Mössbauer spectroscopy and nuclear inelastic scattering studies on polynuclear oxo-bridged iron catalyst—first results

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Published online: 20 February 2009
© Springer Science + Business Media B.V. 2009

Erratum to: Hyperfine Interact (2008) 187:35–41
DOI 10.1007/s10751-008-9869-4

In the Experiment section, the sentence, ‘The isomer shifts were calibrated with respect to α -Fe foil at room temperature (RT).’, should be read as ‘The isomer shifts were calibrated with respect to $^{57}\text{Co/Rh}$ source.’ Therefore, the isomer shifts in Table 1 are also with respect to $^{57}\text{Co/Rh}$ source. The below text and table should be regarded by the reader as the final version.

3 Experiment

The ^{57}Fe Mössbauer spectra were measured at liquid helium temperature (4.2 K; both source and sample in liquid helium) in a bath cryostat, using a gas proportional

The online version of the original article can be found at
<http://dx.doi.org/10.1007/s10751-008-9869-4>.

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Table 1 Mössbauer parameters of all samples

Compound	IS ^a [mm s ⁻¹]	QS [mm s ⁻¹]
Tetranuclear complex (Fig. 3a)	0.308 ± 0.002 ^b 0.290 ± 0.001	0.574 ± 0.003 ^b 0.652 ± 0.001
Active catalyst under N ₂ (Fig. 3b)	0.949 ± 0.002	3.392 ± 0.003
Active catalyst + cyclohexene under N ₂	0.950 ± 0.003	3.372 ± 0.007
Active catalyst under O ₂ (Fig. 3c)	0.947 ± 0.007 0.305 ± 0.005 ^c	3.287 ± 0.013 1.040 ± 0.009 ^c
Reaction after 1 min (Fig. 3d)	0.973 ± 0.002	3.217 ± 0.005
After 5 min (Fig. 3e)	0.952 ± 0.003 0.289 ± 0.005 ^c	3.091 ± 0.006 1.008 ± 0.009 ^c
After 30 min (Fig. 3f)	0.962 ± 0.005 0.304 ± 0.002 ^c	3.203 ± 0.011 1.052 ± 0.004 ^c

Numbers following the symbol ± is the fit error (standard deviation)

^aIS are given with respect to ⁵⁷Co/Rh source

^bValues obtained at RT

^cParameters of the inner doublet 2—Fe(III) species

detector in transmission geometry and a ⁵⁷Co source (Rh-matrix). The isomer shifts were calibrated with respect to ⁵⁷Co/Rh source. In addition, a RT measurement was also performed for the trinuclear complex. In order to study the reaction kinetics, measurements were also done at different times after starting the reaction. The samples were freeze-quenched [6] by taking approximately 0.1–0.2 ml of the oxidation reaction mixture in a syringe, then immediately injecting it into the sample holder and immersing the sample holder in liquid nitrogen. In order to study the influence of different reactants in detail, measurements were also done on different combinations (with or without substrate in inert gas and in O₂). The NIS experiments were performed at ~70 K at the beamline ID 18 of the European Synchrotron Radiation Facility (ESRF) in Grenoble (France), with an energy bandwidth (FWHM) of 1.2 meV. The collection times were ~10 h per spectrum. The probability of recoilless absorption (Lamb–Mössbauer factor, *f*) can be obtained from the inelastic spectra. Details of NIS theory, experimental techniques and data evaluation procedure are described elsewhere [7–9].