*Kirk-Othmer Encyclopedia of Chemical Technology.*

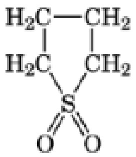
# SULFOLANE AND SULFONES

## 1. Sulfolane

Sulfolane was first described in the chemical literature in 1916. It has been noted for its exceptional chemical and thermal stability and unusual solvent properties. The search for a commercial process to produce sulfolane began about 1940. Market development quantities became available in 1959. Since then, both the use of and the applications for sulfolane have increased dramatically.

### 1.1. Properties

Sulfolane [126-33-0], $C_4H_8SO_2$ **1**, also known as tetrahydrothiophene-1,1-dioxide and tetramethylene sulfone, is a colorless, highly polar, water-soluble compound. Physical properties are given in Table 1 (1).



**(1)**

The thermal stability of sulfolane is summarized as follows (1):

| Temperature, °C | Quantity of $SO_2$ liberated from 250 mL sulfolane, mg/h |
|---|---|
| 180 | 0.6 |
| 200 | 2.8 |
| 220 | 3.3 |
| 240 | 24.1 |

Whereas sulfolane is relatively stable to about 220°C, above that temperature it starts to break down, presumably to sulfur dioxide and a polymeric material. Sulfolane, also stable in the presence of various chemical substances as shown in Table 2 (2), is relatively inert except toward sulfur and aluminum chloride. Despite this relative chemical inertness, sulfolane does undergo certain reactions, for example, halogenations, ring cleavage by alkali metals, ring additions catalyzed by alkali metals, reaction with Grignard reagents, and formation of weak chemical complexes.

**Table 1. Physical Properties of Sulfolane**

| Property | Value |
|---|---|
| molecular weight | 120.17 |
| boiling point, °C | 287.3 |
| melting point, °C | 28.5 |
| specific gravity | |
| 30/30 | 1.266 |
| 100/4 | 1.201 |
| density, 15°C, $g/cm^3$ | 1.276 |
| flash point, °C | 165–178 |
| viscosity, mPa·s(=cP) | |
| at 30°C | 10.5 |
| 100°C | 2.5 |
| 200°C | 1.0 |
| refractive index, $n_D$, at 30°C | 1.48 |
| heat of fusion, kJ/kg[a] | 11.44 |
| dielectric constant | 43.3 |
| surface tension, at 30°C, mN/m(=dyn/cm) | 35.5 |

[a] To convert J to cal, divide by 4.184.

**Table 2. Thermal Stability of Sulfolane in the Presence of Various Substances[a]**

| Additive | Additive quantity,[b] wt % | Reflux time, h | Sulfolane recovery, wt % | Remarks |
|---|---|---|---|---|
| none | | 5 | >98 | darkened after 30 min |
| aluminum chloride | 40 | 5 | 56 | blackened with evolution of heat and HCl |
| potassium carbonate | 40 | 5 | >98 | darkened after 30 min |
| sodium acetate | 40 | 5 | >98 | darkened after 30 min |
| sodium hydroxide[c] | 100 | 6 | >98 | darkened after 2 h |
| sulfur | 10 | 7 | 25 | $H_2S$ evolved, black sludge as residue |
| sulfuric acid[d] | 25 | 6[e] | 89 | darkened after 30 min |

[a] Ref. 2.
[b] Based on sulfolane at 100%.
[c] 25 wt % solution
[d] 93 wt % solution
[e] At 140–150°C

### *1.1.1. Halogenation*

Chlorine can be added to sulfolane by a uv-initiated process to give 3-chlorosulfolane [3844-04-0], 3,4-dichlorosulfolane [3001-57-8], and 3,3,4-trichlorosulfolane [42829-14-1] (3, 4).

Bromination of sulfolane by BrCl under uv irradiation gives 2-bromosulfolane [29325-66-4], which reacts further to give *cis*-2,5-dibromosulfolane [30186-52-8] (5). Continued irradiation converts the *cis*-isomer to *trans*-2,5-dibromosulfolane [30186-54-0], which yields first the *trans*-2,4 isomer [30186-53-9] and then the *trans*-3,4 isomer [15091-30-2] upon further irradiation.

### *1.1.2. Ring Cleavage by Alkali Metals*

The sulfolane ring can be cleaved by sodium or potassium metal in xylene at 66–140°C, by sodium amide in liquid ammonia at −33°C, and by sodium ethoxide at 240–250°C (6). The reaction products for the alkali–metal ring cleavage are dimeric bis-1,8-octanesulfinate salts under static reaction conditions and butanesulfinate salts under stirred reaction conditions. Ring cleavage by sodium amide in liquid ammonia gives sodium

3-butanesulfinate, and ring cleavage by sodium ethoxide gives sodium butadienesulfinate. The C–S bond can also be cleaved by ultrasonically dispersed potassium in toluene, then methylated with iodomethane (7–9).

#### *1.1.3. Ring Additions Catalyzed by Alkali Metals*

The addition of tributyltin chloride and olefins such as styrene, isoprene, or butadiene to sulfolane is catalyzed by alkali metals, including sodium and lithium, and by sodium amide (10–13). The addition of tributyltin chloride to sulfolane in the presence of sodium amide results in the formation of 2,5-bis(tributyltin)sulfolane [41392-14-7]. The addition of styrene to sulfolane in the presence of sodium yields 67% monostyrenated and 17% distyrenated sulfolane. Under similar conditions, isoprene gives 63% mono- and 7% disubstituted product. The reaction of butyllithium and bromoalkyls gives 2-alkyl derivatives of sulfolane (14).

#### *1.1.4. Grignard Reactions*

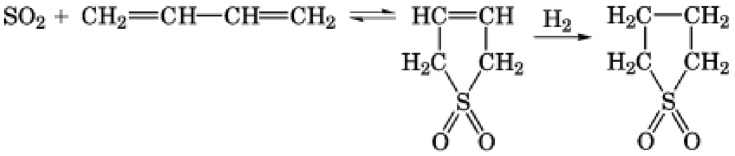
Sulfolane and its alkyl homologues react with ethylmagnesium bromide in ether, benzene, or tetrahydrofuran (15, 16). The reaction products are sulfolanyl 2-monobromide [82770-58-9] and 2,5-dimagnesium bromides [82770-59-0].

#### *1.1.5. Lewis Acid Complexes*

Sulfolane complexes with Lewis acids, such as boron trifluoride or phosphorus pentafluoride (17). For example, at room temperature, sulfolane and boron trifluoride combine in a 1:1 mole ratio with the evolution of heat to give a white, hygroscopic solid which melts at 37°C. The reaction of sulfolane with methyl fluoride and antimony pentafluoride in liquid sulfur dioxide gives crystalline tetrahydro-1-methoxythiophenium-1-oxide hexafluoroantimonate, the first example of an alkoxysulfoxonium salt (18).

### 1.2. Production

Sulfolane is produced domestically by the Phillips Chemical Company (Borger, Texas). Industrially, sulfolane is synthesized by hydrogenating 3-sulfolene [77-79-2] (2,5-dihydrothiophene-1,1-dioxide) **2**, the reaction product of butadiene and sulfur dioxide:



**(2)** **(1)**

Commercially, sulfolane is available as a crystalline anhydrous material, and containing 3 wt % deionized water as a freezing point depressant, as Sulfolane-W.

### 1.3. Toxicity

No mortality in laboratory animals was induced in percutaneous doses up to 3.8 g/kg body weight (19, 20). Subcutaneous administration of sulfolane gives $LD_{50}$ values for rats, mice, and rabbits of 1.606, 1.360, and 1.900–3.500 g/kg body weight, respectively (21). $LD_{50}$ values for sulfolane by oral administration to laboratory animals were 1.9–5.0 g/kg body weight (19–23). In most cases, the cause of death was believed to result from convulsions, which lead to anoxia.

When administered intraperitoneally, sulfolane is excreted both unchanged and as 3-hydroxysulfolane [13031-76-0] (24). Sulfolane injected intraperitoneally in mice and rats at 200–800 mg/kg at ambient temperatures of 15 and 25°C caused a dose-related inhibition of the metabolic rate and hypoactivity, accompanied by hypothermia 60 min after injection. Despite their hypothermic condition these animals did not select a warm ambient temperature. Because sulfolane toxicity appears to be greater upon increased tissue temperature, the behavior of these animals seeking lower environmental temperature appears to enhance their chance of survival (25–28).

Subcutaneous injection of 100–750 mg/kg sulfolane at an ambient temperature of 10°C also caused a dose-dependent decrease in colonic temperature of rabbits. Metabolic rate remained unchanged during the initial phase of the hypothermia for all dose groups; but peripheral vasodilatation, as indicated by an increase in ear skin temperature, was seen at the higher dose levels (29).

Intracerebroventricular injection of sulfolane in dosages of 300, 1000, and 3000 $\mu$g caused the preoptic/anterior hypothalamic area temperature to rise 0.23, 0.47, and 0.56%, respectively. This hyperthermia was considered significant at the 3000-$\mu$g dosage (30).

Sulfolane causes minimal and transient eye and skin irritation (19, 20). Inhalation of sulfolane vapors in a saturated atmosphere is not considered biologically significant. However, when aerosol dispersions have been used to elevate atmospheric concentration, blood changes and convulsions have been observed in laboratory animals (22, 31). Convulsions caused by sulfolane injected intraperitoneally have also been studied (32).

## 1.4. Uses

### *1.4.1. Extractive Solvent*

*1.4.1.1. Aromatic Hydrocarbons.* Sulfolane is used principally as a solvent for extraction of benzene, toluene, and xylene from mixtures containing aliphatic hydrocarbons (33–37). The sulfolane process was introduced in 1959 by Shell Development Company, and that process is licensed by Universal Oil Products. A sulfolane extraction process is also licensed by the Atlantic Richfield Company. In 1994, worldwide consumption was estimated at ca 6974 t/yr of sulfolane for 137 sulfolane extraction units (see BTX processes; Extraction, liquid–liquid ).

In general, the sulfolane extraction unit consists of four basic parts: extractor, extractive stripper, extract recovery column, and water–wash tower. The hydrocarbon feed is first contacted with sulfolane in the extractor, where the aromatics and some light nonaromatics dissolve in the sulfolane. The rich solvent then passes to the extractive stripper where the light nonaromatics are stripped. The bottom stream, which consists of sulfolane and aromatic components, and which at this point is essentially free of nonaromatics, enters the recovery column where the aromatics are removed. The sulfolane is returned to the extractor. The nonaromatic raffinate obtained initially from the extractor is contacted with water in the wash tower to remove dissolved sulfolane, which is subsequently recovered in the extract recovery column. Benzene and toluene recoveries in the process are routinely greater than 99%, and xylene recoveries exceed 95%.

*1.4.1.2. Normal and Branched Aliphatic Hydrocarbons.* The urea-adduction method for separating normal and branched aliphatic hydrocarbons can be carried out in sulfolane (38, 39). The process obviates the necessity of handling and washing the solid urea–normal paraffin adduct formed when a solution of urea in sulfolane is contacted with the hydrocarbon mixture. Overall recovery by this process is typically 85%; normal paraffin purity is 98%.

*1.4.1.3. Fatty Acids and Fatty Acid Esters.* Sulfolane exhibits selective solvency for fatty acids and fatty acid esters which depends on the molecular weight and degree of fatty acid unsaturation (40–42). Applications for this process are enriching the unsaturation level in animal and vegetable fatty oils to provide products with better properties for use in paint, synthetic resins, food products, plastics, and soaps.

*1.4.1.4. Wood Delignification.* The production of wood pulp (qv) for the paper (qv) industry consists of removing lignin (qv) from wood chips, thus freeing the cellulose fibers. An aqueous solution containing 30–70 wt % sulfolane efficiently extracts the lignin from aspen, Western hemlock, and Southern pine wood chips. Pulp yields are from 50–75% (43, 44).

*1.4.1.5. Miscellaneous Extractions.* Additional extractive separations using sulfolane involve (*1*) mercaptans and sulfides from sour petroleum (45); (*2*) *t*-butylstyrene from *t*-butylethylbenzene (46); (*3*) mixtures of close boiling chlorosilanes (47); and (*4*) aromatics from kerosene (48–50), naphtha (49, 51–53), and aviation turbine fuel (54).

### 1.4.2. Extractive Distillation Solvent

Extractive distillation is a technique for separating components in narrow boiling range mixtures which are difficult to separate by ordinary fractionation. The process consists of allowing a higher boiling liquid that has a special affinity for one or more of the components in the mixture to flow downward in a distillation column countercurrent to the ascending vapors and thereby to enhance differences in volatility of components of the mixture. Sulfolane is a suitable extractive–distillation solvent for carrying out the separation of close boiling alcohols (55) and chlorosilanes (56, 57); mono- and diolefins (58), such as isoprene and butadiene; electrochemical fluorination products; water from organic acids (59–62); ethers (63–68); ketones (69, 70); esters (71); cycloalkanes from alkanes (72); and aromatic hydrocarbons (73–84) (see Distillation, azeotropic and extractive).

### 1.4.3. Gas Treating

Another large commercial use for sulfolane is the removal of acidic components, eg, $H_2S$, $CO_2$, COS, $CS_2$, and mercaptans, from sour gas streams. The process, known as the Sulfinol process, was introduced by Shell in 1963 and consists of contacting the gas stream with a mixture of sulfolane, an alkanolamine (usually diisopropanolamine), and water (85–107). The acid gases are absorbed chemically by the amine and absorbed physically by sulfolane, which results in the advantages of high acid gas loading and ease of solvent regeneration.

Other gas-treating processes involving sulfolane are (*1*) hydrogen selenide removal from gasification of coal, shale, or tar sands (qv) (108); (*2*) olefin removal from alkanes (109); (*3*) nitrogen, helium, and argon removal from natural gas (110); (*4*) atmospheric $CO_2$ removal in nuclear submarines; (*5*) ammonia and $H_2S$ removal from waste streams; (*6*) $H_2S$, HCl, $N_2O$, and $CO_2$ removal from various streams (111–120); and (*7*) $H_2S$ and $SO_2$ removal from gas mixtures (121–123). The last process differs from the Sulfinol process in that the $H_2S$ and $SO_2$ are converted directly to high purity, elemental sulfur (see Sulfur removal and recovery).

### 1.4.4. Polymer Solvent

Sulfolane is a solvent for a variety of polymers, including polyacrylonitrile (PAN), poly(vinylidene cyanide), poly(vinyl chloride) (PVC), poly(vinyl fluoride), and polysulfones (124–129). Sulfolane solutions of PAN, poly(vinylidene cyanide), and PVC have been patented for fiber-spinning processes, in which the relatively low solution viscosity, good thermal stability, and comparatively low solvent toxicity of sulfolane are advantageous. Powdered perfluorocarbon copolymers bearing sulfo or carboxy groups have been prepared by precipitation from sulfolane solution with toluene at temperatures below 300°C. Particle sizes of 0.5–100 $\mu$m result.

### 1.4.5. Polymer Plasticizer

Nylon, cellulose, and cellulose esters can be plasticized using sulfolane to improve flexibility and to increase elongation of the polymer (130, 131). More importantly, sulfolane is a preferred plasticizer for the synthesis of cellulose hollow fibers, which are used as permeability membranes in reverse osmosis (qv) cells (131–133) (see Hollow-fiber membranes). In the preparation of the hollow fibers, a molten mixture of sulfolane and cellulose triacetate is extruded through a die to form the hollow fiber. The sulfolane is subsequently extracted from the fiber with water to give a permeable, plasticizer-free, hollow fiber.

### *1.4.6. Polymerization Solvent*

Sulfolane can be used alone or in combination with a cosolvent as a polymerization solvent for polyureas, polysulfones, polysiloxanes, polyether polyols, polybenzimidazoles, polyphenylene ethers, poly(1,4-benzamide) (poly(imino-1,4-phenylenecarbonyl)), silylated poly(amides), poly(arylene ether ketones), polythioamides, and poly(vinylnaphthalene/fumaronitrile) initiated by laser (134–144). Advantages of using sulfolane as a polymerization solvent include increased polymerization rate, ease of polymer purification, better solubilizing characteristics, and improved thermal stability. The increased polymerization rate has been attributed not only to an increase in the reaction temperature because of the higher boiling point of sulfolane, but also to a decrease in the activation energy of polymerization as a result of the contribution from the sulfonic group of the solvent.

### *1.4.7. Electronic and Electrical Applications*

Sulfolane has been tested quite extensively as the solvent in batteries (qv), particularly for lithium batteries. This is because of its high dielectric constant, low volatility, excellent solubilizing characteristics, and aprotic nature. These batteries usually consist of anode, cathode polymeric material, aprotic solvent (sulfolane), and ionizable salt (145–156). Sulfolane has also been patented for use in a wide variety of other electronic and electrical applications, eg, as a coil-insulating component, solvent in electronic display devices, as capacitor impregnants, and as a solvent in electroplating baths (157–161).

### *1.4.8. Miscellaneous Uses*

Textile applications for sulfolane include preparation of concentrated, storage-stable basic dyes; fabric treating prior to dyeing to improve the dye adsorption; and fiber treating to improve the tensile strength, pilling resistance, and drawing properties (162–164) (see Textiles). The curing time of polysulfide-based sealants and fluoropolymer rubbers decreases significantly upon the incorporation of small amounts of sulfolane into the formulation (165, 166). Sulfolane also exhibits catalytic activity for some reactions, increasing both the conversion and the selectivity. Examples of systems where sulfolane functions catalytically are the synthesis of 1,4-dicyanobutene and substituted ketones (167, 168). Microemulsions containing sulfolane together with a cationic surfactant such as cetyltrimethylammonium bromide are useful for the detoxification of pesticides and chemical warfare agents by enabling improved removal or destruction through solubilization, oxidation, or hydrolysis (169). Sulfolane has also been used as a cosurfactant in systems for enhanced petroleum recovery (170).

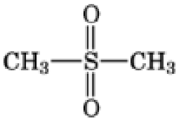
## 2. Sulfones

3-Sulfolene **2** is the next most commercially important sulfone after sulfolane. Besides its precursor role in sulfolane manufacture, 3-sulfolene is an intermediate in the synthesis of sulfolanyl ethers, which are used as hydraulic fluid additives (see Hydraulic fluids). 3-Sulfolene or its derivatives also have been used in cosmetics (qv) and slimicides. Selected physical properties of 3-sulfolene are listed in Table 3.

**Table 3. Physical Properties of 3-Sulfolene**

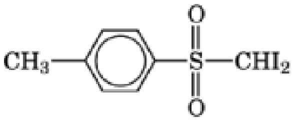
| Property | Value |
|---|---|
| molecular weight | 118.154 |
| specific gravity, 70°C | 1.314 |
| melting point, °C | 65 |
| boiling point | dec |
| flash point, °C | 113 |

Other sulfones with commercial potential include dimethyl sulfone [67-71-0] (sulfonylbismethane) **4**, diiodomethyl *p*-tolylsulfone [20018-09-1] **5**, 4,4′-dihydroxydiphenyl sulfone [80-09-1] (Bisphenol S) **6**, bis(*p*-chlorophenyl) sulfone [80-07-9] **7**, and 4,4′-bis(*p*-chlorophenylsulfonyl) biphenyl [22287-56-5] **8**. Dimethyl sulfone **4** has been patented as a solvent for extractive distillation, electroplating baths, inks, and adhesives (76, 171–173). Diiodomethyl *p*-tolylsulfone **5** has been patented for use as an antifungal preservative (174–176); and 4,4′-dihydroxydiphenyl sulfone **6** has been patented for a variety of applications, eg, as an electroplating solvent, a washfastening agent, and a component in a phenolic resin (177, 178). The two chloro-containing sulfones have been patented in the preparation of high temperature engineering plastics (qv), **7** and **8**, (179, 180).



$CH_3SO_2CH_3$

(**4**)



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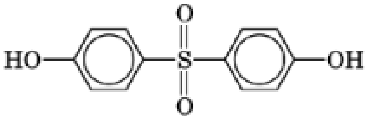

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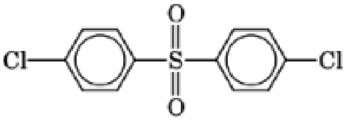

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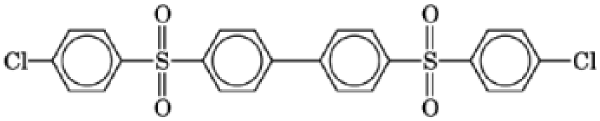

(**8**)

## BIBLIOGRAPHY

"Sulfolane" in *ECT* 2nd ed., Vol. 19, pp. 250–254, by G. S. Morrow, Shell Chemical Co.; "Sulfolanes and Sulfones" in *ECT* 3rd ed., Vol. 21, pp. 961–968, by M. Lindstrom and R. Williams, Phillips Research Center.

## Cited Publications

1. *Technical Information on Sulfolane*, Bulletin 524, Phillips Chemical Co., Bartlesville, Okla.
2. T. E. Jordon and F. Kipnis, *Ind. Eng. Chem.* **41**, 2635 (1949).
3. V. I. Dornov and co-workers, *Khim. Seraorg. Soedin. Soderzh. Naftyakh Nefteprod.* **8**, 133 (1968).
4. V. I. Dornov and V. A. Snegotskaya, *Khim. Geterotsikl. Soedin. Sb.* **3**, 5 (1971).
5. V. I. Dornov and co-workers, *Zh. Org. Khim.* **6**, 2029 (1970).
6. V. I. Dornov and co-workers, *Khim. Seraorg. Soedin. Soderzh. Naftyakh Nefteprod.* **8**, 144 (1968).
7. T. S. Chou and co-workers, *Tetrahedron Lett.* **32**, 3551–3554 (1991).
8. T. S. Chou and co-workers, *J. Chin. Chem. Soc.* **38**, 283–287 (1991).
9. T. S. Chou and M. L. Sin, *Tetrahedron Lett.*, **26**, 4495–4498 (1985).
10. Ger. Offen. 2,246,939 (Apr. 5, 1973), D. J. Peterson, J. F. Ward, and R. A. D'Amico (to Procter & Gamble Co.).
11. U.S. Pat. 3,988,144 (Oct. 26, 1976), D. J. Peterson, J. F. Ward, and R. A. D'Amico (to Procter & Gamble Co.).
12. E. M. Asatryan and co-workers, *Arm. Khim. Zh.* **29**, 553 (1976).
13. E. M. Asatryan and co-workers, *Tezisy Dokl. Molodezhnaya Konf. Org. Sint. Fioorg. Khim.* **44** (1976).
14. L. Frazier and D. P. Claypool, *Synth. Commun.* **18**, 583–588 (1988).
15. T. E. Bezmenova and co-workers, *Khim. Seraorg. Soedin. Soderzh. Neftyakh Nefteprod.* **8**, 140 (1968).
16. U.S. Pat. 2,656,362 (Oct. 26, 1953), H. E. Faith (to Allied Laboratories, Inc.).
17. J. J. Jones, *Inorg. Chem.* **5**, 1229 (1966).
18. M. M. Abdel-Malik and co-workers, *Can. J. Chem.* **62**, 69–73 (1984).
19. V. K. H. Brown, L. W. Ferrigan, and D. E. Stevenson, *Br. J. Industr. Med.* **23**, 302 (1966).
20. H. F. Smyth and co-workers, *Am. Ind. Hyg. Assoc. J.* **30**, 470 (1969).
21. M. E. Anderson and co-workers, *Res. Commun. Chem. Pathol. Pharmacol.* **15**, 571 (1976).
22. M. E. Andersen and co-workers, *Toxicol. Appl. Pharmacol.* **40**, 463 (1977).
23. J. J. Roberts and G. P. Warwick, *Biochem. Pharmacol.* **6**, 217 (1961).
24. G. R. Middleton, R. W. Young, and L. J. Jenkins, Jr., *Annual Research Report*, Armed Forces Radiobiology Research Institute, National Technical Information Service, Springfield, Va., 1974, 77–79.
25. C. J. Gordon and co-workers, *Arch. Toxicol.* **56**, 123–127 (1984).
26. P. H. Ruppert and R. S. Dyer, *Toxicol. Lett.* **28**, 111–116 (1985).
27. C. J. Gordon and co-workers, *J. Toxicol. Environ. Health* **16**, 461–468 (1985).
28. C. J. Gordon and co-workers, *Environ. Res.* **40**, 92–97 (1986).
29. F. S. Mohler and C. J. Gordon, *Arch. Toxicol.* **62**, 216–219 (1988).
30. F. S. Mohler and C. J. Gordon, *Neurotoxicology* **10**, 53–62 (1989).
31. Z. K. Filippova, *Khim. Seraorg. Soedin. Soderzh. Neftyakh. Nefteprod.* **8**, 701 (1968).
32. L. J. Burdette and R. S. Dyer, *Neurobehav. Toxicol. Teratol.* **8**, 621 (1986).
33. D. B. Broughton and G. F. Asselin, *7th World Petrol. Congr. Proc.* **4**, 65 (1968).
34. F. S. Beardmore and W. C. G. Kosters, *J. Inst. Pet. London* **49**(469), 1 (1963).
35. M. A. Plummer, *Hydrocarbon Process.* **52**(6), 91 (1973).
36. M. A. Plummer, *Hydrocarbon Process.* **59**(9), 203 (1980).
37. *Aromatics Extraction Process*, publication EM-9964, Atlantic Richfield Co.
38. U.S. Pat. 3,617,499 (Nov. 2, 1971), D. M. Little (to Phillips Petroleum Co.).
39. U.S. Pat. 3,645,889 (Feb. 29, 1972), D. O. Hanson (to Phillips Petroleum Co.).
40. U.S. Pat. 2,360,860 (Oct. 24, 1944), R. C. Morris and E. C. Shokal (to Shell Development Co.).
41. J. Wisniak, *Br. Chem. Eng.* **15**(1), 76 (1970).
42. Eur. Pat. 576,191 (Dec. 29, 1993), J. B. Cloughley and co-workers (to Scotia Holdings PLC, UK).
43. L. P. Clermont, *TAPPI* **53**, 2243 (1970).
44. L. D. Starr and co-workers, *TAPPI Alkaline Pulping Conference Preprints*, 1975, p. 195.
45. B. B. Agrawal and co-workers, *Erdoel Kohle, Erdgas, Petrochem.* **40**, 489–490 (1987).
46. U.S. Pat. 4,543,438 (Sept. 24, 1985), J. D. Henery and co-workers (to El Paso Products Co.).
47. U.S. Pat. 4,402,796 (Sept. 6, 1983), O. W. Marko and S. F. Rentsch (to Dow Corning Corp.).
48. Ind. Pat. 170,747 (May 9, 1992), S. K. Joshi and co-workers (to Council of Scientific and Industrial Research, India).
49. M. K. Khanna and B. S. Rawat, *Res. Ind.* **37**, 67–72 (1992).

50. A. K. Jain and S. J. Chopra in X. Chou, ed., *Proceedings of an International Conference on Petroleum Refining and Petrochemical Processing*, International Academic Publishers, Beijing, China, 1991, 368–374.
51. F. A. Poposka and co-workers, *J. Serb. Chem. Soc.*, **56**, 489–498 (1991).
52. M. S. H. Fandary and co-workers, *Solvent Extr. Ion Exch.* **7**, 677–703 (1989).
53. R. Krishna and co-workers, *Indian J. Technol.* **25**, 602–606 (1987).
54. R. Mehrotra and co-workers, *Fluid Phase Equilib.* **32**, 17–25 (1986).
55. U.S. Pat. 5,160,414 (Nov. 3, 1992), F. M. Lee and co-workers (to Phillips Petroleum Co.).
56. U.S. Pat. 4,411,740 (Oct. 25, 1983), O. L. Flaningam and R. L. Halm (to Dow Corning Corp.).
57. U.S. Pat. 4,402,797 (Sept. 6, 1983), R. L. Halm and S. F. Rentsch (to Dow Corning Corp.).
58. U.S. Pat. 5,151,161 (Sept. 29, 1992), F. M. Lee and R. E. Brown (to Phillips Petroleum Co.).
59. U.S. Pat. 5,167,774 (Dec. 1, 1992), L. Berg.
60. U.S. Pat. 5,154,800 (Oct. 13, 1992), L. Berg.
61. U.S. Pat. 4,735,690 (Apr. 5, 1988), L. Berg and A. I. Yeh.
62. U.S. Pat. 4,642,166 (Feb. 10, 1987), L. Berg and A. I. Yeh.
63. U.S. Pat. 5,228,957 (July 20, 1993), L. Berg.
64. U.S. Pat. 5,160,414 (Nov. 3, 1992), F. M. Lee and co-workers (to Phillips Petroleum Co.).
65. L. Berg and A. I. Yeh, *AIChE J.*, **30**, 871–874 (1984).
66. U.S. Pat. 4,459,179 (July 10, 1984), L. Berg and A. I. Yeh.
67. L. Berg and A. I. Yeh, *Chem. Eng. Commun.*, **29**, 283–289 (1984).
68. U.S. Pat. 4,459,178 (July 10, 1984), L. Berg and A. I. Yeh.
69. U.S. Pat. 4,957,595 (Sept. 18, 1990), L. Berg and G. Bentu.
70. U.S. Pat. 4,948,471 (Aug. 14, 1990), L. Berg and G. Bentu.
71. U.S. Pat. 5,240,567 (Aug. 31, 1993), L. Berg and R. W. Wytcherley.
72. U.S. Pat. 4,944,849 (July 31, 1990), F. M. Lee (to Phillips Petroleum Co.).
73. U.S. Pat. 2,570,205 (Oct. 9, 1951), C. S. Carlson, E. Smith, and P. V. Smith, Jr. (to Standard Oil Development Co.).
74. B. S. Rawat, K. L. Mallik, and I. B. Gulati, *J. Appl. Chem. Biotechnol.* **22**, 1001 (1972).
75. U.S. Pat. 3,024,028 (May 17, 1977), D. M. Haskell (to Phillips Petroleum Co.).
76. U.S. Pat. 4,054,613 (Oct. 18, 1977), D. M. Haskell, E. E. Hopper, and B. L. Munro (to Phillips Petroleum Co.).
77. U.S. Pat. 4,090,923 (May 23, 1978), D. M. Haskell and C. O. Carter (to Phillips Petroleum Co.).
78. U.S. Pat. 3,689,373 (May 5, 1972), W. M. Hutchinson (to Phillips Petroleum Co.).
79. Brit. Pat. 1,333,039 (Oct. 10, 1973), M. F. Kelly and K. D. Uitta (to Universal Oil Products).
80. Can. Pat. 962,212 (Feb. 4, 1975), H. L. Thompson (to Universal Oil Products).
81. Brit. Pat. 1,392,735 (May 26, 1975), H. L. Thompson (to Universal Oil Products).
82. U.S. Pat. 5,310,480 (May 10, 1994), J. A. Vidueira (to UOP Inc.).
83. U.S. Pat. 5,069,757 (Dec. 3, 1991), R. E. Brown (to Phillips Petroleum).
84. F. M. Lee and D. M. Coombs, *Ind. Eng. Chem. Res.*, **26**, 564–573 (1987).
85. C. L. Dunn, *Hydrocarbon Process. Pet. Refiner* **43**, 150 (1964).
86. U.S. Pat. 3,630,666 (Dec. 28, 1971), L. V. Kunkel (to Amoco Production Co.).
87. U.S. Pat. 4,025,322 (May 24, 1977), E. J. Fisch (to Shell Oil Co.).
88. U.S. Pat. 4,100,257 (July 11, 1978), G. Sartori and D. W. Savage (to Exxon Research and Engineering Co.).
89. U.S. Pat. 3,965,244 (June 22, 1976), (to Shell Oil Co.).
90. F. Murrieta-Guevara and co-workers, *Fluid Phase Equilib.* **95**, 163–174 (1994).
91. F. T. Okimoto, *Proc. Annu. Conv. Gas Process. Assoc.*, **72**, 149–151 (1993).
92. F. Murrieta-Guevara and co-workers, *Fluid Phase Equilib.* **86**, 225–231 (1993).
93. R. J. MacGregor and A. E. Mather, *Can. J. Chem. Eng.* **69**, 1357–1366 (1991).
94. F. J. Littel and co-workers, *J. Chem. Eng. Data* **37**, 49–55 (1992).
95. T. T. Teng and A. E. Mather, *Fas Sep. Purif.* **5**, 29–34 (1991).
96. A. E. Mather and co-workers, *Ind. Eng. Chem. Res.* **30**, 1213–1217 (1991).
97. Eur. Pat. 399,608 (Nov. 28, 1990), V. K. Chopra and S. P. Kaija (to Shell Internationale Research Maatschappij BV, the Netherlands).
98. A. E. Mather and co-workers, *Fluid Phase Equilib.* **56**, 313–324 (1990).
99. K. Yogish, *Can. J. Chem. Eng.* **68**, 511–512 (1990).

100. F. Murrieta-Guevara and co-workers, *Fluid Phase Equilib.* **53**, 1–6 (1989).
101. Brit. Pat. 2,202,522 (Sept. 28, 1988), J. G. Christy and co-workers (to Shell Internationale Research Maatschappij BV, the Netherlands).
102. A. E. Mather and B. E. Roberts, *Chem. Eng. Commun.* **72**, 201–211 (1988).
103. U.S. Pat. 4,749,555 (June 7, 1988), W. V. Bush (to Shell Oil Co.).
104. R. J. Demyanovich and S. Lynn, *Ind. Eng. Chem. Res.* **26**, 548–555 (1987).
105. Brit. Pat. 2,188,455 (Nov. 2, 1983), E. J. Fisch (to Shell Internationale Research Maatschappij BV, the Netherlands).
106. Brit. Pat. 2,072,525 (Oct. 7, 1981), R. Cornelisse (to Shell Internationale Research Maatschappij BV, the Netherlands).
107. Eur. Pat. 13049 (July 9, 1980), E. J. Van De Kraats and R. C. Darton (to Shell Internationale Research Maatschappij BV, the Netherlands).
108. U.S. Pat. 4,792,405 (Dec. 20, 1988), D. C. Baker (to Shell Oil Co.).
109. WO Pat. 8,702,031 (Apr. 9, 1987), Y. R. Mehra (to El Paso Hydrocarbons Co.).
110. WO Pat. 8,700,518 (Jan. 29, 1987), Y. R. Mehra (to El Paso Hydrocarbons Co.).
111. A. E. Mather and Yi-Gui Li, *Fluid Phase Equilib.* **96**, 119–142 (1994).
112. P. G. T. Fogg, *Solubility Data Ser.*, **50**, 358–383 (1992).
113. A. E. Mather and co-workers, *J. Chem. Technol. Biotechnol.* **51**, 197–208 (1991).
114. K. Yogish, *J. Chem. Eng. Jpn.*, **24**, 135–137 (1991).
115. A. E. Mather and co-workers, *Fluid Phase Equilib.* **56**, 313–324 (1990).
116. P. G. T. Foog, *Solubility Data Ser.* **42**, 330–341 (1989).
117. F. Murrieta-Guevara and co-workers, *Fluid Phase Equilib.* **44**, 105–115 (1988).
118. P. G. T. Foog, *Solubility Data Ser.* **32**, 137–326 (1988).
119. D. M. Kassim and co-workers, *Fluid Phase Equilib.* **41**, 287–294 (1988).
120. A. E. Mather and B. E. Roberts, *Can. J. Chem. Eng.* **66**, 519–520 (1988).
121. P. R. Gustafson and R. R. Miller, *Naval Research Laboratories Report 6926*, July 1969.
122. U.S. Pat. 3,551,102 (Dec. 29, 1970), G. R. Hettick and D. M. Little (to Phillips Petroleum Co.).
123. Ger. Pat. 2,108,284 (Aug. 31, 1972), G. Schulze (to Badische Anilin-und-Soda-Fabrik AG).
124. U.S. Pat. 2,706,674 (Apr. 19, 1955), G. M. Rothrock (to E. I. du Pont de Nemours & Co., Inc.).
125. U.S. Pat. 2,548,169 (Apr. 10, 1951), F. F. Miller (to B. F. Goodrich Co.).
126. U.S. Pat. 2,617,777 (Nov. 11, 1952), E. Heisenberg and J. Kleine (to Vereinigte Glanzstoff-Fabriken).
127. U.S. Pat. 2,953,818 (Sept. 27, 1960), L. R. Barton (to E. I. du Pont de Nemours & Co., Inc.).
128. U.S. Pat. 3,474,030 (Oct. 21, 1969), D. M. Little (to Phillips Petroleum Co.).
129. U.S. Pat. 4,540,716 (Sept. 10, 1985), M. J. Covitch and G. G. Sweetapple (to Diamond Shamrock Chemicals Co.).
130. U.S. Pat. 3,361,697 (Jan. 2, 1968), W. E. Garrison and T. J. Hyde (to E. I. du Pont de Nemours & Co., Inc.).
131. U.S. Pat. 2,471,272 (May 24, 1949), G. W. Hooker and N. R. Peterson (to The Dow Chemical Co.).
132. U.S. Pat. 3,619,459 (Nov. 9, 1971), P. G. Schrader (to The Dow Chemical Co.).
133. T. E. Davis and G. W. Skiens, *Polym. Prepr. Am. Chem. Soc. Div. Polym Chem.* **12**, 378 (1971).
134. U.S. Pat. 3,476,709 (Nov. 4, 1969), F. B. Jones (to Phillips Petroleum Co.).
135. U.S. Pat. 2,703,793 (Mar. 8, 1955), (to E. I. du Pont de Nemours & Co., Inc.).
136. U.S. Pat. 3,175,994 (Mar. 30, 1965), A. Katchman and G. D. Cooper (to General Electric Co.).
137. Jpn. Pat. 70 06,533 (Mar. 5, 1970), M. Ikeda and co-workers (to Takeda Chemical).
138. U.S. Pat. 3,784,517 (Jan. 8, 1974), F. L. Hedberg and C. S. Marvel (to U.S. Dept. of the Air Force).
139. Jpn. Pat. 74 02,359 (Jan. 19, 1974), S. Izawa and K. Mizushiro (to Asahi Dow, Ltd.).
140. U.S. Pat. 3,951,914 (Apr. 20, 1976), S. L. Kwolek (to E. I. du Pont de Nemours & Co., Inc.).
141. M. Kakimoto, Y. Oishi, and Y. Imai, *Makromol. Chem., Rapid Commun.* **6**, 557–562 (1985).
142. WO Pat 8,403,891 (Oct. 11, 1984), V. Janson and H. C. Gors (to Raychem. Corp.).
143. Y. Saegusa, S. Iida, S. Nakamura, N. Chau, and Y. Iwakura, *J. Polym. Sci., Polym. Chem. Ed.* **22**, 1017–1023 (1984).
144. M. A. Williamson, J. D. Smith, P. M. Castle, and R. N. Kauffman, *J. Polym. Sci., Polym. Chem. Ed.* **20**, 1875–1884 (1982).
145. U.S. Pat. 5,252,413 (Oct. 12, 1993), M. Alamgir and K. M. Abraham (to EIC Laboratories, Inc.).
146. EP Pat. 529,802 (Mar. 3, 1993), A. Webber (to Eveready Battery Co.).
147. EP Pat. 346,675 (Jan. 10, 1990), N. Furukawa, S. Yoshimura, and M. Takahashi (to Sanyo Electric Co. Ltd., Japan).
148. S. J. Visco, M. Liu, and L. C. De Jonghe, *J. Electrochem. Soc.* **137**, 1191–1192 (1990).

149. EP Pat. 331,342 (Sept. 6, 1989), P. Cheshire and J. E. Przeworski (to Imperial Chemical Industries PLC, U.K.).
150. M. Maxfield, T. R. Jow, M. G. Sewchok, and L. W. Shacklette, *J. Power Sources*, **26**, 93–102 (1989).
151. V. R. Koch, L. A. Dominey, J. L. Goldman, and M. E. Langmuir, *J. Power Sources* **20**, 287–291 (1987).
152. U.S. Pat. 4,670,363 (June 2, 1987), T. A. Whitney and D. L. Foster (to Duracell, Inc.).
153. Y. Matsuda, M. Morita, K. Yamada, and K. Hirai, *J. Electrochem. Soc.* **132**, 2538–2543 (1985).
154. J. S. Foos, L. S. Rembetsy, and S. B. Brummer, Report, LBL-18414, Order No. DE85004345, National Technical Information Service (NTIS), 62 pp. 1984.
155. S. P. S. Yen, B. Carter, D. Shen, and R. Somoano, *Proc. Power Sources Symp.* **30**, 71–73 (1982).
156. G. E. Blomgren, *Proc. Symp. Power Sources Biomed. Implantable Appl. Ambient Temp. Lithium Batt.* **80-4**, 368–377 (1980).
157. Ger. Pat. 2,739,571 (Mar. 23, 1978), L. G. Versberg (to ASEA AB).
158. U.S. Pat. 3,891,458 (June 24, 1975), M. Wisenberg (to Electrochimica Corp.).
159. U.S. Pat. 4,110,015 (Aug. 29, 1978), T. B. Reddy (to American Cyanamid Co.).
160. R. Tobazeon and E. Gartner, *IEE Conf. Publ. London* **129**, 225 (1975).
161. Ger. Pat. 2,207,703 (Aug. 31, 1972), F. Huba and J. E. Bride (to Diamond Shamrock Corp.).
162. Brit. Pat. 1,349,511 (April 3, 1974), J. F. Dawson and J. Schofield (to Yorkshire Chemicals, Ltd.).
163. Jpn. Pat. 77 25,173 (Feb. 24, 1977), M. Ono and co-workers (to Mitsubishi Rayon Co., Ltd.).
164. Brit. Pat. 1,263,082 (Feb. 9, 1972), R. G. Roberts (to Courtaulds, Ltd.).
165. U.S. Pat. Off. T 974,003 (Sept. 1978),
166. Ger. Pat. 2,449,095 (April 17, 1975), R. E. Kolb (to 3M Co.).
167. Ger. Pat. 2,256,039 (May 17, 1973), O. T. Onsager (to Halcon International Inc.).
168. Rus. Pat. 74 25,925 (July 4, 1974), T. Kawaguchi and co-workers (to Kurary Co., Ltd.).
169. R. P. Seiders, *ACS Symp. Ser.* **272**, 265–273 (1985).
170. U.S. Pat. 4,485,872 (Dec. 4, 1984), P. R. Stapp (to Phillips Petroleum Co.).
171. U.S. Pat. 4,046,647 (Sept. 6, 1977), E. P. Harbulak (to M and T Chemicals, Inc.).
172. Jpn. Pat. 80 50,072 (Apr. 11, 1980), K. Hirano (to Pilot Ink Co., Ltd.).
173. Jpn. Pat. 80 66,980 (May 20, 1980), A. Yamada and K. Kimma (to Toagosei Chemical Industry Co., Ltd.).
174. U.S. Pat. 4,078,888 (Mar. 4, 1978), F. W. Arbir and F. C. Becker (to Abbott Laboratories).
175. *Fed. Reg.* **44**, 52189 (Sept. 1979).
176. Rus. Pat. 78 73,434 (June 29, 1978), K. Kariyone and co-workers (to Fujisawa Pharmaceutical Co., Ltd.).
177. Brit. Pat. Appl. 2,001,679 (Feb. 7, 1979), S. A. Lipowski (to Diamond Shamrock Corp.).
178. Rus. Pat. 79 28,357 (Mar. 2, 1979), J. Hiroshima and I. Kai (to Asahi Organic Chemicals Industry Co., Ltd.).
179. U.S. Pat. 4,016,145 (Apr. 5, 1977), R. W. Campbell (to Phillips Petroleum Co.).
180. U.S. Pat. 5,079,079 (Jan. 7, 1992), M. L. Stone, R. L. Bobsein, and H. F. Efner (to Phillips Petroleum Co.).

### General Reference

181. J. A. Reddick and W. E. Bunger, *Organic Solvents*, 3rd ed., Vol. **2**, Wiley-Interscience, New York, 1970, 467–468.

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