Synthesis and solubility measurement in supercritical carbon dioxide of two solid derivatives of 2-methylnaphthalene-1,4-dione (menadione) : 2-(Benzylamino)-3-methylnaphthalene-1,4-dione and 3-(phenethylamino)-2-methylnaphthalene-1,4-dione

Zacconi, F. C., Nuñez, O. N., Cabrera, A. L., Valenzuela, L. M., del Valle, J. M., & Juan, C. (2016). Synthesis and solubility measurement in supercritical carbon dioxide of two solid derivatives of 2-methylnaphthalene-1, 4-dione (menadione): 2-(Benzylamino)-3-methylnaphthalene-1, 4-dione and 3-(phenethylamino)-2-methylnaphthalene-1, 4-dione. The Journal of Chemical Thermodynamics, 103, 325-332. <10.1016/j.jct.2016.08.016> Accessed 08 Jan 2021.

Abstract

Synthesis of two solid derivatives of vitamin K3 (2-methylnaphthalene-1,4-dione or menadione), 2-(benzylamino)-3-methylnaphthalene-1,4-dione and 3-(phenethylamino)-2-methylnaphthalene-1,4-dione was completed using a 1,4 Michael addition reaction at 323 K in an inert atmosphere, with reaction yields of 62% mol·mol–1 and 71% mol·mol–1, respectively, and a purity grade of 98% mol·mol–1 for each component. Isothermal solubility (mole fraction) of each solid derivative in supercritical carbon dioxide was performed using an analytic-recirculation methodology, with direct determination of the molar composition of the carbon dioxide-rich phase by using high performance liquid chromatography, at temperatures of (313, 323 and 333) K and pressures from (8–28) MPa. Results indicated that the range of measured solubilities were from (59 × 10–6 to 368 × 10–6) mol·mol–1 for solid 2-(benzylamino)-3-methylnaphthalene-1,4-dione and from (40 × 10–6 to 205 × 10–6) mol·mol–1 for solid 3-(phenethylamino)-2-methylnaphthalene-1,4-dione. The experimental solubility was validated using three approaches, estimating the combined expanded uncertainty of measurement for each solubility data point, evaluating the thermodynamic consistency of the data utilizing a test based on the Gibbs–Duhem equation, and verifying the self-consistency by correlating the experimental solubility values with a semi-empirical model as a function of temperature, pressure and pure carbon dioxide density..

Keywords

Solubility, Menadione, Derivatives, Supercritical carbon dioxide, Modelling.