

Department of Chemistry
Departmental Seminar:
Physical, Computational & Materials Month

You are cordially invited to a virtual lecture presented by



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Date: Friday, 22 October 2021
Time: 10:30
Venue: [Google Meet](#)
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Characterizing the thermal phase behaviour of fipronil

Fipronil is a broad spectrum, low application rate insecticide that belongs to a class of chemicals known as phenylpyrazoles. It is mostly used for controlling pest organisms of numerous animals and crops such as fleas, weevils, ants, ticks, ants, rootworms, mosquitoes, and termites. When preparing and formulating a large-scale market compound such as fipronil, it is of utmost importance to know if alternate crystalline modifications (polymorphs or solvate pseudo-polymorphs) of the compound exist, how they can be obtained, and what their characteristic properties are. Polymorphs of one compound may have contrasting properties, for instance melting point, colour, degradation stability, crystal size, mechanical and optical properties, and solubility.

The present study was undertaken to identify crystalline polymorphs, solvate pseudomorphs and amorphous forms of fipronil that arise as a result of recrystallization of fipronil.

Analysis by DSC, TGA, single-crystal XRD and powder XRD demonstrated that recrystallisation of fipronil from organic solvents such as acetonitrile, acetone, ethyl acetate yielded fipronil hemi-solvates. TGA analysis shows the solvents are liberated well before

150 °C. DSC analysis shows two sharp melting endotherms located at onset temperatures of ca. 194 °C and 204 °C. The relative proportions of the enthalpies associated with the two melting events vary depending on the nature of the starting material (e.g. solvent used for recrystallization) and its previous thermal history. However, the combined enthalpy measured for the two melting events remains approximately constant suggesting that these events may be associated with the presence of separate crystalline phases. Single-crystal XRD and powder XRD analysis of the samples studied support the thermal observations.