

Combined Experiments to Measure Low Sublimation Pressures and Diffusion Coefficients of Organometallic Compounds

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Vapor pressures and sublimation pressures of organometallic compounds are needed in several processes like chemical vapor deposition (CVD). Thermobalances at ambient pressures are often used to study the evaporation of such compounds. At least three strategies, viz. Knudsen effusion method, transpiration method and thermal gravimetric analysis (TGA), are found in the literature to evaluate the results using different theoretical approaches. In some of the frequently used approaches the diffusion out of a crucible is neglected. We present a simple theoretical approach which describes the interrelation between the observed mass transfer rate and the physical variables of typical TGA set-ups. It turns out that the mass transfer rate at a given total pressure and temperature is mainly a function of the diffusion coefficient and the vapor pressure of the sublimating substance. The vapor pressures at various temperatures may be determined from an independent measurement using the Knudsen cell and combined with the TGA to obtain the diffusion coefficients. Experiments have been performed with two well studied substances naphthalene and phenanthrene to check the present strategy. From a knowledge of the vapor pressure (e.g. via Knudsen effusion method) and the mass loss in TG experiments the binary diffusion coefficients needed for the application of Sherwood number correlations have been evaluated and compared with literature values. Further measurements have been performed for the CVD relevant compounds: ferrocene and 2,2,6,6-tetramethyl-3,5-heptanedionato cobalt complex [Co(tmhd)₃].