SUPPLEMENTARY MATERIAL

Mechanochemical Synthesis of Phthalimides with Crystal Structures of Intermediates and Products.

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List of supplementary material

Figure S1. Powder X-ray diffractograms of solids obtained during synthesis of N-phthaloyl-2,6-dimethylaniline (3ai)
a) starting product (phthalic anhydride) and ground solid of 1a and 2i.
b) solid obtained after grinding of 1a and 2i and heating to 200°C.
c) pattern simulated from the single crystal X-ray structure.

Figure S2. X-ray diffraction analysis of the solid corresponding to 4-carboxylic-N-phthaloyl-2,6-dimethylaniline (3bi), obtained after grinding of 1b and 2i and heating to 200°C. a) Powder diffractograms recorded before and after heating of the ground sample and b) single crystal structure obtained by recrystallization from Toluene/MeOH or c) from a solution in EtOAc. d, e) patterns simulated (Mercury Program) from the single crystal X-ray structures.

Figure S3. Crystal structure of 4-carboxylic-N-phthaloyl-p-anisole (3bn, m.p. >260°C)

Figure S4. Solid-state reaction of 3-nitrophthalic anhydrates (1c) with 2,6-dibromoaniline (2k) leads to an open product of 3-nitro-N-phthaloyl-2,6-dibromoaniline (3ck).

Figure S5. Structure of the intermediate co-crystal observed during the reaction of 4-nitrophthalic anhydride (1d) and 4-methylaniline (2m). Two perpendicular views (a, b) of the π–π stacking 1:1 co-crystal. c) H-bonding network (dashed line).

Nuclear Magnetic Resonance (NMR).

Crystallographic data

Table S1. Selected H-bonding interactions
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b) solid obtained after grinding of 1a and 2i and heating to 200°C.
c) pattern simulated from the single crystal X-ray structure (CCDC Mercury program).
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Nuclear Magnetic Resonance (NMR).

All NMR (H) were recorded on Jeol spectrometer (JNM EX-400) at 25°C. Chemical shifts are reported in parts per million (ppm) using the solvent residual peak as reference (CDCl₃: 7.26 ppm; DMSO-d₆: 2.50 ppm; CD₃OD: 3.31 ppm). Coupling constants (J) are reported in Hertz (Hz). The resonance multiplicity is described as s for singlet, bs for broad singlet, d for doublet, t for triplet, q for quadruplet and m for multiplet.

**N-phthaloyl-2,6-dimethylaniline (3ai):** H NMR (400MHz, CDCl₃) : δ (ppm) = 2.16 (s, 6H), 7.19 (d, 2H, J=7.6Hz), 7.27 (t, 1H, J=7.5Hz), 7.80 (m, 2H), 7.96 (m, 2H)

**N-phthaloyl-2,6-dibromoaniline (3ak):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 6.43 (t, 1H, J=8.0Hz), 7.38 (d, 2H, J=8.0Hz), 7.97 (m, 2H), 8.06 (m, 2H)

**N-phthaloyl-p-anisole (3an):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 3.78 (s, 3H), 7.03 (dt, 2H, J₁=8.9, J₂=2.8), 7.32 (dt, 2H, J₁=8.9, J₂=2.8), 7.86 (m, 2H), 7.91 (m, 2H)

**4-carboxylic-N-phthaloyl-2,6-dimethylaniline (3bi):** H NMR (400MHz, CDCl₃) : δ (ppm) = 2.16 (s, 6H), 7.20 (d, 2H, J=7.6Hz), 7.29 (t, 1H, J=7.6Hz), 8.09 (d, 1H, J=7.8), 8.56 (dd, 1H, J₁=7.8Hz, J₂=1.4), 8.68 (s, 1H)

**4-carboxylic-N-phthaloyl-2,6-dichloroaniline (3bj):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 7.63 (t, 1H, J=8.1Hz), 7.75 (d, 2H, J=8.2Hz), 8.09 (td, 1H, J₁=8.0Hz, J₂=1.8Hz), 8.19 (d, 1H, J=7.8Hz), 8.23 (d, 1H, J=1.6Hz)

**4-carboxylic-N-phthaloyl-2,4-dichloroaniline (3bl):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 7.47 (d, 1H, J=8.5), 7.66 (d, 1H, J=1.8Hz), 7.69 (dd, 1H, J₁=8.3Hz, J₂=2.2Hz), 8.02-8.13 (m, 3H)

**4-carboxylic-N-phthaloyl-2-carboxylicaniline (3bp):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 6.46 (t, 1H, J=8.0Hz), 8.35 (s, 1H), 8.93 (dd, 1H, J=1.7Hz)

**3-nitro-N-phthaloyl-2,6-dimethylaniline (3ci):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 2.26 (s, 6H), 7.02 (s, 2H), 7.09 (s, 1H), 7.76 (t, 1H, J=8.1Hz), 8.13 (d, 1H, J=7.8Hz), 8.20 (d, 1H, J=8.0Hz)

**3-nitro-N-phthaloyl-2,6-dichloroaniline (3cj):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 6.54 (t, 1H, J=7.9Hz), 7.19 (d, 2H, J=8.0Hz), 8.17 (t, 1H, J=8.2Hz), 8.36 (d, 1H, J=7.6Hz), 8.47 (d, 1H, J=8.0Hz)

**3-nitro-N-phthaloyl-2,6-dibromoaniline (3ck):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 7.13 (t, 1H, J=8.0Hz), 7.68 (d, 2H, J=8.0Hz), 7.77 (t, 1H, J=8.0Hz), 8.12 (d, 1H, J=7.8Hz), 8.20 (d, 1H, J=8.2Hz)

**3-nitro-N-phthaloyl-4-methylaniline (3cm):** H NMR (400MHz, DMSO-d₆) : δ (ppm) = 2.34 (s, 3H), 7.29 (dd, 4H, J=8.5Hz, J=2.8Hz), 8.07 (t, 1H, J=7.8Hz), 8.21 (d, 1H, J=7.8Hz), 8.30 (d, 1H, J=7.8Hz)
3-nitro-N-phthaloyl-p-anisole (3cn): $^1$H NMR (400MHz, DMSO-$d_6$): $\delta$ (ppm) = 3.78 (s, 3H), 7.31 (dd, 2H, $J_1$=8.9Hz, $J_2$=2.2Hz), 7.38 (dd, 2H, $J_1$=8.9Hz, $J_2$=2.2Hz), 8.07 (t, 1H, $J$=7.8Hz), 8.19 (dd, 2H, $J_1$=7.8Hz, $J_2$=1.14Hz)

3-nitro-N-phthaloyl-3-nitroaniline (3co): $^1$H NMR (400MHz, DMSO-$d_6$): $\delta$ (ppm) = 7.62 (t, 1H, $J$=8.2Hz), 7.84 (t, 2H, $J$=8.0Hz), 7.95 (dd, 1H, $J_1$=7.9Hz, $J_2$=1.9Hz), 8.29 (dd, 1H, $J_1$=7.9Hz, $J_2$=1.0Hz), 8.37 (dd, 1H, $J_1$=8.2Hz, $J_2$=1.1Hz), 8.60 (t, 1H, $J$=2.1Hz)

4-nitro-N-phthaloyl-2,6-dimethylaniline (3di): $^1$H NMR (400MHz, DMSO-$d_6$): $\delta$ (ppm) = 7.22 (d, 2H, $J$=7.6Hz), 7.31 (t, 1H, $J$=7.7Hz), 8.24 (d, 1H, $J$=8.0Hz), 8.46 (d, 1H, $J$=1.8Hz), 8.69 (dd, 1H, $J_1$=8.1Hz, $J_2$=1.9Hz)

4-nitro-N-phthaloyl-2,6-dibromoaniline (3dk): $^1$H NMR (400MHz, DMSO-$d_6$): $\delta$ (ppm) = 7.47 (t, 1H, $J$=8.1Hz), 7.91 (d, 2H, $J$=8.0Hz), 8.35 (d, 1H, $J$=8.2Hz), 8.49 (d, 1H, $J$=2.1Hz), 8.75 (dd, 1H, $J_1$=8.1Hz, $J_2$=1.9Hz)

4-nitro-N-phthaloyl-4-methylaniline (3dm): $^1$H NMR (400MHz, DMSO-$d_6$): $\delta$ (ppm) = 2.34 (s, 3H), 7.31 (s, 4H), 8.17 (d, 1H, 8.2Hz), 8.54 (m, 1H), 8.64 (m, 1H)

4-nitro-N-phthaloyl-p-anisole (3dn): $^1$H NMR (400MHz, DMSO-$d_6$): $\delta$ (ppm) = 3.79 (s, 3H), 7.06 (d, 2H, $J$=8.7Hz), 7.35 (d, 2H, $J$=8.9Hz), 8.17 (d, 1H, $J$=8.2Hz), 8.54 (d, 1H, $J$=1.8Hz), 8.55 (dd, 1H, $J_1$=8.1Hz, $J_2$=1.9Hz)

4-nitro-N-phthaloyl-3-nitroaniline (3do): $^1$H NMR (400MHz, DMSO-$d_6$): $\delta$ (ppm) = 7.86 (t, 1H, $J$=8.1Hz), 7.85 (t, 1H, $J$=8.0Hz), 8.24 (d, 1H, $J$=8.2Hz), 8.31 (m, 1H), 8.40 (t, 1H, $J$=2.1Hz), 8.60 (d, 1H, $J$=2.1Hz), 8.68 (dd, 1H, $J_1$=8.1 Hz, $J_2$=1.9Hz)
Crystallographic data.

Single crystal X-ray diffraction was performed on a Gemini Ultra R system (4-circle kappa platform, Ruby CCD detector) using Mo ($\lambda = 0.71073$ Å) or Cu Kα radiation ($\lambda = 1.54056$ Å). Selected crystals were mounted on the tip of a quartz pin using cyanoacrylate (Commercial glue). Cell parameters were estimated from a pre-experiment run and full data sets collected at room temperature. Structures were solved by direct methods with sir92 (v.3.0) program and then refined on $F^2$ using SHELXL-97 software. Non-hydrogen atoms were anisotropically refined and the hydrogen atoms (not implicated in H-bonds) in the riding mode with isotropic temperature factors fixed at 1.2 times U(eq) of the parent atoms (1.5 times for methyl groups). Hydrogen atoms implicated in H-bonds were localized by difference maps ($\Delta F$).

**Crystal Data for PA-DMA (3ai) CCDC 1021624.** Prism colourless crystals, orthorhombic, Pbc1, $a = 9.837(2), b = 16.124(4), c = 8.643(3)\AA, \alpha = 90.00, \beta = 90.00, \gamma = 90.00^\circ, V = 1370.9(6) \AA^3, Z = 4, \rho_{calc} = 1.217g \text{ cm}^{-3}, F_{000} = 528, \lambda \text{ Mo Kα} = 0.71073 \AA, \theta_{max} = 25.0^\circ, 4759$ total measured reflections, 1994 independent reflections ($R_{int} = 0.031$), 1322 observed reflections ($I > 2 \sigma(I)$), $\mu = 0.081 \text{ mm}^{-1}, 136$ parameters, $R_I$ (observed data) = 0.0880, $S = \text{GooF} = 1.09, \Delta/s.u. = 0.00$, residual $\rho_{max} = 0.27 \text{ e } \AA^{-3}, \rho_{min} = -0.26 \text{ e } \AA^{-3}$.

**Crystal Data for BTA-DMA 1st form (3bi) CCDC 1021625.** Plate colourless crystals, triclinic, P-1, $a = 8.3591(3), b = 10.4196(7), c = 19.0455(9)\AA, \alpha = 88.407(5), \beta = 88.481(3), \gamma = 76.340(4)^\circ, V = 1610.95(15) \AA^3, Z = 1, \rho_{calc} = 1.311g \text{ cm}^{-3}, F_{000} = 665, \lambda \text{ Mo Kα} = 0.71073 \AA, \theta_{max} = 25.0^\circ, 11980$ total measured reflections, 5663 independent reflections ($R_{int} = 0.024$), 3983 observed reflections ($I > 2 \sigma(I)$), $\mu = 0.093 \text{ mm}^{-1}, 438$ parameters, $R_I$ (observed data) = 0.0468, $S = \text{GooF} = 0.99, \Delta/s.u. = 0.02$, residual $\rho_{max} = 0.16 \text{ e } \AA^{-3}, \rho_{min} = -0.17 \text{ e } \AA^{-3}$.

**Crystal Data for BTA-DMA 2nd form (3bi) CCDC 1021626.** Plate colourless crystals, triclinic, P-1, $a = 8.4040(5), b = 8.4040(5), c = 15.2401(1)\AA, \alpha = 97.881(6), \beta = 93.354(5), \gamma = 96.810(6)^\circ, V = 1468.73(18) \AA^3, Z = 4, \rho_{calc} = 1.335g \text{ cm}^{-3}, F_{000} = 616, \lambda \text{ Mo Kα} = 0.71073 \AA, \theta_{max} = 23.3^\circ, 10179$ total measured reflections, 4188 independent reflections ($R_{int} = 0.078$), 2204 observed reflections ($I > 2 \sigma(I)$), $\mu = 0.096 \text{ mm}^{-1}, 400$ parameters, $R_I$ (observed data) = 0.0705, $S = \text{GooF} = 1.01, \Delta/s.u. = 0.03$, residual $\rho_{max} = 0.19 \text{ e } \AA^{-3}, \rho_{min} = -0.21 \text{ e } \AA^{-3}$.

**Crystal Data for 3NPA-DBA open form (3ck) CCDC 1021627.** Needle colourless crystals, triclinic, $P-1, a = 9.188(5), b = 13.284(7), c = 14.477(8)\AA, \alpha = 68.21(5), \beta = 81.24(4), \gamma = 75.69(4)^\circ, V = 1586.2(16) \AA^3, Z = 2, \rho_{calc} = 1.857g \text{ cm}^{-3}, F_{000} = 862, \lambda \text{ Mo Kα} = 0.71073 \AA, \theta_{max} = 25.0^\circ, 11937$ total measured reflections, 5562 independent reflections ($R_{int} = 0.052$), 3222 observed reflections ($I > 2 \sigma(I)$), $\mu = 5.136 \text{ mm}^{-1}, 415$ parameters, $R_I$ (observed data) = 0.0655, $S = \text{GooF} = 1.04, \Delta/s.u. = 0.00$, residual $\rho_{max} = 0.88 \text{ e } \AA^{-3}, \rho_{min} = -0.59 \text{ e } \AA^{-3}$.

**Crystal Data for 3NPA-pAni (3cn) CCDC 1021628.** Needle yellow crystals, Monoclinic, $P2_1, a = 3.8289(1), b = 23.3314(7), c = 7.4554(2)\AA, \alpha = 90, \beta = 98.153(3), \gamma = 90^\circ, V = 659.3(1) \AA^3, Z = 2, \rho_{calc} = 1.502g \text{ cm}^{-3}, F_{000} = 308, \lambda \text{ Cu Kα} = 1.54184 \AA, \theta_{max} = 66.6^\circ, 5907$ total measured reflections, 2304 independent reflections ($R_{int} = 0.0182$), 2285 observed reflections ($I > 2 \sigma(I)$), $\mu = 0.977 \text{ mm}^{-1}, 200$ parameters, $R_I$ (observed data) = 0.0270, $S = \text{GooF} = 1.08, \Delta/s.u. = 0.001$, residual $\rho_{max} = 0.16 \text{ e } \AA^{-3}, \rho_{min} = -0.14 \text{ e } \AA^{-3}$.

8
Crystal Data for 4NPA-DBA (3dk) CCDC 1021629. Plate colourless crystals, monoclinic, \( I2/a \), \( a = 14.8231(9) \), \( b = 7.7177(6) \), \( c = 25.7142(4) \, \text{Å} \), \( \alpha = 90 \), \( \beta = 97.692(7) \), \( \gamma = 90 \, ^\circ \), \( V = 2915.2(4) \, \text{Å}^3 \), \( Z = 8 \), \( \rho_{\text{calc}} = 1.941 \, \text{g cm}^{-3} \), \( F_{000} = 1648 \), \( \lambda \, \text{Mo K} \alpha = 0.71073 \, \text{Å} \), \( \theta_{\text{max}} = 25.0 \, ^\circ \), 6774 total measured reflections, 2563 independent reflections (\( R_{\text{int}} = 0.079 \)), 1465 observed reflections (\( I > 2 \sigma(I) \)), \( \mu = 5.580 \, \text{mm}^{-1} \), 392 parameters, \( R_I \) (observed data) = 0.0491, \( S = \text{GooF} = 0.96 \), \( \Delta/\text{s.u.} = 0.00 \), residual \( \rho_{\text{max}} = 0.36 \, \text{e Å}^{-3} \), \( \rho_{\text{min}} = -0.40 \, \text{e Å}^{-3} \).

Crystal Data for 4NPA-DBA open form (3dk) Crystal Data for 4NPA-DBA open form (3dk). Plates colourless crystals, monoclinic, \( P2_1/n \), \( a = 14.0897(3) \), \( b = 4.8183(1) \), \( c = 22.6757(3) \, \text{Å} \), \( \alpha = 90 \), \( \beta = 90.555(9) \), \( \gamma = 90 \, ^\circ \), \( V = 1539.3(1) \, \text{Å}^3 \), \( Z = 4 \), \( \rho_{\text{calc}} = 1.916 \, \text{g cm}^{-3} \), \( F_{000} = 864 \), \( \lambda \, \text{Cu K} \alpha = 1.54184 \, \text{Å} \), \( \theta_{\text{max}} = 66.6 \, ^\circ \), 8457 total measured reflections, 2710 independent reflections (\( R_{\text{int}} = 0.0344 \)), 2466 observed reflections (\( I > 2 \sigma(I) \)), \( \mu = 6.946 \, \text{mm}^{-1} \), 1462 parameters, \( R_I \) (observed data) = 0.0465, \( S = \text{GooF} = 1.07 \), \( \Delta/\text{s.u.} = 0.00 \), residual \( \rho_{\text{max}} = 0.53 \, \text{e Å}^{-3} \), \( \rho_{\text{min}} = -0.68 \, \text{e Å}^{-3} \).

Crystal Data for 4NPA-pAni (3dn) CCDC 1021631. Triangular yellow crystals, monoclinic, \( Pn \), \( a = 6.9750(12) \), \( b = 3.876(1) \), \( c = 23.998(3) \, \text{Å} \), \( \alpha = 90 \), \( \beta = 91.227(12) \), \( \gamma = 90 \, ^\circ \), \( V = 648.6(2) \, \text{Å}^3 \), \( Z = 2 \), \( \rho_{\text{calc}} = 1.527 \, \text{g cm}^{-3} \), \( F_{000} = 308 \), \( \lambda \, \text{Mo K} \alpha = 0.71073 \, \text{Å} \), \( \theta_{\text{max}} = 23.3 \, ^\circ \), 1900 total measured reflections, 1462 independent reflections (\( R_{\text{int}} = 0.045 \)), 1224 observed reflections (\( I > 2 \sigma(I) \)), \( \mu = 0.117 \, \text{mm}^{-1} \), 200 parameters, \( R_I \) (observed data) = 0.0724, \( S = \text{GooF} = 1.14 \), \( \Delta/\text{s.u.} = 0.00 \), residual \( \rho_{\text{max}} = 0.32 \, \text{e Å}^{-3} \), \( \rho_{\text{min}} = -0.34 \, \text{e Å}^{-3} \).

Crystal Data for 4NPA-pTol co-crystal (3dm) CCDC 1021632. Triangular yellow crystals, Orthorhombic, \( Fdd2 \), \( a = 59.519(2) \), \( b = 12.942(5) \), \( c = 7.623(2) \, \text{Å} \), \( \alpha = 90 \), \( \beta = 90 \), \( \gamma = 90 \, ^\circ \), \( V = 5872(3) \, \text{Å}^3 \), \( Z = 16 \), \( \rho_{\text{calc}} = 1.440 \, \text{g cm}^{-3} \), \( F_{000} = 2656 \), \( \lambda \, \text{Mo K} \alpha = 0.71073 \, \text{Å} \), \( \theta_{\text{max}} = 25.0 \, ^\circ \), 20263 total measured reflections, 2560 independent reflections (\( R_{\text{int}} = 0.052 \)), 2335 observed reflections (\( I > 2 \sigma(I) \)), \( \mu = 0.113 \, \text{mm}^{-1} \), 217 parameters, \( R_I \) (observed data) = 0.0433, \( S = \text{GooF} = 1.01 \), \( \Delta/\text{s.u.} = 0.04 \), residual \( \rho_{\text{max}} = 0.26 \, \text{e Å}^{-3} \), \( \rho_{\text{min}} = -0.15 \, \text{e Å}^{-3} \).
Table S1. Selected H-bonding interactions 

Crystal Data for 3NPA-DBA (3ck).

<table>
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<th>D-H...A</th>
<th>d D-H (Å)</th>
<th>d H...A(Å)</th>
<th>d D_H...A(Å)</th>
<th>Angle D_H...A (°)</th>
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<td>BTA-DMA 1st form (3ck).</td>
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<tr>
<td>O4 -- H4O .. O23</td>
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<td>1.8500</td>
<td>2.652(2)</td>
<td>168.00</td>
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<td>BTA-DMA 2nd form (3ck).</td>
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<td>O3 -- H3O .. O24</td>
<td>0.8200</td>
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<td>3NPA-DBA (3ck).</td>
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<td>N1 -- H1 .. O21</td>
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<td>4NPA-DBA (3dk) open form.</td>
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<td>O4 -- H4O .. O1</td>
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<td>2.485(3)</td>
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<td>O1 -- H999 .. N20</td>
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<td>1.85(9)</td>
<td>2.824(3)</td>
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