Until recently, it has been believed that during adsorption and desorption cycles of molecular sieve pellets, the pellets have been passive participants in the process. It has lately been shown by others using X-ray diffraction technique that the molecular sieve structure undergoes expansion and contraction as a result of moisture content changes.

A new thermomechanical analyzer (TMA) technique has been developed that allows the dimensional change to be measured continuously as a function of time and, concurrently, moisture content, thus creating a unique method for measuring adsorption rates.

The instrument modifications (DuPont 990 and 943), collection and evaluation of data, and the sensitivity of the adsorption rate due to various parameters are discussed in the paper.

A useful technique has been developed to measure the adsorption rate of water by molecular sieve pellets. This technique involves establishing a relationship between dimensional change and molecular sieve adsorption rates. It has been shown [1–5] that molecular sieves undergo a structural dimension change during adsorption. The change has been observed on a unit-cell scale by X-ray lattice parameter shifts, with corresponding macro-scale detection by dilatometry [3]. This change manifests itself as a volumetric change in a molecular sieve pellet, but it can be estimated by a measurement in one dimension with a commercial dilatometer.

After a molecular sieve pellet has been deactivated, it will expand upon contact with water vapor. This expansion is measured by continuous recording of the dilatometer probe displacement. The expansion continues until the sample has reached an equilibrium state with the moisture available. The curve, plotted by the continuous displacement of the probe, can then be analyzed to give comparative rates.

Experimental

The equipment used in this technique consists of a DuPont 943 Thermomechanical Analyzer (TMA) module, in conjunction with a DuPont 990 Recorder-Controller. The TMA module has a moveable core linear variable differential transformer (LVDT) attached to a probe, which is operated under negligible load. The
movement of the LVDT core through the primary coil generates an electrical signal which is proportional to the distance the core has moved. The signal is amplified and recorded on the calibrated Y-axis of the recorder chart as the absolute displacement of the probe in mils. The X-axis records either temperature or time, or both, if both pens are used.

![Temperature scale calibration for TMA cell.](image)

The manufacturer claims [6] that the linearity of the LVDT should be within 0.5% of the selected scale. This was confirmed by comparing the manual displacement of the probe (by a micrometer graduated in mils), with the recorded displacement. The accuracy of sample temperature measurement in this system was also tested. This was done by scanning a sample of α-quartz and recording the α-β transition, which was observed at 570°, in good agreement with the accepted value of 572°. These data are shown in Figure 1.

A special modification of the DuPont 943 module was necessary to meet experimental requirements for this technique. To insure efficient and reproducible sample-atmosphere contact, the purge system had to be changed. A modified sample holder tube was fabricated, consisting of a coiled Inconel-600 tube (0.056" O.D. - 0.046" I.D.) spiralling downward along the inside wall of the sample holder tube and terminating in the sample area. This system allows the adsorbate-laden gas stream to be thermally equilibrated before coming in contact with the sample, and also insures good sample-atmosphere contact, essential for successful operation.

The samples used in data collecting were a series of laboratory and commercial bonded molecular sieve pellets. All samples were equilibrated at 50% relative hu-

*J. Thermal Anal.* 17, 1979