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**Improved Experimental Setup for
in situ DR UV/Vis/near-IR Spectroscopy of
n-Alkane Isomerization on Sulfated Zirconia***



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The low signal level and thermal radiation are the main problems facing the reflectance spectroscopic investigation of catalyst powders under reaction conditions. In a first setup the hot reactor cell was positioned at a distance of 12 mm from the integrating sphere to avoid its heating and the distance was bridged with highly reflecting ceramics to increase the part of light reflected into the sphere.

The requirements, large distance between integrating sphere and reactor cell, higher signal level, smaller sampling area, to decrease thermal radiation, and a better arrangement of reactor cell and oven in a vertical position to accommodate powder -could be fulfilled by application of a specially formed light conductor made of quartz (2nd setup).

This *in situ* technique was applied to the *n*-butane (5 vol.-% in He) and *n*-pentane (0.13 vol.-% in He) isomerization on sulfated zirconia (c. 1.2 g and 0.34 g, respectively) in the temperature range of 298-523 K. After an induction period one observes strong asymmetrically difference spectra during the *n*-butane isomerization centered at 300 nm at 523 K and the *n*-pentane isomerization centered at 315 nm at room temperature. Using spectral analysis these spectra can be fitted well by two partial spectra. After longer times on stream further absorption bands were observed at 364 nm, 424 nm (*n*-butane) and 378 nm (*n*-pentane). Most absorption bands identified can be assigned to alkenylic (linear and/or cyclo) species and are responsible for the deactivation and/or poisoning of the catalyst. For the *n*-butane isomerization at temperatures ≤ 373 K and for the *n*-pentane isomerization during the induction period at room temperature the difference absorptions are so small that a reasonable identification is possible only by applying the 2nd setup because the signal-to-noise ratio was improved with this setup by the factor 4-5.

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