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Density, Refractive Index, and Kinematic Viscosity of Diesters and Triesters

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Densities at temperatures from 288.15 K and 358.15 K, kinematic viscosity from 293.15 K and 358.15 K, and refractive index from 288.15 K and 323.15 K were measured for six esters: tributyl phosphate, dibutyl suberate, diethyl azelate, diethyl sebacate, diethyl phthalate, and dioctyl phthalate. The results were fitted to various models, and the parameters are reported.

Introduction

Esters of organic diacids, phosphoric acid, dimer acids, and trimellitic acid with linear, branched or mixed alkyl chains, or hindered alkyl chains derived from polyols are very important chemicals, which find extended industrial applications. They are widely employed in the paint and varnish field as plasticizers, mainly in the formulation of chlorinated rubber and cyclorubber binders and as low-volatility polar solvents for liquid-grade epoxies, in the plastics industry as plasticizers, and in the technology of plastisol and organosol and of slash moulding and plastic foams. Besides, they make up an important class of synthetic bases for lubricants, where they are appreciated for their excellent properties concerning viscosity, flow, lubricity, thermal stability, solvency, hydrolitic stability, and, last but not least, biodegradability. Blending with other materials, in particular with poly(alphaolefins), is important for the formulation of special lubricants. Moreover, their full compatibility with mineral oil allows them to be blended in all proportion with mineral lubricant bases. Open literature displays a shortage of density and viscosity data for these compounds (Bried et al., 1947), and no data on their mixtures with the natural lubricant bases are available.

This investigation is aimed at characterizing viscosity and flow behavior of five esters of organic diacids and a triester of phosphoric acid: viscosity, density, and refractive index are measured over a wide temperature range, needed for industrial use. In order to investigate behavior differences connected with differences in the chemical composition, ester selection was directed, when possible, toward chemically pure components. Only low molecular weight esters are commercially available at high-purity grade; low molecular weight involves low viscosity. The esters considered are fully aliphatic, aliphatic−aromatic, and phosphoric esters. This study is preliminary to a characterization of higher molecular weight esters, available at lesser-purity grade, which can be taken into account in the lubricant field for blending with mineral oils, to boost the properties of the latter. It is in light of an investigation on mixtures of a synthetic lubricant basis with high molecular weight esters that some non-highly-pure compounds were considered in this work.

Experimental Section

Materials. All esters considered in this work were supplied by Aldrich and employed as received, without any further purification. The stated purity of the chemicals is the following: tributyl phosphate (99+ mol %), dibutyl suberate (99 mol %), diethyl azelate (technical grade of 90 mol %), diethyl sebacate (98 mol %), diethyl phthalate (99 mol %), and dioctyl phthalate (99 mol %). The purities of nitrogen and water employed for instruments calibration were 99.9999 mol % and 99.9 mol %, respectively. For the calibration of the viscometers at 293.15 K, the Poulten Selfe & Lee standard oil K5 was employed. For the extension of the calibration to higher temperatures, Aldrich dodecane (99+ mol %) was employed.

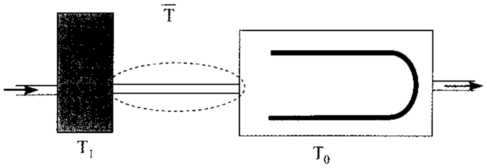

Figure 1. Details of the temperature measurement in the experimental apparatus: T_1 is the chamber temperature, T_0 is the cell (densimeter) temperature, and T is the ambient temperature.

Apparatus and Process. *Density* was measured by means of a vibrating tube digital densimeter, model DMA 602H-DMA 60 (Anton Paar), equipped with calibrated thermometers, suitable to work between 288.15 K and 318.5 K with a precision of ± 0.01 K, connected with a Hetofrig (Heto Birkerød), constant-temperature bath circulator, with a precision of ±0.01 K. Nitrogen and double-distilled water were employed to calibrate the densimeter. Working procedures between 288.15 K and 318.15 K are described in more detail in Fermeglia and Lapasin (1988), Fermeglia et al. (1990), and De Lorenzi et al. (1996). The density estimated precision in this temperature range is higher than 3×10^{-5} g·cm^{-3}. The rather low upper limit of the temperature range of the measuring device, as mentioned above, is due to the availability of a restricted set of Paar calibrated thermometers to be inserted in the appropriate well close to the vibrating tube. In order to extend the measurements to higher temperatures (in this case up to 358.15 K) while still maintaining high accuracy, the following procedure was adopted. A platinum resistance thermometer (PT 100) was inserted in a chamber of the thermostating circuit, about 50 cm in front of the entrance to the Paar measuring cell, and connected with a high-performance 6 Ω digit multimeter (Hewlett Packard HP 34401), suitable for four wire resistance measurements, as is shown in Figure 1. With this assembly, dual temperature measurements (in the cell and in the chamber) have been performed within the original temperature

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Table 1. Temperature Measurements inside and outside the Vibrating Tube

T_1/K	T_0/K	$\Delta T = (T_1 - T_0)$/K	U_0AW^{-1}/J·mol^{-1}·K^{-1}
287.84	287.89	−0.05	0.297
287.84	287.90	−0.06	0.344
293.27	293.30	−0.03	0.300
293.26	293.29	−0.02	0.273
293.28	293.31	−0.03	0.349
303.20	303.20	0.01	0.286
308.25	308.22	0.03	0.330
308.26	308.23	0.02	0.234
313.09	313.05	0.04	0.257
313.11	313.06	0.05	0.307
314.11	314.07	0.03	0.180
314.12	314.08	0.04	0.226
314.11	314.06	0.05	0.267
318.11	318.06	0.05	0.231
318.11	318.05	0.07	0.286
318.15	318.08	0.06	0.269
	average		0.277
	average deviation		0.035
	standard deviation		0.045

range, thus measuring the temperature difference between the two points of the circuit. The results are reported in Table 1 (first three columns).

The extrapolation procedure used to extend the temperature range above 318.15 K is based on the assumption that the heat transfer coefficient between the thermostating fluid and the ambient does not vary with temperature, at least in the range of temperatures considered. An energy balance around the system indicated in Figure 1 can be summarized as follows: heat transferred to the environment (Q)

$$Q = U_0 A \Delta T_{\mathrm{ML}} \tag{1}$$

where U_0 is the overall heat transfer coefficient, A is the heat transfer area, and ΔT_{ML} is an average difference in temperature between the inside fluid (water) and the outside fluid (air) defined as

$$\Delta T_{\mathrm{ML}} = \frac{(T_1 - \bar{T}) - (T_0 - \bar{T})}{\ln\dfrac{(T_1 - \bar{T})}{(T_0 - \bar{T})}} \tag{2}$$

where T_1 and T_0 are the chamber and cell temperature, respectively, and $\bar{T}$ is the room temperature. The entire assembly is contained in a thermostated room that allows a constancy of the temperature of 0.1 K.

The heat transferred to the environment can be expressed by means of the following equation

$$Q = WC_p(T_1 - T_0) \tag{3}$$

where C_p is the constant-pressure heat capacity and W is the flow rate of water.

By equating eqs 1 and 3 the following equation is obtained

$$U_0 A \Delta T_{\mathrm{ML}} = WC_p(T_1 - T_0) \tag{4}$$

from which the nondimensional quantity can be calculated

$$\frac{U_0 A}{WC_p} = \frac{(T_1 - T_0)}{\Delta T_{\mathrm{ML}}} \tag{5}$$

The variation of the heat capacity of the pure water in the liquid state can be evaluated by means of the following expression (Himmelblau, 1989).

Table 2. Estimated Cell Temperatures

T_1/K	T_0/K (estmd)	$\Delta T = (T_1 - T_0)$/K	T_1/K	T_0/K (estmd)	$\Delta T = (T_1 - T_0)$/K
322.89	322.81	0.08	347.51	347.34	0.17
328.03	327.92	0.11	352.42	352.23	0.19
332.85	332.73	0.12	352.34	352.15	0.19
337.92	337.78	0.14	357.44	357.23	0.21
342.45	342.29	0.16			

Table 3. Experimental Density Data

T/K	ρ/g·cm^{-3}	T/K	ρ/g·cm^{-3}	T/K	ρ/g·cm^{-3}
diethyl azelate		tributyl phosphate		dibutyl suberate	
288.04	0.975 46	288.04	0.981 14	288.04	0.952 63
293.22	0.970 97	293.22	0.976 72	293.22	0.948 36
298.15	0.966 70	298.15	0.972 49	298.15	0.944 36
303.17	0.962 34	303.17	0.968 19	303.17	0.940 29
308.19	0.957 99	308.19	0.963 88	308.19	0.936 22
313.05	0.953 77	313.05	0.959 70	313.05	0.932 33
318.07	0.949 40	318.07	0.955 37	318.07	0.928 20
322.8	0.945 3	322.8	0.951 3	322.8	0.924 4
327.9	0.940 8	327.9	0.946 9	327.9	0.920 2
332.7	0.936 6	332.7	0.942 7	332.7	0.916 3
337.8	0.932 4	337.8	0.938 5	337.8	0.912 4
342.3	0.928 3	342.3	0.934 5	342.3	0.908 6
347.3	0.924 7	347.3	0.930 9	347.3	0.905 2
352.2	0.920 5	352.2	0.926 8	357.2	0.897 4
357.2	0.916 3	357.2	0.922 6		
diethyl sebacate		diethyl phthalate		dioctyl phthalate	
288.04	0.967 67	288.04	1.122 76	288.04	0.987 68
293.22	0.963 29	293.22	1.118 18	293.22	0.983 75
298.15	0.959 13	298.15	1.113 81	298.15	0.980 00
303.17	0.954 89	303.17	1.109 35	303.17	0.976 21
308.19	0.950 63	308.19	1.104 89	308.19	0.972 40
313.05	0.946 54	313.05	1.100 59	313.05	0.968 77
318.07	0.942 28	318.07	1.096 15	318.07	0.964 96
322.8	0.938 3	322.8	1.092 0	322.8	0.961 4
327.9	0.933 9	327.9	1.087 4	327.9	0.957 6
332.7	0.929 9	332.7	1.083 2	332.7	0.954 0
337.8	0.925 8	337.8	1.078 9	337.8	0.950 4
342.3	0.921 8	342.3	1.074 8	342.3	0.946 9
347.3	0.918 3	347.3	1.071 2	347.3	0.943 9
352.1	0.914 3	352.1	1.067 1	352.1	0.940 4
357.2	0.910 1	357.2	1.062 8	357.2	0.936 8

$$C_p/\mathrm{J{\cdot}mol^{-1}{\cdot}K^{-1}} = 18.2964 + 0.472\,12T/\mathrm{K} - 0.001\,338(T/\mathrm{K})^2 + 0.000\,001\,314\,2(T/\mathrm{K})^3 \tag{6}$$

The last column of Table 1 and the values of standard deviation reported show that the U_0A/W values can be considered constant in the temperature range between 288.15 and 318.15 K; the mean value of 0.277 (J·mol^{-1}·K^{-1}), consequently, is assumed and maintained constant in the extrapolation to higher temperatures. The calculated values of temperature are reported in Table 2. The precision of the temperature measurements obtained by this extrapolation procedure is estimated to be higher than 0.04 K. Consequently, density precision in the same temperature range is estimated to be 4×10^{-5} g·cm^{-3}.

Viscosity was measured by means of Ubbelohde suspended-level capillary viscometers, coupled with a Schott electronic timer AVS 300, with a precision of ±0.01 s. The thermostat was a Haake F3 instrument, with a precision of ±0.02 K. Working procedures are described in Fermeglia and Lapasin (1988), Fermeglia et al. (1990), and De Lorenzi et al. (1996). Calibration of the capillary viscometer is of paramount importance for obtaining high-accuracy data. Since the temperature range of measurements is rather wide, the calibration performed with the standard oil at 293.15 K was extended to higher temperatures, by using a 99+ purity dodecane. The calibration constant variation with temperature was evaluated by

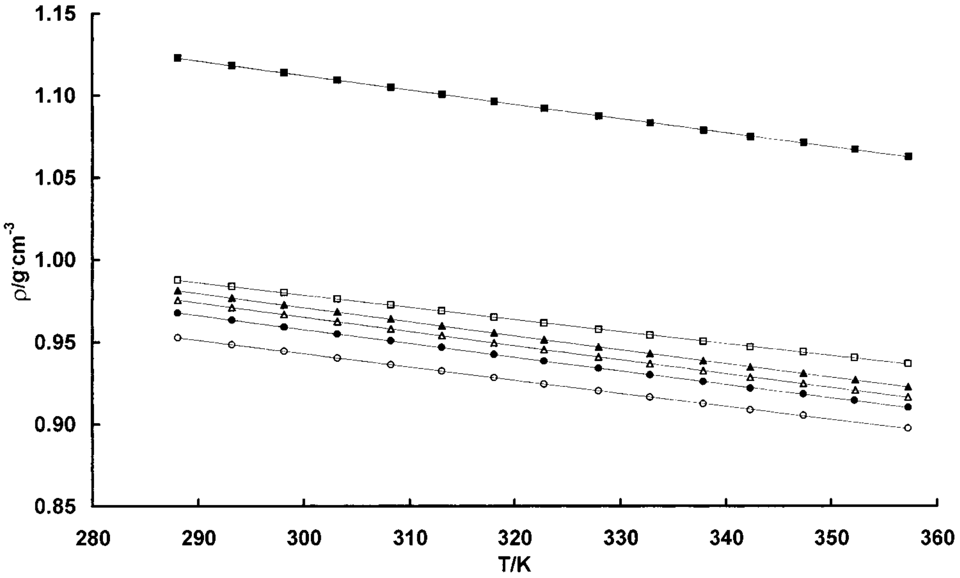

Figure 2. Density vs temperature for (■) diethyl phthalate, (□) dioctyl phthalate, (▲) tributyl phosphate, (△) diethyl azelate, (●) diethyl sebacate, (○) dibutyl suberate.

Table 4. Values of the Coefficients and Relevant Standard Deviation for Eq 7

	diethyl azelate	tributyl phosphate	dibutyl suberate	diethyl sebacate	diethyl phthalate	dioctyl phthalate
a/g·cm^{-3}	1.2506	1.2528	1.2075	1.2341	1.3963	1.2179
b	1.4286	1.4243	1.4095	1.4276	1.3659	1.3791
c/K	7.1210	7.1443	7.1308	7.1794	7.0907	7.3485
d	−0.0181	−0.0180	−0.0180	−0.0179	−0.0181	−0.0175
σ/g·cm^{-3}	1.87×10^{-4}	1.80×10^{-4}	1.92×10^{-4}	1.86×10^{-4}	2.19×10^{-4}	2.53×10^{-4}

Table 5. Experimental Refractive Index and Eykman Constant

T/K	n	C/cm^3·g^{-1}	T/K	n	C/cm^3·g^{-1}
tributyl phosphate			dioctyl phthalate		
288.4	1.4260	0.577	288.4	1.4882	0.652
293.3	1.4240	0.577	293.3	1.4862	0.652
298.2	1.4224	0.577	298.2	1.4846	0.652
303.3	1.4202	0.577	303.3	1.4826	0.652
308.2	1.4183	0.577	308.1	1.4808	0.652
313.4	1.4163	0.577	313.4	1.4787	0.652
317.9	1.4145	0.577	318.0	1.4768	0.652
322.6	1.4127	0.577	322.6	1.4749	0.652
diethyl azelate			diethyl sebacate		
288.4	1.4364	0.594	288.4	1.4384	0.601
293.3	1.4342	0.593	293.3	1.4362	0.601
298.2	1.4326	0.594	298.2	1.4348	0.602
303.3	1.4306	0.594	303.3	1.4326	0.601
308.1	1.4286	0.594	308.2	1.4307	0.602
313.4	1.4266	0.594	313.4	1.4286	0.602
318.0	1.4247	0.594	318.0	1.4267	0.602
322.8	1.4226	0.594	322.8	1.4248	0.602
dibutyl suberate			diethyl phthalate		
288.4	1.4403	0.613	288.6	1.5038	0.590
293.3	1.4382	0.613	293.3	1.5016	0.590
298.2	1.4368	0.614	298.2	1.5001	0.591
303.3	1.4347	0.614	303.3	1.4977	0.591
308.1	1.4328	0.614	308.1	1.4958	0.591
313.4	1.4308	0.614	313.4	1.4936	0.591
317.9	1.4289	0.614	318.0	1.4916	0.591
322.8	1.4270	0.614	322.7	1.4896	0.591

measuring the viscosity of dodecane up to 358.15 K and comparing the values obtained with literature values (Knapstad et al., 1989; Aminabhavi and Gopalkrushna, 1994; TRC Version 1, 1993; Asfour et al., 1990, 1993 Vavanellos et al., 1991; Cooper and Asfour, 1991). The estimated precision in kinematic viscosity measurements in the entire temperature range is approximately 1×10^{-4} mm^2·s^{-1}.

Refractive index was determined for the sodium-D line with an Abbe system, ATAGO type 3 refractometer, connected with the same Hetofrig constant-temperature bath circulator mentioned above. The temperature accuracy is of ±0.02 K. Instrument calibration was carried out with double-distilled water. Measurement precision is estimated to be higher than 10^{-4}.

The temperature probes used in all the measurements were calibrated against a platinum resistance thermometer (Rosemount Model 162 CE) and checked at the water triple point.

The estimated precisions reported in the paper were obtained on the basis of repeated experiments on all pure components at selected temperatures for all the properties measured.

Results and Correlation

Density. The density results at the various temperatures are reported in Table 3.

The Daubert and Danner (DIPPR) equation (Daubert and Danner, 1989) was fit to the experimental results

$$\rho/\text{g}\cdot\text{cm}^{-3} = \frac{a}{b^{1+(1-T/\text{K}c^{-1})^{d}}} \tag{7}$$

where a, b, c, d are adjustable parameters. The parameters obtained in the data regression are reported in Table 4 along with σ, the standard deviation, defined as follows

$$\sigma = \sqrt{\frac{\sum(x_{\text{exp}} - x_{\text{calc}})^2}{N - n}} \tag{8}$$

where N is the number of experimental data, n is the number of equation parameters, and x is the considered property (density in this case).

The values reported in Table 4 show that the standard deviation of the fit is comparable with the experimental

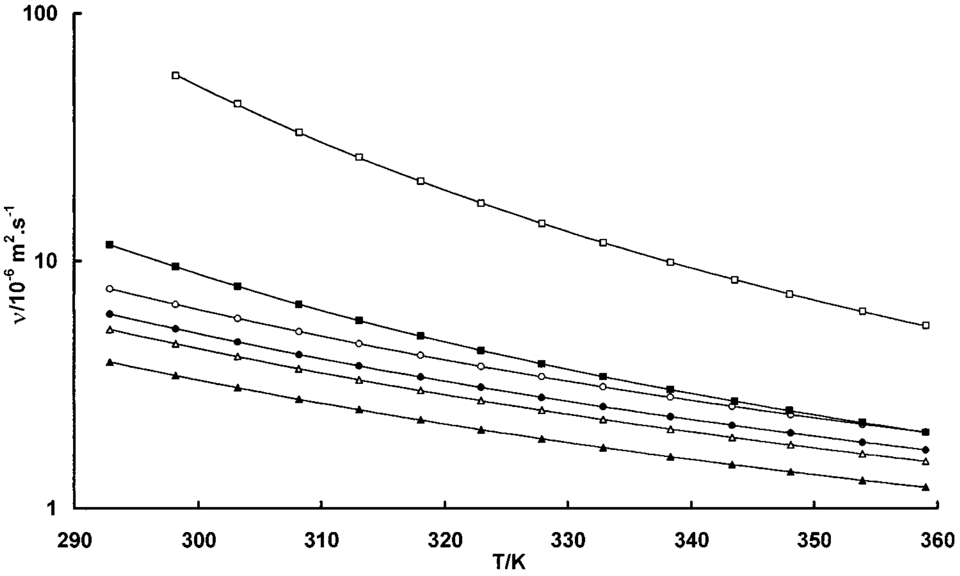

Figure 3. Kinematic viscosity vs temperature for (□) dioctyl phthalate, (■) diethyl phthalate, (○) dibutyl suberate, (●) diethyl sebacate, (△) diethyl azelate, (▲) tributyl phosphate.

Table 6. Values of the Coefficients and Relevant Standard Deviation for Eq 10

	tributyl phosphate	diethyl azelate	dibutyl suberate	dioctyl phthalate	diethyl sebacate	diethyl phthalate
c	1.537 84	1.550 46	1.551 37	1.599 60	1.551 68	1.622 87
q/T^{-1}	-3.88×10^{-4}	-3.96×10^{-4}	-3.85×10^{-4}	-3.86×10^{-4}	-3.93×10^{-4}	-4.13×10^{-4}
σ	6.76×10^{-5}	1.00×10^{-4}	1.12×10^{-4}	9.17×10^{-5}	1.14×10^{-4}	1.09×10^{-4}

Table 7. Experimental Kinematic Viscosity and Calculated Dynamic Viscosity

T/K	$\nu/10^{-6}/\mathrm{m^2 \cdot s^{-1}}$	η/mPa·s	T/K	$\nu/10^{-6}/\mathrm{m^2 \cdot s^{-1}}$	η/mPa·s	T/K	$\nu/10^{-6}/\mathrm{m^2 \cdot s^{-1}}$	η/mPa·s
	diethyl phthalate			dioctyl phthalate			diethyl azelate	
292.88	11.6452	13.0239				292.88	5.2895	5.1376
298.15	9.4667	10.5431	298.15	55.8929	54.7698	298.15	4.6260	4.4717
303.18	7.8822	8.7433	303.18	43.0107	41.9842	303.18	4.0996	3.9448
308.19	6.6525	7.3499	308.19	32.9744	32.0640	308.16	3.6631	3.5088
313.07	5.7310	6.3072	313.07	26.1582	25.3409	313.07	3.3032	3.1501
318.04	4.9644	5.4420	318.04	20.9576	20.2256	318.04	2.9894	2.8381
322.92	4.3442	4.7437	322.92	17.0636	16.4061	322.92	2.7218	2.5726
327.89	3.8330	4.1691	327.89	14.1179	13.5223	327.89	2.4863	2.3394
332.86	3.4040	3.6877	332.86	11.8030	11.2620	332.86	2.2823	2.1378
338.30	3.0211	3.2588	338.30	9.8412	9.3511	338.30	2.0888	1.9470
343.49	2.7075	2.9090	343.49	8.3678	7.9207	343.28	1.9335	1.7941
347.98	2.4794	2.6537	347.98	7.3088	6.8928	348.09	1.8013	1.6641
353.93	2.2192	2.3642	353.93	6.2449	5.8632	353.93	1.6600	1.5254
359.05	2.0268	2.1504	359.05	5.4731	5.1184	359.05	1.5480	1.4160
	diethyl sebacate			dibutyl suberate			tributyl phosphate	
292.88	6.1039	5.8815	292.88	7.7282	7.3314	292.88	3.9053	3.8156
298.15	5.3183	5.1006	298.15	6.6759	6.3042	298.15	3.4388	3.3440
303.18	4.6984	4.4860	303.18	5.8519	5.5021	303.18	3.0706	2.9726
308.19	4.1831	3.9763	308.19	5.1767	4.8462	308.19	2.7603	2.6603
313.07	3.7621	3.5606	313.07	4.6232	4.3098	313.07	2.5039	2.4027
318.04	3.3928	3.1969	318.04	4.1477	3.8499	318.04	2.2774	2.1757
322.92	3.0825	2.8920	322.92	3.7448	3.4613	322.92	2.0747	1.9734
327.89	2.8071	2.6220	327.89	3.3984	3.1276	327.89	1.9048	1.8038
332.86	2.5712	2.3910	332.86	3.0930	2.8343	332.86	1.7582	1.6576
338.30	2.3461	2.1712	338.30	2.8096	2.5625	338.30	1.6162	1.5164
343.28	2.1648	1.9946	343.28	2.5802	2.3431	343.28	1.5001	1.4012
348.09	2.0117	1.8456	348.09	2.3889	2.1604	348.09	1.4019	1.3039
353.93	1.8493	1.6879	353.93	2.1853	1.9663	353.93	1.2971	1.2001
359.05	1.7214	1.5640	359.05	2.0299	1.8184	359.05	1.2174	1.1212

error. Experimental and calculated density values are shown in Figure 2.

Refractive Index. Refractive index was determined in the temperature interval between 288.15 and 323.5 K, and the experimental results are reported in Table 5, together with the relevant Eykman constants (Riddick et al., 1986) evaluated as

$$C/\mathrm{g^{-1} \cdot mm^3} = \frac{(n^2 - 1)}{(n + 0.4)} \frac{1}{\rho/\mathrm{g \cdot mm^{-3}}} \tag{9}$$

where n is the refractive index and ρ is the density, interpolated at the correct temperature by means of eq 7.

Table 5 shows the refractive index measured. The refractive index results were fit to

$$n = c + qT/\mathrm{K} \tag{10}$$

Parameters of equation and relevant standard deviation (eq 8) are reported in Table 6.

Viscosity. The experimental kinematic viscosity data in the temperature range 293.15 K −358.15 K are reported

Table 8. Percent Decrease in the Kinematic Viscosity between 298.15 K and 358.15 K

diethyl phthalate	dioctyl phthalate	diethyl azelate	diethyl sebacate	dibutyl suberate	tributyl phosphate
78.6	90.2	66.5	67.6	69.6	64.6

Table 9. Values of the Coefficients and Relevant Standard Deviation for Eq 12

	$A/\mathrm{mm^2 \cdot s^{-1}}$	B/K	C/K	$\sigma/\mathrm{mm^2 \cdot s^{-1}}$
diethyl phthalate	0.0875	582.42	−173.79	1.03×10^{-3}
dioctyl phthalate	0.0611	799.00	−181.14	5.32×10^{-3}
diethyl azelate	0.0826	657.07	−134.93	5.07×10^{-4}
diethyl sebacate	0.0808	691.77	−132.94	4.25×10^{-4}
dibutyl suberate	0.0841	711.80	−135.42	5.12×10^{-4}
tributyl phosphate	0.0730	634.04	−133.60	1.57×10^{-3}

in Table 7 and Figure 3. Table 7 reports also the dynamic viscosity calculated from the experimental kinematic viscosity and the extrapolated density obtained by eq 7. With the exception of dioctyl phthalate, all esters have a kinematic viscosity of less than 2 $\mathrm{mm^2\ s^{-1}}$ (extrapolated value) at 373.15 K. Consequently, the viscosity index (VI) according to ASTM D 2270 (VI is a parameter describing the decrease of the lubricant viscosity with increasing temperature) could not be evaluated. For a comparison between the temperature behavior of the considered esters, an indication of the viscosity loss with increasing temperature is put forward on the basis of the % ν decrease, i.e., of the index %KVd, defined as

$$\%\mathrm{KVd} = 100\,\frac{\nu_{298.15} - \nu_{358.15}}{\nu_{298.15}} \qquad (11)$$

where $\nu_{298.15}$ and $\nu_{358.15}$ are the kinematic viscosities at 298.15 K and 358.15 K, respectively. Table 8 shows that the compounds considered have markedly different values of %KVd. Kinematic viscosity data were fitted with the Goletz and Tassios equation (Reid et al., 1989)

$$\nu/\mathrm{mm^2 \cdot s} = A\,\exp\!\left(\frac{B}{(T/\mathrm{K} + C)}\right) \qquad (12)$$

where A, B, and C are adjustable parameters. Table 9 reports the values of the parameters and the standard deviation of the fit (eq 8).

Conclusions

For all esters tested, irrespective of the chemical nature, density values regularly decrease with increasing temperature and the Daubert and Danner equation used for the data regression gives standard deviations of the same order of magnitude of the experimental error.

The same behavior vs temperature variations were found for the refractive index for all the esters. A linear relation is suitable for fitting the experimental data. Application of the Eykman correlation gives no variations with temperature for tributyl phosphate, dioctyl phthalate, and diethyl azelate and a $C/\mathrm{cm^3\ g^{-1}}$ variation of 1×10^{-3} for diethyl sabacate, dibutyl suberate, and diethyl phthalate.

The kinematic viscosity percent decrease between 293.15 K and 358.15 K varies in dependence on the chemical composition of the plasticizer tested. The experimental data were fitted by the Goletz and Tassios equation.

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