

6555

STEAM DISTILLATION APPARATUS OPERATION

METHOD #1

Place H₂O and sediment etc. into flask and heat to produce steam and other volatiles.

Prior to operating, place heavy phase (H₂O) in lower section of extractor until it overflows into flask, then add extracting solvent on top of H₂O layer. Needless to say, the solvent must be less dense than the H₂O.

Start distillation, condensing all vapor. The condensate runs down through the extracting solvent (organic) and joins the H₂O layer. The excess H₂O returns to the flask via the overflow tube.

A longer distillation time is required if the H₂O layer is not added first and solvent boil up may occur because there is limited contact with the condenser.

NOTE! If the organic layer boils below 100°C, it will eventually boil within the annulus if the condenser water temperature is high or insufficient water is flowing.

Some organics form azeotropes with H₂O, i.e., toluene, which boils well below H₂O and the vapor temperature will remain below 100°C until these are removed.

METHOD #2

Place a minimal amount of low boiling heavy solvent in the flask and lower part of the annulus, then add a small amount of H₂O sample above.

Extract the H₂O sample with condensate, then drain off solvent and sample, close stopcock, and remove solvent to condensate extracted material.

NOTE! The extracted material must be significantly less volatile than the solvent or it will be lost to the distillate. This can be checked by running at total reflux and sampling the condensate.



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