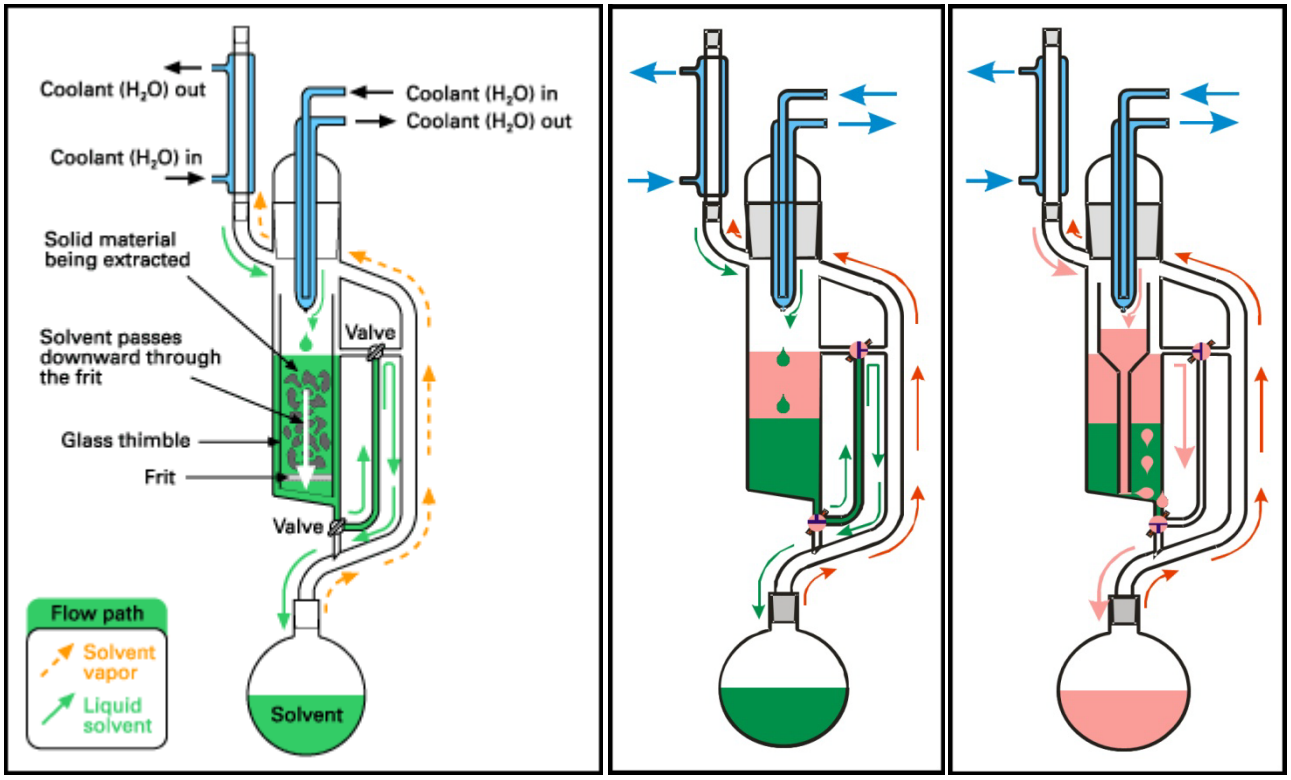
CG-1375 GREGAR EXTRACTOR TECHNICAL NOTES



The Gregar† extractor was designed to perform a variety of different types of extraction, thus eliminating the need for multiple extractors. It is capable of performing solid-liquid (S/L) and liquid/liquid (L/L) extractions, including L/L where the solvent is of either higher or lower density than the (immiscible) liquid being extracted.

The Gregar extractor consists of several elements: a body, a narrow inner side-arm for liquid flow, a broader out vapor arm, a primary condenser (usually a "cold finger" type), a secondary condenser (optional), and a collection flask. There are two glass or Teflon valves, one located below the body at the base of the side arm, and the other at the top of the side arm between the body and the vapor arm. The condensers must be cooled during operation; normally by a slow flow of tap water, but other coolant can also be used if necessary. For solid/liquid extractions, a glass thimble with a porous frit at the base is also used and for (some) liquid/liquid extractions a glass solvent guide (essentially a tall narrow funnel) is used.

For extraction of solids (or in other cases, semi solids), the material to be extracted is first loaded into the glass thimble. For very fine or "gooey" samples, it can be helpful to use a traditional paper thimble inside the glass thimble as this will not impede the extraction and helps to keep the glass frit at the bottom of the thimble clean. For coarse samples, the glass thimble alone is all that is needed. Place the thimble (with sample inside) in the extractor body.

There are two valves on the extractor, one below the body and one in the side arm. Each is able to be configured in several different ways to direct the solvent flows in accordance with the requirements of the extraction. For S/L extractions, the valve below the extractor body is set so that the solvent flows out of the bottom of the body of the extractor and UP the side arm. (If you think of the valve as a "T", then this valve should be configured so that the "T" is inverted. This means that the body and the base of the side arm are connected, but that solvent can't drain out of the body directly back into the collection flask.) The valve in the side arm is configured so that flow moving up the side arm is directed outwards into the vapor arm (with the leg of the "T" facing towards the vapor arm). In this case, the vapor arm also serves as a liquid return path back into the collection vessel (usually a round bottom flask), but that solvent is prevented from flowing out of the body at the level of the top of the side arm.

Used in this way, the extractor operates as follows: Solvent in the collection flask is heated to boiling and the vapor travels up the side arm to the condenser. At the condenser the vapor is converted back into a liquid and drips down into the thimble where it contacts and extracts the sample. As solvent accumulates in the thimble, it is forced out the bottom of the thimble by gravity and fills the body of the extractor and the side arm until it reaches the level of the side arm valve and which point it flows out of the side arm valve and returns to the collection flask.

Unlike traditional Soxhlet extractors, the body of the extractor stays full at all times and flow over/through the sample is continuous. The body does not cyclically fill and empty and hence the sample is not packed by the draining of the solvent; which can result in very inefficient extraction since the solvent simply goes around the sample rather than through it. The

sample is continuously immersed in the solvent which also helps to make extractions more efficient and effective were the rate of extraction is limited by diffusion limitations.

For L/L extractions where the extraction solvent is more dense than the liquid being extracted (e.g., extraction water with a chlorinated solvent such as CHCl3 or CH2Cl2), the extractor is configured exactly the same as for S/L extractions, except that the thimble is unnecessary. Add a little solvent to the body of the extractor to create a liquid barrier at the bottom of the extractor body (to prevent some of the less dense liquid getting into the side arm) then add the liquid to be extracted. As the solvent is boiled in the collecting flask, the vapor passes up the vapor arm and condenses at the condenser and then drips THROUGH the solvent being extracted and will be displaced out of the body via the side arm.

For L/L extractions in which the solvent is less dense than the liquid being extracted (e.g., extraction water with ether or pentane), the system is configured a little differently. In this case, a solvent guide is added and the side arm valve is turned so that solvent in the body is able to flow out of the body at the level of the side arm valve and then flow DOWN the side arm. The valve below the extractor body is also turned so that in this case, there is no flow out of the bottom of the extractor, but rather, flow coming down the side arm is able to flow out of the side arm and return to the collecting flask via this path (With the "T" upright, so to speak). Vapor distilling from the collection flask passes up the vapor arm, condenses at the condenser and drips down into the solvent guide. As solvent accumulates in the solvent guide, the weight of the solvent will displace liquid in the guide out at the bottom of the solvent guide at which point it will float UP through the liquid being extracted and hence extracts the higher density liquid. The valve at the top of the side arm is turned so that the leg of the "T" is towards the body of the extractor. This allows solvent o flow out of the body at the level of the top of the side arm and the flow down the side arm. The solvent collects on top of the liquid being extracted and spills over out of the extractor body via the side arm as described above.

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