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CHAPTER 3

A Urea Synthesis Loop Whose Controlling Ratio Is Never Measured

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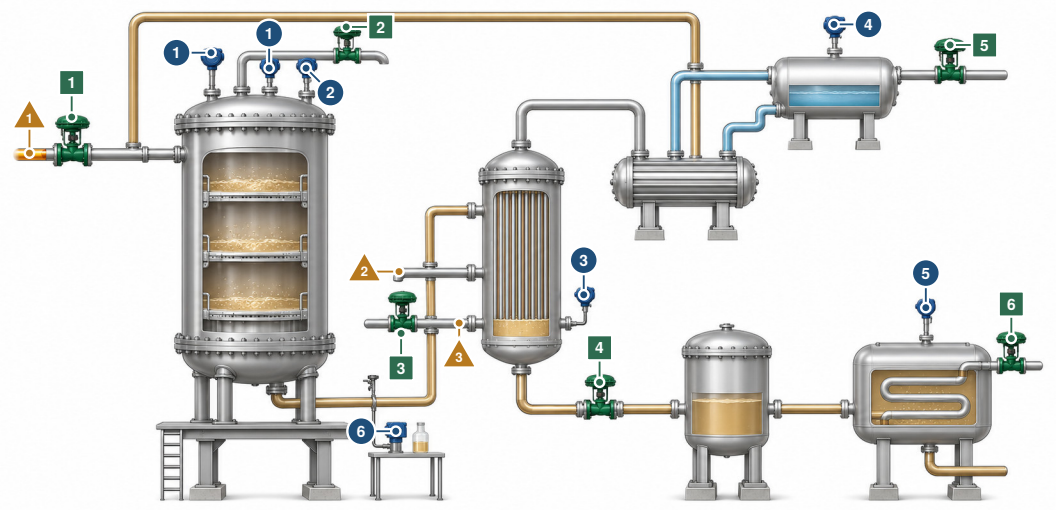
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CHAPTER 3

A Urea Synthesis Loop Whose Controlling Ratio Is Never Measured



The controller moves 1 fresh ammonia, held in ratio to the carbon dioxide 2 inert vent valve 3 stripper steam valve 4 stripper level valve 5 steam drum valve, the reactor's heat 6 evaporator steam valve

The controller reads 1 density and temperature at the reactor top, from which the ratio is inferred 2 reactor pressure 3 stripper level 4 steam drum pressure 5 evaporator temperature 6 laboratory sample every four hours

What disturbs it 1 wetter ammonia 2 a carbon dioxide turndown 3 a dip in the stripper steam

Nothing in the picture reads the ratio of ammonia to carbon dioxide that decides the run, only a density and a temperature on the reactor and a laboratory sample taken hours apart. Illustration, not to scale.

IN PLAIN TERMS A Mixing Ratio Nobody Can See

The main idea. A urea plant joins ammonia and carbon dioxide under high pressure and sends what does not react around the loop again. The amount of spare ammonia in the reactor decides how much urea forms and whether the steel lining stays safe.

The challenge. No instrument can read that amount, so it can drift while both feed meters look normal. The plant guesses it from how heavy and how hot the liquid is, and a slow lab test fixes the guess hours later.

Why it matters. Too little ammonia lets the hot liquid eat the lining, and too much drives up the pressure.

THREE WAYS TO READ THIS CHAPTER

Explore	12 min	the first page; the history and fact boxes of Sections 3.2 and 3.3; the try-it box of Section 3.4; the try-it and fact boxes of Section 3.6; the key points
Study	45 min	the whole chapter with its pause points and the exercises
Practice	10 min	the first page; Section 3.6 with its result chart; Section 3.7; Section 3.8 with its open challenge

The control question The molar ratio of ammonia to carbon dioxide in a urea reactor sets the conversion, the pressure the liquid needs and whether the stainless liner stays passive, and no online instrument measures it. Can a ratio held on the two feed flows hold it, and what do an inference from the reactor's density and temperature, corrected by a laboratory that reports 45 min late, and a predictive controller add?

Learning objectives

- Explain why the reactor N/C is an inventory that integrates every unmeasured error in the fresh feed, and estimate how fast wet ammonia drains it.
- Compute the inferred N/C from density and temperature, and the laboratory bias update made against the inference at the sampling instant.
- Evaluate the corrosion index and say how the reactor temperature and the passivation air move the floor.
- Explain how the carbamate condenser sets the reactor temperature, and why a cascade onto the steam drum restores it at the cost of biuret.
- Compare ratio control on the flows, a PI on the inference and a predictive controller over a twelve-hour shift, including the margin each one leaves.

Prerequisites Sections 1.4.3 (a recycle whose excess has no exit), 1.4.6 (ratio stations, inferred compositions and the bias update), 1.6.2 (velocity form) and 1.6.4 (scenarios and exported files). Section 1.6.3 is needed only for the predictive controller, and Chapter 2 gives a second synthesis loop with the same recycle arithmetic.

The app https://apmonitor.com/apps/urea-plant/

See also Volume 5, Chapter 10 for a soft sensor whose bias a sampled and delayed laboratory corrects, and Volume 5, Chapter 3 for composition control through a slow analyzer. Volume 3, Chapter 7 for a ratio blend trimmed on a composition measurement, and Volume 3, Chapter 6 for a feedforward dose demoted to a slow ratio trim. Volume 6, Chapter 7 for the snowball effect of a reactor and column recycle, and Volume 6, Chapter 8 for a plant whose inventories must close before anything else.



3.1 Introduction

The variable a urea control room cares most about has no transmitter. In the CO_2-stripping loop simulated here, 45.8 t/h of carbon dioxide and 35.83 t/h of ammonia make 62.5 t/h of urea at 140 bar a, and the reactor outlet carries ammonia and carbon dioxide in the molar ratio $N/C = 3.000$ on the initial-mixture basis. Ammonia beyond the two moles each urea molecule takes stays in the loop. When the ammonia arrives with 2.0 instead of 0.2 weight percent water at 1 h, a ratio held on the measured flows lets the reactor's N/C fall to the 2.80 corrosion floor at 7 h 49 min, while the laboratory's first sample, drawn at 4 h, reports 2.918 at 4 h 45 min. A ratio inferred from the reactor's density and temperature had already crossed 2.93 at 3 h 42 min, because free ammonia is the lightest component of the synthesis liquid.

The simulator is aimed at a common misconception, that a ratio held between two measured feeds is a ratio held in the process. A ratio station is a feedforward on the feeds, as in Section 1.4.6, and the quantity downstream of a recycle is an inventory that integrates whatever the feeds get wrong. The general remedy is inferential control, estimating an unmeasured product quality from secondary measurements [1] and maintaining that estimate against slow, accurate analyses, which is the role soft sensors play across the process industries [2]. The milk powder dryer of Volume 5, Chapter 10 corrects one such soft sensor from a sampled and delayed laboratory. Recycles also add dynamics of their own, among them the snowball effects of reactor and separator processes [3] that Volume 6, Chapter 7 measures. The same structure recurs wherever a reactor's feed ratio is held in a loop of unconverted reactants, among them ammonia synthesis (Chapter 2), methanol synthesis, hydrocracker hydrogen and polymerization monomer recovery.

Urea synthesis has been modeled for decades, in a simulation program for urea plants [4], as a thermodynamic framework and reactor model [5, 6], as the synthesis section of an industrial plant [7] and as a high-pressure loop [8], and its phase and reaction equilibria have been brought together in

one thermodynamic model of the synthesis [9]. However, those models are built to answer design and rating questions, and none of them hands a student an N/C that drifts while every flow meter reads its setpoint. This chapter works with a browser simulator of a CO_2-stripping urea loop whose true ratio, inferred ratio and laboratory reports are logged side by side, and it measures what ratio control on the flows, a laboratory-corrected inference and a predictive controller each hold through drift, turndown, a stripper steam dip and a rate cut. The balances, the correlations, the velocity-form controller and the linear predictive controller are standard. The measurements on this one synthetic plant show, among other things, that the corrosion floor is reached by wet ammonia rather than by a CO_2 turndown, which raises the ratio. The model has 39 states whose nitrogen, carbon, hydrogen and oxygen ledgers close to 3×10^{-11} kmol over twelve hours, and every controller reads a measurement packet only. Nine graded cases judge both what the learner does and what the plant does. Chapter 1 treats the recycle and the inferred composition in general terms in Sections 1.4.3 and 1.4.6.

Section 3.2 follows the process from carbamate to the total-recycle loop. Section 3.3 sets out the balances, the teaching correlations, the instruments and the inference. Section 3.4 measures what the loop does before it is controlled: the ratio integrates, the condenser sets the reactor's heat and the recycle stores a disturbance. Section 3.5 describes the loops and the three ways to hold the ratio, and Section 3.6 measures the nine cases and the twelve-hour challenge. Section 3.7 names the hardware and the practice behind the model, Section 3.8 states where the model stops holding, and Section 3.9 collects the measured results.

3.2 From Carbamate to a Total-Recycle Loop

In the industrial route, whose origin the history box at the end of this section tells, ammonia and carbon dioxide combine to ammonium carbamate, which then loses water to give urea. The first step is fast, exothermic and nearly complete above about 135 °C at pressures above the carbamate's decomposition pressure, while the second is slow, slightly endothermic and never complete, so every process separates the unconverted carbamate and returns it to the reactor. Because the carbamate is volatile at these temperatures the synthesis must run at high pressure, typically 170 °C to 200 °C and 130 to 300 bar, and both the corrosion of the liquid and the rate of urea formation rise roughly exponentially with temperature [10].

The equilibrium was measured and modeled in steps. Equilibrium pressures of the CO_2–NH_3–urea–water system were measured as a function of the loading density of the vessel in 1951 [11]. A single overall equilibrium constant for the liquid, proposed in 1948, remained the most widely used model, and a later model with free ammonia and carbon dioxide, carbamate, urea and water in the liquid and a vapor phase was presented in 1972 [10]. The densities of the synthesis solution in chemical equilibrium were measured for N/C from 2.04 to 6.3 and fitted by an empirical equation [12], which is the physical basis for inferring the ratio from a density. A phase model of the same system places minimum bubble points and a ternary azeotrope in the composition plane [13]. The Gorlovskii–Kucheryavyi equilibrium conversions tabulated with a 1993 reactor model [5] and the conversions and bubble pressures of a 2016 thermodynamic model [9] are the published values this chapter compares the simulator's correlations with.

Process design moved from once-through plants to total recycle, in which the reactor effluent passes a decomposer or stripper and the recovered ammonia and carbon dioxide return to the synthesis [10]. In a CO_2-stripping plant both reactants return together with water as carbamate, so the concentration of urea in the reactor liquid, rather than the CO_2 conversion alone, decides the optimum, and that yield passes through a maximum near $N/C = 3$. The same review observes that the reactor is a bubble column of boiling liquid whose endothermic reaction is heated by condensing gas, that the total pressure is a system pressure plus the pressure of inerts that include the passivation air, and that the temperature rise along the reactor indicates the conversion [10]. The worldwide use of urea as a fertilizer has escalated far enough to be studied as a global change that contributes to coastal eutrophication [14]. None of the sources consulted here documents how a commercial plant's

Table 3.1 Variables, Their Roles and Design Values (Case 1, $t = 0$)

Variable	Role	Design	Range
N/C	CV, true (truth only)	3.000	SP 2.7–3.4
$\widehat{N/C}$	CV, inferred	3.014	—
R	MV, fresh NH_3/CO_2 ratio	2.0173	1.80–2.30
S	MV, stripper steam ratio	1.15 t/t	0.8–1.5
p, v	CV, MV: pressure, vent	140.0 bar a, 39.7 %	SP 130–150
L	CV, stripper level	50 %	SP 20–80
p_s	CV, steam drum	4.50 bar a	SP 3.6–6.5
T	CV of the cascade, reactor top	185.02 °C	SP 175–192
T_e	CV, evaporator	138.0 °C	SP 130–145
—	DV, CO_2 load	100 %	50–115
—	DV, ammonia purity	99.8 wt%	96–100
O_2	DV, passivation air	0.6 vol%	0–1
—	DV, HP steam supply	80 t/h	30–100 %
—	DV, CO_2 meter, density gauge	0, 0	±5 %, ±10 kg/m^3
X, H_2O/C	outputs, reactor outlet	61.11 %, 0.406	—
ρ, p_b	outputs	970.6 kg/m^3, 127.6 bar	—
B, w	outputs, product	0.748, 0.300 wt%	—
I_c	output, corrosion index	0.018	stop at 1

control system is configured, so the simulator's loops are a study of the structure above rather than a description of practice.

> ⧖ HISTORY **From Wöhler's Urea to a Loop Under Pressure, 1828 to 1932**
>
> Wöhler reported the artificial formation of urea in 1828 [15], but the industrial route is a different one, through ammonium carbamate. It was first proposed by Basaroff and carried into manufacture by German chemists about 1920 [10]. In his Nobel lecture of 1932, Carl Bosch described the difficulty. Carbon dioxide and ammonia heated to 130 to 140 °C in a closed vessel give urea and water, but stop at about 40 percent of the theoretical yield, and on slow cooling the water would turn most of the urea back, and the use of heat would be very poor. His company therefore separated the mixture at high pressure, drew off the excess ammonia and the carbon dioxide with heated compressors and condensed them again, making about 40,000 tons of urea a year [16]. The total-recycle loop of Figure 3.1 returns, in the same way, what its reactor leaves unconverted.

3.3 The Loop as Modeled

The plant is a generic 1500 t/d CO_2-stripping loop with no licensor's data, as shown in Figure 3.1. The reactor overflows into a falling-film stripper heated by high-pressure (HP) steam, where the fresh CO_2 rises as stripping gas. The stripper gas condenses to carbamate in the HP carbamate condenser, whose shell raises low-pressure (LP) steam in a drum, and the liquid and the uncondensed gas flow into the bottom of the reactor, where the gas finishes condensing and supplies the reactor's heat. The fresh ammonia and the carbamate recycle from the LP section enter the condenser. The stripper's liquid passes the LP section, a urea solution tank and an evaporator that makes the melt. The engine's clock is the minute, species flows are in kmol/min, heat in MJ/min, pressures in bar absolute, and feed flows are reported in t/h.

Table 3.1 lists each variable's role and its value at the design point of case 1. The integrated state holds four apparent species and a temperature in each reactor cell (15), the stripper's bottoms holdup (4), the LP recycle holdup (3), the urea solution tank (2), the inerts in the reactor gas space, the drum pressure, the evaporator temperature and its biuret, seven actuator positions and four element ledgers, 39 in all.

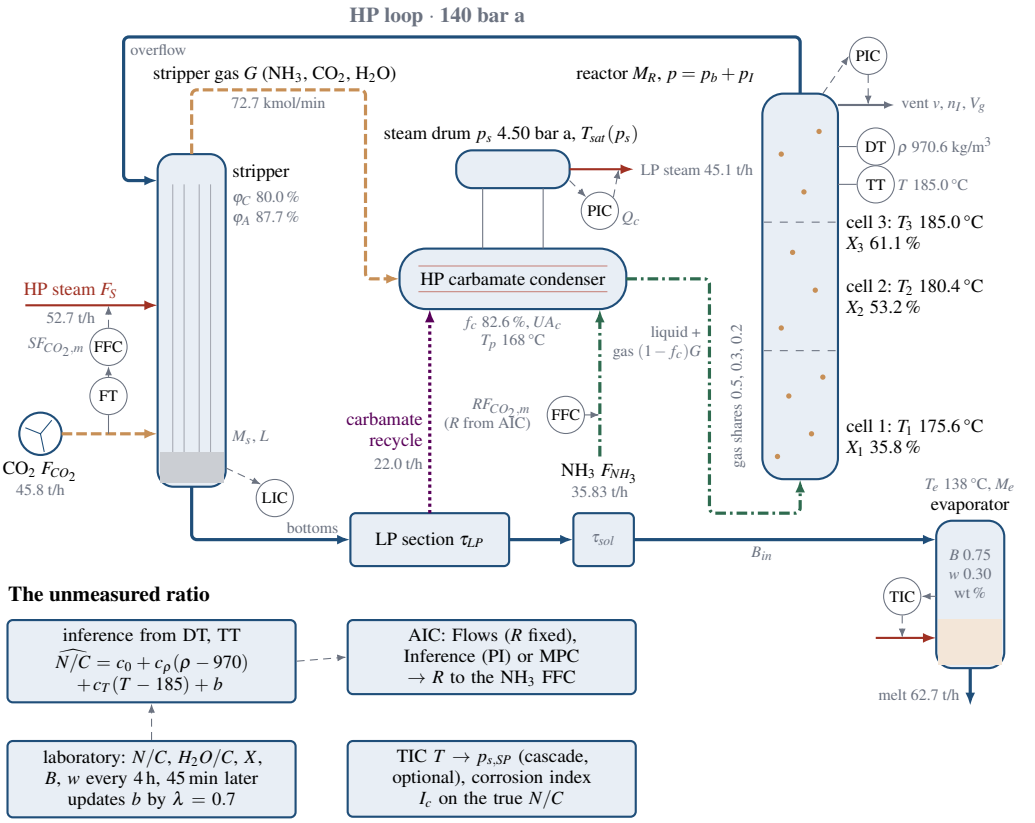


Figure 3.1 The CO_2-stripping synthesis loop as modeled, redrawn from the Loop view of case 1 at $t = 0$, the design point. The stripper gas G (amber, dashed) goes to the HP carbamate condenser, which condenses a fraction f_c and raises LP steam in a drum at p_s. The liquid and the uncondensed gas $(1 - f_c)G$ (green, dash-dotted, as is the fresh ammonia) enter cell 1 of the three-cell reactor, and the overflow (navy, solid) returns to the stripper, which strips fractions φ_C and φ_A with the fresh CO_2 and HP steam F_S. The carbamate recycle (violet, dotted) returns through the LP section with lag τ_{LP}, and the urea solution passes a tank τ_{sol} to the evaporator at T_e. DT and TT on the reactor top feed the inference (lower left), which the laboratory corrects. Process model only, not a piping diagram. Not to scale.

3.3.1 Species, Ratios and the Reactor Cells

Every holdup carries apparent ammonia A, carbon dioxide C, urea U and water W in kmol. Carbamate formation is taken as instantaneous and complete while ammonia is in excess, so the carbamate is $\min(C, A/2)$, and the ratios the industry quotes are those of the initial mixture [10], as shown in Equation (3.1).

$$N/C = \frac{A + 2U}{C + U}, \quad \mathrm{H_2O/C} = \frac{W - U}{C + U}, \quad X = \frac{U}{C + U} \tag{3.1}$$

where X is the CO_2 conversion. Dehydration changes neither ratio, so they describe the feed a cell received. The reactor is three equal cells of 45 t in series, and each balances its species and its heat,

as shown in Equation (3.2).

$$\begin{aligned}
\frac{\mathrm{d}A_k}{\mathrm{d}t} &= A_k^{in} + g_{A,k} - f_k A_k - 2r_k - \delta_{k3}\, n_v \\
\frac{\mathrm{d}C_k}{\mathrm{d}t} &= C_k^{in} + g_{C,k} - f_k C_k - r_k \\
\frac{\mathrm{d}U_k}{\mathrm{d}t} &= U_k^{in} - f_k U_k + r_k \\
\frac{\mathrm{d}W_k}{\mathrm{d}t} &= W_k^{in} + g_{W,k} - f_k W_k + r_k \\
r_k &= k(T_k)\,(C_k + U_k)\,\big(X_{eq,k} - X_k\big) \\
k(T) &= k_{185}\exp\Big[-\frac{E_d}{R}\Big(\frac{1}{T} - \frac{1}{T_{185}}\Big)\Big] \\
M_k c_{pL}\frac{\mathrm{d}T_k}{\mathrm{d}t} &= \dot{m}_k^L c_{pL}(T_k^{in} - T_k) + \dot{m}_k^G c_{pG}(T_p - T_k) \\
&\quad + q_G(g_k) - \Delta H_d\, r_k
\end{aligned} \tag{3.2}$$

where $g_k = s_k(1-f_c)G$ is the share $s_k = 0.5, 0.3, 0.2$ of the condenser's uncondensed gas that condenses in cell k, f_k is the outflow per unit holdup, $\dot{m}_k^L$ and $\dot{m}_k^G$ are the liquid and gas mass flows entering, $T_p = 168\,^{\circ}\mathrm{C}$ is the condenser's process-side temperature, and n_v is the ammonia lost with the vent. The condensation heat is $q_G = \Delta H_c \min(C, A/2) + 20A_{free} + 40W$ in MJ/kmol with $\Delta H_c = 100\,\mathrm{kJ/mol}$, and the dehydration takes $\Delta H_d = 15.5\,\mathrm{kJ/mol}$. Cells 1 and 2 overflow what they receive, and cell 3 overflows $\dot{m}_{out} = \bar{m} + (M_3 - \bar{M}_3)/\tau_{ov}$ with $\tau_{ov} = 0.5\,\mathrm{min}$. At design the cells run at 175.58, 180.40 and 185.02 °C with conversions of 35.80, 53.22 and 61.11 % against an equilibrium of 66.26 % at the top, and the liquid stays 47.7 minutes in the reactor. The rising temperature along the reactor reflects the conversion, as the industrial reactor's does [10].

> **+ DID YOU KNOW? Urea, the Leading Solid Nitrogen Fertilizer**
>
> In 2007 urea was the most used solid nitrogen fertilizer in the world, with an output above 100 million metric tons a year and new plants of typically 2000 tonnes a day, some up to 3500 [17]. In the United States in 2024 it was the derivative of ammonia made in the largest quantity [18]. This chapter's plant makes 1500.

3.3.2 Equilibrium Conversion, a Teaching Correlation

The equilibrium conversion is a polynomial in $a = N/C$, $b = \mathrm{H_2O/C}$ and $\tau = T/100$ with T in °C, as shown in Equation (3.3).

$$\begin{aligned}
X_{eq} = {} & 0.2616a - 0.01945a^2 + 0.0382ab - 0.1160b \\
& - 0.02732a\tau - 0.1030b\tau + 1.640\tau \\
& - 0.1394\tau^3 - 1.869
\end{aligned} \tag{3.3}$$

clamped to $2 \le a \le 5$, $0 \le b \le 1.5$ and 140 °C–210 °C, with a declared shape for a from 2.2 to 4.5. It is a teaching correlation of the published shape, not a fit to any data set. Its conversion rises with N/C (64.10, 66.37 and 75.39 % at 2.8, 3.0 and 4.0 for $\mathrm{H_2O/C} = 0.4$ and 185 °C), falls with water (62.53 % at $\mathrm{H_2O/C} = 0.6$) and peaks in temperature near 190 °C (190.4 °C at $N/C = 3$). Against the 34 Gorlovskii–Kucheryavyi equilibrium conversions tabulated by Isla, Irazoqui and Genoud for N/C 3.5 to 5 at 190 °C and $N/C = 4$ from 180 to 210 °C [5], it runs from 1.5 points below to 2.8 points above, 1.3 points above on average. Against eleven points read off the conversion curves of a process paper whose sources include the Ullmann's encyclopedia [10], for N/C from 2.2 to 5.0 and 165 to 210 °C, it runs 0.6 to 3.1 points above, and 2.4 to 3.1 points above for N/C up to 3. Against Voskov and Voronin's model [9] at N/C 2.5 and 3, 180 and 200 °C and $\mathrm{H_2O/C}$ up to 0.6 it runs 4.4 to 9.1

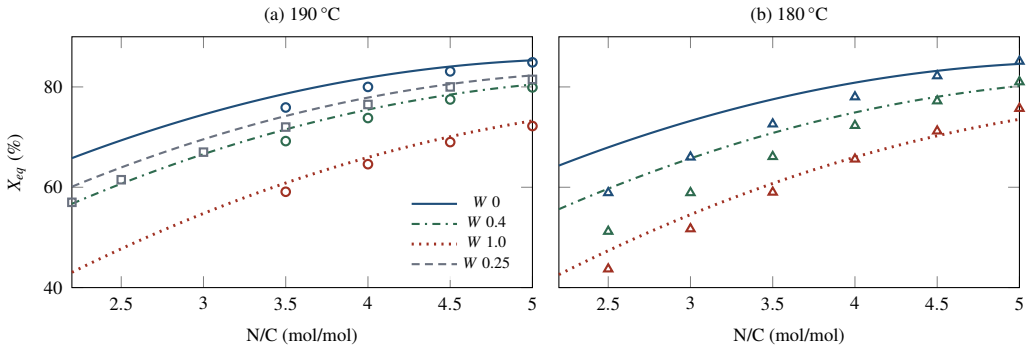


Figure 3.2 The equilibrium conversion correlation (lines) against published values (marks) at $H_2O/C = W$. The water ratios differ by line style and mark fill as well as by color, from a solid line and open marks for the dry liquid to a dotted line and filled marks for the wettest, as in the key. (a) At 190 °C, circles are the Gorlovskii–Kucheryavyi conversions tabulated by Isla et al. and squares the curve reproduced by Brouwer at $W \approx 0.25$ (gray dashed line). (b) At 180 °C, triangles are the Voskov–Voronin model. The correlation's declared range ends at $N/C = 4.5$.

points above, and that model itself lies about 3 to 5 points below the other two near $N/C = 3$. At the design point the correlation gives 66.4 % where the curve reads about 64.5 and Voskov–Voronin 59.3. The comparison is drawn in Figure 3.2.

The shape matters more than the level for the lessons. The plan for the application asked for a conversion maximum near N/C 3 to 3.5, but none of the three sources has one, since the published conversion rises with N/C throughout. The correlation rises too, and the maximum near 3 appears where the published literature puts it, in the urea made per kilogram of synthesis liquid, $X_{eq}M_U/(aM_A + M_C + bM_W)$. It peaks at $N/C = 2.98$ at $H_2O/C = 0.4$ and 185 °C and at 2.56 at $H_2O/C = 0.25$ and 190 °C, where the published yield curve peaks near 3 [10]. The maximum is flat, so its location is sensitive to the correlation. The water slope at $N/C = 3.5$ and 190 °C, −0.178 per unit of H_2O/C, is close to the curve's −0.17, and the temperature maximum at $N/C = 3.5$, $H_2O/C = 0.25$ is 190.6 °C against about 193 °C on the published curve.

3.3.3 Stripper, Condenser and Steam Drum

The stripper decomposes fractions of the reactor overflow's CO_2, ammonia and water from the CO_2 and the steam it receives per unit of liquid, relative to design, and the condenser condenses a fraction of the stripper gas set by the duty its shell can take, as shown in Equation (3.4).

$$\begin{aligned}
\varphi_C &= 1 - \mathrm{e}^{-\eta(k_g r_g + k_q r_q)} \\
\varphi_A &= 1 - (1 - \varphi_C)^{1.3}, \qquad \varphi_W = 0.08\sqrt{r_q} \\
f_c &= 1 - \mathrm{e}^{-2z} \\
z &= \frac{UA_c\big(T_p - T_{sat}(p_s)\big) + Q_f - Q_g}{q_G(G)} \\
Q_c &= f_c\, q_G(G) + Q_g - Q_f \\
C_s \frac{\mathrm{d}p_s}{\mathrm{d}t} &= \frac{Q_c}{\lambda_{LP}} - K_s v_s \sqrt{p_s^2 - p_h^2}
\end{aligned} \tag{3.4}$$

where r_g and r_q are the CO_2 and the steam per kilomole of liquid on the CO_2 basis over their design values, $k_g = 0.5$, $k_q = 1.109$, η is the stripper efficiency factor, Q_f is the heat that warms the fresh ammonia from 60 and the recycle from 100 °C to T_p, Q_g the heat the gas gives up cooling from

185 °C, T_{sat} the IAPWS-IF97 saturation temperature, $\lambda_{LP} = 2.12\,\mathrm{MJ/kg}$, v_s the LP steam valve and $p_h = 3.5\,\mathrm{bar\,a}$ the header. At design the stripper strips 80.0 percent of the CO_2 and 87.7 percent of the ammonia with 52.67 t/h of steam, the gas is 72.72 kmol/min, and the condenser condenses 82.6 percent of it and raises 45.06 t/h of steam at 4.50 bar a ($T_{sat} = 147.91\,^{\circ}\mathrm{C}$). The conductance $UA_c = 84.8\,\mathrm{MJ/(min\,K)}$ was sized so that the top cell sits at 185 °C. The exponential condensed fraction is a teaching closure in the manner of a number of transfer units, which replaced a clipped linear fraction under which the reactor and the condenser oscillated together. The gas the condenser leaves heats the reactor, so the drum pressure is the reactor's temperature handle, 11.6 K per bar from 4.2 to 4.8 bar a and 15.1 K per bar from 4.5 to 5.0 at the lined-out design.

3.3.4 Pressure, Recycle and Evaporator

The reactor pressure is the liquid's bubble pressure plus the inerts that collect in the gas space at the top [10], as shown in Equation (3.5).

$$\begin{aligned} p &= p_b + \frac{n_I R T_3}{V_g} \\ p_b &= 124\,\mathrm{e}^{0.018(T_3-185)+0.40(a-3)-0.30(b-0.5)} \\ \frac{\mathrm{d}n_I}{\mathrm{d}t} &= F_C\left(y_I + \frac{\mathrm{O_2}}{21}\right) - K_v v\sqrt{p}\,\frac{p-p_b}{p} \end{aligned} \tag{3.5}$$

where $V_g = 3\,\mathrm{m^3}$, $y_I = 0.005$, $K_v = 1.4$ and the vent loses 0.3 mole of ammonia per mole of inert. At design $p_b = 127.60\,\mathrm{bar}$ and the inerts hold 12.40 bar. The bubble pressure is a teaching correlation. Against Voskov and Voronin's model, fitted to measured bubble points, it gives 127.8 bar where theirs gives about 135.6 bar at $N/C = 3$, $\mathrm{H_2O/C} = 0.4$ and 185 °C [9]. Its temperature slope, 0.018 per kelvin in $\ln p$, is below their 0.024 (160–180 °C) and 0.028 (180–200 °C), its water slope of -0.30 is three times their -0.10, and its N/C slope of 0.40 is six times their 0.064 from 3.0 to 3.5. Below 3 their bubble pressure falls with N/C (-0.36 from 2.5 to 3.0), consistent with the minimum bubble points of the phase model [13]. The model therefore exaggerates the ammonia's share of a pressure rise and understates the temperature's.

The stripper's liquid holdup drains through a level valve. Its unstripped ammonia (less a 2 percent slip to the evaporator), carbon dioxide and enough water to make a 45 weight percent carbamate solution return to the condenser through a first-order holdup with $\tau_{LP} = 15\,\mathrm{min}$, 21.95 t/h at design. The urea and the remaining water pass a solution tank of $\tau_{sol} = 30\,\mathrm{min}$, a teaching choice that keeps the level loop's transients out of the evaporator. The evaporator balances its heat and its biuret, as shown in Equation (3.6).

$$\begin{aligned} M_e\frac{\mathrm{d}B}{\mathrm{d}t} &= (B_{in} - B)\,\dot{m}_{melt} + M_e k_b \\ k_b &= 0.0585\,\mathrm{e}^{-\frac{E_b}{R}\left(\frac{1}{T_e}-\frac{1}{T_{138}}\right)}\,\frac{1.5}{1+0.5\,n} \\ B_{in} &= 0.30\sqrt{U_0/U}, \qquad w = 0.30\,\mathrm{e}^{-(T_e-138)/4} \end{aligned} \tag{3.6}$$

where B and w are the biuret and moisture in weight percent, $M_e = 8\,\mathrm{t}$, $E_b = 100\,\mathrm{kJ/mol}$, n is the ammonia reaching the evaporator per unit of urea relative to design, and U/U_0 is the urea flow relative to design, with B_{in} capped at three times its design value. The square-root law for the biuret formed upstream is a teaching law, chosen so that the quality window can close at low rate. At design the melt stays 7.66 min and carries 0.748 wt% biuret and 0.300 wt% moisture.

3.3.5 Corrosion Index, Instruments and the Inference

The liner's corrosion is a teaching index on the true N/C at the reactor top, as shown in Equation (3.7).

$$I_c = \mathrm{e}^{(2.8-N/C)/0.05}\sqrt{0.6/\mathrm{O_2}}\;\mathrm{e}^{(T-185)/15} \tag{3.7}$$

The index is 1 at the floor of 2.8 with design passivation air and temperature, and it stops the run once it has stayed at 1 or above for 20 min (the app reports an exercise stop). The floor, the hold and the alarm at $N/C = 3.3$ are teaching values, and real limits come from the licensor's and the liner's corrosion data. The instruments read the CO_2 and ammonia flows, the reactor top's density (lag 0.5 min, noise 0.6 kg/m^3) and temperature (lag 0.3 min, noise 0.08 K), the pressure, the level, the steam flows, the drum pressure and the evaporator temperature. A laboratory samples the reactor outlet's N/C, H_2O/C and conversion and the product's biuret and moisture every 4 h and reports 45 min later, with a standard deviation of 0.01 on the N/C. The density follows the partial molar volumes of free ammonia, carbamate, urea and water, 970.6 kg/m^3 at design, in the spirit of the measured equilibrium densities [12]. The inference and its laboratory correction are given in Equation (3.8), the bias update of Equation (1.11) with $\lambda = \lambda_b$.

$$\begin{aligned} \widehat{N/C} &= c_0 + c_\rho(\rho_m - 970) + c_T(T_m - 185) + b \\ b &\leftarrow b + \lambda\left[N/C_{lab} - \left(\widehat{N/C}_{raw}(t_s) + b\right)\right] \\ \hat{X} &= 0.6041 + 0.1544(\widehat{N/C} - 3) + 0.00431(T_m - 185) \\ &\quad - 0.25(\mathrm{H_2O/C}_{lab} - 0.40) + b_X \end{aligned} \tag{3.8}$$

where $c_0 = 3.018$, $c_\rho = -0.00697$ per kg/m^3, $c_T = -0.01006$ per kelvin, $\lambda = 0.7$, $|b| \le 0.4$, and $\widehat{N/C}_{raw}(t_s)$ is the unbiased inference at the instant t_s the sample was drawn, not at the report. The coefficients are a least-squares fit on 35 lined-out plants of this model at design load and water with N/C from 2.7 to 3.3 and the drum from 3.9 to 5.1 bar a. The fit's worst error is 0.026 over its own plants, and lined-out plants at 70, 85 and 110 % load, with 98 or 99 weight percent ammonia or with steam ratios of 0.95 and 1.30 read within 0.016 (0.0153). At design it reads 3.0137 against a true 3.000, an offset the bias removes, and a gauge 5 kg/m^3 low reads 0.035 high, since the fit gives 14.3 kg/m^3 for each 0.1 of N/C at constant temperature. The fit data reach 215.7 °C, past the correlation's clamp, although no case runs hotter than 192.4 °C.

🕮 **Pause.** Try Exercise 3.3 before reading on.

3.3.6 Parameters, Initialization and Numerics

Table 3.2 lists the parameters. Each case starts from the lined-out plant for its own settings, found by running the model with the fast loops held at their setpoints for 2400 min of pseudo-time, integrating the loop's ammonia toward the N/C target, and then by Newton's method with the conserved modes pinned. Every derivative at the design state is at most 4.2×10^{-16} of its state. The integrator is the engine's fourth-order Runge–Kutta (Section 1.6.1) with a step of 2 s, control executes at 6 s intervals and a log row is written at 10 s intervals. Halving the step changes the N/C at 8 h of the challenge by 6.1×10^{-11} as shipped and 2.8×10^{-10} with the suggested settings. Over the challenge's twelve hours the four element ledgers close to better than 2.9×10^{-11} kmol, and the molar masses are built from the atomic weights so that the overall reaction conserves mass exactly.

3.3.7 Assumptions

Carbamate formation is complete in excess ammonia, so free CO_2 appears only below $N/C = 2$, and carbonate, bicarbonate and the non-ideal electrolyte solution are left out. The reactor is three stirred cells with fixed gas shares, while a real reactor has trays, back-mixing and a vapor phase throughout. The stripper is a decomposition law whose steam enters no energy balance, and the condenser is one duty at a fixed process-side temperature. The LP section and the solution tank are first-order holdups. Every correlation (conversion, bubble pressure, density volumes, stripping, condensing, biuret, moisture, corrosion) is a teaching value of the published shape, and the inference is fitted to this model, so its error comes only from what the fit did not vary and from the instruments. The heat of carbamate formation, 100 kJ/mol, is below the 117 kJ/mol quoted at 160 °C and 110 atm, while

Table 3.2 Parameters. A: Adjustable in the Model Dialog. H: Hidden and Fixed

Symbol	Meaning	Value	
M_R	reactor holdup, three cells	135 t	A
k_{185}, E_d	dehydration rate, activation	0.12 min^{-1}, 90 kJ/mol	A
ΔH_d, ΔH_c	dehydration, carbamate heats	15.5, 100 kJ/mol	A
c_{pL}, τ_{ov}	liquid heat capacity, overflow	2.7 kJ/(kg K), 0.5 min	H
UA_c, T_p	condenser conductance, process side	84.8 MJ/(min K), 168 °C	A
C_s, K_s	drum capacity, LP valve	900 kg/bar, 1200	H
p_h	LP header	3.5 bar a	A
η	stripper efficiency factor	1.00	A
k_g, k_q	stripping exponents	0.5, 1.109	H
—	HP steam supply limit	80 t/h	A
τ_{LP}, τ_{sol}	LP recycle, solution tank	15, 30 min	A
—	water in LP carbamate	45 wt%	A
ε_N	ammonia slip to evaporator	0.02	H
y_I, V_g, K_v	inerts in CO_2, gas space, vent	0.005, 3 m^3, 1.4	H
$p_{b,0}$	bubble pressure at design	124 bar	A
—	ln p_b slopes T, N/C, H_2O/C	0.018, 0.40, −0.30	H
M_e	evaporator melt holdup	8 t	A
B_{in}	biuret upstream at design	0.30 wt%	A
$k_{b,0}$, E_b	biuret rate at 138 °C, activation	0.0585 wt%/min, 100 kJ/mol	A
—	biuret, moisture limits	0.90, 0.50 wt%	A
—	floor, hold, N/C HIGH	2.8, 20 min, 3.3	A

the dehydration's 15.5 kJ/mol is the quoted value [10]. The condenser was sized on the lower value, so changing it would move every run.

3.4 What the Loop Does Before It Is Controlled

3.4.1 The Ratio Integrates

The loop's ammonia is an inventory. A fresh ratio 0.02 too high raises the reactor N/C by 0.0180 per hour at design load, an integrating gain of 0.014 99 per minute per unit of fresh ratio. In case 1 a 2.85 percent step in the ammonia at constant CO_2 takes the N/C from 3.000 to 3.030 in the first 50 min and 3.083 by 110 min, still climbing, as Section 1.4.3 expects of a recycle with no exit for the excess. The density falls with it, from 970.60 to 965.70 and then 956.12 kg/m^3, and the inference follows within 0.024 of the truth over two hours, while the laboratory's first sample is still four hours away. Wet ammonia is the same error with the opposite sign. Ammonia of 98.0 instead of 99.8 weight percent is a shortfall of 0.0363 in the fresh ratio that no flow meter sees.

🕮 **Pause.** Try Exercise 3.1 before reading on.

A CO_2 turndown does not drain the ratio. With the fresh ratio fixed on the flows, a turndown to 80 % raises the N/C from 3.000 to 3.021 by 8 h in case 5, because only an error in the fresh ratio integrates into the inventory. That measurement decided the challenge's design. The application's plan expected the turndown to carry the shipped controller to the floor, but it does not, so the challenge adds wet ammonia at 1 h. Without it the shipped flows hold the N/C between 2.989 and 3.034 for twelve hours, and with it the N/C crosses the floor.

> **TRY IT IN THE APP** **3 · Ratio on the Flows Drifts**
>
> Load scenario 3 and press Run, at a faster playback speed if the clock is too slow. From 1 h the ammonia arrives wetter while both flow meters read as before. Watch the inferred N/C on the Ratio view fall below 2.93 at 3 h 42 min, the laboratory's first report arrive at 4 h 45 min reading 2.918, and the run stop at 8 h 26 min.

3.4.2 The Condenser Sets the Reactor's Heat

The reactor has no heater. Its heat is the gas the condenser does not condense, so at a fixed drum pressure a smaller gas flow loses a larger share to the condenser. In case 5 the turndown raises the

condensed share from 82.6 to 86.1 percent, the reactor top falls from 185.0 to 176.6 °C at 1 h 54 min and the conversion from 61.1 to 58.1 percent at 8 h. The HP steam per tonne of urea stays at 0.84 t/t because the stripper steam follows the CO_2, while the LP steam raised per tonne rises from 0.721 to 0.825 t/t, heat sent to the drum instead of the reactor. The plan's expectation that energy per tonne would climb did not happen in the model, and the case teaches the coupling instead.

Pause. Try Exercise 3.2 before reading on.

3.4.3 Bubble Pressure and a Recycle That Stores a Disturbance

The high side of the ratio costs pressure. Left running after case 1's step, the inert vent is wide open 1 h 58 min after the step and PRESSURE HIGH (measured pressure above 143 bar a) shows 2 h 9 min after it, at $N/C = 3.100$ with the top at 187.28 °C. That alarm is half ammonia and half temperature in this model. The bubble pressure rises from 127.59 to 137.98 bar, of which the N/C alone accounts for 5.2 bar and the 2.3 K warmer reactor alone for 5.3 bar. With the bubble-pressure slopes refitted near design to Voskov and Voronin (0.026, 0.07 and −0.10), the alarm would come 3 h 12 min after the step, the suggested challenge's peak pressure would rise from 142.93 to 144.35 bar a, still under its limit, and no verdict would change, so the shipped slopes were kept with the comparison stated.

A stripper steam dip shows the recycle storing a disturbance. In case 6 the supply falls to 40 % of 80 t/h from 30 to 90 min, so the stripper gets 32.0 t/h and strips 64.5 instead of 80.0 percent of the CO_2. The recycle peaks at 46.9 t/h at 1 h 34 min, the conversion bottoms at 54.68 % at 1 h 36 min, and the reactor's H_2O/C rises from 0.406 to 0.619 at 2 h 17 min, all after the steam's return. On the return the stripper decomposes the backlog at once. The gas to the condenser jumps from 73.3 to 85.2 kmol/min, the condenser at its fixed drum pressure condenses 77.1 instead of 83.7 percent, and the heat the gas brings the reactor rises from 376 to 640 MJ/min. The top reaches 192.37 °C at 2 h 26 min and the measured pressure 143.59 bar a at 2 h 18 min, after the vent saturates. The overshoot is physical. With the ratio on the flows it is 191.76 °C, a pressure loop four times faster leaves it at 192.37, a drum loop five times slower raises it to 194.10 °C, and the drum valve on manual to 194.90 °C.

3.5 Control as Implemented

Every controller reads one measurement packet built by the instrument code, holding the meters, the gauges, the laboratory's reports and their quality, and an automated check rejects any packet holding another key. The header's Manual and Auto switch belongs to the N/C loop alone (Manual hands the operator the ammonia feed), while the pressure, level, drum and evaporator loops each have their own switch, so that an integrating level and a fast pressure are not left unattended in case 1. Every controller not acting tracks the one that is, so mode, architecture and cascade changes are bumpless.

3.5.1 The Shared PI Law

All six loops use the framework's velocity-form PID of Section 1.6.2, Equations (1.14) to (1.17), with the derivative off. Each executes every $\Delta t = 6$ s. The loops marked reverse in Table 3.3 have a measurement that falls as their output rises and run with $a = -1$. The running sum is never clamped, and back-calculation pulls the integral toward the limited output with τ_I as the tracking time. An integral time at its slider's maximum removes integral action. The defaults are listed in Table 3.3.

3.5.2 Three Ways to Hold the Ratio

The ammonia command is always a ratio R to the measured CO_2, $F_{NH_3,SP} = R F_{CO_2,m} M_A / (M_C(1 - 0.002))$, where the ratio station assumes the design purity. With *Flows*, R is the operator's number, 2.0173 at design (2.000 for the urea, 0.0072 for the ammonia slip and 0.0101 for the vent), and nothing looks at the reactor. With *Inference*, the AIC of Table 3.3 moves R on $\widehat{N/C}$. With *MPC*, a receding-horizon plan sets R and the stripper steam ratio S every 10 min over 4 h on the model of

Table 3.3 Loops, Pairings and Default Tunings

Loop	PV → MV	K_c	τ_I	Output range
AIC	$\widehat{N/C} \to R$	1.5 per unit	120 min	1.80–2.30
PIC	$p \to$ vent, reverse	2 %/bar	3 min	0–100 %
LIC	$L \to$ bottoms, reverse	1.5 %/%	30 min	0–100 %
PIC	$p_s \to$ LP valve, reverse	20 %/bar	3 min	0–100 %
TIC	$T \to p_{s,SP}$ (cascade)	0.05 bar/K	20 min	3.6–6.5 bar a
TIC	$T_e \to$ heating steam	1.0 %/K	5 min	0–100 %

Equation (3.9).

$$
\begin{aligned}
\frac{\mathrm{d}a}{\mathrm{d}t} &= g\,(R - R_{bal}) \\
\frac{\mathrm{d}X}{\mathrm{d}t} &= \left[X_0 + k_N(a - a_0) + k_S(S - S_0) - X\right]/\tau_X \\
\frac{\mathrm{d}B}{\mathrm{d}t} &= \left[B_0 + k_B(S - S_0) - B\right]/\tau_B \\
\min\ & \textstyle\sum_k \left[w_N(a_k - \mathrm{SP})^2 - 100 w_X X_k + w_S(S_k - 1.15)^2\right] \\
& + w_{\Delta u} \textstyle\sum_j \left[(\Delta R_j/0.01)^2 + (\Delta S_j/0.05)^2\right] \\
& + 10^4 \textstyle\sum \sigma^2 \\
\text{s.t. } & 2.90 - \sigma \le a_k \le 3.20 + \sigma \\
& B_k \le B_{lim} + \sigma, \qquad \sigma \ge 0
\end{aligned}
\tag{3.9}
$$

where a_0 is the inferred N/C, X_0 the conversion soft sensor of Equation (3.8), B_0 the last laboratory biuret, S_0 the present steam ratio, $g = 0.0149 F_{CO_2,m}/45.8$ per minute, and R_{bal}, the ratio that would hold the N/C still, is estimated from the inferred N/C's motion and filtered over an hour. The step-identified constants are $k_N = 0.154$, $k_S = 0.10$ per t/t, $k_B = 0.27$ wt% per t/t, $\tau_X = 60$ min and $\tau_B = 30$ min. A lined-out step of 0.1 t/t in steam gives 1.00 point of conversion and 0.0273 wt% of biuret, which matches k_S and k_B. The model is converted to discrete time exactly under a zero-order hold, the horizon is 24 steps in six move blocks, two of one step and then 2, 4, 8 and 8 steps, R is bounded by 1.80–2.30 and moves at most 0.04 per step, S lies between 0.8 and the lesser of 1.5 and the supply's limit and moves at most 0.05, and the defaults are $w_N = 200$, $w_X = 1$, $w_S = 50$ and $w_{\Delta u} = 1$. The quadratic program goes to the framework's shared solver of Section 1.6.3, a dual active-set algorithm due to Goldfarb and Idnani [19], and the first move is applied, as in industrial practice [20]. A plan that is not optimal, not finite or out of range is rejected, and the tracked inference PI takes the ratio without a bump. The conversion reward pulls the N/C above its setpoint, and the MPC buys conversion with steam until its biuret constraint binds. Before the first laboratory report the MPC's biuret starts from the shipped default of B_{in} plus 0.45 rather than from the engine's parameter, which moves no shipped run.

3.5.3 Cascade and the Three Models

The optional reactor temperature cascade lets a TIC on the measured top temperature set the drum's setpoint between 3.6 and 6.5 bar a, the handle of Section 3.4. The stripper steam follows the measured CO_2 in the ratio S unless the MPC sets it. The plant is the model of Section 3.3. The control models are the inference fitted to that plant, the conversion soft sensor and the MPC's three-state linear model, none of which knows the ammonia's water, a gauge's error or the corrosion index. The exported file carries every measurement beside its truth, and the columns holding the simulator's truth carry a prefix (`truth_`) that keeps them out of any replayed controller. The corrosion stop reads the truth and is a teaching guardrail outside every controller.

Table 3.4 The Nine Cases

#	Case and events	Teaching point
1	Manual, learner steps the ammonia to 36.85 t/h	the ratio integrates
2	Inference, gauge -5 kg/m^3, lab every 2 h	the laboratory bias
3	Flows, ammonia 98.0 wt % from 1 h	drift on the flows
4	Flows from N/C 2.90, learner cuts R to 1.95	the corrosion floor
5	Flows, CO_2 to 80 % at 30 min	the condenser's heat
6	Inference, HP steam 40 % from 30 to 90 min	a recycle stores it
7	Inference, learner moves T_e	biuret against moisture
8	Inference, CO_2 to 55 % at 30 min	a window that closes
9	Challenge, 12 h, four upsets, Flows as shipped	the whole loop

Table 3.5 Case Outcomes Measured on the Engine

Case and condition	Result
1, step at 5 min	N/C 3.030 / 3.083 at 50 / 110 min after; ρ 956.12 kg/m^3; PRESSURE HIGH 2 h 9 min after
2, bias update on	$\widehat{N/C}$ 3.045 at $t = 0$, truth 2.962 lowest; bias -0.035 at 2 h 45 min; truth 2.997 at 4 h 45 min
2, bias update off	truth 2.952 at 8 h, $\widehat{N/C}$ 3.001
3, Flows	$\widehat{N/C} < 2.93$ at 3 h 42 min; lab 2.918 at 4 h 45 min; floor 7 h 49 min; stop 8 h 26 min
3, Inference at 3 h 42 min	R 2.0955 an hour later; truth $\geq$ 2.913; within 0.03 at 5 h 45 min
3, Inference from $t = 0$	truth 2.969–3.020 for 12 h
4, R 1.95 at 10 min	N/C 2.808 at 2 h; $I_c = 1$ at 2 h 21 min; stop 2 h 41 min
5, operator drum	top 176.56 °C; X 58.12 %; biuret 0.858 wt %
5, cascade	drum SP 5.17 bar a; top $\geq$ 181.02 °C; X 63.23 %; biuret 0.919 wt %
6, steam dip	recycle 46.9 t/h; $X \geq$ 54.68 %; top 192.37 °C; p 143.59 bar a
7, T_e 142.5 / 135 / 138.5	biuret 0.918 / 0.660 / 0.763; moisture 0.097 / 0.638 / 0.267 wt %
8, 55 % load	$X \geq$ 51.26 %; residence 13.38 min; biuret 1.163 wt % at 138 °C
8, best T_e	136.0 °C: biuret 0.968, moisture 0.497 wt %

3.6 Nine Cases on the Running Loop

The cases are listed in Table 3.4 and their main measurements in Table 3.5. Every number comes from the application's own engine, run headless with its 2 s step and its default seed and started from each case exactly as it loads, with any setting the text names entered at the time it names (Section 1.6.4 describes the cases, their completion rules and the export). Two independent measuring scripts reproduce every value with identical output. Times are from $t = 0$ unless a step is named.

3.6.1 An Inferred Ratio Against a Late, Accurate One

Case 2 runs the inference with a density gauge that reads 5 kg/m^3 low, so the inference starts at 3.045 against a true 3.000 and the AIC cuts the ammonia until the truth reaches 2.962. The first report, sampled at 2 h, reads 2.970 at 2 h 45 min and moves the bias to -0.0347, and the truth is 2.997 at the second report. With the update off the error stays and the truth ends at 2.952 at 8 h. The update also inherits the laboratory's noise. A third report 0.018 below the truth moves the bias to -0.0575 at 6 h 45 min. In case 3 the flows let the inferred N/C fall below 2.93 at 3 h 42 min and the truth below 2.90 at 4 h 32 min, before the first report. The truth crosses the floor at 7 h 49 min, but the index reaches 1 only at 8 h 6 min ($N/C = 2.791$) because the wet reactor runs at 182.5 °C, and the run stops at 8 h 26 min. Switched to the inference at 3 h 42 min, the AIC raises R to 2.0955 within the hour and holds the lowest truth at 2.913, and run on the inference from the start the truth stays between 2.969 and 3.020. The state of case 3 at 500 min is shown in Figure 3.5.

3.6.2 Heat, Biuret and a Window That Closes

With the cascade in case 5 the TIC raises the drum to 5.17 bar a, the reactor dips only to 181.02 °C and is back within 1 K of 185 °C at 1 h 54 min, and the conversion settles at 63.23 %. The biuret then rises to 0.919 wt%, over its limit, against 0.858 wt% with the operator's drum. The condenser does

Table 3.6 The Challenge Over 12 h at the Default Seed

Configuration	Samples	Low	X (%)	B (wt%)	p (bar a)	Verdict
Flows, operator, 138 (shipped)	2.949, 2.857	2.774	56.53	0.872	140.75	stop
Inference, cascade, 137 (sugg.)	2.965, 3.021, 3.033	2.964	61.32	0.882	142.93	pass
MPC, cascade, 137	2.976, 3.049, 3.070	2.973	61.98	0.896	142.59	pass
Inference, operator, 138	2.976, 3.012, 3.021	2.967	58.40	0.870	140.98	fail
Inference, cascade, 138	2.965, 3.021, 3.033	2.964	61.32	0.922	142.93	fail
Inference, drum 4.8, 137	2.968, 3.009, 3.022	2.967	60.71	0.858	148.94	fail
Flows, cascade, 137	2.937, 2.839	2.775	60.46	0.880	141.20	stop

not reach the evaporator. The turndown alone lengthens the melt's stay from 7.66 to 9.57 min and raises B_{in} through the square-root law, which moves the biuret from 0.748 through 0.783 to 0.895 in the absence of any ammonia effect. With the operator's drum the cooler reactor converts less and 0.1218 kmol/min of unconverted ammonia reaches the evaporator and holds the biuret back to 0.858. With the cascade the hotter reactor converts more and only 0.0881 kmol/min arrives, so the suppression weakens and the biuret reaches 0.919. With the suppression switched off both settings give 0.895, so the cascade's whole difference is that ammonia term.

> **TRY IT IN THE APP** **5 · A CO_2 Turndown and the Reactor's Heat**
>
> Load scenario 5 and press Run. The carbon dioxide drops to 80 % at 30 min, and the reactor top falls from 185.0 to 176.6 °C. Then set Steam drum setpoint from to Reactor T on the TIC card, press Reset and run again: the drum setpoint climbs to 5.17 bar a, the reactor dips only to 181.02 °C, and the biuret rises to 0.919 instead of 0.858 wt%.

Case 7 finds the evaporator's window at design between the 0.50 wt% moisture limit and the 0.90 wt% biuret limit, 136.00 to 142.10 °C. At 142.5 °C the biuret crosses its limit 19 minutes after the change. Case 8 cuts the CO_2 to 55 %. The urea falls from 62.50 to 35.76 t/h, the reactor top to 163.88 °C and the conversion to 51.26 %, and the fixed melt holdup keeps the melt 13.38 min. On the lined-out plants the window narrows to 136.00–136.60 °C at 65 % load and is gone at 60 percent (smallest excess 0.008 wt%) and 55 percent (0.039 wt%). The case is the honest infeasible one. No evaporator setpoint meets both limits, 136.0 °C giving 0.968 and 0.497 after five hours, while a 5 t melt holdup reopens the window (0.772 and 0.438 at 136.5 °C).

> **DID YOU KNOW?** **Making Less Urea Makes Worse Urea**
>
> From the simulator: with the carbon dioxide cut to 55 %, the melt stays 13.38 min in the evaporator and its biuret reaches 1.163 wt% at 138 °C. The best evaporator temperature, 136.0 °C, gives 0.968 biuret with 0.497 wt% moisture, so no setting meets both limits (Table 3.5).

3.6.3 A Twelve-Hour Shift

The challenge brings 98.0 weight percent ammonia at 1 h, the CO_2 to 80 % at 2 h, the stripper steam to 40 % from 6 to 7 h and the CO_2 back at 9 h. It is met when the true N/C lies within 2.90–3.20 at the samples of 4, 8 and 12 h and never falls below 2.80, the average true conversion is at least 59 %, the biuret never exceeds 0.90 wt% and the reactor pressure never exceeds 145 bar a, judged every step on the truth. The 59 percent and 145 bar a are teaching values chosen by measurement. At the plan's 58 percent the inference alone passed by 0.4 points, so the condenser handle did not matter, and without a pressure limit a drum held at 4.8 bar a all shift passes on conversion (60.71 %) while it reaches 148.94 bar a when the load returns. The configurations are compared in Table 3.6 and the trajectories in Figure 3.4. Figure 3.3 sets the table's last four columns against the limits of the challenge.

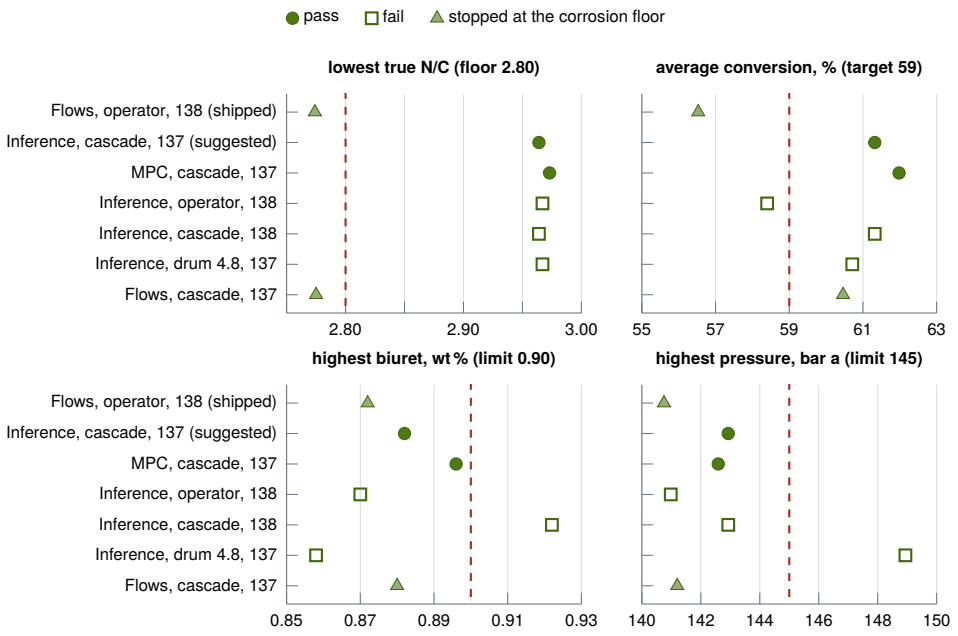


Figure 3.3 Table 3.6 as dots, digit for digit: the lowest true N/C, the average conversion, the highest biuret and the highest pressure of each configuration over the shift, with the floor, target or limit of the challenge dashed. Passing runs are filled circles, failing runs open squares and runs stopped at the corrosion floor filled triangles. Notice that each failing configuration misses on exactly one axis, the conversion, the biuret or the pressure, and that the two runs on the flows are the only ones below the floor.

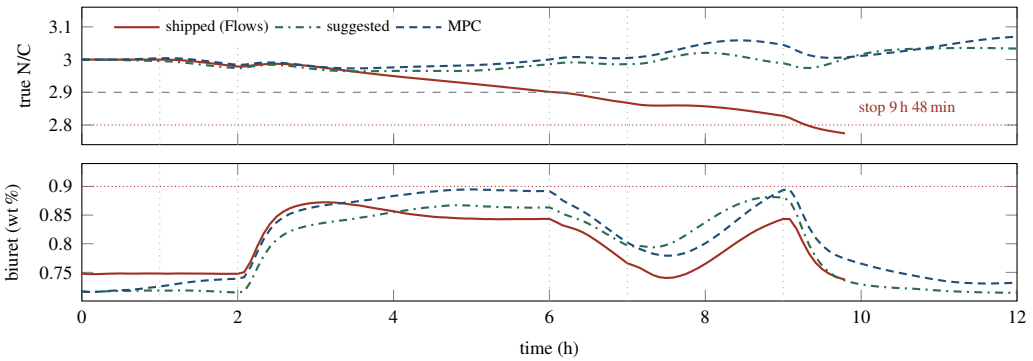


Figure 3.4 The challenge's true reactor N/C and product biuret, shipped (Flows, operator's drum, 138 °C, solid), suggested (Inference, cascade, 137 °C, dash-dotted) and MPC (cascade, 137 °C, densely dashed). Dotted verticals mark the events at 1, 2, 6, 7 and 9 h, dashed lines the 2.90–3.20 band, dotted horizontals the 2.80 floor and the 0.90 wt% limit. Rows every 5 min of the log.

As shipped, the wet ammonia drains the inventory for nine hours, the samples read 2.949 and 2.857, and the run stops at 9 h 48 min. The inference holds the N/C, but with the operator's drum the reactor cools to 174.42 °C through the turndown and the conversion averages only 58.40 %. The cascade raises the drum only while the load is down, which keeps the conversion at 61.32 % and the pressure under 145 bar a when the load returns, and the turndown's longer residence then decides the



Figure 3.5 Case 3 at $t = 500$ min on the Ratio view with the teaching overlay on Show truth and an eight-hour trend window, in Auto with the ratio on the flows, cropped to the Ratio view and two trend strips. Wet ammonia since 1 h has drained the loop. The badges (1) read the inferred N/C 2.777, the last laboratory report 2.918 (sampled at 4 h), the corrosion index 1.14, an estimated conversion of 55.5 %, the reactor top at 182.3 °C and 0.73 wt% biuret, and the status strip (4) N/C LOW, CORROSION, 14 OF 20 MIN. On the conversion plot (2) the inferred point and the truth's × (2.784) sit inside the shaded floor band, and the inference plot (3) places the gauge reading of 1007.5 kg/m^3 beyond the 2.8 line with a bias of −0.007. The trends show the inferred and true N/C falling together through 2.80, the laboratory's single report (5) flat and late, and the fresh ratio unmoved at 2.0173.

evaporator setpoint, 0.922 wt% at 138 °C and 0.882 wt% at 137 °C. Each handle answers one upset. The suggested settings pass at seeds 0 to 19, the default seed and half the step, 22 of 22 runs, with the highest biuret between 0.8807 and 0.8857 wt%, a margin of at least 0.014 wt%. The MPC also passes all 22, but only just. Its highest biuret lies between 0.8946 and 0.8999 wt%, a margin of 0.0001 to 0.0054 wt%, because the biuret is a soft constraint on its own linear model fed by a laboratory reading, and the conversion reward spends stripper steam until that constraint binds. Its samples run higher, 3.049 and 3.070 at the default seed, as the reward pulls the N/C up, and it solves 71 plans with none rejected at seeds 0 to 3, at the default seed and with the step halved. The MPC was left as built, with its margin stated, because holding a soft constraint at its limit is what a reward on the other side of it produces.

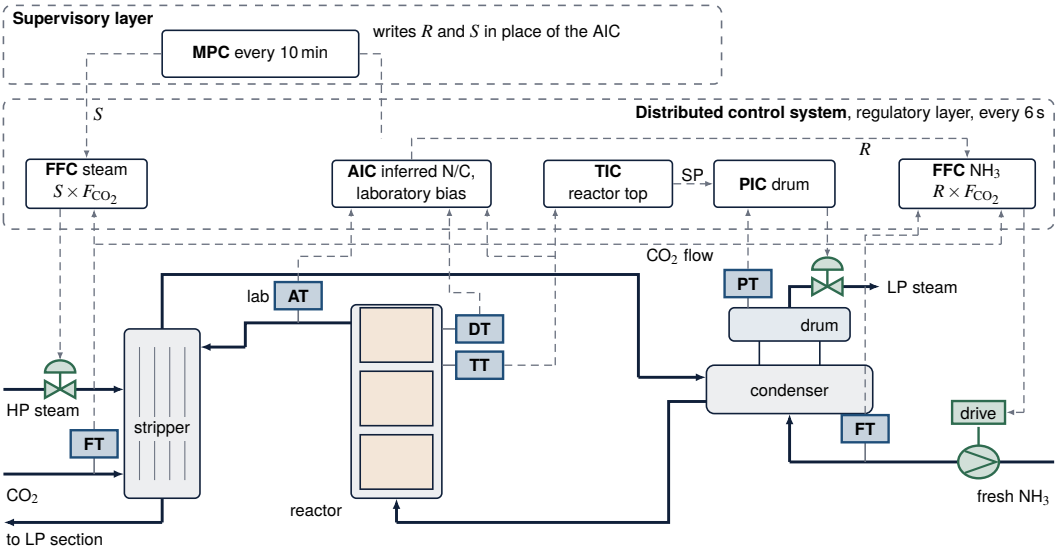


Figure 3.6 The ratio loop and the reactor's heat loop with their hardware. Lettered boxes are sensing devices: flow transmitters on the carbon dioxide and the fresh ammonia (FT), the density and temperature at the reactor top (DT, TT), the drum pressure (PT) and the laboratory sample (AT). Valve symbols and the pump with its drive are final elements, and dashed arrows are signals. Notice that nothing measures the ratio itself: the AIC reads a density, a temperature and a late laboratory result, and the ratio station multiplies its R by the measured carbon dioxide. The reactor's temperature is moved through the drum pressure, a cascade, and the predictive controller writes R and S in place of the AIC. The vent, level and evaporator loops, the LP section and the carbon dioxide compressor are left out. Not to scale, and not a piping diagram.

3.7 Technology in Practice

What Measures It

Figure 3.6 draws the loops of Section 3.5 with their devices. The feed flows, the pressures, the level and the temperatures are read continuously. The ratio that matters has no transmitter. The inference reads a density and a temperature at the reactor top, and the laboratory corrects it every 4 h, 45 min after each sample. Offline analysis is accurate but late, and drawing a sample of hot liquid at synthesis pressure is itself a hazard, which is why plants prefer in-line methods [21]. The model's density lag of 0.5 min and noise of 0.6 kg/m^3 stand for the gauge below.

> **TECHNOLOGY** **Sensor: A Density Gauge on the Reactor Outlet**
>
> No instrument reads the reactor's ratio directly, so plants infer it. A 2026 study lists vibrating-element density sensors, Raman spectroscopy, gas chromatography and laboratory samples, and notes that sensors in contact with the carbamate corrode and foul, so their calibration drifts. A gamma-ray gauge avoids contact: a source and a detector sit on opposite sides of the outlet pipe, and the density of the liquid sets how much radiation gets through. The same study recalibrates it with process water at shutdowns, to remove drift in the pipe, the detector and the decaying source [21]. On a trend, a sensor drift is a slow offset that looks like a change in the process [2]. The gauge 5 kg/m^3 low of case 2 is one.

What Moves It

Two ratios and one pressure move this loop. The ratio station sets the fresh ammonia in proportion to the measured carbon dioxide, and the stripper steam follows the carbon dioxide in its own ratio. In a

stripping plant of this kind the fresh carbon dioxide enters through the high-pressure stripper [17], as it does in Figure 3.1. The reactor has no heater, so its temperature is moved by the pressure of the steam drum on the carbamate condenser, through the low-pressure steam valve. The same handle appears in the reference document's pool reactor, a design in which the pressure of the steam raised controls the rate of condensation [17].

> **TECHNOLOGY Final Element: A Pump at Synthesis Pressure**
>
> The fresh ammonia reaches synthesis pressure through high-pressure pumps [17]. A pumped flow can be moved in two ways. A control valve throttles it, or a variable-frequency drive changes the speed of the motor by changing the frequency of its power, which saves energy and removes a component. A variable-speed machine, however, often cannot change the flow as fast as a valve, cannot stop the flow positively and offers fewer fail-safe states [22]. Either way, the ratio station of Section 3.5 only hands the ammonia flow its setpoint, and the seven actuator positions among the model's 39 states stand in for such hardware.

Where the Controller Runs

A plant runs such loops in a distributed control system, in which a supervisory level oversees local controllers and sends them set points, loops are sometimes nested or cascaded, and cycle times range from milliseconds to minutes [23]. The app executes its six PI loops every 6 s and its predictive controller every 10 min, so both sit inside that range. The ratio stations, the AIC with its laboratory bias and the cascade from the reactor top onto the drum belong to the regulatory layer. The plan belongs to the supervisory layer above it and writes the ammonia ratio and the steam ratio. When a plan is rejected, the tracked inference PI takes the ratio back without a bump.

> **IN PRACTICE The Operating Window and the Biuret Limit**
>
> A 2007 European reference document gives the window urea reactors work in: 140 to 250 bar, 180 to 210 °C and an ammonia to carbon dioxide ratio of 2.8 to 4, for a conversion of 50 to 80 percent of the carbon dioxide. Stripping with carbon dioxide, as this chapter's plant does, is now the most frequent type. Fertilizer urea should preferably carry at most 1.2 percent biuret, which can damage crops sprayed on their leaves, and urea solutions low in ammonia should not stay long at high temperature [17]. The chapter's biuret limit of 0.90 wt% and the evaporator window of case 7 are teaching values of the same trade.

What Is Modernizing It

Three developments bear on this loop. Revamps of older plants have added continuous monitoring of the ratio, along with relined reactors and stripping equipment [17], and gamma-ray gauges, which never touch the carbamate, have gained attention for reading it [21]. Combining condensation and reaction in one vessel, a pool reactor, makes the synthesis much less sensitive to deviations of the ratio and removes the loop's inverse response [17], which would change the dynamics that Section 3.4 measures. And data-driven soft sensors, of which the chapter's least-squares inference is a simple case, are spreading through the process industries, with adaptive versions that update their models as the process drifts [2]. The open challenge after the Limitations takes up what they still lack.

3.8 Limitations

The sizes, rates and limits were chosen to teach, for a generic plant. The conversion correlation has the published shape and lies within about 3 points of two published sources but 4 to 9 points above a third near $N/C = 3$, the bubble pressure overstates the N/C slope sixfold and understates the temperature's, the heat of carbamate formation is 17 kJ/mol below the quoted value, and the stripping, condensing, biuret, moisture and corrosion laws are closures chosen for their shape. The square-root biuret law and the 30 min solution tank were added so that the evaporator's lessons work, and the condensed fraction's exponential form was chosen to remove an oscillation that the clipped form produced. The urea-per-kilogram maximum moves with the correlation, 2.98 at the design's water

and temperature but 2.56 at the conditions of the published curve that peaks near 3. The cases also idealize their upsets. A clean step in ammonia purity or a gauge error that appears at once would not happen on a plant, but each isolates one path into the inventory.

The inference is fitted to the same model it estimates, so its 0.026 worst error is a floor that a real gauge on a real solution would not reach. The reactor is three stirred cells with no vapor phase, the stripper has no energy balance, and the corrosion stop is a guardrail that reads the truth, not a model of a corroding liner. The MPC's challenge pass rests on a biuret margin of thousandths of a weight percent, and its model is identified once on this plant and never updated.

OPEN CHALLENGE **An Inference That Stays Right**

A 2009 review names maintenance as a main open issue of soft sensors in the process industry. After launch, the predictions gradually deteriorate as the process changes, until the model is retrained or rebuilt. Drift comes from the process, such as the purity of the input materials, or from the measuring devices, and telling the two apart is hard and still needs expert judgment. Most soft sensors had no automated maintenance and often no objective measure of their quality [2]. Here one bias, moved by a laboratory report every four hours, does all of that work. Progress would be an inference that separates a drifting gauge from a changing process on its own and states how far it can be trusted.

3.9 Conclusion

A CO_2-stripping urea loop holds its most important variable, the N/C in the reactor, as an inventory that no instrument reports, and this simulator logs the true ratio, the inference from density and temperature and the laboratory's reports side by side from element balances that close to 3×10^{-11} kmol. Two weight percent of water in the ammonia carries a ratio held on the flows from 3.000 to the 2.80 floor in 6 h 49 min, while a CO_2 turndown raises it slightly. A PI on the inferred ratio with a laboratory bias holds the truth between 2.969 and 3.020, and the bias removes a 5 kg/m^3 gauge error within two reports. The condenser, not a heater, sets the reactor temperature, 11.6 K per bar of drum pressure near design, so a turndown cools the reactor from 185.0 to 176.6 °C and a cascade onto the drum restores it, at the cost of biuret through the ammonia that no longer reaches the evaporator. The suggested settings pass the twelve-hour shift at 22 of 22 runs and the MPC passes only by thousandths. A fuller model would fit the teaching correlations to the published equilibria and give the reactor a vapor phase. The simulator shows that a ratio held between two flow meters is a ratio of the meters, not of the process.

KEY POINTS

- No instrument reports the reactor's ratio of ammonia to carbon dioxide, and a ratio held between two flow meters is a ratio of the meters.
- Two weight percent of water in the ammonia carries a ratio held on the flows to the 2.80 corrosion floor.
- A PI on the ratio inferred from density and temperature, with a laboratory bias, holds the truth between 2.969 and 3.020.
- The condenser, not a heater, sets the reactor temperature, and a cascade onto the steam drum restores it at the cost of biuret.
- The suggested settings pass the twelve-hour shift in 22 of 22 runs, the predictive controller only by thousandths.

Where else this applies. Volume 5, Chapter 10 corrects a soft sensor from a delayed laboratory in the same way, and Chapter 2 holds a feed ratio in another loop of unconverted reactants.

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3.10 Exercises

3.1 Concept check · 2 min

In case 3 the ratio is held on the flows: the fresh ammonia follows the measured carbon dioxide in the fixed ratio 2.0173, and the ratio station assumes ammonia of 99.8 wt%. From 1 h the ammonia arrives at 98.0 wt%, and both flow meters keep reading their usual values. What does the true N/C at the reactor top do?

(a) It falls by about two percent within the hour the liquid stays in the reactor, then holds

(b) It falls steadily for hours, because the loop's ammonia integrates the shortfall

(c) It stays near 3.000, because the ratio station already corrects for the water it meters

(d) It rises, because the water dilutes the carbon dioxide more than the ammonia

3.2 Concept check · 2 min

In case 5 the carbon dioxide compressor is turned down to 80 % with the ratio on the flows and the steam drum held at 4.50 bar a by the operator's setpoint. The reactor top falls from 185.0 to 176.6 °C. Why does the reactor cool?

(a) The stripper steam is cut with the carbon dioxide, so less HP steam per tonne of urea reaches the loop

(b) The N/C falls with the turndown, and a leaner liquid converts less and releases less heat

(c) The condenser at a fixed drum pressure condenses a larger share of a smaller gas flow, leaving less gas to heat the reactor

(d) The liquid stays longer in the reactor, so the endothermic dehydration takes more of its heat

3.3 Concept check · 1 min

The laboratory samples the reactor at 2 h and reports at 2 h 45 min. Against which value of the inference does the bias update compare the laboratory result?

(a) The inference at the report, since that is when the result is known

(b) The average of the inference over the 45 min the analysis takes

(c) The inference at the next control execution after the report

(d) The inference at the instant the sample was drawn

3.4 Theory and calculation · 6 min

At design load a fresh ratio 0.02 too high raises the reactor N/C by 0.0180 per hour. In case 3 the ratio station holds the fresh ratio at 2.0173 and assumes ammonia of 99.8 wt%, and from 1 h the ammonia is 98.0 wt%. Give each answer to two significant figures.

(a) Find the integrating gain of the loop in N/C per minute per unit of fresh ratio. *Checkpoint:* 0.015 per minute.

(b) Find the shortfall in the fresh ratio, in moles of ammonia per mole of carbon dioxide, that the wet ammonia causes. *Checkpoint:* About 0.036.

(c) Estimate how long after 1 h the N/C takes to fall from 3.000 to the 2.80 floor. The run measures 6 h 49 min. Give one reason from the chapter's measurements why the estimate is earlier. *Checkpoint:* About 6.1 h. The holdups delay the start of the ramp.

3.5 Theory and calculation · 6 min

The corrosion index of Equation (3.7) stops the run once it has stayed at 1 or above for 20 min. Give N/C values to three decimals.

(a) Evaluate the index at N/C 2.90 with the design passivation air of 0.6 vol% and a reactor top at 185 °C. *Checkpoint:* 0.135.

(b) In case 3 the wet reactor runs at 182.5 °C. At what N/C does the index reach 1 with design air? Compare with the chapter. *Checkpoint:* 2.792, against 2.791 measured at 8 h 6 min.

(c) The passivation air is halved to 0.3 vol% with the top at 185 °C. Find the index at N/C 2.80 and the N/C at which it reaches 1. *Checkpoint:* 1.41, and 2.817.

3.6 Theory and calculation · 7 min

The inference of Equation (3.8) has $c_0 = 3.018$, $c_\rho = -0.00697$ per kg/m^3 and $c_T = -0.01006$ per kelvin, with $\lambda = 0.7$.

(a) At the design point the top reads 970.6 kg/m^3 and 185.02 °C. Find the unbiased inference to three decimals, and its offset from the true 3.000. *Checkpoint:* 3.014, an offset of 0.014.

(b) In case 2 the density gauge reads 5 kg/m^3 low. Predict the inference at $t = 0$, and find the density change the fit expects for a change of 0.1 in N/C at constant temperature. *Checkpoint:* 3.048, and 14.3 kg/m^3.

(c) The first report in case 2 reads 2.970. When that sample was drawn the unbiased inference read 3.0195, and the bias was 0. Find the new bias, and the bias a gain of $\lambda = 1$ would give. *Checkpoint:* -0.0347, and -0.0495.

3.7 App challenge · 9 min

Load case 1, Step the ammonia (Manual), which opens with the header switch on Manual, and change no other setting. Run at 10x with the default seed, pause at about 10 min, set the Ammonia feed on the AIC card from 35.83 to 36.85 t/h and run to 150 min. Export the CSV file, whose time column is in hours, and find the step in column `nh3_cmd_tph`.

(a) Read the true N/C, column `truth_nc`, and the true density, column `truth_rho_kgm3`, 50 min and 110 min after the step. *Checkpoint:* 3.030 and 3.083 within 0.002, 965.7 kg/m^3 and 956.1 kg/m^3 within 0.2 kg/m^3.

(b) The step is 0.0574 in the fresh ratio. From the slope between the two readings, find the integrating gain in N/C per minute per unit of fresh ratio, and compare it with the chapter's 0.01499. *Checkpoint:* 0.0156 within 0.0003, about 4 % above.

(c) Find how long after the step the measured pressure, column `p_meas_bar`, first exceeds 143 bar a, which is when the status strip shows PRESSURE HIGH. *Checkpoint:* 2 h 9 min within 2 min.

3.8 App challenge · 8 min

Load case 2, The inference and the laboratory: the ratio on the inference, the density gauge 5 kg/m^3 low and the laboratory every 2 h. Set the Teaching overlay on the AIC card to Show truth, run to 8 h at 60x with the default seed and export the CSV file, whose time column is in hours.

(a) Read the lowest true N/C, column `truth_nc`, before the first report, and the bias, column `nc_bias`, on the row where `lab_reports` first reads 1. *Checkpoint:* 2.962 within 0.002, and -0.0347 within 0.0005.

(b) Read the true N/C on the row where `lab_reports` first reads 2. *Checkpoint:* 2.997 within 0.002.

(c) Switch to the Advanced level, set Laboratory bias update to Off, press Reset, run to 8 h again and export. Read the true and the inferred N/C at 8 h, and explain their difference. *Checkpoint:* 2.952 and 3.001 within 0.002.

3.9 App challenge · 9 min

Load case 9, the twelve-hour challenge, and keep the default seed. Run each configuration for 12 h at 60x, and read the verdict from the So far tile, whose hover text lists the samples, the lowest N/C, the average conversion, the highest biuret and the highest pressure, and from the note at 12 h.

(a) Run as shipped (ratio on Flows, steam drum setpoint from the Operator, evaporator at 138 °C). When does the run stop, and what do the samples at 4 h and 8 h read? *Checkpoint:* A corrosion stop at 9 h 48 min, samples 2.95 and 2.86.

(b) Set Ratio control to Inference, Steam drum setpoint from to Reactor T and the Evaporator temperature setpoint to 137.0 °C, press Reset and run. Read the three samples, the average conversion, the highest biuret and the highest pressure. *Checkpoint:* 2.97, 3.02 and 3.03; 61.3 %; 0.88 wt%; 142.9 bar a; target met.

(c) Raise the evaporator setpoint to 138.0 °C, press Reset and run again. Which part of the target fails, and why does the evaporator setpoint matter only with the cascade on? *Checkpoint:* The biuret, 0.92 wt%.

3.10 Application area · 4 min

In case 8 the carbon dioxide load is cut to 55 %, the melt holdup of the evaporator stays at 8 t, and no evaporator setpoint meets both the 0.50 wt% moisture limit and the 0.90 wt% biuret limit.

(a) The moisture law is $w = 0.30\,\mathrm{e}^{-(T_e-138)/4}$ in weight percent. Find the lowest evaporator temperature that meets the moisture limit, and say why it is the same at every load. *Checkpoint:* 136.0 °C, because the law has no flow in it.

(b) The melt stays 13.38 min at 55 % load. How long would it stay with a 5 t holdup, and why does that reopen the window? *Checkpoint:* About 8.4 min.

3.11 Application area · 4 min

The ratio runs on the inference with the laboratory every 4 h. A plant manager proposes sampling every 8 h, since the inference already holds the N/C. Using the chapter's measurements, say what the laboratory does for the loop, what the change would risk, and what you would recommend.

3.12 Review the agent · 5 min

An AI assistant reviews Table 3.6 and writes: "Use the MPC for the shift. It passes like the suggested settings, its average conversion is higher (61.98 against 61.32 %) and its samples sit higher in the band (3.049 and 3.070), so it has more margin on every part of the target." Find the flaw, and say how you would check the claim.