Comparison of Catalyst Activity

The problem:
To compare the activity of highly active catalysts for reactions which are so strongly exothermic or endothermic that normal steady-state reactor techniques are impractical.

The solution:
Allow the temperature of the catalyst bed initially at 77°K to increase slowly; a marked deviation in the smooth warming curve denotes the temperature at which detectable reaction occurs. The lower the temperature at which reaction commences, the more active the catalyst.

How it's done:
The apparatus designed for the rising temperature technique is indicated in the diagram. The catalyst sample (usually about 1 ml in volume) is loaded into the apparatus over the coarse glass sintered disc and in contact with the central tube which encloses three thermocouples. Two of the thermocouples are adjusted so that one is above the top of the catalyst bed and the other is 1–2 mm below the top of the catalyst bed; these thermocouples are connected to a differential thermocouple recorder. The other thermocouple is adjusted so that its end is also 1–2 mm below the top of the catalyst bed; it is used to measure the temperature of the catalyst.

When used to compare the activities of platinum catalysts, a standard pretreatment of 10 minutes at 400°C in a 1:1 hydrogen–helium gas mixture is used to dry and reduce the catalyst. Then, the reactor is cooled in a liquid nitrogen bath while the hydrogen–helium gas mixture is passed through the catalyst bed. When the temperature is at 77°K, a reactant stream consisting of 3% hydrogen, 1% oxygen, and 96% helium (by volume) is passed through the catalyst bed at a rate of 400 ml per minute. Exactly one minute later, the liquid nitrogen bath is quickly replaced by a precooled (77°K) insulated air-bath to allow the reactor temperature to rise slowly while the temperature in the central thermowell is recorded as a function of time. The differential thermocouple recorder will indicate when reaction commences.
Notes:
1. Gases are purified by passing them successively through molecular sieves (drying), 0.5% palladium on alumina at 100°C (to oxidize carbon monoxide), and through a carbon dioxide absorbent.
2. No additional documentation is available. Specific questions, however, may be directed to:
   Technology Utilization Officer
   Ames Research Center
   Moffett Field, California 94035
   Reference: B72-10201

Patent status:
No patent action is contemplated by NASA.
Source: Thomas J. Jennings and Hervey H. Voge of Shell Development Company under contract to Ames Research Center (ARC-10493)