

**Supporting Information**

**Mechanochemistry and Cocrystallization of 3-Iodoethynylbenzoic  
Acid with Nitrogen-Containing Heterocycles: Concurrent Halogen  
and Hydrogen Bonding**

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## Experimental Masses and $^{13}\text{C}$ Solid-State NMR Parameters

**Table S1.** Experimental masses used for the synthesis of each cocrystal by slow evaporation.

compound	mass <b>1</b> (mg)	mass acceptor (mg)
<b>1a</b>	100	50.0
<b>1b</b>	100	41.0
<b>1c(i)</b>	100	31.7
<b>1d</b>	100	51.6

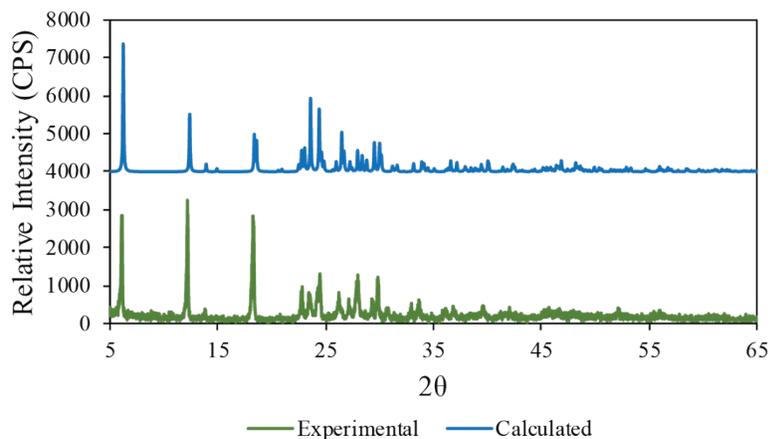
**Table S2.** Experimental masses and yields for the synthesis of each cocrystal by ball milling.

compound	mass <b>1</b> (mg)	mass acceptor (mg)	mass product (mg)	yield (%)
<b>1a</b>	702.5	358.0	832.2	78
<b>1b</b>	209.9	87.7	253.5	85
<b>1c(i)</b>	47.0	17.2	58.3	91
<b>1d</b>	199.8	103.5	197.5	65

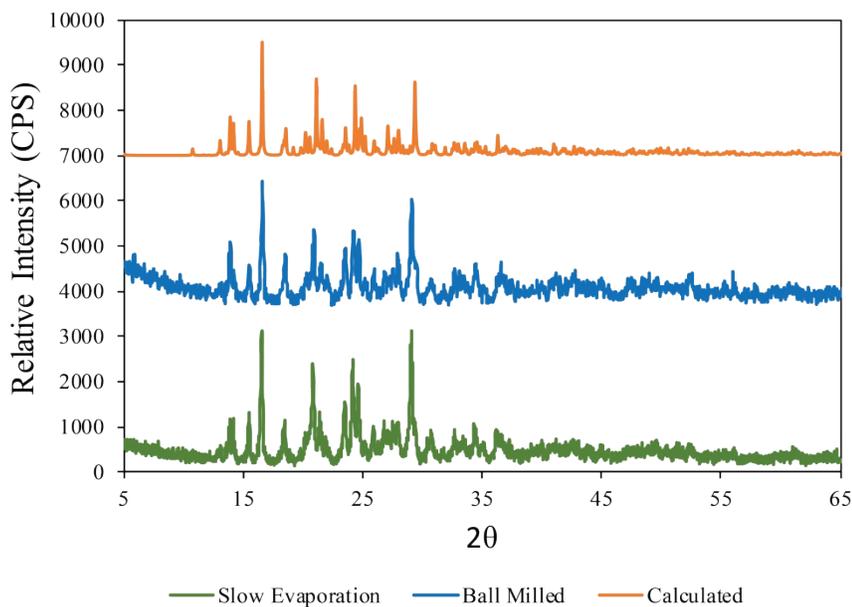
**Table S3.** Selected  $^{13}\text{C}$  solid-state NMR acquisition parameters for the samples obtained by slow evaporation and ball milling.

sample	spinning speeds	recycle delay (s)	number of transients
<b>1</b>	10 kHz	60	256
<b>1a</b> (slow evaporation)	8 kHz, 9 kHz	3	2048
<b>1a</b> (ball milling)	9 kHz	3	2048
<b>1b</b> (slow evaporation)	8 kHz, 10 kHz	5	256
<b>1b</b> (ball milling)	10 kHz	5	1024
<b>1c(i)</b> (slow evaporation)	7 kHz, 8 kHz	10	2048
<b>1c(i)</b> (ball milling)	7 kHz	10	1024
<b>1d</b> (slow evaporation)	8 kHz, 9 kHz	5	2048
<b>1d</b> (ball milling)	9 kHz	5	4096

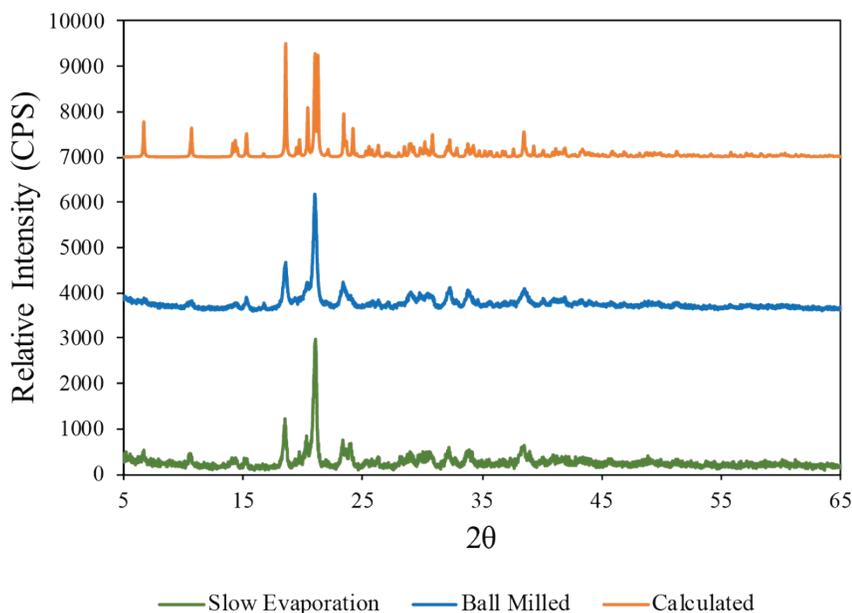
## Powder X-ray Diffractograms



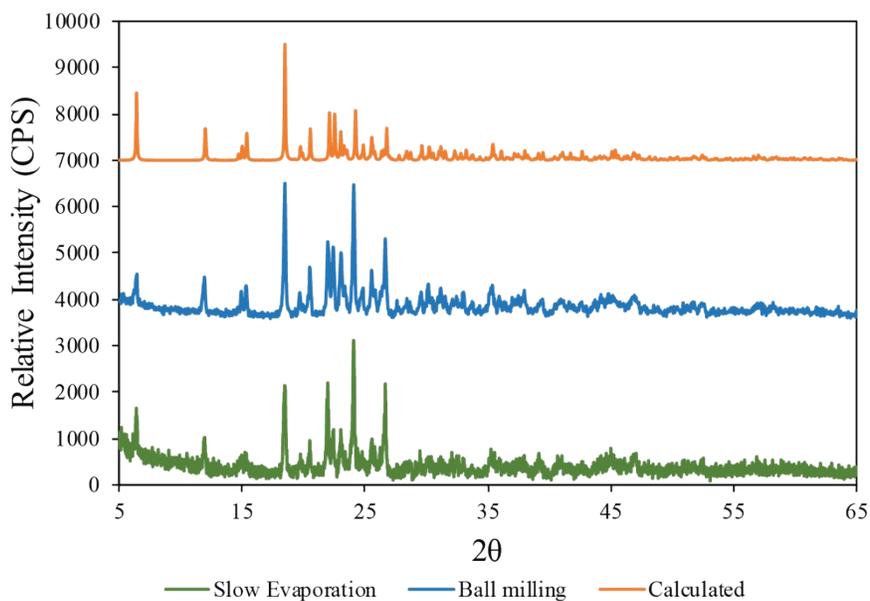
**Figure S1.** Experimental and calculated powder X-ray diffractograms of 3-iodoethynylbenzoic acid (**1**).



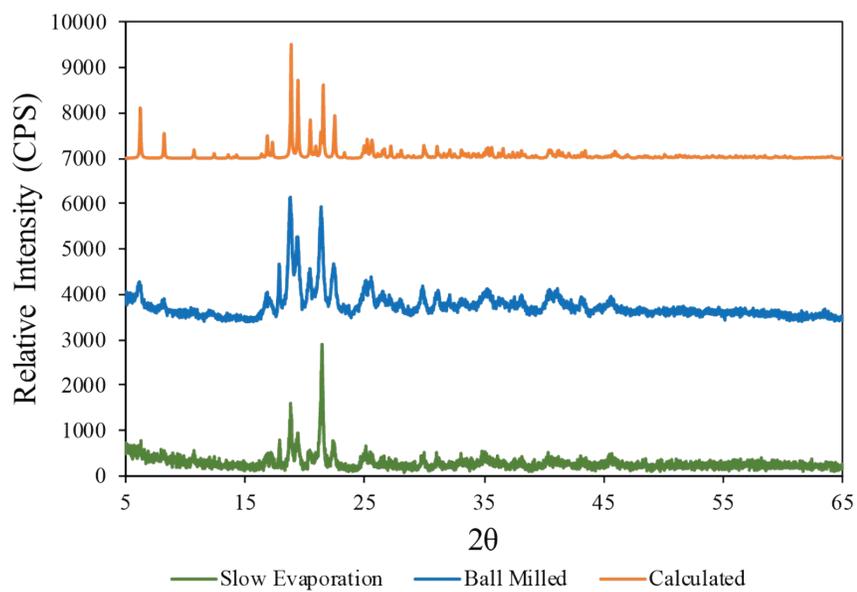
**Figure S2.** Experimental and calculated powder X-ray diffractograms of **1a**, (3-iodoethynylbenzoic acid)(2,3,5,6-tetramethylpyrazine), comparing the sample obtained from slow evaporation and the sample obtained from ball milling.



**Figure S3.** Experimental and calculated powder X-ray diffractograms of **1b**, (3-iodoethynylbenzoate)(1,4-diazabicyclo[2.2.2]octanium), comparing the sample obtained from slow evaporation and the sample obtained from ball milling.

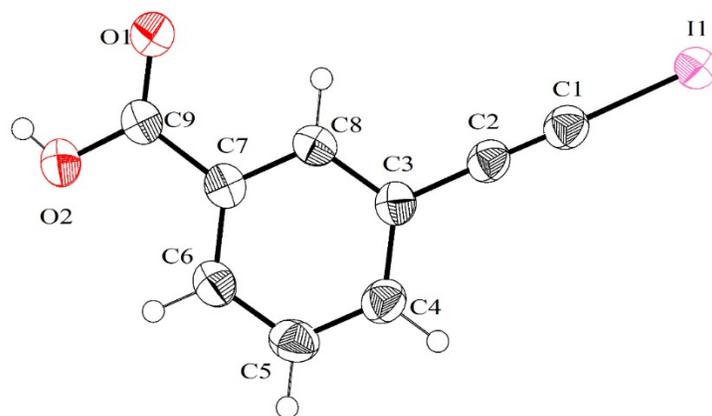


**Figure S4.** Experimental and calculated powder X-ray diffractograms of **1c(i)**, (3-iodoethynylbenzoate)(piperazinium), comparing the sample obtained from slow evaporation and the sample obtained from ball milling.

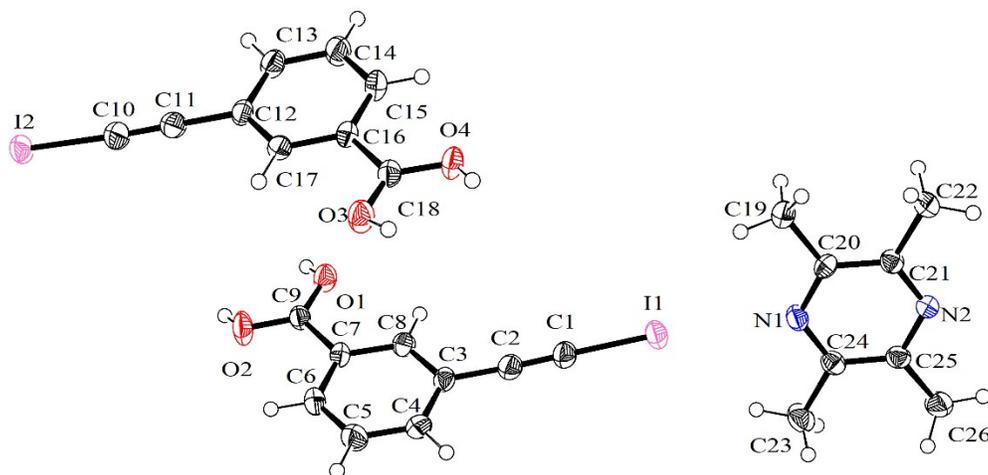


**Figure S5.** Experimental and calculated powder X-ray diffractograms of **1d**, (3-iodoethynylbenzoic acid)(hexamethylenetetramine), comparing the sample obtained from slow evaporation and the sample obtained from ball milling.

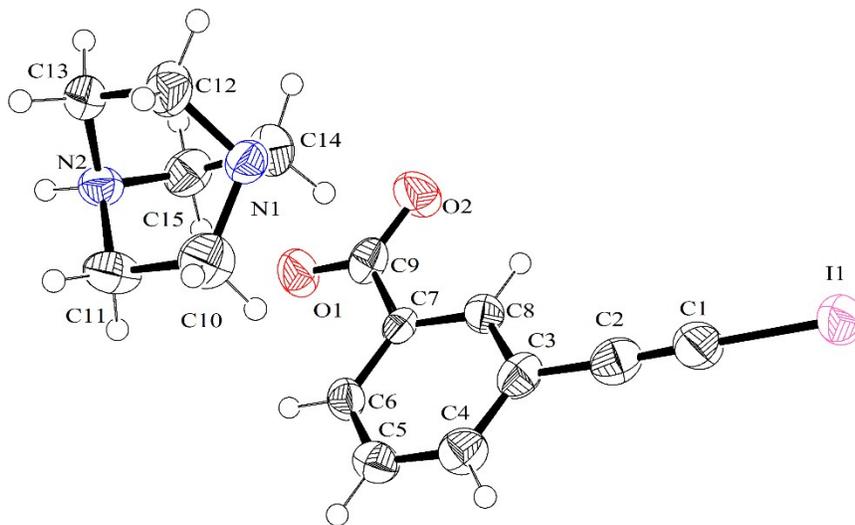
## ORTEP Plots



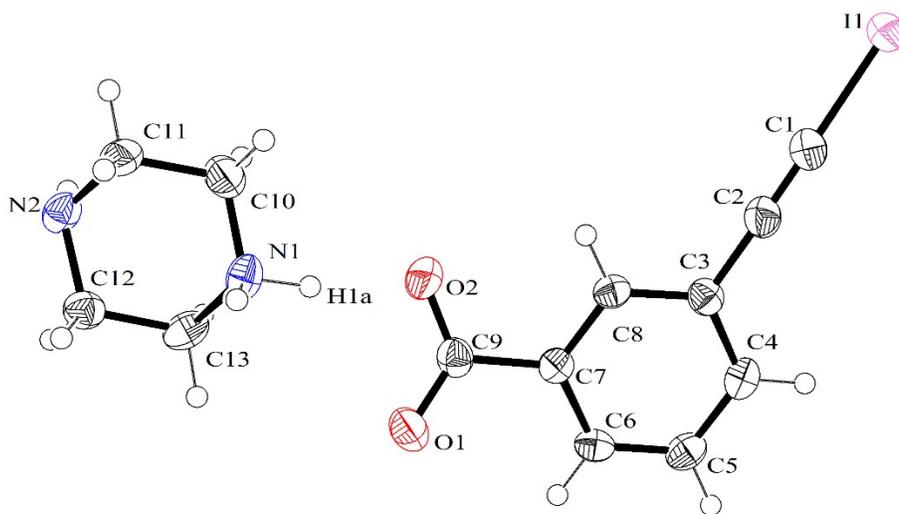
**Figure S6.** Thermal ellipsoid plot of 3-iodoethylbenzoic acid (**1**).



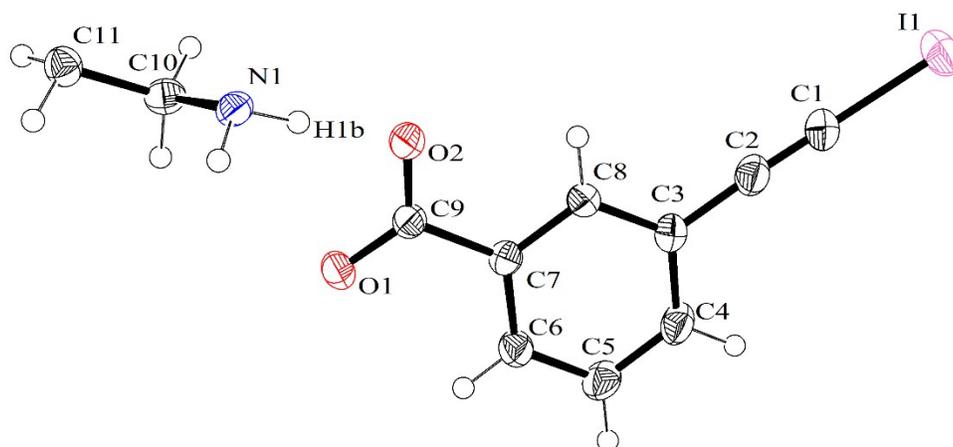
**Figure S7.** Thermal ellipsoid plot of (3-iodoethylbenzoic acid)<sub>2</sub>(2,3,5,6-tetramethylpyrazine) (**1a**).



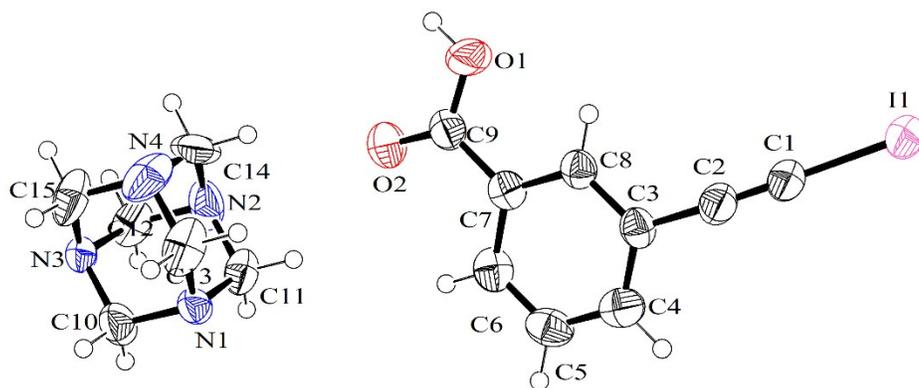
**Figure S8.** Thermal ellipsoid plot of (3-iodoethynylbenzoate)(1,4-diazabicyclo[2.2.2]octanium) (**1b**).



**Figure S9.** Thermal ellipsoid plot of (3-iodoethynylbenzoate)(piperazinium) [**1c(i)**].



**Figure S10.** Thermal ellipsoid plot of (3-iodoethynylbenzoate)<sub>2</sub>(piperazinium) [**1c(ii)**].



**Figure S11.** Thermal ellipsoid plot of (3-iodoethynylbenzoic acid)(hexamethylenetetramine) (**1d**).