

# The first report of the crystal structure of non-solvated $\mu$ -oxo boronsubphthalocyanine and the crystal structures of two solvated forms.

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## **Electronic Supplementary Data.**

1. **General Methods and Materials.** All solvents were purchased from Caledon Laboratories Ltd and used as received unless stated otherwise. Boron tribromide and phthalonitrile were obtained from Sigma Aldrich Canada. All nuclear magnetic resonance (NMR) spectra were acquired on a Bruker 400 MHz system in deuterated chloroform (CDCl<sub>3</sub>) purchased from Cambridge Isotope Laboratories which was used as received. All <sup>1</sup>H NMR spectra were referenced to an internal standard of 0.05% TMS. All ultraviolet-visible (UV-Vis) spectroscopy was performed using PerkinElmer Lambda 25 with a 10.00 mm path length for solution phase samples. High pressure liquid chromatography (HPLC) analysis was conducted using a Waters 2695 separation module with a Waters 2998 photodiode array and a Waters 4.6 mm × 100 mm SunFire™ C18 3.5 μm column. HPLC grade acetonitrile and DMF were eluted at 0.6 mL/min during operation at a composition of 80:20, respectively. Mass spectrometry was performed on a JEOL AccuTOF-DART mass spectrometer with a DART-ion source (no solvent required). The apparatus used and method followed for train sublimation has previously been disclosed (Morse et al, 2011).

## 2. Experimental and Methodology: Preparation

### 2.1. Bromo(subphthalocyanato)boron(III) (Br-BsubPc).

Br-BsubPc was synthesized according to the procedure of Potz *et al.* (2000). On train sublimation (Morse *et al.*, 2011) diffractable crystals were obtained which were determined to be  $\mu$ -oxo-(BsubPc)<sub>2</sub>•0.25H<sub>2</sub>O.

### 2.2. Hydroxy(subphthalocyanato)boron(III) (HO-BsubPc).

Br-BsubPc (1.063 g, 0.0022 mol) was added to DMSO (120 mL), water (8 mL, 0.444 mol) and pyridine (4 mL, 0.0496 mol) in a 250 mL three necked round bottom flask. The mixture was heated to 60°C for 24 hours with stirring. After 24 hours, the heat was turned off and the mixture was added to 360mL of distilled water. This was allowed to stand overnight, and was filtered the next day to remove **HO-BsubPc** as a fine precipitate which was then placed in a vacuum oven overnight (40°C, 10 mmHg). Yield: 0.587 g (61.3%). HPLC R<sub>T</sub> (DMF/ACN): 1.790min; UV-Vis: 557.4 nm; <sup>1</sup>H NMR (400 MHz, CDCl<sub>3</sub>, Me<sub>4</sub>Si) 8.77-8.83 (m, 6H), 7.85-7.91 (m, 6H), -1.13 (s, 1H).

### 2.3. $\mu$ -oxo-bis[(subphthalocyanato)boron(III)] ( $\mu$ -oxo-(BsubPc)<sub>2</sub>) (Method (ii) Figure 1).

1,2-Dichlorobenzene (1.4 mL) was added to Br-BsubPc (0.200 g,  $4.21 \times 10^{-4}$  mol) in a 4 Dram (15 mL) vial fitted with a condenser. One drop of water was added initially and the mixture was heated at reflux (181°C). The reaction progress was monitored by HPLC. A positive pressure of argon was maintained in the vessel throughout the experiment. Additional drops of water were added during the reaction such that the proportion of HO-BsubPc was approximately equal to that of Br-BsubPc by HPLC analysis. After 24 hours Br-BsubPc was no longer detectable by HPLC and the reaction was cooled to room temperature. On standing in the dark for a prolonged period of time, diffractable crystals were formed on the vial walls and on the stir bar. They were determined to be  $\mu$ -oxo-(BsubPc)<sub>2</sub>•2DCB. No further workup was performed.

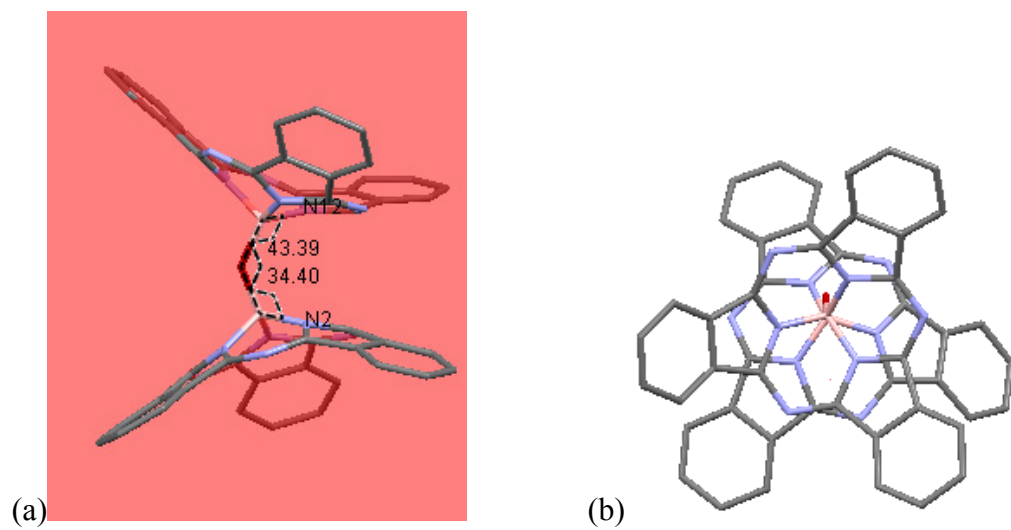
### 2.4. $\mu$ -oxo-bis[(subphthalocyanato)boron(III)] ( $\mu$ -oxo-(BsubPc)<sub>2</sub>) (Method (iii) Figure 1).

Adapted from the method of Mori *et al.* (2008): 1,2-Dichlorobenzene (1.5 mL) was added to HO-BsubPc (0.100 g,  $2.43 \times 10^{-4}$  mol) and Br-BsubPc (0.115 g,  $2.43 \times 10^{-4}$  mol) in a 4 Dram (15 mL) vial fitted with a condenser and heated at reflux (181°C). A positive pressure of argon was maintained in the vessel throughout the experiment. The reaction progress was monitored by HPLC. After 20 hours the reaction was cooled to room temperature. The solvent was removed by rotary evaporation, and the solid was then placed in a vacuum oven overnight to dry (40°C, 10 mmHg). (Yield: 0.298 g (>100 %); HPLC R<sub>T</sub>: 2.88 min (56.5%, at 545 nm)). The crude solid was then purified by Kauffman column using alumina as the stationary phase and dichloromethane as the mobile phase. A first fraction contained an unknown impurity (HPLC R<sub>T</sub>: 2.0 min) and the second fraction (stopped collection after 3 hours) was a mixture of the same impurity (HPLC R<sub>T</sub>: 2.0 min) and  $\mu$ -oxo-(BsubPc)<sub>2</sub>. The solvent of the second fraction was removed by rotary evaporation, and the solid was then placed in a vacuum oven overnight (40°C, 10 mmHg). (Yield: 0.090 g (46 %)). The solid (0.088 g) was train sublimed at 450° C. A

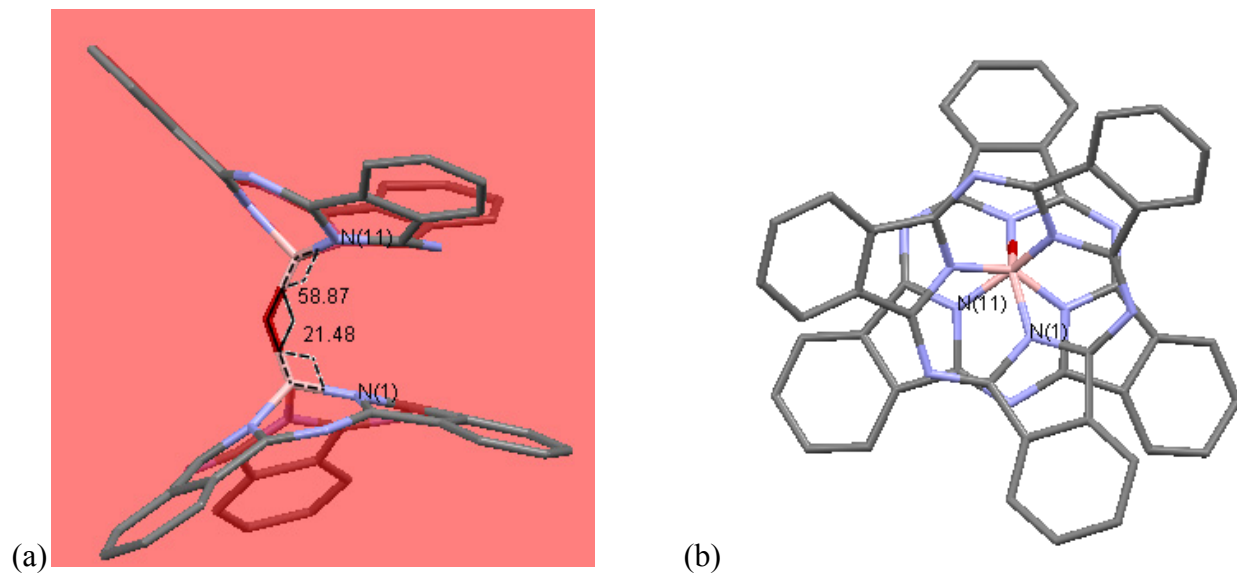
pink film at the end of the tube first became visible at 350°C. Pure crystals were removed by gentle tapping of the glass tube (Yield: 0.031 g (15.8%)). These crystals were confirmed to be  $\mu$ -oxo-(BsubPc)<sub>2</sub><sup>subl</sup>.

### **2.5. $\mu$ -oxo-bis[(subphthalocyanato)boron(III)] ( $\mu$ -oxo-(BsubPc)<sub>2</sub>) (Method (iii), larger scale, Figure 1).**

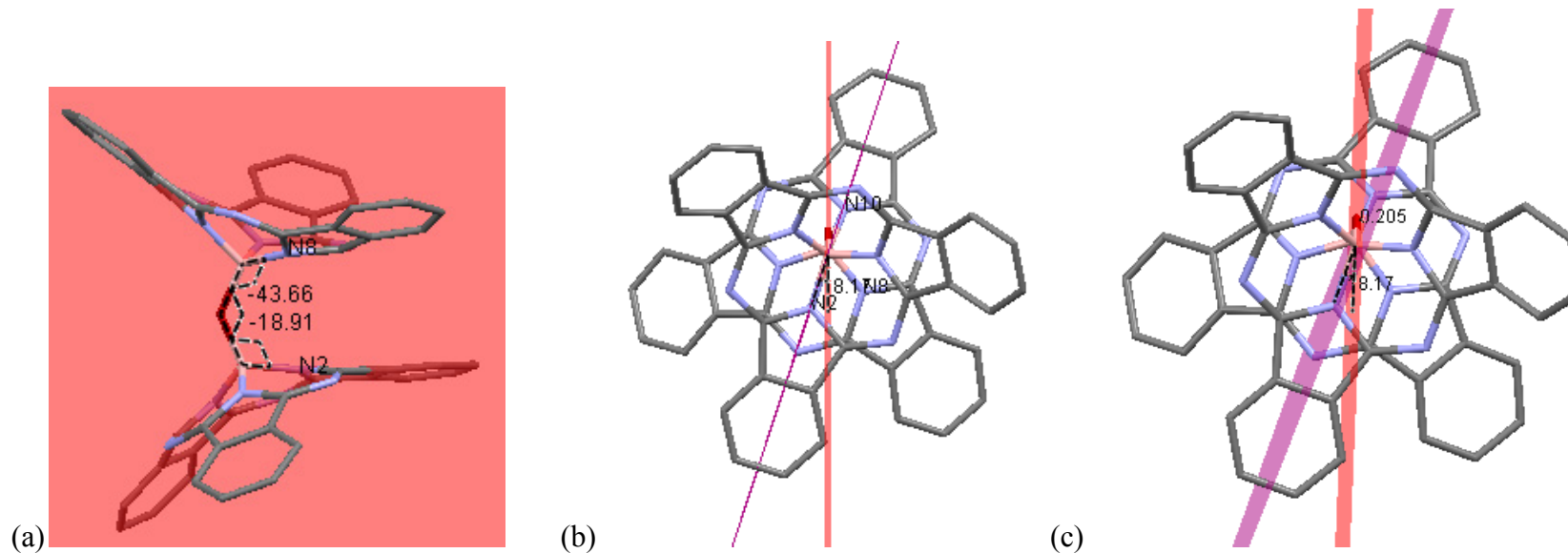
Method (iii) was subsequently scaled-up by a factor of 5 as follows: 1,2-Dichlorobenzene (1.5 mL) was added to HO-BsubPc (0.500 g,  $1.21 \times 10^{-3}$  mol) and Br-BsubPc (0.576 g,  $1.21 \times 10^{-3}$  mol) in a scintillation vial (20 mL) fitted with a condenser and heated at reflux (181°C) for 31 hours. A positive pressure of argon was maintained in the vessel throughout the experiment. It was observed that on cooling and if left stationary in the dark that visible crystals would form on the vial walls and on the stir bar as above. These crystals were determined to be  $\mu$ -oxo-(BsubPc)<sub>2</sub>•2DCB. No further workup was performed.



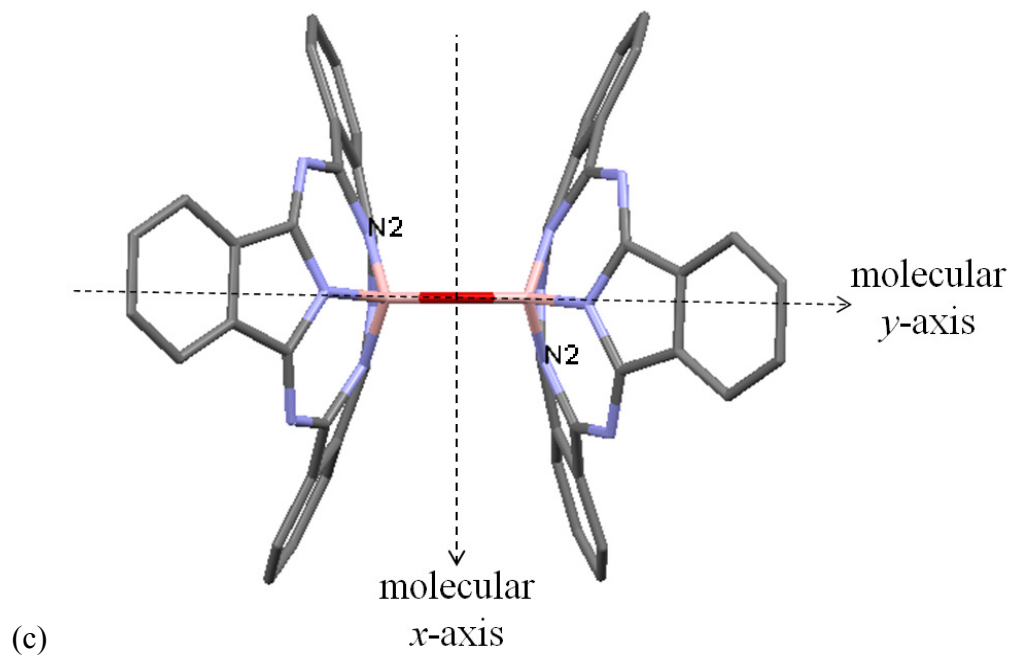
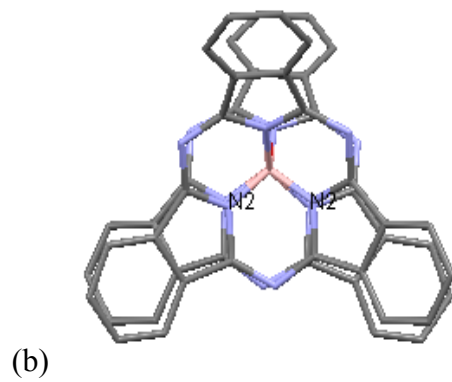
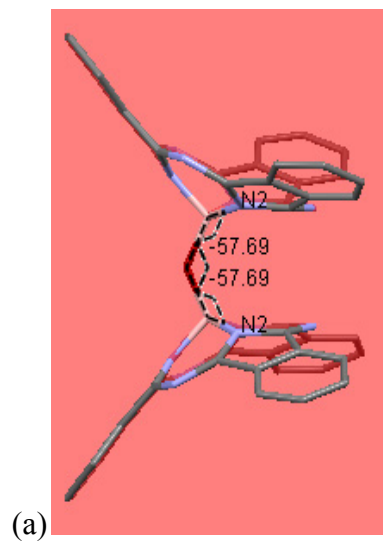
**Figure S1:** Conformation of  $\mu$ -oxo-(BsubPc)<sub>2</sub>•0.25H<sub>2</sub>O showing (a) torsion angles  $\omega_\alpha$  and  $\omega_\beta$ ; (b) a view down the molecular  $\gamma$ -axis.



**Figure S2:** Conformation of  $\mu$ -oxo-(BsubPc)<sub>2</sub>•3toluene (Yamasaki, 2011) showing (a) torsion angles  $\omega_\alpha$  and  $\omega_\beta$ ; (b) a view down the molecular  $y$ -axis.



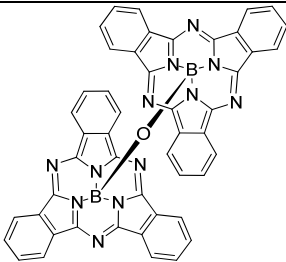
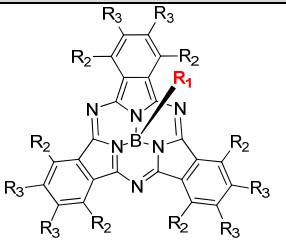
**Figure S3:** Conformation of  $\mu$ -oxo-(BsubPc)<sub>2</sub>•2DCB showing (a) torsion angles  $\omega_\alpha$  and  $\omega_\beta$ ; (b) a view down the molecular  $y$ -axis; (c) perpendicular distance of oxygen from the approximate mirror plane N2-B1...B2-N10 (0.205(4) Å) and dihedral angle between the B1-O-B2 plane and the N2-B1...B2-N10 approximate mirror plane (18.2(4) °).

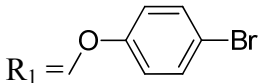
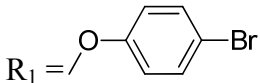


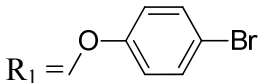
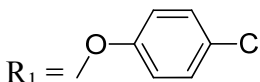
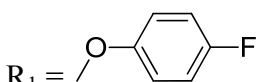
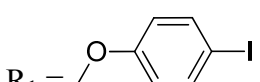
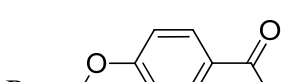


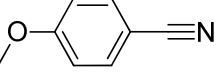
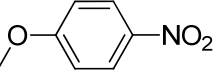
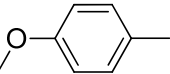
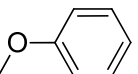
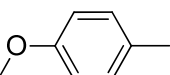
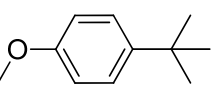
**Figure S4:** Conformation of  $\mu$ -oxo-(BsubPc)<sub>2</sub><sup>subl</sup> showing (a) torsion angles  $\omega_\alpha$  and  $\omega_\beta$ ; (b) a view down the molecular  $y$ -axis; (c) a view down the molecular  $z$ -axis identifying two approximate mirror planes (the  $xz$  molecular plane and the  $yz$  molecular plane).

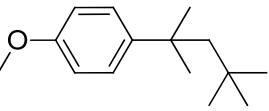
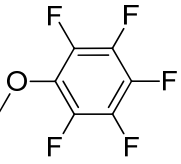
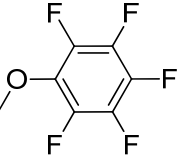
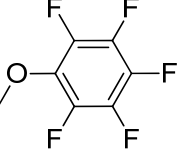
**Table S1.** Density Comparison of BsubPc Crystal Structures.

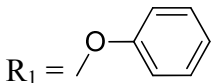
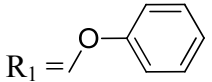
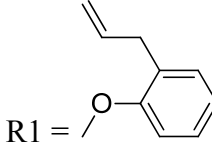
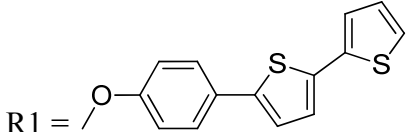
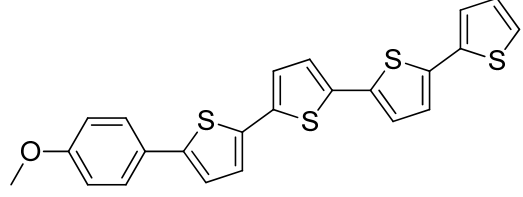
Compound	Reference	Unit Cell Volume, Å <sup>3</sup>	Z	Calculated Density		Calculated Chromophore Density <sup>†</sup>	
				kmol/m <sup>3</sup>	Mg/m <sup>3</sup>	kmol/m <sup>3</sup>	Mg/m <sup>3</sup>
							
$\mu$ -oxo-(BsubPc) <sub>2</sub> •0.25H <sub>2</sub> O	This work	1899.35	2	1.75	1.42	3.50	1.38
$\mu$ -oxo-(BsubPc) <sub>2</sub> •2DCB	This work	2541.89	2	1.31	1.44	2.61	1.03
$\mu$ -oxo-(BsubPc) <sub>2</sub> <sup>subl</sup>	This work	3691.63	4	1.80	1.45	3.60	1.42
$\mu$ -oxo-(BsubPc) <sub>2</sub> •3toluene	CCDC_792081 (Yamasaki & Mori, 2011)	2797.35	2	1.19	1.29	2.37	0.94
							
Halides and related.							

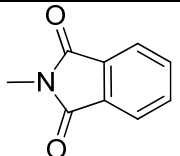
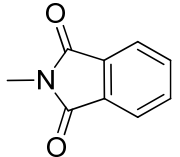
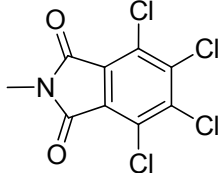
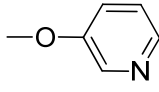
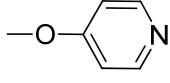
Cl-BsubPc R <sub>1</sub> = Cl R <sub>2</sub> = R <sub>3</sub> = H	PHTLBC (Kietaiabl, 1974)	1865.04	4	3.56	1.53	3.56	1.41
R <sub>1</sub> = OH R <sub>2</sub> = R <sub>3</sub> = H	KAJGIL (Potz <i>et al.</i> , 2000)	3718.69	8	3.57	1.47	3.57	1.41
R <sub>1</sub> = OH R <sub>2</sub> = R <sub>3</sub> = H dihydrate	KAJGOR (Potz <i>et al.</i> , 2000)	1063.69	2	3.12	1.40	3.12	1.23
Br-F12BsubPc R <sub>1</sub> = Br R <sub>2</sub> = R <sub>3</sub> = F	(Morse <i>et al.</i> , 2010b)	2308.33	4	2.88	1.99	2.88	1.14
R <sub>1</sub> = Cl R <sub>2</sub> = R <sub>3</sub> = F	OFUTUE (Fukuda <i>et al.</i> , 2002)	2284.36	4	2.91	1.88	2.91	1.15
R <sub>1</sub> = R <sub>2</sub> = R <sub>3</sub> = F	YIYHUK (Rodríguez-Morgadeet <i>et al.</i> , 2008)	6708.85	12	2.97	1.87	2.97	1.17
Phenoxy and related							
$\alpha$ -4-BrPhO-BsubPc  R <sub>1</sub> =  R <sub>2</sub> = R <sub>3</sub> = H	(Paton <i>et al.</i> , 2011c)	2425.16	4	2.74	1.55	2.74	1.08

$\beta$ -4-BrPhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011c	1184.74	2	2.80	1.59	2.80	1.11
4-ClPhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011c	1166.21	2	2.85	1.49	2.85	1.13
4-FPhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011d	1133.97	2	2.93	1.48	2.93	1.16
4-IPhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011c	1218.85	2	2.72	1.67	2.72	1.08
4-AcPhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Paton <i>et al.</i> , 2010a	1249.54	2	2.66	1.41	2.66	1.05
4-cyanoPhO-BsubPc	Paton <i>et al.</i> , 2011b	5228	8	2.54	1.30	2.54	1.00

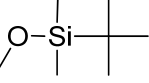
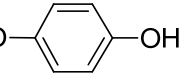
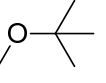
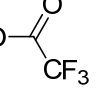
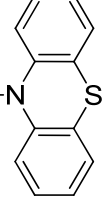
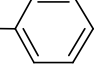
$R_1 = $  $R_2 = R_3 = H$							
4-nitroPhO-BsubPc $R_1 = $  $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011a	2380.82	4	2.79	1.49	2.79	1.10
p-cresol-F12BsubPc $R_1 = $  $R_2 = R_3 = F$	Paton <i>et al.</i> , 2010b	2764.08	4	2.40	1.73	2.40	0.95
PhO-BsubPc $R_1 = $  $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011d	1140.22	2	2.91	1.42	2.91	1.15
p-cresol-BsubPc $R_1 = $  $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011d	1172.88	2	2.83	1.42	2.83	1.12
t-butylPhO-BsubPc $R_1 = $ 	Paton <i>et al.</i> , 2011d	1326.75	2	2.50	1.36	2.50	0.99

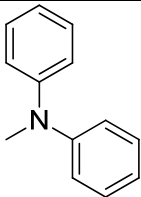
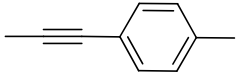
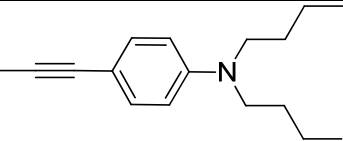
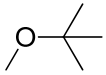


$R_2 = R_3 = H$							
t-octylPhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Paton <i>et al.</i> , 2011d	3288.46	4	2.02	1.29	2.02	0.80
$\alpha$ -F5PhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Morse <i>et al.</i> , 2010a	4786.62	8	2.78	1.60	2.78	1.10
$\beta$ -F5PhO-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Castrucci <i>et al.</i> , 2012	2425.62	4	2.74	1.58	2.74	1.08
F5PhO-F12BsubPc  $R_1 =$ $R_2 = R_3 = F$	Morse <i>et al.</i> , 2010a	2759.29	4	2.41	1.91	2.41	0.95







PhO-F12BsubPc  $R_1 =$ $R_2 = R_3 = F$	Morse <i>et al.</i> , 2010a	2660.01	4	2.50	1.76	2.50	0.99
 $R_1 =$ $R_2 = R_3 = F$	OFUTOY (Claessens & Torres, 2002)	2736.21	4	2.43	1.71	2.43	0.96
 $R_1 =$ $R_2 = R_3 = H$	Mauldin <i>et al.</i> , 2010	8144.51	12	2.45	1.29	2.45	0.97
 $R_1 =$ $R_2 = R_3 = H$	Mauldin <i>et al.</i> , 2010	2949.47	4	2.25	1.47	2.25	0.89
$R_1 =$  $R_2 = R_3 = H$	Mauldin <i>et al.</i> , 2010	1809.29	2	1.84	1.50	1.84	0.73
Phthalimido-BsubPc (sublimation)	Morse <i>et al.</i> , 2011	2490.58	4	2.67	1.44	2.67	1.05

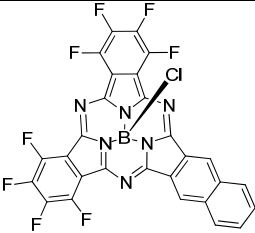
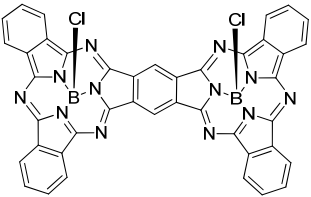
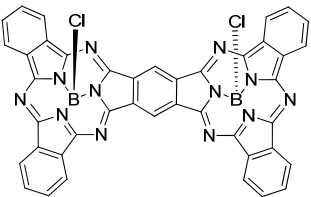
 $R_1 =$ $R_2 = R_3 = H$							
Phthalimido-BsubPc (vapour diffusion)  $R_1 =$ $R_2 = R_3 = H$	Morse <i>et al.</i> , 2011	2910.39	4	2.28	1.51	2.28	0.90
Cl4Phthalimido-BsubPc  $R_1 =$ $R_2 = R_3 = H$	Morse <i>et al.</i> , 2011	2743.27	4	2.42	1.64	2.42	0.96
 $R_1 =$ $R_2 = R_3 = H$	GOHDIR (Xu & Ng, 2008)	1161.97	2	2.86	1.40	2.86	1.13
 $R_1 =$ $R_2 = R_3 = H$	GOHDOX (Xu & Ng, 2008)	2277.7	4	2.92	1.43	2.92	1.15

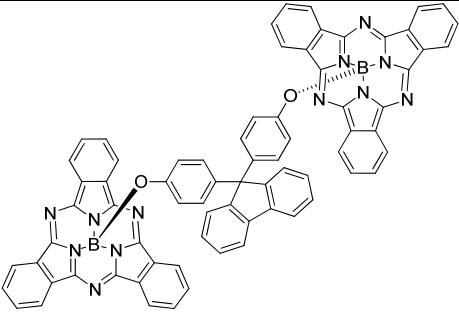
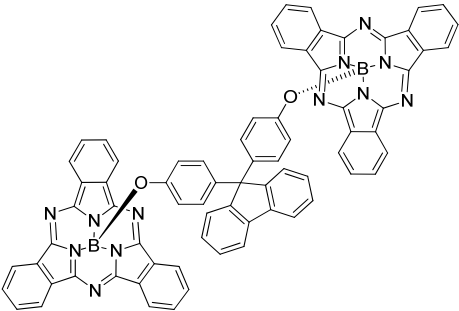


$R_1 =$  $R_2 = R_3 = H$	UCIJUL (Engel <i>et al.</i> , 1997)	2761.23	4	2.41	1.27	2.41	0.95
$R_1 =$  $R_2 = R_3 = H$	Guilleme <i>et al.</i> , 2011	3267.06	4	2.03	1.36	2.03	0.80
$R_1 =$  $R_2 = R_3 = H$	Guilleme <i>et al.</i> , 2011	2268.26	4	2.93	1.37	2.93	1.16
$R_1 =$  $R_2 = R_3 = H$ hydrate	Guilleme <i>et al.</i> , 2011	2206.36	4	3.01	1.58	3.01	1.19
$R_1 =$  $R_2 = R_3 = F$	Guilleme <i>et al.</i> , 2011	6867.51	4	0.97	0.78	0.97	0.38
$R_1 =$  $R_2 = R_3 = H$	Guilleme <i>et al.</i> , 2011	4449.6	8	2.99	1.41	2.99	1.18

 <p>R<sub>1</sub> =</p> <p>R<sub>2</sub> = R<sub>3</sub> = H</p>	Guilleme <i>et al.</i> , 2011	2812.1	4	2.36	1.33	2.36	0.93
 <p>R<sub>1</sub> =</p> <p>R<sub>2</sub> = R<sub>3</sub> = H</p>	CCDC 684062 (Camerel <i>et al.</i> , 2008)	4995.07	8	2.66	1.36	2.66	1.05
 <p>R<sub>1</sub> =</p> <p>R<sub>2</sub> = R<sub>3</sub> = H</p> <p>methanol hydrate</p>	CCDC 691854 (Camerel <i>et al.</i> , 2008)	7868.76	8	1.69	1.16	1.69	0.67
 <p>R<sub>1</sub> =</p> <p>R<sub>2</sub> = R<sub>3</sub> = H</p>	KAJPEQ (Potz <i>et al.</i> , 2000)	2308.93	4	2.88	1.35	2.88	1.14
 <p>R<sub>1</sub> =</p> <p>R<sub>2</sub> = R<sub>3</sub> = H</p> <p>hemihydrate ethanol solvate</p>	KAJQAN (Potz <i>et al.</i> , 2000)	4922.31	8	2.70	1.37	2.70	1.07
 <p>R<sub>1</sub> =</p>	KAJQER (Potz <i>et al.</i> , 2000)	4852.83	8	2.74	1.50	2.74	1.08

R <sub>2</sub> = R <sub>3</sub> = H hydrate pyridine solvate							
R <sub>1</sub> = Cl R <sub>2</sub> = R <sub>3</sub> = H	PHTLBC (Kietaiabl, 1974)	1865.04	4	3.56	1.53	3.56	1.41
R <sub>1</sub> =  R <sub>2</sub> = R <sub>3</sub> = H	RUGJOS (Kasuga <i>et al.</i> , 1996)	2122.4	4	3.13	1.38	3.13	1.24
R <sub>1</sub> =  R <sub>2</sub> = R <sub>3</sub> = H	UCIJOF (Engel <i>et al.</i> , 1997)	2078.71	4	3.20	1.36	3.20	1.26
R <sub>1</sub> =  R <sub>2</sub> = R <sub>3</sub> = H	UCIJOF01 (Engel <i>et al.</i> , 1997)	1988	4	3.34	1.42	3.34	1.32
R <sub>1</sub> =  R <sub>2</sub> = R <sub>3</sub> = H	YOKMUG (Rauschnabel & Hanack, 1995)	4569.83	8	2.91	1.37	2.91	1.15
R <sub>1</sub> =  R <sub>2</sub> = R <sub>3</sub> = H	YOKMUG01 (Potz <i>et al.</i> , 2000)	4529.68	8	2.93	1.39	2.93	1.16
R <sub>1</sub> =  R <sub>2</sub> = R <sub>3</sub> = H	YOKMUG02 (Engel <i>et al.</i> , 1997)	4599.16	8	2.89	1.36	2.89	1.14
Misc.							

 <p>1-methylnaphthalene solvate</p>	XAQQEB (Stork <i>et al.</i> , 1999)	3999.86	4	1.66	1.51	1.66	0.66
<b>Dimers</b>							
<p>(cis)</p>  <p>• dichloromethane solvate</p>	OFUVAM (Fukuda <i>et al.</i> , 2002)	4562.39	4	1.46	1.81	2.91	1.15
<p>(trans)</p>  <p>• dichloromethane solvate</p>	OFUVEQ (Fukuda <i>et al.</i> , 2002)	2360.81	2	1.41	1.81	2.81	1.11
BPF-BsubPc	k08355 (Morse <i>et al.</i> , 2010c)	6943.16	4	0.96	1.39	1.91	0.76

 <p>• cyclohexane &amp; chlorobenzene solvate</p>							
 <p>• cyclohexane &amp; toluene solvate</p>	k08366 (Morse <i>et al.</i> , 2010c)	6941.34	4	0.96	1.35	1.91	0.76

† Density of the BsubPc bowl-shaped unit (without the R<sub>1</sub> substituent, and where R<sub>2</sub> = R<sub>3</sub> = H).

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