Simultaneous TG/DSC measurements performed on mixtures 2Li$_2$CO$_3$-5TiO$_2$ (anatase) subjected to high-energy milling showed that both the temperature and the enthalpy of Li$_2$CO$_3$ decomposition are much lower than in the case of TG/DSC runs performed on a sample of a physical mixture. On the basis of the thermoanalytical evidence a solid-state synthesis of the spinel compound Li$_4$Ti$_5$O$_{12}$ has been proposed that combines mechanical (by high-energy milling) and thermal activation (8 h annealing at 973 K): the obtained compound shows a lattice constant in very good agreement with that expected for the pure phase Li$_{1+x}$Ti$_{2-x}$O$_4$ ($x = 0.333$). The molar heat capacity of Li$_4$Ti$_5$O$_{12}$ has been determined in the temperature range 323–633 K by quasi-isothermal Modulated Differential Scanning Calorimetry (MDSC). The specific surface area of Li$_4$Ti$_5$O$_{12}$ has been determined by gas adsorption.

Key words: Li$_4$Ti$_5$O$_{12}$, Solid-state Synthesis, High-energy Milling, Molar Heat Capacity