# Experimental Report



**Proposal:** **INTER-261** **Council:** 10/2012

**Title:** Internal time on IN10

**This proposal is a new proposal**

**Researh Area:**

**Main proposer:** **MHANNA Ramona**

**Experimental Team:** LE DAIN Guillaume
MORINEAU Denis
LEFORT Ronan
MHANNA Ramona

**Local Contact:** FRICK Bernhard
SEYDEL Tilo

**Samples:** tert-butanol (C4H10O)
hexane(C6H14)
porous silicate

| Instrument | Req. Days | All. Days | From | To |
|---|---|---|---|---|
| IN10 | 9 | 18 | 19/02/2013 | 28/02/2013 |
| | | | 31/03/2013 | 09/04/2013 |
| IN16 | 0 | 10 | 28/02/2013 | 07/03/2013 |
| | | | 29/03/2013 | 01/04/2013 |

**Abstract:**

# Experimental Report : INTER_261

Experimental team: Denis Morineau, Bernhard Frick, Ronan Lefort, Ramona Mhanna.
Local Contact: Bernhard Frick

**Internal time on IN10 (19-28 /2/2013)**

Systematic H/D contrast matching neutron diffraction studies performed on Tert-butanol (TBA)/Toluene (TOL) mixtures confined in the rigid and mono-disperse channels of MCM-41 mesoporous silicates (D=2.5nm) suggest the nano-segregation of the two components (core-shell structure) induced by the preferential interaction of TBA with the silica pore surface of MCM-41. (Fig.1).

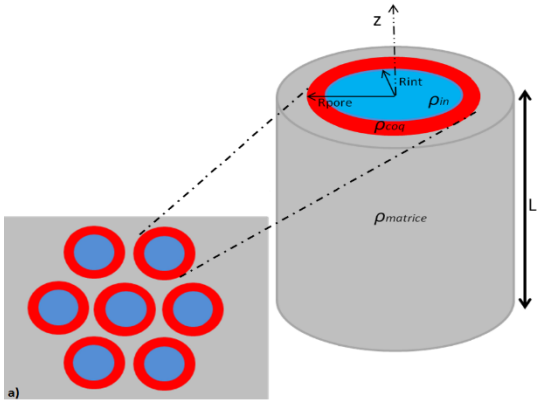


**Fig.1.**Core shell structure with MCM-41 matrix (in grey), TBA (in red) and TOL (in blue).

Fixed window scans were performed on TBA / TOL mixtures confined in MCM-41 in a trial to assess the molecular dynamics of these nano-segregated mixtures, aiming to disentangle the main structural relaxation of the different components.

Different mixtures were measured: TBAx TOL (1-x) with x=1, 0.7, 0.5, 0.3, 0. The motivation for measuring different samples was to study the effect of dilution on the dynamics of both constituents of the core-shell structure.

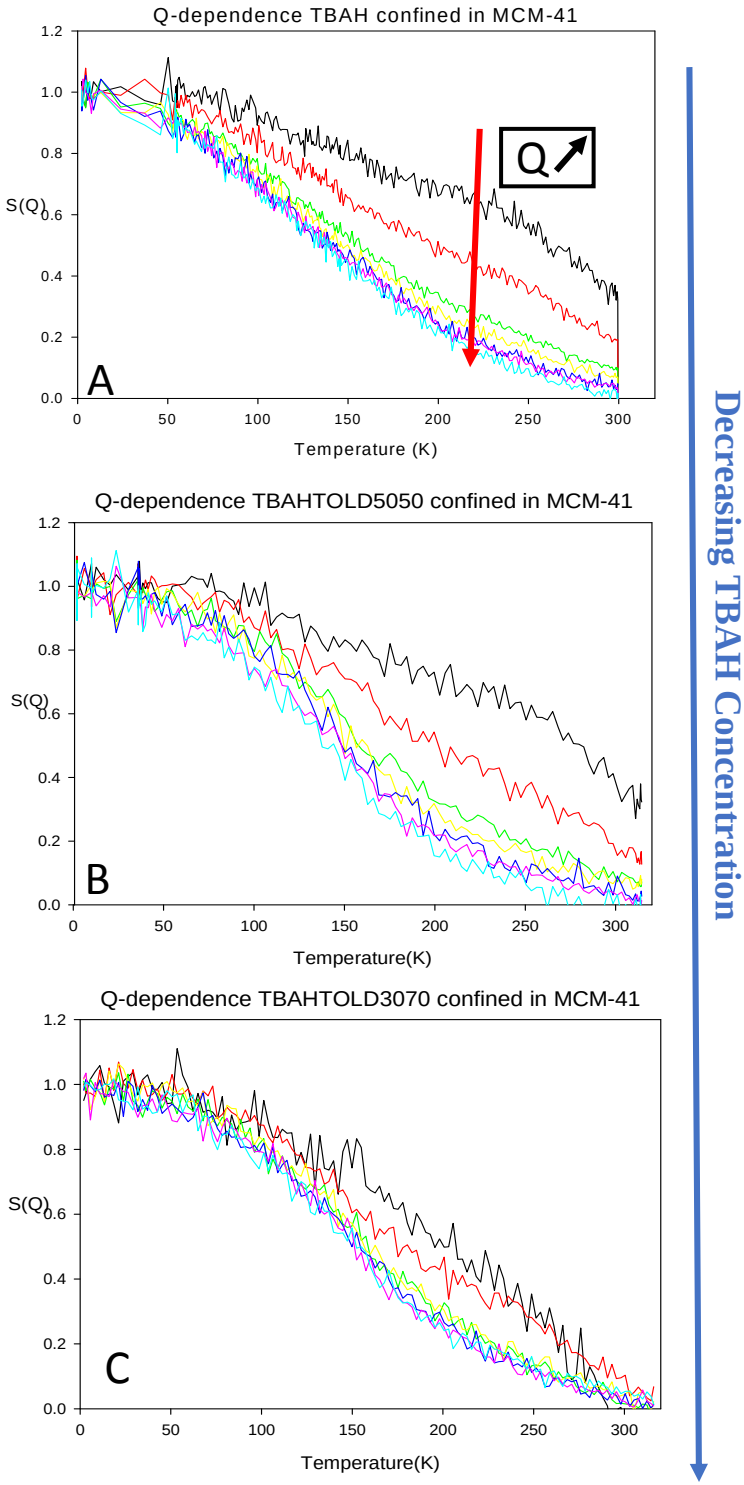


**Fig.2.** Q-dependence of Fixed Window Scans Performed on IN10 for TBAH (A), TBAH50TOLD50 (B) and TBAH30TOLD70 (C) confined in MCM-41.

The FWS were treated by SQWEL where the empty can was subtracted from the raw data.
Subtractions include a proper correction for attenuation factors, with a slab geometry (2theta=135°). The forward cell transmission is taken T=0.95, the attenuation and self- attenuation are calculated by SQWEL, with samples densities and scattering cross section as inputs. Then using an external program (SciLab) the samples were corrected from the empty MCM-41 silicate matrix (already corrected from empty can) and finally normalized to lowest temperature.

FWS performed on confined TBAHTOLD mixtures show that for high/ intermediate TBA concentrations the dynamics of TBA aren't highly affected by the addition of Toluene (**Fig.** 2A, 2B) and show an inflection point at low Q at high Temp which is an indication of probable local dynamics. As for the sample with high Toluene dilution i.e. low TBA concentrations (TBAH<30%) the interaction with Toluene makes TBA dynamics faster which is evident by a fast drop in elastic intensity (**Fig.** 2C).

The same set of experiments were performed on TOLHTBAD systems to highlight the TOL dynamics which don't seem to be highly Q or concentration dependent**.** However, it is evident from the graphs that the larger the TBA concentration the slower the dynamics of TOL probably due the interaction with TBA, seen as a small wing in the elastic intensity at high temperatures in both TBAD50TOLH50 and TBAD70TOLH30 (**Fig.** 3B, C).

It could be noted; however, that the stretching of the decay of the FWS over a wide temperature range makes the dynamical contributions from the different constituents somehow ambiguous and could indicate that the distinction between surface and core is ill-defined as pore size approaches the molecular size.

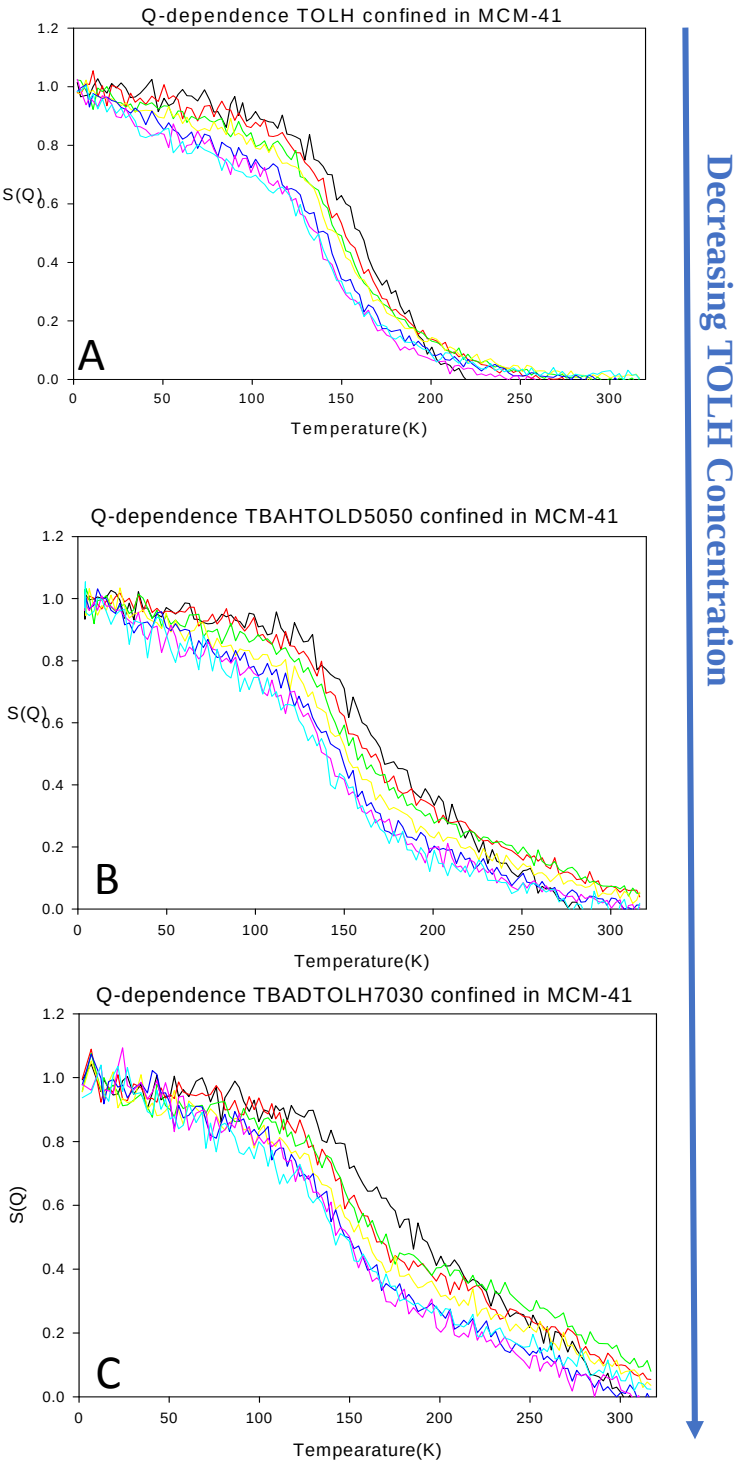


**Fig.3.** Q-dependence of Fixed Window Scans Performed on IN10 for TOLH (A), TBAD50TOLH50 (B) and TBAD70TOLH30 (C) confined in MCM-41.

**Internal time on IN16 (28 /2 to /3/2013) and (30/3 to 1/4/2013)**

Quasielastic measurements were performed on IN16 in the official beam time number 6-02-497 and the internal beam times mentioned in the report.

Different samples with different concentrations and isotopic compositions were measured to understand the effect of composition and to disentangle the different contributions.

Similar to the treatment of IN10, the raw data was treated with SQW, dedicated to quasielastic spectra, which does the same can subtraction taking into account all the attenuation factors. Then using SciLab the samples were corrected from MCM-41(with the correct ratio of masses) and normalized to the 2K measurement.

In the internal beam time, we measured 70/30 HD, DH and HH compositions as well 5050DH and HH compositions to complete the data set measured in the official IN16 beam time.

Different Temperatures were tried based on previous FWS: 320, 220, 170, 140 (and 2K to measure the elastic intensity) in an approach to have the quasielastic contribution within the BS window.

The measurements reveal, at high Q, that for all compositions and all temperatures the dynamics are too fast that the signal runs out of the window of BS into the TOF range, at low Q (**Fig.**4. A, B, C); however, we can still see some slow dynamics with a small quasielastic contribution in the BS window (compare with resolution **Fig.**4. C).

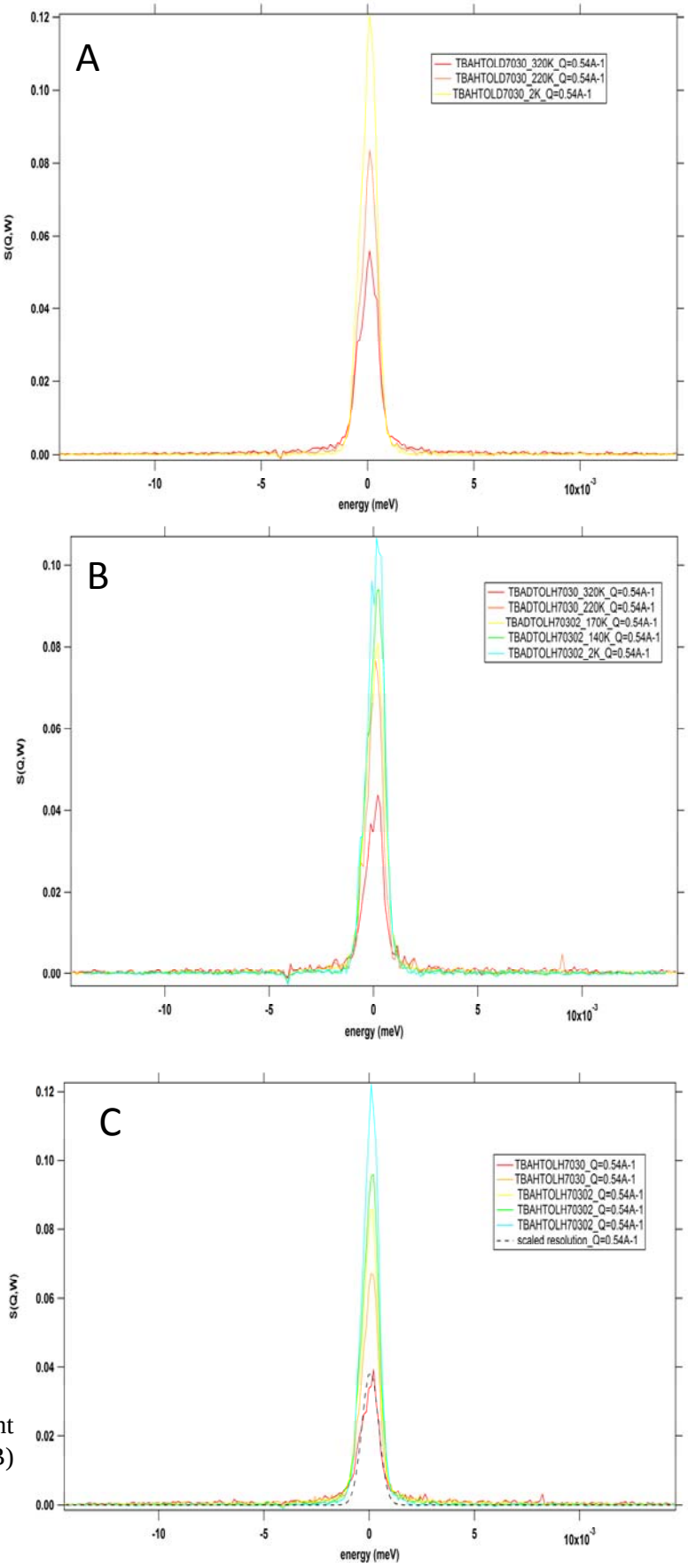


**Fig.4.** Quasielastic measurements performed on IN16 at different temperatures for TBAH70TOLD30 (A), TBAD70TOLH30 (B) and TBAH70TOLH30 (C) samples confined in MCM-41.