

Industrial-Scale Up of Structured Packing for Cryogenic Distillation

Industrial Scale Up of Structured Packing for Cryogenic Distillation

M. A. Kalbassi and I. Zone
Air Products PLC, UK

Synopsis

Air Products has developed and built a unique, large-scale cryogenic test facility, which enables the design and operating boundaries of structured packing and column internals to be determined under true commercial air separation conditions. The data obtained from this unit has been extremely valuable in helping Air Products researchers understand the parameters governing structured packing system fundamentals. An important feature of this facility, despite the scale and inventory involved, is the versatility and speed with which column internals are tested and the data is made available to the design community.

Introduction

Structured packing has gradually become more prevalent in cryogenic air separation over the last 15 years. The need for high thermodynamic efficiency in the cryogenic distillation of the components of air (mainly argon, oxygen, and nitrogen) within an air separation unit (ASU) requires a large number of separation stages operating with small mass transfer driving forces. The pressure drop of structured packing is lower than that of trayed systems and so significantly reduces the energy required for the separation.

However, despite the significant advantages offered by structured packing, data in this field was limited because of the specific nature of the system. The physical properties are significantly different from many commonly used experimental fluids, such as alcohols, air/water, or hydrocarbons. To provide the best possible scale-up data and to understand and remove the technical risk associated with the design and operation of a large industrial air separation unit, a test facility must be built that is representative of the system involved¹.

Structured packings of one type or another have been available since the 1940s, although their widespread use really began in the 1950s. In the mid 1980s air separation companies around the world started to investigate the use of structured packings for the separation of air at cryogenic temperatures. By 1989 Air Products and others^{2,3,4} had obtained patents relating to the use of structured packing for air separation. Agrawal et al. discussed the advantages of low-pressure drop in an air separation plant⁵.

Given the benefits of packing and the need to reduce risk through high-quality performance data, Air Products decided to construct a large cryogenic test unit of a size sufficient to provide representative data for the successful design of a large plant. To complement the existing smaller pilot plant, the column diameter chosen was 900 mm.

The use of cryogenic vapours and liquids causes a number of problems that a researcher must overcome: e.g., safety concerns must be addressed, test units are expensive, and modifications are difficult, time-consuming, and expensive. Consequently, the large experimental facility must be designed and built correctly from the outset. Fortunately, the materials of construction are well known and therefore do not cause a problem. Normally, an air separation unit column is constructed from aluminium or stainless steel, which is contained within an insulating enclosure known as the “cold box.”

Scale-Up Issues

Kister lists 12 criteria that should be met by a distillation test unit that will result in successful scale-up of packing to full-size industrial units⁶. Not all of these are relevant for structured packing, but some are: the packing used, the installation method, and the liquid distributor should be identical to those commercially practised; tests should be performed over the entire operating range while observing for maldistribution and wetting effects; and results should be compared before and after flooding of the packing. Probably the most important criteria are to use a relatively long bed, a diameter that is large enough, and the same fluids that will be separated in the relevant industrial unit.

The first problem facing distillation designers is that much of the data available in the literature is for systems that have physical properties significantly different from those of cryogenic air, such as alcohols, air/water, or hydrocarbons. Changing to a fluorocarbon system gives physical properties that are closer, but they can be up to 60% different from the true system values. Typical values for systems are shown in Table 1.

System	Pressure	Vapour density	Liquid density	Vapour viscosity*	Liquid viscosity*	Surface Tension	$\alpha_{i,i}$
	bara	kg/m ³	kg/m ³	m ² /s	m ² /s	kg/s ²	
air/water	1.0	1.2	1000	15.1	1.03	70.8	3.5x10 ⁶
air/methanol	1.0	1.2	800	13.5	0.85	25.2	5.1x10 ⁴
methanol/ethanol	1.0	1.4	740	7.8	0.59	25.2	1.75
chloro/ethyl benzene	1.0	2.6	1010	3.5	0.46	25.9	91
toluene/octane	1.0	3.3	670	2.5	0.33	14.2	1.6
F124/F218	4.0	33.3	1433	0.4	0.12	8.4	2.2
Ar/O2	1.4	7.0	1250	1.0	0.17	11.9	1.05-1.5
N2/O2	5.2	21.3	750	0.3	0.12	5.5	2.5-2.6

* kinematic viscosity

Table 1 – Typical System Physical Properties

The second problem facing distillation researchers wishing to introduce new technology is how large a demonstration is required to convince those responsible for designing industrial-scale plants that the new technology will truly work on a large scale. In an ideal world, a pilot plant demonstration would be of a size large enough to show that there would be no issues when scaling up to the industrial unit. In the real world, a researcher must balance the size, and hence cost, of a demonstration against the risk and rewards that the technology will bring.

Various packing manufacturers and research programs have published results from column diameters varying from less than 100 mm to over 2000 mm^{6,7}. The larger columns tend to use air/water because of the ease of handling and moving the two fluids. Experiments using system-specific fluids tend to be performed in smaller units. However, as we saw previously, unless the physical properties of the two systems are close, a large test facility will not necessarily be representative of the specific distillation duty in which it will be used. Consequently, the assumption must be made that the smaller column data will scale up predictably.

In the air separation industry, where companies tend to individually perform specific research, information about column size is scarce. The new test column constructed by Air Products is over twice the diameter of air separation columns for which results have been published^{8,9}. Also, by having the ability to test long and short lengths of packing, the effect of bed height on a full size-column with corresponding liquid and vapour distributor system particulars can be investigated.

Test Facility Design

The design of the cryogenic test facility and its technical specification posed a unique challenge to the process designers at Air Products. There are several important aspects of the design of this test plant: it should have a large diameter and hold a long length of packing, it should be possible to change the structured packing and other internals easily, and it should still operate at true industrial conditions, i.e., the correct pressures, fluid compositions, and liquid/vapour (L/V) ratios. A necessity was that it should be possible to easily and quickly change the structured packing within the unit, and other internals such as changing or rotating the liquid distributor, while still maintaining Air Products' highly acclaimed safety standards.

Due to thermodynamic considerations, many of the separations within a cryogenic ASU operate with low mass transfer driving forces and yet highly pure products are normally required. This necessitates a large number of separation stages, so to minimise column heights there is an incentive to use longer bed depths before redistribution. By building a large diameter column, fully representative data can be collected, capturing any important diameter or distributor effects that may occur from scale-up. Also, the capability to take a long length of packing allows investigations on the effect of bed depth to be made.

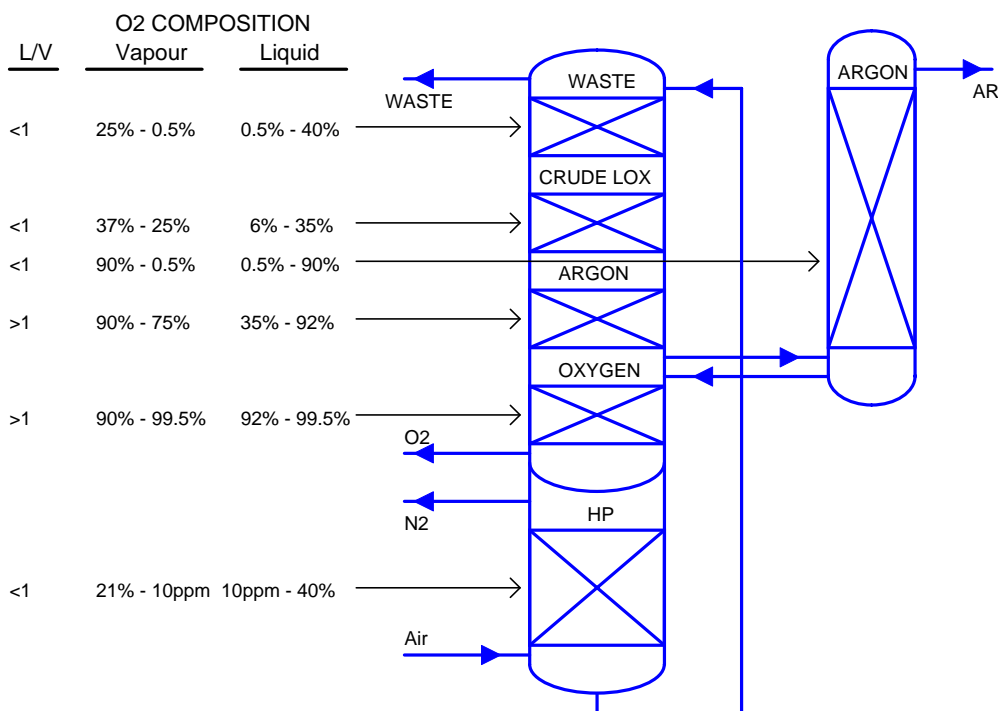


Figure 1 – Typical Oxygen Compositions Within an Air Separation Unit

To operate at true industrial conditions, the process was designed to operate at cryogenic temperatures and all the alternative conditions seen within an air separation plant, Figure 1. The capacity of the equipment within the plant was sized to operate over very wide flow ranges while maintaining highly accurate liquid-to-vapour molar ratio, flows, and compositions. Details of the operating envelopes are given in Table 2. This test unit gives Air Products the capability to test packing over its entire operating range using the exact conditions (binary and ternary) that it will see in service.

	Units	Min	Max
Molar L/V	-	0.3	2.5
Vapour	Nm ³ /h	1,700	25,000
Liquid	Nm ³ /h	1,500	30,000
Pressure	Bara	0	15
Oxygen	-	<1ppm	100%
Argon	-	<100ppm	100%
Nitrogen	-	<100ppm	100%

Table 2 – Operating Range Design

This is achieved by a process that uses two compressors and includes a heat pump to improve efficiency. A simplified schematic of the plant is shown in Figure 2 for just one of the many possible operating modes.

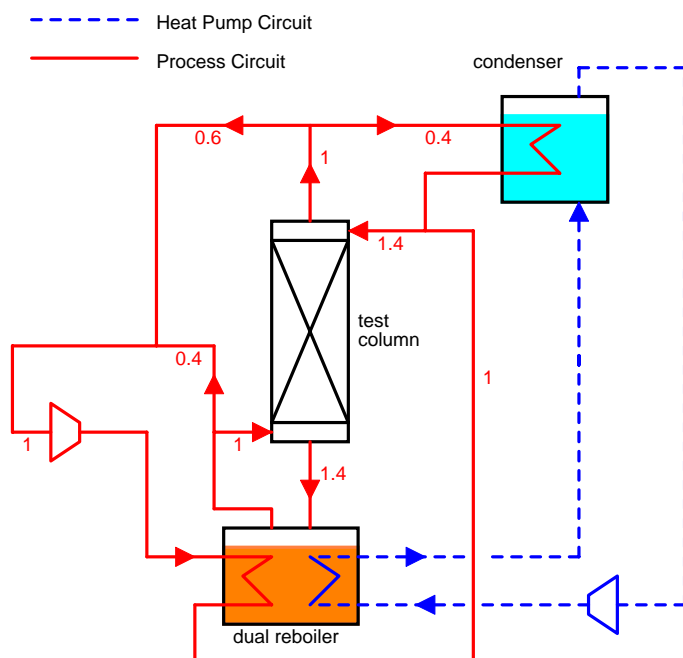


Figure 2 – Simplified Plant Schematic for $L/V > 1$ Operating Mode

Analysis, Control, Data Management, and Operation

By using a mass spectrometer, which gives repeatability of $<0.1\%$ across the entire composition range, highly accurate composition results can be speedily obtained, even in a ternary system. Additionally, the use of multiple parallel flow meters and valves allows optimum control and measurement to be maintained across an extremely wide range of flows and conditions.

Data for many possible conditions are calculated before testing to ensure the lowest possible error bands on the final data. Once the test conditions are finalised, multiple scenarios to cover the desired operating range are produced. When testing begins, data is collected from the plant's Distributed Control System (DCS) and transferred to a workstation where it is analysed for consistency. The iterative separation results are then computed and the results placed into a database. In less than 10 minutes after a test finishes, the results database is updated with the latest values.

Operation of such a large, complicated, and expensive test facility requires all the expertise used in running a normal industrial plant. Standard industrial control systems provide fault tolerance and safe operation, in addition to the R&D-specific installed instrumentation and analysis equipment. Air Products' long and safe operating history allowed us to select a team of highly trained operators with combined experience of over 100 years operating air separation units. Due to the scale of the operation, the plant runs 24 hours a day during a test series.

The size of the investment required in advancing air separation technology through the use of structured packing can be seen in the scale of the completed unit, shown in Figure 3. With a cold box volume of over 750m^3 , liquid inventory of over 20,000 kg, and dedicated compressors consuming $3 \times 10^6\text{W}$ when fully loaded, the total cost of the unit was several million US dollars.



Figure 3 – Air Products Large-Scale Packing Test Facility

Effect of Column Diameter

An important feature of the new test column was that it should be large enough to show any diameter effects over the smaller 200mm column already used. Figure 4 shows that the pressure drop curve for a packing of approximately $500\text{m}^2/\text{m}^3$, when used in two column diameters, is similar.

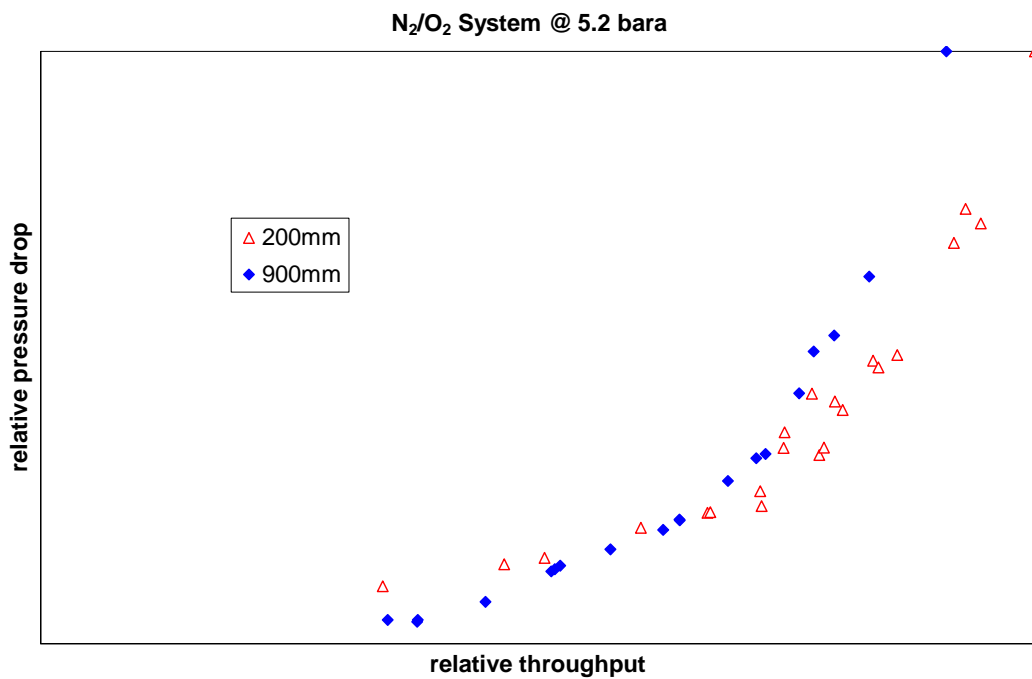


Figure 4 – Pressure Drop for Different Column Diameters, $500\text{m}^2/\text{m}^3$ Packing

When the graph of relative separation efficiency is plotted for the two diameters, Figure 5, a different scenario appears. The larger diameter packing has a Height Equivalent to a Theoretical Plate (HETP) much worse than when it was tested in a smaller column. By increasing the throughput above that of the smaller unit, it is also possible to confirm the flood limit with the 900mm column.

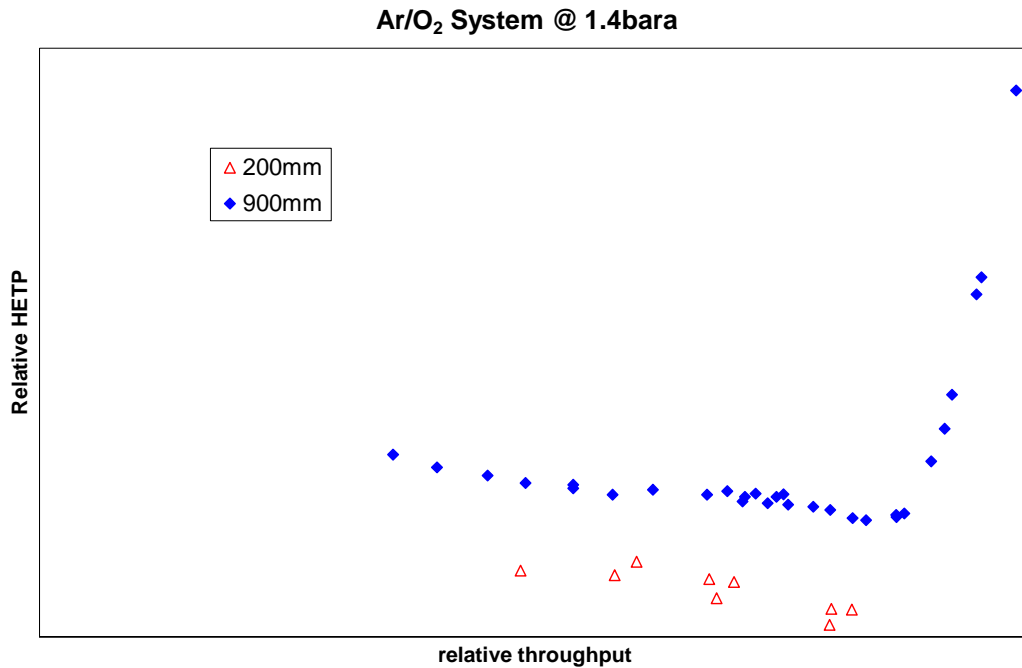


Figure 5 – HETPs for Different Column Diameters, 500m²/m³ Packing

Distribution of Liquid Composition Leaving Packing

One of the features of the large test plant is that it is possible to monitor the composition of the liquid leaving the packing at various locations, as illustrated in Figure 6.

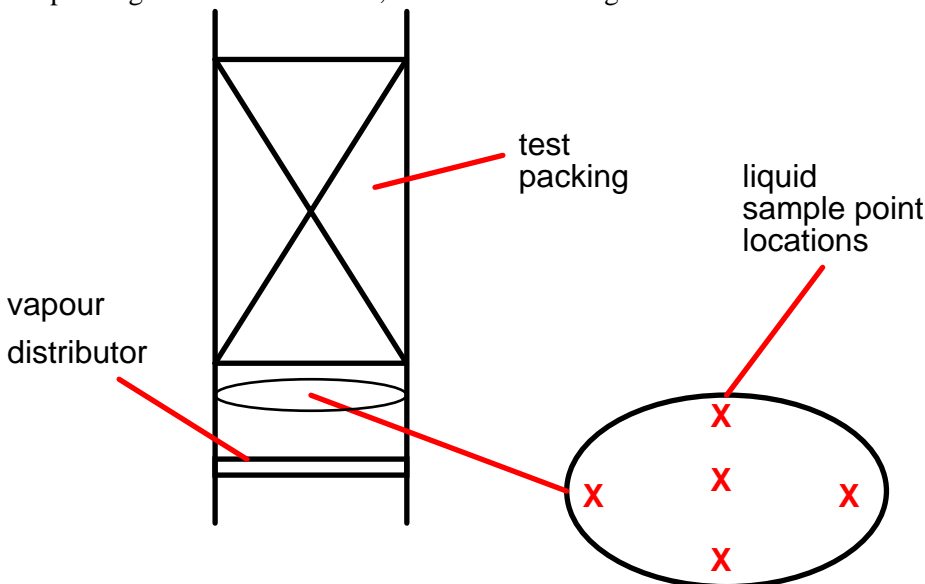


Figure 6 – Liquid Sample Locations Below Packing

This allows the local L/V ratios to be calculated at the five points, giving an indication of how the liquid phase is distributed by the time it leaves the packing. The variations in locally measured liquid argon compositions and the calculated local L/Vs are shown in Figure 7 for a short stack of packing. The same flow condition is shown in Figure 8 but for a long stack of packing, where the local L/V is calculated as:

$$\frac{L}{V} = \frac{y_t - y_b}{x_t - x_b}$$

x_t and y_b are the well-mixed feeds, y_t is assumed to be the same across the whole column when it leaves, and x_b is measured directly below the packing.

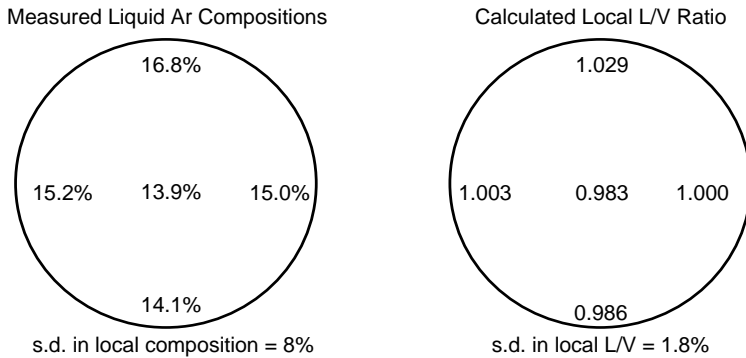


Figure 7 – Measured compositions and calculated local L/V ratios from liquid leaving a short section of 500m²/m³ packing, AR/O₂ under total reflux

In Figure 7 there is only an 8% standard deviation in the compositions of liquid leaving the packing. These compositions translate to the local L/Vs shown, which have a standard deviation of 1.8%. In Figure 8, for the longer stack of packing, the standard deviation of the compositions has risen to 34%, translating into a 3.2% standard deviation in local L/V.

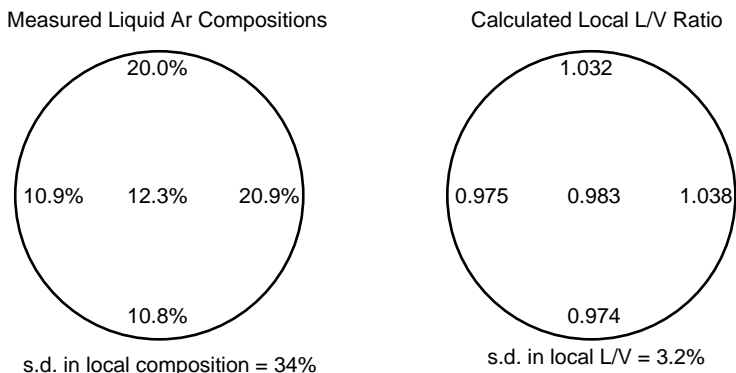


Figure 8 – Measured compositions and calculated local L/V ratios from liquid leaving a long section of 500m²/m³ packing, AR/O₂ under total reflux

For most common separations this would probably give perfectly acceptable separation. However, in the case of air separation, specifically oxygen/argon separation, it can lead to undesired performance. Figure 9 shows a McCabe-Thiele plot of a typical argon column.

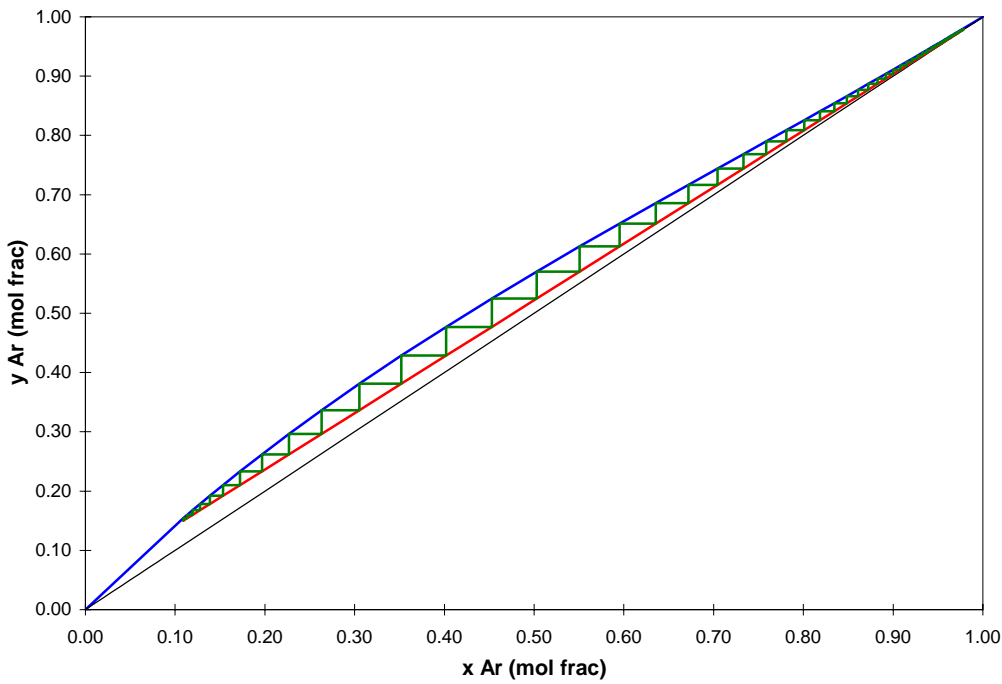


Figure 9 – McCabe-Thiele Plot for Argon Column

In Figure 10 the feed end of the column has been expanded. The solid operating line is now the overall operating line; the local operating lines are a spread of lines between the two bounds. The shaded area represents packing that is performing no separation function at all.

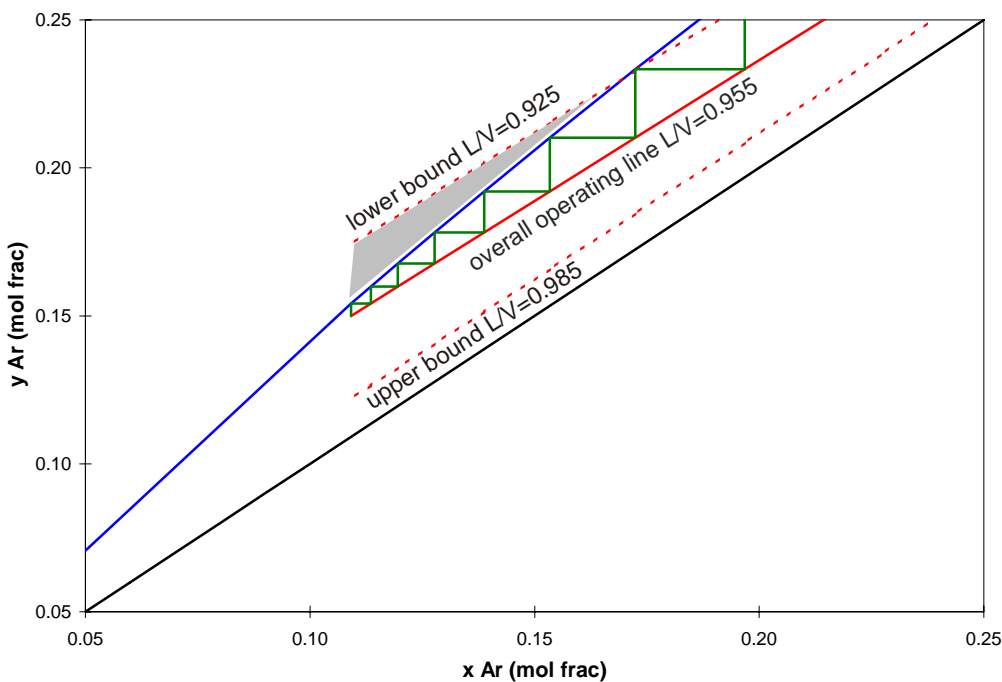


Figure 10 – Detailed Feed McCabe-Thiele Plot for Argon Column

Conclusions

Air Products has developed and built a sophisticated cryogenic test facility that enables true large-scale, structured packing design and operating parameters under exact commercial conditions to be determined.

The real benefits of this unit are its versatility and the speed with which the data is analysed and made available to the process design community. Inevitably, test campaigns have helped Air Products identify the operating ranges and the pitfalls of structured packing systems and internals when applied to a large-scale air separation plant and have further improved the reliability of our already exemplary cryogenic air separation technology.

The choice of a cryogenic fluids to characterise structured packing and being able to control the mass transfer driving force which is the know key determining parameter in structured packing performance has helped Air Products to accurately forecast performances expected from non cryogenic systems e.g. hydrocarbons. It is inevitable that our large scale facility at Carrington U.K could be utilised in helping the process industry to pin point and resolve structured packing and distribution scale up issues or help with understanding the determining features of structured packing geometry or liquid distribution.

References

-
- ¹ Eiden, U. and Kaiser, R., 1997, "Do We Still Need Plant-Scale Measurements in Distillation," IChemE Symp Ser 142.
 - ² Bennett et al., 1989, "Separating argon/oxygen mixtures using a structured packing," US patent 4,836,836.
 - ³ Victor et al., 1989, "Double column air separation process with hybrid upper column," US patent 4,838,913.
 - ⁴ Thorogood et al., 1989, "Air separation process using packed columns for O₂ and Ar recovery," US patent 4,871,382.
 - ⁵ Agrawal, R., et al., 1992, "Impact of Low Pressure Drop Structure Packing on Air Distillation," IChemE Symp Ser 128.
 - ⁶ Kister, H.Z., 1992, "Distillation Design," McGraw-Hill.
 - ⁷ FRI – Fitz et al., 1999, "Performance of Structured Packing in a Commercial-Scale Column at Pressures of 0.02 to 27.6bar," Ind. Eng. Chem. Res, 38(2).
 - ⁸ Krishnamurthy, R.K. and McKeigue, K., 1998, "Application of Structured Packing in High-Pressure Air Distillation Columns for Air Separation," AIChE Annual Meeting, Miami.
 - ⁹ Billingham, J.F. and Lockett, M.J., 1998, "Development of a New Generation of Structured Packings for Distillation," AIChE Annual Meeting, Miami.