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by Richard E. Johnson

Lewis Research Center

Cleveland, Ohio

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SUMMARY

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A screening investigation was conducted to determine the potentialities of organic aliphatic and aromatic sulfur derivatives as solvents for high-energy-density batteries. Side tests included the testing of various other organic compounds to obtain data for comparison with the sulfur derivatives. A literature survey was also made of phosphorus and arsenic organic compounds, but only one experiment was performed because of the high chemical reactivity of these chemicals.

Propylene glycol sulfite had the best electrochemical properties. All the promising sulfur derivatives contained the -SO or the $-SO_2$ radical.

The investigations were limited to those solvents which have a liquidus range similar to that of water (0° to 100° C) and which are readily available from commercial suppliers.

Author

INTRODUCTION

The realization of energy density levels beyond those attainable with state-of-the-art chemical batteries depends principally upon achieving compatibility of an electrolyte possessing desirable electrochemical properties and highly active anode materials, such as lithium or calcium. Consideration of aqueous electrolytes is precluded because of the decomposition of water in the presence of these active materials, and the search has, therefore, been directed to the nonaqueous organic and inorganic solvents. A survey of electrolyte studies indicates that most investigators (e.g., refs. 1 to 3) have concentrated on the aliphatic and aromatic nitrogen and oxygen derivatives with scant attention given to the organic sulfur, phosphorus, and arsenic derivatives. An evaluation of the published properties of these latter derivatives indicates that the sulfur compounds present the most promising possibilities.

The actual screening of the organic aliphatic and aromatic sulfur derivatives was conducted on the bases of the (1) conductivity of the solvents with various solutes and (2) the reactivity with lithium; these properties were measured for the materials obtainable.

The dielectric constant of the solvents was also measured.

SYMBOLS

C_e electrode capacitance (stray), $\mu\mu F$

C_g capacitance to ground, $\mu\mu F$

C_k capacitance of known standard liquid, $\mu\mu F$

C_p capacitance of unknown liquid corrected for losses, $\mu\mu F$

C_v capacitance in air of cell in vacuum, $\mu\mu F$

$C_{v,c}$ capacitance of empty cell corrected for losses, $\mu\mu F$

C_x capacitance of unknown liquid, $\mu\mu F$

K dielectric constant of unknown liquid

K_k dielectric constant of known standard liquid

APPARATUS AND PROCEDURE

All solvent evaluations utilized a tubular glass cell, 1/2 inch in diameter by 3 inches long with a set of platinum electrodes (cleaned in aqua regia, a mixture of hydrochloric and nitric acids), each with an area of 1 square centimeter on each face. The capacitance was first determined with air in the cell to obtain C_v in equation (1). An organic liquid with a known dielectric constant was removed from its bottle with a hypodermic needle and syringe and placed in the tubular cell to avoid contact with air and possible moisture pickup. The capacitance (C_k in eq. (1)) was determined followed by a conductance measurement for the reason that unless the conductivity is less than 5×10^{-6} ohm^{-1} centimeter^{-1} the capacitance readings are in error because of losses associated with the electrical conductivity. Capacitance and conductance measurements were made with a bridge with the use of a signal generator and detector. Although the generator can produce a wide range of frequencies at various voltages that can be used by the bridge in a balancing circuit, all measurements were made at 1000 cps.

The sample of organic liquid to be tested was placed in the cell in the same manner as the aforementioned liquid and its capacitance (C_x in eq. (2)) and conductance were measured. A known amount of potassium hexafluorophosphate (KPF_6) or potassium perchlorate ($KClO_4$) was added to bring the solution to 0.01 molar concentration, and the conductivity measurement was repeated. The solution was then saturated with the same

salt, and the conductivity was again measured (table I). Finally, a piece of lithium (cleaned by filing to bright metal) was placed in the cell for reactivity evaluation.

The dielectric constant was calculated from equations (1), (2), and (4) (given in ref. 4) and equation (3) (derived experimentally):

$$C_e + C_g = \frac{K_k(C_v - C_k)}{K_k - 1} \tag{1}$$

$$C_p = C_x - (C_e + C_g) \tag{2}$$

$$C_{v,c} = C_v - (C_e + C_g) \tag{3}$$

$$K = \frac{C_p}{C_{v,c}} \tag{4}$$

Reference 4 states that

> When liquids are measured in a two-terminal measuring cell, the extra capacitances between electrodes and to ground cannot be directly measured or calculated. The value of their combined capacitances, $C_e + C_g$, can be found by measuring the cell both when filled to the same level with a liquid of known dielectric constant, and when empty, and calculating [in equation (1).] ... these extra capacitances are usually free from loss. ...

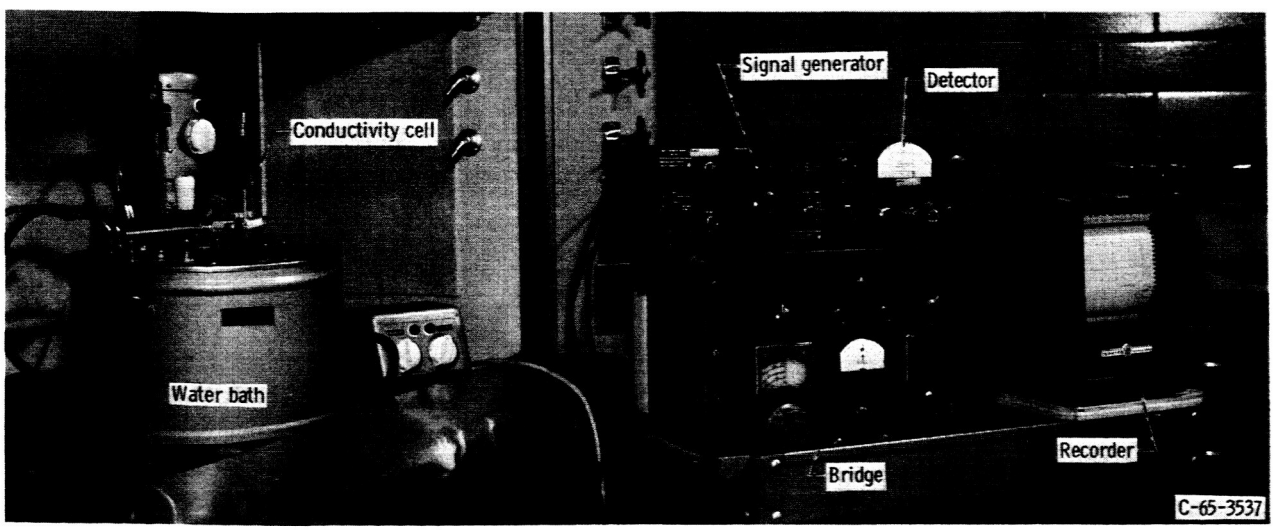

Figure 1. - Organic electrolyte evaluation apparatus.

The calculated results are presented in tables I and II. The accuracy of the capacitances and conductivity measurements was ±2.5 percent with reference to three standard solutions which meet American Chemical Society chemical standards. The experimental setup used is shown in figure 1.

RESULTS AND DISCUSSION

Prior to discussion of the experimental results obtained from the investigation, it may be helpful to review interpretive guidelines for conductivity and dielectric constant values, with a consideration of other characteristics such as viscosity and dipole moment, the values of which were obtained from the literature and are shown in table III. First, although no particular drain rates have been established, it is unlikely that any cell will be developed in the 200 watt-hour per pound class that has an electrolyte conductivity of less than 1×10^{-3} ohm^{-1} centimeter^{-1}; a common dry cell of the Leclanche type has an electrolyte conductivity of 1×10^{-1} ohm^{-1} centimeter^{-1}. The value of 1×10^{-3} ohm^{-1} centimeter^{-1} was thus taken as the threshold of acceptability for electrical conductivity. A minimum value of 20 was arbitrarily selected as the dielectric constant interest level cutoff; the major consideration, however, was the magnitude of the conductivity. The viscosity should be low, about 0.1 to 1 centipoise (water is 0.1 cP).

Dipole moment values vary from 0 to 5 or higher and should be at least as high as that for water (~1.8). Both dielectric constant and dipole moments are a measure of the ability of the solvent to dissolve ionic salts and possibly to yield high conductivity.

Some sulfur organic derivatives are listed in table III(a). These data, as well as those in table III(b), are taken mainly from references 5 and 6. From these data, the most promising compounds were selected for tests as solvents. It is evident that there is a relatively small amount of data for selection of promising compounds which might be good solvents (such as dielectric constant, dipole moments, etc.); a knowledge of chemical and physical properties of these solvents is helpful in selecting the most likely test compounds.

The listing of potential phosphorus and arsenic compounds is given in table III(b). The literature survey indicated that the compounds either are too reactive or have a low dielectric constant. One of the more promising compounds was tested, but the results were discouraging.

The organic sulfur compounds evaluated are given in table I(a). Certain compounds planned for evaluation were unavailable; however, enough information was gathered to allow a prediction of performance. Of the 20 compounds tested, only four passed the two qualification tests and thus warrant further work as electrolytes. The two qualification tests are good conductivity (at least 10^{-3} ohm^{-1}cm^{-1}) and stability towards lightweight

active metals (e.g., lithium).

The dashes in table I indicate that the data were not obtained because of lack of interest or because preliminary data did not warrant further work. No effort was made in the experimental program to purify any of the organic solvents; however, no solvent was abandoned until it failed one of the two qualification tests.

Table I(b) contains the compounds tested that were not sulfur derivatives. While many reasons might have been used for the selection of individual compounds, the reason for this group as a whole was to test compounds similar to those that Lewis contractors were using so as to gain experience with the instruments and testing procedures before the main series of sulfur derivatives was to be tested. The data are presented to show that conductivities in organic compounds other than sulfur derivatives lie in the same range, much below that found in water.

A collection of the data used in the previously described calculation of the dielectric constant is presented in table II. The test cell was designed to make both conductivity and capacitance measurements, which imposed limitations in measuring dielectric constants. Some dielectric constant determinations, thus, were not made because they were beyond the range of the instrumentation. The emphasis was placed on conductivity since that aspect is a more direct measure of a good solvent.

In table II a dashed line indicates that the experiment was not done either because of lack of interest or experimental difficulty. There are a number of dashes in table III that indicate that data could not be found in the literature. Some sulfur compounds tested in table I(a) are not listed in table III(a) because no significant information was found in the literature sources.

Propylene glycol sulfite was the most promising solvent tested. It has a conductivity of 3.3×10^{-3} ohm^{-1} $centimeter^{-1}$ when saturated with lithium perchlorate as the solute and was stable with lithium for 8 hours. The dielectric constant is 33 (unpublished Lewis data obtained by H. F. Leibecki). Its behavior with various cathode materials must yet be determined. Sultones were good solvents but reacted with lithium. While the rate of the reaction was very slow, any reaction at all makes it less desirable as a solvent. Sulfones also show some promise, but high melting points (above room temperature) make these compounds less desirable. It may be possible to make a low-melting sulfone but this will take time.

CONCLUSIONS

The study of the electrolyte solvent properties of a number of organic sulfur, phosphorus, and arsenic derivatives produced the following conclusions:

1. Propylene glycol sulfite was the most promising solvent tested on the basis of the two qualification tests. It has a conductivity of 3.3×10^{-3} ohm^{-1} centimeter^{-1} when saturated with lithium perchlorate as the solute and was stable with lithium for 8 hours. The dielectric constant is 33. Its behavior with various cathode materials must yet be determined.

2. The literature search and experimental work indicated that phosphorus and arsenic derivatives offer no hope of being good electrolyte solvents.

3. Sultones and sulfones were reasonably good solvents but were not so good as propylene glycol sulfite.

Lewis Research Center,
National Aeronautics and Space Administration,
Cleveland, Ohio, June 9, 1966,
123-34-01-09-22.

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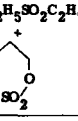
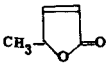
TABLE I. - EXPERIMENTAL ORGANIC SOLVENT DATA

[Temperature, 20° C.]

(a) Sulfur derivatives

Compound	Formula	Pure solvent	Solvent with solute of -				Dielectric constant	Reactivity with -		Viscosity
			Potassium perchlorate		Potassium hexafluorophosphate			Lithium	Calcium	
			0.01 M	Saturated	0.01 M	Saturated				
		Conductivity, $ohm^{-1}\ cm^{-1}$								(a)
2,2' Thiodiethanol	$HOC_2H_4SC_2H_4OH$	4.6×10^{-6}	5.1×10^{-6}	2.4×10^{-5}	7.3×10^{-6}	7.5×10^{-6}	27	Yes	No	High
Ethane dithiol	HSC_2H_4SH	1.5×10^{-7}	1.5×10^{-7}	1.5×10^{-7}	1.5×10^{-7}	1.5×10^{-7}	9.3	No	No	Low
Ethyl thiocyanate	C_2H_5SCN	4.6×10^{-5}	--------	[b] 9.2×10^{-5}	4.6×10^{-5}	--------	29.3	No	No	Low
Thioacetic acid	CH_3COSH	7.2×10^{-6}	--------	1.1×10^{-3}	--------	--------	----	Yes	Yes	Low
Ethane sulfonic acid	$C_2H_5SO_2OH$	1×10^{-2}	--------	---------	--------	--------	----	Yes	Yes	Low
Ethyl ethylxanthate	$C_2H_5OCS_2C_2H_5$	2.1×10^{-6}	--------	---------	--------	--------	----	Yes	---	(c)
Trimethylene disulfide		3.2×10^{-8}	1.5×10^{-7}	3.9×10^{-7}	3.9×10^{-8}	--------	7.9	No	No	Low
Butane sultone		6.5×10^{-7}	5.7×10^{-6}	4.0×10^{-5}	--------	--------	56	Slight	No	Medium
Propane sultone [d]		2.6×10^{-6}	--------	---------	--------	--------	----	Yes	---	High
Propylene glycol sulfite		5.6×10^{-6}	1.0×10^{-4}	3.3×10^{-3}	--------	1.5×10^{-3}	33	No	No	Medium
Sulfolane		9.1×10^{-7}	--------	4.9×10^{-4}	--------	--------	43	No	No	High
20-Percent diethyl sulfone in butane sultone	$C_2H_5SO_2C_2H_5$ + 	6.4×10^{-6}	--------	4.6×10^{-4}	--------	--------	----	Slight	No	--------

(b) Miscellaneous compounds

Aldol	$C_2H_5OHCH_2CHO$	4.4×10^{-5}	--------	5.6×10^{-5}	--------	--------	----	Yes	Yes	High
2-Nitro-1-butanol	$OHC_3H_6NO_2$	1.3×10^{-5}	--------	1.1×10^{-4}	--------	3.0×10^{-5}	----	Yes	No	High
Morpholine		1.1×10^{-8}	--------	---------	2.9×10^{-7}	--------	7.3	No	---	Low
Tropone		1.0×10^{-9}	--------	---------	--------	1×10^{-9}	4.5	---	---	Low
β Angelica lactone		4.0×10^{-6}	--------	---------	1.3×10^{-4}	5×10^{-4}	----	No	---	Low
Triisobutyl phosphate	$(C_4H_9O)_3PO$	2.7×10^{-6}	4.2×10^{-6}	---------	2.7×10^{-6}	3×10^{-6}	39.6	Yes	---	Moderate
Isopropyl nitrate	$(CH_3)_2CHO \cdot NO_2$	4.6×10^{-8}	5.4×10^{-8}	8.7×10^{-8}	1.5×10^{-7}	3.7×10^{-7}	20.1	No	---	Low
Dimethyl carbonate	$(C_2H_5O)_2CO$	1.0×10^{-9}	--------	---------	--------	--------	1.9	---	---	Low

[a] Viscosity determined by visual inspection; viscosities similar to or less than that of water were called low, while those more nearly like that of glycerol were called high.

[b] Solute, ammonium thiocyanate.

[c] Unstable.

[d] Temperature, 30° C.

TABLE II. - DATA USED IN CALCULATION OF DIELECTRIC CONSTANT

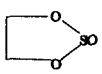
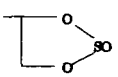
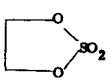
[Temperature, 20° C.]

Compound	Dielectric constant of known standard liquid, K_k	Capacitance in air of cell in vacuum, C_v, $\mu\mu F$ (a)	Capacitance of known standard liquid, C_k, $\mu\mu F$ (a)	Capacitance of unknown liquid, C_x, $\mu\mu F$ (a)
2, 2' Thiodiethanol	20.2	3.6	8	----
Ethane dithiol	20.2	3.3	7.9	5.3
Ethyl thiocyanate	20.2	3.3	7.9	----
Trimethylene disulfide	20.2	3.7	8.0	5.3
Triisobutyl phosphate	20.2	3.7	8.0	12.6
β Angelica lactone	20.2	3.7	8.0	----
Isopropyl nitrate	20.2	3.7	8.0	8.0
Dimethyl carbonate	20.2	3.7	8.0	3.9
Morpholine	20.2	3.7	8.0	4.5
Tropone	20.2	3.7	8.0	4.5
2-Nitro-1-butanol	20.2	3.7	8.0	----
Aldol	20.2	3.7	8.0	----
1, 4-Butane sultone	20.2	2.85	8.0	18.0
Thioacetic acid	20.2	2.90	8.0	----
Ethane sulfonic acid	20.2	2.95	8.1	----
Propylene glycol sulfite	10.3	2.8	5.4	----
Sulfolane	10.3	2.9	5.4	----
Ethyl ethylxanthate	10.3	2.8	5.4	10
Propane sultone	10.3	2.8	5.4	----

[a]At 0° F.

TABLE III. - ORGANIC SOLVENT DATA FROM REFERENCES 5 AND 6

(a) Sulfur compounds

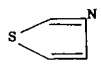
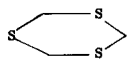
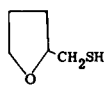
Compound	Formula	Molecular weight	Melting point, °C	Boiling point, °C (a)	Density, g/ml (b)	Refractive index, N (b)	Dielectric constant (b)	Dipole moment, μesu	Solubility in water	Conductivity of pure compound, $ohm^{-1}\ cm^{-1}$
Methanethiol	CH_3SH	48.11	-123	6	0.8665^{20}	---------	----	---------	----------	----------
Ethanethiol	C_2H_5SH	62.13	-144.4	37	$.8391^{20}$	1.4306^{20}	8^{20}	1.58×10^{-18}	Slightly soluble	----------
Propanethiol	C_3H_7SH	76.11	-111.5	68	$.8357^{25}$	1.435^{25}	----	---------	Slightly soluble	----------
Ethane dithiol	$C_2H_4S_2H_2$	94.2	-41.2	146	1.1192	1.5558	----	---------	----------	----------
Methane sulfonic acid	CH_3SO_2OH	96.11	20	167^{10}	1.4812^{18}	1.4317	----	---------	Very soluble	----------
Ethane sulfonic acid	$C_2H_5SO_2H$	110.3	-17	136	1.3341^{25}	1.4340^{16}	----	---------	Soluble	----------
Methyl sulfide	$(CH_3)_2S$	62.14	-83.2	37.3	$.8458^{25}$	---------	----	1.3	Insoluble	----------
Ethyl sulfide	$(C_2H_5)_2S$	90.19	-103.3	92	$.8369^{20}$	1.4424^{20}	----	1.6	Slightly soluble	----------
Dimethyl sulfoxide	CH_3SOCH_3	78.13	6	100	1.1014^{20}	---------	----	---------	Soluble	----------
Diethyl sulfoxide	$(C_2H_5)_2SO$	106.2	15	88-90	---------	---------	----	---------	Soluble	----------
Diethoxy sulfide	$C_2H_3OSOC_2H_5$	122.2	-------	117	$.9940^{20}$	1.4234^{20}	----	---------	Soluble	----------
Dimethyl sulfone	$CH_3SO_2CH_3$	94.14	109	233.5	1.1702^{110}	1.4226	----	4.49	Soluble	----------
Diethyl sulfone	$C_2H_5SO_2C_2H_5$	122.19	73	246	1.357^{20}	---------	----	---------	Soluble	----------
Diisopropyl sulfone	$C_3H_7SO_2C_3H_7$	150.24	36	-------	-------	---------	----	---------	Very soluble	----------
Dimethyl sulfate	$CH_3OSO_2OCH_3$	126.3	-31.75	188.5	1.3222^{20}	1.3874^{20}	55	3.2	Soluble	1.6×10^{-7} (at 0° C)
Diethyl sulfate	$C_2H_5OSO_2OC_2H_5$	154.2	-24.5	208	1.1774^{20}	1.4004^{20}	29.2	---------	Insoluble	2.6×10^{-7} (at 25° C)
Ethylene glycol sulfite		108.2	-11	173	1.4402^{20}	1.4463^{20}	----	---------	Very soluble	----------
Propylene glycol sulfite		------	-70	240	---------	---------	----	---------	Very soluble	----------
Ethylene glycol sulfate		124.12	99	-------	1.735^{13}	---------	----	---------	Decomposes	----------
Methyl sulfite	$CH_3OSOOCH_3$	110.3	-------	126	1.2183^{15}	---------	----	---------	Soluble	----------
Ethyl sulfite	$C_2H_5OSOOC_2H_5$	138.19	-------	157	1.0829^{20}	1.4144^{20}	----	---------	Slightly soluble	----------
Methyl bisulfate	CH_3OSO_2OH	112.1	-30	130	---------	---------	----	---------	Very soluble	----------
Thioacetic acid	CH_3COSH	76.11	-17	88	1.074^{16}	---------	----	---------	Very soluble	----------
Dithioacetic acid	CH_3CSSH	92.19	-------	37^{15}	1.24^{20}	---------	----	---------	Soluble	----------
Ethyl xanthic acid	$C_2H_5OCS_2H$	122.2	-53	-------	---------	---------	----	---------	Slightly soluble	----------
Ethyl ethylxanthate	$C_2H_5OCS_2C_2H_5$	------	-------	91-93	---------	---------	----	---------	----------	----------
Thioglycolic acid	$HSCH_2COOH$	92.12	-16.5	120^{20}	1.3253^{20}	---------	----	---------	Very soluble	----------
Ethyl thioacetic acid	$C_2H_5SCH_2COOH$	120.17	-8.7	164^{83}	1.1497	---------	----	---------	Very soluble	----------

[a]Superscript figures indicate pressure (mm).

[b]Superscript figures indicate temperature (°C).

TABLE III. - Concluded. ORGANIC SOLVENT DATA FROM REFERENCES 5 AND 6

(a) Concluded. Sulfur compounds

Compound	Formula	Molecular weight	Melting point, °C	Boiling point, °C	Density, g/ml (b)	Refractive index, N (b)	Dielectric constant	Dipole moment, μesu	Solubility in water	Conductivity of pure compound, $ohm^{-1}\ cm^{-1}$
Ethyl thiocyanate	C_2H_5SCN	87.14	-85.5	145	0.9964^{25}	1.4684^{15}	29.3	--------	Insoluble	1.2×10^{-6} (at 25° C)
Methyl thiocyanate	CH_3SCN	73.11	-51	132.9	1.0678^{25}	1.4669^{25}	----	--------	Slightly soluble	1.5×10^{-6} (at 25° C)
Ethyl isothiocyanate	C_2H_5NCS	87.14	-5.9	132	0.995^{20}	1.5134^{20}	----	--------	Insoluble	1.3×10^{-7} (at 25° C)
Dimethyl disulfide	CH_3SSCH_3	94.2	-------	116	1.0636^{0}	1.5208^{20}	----	--------	Insoluble	----------
Diethyl disulfide	$C_2H_5SSC_2H_5$	122.25	-------	153	0.9982^{20}	1.5063^{20}	----	--------	Slightly soluble	----------
Thioacetone	CH_3CSCH_3	------	-------	-------	--------	--------	----	--------	----------	----------
Ethane 1,2 bis-methylthio	$CH_3SCH_2CH_2SCH_3$	122.26	-------	182.5	1.0371^{20}	1.5292^{20}	----	--------	Soluble	----------
Trimethylene disulfide		------	-51	175	--------	--------	----	--------	Soluble	----------
Thiazole		------	-------	116.8	--------	--------	----	--------	Slightly soluble	----------
2, 2'-Thiodiethanol	$HOC_2H_4SC_2H_4OH$	------	-10	153	--------	--------	----	--------	Soluble	----------
Trithiane		------	216	-------	--------	--------	----	--------	Insoluble	----------
Furfurylmercaptan		114.17	-------	155	1.1314^{20}	1.5324^{20}	----	--------	Insoluble	----------
Sulfolane		------	81.7 (°F)	545(°F)	1.2590^{30}	1.4814^{30}	----	--------	Soluble	----------
Thiamide	NH_2CSNH_2	76.12	182	(c)	1.405^{20}	--------	----	--------	Soluble	----------
Acetone diethyl sulfone	$(CH_3)CSO_2(C_2H_5)_2$	228.3	128	[c]300	1.260^{25}	--------	----	--------	Soluble	----------

(b) Phosphorus and arsenic compounds

Compound	Formula	Molecular weight	Melting point, °C	Boiling point, °C	Density g/ml (b)	Refractive index, N (b)	Solubility in water
Triethyl phosphine	$(C_2H_5)_3P$	118.16	-----	127.5	0.8006^{19}	1.458^{15}	Insoluble
Diethyl phosphinic acid	$(C_2H_5)_2POOH$	122.1	18.5	92	--------	-------	Very soluble
Diethyl phosphinic anhydride	$\left[(C_2H_5)_2PO\right]_2O$	226.2	-----	188	1.472^{23}	-------	Decomposes
Trimethyl phosphoric ester	$(CH_3O)_3PO$	140.1	-----	197.2	1.2506	1.38	Very soluble
Triethyl phosphoric ester	$(C_2H_5O)_3PO$	182.16	-56.4	215-216	1.0725^{19}	1.4067^{16}	Soluble
Tributyl phosphoric ester	$(C_4H_9O)_3PO$	266.3	-----	289	0.9727^{25}	1.4224^{25}	Soluble
Dimethyl phosphorous acid	$(CH_3O)_2POH$	110.05	-----	170-171	1.2004^{20}	1.4036^{20}	----------
Trimethyl phosphorous ester	$(CH_3O)_3P$	124.08	-----	111-112	1.0520^{20}	1.4095^{20}	----------
Cacodyl oxide	$\left[(CH_3)_2As\right]_2O$	225.9	-25	150	1.4943	1.5255	Soluble

[b]Superscript figures indicate temperature (°C).

[c]Decomposes.

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