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XAS In Situ Cell for Measurements of Mn Promoted Sulfated Zirconia Catalysts

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Promotion of sulfated zirconia (SZ) with Mn (2 wt%) results in a catalyst that can isomerize *n*-butane at a rate two orders of magnitude greater than SZ can. One explanation for this promotion effect, consistent with rapid deactivation observed for these catalysts, suggests that the Mn participates in non-catalytic redox reactions. In order to test this hypothesis, it is necessary to measure the Mn valance in a true in-situ experiment. However, it was found that the catalyst has a reduced activity when pressed into pellets for measurement. Therefore, we designed an in situ cell that could be used to measure spectra of a powdered catalyst sample. The cell consists of an internal powder bed reactor (heatable to 773 K), which is enclosed in a He filled, temperature controlled outer cell to ensure that the window to the inner cell, and the catalyst it contacts, are at a well-defined temperature. Spectra are measured in fluorescence mode with simultaneous measurement of a reference foil in transmission using a section of the beam that bypasses the catalyst bed. Contrary to previous reports, data obtained in situ, show that the Mn valence remains constant during reaction with *n*-butane after the catalyst has been activated in He. However, when activated in 50% O₂ the average Mn valence decreases by about 0.05 during the reaction. These results suggest that the promotion effect of Mn is either catalytic, or is indirect, perhaps through modification of the zirconia bulk.