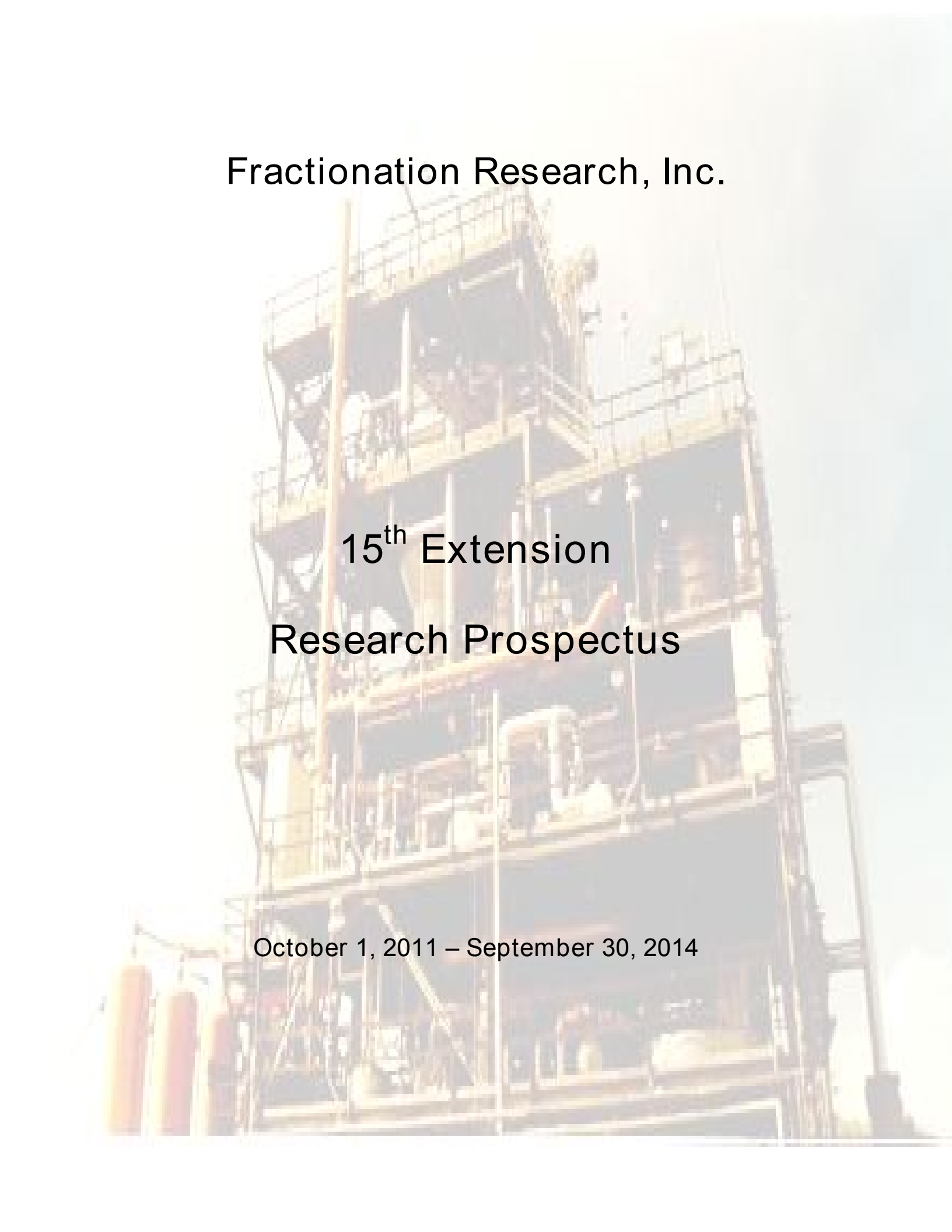


Fractionation Research, Inc.

15<sup>th</sup> Extension

Research Prospectus

October 1, 2011 – September 30, 2014



## Index

Intermediate Bed Limiter Screen Effect for Random Packings .....	6
Inter-Bed Packing Size Change for Structured Packing .....	7
Structured Packings with High Specific Area .....	8
Structured Packing Installation.....	9
Open Area Effect on Entrainment from Packing Distributors .....	10
Gamma Scan Profiles of Packed Towers with Good and Poor Liquid Distribution.....	11
Improving Quality of FRI Test Data Measuring Entrainment from Packed Beds .....	12
Plastic Random Packing.....	14
Small Random Packing Test .....	15
Mixed Packing Test .....	16
Retesting Standard Packings with Modern Distributor and New Test Systems .....	17
Downcomer Anti-Jump and Deflection Baffles .....	20
3-Pass Trays.....	21
System Limit on Trays .....	22
Swept Back (or Extended) Weirs.....	23
Small Downcomers .....	24
Hydraulic Performance of "High" Viscosity Liquids .....	25
Baffles on Dualflow Trays.....	26
Distillation Test on Thinner Decks to Measure Efficiency Increase .....	27
Simulator Study to Improve Tray Pressure Drop Correlation to Predict Flow Distribution .....	28
Packed Downcomers to Stop Vapor Entrainment.....	29
Tray Efficiency Loss with Low Entrainment.....	30
Two-Pass Valve Tray Efficiency and Turndown Evaluation.....	31
Use of Binary Methods on Computer Process Simulator Results.....	33
Valve Tray Efficiency Enhancement with Push Valves .....	34
Improving Quality of FRI Test Data.....	36
Plant Tests.....	39
Improving the Existing Distillation Test Unit to an Innovative/New Research Facility.....	40
Mass Transfer Efficiency – Steam Stripping of Toluene from Water.....	42
Mass Transfer Efficiency – Steam Stripping of an Organic Less Volatile than Toluene ...	43
Suitability of Dynamic Models for Relief Valve Loading .....	44
Column Draw-Offs.....	45
Distillation with Two Liquid Phases Present.....	46
Deentrainment Capacity of Random and Structured Packings At High Vacuum and High Pressure .....	48
Effect of Distribution on Structured Packing Efficiency – The Hump .....	49
Parametric Study of the Effects of Misdistribution on Packing Performance .....	50
Two-Phase Inlets Interaction with Tray Capacity.....	51
Dynamic Model Verification of Tray Performance .....	52
Simulator Test – Effect of Intermediate Pressure Drop Device on Capacity In a Fouling Service .....	53
Fouling Resistance Testing/Studies of the Relative Solids Handling Characteristics and Efficiencies .....	54
Measurement of Entrainment between Dualflow Trays .....	55
Review of State of the Art of Computed Mass Transfer (CMT) and Computational Fluid Dynamics (CFD) .....	56
Partner with Experimental Fluid Dynamics Laboratory.....	57
Rate Based Systems.....	58
Dividing Wall Column Work.....	59

Heat Transfer in Empty Spray Sections in Vacuum Systems .....	60
Study Alternate Separation Process.....	62
Heat Transfer in Pump-Around Zones.....	63
Operation of an Air/Water Simulator .....	64
CO2 Absorption Studies .....	65
High Pressure Valve Tray Testing.....	66
Kettle Reboiler Entrainment .....	67
Mass Transfer Efficiency – Different Physical Properties .....	68

## FRI Funded Proprietary Testing

### Expected Economic Benefit:

The testing of proprietary column internals will allow design engineers to better select the best internals for their needs.

### Present Situation and Proposed Research:

Design engineers must frequently use vendor provided data to help decide which internal will best meet their needs. The cost of doing proprietary device test at FRI has discouraged vendors from testing as many internals as the membership would like. Under this project F.R.I. will test one proprietary tray and one proprietary packing per year.

INTERNALS: One packing and one tray per year. The system will be selected by mutual agreement between the vendor and FRI

ESTIMATED TIME: Three weeks for each packing or tray.

### Background and Discussion:

There are many new contacting devices that have come on the market during the last few years. It is desirable to have these tested by an independent source.

## DEVICE SPECIFIC PROJECTS PACKINGS

DSP



## DSP-1

### Intermediate Bed Limiter Screen Effect for Random Packings

#### Expected Economic Effect:

By quantifying the effect of using successive beds of different sizes of random packing with a bed limiter separating screen, it will be possible to more confidently optimize random packed column performance.

#### Present Situation and Proposed Research:

In many kinds of commercial applications for random packings, the loadings change radically across a packed bed. To match packing sizes to these changes in loading it is desirable to change packing sizes in relatively short height where it would not be necessary or economically desirable to collect and redistribute the vapor and liquid. Standard tests will be run at vacuum and pressure conditions with a composite bed of 1 inch and 2 inch Pall rings separated by level bed limiter screen at the interface. Standard F.R.I. internal bed samplers should be used to quantify the performance of each packing size. The total bed height should be the same as already tested with at least one of the two packing sizes.

INTERNALS: Low pressure column, 1 inch and 2 inch Pall rings.

ESTIMATED TIME: Three weeks.

NON-STANDARD COSTS: None.

#### Background and Discussion:

In commercial practice, a bed limiter screen is used to keep different sizes of random packings from nesting or migrating to form an irregular and/or dense interface that would decrease capacity or efficiency or both. Gas injection support plates are normally not used in this type of service due to the deleterious effect they would have on distribution. Since a flat support plate decreases the capacity potential of a random packed bed, it is reasonable to assume that an internal bed limiter could adversely affect capacity of a bed with two random packing sizes.

## DSP-2

### Inter-Bed Packing Size Change for Structured Packing

#### Expected Economic Effect:

By quantifying the effect of using successive beds of different sizes of structured packings, it will be possible to more confidently optimize performance of columns containing structured packing.

#### Present Situation and Proposed Research:

In many kinds of commercial applications for structured packings, the loadings change radically across a packed bed. To match packing sizes to these changes in loading it is desirable to change packing sizes in relatively short height where it would not be necessary or economically desirable to collect and redistribute the vapor and liquid.

Standard tests will be run at vacuum and atmospheric pressure conditions with a composite bed with two sizes of structured packings. Standard FRI internal bed samplers should be used to quantify the performance of each packing size. The total bed height should be the same as already tested with at least one of the two packing sizes.

INTERNALS: Low pressure column, Mellapak 250Y and 500Y or equivalent.

ESTIMATED TIME: Three weeks

NON-STANDARD COSTS: None.

#### Background and Discussion:

In commercial practice, different sizes of structured packings may form a dense interface that could decrease capacity or efficiency or both. Since structured packings tend to hold up liquid at the element interfaces when approaching flood, it is reasonable to assume that a crimp size change could adversely affect the capacity of a composite bed.

## DSP-4

### Structured Packings with High Specific Area

#### Expected Economic Benefit:

A decrease in capital cost in difficult separations where a large number of theoretical trays are required.

#### Present Situation and Proposed Research:

FRI has collected experimental data on structured packings having specific area of 250 m<sup>2</sup>/m<sup>3</sup> and lower. The proposed research will extend the database of the performance of structured packings up to a specific area of 500 m<sup>2</sup>/m<sup>3</sup>. The effect of pour point density from 10 to 20 pp/ft<sup>2</sup> (100 to 200 pp/m<sup>2</sup>) should be studied and runs should be made at low and high pressures to evaluate the effect of pressure. An adjustable liquid pan distributor should be used so that data on the new structured packings can be compared with the existing data.

INTERNALS: Four foot low pressure column. Structured packings to be supplied by the distillation equipment vendors. The system will be xylenes.

ESTIMATED TIME: Seven weeks

NON-STANDARD COSTS: None.

#### Background and Discussion:

High surface area packings have higher efficiency and are useful for keeping the distillation column height reasonable in difficult separations in which a large number of theoretical trays are required. Such packings may require a higher irrigation density than the 200 to 250 m<sup>2</sup>/m<sup>3</sup> packings studied to date by FRI. In addition, data from higher surface area packings are needed to validate proposed models, all of which use geometric parameters, such as the channel Reynolds Number.

## DSP-6

### Structured Packing Installation

#### Expected Economic Benefit:

Structured packing continues to provide benefit to the industry, but this type of device has caused more unexpected or unexplained performance problems than other types of devices. Information is needed on the sensitivity of this device to installation mistakes.

#### Present Situation and Proposed Research:

Not enough is known about installation tolerance to guide design and field installation to assure good performance. Simulator studies will be done on one packing with various installation flaws. The amount of vapor and liquid maldistribution caused by these flaws will be measured. Distillation tests will then be conducted with installation flaws that appear to have little effect on vapor and liquid flow distribution.

INTERNALS: Structured packing with a small crimp (400 to 500 m<sup>2</sup>/m<sup>3</sup> area). The system will be xylenes.

ESTIMATED TIME: Simulator required. Column: Two weeks if baseline exists, four weeks if not.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Good initial vapor and liquid distribution are known to be important to obtain good performance from structured packing. Liquid flow may be affected by failing to deploy wiper rings, failing to insert filler sheets, leaving gaps between layers, damaging the packing by walking on it, and installing blocks at an angle. The effect of large parting boxes on vapor flow is also unknown. How close can the parting box be to the bottom of a bed before it affects the vapor flow within the bed? How serious a deficiency in each of these areas is necessary before the liquid or vapor distribution is affected?

This study will identify a degree of installation error that appears to have little effect on liquid or vapor distribution. This level of installation error will then be tested under distillation conditions to determine if that level of error can be tolerated.

## DSP-7

### Open Area Effect on Entrainment from Packing Distributors

#### Expected Economic Benefit:

Would improve distributor designs while maximizing packing capacity for both random and structured packings.

#### Present Situation and Proposed Research:

Little is known about liquid entrainment from distributors. During previous FRI research with high capacity structured packing, considerable entrainment was noted. The entrainment was of two types, large droplets and fine mist. It was impossible to determine if the distributor or packing was the major cause of the entrainment generation.

The effect of distributor open area on liquid entrainment would be investigated for trough type distributors. Open area is the area between troughs that is not obstructed by the parting box for vertical upward vapor flow. The objective is to learn how the relative area between the troughs and parting box obstruction influences liquid entrainment from the distributor and the packing.

INTERNALS: Large size structured packing (50 - 120 m<sup>2</sup>/m<sup>3</sup>) and up to four different trough distributor designs for the ortho/para xylene system at 100 mm Hg and atmospheric pressure. An entrainment capture device, similar to that used in prior entrainment studies, would be above the distributor. Also, the bubble cap heat transfer tray would be used to supply saturated reflux, to eliminate possible entrainment from droplets forming on a sub cooled internal reflux pipe. Changes in measured entrainment would be due to differences in distributor open area.

ESTIMATED TIME: Five weeks

NON-STANDARD COSTS: None.

#### Background and Discussion:

There has been a large experimental effort to understand the effect of initial liquid distribution on packing performance that has led to numerous designs to insure an even initial liquid distribution. Some designs sacrifice open area for vapor flow to achieve better initial liquid distribution. There is no clear understanding of the open area tradeoff in distributor design. A poorly designed distributor could have liquid being entrained overhead instead of flowing into the packing. This then becomes a performance limitation. Studying how distributor open area design influences liquid entrainment should lead to better distributor designs.

## DSP-8

### Gamma Scan Profiles of Packed Towers with Good and Poor Liquid Distribution

#### Expected Economic Benefit:

Correct diagnosis of a problem without shutting down the column will save large amounts of money.

#### Present Situation and Proposed Research:

Gamma scanning is a frequently used troubleshooting tool that is well demonstrated for trayed columns. Interpretation of packed tower scans is difficult because of the lack of reference data. Test random packings in the FRI four foot (1.2m) low pressure column with good liquid distribution and with various maldistributions. For each test, have a commercial contractor obtain a gamma scan. The staff will assist the selected contractor; the objective is work typical of the best quality that may be obtained when a contractor operates in an industrial setting. The FRI in-house gamma scanning unit will be used in selected cases for comparison.

INTERNALS: Low Pressure Column: iC4/nC4 at 165 psia, 2 inch (51mm) Pall Rings.

ESTIMATED TIME: Two weeks and one week contingency for one packing type, total three weeks.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Packing is being used extensively to debottleneck trayed columns. Not all packed towers operate as they were designed to. It is important to identify the causes of misoperation as quickly and accurately as possible. There are no tests at FRI where detailed performance data were taken along with chordal gamma scans to assist the troubleshooter to interpret packed tower scans. These data would be very valuable to set a basis for the gamma scanning interpretation of packed towers.

## DSP-9

### Improving Quality of FRI Test Data Measuring Entrainment from Packed Beds

#### Expected Economic Benefit:

Improper rating of a packed or trayed column can cause considerable expense to an operating plant.

#### Present Situation and Proposed Research:

It has been reported that both large-scale and small-scale entrainment exist in many tests performed with both random and structured packing. The standard FRI test condition noted as "flood" is defined as when the column becomes unstable and inoperable. Unfortunately the FRI low pressure column used for many tests has an 8 foot diameter section above the 4 foot diameter section. This possibly allows hydraulic operation at higher rates than if the 8 ft. section were not present. In tests of large capacity packings, video records indicate massive entrainment into the 8ft. diameter section of the low pressure column. Calculations are performed to remove the amount of entrainment to correct the reported flood point. Since all of the entrained liquid does not fall back into the liquid distributor, these calculations may have significant error. There have been no quantitative tests to measure the actual entrainment from the top of a packed bed.

**INTERNALS:** Large-scale and small-scale entrainment are reported and used to adjust the flood point for some of the highest capacity packings. Massive liquid entrainment is seen in video recordings under certain conditions. The vapor rate in the 4 ft. diameter section of the column, with packing and internals in place is obviously high enough to cause this entrainment. As the column expands to an empty vessel in the 8 ft diameter section, the capacity factor ( $C_s$ ) is reduced by a factor of four based on diameter alone. Considering the area taken by the mass transfer equipment in the 4 ft. diameter section, the actual reduction is actually greater than a factor of four.

Using a previously tested high capacity packing, test the same bed moving it down in the FRI column. Install a de-entrainment device above the liquid distributor.

It is proposed that three tests be done.

- Allow entrainment to fall back onto the bed and into the distributor
- Install a de-entrainment device to collect and measure the entrainment above the reflux distributor. The entrained liquid would be returned to the distributor. The distributor would be carefully sized to handle the additional entrainment load.
- Install a de-entrainment device to collect and measure entrainment. In this test, the liquid would be removed from the column and returned to the feed tank. This would simulate liquid that is entrained from the top of a column and go out as overhead product. Composition of the entrained liquid would be measured and compared to that of the reflux stream. If the composition difference is significant, further test configurations may be considered.

**ESTIMATED TIME:** Two weeks of unit operation time and one week of installation are required to measure capacity and effectiveness of an entrainment collection device.

## DSP-9

### Improving Quality of FRI Test Data Measuring Entrainment from Packed Beds

(continued)

When an effective and reliable entrainment capture device is found, it should be considered for use in all future tests in the low pressure column.

NON-STANDARD COSTS: None.

#### Background and Discussion:

During the test of the Koch-Glitsch FLEXIPAC® 4Y structured packing (Progress Report January-February 1994), and more recently in the 2004 Sulzer Mellagrid® 64.X packing test, geysers of liquid were shown in video footage erupting from the top of the packed bed. The liquid entrainment from the top of the bed in both cases was severe at high rates. While different devices were used below the packed beds in each test, in the case of one packing, the efficiency clearly fell off when the bed was severely entraining, and in the other case, the measured sample compositions indicated the packing efficiency remained good during similar entrainment.

The actual liquid flow through the distributor is calculated based on the liquid level in the distributor and the results of the distributor flow test. This calculated value is compared against the measured flow from the reflux flow meter. In addition to dependence on the accuracy of the measurement of the liquid head in the distributor and the actual fluid density, the distributor does not collect all of the entrained liquid. The liquid distributors used in FRI tests have different open areas (<15% to >80%), and complete collection of the entrained liquid is not attempted. Without complete collection of the entrained liquid, the correction based on the measured liquid head in the distributor has a potential for significant error.

The video of the large structured packings demonstrated significant entrainment that was easily visible. However, even with other packings where the massive entrainment was not visible, a significant amount of liquid could be flowing down the wall of the 8 ft section and around the perimeter of the liquid distributor. Some tests have had wall wiper rings installed in the swaged section to conduct such liquid into the distributor. This has not been standard practice for all tests.

## DSP-11

### Plastic Random Packing

#### Expected Economic Benefit:

Plastic packing is common in scrubbers for pollution abatement. Regulatory requirements require performance data in the permits. Poor performance predictions can lead to costly problems in explaining performance deviations from the permit.

Correlations for the predicting the pressure drop and capacity of plastic packing are not available from FRI for plastic packing. A generalized correlation valid for multiple plastic packing would also increase confidence in new designs.

#### Present Situation and Proposed Research:

FRI has not tested any plastic packing. FRI has developed correlations for high void fraction metal packing. A test of plastic packing would show whether those correlations could be extended to plastic packing that have lower void fractions. As stated in the recent FRI Topical Report No. 147 of January 2003, the "new models are for metal random packing only and are not valid for packing made of other materials."

INTERNALS: One plastic packing in the four foot column. (There is some desire for smaller sizes of packing rather than larger sizes.)

ESTIMATED TIME: Three weeks of testing with the C6/C7 system. (No high-pressure data would be obtained.) Flood data would be measured at various liquid rates. Obtain efficiency at total reflux.

NON-STANDARD COSTS: None.

#### Background and Discussion:

The models in Topical Report No. 147 have the desirable characteristic that they avoid any packing specific parameters associated with the packing type. This allows the correlation to be broadly applicable to many metal packing. Extending this correlation to plastic packing would allow better prediction of column size in particular pollution abatement scrubbers. This would also allow proper permitting of pollution abatement columns.

## DSP-12

### Small Random Packing Test

#### Expected Economic Benefit:

Small random packing is often used for the separation of fine chemical systems. They have large specific area and consequently, have a low HETP.

#### Present Situation and Proposed Research:

Carbon steel 5/8" Pall rings were tested with the C6/C7 system at 5 and 24 psia in 1982. The results are documented in Progress Report for July-August 1982 and September-October 1982. These tests found that the measured HETP strongly depends on the liquid distributors used. Multiple cross samplers that was shown to increase HETP in recent studies were used. The combination of poor distributor quality and use of cross samplers might have resulted in higher HETP than expected.

In 1986, stainless steel 5/8" Pall rings was test with the C6/C7 system at atmospheric pressure using the FRI adjustable liquid distributor (Progress Report for January-February 1986). Eight cross samplers were employed. The test results show that the HETP of 5/8" Pall ring was higher than that of 1" Pall ring (Figure 14 in the Progress Report). Again, these results are unexpected.

INTERNALS: Additional tests with 5/8" rings using modern liquid distributors without in bed samplers are necessary to validate the old data and to confirm and extend the TR152 model. For data consistency, 5/8" stainless steel Pall rings may be used. Other packing such as Super ring, Nutter ring, and IMTP can also be considered for practical purposes. Two modern liquid distributors will be used for the test.

ESTIMATED TIME: Three weeks of unit operation time and two weeks of installation are required to measure the HETP, pressure drop and capacity for the C6/C7 system at 5 and 24 psia.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Topical Report No. 152 concluded that all HETP data for 5/8" Pall rings in the FRI database are elevated by using cross samples or low quality liquid distributors. All these data were not used in the TR152 HETP model. TR152 HETP model shows that HETP is inversely proportional to the packing area raised to a power of 0.8. All these findings need to be verified by experimental data.

## DSP-13

### Mixed Packing Test

#### Expected Economic Benefit:

Improved performance of packed towers by retrofitting with mixed beds and improved new tower design.

#### Present Situation and Proposed Research:

The concept of using mixed beds of random packing has been proposed and touted as providing the pressure drop performance of the large packing and the efficiency of the smaller. Patents have been issued in the area and would need to be explored before proceeding. A test of the concept at SRP has been reported, but the results have not been widely used.

#### Proposal

Conduct a patent review to ensure the members can use any experimental work. Test a mixed bed of two sizes of Pall rings and compare against prior tests of those two sizes.

INTERNALS: Standard.

ESTIMATED TIME: Total column time of six weeks as follows:

- One week for initial installation
- Two weeks operation with the first mixed bed
- One week to install a bed with different percentages of the two sizes
- Two weeks operation with the second mixed bed.

NON-STANDARD COSTS: None.

#### Background and Discussion:

The basics of this concept were originally developed by Belko, and patents related to the concept were issued to Belko and later transferred to Amistco. A test was conducted at SRP and appeared to be successful, but has not been widely accepted within the membership. An independent test at FRI would provide a credible gauge of the concept.

## NEW PROSPECTUS ITEM DSP-15

### Retesting Standard Packings with Modern Distributor and New Test Systems

#### Expected Economic Benefit:

Standardized testing of Pall Rings and Mellapak 250.Y structured packings is proposed.

Benchmark capacity-efficiency data collections would be based on:

- P/O-Xylene at 75, 150 and 740 mmHg
- iC<sub>4</sub>/nC<sub>4</sub> at 100 and 165 psia (6.89 and 11.4 bar)
- VKG or equivalent modern high quality distributor.

#### Present Situation and Proposed Research:

At present, new generations of high performance random and structured packings are compared against old Pall-Ring data from 1982-1983 and Mellapak 250.Y data from 1987-1988. C6/C7 was the most common system used. The 'good quality' FRI TDP distributor was employed. Out of 1649 runs for Pall Rings, 30% of the HETP test data are considered suitable for comparisons. Similarly for Mellapak 250.Y, about 20% of the HETP data from 1162 runs are suitable.

In recent years, new high capacity packing tests involve vendor supplied high quality trough type distributors with metering orifices or drip tubes. Although Capacity/HETPs/ $\square$ P advantages are expected, comparisons against baseline data, where old distributors were employed, might be misleading.

INTERNALS For 5/8, 1 and 2 inch (16, 25 and 50 mm) Ring Sizes and Mellapak 250.Y Structured Packing.

- TR Tests with a 10 ft (3.05 m) bed with VKG or equiv. high quality liquid distributor.

ESTIMATED TIME: 3 to 4 weeks per packing type and the five tests systems listed above. In total, at least 4 months.

#### NON-STANDARD COSTS:

#### Background and Discussion:

A common concern with all packing data collections with C6/C7 is adjustment of mid-bed C6 composition to approximately 50%. This is necessary since:

- Low or high mid-bed %C6 impacts the slope of the equilibrium line, m and  $\tilde{\square}$
- Too low or high m-values lead to low or high HETPs that can be misleading
- Nowadays FRI carefully and easily controls mid bed C6 composition at around 50%.
- But a number of FRI members would prefer not to use C<sub>6</sub>/C<sub>7</sub> in future packing tests.
- With xylenes and C4's, composition effects have negligible impacts on HETP's.

## NEW PROSPECTUS ITEM DSP-15

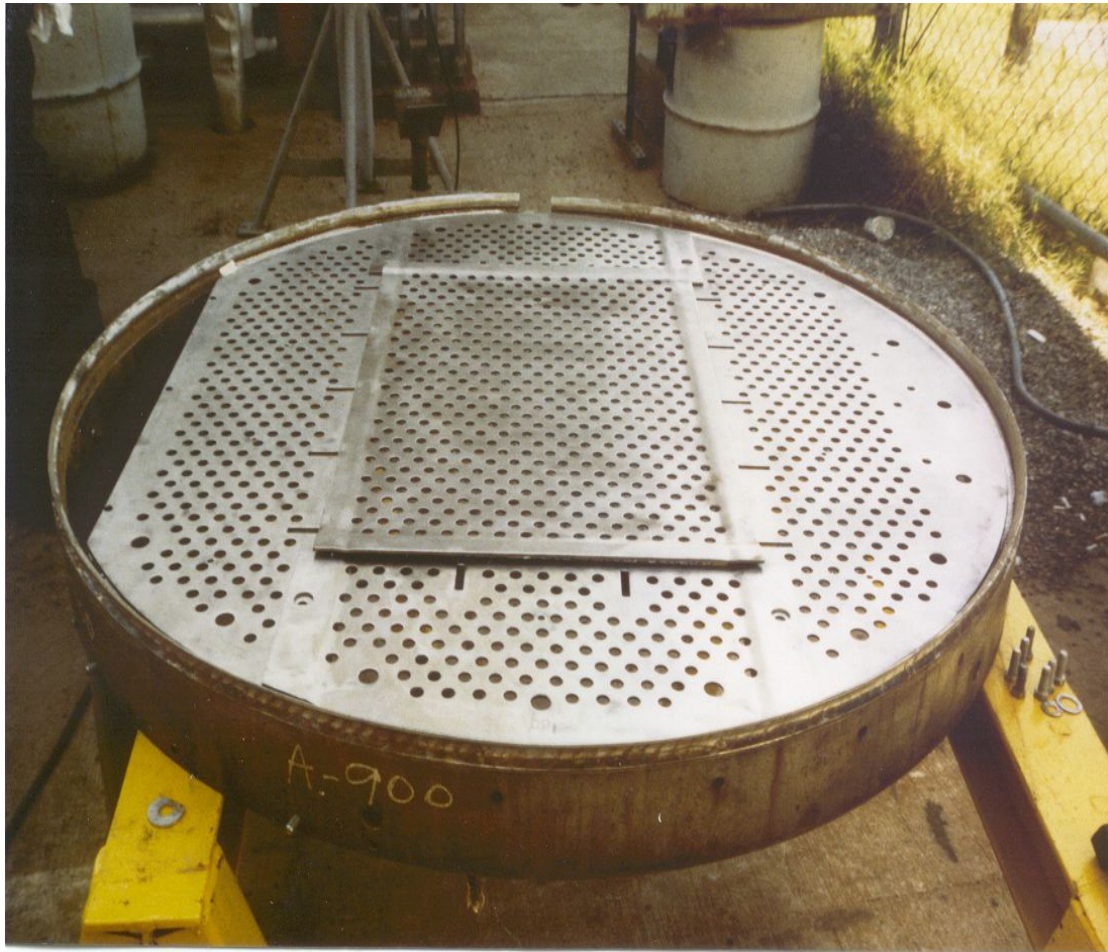
### Retesting Standard Packings with Modern Distributor and New Test Systems

(continued)

Standardized testing of Pall Rings is necessary since there are many column revamps in which they are replaced with a newer generation random packing or where a new distributor is supplied. The opinion of some FRI members is to test a family of IMTP-style 3<sup>rd</sup> generation random packings as they are considered the new benchmark packing specified in the industry. To date FRI has only tested IMTP 25 in 2006 (TR166). The FRI database and DRP do lack a choice of 3<sup>rd</sup> generation random packings for comparison and rating purposes since they have not been tested at FRI. Further tests would significantly increase the scope of the DSP-15 testing program.

## DEVICE SPECIFIC PROJECTS TRAYS

DST



## DST-3

### Downcomer Anti-Jump and Deflection Baffles

#### Expected Economic Benefit:

Increased downcomer capacity for debottlenecking existing trayed towers and lower cost grass roots towers.

#### Present Situation and Proposed Research:

FRI over the years has completed extensive experimental control research on outboard downcomers. The research has included gamma scanning for froth density profiles, observing the hydraulics under actual operating conditions with a transparent downcomer, and studying how variations in downcomer geometry impact downcomer capacity.

Very little research has been conducted on the capacity of inboard downcomers, particularly at the downcomer mouth. The impact of anti-jump baffles on inboard downcomer capacity is unknown. In addition, there have been studies (for example, the report by Mr. Steude of Bayer AG at the May, 1996 TAC Meeting) on using deflection baffles to increase the capacity of downcomers where disengaging is difficult. FRI has not conducted research on the impact of these baffles on capacity.

To evaluate the impact of anti-jump baffles and deflection baffles on downcomer hydraulic capacity, simulator experiments will first be conducted to screen candidate geometry. Testing will then be done in the FRI eight foot column on actual distillation systems for inboard downcomer studies and in the FRI high pressure column for deflection baffles in outboard downcomers.

INTERNALS: Sieve trays with inboard anti-jump baffles and outboard deflection baffles.

ESTIMATED TIME: Simulator required. Column: Two weeks in eight-foot column and two weeks in four-foot high pressure column. System: iC4/nC4 at 165 psia in both columns plus 300 psia in the high pressure column.

NON-STANDARD COSTS: Simulator work would likely be contract ( \$20,000).

#### Background and Discussion:

While anti-jump baffles are used today, the capacity advantage gained (or not gained) is unknown. Means of increasing conventional inboard and outboard downcomer capacity by using relatively inexpensive deflection baffles can offer significant advantages in debottlenecking foam limited or high pressure columns. Testing at FRI with these devices is needed to compare results with the previous data bank that has conventional downcomers.

## DST-4

### 3-Pass Trays

#### Expected Economic Benefit:

Improved design correlations would permit more accurate predictions of column capacity and efficiency. Multi-pass trays could be specified in lieu of two-pass trays to reduce column diameter or tray spacing for a given jet-flood and downcomer backup limit.

#### Present Situation and Proposed Research:

Multi-pass trays are typically specified for high liquid rate applications to reduce weir loadings and to mitigate the negative associated effects such as jet flooding, pressure drop, and downcomer backup. Design methods may be found in the open literature and in suppliers' know-how, but approaches are inconsistent.

It is proposed that three-pass trays be tested in the 8 foot section of the low pressure column. Tests would be performed using the iC4/nC4 system at 165 psia (the limit for the low pressure column) as an example of a high liquid rate system where multi-pass trays would be considered. Data taken would include capacity and efficiency for the overall tray as well as for each panel to attempt to quantify the effects of varying L/V ratios.

Prior to entering into the hardware design, a literature search will be conducted to assimilate the available philosophies and correlations dealing with the design of multi-pass trays. The literature search will also identify gaps in design approaches. Member companies will be surveyed to assess experiences (positive and negative) with multi-pass trays and to invite them to forward any design methods they feel appropriate.

**INTERNALS:** Trays would be commercially fabricated, 1/2 inch hole diameter 11% sieve trays with 2 inch weir height. Two different three-pass tray designs would be tested - 1) a design based on equal flow path length; and, 2) a design based on equal bubbling area. All other parameters (weir height, hole size, fraction hole area, downcomer clearance, etc.) would remain the same for both designs.

**ESTIMATED TIME:** Three weeks per tray design, six weeks total.

**NON-STANDARD COSTS:** To achieve high loadings in the 8 foot section, it is necessary to complete portions of the current capital upgrade program.

#### Background and Discussion:

Various models and rules of thumb for the design of multi-pass trays are available to the designer from suppliers and from the open literature. Designs have been constructed which have met with less than successful operation, to the point that some end users will not accept trays in excess of two flow passes. Conversely, many columns have been designed and successfully operated using three, four, or more liquid passes. Why many columns have exhibited good operation while some columns have not may be largely attributed to the designer's approach.

The proposed program would investigate the two major philosophies of multi-pass tray design, equal bubbling area or equal flow path length, to determine the advantages and disadvantages of each in terms of both capacity and efficiency.

## DST-6

### System Limit on Trays

#### Expected Economic Benefit:

With the rise in popularity of high-capacity trays, the system limit is often approached. The tray designer needs additional information on this limit and factors that affect it on trays.

#### Present Situation and Proposed Research:

FRI only has a handful of data on system limit in trays with downcomers. Recent changes to FRI's system limit correlation produced prediction differences as high as 30-50% for sieve trays. The current tiny system limit data bank provides an insufficient basis to confirm or deny even such large differences in prediction.

There is an absence of data showing how system limit on downcomer trays is affected by liquid rate, hole diameter, and horizontal blowing such as that experienced on several high-capacity trays. There is a debate going on whether the system limit is a function of the superficial or free area in a tray with downcomers - a very large difference! Again, the existing data bank is too tiny to provide an answer.

Simulator work is proposed using air-Isopar® at several tray spacings starting at 36 inches, increasing the tray spacing until capacity no longer increases. Tests are proposed with sieve trays of different hole sizes, hole areas, downcomer top areas, one valve tray, and one proprietary tray (already tested by FRI) with horizontal vapor flow.

INTERNALS: Two tray designs will subsequently be developed and tested at 36 or 48 inch tray spacing in the high-pressure column with systems over the entire pressure range.

ESTIMATED TIME: Four operating weeks for each tray, total eight weeks.

NON-STANDARD COSTS: Simulator work will likely be contracted out ( \$50,000).

#### Background and Discussion:

The proposed program will give insight into the nature of system limit on downcomer trays and into the factors that affect the limit. It will give engineers an idea as to when increasing tray spacing can be used for tower debottlenecking. It will also provide an answer on when a high capacity tray can provide capacities that exceed the system limits in conventional trays. It may also point out regions where use of some high capacity trays may be of limited benefit.

## DST-8

### Swept Back (or Extended) Weirs

#### Expected Economic Benefit:

There are situations where confidence in the benefits of these devices could lead to a smaller tower or the saving of an existing tower shell in a revamp situation.

#### Present Situation and Proposed Research:

Some correlations and literature indicate advantages for swept back and extended weirs. There are very limited FRI data on swept weirs.

**Swept Back Weirs:** A two pass tray with swept back weirs will be tested in the eight foot (2.4m) column duplicating all other tray parameters and operating conditions from the 1989 tray studies.

**INTERNALS:** Two pass sieve trays with swept back weirs.

**ESTIMATED TIME:** Two weeks.

**NON-STANDARD COSTS:** None.

#### Background and Discussion:

**Swept Back Weirs** – The sieve tray capacity correlation indicates situations where capacity increases if the weir length is increased. Swept back weirs can also be used to increase the weir length. This effect should be tested for systems in both the spray and froth regimes. Correlations in the literature indicate that poor tray efficiency can occur with columns that have a weir length that is short relative to column width. Swept back weirs may increase tray efficiency for such conditions.

## DST-10

### Small Downcomers

#### Expected Economic Benefit:

Current design practice may not allow minimization of the downcomer size for systems operating in the spray regime. This study will maximize wall area (to collect the fluid hitting the wall) and minimize downcomer area to increase capacity. This will test the limits of the FRI correlations in a new way.

#### Present Situation and Proposed Research:

For spray systems, FRI data are typically for a downcomer using 5% of the tower area. This means that the bubble area is 90% of the tower area. The lower limit of downcomer size has not been explored by FRI for downcomers operating in the spray regime.

INTERNALS: One sieve tray design in the four foot column with a very small downcomer. (Not an envelope type downcomer)

ESTIMATED TIME: Three weeks of testing. Perhaps the xylene system should be used to stay in the spray regime. Flood data would be measured at various liquid rates. Efficiency data would be gathered.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Downcomers are often not limiting for systems operating in the spray regime. Substantial quantities of liquid sprays are needed against the wall behind the downcomer. This de-aerated liquid runs down the column wall and into the downcomer. Potentially this reduces the disengagement required in the downcomer.

To what extent does this wall effect allow a reduction in downcomer size? These tests will be designed to test the limits of a downcomer operating in the spray regime. The wall effect will be maximized, and the downcomer area will be minimized to allow testing at and above the predicted downcomer-flooding limit.

## DST-11

### Hydraulic Performance of "High" Viscosity Liquids

#### Expected Economic Benefit:

The current FRI correlations for downcomer flooding show reduced capacity for "high" viscosity fluids. The word "high" is in quotes, because high in this case only means fluid with a viscosity above about 1.0 cp. As explained below the FRI correlations may be excessively conservative. Extending the data range may result in a better correlation allowing smaller downcomers for "high" viscosity fluids.

#### Present Situation and Proposed Research:

The highest viscosity data available in the FRI databank is for the absorption oil - CO<sub>2</sub> system. The viscosity of this system is about 1.5cp. Excluding this data reduces the upper limit of the data to only about 0.5 cp.

The absorption oil - CO<sub>2</sub> system was, however, the only FRI system that foamed. This reduced the capacity of the downcomer. Including this data in the correlation's development resulted in a correlation that extrapolates to quite low capacities for any fluid with a higher viscosity.

INTERNALS: One sieve tray design in the four-foot column.

ESTIMATED TIME: Six weeks of testing. Possibly the iC<sub>4</sub>/nC<sub>4</sub> system could be used with a thickening agent added. Primarily flood data would be measured at various liquid rates operating in a stripping mode. The material stripped from the fluid would be totally condensed.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Currently the only "high" viscosity data available is for an absorption oil system. Unfortunately that system was also a foamy system. The FRI correlations for downcomer capacity were +regressed including this data. Extrapolating the current correlation above 1.5 cp shows sharply reduced downcomer capacities. This could cause substantially oversized downcomers for any fluid above about 1.0 cp.

While there may be some correlation between fluid viscosity and foaminess, it is also quite possible that the FRI correlations may be excessively conservative. Better data is needed to extend the correlation and avoid excessive conservatism.

Downcomer problems are more common in high-pressure service. High vapor pressures are typical of small molecules. Small molecules are also more likely to have low viscosity rather than a high viscosity. It may not be possible to find a two component system with a high vapor pressure, which also has a high viscosity. The addition of a non-volatile thickening agent, which is still a fluid at the reboiler the temperatures may be a way to get the desired data. Varying the amount of thickening agent may allow testing a range of viscosity in one experimental program.

## DST-14

### Baffles on Dualflow Trays

#### Expected Economic Benefit:

Dualflow trays are used in some critical services where fouling is an issue. It is uncertain whether baffles on the trays eliminate the known instability of the trays at larger diameter. Elimination of the instability will increase tray efficiency dramatically. This test would allow users to move towards columns with fewer trays with increased confidence.

#### Present Situation and Proposed Research:

FRI has never tested baffles on Dualflow trays. The problem probably occurs at larger diameters (above 4') when the hole area is fairly large (probably above 20%).

INTERNALS: Test six foot trays in a sleeved section of the eight foot FRI column. Users of Dualflow trays will be able to supply suggested designs for the baffles.

ESTIMATED TIME: A total run time of 7 weeks is expected based on the following experimental plan:

Initial installation of sleeve and unbaffled trays	2 weeks
Base operation to establish instability	2 weeks
Modification to incorporate baffles	1 week
Operation with baffles	2 weeks

This assumes operation on one system (C6/C7) at two pressures.

NON-STANDARD COSTS: None.

#### Background and Discussion:

It is probable that as hole area is increased that a Dualflow tray becomes more unstable. There are also reports that as the diameter of a Dualflow tray increases that the flow pattern becomes more unstable. Baffles have been used on Dualflow trays to promote stability, but FRI has never tested whether baffles work or not.

## DST-15

### Distillation Test on Thinner Decks to Measure Efficiency Increase

Expected Economic Benefit:

Custom hole treatments may allow us to create sieve tray designs with higher tray efficiencies.

Present Situation and Proposed Research:

Thin trays provide higher efficiencies than thicker trays, but process conditions often require thick trays. Is there a way to get the ruggedness of thick trays with the benefit of thin trays?

INTERNALS: Sieve trays in 4' section.

ESTIMATED TIME: Unit time is estimated to be 7 weeks as follows:

Initial installation	2 weeks
Operation on C4 system @ 2 pressures	2 weeks
System change	1 week
Operation on C6/C7 @ 2 pressures	2 weeks

NON-STANDARD COSTS: None.

Background and Discussion:

Thin trays have higher efficiency than thicker trays. But thicker trays may be needed to meet certain process conditions. Solicit alternative trays that have the benefits of thinner decks while having the benefits of thicker trays. This test may involve some custom hole treatments.

## DST-17

### Simulator Study to Improve Tray Pressure Drop Correlation to Predict Flow Distribution

#### Expected Economic Benefit:

More confidence is needed in the FRI pressure drop correlations to control flow distribution when designing unequal area trays. This will assist in predicting flow distribution of multi-pass trays. This may also be important to predict flow distribution with the oddly shaped trays that are found in divided wall columns.

#### Present Situation and Proposed Research:

FRI correlations do not now predict the poor flow distribution that FRI achieved in some old three pass tests. A contributing factor for this result may have been the low hole area used in those test. It is not suggested that we model those results, but rather that we obtain a set of good results for hole areas normally found in tray columns.

INTERNALS: Tests could be run in a simulator that is at least 6' in diameter. A fair amount of testing with 8% to 14% trays is probably needed with various three and four pass configurations. Development of a way to measure flow in the various downcomers would assist in analysis of the data.

ESTIMATED TIME: This would probably require about 3-6 months of simulator time.

NON-STANDARD COSTS: Simulator work would probably be contracted out ( \$75,000).

#### Background and Discussion:

Various users of FRI information report that the FRI information does not do well in predicting the flow distribution of liquid on multipass trays. It is thought that the FRI pressure drop equation does not capture all key effects. There is also a need to predict flow and pressure drop of oddly shaped trays common in divided wall columns.

## DST-19

### Packed Downcomers to Stop Vapor Entrainment

#### Expected Economic Benefit:

The addition of random packing to the downcomer may limit the entrainment of vapor in the liquid and allow the downcomer to handle high liquid velocities. This could allow for relatively inexpensive revamps to increase column capacities.

#### Present Situation and Proposed Research:

Downcomers are typically designed with no internals. The swirling liquid that enters the downcomer may have enough kinetic energy to entrain vapor deep into the downcomer, thus decreasing the downcomer capacity.

**INTERNALS:** A previously tested valve tray will be tested but with the downcomer filled with large random packing. The suggested test system is iC4/nC4 at 100 and 165 psia.

**ESTIMATED TIME:** Six weeks of test time is required.

**NON-STANDARD COSTS:** None.

#### Background and Discussion:

This simple modification of column internals could allow inexpensive revamping of columns for higher capacities.

## NEW PROSPECTUS ITEM DST-21

### Tray Efficiency Loss with Low Entrainment

#### Expected Economic Benefit:

Improved tray efficiencies and predictions via an improved understanding regarding the occasions where efficiencies drop steeply – with increasing rates – due to reasons other than entrainment.

#### Previous Situation and Proposed Research:

It has become clear that entrainment may not be the cause of efficiency loss at high loads, for some of the common FRI tray designs. Some sieve tray designs lose efficiency for reasons not related to entrainment – such as jetting, spraying, eddy diffusivity, decreasing  $T_G$ 's, etc.

Additional experimental work is probably required. Before a detailed test plan can be devised, the FRI databank and the literature need to be reviewed.

The Tray Blowing program (DST-1) would certainly impact this study. Also, the on-going OSU valve tray efficiency modeling project will probably impact this study, because, a key component of that project is the impact of jetting on tray efficiency. It is possible that the OSU project will explain, in large part, tray efficiency losses, at high rates, in the absence of entrainment.

INTERNALS: New trays will probably need to be fabricated. Tray spacings will probably be 36 inches. Entrainment will need to be measured – accurately. Jetting will probably need to be created – and measured.

ESTIMATED TIME: Four man-weeks for databank review and literature study. At least four weeks of LP Column Operation.

NON-STANDARD COSTS: None

#### Background and Discussion:

Previous approaches to modeling efficiency (TR79 and TR126) centered on determining the “dry” efficiency and then correcting the efficiency value for entrainment and weeping. Entrainment alone seems unlikely to be the cause of all high-rate efficiency losses. Looking back at some of the entrainment measurements with 24 inch tray spacing, there was a substantial loss of efficiency as the vapor rate was increased even though the measured entrainment rate was less than 7%.

After a review of the existing data base and literature, FRI can more easily determine what data might be needed to improve the FRI efficiency model and to improve tray efficiencies (at high rates).

## NEW PROSPECTUS ITEM DST-22

### Two-Pass Valve Tray Efficiency and Turndown Evaluation

Expected Economic Benefit:

Better understand the efficiency and turndown capability of two-pass valve trays  
Validate published industrial methods for predicting valve tray turndown

Present Situation and Proposed Research:

Test plan

- Collect efficiency and hydraulic data
- FRI 8 ft diameter low pressure column
  - Internals to be studied:
- Test systems: C6/C7 at 5 & 24 psia, iC4/nC4 at 100 and 165 psia, air/water simulator.
- Survey the FRI membership for plant test efficiency data and turndown capability on commercial size towers

INTERNALS: Two-pass valve trays with conventional round valves

ESTIMATED TIME: Four weeks unit time

NON-STANDARD COSTS:

Background and Discussion:

The main objective of this project is to measure the efficiency and turndown capability of two-pass valve trays in the 8 ft section of the low pressure column in the most common standard test systems at FRI. This would include Cyclohexane/n-Heptane system at 5 & 24 psia and the Isobutane/Normal Butane system at 100 and 165 psia. The previous tests with two pass sieve trays (discussed in the Dec 1969 PR) did not include the 100 psia system, so this could be omitted for the sake of cost and time savings. This provides data where trays commonly operate, near atmospheric pressure and at elevated pressures. The 5 psia system data can also be collected without too much additional cost since that does not require an additional column entry. This testing will provide tie points between the current single pass valve tray test program as well as the two pass sieve tray research done in the 1960's.

MODELING

MD

Test Data



Correlation/Modeling



## MD-3

### Use of Binary Methods on Computer Process Simulator Results

#### Expected Economic Benefit:

A better knowledge of the effectiveness of binary/graphical methods will allow engineers to more effectively design distillation columns.

#### Present Situation and Proposed Research:

The ease and speed of rigorous tray to tray calculation has resulted in their use for the optimization of column designs. However, there are still supporters that believe the use of binary/graphical methods reveal important details about a columns nature.

INTERNALS: This is non column work so no hardware is needed. A simulation package such as Aspen Plus is required.

ESTIMATED TIME: No more than 500 person hours of work are expected to be needed for this project. Funding of a student is a possible choice.

NON-STANDARD COSTS: Contract work with a university ( \$20,000).

#### Background and Discussion:

The use of McCabe-Thiele, Hengstebeck and other methods has been recommended for multicomponent distillation. However, their use is presently very limited. In 2001 Billingham and Lockett published a paper on packing mal distribution sensitivity that is based on a binary analysis. A better understanding on the effectiveness of such methods would greatly contribute to fractionation knowledge.

## MD-6/7

### Valve Tray Efficiency Enhancement with Push Valves

#### Expected Economic Benefit:

Developing a rating method of push valve capacity and efficiency will allow member companies to optimize the commercial design of high performance valve trays and tower size (diameter and height).

#### Present Situation and Proposed Research:

This effort would begin with a screening state. No method of comparing the “push tendency” is currently available.

It is proposed that FRI develop a criteria for rating the push tendency of valve types with the Esso jet tab as a 10 and a sieve hole as a 0. This would be incorporated in a Topical Report on how the membership should apply and rate push valve technology from the standpoint of both capacity and efficiency improvements. (Some of Lockett’s modeling work might come in handy here or the Computational Mass Transfer work could be useful if it is shown to be valid.)

At a later stage we may wish to perform plant tests on large scale trays with push valve technology to compare and document improvements in tray efficiency and/or capacity.

INTERNALS: Valve trays with push valves.

ESTIMATED TIME: When tests on commercial size towers are possible.

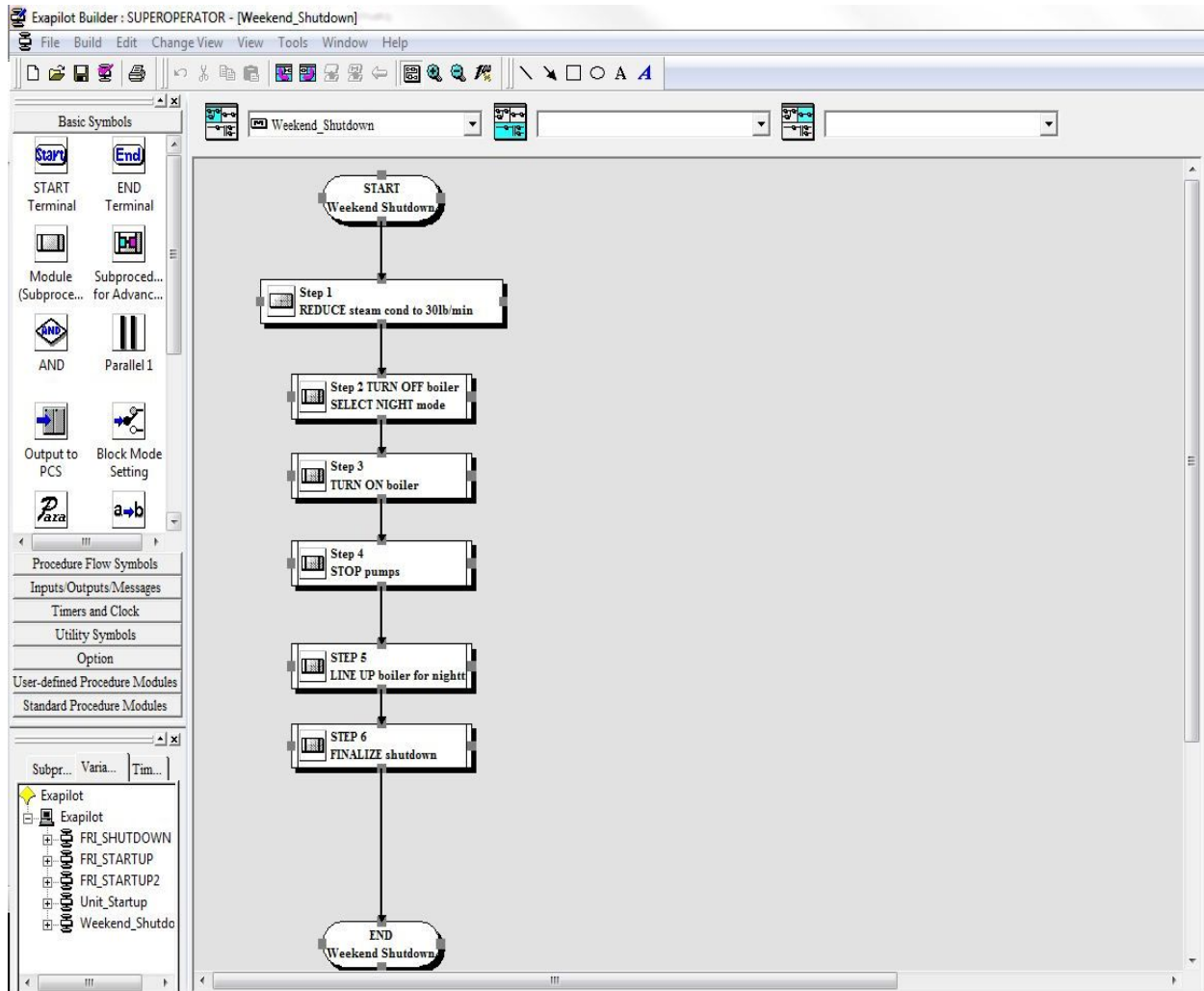
NON-STANDARD COSTS: The first phase is most likely contract work ( \$15,000).

#### Background and Discussion:

The leading tray vendors have experience with push valve technology and may be able to provide guidance and share their expertise on this program.

# OPPORTUNITY RESEARCH PROJECTS

OR



## OR-1

### Improving Quality of FRI Test Data

#### Expected Economic Benefit:

Improper design of auxiliary devices such as collectors, reboiler inlets, and vapor distributors can compromise the performance and/or limit the capacity of an otherwise well designed distillation column. Ensuring that the auxiliary devices are not affecting the test results of the packing or trays being tested will provide improved confidence in the overall test results.

#### Present Situation and Proposed Research:

It has been reported that both large-scale and small-scale entrainment exist in many tests performed with both random and structured packing. The amount of entrainment has been estimated indirectly, but there have been no direct measurements to quantify the amount of entrainment, its dependence on boilup rate, the test system, or pressure.

The proposed program would include the installation of a de-entrainment device to capture all of the reboiler entrainment and measure its quantity as a function of rate and test system.

**INTERNALS:** The quantitative determination of the amount of liquid entrainment from the reboiler is needed to determine whether entrainment from the reboiler is significant and whether this entrainment affects the conclusions reached for packing tests.

**ESTIMATED TIME:** Three weeks of unit operation time and three weeks of installation are required to measure the entrainment from the reboiler at various boilup rates for each FRI test system. Initial tests will be with C6/C7 system at 4 and 24 psia.

It is envisioned that this test would not be completed in one test program, but would continue over time. Depending on the results for C6/C7 tests, tests may be needed for the butanes system.

**NON-STANDARD COSTS:** None.

#### Background and Discussion:

Large-scale and small-scale entrainment has been reported to be present in packing tests, especially for higher capacity structured packing. The quantitative measurement of the entrainment has not been done. Some of the high capacity packing tested show obvious capacity limits for column auxiliaries, while other packing tests may have been affected under certain conditions, but not recognized. A quantitative measurement of the amount of the entrainment from the reboiler for each test system and pressure would help to determine when a de-entrainment device is required and would establish a basis for consideration whether significant entrainment may have affected the results or conclusions of previous test work.

## OR-2

### Improving Quality of FRI Test Data Reboiler De-Entrainment (Part 2)

#### Expected Economic Benefit:

Improper design of auxiliary devices such as collectors, reboiler inlets, and vapor distributors can compromise the performance and/or limit the capacity of an otherwise well designed distillation column.

Ensuring that the auxiliary devices are not affecting the test results of the packing or trays being tested will provide improved confidence in the overall test results.

#### Present Situation and Proposed Research:

It has been reported that both large-scale and small-scale entrainment exist in many tests performed with both random and structured packing. Various devices and configurations have been used in an attempt to prevent entrainment of liquid from the reboiler and/or provide a means to obtain a reliable liquid sample below a packed bed, while none have been thoroughly tested for their effectiveness. The various collectors and de-entrainment devices that have been used over the years have been designs by FRI as well as various packing vendors. In addition to various types and designs of devices, the position of the devices in relation to the reboiler vapor inlet and to the bottom of the packed bed have been widely varied.

In prospectus item "Improving Quality of FRI Test Data (Part 1)", the objective is to quantitatively measure the amount of entrainment from the reboiler and determine whether the entrainment is enough to affect the results or conclusions made for prior or future test work. In Part 2, the capacity and effectiveness of the various collectors/de-entrainment devices are to be measured. Through quantitative testing of the collectors, it will help establish whether the collector/de-entrainment device is actually helping to overcome entrainment from the reboiler, or if the device itself is actually causing additional entrainment. If the collector/de-entrainment device generates additional entrainment, it could lead to erroneous efficiency and/or flooding conclusions.

The quantitative determination of the capacity of various collectors and de-entrainment devices is needed to ensure the conclusions reached for packing tests neither penalize nor exaggerate the device performance due to auxiliary equipment. When extracting data for use in correlations or when test results are used for comparison of different packings, it is important to know if the column auxiliaries may have affected the results.

**INTERNALS:** Various configurations of chimney tray, high rise bubble cap and vane collectors as well as de-entrainment beds of structured packing or grid have been used without a thorough understanding their effect on the results and conclusions reached for packing performance. It is understood that the capacity of the large crimp structured packings is high and the tests can be affected by capacity limiting auxiliaries. The limits of these auxiliary devices often become obvious based on separation performance and/or video observations. Since the limitation of the various devices in different systems and various pressures are not well understood, especially where video records are not available, it is not known when the auxiliary device may affect the measured results and conclusions reached. It is possible that some lower capacity packings may also have been affected to some degree in some systems or pressures.

## OR-2

### Improving Quality of FRI Test Data Reboiler

#### De-Entrainment (Part 2)

(continued)

A proposed program would include quantitative tests of collector and de-entrainment devices that have been used in past experimental work. It is proposed to determine the vapor and liquid capacity of each device under countercurrent flow conditions by using a separate de-entrainment device.

**ESTIMATED TIME:** Six weeks of unit operation time and six weeks of installation are required to measure capacity and effectiveness of various collectors and de-entrainment devices. A high capacity de-entrainment device will be installed with another de-entrainment device above it to determine at what capacity the primary de-entrainment device is failing. The effectiveness of de-entrainment from the reboiler and the capacity of the collector at various boilup rates for each FRI test system should be studied. Initial tests will be with the C6/C7 system at 4 and 24 psia. To provide efficient use of the FRI resources, this test may be combined with the previous prospectus item.

It is envisioned that this test may not be completed in one test program, but could continue over time. Depending on the results for the C6/C7 system, tests may be needed for the butanes system.

**NON-STANDARD COSTS:** None.

#### Background and Discussion:

In the test of the INTALOX® 4T structured packing (Progress Report March-April 1997), it was found during the short bed test that the standard, high rise bubble cap collector tray was entraining heavily and limiting capacity. The collector was changed for the deep bed test (Progress Report July-August 1997) and the useful capacity of the packing increased over the short bed test.

During the last few years of structured packing tests, various combinations of devices of short packed beds and vane or deck type collectors have been used below the packed beds to reduce entrainment from the reboiler. The effect these auxiliary devices have on the measured packing performance is not known because these devices have not been tested independently for effectiveness and/or capacity. There is very little video record of the action above and below any of these devices to even allow a qualitative judgment.

## OR-5

### Plant Tests

#### Expected Economic Benefit:

Increased confidence in the scale up of FRI correlations would provide for more economic designs.

#### Present Situation and Proposed Research:

This item has been attempted in the past and has failed due to the need for an industrial partner.

#### Proposal:

- Identify an industrial partner who is willing to take the steps necessary to allow data collection on a large column.
- Instrument the column as needed for data collection.
- Conduct the plant test.
- Reduce the data using standard FRI data reduction methods.
- Compare the reduced data to comparable FRI data and FRI correlations.

INTERNALS: Whatever is installed in member column.

NON-STANDARD COSTS: Additional instrumentation required for FRI data reduction; staff travel expenses to site ( \$50,000).

ESTIMATED TIME: Conducted in a member unit, so no column time is needed.

Background and Discussion:

## OR-7

### Improving the Existing Distillation Test Unit to an Innovative/New Research Facility

#### Expected Economic Benefits:

The upgrade of the existing test unit to an innovative/new test facility will benefit FRI members in getting more research results per year and equipment suppliers in having less expense.

#### Present Situation and Proposed Research:

The FRI test column first went on-line in 1954 and has been expanded and modified since. Though the present plant is able to test hardware under low pressure and high pressure conditions the unit is not state-of-the-art equipment.

Over the past years suppliers of hardware equipment have marketed and developed more and more high capacity devices that are of interest to FRI membership. The present test equipment is not designed to run these devices up to full capacity.

Recent investigations of the status of the existing unit have verified that corrosion can be monitored to the column shell and heat exchangers and other equipment as pumps and pipe lines are hard to seal. In addition searching for repair equipment of measurement devices as GC analyzer is getting difficult.

INTERNALS: None.

ESTIMATED TIME: The improvement work to raise an innovative/new distillation test unit will take a time frame of more than 1 year.

NON-STANDARD COSTS: Capital costs for this are estimated to be \$8-10MM.

#### Background and Discussion:

The following list should illustrate the targets that can be reached with the innovative test unit:

- Minimize start-up and shut-down time by establishing an approved strategy to reach stable test conditions in a short time depending on the type of test system used and test equipment installed
- Minimize operation time for a test run by the installation of various online concentration measurement devices to indicate constant conditions
- Maximize number of tests per year by optimizing the operation strategy of the unit (i.e. minimize time of changing fluids, minimize time to install/extract test equipment,...).
- Incorporating modern/innovative measurement techniques to maximize information output (video systems, gamma scans ...)
- Installation of a test unit capable to investigate high capacity devices that will come to market in the upcoming years.
- Installation of a test unit capable to examine principle research targets with standard capacity devices
- ....the list of targets does not claim to be complete and further ideas are welcome to be added.

Some of the above mentioned strategies are already in place by running the present test facility but should be reviewed once again in parallel to the other targets for the innovative test unit.

## PHYSICAL PHENOMENA PROJECTS

### PPP



## PPP-2

### Mass Transfer Efficiency – Steam Stripping of Toluene from Water

#### Expected Economic Benefit:

Improved capability to design steam strippers using contactors other than sieve trays, will allow designers to choose the most cost effective device for steam stripping waste water.

#### Present Situation and Proposed Research:

In 1988, FRI measured the efficiency of a sieve tray column in steam stripping toluene from water. The measured tray efficiencies were significantly different from those predicted by existing models, both FRI and the open literature. The data have been useful, not only in designing new strippers, but also in advising the EPA regarding the potential costs of mandated cleanup activities.

For various reasons, a plant may prefer to use a different method of vapor-liquid contacting than sieve trays. Based on the experience with sieve trays, it seems likely that predictive efficiency models for other devices are unsatisfactory in this service.

The test procedure used for sieve trays will be repeated for each tested contacting device.

INTERNALS: A random packing.

ESTIMATED TIME: Three weeks.

NON-STANDARD COSTS: None.

#### Background and Discussion:

The data obtained in 1988 with sieve trays has been very beneficial. To collect it, a number of new techniques were learned and/or developed. That knowledge will be useful in implementing this proposal.

## PPP-3

### Mass Transfer Efficiency – Steam Stripping of an Organic Less Volatile than Toluene

#### Expected Economic Benefit:

More data on steam stripping of water will allow more reliable design with lower safety factors for liquid phase controlled separations.

#### Present Situation and Proposed Research:

In 1988, FRI measured the efficiency of a sieve tray column in steam stripping toluene from water. The measured tray efficiencies were significantly different from those predicted by existing models, both FRI and the open literature. The data have been useful, not only in designing new strippers, but also in advising the EPA regarding the potential costs of mandated cleanup activities.

The slope of the equilibrium line for stripping toluene from water at atmospheric conditions is about 2300. Consequently, the value of  $mV/L$  for these tests ranged from 120 to 2200. Virtually all of the rest of FRI's efficiency data is at values of  $mV/L$  near 1.0. Models that successfully predict the efficiency of systems with  $mV/L = 1$  do not predict the toluene stripping efficiency without including a function of  $mV/L$ ; however, the  $mV/L$  for toluene stripping is so far removed from that of the other data that almost any arbitrary function will work.

In order to obtain efficiency data over a more continuous range of  $mV/L$ , a compound less volatile than toluene will be stripped from water. The compound chosen should have an equilibrium line slope between 80 and 200.

INTERNALS: Sieve trays and a random packing.

ESTIMATED TIME: Four weeks per device, eight weeks total.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Compounds with the desired volatility would likely be partially soluble in water. This could lead to the possibility of foaming and/or the existence of two liquid phases in the tower. These phenomena are the subject of other proposals.

## PPP-5

### Suitability of Dynamic Models for Relief Valve Loading

#### Expected Economic Benefit:

Engineers will be able to size column relief valves and associate header/flare systems with reduced safety margins, resulting in lower costs.

#### Present Situation and Proposed Research:

The major process simulation vendors should have dynamic model simulators operational during the prospectus period. The dynamic simulation vendors along with the operating companies will “tune” and adjust the simulations basic data during normal operation. However, there will be almost no opportunity to verify that dynamic models accurately predict relief valve loading during a severe unsteady state upset that causes a relief valve discharge.

The proposed research involves inviting the major dynamic simulation vendors to participate. Each would generate a model of the FRI test facilities and predict the relief valve loading for common upsets, such as loss of cooling water or reflux pump. FRI would operate one column (possibly the high pressure) with a temporary relief valve (set to relieve before any existing relief valves) discharging into the other column. The other column’s condenser would be operational. FRI would purposely cause an upset by stopping cooling water or reflux pump. The resulting pressure rise and relief rate would be recorded.

FRI members can see how the various dynamic models perform in predicting the relief rate caused by the upset. The dynamic simulation vendors would be free to use the data to improve their simulator (if necessary).

INTERNALS: One of FRI’s sieve trays (or valve trays)

ESTIMATED TIME: Two weeks, one week for steady state tuning of the models followed by one week of upsets for data collection.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Current procedures for column relief valve sizing generally use steady state or over simplified models to approximate the largest expected relief that occurs in an unsteady state system. This is especially true for columns with pump around heat removal systems. Thus, the resulting relief valve and any associated header/flare system are generally oversized.

Some relief valve loadings have been calculated by dynamic models. These have resulted in lower than normal relief loadings. However, the validity of the lower relief valve loadings cannot be easily ascertained.

## PPP-6

### Column Draw-Offs

#### Expected Economic Benefit:

Side and bottom draw offs are seldom-analyzed components of columns which, when improperly designed and specified, may lead to costly startup problems or operating bottlenecks. Well-researched design correlations will aid the designer in correctly sizing these critical components.

#### Present Situation and Proposed Research:

It is proposed that all tests be done in the 4 foot section of the low pressure column. Tests would be performed using the iC4/nC4 system at 165 psia (a high vapor density system) and o/p xylene (a low vapor density system).

The program will include a review of the results of the sponsored research project recently completed with Oklahoma State University to ascertain if any research results obtained in the program might be applicable.

A literature search will be conducted to review the numerous published articles relating to the design of ancillary column details. The FRI Design Practices Committee will be called upon to provide their input and guidance in structuring the details of the program.

No simulator work is proposed due to the uncertain effects of surface tension and viscosity on the experimental results.

INTERNALS: Existing FRI sieve trays and packing with specially constructed draw off sumps.

ESTIMATED TIME: Two weeks per device (trays and packing), total of four weeks.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Side and bottoms draw offs are common components of all columns - both trayed and packed. In the case of bottoms draw offs, there is often sufficient liquid residence time to allow degassing of the liquid so that outflow is predictable and operation does not suffer. In the case of side draw offs, however, the "liquid" being drawn is usually aerated and at equilibrium conditions. These two conditions combine with typically shallow liquid levels so that flashing in the throat of the outlet nozzle is a problem.

Draw off connections are often sized based on other criteria with no consideration given to the physical conditions or internals inside the column. A benefit of this program is that it will draw attention to these details and underscore their criticality. The data and correlations derived from the program will provide the column designer with tools to enable him to easily consider and incorporate side and bottoms sump and draw off nozzle details into his design.

## PPP-7

### Distillation with Two Liquid Phases Present

Expected Economic Benefit:

This test will help reduce uncertainty in designing towers when two liquid phases are present.

Present Situation and Proposed Research:

Non-ideal vapor-liquid equilibrium in a separation sometimes can lead to the formation of two liquid phases in a distillation column. Sometimes a two-liquid-phase mixture may be used as reflux to a tower employing azeotropic distillation. There exists no data to show what effect a second liquid phase has on liquid distribution, downcomer performance, tray capacity or efficiency, etc.

The experimental unit will be run with two liquid phases present in the tower.

INTERNALS: Sieve trays using the C6/C7 system and water.

ESTIMATED TIME: Three weeks.

NON-STANDARD COSTS: None.

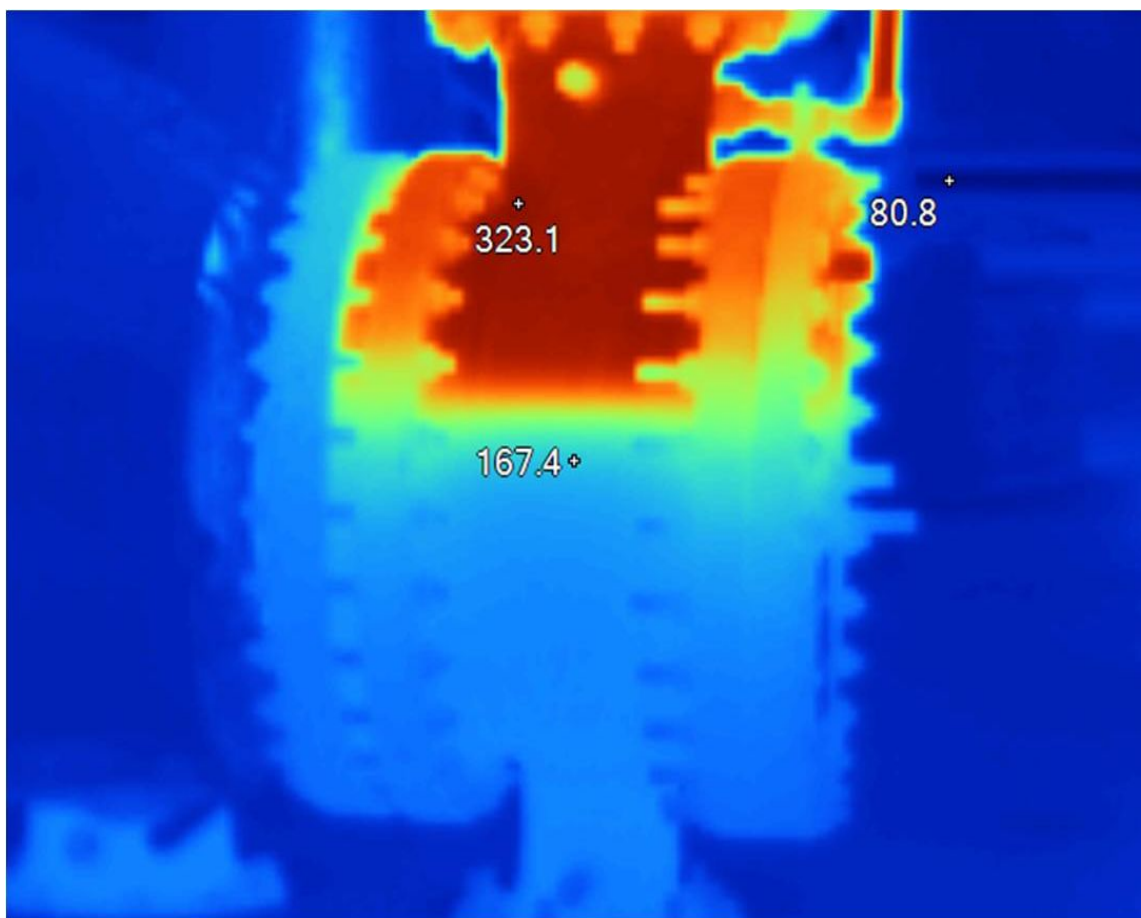
Background and Discussion:

One problem in selecting a system is that a binary two-liquid-phase system always forms an azeotrope, and no further composition change can occur. A second problem is that Ross-type foaming can occur when the composition of a single-liquid-phase system nears the point of phase separation. This test avoids these complications and concentrates on the physical two phase hydraulic problems.

## Developmental Long-Range Projects (DPL)

### Definition:

Developmental Long-Range Projects, also referred to as *DLP Items*, are testing programs generally requiring experimental unit time and resources, but are not intended to produce characteristic operating results for a specific device. Instead, they are intended to gather information about background scientific principles that govern the operation of families of devices. Results from DLP studies are useful for understanding of underlying principles and modeling work rather than for direct use in picking a specific device for a given application. Also, DLP items often depart from standard experimental unit procedures and testing protocols, and may have aspects that require investigation for improved definition before experiments can begin, such as defining a nonstandard chemical system.



## DSP-3

# Deentrainment Capacity of Random and Structured Packings At High Vacuum and High Pressure

### Expected Economic Benefit:

This study will give the designer information on the deentrainment performance of random and structured packings at high and low pressure, enabling more confident design of scrubbers, knockout drums, and other deentrainment services at these extreme conditions.

### Present Situation and Proposed Research:

This is a new area of investigation for FRI. Most public domain and vendor studies of deentrainment performance have been limited to air/water at ambient conditions. However, many applications require design at either high or low pressure with hydrocarbons as the working fluid with substantially different physical properties.

A set of high and low pressure deentrainment data will be collected with both a 2 inch or larger random packing and a 45 ft<sup>2</sup>/ft<sup>3</sup> surface area structured packing or smaller. For low pressure data the o/p xylene system at 100 mm Hg is proposed. For a high pressure test the iC<sub>4</sub>/nC<sub>4</sub> system at 300 psia will be used. Atomizing spray nozzles will be used to introduce the liquid at defined droplet sizes into the packings. The effect of varying inlet droplet size distribution and liquid loading will be investigated as well as the effect of vapor rate on collection efficiency and capacity. The liquid not collected will be determined both by a downstream high efficiency vane/mesh pad collector and fiber collector. Optical or impaction methods may also be used to measure the breakthrough drop sizes and amount of liquid. The liquid collected by the deentrainment bed will be determined by a collector tray draw off.

**INTERNALS:** Two inch or larger metal random packing, 45 ft<sup>2</sup>/ft<sup>3</sup> surface area or less structured packing, atomizing spray nozzles, collector tray, vane/mesh pad collector and fiber collector.

**ESTIMATED TIME:** One week for each packing tested at each pressure level. Total four weeks.

**NON-STANDARD COSTS:** None.

### Background and Discussion:

Deentrainment is becoming a more important physical process in the design of mass transfer contacting equipment as equipment capacity limits are being pushed to new levels. This program is intended to initiate FRI research in this area, utilizing FRI's capabilities in doing high vacuum and high pressure studies with industrially representative chemical systems.

## DSP-5

### Effect of Distribution on Structured Packing Efficiency – The Hump

#### Expected Economic Benefit:

This would allow use of structured packing in high pressure applications should revised liquid distribution eliminate or reduce the efficiency “hump” currently observed.

#### Present Situation and Proposed Research:

Previous FRI research has shown an efficiency decrease or “hump” in the middle of the operating range for structured packing when the pressure is greater than 11.4 bar (165 psia). Changing the distributor height and adding guide pipes did change the observed efficiency decrease or “hump”.

The effect of distributor drip point density and liquid free fall distance to the structured packing on the “hump” would be investigated. The objective is to determine how important these distributor design variables are on the packing performance.

INTERNALS: Mellapak 250Y, and up to four different distributor designs; iC4/nC4 system at 20.7 bar (300 psia).

ESTIMATED TIME: Six weeks plus one week contingency.

NON-STANDARD COSTS: None.

#### Background and Discussion:

While there has been a large experimental effort to understand the effect of liquid distribution on structured packings, little has been done at higher pressure operation. Professor Zuiderweg believes that the efficiency loss develops in the lower part of the bed. Another school of thought is that axial mixing is involved and it is independent of bed height. Previous FRI work indicates that this could be a liquid maldistribution problem, since changing the distributor did change the observed “hump”. (Plus a slight liquid maldistribution problem would then cause a vapor problem.) Studying how distributor design influences the “hump” may also help understand long bed operations at lower pressures.

## DSP-14

### Parametric Study of the Effects of Misdistribution on Packing Performance

#### Expected Economic Benefit:

Greater confidence that new packed column design would meet design objectives. Less overage would be required in a new design to insure design requirements are met, resulting a capital savings.

#### Present Situation and Proposed Research:

Distributor maldistribution is known to cause poor packing performance, but it is difficult to quantify the impact of distributor performance on overall packing performance.

INTERNALS: Either structured or random packing.

ESTIMATED TIME: Twelve weeks operating time.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Conservative design rules are applied when design designing a packed column, often resulting in better than required separation being achieved. (Meaning too much capital was spent.)

## DST-2

### Two-Phase Inlets Interaction with Tray Capacity

#### Expected Economic Benefit:

Improved design criteria for two-phase inlets between trays and for reboiler returns will avoid premature flooding problems and allow satisfactory operation at design rates.

#### Present Situation and Proposed Research:

FRI has not collected any data on the influence of either two-phase flow inlets between trays or reboiler returns on premature jet flood. Commercial data show that premature flooding has been experienced due to the inadequate design of the above internals.

Premature flooding occurs preferentially in debottlenecking cases where existing trays have been either modified or replaced with higher capacity trays without implementing any modifications to either the two-phase inlets between the trays or the reboiler returns.

For both vapor/liquid inlets between trays and reboiler returns, the effect of the following variables on entrainment and premature flooding should be evaluated in an ISOPAR M/air simulator: 1) feed and reboiler inlet distributor type and design; 2) nozzle positioning; 3) typical supports for nozzles and pertinent trays; 4) flow regime; and, 5) vapor/liquid loadings.

Initial tests should be carried out using the most typical designs recommended in the FRI Design Practices Handbook such as using pipe distributors for vapor/liquid inlets between trays. After optimizing the inlet designs in the ISOPAR M/air simulator, they should be tested in the 4 foot diameter tower at FRI using both a low and a high pressure system to confirm or improve their effectiveness in commercial services. These services will exaggerate physical properties (i.e. liquid surface tension, vapor density, liquid viscosity, etc.) significantly different to those of the ISOPAR M/air system.

INTERNALS: Sieve trays with different feed and reboiler arrangements.

ESTIMATED TIME: Simulator Required. Column: Four weeks in the high pressure column with C6/C7 at 24 psia and iC4/nC4 at 300 psia.

NON-STANDARD COSTS: Simulator work most likely contracted out ( \$50,000).

#### Background and Discussion:

With adequate design of inlets, premature flooding problems can be avoided. One typical example of premature flooding in two-phase inlets between trays occurs due to excessive liquid carry over into the tray above the inlet. This excessive liquid carry over could be the result of the generation of small drops in the inlet piping in conjunction with the re-entrainment of liquid (froth) from the tray below. This problem has been eliminated during "squat" turnarounds by removing the tray above the inlet nozzle while simultaneously relocating upwards the existing inlet nozzle.

## DST-13

### Dynamic Model Verification of Tray Performance

#### Expected Economic Benefit:

Dynamic column simulation is used to verify control system strategy and is the heart of a training simulator for operators. Improvements in these models would assist in optimizing control strategy and operator training - before the column is actually started up.

#### Present Situation and Proposed Research:

Simplified tray hold-up models are used by dynamic simulations to insure rapid calculation, since the model needs to run faster than real time in order to be useful. FRI could obtain detailed time dependent composition and holdup data for a trayed column during a step change. This could then be used by the major dynamic simulation vendors to improve their internal models. The membership would also learn how good the initial model predictions were.

INTERNALS: Standard valve or sieve trays.

ESTIMATED TIME: Four weeks calendar time, one week operating time.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Dynamic modeling use is becoming more widespread and frequent. Users need to be assured that the dynamic simulation results truly match reality. And with computers becoming more powerful, more complex models can be used within the dynamic simulation while still allowing the simulation to run at least twice real time speed.

## DST-16

### Simulator Test – Effect of Intermediate Pressure Drop Device on Capacity In a Fouling Service

#### Expected Economic Benefit:

There is a need for high capacity devices that will work in a fouling service. This test attempts to use some principles seen in new high capacity devices. Do those same principles apply to standard devices used in fouling services?

#### Present Situation and Proposed Research:

As the hole area of a sieve tray is increased above about 16% the useful capacity of sieve trays no longer increases. As the hole area of Dualflow trays increases above about 29%, the useful capacity of Dualflow trays no longer increases (the number here may depend on tray diameter). High open area Dualflow trays are known to be unstable at high diameters. Perhaps pressure drop devices between sieve trays and/or Dualflow trays can improve the stability of the flow on the tray and allow the use of higher hole area trays. This will result in higher capacity.

**INTERNALS:** In a simulator, test Dualflow trays or sieve trays with an intermediate pressure drop device.

**ESTIMATED TIME:** One month in a simulator. Determine maximum capacity and observe flow patterns on the tray. Determine the capacity and pressure drop.

**NON-STANDARD COSTS:** Simulator work will probably be contract work ( \$20,000).

#### Background and Discussion:

New high capacity devices have high hole area decks with cyclonic devices above the decks to disengage the vapor and liquid. These cyclonic devices would probably be impossible to use in a fouling service, but does the pressure drop of these device promote increased stability on the tray deck? It may be possible to place intermediate devices such as grid packing or a very high hole area device (40-50% open area) between two operating trays (sieve or Dualflow) and increase the effective capacity while maintaining reasonable liquid distribution on the tray.

## DST-18

### Fouling Resistance Testing/Studies of the Relative Solids Handling Characteristics and Efficiencies

#### Expected Economic Benefit:

Understanding the performance of different contacting devices in fouling services has the potential to improve fractionation performance while running and to extend run lengths between cleanings in these services.

#### Present Situation and Proposed Research:

FRI currently has no system for testing performance in fouling services. The proposed research would identify two fouling systems; one based on “sticky” solids, and the other on hard solids, both in the range of 0.1 – 1.0%. These systems would then be tested with several different types of trays.

INTERNALS: Once the standard systems are identified, a wide variety of tray designs can be tested. However, it is proposed that the initial tests be on the following:

- Provalve trays
- MVG trays
- Dualflow trays
- Baffle trays

ESTIMATED TIME: Phase one of this project would be to identify the two fouling systems, the equipment to be used in the testing (FRI column, small simulator, small lab column...), and the cleaning procedure for the equipment. This phase would not require column time. Subsequent phases of the project would test various trays using the fouling systems. If the FRI column is used for the testing, then it is expected that a test involving all four of the trays mentioned above and one of the two fouling systems would require:

Initial column setup	2 weeks
Device changes after initial setup	3 weeks (3 changes at one week each)
Operation on all four devices	4 weeks
System cleaning	4 weeks

If the FRI column is not used for the testing, then this effort would be contracted out.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Little experimental information exists about the performance of different types of contacting devices in fouling services. Given the number of fouling systems encountered by member companies, there is a desire to develop a standardized test to provide data on relative performance of different devices in these services. At this point, it is not clear whether this work would ultimately be done in the FRI test columns or could be done in either an air/water simulator or in a smaller, easier to clean distillation column. The first steps in developing such a program would be to evaluate potential fouling systems, how equipment would be cleaned after they were used, and then decide what equipment should be used for the testing. After that, testing would proceed in the appropriate equipment.

## DST-20

### Measurement of Entrainment between Dualflow Trays

#### Expected Economic Benefit:

Dualflow trays are used in some critical services. Efficiency normally increases with increasing load. This makes it attractive to push Dualflow trays hard. An understanding of entrainment would assist the designer in finding the optimal design in critical applications. There is little understanding what role entrainment plays in limiting the efficiency of this device.

#### Present Situation and Proposed Research:

The entrainment between or from Dualflow trays has never been measured by FRI. The tests can be done in the FRI column.

INTERNALS: Test Dualflow trays in the low pressure column. May need to measure the entrainment by some innovative technique such as dye concentration. This technique might then be used on other types of trays to verify previous measurements. In previous cases entrainment was measured by using a dry tray where a downcomer could be used to withdraw the liquid from the tray and measure the flow rate. Unfortunately Dualflow trays do not have a convenient way to withdraw liquid from the tray.

ESTIMATED TIME: One hardware configuration (19% open area, 24" tray spacing) with FRI tests at 5 psia, 24 psia and 165 psia.

NON-STANDARD COSTS: None.

#### Background and Discussion:

There are no FRI correlations that predict entrainment for Dualflow trays. This test would be designed to allow extension of the existing sieve tray correlations to Dualflow trays.

## MD-1

### Review of State of the Art of Computed Mass Transfer (CMT) and Computational Fluid Dynamics (CFD)

#### Expected Economic Benefit:

CFD modeling is an alternative to column testing and may have significant cost savings and time advantages.

#### Present Situation and Proposed Research:

The current capabilities of CFD/CMT are not well understood within FRI. The first phase of a project in this area would be to review the state of the art of CFD/CMT and produce a Topical Report of the state of the art in these fields. This report would delineate the current uses of CFD/CMT and the near term/mid term/ long term uses of these tools.

INTERNALS: None required.

ESTIMATED TIME: Not required.

NON-STANDARD COSTS: Work would likely be contracted out ( \$15,000).

#### Background and Discussion:

CFD modeling has been applied to quite a few systems of interest to chemical engineers. At some point it should be interesting in analyzing industrial scale mass transfer. It would clearly be nice to have CFD capabilities through consultants that could at least screen FRI tests

Reference: Sun, Z.M., Yu, K.T., Yuan, X.G., and Liu, C.J., "A Modified Model of Computational Mass Transfer for Distillation Column", Chemical Engineering Science, Vol. 62, 2007, p. 1839-1850.

## MD-2

### Partner with Experimental Fluid Dynamics Laboratory

#### Expected Economic Benefit:

Partnering with an experimental fluid dynamics laboratory will streamline research efforts, reducing the time and cost to understand distillation phenomena.

#### Present Situation and Proposed Research:

Experimental fluid dynamics labs have developed instrumentation that would aid our understanding of basic fluid dynamics involved in our trays, packing and vapor distribution in large scale spray chambers. More specifically, it is recommended that FRI develop the capability to determine droplet size distribution so that de-entrainment studies can have a more rigorous background. This first phase would be a screening study to determine the available techniques for droplet size distribution and whether or not they can be applied in the FRI test columns.

INTERNALS: Trays, packing, and open spray chamber.

ESTIMATED TIME: No column time is required for this first phase. Approximately 6 months calendar time for staff to discuss with the experimental fluid dynamics labs and determine applicability to FRI systems.

NON-STANDARD COSTS: None for the first phase of the project. Any subsequent work would be contract work with the chosen lab.

#### Background and Discussion:

Background on one recommended experimental fluid dynamics lab, Coanda Research & Development Corporation, can be found online at: [www.coanda.ca](http://www.coanda.ca).

## MD-4

### Rate Based Systems

#### Expected Economic Benefit:

Reduced number of trays or reduced packing height due to ability to more accurately predict the component separation.

#### Present Situation and Proposed Research:

FRI are based on predicting how closely the mass transfer approaches equilibrium. It is possible that another conceptual approach would provide better predictions of distillation performance. Once such approach is the use of rate based mass transfer. FRI has not investigated in any major way this alternative computation approach. Perhaps it provides a more accurate framework to allow the modeling of mass transfer.

We propose to obtain a rate based computation tool and test it against FRI data. This may mean licensing a process simulator or working in partnership with a process simulator vendor. The tool would be used to investigate how well it does in predicting the existing FRI test data. It is expected that all the data necessary for the computations may not be available. Correlation from the literature will be tested to see if they are sufficient or if additional FRI experimental work is needed. A separate prospectus item would cover that work.

INTERNALS: None.

ESTIMATED TIME: No column time is needed. The initial phases suggested here are simple paper studies. They would identify what data is needed by a rate based distillation computation. The result of this work would be to propose specific tests to be conducted to get the necessary data to support input to the rate based distillation computations. In other words more prospectus items would be generated from this paper study.

NON-STANDARD COSTS: Most likely contract work ( \$25,000).

#### Background and Discussion:

There are a number of approaches to determining the amount of mass transfer that takes place during distillation. FRI's effort has primarily been towards how far the mass transfer extends towards obtaining complete equilibrium. Would a different approach provide a better prediction of the mass transfer?

There are also issues in applying the FRI correlations. The FRI work was primarily with binary systems. How does one apply these correlations to a multicomponent system to get proper results?

Please note that the information proposed here may also be required by a CFD approach to performing distillation computations. This approach here would use relatively simple computations for the flow pattern on a tray, but would investigate issues such as contact area. That information would most likely also be required by the CFD approach to solving the computations.

## MD-5

### Dividing Wall Column Work

#### Expected Economic Benefit:

Providing sample design/optimization procedures for Dividing Wall Column (DWC) systems will allow engineers, new to this area, to be more efficient with their time. Demonstrating the effects of vapor/liquid distribution around the dividing wall and demonstrating the ability to predict that distribution will greatly add to the comfort level when designing such system.

#### Present Situation and Proposed Research:

The use of DWC systems has become more popular over the last couple of decades. Modeling of these systems is normally done by stacking together multiple independent column models. The appropriate arrangement of these columns is non obvious. Convergence difficulties are common. In addition, the nature of these systems provides an extra degree of freedom that need to be used for optimization.

Over the long term, it is envisioned that this project will include both experimental and non-experimental work on DWC systems. If work on this project is desired, the FRI membership will decide if either or both are desired. The initial work will be a literature survey. After this, non-experimental work includes the use of computer simulation (such as Aspen Plus) to model several DWC systems. Recommended procedures would be produced that discuss the appropriate specification and optimization of these systems.

Experimental work would include the use of a dividing wall in the FRI unit. The ability to predict vapor/liquid distribution around the dividing wall and the effects of these distributions will be explored. The need for controllable vapor/liquid distribution around the dividing wall will be reviewed. If control is considered important, methods of control will be investigated.

**INTERNALS AND SOFTWARE:** A computer simulation package such as Aspen Plus is required for much of this work. The experimental work requires a dividing wall that does not need welding to the vessel wall. Possible column nozzle addition may be required.

**ESTIMATED TIME:** No more than 500 person hours of work are expected to be needed for non experimental part of this project. Funding of a student is a possible choice. The column work will be defined later.

**NON-STANDARD COSTS:** Most likely work contracted to a university ( \$20,000).

#### Background and Discussion:

Many papers have been published on DWC systems. An initial review of published information is the first step of this project. Several three product systems should be identified that cover a range of fluids and product ratios. Effective simulation methods should be produced and used to optimize the design variables such as the number of trays in each column section and the splitting of the vapor and liquid around the dividing wall.

## OR-3

### Heat Transfer in Empty Spray Sections in Vacuum Systems (Also See MD-2, Possible Combine)

#### Expected Economic Benefit:

Use in heat transfer sections of industrial columns, allowing reduction in pressure drop with increase in vaporization of distillates, reducing coke formation, maintenance and packing costs.

#### Present Situation and Proposed Research:

In 1987, FRI did experiments with empty spray sections. At that time it was shown that sprays are very good heat transfer devices and that the heat transfer occurred in a short distance from the distributor.

The proposed research is that analysis of the empty spray sections, by measurements of the pressure drop, heat transferred, height necessary and determination of the heat transfer coefficient. Sprays with different spray angles (90 and 120 degrees), different pressure drops (changing liquid reflux rates), and different types (full cone and hollow cone) will be tested.

The studies may reproduce the same behavior of the pumparounds, that is, a subcooled reflux liquid that condenses the upper vapor (total or not) forming the liquid product of same composition of the reflux liquid.

#### If possible, three other studies may be done:

- Two streams and one spray distributor: these two streams would be pumped from two individual pumps into one sole header, upstream of the spray distributor. The streams would be of different volatility. The heavier one would play the role of the pumparound in an industrial vacuum column, whereas the lighter one would simulate a lighter sidecut from the crude column. The purpose of this case study is to observe the feasibility of introducing two different feeds into one spray distributor: how this affects the heat transfer coefficient and whether the cone collapses from the vaporizing lighter feed.
- Two streams and two spray distributors: again two streams of different volatility, this time would be pumped from two individual pumps into two individual spray distributors, placed one above the other. The same aspects as in the previous case study should be studied.
- Two streams, one spray distributor and one gravitational distributor: again two streams of different volatility, this time would be pumped from two individual pumps into two individual distributors, the heavier feed going to the spray distributor whereas the lighter one to the gravitational distributor, just above the former. The same aspects should be studied. In a paper by Cai and Kunesh presented at AIChE in 1999, a pan distributor was employed, and just as with sprays, the heat transfer took place in a short height.

INTERNALS: No INTERNALS, just a spray distributor and the collector pan.

ESTIMATED TIME: 10-12 weeks of column time.

NON-STANDARD COSTS: None.

## OR-3

### Heat Transfer in Empty Spray Sections in Vacuum Systems (Also See MD-2, Possible Combine) (continued)

#### Background and Discussion:

The results obtained by FRI were very important, but the pressure drop was not measured. Using different types of spray distributors will allow better understanding of the behavior of empty spray sections and their use in industrial columns.

## OR-4

### Study Alternate Separation Process

#### Expected Economic Benefit:

As energy costs increase other low cost separation techniques will become more economic. This study will determine if there are devices that FRI should research beyond distillation.

#### Present Situation and Proposed Research:

FRI's work has primarily been with distillation. Perhaps it is time to begin investigating what research is needed in other separation techniques. In particular separation via partial freezing of liquid solutions should be investigated.

The first phase of this would be a literature review and paper study to determine what factors might make alternative fractionating techniques more attractive than distillation, what candidate technologies should be studied, and whether or not they can be studied in the existing FRI equipment. Later phases of the project would do the actual experimental work.

INTERNALS: None for this phase

ESTIMATED TIME: Probably requires three months of staff time. Hire a consultant to do the literature review and prepare the paper including proposed experimental work.

NON-STANDARD COSTS: Contract consultant for the first phase ( \$15,000).

#### Background and Discussion:

FRI work has primarily researched distillation as a fractionating technique. There are, however, other fractionating techniques. As energy costs continue to increase and equipment becomes more expensive, other separations methods which do not involve handling large quantities of vapor will become more competitive. This is only a first step towards investigating other separation techniques.

## OR-6

### Heat Transfer in Pump-Around Zones

Expected Economic Benefit:

Improved understanding and design of direct contact heat transfer zones of distillation columns.

Present Situation and Proposed Research:

Currently used correlations for sizing pump-around zones in distillation are 40+ yrs old and are thought to be unduly conservative. The impact of the spray contacting zone on overall performance is not adequately described, and generally the existing correlations are too simplistic.

There is very little independent research data on this topic. The data that is available tends to be only applicable for close boiling separations – and in this service very high heat transfer coefficients are obtainable.

A new research program to be undertaken by FRI is proposed as follows:

- Determine the heat transfer performance for spray and also packed column internals.
- Test systems to cover typical crude oil and crude vacuum distillates, with varying quantities of non-condensable (steam).
- Main parameters for the test program to include:
  - impact of process conditions (vapor, liquid loads, and MTD)
  - impact of non-condensable
  - impact of internals (bed depth, packing type, spray system)

It is likely that this program will require testing with crude oil distillates, and this will require FRI to build a new pilot plant facility with an electrically heated feed heater. It is envisaged that these tests could be carried out in a 12” diameter column with a 5 – 10 ft contacting zone.

**INTERNALS:** New experimental equipment; both sprays and structured packing internals would be tested.

**ESTIMATED TIME:** Approximately three calendar months would be required for a staff member to investigate whether equipment is available where the experimental work could be done on a contract basis or new experimental equipment is needed.

**NON-STANDARD COSTS:** This would be contract experimental work. Cost not estimated because it is not know where the work could be done.

Background and Discussion:

## OR-8

### Operation of an Air/Water Simulator

#### Expected Economic Benefits:

Air/water experiments are easy to monitor, provide fast information to fluid dynamic flow behavior and can supply additional information to FRI database. In addition to fluid dynamic research heat transfer investigations can be performed and are of high interest to the industry using aqueous systems.

#### Present Situation and Proposed Research:

The list of FRI experimental research projects includes numerous interesting topics that could be investigated using an air/water system. These projects currently compete for experimental unit time with interesting but time consuming proprietary equipment tests. This conflict can be resolved through the operation of an air/water simulator.

INTERNALS: None.

ESTIMATED TIME: The installation of a new/innovative air/water test unit will take a time frame of more than 1 year.

NON-STANDARD COSTS: Construction of an air/water test unit would cost roughly \$1MM. A likely alternative is to contract the work.

#### Background and Discussion:

There are research topics that can be also investigated in an air/water simulator instead of a distillation unit. Air/water test results will also enlarge the FRI data base and will allow fitting the FRI correlations to a wider range of physical properties. Samples are:

- Investigation of downcomer sealing predictions
- Capacity benefits of truncated downcomers for various valve designs
- Verification of CFD modeling of fluid flow on trays, packings and tower internals
- Performing CO<sub>2</sub> absorption tests in caustic solutions to determine effective mass transfer surface areas
- Studying CO<sub>2</sub> absorption in "high" viscosity liquids
- Investigation of fouling systems\
- Performing absorption tests with various chemical reactions in the liquid phase
- Studying heat transfer efficiencies of spray distributors or packing/trays devices
- Studying entrainment dynamics of tower internals.
- ----the list does not claim to be complete and further ideas are welcome to be added.

Running an air/water simulator in parallel to a distillation unit will maximize the information for FRI members and will reduce cost in bypassing distillation tests.

FRI has contracted two air/water tests at the Zhejiang University of Technology (ZUT) in China using their 4 foot air/water simulator. The cost of the tests was somewhat less than the labor and operating costs for a similar unit in the US. This represents a viable alternative to complete at least some of this work.

## OR-9

### CO2 Absorption Studies

#### Expected Economic Benefit:

Acid gas absorption is a huge and growing area. The economic benefits of any technical advances could be very large.

#### Present Situation and Proposed Research

Dr. Ralph Weiland, of Optimized Gas Treating, was consulted. Dr. Weiland suggested three ideas for research as follows:

1. Testing of new solvents such as potassium dimethyl glycinate promoted with piperazine
2. Amine regeneration, which has received far less attention than amine absorption
3. Heat transfer between phases during the quenching operation that will have to be done on flue gas before it passes to the absorption system

Dr. Weiland would be willing to consult occasionally on any such project.

FRI could collaborate with SRP, OSU or an industrial partner on such work. It is also probable that federal funding could be obtained, if FRI was willing to accept the associated paperwork difficulties.

INTERNALS: Tray and packing performances could be compared. High-cost internals would not be required.

ESTIMATED TIME: It might take 6 months and appreciable capital to convert the Stillwater Unit for absorption research.

NON-STANDARD COSTS: As a guesstimate, \$150K would be required to convert the Stillwater Unit for absorption research.

#### Background and Discussion

Many years ago, the Alhambra Unit was utilized, with difficulty, for absorption research.

It is possible that OSU and ConocoPhillips will soon be embarking on a joint CO<sub>2</sub> project.

It is believed that SRP is already working on regeneration research.

The conversion of the Stillwater Unit for absorption research is not recommended by the Technical Director (MRR) who feels that such a conversion would stop necessary distillation development for many years – if not forever.

## NEW PROSPECTUS ITEM OR-10

### High Pressure Valve Tray Testing

#### Expected Economic Benefit:

Extending the FRI valve tray database to high pressure distillation systems will reduce uncertainty on performance when choosing between different trays. Like packings, high pressure distillation testing of a moving or fixed valve tray will serve as a valuable reference against which new high performance valve trays can be compared.

#### Present Situation and Proposed Research:

Previous high pressure tests were performed on sieve trays only from 1964 to 1969 and 1987. To date there are no valve tray test data at pressure above 165 psia (11.4 bara). Testing of a generic valve tray with iC4/nC4 at 300 and 400 psia (20.7 and 27.6 bara) will:

- provide capacity, efficiency and pressure drop data to extend the physical properties range in the valve tray database.
- enable testing of the new FRI Valve Tray Capacity / Efficiency Models at High Pressures

**INTERNALS:** Six Phase-2 Moving Valve Trays with 17.8% Open Area (13.7% Vertical Curtain Area) 13% straight downcomers - an extension of the DST-7 valve tray program. Testing in the 4 foot (1.22 m) HP column of the Stillwater FRI hydrocarbon experimental unit.

Among FRI members there is a concern that 13% downcomers will choke flood, FRI will investigate a moving valve tray design with sloped downcomers, e.g. 3-to-1 sloping. The opinion of some members is that fixed valve trays are preferred as a more suitable 'reference tray' when comparing new high capacity mini-fixed valve trays (see Non-Standard Costs below).

**ESTIMATED TIME:** 3 to 4 weeks for one valve tray or 6 to 8 weeks if both a moving and a fixed valve tray are tested.

**NON-STANDARD COSTS:** If the consensus is to test a fixed valve tray, FRI will send an invitation letter for bids to the Vendor members (similar to the procedure for the High Pressure Random Packing DSP-10 program).

#### Background and Discussion:

As far back as 1998, the consensus was to establish a valve tray database along similar lines to sieve trays. Considerable progress was made during the DST-7 valve tray program when five generic round moving valve tray designs were tested in 2008-2011. High pressure testing of a moving valve tray is an extension of the DST-7 program. It would provide high pressure data relevant to TR 137s (valve tray capacity correlation)

In the case of fixed valve trays, high pressure test data would replace sieve trays as the baseline reference for comparing proprietary high performance trays.

## NEW PROSPECTUS ITEM OR-11

### Kettle Reboiler Entrainment

#### Expected Economic Benefit:

Distillation is the primary separation technology in the refining and chemical industries. Roughly \$100B/yr is expended producing heat to drive distillation columns across the world.

#### Present Situation and Proposed Research

The kettle reboiler of the LP Column was modified during the May 2011 turnaround. Two 6-inch windows were added. Also, 8 new taps were added so that additional pressure, temperature and hydraulic information can be collected. Overall heat transfer coefficients will be calculable using 3 different hydrocarbon systems at pressure from 75 mmHg to 165 psia. Steam pressure/temperature changes will yield film and nucleate boiling.

In the future, gamma scanning could be used to determine quantitative entrainment rates in the 18-inch vapor product nozzle. The deentrainment capabilities of mesh, packings and Eaton separators could be studied. The U values of new tubes or enhanced (e.g., coated) tubes could be determined. A borescope could be purchased. Asymmetric reboiling, bundle vibration and thermosiphon operation could be studied.

INTERNALS: No trays or packings would need to be purchased.

ESTIMATED TIME: Four years to study everything in the above discussion.

NON-STANDARD COSTS: Roughly \$30K per year for four years.

#### Background and Discussion

Fractionation Research Inc. (FRI<sub>(SM)</sub>) operates an industrial scale distillation research unit. The FRI Experimental Unit includes two kettle reboilers. Kettle reboilers are not well understood. HTRI's analysis of October 15, 2008 warned about high entrainment levels from the reboilers, but the impacts of the impingement baffles were unquantifiable. Entrainment levels in such exchangers are a strong function of the bi-phase pool height, and the flow over the internal weir. That bi-phase height is not accurately calculable. During the November 2009 Empty Column Test, gamma scanning showed little entrainment from the reboilers.

The goal of the proposed research is to gain a fundamental understanding of kettle reboiler operation and to obtain data for improving rating correlations.

## PPP-1

### Mass Transfer Efficiency – Different Physical Properties

#### Expected Economic Benefit:

More reliable design with lower safety factors for distillations involving non-hydrocarbon chemicals.

#### Present Situation and Proposed Research:

Fundamental theories for modeling of mass transfer efficiency predict that an increase in surface tension should reduce efficiency. However, the chemical systems tested by FRI (mostly hydrocarbons) are such that the effect of surface tension cannot be distinguished from the effects of other physical properties. In order to predict efficiency reliably, the model should be based on data that allows the effects of the various physical properties to be determined independently.

Efficiency data for a system whose physical properties relate differently from hydrocarbons will be collected at total reflux ( $L/V=1$ ).

INTERNALS: Sieve trays and a packing

ESTIMATED TIME: Three weeks for each contacting device or six weeks total.

NON-STANDARD COSTS: None.

#### Background and Discussion:

Hydrocarbons have been preferred as test fluids by FRI because of cost, corrosion, health, safety, and environmental considerations. However, the relationship between the various physical properties of hydrocarbons is very similar, so that it is difficult to determine which property causes any particular observed effect. Data on systems with a different physical property correlation are needed for model improvement.

Choosing an appropriate test system will be difficult, because many otherwise suitable systems contain chemicals that are toxic and/or corrosive. FRI does, however, have experience with some non-ideal systems such as propylene glycol and water.