

Gas-Circulating Apparatus (August 2019 Update)

This apparatus is utilized to flow air/oxygen through the reaction mixture to promote oxidation. One would commonly use in reactions where oxidation is desired such as $[\text{Co}(\text{dpen})_3]^{3+}$ synthesis.



1. Make sure that all parts (flask, stirrer, and lid) are labeled with the same letter. This signifies that they all belong together.
2. Once contents are in flask (may be easier to “pre-dissolve” to prevent contents from being buried beneath stirrer), make sure solvent level is above T-shape, begin stirring slowly, and ramp up to at least 500 rpm to induce bubbling of solvent in the flask.
3. If apparatus is not stirring smoothly, one can wedge a Kimwipe or Teflon ring in between the lid and flask to relieve pressure between the lid and stirrer.

How Not to Break: Do not drop apparatus, do not put lid on tightly, in a manner that it cannot be removed easily. If new apparatus is needed, bring attached drawing to Bill Merka/glassblower.

From SI of Ghosh, S. K.; Lewis, K. G.; Kumar, A.; Gladysz, J. A. *Inorg. Chem.* **2017**, *56*, 2304-2320.

Gas circulating flask. A three neck flask and hollow glass stopper is configured to hold a horizontal hollow glass rod that reaches from the bottom of the flask to the apex of the stopper (Figure s2). The hollow rod has a hole at the top and a short perpendicular T-segment with two holes at the bottom. The T-segment must be beneath the surface of solvent. A magnetic stir bar is embedded in the rod immediately below the T-segment. When rotation is driven by a magnetic stirrer, an aspirator effect draws the ambient atmosphere down the rod and into the solution, producing a vigorous stream of bubbles out of the T-segment.

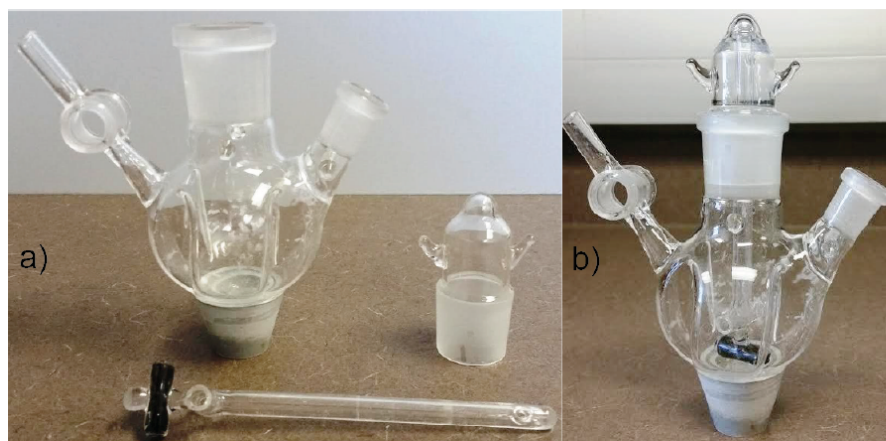


Figure s2. (a) Components of the gas circulating flask; (b) The assembled flask.

Alternative: Charge contents in a Schlenk flask and pull slight vacuum with water. Make sure flask is stoppered with a septum and pierce with long needle so that the tip is below solvent surface (see below).

