Using Separatory Funnels

This SOP is for the use of the separatory funnels and other similar equipment.

Minimum PPE is standard plus safety goggles. Always use separatory funnels in a fume hood. Appropriate gloves suitable for the contents should be worn. The gloves must permit sufficient manual dexterity and grip to safely handle the full funnel. Separatory funnels commonly come from 30 mL to 6L in size. Before using a large separatory funnel full of hazardous materials, verify that the worker can safely handle the size and weight of the intended funnel full of DI water.

Before use, make sure the stopper fits properly and is liquid tight. Filling the funnel 50% full with DI water is a good way to conduct the test. Make sure the stopcock is assembled correctly. If the stopcock plug is Teflon, make sure the Teflon ring goes against the glass of the stopcock body followed by the rubber O-ring (SS spring in some cases). The nut is then tightened until the plug turns smoothly without any side-to-side movement. This is working state of a Teflon stopcock, not the storage state. Teflon stopcock would allow flow under even moderate pressure. Therefore, when the stopcock is not in use, loosen the plug significantly. If the funnel is fitted with a glass stopcock, make sure the plug is greased adequately. If the plug turns smoothly in the stopcock (which should have a clip to help hold the plug in place) then it is ready to test. Shaking the funnel 50% full of DI water should not cause any leakage at either the stopper or the stopcock.

The ring and support stand should hold the funnel securely and not be sagging under its weight. Larger funnels require heavier support rods (5/8 instead of ½) and similarly very heavy support rings. Adjust the height of the ring and separatory funnel appropriately for proper discharge of the two immiscible liquids.

To use the funnel, add sequentially the solvent and the aqueous solution, using a funnel of appropriate size. Fit the stopper to the funnel. Grasp the funnel firmly and remove it from the support ring. Invert the funnel (double check the stopper as this operation is performed) and vent the stopcock by opening it slowly while pointing the funnel towards the back of the hood. Close the stopcock, shake the funnel vigorously several times (holding a finger over the stopper) and vent the funnel again. If pressure is noted continuing shaking and venting until no excess pressure is released (5-6 times is normal). Place the funnel back in the support ring and let the layers separate. This may take a fairly long time, especially on larger scales, where the layer densities are closer than average, or when surfactants form part of the mixture. Once the layers are separate remove the stopper place the vessel intended to hold the denser layer under the funnel and drain that layer off. Larger funnels can develop a whirlpool effect, remixing the layers, if they are drained too quickly. Slowing the drain rate will eliminate that problem. Close the stopcock when the denser layer is entirely taken off. Gently tap the funnel to make sure none of the denser layer adhered to the glass. This is more often an issue with
some aqueous layers, but can occur with organic solvents as well. If the less dense layer is to be extracted with a fresh portion of the denser layer, add the fresh aliquot through the top using a funnel and repeat the agitation process. Repeat as necessary. If the denser layer is to be extracted again, drain the less dense layer into a separate vessel, close the stopcock and add the denser layer back to the separatory funnel. Add a fresh aliquot of the less dense layer and agitate as before. Repeat as necessary. Continue as necessary taking all appropriate safeguards for the reagents involved.

Special precautions need to be taken when a carbonate or bicarbonate wash is used after an acid wash to completely neutralize the solution. A significant amount of carbon dioxide gas could be generated by this process, enough to burst the funnel in some cases. Always use a water wash between the acid wash and the carbonate wash to help mitigate this potential problem. Also once the carbonate solution has been added to the funnel (already containing the solution to be neutralized) swirl the funnel in the support ring with the stopper off until no more gas evolution is observed. When the funnel is stoppered and then inverted, vent it immediately. Extraction can continue as normal from this point.