J. Chem. Eng. Data **1981**, *26*, 323-332 **323**

Table IV. Henry's Constants of H_2 in 9-Methylanthracene, 2-Ethylanthraquinone, and Dibenzofuran

temp, °C	Henry's constant, atm 9-methyl-anthracene	 2-ethylan-thraquinone	 dibenzo-furan
100	4140		3750
125		3520	
150	3240	3175	2925
200	2525	2550	2395
250	2025		1920

where subscript 2 stands for the solute and 1 for the solvent; x_2 is the mole fraction of the solute in the solvent; f_2 is the fugacity of the solute in the gas phase. Henry's constants for hydrogen in the three aromatic compounds were evaluated by extrapolating a plot of f_{H_2}/x_{H_2} to $x_H = 0$. The fugacity of hydrogen in the gas phase, f_{H_2}, was calculated from the Soave-Redlich-Kwong equation of state (*4*). Figure 2 shows the plot of Henry's constants vs. temperature. The values of these constants are tabulated in Table IV. For comparison purposes, the Henry's constant of hydrogen in 9,10-dihydrophenanthrene, calculated from data of Sebastian et al. (*3*), are also shown in Figure 2.

Acknowledgment

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Density and Surface Tension of 83 Organic Liquids

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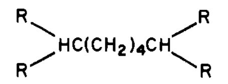
Densities and surface tensions of organic liquids of known purity were measured in the temperature range of 20-80 °C. In many cases literature data from apparently reliable sources conflicted with each other. Our measurements were conducted with the aim of making a choice. Also included are data of a few compounds of higher molecular weight measured in a broader temperature range.

In the present paper densities and surface tensions are given for 75 pure liquids and 8 purified polymers. The surface tensions of about half of the substances have been reported in the excellent critical review of Jasper (*1*), and his recommended values are compared with our data in Table II. In most cases the surface tensions agree within the experimental error, but, where significant deviations were found, the substances were reexamined with special care in order to obtain the most reliable values.

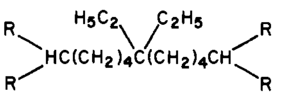
1. Compounds

In Table I information is given about origins, methods of purification, purities, refractive indexes, and melting points of the compounds examined. Compounds **16-24** are branched hydrocarbons of the general structures **A1** and **A2** with following formulas:



A1

A1-C_6, R = hexyl
A1-C_{10}, R = decyl
A1-C_{14}, R = tetradecyl
A1-C_{18}, R = octadecyl



A2

A2-C_{11}, R = hendecyl
A2-C_{13}, R = tridecyl
A2-C_{15}, R = pentadecyl
A2-C_{18}, R = octadecyl
A2-C_{22}, R = docosyl

Squalane (**15**) is a mixture of the diastereomers of 2,6,10,15,19,23-hexamethyltetracosane. Compounds **69-76** are poly(ethylene glycols) (PEGs) and their methoxylated derivatives (PEG-M) with nominal molecular masses of 600, 1000, 2000, and 20 000. The average molecular masses of the

$HO(CH_2CH_2O)_xH$ PEG
$H_3CO(CH_2CH_2O)_xCH_3$ PEG-M

PEGs determined by the method of ref *2* are as follows: PEG-600, 6.10 × 10^2; PEG-1000, 1.13 × 10^3; PEG-2000, 2.15 × 10^3; PEG-20000, 1.71 × 10^4. The derivatives PEG-M were prepared from the PEGs; consequently, their average molecular masses are MW(PEG-M) = MW(PEG) + 24. Under the synthetic conditions no degradation is to be expected.

1.1. Origin. In the third column of Table I, the origin of the compounds is indicated by using the following abbreviations: Fl, Fluka AG, Buchs, Switzerland; EGA, EGA-Chemie, Steinheim, Germany; Sh, Shell Research Laboratories, Amsterdam, Holland; Me, Merck AG, Darmstadt, Germany; Sy1, Synthetized in our laboratory, see ref *3*; Sy2, Synthetized in our laboratory, see ref *2*.

1.2. Purification. Commercially available research-grade compounds have been further purified. The method of purification is indicated in the fourth column of Table I by using the following abbreviations.

Di: Distillation of the product in a Vigreux column discarding a forerun and a residue, each representing 25% of the distilled material. This operation was capable of removing other members of homologous series to less than 0.2%.

Di/Na: Distilled over sodium.

Di/Hg: Distilled over mercury.

Cr: Recrystallization from diethyl ether (DE), propanol (Pr), cyclohexane (CH), ethanol (E), or *n*-hexane (H) or from a mixture of them. For example, the symbol "Di; 3×Cr(E/DE)" means that the middle fraction of the distillation was recrystallized three times from a mixture of ethanol and diethyl ether.

Fi: Filtration on a silica gel column (Merck: Kieselgel 60)

0021-9568/81/1726-0323$01.25/0

Table I Compounds. For a detailed description of the table see section 1.

No	Compound	Molecular weight	Origin	Method of purification	Gas chromatographic analysis				n_d^{20}	m.p.	Remarks
					T_c °C	Main component I^A	Impurities retention index (I^A)/% in chromatogram	total impurities %		°C	see section 1.5.
	Hydrocarbons										
	Alkanes										
1.	Decane	142.29	Fl	Di; Fi	140	1000	1034/.02; 1065/.02	.04	1.4124		a
2.	Hendecane	156.32	Fl	Di; Fi	140	1100	999/.37h; 1200/.19h; 1300/.04h; 1399/.03h	.63	1.4171		a
3.	Dodecane	170.34	Fl	Di; Fi	140	1201	1001/.02h; 1099/.04h; 1299/.05h	.11	1.4221		a
4.	Tridecane	184.37	Fl	Di; Fi	180	1300	1099/.10h; 1146/.02; 1173/.02	.14	1.4260		a
5.	Tetradecane	198.40	Fl	Di; Fi	210	1399	1299/.02h; 1346/.04; 1370/.20; 1461/.02; 1499/ 19h	.47	1.4289		a
6.	Pentadecane	212.42	Fl	Di; Fi	210	1500	1324/.02; 1388/.02; 1599/.02h	.06	1.4316		a
7.	Hexadecane	226.45	Fl	Di; Fi	210	1601	1399/.02h; 1499/.03h; 1701/.02h	.07	1.4347		a
8.	Heptadecane	240.48	Fl	Di; Fi	250	1698	1200/.05h; 1301/.07h; 1402/.04h; 1599/.02/; 1801/.04h	.22		22.0[27]	a
9.	Octadecane	254.51	Fl	Di; Fi	260	1800	1501/.02h; 1600/.01h; 1739/.22; 1772/.25; 1862/.02	.52		28.2[27]	a
10.	Nonadecane	268.53	Fl	Di; Fi	260	1901	1598/.04h; 1648/.02; 1744/.02; 1933/.08; 1950/.02; 1962/.11	.29		32.1[27]	a
11.	Eicosane	282.56	Fl	Di; Fi	260	2000	1564/.05; 1841/.03; 1933/.08; 2036/.02	.18		36.8[27]	a
	Isoalkanes										
12.	3,8-Diethyldecane	198.40	Sy1	Di; Fi	180	1324	930/.04; 1099/.02; 1232/.10; 1301/.10; 1385/.09	.35	1.4347		a
13.	2,6,10,14-Tetramethylpentadecane	268.53	Sh	Di; Fi	260	1686	1273/.04; 1332/.01; 1489/.04 1631/.01; 1648/.03; 1792/.25	.38	1.4380		a,b
14.	2,2,4,4,6,8,8-Heptamethylnonane	226.45	Sh	Di; Fi	210	1337	904/.05; 1382/.23; 1404/.04; 1500/.03	.35	1.4396		a
15.	Squalane	422.83	Fl	Di; Fi					1.4521		a,b,c
16.	A1-C_6 ($C_{30}H_{62}$)	422.83	Sy1	2xCr(DE/E)							
17.	A1-C_{10}($C_{46}H_{94}$)	647.26	Sy1	4xCr(DE/E)						46.0-46.5	a,d
18.	A1-C_{14}($C_{62}H_{126}$)	871.69	Sy1	4xCr(DE)						65.0-66.5	a,d
19.	A1-C_{18}($C_{78}H_{158}$)	1096.12	Sy1	4xCr(CH)						72-78	a,d
20.	A2-C_{11}($C_{59}H_{120}$)	829.61	Sy1	Cr(Pr/H/E)						5,5-7	a,d
21.	A2-C_{13}($C_{67}H_{136}$)	941.83	Sy1	Cr(Pr/H/E)						13,5-14.5	a,d
22.	A2-C_{15}($C_{75}H_{152}$)	1054.04	Sy1	Cr(Pr/H/E)						22.5-23.5	a,d
23.	A2-C_{18}($C_{87}H_{176}$)	1222.37	Sy1	Cr(Pr/H/E)						37.5-38.5	a,d
24.	A2-C_{22}($C_{103}H_{208}$)	1446.80	Sy1	Cr(Pr/H/E)						47.5-48.5	a,d
	Alkenes										
25.	1-Tetradecene	196.38	Fl	Di; Fi	210	1388	1096/.02; 1299/.04; 1355/.02; 1324/.07	.15	1.4366		a
26.	1-Octadecene	252.49	Fl	Di; Fi	250	1784	1598/.03; 1691/.09; 1754/.01; 1900/.05	.18	1.4451		a
	Cyclic hydrocarbons										
27.	Cyclohexane	84.15	Me	Di; Fi	80	678	599/.02; 637/.01	.03	1.4268		a
28.	*cis*-Decalin	138.25	Fl	Di; Fi	140	1162	1119/.52; 1198/.05; 1225/.01	.58	1.4811		a,e
29.	*trans*-Decalin	138.25	Fl	Di; Fi	140	1121	1000/.01; 1108/.01	.02	1.4693		a
	Aromatic hydrocarbons										
30.	Benzene	78.12	Me	Di/Na	80	663	497/.01; 739/.08	.09	1.5011*		f
31.	Toluene	92.15	Me	Di/Na	80	769	664/.02	.02	1.4969		

Table I (continued)

No	Compound	Molecular weight	Origin	Method of purification	Gas chromatographic analysis				n_d^{20}	m.p.	Remarks
					T_c °C	Main component I^A	Impurities retention index (I^A)/% in chromatogram	total impurities %		°C	see section 1.5.
Alkane derivatives											
	1-Halogenoalkanes										
32.	1-Chlorotetradecane	232.85	Fl	Di; Fi	240	1674	1376/.02; 1468/.04; 1548/.02; 1570/.02h; 1595/.12; 1616/.03; 1631/.03; 1774/.05h; 1805/.03	.38	1.4475		
33.	1-Chlorohexadecane	260.90	Fl	2xCr(DE/E); Di	250	1878	1584/.03; 1777/.18h; 1840/.32; 1940/.07	.60	1.4503		
34.	1-Chloroöctadecane	288.95	Fl	2xCr(DE/E); Di	260	2080	1781/.01h; 1878/.16h; 1940/.14; 1981/.25h; 1991/.05; 2026/.15; 2051/.11; 2129/.03	.90	1.4511	28.6[27]	
35.	1-Bromotetradecane	277.30	Fl	Di; Fi	250	1781	1380/.07h; 1579/.23h; 1630/.02; 1679/.09h; 1697/.02; 1823/.02; 1883/.03h	.48	1.4609		
36.	1-Bromohexadecane	305.35	Fl	2xCr(DE/E); Di	260	1986	1584/.13h; 1784/.05h; 1877/.15; 1847/.07; 1935/.07; 1948/.07	.54	1.4620		
37.	1-Bromoöctadecane	333.41	Fl	2xCr(DE/E); Di	260	2184	1800/.11; 2119/.02; 2129/.25; 2148/.09; 2209/.05	.52	-	28.2[27]	
	1-Cyanoalkanes										
38.	Acetonitrile	41.06	Me	Sp. ref.[4]					1.3442		
39.	Cyanoethane	55.08	Fl	Di					1.3658		
40.	1-Cyanobutane	83.13	Fl	Di	130	721	595/.45	.45	1.3970		
41.	1-Cyanohendecane	181.33	Fl	Di	230	1425	1225/.39h; 1327/.17h; 1483/.04	.60	1.4361		
42.	1-Cyanotridecane	209.38	Fl	Di	250	1641	1097/.02; 1332/.05h; 1420/.05; 1530/.08h; 1595/.02; 1833/.03h	.25	1.4413		
43.	1-Cyanohexadecane	251.46	Fl	2xCr(H); Di	260	1937	1581/.04; 1630/.03h; 1733/.02h; 1777/.08; 1837/.25h; 1985/.05; 2065/.14	.61		34[27]	
44.	1-Cyanoheptadecane	265.49	EGA	2xCr(H); Di				~.2		41[27]	
45.	1 Cyanoöctadecane	279.52	Fl	2xCr(H); Di				~.2		42	
	Alkanols										
46.	Methanol	32.04	Me					<.02			
47.	Ethanol	46.07	Me					<.02			
48.	2-Butanol	74.12	Fl	Di	60	563		<.02	1.3976		
49.	1-Hexanol	102.17	Fl	Di	140	811	764/.02; 799/.08	.10	1.4179		
Benzene and naphthelene derivatives											
50.	Fluorobenzene	96.11	Fl	Fi	80	657		<.02	1.4660		
51.	Chlorobenzene	112.56	Fl	Fi	130	869		<.02	1.5248		
52.	Bromobenzene	157.02	Fl	Fi	140	963		<.02	1.5601		
53.	Iodobenzene	204.01	Fl	Di/Hg; Fi	180	1122	1091/.10	.10	1.6201		
54.	Nitrobenzene	123.11	Fl	Di	140	1077		<.02	1.5521		
55.	Methoxybenzene	108.15	Fl	Di	140	920		<.02	1.5172		
56.	N,N-Dimethylanilin	121.18	Fl	Di				<.02	1.5582		
57.	Dimethylphtalate	194.19	Fl	Di	220	1384	945/.02; 1156/.09; 1518/.01	.12	1.5150		
58.	1-Chloronaphtalene	162.62	Fl	Di	220	1497	1295/.12; 1429/.07	.19	1.6321		
Methane derivatives											
59.	Nitromethane	61.04	Fl	Di				<.02	1.3817		
60.	Dichloromethane	84.93	Fl	Sp. ref.[4]	60	509	605/.04; 663/.09	.13	1.4246		
61.	Tetrachloromethane	153.82	Me	Di; Fi	60	663		<.02	1.4601		
62.	Dibromomethane	173.85	Fl	Di; Fi	60	709		<.02	1.5419		
63.	Diiodomethane	267.84	Fl	Di/Hg; Fi				~.2	*(1.7425)*		

Table I (continued)

No	Compound	Molecular weight	Ori-gin	Method of purification	Gas chromatographic analysis				n_d^{20}	m.p.	Remarks
					T_c °C	Main component I^A	Impurities retention index (I^A)/% in chromatogram	total impurities %		°C	see section 1.5.
Oligo-and poly-(ethyleneglycols)											
64	1,2-Ethanediol	62.07	Fl	Di				<.02	1.4281		
65	1,2-Dimethoxyethane	90.12	Fl	Di; Fi	60	601	579/.27	.27	1.3815		
66	2,5,8-Trioxanonane	134.18	Fl	Di; Fi	140	867	797/.01	.02	1.4090		
67	2,5,8,11-Tetraoxadodecane	178.24	Fl	Di; Fi	140	1126	600/.01; 776/.01; 829/.01;847/.01	.05	1.4235		
68	2,5,8,11,14-Pentaoxapentadecane	222.29	Fl	Di; Fi	180	1379	1004/.01; 1102/.01; 1219/.02	.05	1.4326		
69	PEG-600	610	Fl	Sp ref[2]						20-22	
70	PEG-1000	$1.13 \cdot 10^3$	Fl	Sp ref[2]						42-45	
71	PEG-2000	$2.15 \cdot 10^3$	Fl	Sp ref[2]						52-55	
72	PEG-20000	$1.71 \cdot 10^4$	Fl	Sp ref[2]						60-65	
73	PEG-M-600	638	Sy2	Sp ref[2]						22-25	
74	PEG-M-1000	$1.16 \cdot 10^3$	Sy2	Sp ref[2]						43-45	
75	PEG-M-2000	$2.18 \cdot 10^3$	Sy2	Sp ref[2]						52-55	
76	PEG-M-20000	$1.71 \cdot 10^4$	Sy2	Sp ref[2]						60-66	
Miscellaneous											
77	Formamide	45.04	Fl	Di				<.02	1.4462		
78	1,4-Dioxane	88.12	Me	Sp ref[4]				<.02	1.4224		
79	Pyridine	79.10	Me	Sp ref[4]				<.02	1.5100		
80	Tetrahydrofurane	72.12	Fl	Di/Na [4]				<.02	1.4072		
81	Cyclohexanone	98.15	Me	Di	140	880		<.02	1.4501		
82	Dimethyladipate	174.20	Fl	Di	220	1132		<.02	1.4281		
83	Dimethylsulfoxide	78.13	Fl	Di				<.02	1.4776		

prepared with ~$^1/_4$ of the weight of the substance to be purified.

Sp: Special purification method described in the reference indicated in the table.

1.3. ***Purity.*** Gas-chromatographic analysis, the results of which are summarized in the fifth column of Table I, was performed under the following experimental conditions: Packard Becker (Delft, Holland) gas chromatograph Model 419, with flame ionization detector; column, 2.8-m Pyrex glass tube of 0.4-cm i.d.; stationary phase, 5% (weight/total weight) Apolane-87 on Chromosorb G (Apolane-87 is identical with substance **23** of Table I); carrier gas, helium; chromatograms at the indicated temperature, T_c (°C); retention indexes were determined at the temperature T_c.

Impurities. The retention index and the amount of each impurity are given (the percentages are surface percents estimated from the areas in the chromatogram); the symbol h after the percentage of an impurity indicates that it is probably a homologue of the main component.

1.4. ***Refractive Index and Melting Point.*** The refractive indexes of the pure compounds were determined with a thermostated (20.0 ± 0.2 °C) Zeiss refractometer (Abbé system); $\Delta_{95} = \pm 0.0003$. Melting points are literature data from the references indicated.

1.5. ***Remarks (Table I).*** (a) The filtration over silica gel eliminates all polar compounds. Consequently, any impurities are hydrocarbons. (b) Mixture of diastereomers. (c) The commercial squalane was first fully hydrogenated with Pt as a catalyst. (d) For details of purification, see ref *3*. The purity of the hydrocarbons of the series **A1** and **A2** could be determined neither by gas chromatography (too low volatility) nor by calorimetry. The purity is estimated to be better than 99.5%. (e) The impurity with $I^A{}_{140} = 1119$ IU is *trans*-decalin. (f) The refractive index of benzene from ref *6* and that of carbon tetrachloride from ref *27* were accepted for the calibration of the refractometer.

2. Experimental Section

2.1. ***Density.*** Pycnometers were thermostated to ±0.05 K in a water bath up to 70 °C (Tamson, Zoetermeer, Holland; thermostat Model TX9-100) and in a stirred silicon oil bath (±0.3 K) up to 200 °C. The temperature of the bath was controlled with calibrated precision thermometers. All weights were corrected to vacuum; the weighing error was 0.0002 g.

The density of the pure compounds was determined with ASTM-pycnometers (Model 0941-47T) on a sample of ~5 cm^3. The calibration with distilled mercury was made between 20 and 200 °C; the error of the volume of the pycnometer, V_{py}, was estimated to ~±0.0002 cm^3. The density is calculated by

$$d = (w_{tot} - w_{py})/V_{py} \qquad (1)$$

where w_{tot} and w_{py} are for the weight of the filled and empty pycnometer. The estimation of the variance of the density, V(d), gives

Table II Density and surface tension between 20°C and 70°C. For detailed discussion see section 3.
Data cited from literature in italics

Compound	Temp. domain	Density, d [g.cm^{-3}] and coefficient of thermal expansion, κ [K^{-1}]							Surface tension, γ, [dyn.cm^{-1}] and its temperature dependence, b = ∂γ/∂T [dyn.cm^{-1} K^{-1}]							Remarks see section 3.3.
		d at		10^4x κ	10^4x Δ_{95}		Literature data	Further literature	γ at		-b	Δ_{95}		Literature data	Further literature	
No.	°C	20°C	80°C	K^{-1}	min.	max.	d_{20}; κ [ref.]		20°C	80°C		min.	max.	γ_{20}; -*b* [ref.]		
1.	20-70	*.7301*[6]	*.6838*[6]						23.74	18.24	.0918	.034	.060	*23.83; .0917* [1][7]		a
2.	20-70	*.7402*[6]	*.6953*[6]						24.65	19.23	.0904	.052	.092	*24.64; .0910* [1][7]		a
3.	20-70	*.7488*[6]	*.7051*[6]						25.58	19.97	.0894	.030	.053	*25.35; .0884* [1][7]		a,d
4.	20-70	*.7561*[6]	*.7141*[6]						25.95	20.67	.0880	.047	.083	*25.99; .0872* [1][7]		a
5.	20-70	*.7626*[6]	*.7220*[6]						26.69	21.28	.0869	.034	.060	*26.55; .0869* [1][7]		a
6.	20-70	*.7684*[6]	*.7269*[6]						27.07	21.93	.0858	.060	.107	*27.07; .0857* [1][7]		
7.	30-70	*.7735*[6]	*.7325*[6]						27.49	22.34	.0857	.038	.066	*27.47; .0854* [1][7]		
8.	30-70	*.7780*[6]	*.7372*[6]						27.88	22.77	.0851	.049	.085	*27.91; .0846* [1][7]		c
9.	30-70	*.7828*[6]	*.7415*[6]						28.27	23.22	.0843	.039	.068	*28.29; .0843* [1][7]		c
10.	40-70	*.786* [6]	*.746* [6]						28.60	23.61	.0833	.025	.042	*28.59; .0837* [1][7]		c
11.	40-70	*.7843*[6]	*.7491*[6]						28.86	23.91	.0825	.049	.082	*28.87; .0838* [1][7]		c
12.	20-70	.7770	.7340	9.49	1.3	2.1	*.7778; 8.82*[8]		26.10	21.22	.0814	.037	.065			
13.	20-70	.7828	.7415	9.04	1.9	3.2	*.7853;* - [9]	[10]*	26.24	21.50	.0790	.035	.062			
14.	20-70	.7850	.7447	8.79	1.4	2.3	-		24.32	19.83	.0747	.023	.042			
15.	20-80	.8092	.7701	8.25		~ 2.0	*.8090;* - [11]	[12]	28.15	23.85	.0721	.054	.102			
16.	20-80	.8137	.7730	8.55		~ 2.0	-	-	28.40	24.04	.0728	.051	.097			b
17.	50-80	-	.7883	7.79		~ 2.0	-	-	-	26.36	.0689	.059	.110			b
18.	70-80	-	.7961	7.84		~ 2.0	-	-	-	27.27	.0633	.078	.143			b
19.	80	-	.8006	7.48		~ 2.0	-	-	-	27.87	.0618	.097	.177			b
20.	20-80	.8365	.8002	7.40		~ 2.0	-	-	30.96	26.97	.0665	.058	.109			b
21.	20-80	.8383	.8021	7.37		~ 2.0	-	-	31.24	27.32	.0653	.053	.100			b
22.	50-80	-	.8047	7.45		~ 2.0	-	-	31.41	27.62	.0631	.047	.088			b
23.	50-80	-	.8058	7.41		~ 2.0	-	-	-	28.03	.06095	.041	.078			b
24.	60-80	-	.8071	7.44		~ 2.0	-	-	-	28.46	.0595	.049	.091			b
25.	20-70	.7714	.7290	9.42	.9	1.6	*.7716; 9.55* [6]	[13]	26.75	21.56	.0865	.041	.072	*26.79; .0878* [1][7]	[14]	
26.	20-70	.7891	.7482	8.86	1.3	2.3	*.7888; 9.06* [6]		28.45	23.31	.0858	.044	.078	*28.49; .0852* [1][6]		
27.	20-60	*.7786*[6]	*.7212*[6]						25.16	17.99	.1195	.036	.060	*25.24; .1188* [1][15]	[16]*[17]	a
28.	20-70	.8966	.8511	8.67	.7	1.2	*.8968; 8.68*[18]	[13]	32.18	26.41	.0961	.029	.051	*32.06; .1086* [1][15]	[16]*	d
29.	20-70	.8690	.8250	8.65	.8	1.5	*.8698; 8.83*[18]	[13]	30.15	24.59	.0928	.040	.071	*29.87; .1015* [1][15]	[16]*	d
30.	20-60	*.8790*[6]	*.8134*[6]						28.81	21.07	.1290	.059	.104	*28.86; .1296* [1][6]	[16][19]	a
31.	20-70	*.8670*[6]	*.8115*[6]						28.52	21.43	.1181	.046	.081	*28.52; .1189* [1][6]	[16][19]	a

Table II (continued)

Compound	Temp. domain	Density, d [g.cm^{-3}] and coefficient of thermal expansion, κ [K^{-1}]							Surface tension, γ, [dyn.cm^{-1}] and its temperature dependence, b = ∂γ/∂T [dyn.cm^{-1} K^{-1}]							Remarks see section 3.3.
No.	°C	d at 20°C	80°C	$10^4\times\kappa$ K^{-1}	$10^4\times\Delta_{95}$ min.	max.	Literature data d_{20}; κ [ref.]	Further literature	γ at 20°C	80°C	−b	Δ_{95} min.	max.	Literature data γ_{20}; −b [ref.]	Further literature	
32.	20-70	.8662	.8214	8.85	2.1	3.5	.8657; - [20]	[21]*	30.36	25.19	.0861	.025	.046			
33.	20-70	.8648	.8214	8.59	2.0	3.4	.8649; - [22]	[20];[21]*	30.98	25.95	.0839	.035	.065	31.10 - [1][22]		d
34.	30-70	.8628	.8204	8.41	1.4	2.5	.8633; - [20]		31.51	26.51	.0832	.033	.061			
35.	20-70	1.0173	.9660	8.63	2.1	3.7	1.0176; 8.19[23]	[24]	31.17	26.03	.0856	.038	.070	31.18; .0873[1][23]		
36.	20-70	.9989	.9496	8.44	2.7	4.4	.9998; 8.10[23]	[22]	31.65	26.65	.0832	.036	.067	31.65; .0861[1][23]	[24]	
37.	30-70	.9850	.9374	8.26	2.1	3.7	.9841; 8.30[25]		32.01	27.27	.0800	.043	.080			
38.	20-60	.7824	.7208	13.68	.8	1.3	.7843;13.42[14]		29.29	21.77	.1252	.072	.120	29.29; .1263[1][14]		
39.	20-60	.7823	.7324	13.04	.9	1.6	.7828;12.84[14]		27.24	20.56	.1114	.067	.113	27.23; .1135[1][14]		
40.	20-60	.7995	.7491	10.79	.9	1.6	.7992;10.81[14]	[22]	27.39	21.89	.0916	.046	.081	27.43; .0932[1][14]	[26]	
41.	20-70	.8239	.7789	9.35	1.7	3.0	.8240; - [27]		29.82	25.24	.0763	.053	.095			
42.	20-70	.8278	.7855	8.74	1.4	2.2	.8281; - [27]		30.70	26.18	.0758	.053	.095			
43.	40-70	.8319	.7904	8.50	1.1	1.9	.8355; - [22]		31.37	26.88	.0749	.051	.092	30.60 [22]		c
44.	45-70	.8380	.7971	8.34	-	-	.8325; - [27]		31.50	27.10	.0738	-	-			c
45.	45-70	.8400	.7995	8.24	.9	1.5			31.60	27.22	.0730	.023	.040			c
46.	20-50	.7912[6]							22.49	17.78	.0786	.159	.266	22.51; .0744[1][28]		a
47.	20-50	.7893[6]							22.32	17.39	.0823	.095	.159	22.39; .0832[1][28]	[29]	a
48.	20-60	.8077	.7558	11.07	.3	.5	.8073;11.54[30]	[31]	23.37	18.23	.0856	.042	.072	23.47; - [26]	[29]	
49.	20-70	.8205	.7765	9.16	.9	1.5	.8205; 9.34[28]	[22][32]	26.20	21.44	.0792	.070	.121	26.21; .0801[1][33]	[22]*[32]*	
50.	20-60	1.0246	.9539	11.92	3.1	5.2	1.0240;11.75[34]	35	27.28	20.03	.1207	.052	.091	27.26; .1204[1][34]	[35]*	
51.	20-70	1.1066	1.0419	10.00	1.6	2.6	1.1064; 9.99[36]	[37][38][39]	32.96	26.00	.1160	.045	.080	33.59; .1191[1]	[22]*[19]*[26]*[38]	d
52.	20-70	1.4959	1.4144	9.33	3.1	4.4	1.4956; 9.15[36]	[38]*[39][40]	35.80	28.66	.1158	.026	.046	35.82; .1160[1][38]	[26]* [19]*	
53.	20-40	1.8316	1.7548	8.56	5.6	9.4	1.8313; - [38]	[41]	39.57	-	.1020	.418	.660	39.34; .1132[1][38]		d
54.	20-70	1.2026	1.1446	8.24	.8	1.5	1.2031; 7.90[34]	[39][42]	42.70	36.08	.1104	.101	.161	44.03; .1157[1][43]	[42]*[34]*[44]*	d
55.	20-70	.9939	.9391	9.51	2.0	3.3	.9939; - [45]	[46]*	35.00	27.98	.1171	.028	.046	35.70; .1204[1][46] (35.45; .1346 [45])	[45]*	d
56	20-40	.9559	-	8.65	1.3	2.0	.9571; 7.99[47]	[42]	36.04	-	.0974	.051	.092	36.04; .1049[1][47]		f
57	20-70	1.1917	1.1369	7.85	.7	1.3	1.1899; - [48]		41.84	35.51	.1054	.024	.041			
58	20-70	1.1916	1.1426	6.99	1.5	2.7	1.1910;6.79 [34]		41.04	35.57	.0912	.080	.135	42.05; .1035[1][49]	[34]	d
59	20-60	1.1381	1.0594	11.95	1.3	2.4	1.1371;12.25[50]	[41][51] [52]*[53]*[54]	37.36	27.33	.1673	.107	.185	37.48; .1678[1][55]	[41][50]*; [56]*[32]*	
60	20-35	1.3264	-	13.91	19.6	31.4	1.3255;13.98[57]	[58]*[59][37]	27.89	-	.1309	-	-	27.85; .1308 [1][58]	[22];[59]*	
61	20-60	1.5938	1.4809	12.25	2.3	4.1	1.5941;12.11[58]	[52][16][60] [61]*	26.92	19.50	.1236	.096	.167	27.04; .1224[1]	[61]*[62]*[26]*	d
62	20-70	2.4965	2.3365	11,04	5.6	9.4	2.4956;10.59[58]	[57];[62]	39.74	30.88	.1478	.078	.139	39.79; .1478 [1][58]	[63]*	
63	20-40	3.2992	-	8.33	3.9	6.1	3.3212; 7.94[57]	[58]*	50.88	-	.0971	.242	.383	66.98; .1613[1][64] 50.8 [65]		d,f

Table II (continued)

Compound	Temp. domain	Density, d [g.cm^{-3}] and coefficient of thermal expansion, κ [K^{-1}]							Surface tension, γ, [dyn.cm^{-1}] and its temperature dependence, b ≡ ∂γ/∂T [dyn.cm^{-1} K^{-1}]							Remarks see section 3.3.
		d at		10^4 x κ	10^4 x $Δ_{95}$		Literature data	Further literature	γ at		-b	$Δ_{95}$		Literature data	Further literature	
No.	°C	20°C	80°C	K^{-1}	min.	max.	d_{20}; κ [ref.]		20°C	80°C		min.	max.	$γ_{20}$; -b [ref.]		
64	20-70	*1.1126*[66]	*1.0716*[66]	6.26					48.49	43.16	.0889	.057	.102	*48.43; .0890* [1][67]	[68]*	a
65	20-60	.8691	.8092	11.90	1.9	3.1	*.8689;* - [69]	[70]*	24.61	17.71	.1150	.021	.037			
66	20-70	.9439	.8848	10.78	.5	.8	*.946; 10.1* [70]		29.36	23.13	.1037	.035	.062			
67	20-70	.9867	.9305	9.77	.5	.7	*.9859; 9.5* [70]	[71]*	32.22	26.21	.1001	.029	.052			
68	20-70	1.0121	.9571	9.30	.4	.6	*1.0119; 9.1* [70]	[72]*	34.16	28.19	.0995	.059	.105	*33.89; .1017* [72]		
69	20-80	1.1273	1.0794	7.23		~ 2.0			45.76	39.86	.0984	.052	.092			b
70	40-80	-	1.0799	7.34		~ 2.0			-	39.51	.0951	.042	.082			b
71	60-80	-	1.0813	7.35		~ 2.0			-	39.41	.0947	.092	.160			b
72	80	-	1.0817	7.37		~ 2.0			-	39.35	.0914	.065	.115			b
73	20-80	1.1071	1.0599	7.25		~ 2.0			42.23	36.67	.0928	.104	.185			b
74	60-80	-	1.0608	7.33		~ 2.0			-	37.66	.0910	.100	.174			b
75	60-80	-	1.0634	7.29		~ 2.0			-	38.39	.0888	.040	.070			b
76	70-80	-	1.0646	7.20		~ 2.0			-	38.73	.0870	.087	.151			b
77	20-70	1.1338	1.0823	7.75	.7	1.1	*1.1332; 7.43*[73]	[66]	57.46	52.39	.0845	.043	.076	*57.45; .0842*[1][22]		
78	20-70	1.0343	.9674	11.15	2.0	3.6	*1.0339;11.33*[74]		33.26	25.27	.1331	.063	.112	*33.45; .1391*[1][67]	[75]*	e
79	20-70	.9837	.9225	10.70	1.3	2.0	*.9835;10.72*[76]	[77];[78]	37.24	29.03	.1369	.081	.144	*37.21; .1306*[1][79]	[77]*	
80	20-50	.8884	-	11.38	3.7	5.5	*.8884;* - [80]	[81];[56]	27.31	19.73	.1262	.154	.258	*28.0;* - [65]		
81	20-70	.9452	.8926	9.55	.9	1.3	*.9455; 9.15*[82]	[83]	35.05	27.79	.1210	.061	.108	*35.32; .1243*[1][82]	[83]*	e
82	20-70	1.0620	1.0051	9.28	1.8	2.7	*1.0625: 9.25*[84]		35.86	29.14	.1120	.043	.085	*35.98; .1137*[1][84]		e
83	20-60	1.1005	1.0414	9.28	1.8	2.5	*1.1007; 9.36*[85]	[86]	43.72	37.25	.1078	.080	.141	*43.54; .1145*[1][86]		e

$$V(d) = (\partial d/\partial w_{tot})^2 V(w) + (\partial d/\partial w_{py})^2 V(w) + (\partial d/\partial V_{py})^2 V(V_{py}) + (\partial d/\partial T)^2 V(T) = \left(\sqrt{2}/V_{py}\right)^2 V(w) + [(w_{tot} - w_{py})/V_{py}^2]^2 V(V_{py}) + \kappa^2 V(T) \simeq 3 \times 10^{-8}\ (\text{g cm}^{-3})^2 \tag{2}$$

for measurements up to 80 °C, giving for the confidence limit of a single measurement at the 95% confidence level $\Delta_{95} = \pm 3 \times 10^{-4}$ g cm^{-3}. An analogous estimation gives $\Delta_{95} = \pm 6 \times 10^{-4}$ g cm^{-3} for measurements between 80 and 200 °C.

The density was determined at more or less equidistant temperatures (ca. every 10 °C). In case the experimental temperature range was less than 60 K (in general 20–70 °C), eq 3 was fitted on the experimental points by the method of least squares:

$$\ln d_T = \ln d^\dagger - \kappa_T(T - T^\dagger) \tag{3}$$

where $T^\dagger$ is a standard temperature and κ_T is the mean coefficient of thermal expansion in the experimental domain. For each regression the standard temperature was the average of the experimental temperature domain. At this temperature the confidence limit is the smallest, and it is the largest at the lowest and upper temperatures of the experimental range. For the determinations in a larger domain, it was necessary to put

$$\kappa_T = \kappa^\dagger + \alpha(T - T^\dagger) \tag{4}$$

by choosing $T^\dagger$ = 273.16 K. Consequently eq 5 was fitted on the experimental points

$$\ln d_T = \ln d^\dagger - \kappa^\dagger (T - T^\dagger) - (\alpha/2)(T^2 - T^{\dagger 2}) \tag{5}$$

The confidence limit is a quadratic function of the temperature, being largest at the limits and in the middle of the temperature range.

2.2. Surface Tension. The surface tension was measured with the capillary rise method by using *n*-heptane as secondary standard. Capillaries of 0.27 ± 0.02 mm i.d. were drawn from precision Pyrex tubes (i.d., 3.0 mm; o.d., 7.0 mm) with a drawing machine from Hupe and Busch (Karlsruhe, Germany). The capillary was cut to pieces of 1 m, and the inner diameter determined by measuring the length of the column of a known amount of mercury. Pieces the inner diameter of which deviated more than 1% from the average were discarded. The accepted capillaries were now cut to pieces of 10.0-cm length and digested in nitric acid (65%) for 48 h at room temperature, then washed with distilled water, digested in water for 1 h, and finally dried at 70 °C in a nitrogen flow. The ready-to-use capillaries were stored in sealed ampules in an argon atmosphere. All liquids examined gave a contact angle $\theta = 0°$ on these capillary surfaces.

The capillary rise was measured as described in ref *4*. The height of capillary rise, h_L, was determined in a nitrogen atmosphere on a set of three capillaries placed circularly in a cylindrical closed vessel, made from precision glass in order to avoid parallax effects. The heights were measured with a cathetometer from Pindler and Hoyer (Göttingen, Germany) in sodium light. The height used to calculate the surface tension was the average of the three determinations.

A sample of *n*-heptane was carefully purified until impurities were not detectable by gas chromatography. The surface tension of this sample was measured at regular intervals in

Table III Density and surface tension of substances between 20°C and 200°C

For a detailed discussion see section 3. Symbols and units as in Table II

No.	Compound	Temp. domain °C	Density, d, [g.cm^{-3}] For coefficient of thermal expansion see section 3								Surface tension, γ, [dyn.cm^{-1}] and its temperature dependence, b = ∂γ/∂T[dyn-cm^{-1} K^{-1}]						
			d at κ *(in italics)* 20°C	80°C	140°C	200°C	κ^{+}x10^{4} K^{-1}	αx10^{7} K^{-2}	10^{4}xΔ$_{95}$ min.	max.	γ at 20°C	80°C	140°C	200°C	-b dyn.cm^{-1}K^{-1}	Δ$_{95}$ min.-	max.
	Isoalkanes																
15.	Squalane	20-200	.8092 *8.07*	.7701 *8.43*	.7314 *8.79*	.6930 *9.15*	7.95	5.98	2.3	4.0	28.18	23.85	19.53	15.20	.0721	.054	.102
16.	A1-C$_6$ (C$_{30}$H$_{62}$)	20-200	.8137 *8.34*	.7730 *8.75*	.7326 *9.15*	.6920 *9.56*	8.21	6.73	3.7	7.2	28.40	24.04	19.67	15.30	.0728	.051	.097
17.	A1-C$_{10}$(C$_{46}$H$_{94}$)	50-200	-	.7883 *7.88*	.7511 *8.22*	.7142 *8.57*	7.41	5.82	3.4	6.4	-	26.36	22.32	18.10	.0689	.059	.110
18.	A1-C$_{14}$(C$_{62}$H$_{126}$)	70-200	-	.7961 *7.85*	.7591 *8.02*	.7231 *8.20*	7.62	2.88	3.3	6.5	-	27.27	23.47	19.67	.0633	.078	.143
19.	A1-C$_{18}$(C$_{78}$H$_{158}$)	80-200	-	.8006 *7.48*	.7651 *7.64*	.7305 *7.80*	7.27	2.64	3.2	6.9	-	27.87	24.16	20.46	.0618	.097	.177
20.	A2-C$_{11}$(C$_{59}$H$_{120}$)	20-200	.8365 *7.23*	.8002 *7.56*	.7639 *7.90*	.7278 *8.23*	7.12	5.55	2.7	7.4	30.96	26.97	22.98	18.99	.0665	.058	.109
21.	A2-C$_{13}$(C$_{67}$H$_{136}$)	20-200	.8383 *7.21*	.8021 *7.52*	.7660 *7.84*	.7302 *8.15*	7.10	5.26	2.1	5.0	31.24	27.32	23.40	19.48	.0653	.053	.100
22.	A2-C$_{15}$(C$_{75}$H$_{152}$)	40-200	-	.8047 *7.54*	.7685 *7.80*	.7328 *8.07*	7.18	4.44	3.3	6.0	31.41	27.62	23.83	20.04	.0631	.047	.088
23.	A2-C$_{18}$(C$_{87}$H$_{176}$)	50-200	-	.8058 *7.46*	.7700 *7.67*	.7349 *7.88*	7.18	3.51	2.2	3.1	-	28.03	24.38	20.72	.06095	.041	.078
24.	A2-C$_{22}$(C$_{103}$H$_{208}$)	60-200	-	.8071 *7.45*	.7716 *7.52*	.7374 *7.59*	7.35	1.21	2.9	5.1	-	28.46	24.88	21.31	.0595	.049	.091
	Poly(ethylene glycols)																
69.	PEG-600	20-150	1.1273 *7.03*	1.0794 *7.43*	1.0311 *7.82*	-	6.90	6.60	3.9	9.0	45.76	39.86	33.95	-	.0984	.052	.092
70.	PEG-1000	40-150	-	1.0799 *7.44*	1.0319 *7.74*	-	7.04	5.01	5.4	8.2	-	39.51	33.81	-	.0951	.042	.082
71.	PEG-2000	60-150	-	1.0813 *7.41*	1.0331 *7.78*	-	6.92	6.14	3.4	6.0	-	39.41	33.72	-	.0947	.092	.160
72.	PEG-20000	80-150	-	1.0817 *7.37*	1.0330 *7.68*	-	6.96	5.17	3.6	5.5	-	39.35	33.87	-	.0914	.065	.115
73.	PEG-M-600	20-150	1.1071 *7.09*	1.0599 *7.41*	1.0128 *7.74*	-	6.98	5.43	2.5	4.8	42.23	36.67	31.10	-	.0928	.104	.185
74.	PEG-M-1000	60-150	-	1.0608 *7.39*	1.0138 *7.73*	-	6.93	5.70	2.7	5.6	-	37.66	32.20	-	.0910	.100	.174
75.	PEG-M-2000	60-150	-	1.0634 *7.33*	1.0169 *7.58*	-	7.00	4.12	3.7	6.9	-	38.39	33.07	-	.0888	.040	.070
76.	PEG-M-20000	70-150	-	1.0646 *7.23*	1.0183 *7.58*	-	6.76	5.88	3.3	6.8	-	38.73	33.51	-	.0870	.087	.151

capillaries of known diameter. The observed deviation from the value recommended by Jasper (*1*) was always less than 0.03 dyn cm^{-1}. This sample was used as a secondary standard.

The capillary rise is given by eq 6, where h_L is the height of

$$h_L = 2\gamma_L \cos \theta_{L/S}/gr_jd_L \tag{6}$$

the capillary rise (cm) of the Lth liquid, g is the acceleration due to gravity (cm s^{-2}), γ_L is the surface tension of the liquid (dyn cm^{-1}), $\theta_{L/S}$ is the contact angle of the liquid on the solid, r_j is the radius of the jth capillary (cm), and d_L is the density of the liquid (g cm^{-3}). For liquids with $\theta_{L/S} = 0°$, eq 7 simplifies to

$$h_{L,j} = 2\gamma_L/gr_jd_L = \gamma_L/k_jd_L \tag{7}$$

where $k_j = gr_j/2$ is the constant for the jth capillary.

With the aid of the standard n-heptane ($\theta = 0$), a capillary constant was determined for each capillary before the measurement:

$$k_j = \gamma_{hep}/h_{hep,j}d_{hep} \tag{8}$$

It is important to note that, for the determination of the surface tension of nonvolatile substances with surface tension higher than ~30 dyn cm^{-1}, new capillaries had to be used for each measurement and that in such cases the calibration had to be made after the measurement.

The variance of the capillary constant k_j is

$$V(k_j) = \left(\frac{1}{h_{hep}d_{hep}}\right)^2 V(\gamma_{hep}) + \left(\frac{\gamma_{hep}}{d_{hep}h_{hep}^2}\right)^2 V(h_{hep}) + \left(\frac{\gamma}{d_{hep}^2h_{hep}}\right)^2 V(d_{hep}) \tag{9}$$

By substituting approximate values ($h_{hep} \approx 4.4$. cm; $d_{hep} \approx 0.67$ g cm^{-3}; $\gamma_{hep} \approx 19.2$ erg cm^{-2}; $V(\gamma_{hep}) \approx 9 \times 10^{-4}$ (erg cm^{-2})^2; $V(h_{hep}) \approx 9 \times 10^{-6}$ cm^2; $V(d_{hep}) \approx 10^{-8}$ (g cm^{-3})^2), we get

$$V(k_j) = (1.03 + 0.20 + 0.01) \times 10^{-4} = 1.24 \times 10^{-4}\ (\text{cm}^2\ \text{s}^{-2})^2$$

for the confidence limit

$$\Delta_{95}(k_j) = \pm 0.022\ \text{cm}^2\ \text{s}^{-2}$$

$$\Delta_{95,rel}(k_j) = \pm 0.30\%$$

The surface tensions of liquids with $\theta = 0°$ can now be determined if their densities are known as

$$\gamma_{\mathrm{L}} = d_{\mathrm{L}} h_{\mathrm{L},j} k_j \tag{10}$$

Surface tensions were measured in general at equidistant temperature intervals (every 10 °C). On the experimental points a linear equation was fitted:

$$\gamma_{\mathrm{L}} = \gamma_{\mathrm{L},T^\dagger} + b(T - T^\dagger) \tag{11}$$

$T^\dagger$ is the average of the lowest and highest temperatures of the experimental range. Deviation from linearity was not observed, even in cases when the surface tension was determined up to 200 °C.

The variance of a single determination can be estimated with eq 12 to be $V(\gamma_{\mathrm{L}}) \approx 0.0025$ (dyn cm^{-1})^2 giving for the confidence limit of a single determination $\Delta_{95}(\gamma_{\mathrm{L}}) = 0.12$ dyn cm^{-1}. The confidence limit of the regression equation (eq 11) is a quadratic function with maximum values at the limits of the experimental temperature range.

$$V(\gamma_{\mathrm{L}}) = (d_{\mathrm{L}}k_j)^2 V(h_{\mathrm{L}}) + (h_{\mathrm{L}}k_j)^2 V(d_{\mathrm{L}}) + (d_{\mathrm{L}}h_{\mathrm{L}})^2 V(k_j) \tag{12}$$

3. Results

3.1. Table II. In the second column the experimental temperature range is given. It is the same for the density determinations as well as for those of the surface tension in almost every case.

Data concerning density are listed in the following order: density at 20 and 80 °C, mean coefficient of thermal expansion in the experimental temperature range, minimum and maximum of the confidence limit of the regression (95% confidence level) for interpolated data, and finally data from the literature. The confidence limit is calculated from the scatter around the regression; it includes also the error from calibration. Literature data cited are those which most closely confirm our results. Under the heading "further literature" two kinds of references are given: the first kind also confirms our data; the second kind, marked by an asterisk, describes results of careful determinations on (presumably) impure samples.

Data concerning surface tension are listed in the following order: surface tension at 20 and 80 °C, temperature dependence of the surface tension (coefficient *b* in eq 11), and literature data. Our data are always confronted with the "recommanded values" of Jasper (*1*) together with the original work that Jasper refers to; further references are given, as for the density.

3.2. Table III. The densities and surface tensions of the high molecular weight hydrocarbons of the series **A1** and **A2** (**15–24**) and those of the poly(ethylene glycols) of the series PEG and PEG-M (**69–76**) were measured in a wider temperature range. The coefficient of thermal expansion is given as a linear function of the temperature (eq 4 and 5). In the second column of the table the densities and the coefficients of thermal expansion are given at 20, 80, 140, and 200 °C. In the third column the coefficients of thermal expansion at 0 °C and their temperature dependence, α, are given. Otherwise, the organization of the table is similar to that of Table II.

3.3. Remarks (Tables II and III). The surface tensions of the following substances have shown significant differences from those recommended by Jasper (*1*): dodecane (**3**), *cis*-decalin (**28**), *trans*-decalin (**29**), 1-chlorohexadecane (**33**), chlorobenzene (**51**), iodobenzene (**53**), methylene iodide (**63**), 1,4-dioxane (**78**), nitrobenzene (**54**), anisol (**55**), 1-chloronaphthalene (**58**), cyclohexanone (**81**), dimethyl adipate (**82**), and dimethyl sulfoxide (**83**). All deviating data have been controlled, and we believe that our results are superior. The big difference observed in the case of methylene iodide (**63**) is probably due to the different method of determination (maximum bubble pressure (*1*, *22*)). (a) The densities of these compounds were not measured by us. For the determination of the surface tension, densities from the references indicated were used. The coefficient of the thermal expansion is not given because, even in the narrow temperature range, it is a function of the temperature. (b) The confidence limit of the density in this temperature range is a rough estimate. For exact data, see Table III. (c) Density at 20 °C: extrapolated value for a hypothetical undercooled liquid. (d) The surface tensions are significantly different from those proposed by Jasper (*1*) in the entire temperature domain. (e) The surface tension deviates in a part of the temperature range from the values proposed by Jasper (*1*) (the coefficient *b* is significantly different). (f) Above 40 °C the *N*,*N*-dimethylaniline and the diiodomethane decomposed during the measurements.

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Some Methyl 2,5- and 5,6-Dihalonicotinates

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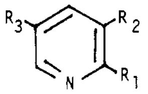
The preparation of the methyl esters of eight dihalonicotinic acids is described. The esters were synthesized either by the methanolysis of their respective acid chlorides or by treatment of the appropriate acid with diazomethane in ether. Experimental and spectral data for the methyl dihalonicotinates are presented.

We have previously reported the synthesis of a series of 2,5- and 5,6-dihalonicotinic acids of potential medicinal interest (*1–5*). As an extension of that work, we now wish to report the preparation and characterization of the methyl esters (I–VIII) of eight of the aforementioned acids.



	R_1	R_2	R_3
I	Cl	Br	CO_2CH_3
II	Cl	F	CO_2CH_3
III	Cl	CO_2CH_3	Br
IV	Cl	CO_2CH_3	Cl
V	Br	Cl	CO_2CH_3
VI	Br	Br	CO_2CH_3
VII	Br	CO_2CH_3	Br
VIII	Cl	CO_2CH_3	I

We found that two standard esterification procedures could be employed. Conversion of the acid to the acid chloride followed by rapid treatment of the latter with methanol proved to be a successful procedure in those instances attempted. Alternatively, direct methylation of the acid with diazomethane in ether was employed in some cases. As indicated in Table I several of the esters were prepared by both methods.

Elemental analyses (C, H, N) for the methyl dihalonicotinates in agreement with theoretical values were obtained and submitted for review as supplementary material. (See paragraph at end of text regarding supplementary material.) Experimental and physical data for the esters reported herein are presented in Table I.

Experimental Section

Elemental analyses were performed by Galbraith Laboratories, Knoxville, TN. Melting points were taken on a Mel-Temp apparatus and are uncorrected. Infrared spectra were obtained on a Perkin-Elmer 337 spectrophotometer with samples prepared as KBr disks. Proton nuclear magnetic resonance spectra were obtained in deuteriochloroform on a Jeolco C-60 HL instrument with tetramethylsilane as internal standard.

Acid Chloride Method. Typical Procedure. A mixture of 5-bromo-6-chloronicotinic acid (*1*) (1.2 g, 0.005 mol) and thionyl chloride (5 mL) was stirred magnetically under gentle reflux for 1 h. The excess thionyl chloride was then removed under reduced pressure (rotary evaporator) leaving the crude acid chloride as a heavy yellow oil. The oil was dissolved immediately in dry benzene (10 mL), and anhydrous methanol (5 mL) was added. The resulting solution was heated under gentle reflux for 1 h. Evaporation of the volatile solvents afforded the

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