NOMENCLATURE

A_n = fitting parameter in excess properties equation
H = enthalpy of forming new surface, ergs per sq. cm.
n = fitting parameter in excess properties equation
R = gas constant
S = entropy of forming new surface, ergs per deg. per sq. cm.
t = temperature, ° C.
T = temperature, ° K.
X_i = mole fraction of component i
d = density, g. per cc.
Γ_i = individual Gibbs surface excess of component i, moles per sq. cm.
Γ_2^N = composite Gibbs surface excess, moles per sq. cm.
Σ = summation sign
σ = surface tension, dynes per cm. or free energy of forming new surface, ergs per sq. cm.

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Physical Properties of Some Sulfur and Nitrogen Compounds

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Physical properties were determined on purified samples of 38 organic sulfur compounds and 18 organic nitrogen compounds. Most of the compounds were donated by interested laboratories for augmenting the spectral data on these types of compounds. Special handling techniques to prevent contamination or decomposition were employed during purification, storage, and property measurement. Boiling point, freezing point, density at 20° C., and refractive index (sodium *D* and mercury *g* lines) at 20° C. were determined. Derived functions calculated are refractivity intercept, specific dispersion, and molecular refraction.

TO SUPPLY fundamental data on the sulfur and nitrogen compounds that occur in petroleum, the Bureau of Mines prepared standard samples of organic sulfur and nitrogen compounds as reference materials and carried out intensive studies of their properties (*4, 5, 6, 8*).

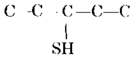
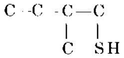
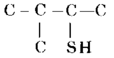
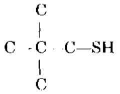
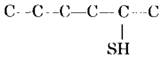
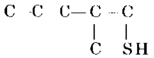
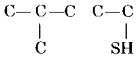
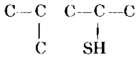
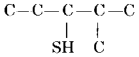
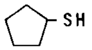
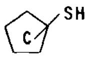
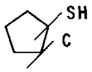
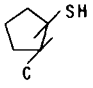
An important adjunct of this work was a program to speed the acquisition and publication of spectral data on additional compounds of these types. A bank of over 350 sulfur and nitrogen compounds of reliable purity, ranging in amounts from a few milligrams to several grams, was accumulated. Most of the compounds in this phase of the work were donated, although a few were purchased. Spectral data on these compounds were determined by cooperating oil company laboratories and the Bureau of Mines and were published in the Catalogs of Selected Spectral Data edited by American Petroleum Institute Research Project 44 (*1*).

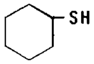
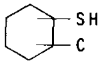
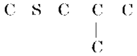
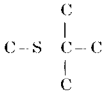
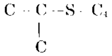
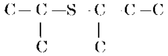
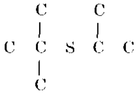
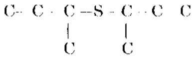
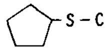
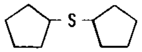
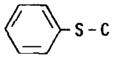
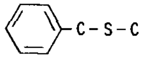
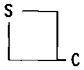
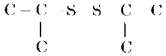
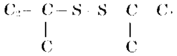
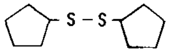
This paper reports the values for the physical properties of 56 of these small-scale samples. Included in the tables are the catalog serial numbers for the mass, ultraviolet, infrared, and Raman spectra obtained on these samples.

EXPERIMENTAL

Purification. Purification procedures were similar to those reported previously by this laboratory (*4, 5, 6, 8*) on organic sulfur and nitrogen compounds. Three distillation columns were used: a column 30 cm. in length and 1.1 cm. in diameter packed with stainless steel helices, a spinning band column 30 cm. in length and 1.2 cm. in diameter, and a concentric tube column approximately 100 cm. in length and 1.0 cm. in diameter. Each column was vacuum-jacketed and equipped with automatic take-off and reflux ratio control.

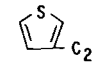
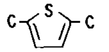
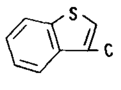
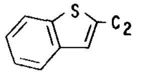
Table I. Properties of Organic Sulfur Compounds

Compound		Freezing Point, ° C.	Purity, Mole %	Boiling Point, ° C., 760 Mm.	Density G./Ml. d_4^{20}	Refractive Index		Derived Functions			Spectra Catalog Serial Numbers					
						n_D^{20}	n_F^{20}	Refractivity intercept, $n_D^{20} - d_4^{20}/2$	Specific dispersion, $(n_F^{20} - n_D^{20})10^4/d_4^{20}$	Molecular refraction, $M(n^2 - 1)/d(n^2 + 2)$	UV	IR 2–5μ	IR 2–15μ	IR 15–25μ	Mass	Raman
Thiols																
3-Pentanethiol[a]	 $C-C-\underset{SH}{C}-C-C$		b	113.9	0.8410	1.4447	1.4566	1.0242	141.5	32.961	530	1527	1526	1528	921	
2-Methyl-1-butanethiol[a]	 $C-C-\underset{C}{C}-\underset{SH}{C}$		b	119.1	0.8467	1.4477	1.4598	1.0243	142.9	32.985	770		2029		1413	
3-Methyl-2-butanethiol[a]	 $C-\underset{C}{C}-\underset{SH}{C}-C$		b	109.8	0.8408	1.4446	1.4566	1.0242	142.7	32.969	554		1548	1549	943	219
2,2-Dimethyl-1-propanethiol[a]	 $C-\overset{C}{\underset{C}{C}}-C-SH$		b	103.7	0.8298	1.4393	1.4512	1.0244	143.4	33.029	527	1519	1518		918	
2-Hexanethiol[a]	 $C-C-C-C-\underset{SH}{C}-C$		b	139.1	0.8330	1.4448	1.4565	1.0283	140.5	37.757	726		1942			
2-Methyl-1-pentanethiol[a]	 $C-C-C-\underset{C}{C}-\underset{SH}{C}$		b	143.9	0.8455	1.4501	1.4620	1.0273	140.5	37.618	641		1771		1242	
4-Methyl-1-pentanethiol[a]	 $C-\underset{C}{C}-C-C-\underset{SH}{C}$		b	144.9	0.8383	1.4477	1.4596	1.0285	142.0	37.800	607		1775		1235	
4-Methyl-2-pentanethiol[a]	 $C-\underset{C}{C}-C-\underset{SH}{C}-C$		b	c	c	1.4409	1.4527				642		1772		1243	
2-Methyl-3-pentanethiol[a]	 $C-C-\underset{SH}{C}-\underset{C}{C}-C$		b	135.6	0.8480	1.4499	1.4617	1.0259	139.2	37.507	643		1773		1244	
1-Heptanethiol[d]	C_7—SH		b	110.4[d]	0.8429	1.4519	1.4638	1.0304	141.2	42.367	451		1425	1260	726	
Cyclopentanethiol[a]			b	132.2	0.9553	1.4904	1.5040	1.0627	142.4	30.917	528	1521	1520	1522	919	
1-Methylcyclopentanethiol[a]			b	137.6	0.9269	1.4800	1.4932	1.0165	142.4	35.610	608		1776		1236	
cis-2-Methylcyclopentanethiol[a]			b	86.9[d]	0.9474	1.4892	1.5023	1.0155	138.3	35.453	664		1849		1371	
trans-2-Methylcyclopentanethiol[a]			b	81.1[d]	0.9236	1.4799	1.4932	1.0181	144.0	30.578					1414	

Cyclohexanethiol[a]			[b]	158.8	0.9485	1.4936	1.5072	1.0193	143.4	35.674	555		1550	1551	944	
cis-2-Methylcyclohexanethiol[c]			[b]	109.2[d]	0.9498	1.4945	1.5076	1.0196	137.9	39.905	665		1850		1372	
Sulfides																
2-Thiahexane[e]	C—S—C C C C	−97.9	99.8	123.3	0.8424	1.4478	1.4601	1.0266	146.0	33.154	449		1423	1258	724	207
4-Methyl-2-thiapentane[a]	 $\mathrm{C\ \ S\ \ C\ \ \underset{C}{C}\ \ C}$	−109.1	98.2	113.1	0.8349	1.4437	1.4559	1.0262	146.1	33.077	546	1541	1540	1541	945	
3,3-Dimethyl-2-thiabutane[a]	 $\mathrm{C{-}S\ \ \overset{C}{\underset{C}{C}}{-}C}$	−82.3	99.98	98.9	0.8255	1.4403	1.4526	1.0275	149.0	37.813	529	1524	1523	1525	920	
2-Thiaoctane[c]	C—S—C_6	−66.8	99.1	107.2[d]	0.8435	1.4529	1.4649	1.0311	142.3	42.337	545		1467		925	
2-Methyl-3-thiaheptane[f]	 $\mathrm{C{-}\underset{C}{C}{-}S{-}C_4}$	−92.4	99.0	92.1[d]	0.8291	1.4470	1.4589	1.0324	143.5	42.593	730		1946			
2,4-Dimethyl-3-thiahexane[f]	 $\mathrm{C{-}\underset{C}{C}{-}S{-}\underset{C}{C}{-}C{-}C}$		[g]	144.6	0.8253	1.4453	1.4572	1.0326	144.2	42.630	687		1911		1409	
2,2,4-Trimethyl-3-thiapentane[f]	 $\mathrm{C\ \ \overset{C}{\underset{C}{C}}\ \ S\ \ \overset{C}{C}\ \ C}$	−70.3	99.0	132.7	0.8132	1.4404	1.4525	1.0338	148.8	42.939	688		1912		1410	
2-Thiadodecane[e]	C S C_7	−63.4	99.7	127.7[d]	0.8437	1.4548	1.4666	1.0329	139.9	46.989	542		1426	1261	727	210
3,5-Dimethyl-4-thiaheptane[f]	 $\mathrm{C{-}C{-}\underset{C}{C}{-}S{-}\underset{C}{C}{-}C{-}C}$		[g]	101.2[d]	0.8336	1.4504	1.4622	1.0336	141.6	47.207	663		1851		1373	
2-Thiadodecane[e]	C S C_{10}	−18.5	99.8	180.5[d]	0.8451	1.4592	1.4707	1.0366	136.1	59.235	531	1530	1529	1531		
1-Cyclopentyl-1-thiaethane[a]		−103.3	99.8	155.9	0.9419	1.4892	1.5026	1.0182	142.3	35.660	547	1543	1542	1543	946	
Dicyclopentylthiamethane[a]			99.8	169.0[d]	0.9752	1.5099	1.5233	1.0223	137.4	52.218	504		1499		860	
1-Phenyl-1-thiaethane[a]		−16.7	99.9	125.9[d]	1.0579	1.5873	1.6187	1.0583	296.8	39.447	561		1636	1883	969	
1-Phenyl-2-thiapropane[a]		−28.3	99.7	141.6[d]	1.0274	1.5634	1.5880	1.0497	239.4	43.726	562		1637	1884	970	
Cyclic sulfides																
3-Methylthiacyclobutane[a]		−83.5	99.0	51.9[d]	0.9588	1.4873	1.5020	1.0079	153.3	26.484	769		2028		1412	
Disulfides																
2,5-Dimethyl-3,4-dithiahexane[i]	 $\mathrm{C{-}\underset{C}{C}{-}S\ \ S\ \ \underset{C}{C}\ \ C}$	−70.5	99.8	109.5[d]	0.9427	1.4918	1.5074	1.0204	165.5	46.238	689		1913		1411	
3,6-Dimethyl-4,5-dithiaoctane[i]	 $\mathrm{C_2{-}\underset{C}{C}{-}S\ \ S\ \ \underset{C}{C}\ \ C_2}$		[g]	145.0[d]	0.9363	1.4928	1.5076	1.0246	158.1	55.433	727		1943		1502	
1,2-Dicyclopentyl-1,2-dithiaethane[i]		−19.1	99.7	205.9[d]	1.0655	1.5479	1.5642	1.0151	153.0	60.399	728		1944			

(*Continued on page 96*)

Table I. Properties of Organic Sulfur Compounds (*Continued*)

Compound		Freezing Point, ° C.	Purity, Mole %	Boiling Point, ° C., 760 Mm.	Density G./Ml. d_4^{20}	Refractive Index n_D^{20}	Refractive Index n_g^{20}	Derived Functions: Refractivity intercept, $n_D^{20} - d_4^{20}/2$	Derived Functions: Specific despersion, $(n_g^{20} - n_D^{20})\,10^4/d_4^{20}$	Derived Functions: Molecular refraction, $M(n^2-1)/d(n^2+2)$	Spectra Catalog Serial Numbers: UV	IR 2–5μ	IR 2–15μ	IR 15–25μ	Mass	Raman
Thiophenes																
3-Ethylthiophene[f]		−89.1	99.9	140.9	0.9980	1.5146	1.5346	1.0156	200.4	33.837	503		1498		859	
2,5-Dimethylthiophene[e]		−62.6	99.9	136.8	0.9852	1.5130	1.5340	1.0204	213.2	34.276	544		1466		924	
3-Metbyl-1-thiaindene[a]		−39.8	[g]	168.2[d]	1.1292	[c]	[c]				666		1853		1027	
2-Ethyl-1-thiaindene[a]		9.2	99.9	180.4[d]	1.0883	1.6083	1.6437	1.0641	325.3	51.582						

[a] Original sample donated by Northwestern University, Evanston, Ill. [b] Purity certified 99.0 mole % or better by donor. [c] Insufficient sample for this measurement. [d] Boiling point at 100 mm. [e] Original sample purchased from Columbia Organic Chemicals, Columbia, S. C. [f] Original sample donated by Bureau of Mines, Bartlesville, Okla. [g] Purity estimated 99.0 mole % or better (8). [h] Original sample purchased from Eastman Organic Chemicals, Rochester, N. Y. Original sample donated by Bureau of Mines, Laramie, Wyo. [k] Original sample donated by Socony-Vacuum Oil Co., Paulsboro, N. J.

Special procedures were used to increase the purity and to protect the purity of the samples during storage. To minimize decomposition, the compounds were distilled under an atmosphere of dry nitrogen containing less than 2 p.p.m. of oxygen (*7*). The nitrogen compounds after distillation were treated with calcium hydride to remove traces of water. Silica gel treatment was used on some of the thiols to remove polar materials, including water. All compounds were degassed and sealed in borosilicate glass ampoules either under their own vapor pressure or under an atmosphere of dry, deoxygenated nitrogen.

Property Determinations. The properties determined and calculated are shown in Tables I and II; Table I gives the properties of the 38 sulfur compounds, and Table II lists the properties of the 18 nitrogen compounds. During property measurements, the compounds were protected by an atmosphere of dry, deoxygenated nitrogen.

Freezing points were determined using a platinum resistance thermometer and a Mueller bridge (Leeds and Northrup, Type G-2), as described by Glasgow *et al.* (*3*). Less than optimum amounts of sample were available for most of the determinations, reducing the precision to an estimated ±0.02° C. The values in the table have been rounded to the nearest 0.1° C.

Purities were calculated from the freezing point curves according to the method of Glasgow *et al.* (*3*). For compounds where freezing point data were lacking in precision, estimates of purity were calculated, using a combination of spectral techniques described by Davis and Morris (*2*). The purity values reported in the table are calculated to within ±0.1 mole % of the true value.

Boiling point measurements were made on a Cottrell ebulliometer, using a platinum resistance thermometer and precision Mueller bridge. Pressure was maintained at 760 torr by means of a Cartesian manostat, using dry, deoxygenated nitrogen for pressurization. Measurements for high-boiling compounds, especially those subject to decomposition, were made at 100 torr. The platinum resistance thermometer was calibrated and certified by the National Bureau of Standards. The manostat was calibrated from the boiling point of triple-distilled water at 760 and 100 torr; the estimated variability was ±0.05 torr.

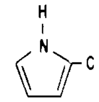
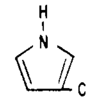
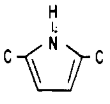
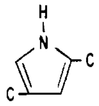
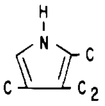
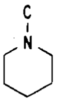
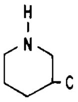
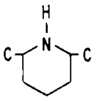
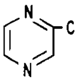
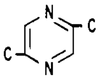
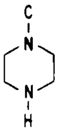
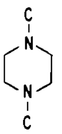
Density was obtained at 20.00° C. with a Christian Becker Chainomatic five-place density balance, using a 5-gram plummet. The balance was calibrated with a set of standard weights and with hydrocarbon standards certified by the National Bureau of Standards. The measurements were made in a vacuum-jacketed bath, through which water thermostated to a maximum variation of 0.005° C. was circulated. Temperature was measured near the plummet by means of a calibrated precision thermometer.

Refractive indices were measured with a Bausch and Lomb Precision oil refractometer, Abbe type, enclosed in a special housing. Temperature within the housing was controlled by circulating nitrogen through a water-cooled radiator within the housing. Temperature in the prisms was maintained at 20.00° C. by circulating water thermostated to a maximum variation of 0.005° C.

Derived functions were calculated from the properties determined (Tables I and II). Specific dispersion was calculated from the sodium *D* and mercury *g* lines according to Thorne *et al.* (*9*). Refractivity intercept and molecular refraction were calculated using methods discussed by Weissberger (*10*).

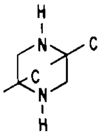
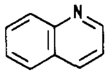
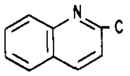
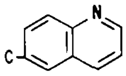
The spectra obtained on the samples are noted in the tables according to the serial numbers assigned in the catalogs (*1*). The spectra were obtained by laboratories cooperating with API Research Projects 48 and 52 to speed the publication of the spectral data of these samples. The laboratories cooperating to obtain the spectra are identified in the catalogs.

Table II. Properties of Organic Nitrogen Compounds

Compound		Freezing Point, °C.	Purity, Mole %	Boiling Point, °C., 760 Mm.	Density G./Ml., d_4^{20}	Refractive Index		Derived Functions			Spectra Catalog Serial Numbers				
						n_D^{20}	n_F^{20}	Refractivity intercept $n_D^{20} - d_4^{20}/2$	Specific dispersion, $(n_F^{20} - n_D^{20})10^4/d_4^{20}$	Molecular refraction, $M(n^2-1)/d(n^2+2)$	UV	IR 2–15μ	IR 14–25μ	Mass	Raman
2-Methylpyrrole[a]		−35.6	[b]	[c]	[c]	1.5035	1.5229				729	1945		1536	
3-Methylpyrrole[a]		−48.4	[b]	[c]	[c]	1.4986	1.5176					2474	2475		
2,5-Dimethylpyrrole[a]			99.8	108.3[d]	0.9361	1.5066	1.5257	1.0385	204.04	30.22			2594		
2,4-Dimethylpyrrole[e]			[b]	165.3	0.9194	1.4965	1.5149	1.0368	200.13	30.26	633	1761	1762[f]	1347	247
1-(1-Butyl)pyrrole[g]		−66.1	99.5	106.7[d]	0.8747	1.4741	1.4901	1.0367	178.83	39.59		2478	2479		
2,4-Dimethyl-3-ethylpyrrole[e]		13.6	[b]	197.3	0.9115	1.4974	1.5147	1.0416	189.80	39.57	634	1763	1764[f]	1349	248
1-Methylpyrrolidine[a]			[b]	79.3	0.7992	1.4236	1.4351	1.0240	143.89	27.17		2731			
1-Methylpiperidine[h]			[b]	105.7	0.8157	1.4372	1.4489	1.0298	137.31	31.87					
3-Methylpiperidine[i]		−27.2	99.7	64.9[d]	0.8433	1.4472	1.4583	1.0295	131.63	31.43	644	1744		1245	
2.6-Dimethylpiperidine[i]		−63.6	99.9	127.3	0.8201	1.4395	1.4503	1.0294	131.69	36.34	668	1871		1348	
2-Methylpyrazine[j]		−28.8	99.7	135.7	1.0246	1.5053	1.5297	0.9930	238.14	27.25	691	1929	1930	1417	
2,5-Dimethylpyrazine[j]		−15.5	[b]	89.1[d]	0.9884	1.5007	1.5247	1.0065	242.82	32.22		2476	2477		
1-Methylpiperazine[k]		−5.33	[b]	137.5	0.9021	1.4658	1.4793	1.0147	149.65	30.74		2596			
1.4-Dimethylpiperazine[h]		−1.0	[b]	131.0	0.8509	1.4471	1.4602	1.0216	153.95	35.86		2597			

(*Continued on page 98*)

Table II. Properties of Organic Nitrogen Compounds (*Continued*)

Compound		Freezing Point, °C.	Purity, Mole %	Boiling Point, °C., 760 Mm.	Density G./Ml., d_4^{20}	Refractive Index		Derived Functions			Spectra Catalog Serial Numers				
						n_D^{20}	n_g^{20}	Refractivity intercept $n_D^{20} - d_4^{20}/2$	Specific dispersion, $(n_g^{20} - n_D^{20})10^4/d_4^{20}$	Molecular refraction, $M(n^2-1)/d(n^2+2)$	UV	IR 2–15μ	IR 14–25μ	Mass	Raman
cis-2,5-Dimethylpiperazine[j]		18.9	[b]	101.1[d]	0.9218	1.4731	1.4853	1.0122	132.35	34.76	772	2031	2032	1559	
Quinoline[e]		−14.9	[b]	161.9[d]	1.0941							2598	2599		
2-Methylquinoline[i]			[b]	171.5[d]	1.0636										
6-Methylquinoline[i]		−26.0	[b]	181.4[d]	1.0664	1.6165	1.6577	1.0833	386.35	46.95	773	2033	2034	1560	

[a] Original sample donated by University of Kansas, Lawrence, Kan. [b] Purity estimated 99.0 mole % or better (*8*). [c] Insufficient sample for this measurement. [d] Boiling point at 100 mm. [e] Original sample donated by Mellon Institute of Research, Pittsburgh, Pa. [f] 14—40μ. [g] Original sample donated by Bureau of Mines, Laramie, Wyo. [h] Original sample purchased from Aldrich Chemical Co., Milwaukee, Wis. [i] Original sample purchased from Du Pont Organic Chemicals, Wilmington, Del. [j] Original sample purchased from Wyandotte Chemical Corp., Wyandotte, Mich. [k] Original sample purchased from Union Carbide Corp., New York, N. Y. [l] Original sample purchased from Matheson Co., Inc., East Rutherford, N. Y.

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Vapor Pressure of Primary *n*-Alkyl Chlorides and Alcohols

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The vapor pressures of 1-alkyl chlorides and of 1-*n*-alkanols were determined by differential thermal analysis between 5 and 760 Torr. The experimental data were correlated by the Antoine vapor pressure equation, and the Antoine constants for the compounds are presented.

DIFFERENTIAL thermal analysis provides a relatively new technique for determining the vapor pressure of compounds over a wide range of temperature and pressure (*8*). The dynamic nature of the technique avoids problems of decomposition and decontamination of the compounds under test and the method can provide data of excellent accuracy.

Experimental determinations of vapor pressure of the primary alcohols and the corresponding alkyl chlorides have been reported in many places, but the data available are neither complete nor entirely reliable. For example, Winslow (*14*) shows that fair amounts of data are available for the first eight members of the series, especially for the even numbered homologs, but above this point the data are sparse and of unknown reliability. Li and Rossini (*10*) have shown that most of the reliable data for the alkyl chlorides apply to the first four homologs, but even within these data there are many discrepancies. For instance,

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