DISTILLATION: Revisiting Some Rules of Thumb

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Distillation is the most common unit operation for separating liquid mixtures into valuable and/or high purity products. It is also one of the most energy-intensive operations. Hence, optimization of distillation-column design and operation should get high priority.

Numerous distillation heuristics (rules of thumb) for quick optimization have emerged over the years. For instance, heuristics on optimal reflux ratio as a certain multiple of the minimum reflux ratio have been widely used as quick tools to estimate optimum reflux ratio.

However, changes over time in the relative cost of equipment and energy (which affects operating cost) can affect the validity of such rules of thumb. Meanwhile, it now becomes more feasible to assess their validity, as today's availability of commercial simulators and high-speed computers allows rigorous and thus more-accurate distillation calculations be carried out with relative ease.

This article assesses the validity of optimal-reflux-ratio and other heuristics in light of recent cost data, by considering seven binary and six multi-component systems. Distillation columns for each of the 13 have been designed and optimized by both shortcut (heuristics-based) calculations and rigorous simulations. In addition to the reassessment, a key observation emerges: that the cost of a column designed by shortcut calculations can be reduced substantially by optimizing the location of the feed stage.

Laying the groundwork
The reflux ratio is a key variable, affecting both the capital cost and the operating cost of a column. As the reflux increases, the number of stages and the column height both decrease but the flowrates in the column and, consequently, its diameter increase.

Despite that diameter increase, the capital cost of the column generally decreases as the reflux increases, because the savings in tower height more than offset the cost of the increase in diameter. However this is not the case at very high reflux ratios. And as alternatives having successively higher reflux ratios are compared with each other, there is a particular, high ratio at which the capital cost of the column begins to rise again [7]. In addition, the capital as well as the operating costs for the reboiler and condenser will rise in proportion to the vapor rate in the column.

Column optimization, therefore, reflects a balance between (1) the capital cost, which decreases (to a certain point, as just discussed) as reflux increases, and (2) the operating cost, which increases as the reflux increases. The total cost is minimum at an intermediate reflux ratio.

Generally speaking, the number of theoretical stages at the optimal reflux has been stated as being on the order of twice the minimum number of theoretical plates (corresponding to total reflux), and the optimal reflux ratio, \( R_{\text{opt}} \), as being in the range of 1.1 to 1.5 times the minimum reflux ratio, \( R_{\text{min}} \) [1]. A study described in this magazine over 30 years ago [19] evaluated a large number of cases, mainly via shortcut methods, and stated that \( R_{\text{opt}} \) lies between 1.1 and 1.6 times \( R_{\text{min}} \), the lower value being favored by high relative volatilities. Conversely, relative volatilities closer to unity and sharper separations were said to require higher values of \( R_{\text{opt}}/R_{\text{min}} \) within the above range. Since then, many articles and books have recommended estimates of \( R_{\text{opt}}/R_{\text{min}} \) for various situations, as summarized in Table 1. The range of recommended \( R_{\text{opt}}/R_{\text{min}} \) values in the open literature is 1.05 to 1.6, with the lower value for systems involving refrigerants and the higher value for systems using cooling water.

Despite the diversity in ranges in Table 1, the use of a rule-of-thumb on optimal reflux ratio as a certain multiple of the minimum reflux ratio has been widespread and, indeed, has proved beneficial over recent decades as a quick method to estimate optimum reflux ratio. But as mentioned earlier, the relative costs of equipment and energy (which affects utilities)
have been changing, particularly during the latter years of that time period. Furthermore, some of the early studies on optimal reflux ratio were based on shortcut calculation methods or graphical correlations, whereas today, rigorous calculations (with more-accurate results) can be made with ease.

Such calculations can assess the suitability of the heuristics on optimum reflux ratio with current cost data and, if necessary, update those heuristics. Furthermore, it is possible to determine whether, and how, the capabilities of commercial simulators for rigorous distillation simulation can also be used for optimizing reflux ratio. Both of these questions are addressed in what follows, by considering industrially relevant applications that involve both binary and multi-component mixtures. Along the way, we also scrutinize the validity of some other heuristics for distillation-column design.

Equations and data for sizing and costing of columns, including reboilers and condensers, are taken from the open literature. This study is limited to simple (but not necessarily binary) columns, each with a single feed stream and two product streams.

**Examples and procedures**

The 13 distillation examples also come from the open literature, for the most part. Seven examples have two components (Table 2); the others involve multiple components (Table 3). Besides showing the components, Tables 2 and 3 specify feed conditions, column pressure and product specifications for each system. In a few cases, the specifications were either unavailable in the original references or were modified to suit the needs of this study (for instance, the reflux ratio should not have a specified value).

The selected examples cover a wide range of design and operating conditions. Some operate at high pressures, others at atmospheric pressure. A few require a refrigerant as the cold utility. The number of stages for the examples ranges from 9 (short columns) to more than 100 (tall columns).

Steady state simulation and design of column for each example is done using HYSYS, the simulation system.
TABLE 3. DETAILS OF MULTICOMPONENT EXAMPLES

<table>
<thead>
<tr>
<th>Example No.*</th>
<th>Components</th>
<th>Feed Mole Fraction</th>
<th>Feed Conditions</th>
<th>Column Pressure</th>
<th>Product Purity Specifications (Mole %)</th>
</tr>
</thead>
<tbody>
<tr>
<td>8</td>
<td>Nitrogen CO₂ Methane Ethane Propyne n-Butane n-Butane</td>
<td>0.0020 0.0046 0.2412 0.2576 0.2561 0.1219 0.1166</td>
<td>140.85 kmol/h, 4,000 kPa, sat liq.</td>
<td>$P_{\text{cond}} : 1,378 \text{ kPa}$ (refrigerant) partial condenser (vapor distillate) $P_{\text{reb}} : 1,413 \text{ kPa}$</td>
<td>Top : 0.6% n-Butane Btm : 2% Propane</td>
</tr>
<tr>
<td>9</td>
<td>Propylene Oxide Propylene Glycol Water</td>
<td>0.0129 0.2296 0.7575</td>
<td>618.5 kmol/h, 120 kPa, sat liq.</td>
<td>$P_{\text{cond}} : 103 \text{ kPa}$ $P_{\text{reb}} : 117 \text{ kPa}$</td>
<td>Top : 2 X $10^{-5}$ % Propylene Glycol Btm : 0.5% Water</td>
</tr>
<tr>
<td>10</td>
<td>Propene Propane 1-Butene n-Butane n-Pentane</td>
<td>0.2158 0.1817 0.2010 0.2312 0.1703</td>
<td>1,000 lb-mol/h, 100 psia, sat liq.</td>
<td>$P_{\text{cond}} : 97 \text{ psia}$ (refrigerant) $P_{\text{reb}} : 100 \text{ psia}$</td>
<td>Top : 4.74% 1-Butene Btm : 2.54% Propane</td>
</tr>
<tr>
<td>11</td>
<td>Acetone Methanol Ethanol Water 1-Butanol</td>
<td>0.20 0.20 0.20 0.20 0.20</td>
<td>1,000 kmol/h, 101.3 kPa, sat liq.</td>
<td>$P_{\text{cond}} : 0.98 \text{ atm}$ $P_{\text{reb}} : 1 \text{ atm}$</td>
<td>Top : 2% Ethanol Btm : 2% Methanol</td>
</tr>
<tr>
<td>12</td>
<td>Propylene Propane 1,3-Butadiene n-Butane n-Pentane</td>
<td>0.0005 0.0002 0.0306 0.4160 0.2773</td>
<td>538 m.t/d, 6.29 atm, sat liq.</td>
<td>$P_{\text{cond}} : 431.5 \text{ kPa}$ $P_{\text{reb}} : 470.7 \text{ kPa}$</td>
<td>Top : 1% n-pentane Btm : 1% n-butane</td>
</tr>
<tr>
<td>13</td>
<td>Ethane Propylene Propane Propadiene n-Butane</td>
<td>0.0005 0.9500 0.0450 0.0030 0.0015</td>
<td>15 m.t/h, 1,457.4 kPa, sat liq.</td>
<td>$P_{\text{cond}} : 1,380 \text{ kPa}$ $P_{\text{reb}} : 1,450 \text{ kPa}$</td>
<td>Top : 2% Propane Btm : 50% Propane</td>
</tr>
</tbody>
</table>

*Sources of Examples: 8 and 9 from HYSYS Documentation; 10 from Van Winkle and Todd (1971); 11 from Ishii and Otto (2001); 12 and 13 from typical petrochemical industries.

**Items in italics indicate unavailable specifications, or ones modified to allow column optimization by varying the reflux ratio.**

**Thermodynamic package used:** Peng-Robinson for Examples 8, 10, 12 and 13; UNIQUAC for Example 9; and NRTL for Example 11.

**Frac**

$P_{\text{cond}}$ = pressure at condenser; $P_{\text{reb}}$ = pressure at reboiler.

**Coil**

- Cooling water for cold utility unless stated otherwise.

- Thermodynamic package used: Peng-Robinson for Examples 8, 10, 12 and 13; UNIQUAC for Example 9; and NRTL for Example 11.

- Items in italics indicate unavailable specifications, or ones modified to allow column optimization by varying the reflux ratio.

- Footnotes to Tables 2 and 3 spell out the thermodynamic models thus selected.

- For each example, the shortcut column in HYSYS is first used to estimate $D_{\text{min}}$, and the number of theoretical stages and the feed stage location for the chosen reflux ratio. These values then serve as the basis for rigorous simulation of the column with reboiler and either total or partial condenser (the latter is the choice when the feed contains non-condensable components). To satisfy the product specifications of each example in Tables 2 and 3, HYSYS adjusts the reflux ratio and other quantities suitably. Thus, the reflux ratio obtained by rigorous simulation is slightly different from that obtained earlier by shortcut calculations.

- After each rigorous simulation, the column, condenser and reboiler are sized, and their combined cost is estimated for optimization. The sizing pertains to the height and diameter of the distillation column and the design of the condenser and reboiler. The column diameter depends mainly on the velocity of the vapor stream within the column: to avoid excessive liquid entrainment or a high pressure drop, the maximum gas velocity, $V_{\text{max}}$, is calculated in meters per second by the following equation [14]:

$$V_{\text{max}} = \frac{0.171S_{\text{liq}}^2 + 0.27S - 0.047}{(C_{\text{liq}} - C_{\text{vap}})/C_{\text{vap}}}^{1/2}$$

where $S$ is tray spacing in meters and $C_{\text{liq}}$ and $C_{\text{vap}}$ are the liquid and vapor density, respectively.

- In our examples, the vapor velocity used for actual design is typically 80% of $V_{\text{max}}$. Because columns are customarily fabricated in increments of 0.5 ft in diameter, D, the diameters calculated are rounded up to the nearest half foot. This practice results in a lower vapor velocity and, hence, a more conservative estimate.

- Tray spacing, S, depends on the column diameter, and is at least 0.5 m for the sake of cleaning the trays [16]. Our designs take into account recommendations [18] that the tray spacing should be 0.5 m for columns with diameters up to 1 m, and that for wider columns, spacing should be a function of column diameter:

$$S = 0.5D^{0.3}$$

- The column height, H, is determined by multiplying the number of real trays by S and adding an extra space of 1.5 to 3 m (5 to 10 ft) both at the top of the tower for vapor-liquid disengagement and at the bottom for a liquid sump [3]. An overall efficiency of 70% is used to calculate number of real trays from the number of ideal trays in the simulation.

- The heat transfer areas of the condensers is estimated assuming an overall heat transfer coefficient of 510 W/(m²)K [13]. For the reboilers, a
conservative heat flux of 35,490 W/m², suggested by Reference [3], is used to estimate the required areas.

**Estimates of the costs**

Fixed capital is the capital needed for the plant to be ready for startup, and it represents the capital cost of all equipment, including installation and auxiliaries, that are needed for the complete process operation. Bare-module cost equations, expressed as a function of characteristic size of equipment by Reference [17], are used for estimating the capital cost of the columns, condensers and reboilers. However, these correlations are in many cases applicable for certain size ranges only. In examples where the size of the equipment exceeds the upper limit, then the usage of the minimum number of multiple units of that upper-limit size within the applicable range is assumed, for a conservative estimate.

As the cost data are historical and subject to inflation, the Chemical Engineering Plant Cost Index (CEPCI) is used to update capital and operating costs to January 2002 (CEPCI = 390.3). Annualized capital costs are found using an annualization factor of 15% to account for depreciation, interest and maintenance associated with the equipment.

The operating cost for distillation columns consists mainly of utility costs for heating in the reboiler and cooling in the condenser. In the examples, utility costs are estimated using cost equations given in Reference [18], which contain two separately escalating components. One is due to materials and labor, which inflates at a rate typified by the CEPCI, and the other is energy (fuel) cost, which escalates at a different rate. In this study, fuel price is taken to be $2.516/GJ based on a typical price of $0.40/gal for residual fuel oil in January 2002 (from http://www.eia.doe.gov/oil_gas/petroleum/data_publications/petroleum_marketing_monthly/pmm.html) with a heating value of 42 GJ/m³ [18]. All other cost data are also in U.S. dollars, and the column is assumed to operate for 8,500 hours per year (97% onstream time).

**Varying the reflux ratio**

We wish to find the reflux ratio that is optimal while continuing to meet the given product specifications, but the only way to do so in the rigorous simulation is by changing number of stages and feed stage. It was found that these two quantities could not be used as decision variables in the built-in optimizer of HYSYS. Following a suggestion from Hyprotech’s support group, Visual Basic programs were developed for optimizing the column by varying the number of stages and/or the location of the feed stage (in larger steps initially over a wider range, and then in single steps over a shorter range). The steps in the Visual Basic Program are as follows:

1. Select total number of stages, \( N_t \), and the feed stage, \( N_f \).
2. Transfer \( N_t \) and \( N_f \) to HYSYS, and instruct HYSYS to perform a rigorous simulation
3. Collect column data (for example, temperatures, flowrates, exchanger duties) in Excel
4. Based on those data, find the size and the cost the column, reboiler and condenser in Excel
5. Sort the costing results for the user to identify the optimal point.

**What was found**

The results of minimizing the total cost of each column by varying both the number of stages and feed stage are summarized in Table 4. In this table and Table 5, the number of stages excludes the reboiler and the condenser; they and the feed stage refer to theoretical or equilibrium stages. The feed stage is counted from the column top, with the condenser counted as zero.

Values of \( R_{\text{opt}} / R_{\text{min}} \) for many of the examples fall within the range of 1.05 to 1.6 as suggested in the literature (Table 1); the exceptions are Examples 2 and 7 with \( R_{\text{opt}} / R_{\text{min}} \) equaling 1.04 and 1.65 respectively.

Examples 1, 4 to 10, and 12 require short towers with 9 to 25 theoretical stages, which results in low capital cost. Example 2 entails a very high operating cost, as the separation requires a refrigerant and very large exchanger duties; also, the tall column and multiple heat exchangers for the large feedrate of 30,000 bbl/day mean a high capital cost.

Example 3 involves the difficult separation of propylene and propane, thus requiring a tower of over 100 ideal stages and hence incurring a large capi-

### Table 4: Selected Results from Rigorous Simulation and Optimization of All 13 Examples

<table>
<thead>
<tr>
<th>Example</th>
<th>Number of Stages*</th>
<th>Feed Stage#</th>
<th>Annualized Capital Cost, $/yr</th>
<th>Operating Cost, $/yr</th>
<th>Total Cost, $/yr</th>
<th>( R_{\text{opt}} )</th>
<th>( R_{\text{min}} )</th>
<th>( R_{\text{opt}} / R_{\text{min}} )</th>
<th>( N_{\text{min}} )</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>21</td>
<td>10</td>
<td>57,741</td>
<td>364,533</td>
<td>422,274</td>
<td>1.362</td>
<td>1.261</td>
<td>1.08</td>
<td>5.3</td>
</tr>
<tr>
<td>2</td>
<td>65</td>
<td>34</td>
<td>316,353</td>
<td>10,752,075</td>
<td>11,068,428</td>
<td>10.36</td>
<td>10.00</td>
<td>1.04</td>
<td>13.3</td>
</tr>
<tr>
<td>3</td>
<td>102</td>
<td>72</td>
<td>199,542</td>
<td>197,489</td>
<td>397,031</td>
<td>18.63</td>
<td>15.08</td>
<td>1.24</td>
<td>47.7</td>
</tr>
<tr>
<td>4</td>
<td>9</td>
<td>8</td>
<td>31,145</td>
<td>59,013</td>
<td>90,158</td>
<td>0.365</td>
<td>0.348</td>
<td>1.05</td>
<td>1.7</td>
</tr>
<tr>
<td>5</td>
<td>9</td>
<td>5</td>
<td>29,018</td>
<td>94,197</td>
<td>123,215</td>
<td>0.540</td>
<td>0.381</td>
<td>1.42</td>
<td>2.2</td>
</tr>
<tr>
<td>6</td>
<td>23</td>
<td>21</td>
<td>46,517</td>
<td>110,516</td>
<td>157,034</td>
<td>1.135</td>
<td>0.764</td>
<td>1.49</td>
<td>6.1</td>
</tr>
<tr>
<td>7</td>
<td>23</td>
<td>19</td>
<td>57,188</td>
<td>296,692</td>
<td>353,880</td>
<td>0.798</td>
<td>0.484</td>
<td>1.65</td>
<td>6.1</td>
</tr>
<tr>
<td>8</td>
<td>25</td>
<td>11</td>
<td>54,695</td>
<td>170,794</td>
<td>225,489</td>
<td>0.491</td>
<td>0.441</td>
<td>1.11</td>
<td>8.7</td>
</tr>
<tr>
<td>9</td>
<td>21</td>
<td>18</td>
<td>77,954</td>
<td>795,940</td>
<td>873,900</td>
<td>0.080</td>
<td>0.050</td>
<td>1.60</td>
<td>5.3</td>
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<tr>
<td>10</td>
<td>18</td>
<td>8</td>
<td>55,025</td>
<td>902,703</td>
<td>957,729</td>
<td>0.961</td>
<td>0.778</td>
<td>1.24</td>
<td>3.9</td>
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<tr>
<td>11</td>
<td>48</td>
<td>19</td>
<td>194,885</td>
<td>1,343,503</td>
<td>1,538,388</td>
<td>2.019</td>
<td>1.730</td>
<td>1.17</td>
<td>10.0</td>
</tr>
<tr>
<td>12</td>
<td>25</td>
<td>13</td>
<td>75,286</td>
<td>428,830</td>
<td>504,117</td>
<td>0.771</td>
<td>0.727</td>
<td>1.06</td>
<td>8.0</td>
</tr>
<tr>
<td>13</td>
<td>105</td>
<td>42</td>
<td>452,312</td>
<td>1,617,538</td>
<td>2,069,850</td>
<td>6.081</td>
<td>5.215</td>
<td>1.17</td>
<td>37.0</td>
</tr>
</tbody>
</table>

* Excluding reboiler and condenser.
# Counted from the top with condenser as zero.
@ Minimum reflux ratio and minimum number of stages (excluding reboiler and condenser) obtained from shortcut calculations.
Cost totals may not agree with cost components due to rounding.
Revelations about the feed stage

In addition to the above findings, a closer analysis of the results for various \( R_{opt}/R_{min} \) values indicated that the feed stage given by shortcut column calculations can be inappropriate. The most extreme case is Example 4, for which increase in the total cost ranged from 70 to 260% with \( R_{opt}/R_{min} \) in the range 1.1 to 1.6. One can see from the optimized results that the feed stage from the shortcut calculations (for instance, for \( R_{opt}/R_{min} \) equaling 1.2 in Table 5) is very different from the feed stage in Table 4, even if the total number of stages is comparable.

In fact, Reference [7] points out that the guideline for optimal feed stage is that the ratio of key-component mole fractions in the liquid on the feed stage should be close to the corresponding ratio in the liquid part of the feed. The key-ratio plot in Figure 1 for Example 4 indicates that the feed-stage location should be closer to the reboiler. The feed stage in the optimized design is consistent with the heuristic given in Reference [7].

A recent reference [8] states that the optimal feed location for a specified total number of stages and separation minimizes the reflux ratio (and therefore the reboiler and condenser duties). In accordance with this guideline, the feed stage for the case of \( R_{opt}/R_{min} \) equaling 1.2 in Table 5 is optimized by varying the feed stage in the rigorous simulation and finding the reflux ratio to achieve the desired separation. These optimized results after feed stage optimization are shown in the last three columns of Table 5.

A separate exercise was carried out to optimize the feed stage by minimizing the total cost for the case of \( R_{opt}/R_{min} \) equaling 1.2 for all examples. These results are identical to those obtained by feed stage optimization via minimizing the reflux ratio. This equivalence is to be expected, as the total cost is often dominated by operating cost when the total number of stages is fixed.

<table>
<thead>
<tr>
<th>Example</th>
<th>Results for ( R_{opt}/R_{min} = 1.2 )</th>
<th>Results for ( R_{opt}/R_{min} = 1.2 ) after feed stage optimization</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Number of stages</td>
<td>Feed stage</td>
</tr>
<tr>
<td>1</td>
<td>15</td>
<td>7</td>
</tr>
<tr>
<td>2</td>
<td>29</td>
<td>20</td>
</tr>
<tr>
<td>3</td>
<td>97</td>
<td>58</td>
</tr>
<tr>
<td>4</td>
<td>8</td>
<td>3</td>
</tr>
<tr>
<td>5</td>
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</tr>
<tr>
<td>6</td>
<td>18</td>
<td>10</td>
</tr>
<tr>
<td>7</td>
<td>18</td>
<td>8</td>
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</tr>
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<td>12</td>
<td>22</td>
<td>10</td>
</tr>
<tr>
<td>13</td>
<td>78</td>
<td>42</td>
</tr>
</tbody>
</table>

Note: \% increase in total cost is from the minimum total cost shown in Table 4.
Summarizing the conclusions
Column optimization through rigorous simulation, sizing and costing commonly gives an $R_{\text{opt}}/R_{\text{min}}$ value in the range of 1.1 to 1.6. Also, the heuristic that the optimal number of stages is twice the minimum number is generally not valid.

Shortcut (as opposed to rigorous) calculations using the heuristic, $R_{\text{opt}}/R_{\text{min}} = 1.1$ to 1.6, produce columns whose total cost is generally more than the minimum. For the specific case of $R_{\text{opt}}/R_{\text{min}}$ equaling 1.2, the total cost of a column by shortcut calculations (followed by rigorous simulation, sizing and costing) is on average 14% higher than the minimum attainable by rigorous simulation and optimization. However, the design in this case can often be improved substantially by optimizing the feed stage (for a specified number of stages and separation), and the total cost of a column can be reduced to within 4% of the minimum.

In a few cases, potential exists for further cost reduction by varying both the number of stages and feed stage, and simulating the column rigorously. These findings are applicable to simple columns with a single feed stream and two product streams only.

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References