Montreal Protocol

Process Agents Task Force

Case Study #15

Reduction of perfluoropolyetherpolyperoxide intermediate for production of perfluoro-polyether (PFPE) diesters.

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CS-15 Reduction of perfluoropolyetherpolyperoxide intermediate for production of perfluoro-polyether (PFPE) diesters.

ODS type: **CFC 113**

Application

Reduction of perfluoropolyetherpolyperoxide intermediate for production of perfluoro-polyether (PFPE) diesters.

Process description

This process is for the production of PFPE diesters by reduction of Z-perfluoropolyether-polyperoxides (raw peroxi-oil). The reaction comprises the reduction of peroxy bonds of the raw peroxi-oil to carbonyl fluorides and the esterification of the latter with ethanol.

Process flow diagram is reported in fig. 3. The process is carried out in a single step in a batch reactor. A solution of the raw peroxi-oil in CFC 113 is added to a solution of a catalytic amount of elemental iodine in ethanol/water mixture. The resulting mixture is separated in two phases. Sulfur dioxide is then fed at a proper rate to support the reduction of iodine to iodide, which in turn reduces the peroxy bonds in the fluorinated phase, yielding elemental iodine, which returns to the ethanol phase to continue the catalytic cycle. The reaction conditions are 40 °C, at atmospheric pressure.

The off gas (COF_2, unreacted SO_2, CFC 113, HF) is collected to an alkaline scrubber and then sent to a high temperature destruction unit. The liquid reaction mixture is fractioned in a batch distillation column in order to separate CFC 113 from the other components. CFC 113 is recovered and recycled.

Reason why it is needed

The presence of CFC 113 in necessary because this substance is the only known process agent compatible with both phases: the perfluoropolyetherpolyperoxide (phase 1) and the ethanol-water mixture (phase 2). Compatibility of the two phases is essential since the reaction proceeds thanks to the continuous rapid exchange of I_2 from phase 2 to phase 1 and HI from phase 1 to phase 2.

Quantity used

37 t was used in 1997
A consumption of 80 t is expected in year 2000.

Emissions

Emissions are practically zero. CFC 113 is separated from the diester and recycled. All unrecycled gaseous emissions are conveyed to a specially designed unit where they react with water at very high temperature and are fully converted to CO_2, HF and HCl. Total (organic and inorganic) fluorine emission allowed from this unit by local authorities amounts to less than one gram per hour (< 1 g/h)

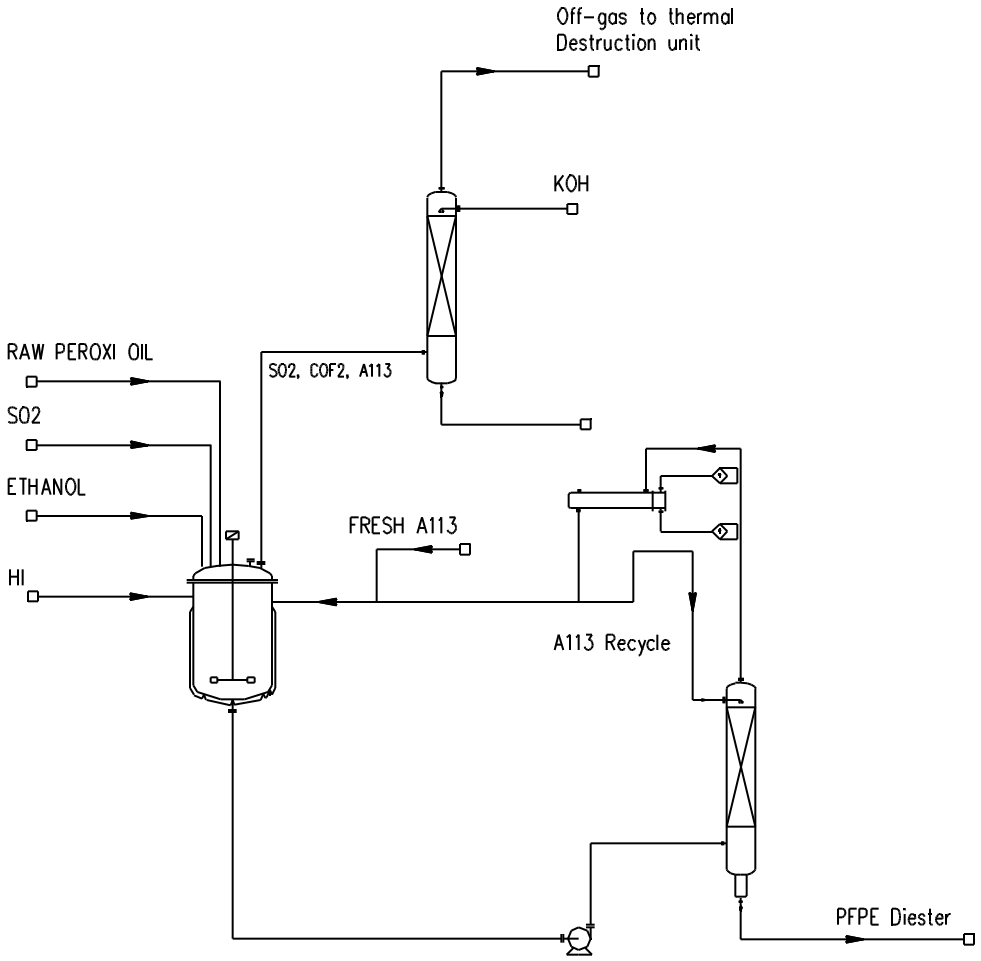

Fig. 3: PFPE diester process