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CHAPTER 9

Two Time Scales and a Late Laboratory in Rare Earth Solvent Extraction

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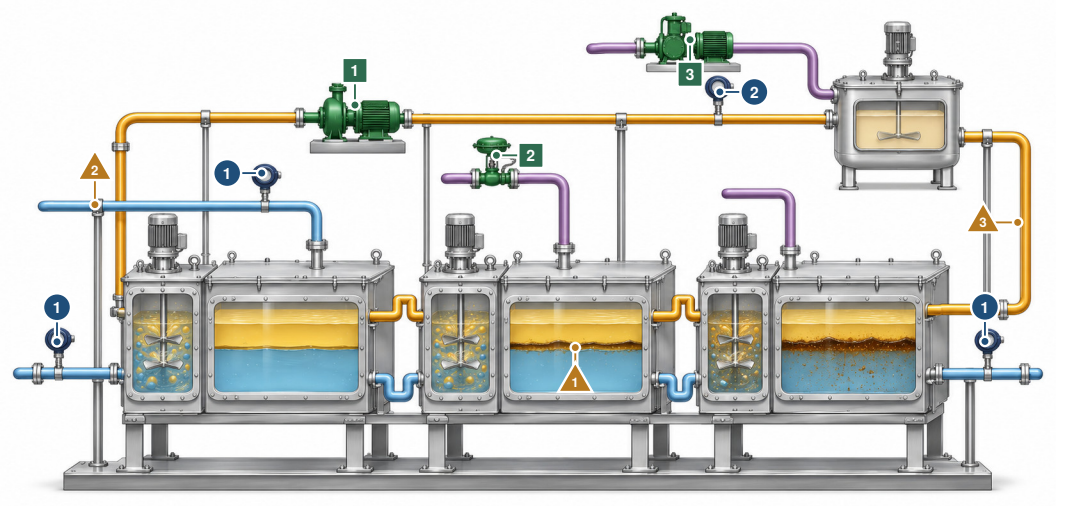
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CHAPTER 9

Two Time Scales and a Late Laboratory in Rare Earth Solvent Extraction



The controller moves 1 organic flow, held in ratio to the feed 2 scrub acid, held in ratio to the feed 3 caustic soda dose to the saponifier

The controller reads 1 feed, raffinate and product samples, analyzed every four hours 2 saponifier pH

What disturbs it 1 crud in one scrub stage 2 richer or leaner feed 3 extractant capacity loss

The organic picks up neodymium a little more readily than praseodymium in every unit, so the purity is made by the number of stages, and the days of metal they hold make the product slow to answer a change of feed. Illustration, not to scale.

IN PLAIN TERMS **Separating Two Nearly Identical Metals**

The main idea. Two rare metals used in strong magnets, neodymium and praseodymium, behave almost alike. A plant parts them in a row of seventy small tanks. In each tank an oily liquid takes up a little more neodymium than the watery liquid it meets.

The challenge. The tanks hold days of metal, so a change in what is fed reaches the product only after a day. It then keeps drifting for weeks. The only check on purity is a lab test every four hours that comes back late.

Why it matters. A plant that waits for that late test has lost days, so the flows must follow what is fed.

THREE WAYS TO READ THIS CHAPTER

Explore	12 min	The first page, the fact box of the Introduction, the history box of Section 9.2, the try-it box of Section 9.4, the try-it and fact boxes of Section 9.6, and the key points
Study	50 min	The whole chapter with its pause points and the twelve exercises
Practice	15 min	The first page, Section 9.6, Section 9.7, Section 9.8 and the open challenge after it

The control question A seventy-stage neodymium and praseodymium cascade answers a change of scrub acid in about a day and a change of feed grade in weeks, and its only composition measurement is a laboratory analysis every four hours, reported an hour late. Which ratios, pairing, feedforward and loop speeds hold the product's purity and recovery, and where does the cascade's own capacity stop every controller?

Learning objectives

- Compute the relative gain of purity and recovery on the scrub and organic ratios, and show how it changes when the scrub ratio is keyed to the organic instead of the feed.
- Estimate the time scale of a feed-grade change from the inventory over the imbalance, and contrast it with the reflux response to a scrub change.
- Derive the material-balance feedforward from the feed analysis and say what the purity loop must still correct.
- Tune a product loop that acts once per laboratory report to the plant's day, and name its action in the convention of Chapter 1.
- Explain why 99.9 % purity at 97 % recovery is a capacity limit and not a stage limit.

Prerequisites Sections 1.4.4 (Kremser, Fenske, fast and slow modes), 1.4.5 (sampled assays), 1.4.6 (ratio and inferential control), 1.6.2 (velocity form and action) and 1.6.4 (scenarios). Section 1.6.3 is needed only for the predictive supervisor. Chapter 7 meets a late assay first.

The app https://apmonitor.com/apps/rare-earth-sx/

See also Volume 6, Chapter 3 for two quality loops on a sampled gauge with a negative relative gain on one pairing, and Volume 6, Chapter 2 for a pairing decided before any loop is tuned. Volume 5, Chapter 3 for composition control through a slow analyzer. Volume 3, Chapter 6 for a feedforward from the raw feed with the late product measurement as a slow trim.

9.1 Introduction

A 99.67 % neodymium product leaves the strip section of the simulated cascade at 74.8 kg/h. It is made from 2 m^3/h of a rare earth chloride solution that is 75 % neodymium and 25 % praseodymium, by 50 m^3/h of an organic phase that meets the aqueous in seventy mixer-settlers. In any one stage the organic carries neodymium in preference to praseodymium by a factor of 1.5, so a single contact of the feed would give an organic only about 82 % neodymium. The purity comes from the stage count, and the stage count makes the plant slow. The settlers and mixers hold 76.0 kmol of rare earth, 108.6 hours of feed.

The hard magnets that electric motors rely on include rare earth intermetallics, and their raw materials are expected to become a bottleneck that recycling is asked to relieve [1]. The separation of adjacent lanthanides is done industrially by solvent extraction, with up to hundreds of mixers and settlers assembled in series [2]. A dynamic equilibrium-stage model of a praseodymium and neodymium flowsheet predicted the steady profiles of a laboratory cascade well and its transients less well [3]. Control of industrial rare earth cascades has been posed as the control of the distribution profile of a component content along the cascade and studied with identified delays [4, 5], and dynamic models of mixer-settler units have been written for copper as well [6]. The control problem underneath has the shape of two-point composition control of a distillation column, where the choice of the configuration, which flows are manipulated and in what ratio, decides the interaction the controllers meet [7] and the relative gain array is the standard screen for it [8]. Volume 5, Chapter 3 closes a column's composition loop on a slow online analyzer, the same difficulty with minutes in place of hours.

DID YOU KNOW? Rare Earths Mined in 2024

In 2024 the world mined an estimated 390 000 tons of rare earths, counted as oxide, 270 000 of them in China. Neodymium oxide is priced at a purity of at least 99.5 %, the specification of this chapter, and averaged $56 per kilogram that year [9].

The simulator takes aim at two beliefs. The first is that a plant has one speed. Here a step in the scrub acid moves the purity 63 % of its way in 25.3 h at fixed flows, but a feed-grade step changes the material balance itself, and the purity of case 4 is still falling five days later toward a steady state of 90.9 %. The second is that the relative gain belongs to the plant. Keyed to the feed, the scrub ratio gives purity a relative gain of 3.92 on it and the crossed pairing −2.92, and keyed to the organic the same cascade gives 0.60 with both pairings positive. The same two lessons, a fast reflux mode beside a slow inventory mode and a pairing that depends on how each ratio is defined, apply to two-product distillation [7] and to other counter-current separations whose product is a small difference of two large flows.

These pieces seldom appear side by side. The industrial control studies work on plant data and identified models [4, 5], the laboratory model was checked on laboratory mixer-settlers [3], and the distillation results come from a different unit operation. This chapter tests what ratio stations, sampled proportional-integral (PI) loops, a pairing choice and a feedforward from the feed analysis can hold through feed changes, an extractant loss and crud, and where the cascade's own capacity stops every controller, using a 40/20/10 praseodymium and neodymium cascade simulated in a browser with its stage profile, laboratory samples and true products logged side by side. The control of rare earth cascades on monitoring stages and the dependence of the relative gain on the control configuration are both documented [4, 7], and the measurements of this chapter are properties of one teaching plant. The simulator is a two-phase stage stack with 2243 integrated quantities whose metal balances close to 10^{-11}, a steady-state solver that the dynamic stack reproduces to five decimals, and controllers confined to the pH, the flows and the delayed analyses. Section 9.2 follows the domain from ion exchange to counter-current cascades. Section 9.3 sets out the stage chemistry, the mixers, settlers and saponifier, the products and the measurements. Section 9.4 measures what each stage adds, the two time scales, the interaction of the two ratios and the capacity limit. Section 9.5 describes the ratio stations, the two sampled product loops, the feedforward and the optional predictive supervisor. Section 9.6 measures the ten cases, nine of which carry rules that grade them, with the challenge graded on its own verdict. Section 9.7 names the hardware of a real cascade, Section 9.8 states the limitations and Section 9.9 concludes.

9.2 From Ion Exchange to Counter-Current Cascades

The way from ion exchange to solvent extraction is told in the history box below, and the rest of this section follows the cascade theory, the extractant and the models.

HISTORY From Ion Exchange to Solvent Extraction (1947 to 1957)

The lanthanides were first isolated and purified by fractional crystallization, and ion exchange later replaced it as the way to make usable quantities of pure rare earths [10]. A paper of 1947 opened a series on the separation of rare earths by ion exchange with cerium and yttrium [11]. In the same years the solvent extraction of the trivalent rare earths was studied, and their order and degree of fractionation were reported in 1953 [12]. In 1957 the lanthanides were fractionally extracted as their dialkyl orthophosphates [13], the family of acidic organophosphorus cation exchangers to which the extractant of this chapter belongs. As demand grew, the low throughput and rising cost of ion exchange moved industrial separation to solvent extraction, which makes pure rare earths in large quantities, continuously and economically [10].

The design of a counter-current cascade for a pair with a small separation factor became a theory of its own, and a theory of counter-current extraction and its applications in the rare earth industry

Table 9.1 Variables, Their Roles and Their Values at the Design Point

Symbol	Role	Design	Range
x	CV, Nd purity, strip liquor	99.671 %	SP 99.0–99.95
R	CV, Nd recovery	98.75 %	SP 90–99.9
s	CV, saponified share	16.7 %	SP 10–18
W/F	MV, scrub per feed	1.173	0.8–1.6
O/A	MV, organic per feed	25	15–28
n_{OH}	MV, NaOH per organic (M)	0.167	0–0.18
S/O	set, strip per organic	0.012	0.010–0.03
z_{Nd}	DV, feed Nd share of the metals	75 %	60–90
F, c_{RE}	DV, feed flow, concentration	2 m^3/h, 0.35 M	1.5–2.5, 0.25–0.45
ℓ	DV, extractant capacity loss	0	0–30 %
κ	DV, crud in stage 55	0	0–90 %
$N_{k,n}$	states, mixer holdups (kmol)	profile	70 stages
$a_{k,n,j}$, $o_{k,n,j}$	states, settler cells (M)	profile	4 cells
$o_{k,sap}$	states, saponifier organic (M)	—	—
pH_n	output, measured pH of stage n	1.26 to −0.60	—
P	output, Nd product (kg/h)	74.8	—

was published in English in 1985 [14]. The simulator uses only the material balance any such cascade obeys, that the product draw is the extraction amount less the reflux returned by the scrub section. The phosphonic-acid extractant of the model, 2-ethylhexyl phosphonic acid mono-2-ethylhexyl ester, is the public name of a type sold as PC88A or P507, and it is described as the extractant of industrial rare earth separation facilities [3]. Reviews of rare earth solvent extraction list it among the commercially used extractants with D2EHPA [2], and separation factors of praseodymium and neodymium on it have been measured from process loadings down to trace concentrations [10, 15].

Process models followed the plants. A dynamic model of a praseodymium and neodymium flowsheet coupled equilibrium data with an acid balance and was checked against laboratory mixer-settlers [3]. Control of industrial cascades has been formulated as control of the profile of a component content along the cascade [4], and the identification of the process's delays for a neodymium content model has become a problem in its own right [5]. None of the sources consulted here describes the purity and recovery loops of a particular commercial plant, so the simulator's loop structure is a teaching design built on the column analogy rather than an account of how any plant is run.

9.3 The Cascade as Modeled

The cascade is a generic teaching design, drawn in Figure 9.1(a) in the arrangement of the application's Settlers view. Stages are numbered 1 at the raffinate end to 70 at the strip end, with 40 extraction, 20 scrub and 10 strip stages. The organic enters stage 1 from the saponifier and flows to stage 70, returning to the saponifier. The feed enters the mixer of stage 40 together with the scrub liquor leaving stage 41, and the aqueous flows down the extraction section to leave stage 1 as the raffinate. Scrub acid enters stage 60 and flows down the scrub section, and strip acid enters stage 70 and leaves stage 61 as the strip liquor, the neodymium product. The engine integrates in hours, flows are in m^3/h and concentrations in mol/L (kmol/m^3).

Table 9.1 gives every variable its role. The state is a holdup of praseodymium, neodymium, sodium and chloride in each mixer, four settler cells per phase per stage (four species in the aqueous, three in the organic) and the saponifier's organic, 2243 quantities in all, plus ten running totals of the flows and costs that cross the boundary.

9.3.1 Cation Exchange on a Dimeric Extractant

The extractant is held as dimer sites E, half its monomer concentration C_{HA}, and a trivalent metal takes three dimers and releases three hydrogen ions, while sodium takes one and releases one, as

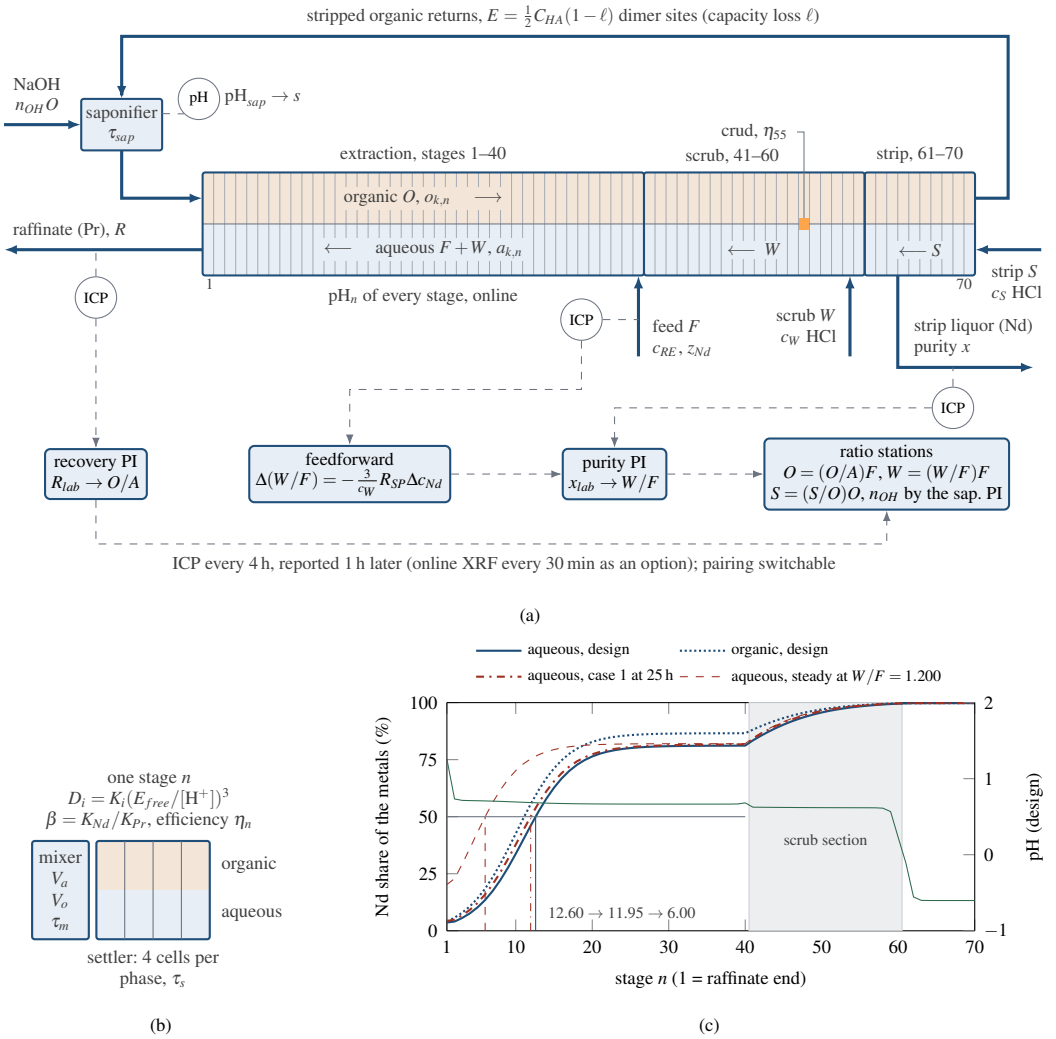


Figure 9.1 The 40/20/10 cascade as modeled. (a) The mixer-settler bank after the application's Settlers view, with the organic O from stage 1 to 70 and back through the saponifier, where NaOH at $n_{OH}O$ converts part of the extractant to its sodium form, the aqueous counter-current, the feed F at stage 40, scrub acid W at stage 60 and strip acid S at stage 70, the inductively coupled plasma (ICP) sample points on the feed, raffinate and strip liquor, the crud at stage 55, and the two sampled product loops with the feedforward (signals dashed). (b) One stage enlarged. (c) The neodymium share of each phase along the cascade at the design steady state (aqueous solid, organic dotted), the aqueous profile 24 hours after the scrub ratio of case 1 is raised from 1.173 to 1.200 (dash-dotted), and the steady state at 1.200 (dashed), with the stage where the aqueous is half neodymium (12.60, 11.95, 6.00, on the thin horizontal line at half) and the design pH (thin line, right axis). The scrub section is shaded. Process model only. Not to scale.

shown in Equation (9.1).

$$
\begin{aligned}
D_M &= \frac{o_M}{a_M} = K_M r^3, \qquad D_{Na} = K_{Na} r, \qquad r = \frac{E_{free}}{[\mathrm{H}^+]} \\
E_{free} &= E - 3\sum_M o_M - o_{Na}, \qquad E = \tfrac{1}{2}C_{HA}(1-\ell) \\
[\mathrm{H}^+] &= [\mathrm{Cl}^-] - [\mathrm{Na}^+] - 3\sum_M a_M
\end{aligned}
\tag{9.1}
$$

where M is Pr or Nd, a and o are aqueous and organic concentrations, chloride stays in the aqueous and the hydrogen ion follows from the aqueous charge balance. The separation factor $\beta = D_{Nd}/D_{Pr} = K_{Nd}/K_{Pr}$ is therefore the same at every stage, every acidity and every loading, and the organic holds at most $E/3 = 0.167\,\mathrm{M}$ of rare earth. Given a mixer's totals in its two phase volumes, the function $g(r) = E_{free}(r) - r[\mathrm{H}^+](r)$ is strictly decreasing, so the equilibrium is one root, found by a bracketed Newton iteration to a relative tolerance of 10^{-13} from the stage's last root. At the feed stage of the design point the root gives $[\mathrm{H}^+] = 0.208\,\mathrm{M}$ (pH 0.68), $E_{free} = 0.339\,\mathrm{M}$ and $D_{Nd} = 0.0906$ against $D_{Pr} = 0.0604$, and Equation (9.1) evaluated by hand from those concentrations agrees with the solver to six digits.

The neodymium constant comes from a laboratory measurement. A single-metal contact of 12.5 g/L neodymium with a 1 M extractant extracted 38 % at an equilibrium acidity of 0.13 M, a distribution ratio of 0.619 [10], and with the free dimer $(1 - 6 \times 0.0329)/2 = 0.40\,\mathrm{M}$ the expression $K_{Nd} = D[\mathrm{H}^+]^3/E_{free}^3$ gives 0.0212, used as 0.021. The same table's rows at 0.12 to 0.18 M acid give 0.019 to 0.044, and the constant falls as the acid rises, a dependence the model leaves out. The value $\beta = 1.5$ sits within the measured range. The thesis cites 1.5 to 1.6 from the literature for the pair, reports 1.29 to 1.59 in batch contacts across acidities and loadings (1.86 at 0.3 M acid) and 1.46 to 1.79 stage by stage in an 18-stage mixer-settler test [10], and contacts at trace concentrations in hydrochloric acid reached 1.75 to 2.05 [15].

9.3.2 Mixers, Settlers and the Saponifier

Each mixer holds the two phases at equilibrium in fixed volumes V_a and V_o, and a stage efficiency η acts as a bypass, as shown in Equation (9.2).

$$\begin{aligned} \frac{\mathrm{d}N_{k,n}}{\mathrm{d}t} &= \eta_n \left[A_n (a_{k,in} - a_k^*) + O(o_{k,in} - o_k^*) \right] \\ a_{k,out} &= \eta_n a_k^* + (1 - \eta_n)\, a_{k,in} \\ o_{k,out} &= \eta_n o_k^* + (1 - \eta_n)\, o_{k,in} \\ \frac{V_s}{4} \frac{\mathrm{d}c_j}{\mathrm{d}t} &= Q\,(c_{j-1} - c_j), \quad j = 1, \ldots, 4, \quad c_0 = c_{out} \end{aligned} \tag{9.2}$$

where $N_{k,n} = V_a a_k^* + V_o o_k^*$ is the mixer's holdup of species k, a^* and o^* its equilibrium, A_n the aqueous flow of the stage's section ($F + W$ in extraction, W in scrub, S in strip), and the settler of each phase is four well-mixed cells in series at constant volume V_s with $Q = A_n$ or O. Stage n takes its organic from the last organic cell of stage $n-1$ and its aqueous from the last aqueous cell of stage $n+1$, except that stage 70 takes the strip acid, stage 60 the scrub acid and stage 40 the mixture $(F a_F + W a_{41})/(F + W)$, with the feed composition $a_F = \left(c_{RE}(1 - z_{Nd}),\, c_{RE} z_{Nd},\, 0,\, 3c_{RE} + h_F\right)$ for Pr, Nd, Na and Cl. Volumes are sized at the design flows, $V_a = A_n \tau_m$, $V_o = O \tau_m$ and $V_s = Q \tau_s$. The crud event lowers one stage's efficiency, $\eta_{55} = \eta\,(1 - \kappa)$. The saponifier mixes the returning organic with the sodium that the NaOH loads, as shown in Equation (9.3).

$$\begin{aligned} V_{sap} \frac{\mathrm{d}o_{k,sap}}{\mathrm{d}t} &= O\,(o_{k,70} - o_{k,sap}) + \delta_{k,Na}\, \dot{n}_{NaOH} \\ \dot{n}_{NaOH} &= \min\left(n_{OH} O,\; 20 V_{sap} E_{free,sap}\right) \end{aligned} \tag{9.3}$$

where the cap keeps the dose within the free sites of about three minutes of organic, and the saponified share is $s = o_{Na,sap}/(2E)$, the fraction of the extractant monomer in the sodium form. The sodium form exchanges for rare earth in the first extraction stages without releasing acid, so the NaOH dose fixes how much rare earth the organic can take up.

9.3.3 Products, the Reagent Bill and the Steady State

The products are read at the last aqueous cell of stage 61 and of stage 1, as shown in Equation (9.4).

$$x = \frac{a_{Nd,61}}{a_{Nd,61} + a_{Pr,61}}, \qquad R = 1 - \frac{(F+W)\,a_{Nd,1}}{F\,a_{F,Nd}}$$
$$\frac{\$}{\mathrm{kg}} = \frac{p_N \dot{m}_{NaOH} + p_H \dot{m}_{HCl} + p_o e_o (F+W+S)}{S\,a_{Nd,61}\,M_{Nd}} \tag{9.4}$$

where $\dot{m}_{HCl} = M_{HCl}(W c_W + S c_S)$, e_o is the organic entrained per cubic meter of aqueous leaving and the prices p are teaching values. Recovery is measured from the raffinate's loss rather than from the product's neodymium, because the product-based yield counts the inventory the cascade gives up. In case 4 it reads up to 110.0 % at hour 26 while the raffinate-based recovery stays between 98.60 and 99.37 %. At the design point the bill is \$150/h of NaOH (8.35 kmol/h), \$69/h of hydrochloric acid and \$0.30/h of organic, \$2.93/kg of neodymium.

The same stage equations with the holdups removed give the steady state the cases start from and the Map view draws. Each stage's outlets are a function of its two inlets, and the residual $x - \Phi(x)$ over all stages is solved by pseudo-transient continuation with a block-tridiagonal Newton step whose Jacobian blocks are finite differences, with the organic recycle lagged one iteration. The design point converges to a residual of 1.4×10^{-13} in 49 iterations, and the steady balances close to 2.5×10^{-11} of the feed for praseodymium, 8.1×10^{-12} for neodymium and 2.1×10^{-13} kmol/h for the acid. The dynamic stack integrated for 6000 h after a scrub step and after a grade step reaches the solver's answers to five decimals, 99.69096 and 97.4675 % for the first and 90.89741 and 99.9998 % for the second.

9.3.4 Measurements

The controllers see a packet of measurements and nothing else, and a grep-level test in the application rejects controller code that reads a truth field. The pH of every stage's aqueous outlet is read every 5 min with a noise of 0.02, and the flows are metered exactly. The saponifier's pH carries 0.005 of noise and is converted to the saponified share through a declared titration curve, as shown in Equation (9.5).

$$\mathrm{pH}_{sap} = 6.5 + \log_{10}\frac{f}{1-f}, \quad f = \frac{o_{Na,sap}}{E}, \quad s_m = \frac{f}{2}$$
$$y_{lab}(t) = y(t_i) + \varepsilon_i, \qquad t_i + \theta \le t < t_{i+1} + \theta \tag{9.5}$$

where $t_i = i T_{lab}$ are the sampling instants, $T_{lab} = 4$ h and $\theta = 1$ h. The ICP analysis of the feed, raffinate and strip liquor reports the strip liquor's purity with 0.01 points of noise, the recovery computed from the sampled raffinate and feed with the metered flows (0.03 points), the feed's neodymium share and its neodymium concentration (0.5 %), each held until the next report. An online X-ray fluorescence option samples every 30 min, reports 6 min later and carries five times the ICP's purity noise. Section 1.4.5 gives the age of such a report. Every sample is kept with its true value at the drawing instant, and the challenge is graded on those true values.

9.3.5 Parameters and Numerics

Table 9.2 lists the parameters with their sources. The engine advances the stack by explicit Euler at $\Delta t = 20$ s, with concentrations clipped at zero and each stage's equilibrium root warm-started, and the ratio stations and the saponification loop act every 5 min. Halving the step moves the true purity and recovery of case 4 at hour 96 by 0.0012 and 0.0050 points. With one or four settler cells the steady purity agrees to eight digits, and the purity's 63 % time after a scrub step stays between 25.26 and 25.27 h at two, four and eight cells and at a 10 s step. Over 24 h with the capacity loss the sodium closes to 4.5×10^{-15} of the dose. With $\beta = 1$ both products carry the feed's 75 % to six decimals. Headless, the 120-hour challenge takes about 1 s (47 to 53 µs per step on two runs), and in the browser it plays in about 40 s at the top speed of 3 h per second.

Table 9.2 Parameters. S: From a Source. T: Teaching Value. A: Adjustable in the Parameters Panel or a Control

Symbol	Meaning	Value	
β	separation factor Nd/Pr	1.5	S, A
K_{Nd}	Nd constant of Eq. (9.1)	0.021	S, A
K_{Na}	sodium exchange constant	0.001	T
C_{HA}	extractant, as monomer	1.0 M	S, A
η	stage efficiency	0.95	T, A
τ_m, τ_s	mixer, settler residence	3, 12 min	S, A
τ_{sap}	saponifier residence	15 min	T, A
s	design saponification	16.7 %	S
c_W, c_S	scrub, strip acid (HCl)	3, 4 M	T, A
c_{RE}, h_F	feed metals, free acid	0.35, 0.01 M	S, T
z_{Nd}	feed Nd share	75 %	S
F	feed flow	2 m^3/h	T
—	organic capacity of the settlers	56 m^3/h (O/A 28)	T
—	saponification cap	18 %	T
—	titration center	pH 6.5	T
p_N, p_H, p_o	NaOH, HCl, organic prices	0.45, 0.20 $/kg, 3 $/L	T, A
e_o	organic entrained	20 mL/m^3	T, A
T_{lab}, θ	laboratory period, delay	4, 1 h	T, A
—	specification (purity, recovery)	99.5, 97 %	T

Sources: β, K_{Nd} (Table 4 of the thesis through its Eq. 4), the 1 M extractant, the 3-minute mixer needed for equilibrium, the 12-minute settler (a 480 mL settler at 40 mL/min of total flow), the 16.7 % saponification and the 50 g/L feed are taken from the thesis [10], and the 75/25 feed from the companion paper [3]. The thesis also states the manufacturers' guideline of at most 30 % of the stoichiometric loading [10]. On the 3:1 capacity of the model the design point loads 32.2 %, 15 % saponification would load 29.5 and the 18 % cap 34.2, so the cap is a teaching value near the guideline rather than the guideline itself.

9.3.6 Assumptions

Two metals stand for a plant that separates a dozen lanthanides pair by pair, and cerium, lanthanum and the heavy rare earths are absent. The stages are at equilibrium with an efficiency, so mass-transfer kinetics, a third phase, emulsions and aqueous carried in the organic are not modeled, and crud is only a loss of one stage's efficiency. Activities are concentrations and the separation factor and K_{Nd} are constants, while the measured constant falls with acidity. The scrub is hydrochloric acid, while the thesis's own flowsheet scrubbed with a neodymium solution in a 12-stage scrub circuit [10]. The settlers are four tanks in series at fixed volumes, an approximation of a plug delay that keeps the inventory closed when a flow changes. The titration curve, the prices and the entrainment are teaching values, and nothing in the model is plant data.

9.4 One Stage, Seventy Stages and Two Speeds

9.4.1 What Each Stage Adds

At total reflux the Fenske relation [16] of Section 1.4.4 would add $\ln\beta = 0.405$ to the log-odds $\ln(x/(1-x))$ of the purity at each stage. At the design ratios the steady-state solver gives 95.49, 97.69, 98.79, 99.37, 99.67, 99.83 and 99.91 % for 4, 8, 12, 16, 20, 24 and 28 scrub stages, a nearly straight line of 0.167 per stage. The scrub section runs far from total reflux. Its organic carries 2.681 kmol/h of metal into stage 41 and its aqueous returns 2.161 kmol/h as reflux, so the product is 0.520 kmol/h, a fifth of the extraction amount, and the reflux ratio is 4.15. The Kremser factor of a section at those flows (Equation (1.7) uses the same factor), $\beta W_m/O_m = 1.209$, gives $\ln 1.209 = 0.190$ per stage, and 0.181 with the stage efficiency taken as a bypass, against an organic log-odds gain of 0.185 to 0.195 per stage inside the section. The staircase's 0.167 is lower, likely because adding scrub stages at fixed flows also shifts the profile at the section's two ends, where the feed and strip entries break the

constant-factor pattern. At $\beta = 1.3$ the cascade lines out at 95.98 % and each stage adds 0.045, close to $\ln(1.3 \times 0.806) = 0.047$, and at $\beta = 1$ both products are the feed.

9.4.2 A Fast Mode and a Slow Mode

The cascade holds 76.0 kmol of rare earth. A change of the scrub ratio redistributes the reflux without changing the neodymium balance at the moment of the step. Raising W/F from 1.173 to 1.178 leaves the neodymium fed and drawn at 0.5250 kmol/h each, and the neodymium inventory needs only 2.57 kmol more to reach the new steady state. At fixed flows the purity moves 63 % of its way in 25.3 h and 95 % in 76 h, overshooting its final value by 1.3 % of the step near 200 h, while the recovery takes 390 h to move 63 %, as shown in Figure 9.2(a). On the engine, with the saponification loop and the instruments, the purity's 63 % time is 27.1 h.

A feed-grade step changes the balance itself. At 67.5 % neodymium the feed brings 0.4725 kmol/h while the unchanged draw takes 0.5250 kmol/h, and the new steady state holds 37.7 kmol less neodymium. Inventory over imbalance is 718 h. Measured, the purity of case 4 is flat for a day, crosses 99.5 % 81.7 h after the change, reaches 63 % of its fall at 877 h and is 91.80 % at 1000 h on its way to 90.90 %, as shown in Figure 9.2(b). The recovery moves first and the wrong way, because the feed changes at once and the raffinate does not. A settling time of about 30 h for a 10 % feed step would follow from the 18-stage laboratory rig of the thesis, which was run that long to reach steady state [10], but it does not carry to seventy stages with 108.6 hours of feed in them, so the cases teach the day-long reflux response and the weeks-long inventory response instead.

> **TRY IT IN THE APP** **4 · The Long Transient: A Leaner Feed Takes Days to Arrive**
>
> Load the scenario and press Run. Watch the purity trend. The feed turns leaner at hour 4, yet the purity stays flat for a day and only then begins to slide. The true purity crosses 99.5 % at hour 85.7, and the first laboratory report below it comes from the sample of hour 88.

The profile shows the slow mode too. In case 1 the scrub ratio is raised from 1.173 to 1.200 at hour 1. A day later the purity has risen from 99.671 to 99.733 %, most of its way, while the stage where the aqueous is half neodymium has moved only from 12.60 to 11.95 toward the 6.00 of the new steady state, as shown in Figure 9.1(c). The parting stage lies in the extraction section and follows the inventory, and it reads 11.24 at hour 49.

Pause. Try Exercise 9.2 before reading on.

9.4.3 Interaction, and What the Ratio Is Keyed To

The steady-state gains at the design point, from upward steps of 0.0005 in W/F and 0.02 in O/A, are $\partial x/\partial(W/F) = 0.0416$, $\partial x/\partial(O/A) = -0.00173$, $\partial R/\partial(W/F) = -2.55$ and $\partial R/\partial(O/A) = 0.142$ in fractions per unit ratio, and the relative gain of purity on the scrub ratio follows as shown in Equation (9.6).

$$\lambda_{11} = \left(1 - \frac{K_{12}K_{21}}{K_{11}K_{22}}\right)^{-1} = 3.92 \tag{9.6}$$

where K_{ij} is the gain of output i (purity, recovery) on input j (W/F, O/A). The knee of the purity and recovery trade is curved, so downward steps give 3.60 and central differences 3.76, and four other points on the knee give 3.72 to 3.82. The pairing of purity with the scrub ratio and recovery with O/A is the one to use, and the crossed pairing has $1 - \lambda_{11} = -2.92$. The sign test agrees. The gain of purity on O/A is -0.00172 open and $+0.00062$ with the recovery held by the scrub ratio, so it changes sign when the other loop closes [8]. Volume 6, Chapter 3 meets the same structure on a paper machine, two quality loops that act only when a sampled gauge reports and a relative gain that is negative on one pairing.

The number belongs to the handles as well as to the plant. With the scrub keyed to the feed, more organic raises the extraction amount while the reflux stays, so the product draw, their small difference,

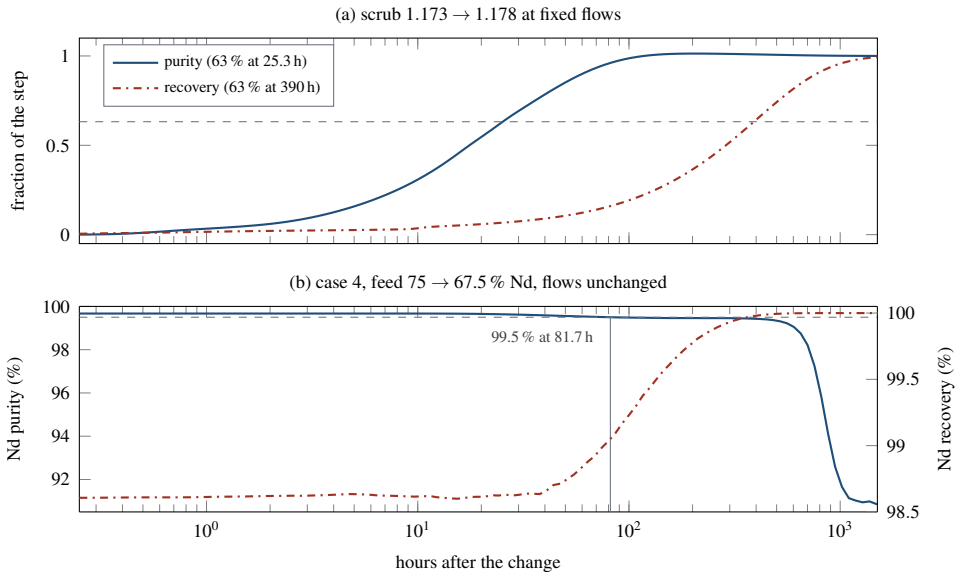


Figure 9.2 The two time scales. (a) The fraction of its step that the purity and the recovery have moved after the scrub ratio is raised from 1.173 to 1.178 at fixed flows and NaOH, on the dynamic stack alone, purity solid and recovery dash-dotted, with the 63 % line dashed. (b) Case 4 on the engine, the feed cut from 75 to 67.5 % neodymium with every flow unchanged, the true purity (solid, left axis) and recovery (dash-dotted, right axis) to 1500 h, with the 99.5 % line dashed.

grows and both products respond. Keyed to the organic, as scrub per cubic meter of organic, more organic brings more reflux with it, and the same cascade gives $\lambda_{11} = 0.62$ with upward steps and 0.60 by central differences, both pairings positive and neither rejected. Keying the ratio is the configuration choice of two-point distillation control, where the steady-state relative gain of the same column changes with the choice of flows and their ratios [7]. The application keys the scrub to the feed, the NaOH and the strip acid to the organic, and its pairing lesson is honest for those handles.

9.4.4 A Capacity Limit, Not a Stage Limit

The scrub ratio buys purity with recovery along each O/A curve, as shown in Figure 9.3. At 97 % recovery the purity is 99.04, 99.63, 99.70 and 99.82 % at O/A 20, 24, 25 and 28, and 99.87 % at O/A 28 with the 18 % saponification cap. A sweep of 49,771 steady states over O/A from 15 to 36, saponification from 10 to 24 % and four strip ratios finds the best purity at 97 % recovery within $O/A \leq 28$ and 18 % to be 99.866 %, so 99.9 % at 97 % is out of reach within the equipment limits. Past them it is reachable, at O/A 33 and the design saponification with $W/F = 1.625$ (99.902 %) or at 21 % saponification and O/A 26 (99.903 %). The limit is the extractant's and the settlers' capacity, not the stage count. The NaOH station stops at 0.18 kmol/m^3, the cap on the nameplate extractant, so the second point cannot be reached by hand. A station without that stop would reach it. The cheapest steady state that just meets 99.5 % and 97 % at the design saponification sits at O/A 23 and \$2.69/kg, against \$2.93/kg at the design point with its margins.

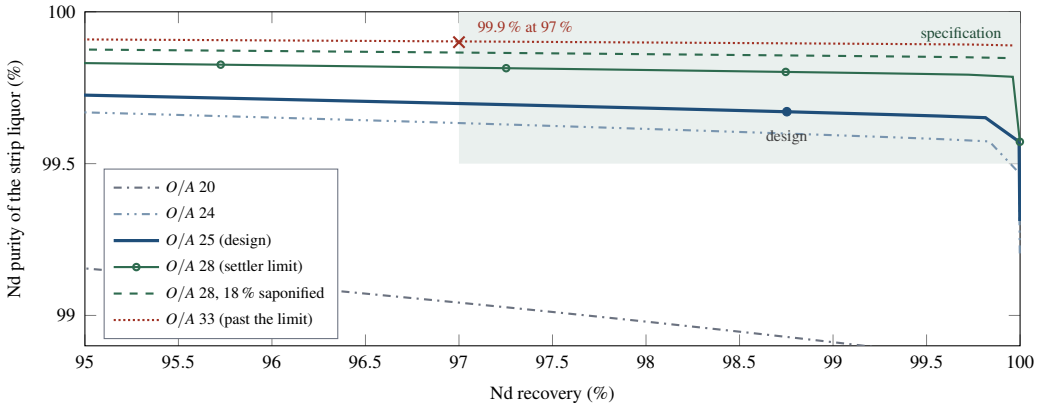


Figure 9.3 Purity against recovery from the steady-state solver at the design feed, each curve one O/A swept across the scrub ratio at 16.7 % saponification unless marked. The shaded corner is the teaching specification. The cross at 99.9 % purity and 97 % recovery lies above every curve inside the settlers' organic capacity and the saponification cap and below the curve at O/A 33.

9.5 Control as Implemented

9.5.1 Ratio Stations and Saponification

Every 5 min the first layer sets the flows from the metered feed, as shown in Equation (9.7).

$$\begin{aligned} O &= \min\big(56,\ (O/A)\,F_m\big), \qquad W = (W/F)\,F_m \\ S &= (S/O)\,O, \qquad n_{OH} = \mathrm{PI}_s(s_{SP} - s_m) \end{aligned} \tag{9.7}$$

with the NaOH commanded at $n_{OH}O$, so the NaOH and the strip acid follow the organic at once and the saponification PI ($K_c = 0.1$ kmol/m^3 per unit, $\tau_I = 0.3$ h) trims the NaOH per cubic meter between 0 and 0.18 on the share read from the saponifier's pH. The stations clamp O/A to 15–28 and W/F to 0.8–1.6, and the organic stops at 56 m^3/h, the settlers' capacity at the design feed.

9.5.2 Two Sampled Product Loops

Each product loop is the velocity-form PID of Section 1.6.2 with the derivative off, executed once per fresh analysis and holding between analyses, as shown in Equation (9.8).

$$\begin{aligned} e_k &= \sigma\,(y_{SP} - y_{lab,k}), \qquad \Delta t_k = t_k - t_{k-1} \\ S_k &= S_{k-1} + K_c(e_k - e_{k-1}), \qquad \hat{u}_k = S_k + I_{k-1} \\ u_k &= \min\big(u_{max} - u_{ff},\ \max(u_{min} - u_{ff},\ \hat{u}_k)\big) \\ I_k &= I_{k-1} + \left[\frac{K_c}{\tau_I}e_k + \frac{u_k - \hat{u}_k}{T_t}\right]\Delta t_k \\ T_t &= \max(\tau_I,\ \Delta t_k) \end{aligned} \tag{9.8}$$

where t_k is the report time, σ is the action a of Equation (1.10), the error is in fractions with K_c entered per point, u is the ratio before the feedforward u_{ff}, and the applied ratio is $u_k + u_{ff}$. The integral used at report k was accumulated through report $k-1$, so a report's error reaches the ratio through the proportional term at once and through the integral four hours later. In the pairing of the relative gain, purity drives W/F and recovery O/A, both with $\sigma = +1$, reverse acting in the convention of Section 1.6.2, because each product rises with its ratio. The crossed pairing drives O/A from purity and W/F from recovery, both with $\sigma = -1$. Both are direct acting, because purity falls as O/A rises and recovery falls as W/F rises. The application's source labels these two loops direct

acting too, and its control panel names the pairing Purity→O/A. The shipped tuning is $K_c = 0.01$ per point with $\tau_I = 40\,\text{h}$ for purity and 0.05 with 400 h for recovery, and the cases set their own.

Pause. Try Exercise 9.3 before reading on.

Three details guard paths that no shipped case takes. The tracking time is $\max(\tau_I, \Delta t_k)$. With τ_I alone, a loop with $\tau_I = 1\,\text{h}$ at its scrub limit would be kicked from 1.6 to 1.23 at the next report while that report was still below setpoint. A pairing switch during a run re-initializes each PI on its new loop's error at the applied ratio. Without it, the first report after a switch would apply the jump between the two loops' proportional terms, $\Delta(W/F) = -0.1125$, against the -0.0130 applied. The NaOH station stops at its cap, as described above.

9.5.3 Feedforward From the Feed Analysis

The neodymium the product must carry is the neodymium fed times the recovery. Each mole of neodymium that the scrub acid returns down the scrub section takes three moles of hydrochloric acid, so at each feed analysis the scrub ratio moves as shown in Equation (9.9).

$$u_{ff} = -\frac{3}{c_W} R_{SP} \left(c_{Nd,lab} - c_{Nd,ref} \right) \tag{9.9}$$

where $c_{Nd,ref}$ is the feed analysis when the feedforward is switched on, which keeps the draw in step with the neodymium fed while the organic and the extraction amount stay. For the leaner feed of cases 4 and 6 the law asks $+0.0259$, and the part measured in a run is 0.0246 because the feed analysis carries its noise. The switch is bumpless, because the PI's integral takes over the feedforward's part when it is turned off. Volume 3, Chapter 6 makes the same move, a dose computed from the raw feed with the late product measurement demoted to a slow trim.

9.5.4 Predictive Ratio Supervision

The optional model predictive controller follows Section 1.6.3 and uses four local lag paths, rather than identifying a model inside a five-day case. Central steps of $\Delta W/F = \pm 0.0005$ and $\Delta O/A = \pm 0.02$ were observed for 1000 h at nominal chemistry with product loops manual and saponification automatic. Instrument precursors provide the identification outputs only. Online control uses the selected reported assays and metered flows. In input order $(W/F, O/A)$ and output order (purity, recovery), the fitted percentage-point gains and hour time constants are

$$\begin{aligned} K &= \begin{bmatrix} 4.1994 & -0.17229 \\ -249.108 & 13.6935 \end{bmatrix}, \\ \tau &= \begin{bmatrix} 26.9067 & 25.5122 \\ 507.155 & 484.893 \end{bmatrix}, \qquad G_{ij}(s) = \frac{K_{ij}}{1 + \tau_{ij} s}. \end{aligned} \tag{9.10}$$

These are finite-window dynamic fits, not established infinite-time gains. A fitted 0.2365 h delay on purity from scrub is omitted. RMS errors of the implemented paths are 0.00000765 and 0.0000169 purity points, and 0.00116 and 0.00292 recovery points. Saponifier noise remains in the experiments and cancels only in the central difference. The four states advance by exact exponential lags on measured ratios every 5 min.

Each fresh selected assay corrects an output bias by $b_i^{+} = b_i + 0.3[y_{i,lab} - \hat{y}_i(t_{sample}) - b_i]$, limited to ± 2 purity points and ± 5 recovery points. The nominal output at the sample time is interpolated from saved five-minute predictions. Comparing at report time would mix process motion with model error, the point of Equation (1.8). This is a bounded residual correction rather than a convergence guarantee for a changed gain [17]. No current stage profile, chemistry, capacity loss or unreported feed analysis enters the predictor.

Every 4 h, the quadratic program covers 480 h in 120 steps, with twelve graduated blocks. In percentage points and unscaled ratio units, the objective has output weights $(100, 1)$ and block-change

Table 9.3 The Ten Cases

#	Settings and events	Teaching point
1	Manual at design, learner raises W/F to 1.200	the profile and its pinch
2	Manual, β set to 1.30 and Reset	stages and separation factor
3	Manual, learner raises W/F to 1.185	the trade and its limit
4	Manual, feed 75 to 67.5 % Nd at 4 h	the long transient
5	Purity loop 1.5 per point, 4 h, feed to 82.5 % at 6 h	a loop on stale reports
6	Purity loop 0.05, 24 h, feed to 67.5 % at 6 h	the feedforward
7	Both loops, crossed pairing (1, 24 h and 0.05, 24 h)	the relative gain
8	Feedforward, purity loop 0.05, capacity −10 % at 6, crud at 30 h	the bank before the laboratory
9	Higher purity and lower recovery requested at 8 h	coordinated ratios
10	Challenge, 120 h, feed 82.5 % at 12 and 67.5 % at 48, crud at 72 h	the whole problem

Table 9.4 Case Outcomes Measured on the Engine

Case and condition	Result
1, W/F 1.200 at 1 h	parting 12.60 / 11.95 / 11.24 at 1, 25, 49 h, x 99.733 %, R 98.22 % at 25 h
2, β 1.5 / 1.3 / 1.0	x 99.67 / 95.98 / 75.00 %
3, W/F 1.185 at 1 h	x 99.700 / 99.710 %, R 98.54 / 98.43 % at 25 / 49 h, \$2.93 to \$3.04/kg
4, as shipped	first report after the step R 98.63 against 98.78 %, x below 99.5 % at 85.7 h, 99.476 % at 120 h
5, shipped / 0.05, 24 h	W/F 1.015–1.201 / 1.1665 to 1.1449, x 99.52–99.67 / 99.74 %
6, feedforward off / on	x 99.510 / 99.591 % at 96 h, \$2.94 / \$3.08 per kg over 96 h
7, crossed / paired	W/F 1.2013 / 1.155, O/A 25.472 / 24.646, x 99.702 / 99.661 % at 96 h
8, capacity loss	s_m 18.4 % at once, ICP R 98.20 % at 9 h, true R 87.17 % at 48 h

weights $(10^4, 10)$. Predicted purity and recovery floors are 99.5 and 97 %. One slack per output, shared across the horizon, costs $10^5\xi_x^2 + 10^4\xi_R^2 + 10^3(\xi_x + \xi_R)$, with ξ_x and ξ_R the slacks. The ridge is 10^{-8}. Ratio limits are $0.8 \le W/F \le 1.6$ and $15 \le O/A \le 28$, with the organic hydraulic cap retained, and block changes are at most 0.002 and 0.05. A manual product loop fixes its assigned handle. There is no reagent objective or feed-composition feedforward in this MPC, unlike the classical balance correction.

The ratio PIs track the applied commands. A rejected solver result holds both ratios on the first failed scan, then returns to sampled PI. Validation checks the whole planned sequence, diagnostics and bounds. A valid solve resumes prediction, and leaving the option clears its model history. These choices implement receding-horizon supervision [18] while the ratio stations and saponification loop remain active.

9.6 Ten Cases on the Running Cascade

The cases are listed in Table 9.3 and their outcomes in Table 9.4. Every value was measured with the application's engine of Section 1.6.1 stepped headless at its 20 s step and the default seed, with each case loaded as the browser loads it and every learner move made at the hour its text names. A measuring script reproduces every number the application's texts quote.

9.6.1 The Profile, the Staircase and the Trade

Case 1 opens at the design point with the metals parting at stage 12.60, the organic loaded to 32.2 % of its capacity at stage 40, and the measured pH at 1.26 in stage 1, about 0.7 through the extraction section, 0.22 at the top of the scrub section and below zero in the strip. Raising the scrub ratio to 1.200 moves the parting toward the raffinate end, to 11.95 a day later and 11.24 at hour 49, while the pH profile hardly changes, because the separation is an exchange of one metal for another at a nearly constant loading. Case 2 reads the staircase of the previous section on the Map and then lowers the separation factor to 1.30, where the shipped cascade lines out at 95.98 %. Case 3 raises the scrub ratio by one percent, to 1.185. The purity rises by four hundredths of a point, to 99.710 % at hour

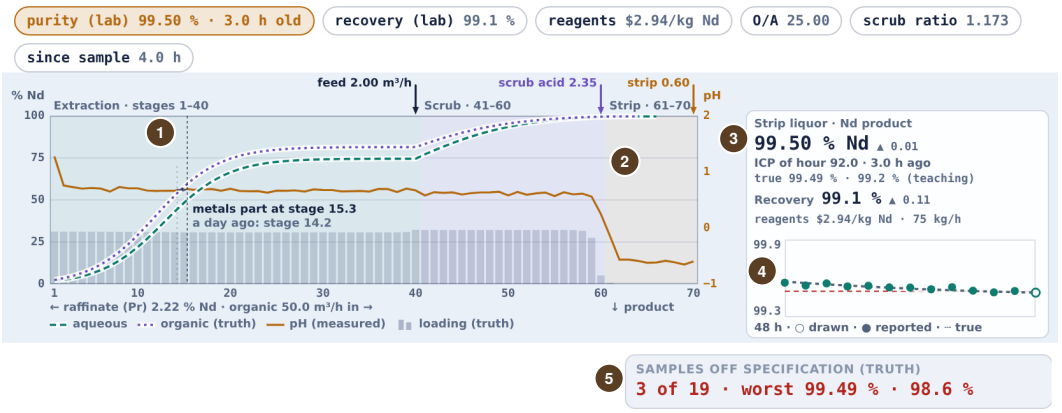


Figure 9.4 Case 4 at its hour-96 pause on the Cascade view, in Manual with every flow at the design ratios (O/A 25.00, scrub ratio 1.173), light theme, 1366 × 768, cropped to the plant view and the samples tile of the Learn panel. The feed fell from 75 to 67.5 % neodymium at hour 4. The profile shows the metals parting at stage 15.3 against 14.2 a day earlier (1), the organic loading near a third of its capacity and the measured pH, which falls below zero in the strip section (2). The products panel gives the ICP purity of 99.50 %, up 0.01 on the previous report, from the sample of hour 92 reported 3.0 h ago, the true 99.49 % and 99.2 %, and the recovery of 99.1 %, up 0.11 (3), with the samples of the last two days sliding toward the 99.5 % line (4). The badges give $2.94 of reagents per kilogram, and the scorecard three of 19 samples off specification with the worst at 99.49 % (5).

49, while the recovery falls to 98.43 % and is still falling at hour 121 (97.98 %), because one percent more reflux cuts a draw that is a fifth of the extraction amount by several percent.

9.6.2 A Leaner Feed by Hand

Case 4 cuts the feed to 67.5 % neodymium at hour 4 and leaves every flow alone. The first report after the change, the sample of hour 8 reported at hour 9, reads a recovery of 98.63 % against 98.78 % before, the wrong way, because the feed carries less neodymium before the raffinate has changed. The purity reads 99.666 % at hour 25, the true purity crosses 99.5 % at hour 85.7, and the first report below it is the sample of hour 88 (99.492 %). At the pause at hour 96 the true purity is 99.490 % with the recovery at 99.16 %, the parting has walked from 14.25 at hour 72 to 15.35, and three of the 19 samples drawn after the first day have fallen short of the specification, as shown in Figure 9.4. The unchanged draw takes more neodymium than arrives, the cascade gives up its inventory, and praseodymium creeps up the scrub section into the product.

🕮 **Pause.** Try Exercise 9.1 before reading on.

9.6.3 Loops on a Four-Hour Laboratory

Case 5 runs the purity loop at 1.5 per point with a 4 h integral time and raises the feed grade to 82.5 % at hour 6. Each report sees the result of moves made a day before and responds with another move. The moves do not grow from report to report, but the scrub ratio keeps swinging. From hour 6 to 96 the scrub ratio swings between 1.015 and 1.201, about 17 % of its middle, the pH of stage 1 between 1.18 and 2.08, and the true purity between 99.52 and 99.67 %. At 0.05 per point and 24 h the ratio moves smoothly from 1.1665 at hour 24 to 1.1449 at hour 96 and the purity settles near 99.74 %. The swinging loop stays inside the specification only because the plant is slow. On the online analyzer the same gain acts eight times as often and the scrub ratio swings between 0.899 and 1.304.

Case 6 repeats case 4's leaner feed at hour 6 with the detuned purity loop in Auto. Without the feedforward the loop sees nothing for a day and then chases the drift, the purity falling from 99.668 to

99.510 % by hour 96 (worst sample 99.509 %) while the scrub ratio creeps from 1.169 to 1.180. With it the scrub ratio steps to 1.197 when the leaner feed is reported at hour 9, the purity rises to 99.693 % at hour 24 and eases to 99.591 % at hour 96. The reagents over the 96 hours rise from $2.94 to $3.08 per kg, because without the feedforward the cascade was selling neodymium out of its own inventory.

> **TRY IT IN THE APP** **6 · A Feed Change With the Feedforward**
>
> Run to the pause at hour 96 with Feed feedforward Off, then switch it On, press Reset and run again. Off, the purity slides to 99.510 % by hour 96. On, the scrub ratio steps to 1.197 when the leaner feed is reported at hour 9, the purity ends at 99.591 %, and the reagents rise from $2.94 to $3.08 per kilogram.

9.6.4 The Pairing

Case 7 closes both loops with purity on O/A and recovery on the scrub ratio. Each loop undoes the other's move and asks for more. By hour 96 the scrub ratio has climbed from 1.173 to 1.2013 and O/A from 25.00 to 25.472 (1.2164 and 25.769 by hour 168), the purity sits at 99.702 % against its 99.6 % setpoint and the reagents cost $3.00/kg. Paired as the relative gain says, with gains of 0.05 and 1, the scrub ratio eases to 1.155 and O/A to 24.646, the purity comes down to 99.661 % with the recovery at 98.53 %, and the reagents fall to $2.90/kg. Along the crossed run the Map's relative gain, recomputed at the plant's present ratios, stays between 3.90 and 4.11. The drift is slow because the recovery answers in weeks, and slow is not stable.

9.6.5 Capacity Loss and Crud

Case 8 removes a tenth of the extractant's sites at hour 6. The saponifier reads 18.4 % within minutes against its 16.7 % setpoint, because the same sodium is a larger share of fewer sites, and the loop cuts the NaOH from 8.36 to 7.51 kmol/h by hour 24, which also cuts the rare earth the organic can take up. The pH of stage 1 passes below 1.20 at hour 7.1 and reads 1.09 at hour 9, when the first ICP report with the loss gives a recovery of 98.20 %. The true recovery is 97.20, 94.79 and 87.17 % at hours 12, 24 and 48, while the purity rises to 99.83 %. The crud at hour 30 costs about five hundredths of a point of purity (99.624 against 99.670 % at hour 48 on case 1's plant). Its stage matters. A 70 % efficiency loss in any of stages 5 to 40 costs 0.0001 points of steady purity, because the feed stage pinch leaves the extraction section stages to spare, while in stages 45 to 55 it costs 0.047 points of purity and 0.045 of recovery. Stage 30 is no praseodymium-only zone (its aqueous is 80.9 % neodymium), and it lies in the feed-stage pinch, so the crud event sits at stage 55.

9.6.6 Five Days, Two Feed Changes and Crud

The challenge runs 120 h with the feed at 82.5 % neodymium from hour 12, at 67.5 % from hour 48 and crud in stage 55 at hour 72. It is met when every ICP sample drawn after hour 24 has a true purity of at least 99.5 % and a true recovery of at least 97 %, and the reagents over the run cost at most $2.95/kg of neodymium. Changing a model parameter or switching the loops to the online analyzer voids the attempt. The configurations are compared in Table 9.5. As shipped, the purity loop alone at 0.5 per point and 6 h puts two of 25 samples short (worst 99.47 % at hour 76) at $2.85/kg. The suggested settings, the feedforward with the purity loop at 0.05 and 24 h, keep every sample at or above 99.55 % at $2.93/kg. The feedforward alone holds the purity but fails the cost at $3.01/kg, because nothing brings the scrub back down once the richer feed makes purity cheap, and the detuned loop alone is too slow for the leaner feed, with seven samples short. Adding the recovery loop to the suggested settings puts four samples short, because in five days it can only trade purity for NaOH. With the feedforward on, every purity gain from 0.05 to 0.3 passed at integral times of 12 and 24 h and every gain from 0.05 up failed at 6 h, so the integral time is what has to match the plant's day. At seeds 7 and 11 and at half the step the verdicts hold, the suggested settings passing at 99.547 to 99.557 % and $2.925 to $2.935 per kg, a margin of $0.015 to $0.025 per kg under the cap. On the

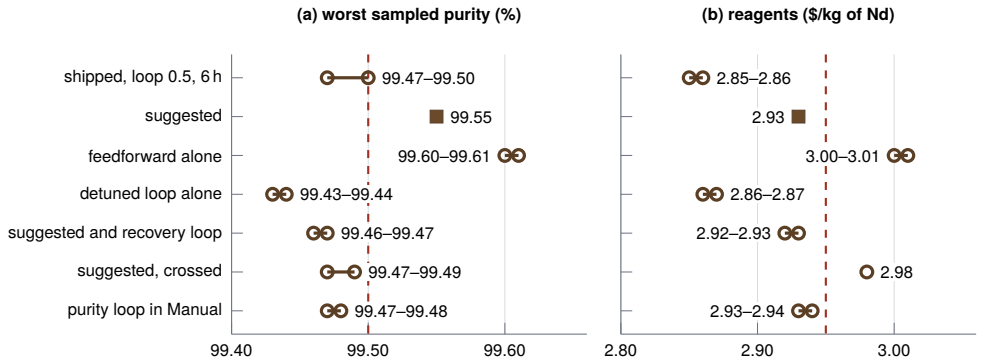


Figure 9.5 The challenge of Table 9.5 as a chart, the range over five seeds drawn from its low to its high value. Only the suggested settings (filled square) sit right of the purity floor and left of the cost cap (dashed lines). The feedforward alone holds the purity with the widest margin of any policy and fails on cost, while the shipped loop and the detuned loop alone, both without the feedforward, are the cheapest rows and fall short of the purity floor.

Table 9.5 The Challenge Over 120 h, Five Seeds Each

Policy (ICP only)	Pass	Short	Worst x (%)	$/kg
shipped, purity loop 0.5, 6 h	0/5	1–2	99.47–99.50	2.85–2.86
suggested, feedforward and 0.05, 24 h	5/5	0	99.55	2.93
feedforward alone (gain 0)	0/5	0	99.60–99.61	3.00–3.01
detuned loop alone	0/5	7	99.43–99.44	2.86–2.87
suggested and recovery loop (1, 24 h)	0/5	4	99.46–99.47	2.92–2.93
suggested, crossed pairing	0/5	2–3	99.47–99.49	2.98
purity loop in Manual	0/5	3	99.47–99.48	2.93–2.94

online analyzer even the shipped loop passes (worst 99.53 %, $2.88/kg), which is why the attempt is graded on the ICP. Figure 9.5 draws the table as a chart.

> ✦ DID YOU KNOW? **On Specification and Still Failing**
>
> From the simulator: in Table 9.5 the feedforward alone keeps the purity of the challenge highest, its worst sample at 99.60 to 99.61 %, yet fails all five seeds at $3.00 to $3.01 per kilogram. The suggested settings pass every seed with a worst sample of 99.55 % at $2.93.

9.6.7 Coordinated Ratios Against Sampled PI

Case 9 raises requested purity from 99.6 to 99.8 % and lowers requested recovery from 98.5 to 97.5 % at hour 8. Both loops are automatic, with identical seed, instruments and initial conditions for the two methods. The 120 h comparisons are in Table 9.6, whose rows run cases 9, 5, 6, 7 and 8 as loaded and the challenge with its suggested settings, with only the supervision changed. IAE integrates each reported product through the same metric used by the scorecard. Off-specification hours use the true purity, and cost is cumulative reagent expense per kilogram of Nd produced.

On the new requests, purity IAE falls from 13.11 to 9.24 point h and recovery IAE from 124.31 to 80.93 point h, but reagent cost rises from $2.977 to $3.097/kg. Neither run crosses the purity specification. The different response rates appear in Figure 9.6. Halving the integration step retains the ranking, with purity IAE 13.13 versus 9.26 point h. A six-setting hindsight search of the classical gains is retained in the evidence without claiming a globally optimal PI comparator.

Table 9.6 Matched 120-Hour Supervision Comparisons

Case, law	Purity IAE (point h)	Recovery IAE (point h)	Off (h)	$/kg
Setpoints, PI	13.11	124.31	0.00	2.977
Setpoints, MPC	9.24	80.93	0.00	3.097
Fast PI case, PI	3.16	120.42	0.00	2.520
Fast PI case, MPC	18.11	34.03	0.00	2.910
Feed change, PI	7.63	47.08	0.00	2.954
Feed change, MPC	6.47	33.25	0.00	2.991
Crossed PI, PI	11.92	15.20	0.00	3.012
Crossed PI, MPC	6.33	22.64	0.00	2.865
Capacity/crud, PI	24.74	1659.68	0.00	3.711
Capacity/crud, MPC	26.34	1899.15	0.00	3.864
Shift, suggested, PI	6.57	42.35	0.00	2.927
Shift, suggested, MPC	11.86	27.83	17.78	2.925

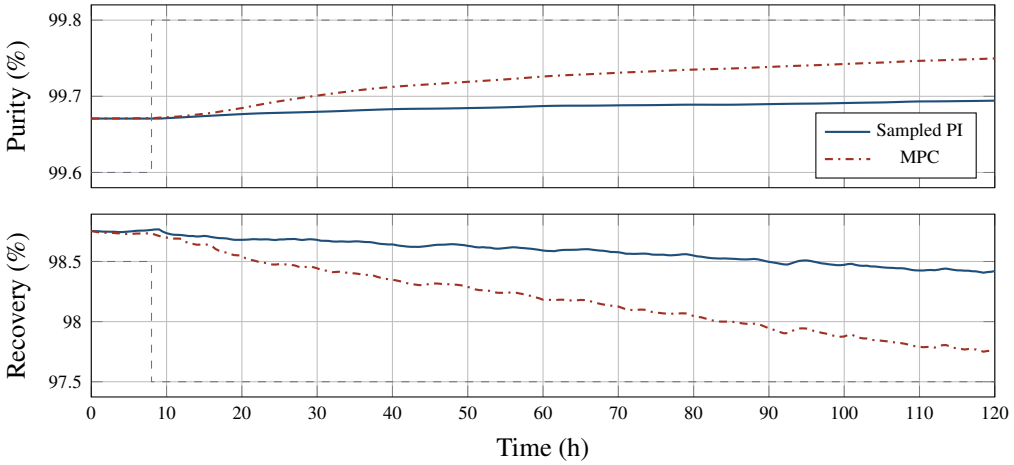


Figure 9.6 Purity and recovery following the paired setpoint changes of case 9, sampled PI solid and MPC dash-dotted. Thin dashed lines are the requests.

The benefit does not carry to the suggested shift. Compensated PI passes with no purity-specification crossing, while MPC spends 17.78 h below it and has worst sampled purity 99.443 %. The model has no feed-composition input. On the single feed change, adding the classical feedforward gives purity IAE 4.82 rather than MPC's 6.47 point h. With capacity loss and crud, worst sampled recovery is 69.93 versus 73.48 %. Bias correction does not repair the wrong dynamic gain. The soft product floors therefore cannot be interpreted as actual specification guarantees.

All ordinary comparison solves are accepted, 30 per 120 h active run. Chrome averages 3.98–4.67 ms per solve in the six layout captures, at most 0.117 % of one core at unit playback and 0.350 % at the top 3 h/s rung. Independent tests reject ten injected malformed or invalid solver results and reproduce two measurement rows exactly.

9.7 Technology in Practice

Figure 9.7 draws the loops of Section 9.5 with their hardware.

What Measures It

The cascade's composition is known only from analyses of samples. The model reports the purity, the recovery and the feed's neodymium from samples drawn every 4 h and reported 1 h later, reads

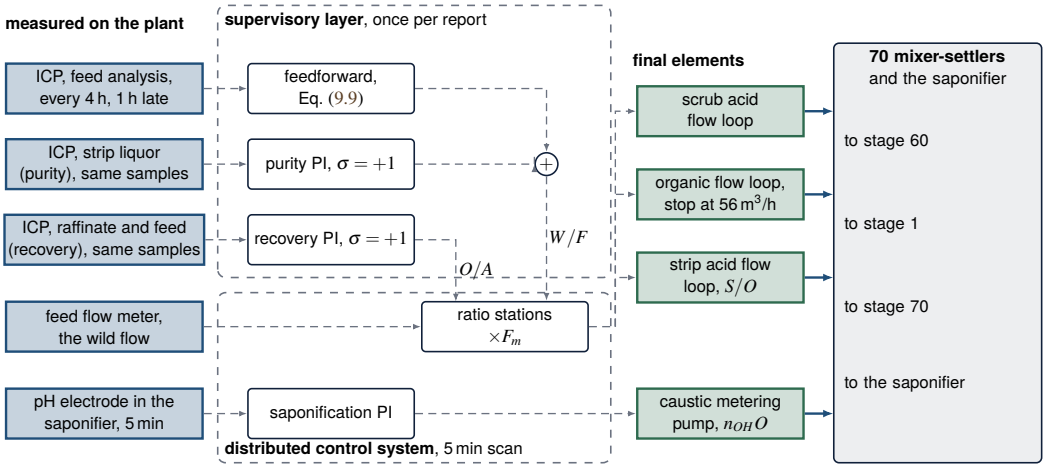


Figure 9.7 The loops of the simulated cascade drawn with their hardware. Three laboratory analyses of the same samples feed the supervisory layer, which acts once per report: the feedforward and the purity PI add up to the scrub ratio, and the recovery PI sets the organic ratio. The ratio stations of the regulatory layer multiply both by the metered feed, the wild flow, every 5 min, and the saponification PI on the saponifier's pH trims the caustic dose.

the pH of every stage every 5 min, and offers an online X-ray fluorescence option that samples every 30 min with five times the purity noise.

> **TECHNOLOGY Sensor: Plasma Emission Analysis in the Laboratory**
>
> The purity and recovery of this chapter come from inductively coupled plasma optical emission spectroscopy. A sample is sprayed into a plasma sustained by a radio-frequency coil, the excited atoms and ions emit light at discrete wavelengths, and optics separate the lines for a detector. Neodymium can be read at 430.358 nm and praseodymium at 417.939 nm. An organic sample cannot be analyzed directly, so its metal is first stripped into acid. In one laboratory study the samples were diluted 2 to 500 times into a calibration of 10 to 100 000 µg/L, a control standard every ten samples guarded against drift, spiked samples checked for interference, and mass balances on the stripped organic closed within 5 % [10].

The one-hour delay of Equation (9.5) stands for all of that work, the dilution, the stripping of an organic sample and the queue at the instrument, and the 0.01 points of purity noise stand for its error.

What Moves It

The model sets the organic, scrub, strip and caustic flows from the metered feed every 5 min, meters them exactly and stops the organic at the settlers' 56 m^3/h.

> **TECHNOLOGY Final Element: Ratio Stations and Metering Pumps**
>
> A ratio station takes the signal of the uncontrolled, or wild, flow, multiplies it by an adjustable constant and sends the product as the setpoint of the controller on the captive flow, so the proportion holds whatever the total [19]. In this cascade the feed is the wild flow, and the organic, the scrub acid and the strip acid are captive. Small flows of acid or caustic are commonly delivered by metering pumps, a pump, motor and drive in one package whose output runs from no flow at 4 mA to full flow at 20 mA. Many use a plunger driven by a solenoid and deliver discrete pulses rather than a continuous flow [19].

In the model each captive flow follows its setpoint at once, so the lag of a real flow loop and the pulses of a plunger pump are left out.

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 [20]. In the app the ratio stations and the saponification PI act every 5 min, while the two product loops act once per laboratory report, every 4 h, on a plant whose purity needs about a day to answer the scrub. On such a platform the ratio stations and the saponification loop are function blocks of the regulatory layer, and the product loops with the feedforward form a supervisory application that writes the two ratios.

> **IN PRACTICE** **Monitoring Stages and Experience**
>
> In industrial rare earth extraction, monitoring stages are set in each extraction and scrub section, and the flows of extractant and scrub liquor are adjusted from the deviation between the measured and the target content there. In one plant that content came from a daily laboratory analysis of field samples [21]. Dosing has been described as offline analysis, manual adjustment and experience control: the operator sets the flow rates from experience and inner PID loops hold them [22]. Saponification is among the largest reagent costs of a separation plant, and extractant makers recommend loading the organic to no more than 30 % of its stoichiometric capacity [10].

What Is Modernizing It

The first development is the soft sensor. Data-driven models estimate a hard-to-measure quality from variables that are easy to measure, and a recent one estimates the content at a monitoring stage from the extractant, scrub and feed flows and the feed composition, recorded every 30 min, against a laboratory that reports daily [21]. The second is machine vision: because the solution changes color with its praseodymium and neodymium content, color features of its image have been used to model the neodymium content of a production cascade [5]. The third is predictive control. An outer model predictive controller that sets the extractant and scrub flows over inner PID loops has been proposed in place of manual dosing, with an event trigger that updates the controller only when the process calls for it [22]. The predictive supervisor of Section 9.5.4 is a version of that idea, and its failure on the feed shift shows why such a controller still needs the feed analysis.

9.8 Limitations

Every flow, price and limit is a teaching value, and only the chemistry constants, the residence times, the saponification and the feed come from a source, a laboratory thesis rather than a plant. The constant K_{Nd} is held at one acidity while the measured value spans 0.019 to 0.044 over the process range. At the shipped ratios half of it gives 99.72 and 96.1 % and twice it 97.9 and 100 %, so the design ratios belong to the constant, and a re-tuned cascade would teach the same lessons at other ratios. The separation factor is the same at every stage and loading, while the sources measure 1.29 to 2.05 as those change [10, 15]. Two metals, equilibrium stages, settlers as four tanks and a hydrochloric acid scrub stand for a plant of many pairs, finite kinetics, real settler hydraulics and product reflux. Real feed tanks blend over hours, so a grade that steps at once is unrealistic, but the step isolates the inventory's response from everything else.

The organic loading has no consequence of its own. The 30 % guideline is met only approximately at design (32.2 %), and with the NaOH station at its cap on an extractant that has lost 30 % of its sites the loading reaches 50 % with no third phase or solubility penalty. The infeasibility of 99.9 % at 97 % holds only within the declared limits of $O/A \leq 28$ and 18 % saponification. The recovery loop barely acts within a case because the recovery responds in weeks, so the challenge is won without it. The relative gain is the model's at one feed and moves from 3.60 to 3.92 with the step direction. The laboratory has no sample-line lag and no outliers except a scripted one, and the classical controllers retain only their material balance. The added predictive supervisor uses the clean local model of Equation (9.10), which cannot infer changing gains or the current extraction inventory.

OPEN CHALLENGE **Measuring Composition Along the Cascade**

Rare earth component content is hard to measure quickly in a plant, where slow dynamics and a harsh environment stand in the way, and the offline analyses in wide use delay control and cost product consistency [21]. The solution changes color as its praseodymium and neodymium content changes, and color features of its image have served as inputs to a neural network model of neodymium content in a 30-stage plant, where each group of tanks adds a delay that most models ignore or hold constant [5]. Progress would be an online estimate of the stage profile, checked against the laboratory, that a controller could act on within hours rather than a day.

9.9 Conclusion

A praseodymium and neodymium cascade of 40 extraction, 20 scrub and 10 strip mixer-settlers is modeled here as a two-phase stage stack with cation exchange on a dimeric extractant, a charge-balance pH and a saponifier, whose metal balances close to 10^{-11} and whose steady-state solver the dynamic stack reproduces to five decimals. A separation factor of 1.5 adds 0.167 to the purity's log-odds per scrub stage at the design ratios, against 0.405 at total reflux. The scrub ratio moves the purity 63 % in about a day, while a feed-grade step drains an inventory of 108.6 hours of feed whose ratio to the new imbalance is 718 h, and the purity of case 4 crosses 99.5 % only at hour 86. Keyed to the feed the scrub gives a relative gain of 3.92 that rejects the crossed pairing, which in case 7 drives both ratios up for days, and keyed to the organic the same plant gives 0.60. The 99.9 % and 97 % pair is a capacity limit, reached at O/A 33 or at 21 % saponification. A feedforward from the feed analysis with a purity loop detuned to the plant's day passes five days at \$2.93/kg, where either alone fails. The chemistry constants come from laboratory data and the rest are teaching values. The predictive option reduces paired-setpoint IAE at greater reagent cost, but fails a shift the compensated PI passes. A fuller model would add an acidity-dependent extraction constant, product reflux in the scrub, a third rare earth and a soft sensor on the stage pH. The simulator shows that a separation with a fast and a slow end has to be controlled at the speed of each end, on what is measured, when it is measured.

KEY POINTS

- The purity comes from the stage count, and the stage count makes the plant slow.
- The scrub ratio moves the purity 63 % in about a day, while a feed-grade step drains an inventory whose ratio to the new imbalance is 718 h.
- Keyed to the feed, the scrub gives a relative gain of 3.92 that rejects the crossed pairing, and keyed to the organic the same plant gives 0.60.
- The 99.9 % and 97 % pair is a capacity limit, not a stage limit.
- A feedforward from the feed analysis with a purity loop detuned to the plant's day passes five days at \$2.93/kg, where either alone fails.

Where else this applies. Volume 5, Chapter 3 closes a composition loop on a slow analyzer, and Volume 3, Chapter 6 doses from the raw feed with the late product measurement as a slow trim.

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9.10 Exercises

9.1 ○ Concept check · 2 min

In case 4 the feed grade falls from 75 to 67.5 % neodymium at hour 4 and no flow changes. The first laboratory report after the change, the sample of hour 8 reported at hour 9, reads a recovery of 98.63 % against 98.78 % before, although the recovery rises over the following days. Why does the first report move the wrong way?

(a) The leaner feed loads less neodymium on the organic, so more neodymium leaves in the raffinate at once

(b) The saponification loop cuts the NaOH when the feed changes, so the organic takes up less rare earth

(c) The recovery divides the raffinate's neodymium by the feed's, and the feed changes at once while the raffinate has not

(d) The recovery analysis carries noise, and the true recovery rises from the first hour while the report lags behind it

9.2 Concept check · 1 min

Why does the purity answer a step of the scrub ratio within about a day, but a step of the feed grade only over weeks?

(a) The scrub acid reaches the strip end at once, while the feed must travel through forty extraction stages

(b) The laboratory reports the scrub change at once, while the feed analysis is held for four hours

(c) The scrub step changes the separation factor, while the feed step leaves the separation factor unchanged

(d) The scrub step leaves the neodymium balance unchanged, while the feed step must drain tens of kilomoles at a small imbalance

9.3 Concept check · 2 min

In the crossed pairing of case 7 the purity analysis drives the O/A ratio. With the other loop open, the steady-state gain of purity on O/A is -0.00173 per unit ratio. In the convention of Section 1.6.2, what is this loop's action?

(a) Direct acting, $\sigma = -1$, because the purity falls as the organic ratio rises

(b) Reverse acting, $\sigma = +1$, because the purity loop on the scrub ratio is reverse acting

(c) Reverse acting, $\sigma = -1$, because the app names this pairing reversed

(d) Direct acting, $\sigma = +1$, because a negative relative gain flips the sign of the loop

9.4 Theory and calculation · 10 min

At the design point ($W/F = 1.173$, $O/A = 25$) the steady-state gains in fractions per unit ratio are $\partial x/\partial(W/F) = 0.0416$, $\partial x/\partial(O/A) = -0.00173$, $\partial R/\partial(W/F) = -2.55$ and $\partial R/\partial(O/A) = 0.142$. Give each relative gain to within 0.05.

(a) Compute λ_{11} of purity on the scrub ratio from Equation (9.6), and the relative gain of the crossed pairing. *Checkpoint:* $\lambda_{11} \approx 3.95$, crossed -2.95

(b) Key the scrub to the organic instead: $v = W/O$, so $W/F = v\,(O/A)$. Use the chain rule to find the gains of x and R on v at fixed O/A and on O/A at fixed v at the design point, and compute λ_{11} of purity on v. *Checkpoint:* gains 1.04, 0.000222, -63.75, 0.0224; $\lambda_{11} \approx 0.62$

(c) Which pairing does each keying reject, and why do the two numbers differ for one plant? *Checkpoint:* feed keyed rejects the crossed pairing, organic keyed rejects neither

9.5 Theory and calculation · 6 min

The design feed is 2 m^3/h at 0.35 M rare earth, 75 % of it neodymium, and the cascade holds 76.0 kmol of rare earth. Give each answer to within 1 %.

(a) How many hours of feed does the cascade hold? *Checkpoint:* 108.6 h

(b) The feed grade falls to 67.5 % with every flow unchanged, so the draw still takes 0.5250 kmol/h of neodymium. What is the imbalance, and what time does inventory over imbalance give for the 37.7 kmol the new steady state gives up? *Checkpoint:* 0.0525 kmol/h, 718 h

(c) A scrub step from 1.173 to 1.178 needs only 2.57 kmol more neodymium. Why does the same estimate not apply to it, and what does the chapter measure instead? *Checkpoint:* no imbalance at the step; 63 % in 25.3 h

9.6 Theory and calculation · 6 min

The feedforward of Equation (9.9) uses scrub acid at $c_W = 3\,\mathrm{M}$ and a recovery setpoint $R_{SP} = 98.5\,\%$. The feed is 0.35 M rare earth, and its grade falls from 75 to 67.5 % neodymium.

(a) Evaluate u_{ff} to four decimals. *Checkpoint:* $+0.0259$

(b) Show where the factor $3/c_W$ comes from, starting from the cut in the neodymium draw that keeps it equal to R_{SP} times the neodymium fed. *Checkpoint:* $\Delta W = 3R_{SP}F\,|\Delta c|/c_W$

(c) A run measures a feedforward part of 0.0246. The feed analysis reports its neodymium concentration with 0.5 % noise. How large is the noise of one reading of the design feed, 0.2625 M of neodymium, in M, and in the scrub ratio? Give two significant figures. *Checkpoint:* 0.0013 M, 0.0013 in the ratio

9.7 App challenge · 8 min

Open scenario 5, "The laboratory is four hours late: tuning a loop on stale reports", at the default seed. It runs in Auto with Purity gain 1.5 per point and Purity integral time 4 h, and the feed grade rises to 82.5 % neodymium at hour 6. Run at 3 h per second to the pause at hour 96 (about 32 s) and export the CSV file. Read ratios within 0.002 and the purity within 0.01 points.

(a) Read the lowest and the highest `WF_cmd` from Time (h) 6 to 96, and give the swing as a percentage of their mean, to 1. *Checkpoint:* 1.015 and 1.201, 17 %

(b) Set Purity gain to 0.05 and Purity integral time to 24 h, press Reset, run to the pause at hour 96 and export again. Read `WF_cmd` at Time (h) 24 and 96, and the true purity at hour 96 on the tile "Nd product: purity · recovery · reagents". *Checkpoint:* 1.1665, 1.1449, 99.74 %

(c) Both loops act once per report. Why does the second settle? *Checkpoint:* its integral time matches the plant's day, not the report period

9.8 App challenge · 8 min

Open scenario 7, "The pairing: why the relative gain says purity goes on the scrub", at the default seed. Purity drives O/A at gain 1 per point and recovery drives the scrub ratio at 0.05, both with 24 h integral times and both in Auto. Run at 3 h per second to the pause at hour 96 and export the CSV file. Read the scrub ratio within 0.0005, O/A within 0.01, and the true purity and recovery on the tile "Nd product: purity · recovery · reagents".

(a) Read `WF_cmd` and `OA_cmd` at Time (h) 96 and the true purity. Which way did each ratio move from 1.173 and 25.00? *Checkpoint:* 1.2013 and 25.47, both up; 99.70 %

(b) Set Pairing to Purity→scrub, Purity gain to 0.05 and Recovery gain to 1, press Reset and run to the pause at hour 96. Read the same values and the true recovery. *Checkpoint:* 1.1550, 24.65, 99.66 %, 98.5 %

(c) Use the relative gain to explain why the crossed loops walked both ratios up while the purity moved away from its 99.6 % setpoint. *Checkpoint:* purity's gain on O/A changes sign when the recovery loop closes

9.9 App challenge · 10 min

Open scenario 10, "Challenge · five days, two feed changes and crud", at the default seed. It is met when every ICP sample drawn after hour 24 has a true purity of at least 99.5 % and a true recovery of at least 97 %, with the reagents at most $2.95 per kg of neodymium. Each run takes 40 s at 3 h per

second and pauses at hour 120 with its verdict on the Challenge target tile. Read the worst sample within 0.01 points and the cost within $0.01 per kg.

(a) Run it as loaded (purity loop at 0.5 per point and 6 h, feedforward off). Record the verdict, the samples short, the worst sample purity and the cost. *Checkpoint:* not met, 2 of 25, 99.47 %, $2.85/kg

(b) Set Feed feedforward to On, Purity gain to 0.05 and Purity integral time to 24 h, press Reset and run. *Checkpoint:* met, 0 of 25, 99.55 %, $2.93/kg

(c) Keep the feedforward and the gain of 0.05. Run once with the integral time at 6 h and once at 12 h, pressing Reset before each. Which passes, and which setting decides it? *Checkpoint:* 6 h: not met, 5 of 25, 99.49 %; 12 h: met, 99.52 %, $2.89/kg

9.10 Application area · 3 min

In case 8 a tenth of the extractant's sites are lost at hour 6. Put in order the three signals that show it, with the hour each first does: the saponified share read from the saponifier's pH, the pH of stage 1, and the laboratory's recovery. Then explain why the saponification loop, acting correctly on its own measurement, makes the recovery worse, and name one corrective move an engineer could make before the laboratory confirms the loss.

9.11 Application area · 3 min

A plant manager asks for 99.9 % neodymium at 97 % recovery from the shipped cascade. Using Figure 9.3 and the sweep of Section 9.4, say why no setting of the ratios meets it within the equipment limits, name the two ways past those limits, and say what the model leaves out that an engineer must weigh before either.

9.12 Review the agent · 5 min

An AI assistant reviewing the challenge writes: "The suggested settings leave the recovery loop in Man. Close it too, at Recovery gain 1 per point and a 25 h Recovery integral time, so that both products are under feedback. Two loops always hold the specification better than one, and the reagent cost will barely change." Find the flaw, and say how you would check it in the app.