Journal of Inclusion Phenomena and Macrocyclic Chemistry **37:** 131–136, 2000.

Mechanochemical Synthesis of Urea Adducts with Long Chain Alkyl Derivatives

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(Received: 12 February 1999; in final form: 26 August 1999)

Abstract. The adducts of urea and solid alkyl derivatives can be obtained mechanochemically in good yields. The products synthesized by grinding the solid reagents have identical IR spectra and XRD powder patterns to those obtained by crystallization from ethanolic solutions.

Key words: urea adducts; alkyl derivatives; mechanochemical synthesis

1. Introduction

When solutions of urea and straight-chain alkyl derivatives are mixed, colorless crystals of urea adducts are obtained [1]. These adducts correspond to hexagonal urea as a host for the alkyl chains which occupy parallel channels in the matrix [2]. The structure has been confirmed by XRD and neutron diffraction [2–5] and by NMR spectroscopy [6–7]. Infrared [8–10] and Raman [11–13] spectra are useful in detecting complex formation due to changes in the position and intensity of the urea bands.

The most general method of synthesis of urea clathrates is the use of a common solvent for the urea and alkyl derivative [1, 14]. A practical method is the addition of the alkyl derivative to a slurry of urea in a small amount of methanol, followed by stirring of the mixture [15]. Hollingsworth has reported that liquid suberonitrile can be clathrated by strong stirring with urea [16]. This method of synthesis can be expedited by the use of "activated" or "expanded" urea obtained by previous treatment with acetone [17–20].

In the pharmaceutical industry, certain alkyl derivatives, which are difficult to handle, are transformed into a suitable dry powder by grinding and tableting with urea [21]. One can infer that, here, a complex with urea has been formed mechanochemically. It is the purpose of this paper to present evidence, based on IR and

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