Summary - Solvent Recovery Validation Example

This guidance provides an example of the contents that may be found in solvent recovery validation documentation, including appropriate acceptance criteria for solvent recovery validation. The processing addressed by this type of validation includes the recovery of used solvent to provide solvent that is acceptable for use.

If the potential for significant variability in the feedstock of used solvent exists, it is recommended that validation batches be prepared over a significant period of time to encompass the likeliness for variability in the used solvent feedstock. A good practice that has been used is to include validation batches made with an intervening time interval between each, as shown in the example provided later in this guidance (see acceptance criteria section). The increased risk introduced by potential variability in feedstock is therefore addressed by evaluating more validation batches over a longer time period.

The remainder of this guidaqnce provides one example of validation of a batch solvent recovery process that of the recovery of tetrahydrofuran (THF) from a used THF/toluene mixed solvent . The used THF may be derived from multiple process streams and the recovered THF will be available for mult-process use. While this example includes the use of computer simulation to predict when to make cuts for collection of distillate fractions, validation of the process may also be achieved with appropriate in-process monitoring.

In preparation for the final distillation, information on the composition of each batch of used solvent to be distilled is needed. For THF recovery, the computer simulation does not account for isopropyl alcohol (IPA) in the stream, so it is critical that the amount of IPA is NMT 0.1 vol-%. Feed composition that is higher in IPA will negatively impact the quality of recovered THF by being unacceptably high in the total alcohols (C1-C4) specification. Used THF/toluene streams with higher amounts of IPA should be directed through the optional processing step for removal of alcohols.

To validate this solvent recovery process, THF will be recovered from three sets of at least three consecutive batches of used THF. The time period between the beginning of the last batch in a set and the first batch of the next set will be at least one month. The total number of batches will therefore be no less than nine.

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