DISTILLATION EQUIPMENT FOR REACTORS

- Boiling under reflux -
- Distillation -
- Hydrodistillation -
- Rectification -

De Dietrich
PROCESS SYSTEMS

Syntheses and product separation in one single apparatus

INTRODUCTION

In practice the processes “Reaction” and “Distillation” frequently will be executed consecutively, so that within the same equipment the starting materials react to the desired products and these products will be separated by distillation.

The combination of big stirred reactors made of glass-lined steel or stainless steel with distillation attachments made of borosilicate glass 3.3 represents an advantageous solution, which has been used successfully in the chemical and pharmceutical industries for many years. Specifically in the production of highly refined and highly pure products such as for example fine chemicals, plant protection agents, vitamins and pharmaceuticals, borosilicate glass 3.3 as an inert, metal-free and highly corrosion resistant material has an important position. Overhead construction made of borosilicate glass 3.3 suitable for reactions and distillation/rectification can be realized for reactions vessels up 10.000l (stainless steel or glass-lined).

Vessel attachments are suitable for different processes, such as, for example:

- Boiling under Reflux
- Distillation
- Hydro Distillation
- Rectification

The plants are operated at atmospheric pressure or under vacuum mostly in batch but also in continuous mode.

The vessel attachments can be flexibly connected via PTFE bellows directly to or laterally offset from the vapour outlet of the reactor. Vessel attachments are mounted in separate structures next to the vessels. The structure can be adapted individually to the available space conditions and leaves the required free space on the vessel cover for the stirrer unit, filling neck, illumination, sight glass, safety valves etc. Since borosilicate glass 3.3 is transparent, the process can be visually controlled at any time improving the safety and reliability of the production.

APPLICATION EXAMPLES

The are various design examples for different applications:

- Boiling under reflux and distillation with coil heat exchanger
- Boiling under reflux and distillation with shell&heat exchanger

- Distillation with phase separation
- Vacuum distillation
- Rectification

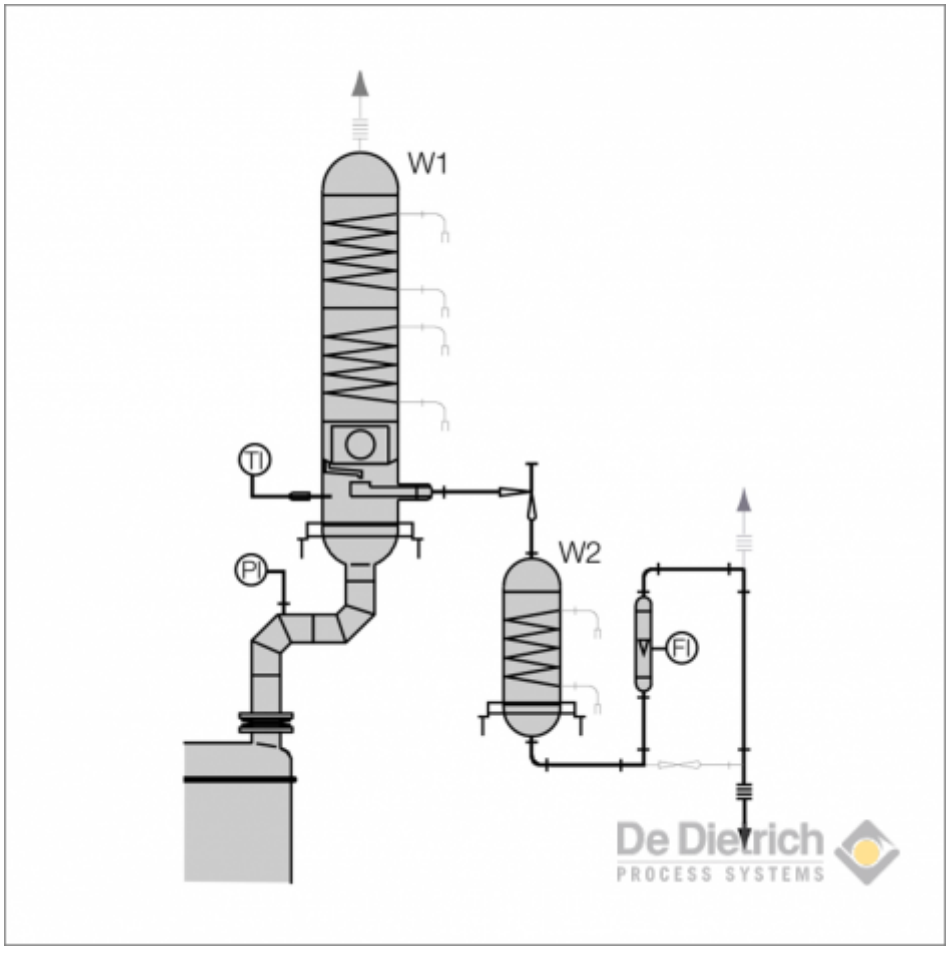
Boiling under Reflux and Distillation with Coil Heat Exchanger

This example shows a vessel attachment with uprising condensation means the vapour are moving upwards along the heat excanger W1 and condensing. The down flowing condenstate leaves the condenser W1 at its boiling temperature. Depending on the on/off postion of the valve above W2 the condensate is either guided backwards into the reactor or withdrawn.

The vessel attachment can be equipped with measuring and control devices according to the process requirements.

Mostly the following measuring instruments are sufficient:

- Thermometer (Pt100) - vapour line
- Flow meter with local display - distillate flow
- Pressure gauge with local display - vapour line

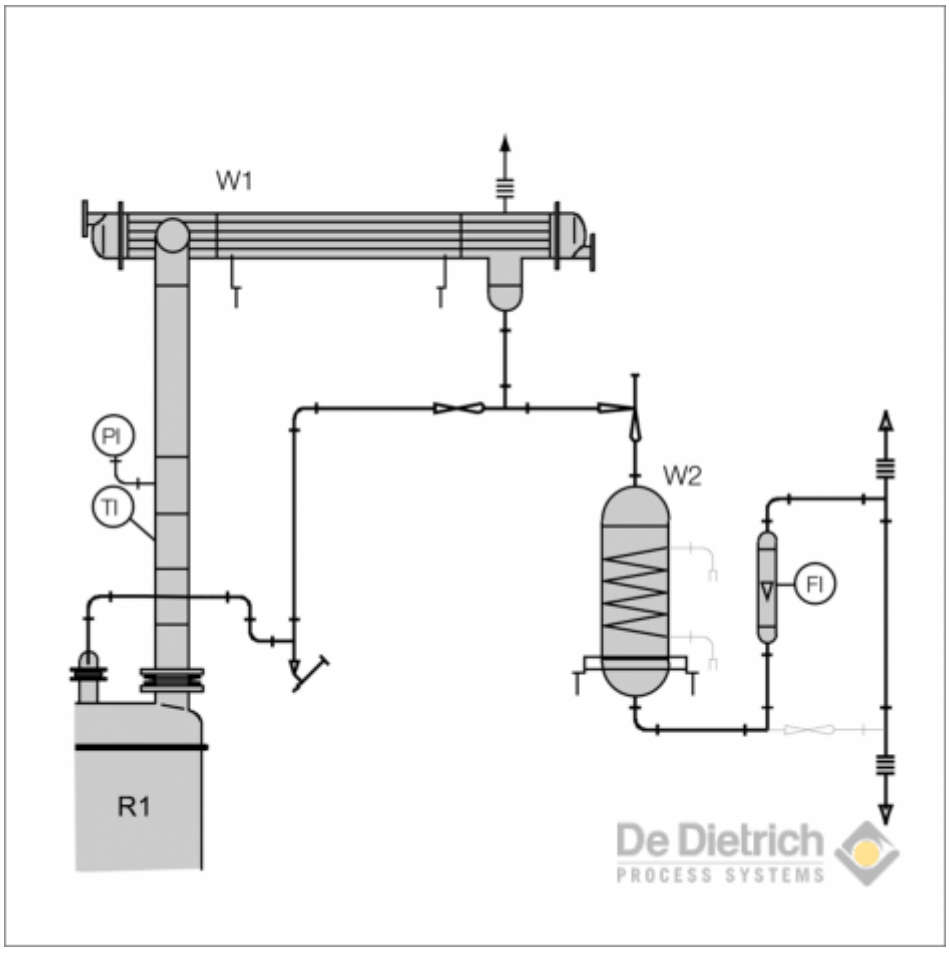


Boiling under Reflux and Distillation with Shell&Tube Heat Exchanger

The same process funtionality can be realised with a shell&tube heat exchanger. They can also be installed in an horizontal position to minimise the required height. The heat transfer rates are higher and the sizes of shell&tube heat exchangers can be bigger so that they are also suitable for bigger vessels than can be covered with coil type heat exchangers.

The vessel attachment can be equipped with measuring and control devices according to the process requirements. Mostly the following measuring instrumentation is sufficient:

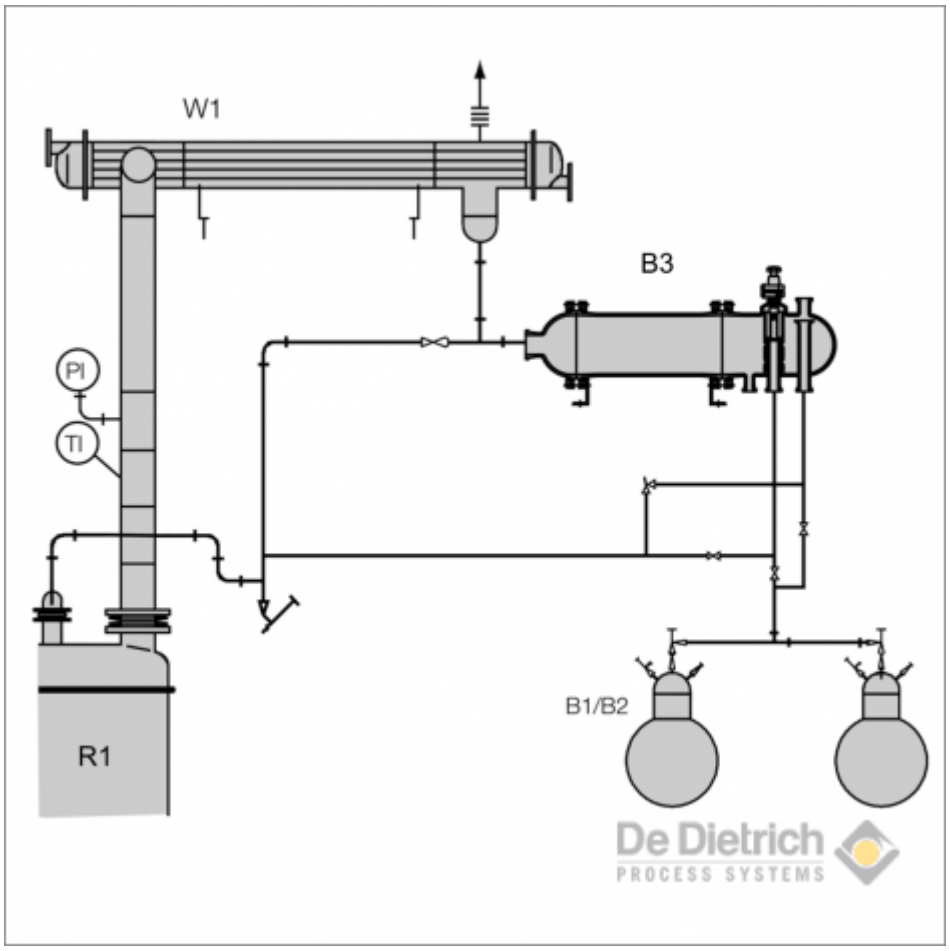
- Thermometer (Pt100) - vapour line
- Flow meter with local display - distillate flow
- Pressure gauge with local display - vapour line



Distillation with Phase Separation

In addition to the apparatus described above this setup includes a phase separation vessel for the distillate. Such setups are used when the distillate forms 2 phases as distillates e.g. for water removal during esterification, hydrodistillations and heterogenous azeotrops.The phase

separator permits the adjustment of the interphase and the separate withdrawal of the light and heavy phase. Both the light phase or the heavy phase can selectively be guided back into the vessel or withdrawn.



Vacuum Distillation

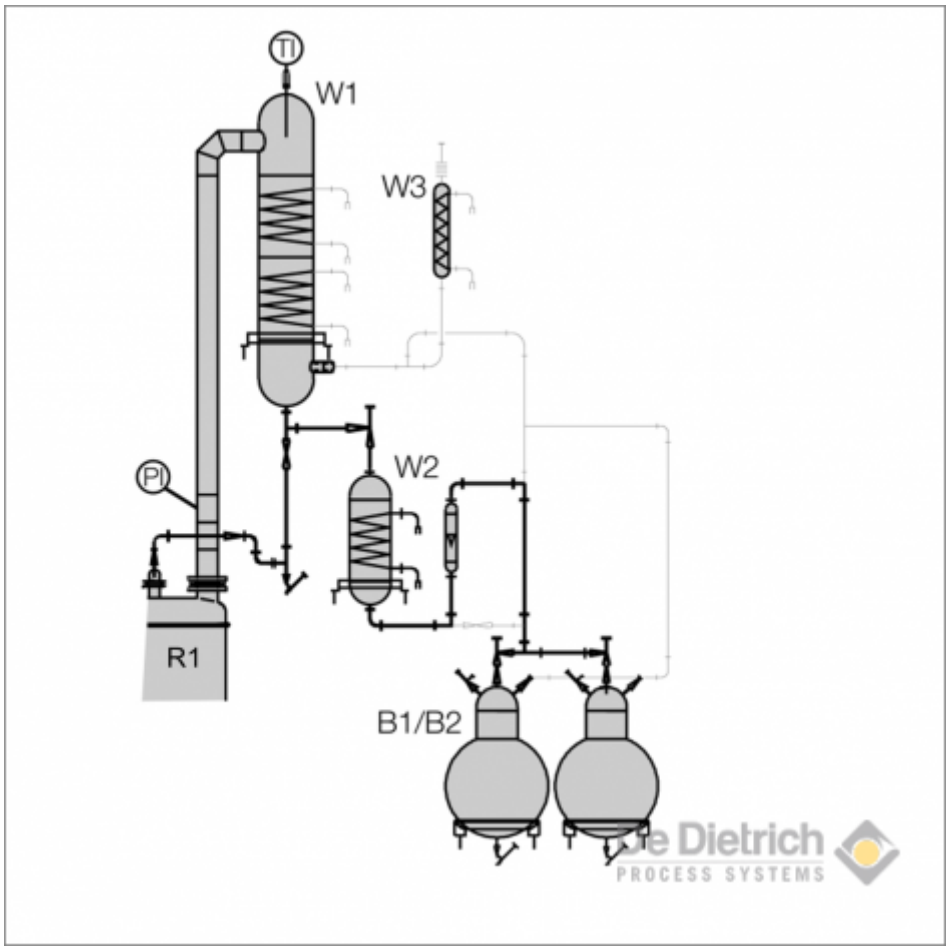
In this setup the vapours enter the condenser W1 and flow downwards along the heat exchanger W1. Contrary to an uprising condensation the principle of the descending condensation has the the condensate runs off with a temperture below its boiling point. This is especially advantageous for vacuum distillations as it reduces the amount of vapours leaving the condenser so that the distillate cooler W2 and the aftercooler (W3) operated at a lower temperature than W1 can be smaller if not even skipped. The condensate can either be withdrawn or sent back to the reactor. Boiling under reflux is of course possible but not always advantageous since the reflux is cooled below the boiling point of the distillate and cools down the reactor content. On the other side this can be an advantage if heat of reaction has to be taken out from the reaction mixture to control the reaction.

Additional alternate receivers are integrated, so that the distillate can be taken off during vacuum distillation.

The selected attachment can be equipped with measuring and control devices according to customer’s request and process request.

The following measuring instruments are normally included

- Thermometer (Pt100) - vapour line
- Flow meter with local display - distillate flow
- Pressure gauge with local display - vapour line



Rectification

Using a packed column C1 with a reflux divider between the condenser W1 and the reactor R1 volatile substances with closer boiling points can be separated by rectification from each other. The column height and the type of column internals are fixed in accordance with the separation problem and the available height. In order to maintain the advantage of a universal corrosion resistance they are advantageously made of borosilicate glass 3.3 or PTFE. We offer a wide range of such internals as:

- Bubble cup trays
- Raschig ring

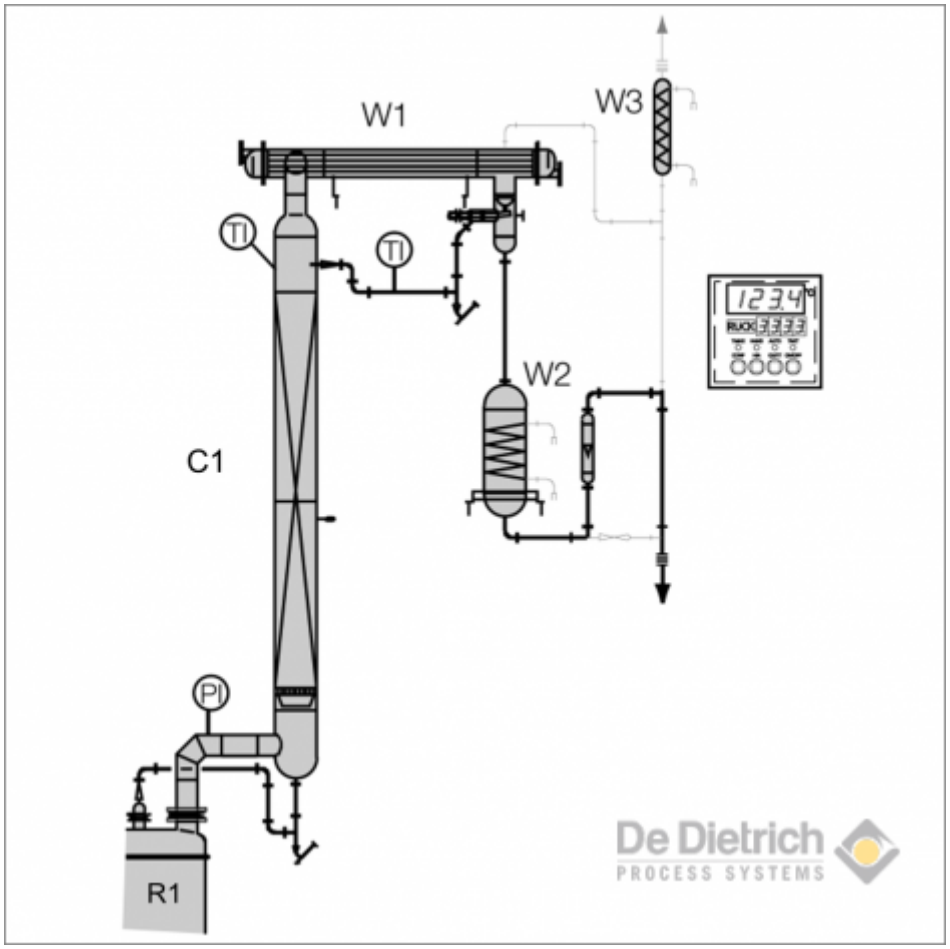
- Structure packing DURAPACK®
- Alternatively and depending on the process requirements also other types of random and structured packings made of other materials can be used (e.g. metals, plastics [PP, PVDF etc.], ceramics).

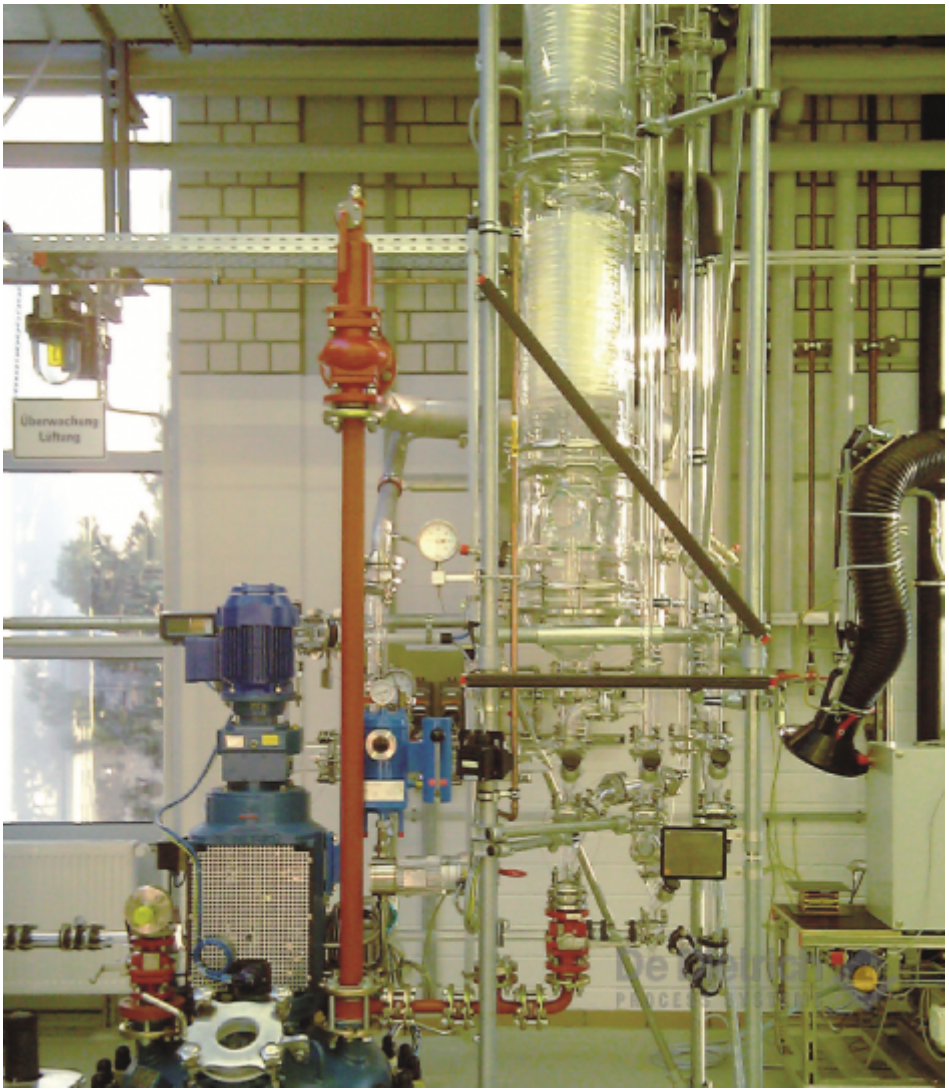
The reflux is fed into the column above the packed bed. The withdrawn distillate will be cooled down through the subsequent distillate cooler (W2) and finally taken captured by the receivers. The ratio between the reflux and he distillate take off required for the rectification is set by means of a magnetically controlled reflux divider in connection with either an electronic timer or a process control system.

Depending on availble height and required condensing capacity either tube & shell or coil type heat exchangers are used.

The standard instrumentation covers most applications:

- 3 Temperature measurements (Pt100) - vapour line entry, vapour line exit, distillate flow
- Flow measurment - distillate flow
- Pressure - vapour line
- Electronic timer for the reflux ratio

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