**Assessing the Detection Limit of a Minority Solid-State Form of a Pharmaceutical by 1H Double-Quantum MAS NMR Spectroscopy**

Keisuke Maruyoshi,1,2 Dinu Iuga,1 Abigail E. Watts,3 Colan E. Hughes,3 Kenneth D. M. Harris,3 and Steven P. Brown\*1

*1Department of Physics and †Department of Chemistry, University of Warwick, Coventry CV4 7AL, U.K.*

*2Analytical & Quality Evaluation Research Laboratories, Daiichi Sankyo Co. Ltd., 1-12-1, Shinomiya, Hiratsuka-shi, Kanagawa, 254-0014, Japan*

*3School of Chemistry, Cardiff University, Park Place, Cardiff, Wales, CF10 3AT, U.K.*

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**Figure 1** 1H (850 MHz) DQ MAS (30 kHz) spectra of (a) the anhydrous HCl salt of cimetidine, (b) polymorph A of cimetidine, and (c) a 10% w/w physical mixture of polymorph A with the anhydrous HCl salt

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| samples | NMR data (850 MHz) |
| (a) cimetidine HCl salt | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_HCl |
| (b) cimetidine | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_free |
| (c) 10% w/w physical mixture | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_10% |

**Figure 2** Slices corresponding to a 1H DQ frequency of 19.0 ppm, as extracted from two-dimensional 1H (850 MHz) DQ MAS (30 kHz) spectra of (a) the anhydrous HCl salt of cimetidine, (b) polymorph A of cimetidine, and (c-f) physical mixtures of polymorph A with the anhydrous HCl salt with (c) 10%, (d) 5%, (e) 1%, and (f) 0.5% w/w of polymorph A.

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| samples | NMR data (850 MHz) |
| (a) cimetidine HCl salt | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_HCl |
| (b) cimetidine | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_free |
| (c) 10% w/w physical mixture | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_10% |
| (d) 5% w/w physical mixture | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_5% |
| (e) 1% w/w physical mixture | Recorded 14/03/2012 Dataset: 1H\_120314\_cimetidine\_1% |
| (f) 0.5% w/w physical mixture | Recorded 04/06/2012 Dataset: 1H\_120604\_cimetidine\_0.5% |