

F DESIGN PROJECTS II

The design exercises given in this appendix are somewhat more structured than those given in Appendix E. They have been adapted from design projects set by the Institution of Chemical Engineers as the final part of the Institution's qualifying examinations for professional chemical engineers.

F.1 ETHYLHEXANOL FROM PROPYLENE AND SYNTHESIS GAS

The Project

Design a plant to produce 40,000 metric tons (tonnes)/year of 2-ethylhexanol from propylene and synthesis gas, assuming an operating period of 8,000 hours on stream.

The Process

The first stage of the process is a hydroformylation (oxo) reaction from which the main product is n-butyraldehyde. The feeds to this reactor are synthesis gas (CO/H₂ mixture) and propylene in the molar ratio 2/1, and the recycled products of isobutyraldehyde cracking. The reactor operates at 130°C and 350 bar, using cobalt carbonyl as catalyst in solution. The main reaction products are n- and isobutyraldehyde in the ratio of 4:1, the former being the required product for subsequent conversion to 2-ethylhexanol. In addition, 3% of the propylene feed is converted to propane while some does not react.

Within the reactor, however, 6% of the n-butyraldehyde product is reduced to n-butanol, 4% of the isobutyraldehyde product is reduced to isobutanol, and other reactions occur to a small extent yielding high molecular weight compounds (heavy ends) to the extent of 1% by weight of the butyraldehyde/butanol mixture at the reactor exit.

The reactor is followed by a gas-liquid separator operating at 30 bar from which the liquid phase is heated with steam to decompose the catalyst for recovery of cobalt by filtration. A second gas-liquid separator operating at atmospheric pressure subsequently yields a liquid phase of aldehydes, alcohols, heavy ends, and water, which is free from propane, propylene, carbon monoxide, and hydrogen.

This mixture then passes to a distillation column which gives a top product of mixed butyraldehydes, followed by a second column which separates the two

butyraldehydes into an isobutyraldehyde stream containing 1.3% mole n-butyraldehyde and an n-butyraldehyde stream containing 1.2% mole isobutyraldehyde.

A cracker converts isobutyraldehyde at a pass yield of 80% back to propylene, carbon monoxide, and hydrogen by passage over a catalyst with steam. After separation of the water and unreacted isobutyraldehyde the cracked gas is recycled to the hydroformylation reactor. The isobutyraldehyde is recycled to the cracker inlet. The operating conditions of the cracker are 275°C and 1 bar.

The n-butyraldehyde is treated with a 2% w/w aqueous sodium hydroxide and undergoes an aldol condensation at a conversion efficiency of 90%. The product of this reaction, 2-ethylhexanol, is separated and then reduced to 2-ethylhexanol by hydrogen in the presence of a Raney nickel catalyst with a 99% conversion rate. In subsequent stages of the process (details of which are not required), 99.8% of the 2-ethylhexanol is recovered at a purity of 99% by weight.

Feed Specifications

- i. Propylene feed: 93% propylene, balance propane.
- ii. Synthesis gas: from heavy fuel oil, after removal of sulfur compounds and carbon dioxide:

H₂ 48.6%; CO 49.5%; CH₄ 0.4%; N₂ 1.5%.

Utilities

- i. Dry saturated steam at 35 bar.
- ii. Cooling water at 20°C.
- iii. 2% w/w aqueous sodium hydroxide solution.
- iv. Hydrogen gas: H₂ 98.8%; CH₄ 1.2%.

Scope of Design Work Required

1. Process Design

- a. Prepare a material balance for the complete process.
- b. Prepare a process diagram for the plant showing the major items of equipment. Indicate the materials of construction and the operating temperatures and pressures.
- c. Prepare energy balances for the hydroformylation reactor and for the isobutyraldehyde cracking reactor.

2. Chemical Engineering Design

Prepare a chemical engineering design of the second distillation unit, i.e., for the separation of n- and isobutyraldehyde. Make dimensioned sketches of the column, the reboiler, and the condenser.

3. Mechanical Design

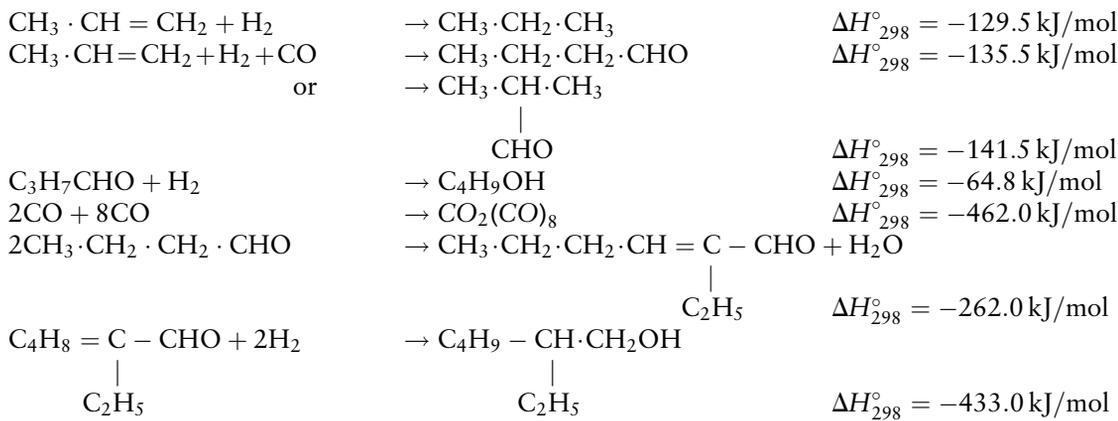
Prepare a mechanical design with sketches suitable for submission to a drawing office of the n- and isobutyraldehyde distillation column.

4. Control System

For the hydroformylation reactor prepare a control scheme to ensure safe operation.

Data

1. Reactions



2. Boiling Points at 1 bar

Propylene	- 47.7°C
Propane	- 42.1°C
n-Butyraldehyde	75.5°C
Isobutyraldehyde	64.5°C
n-Butanol	117.0°C
Isobutanol	108.0°C
2-Ethylhexanol	184.7°C

3. Solubilities of Gases at 30 Bar in the Liquid Phase of the First Gas-Liquid Separator

H ₂	0.08×10^{-3}	kg dissolved/kg liquid
CO	0.53×10^{-3}	kg dissolved/kg liquid
Propylene	7.5×10^{-3}	kg dissolved/kg liquid
Propane	7.5×10^{-3}	kg dissolved/kg liquid

4. Vapor-Liquid Equilibrium of the Butyraldehydes at 1 atm (Reference 7)

T°C	x	y
73.94	0.1	0.138
72.69	0.2	0.264
71.40	0.3	0.381
70.24	0.4	0.490
69.04	0.5	0.589
68.08	0.6	0.686
67.07	0.7	0.773
65.96	0.8	0.846
64.95	0.9	0.927

where x and y are the mole fractions of the more volatile component (isobutyraldehyde) in the liquid and vapor phases, respectively.

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F.2 CHLOROBENZENES FROM BENZENE AND CHLORINE

The Project

Design a plant to produce 20,000 metric tons/year of monochlorobenzene together with not less than 2,000 metric tons/year of dichlorobenzene, by the direct chlorination of benzene.

The Process

Liquid benzene (which must contain less than 30 ppm by weight of water) is fed into a reactor system consisting of two continuous stirred tanks operating in series at 2.4 bar. Gaseous chlorine is fed in parallel to both tanks. Ferric chloride acts as a catalyst, and is produced *in situ* by the action of hydrogen chloride on mild steel. Cooling is required to maintain the operating temperature at 328°K. The hydrogen chloride gas leaving the reactors is first cooled to condense most of the organic impurities. It then passes to an activated carbon adsorber where the final traces of impurity are removed before it leaves the plant for use elsewhere.

The crude liquid chlorobenzenes stream leaving the second reactor is washed with water and caustic soda solution to remove all dissolved hydrogen chloride. The product recovery system consists of two distillation columns in series. In the first column (the “benzene column”) unreacted benzene is recovered as top product and recycled. In the second column (the “chlorobenzene column”) the mono- and dichlorobenzenes are separated. The recovered benzene from the first column is mixed with the raw benzene feed, and this combined stream is fed to a distillation column (the “drying column”) where water is removed as overhead. The benzene stream from the bottom of the drying column is fed to the reaction system.

Feed Specifications

- i. Chlorine: 293°K, atmospheric pressure, 100% purity.
- ii. Benzene: 293°K, atmospheric pressure, 99.95 wt percent benzene, 0.05 wt percent water.

Product Specifications

- i. Monochlorobenzene: 99.7 wt percent.
- ii. Dichlorobenzene: 99.6 wt percent.
- iii. Hydrogen chloride gas: less than 250 ppm by weight benzene.

Utilities

- i. Stream: dry saturated at 8 bar and at 28 bar.
- ii. Cooling water: 293°K.
- iii. Process water: 293°K.
- iv. Caustic soda solution: 5 wt percent NaOH, 293°K.

Scope of design work required

1. Process Design

- Prepare a materials balance for the process including an analysis of each reactor stage (the kinetics of the chlorination reactions are given below). Onstream time may be taken as 330 days per year.
- Prepare energy balances for the first reactor and for the chlorobenzene column (take the reflux ratio for this column as twice the minimum reflux ratio).
- Prepare a process flow diagram for the plant. This should show the major items of equipment with an indication of the materials of construction and of the internal layout. Temperatures and pressures should also be indicated.

2. Chemical Engineering Design

Prepare a sieve-plate column design for the chlorobenzene distillation and make dimensioned sketches showing details of the plate layout including the weir and the downcomer.

3. Mechanical Design

Prepare a mechanical design of the chlorobenzene column, estimating the shell thickness, the positions and sizes of all nozzles, and the method of support for the plates and the column shell. Make a dimensioned sketch suitable for submission to a drawing office.

4. Safety

Indicate the safety measures required for this plant, bearing in mind the toxic and flammable materials handled.

Data

1. The Reactions

- $\text{C}_6\text{H}_6 + \text{Cl}_2 \rightarrow \text{C}_6\text{H}_5\text{Cl} + \text{HCl}$
- $\text{C}_6\text{H}_5\text{Cl} + \text{Cl}_2 \rightarrow \text{C}_6\text{H}_4\text{Cl}_2 + \text{HCl}$

The dichlorobenzene may be assumed to consist entirely of the para-isomer and the formation of trichlorobenzenes may be neglected.

The rate equations can be written in first-order form when the concentration of dissolved chlorine remains essentially constant. Thus:

$$\begin{aligned} r_B &= -k_1 x_B \\ r_M &= k_1 x_B - k_2 x_M \\ r_D &= k_2 x_M \end{aligned}$$

where r is the reaction rate,

k_1 is the rate constant for reaction (1) at $328^\circ\text{K} = 1.00 \times 10^{-4} \text{s}^{-1}$,
 k_2 is the rate constant for reaction (2) at $328^\circ\text{K} = 0.15 \times 10^{-4} \text{s}^{-1}$
 and x denotes mole fraction.

The subscripts B, M, and D denote benzene, monochlorobenzene, and dichlorobenzene respectively.

Yields for the reactor system should be calculated on the basis of equal liquid residence times in the two reactors, with a negligible amount of unreacted chlorine in the vapor product streams. It may be assumed that the liquid product stream contains 1.5 wt percent of hydrogen chloride:

2. Solubilities

Solubility of the water/benzene system (taken from Seidell (1941)).

Temperature (K)	293	303	313	323
g H ₂ O/100 g C ₆ H ₆	0.050	0.072	0.102	0.147
g C ₆ H ₆ /100 g H ₂ O	0.175	0.190	0.206	0.225

3. Thermodynamic and Physical Properties

	C ₆ H ₆ liquid	C ₆ H ₆ gas	C ₆ H ₅ Cl liquid	C ₆ H ₅ Cl gas	C ₆ H ₄ Cl ₂ liquid	C ₆ H ₄ Cl ₂ gas
Heat of formation at 298°K (kJ/kmol)	49.0	82.9	7.5	46.1	-42.0	5.0
Heat capacity (kJ/kmol K)						
298°K	136	82	152	92		103
350°K	148	99	161	108	193	118
400°K	163	113	170	121	238	131
450°K	179	126	181	134	296	143
500°K	200	137	192	145	366	155
Density (kg/m ³)						
298°K	872		1100			
350°K	815		1040		1230	
400°K	761		989		1170	
450°K	693		932		1100	
500°K	612		875		1020	
Viscosity (Ns/m ²)						
298°K	0.598 × 10 ⁻³		0.750 × 10 ⁻³			
350°K	0.326 × 10 ⁻³		0.435 × 10 ⁻³		0.697 × 10 ⁻³	
400°K	0.207 × 10 ⁻³		0.305 × 10 ⁻³		0.476 × 10 ⁻³	
450°K	0.134 × 10 ⁻³		0.228 × 10 ⁻³		0.335 × 10 ⁻³	
500°K	0.095 × 10 ⁻³		0.158 × 10 ⁻³		0.236 × 10 ⁻³	
Surface tension (N/m)						
298°K	0.0280		0.0314			
350°K	0.0220		0.0276		0.0304	
400°K	0.0162		0.0232		0.0259	
450°K	0.0104		0.0177		0.0205	
500°K	0.0047		0.0115		0.0142	

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F.3 METHYL ETHYL KETONE FROM BUTYL ALCOHOL

The Project

Design a plant to produce 1×10^7 kg/year of methyl ethyl ketone (MEK).

Feedstock: Secondary butyl alcohol.

Services available:

Dry saturated steam at 140°C.

Cooling water at 24°C.

Electricity at 440 V three-phase 50 Hz.

Flue gases at 540°C.

The Process

The butyl alcohol is pumped from storage to a steam-heated preheater and then to a vaporizer heated by the reaction products. The vapor leaving the vaporizer is heated to its reaction temperature by flue gases that have previously been used as reactor heating medium. The superheated butyl alcohol is fed to the reaction system at 400°C to 500°C where 90% is converted on a zinc oxide–brass catalyst to methyl ethyl ketone, hydrogen, and other reaction products. The reaction products may be treated in one of the following ways:

- a. Cool and condense the MEK in the reaction products and use the exhaust gases as a furnace fuel.
- b. Cool the reaction products to a suitable temperature and separate the MEK by absorption in aqueous ethanol. The hydrogen off gas is dried and used as a furnace fuel. The liquors leaving the absorbers are passed to a solvent extraction column, where the MEK is recovered using trichlorethane. The raffinate from this column is returned to the absorber and the extract is passed to a distillation unit where the MEK is recovered. The trichlorethane is recycled to the extraction plant.

Scope of Design Work Required

1. Prepare material balances for the two processes.
2. On the basis of the cost data supplied below, decide which is the preferable process.
3. Prepare a material flow diagram of the preferred process.

4. Prepare a heat balance diagram of the preheater–vaporizer–superheater–reactor system.
5. Prepare a chemical engineering design of the preheater–vaporizer–superheater–reactor system and indicate the type of instrumentation required.
6. Prepare a mechanical design of the butyl alcohol vaporizer and make a dimensioned sketch suitable for submission to a drawing office.

Data

Process Data

Outlet condenser temperature = 32°C.

Vapor and liquid are in equilibrium at the condenser outlet.

Calorific value of MEK = 41,800 kJ/kg.

Cost Data

Selling price of MEK = \$2.0 per kg

Reactor Data

The “shortcut” method proposed in Reference 1 may be used only to obtain a preliminary estimate of the height of catalyst required in the reactor. The reactor should be designed from first principles using the rate equation, below, taken from Reference 1:

$$r_A = \frac{C(P_{A,i} - P_{K,i}P_{H,i}/K)}{P_{K,i}(1 + K_A P_{A,i} + K_{AK} P_{A,i}/P_{K,i})}$$

where $P_{A,i}$, $P_{H,i}$, and $P_{K,i}$ are the interfacial partial pressures of the alcohol, hydrogen, and ketone in bars, and the remaining quantities are as specified by the semi-empirical equations below:

$$\log_{10} C = -\frac{5964}{T_i} + 8.464$$

$$\log_{10} K_A = -\frac{3425}{T_i} + 5.231$$

$$\log_{10} K_{AK} = +\frac{486}{T_i} - 0.1968$$

In these equations, the interfacial temperature T_i is in Kelvin, the constant C is in $\text{kmol/m}^2\text{h}$, K_A is in bar^{-1} , and K_{AK} is dimensionless.

The equilibrium constant, K , is given in Reference 1 (although the original source is Reference 2) by the equation:

$$\log_{10} K = -\frac{2790}{T_i} + 1.510 \log_{10} T_i + 1.871$$

where K is in bar.

Useful general information will be found in Reference 3.

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F.4 ACRYLONITRILE FROM PROPYLENE AND AMMONIA

The Project

Design a plant to produce 1×10^8 kg/year of acrylonitrile ($\text{CH}_2\text{:CH.CN}$) from propylene and ammonia by the ammoxidation process.

Feedstock:

Ammonia: 100% NH_3 .

Propylene: Commercial grade containing 90% C_3H_6 , 10% paraffins, etc., which do not take any part in the reaction.

Services available:

Dry saturated steam at 140°C .

Cooling water at 24°C .

Other normal services.

The Process

Propylene, ammonia, steam, and air are fed to a vapor-phase catalytic reactor (item A). The feed stream composition (molar percent) is propylene 7; ammonia 8; steam 20; air 65. A fixed-bed reactor is employed using a molybdenum-based catalyst at a temperature of 450°C , a pressure of 3 bar absolute, and a residence time of 4 seconds. Based upon a pure propylene feed, the carbon distribution by weight in the product from the reactor is

Acrylonitrile	58%
Acetonitrile	2%
Carbon dioxide	16%
Hydrogen cyanide	6%
Acrolein	2%
Unreacted propylene	15%
Other byproducts	1%

The reactor exit gas is air-cooled to 200°C and then passes to a quench scrubber (B) through which an aqueous solution containing ammonium sulfate 30 wt percent and sulfuric acid 1 wt percent is circulated. The exit gas temperature is thereby reduced to 90°C .

From the quench scrubber (B) the gas passes to an absorption column (C) in which the acrylonitrile is absorbed in water to produce a 3 wt percent solution. The carbon

dioxide, unreacted propylene, oxygen, nitrogen, and unreacted hydrocarbons are not absorbed and are vented to atmosphere from the top of column (C).

The solution from the absorber (C) passes to a stripping column (D) where acrylonitrile and lower boiling impurities are separated from water. Most of the aqueous bottom product from the stripping column (D), which is essentially free of organics, is returned to the absorber (C), the excess being bled off. The overhead product is condensed, and the aqueous lower layer returned to the stripping column (D) as reflux.

The upper layer which contains, in addition to acrylonitrile, hydrogen cyanide, acrolein, acetonitrile, and small quantities of other impurities, passes to a second reactor (E) where, at a suitable pH, all the acrolein is converted to its cyanohydrin. (Cyanohydrins are sometimes known as cyanhydrins.) The product from the reactor (E) is fed to a cyanohydrin separation column (F), operating at reduced temperature and pressure, in which acrolein cyanohydrin is separated as the bottom product and returned to the ammoxidation reactor (A) where it is quantitatively converted to acrylonitrile and hydrogen cyanide.

The top product from column (F) is fed to a stripping column (G) from which hydrogen cyanide is removed overhead.

The bottom product from column (G) passes to the hydroextractive distillation column (H). The water feed rate to column (H) is five times that of the bottom product flow from column (G). It may be assumed that the acetonitrile and other byproducts are discharged as bottom product from column (H) and discarded. The overhead product from column (H), consisting of the acrylonitrile water azeotrope, is condensed and passed to a separator. The lower aqueous layer is returned to column (H).

The upper layer from the separator is rectified in a column (I) to give 99.95 wt percent pure acrylonitrile.

Scope of design work required

1. Prepare a material balance for the process.
2. Prepare a material flow diagram of the process.
3. Prepare a heat balance for the reactor (A) and quench column (B).
4. Prepare a chemical engineering design of reactor (A) and either column (B) OR column (D).
5. Prepare a mechanical design of the condenser for stripping column (D) and make a dimensioned sketch suitable for submission to a drawing office.
6. Indicate the instrumentation and safety procedure required for this plant bearing in mind the toxic and flammable materials being handled.

REFERENCES

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F.5 UREA FROM AMMONIA AND CARBON DIOXIDE

The Project

A plant is to be designed for the production of 300,000 kg per day of urea by the reaction of ammonia and carbon dioxide at elevated temperature and pressure, using a total-recycle process in which the mixture leaving the reactor is stripped by the carbon dioxide feed (DSM process, References 1 to 4).

Materials Available

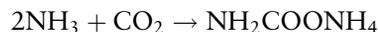
1. Liquid ammonia at 20°C and 9 bar, which may be taken to be 100% pure.
2. Gaseous carbon dioxide at 20°C and atmospheric pressure, also 100% pure.

All normal services are available on site. In particular, electricity, 440-V three-phase 50 Hz; cooling water at a maximum summer temperature of 22°C; steam at 40 bar with 20°C of superheat.

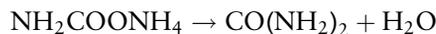
The on-stream time is to be 330 days/year, and the product specification is fertilizer-grade urea prills containing not more than 1.0% biuret.

The Process

The reaction that produces urea from ammonia and carbon dioxide takes place in two stages; in the first, ammonium carbamate is formed:



In the second, the carbamate is dehydrated to give urea:



Both reactions are reversible, the first being exothermic and going almost to completion, while the second is endothermic and goes to 40 to 70% of completion.

Ammonia and carbon dioxide are fed to the reactor, a stainless steel vessel with a series of trays to assist mixing. The reactor pressure is 125 bar and the temperature is 185°C. The reactor residence time is about 45 minutes, a 95% approach to equilibrium being achieved in this time. The ammonia is fed directly to the reactor, but the carbon dioxide is fed to the reactor upwardly through a stripper, down which flows the product stream from the reactor. The carbon dioxide decomposes some of the carbamate in the product stream, and takes ammonia and water to a high-pressure condenser. The stripper is steam heated and operates at 180°C, while the high-pressure condenser is at 170°C and the heat released in it by recombination of ammonia and carbon dioxide to carbamate is used to raise steam. Additional recycled carbamate solution is added to the stream in the high-pressure condenser, and the combined flow goes to the reactor.

The product stream leaving the stripper goes through an expansion valve to the low pressure section, the operating pressure there being 5 bar. In a steam-heated rectifier, further ammonia and carbon dioxide are removed and, with some water vapor, are

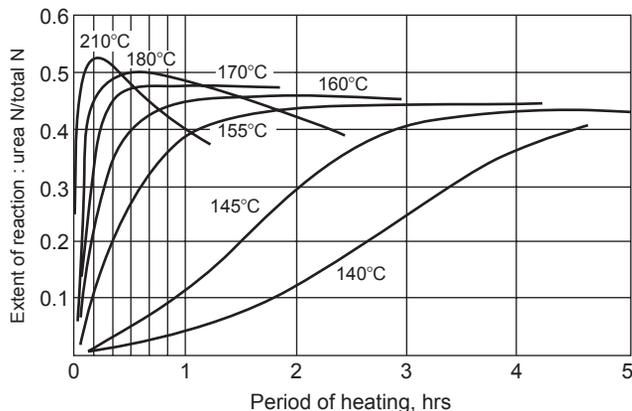


Figure F1. Rate of dehydration of carbamate.

condensed to give a weak carbamate solution. This is pumped back to the high-pressure condenser.

A two-stage evaporative concentration under vacuum, with a limited residence-time in the evaporator to limit biuret formation, produces a urea stream containing about 0.5% water which can be sprayed into a prilling tower.

Physico-chemical data



Properties of urea:

Density at 20°C = 1.335 g/cm³

Heat of solution in water = -250 J/g

Melting point = 133°C

Specific heat = 1.34 J/g at 20°C

Reactor and Stripper Design

The relationships between temperature, pressure, and composition for the Urea—CO₂—NH₃—H₂O system are given in References 5 and 6. These are equilibrium relationships. The reaction velocity may be obtained from the graph in Figure 5 of Reference 5, which is reproduced below for ease of reference (Figure F1). Some stripper design data appear in Reference 7.

Scope of Design Work Required

1. Prepare a mass balance diagram for the process, on a weight per hour basis, through to the production of urea prills.

2. Prepare an energy balance for the reactor–stripper–high-pressure condenser complex.
3. Prepare a process flow diagram, showing the major items of equipment in the correct elevation, with an indication of their internal construction. Show all major pipelines and give a schematic outline of the probable instrumentation of the reactor and its subsidiaries.
4. Prepare an equipment schedule, listing the main plant items with their size, throughput, operating conditions, materials of construction, and services required.
5. Prepare an outline design of the reactor and carry out the chemical engineering design of the stripper, specifying the interfacial contact area that will need to be provided between the carbon dioxide stream and the product stream to enable the necessary mass transfer to take place.
6. Prepare a mechanical design of the stripper, which is a vertical steam-heated tube bundle rather like a heat exchanger. Show how liquid is to be distributed to the tubes, and how the shell is to be constructed to resist the high pressure and the corrosive process material.
7. Prepare a detailed mechanical design of the reactor in the form of a general arrangement drawing with supplementary detail drawings to show essential constructional features. Include recommendations for the feed of gaseous ammonia, carbon dioxide, and carbamate solution, the latter being very corrosive. The design should ensure good gas-liquid contact; suitable instrumentation should be suggested, and provision included for its installation. Access must be possible for maintenance.
8. Specify suitable control systems for the maintenance of constant conditions in the reactor against a 15% change in input rate of ammonia or carbon dioxide, and examine the effect of such a change, if uncorrected, on the steam generation capability of the high-pressure condenser.

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F.6 HYDROGEN FROM FUEL OIL

The Project

A plant is to be designed to produce 20 million standard cubic feet per day (0.555×10^6 standard m^3/day) of hydrogen of at least 95% purity. The process to be employed is the partial oxidation of oil feedstock (References 1–3).

Materials Available

1. Heavy fuel oil feedstock of viscosity 900 seconds Redwood One (2.57×10^{-4} m^2/s) at 100°F with the following analysis:

Carbon	85% wt
Hydrogen	11% wt
Sulfur	4% wt
Calorific value	18,410 Btu/lb (42.9 MJ/kg)
Specific gravity	0.9435

The oil available is pumped from tankage at a pressure of 30 psig (206.9 kN/m^2 gauge) and at 50°C.

2. Oxygen at 95% purity (the other component assumed to be wholly nitrogen) and at 20°C and 600 psig (4140 kN/m^2 gauge).

Services Available

1. Steam at 600 psig (4140 kN/m^2 gauge) saturated.
2. Cooling water at a maximum summer temperature of 25°C.
3. Demineralized boiler feed water at 20 psig (138 kN/m^2 gauge) and 15°C suitable for direct feed to the boilers.
4. Waste low-pressure steam from an adjacent process.

On-Stream Time

8050 hours/year.

Product Specification

Gaseous hydrogen with the following limits of impurities:

CO	1.0% vol maximum (dry basis)
CO ₂	1.0% vol maximum (dry basis)
N ₂	2.0% vol maximum (dry basis)
CH ₄	1.0% vol maximum (dry basis)
H ₂ S	Less than 1 ppm

The gas is to be delivered at 35°C maximum temperature, and at a pressure not less than 300 psig (2060 kN/m² gauge). The gas can be delivered saturated, i.e., no drying plant is required.

The Process

Heavy fuel oil feedstock is delivered into the suction of metering-type ram pumps that feed it via a steam preheater into the combustor of a refractory-lined flame reactor. The feedstock must be heated to 200°C in the preheater to ensure efficient atomization in the combustor. A mixture of oxygen and steam is also fed to the combustor, the oxygen being preheated in a separate steam preheater to 210°C before being mixed with the reactant steam.

The crude gas, which will contain some carbon particles, leaves the reactor at approximately 1300°C and passes immediately into a special waste-heat boiler where steam at 600 psig (4140 kN/m² gauge) is generated. The crude gas leaves the waste heat boiler at 250°C and is further cooled to 50°C by direct quenching with water, which also serves to remove the carbon as a suspension. The analysis of the quenched crude gas is as follows:

H ₂	47.6	percent vol (dry basis)
CO	42.1	percent vol (dry basis)
CO ₂	8.3	percent vol (dry basis)
CH ₄	0.1	percent vol (dry basis)
H ₂ S	0.5	percent vol (dry basis)
N ₂	1.40	percent vol (dry basis)
	100.0	percent vol (dry basis)

For the primary flame reaction, steam and oxygen are fed to the reactor at the following rates:

Steam	0.75 kg/kg of heavy fuel oil feedstock
Oxygen	1.16 kg/kg of heavy fuel oil feedstock

The carbon produced in the flame reaction, and which is subsequently removed as carbon suspension in water, amounts to 1.5% by weight of the fuel oil feedstock charge. Some H₂S present in the crude gas is removed by contact with the quench water.

The quenched gas passes to an H₂S removal stage where it may be assumed that H₂S is selectively scrubbed down to 15 parts per million with substantially no removal of CO₂. Solution regeneration in this process is undertaken using the waste low-pressure steam from another process. The scrubbed gas, at 35°C and saturated, has then to undergo CO conversion, final H₂S removal, and CO₂ removal to allow it to meet the product specification.

CO conversion is carried out over chromium-promoted iron oxide catalyst employing two stages of catalytic conversion; the plant also incorporates a saturator and desaturator operating with a hot water circuit.

Incoming gas is introduced into the saturator (a packed column) where it is contacted with hot water pumped from the base of the desaturator; this process serves

to preheat the gas and to introduce into it some of the water vapor required as reactant. The gas then passes to two heat exchangers in series. In the first, the unconverted gas is heated against the converted gas from the second stage of catalytic conversion; in the second heat exchanger the unconverted gas is further heated against the converted gas from the first stage of catalytic conversion. The remaining water required as reactant is then introduced into the unconverted gas as steam at 600 psig (4140 kN/m² gauge) saturated and the gas/steam mixture passes to the catalyst vessel at a temperature of 370°C. The catalyst vessel is a single shell with a dividing plate separating the two catalyst beds that constitute the two stages of conversion. The converted gas from each stage passes to the heat exchangers previously described and thence to the desaturator, which is a further packed column. In this column the converted gas is contacted countercurrent with hot water pumped from the saturator base; the temperature of the gas is reduced and the deposited water is absorbed in the hot-water circuit. An air-cooled heat exchanger then reduces the temperature of the converted gas to 40°C for final H₂S removal.

Final H₂S removal takes place in four vertical vessels each approximately 60 feet (18.3 m) in height and 8 feet (2.4 m) in diameter and equipped with five trays of iron oxide absorbent. Each vessel is provided with a locking lid of the autoclave type. The total pressure drop across these vessels is 5 psi (35 kN/m²). Gas leaving this section of the plant contains less than 1 ppm of H₂S and passes to the CO₂ removal stage at a temperature of 35°C.

CO₂ removal is accomplished employing high-pressure potassium carbonate wash with solution regeneration (Reference 4).

Data

1. Basic Data for CO Conversion Section of the Plant

a. Space velocity

The space velocity through each catalyst stage should be assumed to be 3500 volumes of gas plus steam measured at NTP per volume of catalyst per hour. It should further be assumed that use of this space velocity will allow a 10°C approach to equilibrium to be attained throughout the possible range of catalyst operating temperatures listed below.

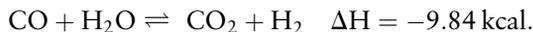
b. Equilibrium data for the CO conversion reaction

For

$$K_p = \frac{p_{\text{CO}} \times p_{\text{H}_2\text{O}}}{p_{\text{CO}_2} \times p_{\text{H}_2}}$$

Temp. (K)	K_p
600	3.69×10^{-2}
700	1.11×10^{-1}
800	2.48×10^{-1}

- c. Heat of reaction



2. Basic Data for CO₂ Removal Using Hot Potassium Carbonate Solutions

The data presented in Reference 4 should be employed in the design of the CO₂ removal section of the plant. A solution concentration of 40% wt equivalent K₂CO₃ should be employed.

Scope of Design Work Required

1. Process Design

- Calculate, and prepare a diagram to show, the gas flows, compositions, pressures, and temperatures, at each main stage throughout the processes of gasification and purification.
- Prepare a mass balance diagram for the CO conversion section of the plant including the live steam addition to the unconverted gas. Basic data that should be employed for the CO conversion process are presented in the references below.
- Prepare an energy-balance diagram for the flame reactor and for the associated waste-heat boiler.
- Prepare a process flow-diagram showing all major items of equipment. This need not be to scale, but an indication of the internal construction of each item (with the exception of the flame reactor, waste-heat boiler, and quench tower) should be given. The primary H₂S removal stage need not be detailed.
- Prepare an equipment schedule for the CO conversion section of the plant, specifying major items of equipment.

2. Chemical Engineering Design

- Prepare a detailed chemical engineering design of the absorber on the CO₂ removal stage.
- Prepare a chemical engineering design for the saturator on the CO conversion section.

3. Mechanical Design

Make recommendations for the mechanical design of the CO₂ removal absorber, estimating the shell and end-plate thickness and showing, by means of sketches suitable for submission to a design office, how

- the beds of tower packing are supported;
- the liquid is distributed.

Develop a detailed mechanical design of the CO conversion reactor, paying particular attention to the choice of alloy steels versus refractory linings, provisions for thermal expansion, inlet gas distribution, catalyst bed-support design, facilities for charging and discharging catalyst, and provisions for instrumentation.

4. Control

Prepare a full instrumentation flowsheet of the CO conversion section of the plant, paying particular attention to the methods of controlling liquid levels in the circulating water system and temperatures in the catalyst beds. Derive the unsteady-state equations that would have to be employed in the application of computer control to the CO conversion section of the plant.

REFERENCES

1. Garvie, J. H. (November 1967) Synthesis gas manufacture. *Chem Proc Engng*, pp. 55–65.
2. *Hydrocarbon Processing—Refining Processes Handbook. Issue A* (September 1970), p. 269.
3. Singer, S. C. and Ter Haar, L. W. (1961) Reducing gases by partial oxidation of hydrocarbons. *Chem Eng Prog*, 57, pp. 68–74.
4. Benson, H. E., Field, J. H., and Haynes, W. P. (1956) Improved process for CO₂ absorption uses hot carbonate solutions. *Chem Eng Prog*, 52, pp. 433–438.

F.7 CHLORINE RECOVERY FROM HYDROGEN CHLORIDE

The Project

A plant is to be designed for the production of 10,000 metric tons per year of chlorine by the catalytic oxidation of HCl gas.

Materials Available

1. HCl gas as byproduct from an organic synthesis process. This may be taken to be 100% pure and at 20°C and absolute pressure of 14.7 psi (100 kN/m²).
2. Air. This may be taken to be dry and at 20°C and absolute pressure of 14.7 psi (100 kN/m²).

Services Available

1. Steam at 200 psig (1400 kN/m²).
2. Cooling water at a maximum summer temperature of 24°C.
3. A limited supply of cooling water at a constant temperature of 13°C is also available.

On-Stream Time

8000 hours/year.

Product specification

Gaseous chlorine mixed with permanent gases and HCl. The HCl content not to exceed 5×10^{-5} part by weight of HCl per unit weight of chlorine.

The Process

HCl is mixed with air and fed into a fluidized bed reactor containing cupric chloride/pumice catalyst and maintained at a suitable temperature in the range 300–400°C. The HCl in the feed is oxidized, and the chlorine and water produced in the reaction, together with unchanged HCl and permanent gases, are passed to a packed tower cooler/scrubber, operating somewhat above atmospheric pressure, where they are contacted with aqueous HCl containing 33–36% by weight of HCl. This acid enters the cooler/scrubber at about 20°C. Most of the water and some of the HCl contained in the gases entering the cooler/scrubber are dissolved in the acid. The liquid effluent from the base of the cooler/scrubber flows to a divider box from which one stream passes to the top of the cooler/scrubber, via a cooler that lowers its temperature to 20°C, and another stream passes to a stripping column (“expeller”). Gas containing 98% by weight of HCl (the other constituents being water and chlorine) leaves the top of the expeller and is recycled to the reactor. A mixture of water and HCl containing 20–22% by weight of HCl leaves the base of the expeller. This liquid passes, *via* a cooler, to the top of an HCl absorber, which is required to remove almost the whole of the HCl contained in the gases leaving the cooler/scrubber. The liquid leaving the base of the HCl absorber, containing 33–36% by weight of HCl, is divided into two streams, one of which flows to the expeller, while the other is collected as product. The gaseous chlorine leaving the top of the HCl absorber passes to a drier.

Data

Reactor

Catalyst particle size distribution (U.S. Patent 2 746 844/1956)

Size range (μm)	Cumulative weight percentage undersize (at upper limit)
50–100	0.39
100–150	15.0
150–200	58.0
200–250	85.0
250–300	96.6
300–350	99.86

Density of catalyst: 40 lb/ft³ (640 kg/m³).
Voidage at onset of fluidization: 0.55.
Particle shape factor: 0.7.
Heat of reaction: 192 kcal/kg of HCl ($\Delta H = -29,340$ kJ/kmol).
(See Reference 1.)
Gas residence time in reactor: 25 seconds,
(See Reference 3.)

Cooler/Scrubber and Expeller

The overall heat transfer coefficient between the gas and liquid phases can be taken to be 5.0 Btu/h ft² degF (28 W/m² °C).

Scope of Design Work Required

1. Prepare a mass balance diagram for the process, up to but not including the drier, on the basis of weight/hour. Base the calculation on 10,000 long tons/year of chlorine entering the drier together with permanent gases, water, and not more than 5×10^{-5} parts by weight of HCl per unit weight of chlorine.
2. Prepare an energy balance diagram for the reactor and cooler/scrubber system.
3. Prepare a process flow diagram, up to but not including the drier, showing all the major items of equipment, with indications of the type of internal construction, as far as possible in the corrected evaluation. The diagram should show all major pipelines and the instrumentation of the reactor and the cooler/scrubber system.
4. Prepare an equipment schedule listing all major items of equipment and giving sizes, capacities, operating pressures and temperatures, materials of construction, etc.
5. Present a specimen pipeline sizing calculation.
6. Work out the full chemical engineering design of the reactor and cooler/scrubber systems.
7. Calculate the height and diameter of the expeller.
8. Prepare a mechanical design of the cooler/scrubber showing by dimensioned sketches, suitable for submission to a draftsman, how
 - a. The tower packing is to be supported;
 - b. The liquid is to be distributed in the tower;
 - c. The shell is to be constructed so as to withstand the severely corrosive conditions inside it.
9. Discuss the safety precautions involved in the operation of the plant, and the procedure to be followed in starting the plant up and shutting it down.
10. Develop the mechanical design of the reactor and prepare a key arrangement drawing, supplemented by details to make clear the essential constructional

features. The study should include recommendations for the design of the bed and means of separation and disposal of dust from the exit gas stream, and should take account of needs connected with thermal expansion, inspection, maintenance, starting and stopping, inlet gas distribution, insertion and removal of catalyst, and the positioning and provision for reception of instruments required for control and operational safety. Written work should be confined, as far as possible, to notes on engineering drawings, except for the design calculations, the general specification and the justification of materials of construction.

11. Assuming that the plant throughput may vary by 10% on either side of its normal design value due to changes in demand, specify control systems for
 - i. Regulation of the necessary recycle flow from the cooler/scrubber base, at the design temperature; and
 - ii. Transfer of the cooler/scrubber to make liquor to the expeller.

REFERENCES

1. Arnold, C. W. and Kobe, K. A. (1952) *Chem Engng Prog.* 48, p. 293.
2. Fleurke, K. H. (1968) *Chem Engr, Lond.*, p. CE41.
3. Quant, J., Van Dam, J., Engel, W. F., and Wattimena, F. (1963) *Chem Engr, Lond.*, p. CE224.
4. Sconce, J. S. (1962) *Chlorine: Its Manufacture, Properties, and Uses* (New York: Reinhold Publishing Corporation).

F.8 ANILINE FROM NITROBENZENE

The Project

Design a plant to make 20,000 metric tons per year of refined aniline by the hydrogenation of nitrobenzene. The total of on-stream operation time plus regeneration periods will be 7500 hours per year.

Materials Available

Nitrobenzene containing <10 ppm thiophene.
Hydrogen of 99.5% purity at a pressure of 50 psig (350 kN/m²).
Copper on silica gel catalyst.

Services Available

Steam at 200 psig (1400 kN/m²) 197°C, and 40 psig (280 kN/m²) 165°C.
Cooling water at a maximum summer temperature of 24°C.
Town water at 15°C.

Product Specification

Aniline	99.9% w/w min.
Nitrobenzene	2 ppm max.
Cyclohexylamine	100 ppm max.
Water	0.05% w/w max.

The Process

Nitrobenzene is fed to a vaporizer, where it is vaporized in a stream of hydrogen (three times stoichiometric). The mixture is passed into a fluidized bed reactor containing copper on silica gel catalyst, operated at a pressure, above the bed, of 20 psig (140 kN/m²). The contact time, based on superficial velocity at reaction temperature and pressure and based on an unexpanded bed, is 10 seconds. Excess heat of reaction is removed to maintain the temperature at 270°C by a heat transfer fluid passing through tubes in the catalyst bed. The exit gases pass through porous stainless-steel candle filters before leaving the reactor.

The reactor gases pass through a condenser/cooler, and the aniline and water are condensed. The excess hydrogen is recycled, except for a purge to maintain the impurity level in the hydrogen to not more than 5% at the reactor inlet. The crude aniline and water are let down to atmospheric pressure and separated in a liquid/liquid separator, and the crude aniline containing 0.4% unreacted nitrobenzene and 0.1% cyclo-hexylamine as well as water is distilled to give refined aniline. Two stills are used, the first removing water and lower boiling material, and the second removing the higher boiling material (nitrobenzene) as a mixture with aniline. The vapor from the first column is condensed, and the liquid phases separated to give an aqueous phase and an organic phase. A purge is taken from the organic stream to remove the cyclo-hexylamine from the system, and the remainder of the organic stream recycled. The cyclo-hexylamine content of the purge is held to not greater than 3% to avoid difficulty in phase separation. In the second column, 8% of the feed is withdrawn as bottoms product.

The purge and the higher boiling mixture are processed away from the plant, and the recovered aniline returned to the crude aniline storage tank. The aniline recovery efficiency in the purge unit is 87.5%, and a continuous stream of high-purity aniline may be assumed.

The aqueous streams from the separators (amine-water) are combined and steam stripped to recover the aniline, the stripped water, containing not more than 30 ppm aniline or 20 ppm cyclo-hexylamine, being discharged to drain.

Regeneration of the catalyst is accomplished in place using air at 250–350°C to burn off organic deposits. Regeneration takes 24 hours, including purging periods.

The overall yield of aniline is 98% theory from nitrobenzene; i.e., from 100 moles of nitrobenzene delivered to the plant, 98 moles of aniline pass to final product storage.

SCOPE OF DESIGN WORK REQUIRED

1. Prepare a material balance on an hourly basis for the complete process in weight units.
2. Prepare a heat balance for the reactor system, comprising vaporizer, reactor, and condenser/cooler.
3. Draw a process flow diagram for the plant. This should show all items of equipment approximately to scale and at the correct elevation. The catalyst regeneration equipment should be shown.
4. Chemical engineering design.
 - a. Vaporizer

Give the detailed chemical engineering design and give reasons for using the type chosen. Specify the method of control.
 - b. Reactor

Give the detailed chemical engineering design for the fluidized bed and heat transfer surfaces. Select a suitable heat transfer fluid and give reasons for your selection. Do *not* attempt to specify the filters or to design the condenser/cooler in detail.
 - c. Crude aniline separator

Specify the diameter, height, and weir dimensions and sketch the method of interface level control proposed.
 - d. Amine water stripper

Give the detailed chemical engineering design of the column.
5. Prepare a full mechanical design for the reactor. Make a dimensioned sketch suitable for submission to a drawing office, which should include details of the distributor and show how the heat transfer surfaces will be arranged. An indication of the method of supporting the candle filters should be shown, but do not design this in detail.
6. Prepare an equipment schedule detailing all major items of equipment, including tanks and pumps. A specimen pipeline sizing calculation for the reactor inlet pipe should be given. All materials of construction should be specified.
7. Describe briefly how the plant would be started up and shut down, and discuss safety aspects of operation.
8. Write a short discussion, dealing particularly with the less firmly based aspects of the design, and indicating the semitechnical work that is desirable.

Data

1. Catalyst properties:

a. Grading:

0–20 μm	Negligible
20–40 μm	3% w/w
40–60 μm	7% w/w
60–80 μm	12% w/w
80–100 μm	19% w/w
100–120 μm	25% w/w
120–140 μm	24% w/w
140–150 μm	10% w/w
>150 μm	Negligible

b. Voidage at minimum fluidization, 0.45.

c. Shape factor, 0.95.

d. Bulk density at minimum fluidization, 50 lb/ft³ (800 kg/m³).

e. Life between regenerations 1500 metric tons of aniline per ton of catalyst, using the feedstock given.

2. Exothermic heat of hydrogenation. $-\Delta H_{298} = 132,000$ BTU/lb mol (552,000 kJ/k mol).

3. Mean properties of reactor gases at reactor conditions:

Viscosity	0.02 centipoise (0.02 mNs/m ²)
Heat capacity at constant pressure	0.66 BTU/lb ^o C (2.76 kJ/kg ^o C)
Thermal conductivity	0.086 BTU/h ft ² (0.15 W/m ^o C)

4. Pressure drop through candle filters = 5 psi (35 kN/m²).

5. Density of nitrobenzene:

Temp. ^o C	Density g/cm ³
0	1.2230
15	1.2083
30	1.1934
50	1.1740

6. Latent heat of vaporization of nitrobenzene:

Temp. °C	Latent heat BTU/lb	(kJ/kg)
100	104	(434)
125	101	(422)
150	97	(405)
175	92.5	(387)
200	85	(355)
210	79	(330)

7. Latent heat of vaporization of aniline:

Temp. °C	Latent heat BTU/lb	(kJ/kg)
100	133.5	(558)
125	127	(531)
150	120	(502)
175	110	(460)
183	103.7	(433)

8. Specific heat of aniline vapor = 0.43 BTU/lb°C (1.80 kJ/kg°C).

9. Solubility of aniline in water:

Temp. °C	Percent w/w aniline
20	3.1
40	3.3
60	3.8
100	7.2

10. Solubility of water in aniline:

Temp. °C	Percent w/w water
20	5.0
40	5.3
60	5.8
100	8.4

11. Density of aniline/water system:

Temp. °C	Density g/cm ³	
	Water layer	Aniline layer
0	1.003	1.035
10	1.001	1.031
20	0.999	1.023
30	0.997	1.014
40	0.995	1.006
50	0.991	0.998
60	0.987	0.989
70	0.982	0.982

12. Partition of cyclo-hexylamine between aniline and water at 30°C:

w/w percent cyclo-hexylamine in aniline	w/w percent water in aniline	w/w percent cyclo-hexylamine in water	w/w percent aniline in water
1.0	5.7	0.12	3.2
3.0	6.6	0.36	3.2
5.0	7.7	0.57	3.2

13. Partition coefficient of nitrobenzene between aniline layer and water layer:

$$C_{a.l.}/C_{w.l} = 300.$$

14. Design relative velocity in crude aniline-water separator: 10 ft/h (3 m/h).

15. Equilibrium data for water-aniline system at 760 mm Hg abs:

Temp. °C	Mole Fraction Water	
	Liquid	Vapor
184	0	0
170	0.01	0.31
160	0.02	0.485
150	0.03	0.63
140	0.045	0.74
130	0.07	0.82
120	0.10	0.88
110	0.155	0.92
105	0.20	0.94
100	0.30	0.96
99	0.35–0.95	0.964
	0.985	0.9641
	0.9896	0.9642
	0.9941	0.9735
	0.9975	0.9878
	0.9988	0.9932

16. Equilibrium data for cyclo-hexylamine-water system at 760 mm Hg abs:

Mole Fraction Cyclo-Hexylamine	
Liquid	Vapor
0.005	0.065
0.010	0.113
0.020	0.121
0.030	0.123
0.040	0.124
0.050	0.125
0.100	0.128
0.150	0.131
0.200	0.134
0.250	0.137

17. Temperature coefficient for aniline density— 0.054 lb/ft^3 (0.86 kg/m^3 °C) (range 0–100°C).

REFERENCES

1. U.S. Patent 2,891,094 (American Cyanamid Co.).
2. Perry, R. H., Chilton, C. H., and Kirkpatrick, S. D. (eds). (1963) *Chemical Engineers' Handbook*, 4th ed., Section 3 (New York: McGraw-Hill Book Company, Inc.).
3. Leva, M. (1959) *Fluidization* (New York: McGraw-Hill Book Company, Inc.).
4. Rottenburg, P. A. (1957) *Trans Instn Chem Engrs*, 35, p. 21.

As an alternative to Reference 1 above, any of the following may be read as background information to the process:

5. *Hyd Proc and Pet Ref* (1961) 40, No. 11, p. 225.
6. Stephenson, R. M. (1966) *Introduction to the Chemical Process Industries* (New York: Reinhold Publishing Corporation).
7. Faith, W. L., Keyes, D. B., and Clark, R. L. (1965) *Industrial Chemicals*, 3rd ed. (New York: John Wiley & Sons Inc.).
8. Sittig, M. (1962) *Organic Chemical Processes* (New York: Noyes Press).