

Supporting Materials

for

New Solid Forms of Nitrofurantoin and 4-aminopyridine Salt: Influence of Salt Hydration Level on Crystal Packing and Physicochemical Properties

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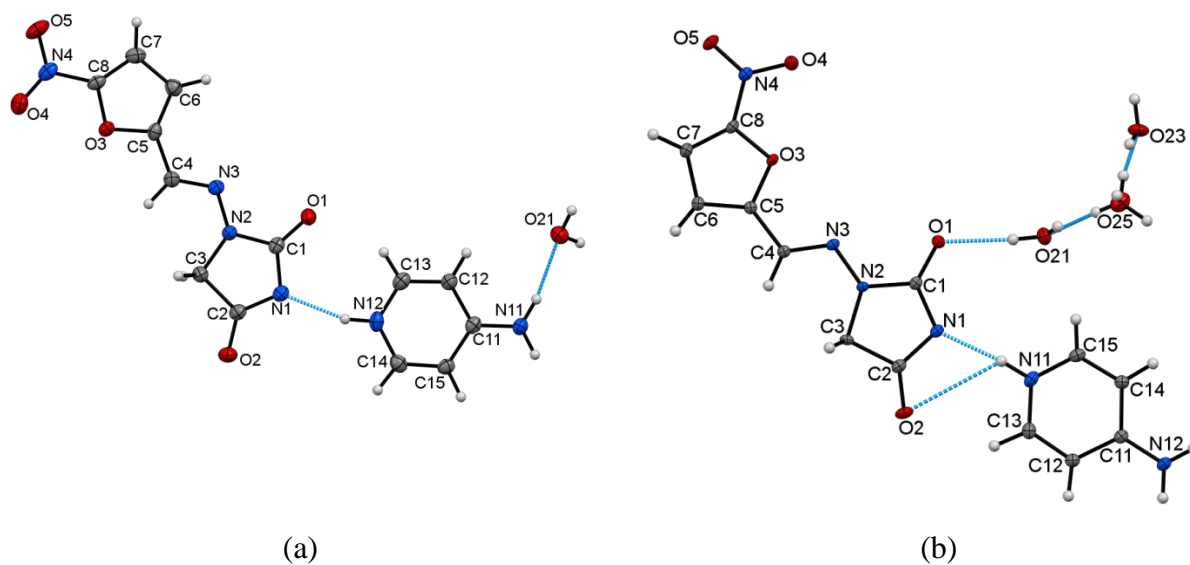


Figure S1. The ORTEP diagram of (a) [NFT+4AmPy+H₂O] salt (1:1:1) and (b) [NFT+4AmPy+H₂O] salt (1:1:4). Thermal ellipsoids are drawn at the 50% probability level. Intermolecular interactions are drawn as blue dashed lines.

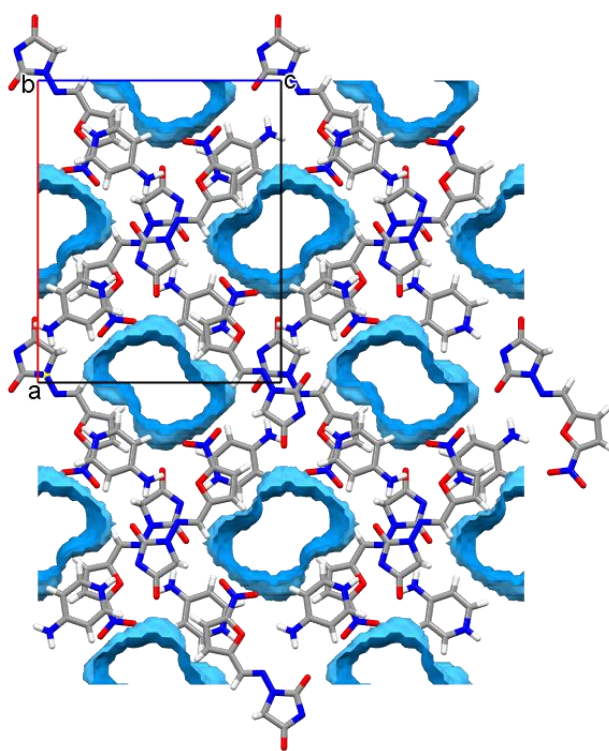


Figure S2. Water interaction map for the [NFT+4AmPy+H₂O] salt (1:1:4).

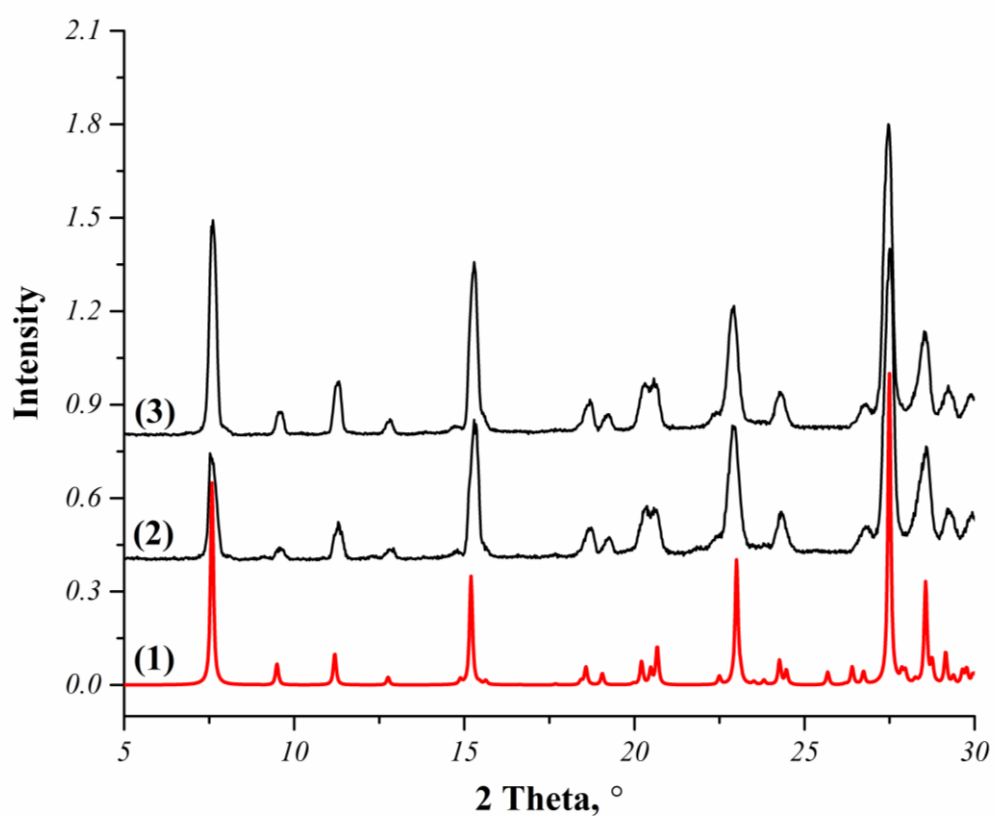


Figure S3. Comparison of the simulated PXRD calculated for the [NFT+4AmPy+H₂O] salt (1:1:4) (1) with the experimental PXRD patterns of the powder samples obtained by LAG of the [NFT+4AmPy] salt (1:1) (2) and the [NFT+4AmPy+H₂O] salt (1:1:1) (3) in the presence of water.

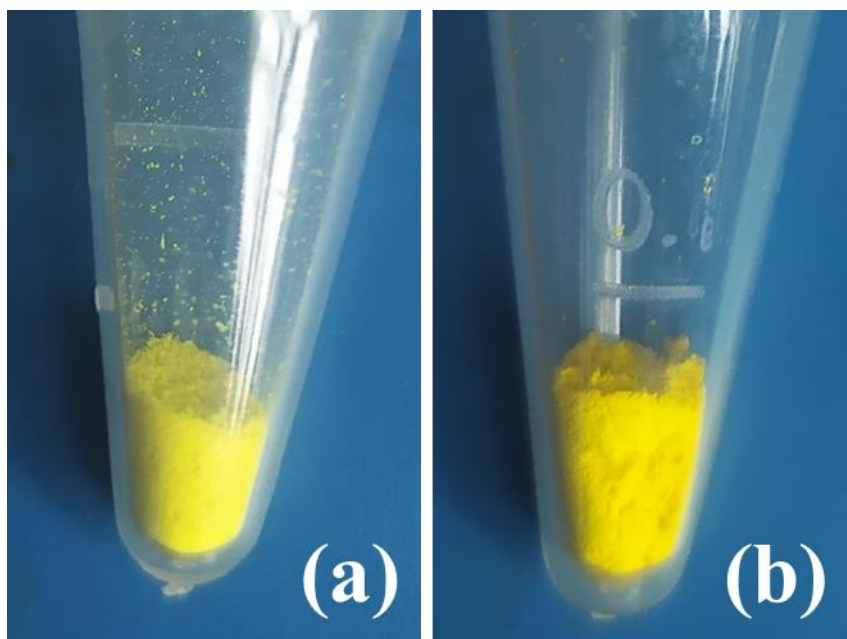
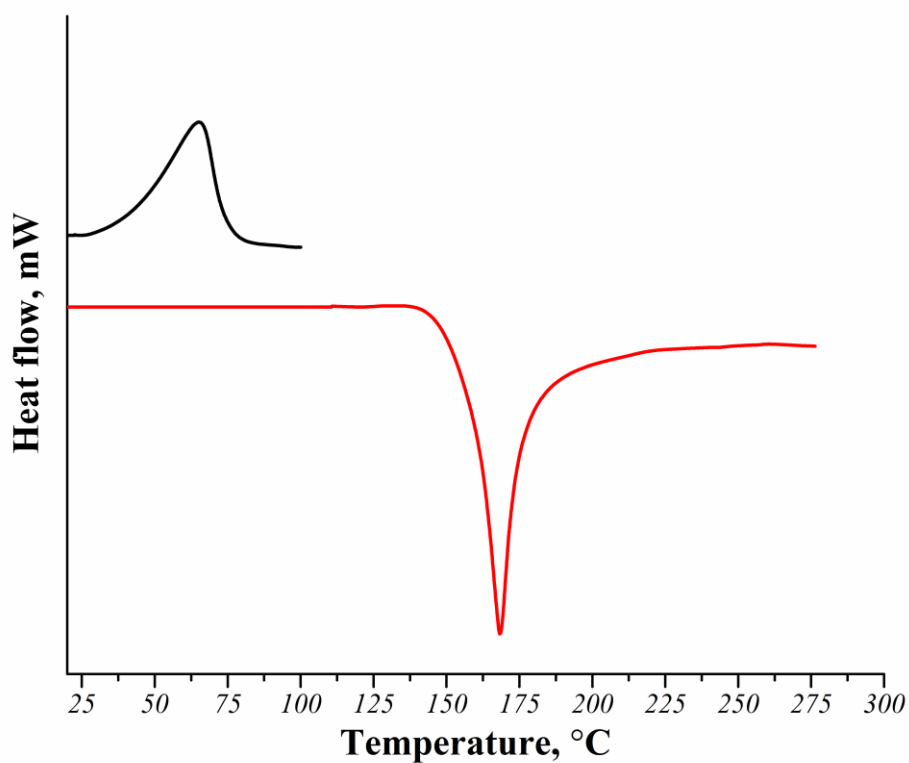
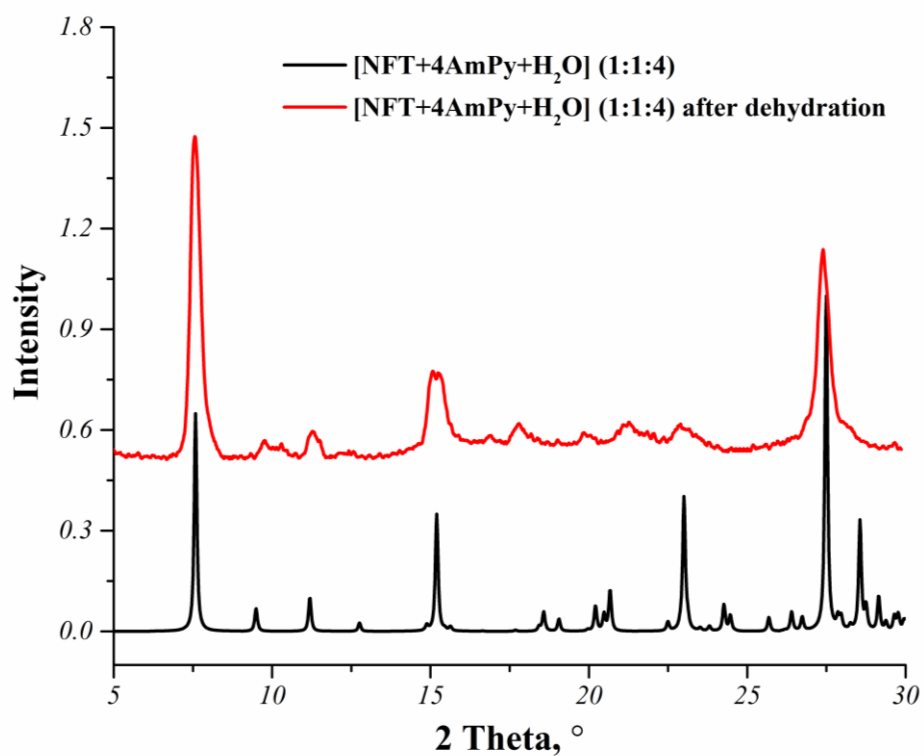


Figure S4. Color difference between (a) [NFT+4AmPy+H₂O] salt (1:1:1) and (b) [NFT+4AmPy+H₂O] salt (1:1:4) prepared by LAG method.



(a)



(b)

Figure S5. (a) DSC curves of the [NFT+4AmPy+H₂O] salt (1:1:4): the black line shows the first heating, the red one – the second heating; (b) comparison of the experimental PXRD pattern of [NFT+4AmPy+H₂O] salt (1:1:4) after dehydration and the simulated PXRD pattern calculated from the SCXRD data.

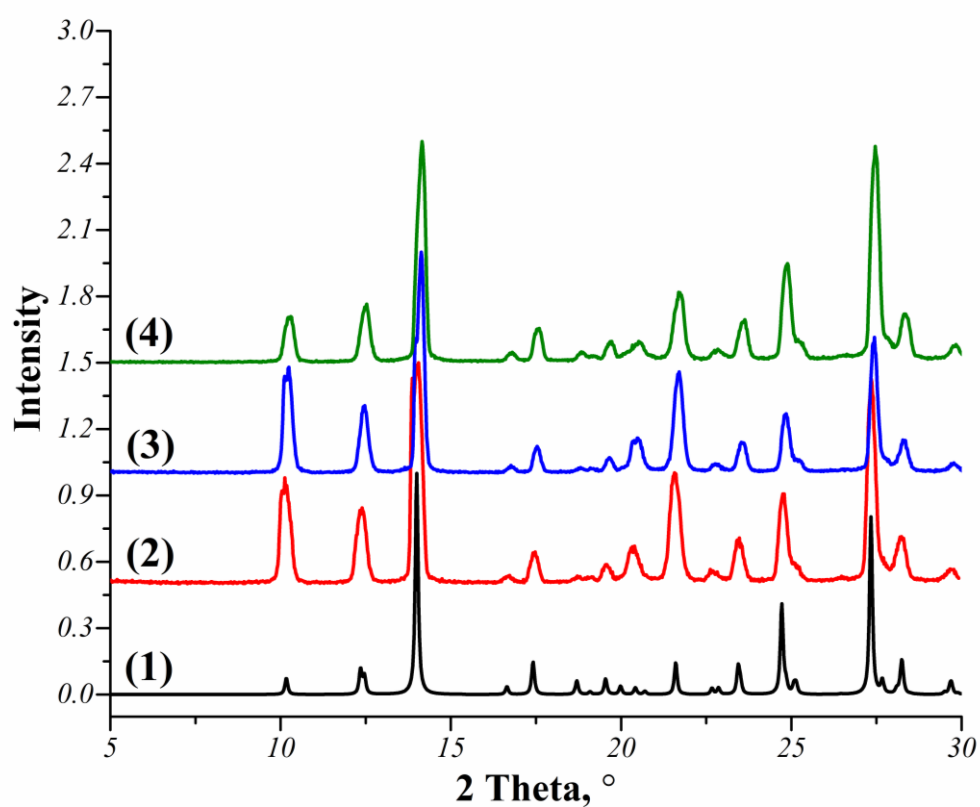


Figure S6. Results of PXRD analysis of the residual materials collected at the end of the dissolution experiments: (1) calculated PXRD patterns of NFT monohydrate; (2) [NFT+4AmPy] (1:1); (3) [NFT+4AmPy+H₂O] (1:1:1) and (4) [NFT+4AmPy+H₂O] (1:1:4).

Table S1. Hydrogen bond geometries for the NFT hydrated salts

| $D-H\cdots A$ | $D-H, \text{\AA}$ | $H\cdots A, \text{\AA}$ | $D\cdots A, \text{\AA}$ | $D-H\cdots A, ^\circ$ |
|---|-------------------|-------------------------|-------------------------|-----------------------|
| [NFT+4AmPy+H₂O] salt (1:1:1) | | | | |
| N12 ⁺ —H1 ⁺ ...N1 ⁻ | 0.904 (19) | 1.963 (19) | 2.8396 (17) | 162.9 (16) |
| N11—H10...O21 | 0.91 (2) | 2.03 (2) | 2.9241 (18) | 167.7 (17) |
| N11—H11...O1 ⁱ | 0.905 (19) | 2.030 (19) | 2.8973 (17) | 160.2 (16) |
| O21—H20...O2 ⁱⁱ | 0.90 (2) | 1.88 (2) | 2.7669 (16) | 169 (2) |
| O21—H21...O2 ⁱⁱⁱ | 0.87 (2) | 1.97 (2) | 2.8286 (17) | 169 (2) |
| Symmetry codes: (i) $x+1/2, -y+1/2, z-1/2$; (ii) $x+1/2, -y+1/2, z+1/2$; (iii) $-x+3/2, y-1/2, -z+1/2$ | | | | |
| [NFT+4AmPy+H₂O] salt (1:1:4) | | | | |
| N11 ⁺ —H1 ⁺ ...N1 ⁻ | 0.91 (3) | 1.86 (3) | 2.7669 (18) | 174 (3) |
| O21—H21...O1 | 0.82 (3) | 1.94 (3) | 2.7645 (18) | 176 (3) |
| O21—H22...O25 | 0.75 (4) | 2.08 (4) | 2.7837 (15) | 156 (4) |
| O23—H24...O2 ⁱ | 0.82 (4) | 1.93 (4) | 2.7523 (18) | 178 (3) |
| O23—H23...O25 | 0.81 (4) | 1.99 (4) | 2.7496 (15) | 156 (4) |
| O25—H28...O21 | 0.78 (4) | 2.02 (4) | 2.7837 (15) | 166 (3) |
| O25—H25...O23 | 0.81 (4) | 1.99 (4) | 2.7496 (15) | 158 (3) |
| O25—H26...O25 ⁱⁱ | 0.86 (3) | 1.94 (3) | 2.793 (2) | 172 (3) |
| O25—H27...O25 ⁱⁱⁱ | 0.84 (4) | 1.95 (4) | 2.754 (2) | 160 (4) |
| N12—H10...O3 ^{iv} | 0.87 (2) | 2.36 (2) | 3.1269 (17) | 148 (2) |
| N12—H11...O1 ^{iv} | 0.89 (2) | 2.09 (3) | 2.9628 (18) | 168 (2) |
| Symmetry codes: (i) $x+1/2, y, -z+3/2$; (ii) $-x+1, -y+2, -z+2$; (iii) $x, -y+5/2, z$; (iv) $x-1/2, y, -z+3/2$ | | | | |