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Separation of Isopropyl Ether from Acetone by Extractive Distillation

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Isopropyl ether cannot be completely removed from isopropyl ether–acetone mixtures by distillation because of the presence of the minimum binary azeotrope. Isopropyl ether can be readily removed from mixtures containing this and acetone by using extractive distillation in which the extractive distillation agent is a higher boiling oxygenated, nitrogenous- and/or sulfur-containing organic compound or a mixture of these. Typical examples of effective agents are: dimethyl sulfoxide; sulfolane and propylene glycol; and glycerine, ethylene glycol and adiponitrile.

Extractive distillation is the method of separating close boiling compounds or azeotropes by carrying out the distillation in a multiplate rectification column in the presence of an added liquid or liquid mixture—the liquid(s) having a boiling point higher than the compounds being separated. The extractive agent is introduced near the top of the column and flows downward until it reaches the stillpot or reboiler. Its presence on each plate of the rectification column alters the relative volatility of the close boiling compounds in a direction to make the separation on each plate greater and thus require either fewer plates to effect the same separation or make possible a greater degree of separation with the same number of plates.

When the compounds to be separated normally form an azeotrope, the proper agents will cause them to boil separately during extractive distillation and thus make possible a separation in a rectification column that cannot be done at all when no agent is present. At the bottom of a continuous column, the less volatile components of the close boiling mixtures and the extractive agent are continuously removed from the column. The usual methods of separation of these components are the use of another rectification column, cooling and phase separation, or solvent extraction.

One of the commercially important ways to manufacture acetone is by the catalytic dehydrogenation of isopropanol. Since the acetone does not form an azeotrope with isopropanol and boils 26C° below it, acetone is relatively easy to separate from the unreacted isopropanol by rectification. However, a concurrent reaction takes place in which some of the isopropanol dehydrates to form isopropyl ether. Acetone and isopropyl ether form a minimum azeotrope boiling at 54.2°C and containing 61 wt.% acetone. It is, therefore, impossible to produce pure acetone from acetone–isopropyl ether mixtures by rectification. Extractive distillation would be an attractive method of effecting the separation of acetone from isopropyl ether if agents can be found that will break the acetone–isopropyl ether azeotrope and are easy to recover from the acetone.

The breaking of this azeotrope by extractive distillation is a new concept. The closest application of this concept might be the breaking of the ethanol-water azeotrope. Schneible (1923) used glycerol; Smith (1951) employed ethoxyethanol and butoxyethanol for this purpose; and Catterall (1952) reported gasoline as being effective. These are dehydrations and operate more conventionally as a solvent extraction process. Snyder (1978) has suggested that the selection of the extractive distillation agents be made on the basis of their properties of proton acceptance and dipole moment. To this we would add strong hydrogen bond capability as a major factor. However, even with these guidelines, the search for effective extractive distillation agents must be directed to a large number of compounds and mixtures.

We have ascertained that certain oxygenated, nitrogenous- and/or sulfur-containing compounds, some individually but principally as mixtures, will effectively negate the isopropyl ether–acetone azeotrope and permit the separation of pure isopropyl ether from acetone by rectification when employed as the agent in extractive distillation. Table 1 lists the compounds, mixtures and approximate proportions that we have found to be exceptionally effective. Table 2 lists those that are reasonably successful and Table 3, those that were relatively unsuccessful. The data in Tables 1, 2 and 3 were obtained in a vapor-liquid equilibrium still. In each case, the starting material was the isopropyl ether–acetone azeotrope. The ratios are the parts by weight of extractive agent used per part of isopropyl ether–acetone azeotrope.

The relative volatilities are listed for each of the two ratios employed. For example, in Table 1, one part of ethylene glycol with one part of isopropyl ether–acetone azeotrope gives a relative volatility of 3.80, 6/5 parts of ethylene glycol gives 5.62. One half part of sulfolane mixed with one half part of adiponitrile with one part of isopropyl ether–acetone azeotrope gives a relative volatility of 3.98, 3/5 parts of sulfolane plus 3/5 parts of adiponitrile gives 3.08. One-third part of glycerine plus 1/3 part of DMSO plus 1/3 part of 1,4-butanediol mixed with one part of isopropyl ether–acetone azeotrope gives a relative volatility of 3.46, with 2/5 parts, these three give 3.40.

Several of the compounds and mixtures listed in Tables 1 and 2 and whose relative volatility had been determined in the vapor-liquid equilibrium still were then evaluated in a glass-perforated-plate rectification column possessing 4.5 theoretical plates. The results are listed in Table 4. The data were obtained in the following way. A solution of 460 g of acetone and 40 g of isopropyl ether was placed in the stillpot and heated. When refluxing began, an extractive agent containing pure sulfolane was pumped into the column at a rate of 20 ml/min. The temperature of the extractive

TABLE 1. EXTRACTIVE DISTILLATION AGENTS WHICH ARE EXCEPTIONALLY EFFECTIVE IN SEPARATING ACETONE FROM ISOPROPYL ETHER

Compounds	Ratios	Relative Volatilities
Ethylene glycol	1 6/5	3.80 5.62
Dimethylsulfoxide (DMSO)	1 6/5	3.78 4.92
1,6-Hexanediol	1 6/5	3.82 4.45
Adiponitrile	1 6/5	3.82 4.07
Sulfolane	1 6/5	3.09 3.80
Glycerine	1 6/5	3.13 —
Glycerine, DMSO	$(3/5)^2$	— 3.82
Glycerine, Dimethylformamide (DMFA)	$(1/2)^2(3/5)^2$	2.91 3.63
Glycerine, Sulfolane	$(1/2)^2(3/5)^2$	3.85 4.36
Glycerine, 1,6-Hexanediol	$(1/2)^2(3/5)^2$	3.26 4.42
Glycerine,3-Chloro-1,2-propanediol	$(1/2)^2(3/5)^2$	3.57 —
Ethylene glycol, DMSO	$(1/2)^2(3/5)^2$	3.14 3.31
Ethylene glycol, Sulfolane	$(1/2)^2(3/5)^2$	4.01 4.57
Ethylene glycol, 1,4-Butanediol	$(1/2)^2(3/5)^2$	3.26 3.77
Ethylene glycol, 3-Chloro-1,2-propanediol	$(1/2)^2(3/5)^2$	3.53 4.29
DMSO, Adiponitrile	$(1/2)^2(3/5)^2$	3.71 3.81
DMSO, 1,4-Butanediol	$(1/2^2(3/5)^2$	3.08 3.50
DMSO, 3-Chloro-1,2-propanediol	$(1/2)^2(3/5)^2$	2.74 4.73
Sulfolane, Adiponitrile	$(1/2)^2(3/5)^2$	3.98 3.08
Sulfolane, DMSO	$(1/2)^2(3/5)^2$	3.57 4.10
Sulfolane, Propylene glycol	$(1/2)^2(3/5)^2$	3.07 3.16
Sulfolane, Triethylene glycol	$(1/2)^2(3/5)^2$	3.07 3.10
Sulfolane, 1,6-Hexanediol	$(1/2)^2(3/5)^2$	3.56 4.34
Sulfolane, 3-Chloro-1,2-propanediol	$(1/2)^2(3/5)^2$	3.07 3.29
Glycerine, Ethylene glycol, DMSO	$(1/3)^3(2/5)^3$	4.82 4.05
Glycerine, Ethylene glycol, Dipropylene glycol	$(1/3)^3(2/5)^3$	2.59 3.69
Glycerine, Ethylene glycol, Adiponitrile	$(1/3)^3(2/5)^3$	4.04 4.60
Glycerine, Ethylene glycol, Sulfolane	$(1/3)^3(2/5)^3$	3.60 3.06
Glyercine, DMSO, Propylene glycol	$(1/3)^3(2/5)^3$	2.93 3.59
Glycerine, DMSO, 1,4-Butanediol	$(1/3)^3(2/5)^3$	3.46 3.40
Glycerine, DMSO, Tetraethylene glycol	$(1/3)^3(2/5)^3$	3.70 3.98
Glycerine, DMSO, Polyethylene glycol 300	$(1/3)^3(2/5)^3$	3.60 4.36
Glycerine, DMSO, 3-Chloro-1,2-propanediol	$(1/3)^3(2/5)^3$	3.20 3.64
Glycerine, DMSO, Diisooctyl phthalate	$(1/3)^3(2/5)^3$	3.95 3.13
Glycerine, DMSO, Adiponitrile	$(1/3)^3(2/5)^3$	4.19 4.00
Glycerine, DMSO, Sulfolane	$(1/3)^3(2/5)^3$	3.84 3.79
Ethylene glycol, DMSO, Propylene glycol	$(1/3)^3(2/5)^3$	4.03 3.66
Ethylene glycol, DMSO, 1,4-Butanediol	$(1/3)^3(2/5)^3$	2.98 3.47
Ethylene glycol, DMSO, 1,5-Pentanediol	$(1/3)^3(2/5)^3$	3.12 3.42
Ethylene glycol, DMSO, Diethylene glycol	$(1/3)^3(2/5^3$	3.21 3.34
Ethylene glycol, DMSO, Triethylene glycol	$(1/3^3)(2/5)^3$	3.04 3.83
Ethylene glycol, DMSO, Tetraethylene glycol	$(1/3)^3(2/5)^3$	3.30 3.54
Ethylene glycol, DMSO, 3-Chloro-1,2-propanediol	$(1/3)^3(2/5)^3$	3.01 3.87
Ethylene glycol, DMSO, Adiponitrile	$(1/3)^3(2/5)^3$	3.80 4.39
Ethylene glycol, DMSO, Sulfolane	$(1/3)^3(2/5)^3$	4.23 6.39
DMSO, Sulfolane, Propylene glycol	$(1/3)^3(2/5)^3$	3.17 3.53
DMSO, Sulfolane, 1,4-Butanediol	$(1/3)^3(2/5)^3$	4.88 —
DMSO, Sulfolane, Adiponitrile	$(1/3)^3(2/5)^3$	3.46 3.32
DMSO, Sulfolane, Adiponitrile, Glycerine	$(1/4)^4(1/3)^4$	4.38 4.32
DMSO, Sulfolane, Adiponitrile, Ethylene glycol	$(1/4)^4(1/3)^4$	4.53 5.04
DMSO, Sulfolane, Adiponitrile, Propylene glycol	$(1/4)^4(1/3)^4$	3.60 3.80
DMSO, Sulfolane, Adiponitrile, 1,5-Pentanediol	$(1/4)^4(1/3)^4$	2.96 3.27
DMSO, Sulfolane, Adiponitrile, 1,6-Hexanediol	$(1/4)^4(1/3)^4$	3.24 3.28
DMSO, Sulfolane, Adiponitrile, Diethylene glycol	$(1/4)^4(1/3)^4$	3.01 3.10
DMSO, Sulfolane, Adiponitrile, Triethylene glycol	$(1/4)^4(1/3)^4$	3.57 3.55
DMSO, Sulfolane, Adiponitrile, Tetraethylene glycol	$(1/4)^4(1/3)^4$	3.27 3.20
DMSO, Sulfolane, Adiponitrile, Dipropylene glycol	$(1/4)^4(1/3)^4$	2.87 3.17
DMSO, Sulfolane, Adiponitrile, 3-Chloro-1,2-propanediol	$(1/4)^4(1/3)^4$	3.16 3.38

agent as it entered the column was 52°C. After establishing the feed rate of the extractive agent, the heat input to the isopropyl ether and acetone in the stillpot was adjusted to give a total reflux rate of 10–20 ml/min. After one-half hour of operation, overhead and bottoms samples of approximately 2 ml were collected and analyzed by gas chromatography. The overhead analysis showed 91.3% isopropyl ether, 8.7% acetone. The bottoms analysis indicated 4.7% isopropyl ether, 95.3% acetone. Using these compositions in the Fenske equation, with the number of theoretical plates in the column being 4.5, gave an average relative volatility of 3.29 for each theoretical plate. After one hour of total operating time, analysis of overhead and bottoms indicated an average relative volatility of 4.53; after 1.5 hours of total operating time, these analyses gave an average relative volatility of 4.52. These data indicate that about an hour of operating time at constant conditions are required for this column to come to equilibrium.

The charge designated "blank" in Table 4 was 8% isopropyl ether, 92% acetone; after 1.5 hours of operation in the 4.5 theoretical plate column, the relative volatility of the separation between the isopropyl ether–acetone azeotrope and acetone was 1.42. The remaining data are for the extractive distillation agents designated. Here we not only have negated the azeotrope but the isopropyl ether has become the more volatile component. The temperature of the overhead approaches 63°C; the boiling point

TABLE 2. EXTRACTIVE DISTILLATION AGENTS WHICH ARE EFFECTIVE IN SEPARATING ACETONE FROM ISOPROPYL ETHER

Compounds	Ratios	Relative Volatilities
Propylene glycol	1 (6/5)	2.10 2.76
1,3-Butanediol	1 (6/5)	2.55 2.45
1,4-Butanediol	1 (6/5)	2.23 2.20
Diethylene glycol	1 (6/5)	2.38 2.15
Triethylene glycol	1 (6/5)	2.50 1.83
Tetraethylene glycol	1 (6/5)	1.97 2.51
3-Chloro-1,2-propanediol	1 (6/5)	2.62 3.13
Dimethylformamide (DMFA)	1 (6/5)	2.05 2.08
Glycerine, Propylene glycol	$(1/2)^2(3/5)^2$	2.32 3.72
Glycerine, 1,5-Pentanediol	$(1/2)^2(3/5)^2$	2.01 2.62
Ethylene glycol, Tetraethylene glycol	$(1/2)^2(3/5)^2$	2.67 2.77
DMSO, Propylene glycol	$(1/2)^2(3/5)^2$	2.68 2.74
DMSO, 1,3-Butanediol	$(1/2)^2(3/5)^2$	2.76 2.38
DMSO, 1,5-Pentanediol	$(1/2)^2(3/5)^2$	2.37 2.70
DMSO, 1,6-Hexanediol	$(1/2)^2(3/5)^2$	1.74 2.51
DMSO, Hexylene glycol	$(1/2)^2(3/5)^2$	2.01 2.04
DMSO, Diethylene glycol	$(1/2)^2(3/5)^2$	2.41 3.23
DMSO, Triethylene glycol	$(1/2)^2(3/5)^2$	2.57 2.86
DMSO, Tetraethylene glycol	$(1/2)^2(3/5)^2$	2.56 2.78
DMSO, Dipropylene glycol	$(1/2)^2(3/5)^2$	2.36 2.45
DMSO, 3-Chloro-1,2-propanediol	$(1/2)^2(3/5)^2$	2.52 2.87
DMSO, Benzyl alcohol	$(1/2)^2(3/5)^2$	2.19 3.00
Sulfolane, 1,3-Butanediol	$(1/2)^2(3/5)^2$	2.79 2.99
Sulfolane, 1,4-Butanediol	$(1/2)^2(3/5)^2$	2.83 3.05
Sulfolane, 1,5-Pentanediol	$(1/2)^2(3/5)^2$	2.56 2.74
Sulfolane, Diethylene glycol	$(1/2)^2(3/5)^2$	2.93 2.82
Sulfolane, Tetraethylene glycol	$(1/2)^2(3/5)^2$	2.87 2.93
Sulfolane, Dipropylene glycol	$(1/2)^2(3/5)^2$	2.47 2.85
Sulfolane, Phenol	$(1/2)^2(3/5)^2$	2.32 2.63
DMFA, 1,3-Butanediol	$(1/2)^2(3/5)^2$	2.52 2.55
DMFA, 3-Chloro-1,2-propanediol	$(1/2)^2(3/5)^2$	2.29 2.38
Sulfolane, 1,6-Hexanediol	$(1/2)^2(3/5)^2$	2.51 2.83
Sulfolane, Hexylene glycol	$(1/2)^2(3/5)^2$	2.11 2.16
Glycerine, Ethylene glycol, Propylene glycol	$(1/3)^3(2/5)^3$	2.65 2.96
Glycerine, Ethylene glycol, 1,5-Pentanediol	$(1/3)^3(2/5)^3$	2.26 3.52
Glycerine, Ethylene glycol, Diethylene glycol	$(1/3)^3(2/5)^3$	2.03 3.35
Glycerine, Ethylene glycol, Triethylene glycol	$(1/3)^3(2/5)^3$	2.32 3.76
Glycerine, Ethylene glycol, Formamide	$(1/3)^3(2/5)^3$	2.48 2.98
Glycerine, DMSO, Propylene glycol	$(1/3)^3(2/5)^3$	2.97 2.13
Glycerine, DMSO, 1,3-Butanediol	$(1/3)^3(2/5)^3$	2.95 1.95
Glycerine, DMSO, 1,4-Butanediol	$(1/3)^3(2/5)^3$	2.84 3.05
Glycerine, DMSO, 1,5-Pentanediol	$(1/3)^3(2/5)^3$	2.56 3.40
Glycerine, DMSO, 1,6-Hexanediol	$(1/3)^2(2/5)^3$	2.37 2.93
Glycerine, DMSO, Hexylene glycol	$(1/3)^3(2/5)^3$	2.44 2.70
Glycerine, DMSO, Triethylene glycol	$(1/3)^3(2/5)^3$	2.33 2.00
Glycerine, DMSO, Diethylene glycol	$(1/3)^3(2/5)^3$	2.60 3.37
Glycerine, DMSO, Dipropylene glycol	$(1/3)^3(2/5)^3$	2.40 2.76
Glycerine, DMSO, 3-Chloro-1,2-propanediol	$(1/3)^3(2/5)^3$	2.26 2.97
Glycerine, DMSO, Nitrobenzene	$(1/3)^3(2/5)^3$	3.36 2.11
Glycerine, DMFA, 1,5-Pentanediol	$(1/3)^3(2/5)^3$	2.21 32.5
Ethylene glycol, DMSO, 1,3-Butanediol	$(1/3)^3(2/5)^3$	2.60 3.38
Ethylene glycol, DMSO, 1,6-Hexanediol	$(1/3)^3(2/5)^3$	2.33 3.09
Ethylene glycol, DMSO, Hexylene glycol	$(1/3)^3(2/5)^3$	2.42 2.65
Ethylene glycol, DMSO, Dipropylene glycol	$(1/2)^2(3/5)^2$	2.45 3.18
Ethylene glycol, DMSO, Polyethylene glycol 300	$(1/3)^3(2/5)^3$	2.73 2.90
Propylene glycol, DMSO, 3-Chloro-1,2-propanediol	$(1/3)^3(2/5)^3$	2.38 2.19
DMSO, Sulfolane, Adiponitrile, 1,4-Butanediol	$(1/4)^4(1/3)^4$	2.92 2.82
DMSO, Sulfolane, Adiponitrile, Hexylene glycol	$(1/4)^4(1/3)^4$	2.32 2.49

TABLE 3. EXTRACTIVE DISTILLATION AGENTS WHICH ARE RELATIVELY INEFFECTIVE IN SEPARATING ACETONE FROM ISOPROPYL ETHER

Compounds	Ratios	Relative Volatilities
1,5-Pentanediol	1 6/5	1.82 1.79
Hexylene glycol	1 6/5	1.24 1.38
Glycerine, Ethylene glycol	$(1/2)^2(3/5)^2$	0.90 0.73
Glycerine, 1,4-Butanediol	$(1/2)^2(3/5)^2$	1.91 1.52
Glycerine, 1,5-Pentanediol	$(1/2)^2(3/5)^2$	2.31 1.52
Glycerine, Tetraethylene glycol	$(1/2)^2(3/5)^2$	2.73 1.18
DMSO, Diisooctyl phthalate	$(1/2)^2(3/5)^2$	1.61 1.80
1,5-Pentanediol, Tetraethylene glycol	$(1/2)^2(3/5)^2$	2.12 1.84
3-Chloro-1,2-propanediol, Nitrobenzene	$(1/2)^2(3/5)^2$	1.43 1.89
Glycerine, Ethylene glycol, 1,4-Butanediol	$(1/3)^3(2/5)^3$	2.26 1.25
Glycerine, Ethylene glycol, Diisobutyl phthalate	$(1/3)^3(2/5)^3$	1.96 0.94

TABLE 4. DATA FROM RUN MADE IN RECTIFICATION COLUMN

Compounds	Overhead Temp., °C.	Stillpot Temp., °C at start	After 1.5 h	Relative Volatility	Time to Reach Equil., h
Blank	49	53	53	1.42	0.5
DMSO	63	54	86	5.97	1
DMSO (*R*)	63	54	84	5.90	1
Adiponitrile	62	54	85	5.43	1
Sulfolane	62	54	84	4.52	1
Sulfolane (*R*)	59	55	84	4.43	1
Glycerine	49	53	69	1.67	0.5
Ethylene glycol	56	54	77	2.72	1
DMSO + Adiponitrile	62	54	87	4.89	1
DMSO (*R*) + Sulfolane (*R*)	62	54	84	4.62	1
Sulfolane (*R*) + Adiponitrile (*R*)	61	55	82	4.27	1
DMSO + Glycerine	57	56	70	2.89	1
Sulfolane + Glycerine	56	55	76	2.74	1
DMSO (*R*) + Sulfolane (*R*) + Adiponitrile (*R*)	61	55	83	4.06	1
DMSO (*R*) + Sulfolane (*R*) + Glycerine (*R*)	60	55	76	3.19	1

of pure isopropyl ether at 630 mm Hg (84 kPa); and the acetone goes to the stillpot with the extractive distillation agent. The designation *R* by the extractive distillation agent means that the same material was recovered and reused to show its stability in repeated operation. When the acetone–extractive distillation agent mixture taken from the stillpot is redistilled, acetone comes off in the usual way at its normal boiling point, 56°C.

The results obtained in the rectification column and presented in Table 4 show that vapor-liquid equilibrium data obtained in the Othmer still can be successfully used to demonstrate the effectiveness of extractive distillation in the separation of pure isopropyl ether from acetone.

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Solventing out of Electrolytes from their Aqueous Solution

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INTRODUCTION

It has been found that miscible organic solvents can precipitate a large fraction of some electrolytes from saturated aqueous solutions. Hull and Owens (1975) showed that KI and KIO_3 can be separated in an aqueous solution by an addition of large amount of 1,4 dioxane which precipitates KIO_3 while the KI remains in solution. A year later, Alfassi and Feldman (1976) used acetone to separate KBr and $KBrO_3$ to get carrier-free radiobromide. Mosseri and Alfassi (1982) studied the separation of the KX-KXO_3-KXO_4 system (when X = Cl, Br or I) by using several miscible organic solvents. Further study has been done also by Alfassi (1979).

The precipitation of salts from aqueous solution using miscible organic solvents can be named "solventing out" process by being of a similar origin as the reverse process "salting out" summarized by King (1969), where the addition of electrolytes is used to remove water from miscible organic solvents. In both cases there is a competition between the electrolytes and the organic solvents molecules on the water molecules.

This study was done to point out the possibility of using low

TABLE 1. PHYSICAL AND THERMODYNAMIC DATA FOR WATER AND LOW BOILING POINT MISCIBLE ORGANIC SOLVENT. HEAT EVAPORATION ΔH_v FROM DEAN (1979) BOILING POINTS AND DENSITY FROM WEAST (1974)

Solvent	Temp. Boiling Point (°C) at 760 mm Hg	Pres. Boiling Point (mm Hg) at 20°C*	ΔH_v kcal/mol	Density g/cm^3 at 20°C	ΔH_v kcal/L
Water	100	17.54	9.945	1.00	552.5
Acetone	56.2	184.80	6.952	0.7899	94.5
Methanol	64.96	92.68	8.240	0.7914	203.5
Propylamine	47.8	258.90		0.7173	
Isopropylamine	32.4	473.32		0.8889	

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* Approximated value based on correlation from Weast (1974).