

Solutions to Towler & Sinnott Chemical Engineering Design 2nd edition

Part I: Process Design

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Note that most of the problems involve design and so have no single unique answer. Credit should be given to students who have followed the right method and found similar solutions. Indeed, the probability of any student independently coming up with the exact answers given in the solution set for more than a few problems should be vanishingly small and this event should cause the grader to be suspicious. The “optimal” solutions presented are usually not numerically optimal and are merely close enough to optimal to be good enough for engineering purposes. This reflects the optimization philosophy described in Chapter 12.

Chapter 1

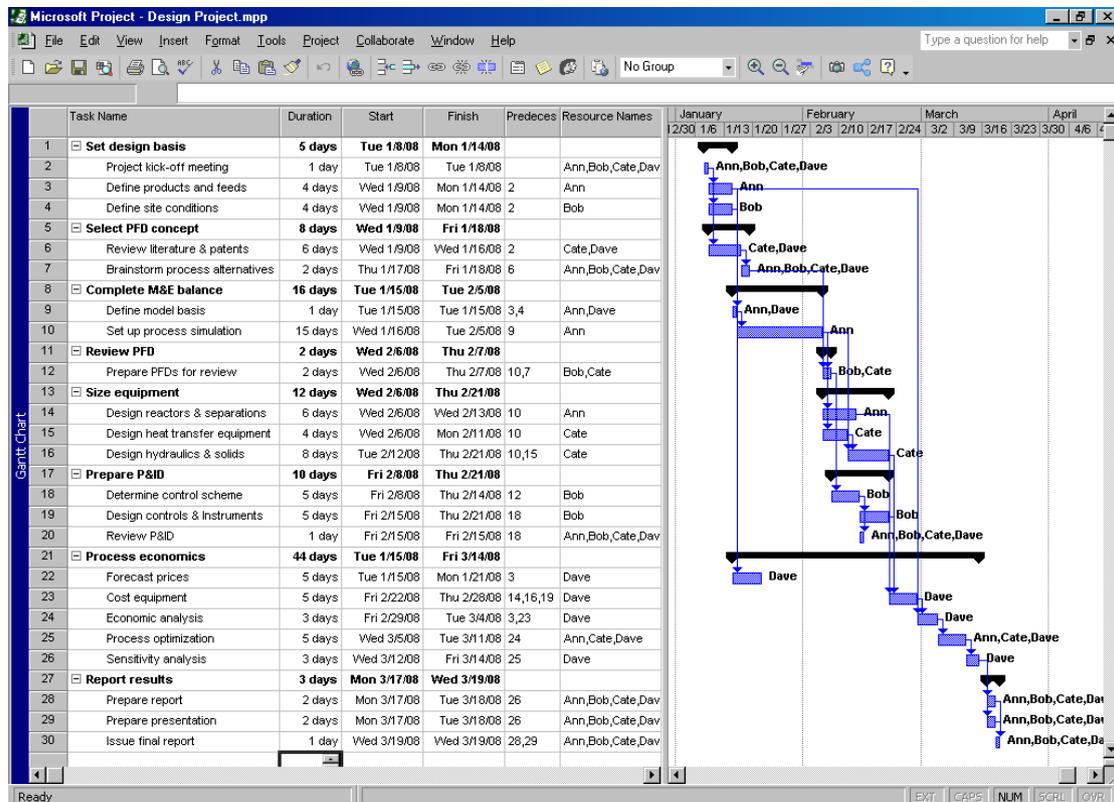
Problem 1.1

There are many possible correct answers to this question and it can be answered in varying levels of detail. The key steps that should be included for each process with typical required times are listed below. The project plan can be sketched using a spreadsheet or drawn up using a project planning tool such as MS Project (as in Problem 1.2).

- a) A petrochemical process using established technology, to be built on an existing site. Since the technology is established, there will be no need to generate design concepts and carry out R&D. The steps are then:
- Set design basis (1 week)
 - Evaluate economics, optimize and select design (typically 10-30 weeks, depending on project scope)
 - Detailed design and equipment selection (typically six months to one year)
 - Procurement and construction (typically one year)
 - Shakedown and start-up (typically one month)
- These steps are usually more or less sequential, although some procurement of long lead-time items may be started during detailed design.
- b) A process for full-scale manufacture of a new drug, based on a process currently undergoing pilot plant trials. Since the pilot plant is already operating the designer already has a good idea of the process flowsheet and the goal is to be prepared to ramp up production to full scale once the drug is approved. The steps are:
- Set design basis (1 week)
 - Confirm performance/scale-up of pilot plant operations (2-20 weeks, depending on how smoothly pilot plant runs)
 - Optimize and select design (10-20 weeks)
 - Detailed design and equipment selection (about six months)
- In parallel to these process design activities there will be activities related to getting approval for the new drug:
- Conduct clinical trials (6 months to 2 years)
 - Review clinical trial results (typically 3 to 6 months)
 - Obtain FDA approval
- Some of the procurement and construction activities will be started as soon as the first clinical results look promising, but final construction and shakedown will not occur until the review of clinical trials is completed.
- c) A novel process to convert cellulosic waste to fuel. The technology and flowsheet will need considerable development, so a schedule might be:
- Set design basis (1 week)
 - Generate design concepts & carry out R&D (one to five years)
 - Evaluate economics, optimize and select design (six months, but could run parallel to generating design concepts for up to five years)
 - Detailed design and equipment selection (six months to one year)
 - Procurement and construction (about one year)
 - Shakedown and start-up (one month to one year, as there may be start-up hiccups with a new technology)
- d) A spent nuclear fuel reprocessing facility. There is established technology for nuclear fuel reprocessing, but new processes are always possible. For an established technology the schedule would look much like problem (i) and for new technology it would look like problem (iv). All of the steps would probably take longer because of the scale of the plant and additional steps would be needed for obtaining local, state and federal permits and revising them after setting the design basis, selecting the design, and completing detailed design. The time taken to obtain permits could be several years and the total time to operation would probably exceed ten years.
- e) A solvent recovery system for electronics production. This is a relatively small project, so the steps would be:
- Set design basis (1 – 2 days)
 - Generate design concepts (1 to 2 months)
 - Evaluate economics, optimize and select design (ten weeks or less)
 - Detailed design and equipment selection (2 to 3 months)
 - Procurement and construction (3 to 6 months)
 - Shakedown and start-up (one month)

Problem 1.2

This requires a more detailed breakdown than problem 1.1. A sample project plan is given in the lecture slides and shown below (in MS Project format):



Suitable intermediate deliverables could include:

- The design basis
- A completed PFD (or PFD review)
- A completed process simulation
- A completed PID (or review)

Problem 1.3

a) The list of product requirements will be somewhat qualitative and depend on the preferences of the “customer” group. The required properties of the dough must consider properties of the dough itself, as well as properties of the final (home-baked) product. Some properties of the dough that might be considered include:

- Shelf life
- Calorie content
- Chocolate chip content
- Stiffness (do you scoop it or is it preformed in cookie shapes?)
- Baking time

Properties of the end cookies are perhaps more obvious:

- Chewiness
- Crunchiness
- Sweetness
- Saltiness
- Mouth feel
- Serving size (if pre-formed)

b) The product specifications could include the following:

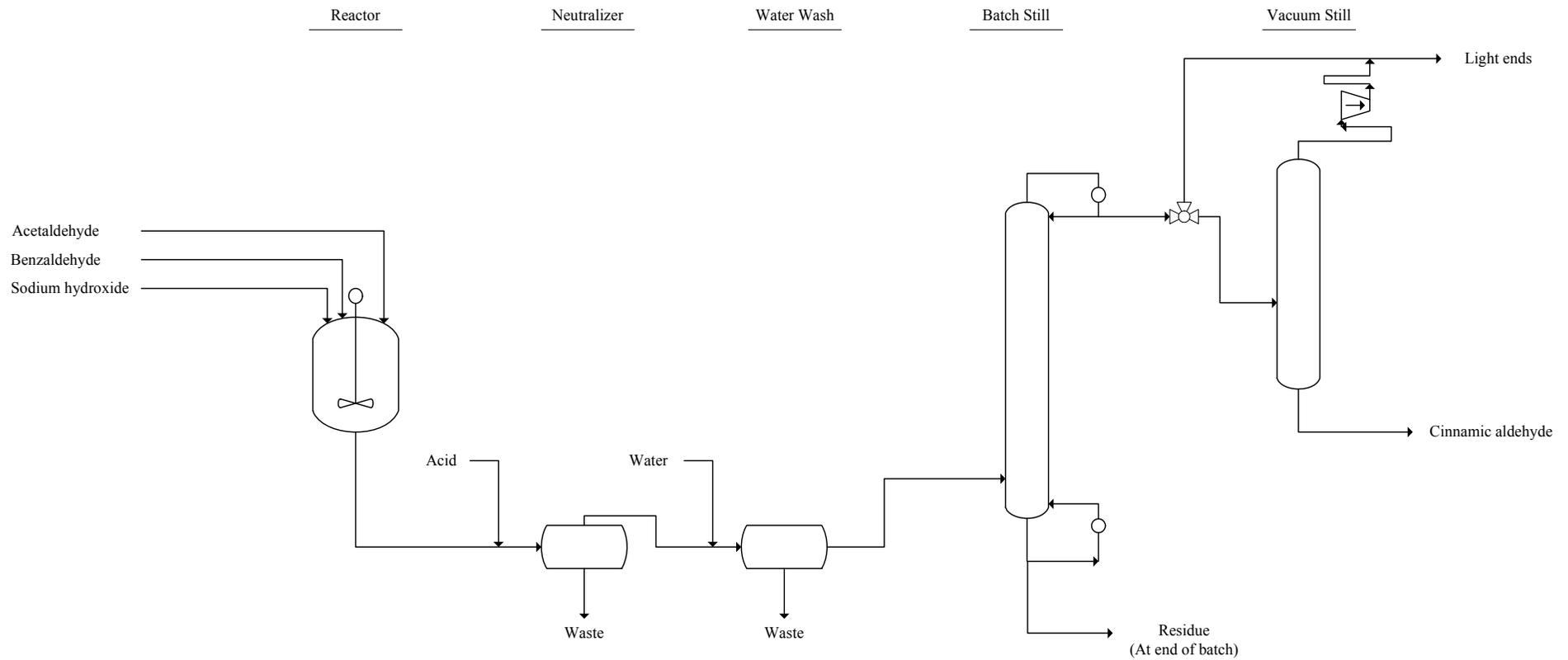
- Composition of major ingredients (see any cookie dough: flour, fat or oil, water, etc.)
- Composition of chocolate chips

- Size of chocolate chips
- Composition of minor flavors (salt, vanilla, etc.)
- Composition of baking soda?
- Type and composition of sweetening agent
- Type and composition of preservatives, stabilizers
- Type and composition of viscosity modifiers?
- Mixing order
- Mixing time, speed, temperature
- Dough aging / forming processes (extrusion, cutting, rolling, etc.)

Chapter 2

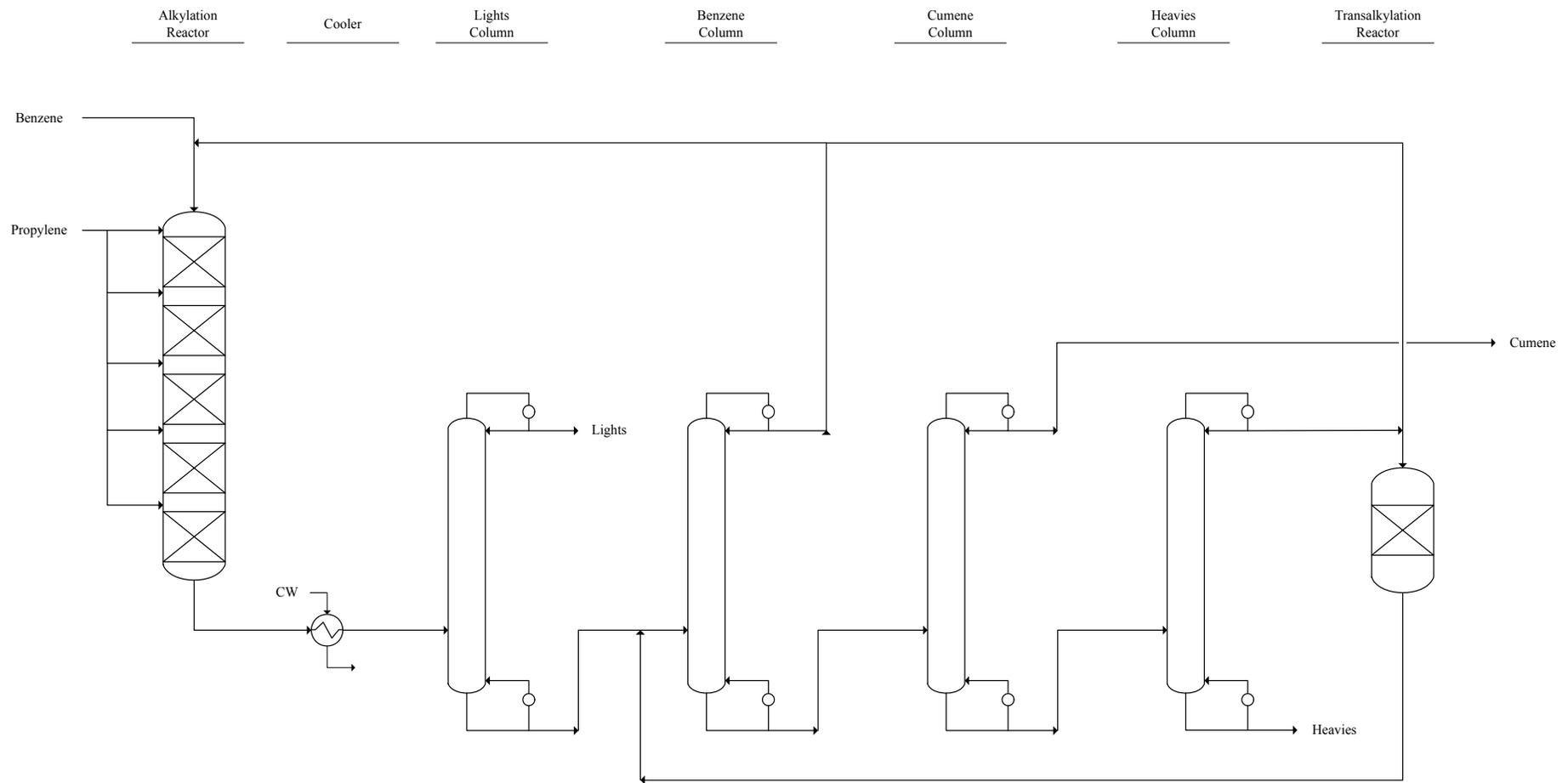
Solution 2.1

This process can be drawn in more detail, but a simple block-flow diagram is adequate. There may be a need for some heaters and coolers in the plant (e.g. after neutralization), but these are not described in the problem statement and would not need to be shown in a block-flow diagram. Since the process involves batch distillation, it would also be possible to operate the reactor in batch mode and then carry out the neutralization and wash stages in batch mode in the same vessel.



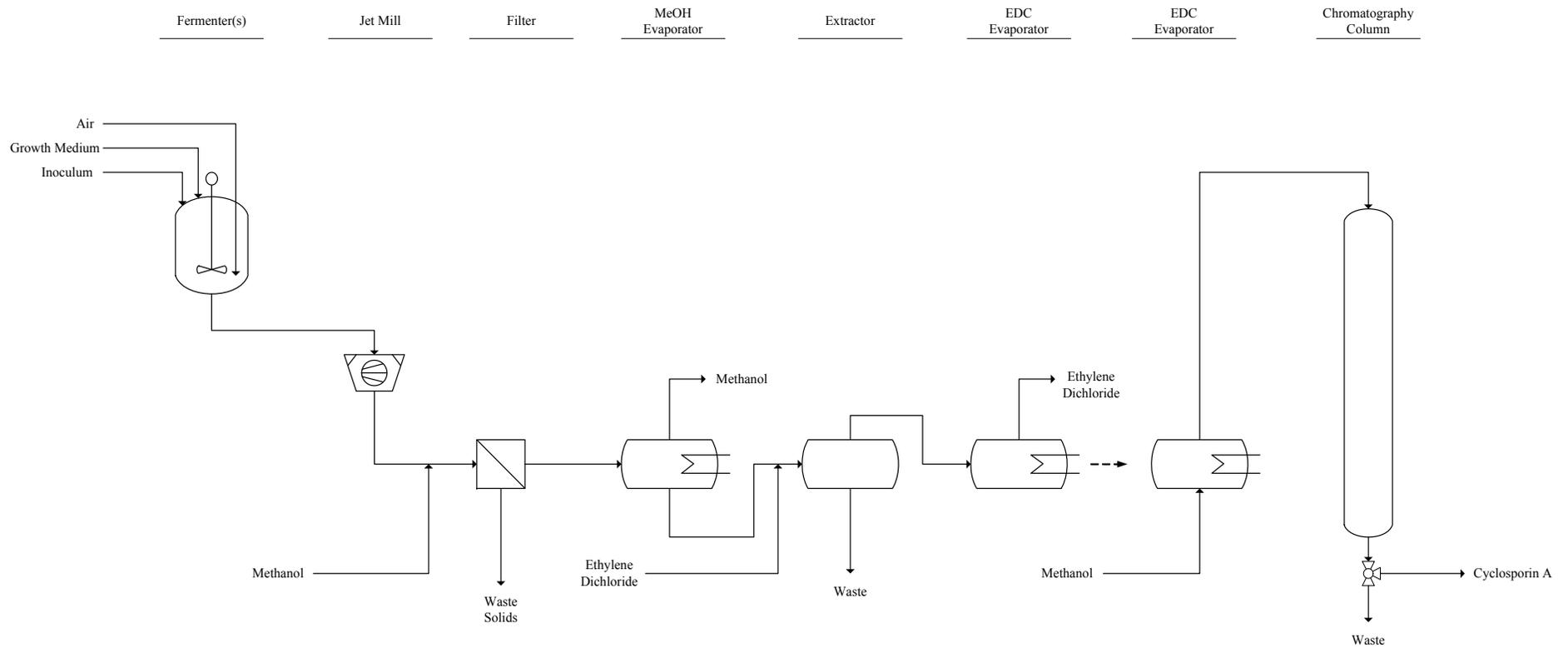
Solution 2.2

This is a continuous process and has a more complex flowsheet incorporating two recycles:



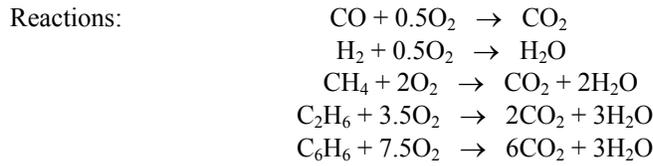
Solution 2.3

This is also a batch process in which some of the extraction steps could be carried out reusing the same equipment. The dashed line shows the reuse of the EDC evaporator to dissolve the product in MeOH for chromatography.



Solution 2.4

Basis for calculation: 100 kmol dry gas



	REACTANTS		PRODUCTS		
	Syn. Gas	O ₂	CO ₂	H ₂ O	N ₂
CO ₂	4		4		
CO	16	8	16		
H ₂	50	25		50	
CH ₄	15	30	15	30	
C ₂ H ₆	3	10.5	6	9	
C ₆ H ₆	2	15	12	6	
N ₂	10				10
Totals	100	88.5	53	95	10

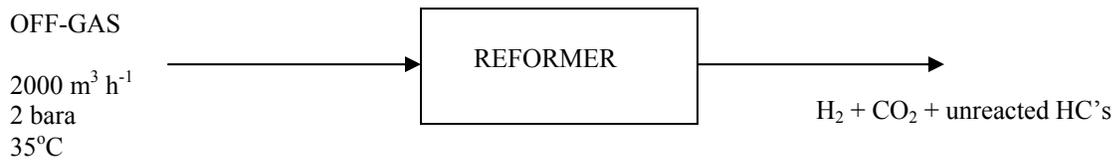
If Air is N₂:O₂ = 79:21

N₂ with combustion air = 88.5 x 79/21 = 332.9 kmol
 Excess O₂ = 88.5 x 0.2 = 17.7 kmol
 Excess N₂ = 17.7 x 79/21 = 66.6 kmol
 Total = 417.2 kmol

- (i) Air for combustion = 417.2 + 88.5 = 505.7 kmol
- (ii) Flue Gas produced = 53 + 95 + 10 + 417.2 = 575.2 kmol
- (iii) Flue Gas analysis (dry basis):

N ₂	409.5 kmol	85.3 mol %
CO ₂	53.0 kmol	11.0 mol %
O ₂	17.7 kmol	3.7 mol %
	<hr/> 480.2 kmol	<hr/> 100.0 mol %

Solution 2.5



At low pressures vol% = mol%

- (i) Basis: 1 kmol of off-gas

Component	mol%	MW	mass (kg)
CH ₄	77.5	16	12.40
C ₂ H ₆	9.5	30	2.85
C ₃ H ₈	8.5	44	3.74

C ₄ H ₁₀	4.5	58	$\frac{2.61}{21.60}$
			Σ

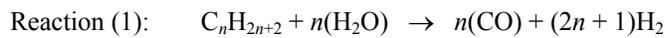
So the average molecular mass = 21.6 kg kmol⁻¹

(ii) At STP, 1 kmol occupies 22.4 m³

$$\text{Flow rate of gas feed} = \left(\frac{2000}{22.4} \right) \left(\frac{2 \times 10^5}{1.013 \times 10^5} \right) \frac{273}{(273 + 35)} = 156.248 \text{ kmol h}^{-1}$$

$$\text{Mass flow rate} = (156.248)(21.60) = 3375 \text{ kg h}^{-1}$$

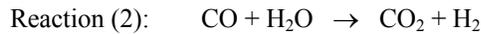
(iii) Basis: 100 kmol of feed



Component	<i>n</i>	Amount CO	H ₂	
CH ₄	1	77.5	77.5	232.5
C ₂ H ₆	2	9.5	19.0	47.5
C ₃ H ₈	3	8.5	25.5	59.5
C ₄ H ₁₀	4	4.5	18.0	40.5
		Σ	140.0	380.0

$$\text{If the conversion is 96\%, then: } H_2 \text{ produced} = (380.0)(0.96) = 364.8 \text{ kmol}$$

$$CO \text{ produced} = (140.0)(0.96) = 134.4 \text{ kmol}$$



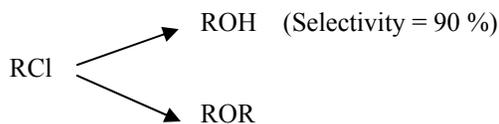
$$\text{If the conversion is 92\%, then: } H_2 \text{ from CO} = (134.4)(0.92) = 123.65 \text{ kmol}$$

$$\text{Total } H_2 \text{ produced} = 364.8 + 123.65 = 488.45 \text{ kmol/100 kmol feed}$$

If the gas feed flow rate = 156.25 kmol h⁻¹, then

$$H_2 \text{ produced} = 156.25 \left(\frac{488.45}{100} \right) = 763.20 \text{ kmol h}^{-1} \equiv (763.2)(2) = 1526 \text{ kg h}^{-1}$$

Solution 2.6



(Conversion = 97 %)

Basis: 1000 kg RCl feed

Relative molecular masses:

CH ₂ =CH-CH ₂ Cl	76.5
CH ₂ =CH-CH ₂ OH	58.0
(CH ₂ =CH-CH ₂) ₂ O	98.0

$$\begin{aligned}
 \text{RCl feed} &= \frac{1000}{76.5} = 13.072 \text{ kmol} \\
 \text{RCl converted} &= (13.072)(0.97) = 12.68 \text{ kmol} \\
 \text{ROH produced} &= (12.68)(0.9) = 11.41 \text{ kmol} \\
 \text{ROR produced} &= 12.68 - 11.41 = 1.27 \text{ kmol} \\
 \text{Mass of allyl-alcohol produced} &= (11.41)(58.0) = 661.8 \text{ kg} \\
 \text{Mass of di-ally ether produced} &= (1.27)(98.0) = 124.5 \text{ kg}
 \end{aligned}$$

Solution 2.7

Basis: 100 kmol nitrobenzene feed.

(a, b)

The conversion of nitrobenzene is 96% and so $100(1 - 0.96) = 4$ kmol are unreacted.

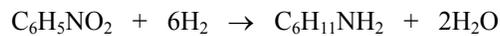
The selectivity for aniline is 95% and so aniline produced = $(96)(0.95) = 91.2$ kmol

Therefore, the balance is to cyclohexylamine = $96 - 91.2 = 4.8$ kmol

From the reaction equations:



1 mol of aniline requires 3 mol of H₂



1 mol of cyclohexylamine requires 6 mol of H₂

Therefore, H₂ required for the reactions = $(91.2)(3) + (4.8)(6) = 302.4$ kmol

A purge must be taken from the recycle stream to maintain the inerts below 5%. At steady-state conditions:

$$\text{Flow of inerts in fresh H}_2 \text{ feed} = \text{Loss of inerts from purge stream}$$

Let the purge flow be x kmol and the purge composition be 5% inerts.

$$\text{Fresh H}_2 \text{ feed} = \text{H}_2 \text{ reacted} + \text{H}_2 \text{ lost in purge}$$

$$= 302.4 + (1 - 0.05)x$$

$$\text{Inerts in the feed at 0.005 mol fraction (0.5\%)} = (302.4 + 0.95x) \frac{0.005}{1 - 0.005}$$

$$= 1.520 + 4.774 \times 10^{-3}x$$

$$\text{Inerts lost in purge} = 0.05x$$

$$\text{So, equating these quantities: } 0.05x = 1.520 + 4.774 \times 10^{-3}x$$

$$\text{Therefore: } x = 33.6 \text{ kmol}$$

The purge rate is 33.6 kmol per 100 kmol nitrobenzene feed.

$$\text{H}_2 \text{ lost in the purge} = 33.6(1 - 0.05) = 31.92 \text{ kmol}$$

$$\text{Total H}_2 \text{ feed} = 302.4 + 31.92 = 334.3 \text{ kmol}$$

b) Yield after reactor = feed rate \times 0.938 = 0.938 (100/0.87) = 107.82 kg/day

So losses in evaporator = 107.82 – 100 = 7.82 kg/day

c) It is not necessary to read the patent to understand this part. The clue is that the catalyst is NaOH. The purpose of the evaporator must therefore be to evaporate the guaifenesin from the nonvolatile catalyst (which is actually neutralized with acid before the evaporator). The product that is lost is therefore the product that remains in the liquid phase in the evaporator.

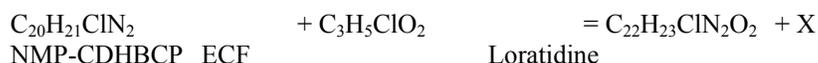
Possible ways to increase product recovery could include:

- Reducing the evaporator pressure
- Adding a second stage evaporator operated at lower pressure
- Stripping the reactor product using a suitable volatile compound (the patent says steam is no good, but glycidine could be investigated).

Solution 2.9

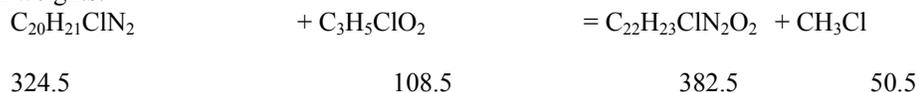
It is not necessary to read the patent to answer this question.

a) Start by writing a stoichiometric equation:



By difference, X has formula CH₃Cl, i.e. is chloroform

b) Molar weights:



If conversion is 99.9%, then for 16.2g of feed NMP-CDHBCP, 16.2 \times 0.999 = 16.184 g are converted

So required feed of ECF = 16.184 (108.5/324.5) = 5.411 g

Actual feed of ECF = 10.9g, so excess at end of reaction = 10.9 – 5.411 = 5.489 g

c) If ECF is not recovered or recycled then 5.489/10.9 = 50.4% is lost to waste products (spent solvents). Since the selectivity is 100%, almost all of this lost material could in principle be recovered.

d) The feed of 16.2 g of NMP-CDHBCP gives a yield of 16.2 \times 0.999 \times (382.5/324.5) = 19.076 g of product, so per kg of API we require 200 ml \times 1000/19.076 = 10.484 liters of each solvent at each step.

Step	solvent	density (kg/m ³)	mass (kg/200ml)	mass (kg/kg API)
Reaction	benzene	879	0.176	9.216
Quench	water	1000	0.200	10.484
Wash	water	1000	0.200	10.484
Recrystallization	isopropyl ether	724	0.145	7.590

In addition to the waste solvent, we also have chloroform produced by the reaction.

Chloroform yield = 50.5 g per 382.5 g of API = 132.0 g/kg of API.

The ECF waste is 5.489 g per 19.076 g product, i.e., 5.489 \times 1000/19.076 = 287.7 g per kg product.

The trituration step also uses petroleum ether, but the amount of this is much smaller than the amounts of solvents used. Allow 1 kg per kg product as an initial estimate.

So total mass of waste produced is:

Water	20.968
Benzene	9.216
Chloroform	0.132
Ethyl chloroformate	0.288
Petroleum ether	1.0
<u>Isopropyl ether</u>	<u>7.59</u>
Total	39.2 kg/kg API

Note: this made no allowance for recovery yields on the washing and recrystallization steps, so the actual waste produced would be higher.

e) 16.2 g of NMP-CDHBCP + 10.9 g of ECF give 19.076 g of product

If 92% of the product is recovered after separation then overall yield = $0.92 \times 19.076 = 17.550$ g

So to produce 10kg of API we require:

Feed rate of NMP-CDHBCP = $16.2 \times (10/17.55) = 9.23$ kg/batch

Feed rate of ECF = $10.9 \times (10/17.55) = 6.21$ kg/batch

f) The reactor should be operated no more than 2/3 full, to allow for swirling of the contents, vessel internals, etc., so available volume = $3800 \times 2/3 = 2533$ liters. Since the preparation involved decanting the benzene mixture into water, the reaction step must use only half of the available volume (so that the other equal volume of water can be added later), so available volume for reaction = 1267 liters

The preparation method describes dissolving 16 g of NMP-CDHBCP in 175g of benzene (200ml), so the solution is roughly 10 wt%. We have no information on the volume of mixing, so as a first approximation we can assume the mixed volume is 200ml. The lab recipe makes a 200ml batch, that ultimately yields 17.55 g of API.

If 200ml gives 17.55 g, then:

1267 liters gives $17.55 \times (1267/0.2) = 111.2$ kg per batch

Reaction time is 18 hours. Estimated times for the other processing steps might be:

Cooling & quench:	2 hours
Decanting	1 hour
Washing & evaporation	2 hours
Trituration	1 hour
<u>Recrystallization</u>	<u>2 hours</u>
Total	8 hours

If all the steps were carried out in the same vessel we would also need to allow time for cleaning and refilling at the end of the batch, say 2 more hours, giving a total batch time of 28 hours and production rate of $111.2/28 = 3.97$ kg/hr.

If only the reaction step is carried out in the reactor then we still need to allow some time for filling, emptying and cleaning, say 3 hours, giving a batch time of 21 hours and a maximum production rate of $111.2/21 = 5.3$ kg/hr.

g) Advantages of carrying out all steps in same vessel:

- Less equipment to clean between batches
- Possibly higher yields during washing and decanting steps

Disadvantages of carrying out all steps in same vessel:

- Could be impractical for trituration
- Could give poor separation during decanting, washing
- Difficult for ice-water quench unless the effect of pouring ice water into benzene mixture is the same as pouring benzene mixture into ice water (scale-up question).
- Would be difficult to get good recovery in final recrystallization step as material would coat vessel internals – easiest to send to a crystallizer.

Solution 2.10

The descriptions below were based on the Kirk Othmer encyclopedia entries. More detail could be added, but the summaries below are adequate to sketch a block flow diagram of the process.

a) Phosphoric acid

Most phosphoric acid is made by the wet process and is then used for making fertilizers. In the wet process, phosphate rock containing mainly calcium phosphate is ground up and treated with concentrated sulphuric acid at 80 – 90 °C. The calcium sulphate is filtered from the acid. If higher purity phosphoric acid is needed then it can be purified by countercurrent extraction with an organic solvent. The highest purity phosphoric acid is made by the dry process, in which elemental phosphorus is burned in excess air.

b) Adipic acid

Adipic acid is manufactured in a two-step process starting from either cyclohexane (by oxidation) or phenol (by hydrogenation). The first step produces a mixture of cyclohexanol and cyclohexanone known as KA oil (ketone and alcohol oil). There are several variations on the cyclohexane oxidation process using different catalysts, but all operate at relatively low CH conversion and require a large recycle of unconverted feed. The oxidation is carried out at moderate pressure (~10 bar) and temperature (~150°C) in a liquid-vapor reactor. The product is washed with water to remove acids (which can be recycled or used as fuel), then distilled to recover unconverted cyclohexane for recycle to the reactor. The KA oil is then sent to a stripper and re-run column sequence to remove light impurities and recover the KA oil as overhead product of the re-run column. The heavies from the re-run column are usually incinerated.

The KA oil is oxidized to adipic acid using nitric acid, at about 4 bar and 75 °C. The reaction is highly exothermic and the reactors use pump-arounds through heat exchangers to provide the necessary surface area for cooling. The reactor product is sent to crystallizers for adipic acid recovery. The crystals are washed, then redissolved in hot water and recrystallized to purify the adipic acid.

c) Polyethylene terephthalate

PET is a polyester, formed by condensation polymerization of ethylene glycol (EG) and terephthalic acid (TA). Byproduct water is removed by evaporation. The polymerization reaction is typically carried out in two or three stages, at 250 – 300 °C, and at successively lower pressure, to allow a longer chain length polymer to form. EG is added to each reactor, and the final reactor is operated under vacuum. Polymer from the last reactor is pumped direct to spinnarets for producing fiber, or is pelletized.

d) Insulin

Insulin is a hormone that regulates glucose homeostasis. It is manufactured by fermentation in genetically modified *E. coli* or yeast. At the end of the fermentation the cells are homogenized, and the broth is filtered to remove debris. In the most common route, the cells produce an insulin precursor called proinsulin, which can be converted to insulin by adding an enzyme to splice away the connecting sequence between insulin proteins. The resulting insulin can be precipitated by pH adjustment, but is more commonly purified by ion exchange and gel chromatography.

e) Sorbitol

Sorbitol is a sugar alcohol and is made by hydrogenation of glucose. The reaction is carried out in aqueous solution slurried with a nickel catalyst at high pressure (125 bar) and moderate temperature (140°C). The catalyst is recovered by filtration for reuse, and the solution is purified by ion exchange followed by activated carbon adsorption to remove organic impurities. The purified sorbitol solution is concentrated by evaporation to 70% solids for sale as solution, or sent to a crystallizer to form a solid product.

Solution 2.11

To begin we need to calculate the temperatures that can be handled by the heater and cooler in the revamped case.

Starting with the steam heater, E102:

New overall heat transfer coefficient = 513 W/m²K (from the example)

Area = 27.5 m²

So if the temperature into the heater is T_i , then:

$$Q_{102} = 1.5 \times 10^4 \times (160 - T_i) = 513 \times 27.5 \times \left(\frac{(180-160)-(180-T_i)}{\ln \left(\frac{180-160}{180-T_i} \right)} \right)$$

Which can be solved to give $T_l = 128.8\text{ }^\circ\text{C}$

Note that we have increased the outlet temperature from the plate exchanger, and so we have increased the amount of heat that is transferred in this exchanger. Since the heat capacity is the same on both sides of this exchanger $(128.8 - 40) = (140 - T_{out})$, so the outlet temperature from the plate exchanger is $51.2\text{ }^\circ\text{C}$. By inspection, we can see that the duty of the cooler must therefore be reduced (temperature difference roughly halved while flow increased 50%) and hence the cooler will not limit the revamp design.

For the plate heat exchanger E101, we now have:

$$\text{Duty } Q_{101} = 1.5 \times 10^4 \times (128.8 - 40) = 1.33 \text{ MW}$$

Overall heat transfer coefficient = $350\text{ W/m}^2\text{K}$ (assuming we keep the same plate velocities and pressure drop)

$$\text{Mean temperature difference} = (140 - 128.8) = (51.2 - 40) = 11.2\text{ }^\circ\text{C}$$

$$\text{So area} = Q/U\Delta T = 1.33 \times 10^6 / (350 \times 11.2) = 340\text{ m}^2$$

So the additional area needed would be $340 - 114 = 226\text{ m}^2$.

This is quite a large increase relative to the initial area of 114 m^2 and it is not clear that the plate and frame exchanger would have been sized large enough to accommodate such a large increase in area. We could reduce the additional area required if we allowed an increase in plate velocities and hence the overall heat transfer coefficient of the plate exchanger.

Solution 2.12

Compound	Formula	Molecular weight
Benzene	C_6H_6	78
Ethylene	C_2H_4	28
Ethylbenzene	C_8H_{10}	106
Styrene	C_8H_8	104

So to make 1 ton of styrene we require $106/104 = 1.019$ tons of ethylbenzene (EB)

To make 1.019 tons of (EB) we need $1.019 \times (78/106) = 0.75$ tons of benzene and $1.019 \times (28/106) = 0.269$ tons of ethylene.

So the stoichiometric feed cost per ton of styrene = $0.75(500) + 0.269(800) = \590.2

If we use the rule of thumb given in Section 2.6.1 and assume that the plant will be able to pay off the capital invested if the target feed cost is 80% of the total cost of production then:

$$\text{Target feed cost} = 0.8 \times 800 = \$640 / \text{ton styrene}$$

and

$$\text{Target plant yield} = \text{stoichiometric cost}/\text{target feed cost} = 590.2/640 = 92.2\%$$

As a first pass, we can allocate the yield equally between the two process steps, so

$$Y_1 = Y_2 = \sqrt{(0.922)} = 0.960$$

Alternatively, we could use our knowledge of the process chemistry to require a higher yield of one process than the other.