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CONTROLLED ATMOSPHERE-CONTROLLED PRESSURE THERMOGRAVIMETRY

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Reversible dehydrations and decompositions are seldom studied under equilibrium or near equilibrium conditions because of the need to provide the special atmospheres. Self-generated atmospheres provide only a limited approach to a controlled atmosphere at a controlled pressure; reveersibility must be inferred from the variation of decomposition temperature with pressure. Monotropic processes cannot be identified and close-lying processes evolving different gases cannot ordinarily be resolved. Controlled atmospheres in the study of processes near the boiling point of the adduct (water or a complexing agent, for example) are particularly troublesome.

These authors have constructed a thermobalance based upon a Cahn 1000 automatic balance especially for studies of hydrates and of coordination compounds whose ligands are liquid at room temperature. The glass dome was replaced with a properly secured steel dome fitted with heaters to prevent condensation. Twin furnaces were constructed using stainless steel tubes each fitted with flanges and a pair of (insulated) hemicylindrical heating elements. The atmosphere is generated by vaporization from the surface of the liquid adduct admitted to the furnaces from a reservoir; this reservoir is kept at the chosen pressure so that the vapor inside the furnace is that same pressure, in the manner previously reported for DTA.

Equilibrium can be achieved over a range of pressure/temperature combinations. Not only decomposition/dehydration processes but also the reverse processes can be studied because the water or ethylene diamine or other solvate vapor is present. Resolvations generally start rapidly but become very slow because of the need for entering vapor to diffuse through the rehydrated material. The slowing effect is less for layered minerals.

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