Overall setup description

A compact capillary based reaction cell (originally designed by Peter J. Chups from Argonne National Laboratory) is used for in-situ flow/heated diffraction measurement at the HXMA beamline. The details of this design (Figure 1) can be found in the paper J.Appl.Cryst. (2008). **41**, 822-824.

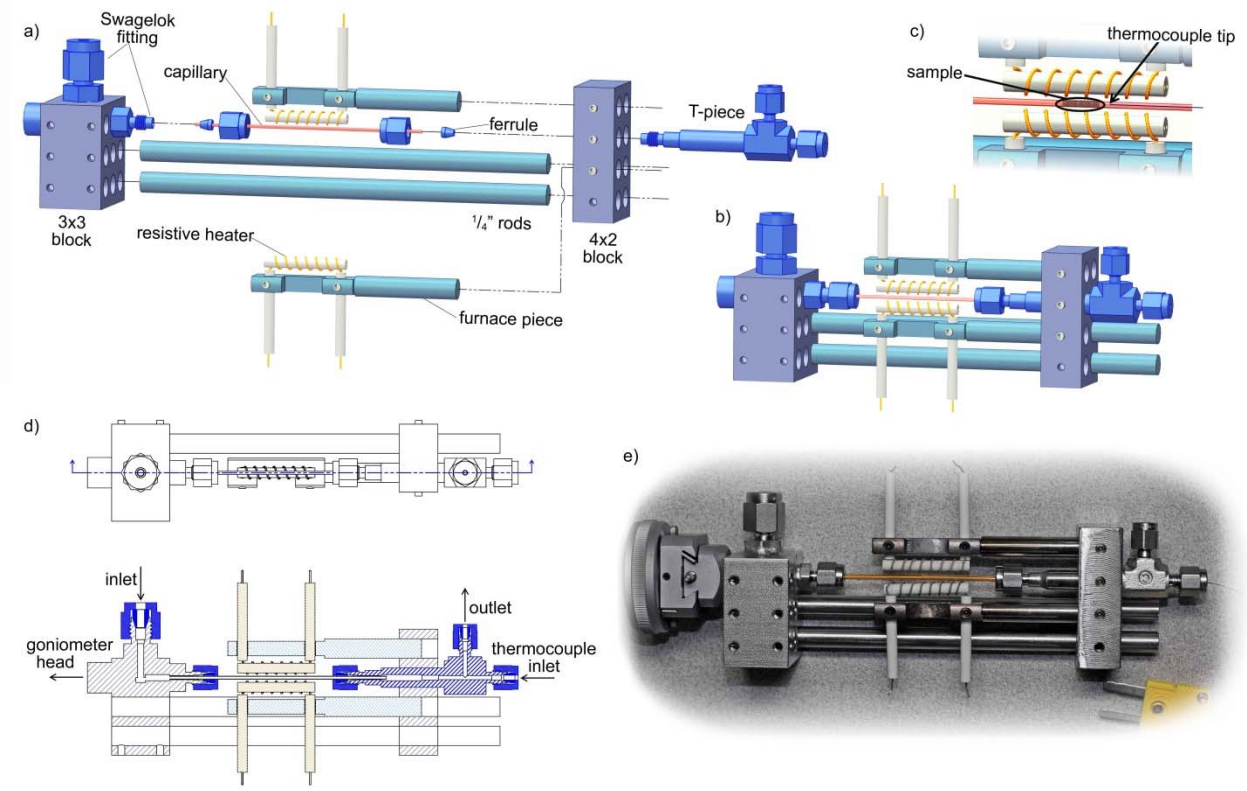


Figure 1 (Courtesy from Peter J. Chups)

(a) An 'exploded' representation of the flow-cell/furnace components, indicating how they fit together; (b) the fully assembled flow-cell/furnace; (c) an expanded view of the sample region, indicating the relative position of the sample and thermocouple tip within the furnace hot zone; (d) a top view of the flow-cell/furnace, with a corresponding cross section through the sample plane showing the gas/fluid path; (e) a photograph of the flow-cell/furnace mounted in a goniometer head.

For mounting, two 'mounting' blocks, containing an array (3×3 and 4×2) of holes, are connected by two rods that can be secured by set screws. The 3×3

block, which mounts on the goniometer head, contains welded Swagelok fittings: a gas/fluid inlet ($\frac{1}{8}$" Swagelok) and a $\frac{1}{16}$" Swagelok fitting that holds one end of the capillary. The 4×2 block holds a T-shaped component which holds the other end of the capillary, allowing a thermocouple to be placed inside the capillary and the gas/liquid outlet.

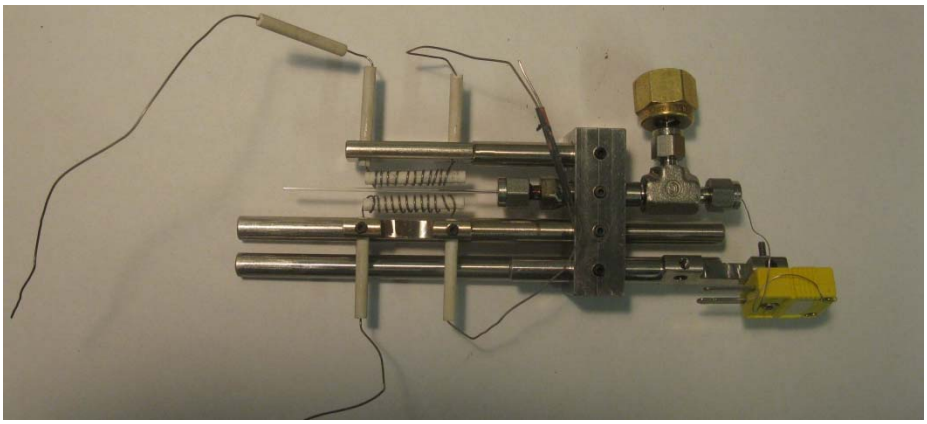
For heating, two furnace pieces, which hold resistive heaters, are placed immediately above and below the sample capillary and T-piece in the 4×2 block. The heating elements are sheathed in ceramic tubes for electrical insulation from the stainless steel furnace pieces. Power is provided to the resistive heaters by a Sorenson DC power supply, regulated by a Eurotherm temperature controller based on the sample temperature as measured by a thermocouple within the capillary in close proximity to the sample. Heat shields, which minimize air currents around the sample and heating elements, can be attached using the tapped set-screw holes to improve further temperature stability.

System assembly

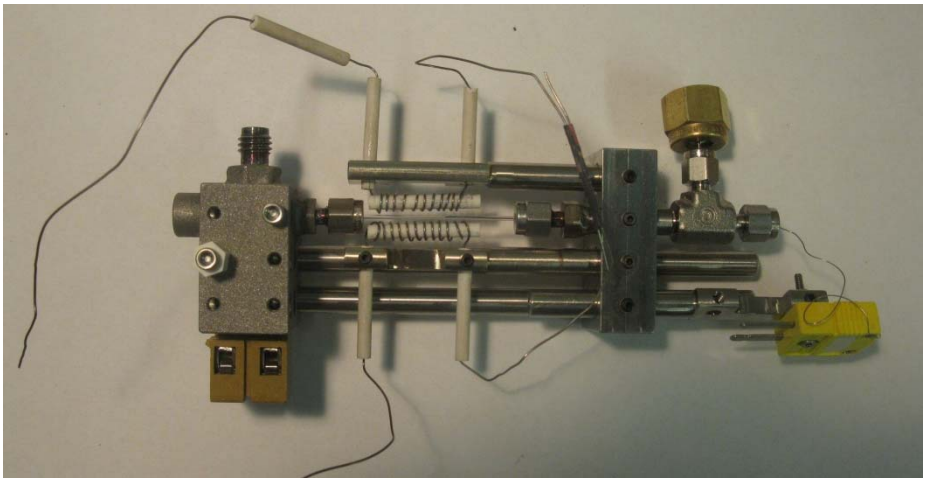
Please refer to the Figure 1 for assembly details.

Step 1, fill the capillary with sample and put the capillary through the ferrule.

Step 2, slide the thermocouple wire through the capillary and then mount the capillary to one end of T-piece in the 4×2 block.



Step 3, slide the 3×3 block to the other ends of mounting rods, align the other end of capillary to the Swagelok fitting cap and make sure the capillary through the ferrule.



Step 4, adjust the thermocouple wire position in the capillary, tight the Swagelok caps and the set screws.

Step 5, mount the whole cell piece to the goniometer head. Connect the resistive heating wires and power supply wires to the terminal block attached to the 3×3 block. Connect the thermocouple plugins. Further step is to connect the Swagelok inlet gas fitting (at the 3×3 block) to the gas supply pipe end and gas pumping pipe to the Swagelok outlet fitting (at the 4×2 block). If no gas flow is required, the gas inlet fitting can be capped and the gas outlet side can be pumped to make a rough vacuum environment for the sample.