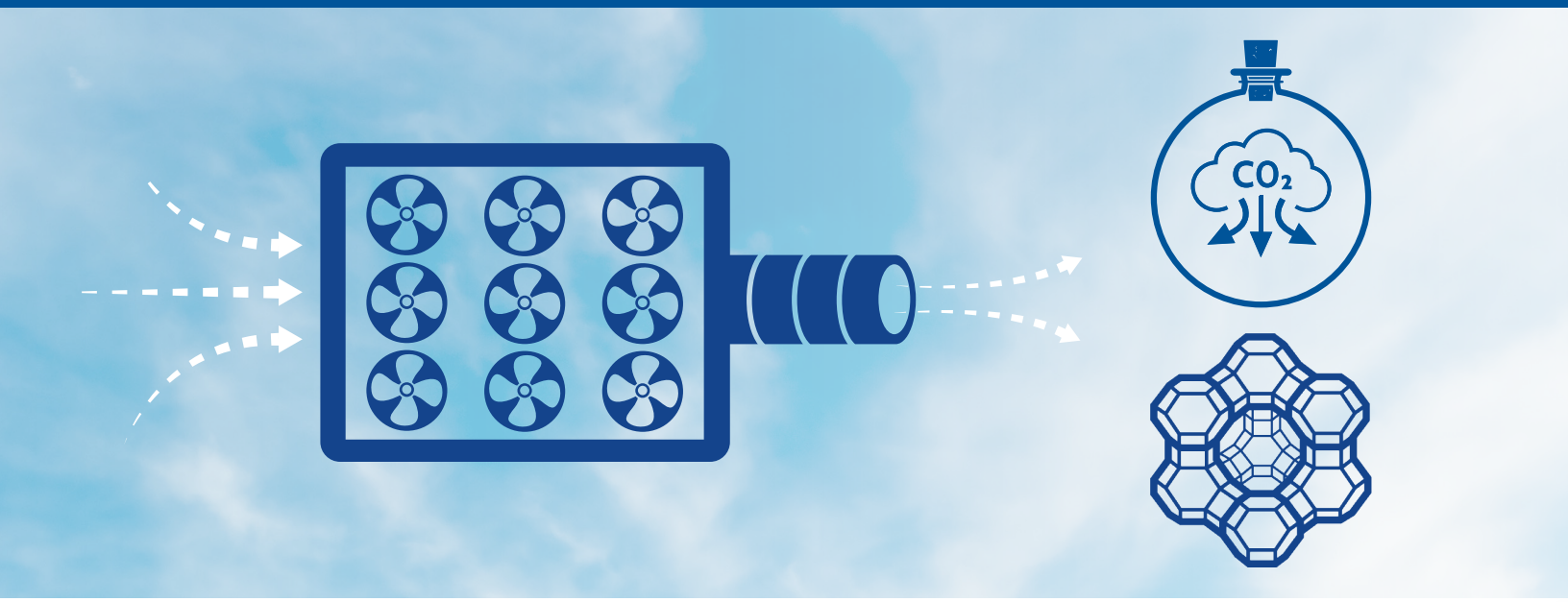
micromeritics®

BREAKTHROUGH ANALYSIS ON THE AUTOCHEM



INTRODUCTION

The AutoChem is a flow-through system that allows for the characterization of catalysts for surface activity. The AutoChem can also function as a basic single component breakthrough unit allowing for carrier, preparation, and analysis gas flows. This document will describe how to perform a breakthrough analysis using the AutoChem alongside a mass spectrometer.

EXPERIMENTAL

Zeolite 13X was used as a standard reference material for carbon dioxide breakthrough analysis. **Figure 1** below shows the instrument schematic that was used for the breakthrough analysis. Prior to analysis, the sample was heated to 200 °C at 10 °C/min under nitrogen gas flow at a rate of 20 sccm overnight. The sample temperature was then cooled to the analysis temperature of 30 °C. Next the mass spectrometer was turned on while continuing to flow nitrogen gas through the carrier line.

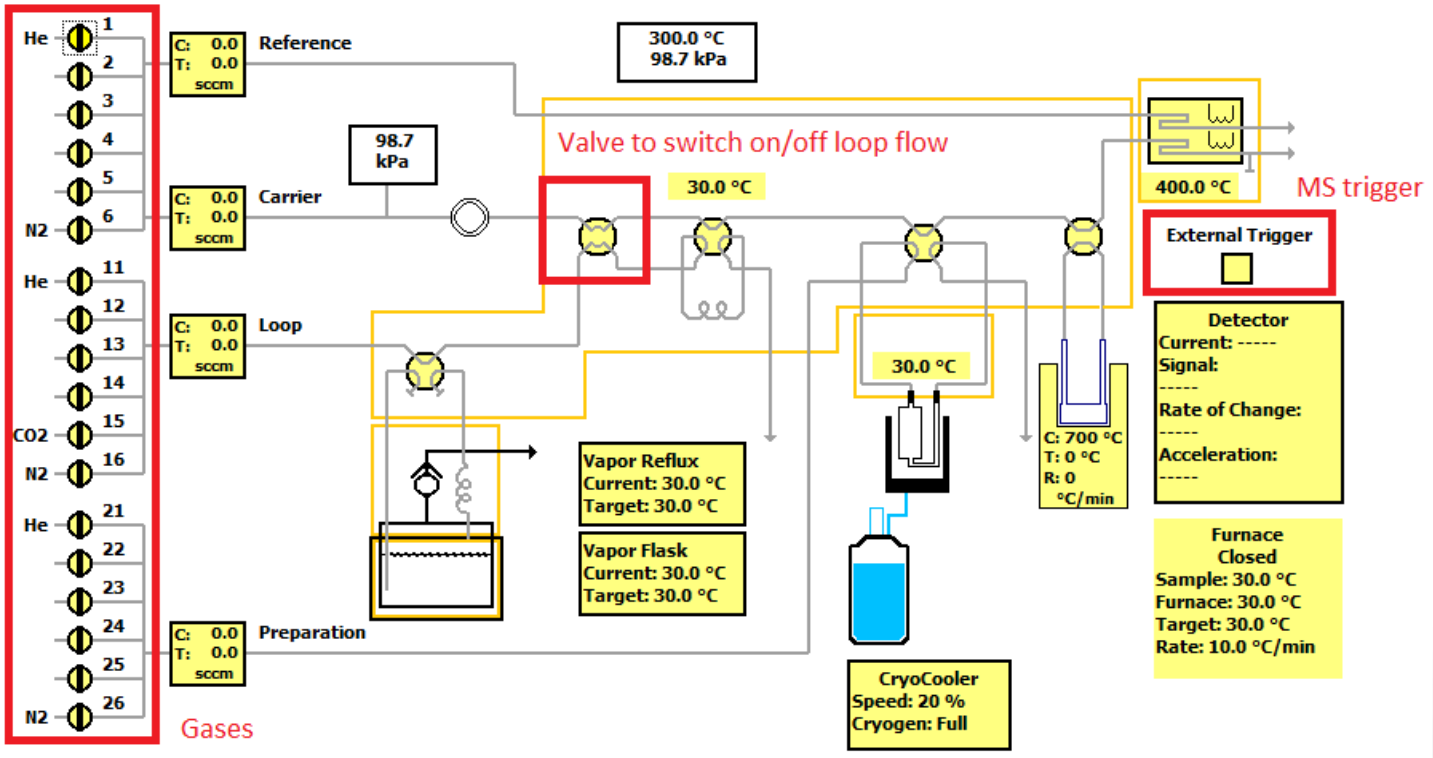


Figure 1. AutoChem CO_2 breakthrough analysis.

Before breakthrough measurements, the deadtime of the system was calculated. The deadtime is the time that it takes for the gas to flow through the system and reach the mass spectrometer. The deadtime was determined by flowing nitrogen through the carrier gas line (20 sccm) and helium through the loop gas line (20 sccm). The flows were alternated and the time was recorded between when the valve was turned on and a difference in mass signal was detected by the mass spectrometer. After several measurements, the deadtime was determined to be 14.5 seconds for a flowrate of 20 sccm.

Following the determination of the deadtime, the flow of gas through the loop was switched from helium to CO_2 (20 sccm). After establishing the flow for several minutes, breakthrough began by switching on the flow of CO_2 from the loop. Following breakthrough measurements, the sample furnace was heated to 200 °C for several hours to ensure the complete desorption of carbon dioxide from zeolite 13X.

RESULTS

The results of the breakthrough experiment are shown below. Zeolite 13X showed strong adsorption for CO_2 at ambient conditions reaching a capacity of 4.53 mmol/g. Additionally, the breakthrough curve is steep signifying that there are little to no mass transfer limitations in the system.

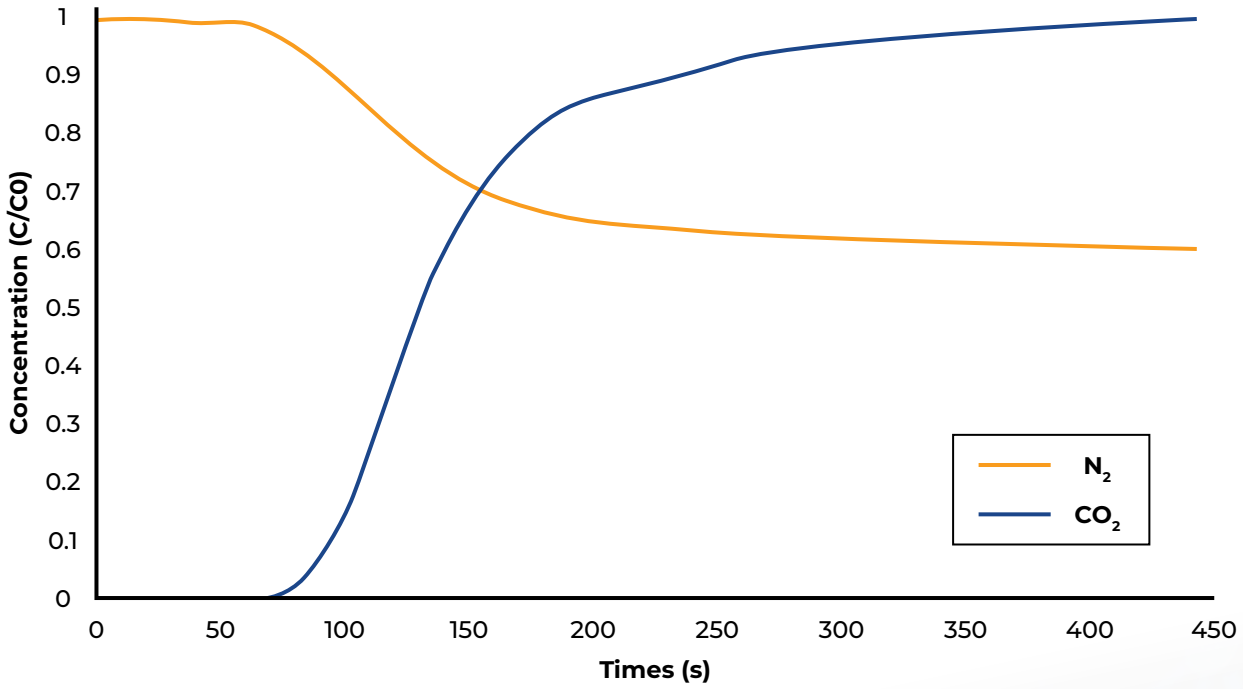


Figure 2. CO_2 breakthrough adsorption in zeolite 13X.

CONCLUSIONS

The AutoChem is a capable unit for performing simple single component breakthrough analysis. This was confirmed via analysis of zeolite 13X, achieving a CO_2 adsorption capacity of 4.53 mmol/g at 30 °C and a flowrate of 20 sccm.

Micromeritics Instrument Corp.
4356 Communications Drive
Norcross, GA 30093

Tel: +1 770 662-3636
info@micromeritics.com
micromeritics.com