Supplementary Material (ESI) for Green Chemistry

"Supercriticality"; A Dramatic but Safe Demonstration of The Critical Point – Supplementary Information.

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Abstract

The image of a fluid passing up through its critical point and back down again is one of the most beautiful and fascinating physical transformations that can be demonstrated to an audience. Unfortunately this image is not often seen; safety issues often restrict educators from carrying out such demonstrations in the public arena. In this article we describe a self contained demonstration apparatus that may be used to illustrate this transition in a very safe and controlled way. The demonstration apparatus may be used as a hands-on teaching instrument in the classroom, or alternatively with the aid of an LCD projector and screen, be used as an interactive demonstration during larger lectures or exhibitions.

Safety Note

Each piece of high-pressure equipment is unique and must be subjected to its own individual safety assessment. Neither the Authors nor The University of Nottingham accepts liability for the safety of any equipment or apparatus built upon the basis of this article.

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Optical Cell

The optical cell used in our demonstration was donated to our laboratory by François Cansell of L' Institut de Chemie de la Matière Condensée de Bordeaux. To the best of our knowledge, the cell is constructed from two opposing steel plates that each house a bonded sapphire window (Ø = 12 mm, d = 5 mm). The plates are secured by 4 cap-head bolts (M4, 12mm depth). A pressure seal is made between the two plates by way of a elastomeric ring. The optical cell is permanently mounted in a heat sink block, fabricated from oxygen free copper.

Heat Exchanger

The heat exchanger unit employed in our demonstration is comprised of three main components; a pair of Peltier heat pumps (23 mm × 23 mm, 18.7 W, from UK company RS, part number 189-1627) and associated electronics (directional 7 volt DC supply); A pair of channeled oxygen free copper heat transfer blocks; and a standard thermostatic re-circulation bath (Grant Ltd Model 6G) using water as the circulation fluid.

The temperature of the re-circulation bath is set at 313 K, additional heating up to the critical point (318 K) is provided by the Peltier heat pumps in "heat" mode. Similarly, cooling of the cell is carried out by the Peltiers running in the "cool" mode.

Operation

Connect the recirculation bath to the copper heat exchangers using a suitable length of tubing and make sure coolant level is sufficient to run the system. Turn on the bath and adjust the fluid temperature to 313 K. The system temperature should be allowed to stabilize before continuing.

Turn on the power to the Peltier heat pump, with the directional current switch in the "cool" position. Take note of the optical cell, two phases (a liquid and a gas) should be seen in cell.

To elevate the cell temperature, making the contents supercritical, turn the directional switch to the "heat" position, the demonstration should now cycle until a single phase is observed inside the optical cell. Return the directional switch to the "cool" position to re-establish the original two-phase system.

Troubleshooting Guide

If cell does not cycle, please check that:

1 Power supply is on and fuses have not blown

2 All electronic connections are made

3 Recirculation bath is connected to heat exchangers

4 Fluid level in recirculation bath is sufficient.

Further troubleshooting will be apparatus dependant.