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# APPLICATION OF PULSE THERMAL ANALYIS FOR INVESTIGATING GAS-SOLID REACTIONS

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Pulse thermal analysis (PulseTA®) is based on the injection of a specific amount of gaseous or liquid reactant into a carrier gas stream while monitoring changes in mass, enthalpy and gas composition resulting from.the incremental reaction extent [1,2]. In contrast to conventional thermal analysis (TA) and all its modifications, the course of the reaction is controlled not only by temperature but also by transient change in the composition of the reactive atmosphere.

PulseTA® offers three principle opportunities of thermoanalytical studies, depending on the kind of injected gas: (i) injection of an inert gas, facilitating quantitative calibration of the mass spectrometric signals, allows to increase the sensitivity of TA measurements to such an extent that species in amounts lower than 0.01 wt% can be detected; (ii) injection of a gas which reacts with the solid sample provides the opportunity of investigating all types of gas-solid reactions; and, (iii) injection of a gas which adsorbs onto the sample surface facilitates the study of adsorption phenomena under atmospheric pressure and at a required temperature.

The most distinct feature of PulseTA® is the fact that changes of the gaseous atmosphere during experiments occur in a limited, short period of time after each injected pulse of the gas. PulseTA® allows monitoring gas-solid processes corresponding to a specific extent of reaction with desired temperature ramp (iso- or non-isothermally). The reaction can be stopped at any point between pulses, enabling elucidation of the relationship between the composition of the solid and the reaction progress. The potential of simultaneous monitoring changes in mass, thermal effects, composition and amount of gaseous reactants and products under pulse conditions enables to gain simultaneously information concerning both gas and solid phases.

The conventional TA methods generally do not allow investigating processes in differential steps of reaction extent. The transient character of the pulse technique offers interesting opportunities which are important when studying in-situ gas-solid reactions and catalytic processes in transient mode.

The application of PulseTA® will be illustrated by results obtained by means of combined TA-MS and TA-FTIR systems. A variety of different gas-solid interactions will be addressed such as decomposition of simple and complex inorganic salts, oxidation and reduction (by hydrogen, methane, carbon monoxide) of solids, adsorption and desorption phenomena, and catalytic heterogeneous reactions. The studies clearly demonstrate that the pulse technique offers new interesting opportunities for investigating mechanism of gas-solid processes.

**References**

1. M. Maciejewski, C.A. Müller, R. Tschan, W.-D. Emmerich and A. Baiker, Thermochim. Acta, 295 (1997) 167.
2. M. Maciejewski, W.D. Emmerich and A. Baiker, Proc. 25-th North American Thermal Society Conference, McLean, Virginia, USA, 1997, Ed. R.J. Morgan, p. 508.