Surface Science
CATLAB PCS Tech Info Sheet 160

CATLAB PCS

Pulse Chemisorption With the Hiden CATLAB

Pulse Chemisorption Overview

The technique of pulse chemisorption involves passing known quantities of gas over an activated catalyst and quantifying the amount of uptake during each pulse. To ensure accurate results an electrically actuated valve with sample loop of known volume is used (typically 100 µl, volumes from 15 µl to 10 ml are available). The pulse is injected into an inert carrier gas before passing over the catalyst.

The technique generally uses several pulses, the first few pulses either completely consumed by the catalyst or partially consumed. Gas that does not react with the sample is detected by the mass spectrometer. Eventually the detected peaks will reach a constant level (integrated area). These saturation pulses are used as calibration pulses to relate the mass spec response to a quantity of gas, *i.e.* the area under these peaks is proportional to the number of moles of gas in the sample loop. The uptake of the adsorbate can be calculated from the areas of peaks fully consumed plus the difference in the areas of the peaks of the partially consumed pulses compared with the calibration peaks.

Once the uptake has been determined for each pulse information such as adsorption isotherms, monolayer coverage and catalyst dispersion can be determined.

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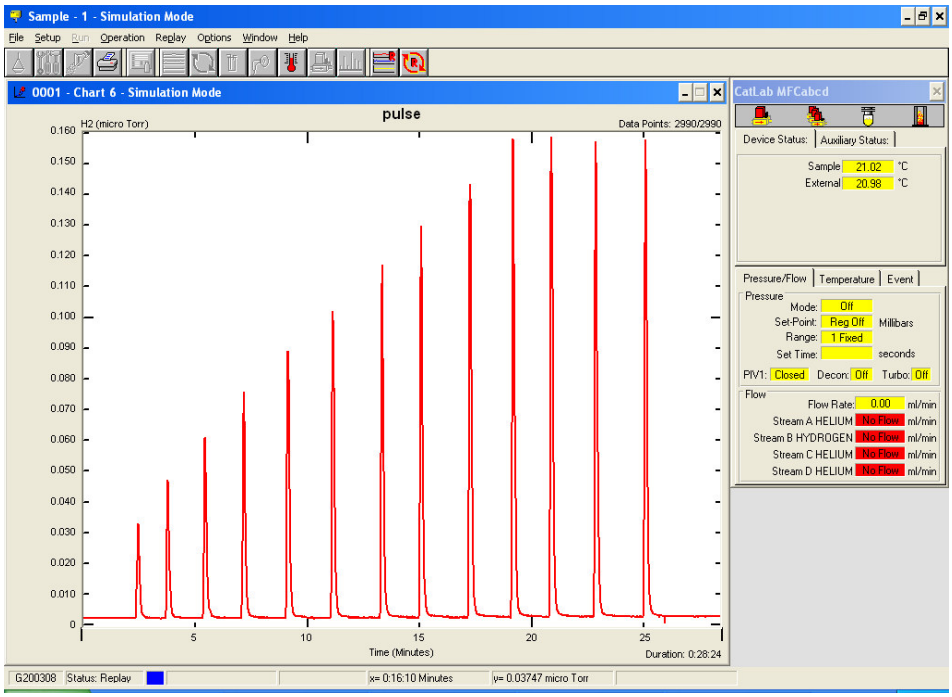

Figure 1 Typical Pulse Adsorption Profile

System Description

The Hiden CATLAB is available with an option to allow pulse chemisorption measurements (CATLAB-PCS). An integrated 2-position low dead volume valve with changeable sample loop volume is provided to allow known volumes of gas to be pulsed over the activated catalyst. Valve control is provided automatically via the Hiden CATLAB software.

Figure 1 above shows a pulse profile with a total of 14 pulses passed over the catalyst. Pulse 1 is completely adsorbed by the sample. Pulses 2 – 10 are partially adsorbed. Peaks 11 – 14 represent pulses passed over the catalyst after saturation has occurred. These are used as calibration pulses to relate the mass spec response to a quantity of gas, *i.e.* the area under these peaks is proportional to the number of moles of gas in the sample loop (100 µl volume). The uptake of the adsorbate is calculated from the areas of peaks fully consumed plus the difference in the areas of the peaks of the partially consumed pulses compared with the calibration pulses.

Once the amount of uptake has been determined for each pulse, information such as adsorption isotherms, monolayer coverage and dispersion can be determined. An example of a pulse adsorption isotherm is shown below. The adsorption isotherm relates the uptake to the exposure the catalysts has had of the adsorbate.

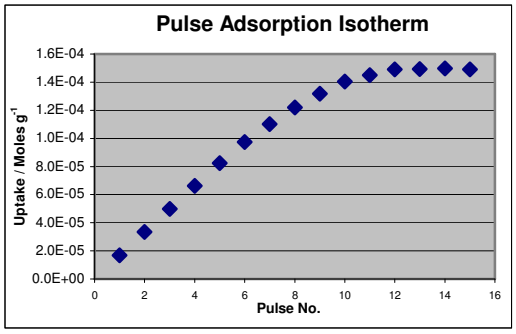

Figure 2 Pulse Adsorption Isotherm

The CATLAB also has the capability of sending pulses of reaction mixtures over the catalyst. The CATLAB is supplied with a mass spec as standard. This means that several species can be followed at a time. Disadvantages of systems with a TCD (thermal conductivity detector) mean they can only follow a change in gas flow

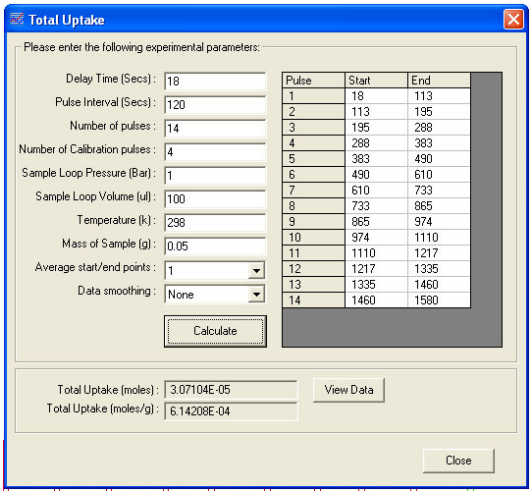

Figure 3 TPD Analysis Software Screen

composition and water must be removed before reaching the TCD and so are only useful for single component adsorption experiments. Careful choice of adsorbate and carrier gas for use with TCDs is also important, as they must have significant differences in their thermal conductivity. This is not a problem for mass specs, although spectral overlaps should be accounted for.

By passing pulses of mixtures over the catalyst, reactions can be followed providing information such as catalyst retention of reactant, product distribution and mass balance information. This can be especially useful for following the early stages of a reaction where the active sites on the catalyst are created by the reactants (e.g. through carbon laydown). This type of information is lost in continuous flow measurements. It is also possible to observe how components of mixtures may adsorb preferentially in pulse experiments. Again this is useful for understanding the surface chemistry on the catalyst.