



# Distillation Diagnostics

An Engineer's Guidebook

Henry Z. Kister

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## An Engineer's Guidebook

**Henry Z. Kister**

*Fluor Corporation, USA*

**AIChE**   
The Global Home of Chemical Engineers

**WILEY**

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*To my son, Abraham, and my daughter, Helen, who have been my love, blessings, and the lighthouses illuminating my path*



# Contents

**Preface**     **xv**

**List of Acronyms**     **xix**

**Acknowledgments**     **xxi**

---

**1. Troubleshooting Steps** **1**

- 
- 1.1 Causes of Column Malfunctions     2
  - 1.2 Column Troubleshooting – a Case History     4
  - 1.3 Strategy for Troubleshooting Distillation Problems     6
  - 1.4 Dos and Don'ts for Formulating and Testing Theories     12
  - 1.5 Learning to Troubleshoot     17
  - 1.6 Classification of Column Problems     18

---

**2. Troubleshooting for Flood** **21**

- 
- 2.1 Flooding: The Most Common Tower Throughput Limitation     21
  - 2.2 Flood Mechanisms in Tray and Packed Towers     22
  - 2.3 Flood and Flood Mechanism Determination: Hydraulic Analysis     25
  - 2.4 Operating Window (or Stability) Diagrams     27
  - 2.5 Flood Point Determination: Field Testing     29
  - 2.6 Flood Point Determination in The Field: The Symptoms     31
  - 2.7 Flood Mechanism Determination: Vapor and Liquid Sensitivity Tests     43
    - 2.7.1 Extension of Vapor/Liquid Sensitivity Tests to Complex Fractionators     44
  - 2.8 Gaining Insight into The Cause of Flood From dP Versus Vapor Rate Plots     46
  - 2.9 Diagnosing Floods that Give Small dP or No dP Rise     51
    - 2.9.1 Flood Diagnosis in a Chemical Vacuum Tower with no dP Rise     53
    - 2.9.2 Flood Diagnosis in Packed Pumparound (PA) Beds     54
  - 2.10 Foam Flooding Symptoms and Testing     55
  - 2.11 Downcomer Unsealing Floods at Low Liquid Loads     64
  - 2.12 Channeling-Induced Premature Floods at High Liquid Loads     66
  - 2.13 Floods by High Base Level or Entrainment From the Tower Base     67
  - 2.14 Troubleshooting Intermediate Component Accumulation     71
  - 2.15 Troubleshooting Liquid Side Draw Bottlenecks     74
  - 2.16 Twelve Useful Rules of Thumb     77

### 3. Efficiency Testing and Separation Troubleshooting 81

---

- 3.1 Efficiency Testing for Troubleshooting 81
  - 3.1.1 Purpose and Strategy of Efficiency Testing for Troubleshooting 81
  - 3.1.2 Planning and Execution of Efficiency Testing for Troubleshooting 83
  - 3.1.3 Preparations for Efficiency Testing 85
  - 3.1.4 Last-Minute Preparations 97
  - 3.1.5 The Test Day(s) 100
  - 3.1.6 Processing the Results 100
  - 3.1.7 Determining Hydraulic Loads 113
  - 3.1.8 Hennigan's Rules 115
- 3.2 Diagnosing Poor Separation 116
  - 3.2.1 Troubleshoot for Process Leaks 116
  - 3.2.2 Troubleshoot for Tray Weeping 120
  - 3.2.3 Diagnosing Side Draw Liquid Starvation 122
  - 3.2.4 Diagnosing Once-Through Reboiler Liquid Starvation 124
  - 3.2.5 Troubleshoot for Liquid in Vapor Side Draws 125
  - 3.2.6 Troubleshoot for Missing or Damaged Trays or Packing 125
  - 3.2.7 Poor Material Balance Control Produces High Impurities 127
  - 3.2.8 Limitations on Reflux or Reboil Generation 128
  - 3.2.9 Control Instability Increases Impurities 129
  - 3.2.10 Impurities and Contaminants Affecting Azeotroping and Product Purities 129
  - 3.2.11 Absorption of Sparingly Soluble Gases Affecting Product Purity and Downstream Venting 130
  - 3.2.12 Reactions and Contaminants Can Affect Product Purity 130

### 4. Diagnosing Packed Tower Maldistribution 135

---

- 4.1 Diagnosing Packing Maldistribution: an Overview 135
- 4.2 Expected Packing HETPS 137
- 4.3 Small-Scale Versus Large-Scale Maldistribution: Do They Equally Raise HETP? 137
- 4.4 By How Much does Maldistribution Reduce Packing Efficiency? 138
- 4.5 Diagnosing Packing and Distributor Plugging 141
- 4.6 Troubleshooting for Distributor, Collector, and Parting Box Overflows 145
  - 4.6.1 Overflows and Their Impact 145
  - 4.6.2 Overflows in Distributors, Redistributors, and Parting Boxes 145
  - 4.6.3 Overflows in Collectors 148
  - 4.6.4 Overflows due to Plugging, Foaming, Impingement 150
- 4.7 Troubleshooting Maldistribution at Turndown 150
- 4.8 Troubleshooting Distributor Out-of-Levelness 151
- 4.9 Troubleshooting Distributor Feeds 153
- 4.10 Evaluation of Distributor Irrigation Quality 154
- 4.11 Troubleshooting for Vapor Maldistribution 158
- 4.12 Vapor Maldistribution in The Feed Zone to Refinery Vacuum Towers 161
- 4.13 Troubleshooting Flashing Feeds Entry 164
- 4.14 Troubleshooting Notched Distributors 167
- 4.15 Troubleshooting Spray Nozzle Distributors 169

4.16	Distributor Water Tests	173
4.16.1	Gravity Distributor Water Tests in the Supplier Shop	173
4.16.2	Gravity Distributor Water Tests In situ	175
<b>5.</b>	<b>Qualitative Gamma Scans Troubleshooting: The Basic Diagnostics Workhorse</b>	<b>179</b>
5.1	Gamma-Ray Absorption	179
5.2	Qualitative Gamma Scans	180
5.2.1	What Do Qualitative Gamma Scans Show in Tray Towers	181
5.2.2	Entrainment versus Weeping	184
5.2.3	What Do Qualitative Gamma Scans Show in Packed Towers	185
5.2.4	Stationary Monitoring (“Time Studies”)	190
5.2.5	What is in the Tower Inlet/Outlet Pipes?	191
5.3	Gamma Scans Pitfalls and Watchouts	191
5.4	Gamma Scan Shortcuts: Cost Versus Benefits	195
5.4.1	Dry (Empty Column) Scans: Yes or No?	195
5.4.2	Initial Operation Scans (Often Referred to as Baseline Scans): Yes or No?	196
5.4.3	Changed Conditions Monitoring Scans	196
5.4.4	Performing Downcomer Scans: Yes or No?	196
5.4.5	Can We Learn More about the Bottleneck?	197
5.4.6	Would Our Proposed Modification Solve the Problem?	197
5.5	Some Applications of Qualitative Gamma Scans	197
5.5.1	Distinguishing Fact from Interpretation	197
5.5.2	Is the Flood a Jet Flood?	199
5.5.3	Diagnosing the Correct Flood Mechanism and Arriving at the Effective Fix	199
5.5.4	Does the Tower Flood Occur at the Expected Location?	202
5.5.5	Foaming or Not Foaming?	204
5.5.6	Insights into Fouling Patterns/Shortcuts Lead to Misinterpretations	209
5.5.7	Multipass Trays Maldistribution	211
5.5.8	Fouling-Induced Maldistribution in 2-Pass Trays	212
5.5.9	Weeping/Dense Liquid/Missing Trays	214
5.5.10	Plugging, Flood, and Fouling Monitoring in a Packed Bed	216
5.5.11	More Plugging and Flooding in a Packed Bed	221
5.5.12	Flood due to Crushed or Damaged Random Packings	221
5.5.13	Missing Random Packings	223
5.5.14	Displaced and Damaged Structured Packings	225
5.5.15	Flooding Induces Channeling in Deep Vacuum Packed Tower	225
5.5.16	Distributor Overflow and Maldistribution	228
5.5.17	Overflow, Entrainment, and Maldistribution from Flashing Feed Distributor	230
5.5.18	Tower Overfill due to Excessive Pressure Drop in Kettle Reboiler Piping	231
5.5.19	Is a Collector (or Chimney Tray) Overflowing? Does this Initiate Flooding?	233
5.5.20	Damaged or Dry Trays?	233

<b>6.</b>	<b>Advanced Radioactive Techniques for Distillation Troubleshooting</b>	<b>237</b>
6.1	Quantitative Multi-Chordal Tray Gamma Scans Analysis	237
6.1.1	Harrison's Method for Froth Height and Flood Determination	238
6.1.2	Application of Harrison's Method Prevents Unnecessary Shutdown	241
6.1.3	Entrainment Index	243
6.1.4	Kistergrams and their Application	244
6.1.5	Froth (or Spray) Density and Liquid Head Determination	246
6.1.6	Quantitative Analysis for High-Capacity Trays with Truncated Downcomers	248
6.1.7	Scan Chord Selection	251
6.2	Quantitative Analysis of Packing Gamma Scans	256
6.2.1	Packed Tower Quantitative Analysis Techniques	256
6.2.2	Flooding at the Bottom or Missing Packing Tower at the Top?	258
6.2.3	Dense Grid or Flooding/Coking in a Wash Bed?	259
6.2.4	Good or Bad Distribution Quality?	260
6.3	Neutron Backscatter Techniques Application	261
6.3.1	Detecting Flood and Seal Loss in Downcomers	263
6.3.2	Distinguishing Dry from Full Downcomers	264
6.3.3	Detecting Maldistribution in a Kettle Reboiler	264
6.4	CAT Scans	266
6.4.1	Identifying Unexpected Flashing in Reflux Distributor	268
6.4.2	Diagnosing Unexpected Parting Box Overflow	269
6.4.3	Monitoring Coking in a Refinery Vacuum Tower	269
6.5	Tracer Techniques	273
6.5.1	Measuring Flow Rates, Internal Leaks, and Spray Entrainment in Refinery Vacuum Tower	274
6.5.2	Observing Downward Vapor Flow in Packed Bed	275
6.5.3	Quantitative Determination of Entrainment from a Kettle Reboiler	277
6.6	Slant Scanning	280
6.7	Useful Case Histories Literature	283
6.7.1	Qualitative Gamma Scans of Trays	283
6.7.2	Qualitative Gamma Scans of Packings	284
6.7.3	Gamma Scans of Points of Transition and Inlet and Outlet Lines	284
6.7.4	Quantitative Gamma Scans of Trays	284
6.7.5	Quantitative Gamma Scans of Packings	285
6.7.6	Neutron Backscatter	285
6.7.7	CAT Scans	285
6.7.8	Tracer Technique	285
<b>7.</b>	<b>Thermal and Energy Troubleshooting</b>	<b>287</b>
7.1	Wall Temperature Surveys	287
7.1.1	Dos and Don'ts for Temperature Surveys	288
7.1.2	Diagnosing Internals Damage	291
7.1.3	Diagnosing Packing Maldistribution	293
7.1.4	Time Study for Diagnosing the Nature of Instability	296
7.1.5	Diagnosing an Unexpected Second Liquid Phase	301
7.1.6	Diagnosing Poor Mixing in a Packing Distributor	304
7.1.7	Diagnosing Flashing in a Packing Distributor	305

7.1.8	Identifying Various Boiling Regions in a Kettle Reboiler	307
7.1.9	Identifying Plugging Zone in a Ladder Pipe Distributor	308
7.1.10	Identifying Uneven Quench Distribution in a Bottom Sump	309
7.1.11	Identifying Flood	309
7.2	Thermal Camera (Thermography) Applications	310
7.2.1	Dos and Don'ts for Thermal Camera Imaging	310
7.2.2	Diagnosing Packing Maldistribution	312
7.2.3	Diagnosing Vapor or Liquid Maldistribution in Insulated Towers	312
7.2.4	H <sub>2</sub> S Amine Absorber Temperature Profile, Foam, and Flooding	314
7.2.5	Temperature Bulges in CO <sub>2</sub> Amine Absorbers	316
7.2.6	Excessive Liquid Levels	318
7.2.7	Diagnosing a Flood due to Bottom Baffle Malfunction	318
7.2.8	Diagnosing a Damaged Draw Pan	321
7.2.9	Liquid Levels in Condensers and Reflux Drums	322
7.2.10	Troubleshooting Condensers and Reboilers	322
7.2.11	Thermal Video Diagnoses Cause of Pressure Instability	327
7.3	Energy Balance Troubleshooting	329
7.3.1	Energy Balance Application for Correctly Validating Simulations and Correctly Diagnosing Tower Problems	329
7.3.2	Energy Balance Troubleshooting to Detect Internal Leaks	332
7.3.3	Energy Balance Troubleshooting to Eliminate Overflows or Leaks in the Upper Parts of Chimneys	335
7.3.4	Energy Balance Troubleshooting of a Two-Compartment Chimney Tray	337
7.3.5	Leaks from Heat Exchangers: The Role of Mass and Energy Balance in their Troubleshooting	338

## 8. Point of Transition Troubleshooting: You Do Not Need an Expert, You Need a Sketch

343

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8.1	Guidelines for Points of Transition Sketches	343
8.2	Flashing Feed Entry Causing a 12-Year Bottleneck	344
8.2.1	Upward Component of Flashing Feed Entry Causes Damage and Flooding	346
8.3	Feed Maldistribution to 4-Pass Trays Causing Poor Separation	347
8.4	Feed Pipes Blocking Liquid Entrance to Downcomers	349
8.5	Draw Sump Blocking Liquid Entrance to Downcomers	350
8.6	Unsealed Downcomers or Overflow Pipes Can Lead to Premature Flood	351
8.7	Excessive Downcomer Submergence Can Lead to Premature Flood	353
8.8	“Leak-Proof” Chimney Trays in an FCC Main Fractionator	354
8.9	More “Leak-Proof” Chimney Trays	355
8.10	Hydraulic Gradients Generating Chimney Tray Overflows	358
8.11	Look for the Possibility of a System Limit Setting In	359
8.12	Vapor Maldistribution at the Tower Base and Chimney Tray	362
8.13	Entrainment from a Gallery Flashing Feed Distributor	364
8.14	Vapor Impinging on Liquid at the Tower Base	365
8.15	More Vapor Impinging on Liquid at the Tower Base	366
8.16	V-Baffles Produce Unexpected Flow Pattern at the Tower Base	368
8.17	Baffling Tower Base Baffles	370

8.18	Liquid Maldistribution at a Feed or a Product Draw	372
8.19	Poor Solvent/Reflux Mixing Gives Poor Separation in Extractive Distillation (ED) Tower	376
8.20	Two Seemingly Well-Designed Pieces May Not Work Well When Combined	378
8.21	Another Two Seemingly Well-Designed Pieces that Did Not Work Well When Combined	380
8.22	Liquid Maldistribution of Internal Reflux Below a Side Draw	381
8.23	Would You Believe this was a Real Troubleshooting Assignment?	381

**9. Making the Most of Field Data to Analyze Events and Test Theories 383**

---

9.1	Event Timing Analysis	383
9.1.1	General Application Guidelines for Event Timing Analysis	383
9.1.2	Diagnosing the Unexpected Cause of Off-Spec Product	384
9.1.3	Diagnosing Reboiler Surging	386
9.1.4	Loss of Condensate Seal in a Demethanizer Reboiler	387
9.1.5	Figuring out Hot Vapor Bypass (HVB) Instability	388
9.1.6	Can Multiple Steady States Occur in a Reboiler System?	392
9.1.7	Can a Plugged Packing Distributor Generate Two Steady States?	395
9.1.8	What Caused Tray Damage in Refinery Atmospheric Crude Fractionator?	397
9.1.9	Event timing Analysis of Startup Instability Leads to Improved Startups	401
9.2	Field Testing	404
9.2.1	General Application Guidelines for Field Testing	404
9.2.2	Narrowing Down from 12 Theories to the Root Cause	405
9.2.3	Making Sense of Plant Data and Operation Experience in a Packed Tower	407
9.2.4	Downcomer Unsealing: A Correct Diagnosis Brings a Correct Cure	409
9.2.5	Well-Targeted Field Testing Brings a Correct Diagnosis and an Unexpected Simple Solution	413
9.2.6	Field Testing Brings a Correct Diagnosis where Engineering Analysis Failed	419
9.2.7	Operating Team Observations and Field Testing Brings a Correct Diagnosis where Engineering Analysis Failed	421
9.2.8	Lessons Learnt from Packed Tower History and Testing	422

**10. Troubleshooting by Inspection 425**

---

10.1	Safety Precautions for Work Inside The Column	426
10.2	Troubleshooting Starts with Preventive Practices During Installation	427
10.2.1	Preinstallation Dos and Don'ts for Tray Columns	427
10.2.2	Preinstallation Dos and Don'ts for Packed Towers	429
10.2.3	Removal of Existing Trays and Packings	432
10.2.4	Tray Installation	433
10.2.5	Dry versus Wet Random Packing Installation	435
10.2.6	Dos and Don'ts for Random Packing Installation	436
10.2.7	Dos and Don'ts for Structured Packing Installation	438

10.2.8	Some Considerations for Towers Out of Service for a Time	442
10.3	Tower Inspection: What to Look For	442
10.3.1	Strategy	442
10.3.2	Should the Tower be Entered at the Turnaround?	444
10.3.3	Inspector's Checklist	445
10.3.4	Packing Distributor Checks	449
10.3.5	Packing Assembly Checks – Existing Columns	454
10.3.6	Untightened Nuts, Bolts, Clamps, and Downcomer Panel Assembly	455
10.3.7	Tray Assembly	457
10.3.8	Feeds/Draws Obstruction, Misorientation, and Poor Assembly	472
10.3.9	Cleanliness of Internals	481
10.3.10	Final Inspection	482
10.3.11	Externals Inspection	484

**References 487**

**Index 513**



# Preface

For over a century, distillation, the “king of separations,” has been by far the most common separation technique in refineries, petrochemical, chemical, and natural gas plants and will remain the prominent separation technique in the foreseeable future. Every chemical process has a reaction section and a separation section, with distillation usually dominating the latter. Three percent of the world’s energy is tied in distillation. The dominance of distillation stems from its low capital compared to other techniques, scaling up well, and not introducing an extra agent that requires removal later.

The past half-century has seen tremendous advances in distillation technology. High-speed computers revolutionized the design, control, and operation of distillation towers. Invention and innovation in tower internals enhanced tower capacity and efficiency beyond previously conceived limits. Despite all these advances, the distillation failure rate has been on the rise and growing.

The great progress has not bypassed the distillation troubleshooting tools. Reliable pressure drop measurements, gamma scans, laser-guided pyrometers, and thermal cameras are tools that not-so-long-ago troubleshooters would only dream of. But again, despite this great progress, the ability of engineers to effectively diagnose and solve plant problems appears to be on the decline.

Why are we losing the problem-solving war? Despite the prominence of distillation in industry, academics prefer to focus on more trendy “cool” fields. The *Chemical Processing* editorial from March 2018 decries the lack of practical research on distillation in chemical engineering departments. In industry, mergers, cost-cuts, and the pandemic have retired most of the experienced troubleshooters and thinly spread the others. The literature offers little to bridge the experience gap. In the era of information explosion, databases, and computerized searches, finding the appropriate information in due time has become like finding a needle in an ever-growing haystack. To locate a useful reference, one needs to click away a huge volume of wayward leads.

The result is that distillation diagnostics is becoming a lost art. Engineers are given little practical guidance on how to correctly apply and get the most out of the diagnostic tools, so the tools are utilized sparsely and only on a very basic level. Problems are left undiagnosed or misdiagnosed. Solutions based on misdiagnoses are doomed to fail. Lacking a proper diagnosis, some engineers turn to so-called experts and end up being sold a bill of goods or sledge hammers to crack nuts. It is not uncommon to see an expensive new tower solving a problem that can be solved by replacing a faulty feed pipe for nickels and dimes.

The trend away from diagnosis could not have come at a worse time. We are being alarmed by adverse climate and environmental changes, are recognizing the importance of saving our planet, and are striving for a cleaner tomorrow. The engineering community is engaged in developing new technologies for reducing the carbon footprint, aspiring to “zero carbon” energy transition, carbon capture, and replacement of fossil fuels by renewables. Unfortunately, many of these technologies require massive new equipment and additional energy usage, neither of which helps achieve the prescribed goals of reducing carbon footprints.

One simple technology is being forgotten: correct diagnostics. In his book *Process Engineering for a Small Planet* (Wiley, 2010), Norman Lieberman demonstrates how poor troubleshooting and wasteful practices guzzle energy, generate carbon dioxide, and waste the earth's precious resources. In Chapter 1 of his book, Lieberman describes a case in which a correct diagnosis would have led to modifying trays and downcomers in a fractionator, and adding mist injection to the overhead compressor, which could have circumvented erecting a giant new fractionator with a new oversized overhead compressor. Just the energy usage due to the compressor oversizing was estimated to waste the amount of crude oil that 400 families use daily. Fabricating the new steelwork and structures consumed additional immense amounts of energy and emitted tons of carbon dioxide, all of which were unnecessary.

In another example (Chapter 2), four uninstalled tray manways induced the escape of a large quantity of diesel in the bottom of a crude fractionator. To vaporize it took 40 MM Btu/h, which is equivalent to the fuel consumption of a quarter of a million 400 horsepower motor cars driving around the clock. Lieberman diagnosed the problem and recommended re-installing the manways, but the implemented solution was to build a new larger heater, wasting not only energy but also concrete, steel, nickel, and chromium needed for the heater and its tubes, the mining and production of which only adds to the carbon footprint. Lieberman presents a book full of similar experiences.

This book builds up on Lieberman's initiative with a multitude of distillation examples. In one (Chapter 8), a packed stripper was erected to turn a wastewater stream into cooling water makeup. The entering wastewater poured above the oversized hats of the liquid distributor. In the little space between the hats, the vapor sped up and carried the liquid over. Two different consultants studied the problem. The first offered an incorrect diagnosis and a failed fix. The second performed an extensive simulation study, incorrectly concluding that a larger-diameter tower was needed. The plant gave up and junked the tower, with the water still going to the sewer. Good troubleshooting and hat or feed pipe modifications costing nickels and dimes would have saved precious water, reduced sewage, and prevented turning a good tower into scrap metal.

My previous book, *Distillation Troubleshooting*, focused on case studies and the lessons learned. This book goes further, providing the tools to correctly diagnose and solve operating problems, avoid the wasteful practices, and make the most of existing equipment. It travels through the multitude of invaluable tools available for diagnosing tower problems, providing application guidelines derived from the school of hard knocks. The book describes what each tool can and cannot do, provides insight into how different ideas and theories can be tested using these tools, shares the experience of how to correctly interpret the results provided by each technique, and guides troubleshooters to a multitude of additional tests that they can perform to get closer to a correct diagnosis and an effective fix. The only other place where its information is available is in the heads of experienced troubleshooters.

The techniques are illustrated with real case studies from my experience of 45 years as well as from the experiences of many of my colleagues and those presented in the literature. Every technique is accompanied with an extensive list of "do and don'ts" based on the author's and industry's experience. This guidance can make the difference between success and failure, and between good and bad results.

The case studies and lessons learned are described to the best of the author's and the contributors' knowledge and in good faith, but may not always correctly reflect the problems and solutions. Many times I thought I had the answer, only to be humbled by a new light or another experience later. The experiences and lessons in this book are not meant to be followed blindly. They are meant to be stories told in good faith and to the best of knowledge and understanding of the author or contributors, which hopefully give troubleshooters ideas to think about. We welcome any comments that either affirm or challenge our perception or understanding.

If an incident that happened in your plant is described, you may notice that some details could have changed. Sometimes, this was done to make it more difficult to tell where the incident occurred. At times, it was done to simplify the story without affecting the key lessons. Sometimes, it happened long ago and memories of some details faded away. Sometimes, and most likely, this incident did not happen in your plant at all. Another plant had a similar incident.

The book emphasizes the “do-it-yourself” approach. It endeavors to place the tools and good application guides in the practitioner’s hands, eliminating reliance on the so-called experts. People who attend my seminar classes come out with knowledge on how to apply the tools and express concern about forgetting this knowledge over the years. They express wishes to have a book like this so they can refresh their memories in the future. This is that book. It is my hope that the book will lead the readers to correct diagnostics and effective fixes to tower problems, and to trouble-free operation.

If you can successfully solve only one problem using the techniques in this book, the savings to your company will be enough to pay for thousands of copies of the book. But far beyond the savings, you will be taking a step in the right direction of saving our small planet and ushering in a cleaner tomorrow.

HENRY Z. KISTER

*May 2024*



# List of Acronyms

AIChE	American Institute of Chemical Engineers
AGO	Atmospheric gas oil
API	American Petroleum Institute
ASTM	American Society for Testing and Materials
ATB	Atmospheric tower bottoms
BPA	Bottom pumpharound
BPD	Barrels per day
BPH	Barrels per hour
BPSD	Barrels per stream day
BTM	Bottom
CAT	Computer aided tomography
CFD	Computational fluid dynamics
COND	Condensate
COT	Coil outlet temperature
COV	Coefficient of variation
CW	Cooling water
dP	Differential pressure (pressure drop)
ED	Extractive distillation
FCC	Fluid catalytic cracking
FI	Flow indicator
FRI	Fractionation Research Inc (or Institute)
FRN	Full range naphtha
HAZOP	Hazard and operability analysis
HCO	Heavy cycle oil
HCU	Hydrocracker Unit
HETP	Height equivalent to a theoretical plate
HF	Hydrogen fluoride
HN	Heavy naphtha
HP	High pressure
HSE	Health, Safety, and Environment
HVB	Hot vapor bypass
HVGO	Heavy vacuum gas oil
ID	Internal diameter
IPA	Intermediate pumpharound
IR	Infrared
LCO	Light cycle oil
LED	Light Emitting Diode
LI	Level indicator

## xx List of Acronyms

LN	Light naphtha
LNG	Liquefied natural gas
LV	Liquid volume
LVGO	Light vacuum gas oil
MBPD	Thousand BPD
MCB	Main Column Bottoms
MDEA	Methyldiethanol amine
MEA	Monoethanol amine
MP	Medium pressure
MPA	Mid pumparound
MRI	Magnetic resonance imaging
MSCFH	Standard cubic feet per hour $\times$ 1000
OSHA	The Occupational Safety and Health Administration
OVHD	Overhead
P&ID	Process and instrumentation diagram
PA	Pumparound
PFD	Process flow diagram
PI	Pressure indicator
PPR	Polypropylene return
STM	Steam
TAR	Turnaround
TI	Temperature indicator
TPA	Top pumparound
TRC	Temperature recorder/controller
VCFC	Vapor crossflow channeling
VLE	Vapor-liquid equilibrium
VLLE	Vapor-liquid-liquid equilibrium
VTB	Vacuum tower bottoms

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## Troubleshooting Steps

*“In our industry we have many more troublemakers than troubleshooters”*

—(Professor Zarko Olujic, presentation to AIChE)

Troubleshooting, revamping, and eliminating waste offer huge benefits in reducing capital investment, downtime, carbon dioxide emissions, and energy consumption. Unfortunately, the attention paid to this resource in the energy-transition era has been too little to reflect its tremendous potential.

A multitude of examples in Norman Lieberman’s book *Process Engineering for a Small Planet* (287) demonstrate how poor troubleshooting leads to wasteful practices that guzzle energy, increase the carbon footprint, and deplete the earth’s precious resources. The chemical process industry (CPI) has been paying dearly for downtime, lost production, substandard product quality, raw material problems, safety and environmental issues, and excessive energy consumption due to ineffective troubleshooting of abnormal situations (16). In 1997, a consortium estimated that the annual loss for the CPI due to ineffective abnormal situation management was US \$10 billion (16).

Correct diagnosis is at the heart of problem identification and implementing a correct, cost-effective solution. An incorrect diagnosis breeds ineffective solutions that prolong the agony and escalate the costs. This chapter focuses on the systematic diagnostic steps. The following chapters will describe the multitude of techniques that have been found effective for diagnosing distillation problems.

A well-known sales axiom states that 20% of the customers bring in 80% of the business. A sales strategy tailored for this axiom concentrates the effort on these 20% without neglecting the others. Distillation diagnostics follow an analogous axiom. A person engaged in diagnosing column problems must develop a good understanding of the factors that cause the vast majority of column malfunctions and the techniques available for narrowing in on their root causes. While a good knowledge and understanding of the broader field of distillation is beneficial, the diagnosis often requires only a shallow knowledge of this broader field.

It is well accepted that diagnosing problems is a primary job function of operating engineers, supervisors, and process operators. Far too few realize that distillation diagnostics start at the design phase. Any designer wishing to achieve a trouble-free column design and operation must be as familiar with diagnostic techniques, many of which are applicable during the design (and more so at the revamp) phase.

Expansive surveys of the causes of column malfunctions were described in previous studies (196, 198, 201). Abundant resources are available to distinguish good from poor practices and to

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*Distillation Diagnostics: An Engineer’s Guidebook*, First Edition. Henry Z. Kister.

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avoid and overcome troublesome design and operations (e.g., 192, 285, 286, 293). What is often missing is the connect. How does one link field observations with the known tower malfunctions in order to develop an effective remedy? This book is all about the link: translating field observations into diagnoses and cures.

Following a brief survey of the primary causes of tower malfunctions, this chapter looks at the basic diagnostic: the systematic strategy for diagnosing distillation problems and the dos and don'ts for formulating and testing theories. Finally, it reviews the techniques for testing these theories and for focusing on the most likely root cause.

### 1.1 CAUSES OF COLUMN MALFUNCTIONS

Close to 1500 case histories of malfunctioning columns were extracted from the literature and abstracted in Ref. 201. Most of these malfunctions were analyzed in Ref. 198 and classified according to their principal causes. A summary of the common causes of column malfunctions is provided in Table 1.1. If one assumes that these case histories make up a representative sample, then the analysis presented below has statistical significance. Accordingly, Table 1.1 can provide a useful guide to the factors most likely to cause column malfunctions and can direct troubleshooters toward the most likely problem areas.

The general guidelines in Table 1.1 often do not apply to a specific column or even plant. For instance, foaming is not high up in the table; however, in amine absorbers, it is a very common trouble spot. The author therefore warns against blindly applying these guidelines in any specific situation.

The total number of cases in each category is shown in the column headed "Cases." The other three columns show the split of these cases according to industry categories, namely refining, chemicals, and olefins/gas plants.

An analysis of Table 1.1 suggests the following:

- Plugging, tower base, tower internals damage, instrument and control problems, startup and/or shutdown difficulties, points of transition (tower base, packing distributors, intermediate draws, feeds), and assembly mishaps are the major causes of column malfunctions. They make up nearly two-thirds of the reported incidents. Familiarity with these problems, therefore, constitutes the "bread and butter" of distillation and absorption troubleshooters.
- Primary design is a very wide topic, encompassing vapor-liquid equilibrium, stage-to-stage calculations, reflux-stages relationship, unique features of multicomponent distillation, tray and packing capacities and efficiencies, scale-up, column diameter and height determination, type of tray, and size and material of packing. This topic represents most of our distillation know-how and occupies the bulk of most of the distillation texts (e.g., 34, 193, 299, 451, 492). While this topic is paramount for designing and optimizing distillation columns, it plays only a minor role in operations and troubleshooting in distillation. As shown in Table 1.1, only one column malfunction among fourteen is incurred in the primary design. The actual figure is probably higher for a first-of-its-kind separation, but lower for an established separation. Due to the bulkiness of this topic and its low likelihood to cause malfunctions, and due to the excellent coverage that the topic receives in several texts (e.g., 34, 193, 299, 451, 492), it is only lightly touched upon in this book.
- The above statements must not be interpreted to suggest that troubleshooters need not be familiar with the primary design. Quite the contrary. A good troubleshooter must have a solid understanding of primary design because it provides the foundation of distillation

**Table 1.1** Most common causes of column malfunctions. (From Kister, H. Z., Transactions of the Institution of Chemical Engineers, 81, Part A, p. 5, January 2003. Reprinted Courtesy of the Institution of Chemical Engineers in the UK.)

No.	Cause	Total cases	Refinery cases	Chemical cases	Olefins/gas cases
1	Plugging, coking	<b>121</b>	68	32	16
2	Tower base and reboiler return	<b>103</b>	51	22	11
3	Tower internals damage (excluding explosion, fire, implosion)	<b>84</b>	35	33	6
4	Abnormal operation incidents (startup, shutdown, commissioning)	<b>84</b>	35	31	12
5	Assembly mishaps	<b>75</b>	23	16	11
6	Packing liquid distributors	<b>74</b>	18	40	6
7	Intermediate draws (including chimney trays)	<b>68</b>	50	10	3
8	Misleading measurements	<b>64</b>	31	9	13
9	Reboilers	<b>62</b>	28	13	15
10	Chemical explosions	<b>53</b>	11	34	9
11	Foaming	<b>51</b>	19	11	15
12	Simulations	<b>47</b>	13	28	6
13	Leaks	<b>41</b>	13	19	7
14	Composition control difficulties	<b>33</b>	11	17	5
15	Condensers that did not work	<b>31</b>	14	13	2
16	Control assembly	<b>29</b>	7	14	7
17	Pressure and condenser controls	<b>29</b>	18	3	2
18	Overpressure relief	<b>24</b>	10	7	2
19	Feed inlets to tray towers	<b>18</b>	11	3	3
20	Fires (excluding explosions)	<b>18</b>	11	3	4
21	Intermediate component accumulation	<b>17</b>	6	4	7
22	Chemicals release to the atmosphere	<b>17</b>	6	10	1
23	Subcooling problems	<b>16</b>	8	5	1
24	Low liquid loads in tray towers	<b>14</b>	6	2	3
25	Reboiler and preheater controls	<b>14</b>	6	–	5
26	Two liquid phases	<b>13</b>	3	9	1
27	Heat integration issues	<b>13</b>	5	2	6
28	Poor packing efficiency (excluding maldistribution/support/hold-down)	<b>12</b>	4	3	2
29	Troublesome tray layouts	<b>12</b>	5	2	–
30	Tray weep	<b>11</b>	6	1	3
31	Packing supports and hold-downs	<b>11</b>	4	2	2

know-how. However, the above statements do suggest that in general, when troubleshooters examine the primary design for the cause of a column malfunction, they have less than one chance out of ten of finding it there.

## 1.2 COLUMN TROUBLESHOOTING – A CASE HISTORY

In Sections 1.3 and 1.4, the systematic approach recommended for diagnosing distillation problems is presented. The recommended sequence of steps is illustrated with reference to the case history described below.<sup>1</sup>

*The following story is not a myth; it really happened. One morning as I sat quietly at my desk in corporate headquarters, the boss dropped by to see me. He had some unpleasant news. One of the company's refinery managers was planning to visit our office to discuss the quality of some of the new plants that had been built in his refinery. As an example of how not to design a unit, he had chosen a new gas plant for which I had done the process design. The refinery manager had but one complaint: "The gas plant would not operate."*

*I was immediately dispatched to the refinery to determine which aspect of my design was at fault. If nothing else, I should learn what I did wrong so as not to repeat the error.*

*Upon arriving at the refinery, I met with the operating supervisors. They informed me that, while the process design was fine, the gas plant's operation was unstable because of faulty instrumentation. However, the refinery's lead instrument engineer would soon have the problem resolved.*

*Later, I met with unit operating personnel. They were more specific. They observed that the pumparound circulating pump (see Figure 1.1a) was defective. Whenever they raised hot oil flow to the debutanizer reboiler, the gas plant would become destabilized. Reboiler heat-duty and reflux rates would become erratic. Most noticeably, the hot-oil circulating pump's discharge pressure would fluctuate wildly. They felt that a new pump requiring less net positive suction head was needed.*

*Both these contradictory reports left me cold. Anyway, the key to successful troubleshooting is personal observation. So I decided to make a field test.*

*When I arrived at the gas plant, both the absorber and debutanizer towers were running smoothly but not well. Figure 1.1b shows the configuration of the gas plant. The debutanizer reflux rate was so low it precluded significant fractionation. Also, the debutanizer pressure was about 100 psi below design. Only a small amount of vapor, but no liquid, was being produced from the reflux drum. Since the purpose of the gas plant was to recover propane and butane as a liquid, the refinery manager's statement that the gas plant would not operate was accurate.*

*As a first step, I introduced myself to the chief operator and explained the purpose of my visit. Having received permission to run my test, I switched all instruments on the gas-plant control panel from automatic over to local manual. In sequence, I then increased the lean oil flow to the absorber, the debutanizer reflux rate, and the hot-oil flow to the debutanizer reboiler.*

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<sup>1</sup> Reproduced from Norman P. Lieberman, *Troubleshooting Process Operations* (4th ed., PennWell Books, Tulsa 2009). This case history is a classic example of how to perform a systematic troubleshooting investigation. The permission of PennWell Books and Norman P. Lieberman for reproducing this material is gratefully acknowledged.



*The gas plant began to behave properly. The hot-oil circulating pump was putting out a steady flow and pressure. Still, the plant was only producing a vapor product from the debutanizer reflux drum. This was because the debutanizer operating pressure was too low to condense the  $C_3$ – $C_4$  product. By slowly closing the reflux drum vapor vent valve, I gradually increased the debutanizer pressure from 100 psig toward its design operating pressure of 200 psig.*

*Suddenly, at 130 psig, the hot-oil flow to the debutanizer's reboiler began to waiver. At 135 psig the debutanizer pressure and the hot-oil flow plummeted. This made absolutely no sense. How could the debutanizer pressure influence hot-oil flow?*

*To regain control of the gas plant, I cut reflux to the debutanizer and lean-oil flow to the absorber. I was now back where I started. The thought of impending failure loomed.*

*I repeated this sequence twice more. On each occasion, all went well until the debutanizer pressure increased. By this time it was 3 a.m. Was it also time to give up and go home?*

*Just then, I noticed a commotion at the main fractionator control panel. The operators there stated that the fractionator was flooding again—for the third time that night. The naphtha production from the fractionator had just doubled for no apparent reason.*

*In every troubleshooting assignment there always occurs that special moment, the moment of insight. All of the bits and pieces fall into place, and the truth is revealed in its stark simplicity.*

*I cut the debutanizer pressure back to 100 psig and immediately the flooding in the main fractionator subsided. The operators then closed the inlet block valve to the hot-oil side of the reboiler and opened up a drain. Naphtha poured out instead of gas oil. This showed that the debutanizer reboiler had a tube leak.*

*Whenever the debutanizer pressure reached 130 psig, the reboiler pressure exceeded the hot-oil pressure. The relatively low-boiling naphtha then flowed into the hot oil and flashed. This generated a large volume of vapor that then backed hot oil out of the reboiler. The naphtha vapors passed on into the main fractionator and flooded this tower. Thus, the cause of the gas plant instability was neither a process design error, instrument malfunction, nor pumping deficiency. It was a quite ordinary reboiler tube failure.*

### 1.3 STRATEGY FOR TROUBLESHOOTING DISTILLATION PROBLEMS

In almost any troubleshooting assignment, it is desirable to solve a problem as fast as possible with the least expense. Surprisingly, this objective is often only partially achieved, the obstacle being a poor (often nonexistent) strategy for diagnosing the problem.

While devising a troubleshooting strategy, it is useful to think in terms of a “doctor and patient” analogy. The doctor’s strategy for diagnosing an illness is well established and easily understood by most people. Applying a similar strategy to diagnosing distillation problems often constitutes the most effective and least expensive course of action.

The sequence of steps listed below is often considered optimum for tackling a troubleshooting problem. It is based on the author’s experience as well as the experience of others (112, 116, 161, 166, 286, 293, 318, 411, 498). The step headings refer to the doctor and patient analogy. Actions described in Lieberman’s case history (Section 1.2) are referenced to demonstrate the optimum sequence of steps. A good troubleshooting strategy always proceeds stepwise, starting with the simple and obvious.