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CHAPTER 10

A Lithium Hydroxide Refinery Simulator for Interacting Loops on Three Units in Series

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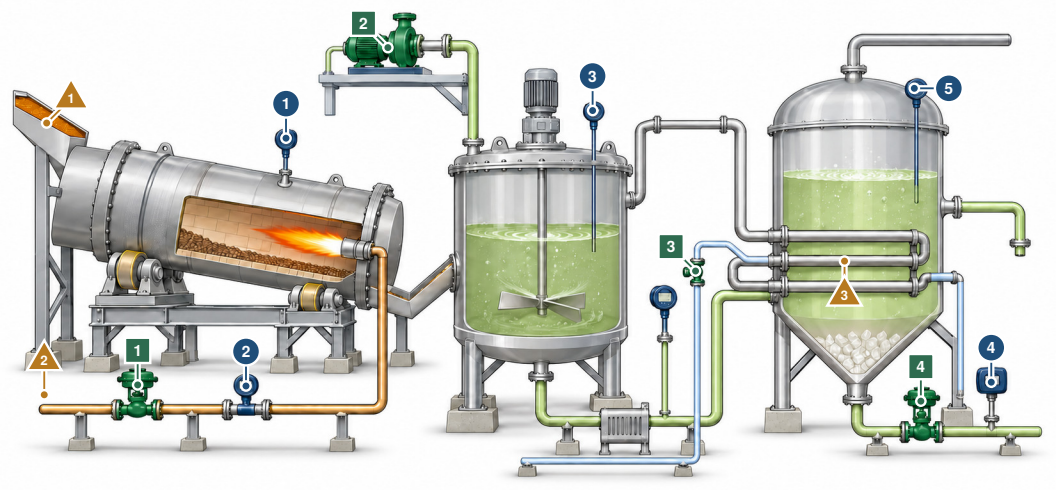
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CHAPTER 10

A Lithium Hydroxide Refinery Simulator for Interacting Loops on Three Units in Series



The controller moves 1 the fuel valve of the kiln burner 2 the lime slurry pump 3 the steam valve that heats the evaporator 4 the product draw, which holds the level

The controller reads 1 the kiln bed temperature 2 the fuel flow, for the cascade 3 the pH electrode in the leach 4 the product strength analyzer, four minutes late 5 the evaporator level

What disturbs it 1 the ore grade of the feed 2 the gas supply pressure and heating value 3 scale on the heating tubes

Notice that one change in ore grade at the kiln feed reaches the kiln, the leach and the product at three different times, while each loop holds its own reading. Illustration, not to scale.

IN PLAIN TERMS **Three Steps in a Row, One Ore**

The main idea. A refinery turns crushed lithium ore into a strong lithium solution in three steps. A turning oven heats the ore, a stirred tank dissolves the lithium with lime, and a boiler drives off water. Each step has its own controller.

The challenge. Nothing is stored between the steps, so a change at the front reaches each controller at a different time. When the ore gets poorer, every controller holds its reading, yet the plant makes 27 percent less lithium. A probe that misreads the tank can even make the plant look better while it wastes lime.

Why it matters. A plant that watches only its controllers can lose output or money with no alarm.

THREE WAYS TO READ THIS CHAPTER

Explore	12 min	The first page, the fact box of the Introduction, the history box of Section 10.2, the try-it boxes and the fact box of Section 10.6, and the key points
Study	45 min	The whole chapter with its pause points and the twelve exercises
Practice	15 min	The first page, Section 10.6, Section 10.7, Section 10.8 and the open challenge in it

The control question A calciner, a leach and an evaporative crystallizer run in series with no storage between them, so one disturbance reaches three loops at three different times. What does each loop see, what is a fuel flow cascade worth, and what does the plant lose while every loop holds its setpoint?

Learning objectives

- Identify each unit as a first-order plus dead-time plant from one manual step and turn the model into SIMC tunings.
- Predict which fuel-side disturbances a fuel flow cascade rejects, from what its inner loop measures.
- Trace an ore grade change through the three loops, with the sign and arrival time at each, and explain why the output falls with every loop on setpoint.
- Explain the interior maximum of the pH loop gain and where it makes a loop safe to tune.
- Detect a biased electrode or a scaling evaporator from evidence outside the loop that hides it.

Prerequisites Sections 1.6.2 (velocity form), 1.6.4 (scenarios and exports), 1.4.4 (leaching), 1.4.6 (cascade) and 1.4.7 (control layers of a circuit). Chapter 4 sorts a cascade's disturbances the same way.

The app https://apmonitor.com/apps/lithium-refinery/

See also Volume 6, Chapter 7 and Volume 6, Chapter 8 for plantwide structure, Volume 6, Chapter 4 for an evaporator held through a late analyzer. Volume 3, Chapter 2 and Volume 3, Chapter 4 for cascade by disturbance entry and fuel control on a fired heater. Volume 5, Chapter 3 and Volume 5, Chapter 11 for a slow analyzer and an unmeasured kiln product.

10.1 Introduction

A hard-rock lithium refinery converts spodumene concentrate into a lithium hydroxide solution in three steps. A rotary calciner heats the concentrate above 1000 °C, where the mineral converts from its dense alpha form to a porous beta form that reagents can reach [1, 2]. An alkaline leach dissolves the lithium out of the converted solid. An evaporative crystallizer concentrates the resulting liquor to the strength the downstream crystallization needs [3].

> **DID YOU KNOW? Lithium Mined in 2024**
>
> In 2024 the world produced an estimated 240 000 tons of lithium, counted as lithium content, 18 % more than in 2023. Australia, where the lithium comes from mineral operations, produced 88 000 tons of it. Batteries took an estimated 87 % of the lithium used [4].

Each unit has one controlled variable and one actuator, and each of the three loops is straightforward in isolation. The three are in series with no intermediate storage, so a disturbance entering at the front reaches each loop in turn at a time set by the units upstream of it. One fuel valve step reaches the spodumene conversion in 14.0 min, the kiln temperature the valve is nominally controlling in 29.3 min, and the product concentration two units downstream in 112.8 min, a factor of 8.1 between the first and the last. A controller tuned against the kiln loop alone therefore addresses one of three response times.

The model rests on standard process fundamentals. Conservation of energy on the kiln charge and conservation of species in the leach and the evaporator give one state equation per inventory, and the number of inventories in series sets the order of the response and the phase lag a controller has to survive [5, 6]. Each rate is written against a driving force, so the conversion follows an Arrhenius rate in bed temperature, the leach rate is first order in both unleached lithium and free hydroxide, and the evaporation rate is proportional to duty, which makes every loop gain a function of the operating point rather than a constant. Time-scale separation orders the resulting lags, with a 40 min solids residence in the kiln and a 44 min liquor residence in the evaporator against a 0.3 min valve and a 0.25 min electrode, which is what permits each unit to be identified as a first-order plus dead-time model [7].

The model and its scenarios teach open-loop identification from a reaction curve, process order and dead time, cascade structure and the disturbance classes an inner loop does and does not reject, nonlinear loop gain and the case for detuning, sampled and delayed measurements, integrating level control, anti-windup and bumpless transfer, the effect of instrument bias on a closed loop, and the distinction between a loop settling time and a plant settling time [5, 7]. Industry runs fired equipment on the flow cascade studied here [8], operates hydrometallurgical circuits against feed grade as the dominant unmeasured disturbance [9, 10], and pays for reagent consumption that no controlled variable reports.

This chapter describes a browser-hosted lithium hydroxide refinery simulator in enough detail to be rebuilt, measures what a fuel flow cascade is worth against three different fuel-side disturbances on the same kiln, and follows a single upstream ore grade change through three loops that all return to setpoint. Three measured outcomes carry the argument. The cascade reduces the integral error from a 20 % header pressure loss by a factor of 52 to 93, depending on where in the controller scan the loss arrives, and the same error from a 7 % heating value loss not at all. A grade change from 5.5 to 4.0 % Li_2O moves no controlled variable by more than a sixth of a pH unit and removes 27 % of the plant output over six hours. A pH electrode reading 0.70 low raises the extraction, the yield and the production while raising the reagent consumption by 24 %. The unit models, the cascade structure and the tuning rules are standard. What the app adds is an eleven-state model of three units in series, the identified model of each unit, and eleven scenarios with a completion requirement each, the last a graded challenge whose clauses are separable.

Section 10.2 reviews lithium refining, kiln control and plantwide control. Section 10.3 gives the equations of the calciner, the leach and the evaporative crystallizer. Section 10.4 identifies each unit from a step test, measures how fast one fuel valve reaches three plant variables and how the pH loop gain moves with the operating point. Section 10.5 sets out the loops and their tunings. Section 10.6 reports the cascade against three fuel-side disturbances, the grade change walking through the train, a throughput increase, evaporator scaling, a biased pH electrode and the graded run. Section 10.7 names the real instruments, final elements and platform of the loops. Sections 10.8 and 10.9 state what the model leaves out and which results carry over to other plants, and the Exercises close the chapter.

10.2 Background

Spodumene, $LiAlSi_2O_6$, carries 8.03 % Li_2O when pure, and a commercial concentrate assaying 5.5 % Li_2O is therefore roughly two thirds spodumene and one third gangue. The alpha form is essentially inert to acid and alkaline attack, and the industrial route begins by decrepitating it to the beta form above about 1000 °C [2]. The transformation proceeds through an intermediate gamma phase, and the reported activation energies vary with particle size, heating rate and impurities [1]. Downstream of the calciner the sulfate route and the alkaline route compete, and the flowsheets, reagent choices and impurity removal steps are surveyed for the lithium market as a whole [3]. Alkaline leaching of spodumene with hydroxide has been characterized against thermodynamic and surface data [11], and first-order dissolution kinetics of the form used here are standard for hydrometallurgical circuits [12].

Kiln control has a long history in the process industries. The first industrial application of fuzzy logic control was a cement kiln, chosen because the plant was too nonlinear, too slow and too poorly instrumented for the classical methods available at the time [13]. Model predictive control later displaced rule-based schemes on the same equipment, handling the kiln with the calciner and the cooler in one calculation [14], and Chapter 11 treats a rotary cement kiln whose air follows its fuel. On the modeling side, one-dimensional axial models of the bed and the counter-current gas have been developed and validated against plant data [15] and extended to the integrated preheater, calciner, kiln and cooler so that the recycles between them can be represented [16]. Those models are the reference against which the reduced kiln used here should be read.

> ⧖ HISTORY **Heat First, Then Acid (1948 to 1950)**
>
> A patent filed in 1948 and granted in 1950 describes the route that hard-rock lithium still follows. Its inventors found no acid strength, time or temperature that could take lithium out of spodumene as mined. They first heated the crushed ore to between about 1000 and 1350 °C, in a muffle, rotary or direct-fired furnace, to turn alpha-spodumene into beta, and found 1050 to 1100 °C ample. Any furnace would serve so long as uniform and adequate temperature control was possible. The beta form was then roasted with strong sulfuric acid at about 250 °C, never much above 300 °C, and the lithium sulfate leached out with water [17]. In 2019 this acid route was still the only one practiced on an industrial scale, and alkaline leaching was one of the alternatives proposed [18].

Plantwide control is the branch of the field that treats the interaction between units rather than the tuning of any one loop. The control structure design problem asks which variables to control, which to manipulate and how to pair them so that the plant, rather than the individual loop, meets its economic objective [5, 19]. Volume 6, Chapter 7 poses the same question for a reactor and a column joined by a recycle. Mineral processing plants make the question concrete, because feed grade and mineralogy vary on a time scale of hours, the measurements that would resolve them are sparse or offline, and reagent consumption is one of the two largest operating costs [9, 10]. The cascade structure is the standard local remedy for an actuator-side disturbance, and the fired-heater arrangement in which a temperature controller sets a fuel flow setpoint is the textbook case [5, 8].

However, the textbook justification for a cascade is usually presented as a single benefit, without separating the disturbances that enter inside the inner loop from those that do not. A fuel flow cascade rejects a header pressure change almost completely, because the inner loop measures the flow that the pressure changes. It rejects nothing at all of a heating value change, because the mass flow of gas is unaltered and no instrument in either loop observes the energy content. Volume 3, Chapter 2 sorts disturbances the same way on a level loop, and Chapter 4 on the feed pressure of a cyclone cluster. The application is built so that both disturbances can be applied to the same kiln under the same tuning, and so that the resulting numbers can be quoted separately.

10.3 Process Model

Figure 10.1 shows the train and its four loops.

10.3.1 Design Point and Overall Balance

The clock is in minutes. The plant takes 500 kg/min of concentrate at 5.5 % Li_2O and 3 % moisture. At design the calciner runs at 1090 °C on 26.5 kg/min of fuel and converts 93.5 % of the spodumene, the leach runs at pH 12.500 on 279.1 L/min of lime slurry, and the crystallizer holds 12.00 kg Li/m^3 at 77.1 % of steam duty on a 3.33 m level.

The lithium balance closes at every stage. The concentrate carries 12.39 kg/min of lithium, the calciner converts 11.59 kg/min of it into leachable form, the leach dissolves 11.04 kg/min, and the filter recovers 98.5 % of that as 10.87 kg/min of lithium in product liquor, an overall yield of 87.7 %. The application reports a leach extraction of 95.7 % on a concentration basis, which is 95.2 % on a mass basis because the reagent flow dilutes the slurry between the inlet and the outlet of the leach vessel.

10.3.2 Calciner

The kiln is one well-mixed zone carrying an energy balance on the charge and a first-order conversion. The energy balance is Equation (10.1).

$$M_k c_{p,k} \frac{\mathrm{d}T_k}{\mathrm{d}t} = \eta_f \Delta H_f \dot{m}_f - F_s c_{p,s}(T_k - T_{\mathrm{in}}) - UA_k(T_k - T_{\mathrm{amb}}) - \Delta H_c F_s \phi X_k - F_s w h_{\mathrm{vap}} \mu(T_k) \quad (10.1)$$

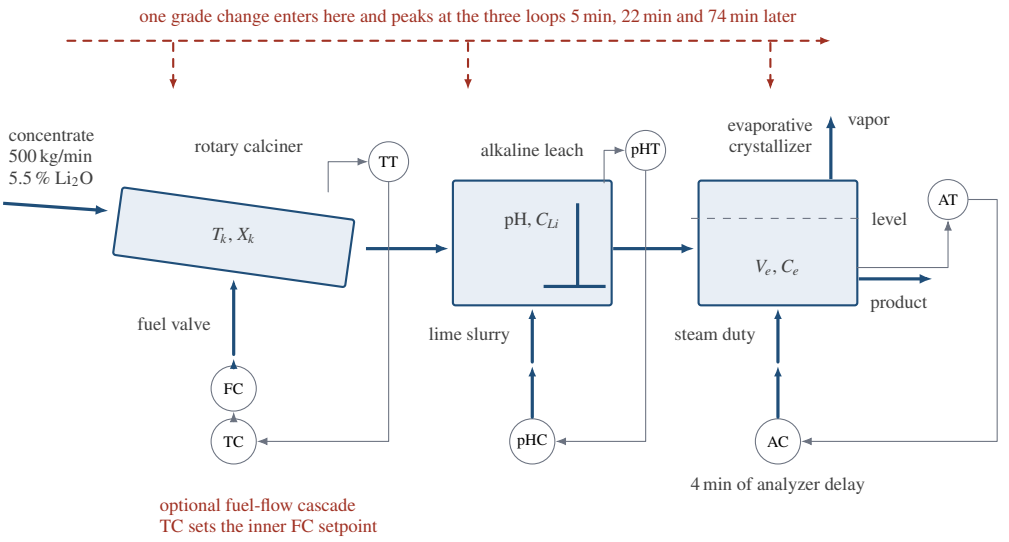


Figure 10.1 The simulated refinery. Three units in series, each with one loop. Kiln temperature goes to the fuel valve, optionally through a fuel flow controller in cascade. Leach pH goes to the lime slurry. Product concentration goes to the evaporator steam duty, through an analyzer with four minutes of transport delay. A fourth loop holds the crystallizer level. The dashed red path is the ore grade, which enters at the front and reaches the three controlled variables at three different times.

where $M_k c_{p,k} = 22\,000\,\mathrm{kJ/K}$ is the lumped thermal mass of the shell and charge, $\eta_f = 0.65$ is the burner efficiency to the bed, $\Delta H_f = 50\,000\,\mathrm{kJ/kg}$ is the fuel heating value, $\dot{m}_f$ is the fuel mass flow, F_s is the concentrate rate, $c_{p,s} = 1.05\,\mathrm{kJ/(kg\,K)}$, $UA_k = 220\,\mathrm{kJ/(min\,K)}$ is the shell loss coefficient, $\Delta H_c = 100\,\mathrm{kJ/kg}$ is the decrepitation enthalpy, $\phi = 0.685$ is the spodumene fraction of the concentrate at 5.5 % Li_2O, w is the moisture fraction, $h_{\mathrm{vap}} = 2450\,\mathrm{kJ/kg}$ and $\mu(T_k)$ is the fraction of the moisture driven off, equal to 1 at 100 °C and above and falling linearly to 0 at T_{in}, so that a kiln without fuel settles at the feed temperature. Every term is in kJ/min. At design the burner delivers 862 MJ/min against 559 of sensible heat, 234 of shell loss, 37 of moisture and 32 of reaction. The linearized thermal time constant is $M_k c_{p,k}/(F_s c_{p,s} + UA_k) = 29.5\,\mathrm{min}$.

Only the spodumene fraction of the concentrate absorbs the decrepitation enthalpy, so a lower ore grade is a smaller heat sink and drives the bed temperature up rather than down. That sign is the first of the three different disturbances one grade change delivers to this plant.

The conversion is a first-order reaction against a solids residence time, as shown in Equation (10.2).

$$\frac{\mathrm{d}X_k}{\mathrm{d}t} = k(T_k)\,(1 - X_k) - \frac{X_k}{\tau_s}, \qquad \tau_s = \frac{M_s}{F_s} \tag{10.2}$$

$$k(T_k) = k_{\mathrm{ref}}\,\mathrm{e}^{-\frac{E}{R}\left(\frac{1}{T_k} - \frac{1}{T_{\mathrm{ref}}}\right)} \tag{10.3}$$

where X_k is the fraction of the spodumene converted to the beta form, $M_s = 20\,000\,\mathrm{kg}$ is the kiln solids hold-up, $k_{\mathrm{ref}} = 0.475/\mathrm{min}$ at $T_{\mathrm{ref}} = 1100\,^{\circ}\mathrm{C}$, $E/R = 50\,500\,\mathrm{K}$ and both temperatures in (10.3) are absolute. The washout term X_k/τ_s is what makes the conversion less than unity, because material leaves the kiln after $\tau_s = 40\,\mathrm{min}$ whether or not it has reacted. At steady state $X_k = k\tau_s/(1 + k\tau_s)$, which gives 0.9355 at 1090 °C on $k = 0.363/\mathrm{min}$.

The conversion saturates against temperature over the operating range, at 0.780 at 1040 °C, 0.935 at 1090 °C and 0.982 at 1140 °C, and the overall plant yield follows it from 73.3 % to 92.0 % across the same span. The refractory limit is 1160 °C. Below about 1050 °C the kiln converts too little for

the leach to recover, and the loss is permanent because no downstream unit acts on unconverted alpha spodumene.

The fuel valve is a first-order lag of 0.3 min and the bed thermocouple carries 1.6 min of transport delay.

10.3.3 Leach

The leach is one stirred tank carrying three species balances and an energy balance. Extraction is first order in the unleached lithium and first order in the free hydroxide, as shown in Equations (10.4) to (10.6).

$$r = V_L\, k_L(T_L)\, C_s\, C_R \tag{10.4}$$

$$V_L \frac{\mathrm{d}C_s}{\mathrm{d}t} = F_{\text{in}} C_{s,\text{in}} - F_{\text{out}} C_s - r$$

$$V_L \frac{\mathrm{d}C_{Li}}{\mathrm{d}t} = r - F_{\text{out}} C_{Li} \tag{10.5}$$

$$V_L \frac{\mathrm{d}C_R}{\mathrm{d}t} = F_R C_{R,f} - F_{\text{out}} C_R - \nu\, r \tag{10.6}$$

where $V_L = 150\,\mathrm{m}^3$ is the reactor volume, C_s is the unleached lithium in the solids in kg Li/m^3, C_{Li} is the dissolved lithium, C_R is the free hydroxide in mol/m^3, $k_L = 0.0111\,\mathrm{m}^3/(\mathrm{mol\,min})$ at 95 °C with $E/R = 7000\,\mathrm{K}$, F_R is the lime slurry flow, $C_{R,f} = 6000\,\mathrm{mol/m}^3$ is its strength, and $\nu = 144.1\,\mathrm{mol/kg}$ is the hydroxide consumed per kilogram of lithium taken into solution, which is one mole of hydroxide per mole of lithium. The rate r is in kg/min, and $F_{\text{out}} = F_{\text{in}} + F_R$ carries the reagent dilution that separates the concentration-basis and mass-basis extractions.

The measurement is the logarithm of the free hydroxide, as shown in Equation (10.7).

$$\mathrm{pH} = 14 + \log_{10}\left(\frac{C_R + C_{\text{buf}}}{1000}\right) \tag{10.7}$$

where $C_{\text{buf}} = 0.5\,\mathrm{mol/m}^3$ is the residue alkalinity floor and the divisor converts mol/m^3 to mol/L. Extraction against pH saturates sharply, at 84.7 % at pH 11.9, 95.7 % at 12.5 and 98.9 % at 13.1, on lime rates of 235, 279 and 332 L/min. Above about pH 12.8 the extraction is within two points of its ceiling and further reagent buys almost nothing, which makes the pH setpoint an operating cost decision rather than a quality one.

The leach temperature is held near 95.8 °C by a jacket steam setting that is an operator input rather than a controlled variable. The electrode is a 0.25 min first-order lag with optional bias and noise.

10.3.4 Evaporative Crystallizer

The evaporator is a volume and a solute inventory, as shown in Equation (10.8).

$$\begin{aligned} \frac{\mathrm{d}V_e}{\mathrm{d}t} &= F_e - F_p - F_v, \qquad F_v = k_v Q_a \\ \frac{\mathrm{d}(V_e C_e)}{\mathrm{d}t} &= F_e C_{e,\text{in}} - F_p C_e \end{aligned} \tag{10.8}$$

where F_e is the filtrate feed, F_p is the product draw, F_v is the vapor rate, Q_a is the applied duty in MW behind a 0.8 min lag, and $k_v = 0.0955\,\mathrm{m}^3/(\mathrm{min\,MW})$. The vapor carries no lithium, which is why the liquor concentrates. Expanding the product rule gives the form integrated by the application, $\dot{C}_e = (F_e C_{e,\text{in}} - F_p C_e - C_e \dot{V}_e)/V_e$.

The level is an integrating state held by a dedicated draw controller that runs in both manual and automatic modes. It completes 63 % of a 0.3 m setpoint change in 2.5 min and 95 % in 4.3 min, then overshoots by 13.5 % and stays within 5 % of the new setpoint from 17.2 min. The concentration is

Table 10.1 First-order plus dead-time models identified from one step per unit (2 % fuel, 5 L/min lime, 1 % duty), measured at the transmitter. The two-point fit of the leach returns a negative dead time, so its entry bounds the delay before the transmitter first moves.

Loop	Gain	τ_p (min)	θ_p (min)
Kiln temperature	18.7 °C/%	28.4	1.86
Leach pH	0.0159 pH per L/min	55.0	< 0.3
Product strength	0.258 kg Li/m^3 per %	45.1	5.96

far slower. Its open-loop time constant is the liquor residence $V_e/F_p = 44$ min, and a step in duty moves it with a measured time constant of 45.1 min through an analyzer that adds 4.0 min of transport delay and a 1.2 min lag. Product below 11.5 kg Li/m^3 is unsaleable, and above the 15.5 kg Li/m^3 saturation line lithium hydroxide monohydrate deposits on the heating surfaces rather than in the crystallizer, so the concentration loop approaches a hard constraint through five minutes of dead time.

Scaling on those surfaces reduces the evaporation obtained per unit of duty over weeks of operation, which the application applies as a step in k_v within a single run.

10.3.5 Initialization, Numerics and Assumptions

The eleven states are the kiln temperature and conversion, the fuel valve position, the three leach concentrations, the leach temperature, the evaporator volume and concentration, the applied evaporator duty and the applied jacket steam. Initialization solves the three units in flow order at the given actuator settings, iterating the kiln because the reaction is a heat sink whose size depends on the temperature that drives it, and bisecting the leach hydroxide balance because the residual is monotone in C_R while a fixed-point substitution is not. The result is an exact equilibrium, so a run that is left alone does not drift. A manual-mode helper inverts the same steady-state solution to return the fuel, lime and duty that place all three controlled variables on their setpoints at the present feed, as far as the actuator ranges allow.

The engine of Section 1.6.1 integrates the model by fixed-step fourth-order Runge-Kutta at 0.01 min (0.6 s) and runs the controllers on a 0.1 min (6 s) scan, so every measurement is a sampled one and each loop carries half a scan of additional lag.

The calciner is one well-mixed zone rather than an axial profile, which is adequate for a control argument and not for a kiln design [15]. Impurity removal, the sulfate route and the final crystallization to lithium hydroxide monohydrate are outside the boundary.

10.4 Dynamic Analysis

10.4.1 Identified Models of the Three Units

Each unit is identified the way a commissioning engineer identifies one, by stepping its actuator in manual and reading the response off the transmitter. The two-point fits are given in Table 10.1.

None of the three identified plants is difficult to control in isolation. The kiln is close to a textbook first-order plus dead-time process with a dead-time fraction of 0.07. The leach carries no transport delay at all, only the electrode lag. The product loop has a dead-time fraction of 0.13 and approaches a hard saturation constraint rather than a graded one, and it is the loop that has to be detuned.

10.4.2 Propagation Speed Through the Train

The same actuator reaches three plant variables at three different times, as listed in Table 10.2.

Table 10.2 carries the measurement the application is organized around. The fuel valve reaches the conversion in fourteen minutes, the temperature it is nominally controlling in twenty-nine, and the product concentration two units downstream in nearly two hours, with ninety-five percent of the last taking four hours.

Table 10.2 Measured open-loop response of three plant variables to the same 5 % fuel valve step. The actuator is one valve. The step carries the bed past the 1160 °C refractory limit 38.8 min after it is made, which the application flags, so scenario 1 uses a 3 % step.

Variable	Unit	τ_{63} (min)	τ_{95} (min)
Spodumene conversion X_k	calciner	14.0	51.8
Kiln temperature T_k	calciner	29.3	87.8
Product concentration C_e	crystallizer	112.8	239.7

The conversion is faster than the temperature that drives it, which is a consequence of the saturation in Equation (10.2) rather than of any extra dynamics. The conversion has a time constant of $1/(k+1/\tau_s) = 2.6$ min about the design point, so it tracks the quasi-steady value $k\tau_s/(1+k\tau_s)$ with little lag. That value is a concave function of temperature, and when the bed has completed 63 % of a 95 °C rise the quasi-steady conversion has already completed 86 % of its own change. Approaching a saturating characteristic makes a downstream variable look faster than its driver, and the effect is not a modeling artifact.

10.4.3 Operating-Point Dependence of the pH Loop Gain

The pH loop is the one whose gain is not a constant, and the way it varies is not the textbook titration curve. Measured open-loop gains for a 5 L/min step are 0.0104 pH per L/min at pH 11.9, 0.0159 at 12.5, 0.0116 at 12.8, 0.0067 at 13.1 and 0.0034 at 13.4. The gain rises to a maximum at the design point and then falls by a factor of 2.4 by pH 13.1 and 4.7 by pH 13.4.

Two mechanisms compete. Below the design point almost all of the added hydroxide is consumed by (10.4), because the extraction is still climbing, so reagent buys extraction rather than free alkalinity and the pH moves little. Above it the extraction is saturated, the added hydroxide accumulates as C_R, and the logarithm of (10.7) compresses a tenfold concentration change into one pH unit. A controller tuned at the design point therefore carries about one fifth of its design loop gain by pH 13.4, which is where an instrument bias or a low ore grade will put it. The interior maximum makes the design point the safe place to tune, because the loop gain falls in both directions and the loop grows sluggish rather than oscillatory. Tuning the same loop at pH 13.1 and returning the plant to the design point reverses the argument, because the loop gain is then 2.4 times the value it was tuned for.

Scenario 2 shows the falling gain from two 30 L/min lime steps, from 279 to 309 and then to 339 L/min, each held 150 min. The hold has to be that long, because the pH covers two thirds of the first step only after 47 min. Read after 45 min, the two steps give 0.25 and 0.26 pH and the comparison reverses, while the 150 min holds give 0.38 and 0.25 against settled changes of 0.40 and 0.24.

10.5 Controller Design

10.5.1 Velocity Form PID Algorithm

All four loops, and the inner fuel flow loop when the kiln is in cascade, run the velocity-form PID of Section 1.6.2, Equations (1.10) to (1.13), with the derivative on the measurement [20] and a control period $\Delta t = 0.1$ min. The kiln, pH and product strength loops, and the inner fuel flow loop, are reverse acting with $a = +1$, because more fuel, lime or duty raises the measurement. The level loop is direct acting with $a = -1$, because more product draw lowers the level. The derivative of the kiln loop acts on a measurement filtered with $\tau_f = 0.5$ min, and τ_D is zero in every shipped and recommended tuning. The output is clipped to the actuator span and back-calculation with a tracking time $T_t = \tau_I$ corrects the integral alone. No rate limit and no further clamp act between the controller output and the actuator.

Among the runs reported here only the scaling case of scenario 9 puts an actuator on a limit, the evaporator duty at 227.3 min, and no reported number depends on the tracking time. Transfer

Table 10.3 Recommended and cautious tunings. The kiln loop follows the SIMC rules at $\tau_c = \theta_p$ and the other two are detuned from it.

	Recommended		Cautious	
Loop	K_c	τ_I (min)	K_c	τ_I (min)
Kiln temperature	0.42	15	0.08	40
Leach pH	240	25	40	60
Product strength	13	35	3	80
Inner fuel flow	3	0.4	3	0.4
Level	6	8	6	8

between Manual and Auto is bumpless in both directions, as Section 1.6.2 describes. With the kiln in single-loop mode the inner flow controller tracks the valve command in the same way. Output limits are 0 % to 100 % on the fuel valve and the evaporator duty, 0 L/min to 900 L/min on the lime slurry and 0 m³/min to 4 m³/min on the product draw.

10.5.2 Loop Pairing and Tunings

Kiln temperature goes to the fuel valve, leach pH to the lime slurry, product concentration to the steam duty and crystallizer level to the product draw. The level loop runs in both modes, because an inventory that nobody holds does not wait for an operator to select automatic. The recommended tunings are listed in Table 10.3 beside the cautious set that the application loads at start-up, in the three manual scenarios and in the challenge.

The SIMC rules give $K_c = \tau_p/(K_p(\tau_c + \theta_p))$ and $\tau_I = \min(\tau_p, 4(\tau_c + \theta_p))$ [21]. Applied to the kiln row of Table 10.1 with the standard aggressive choice $\tau_c = \theta_p$, they return $K_c = 0.41$ and $\tau_I = 14.9$ min against the 0.42 and 15 min recommended. The product loop is detuned to $\tau_c = 7.5$ min against a dead time of 5.96 min because it approaches the saturation constraint through that dead time, and the pH loop to $\tau_c = 14.1$ min because its identified dead time is under 0.3 min, where the aggressive choice $\tau_c = \theta_p$ would ask for a controller gain near 5800 L/min per pH unit against an actuator that spans 900 L/min. The integral times shipped for the two detuned loops, 25 and 35 min, are shorter than the 55 and 45 min that $\min(\tau_p, 4(\tau_c + \theta_p))$ returns for them.

The kiln loop is offered two ways. Single-loop, the temperature controller positions the fuel valve directly. In cascade, it sets a fuel flow setpoint and a fast flow controller positions the valve, which is the classical arrangement for a fired heater [8] and the one rotary kilns use [13, 14]. The inner loop settles in well under a minute against an outer time constant of 28.4 min, so the separation of time scales that cascade control requires is present by a factor of more than thirty [5].

10.6 Results

10.6.1 Cascade Against Three Fuel-Side Disturbances

Each disturbance is applied alone to the plant at its design steady state and followed for 300 min under both architectures at the same outer tuning. The runs use scenario 5 with the architecture selected before the run and the disturbance slider or setpoint moved at 20 min, on a controller scan. Results are given in Table 10.4.

Table 10.4, drawn as a chart in Figure 10.2, separates the two halves of the classical cascade argument, and the two are of very different size. Against a header pressure loss the cascade reduces the peak by a factor of 34 and the integral error by a factor of 93, because the inner loop measures the flow that the pressure changed and restores it to within 0.5 % in 0.6 min, before the bed temperature has moved more than 0.85 °C. The inner loop acts only at its 0.1 min scan, so a loss arriving just after a scan goes uncorrected for nearly a whole scan, and the factors fall to 18.9 and 52. Against a heating value loss the cascade reduces the peak by a factor of 1.05 and the integral error not at all, because

Table 10.4 Kiln temperature loop, single loop and cascade, one disturbance at a time from the design steady state. Peak is the largest absolute deviation from setpoint in °C and IAE is in °C min over 300 min. The setpoint row gives the overshoot past the new setpoint, because the peak error of a setpoint step is the step itself. A change that arrives one physics step after a scan raises the cascade header entries to 1.52 and 10.2 (1.56 and 28.8 at the cautious tuning) and each setpoint IAE by 2.7, and leaves the other entries unchanged.

	Single loop		Cascade	
Disturbance	Peak	IAE	Peak	IAE
Recommended tuning				
Setpoint +30 °C	5.15	117.7	3.75	**107.2**
Header pressure −20 %	28.79	526.3	**0.85**	**5.7**
Heating value −7 %	9.38	158.4	8.96	158.4
Cautious tuning				
Header pressure −20 %	79.25	7311	**0.88**	**16.0**

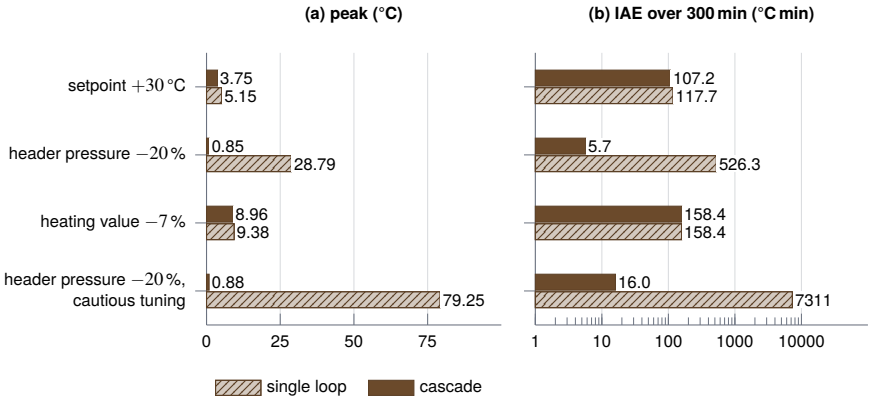


Figure 10.2 The kiln cascade of Table 10.4 as a chart, single loop hatched and cascade solid; the setpoint row's peak is the overshoot. Against a header pressure loss the cascade bar nearly vanishes at either tuning, while against a heating value loss the two bars are almost equal and the integral errors identical: the inner loop rejects only what its flow meter can see.

the mass flow of gas is unchanged, the inner loop sees nothing, and the whole energy error has to be found by the outer loop through the 1.86 min identified dead time. On a setpoint change it improves the integral error by a factor of 1.10, because removing the valve lag from the outer loop path is all that is left to remove and the 28.4 min thermal lag is untouched.

🕮 **Pause.** Try Exercise 10.1 before reading on.

The inner loop's rejection is nearly independent of the outer tuning. At the cautious tuning the same header dip costs 79.25 °C single loop and 0.88 °C in cascade, so detuning the outer loop by a factor of 5.3 in gain multiplies the single-loop peak by 2.8 and leaves the cascade peak within 0.03 °C of where it was. The two effects do not compose, because they act on different paths. Scenario 4 delivers the same comparison as one scripted sequence, a 25 % header loss at 20 min, its recovery at 90 min and a 10 % heating value loss at 130 min. The kiln falls 37.2 °C and then overshoots 43.1 °C single loop against 1.2 °C and 1.0 °C in cascade, while the heating value loss costs 11.7 °C single loop and 13.0 °C in cascade. The single-loop figure is the smaller only because that kiln is still 2.9 °C above setpoint from the overshoot when the loss arrives.

Table 10.5 A single upstream grade change walking through the train. Every controlled variable returns to setpoint.

t (min)	T_k (°C)	pH	Lime (L/min)	C_e (kg Li/m^3)	Output (kg/min)
19	1090.00	12.500	279.1	12.000	10.870
25	1091.26	12.562	264.1	12.000	10.774
40	1090.42	12.662	221.1	11.967	10.187
90	1090.01	12.531	203.0	11.787	8.773
220	1090.00	12.500	207.1	11.961	7.988
400	1090.00	12.500	207.1	11.998	7.935

> **TRY IT IN THE APP** **4 · Cascade on the Kiln**
>
> Run the four hours with Kiln architecture on Single loop. Then press Reset, switch it to Cascade and run again. The 25 % header pressure loss at minute 20 drops the kiln 37.2 °C on the single loop and 1.2 °C in cascade. The 10 % heating value loss at minute 130 costs 11.7 °C and 13.0 °C: the inner loop cannot see it.

10.6.2 Propagation of an Ore Grade Change

Scenario 6 drops the ore grade from 5.5 to 4.0 % Li_2O at twenty minutes with every setpoint and tuning unchanged. The trajectory is given in Table 10.5.

Table 10.5 records the central plantwide experiment, and every excursion in it is small. The kiln temperature peaks 1.26 °C high five minutes after the change and comes back. The pH peaks 0.163 high after twenty-two minutes and comes back. The product concentration reaches 0.214 kg Li/m^3 low after seventy-four minutes and comes back. Each loop does what it was designed to do, the largest excursion anywhere on the plant is a sixth of a pH unit, and the plant output falls from 10.87 to 7.93 kg/min, a reduction of 27 %.

> **TRY IT IN THE APP** **6 · The Ore Grade Falls**
>
> Load the scenario and press Run for four hours. Watch the lithium-through-the-plant trend. The grade drops at minute 20. The kiln temperature rises 1.26 °C and comes back, the pH rises 0.163 and comes back, and the product strength dips 0.214 kg Li/m^3 and comes back, yet the lithium recovered as product falls from 10.87 toward 7.93 kg/min and stays down.

The order of arrival is the instructive part. The kiln moves first, and it moves up, because the reaction is a heat sink and less spodumene is a smaller sink. The lime flow follows within about a minute, because less lithium in the feed is less hydroxide demand and the free alkalinity climbs, so the pH controller correctly cuts reagent by 26 % at the new steady state. The evaporator is last and is the unit under pressure, because weaker liquor has to be boiled harder to reach the same product strength, and its duty climbs from 77.1 to 86.6 %. The plant is still moving at 220 min and settles at about 400 min, six hours after an instantaneous change at the plant inlet.

Pause. Try Exercise 10.2 before reading on.

Nothing in this scenario is a control failure and nothing in it is remedied by a controller. What the scenario demonstrates is that a plant has a settling time that is not any loop's settling time, and that the correct response to a grade change is a production plan rather than a tuning change.

10.6.3 Grade Increase and Throughput Increase

Scenario 7 runs the same experiment upward, to 6.6 % Li_2O, and dilutes the lime slurry to 70 % strength at 150 min. Production settles at 12.93 kg/min and the product concentration peaks at 12.15 kg Li/m^3, well below the 15.5 kg Li/m^3 saturation line. The pH excursion of 0.251 is the largest

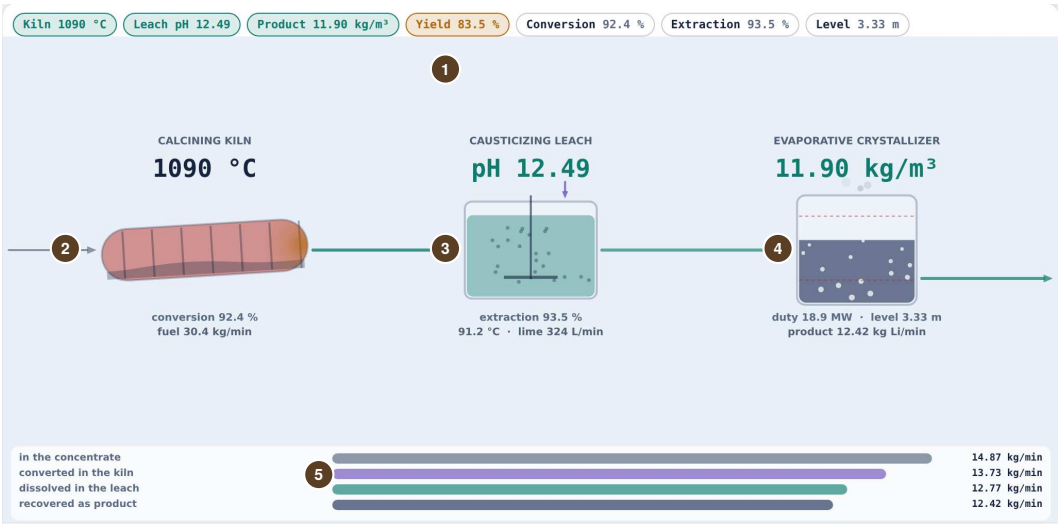


Figure 10.3 Scenario 8 at 2.08 h (124.8 min), 100 min after a twenty percent throughput increase, cropped to the plant view. All three loops are back at or near setpoint, with the kiln at 1090.0 °C, the leach at pH 12.492 and the product at 11.90 kg Li/m^3. (1) The badge row carries the three controlled variables and the yield. (2) The calcining kiln shows its bed temperature, conversion and fuel, (3) the leach its pH, extraction and lime flow, and (4) the evaporative crystallizer its product strength, duty and level. (5) The lithium balance beneath the flowsheet accounts for the metal at each stage: 14.87 kg/min in the concentrate, 13.73 converted in the kiln, 12.77 dissolved in the leach and 12.42 recovered as product, for a yield of 83.5 % against 87.7 before the change.

of the three feed-side scenarios, because the reagent loop is being asked for more lime at the moment each liter carries less hydroxide, and the lime flow ends at 472 L/min against 279 L/min at design. The flow controller has no defense against a reagent concentration change, because the disturbance is in the strength of the stream rather than in its rate.

Scenario 8 raises the concentrate feed by 20 % instead. The kiln temperature dips 17.71 °C while the fuel catches up and rises 17.95 °C when the feed is restored, the pH holds to 0.084 and the product concentration moves 0.921 kg Li/m^3, which is the largest single deviation the application produces from a normal operating change and nearly the whole of the graded run's ±1.0 kg Li/m^3 band. The dip that follows the push holds the product below the 11.5 kg Li/m^3 sales limit for 39.3 min. The kiln temperature loop holds 1090 °C without difficulty, and the conversion falls from 0.9355 to 0.9236 anyway, because the solids residence time falls from 40 min to 33.3 min and no controller measures conversion. That unmeasured loss carries through the train, and the yield falls from 87.7 % to 84.4 % with every controlled variable on setpoint, as shown in Figure 10.3. The crystallizer is the unit that reaches its capacity first, which is information required before a throughput increase rather than after one.

10.6.4 Evaporator Scaling

Scenario 9 steps the evaporator surface to 94, 86 and 72 % of clean duty at 30, 120 and 210 min. The concentration loop absorbs the first two steps, and for three hours the concentration stays within 0.41 kg Li/m^3 of setpoint while the duty command climbs. The duty command is the variable that carries the information. After the third step the product leaves the saleable range, below 11.5 kg Li/m^3, at 217.2 min, ten minutes before the duty reaches its limit at 227.3 min. The saturation flag therefore

Table 10.6 A pH electrode reading low. Every reported number improves. Values are instantaneous readings with the shipped noise seed, and the noise moves the lime flow by 6.9 L/min (one standard deviation).

t (min)	True pH	Measured	Lime (L/min)	Yield (%)
35	12.508	12.515	276.3	87.95
80	12.863	12.526	310.8	89.24
200	13.170	12.471	369.8	90.98
500	13.203	12.470	356.7	**91.26**

arrives too late to serve as the warning, and a plant that trends the manipulated variable against its clean value sees the loss of capacity hours earlier.

10.6.5 Bias on the pH Electrode

Scenario 10 puts a bias on the pH electrode, 0.35 low at forty minutes and 0.70 low at one hundred and sixty, on top of 0.04 of electrode noise from a fixed seed. The consequences are given in Table 10.6.

Table 10.6 records the effect of an electrode bias on the reported performance of the plant. The controller holds the reading at 12.50, so it adds lime until the true pH reaches 13.20, which is five times the free hydroxide the leach needs. Averaged over the first 40 min and over 300 min to 500 min, extraction rises from 95.8 to 99.1 % because the leach is in fact operating at a higher free hydroxide concentration. The yield rises from 87.8 to 91.2 % and production from 10.88 to 11.30 kg/min.

> ✦ DID YOU KNOW? **A Faulty Electrode That Looks Like an Improvement**
>
> From the simulator: at minute 500 of Table 10.6 the electrode shows pH 12.470, close to its setpoint, while the true pH is 13.203 and the lime runs at 356.7 L/min against 276.3 before the fault. The yield column climbs from 87.95 to 91.26 %, so the fault reads as a gain.

Every number on the daily report improves. The reagent consumption rises 24 %, from 280 to 348 L/min on the same averages (24.1 % to 25.1 % over eight other noise seeds), and it is the only symptom. The pH trend itself is flat, and a plant judged on yield alone records an improvement. Detecting the fault requires evidence from outside the loop, from a reagent consumption per tonne figure, a laboratory titration or a second electrode.

🕮 **Pause.** Try Exercise 10.3 before reading on.

10.6.6 Graded Run

The challenge starts from the cautious tunings with a single-loop kiln and applies a grade change to 4.6 % Li_2O at 20 min, a 7 % gas heating value drop at 70 min, an evaporator scaling step to 86 % of clean duty at 120 min and a grade recovery to 5.9 % at 170 min. The thermocouple, electrode and analyzer carry noise of 1.2 °C, 0.03 pH and 0.06 kg Li/m^3 from a fixed seed. The run is judged at 300 min on the largest deviation of each true controlled variable over the whole run, with the kiln within ±12 °C, the leach within ±0.15 pH, the product within ±1.0 kg Li/m^3, the kiln below the 1160 °C refractory limit and the product below the 15.5 kg Li/m^3 saturation line. Results with one setting changed at a time are given in Table 10.7.

Table 10.7 isolates each clause to one decision. The kiln loop's tuning owns the kiln clause, at 9.29 °C against 24.53 °C. The pH loop's tuning owns the pH clause, at 0.135 against 0.315, and the concentration loop's tuning owns the concentration clause, at 0.789 kg Li/m^3 against 1.942 kg Li/m^3. Influence runs only downstream, which is the one-way material coupling of Figure 10.1. The concentration loop leaves the pH clause unchanged to the third decimal, while the cautious pH loop moves the product deviation by 0.023 kg Li/m^3 and the cautious kiln loop moves the pH and product deviations by 0.006 and 0.037 kg Li/m^3.

Table 10.7 The graded run. Largest deviation from setpoint over the 300 min judged run, against bands of ±12 °C, ±0.15 pH and ±1.0 kg Li/m^3. Rows below Recommended change one setting of it.

Configuration	T_k (°C)	pH	C_e (kg Li/m^3)	Result
As loaded	24.66	0.358	1.980	fail
Recommended	**9.29**	**0.135**	**0.789**	pass
single-loop kiln	9.59	0.135	0.789	pass
cautious kiln loop	24.53	0.141	0.826	fail
cautious pH loop	9.29	0.315	0.766	fail
cautious concentration loop	9.29	0.135	1.942	fail

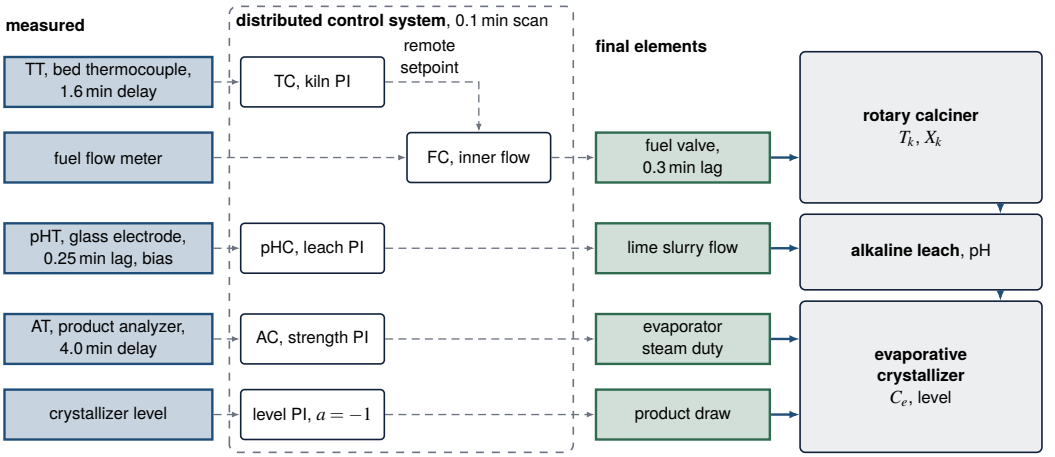


Figure 10.4 The loops of the simulated refinery drawn with their hardware. In cascade the kiln controller TC writes the setpoint of the fuel flow controller FC, which moves the valve; in single loop TC moves the valve directly. The three units downstream of the valve are joined in series by the material, with no storage between them, and every block runs on the same 0.1 min scan.

The cascade row is the one that qualifies Table 10.4. On this disturbance sequence the cascade changes the peak kiln excursion from 9.59 °C to 9.29 °C and nothing else, because the sequence contains a heating value change and no fuel supply disturbance, and because the outer loop at its recommended tuning absorbs what remains. The difference is smaller than the spread of the kiln deviation over eight other noise seeds, 8.36 °C to 10.29 °C in cascade. The cascade is worth a factor of 52 or more on integral error in Table 10.4 and three tenths of a degree here. Both numbers are correct and what differs is the disturbance.

The recommended configuration passes, and the pH clause is the one with the least margin. Its largest pH deviation, 0.135 at 190.5 min, comes twenty minutes after the grade recovery, and over eight other noise seeds it lies between 0.135 and 0.142, so the configuration passes on every seed tried with as little as 0.008 to spare. The product deviation peaks at 141 min, 21 min after the scaling step, because the evaporator's own response takes 45 min while the scaling step removes evaporation capacity in one move. As loaded, the run fails all three bands, the kiln by 12.7 °C after the heating value drop, the pH by 0.21 and the product by 0.98 kg Li/m^3.

10.7 Technology in Practice

Figure 10.4 draws the four loops of Section 10.5 and the inner fuel flow loop with their hardware.

What Measures It

Each unit has one measurement that its loop acts on. The kiln bed is read by a thermocouple. Its most common failure is to fail open, or burn out, and a furnace transmitter is set to read high when that happens, so the controller cuts the fuel rather than adding it [22]. The 1.6 min of transport delay on the bed reading in Section 10.3.2 stands in for the thermowell and the bed between the hot charge and the junction. The leach is read by a pH electrode, drawn in Figure 10.5.

> **TECHNOLOGY** **Sensor: The Glass pH Electrode**
>
> A thin bulb of glass that hydrogen ions can penetrate develops a voltage proportional to the difference between the pH of the process and that of a buffer sealed inside it, about 59 mV per pH unit at 25 °C and more when hot, so the transmitter needs the solution temperature. A reference electrode with a porous junction closes the circuit. The instrument is calibrated with two buffers, and a clean probe settles in each within about 30 s. The glass wears as it hydrates, so the slope falls with age, while stray potentials at the junction shift the zero, an offset of the kind scenario 10 applies. Sodium, potassium and lithium ions interfere at high pH [22].

The 0.25 min lag of the model's electrode stands for the response of the glass, and its optional bias for the shifted zero of an aged or fouled junction. The product strength comes from an analyzer that the model places 4.0 min downstream with a 1.2 min lag of its own.

What Moves It

The kiln's final element is the fuel valve, with a 0.3 min lag in the model, and in cascade a flow controller sits between it and the temperature controller.

> **TECHNOLOGY** **Final Element: The Fuel Valve Under a Flow Loop**
>
> The fuel enters through a control valve whose throttling changes with the pressure drop across it, more responsive near closed than near open. Placing a flow controller under the temperature controller confines that distortion to the flow loop, and a change in supply pressure is countered by the flow controller before the master sees it. The flow meter measures quantity, not energy: a heating value is computed from the composition of the gas. An air-to-open valve fails closed on loss of air, and the risks of the process, not habit, should decide the failure mode [22].

The lime slurry and the evaporator steam duty are the other two manipulated flows, and a draw on the product holds the crystallizer level. The model moves each of them through its own lag and clips it to its range, with no dead band or stiction.

Where the Controller Runs

Control loops in a plant are often nested or cascaded, with cycle times from milliseconds to minutes, and a distributed control system uses a supervisory loop to mediate a group of localized controllers [23]. In the app every controller, the inner flow loop included, acts on a 0.1 min scan against a kiln time constant of 28.4 min. On such a platform the four loops and the inner flow loop are PID function blocks of the regulatory layer, the cascade a remote setpoint from one block to the next.

> **IN PRACTICE** **Commissioning a Cascade**
>
> The secondary controller is tuned first, whenever a cascade is commissioned or optimized, because the master depends on it and it does not depend on the master. With the master in manual the secondary simply holds a constant setpoint. The secondary then has three modes: manual, automatic on a local setpoint, and cascade on the master's remote setpoint. A secondary left in automatic defeats the cascade, so it must stay in cascade mode for the strategy to work as designed [22].

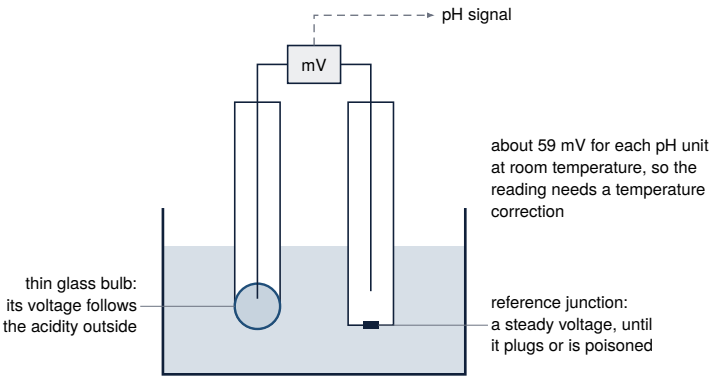


Figure 10.5 The glass pH electrode and its reference. Notice that the reading is a small voltage across the glass bulb, referred to a junction that can drift. A shift at the junction moves every reading by the same amount, which is the low bias of scenario 10 that the pH loop then hides.

What Is Modernizing It

The first development measures what the cascade cannot. A process gas chromatograph reads the composition of natural gas and a computer multiplies each part by its own heating value to give the heating value of the stream, which pipelines already use to price the gas [22]. Such a reading would show the heating value loss of Table 10.4 before the bed temperature does. The second is a new calciner. Fluidized heating of finely ground concentrate and microwave heating have been suggested to cut the excess heat of the rotary kiln [18]. The third is predictive control and estimation over the whole train, which Section 10.9 sets out: one calculation that holds the refractory limit and the saturation line as constraints, and an estimator that reconstructs the ore grade and the electrode bias [24].

10.8 Limitations

The calciner is one well-mixed zone with a single lumped conversion. A real rotary kiln has an axial temperature and conversion profile, a freeboard gas in counter-current, and a solids bed whose depth and residence time depend on rotation and slope, and integrated kiln simulators exist to capture those [15, 16]. Nothing here describes the condition of a kiln's hot end.

> **OPEN CHALLENGE** **The Energy of the Calciner**
>
> Calcination, acid digestion and purification are the stages that decide the energy use of a spodumene refinery, and the high energy consumption of the calcination step remains unsolved. A typical kiln is a fairly inefficient furnace fired on gas or coal, and its heat load has been put at 82 % above the thermodynamic need of the phase change, with the carbon dioxide that goes with it. Fluidized and microwave heating have been suggested [18]. Progress would be a calciner that reaches the same conversion of the ore on far less fuel, with the temperature control the chapter's kiln loop provides.

The phase transformation is one Arrhenius rate. The real conversion proceeds through an intermediate gamma phase whose extent depends on particle size, heating rate and impurities, and the reported activation energies span a wide range [1]. The application reproduces the saturating shape of Equation (10.2) and no particular kiln's numbers.

The leach is one tank with one lumped rate constant and no solids handling. A real circuit is several tanks with a filtration step, and a badly leached solid is the one that is difficult to filter. Extraction is first order in the manner used for other hydrometallurgical circuits [12] rather than shrinking-core. The reported extraction is on a concentration basis and therefore overstates the mass-basis extraction

by 0.5 percentage points at the design reagent rate, while the reported yield is computed from the mass balance and carries no such offset.

Impurity removal is entirely absent. On a real refinery the removal of aluminum, iron, calcium and magnesium between the leach and the crystallization is a substantial plant in its own right, and the product specification is dominated by those impurities rather than by concentration [3]. The product quality here is one number and should be read as a proxy for a duty rather than as a specification.

The crystallizer produces a concentrated liquor rather than crystals. There is no supersaturation, no nucleation and no crystal size distribution, so the saturation line is a limit rather than a mechanism, and the scaling disturbance is a step in evaporation per unit duty rather than a fouling model.

10.9 Discussion

Three results generalize beyond this refinery.

The first is that the value of a cascade depends on which disturbance it is asked to reject, and the two halves of the classical justification should be quoted separately. Table 10.4 shows the same inner loop worth a factor of 52 to 93 on integral error for a header pressure loss, 1.10 for a setpoint change and 1.00 for a heating value loss, and Section 10.6.6 shows it worth three tenths of a degree on a disturbance sequence containing neither. An inner loop rejects what enters inside its own measurement and nothing else. The corollary is that specifying a cascade requires naming the disturbances a plant actually faces, because the same structure is worth a factor of fifty or more against one of them and nothing against another.

The second is that a plant's settling time is not any loop's settling time. Table 10.5 has three loops with excursions of 1.26 °C, 0.163 pH and 0.21 kg Li/m^3, all returning to setpoint, on a plant that takes six hours to reach a new steady state 27 % below the old one. A regulatory scorecard records that day as compliant. Plantwide control exists because the scorecard is insufficient, and because the remedy is a change to the production plan that no local loop is permitted to make [5, 9, 19].

The third concerns instrument validation. Table 10.6 has a drifting electrode improving every figure the plant is judged on, because the controller compensating for it happens to push the process in a direction that helps. The only evidence is the reagent consumption, which is reported outside the control system and on a longer time scale. Instrument validation is therefore a control activity rather than a maintenance one [7], and reagent consumption is one of the largest operating costs a hydrometallurgical plant carries [10].

The documented model also supports control methods beyond the PID and cascade loops the application implements. The eleven differential states, the actuator limits and the two hard constraints match the problem form solved by nonlinear model predictive control, which would coordinate the fuel valve, the lime flow and the evaporator duty in one calculation, hold the 1160 °C refractory limit and the 15.5 kg Li/m^3 saturation line as constraints rather than as alarms, and run an economic objective on the gas and reagent bills against the lithium recovered, which is precisely the trade-off the pH setpoint sweep of Section 10.3.3 exposes and no local loop can resolve [24]. Moving horizon estimation over the same equations would reconstruct the ore grade and the evaporator surface condition from the kiln temperature, pH and product concentration histories, converting the two unmeasured disturbances of Table 10.5 and the scaling event into estimated quantities available to the controller. The same estimator would supply a continuous soft sensor for the spodumene conversion, which is measured nowhere and which falls by 0.012 during the throughput increase with every controlled variable on setpoint, and estimating the electrode bias of Table 10.6 as a parameter would surface that fault from inside the control system rather than from the reagent report. The Gekko Optimization Suite implements these methods in Python from model statements close to (10.1) through (10.8) [25].

10.10 Conclusion

A lithium hydroxide refinery puts three units in series with no storage between them, so one fuel valve reaches the spodumene conversion in 14.0 min, the kiln temperature it is controlling in 29.3 min and the product concentration two units downstream in 112.8 min. Each unit is identified as a first-order plus dead-time plant from one step test, and the three identified models set the tunings through the SIMC rules. A cascade on the kiln reduces the integral error of a header pressure loss by a factor of 52 to 93, a setpoint change by 1.10, a heating value loss by 1.00 and a graded disturbance sequence by three tenths of a degree of peak, so the disturbance rather than the structure decides what the cascade is worth. A single grade change at the front takes six hours to walk through the train, moves no controlled variable by more than a sixth of a pH unit, and removes 27 % of the plant output. An electrode reading 0.70 low raises the extraction, the yield and the production while raising the reagent consumption by 24 %, which is a failure that a plant measuring only its controlled variables is structurally unable to see. The app lets a reader run each of these results on a train whose constraint moves between units.

KEY POINTS

- With no storage between the units, one fuel valve reaches the conversion in 14.0 min, the kiln temperature in 29.3 min and the product in 112.8 min.
- A fuel flow cascade cuts the integral error of a header pressure loss by a factor of 52 to 93 and of a heating value loss not at all.
- A grade change to 4.0 % Li_2O moves no controlled variable by more than a sixth of a pH unit and removes 27 % of the output.
- An electrode reading 0.70 low raises the yield while the reagent rises 24 %.

Where else this applies. Chapter 4 and Volume 3, Chapter 2 sort a cascade's disturbances the same way, and Volume 6, Chapter 7 poses the plantwide question for a reactor and a column.

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10.11 Exercises

10.1 ⌕ Concept check · 2 min

The kiln runs with the fuel flow cascade at the recommended tuning. Which fuel-side disturbance does the cascade remove almost entirely, and why?

(a) A loss of gas heating value, because the inner loop acts ten times a minute

(b) A loss of header pressure, because the inner loop measures the flow it changes

(c) Both losses equally, because the cascade takes the valve lag out of the outer loop

(d) Neither loss, because the outer loop still sets the fuel flow setpoint

10.2 ⌕ Concept check · 1 min

The concentrate grade falls from 5.5 to 4.0 % Li_2O with every loop in Auto. How do the kiln temperature and the lime flow first respond?

(a) The kiln cools and the lime flow rises

(b) The kiln cools and the lime flow falls

(c) The kiln heats and the lime flow falls

(d) The kiln heats and the lime flow rises

10.3 Concept check · 2 min

The pH electrode starts to read 0.70 low while the pH loop is in Auto at a setpoint of 12.50. Which describes the plant some hours later?

(a) The pH trend dips by 0.70 and the lime flow falls to restore it

(b) The true pH falls, so the extraction and the yield fall with it

(c) The true pH rises and the overdosed leach extracts less lithium

(d) The true pH rises, and extraction, yield and production rise with the lime

10.4 Theory and calculation · 6 min

The calciner conversion follows Equations (10.2) and (10.3) with $k_{\text{ref}} = 0.475/\text{min}$ at 1100 °C, $E/R = 50\,500\,\text{K}$ and a solids hold-up $M_s = 20\,000\,\text{kg}$.

(a) Compute k at 1090 °C to three significant figures and the steady conversion at a feed of 500 kg/min to four decimals. *Checkpoint:* 0.363/min; 0.9355

(b) The feed rises to 600 kg/min and the temperature loop holds 1090 °C. Compute the new residence time and steady conversion. *Checkpoint:* 33.3 min; 0.9236

(c) Linearize Equation (10.2) at fixed temperature about the design point and give the time constant of the conversion, to one decimal. Use it to explain why the conversion reaches 63 % of its change before the bed temperature does. *Checkpoint:* 2.6 min

10.5 Theory and calculation · 8 min

Use the identified models of Table 10.1 and the SIMC rules $K_c = \tau_p/(K_p(\tau_c + \theta_p))$ and $\tau_I = \min(\tau_p, 4(\tau_c + \theta_p))$.

(a) Tune the kiln loop with $\tau_c = \theta_p$. Give K_c to two decimals in % per °C and τ_I to one decimal. *Checkpoint:* 0.41; 14.9 min

(b) Tune the product strength loop with $\tau_c = 7.5\,\text{min}$. Give K_c to one decimal and τ_I to one decimal, and compare them with the recommended 13 and 35 min of Table 10.3. *Checkpoint:* 13.0; 45.1 min

(c) Tune the pH loop with $\tau_c = \theta_p = 0.3\,\text{min}$ (the bound of the table) and give K_c to the nearest 10 L/min per pH. What lime move would a pH error of 0.1 demand at once, and why is this τ_c unusable? *Checkpoint:* about 5770; about 577 L/min

10.6 Theory and calculation · 6 min

At design the calciner burns 26.53 kg/min of fuel with $\eta_f = 0.65$ and $\Delta H_f = 50\,000\,\text{kJ/kg}$. The feed is 500 kg/min at 25 °C with $c_{p,s} = 1.05\,\text{kJ/(kg K)}$ and 3 % moisture, $UA_k = 220\,\text{kJ/(min K)}$ with ambient at 25 °C, $T_k = 1090\,°\text{C}$, $X_k = 0.9355$ and $\Delta H_c = 100\,\text{kJ/kg}$ on a spodumene fraction $\phi = 5.5/8.03$.

(a) Compute the burner heat and the four sinks of Equation (10.1) in MJ/min, to one decimal, and check that they balance. *Checkpoint:* 862.2 in; 559.1, 234.3, 36.8, 32.0 out

(b) The grade falls to 4.0 % Li_2O ($\phi = 4.0/8.03$). At the same conversion, by how much does the reaction sink fall, and how far would the bed rise at fixed fuel if the sensible heat and the shell loss absorbed the difference? Give both to one decimal. *Checkpoint:* 8.7 MJ/min; 11.7 °C

(c) A run in Manual settles about 11 °C hot. Why is the true rise smaller than the estimate of (b)? *Checkpoint:* the hotter bed converts more

10.7 App challenge · 8 min

Open scenario 6, "The ore grade falls (5.5 → 4.0% Li_2O)", at the default seed, with every setting as loaded. Run it at 50 min per second to 400 min (the clock reads 6.67 h), then export the CSV. Its time column is Time (h), one row every 0.25 min.

(a) From the columns `kiln_C`, `ph` and `conc_kgm3`, give the largest deviation of each controlled variable from its setpoint after the 20 min grade change, with its sign and the time after the change at which it occurs (deviations within 0.01 in their own units, times within 0.5 min). *Checkpoint:* $+1.26\,^{\circ}\mathrm{C}$ at 5.3 min; $+0.163$ pH at 22.3 min; $-0.21\,\mathrm{kg\,Li/m^3}$ at 74.3 min

(b) Read `production_kgmin` and `li_feed_kgmin` at 19 min and at 400 min (within 0.005) and give the fall of each as a percentage of its 19 min value, to 0.1. *Checkpoint:* 10.870 to 7.935 kg/min, 27.0 %; 12.391 to 9.012 kg/min, 27.3 %

(c) Explain the order in which the three loops are disturbed, and why no retuning would recover the lost production. *Checkpoint:* kiln, leach, evaporator; the feed carries less lithium

10.8 App challenge · 8 min

Open scenario 4, whose name begins "Cascade on the kiln", at the default seed, and run at 20 min per second. The header pressure falls 25 % at 20 min and recovers at 90 min, and the heating value falls 10 % at 130 min.

(a) With Kiln architecture on Single loop as loaded, run to 240 min and export the CSV. From `kiln_C`, give the kiln deviation from 1090 °C at its lowest between 20 and 90 min, its highest between 90 and 130 min, its value at 130 min and its lowest after 130 min, each within 0.1 °C. *Checkpoint:* -37.2, $+43.1$, $+2.9$, -11.7

(b) Press Reset, select Cascade before pressing Run, run to 240 min and read the same four values. *Checkpoint:* -1.2, $+1.0$, 0.0, -13.0

(c) The heating value drop looks smaller under the single loop. Is the single loop better against it? Use the values at 130 min to explain. *Checkpoint:* no; it starts 2.9 °C hot

10.9 App challenge · 9 min

Open the challenge, "Challenge · take the plant through a grade change", at its default seed, and run each part at 20 min per second until it is judged at 300 min. Read the verdict line in the progress panel (kiln, pH and product deviations as it prints them).

(a) Run it as loaded. *Checkpoint:* fail: 24.7 °C, 0.36, 1.98 kg Li/m^3

(b) Reload it with its reload button, press Reveal suggested tuning and run. *Checkpoint:* pass: 9.3 °C, 0.13, 0.79 kg Li/m^3

(c) Reload, press Reveal suggested tuning, then set pH loop gain to 40 L/min per pH and pH loop integral time to 60 min before Run. Run, and explain which clauses moved and why the others did not. *Checkpoint:* fail: 9.3 °C, 0.31, 0.77 kg Li/m^3

10.10 Application area · 4 min

Production asks for 20 % more concentrate. Scenario 8 shows every loop back on setpoint while the yield falls from 87.7 to 84.4 %. As the process engineer, explain where the lithium is lost, which unit limits a further increase, and what you would measure or estimate before agreeing to the push.

10.11 Application area · 3 min

In scenario 9 the evaporator scales in three steps. An operator watches the product strength trend and the duty saturation flag. Why is that not enough, and what should the plant watch instead?

10.12 Review the agent · 5 min

An AI assistant reviews the pH loop and writes: "The process gain is 0.0159 pH per L/min at the design point but only 0.0034 at pH 13.4. A biased electrode can move the leach there, where the loop tuned at design will be 4.7 times too aggressive and oscillate. Retune at pH 13.4 so that the loop is safe everywhere." Find the flaw and say how you would check it in the app.