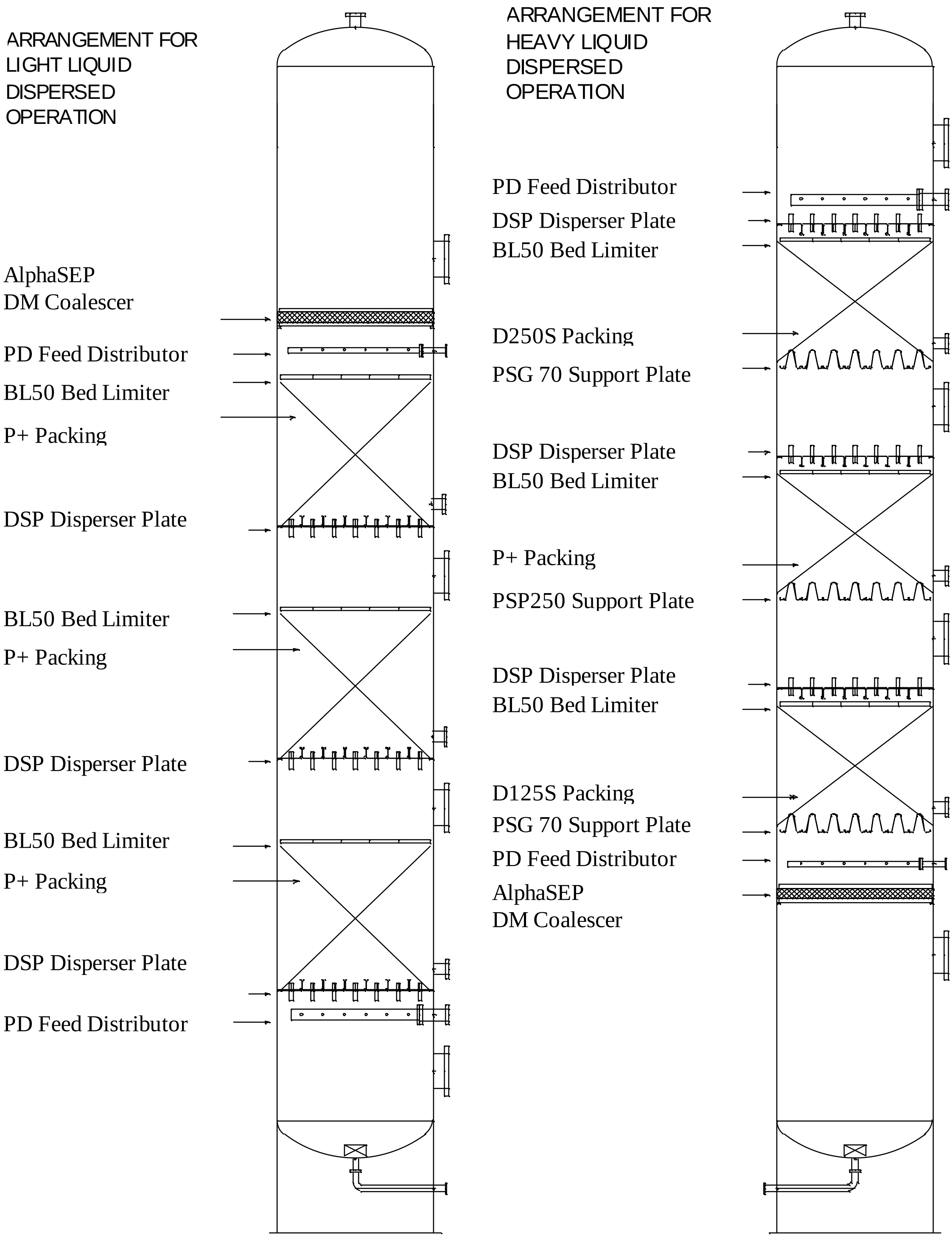
Liquid-Liquid Extraction

Typical Column Arrangements



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PACKED BEDS IN LIQUID-LIQUID EXTRACTION

General Arrangement

The typical arrangement above indicates the configuration of packing and internals in a column to perform in liquid-liquid extraction duty. The arrangement for operation with either heavy phase or light phase as the dispersion are shown separately. The actual number of packed beds and type of packing will vary according to the specific duty.

The operation of a packed liquid-liquid contactor is best understood by considering the unit as a series of mixer-settlers. The light phase is introduced to the column at the bottom and the heavy phase at the top to effect countercurrent flow. The dispersed phase is distributed evenly across each packed bed. The packed beds may be considered to be mixers which promote the transfer of the solute through the interfacial boundary formed by the dispersed phase droplets. The transfer of solute eventually results in a reduced concentration gradient at the interfacial boundary thus reducing the rate of mass transfer. In order to maintain mass transfer efficiency, properly designed disperser plates installed between each bed act as settlers by collecting, coalescing and then redistributing the dispersed phase droplets whilst at the same time allowing the continuous phase to pass in the opposite direction.

Liquid-liquid contactors can be operated with either light phase or heavy phase dispersed. In most cases it is more effective to disperse the phase having the larger volumetric flowrate in order to achieve maximum droplet surface area. However other factors should be considered in optimising the choice of dispersed phase such as column capacity and wetability of the packing surface as discussed below.

Where the heavy phase is chosen to be dispersed, the disperser plates are located above each packed bed. Alternatively, if the light phase is to be dispersed, the disperser plates are located below each packed bed and can be designed to function as supports plates for the packed bed.

Packed Bed Capacity

Capacity limitations in packed beds installed in liquid-liquid contactors can be considered analogous to those installed in vapour-liquid service. Increasing the superficial velocity of the continuous phase eventually causes entrainment of dispersed phase droplets which in turn leads to a rapid increase in dispersed phase hold-up. Increasing the dispersed phase superficial velocity displaces continuous phase within the packed bed until dispersed phase is forced to coalesce. The packed bed is considered to be flooding when either of these effects occur and the column will cease to function effectively.

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A number of correlations have been developed to predict the flood point of both random packing and structured packing in liquid-liquid service, the most widely used in the industry being that developed by Crawford & Wilke. Experience shows that at best a very wide margin of safety should be applied to designs which rely on classical flood correlations and that the influence of the design of associated internals, particularly the distributors and disperser plates, should not be ignored.

HAT have developed integrated design software to rate the capacity of packed beds in liquid-liquid service together with the hydraulic design of the distributors and disperser plates.

Packed Bed Efficiency

The mass transfer efficiency of a packed bed is as much dependent upon the bed height as the actual packing element size and surface area. Unlike vapour-liquid service where the packing surface is wetted by the dispersed (liquid) phase to provide optimum contact surface as well as surface renewal, in liquid-liquid service the packing surface is wetted by the continuous phase and mass transfer occurs only across the surface of individual droplets. The packed bed provides an increased flow path for the dispersed droplets (hence longer residence time for mass transfer) whilst promoting sufficient turbulence to reduce the tendency for the droplets to coalesce. Since there is little surface renewal, the rate of mass transfer decreases through the bed height.

Bed height optimisation will normally result in several packed beds each about 2 to 3 metres high with disperser plates installed between each bed rather than fewer taller beds

The packing should always be of material which is preferentially wetted by the continuous phase to reduce the tendency of the dispersed phase droplets from coalescing on the surface thus contributing to further loss of efficiency. In this regard thermoplastic packing are preferentially wetted by hydrocarbon liquids and ceramic packing is preferentially wetted by aqueous liquids. Metal packings are wetted by either hydrocarbon or aqueous liquids.

Most types of random and structured packing have been used successfully in liquid-liquid service. However it should be remembered that column packing is in most cases developed and rated based on performance in vapour-liquid service. Whilst hydraulic similarities between liquid-liquid and vapour-liquid are generally reflected in packing capacity, the mechanism of mass transfer and therefore the influence of packing surface on mass transfer efficiency is entirely different. Efficient distillation and absorption packings may not be efficient extraction packings.

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Column Internals

The specification, design and location of associated vessel internals is critical to the overall performance of a packed liquid-liquid contactor due to the close proximity of physical properties of the two phases. The need to optimise dispersed droplet size must be taken into account. As well as ensuring uniform fluid velocities across the column cross section, dispersed phase orifice discharge velocities must be limited to avoid the formation of emulsions due to shearing which are difficult to coalesce out.

An **AlphaPACK DSP** liquid/liquid distributor with 6mm orifices on a 60mm pitch will provide an optimum dispersion for most applications. However the orifice size and pitch must ensure that discharge velocity will not lead to emulsion formation whilst maintaining sufficient head of dispersed phase to ensure that it is completely coalesced.

The heavy feed liquid is introduced to the column through a **PD** orifice pipe distributor with orifices at the bottom of the pipes for downwards flow and continuous feed liquid is introduced to the column through an orifice pipe distributor with the orifices at the top of the pipes. The dispersed phase pipe distributor must be designed and located to function as an effective pre-distributor for the inlet disperser plate.

Sufficient space in the vessel must be allowed for separation of dispersed and continuous liquids to avoid product contamination and losses. The residence time required for separation is a function of interfacial tension. To improve separation, a knitted wire mesh coalescer pad should be installed between the continuous feed inlet distributor and the interface.

Amine Contactors

One of the more common liquid-liquid extraction applications is the removal of sulphur compounds from LPG using an amine solution.

A rough tower sizing may be based on an amine superficial velocity of 5 m^3/m^2 but a final check on column diameter should take into consideration all physical and transport properties at both the top and the bottom of the column. The actual ratio of amine to LPG is determined by the amount of H_2S and CO_2 removed but normally the LPG will be dispersed.

Careful design of the amine & LPG inlet distributors is required to avoid excessive amine carry over. Our experience tends to show that an orifice velocity of around 0.5m/s is about optimum in both cases. The LPG inlet should be located as close as possible to the disperser plate below the bottom packed bed and the amine Inlet below the interface at the top of the column.

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At least 2 metres of tower space and preferably at least 15 minutes residence time is required above the amine inlet to allow the dispersed phase to coalesce and disengage. An **AlphaSEP DM** PTFE coalescer pad should be installed below the interface. Sludge build up at the interface is detrimental to settling and can lead to amine carry over. A small draw-off nozzle located at the interface can be used to withdraw the sludge and avoid this problem.