

Annex I: Response to Query B2.3 – Waste Recovery Plant  
Description

## ANNEX G

### RECTIFICATION PLANT

#### 1. Overall concept and utilisation of the plant

A rectification unit is studied in order to be able to separate most of the solvent mixture coming from the industrial processes applied for the production of active pharmaceutical ingredients. Mixtures like Acetonitrile/water, tetrahydrofuran/ethyl acetate, sec-butanol/acetone, isopropanol/ water, etc, must be disposed of and sent abroad for incineration because they cannot be recycled in the process. After distillation of these mixtures in the rectification unit, pure solvents can be obtained with the same technical specifications of the fresh solvent and they can be used again during the synthesis of new batches of API reducing considerably the amount of waste.

The rectification unit can be used to concentrate waste water solution coming from the production process as well in order to separate residue of solvent and inorganic compounds that can be destroyed abroad, from water that can be discharged into the sewage according to the local regulation. In this case too the reduction of the amount of waste is significant with the consequence of decreasing of the stocked wastes in house and the disposing expenses.

#### 2. Description of the individual components

The rectification unit consists of:

- 10m<sup>3</sup> vessel complete with stirring (boiler)
- Stainless steel distillation column
- Condenser
- Post condenser
- Phases separator
- N° 2 of 2m<sup>3</sup> collecting tank
- N° 2 of 5 m<sup>3</sup> storage tank
- No 3 of 35m<sup>3</sup> storage tank

2.1 10m<sup>3</sup> vessel complete of stirring: it is a stainless steel (AISI-316) vessel Equipped with an external jacket for heating with steam.  
Working pressure: 3 bar / vacuum; working temperature: 150°C  
Category: PED / IV.

2.2 Stainless steel distillation column: it is a 600 mm of diameter and 15 m long stainless steel column (AISI-316); it is filled with a high efficiency packing.  
Working pressure: 3 bar / vacuum; working temperature: 60°-150°C.  
Category: PED / IV.

2.3 Condenser: it is a stainless steel shell/pipe condenser with 30 m<sup>2</sup> of exchange surface area cooled with cooling water; working pressure: 3bar / vacuum; working temperature: max 150°C.

Category: PED I

2.4 Post condenser: it is a stainless steel shell/pipe condenser with 10 m<sup>2</sup> of exchange surface area cooled with ethylene glycol/water solution; working pressure: 3bar / vacuum; working temperature: max 100°C.  
Category: PED I

2.5 Phase separator: it is a 1.6 m<sup>3</sup> stainless steel tank used to separate not miscible solvents

2.6 Collecting tank: it is a 2 m<sup>3</sup> stainless steel tank (AISI-316) used to collect the distilled solvent.  
Category: PED I

2.7 Storage tank: it is a 5 m<sup>3</sup> stainless steel tank (AISI-316) used to store the distilled solvent during the analysis time.  
Category: PED I

2.8 Storage tank: it is a 35 m<sup>3</sup> stainless steel tank (AISI-316) used to store the distilled solvent before use.  
Category: PED I

### 3 Safety features of the plant with respect to any gas leaks and/or liquid leaks or spillage

The rectifying unit will be constructed utilising only stainless steel materials and PTFE gaskets (Teflon) which are very strong resistant materials regarding the corrosion in neutral or alkaline condition. The plant has been designed to reach 3 bar of pressure and -1 bar of vacuum and it will be tested at this condition, but in standard condition it will operate at atmospheric pressure (max 50 mbar of overpressure), hence the possibility to have a gas leakage in the plant is very low. All the vents of the unit will be connected to the scrubbing tower in order to minimize the residual solvent in the emissions. Different blowout disks and safety valves will be installed in specific positions and the piping will be connected to the existing blow-down tank in order to prevent the loss of solvents in case of emergency. Level cut off switches will be installed on the collecting tanks and on the storage tanks to avoid the possibility to fill up too much the tanks. The plant will be installed on a concrete platform with an around containment sufficient to contain any accidental liquid spillage: the eventual spilled liquid will be directly collected in a drain and it will be easily recovered using a pump.



The 35 m<sup>3</sup> storage tanks are contained in an external concrete reservoir able to contain the full volume of one tank in case of leakage.

#### 4 Safety measures with respect to safety in use

All the operations will be performed using nitrogen (loading/unloading of solvents mixture, distillation, transferring from tank to tank), in order to prevent the risk of fire and explosion. An overpressure valve (calibrated at 50 mbar) warranties a constant pressure in the system and a parallel pressure reducing valve regulates the introduction of nitrogen when is required. Temperatures, pressures, heating, cooling and safety systems will be monitored and controlled by a PLC system; this controller unit is just installed and at the present time is used to control the same parameters in the production plant. All the control systems will be validated and calibrated; all the instruments will controlled and calibrated periodically (as per internal SOP) in order to maintain them in good order of functioning.

If any anomaly occurs during the normal operation an alarm will appear on the PLC monitor and the boiler heating will be automatically stopped.

A fire fighting system will be installed in the distillation area starting from the existing fire fighting station according to the local regulation.