The 1998
National Student
Design Competition

American Institute of
Chemical Engineers

If there are any questions about the design problem, Student Chapter Advisors and design course instructors are asked to contact:

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Please read the rules on the following pages carefully before submitting a solution to AIChE.

AMERICAN INSTITUTE OF CHEMICAL ENGINEERS
345 East 47th Street, New York, NY 10017-2395

AIChE NATIONAL STUDENT DESIGN COMPETITION
1998

Solvent Recycle From a Siloxane Process

DEADLINE FOR MAILING:
Solutions must be postmarked no later than midnight, June 5, 1998.

RULES OF THE CONTEST

Solutions will be graded on (a) substantial correctness of results and soundness of conclusions, (b) ingenuity and logic employed, (c) accuracy of computations, and (d) form of presentation. Accuracy of computations is intended to mean primarily freedom from mistakes; extreme precision is not necessary.

It is to be assumed that the statement of the problem contains all the pertinent data for those available in handbooks and literature references. The use of textbooks, handbooks, journal articles, and lecture notes is permitted.

Students may use any available commercial or library computer programs in preparing their solutions. Students are warned, however, that physical property data built into such programs may differ from data given in the problem statement. In such cases, as with data from other literature sources, values given in the problem statement are most applicable. Students using commercial or library computer programs or other solution aids should so state in their reports and include proper references and documentation. Judging, however, will be based on the overall suitability of the solutions, not on skills in manipulating computer programs.

The 1998 National Student Design Competition is designed to be solved either by an individual chemical engineering student working entirely alone, or a group of no more than three students working together. Solution will be judged in two categories: individual and team. There are, however, other academically sound approaches to using the problem, and it is expected that some Advisors will use the problem as classroom material. The following confidentiality rules therefore apply:

1. For individual students or teams whose solutions may be considered for the contest:
The problem may not be discussed with anyone (students, faculty, or others, in or out of class) before or during the period allowed for solutions. Discussion with faculty and students at that college or university is permitted only after complete final reports have been submitted to the Chapter Advisor.

2. For students whose solutions are not intended for the contest:
Discussion with faculty and with other students at that college or university who are not participating in the contest is permitted.

3. For all students:
The problem may not be discussed with students or faculty from other colleges or universities, or with individuals in the same institution who are still working on the problem for the contest, until after June 5, 1998. This is particularly important in cases where neighboring institutions may be using different schedules.

Submission of a solution for the competition implies strict adherence to the following conditions:

(Failure to comply will result in solutions being returned to the appropriate Faculty Advisor for revision. Revised submissions must meet the original deadline.)

ELIGIBILITY

- ✓ ONLY AIChE NATIONAL STUDENT MEMBERS MAY SUBMIT A SOLUTION. Non-member entries will not be considered.
- ✓ Entries may be submitted either by individuals or by teams of no more than three students. Each team member must meet all eligibility requirements.
- ✓ Each Faculty Advisor should select the best solution or solutions, not to exceed two from each category (individual and team), from his or her chapter and send these by registered mail, as per the below instructions, to the Institute.

TIMELINE FOR COMPLETING THE SOLUTION

- ⌛ A period of no more than thirty days is allowed for completion of the solution. This period may be selected at the discretion of the individual advisor, but in order to be eligible for an award, a solution must be postmarked no later than midnight, June 5, 1998.
- ⌛ **The finished report must be submitted to the Faculty Advisor WITHIN THE 30-DAY PERIOD.**

REPORT FORMAT

- ✎ The body of the report must be suitable for reproduction, that is, typewritten or computer-generated. Tables may be written in ink. Supporting calculations and other appendix material may be in pencil.
- ✎ The solution itself must bear no reference to the students' names or institution by which it might be identified. In this connection, graph paper bearing the name of the institution should not be used.

SENDING THE SOLUTION TO AIChE

- 📪 Two copies of each of the solution(s) must be sent to the address below; original manuscript(s) must remain in the possession of the Student Chapter Advisor, or Faculty Advisor, sponsoring the student(s)
- 📪 There should not be any variation in form of content between the solution submitted to the Faculty Advisor and that sent to the AIChE office.
- 📪 Each copy must be accompanied by the enclosed ENTRY FORM giving each contestant's name, AIChE membership number, college or university, Faculty Advisor name, address, home address, home telephone number, and student chapter, lightly attached to the report. This form will be retained for identification by the executive director of the Institute.
- 📪 DEADLINE: Entries must be postmarked no later than midnight, June 5, 1998. As soon as the winners have been notified, original manuscripts must be forwarded to the office of the executive director as soon as possible.

SEND TO:

Coordinator, AIChE Awards
American Institute of Chemical Engineers
345 East 47th Street
New York, New York 10017-2395

DEADLINE: JUNE 5, 1998

SOLVENT RECYCLE FROM A SILOXANE PROCESS

I. OBJECTIVE AND SCOPE

Waste minimization has become increasingly important in chemical processing due to rising disposal costs and regulations. A future siloxane unit will require acetonitrile and toluene as processing aids. Total disposal of these solvents is environmentally and economically unacceptable. You are asked to design a solvent separation process for recycling these solvents back into the siloxane polymerization unit. In addition to minimizing waste and optimizing economics, the design should include a safety risk assessment.

The following should be provided with the solution in the format specified:

A. Title page.

B. Table of contents.

C. Executive summary - one to two page condensation of the report.

D. Introduction - restatement of the problem including background and objectives.

E. Discussion - include what alternatives were considered, the advantages / disadvantages of each alternative, results (tables and graphs), conclusions and recommendations. **All results and supporting calculations must be submitted in the following units (kg, kmol, °C, kJ, kPa, m, hr).**

F. Process - provide a detailed description of the process flow scheme, the function of major equipment, stream connectivity, cost savings, safety and other design features.

G. Equipment summary - provide a list of all the equipment used in the process, including equipment type and description, operating conditions, sizes, duties, estimated purchase cost, and all important specifications.

H. Utilities summary - provide a table showing duty, consumption and cost of utilities for each piece of equipment. Show also the total consumption and cost for each utility category.

I. Process flow diagram (PFD) - show a flow scheme with all major process equipment, and with a stream number for each major process stream.
Show the basic process control instrumentation including points of

measurement for temperatures, pressures, and flow rates, and the associated control devices.

J. Mass and energy balance - provide a mass and energy balance summary which includes heat input and coolingl duties. For each stream on the PFD, report the stream number, description, composition (weight fraction), temperature, pressure, and flow rate (overall and by individual component).

K. Safety, health, and environmental issues - address material hazards and the safety of the selected process alternative.

L. Economic analysis - include a discussion of the economic methods and analysis. Include appropriate cash flow tables.

M. Computer process simulators and other programs - the use of generally available process simulators (Aspen®, Hysys®, Chemcad®, Provision®, etc.) together with spreadsheets and student developed computer programs is acceptable.

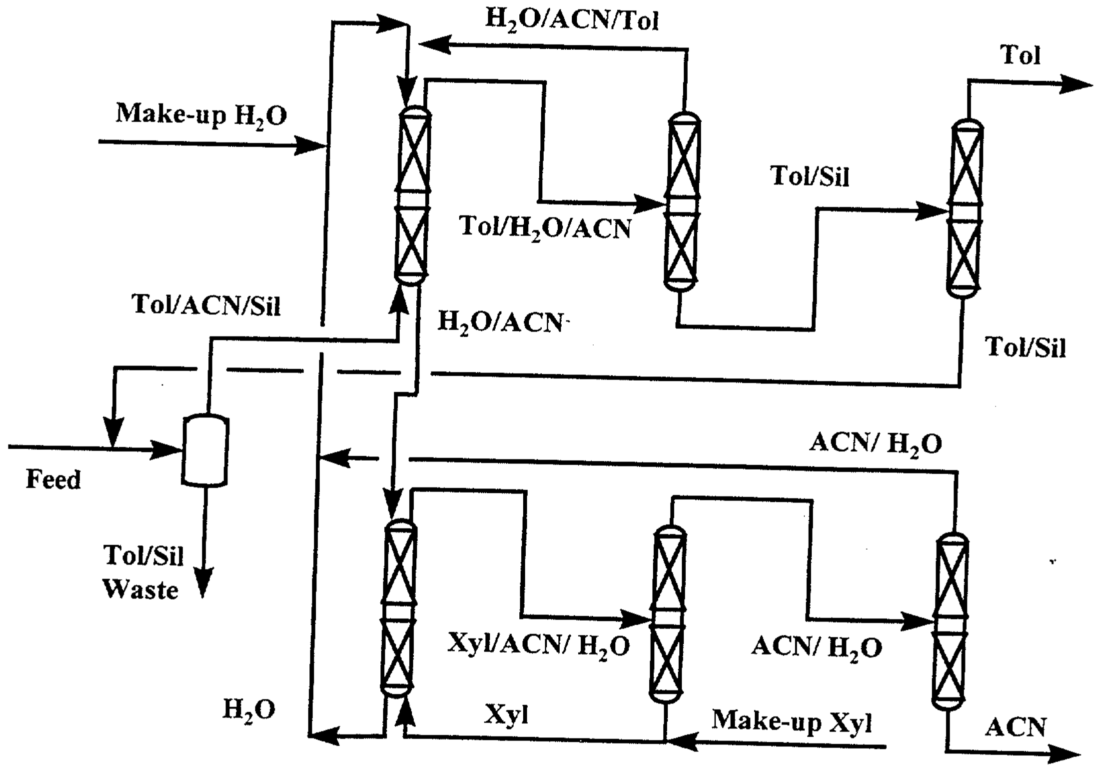
N. Back-up data and calculations - provide documentation for all hand calculations. Include flow charts, input files, and output reports (edited and condensed) from computer programs and process simulators. Provide an explanation of the computer model(s) used and the nomenclature and relevant information to familiarize the reader with the capability and acceptability of the computer program utilized. Provide assumptions and details which are not included elsewhere in the report, and include all references.

II. PROCESS DESCRIPTION AND DESIGN CONSIDERATIONS

A. A siloxane unit generates three solvent streams composed of toluene and acetonitrile. Siloxane is a minor component in these streams. While the recovery of the siloxane is beyond the scope of this problem, it must be removed prior to solvent recovery. The removal must be done in a way to keep the siloxane in solution since it is a solid in its pure form at ambient conditions. The maximum acceptable level of siloxane in the solvent(s) is 25 wt% siloxane.

B. It is desirable to recycle as much of the toluene and acetonitrile as possible, especially the acetonitrile due to the high cost of the special grade required by the process. They must be separated as components, a >99.92 wt% toluene stream and a 99.87 wt% acetonitrile stream.
Unfortunately, acetonitrile and toluene form a low pressure minimum boiling azeotrope which makes them impossible to separate to the necessary degree of purity using ordinary distillation.

C. There may be a number of ways to perform the separation, but as a start, U.S. patent 3,281,450 on the purification of acetonitrile can be considered. One possible scheme of a system using this patent would be to separate the acetonitrile from the toluene by extraction using water as solvent, and then use the patented process to recover pure acetonitrile from the water.



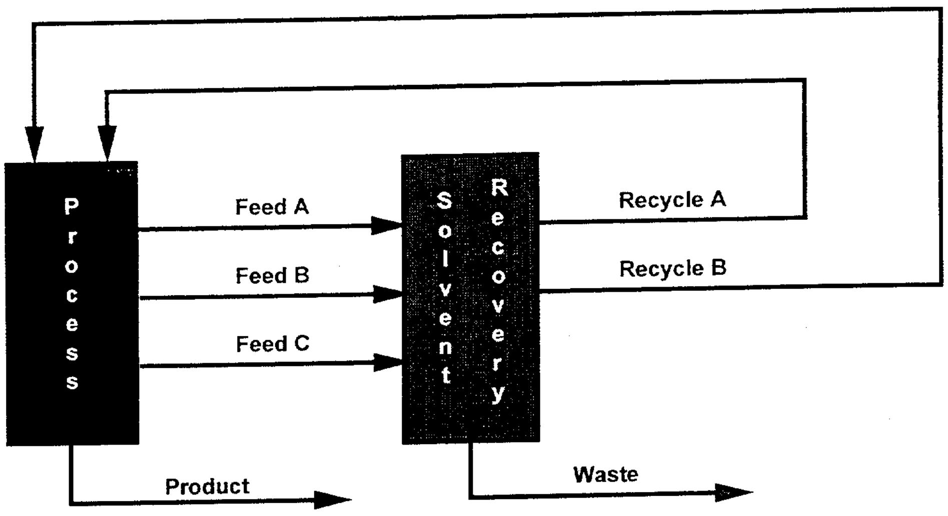
Legend:

Tol is toluene
ACN is acetonitrile
Xyl is p-xylene
Sil is siloxane

III. STREAM SPECIFICATIONS

A simplified schematic and table is shown below depicting the desired outcome of the project.

FLOW SCHEMATIC



Feed Stream Information						
Stream	Flow Rate (kg/hr)	Composition (wt%)				
		Toluene	Acetonitrile	Siloxane	Water	p-Xylene
Feed A	270	98.50		1.50		
Feed B	60	96.50	2.00	1.50		
Feed C	200	19.50	78.50	2.00		
Recycle Stream Information						
Stream	Purch. Cost ($/kg)	Composition (wt%)				
		Toluene	Acetonitrile	Siloxane	Water	p-Xylene
Recycle A	1.90	> 99.92	< 0.05	< 0.01	< 0.01	< 0.01
Recycle B	3.30	< 0.10	> 99.87	< 0.01	< 0.01	< 0.01
Waste Stream Information						
Waste	Incin. Cost ($/kg)	Composition (wt%)				
		Toluene	Acetonitrile	Siloxane	Water	p-Xylene
Option A	0.50		< 20	< 1	< 5	
Option B	1.50		> 20	> 1	> 5	

IV. TECHNICAL DATA

A. Tables 1 through 4 contain the available technical data. General physical properties and temperature dependent data are listed in Tables 1 and 2, respectively. For vapor-liquid equilibrium, the gamma-phi method[1] applies (i.e., $\hat{\phi}_i y_i P = \gamma_i x_i \varphi_i^{sat} P_i^{sat} P_f$). At low to moderate pressures, the ratio, $\phi_i^{sat} P_f / \hat{\varphi}_i$, may be taken as unity while for higher pressures, the Redlich-Kwong equation of state (Table 3) is required. Activity coefficients are to be calculated from the Non-Random-Two-Liquid (NRTL) model. Table 4 contains the interaction parameters, A_{ji}, and the solution *nonrandomness* parameters, α_{ji} to be used in the model. These parameters may be assumed to hold up to 2600 kPa. As it is assumed that the siloxane exhibits ideal solution behavior and does not affect solution excess properties, no parameters are given for this compound. The NRTL model applies for liquid-liquid equilibrium; however, graphical presentation of liquid-liquid equilibrium data, such as the ternary system: acetonitrile/p-xylene/water, is also available (U.S. patent 3,281,450).

Table 1. Physical Properties

	Acetonitrile **(C_2H_3N)**	**Toluene** **(C_7H_8)**	**p-Xylene** **(C_8H_{10})**	**Water** **(H_2O)**	**Siloxane**
Molecular Weight	41.05	92.14	106.17	18.02	593.23
Normal Boiling Pt. (°C)	81.65	110.65	138.36	100.00	303.2
Melting Pt. (°C)	-43.83	-94.97	13.26	0.00	31.5
Critical Temperature (°C)	272.35	318.65	343.11	374.15	416.05
Critical Pressure (kPa)	4820	4100	3510	22120	691
Critical Volume (m^3/kmol)	0.173	0.316	0.378	0.0559	1.856
Acentric Factor	0.327	0.260	0.326	0.344	0.869
Density, 25 °C (kg/m^3)	782	870	864	998	1177
Heat of Fusion (kJ/kmol)					(solid) 6.32 x 10^4

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[1] Smith, J.M.; Van Ness, H.C.; Abbott, M.M. *Introduction to Chemical Engineering Thermodynamics*. 5th edition: McGraw-Hill (1996).

Table 2. Temperature Dependent Properties

		Acetonitrile **(C_2H_3N)**	**Toluene** **(C_7H_8)**	**p-Xylene** **(C_8H_{10})**	**Water** **(H_2O)**	**Siloxane**
Vapor Pressure, P^{sat} (kPa)		$\ln P^{sat} = A + \frac{B}{T+C} + D\ln T + ET^F$				
	A	67.81	76.45	91.25	65.93	113.29
	B	-6321	-6995	-8121	-7228	-13889
	C	0	0	0	0	0
	D	-7.854	-9.163	-11.32	-7.177	-13.328
	E	5.898×10^{-6}	6.225×10^{-6}	7.306×10^{-6}	4.031×10^{-6}	4.646×10^{-18}
	F	2	2	2	2	6
Heat of Vaporization, H^v (kJ/kmol)		$A(1-T_r)^{(B+C\,T_r+D\,T_r^2+E\,T_r^3)}$				
	A	4.351×10^4	5.014×10^4	5.374×10^4	5.205×10^4	1.683×10^5
	B	3.477×10^{-1}	3.859×10^{-1}	3.656×10^{-1}	3.199×10^{-1}	4.118
	C	0	0	0	-2.12×10^{-1}	-9.075
	D	0	0	0	2.580×10^{-1}	7.368
	E	0	0	0	0	0
Ideal Gas Heat Capacity, c_p^{ig} (kJ/kmol-K)		$A + B\left[\frac{C/T}{\sinh(C/T)}\right]^2 + D\left[\frac{E/T}{\cosh(E/T)}\right]^2$ $A + BT + CT^2 + DT^3 + ET^4$ (second expression applies to siloxane only)				
	A	41.914	58.140	75.120	33.363	-15.204
	B	88.760	2.863×10^2	3.397×10^2	26.790	2.713
	C	1581	1440.6	1492.8	2610.5	-1.632×10^{-3}
	D	50.320	1.898×10^2	2.247×10^2	8.896	3.63×10^{-7}
	E	699.8	-650.43	-675.1	1169	0

T is expressed in Kelvin

T_r is the reduced temperature, T/T_c

Table 3. Equation of State and Fugacity Coefficients

Redlich-Kwong

$$P = \frac{RT}{V-b} - \frac{a}{V\sqrt{T}(V+b)}$$

$$\ln \hat{\phi}_i = \frac{b_i}{b}(z-1) - \ln\left[z\left(1-\frac{b}{V}\right)\right] + \frac{1}{bRT^{1.5}}\left[\frac{ab_i}{b} - 2\sqrt{aa_i}\right]\ln\left(1+\frac{b}{V}\right)$$

$$a = \left(\sum_i y_i \sqrt{a_i}\right)^2$$

$$b = \sum_i y_i b_i$$

$$a_i = 0.42748\, R^2 T_c^{2.5} \,/\, P_c$$

$$b_i = 0.08664\, R\, T_c \,/\, P_c$$

Table 4. Activity Coefficients and Interaction Parameters

Non-Random-Two-Liquid (NRTL)

$$\ln\gamma_i = \frac{\sum_j \tau_{ji} G_{ji} x_j}{\sum_k G_{ki} x_k} + \sum_j \frac{x_j G_{ij}}{\sum_m G_{mj} x_m}\left(\tau_{ij} - \frac{\sum_n x_n \tau_{nj} G_{nj}}{\sum_{k=1} G_{kj} x_k}\right)$$

$$\tau_{ji} = \frac{A_{ji}}{RT} \qquad G_{ji} = e^{-\alpha_{ji}\tau_{ji}} \qquad R = 1.987\ cal/mol - K$$

A_{ji}

Row ≡*j*	Column ≡ *i* Acetonitrile	Toluene	Water	p-Xylene
Acetonitrile	0	790	1100	1413
Toluene	724	0	5680	226
Water	1217	4375	0	13872
p-Xylene	-210	-242	-2456	0

α_{ji}

Row ≡*j*	Column ≡ *i* Acetonitrile	Toluene	Water	p-Xylene
Acetonitrile	0	0.9353	0.5654	0.294
Toluene	0.9353	0	0.2	0.287
Water	0.5654	0.2	0	0.3
p-Xylene	0.294	0.287	0.3	0

I. PROCESS DESIGN GUIDELINES:

A. INVENTORY - Provide storage for all isolated streams entering and leaving the process. (A minimum of 24 hour surge capacity is required. However, more capacity should be provided where a higher risk to process on-line time is an issue.) Waste streams are to be disposed by 19,000 liter (or 5,000 gallon) trailer.

B. MATERIALS OF CONSTRUCTION - 316SS is required as a material of construction for process piping and equipment. Carbon steel is acceptable for utility service.

C. EQUIPMENT RATINGS - A minimum of 345 kPa (or 50 psig) pressure rating is required for all pressure vessels (see below section "SAFETY, HEALTH, AND ENVIRONMENTAL CONSIDERATIONS" for more information).

D. PROCESS STRUCTURE - Document the overall size and cost of the process structure needed to safely support the process, and list any applicable assumptions used (e.g. cost per square meter of structure area).

E. Toxic air contaminant vent load is not to exceed 277 kg/month (or 500 lb/month). (acetonitrile, toluene, p-xylene, and other volatile organic carbon compounds are considered toxic air contaminants.)

F. Cooling tower water, high pressure steam, low pressure steam, low temperature heat transfer fluid (Syltherm XLT®), high temperature heat transfer fluid (Syltherm 800®), nitrogen, air, and electricity will be available as plant utilities.

G. Assume the following for design:

Cooling tower water: 35°C supply temperature, max delta 10°C

High pressure steam: 1034 kPa (or 150 psig) supply pressure

Low pressure steam: 172 kPa (or 25 psig) supply pressure

Low temperature heat transfer fluid (Syltherm XLT®): -29°C supply temperature, maximum temperature rise 10°C

High temperature heat transfer fluid (Syltherm 800®): 320°C supply temperature

Plant nitrogen: 689 kPa (or 100 psig) supply pressure

Plant air: 689 kPa (or 100 psig) supply pressure

II. SAFETY, HEALTH, ENVIRONMENTAL DESIGN GUIDELINES

A. PROCESS PRESSURE ANALYSIS - An analysis should be done to determine the maximum pressure that is likely to occur under normal conditions, startup, shutdown, turnaround, clean-out, maintenance, upset, utility failure, etc. Wherever practical, equipment and piping should be designed to withstand this pressure to minimize the risk of a release of hazardous materials to the environment. In cases where this would incur excessive costs, special measures should be taken to contain overpressure vents prior to equipment or piping failure and impact to the environment. The analysis should include a listing of the Maximum Allowable Working Pressure (MAWP) and any overpressure protection systems for key equipment.

B. FIRE PROTECTION - The project cost estimate should include an adequate allowance for fire protection of the process structure. Protection should be provided to a minimum of 10.7 meters (35 feet) above grade.

C. HEALTH CONSIDERATIONS - The project cost estimate should include an adequate allowance for health protection systems. Some include eye wash / safety showers (to be provided at a minimum of every 15.2 meters (50 feet) walking distance in all areas where there is a possibility of exposure to hazardous substances), hearing protection, and ventilation.

D. SPILL CONTAINMENT - The project cost estimate should include an adequate allowance for spill containment. Provide a minimum containment volume of 1.5 times the volume of the largest storage vessel.

III. COST OF RAW MATERIALS, UTILITIES, AND WASTE DISPOSAL

A. Cost of Raw Materials:

1.	p-xylene	$ 1.70 per kg.	(special grade)
2.	toluene	$ 1.90 per kg.	(special grade)
3.	acetonitrile	$ 3.30 per kg.	(special grade)
4.	Water	$ 0.40 per 1000 liter	

B. Disposal Costs of Waste Streams:

1. Waste A: $ 0.50 per kg. if less than 20% acetonitrile and less than 5% water and less than 1% siloxane.

2. Waste B: $ 1.50 per kg. if greater than 20% acetonitrile or greater than 5% water or greater than 1 % siloxane.

C. Cost of Utilities:

1.	Cooling water	$ 0.03 per 1000 liter
2.	Syltherm XLT®	$19.70 per 1,000,000 kJ/hr
3.	Steam (saturated conditions):	
	a) 172 kPa	$21.10 per 1000 kg
	b) 1034 kPa	$24.40 per 1000 kg
4.	Syltherm 800®	$22.20 per 1,000,000 kJ/hr
5.	689 kPa Air	$11.30 per 1000 m^3
6.	689 kPa Nitrogen	$47.00 per 1000 m^3
7.	Electricity	$ 0.02 per 1000 kJ

IV. EQUIPMENT SIZING GUIDELINES

A. There are several references available to assist in equipment sizing. Two recommended references follow:

1. Perry, R. H., Green, D. W., Perry's Chemical Engineer's Handbook, Latest edition, McGraw-Hill.

2. Peters, M. S., Timmerhaus, K. D., Plant Design and Economics for Chemical Engineers, Latest edition, McGraw-Hill.

B. Following is a broad list of chemical processing equipment and the key parameters needed to adequately define each for initial cost estimates.

1. AGITATORS - Type, Power, Material of Construction, Pressure & Temperature rating

2. BLOWERS - Power, Volume Flow Rate, Pressure & Temperature Rating

3. COMPRESSORS - Type (Reciprocating, Centrifugal, Screw, Turboblower, etc.), Power, Volume Flow, Differential Pressure requirement., Pressure & Temperature Rating

4. DISTILLATION - Diameter, No. of trays & Spacing or Packing Height, Materials of Construction, Pressure & Temperature Rating

5. DRYER - Volume Flow, Power (if applicable), Steam Flow (if applicable), Pressure & Temperature Rating

6. FILTERS - Type, Surface Area, Material of Construction, Pressure & Temperature Rating

7. HEAT EXCHANGERS - Type, Area of Tube Surface, Material of Construction, Pressure & Temperature Rating (for Air Cooled Heat Exchangers - Outside Bare Tube Surface Area, Power, Material of Construction, Pressure & Temperature Rating)

8. PUMPS - Volume Flow Rate, Power, Material of Construction, Total Differential Head (if known or assumed)

9. REACTOR - Volume, Material of Construction, Jacket (Y/N) Pressure & Temperature Rating (for vessel and jacket)

10. PRESSURE VESSELS - Volume, Material of Construction, Pressure & Temperature Rating

11. REFRIGERATION UNIT - Duty, Temperature Range, Electrical Classification

12. VACUUM PUMPS - Type, Power, Material of Construction, Pressure & Temperature Rating

V. ECONOMIC ANALYSIS GUIDELINES

A. The separation costs must be added to the product costs using a value of 15% for the cost of capital (minimum acceptable return), and should be based on after tax discounted cash flow and a plant service life cf 20 years. Use the "Sum of the Years" depreciation method (page 25-8, Perry's Chemical Engineering Handbook,6th edition) and 40% corporate tax rate.

B. Estimate purchased equipment cost and projected capital costs using methods from Peters, M. S., Timmerhaus, K. D., Plant Design and Economics for Chemical Engineers, latest edition, McGraw-Hill, or from process simulators or other generally acceptable and properly documented sources.

C. Include in the capital estimate an open process tower and tank farm with fire protection and spill containment.

OTHER REFERENCES

1. U.S. Patent 3,281,450 , September 27, 1963.
2. Hazards of Process Chemicals:
    - Sax, N.I., <u>Dangerous Properties of Industrial Materials</u>, 6th Edition. Van Nostrand-Reinhold, New York, (1984).
    - de Renzo, D.J. (Ed.), <u>Solvents Safety Handbook</u>. Noyes Data Corporation, Park Ridge, N.J., (1986).
3. Pure Component Property Data:
    - DIPPR (AIChE): Acetonitrile, H2O, Toluene, p-Xylene
4. Syltherm® Heat Transfer Fluids: DOW CHEMICAL COMPANY, http:\www.dow.com, 800-447-4369
5. VLE and LLE Data:
    - Acetonitrile /H2O: Blackford D. S., York R. J.; Chem. Eng. Data 10, 313 (1965).
    - Acetonitrile /Toluene: Krishna S., Tripathi R. P., Rawat B. S., J. Chem. Eng. Data 25, 11 (1980).
    - Acetonitrile /p-Xylene: Dechema Vol. 1, Part 7, p. 499 Bagga O. P., Katyal R.C., Raju K.S., Chem. Eng. Data 22, Xylene, (1977).
    - Acetonitrile /Xylene/H2O: Goodrich Co. U.S. Patent 3,281,450, Sep. 27, 1963.
    - Toluene/H2O: Brown, R. L.; Wasik, S. P. J. Res. Natl. Bur. Stand., Sect. A, 78, 453, (1974).
    - p-Xylene/H2O: Guseva, A. N.; Parnov, E. I. Vestn. Mosk. Univ.; Ser. 2; Khim.; 18, 76-9, (1963).
    - p-Xylene/Toluene: Dechema Vol. 1, Part 7, p. 444 Wichterle collect., Czech. Chem. Commun., 30, 3388, (1965).
6. Horwitz, B.A., Optimize Pressure Sensitive Distillation, Chemical Engineering Progress, April, page 47, (1997).
7. Frank, T.C., Break Azeotropes with Pressure Sensitive Distillation, Chemical Engineering Progress, April, page 52 (1997).