11.339	8.586	2.5	12.104	−11.80
Tripropylamine	−11.131	9.291	4.6	14.060	−16.87
Tributylamine	−12.160	13.627	18.6	16.410	−9.34
Trihexylamine	−13.070	18.623	10.7	20.100	−4.87
Trioctylamine	−13.538	21.595	4.5	22.560	−3.1
Triisooctylamine	−13.845	23.071	3.3	23.270	−0.7
Tridodecylamine	−14.575	26.860	10.5	26.120	2.43

[a] At 303.15 K.

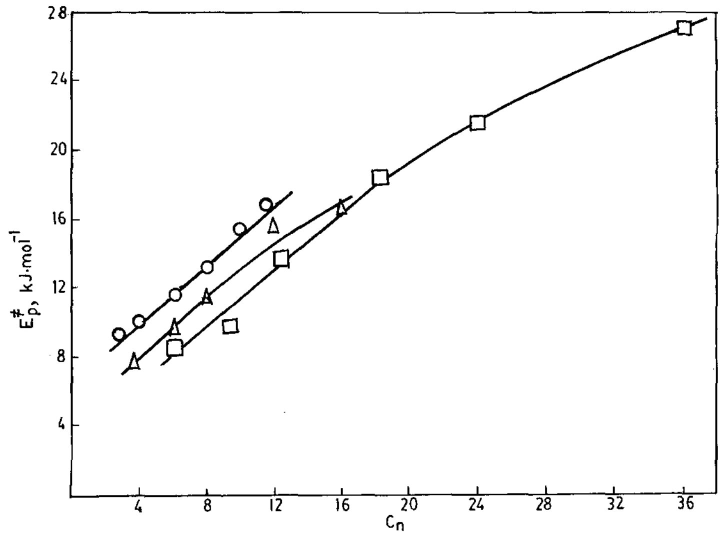

Fig. 1. Dependence of $E_P^{\ddagger}$ on the number of carbon atoms in alkylamine. Primary amine (○), secondary amine (△), and tertiary amine (□).

On the basis of the transition state theory, the values of free energy and entropy of activation ($\Delta G^{\ddagger}$ and $\Delta S^{\ddagger}$) of viscous flow [27] can be estimated from Eqs. (2) and (3) in combination with Eq. (1).

$$\Delta G^{\ddagger} = RT \ln(\eta V/Nh) \tag{2}$$

and

$$\Delta S^{\ddagger} = (E_P^{\ddagger} - \Delta G^{\ddagger})/T \tag{3}$$

where V is molar volume and N and h have their usual significance. The values of $\Delta G^{\ddagger}$ and $\Delta S^{\ddagger}$ are also included in Table III.

Table III shows that the values of energy of activation $E_P^{\ddagger}$ of viscous flow follow the sequence primary > secondary > tertiary amines for isomer compounds of the same molecular weight.

Figure 1 shows that the value of $E_P^{\ddagger}$ increases with an increase in the alkyl chain of all the amines. With the increase in alkyl chain length of the amine, the orientational order goes on increasing quite substantially, reflecting an increase in $E_P^{\ddagger}$ values. A similar trend was also observed by Klofutar et al. [9] in the study of tri-*n*-alkylamines from ethyl to dodecyl members.

There is an increase in $\Delta G^{\ddagger}$ values with an increase in the number of carbons in the alkyl chain of amines and values of $\Delta G^\ddagger$ also increase with temperature. A parallel observation was made for the *n*-alkane liquids [27].

3.3. Free Volume Theory

On the basis of the free volume theory [28, 29], the viscosity related to density ρ is presented as

$$\eta^{-1} = b(\rho^{-1} - \rho_0^{-1}) \tag{4}$$

where b and ρ_0 are empirical constants. ρ_0 is also known as the intrinsic density. Values of the parameters in the empirical Eq. (4) are given in Table IV for the present alkylamines.

It was shown that the energy of activation $E_\mathrm{P}^\ddagger$ for viscous flow at low pressure can be given by [30]

$$E_\mathrm{P}^\ddagger = E_\mathrm{V}^\ddagger + \pi \Delta V^\ddagger \tag{5}$$

Table IV. Coefficients of Eqs. (4) and (6) and Standard Deviations σ_ρ, Where E_ρ is the Expansion Energy

	For Eq. (4)			For Eq. (6)			
Compound	$b \times 10^3$ ($\mathrm{mm^2 \cdot s^{-1}}$)	ρ_0 ($\mathrm{kg \cdot m^{-3}}$)	$10\sigma_\rho$ ($\mathrm{kg \cdot m^{-3}}$)	ρ_* ($\mathrm{kg \cdot m^{-3}}$)	a	E_ρ ($\mathrm{kJ \cdot mol^{-1}}$)	$10\sigma_\rho$ ($\mathrm{kg \cdot m^{-3}}$)
Propylamine	18.000	798	3.3	828	8.216	7.28	0.2
Butylamine	17.749	804	10.0	844	8.138	7.32	0.5
Hexylamine	15.622	812	2.2	857	7.809	6.98	0.1
Octylamine	12.820	818	3.0	868	7.668	6.91	0.1
Decylamine	10.618	821	2.0	879	7.668	6.89	1.5
Dodecylamine	9.070	822	1.2	879	7.788	6.84	1.4
Diethylamine	15.768	826	6.9	825	8.588	7.96	0.4
Dipropylamine	16.730	804	8.4	834	8.129	7.55	0.6
Diisopropylamine	16.374	808	7.6	828	8.178	7.28	0.2
Dibutylamine	15.134	804	9.9	849	7.838	7.08	0.6
Dihexylamine	10.354	815	1.1	869	7.597	6.94	0.8
Dioctylamine	6.560	823	15.0	883	7.572	6.83	0.2
Triethylamine	17.050	814	7.8	832	8.255	7.58	0.1
Tripropylamine	14.836	821	7.7	851	7.801	6.89	0.3
Tributylamine	14.207	808	9.9	863	7.582	6.68	0.7
Trihexylamine	7.966	818	1.8	878	7.427	6.65	0.6
Trioctylamine	4.964	523	3.0	892	7.404	6.58	0.2
Triisooctylamine	4.835	825	15.6	885	7.408	6.98	0.7
Tridodecylamine	2.684	830	5.6	890	7.391	7.05	1.4

where $E_V^{\ddagger}$ is the energy of activation for viscous flow at constant volume, $\Delta V^{\ddagger}$ is the activation volume, and π is the internal pressure of the liquid, defined as $\pi = \alpha T/K_T$, where K_T is the isothermal compressibility, and α is the thermal expansion coefficient.

A volume-restrained liquid is characterized by $\pi \Delta V^{\ddagger} \gg E_V^{\ddagger}$, while an energy-restrained liquid has $\pi \Delta V^{\ddagger} \ll E_P^{\ddagger}$ [31].

At low pressure, $\pi \Delta V^{\ddagger}$ is at least to the expansion energy, E_ρ, representing the work done against the internal pressure π of the liquid. The expansion energy E_ρ can be estimated from the Van't Hoff-type dependence of free volume on temperature,

$$\ln(\rho^{-1} - \rho_*^{-1}) = a - E_\rho/RT \tag{6}$$

The values of E_ρ for the present alkylamines were derived from fitting Eq. (6) to the density data by the least-squares method in the manner recommended by Stairs [31]. Table IV contains the results of applying this procedure to the alkylamines under investigation. Comparison of values of $E_P^{\ddagger}$ and E_ρ given in Tables III and IV shows that propylamine, butylamine, diethylamine, dipropylamine, diisopropylamine, triethylamine, and tripropylamine can be termed volume-restrained, while the others are energy-restrained liquids.

4. ESTIMATION OF VISCOSITIES BY THE GROUP CONTRIBUTION METHOD

Van Velzen et al. [5], from detailed study of the effect of structure on liquid viscosities, developed a group contribution method to estimate the viscosities of a variety of liquids. They [5] proposed a modification of Eq. (1),

$$\log \eta = B(T^{-1} - T_0^{-1}) \tag{7}$$

where B and T_0 are parameters related to the structure of the liquid and η is the viscosity of liquids in mPa · s. The values of these parameters were obtained first by finding the equivalent chain length N^*, where

$$N^* = N + \sum \Delta N_i \tag{8}$$

N is the actual number of carbon atoms in the molecule and ΔN_i represents structural group contribution increments (SGCI).

The value of N^* was then used to determine constants B and T_0. T_0 for $N^* \leqslant 20$ is given by

$$T_0 = 28.86 + 37.439N^* - 1.3547(N^*)^2 + 0.02076(N^*)^3 \tag{9}$$

while for $N^* > 20$

$$T_0 = 8.164N^* + 238.59 \tag{10}$$

Similarly, B is obtained by

$$B = B_A + \sum \Delta B_i \tag{11}$$

where B_A for $N^* \leqslant 20$ is

$$B_A = 24.79 + 66.885N^* - 1.3173(N^*)^2 - 0.00377(N^*)^3 \tag{12}$$

and B_A for $N^* > 20$ is

$$B_A = 530.59 + 13.740N^* \tag{13}$$

and $\sum \Delta B_i$ can be determined by summing the contributions for appropriate groups in the liquid.

Van Velzen et al. [5] determined ΔN_i and ΔB_i for a large number of liquids having different functional groups including those for primary, secondary, and tertiary amines. It appears [21, 32] that the alkylamines they used to determine ΔN_i and ΔB_i were mainly methylamine, ethylamine, *n*-propylamine, *n*-butylamine, isobutylamine, diethylamine, ethylpropylamine, di-*n*-butylamine, triethylamine, and tri-*n*-butylamine. For most of the liquids, the viscosity data were available only at either one or two temperatures, except for the diethylamine and triethylamine. For the latter two liquids, the viscosity data were available in the temperature range 240 to 313 K. From comparison of viscosity data for the compounds that they used with the other available precise viscosity data in the literature, it seems that the values of viscosities for *n*-butylamine, diethylamine, ethylpropylamine, and di-*n*-butylamine are considerably higher. These aforesaid factors lead to errors in estimation of ΔN_i and ΔB_i for the alkylamines [5]. Further, the use of these SGCI values results in considerable errors in estimation of viscosities for higher alkylamine liquids. In light of this, we thought it appropriate to reevaluate SGCI for alkylamines, as precise values of viscosities for a large number of alkylamines having up to 36 carbon atoms and at several temperatures are now available [7, 9, 17–24].

In all, we have used eight primary amines, seven secondary amines, and eight tertiary amines with nearly 220 data points in the temperature range 288 to 333 K and a careful statistical evaluation was made. The values of ΔN_i and ΔB_i were determined by the same statistical method as proposed by Van Velzen et al. [5]. The new values of ΔN_i and ΔB_i are given in Table V.

Table V. Structural Group Contribution Increments ΔN_i and ΔB_i for the Van Velzen et al. [5] Method

Liquid	ΔN_i	ΔB_i
Primary amine	$4.5 - 0.12N$	$-119 + 21.0N^*$
Secondary amine	$3.2 - 0.11N$	$-26 + 5.8N^*$
Tertiary amine	$3.6 - 0.37N$	$-166 + 24.2N^*$

Table VI. Comparison of Experimental and Calculated Viscosities of Primary Amines

			Percentage standard error calculated using	
Compound	Data points	Temp. range (K)	Van Velzen et al. [5]	Present work
Propylamine	8	293.15–333.15	2.89	2.98
Butylamine	10	293.15–333.15	23.31	2.94
Pentylamine	2	298.15–308.15	22.83	13.80
Hexylamine	8	298.15–333.15	58.31	1.70
Octylamine	8	298.15–333.15	83.88	1.48
Decylamine	8	298.15–333.15	99.01	1.17
Dodecylamine	8	298.15–333.15	120.45	5.85
Total	52		71.53	6.08

Table VII. Comparison of Experimental and Calculated Viscosities of Secondary Amines

			Percentage standard error calculated using	
Compound	Data points	Temp. range (K)	Van Velzen et al. [5]	Present work
Dimethylamine	2	288.15–298.15	90.02	11.51
Diethylamine	9	293.15–308.15	19.15	24.77
Dipropylamine	10	293.15–333.15	61.26	13.21
Dibutylamine	13	293.15–333.15	116.28	13.51
Dihexylamine	8	298.15–333.15	177.54	4.24
Dioctylamine	8	298.15–333.15	212.98	1.30
Total	50		144.27	13.68

Table VIII. Comparison of Experimental and Calculated Viscosities of Tertiary Amines

Compound	Data points	Temp. range (K)	Percentage standard error calculated using: Van Velzen et al. [5]	Percentage standard error calculated using: Present work
Trimethylamine	2	288.15–298.15	93.74	59.27
Triethylamine	23	288.15–333.15	71.75	15.26
Tripropylamine	8	298.15–333.15	126.00	7.79
Tributylamine	23	293.15–333.15	130.00	8.97
Trihexylamine	17	298.15–333.15	132.70	17.28
Trioctylamine	17	298.15–333.15	200.22	7.54
Tridecylamine	9	298.15–333.15	319.00	4.76
Tridodecylamine	17	298.15–333.15	633.00	54.13
Total	116		296.57	25.32

In order to check the improvement due to the new values of SGCI, the absolute viscosities were calculated using the present SGCI ΔN_i and ΔB_i given in Table V and those given by Van Velzen et al. [5]. The preditive ability of the new set of SGCI and those given by Van Velzen has been tested by means of comparing the percentage deviations $\sigma\%$ between the experimental values and those calculated by each of the methods. The percentage deviation for each amine has been estimated as

$$\sigma(\%) = 100\left[\sum\left(\frac{\eta_{\text{exp}} - \eta_{\text{cal}}}{\eta_{\text{exp}}}\right)^2 \Big/ n\right]^{1/2}$$

A careful perusal of Tables VI to VIII indicates that the present SGCI estimates the absolute viscosities with percentage deviations $\sigma\%$ from 1.2 to 13.8 for primary amines, from 1.3 to 24.8 for secondary amines, and from 4.8 to 59.3 for tertiary amines, while considerably higher values of $\sigma\%$ for higher alkylamines were observed when the ΔN_i and ΔB_i given by Van Velzen [5] were used.

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