FIFTY YEARS OF FRI$_{(SM)}$ CONTRIBUTIONS

By

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Introduction/Background/History

In the early 1950's, Dr. Karl Hachmuth, of Phillips Petroleum Company, was studying the effectiveness of distillation devices and decided that too little was known for design engineers to properly predict distillation results. He concluded that research was needed on plant-scale equipment and also concluded that such research was too expensive for any one company. He then promoted the concept of cooperative research to technical representatives of other companies, who found the idea to be a good one. Mr. T.B. Hudson, of Phillips, did much of the organizational work that developed the structure of Fractionation Research, Inc. (FRI) and became its first President. By February 1, 1952, 15 companies had executed the FRI agreement and incorporation was completed on April 4, 1952. By the first stockholder meeting of November 10, 1952, there were 37 member companies.

From the start, FRI was organized as a corporation with a limited life unless extended by the membership. The first operation was envisioned as a 5 year research program. Since then, FRI's life has been extended, in periods of 3 or 5 years, because the members continued to see important research projects of interest to a large group of member companies. The research proposed for each extension is outlined in prospectus form for membership approval. Since the research is membership directed, the research program can and has been modified during an extension by vote of the Technical Advisory Committee.

Dr. E.H. Amick of Columbia University was the first Technical Director. He was followed by Mr. F.W. Winn. Mr. George Keller was employed in August, 1954, and became Technical Director in January, 1959. Dr. John Kunesh became Technical Director in October, 1984. Dr. G.X. Chen was employed in 1993 and served as Technical Director after retirement of Dr. John Kunesh in 2004. Mr. Michael Resetarits is the current Technical director.

In January, 1954, FRI entered into a contract with C.F. Braun & Co., Alhambra, California, to use their simulator. The first four foot (1.2m) diameter FRI column was contracted in March, 1954, and operations began September 30, 1954. An eight foot (2.4m) diameter extension to the research column was made in 1960. A four foot diameter high pressure column was added in 1963. Operations continued at C.F. Braun until November, 1989. The equipment was then moved to a site at Oklahoma State University, Stillwater, Oklahoma, and began operating in August, 1991.

Table 1 presents a listing of some key events that occurred during FRI’s history.

Organization

To oversee its business, FRI has a Board of Directors. Each member company may have a representative on this Board if it desires. The Board elects an Executive Committee which directs the detailed business of the Corporation through the President and the Secretary of the Corporation, subject to Board approval.

To oversee the research, there is a Technical Advisory Committee which plans, approves, and receives the reports on the research program. It meets two times per year. Each member company has a representative on the Committee. The TAC elects a Chairman and a Technical Committee from its membership. The Chairman chairs both Committees. The Technical Committee has three representatives from each industry classification - petroleum, chemical and engineering. One representative from each category is elected each year. The Chairman serves throughout a contract period. The Chairman and the Technical Committee meet four times per year and work closely with the FRI Technical Director to execute the research program approved by the TAC. The Technical Director leads the engineers and technicians regarding the executing of the research program.

Test Columns

Figure 1 shows a Process Flow Diagram of the Stillwater Unit. Figure 2 provides a photograph. The low pressure (LP) double-diameter column is capable of operating from 16 mm Hga to 165 psia. The lower diameter is 4 ft (1.2m) and the upper diameter is 8 ft (2.4m). The high pressure (HP) column is capable of operating from atmospheric pressure to 500 psia. Its diameter is 4 ft (1.2m). The outputs of the two reboilers can be combined to drive either the LP Column or the HP column. Similarly, the condensers can be piped together to support either the LP Column or the HP Column.

These columns contain windows which allow engineers and technicians to observe the hydraulic functioning of the internals being tested. FRI's technicians routinely videotape the hydraulic performances.

Attached to both columns are gamma scan apparatuses which automatically move radiation sources and detectors up and down alongside the columns to determine the densities of the bi-phases on the trays and within the packings.

Technical Staff

In Stillwater, eight highly-trained and experienced technicians perform all of the following duties:

1. Unit start-ups, operation and shut-downs
2. Data collections
3. Column revamps
4. Unit maintenance and upgrading

Their work is unlike that of most chemical plant operators in that steady states are rarely held for more than a few hours. Operation changes are a constant. The operators recently set an FRI record by going 2466 consecutive days without a lost-time accident.

Five staff engineers perform a multiplicity of functions, including the following:

1. Project and safety engineering
2. Column tray/packing layouts and data planning
3. Data collection oversight and database management
4. Data reporting, especially Topical Reports and Progress Reports
5. Hydraulic and mass transfer modeling and correlating
6. Web site and Device Rating Program (DRP) computer programming

Historically, the majority of FRI's engineers have held PhD's in chemical engineering.

Everyday, and all day, the #1 Priority of the technicians and the engineers is Safety, including the protection of the environment.

Member versus Public Benefits

In 1995, FRI's membership peaked at 94 companies. Although, the merger mania of the last few decades impacted that membership negatively, distillation globalization has had a positive effect. There are presently 67 global companies who belong to the organization.

Admittedly, members of FRI gain more direct benefits from FRI than do non-members and the public. Nevertheless, FRI regularly releases information to the public, especially data and reports that are more than 30 years old. The public also benefits indirectly when FRI information is used by FRI members to design or revamp plants. Additionally, with the approval of the Technical Committee, articles are produced by FRI's staff engineers and by member company engineers (13). The bibliography of this paper includes the most recent 20 articles authored by FRI staff engineers (1-20).

The information and correlations developed by FRI enable refining, chemical, petrochemical, and engineering member companies to design with greater confidence and precision resulting in lower capital cost plants. The value to the public from FRI's research is greater energy efficiency and lower cost production of chemical, petrochemical, and petroleum products. The lower energy consumption also lowers greenhouse gas emissions to the benefit of all.

FRI sometimes utilizes university apparatuses and consults with professors from around the world. The graduate students that sometimes perform the university work often become the next generation of distillation experts.

Database

Table 2 shows the large number of vapor-liquid systems that have been tested by FRI. As shown in bold in the table, the three primary systems are C6/C7, iC4/nC4, and ortho/para Xylene. FRI's database divides the 54 years worth of data into seven major categories which presently contain a total of 23,542 runs. Of these total runs, 44.6 % are with the C6/C7 system, 22.2 % are with butane, and 8.3 % are with the xylene system. All other systems make up 24.9 %.

The numbers of runs in the seven major categories of the F.R.I database are as follows:

- Baffle Trays 280
- DualFlow Trays 1027
- Bubble Cap Trays 1925
- Sieve Trays 7125
- Structured Packing and Grid 2611
- Random Packing 2839
- Valve and Proprietary Trays 2461

Table 3 provides a breakdown of the Topical Reports that have been written since 1954. Note how attention paid to trays has been steady. More interesting, see how attention paid to packing, and especially structured packing, increased by a quantum step - around 1980.

Table 4 presents a list of Topical Reports that were regarded as "milestones" by the membership.

Table 5 provides a list of random packings that were tested at FRI. Table 6 addresses structured packings.

Technical Contributions – Trays

Tray vendors have done an outstanding job over the last 30 years regarding the development of new generations of trays that are capable of grossly outperforming the early generations of trays. FRI obtained data and correlations regarding a wide range of trays, as follows:

1. Data were collected and correlations were developed for sieve, valve, baffle, dualflow and bubble cap trays (1, 6, 8). FRI's Device Rating Program (DRP) includes those correlations. Publicly-available design tools for the somewhat-unusual trays are not plentiful.

2. Data were collected on conventional crossflow trays, countercurrent trays, high-capacity crossflow trays with and without truncated downcomers, and cocurrent flow trays with and without cyclonic separators. These new generations of trays have been highly successful industrially. Thousands of columns have been revamped to effect processing improvements

3. On crossflow trays, retrograde liquid flow have been demonstrated to the industry via dye lines and film footage. The associated stagnant froth areas reduce tray efficiencies (4, 7). At least three vendors offer technologies that eliminate those stagnant areas.

4. The deleterious impacts of foaming on trays has been studied and documented. The <u>efficiency</u> detriments of foaming were not generally known before FRI's work was performed.

Technical Contributions – Packings

FRI's first packing test program occurred in 1962 and addressed ceramic Raschig Rings. Around 1980, random packings experienced a "renaissance" - and - structured packings were "born." Working alongside many excellent packing vendor researchers, FRI contributed the following:

1. The importance of good initial distribution and redistribution were demonstrated (5, 9, 18). FRI's test work included some very long beds - 35 ft (11m). Separation and gamma scan data showed that, with such beds, liquid migrates towards column walls and separations deteriorate

2. Data were collected on random packing sizes ranging from 5/8 inch to 3.5 inch. For the larger packings, the reduced efficiencies, reduced pressure drops and increased capacities that were expected indeed occurred - and were correlated

3. Data were collected on new high-capacity random packings

4. An efficiency detriment, or "HETP hump," was observed with random packings at high pressures and high volumetric liquid-to-vapor ratios

5. Working with University of Alberta personnel, the deleterious impacts of foaming on random packings was demonstrated (3). Some engineers had hypothesized that random packings would resist foaming because, with random packings, vapor streams do not bubble through the liquid streams. Although this hypothesized foaming reduction proved not to be the case, random packing proved to be the best of the three major types of contacting devices with regards to foam handling.

6. Structured packings exploded onto the scene in the 1980's, especially in very-large diameter styrene purification applications. FRI tests corroborated the claims of the vendors regarding high efficiencies, high capacities and pressure drops much lower than trays (5, 11, 12, 14, 15, 16). The combination proved

too hard to resist for owners of vacuum towers – and a revolution was underway. FRI's first structured packing study was in 1981. Subsequently, there were 31 other studies.

7. FRI showed that, just like random packings, structured packings are detrimentally affected by foaming. The anti-foaming benefits of the film contacting were not as large as had been expected by the vendors and users of structured packings.

8. At high pressures, structured packing exhibits an even larger deleterious HETP hump than does random packing (16, 17). FRI engineers correlated the starting vapor rate, ending vapor rate and HETP increase of such humps.

9. FRI gamma scan work revealed that, with the first generation of structured packings, liquid was building up at the horizontal interfaces between packing levels. This build-up was reducing capacity. In response, structured packing vendors developed a second generation that virtually eliminated the problem. To date, FRI has tested the following such high-performance packings: Sulzer MellaPak 252Y, 452Y and 752Y - and Koch-Glitsch FlexiPac HC 1.6Y. See Table 6.

10. FRI testing also showed that the vertical gaps between structured packing blocks have no impact on structured packing performance

Technical Contributions – General

Some of FRI's contributions to the distillation world were of a general nature. Examples follow:

1. To the dismay of all distillation engineers – foams just won't go away (3). Foams caused by positive surface tension gradients were possibly first identified as the so called "tears of wine" by physicist James Thomson (Lord Kelvin's brother) in 1855. The general effect is named after Italian physicist Carlo Marangoni, who studied it for his doctoral dissertation at the University of Pavia and published his results in 1865. The most complete early treatment of the subject is due to Willard Gibbs.

   Thereafter, Dr. Sydney Ross's Topical Report 68 described another source of foaming – the imminent formation of a second liquid phase in a column. His research and analysis work aided with the understanding of the sources and also the solutions to column foams.

2. Gamma scanning first impacted distillation column understanding in the early '70's. FRI was quick to adopt this technique for looking inside columns using gamma rays and detectors (20). For the last 30 years or so, FRI has routinely scanned trayed and packed towers, identifying the locations/elevations of high- and low-density biphases. Today, gamma scanning is used routinely industrially to troubleshoot columns, especially by highly experienced vendors of gamma scan services.

Technical Contributions – DRP

FRI's members have access to FRI's Device Rating Program (DRP). This computer program includes correlations and models regarding the hydraulic and mass transfer performance of trays and packings. FRI's engineers are very aware of the vast global collection of papers and articles that exist regarding such equipment, but FRI has a tendency to use its own data to develop its own models and correlations. As a result, FRI members can use DRP to check/corroborate literature and vendor information. As of this writing, almost 400 global engineers are using DRP to design and rate distillation, absorption and stripping columns.

Design Practices Committee

The Design Practices Committee of FRI functions as a body where general distillation industry design practices are documented and guidelines for their application are developed. Design Practice Committee members are not always Technical Representatives from their member companies. They are appointed to the committee from Member Companies based on their expertise in the industry. Their work is documented in FRI's Fractionation Tray Design Handbook: Volume 5; Design Practices. Among the many topics that have been addressed by the DPC are the following:

General Topics: Troubleshooting, scanning and tracing, fouling services, instrumentation, small columns, start-ups and shutdowns, mist elimination, heat exchangers, inspections

Tray Topics: Inlets and outlets, reboiler circuits, transitions, weir types, blanking, gasketing, construction and assembly, mechanical strength, low liquid rates

Packing Topics: Installation, removal, distributors, redistributors, hold-down grids, support plates, inlets and outlets, fires

Technical Contributions – Miscellaneous

FRI's first membership list included zero non-US members. At the present time, FRI membership breaks down geographically as follows:

- U.S. – 26
- Asia – 19
- Europe – 22

In the last thirty years, hundreds of new separation plants (including refineries) have been commissioned in the Far East, South America and the Middle East. There has been a globalization of distillation and distillation vocabulary. Many long-time FRI members believe that FRI has contributed to that globalization.

Additionally –

Not all of FRI's staff members have remained with FRI. Some have moved on to other distillation "pastures." Among those engineers are the following: Simon Chambers, G.X. Chen, Carl Fitz, Henry Kister, Tom Ognisty, Frank Silvey, Walt Stupin and Tak Yanagi. Their contributions to FRI have not been forgotten.

Also, FRI provides monetary support to consultants and universities. Among the consultants who have performed work for FRI are the following: Khaled Gasem, Shuzo Ohe, Sydney Ross, Fritz Zuiderweg. Among the universities who have performed work for FRI are the following: Alberta, Zhejiang, Delft, Tokyo and Oklahoma State. Their contributions to FRI have not been forgotten either.

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Table 1 KEY EVENTS - ESPECIALLY FROM Topical Report 120 BY DR. JOHN G. KUNESH

1961 First packing tests (wet packed) – 1.5-inch Raschig Rings

1962 Long (35-foot) bed of rings tested

1962 Cross-type in-bed sampler introduced for packed beds

1964 Visible High Pressure Downcomer shows entrance choking (OR 1974)

1969 Performed studies on impact of flow path length, residence time and hold-up on tray efficiencies

1973 First FRI dry packing of Pall-type rings

1978 Absorption studies reveal mass transfer in tray downcomers

1982 Tubed Drip Pan Distributor shows that laboratory HETP's can be obtained in commercial columns

1982 New appreciation for importance of distributors over packed beds; importance of near-wall irrigation identified

1985 Professor Fritz Zuiderweg reports on zone/stage model of packed bed behavior

1985 Severe vapor maldistribution had no impact on 1" Pall rings.

1985 Spray collapse observed

1987 Gamma scanning initiated; empty columns scanned; very quantitative results when compared to commercial scanning

1987 PC based data acquisition system was installed

1987 First structured packing testing (Glitsch Gempak 2AT); first use of bayonet sampler

1990 High-performance random packings tested

1996 Database documented

1997 Effect of weeping and entrainment on tray efficiency

1998 High-performance crossflow trays tested

2000 Most recent ultimate capacity correlation released

2001 High-capacity structured packing tested

2001 First FRI IsoPar tests

2002 Sieve and valve tray capacity models updated

2002 Structured packing humps in HETP graphs at high pressure documented

2004 First cocurrent flow tray tested

2006 Tray spray fluidization and downcomer seal models established

2008 Large valve tray study initiated

Table 2 FRI DATABASE SYSTEMS:

System	BC	BT	DF	RP	SP	ST	VT
A-OIL/CO2						X	
A-OIL/N2						X	
A-OIL/N-GAS						X	
A-OIL/STEAM	X	X				X	
C10OH/CO2						X	
C10OH/HE/N2						X	
C10OH/N2						X	
C3			X			X	
C3(OH)2/C2(OH)2				X			
C3/C4/C5	X						
C3/C4/C5/C6	X						
C3/C4/C5/C6/C7	X						
C3=						X	
C3=/C3				X		X	
C3=/C3/I-NC4				X			
C6						X	
C6/C7	**X**	**X**	**X**	**X**	**X**	**X**	**X**
C6/TOL						X	
C8/C10	X		X	X		X	X
CCL4/C3H6CL2	X					X	
CO2/PIB						X	
FREON-11	X		X			X	
H20/AIR						X	
H20/C2(OH)2						X	
H20/CO2						X	
H20/HE/N2						X	
H20/STEAM	X	X	X	X		X	X
HEXANE/CO2						X	
HEXANE/N2						X	
HEXANE/N-GAS						X	
IC4			X				
IC4/NC4	**X**	**X**	**X**	**X**	**X**	**X**	**X**
IC8/TOL			X	X		X	
IPA/H2O	X		X	X	X	X	X
MEOH/HOH				X		X	X
MEOH/HOH/ACE				X		X	
N2/PIB						X	
NC4/NC7						X	
NC5						X	
NC5/A-OIL/ST	X		X			X	
N-GAS/PIB						X	
O-P XYLENE	**X**		**X**	**X**	**X**	**X**	**X**
TOL/H20						X	

BC >> Bubble Cap
BT >> Baffle Tray
DF >> DualFlow
RP >> Random Packing
SP >> Structured Packing
ST >> Sieve Tray
VT >> Valve Tray

Table 3 TOPICAL REPORTS BY DEVICES

	Trays	Random Packing	Structured Packing	Other	Total
50's	14	0	0	9	23
60's	17	6	0	7	30
70's	22	2	0	13	37
80's	12	5	3	8	28
90's	10	5	4	11	30
00's	18	3	12	4	37

- "SP" includes grid packing
- "Other" includes VLE thermodynamics, transitions, physical properties, ultimate capacity, etc.

Table 4 TOPICAL REPORT MILESTONES

1954	First Topical Report – "Simulator Studies of a Bubble Cap Tray
1956	First Model – "Prediction of Tray Efficiency Two–Film Theory
1956	"Koch Flexitray"
1956	"Glitsch Ballast Tray"
1959	"Studies on Ultimate Capacity of Fractionators"
1962	"Nutter Float Valve Trays"
1964	"Two Inch Carbon Steel Pall Rings"
1965	"Review of Packing Literature and FRI Design Methods"
1972	"Direct Contact Thermal Efficiency of Reflux Distribution Systems"
1974	"A Review of Foaming in Fractionation Towers"
1978	"Performance of 2-Inch Ceramic Intalox Saddles"
1981	"Test of Glitsch 2-Z Grid"
1983	"The Effect of Entrainment on Efficiency"
1983	"The Effect of Weeping on Sieve Tray Efficiency"
1984	"Packed Column Flow Phenomena and Performance", by Prof. F.J. Zuiderweg
1986	"Counter-flow Tray Capacity Study"
1987	"Tests of Glitsch Gempak 2AT"
1990	"Tests of No. 2.5 Nutter Ring with Mobil Pour Pan Distributor and FRI TDP Distributor "
1996	"Documentation of PC Data Base"
2000	"Test of Norton Triton Tray"
2002	"Structured Packing Performance in the Hump Region"
2003	"Bubble Cap Tray Flood Models"
2004	"Test of Shell ConSep Tray"
2007	"Surge Volumes Survey Results"

Table 5 FRI RANDOM PACKING

Tower Type	YEAR	PACKING TYPE	MATERIAL	SIZE (INCH)
4 FT LP	1962	Raschig Ring	Ceramic	1.5
4 FT LP	1962	Raschig Ring	Ceramic	1.5
4 FT LP	1962	Raschig Ring	Ceramic	3
4 FT LP	1962	Raschig Ring	Ceramic	0.75
4 FT HP	1964	Pall Ring	Carbon Steel	2
4 FT LP	1966	Raschig Ring	Ceramic	3
4 FT LP	1966	Raschig Ring	Ceramic	3
4 FT LP	1966	Raschig Ring	Ceramic	3
4 FT LP	1967	Perforated Ring	Carbon Steel	3
4 FT LP	1967	Perforated Ring	Carbon Steel	2
4 FT LP	1968	Berl Saddle	Ceramic	2
4 FT LP	1968	Berl Saddle	Ceramic	2
8 FT	1973	Slotted Ring	Carbon Steel	2
4 FT HP	1978	Intalox Saddle	Ceramic	2
4 FT LP	1982	Pall Ring	Carbon Steel	5/8
4 FT LP	1982	Pall Ring	Carbon Steel	5/8
4 FT LP	1983	Pall Ring	Carbon Steel	1
4 FT LP	1983	Pall Ring	Carbon Steel	2
4 FT LP	1983	Pall Ring	Carbon Steel	3.5
4 FT HP	1983	Pall Ring	Carbon Steel	2
4 FT HP	1983	Pall Ring	Carbon Steel	1
4 FT HP	1984	Pall Ring	Carbon Steel	1
4 FT HP	1985	Pall Ring	Stainless Steel	1
4 FT HP	1985	Pall Ring	Stainless Steel	1
4 FT HP	1985	Pall Ring	Stainless Steel	1
4 FT HP	1985	Pall Ring	Stainless Steel	2
4 FT LP	1985	Nutter ring 2.5	Stainless Steel	2
4 FT HP	1986	Pall Ring	Stainless Steel	5/8
4 FT HP	1986	Intalox Saddle	Ceramic	1
4 FT HP	1990	Nutter ring 2.5	Stainless Steel	2.5
4 FT LP	1993	Pall Ring	Stainless Steel	1
4 FT HP	1993	Pall Ring	Stainless Steel	1
4 FT LP	1993	Pall Ring	Stainless Steel	1
4 FT LP	1994	Pall Ring	Stainless Steel	1
8 FT	1994	Pall Ring	Stainless Steel	1
4 FT LP	1998	Pall Ring	Stainless Steel	1
4 FT LP	1998	Pall Ring	Stainless Steel	1
4 FT HP	1999	Raschig Super Ring No. 2	Stainless Steel	2
4 FT LP	2004	Pall Ring	Stainless Steel	2
4 FT HP	2006	Raschig Super Ring No. 2	Stainless Steel	2
4 FT LP	2006	IMTP 25	Stainless Steel	1

Table 6 FRI STRUCTURED PACKING

Tower	YEAR	VENDOR	NAME
4 FT LP	1981	GLITSCH	2-Z GRID
4 FT LP	1981	GLITSCH	2-Z GRID
4 FT LP	1987	GLITSCH	Gempak 2AT
4 FT LP	1987	SULZER	Mellapak 250.Y
4 FT LP	1988	SULZER	Mellapak 250.Y
4 FT HP	1989	NORTON	Intalox 2T
4 FT LP	1990	MONTZ	B1-200
4 FT LP	1990	MONTZ	BSH-250
4 FT HP	1991	SULZER	Mellapak 250.Y
4 FT HP	1992	SULZER	Mellapak 250.Y
4 FT HP	1992	SULZER	Mellapak 250.Y
4 FT LP	1993	JAEGER	Max-Pak
4 FT LP	1994	KOCH	4Y
4 FT LP	1994	SULZER	Mellapak 250.Y
4 FT LP	1997	NORTON	Intalox 4T
4 FT LP	1997	NORTON	Intalox 4T with gas injection
4 FT LP	1997	NORTON	Intalox 4T
4 FT LP	1997	NUTTER	No.3 Snap Grid
4 FT HP	1999	SULZER	HP Mellapak 250.Y
4 FT LP	2000	SULZER	Mellapak 250.Y
4 FT LP	2001	SULZER	Optiflow
4 FT LP	2001	SULZER	Mellapak 252.Y
4 FT LP	2002	SULZER	Mellapak 250.Y
4 FT LP	2003	SULZER	Mellapak 752.Y
4 FT LP	2004	KOCH GLITSCH	Flexipac HC 1.6Y
4 FT LP	2004	SULZER	Mellapak 250.X
4 FT LP	2004	SULZER	Mellapak 64.X
4 FT LP	2005	SULZER	Mellapak 250.Y
4 FT LP	2005	SULZER	Mellapak 250.Y
4 FT LP	2005	SULZER	Mellapak 452.Y

Figure 1 FRI PROCESS FLOW DIAGRAM

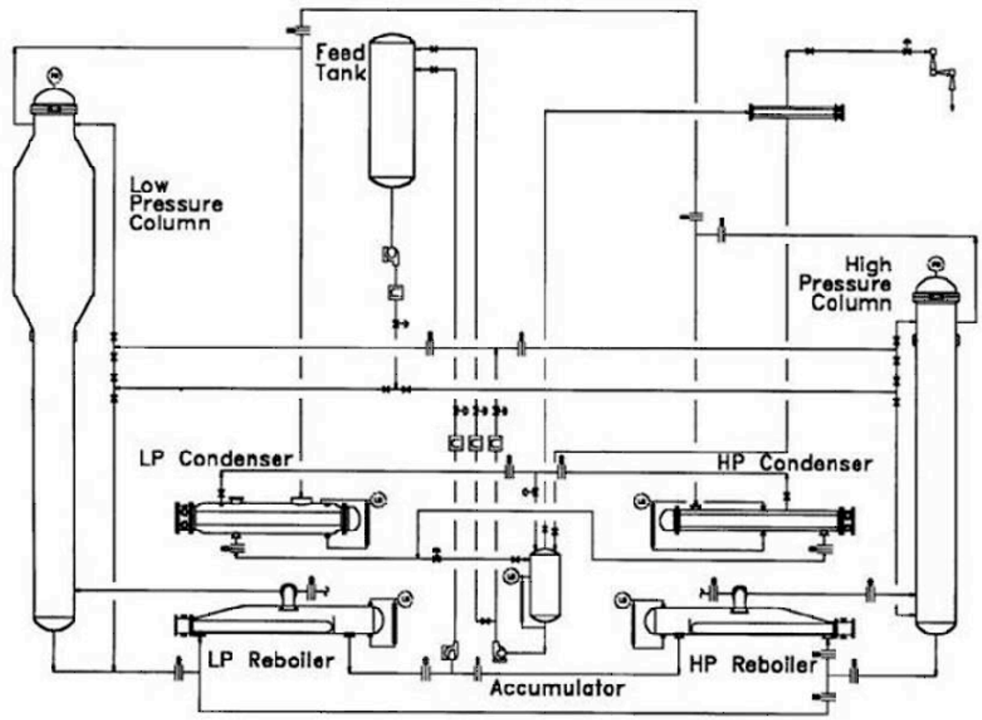

Figure 2 FRI EXPERIMENTAL UNIT, STILLWATER, OK