

HETEROGENEOUS CATALYSIS INVESTIGATION BY MEANS OF SETARAM DSC 111 LINKED TO GAS CHROMATOGRAPH

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Hydrogen adsorption on a Pt/SiO₂ catalyst during an isothermal process and then its desorption by a temperature treatment can be achieved successfully by means of a SETARAM DSC 111 linked to a gas chromatograph. The latter monitors the amount of Hydrogen in the carrier gas after passing on the catalyst.

The design of the SETARAM DSC 111 allows a flow of gas in the sensitive zone. The sensitivity is not modified by the flow rate and the nature of gas, owing to the surrounding structure of the fluxmetric detector.

Two quartz reactors pass through the measuring and reference tubes of the calorimeter (Fig. 1). The catalyst sample is placed on a piece of sintered glass in the middle of the reactor on which gas can sweep. The outlet of the DSC 111 is connected to a gas chromatograph with catharometric detection.

ISOTHERMAL ADSORPTION (20°C)

Injections of 0,5 ml of Hydrogen diluted at 5,4 % in argon are made every 8 minutes. The exothermic peaks of adsorption decrease as the active sites of the catalyst are occupied (Fig. 2). From the 10th injection endothermic effects of desorption are noticed on the calorimeter signal. It is corroborated by the chromatograph which detects the hydrogen coming from the sample : desorbed or non adsorbed.

The total amount of adsorbed hydrogen is $12,3 \cdot 10^{-5}$ mole per g. of catalyst. The adsorption heat of hydrogen on platinum varies from 42 to 9 k cal .mole⁻¹ of H₂ (Fig. 3).

The mean adsorption heat is $27,7 \pm 2$ k.cal mole⁻¹ H₂.

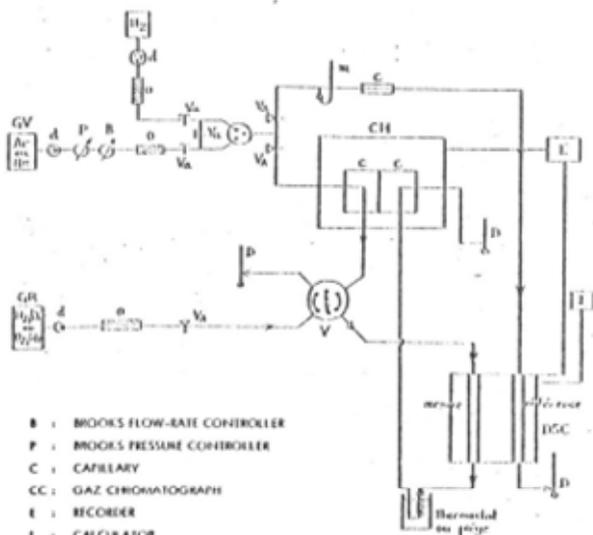
DESORPTION

It is made by scanning the temperature at 10°C.mn⁻¹ between 20°C and 430°C. The recorded thermogram has a maximum at 117°C and comes back to base line at 267°C.

The volume of desorbed hydrogen is $12,05 \cdot 10^{-6}$ mole.

The mean desorption heat is 28 ± 2 k cal.mole⁻¹.

A good agreement between the adsorption and desorption heats and between the adsorbed and desorbed amounts of hydrogen can be noticed.



- B : BROOKS FLOW-RATE CONTROLLER
- P : BROOKS PRESSURE CONTROLLER
- C : CAPILLARY
- CC : GAS CHROMATOGRAPH
- E : RECORDER
- I : CALCULATOR
- # : DOUBLE EXPANSION MANOMETER
- DSC : DIFFERENTIAL SCANNING CALORIMETER
- GR : REACTIVE GAS
- GV : CARRIER GAS
- m : MANOMETER
- O : OXYGEN TRAP
- V : 6 WAYS GAP
- D : SOAP BUBBLE FLOWMETER

APPARATUS SCHEME

FIGURE 1

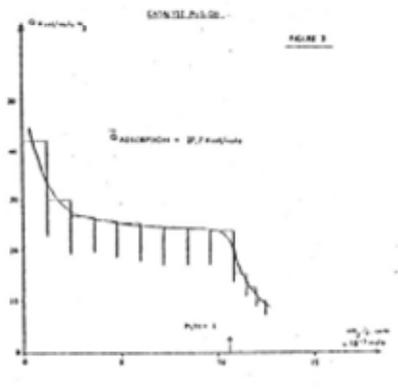
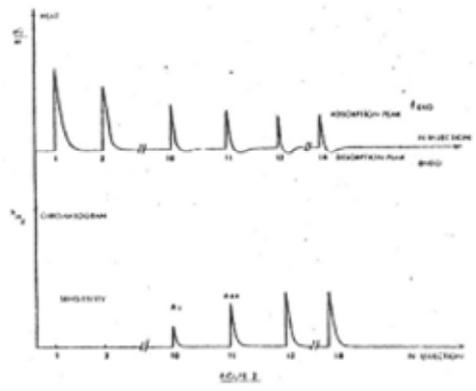


FIGURE 3