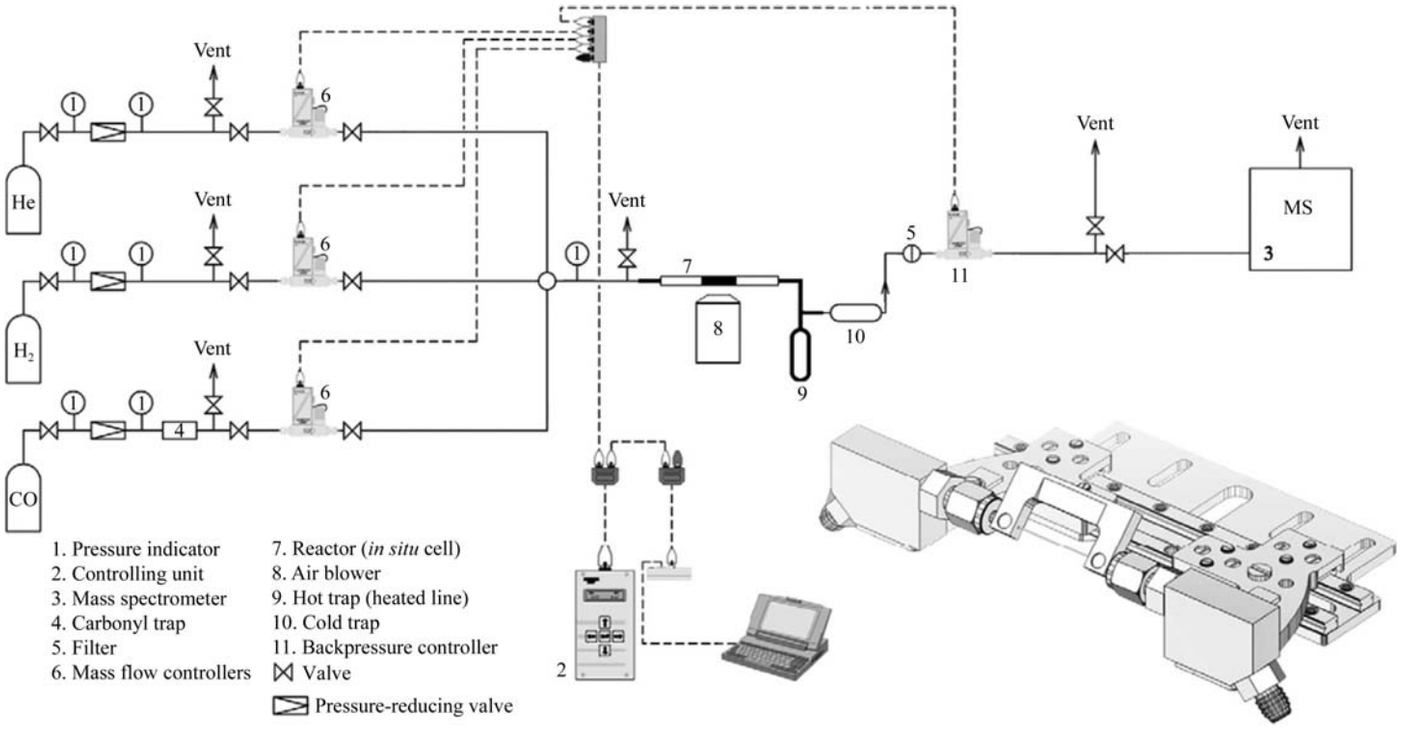

Figure 2.9.3
Sketch of a typical experimental setup and a three-dimensional drawing of the *in situ* flow cell. Note the strain-relief bracket over the capillary. Adapted from Tsakoumis *et al.* (2012), with permission from Elsevier.

Above 800 K, open capillary systems have severe heat loss and the use of insulation or reflectors around the sample or mirror furnaces is more appropriate. See Lorenz *et al.* (1993), Margulies *et al.* (1999), Proffen *et al.* (1995), Riello *et al.* (2013) and Yashima & Tanaka (2004) for special designs to minimize heat loss. Finally, it is worth mentioning the recent work of Figueroa *et al.* (2013), which combines the strong points of the various capillary designs, and also work by Johnsen & Norby (2013), who managed to create and study a working battery in a capillary.

In any powder-diffraction experiment (Warren, 1990), but particularly when using capillary cells, the experimentalist needs to take special care in order to obtain sufficient averaging in terms of grain statistics and to avoid preferred orientation. Typically *ex situ* capillaries are spun, but when gas lines are attached to the sample, spinning is not possible and only rocking or stationary geometry can be used. In addition, a fine (ground) polycrystalline powder giving perfect homogenous Debye–Scherrer rings, even without spinning, often results in an excessive pressure drop due to its high density and packing. In such cases, the sample may need to be pressed into a pellet and then crushed again to obtain larger agglomerates that allow sufficient gas flow through the sample (Jacques *et al.*, 2009). However, relatively large agglomerates, while reducing the packing density, might still give nonhomogeneous powder rings, which affects the intensities, especially on one-dimensional detector systems (strip detectors or crystal-analyser high-resolution systems). For all of these reasons it is not always straightforward to acquire reliable intensities under *in situ* conditions. If, however, proper care is taken, then precise structural parameters can indeed be refined from *in situ* data. For example, Milanesio *et al.* (2003) obtained a detailed view of the structural rearrangements induced by the template-burning process from 350 to 1000 K on a zeolitic MFI framework. Oxygen flowed through a rocking sample and diffraction data were collected on a translating two-dimensional image plate capable of verifying the reliability of the measured

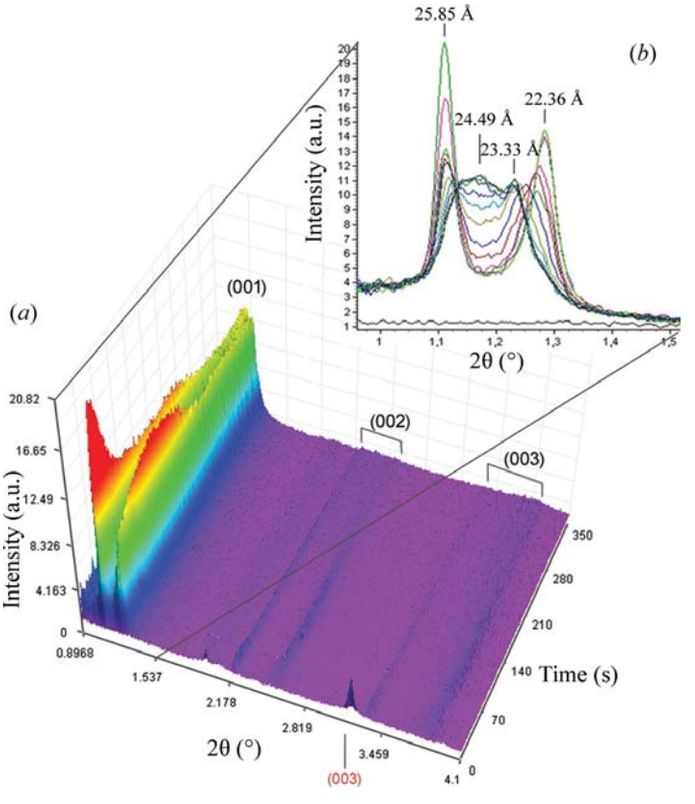

Figure 2.9.4
(*a*) *In situ* three-dimensional stacked plot of the intercalation of ibuprofen into an LDH. Miller indices are shown in black for the intercalation product peaks and in red for the LDH nitrate starting material peaks. (*b*) Two-dimensional patterns showing the low-angle peaks during the first instance of the reaction. Adapted with permission from Conterosito *et al.* (2013). Copyright (2013) American Chemical Society.