*Supplementary Material for the article entitled*

# Synthesis of an octasubstituted monohydroxylated phthalocyanine designed to investigate the effect of the presence of active moieties

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Vefa Ahsen,[a] Fabienne Dumoulin[a]*

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## Content

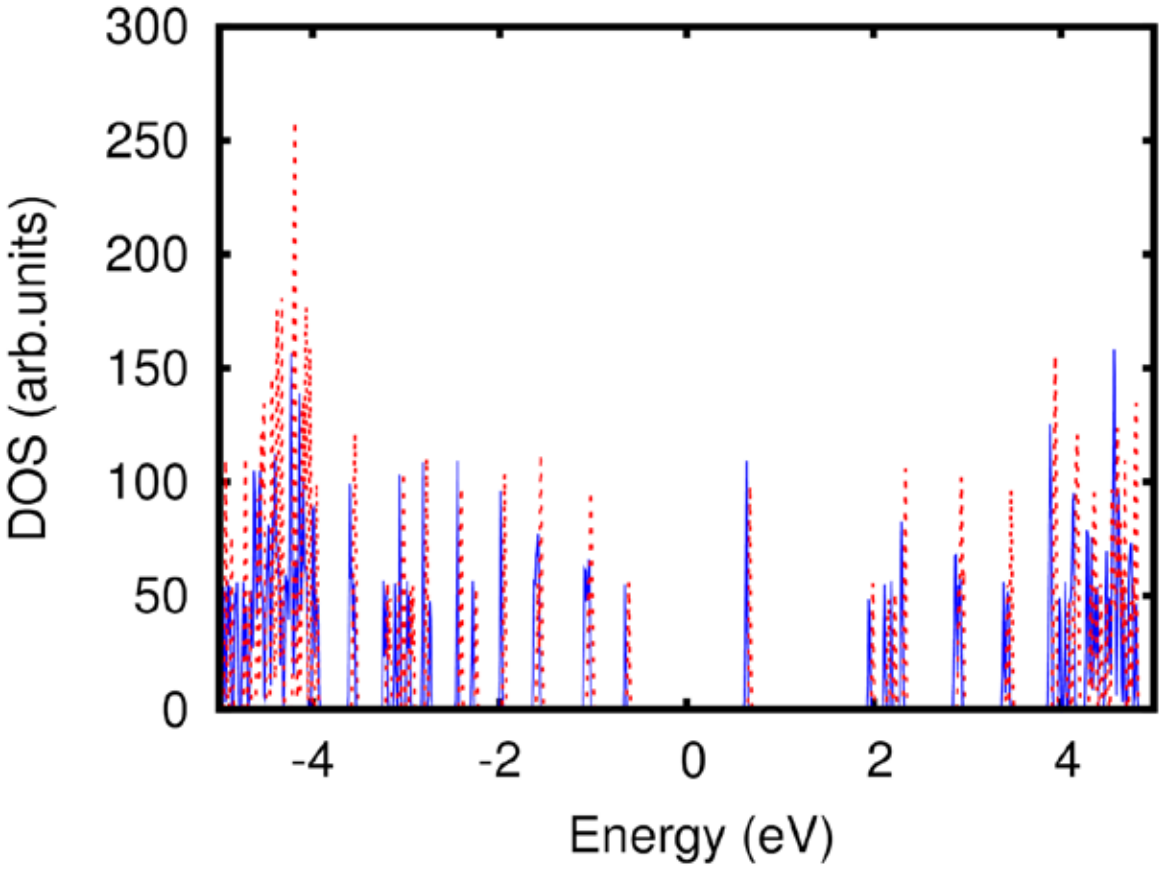


**Fig. S1** Comparison of the electronic density of states (DOS) for phthalocyanines **1** and **2**. Solid (blue) lines are used for **1** and dashed (red) lines for **2**. The energy levels are given with respect to the Fermi level of each molecule.

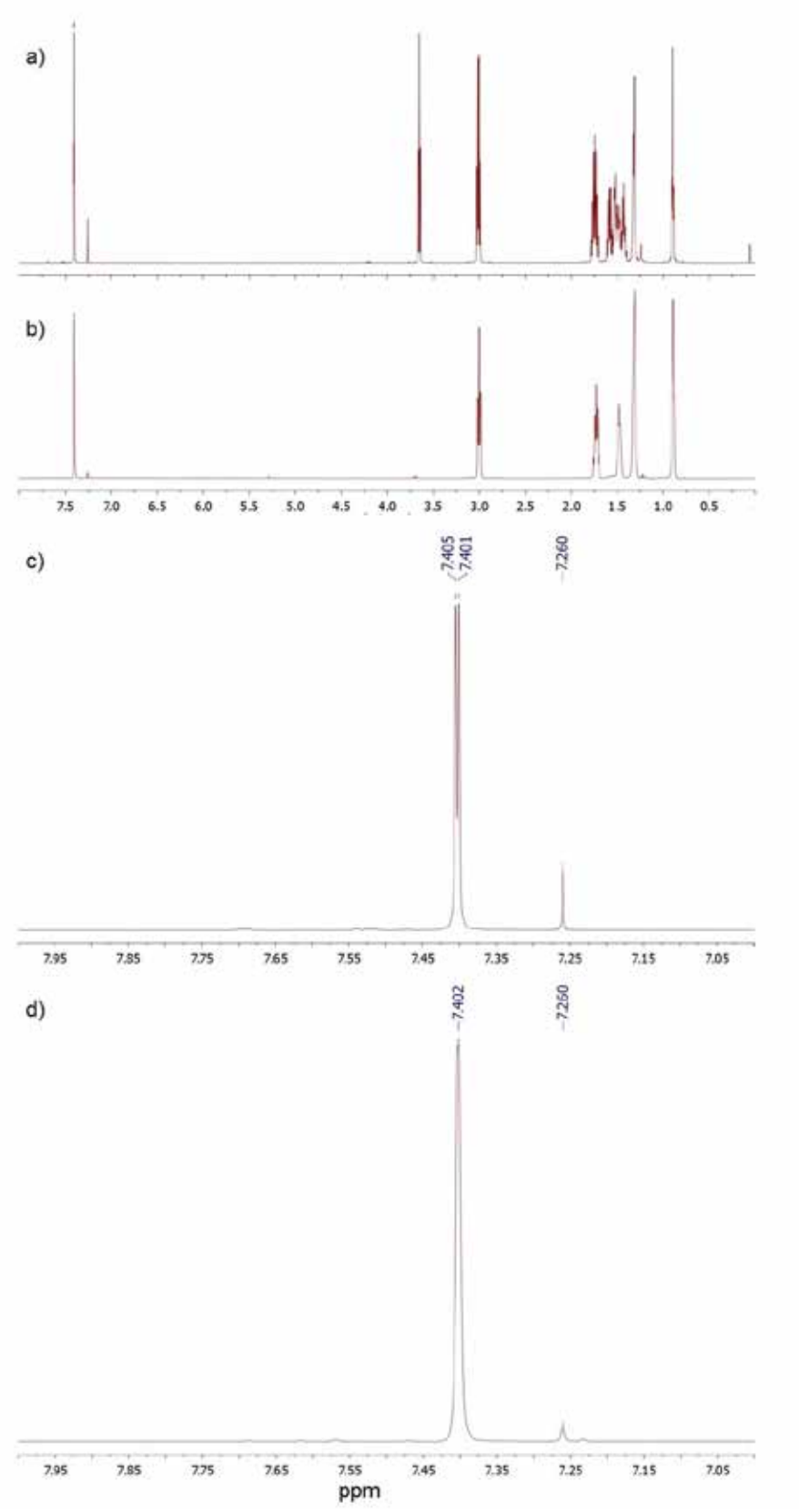


**Fig. S2** Full $^{1}$H NMR spectra of phthalonitriles **3** (a) and **4** (b) in $CDCl_3$. Magnification between 8 and 7 ppm respectively for spectra of phthalonitriles **3** (c) and **4** (d).

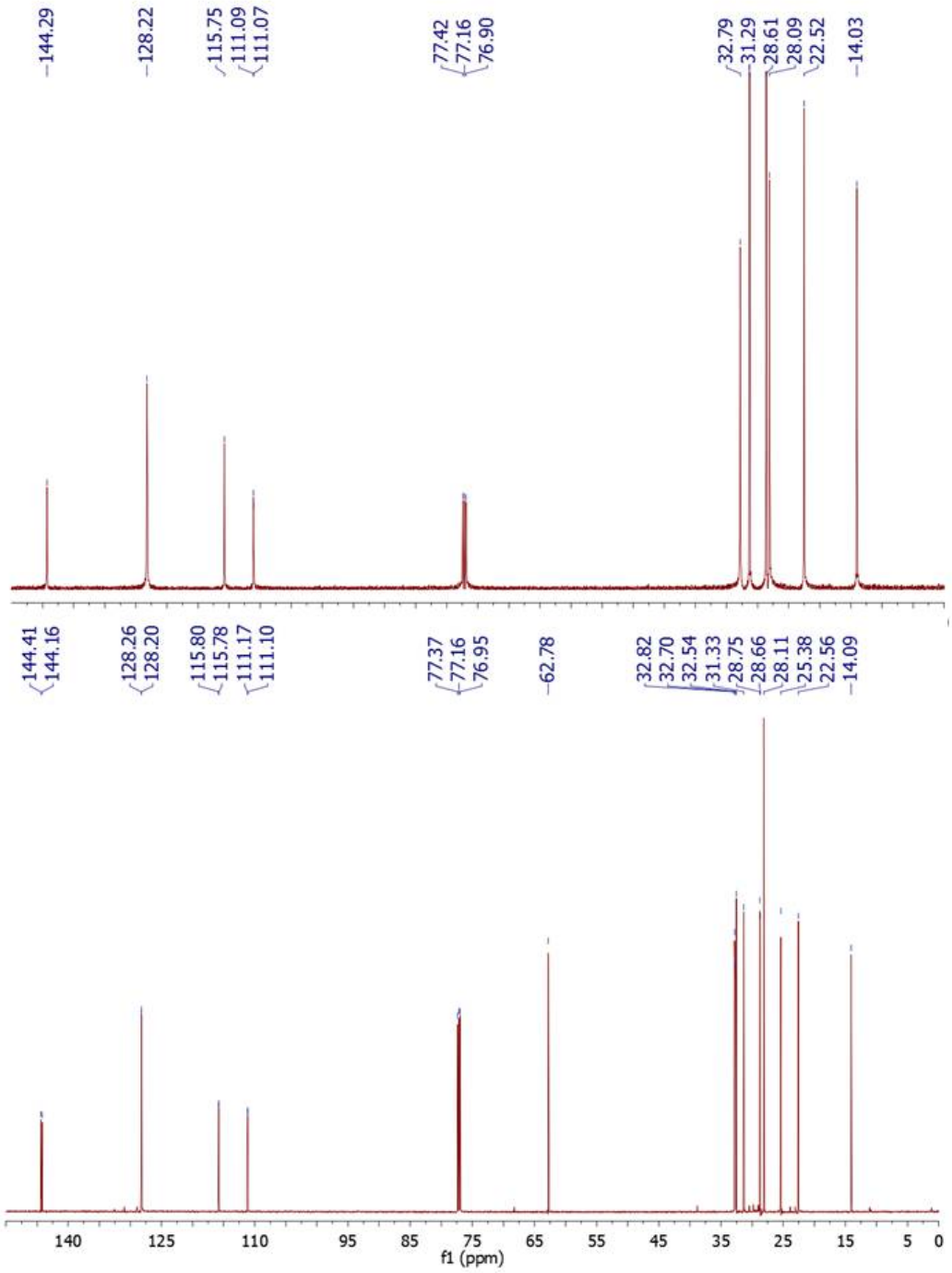


**Fig. S3** Full $^{13}C$ NMR spectra of phthalonitriles **3** (bottom) and **4** (top) in $CDCl_3$.

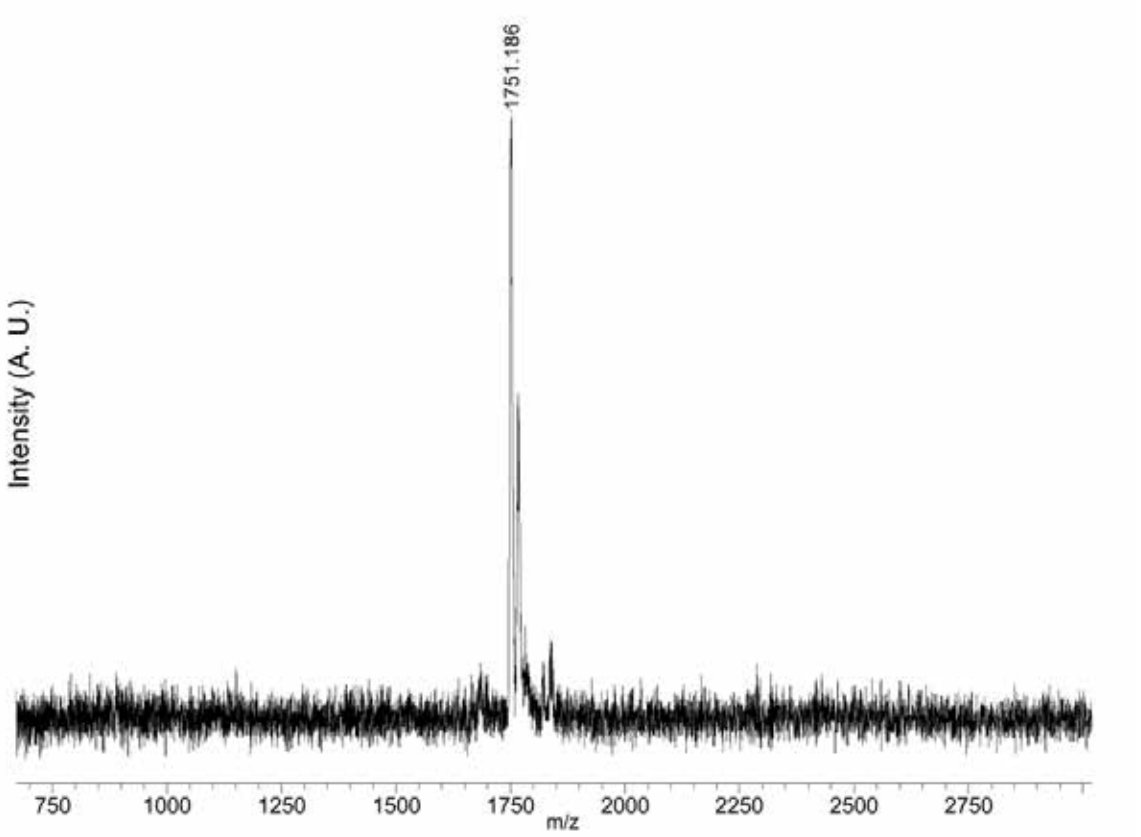


**Fig. S4** MALDI spectrum of phthalocyanine-biotin conjugate **5**. $[M+H]^+$ and $[M+Na]^+$ are observed. Matrix: 2,5-dihydroxybenzoic acid.

**Table S1.** Crystal data and refinement parameters for phthalonitrile **3**

| | |
|---|---|
| **CCDC** | 968416 |
| **Empirical Formula** | $C_{20}H_{28}N_2O_4S_2$ |
| **Formula weight (g. mol$^{-1}$)** | 376.56 |
| **Temperature (K)** | 150(2) |
| **Wavelength (Å)** | 0.71073 |
| **Crystal system** | triclinic |
| **Space group** | P -1 |
| ***a* (Å)** | 8.9476(8) |
| ***b* (Å)** | 10.2758(9) |
| ***c* (Å)** | 12.1482(11) |
| **α(°)** | 98.043(4) |
| **β(°)** | 95.114(4) |
| **γ(°)** | 110.648(4) |
| **Crystal size (mm)** | 0.050 x 0.160 x 0.540 |
| ***V* (Å$^3$)** | 1023.30(16) |
| ***Z*** | 2 |
| **$\rho_{calcd}$ (g. cm$^{-3}$)** | 1.222 |
| **μ (mm$^{-1}$)** | 0.270 |
| ***F*(000)** | 404 |
| **θ range for data collection (°)** | 1.71 - 25.00 |
| ***h*/*k*/*l*** | -10/10, -12/12, -14/14 |
| **Reflections collected** | 16083 |
| **Independent reflections** | 3573 [R(int) = 0.0564] |
| **Data/restraints/parameters** | 3573 / 0 / 228 |
| **Goodness-of-fit on $F^2$** | 1.059 |
| **Final *R* indices [$I > 2\sigma(I)$]** | $R_1$= 0.0512, $wR_2$= 0.1429 |
| ***R* indices (all data)** | $R_1$= 0.0557, $wR_2$= 0.1467 |
| **Largest diff. peak and hole (e.Å$^{-3}$)** | 1.379 and -0.421 |

# checkCIF/PLATON report

You have not supplied any structure factors. As a result the full set of tests cannot be run.

THIS REPORT IS FOR GUIDANCE ONLY. IF USED AS PART OF A REVIEW PROCEDURE FOR PUBLICATION, IT SHOULD NOT REPLACE THE EXPERTISE OF AN EXPERIENCED CRYSTALLOGRAPHIC REFEREE.

No syntax errors found. CIF dictionary Interpreting this report

# Datablock: I

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```
Bond precision:  C-C = 0.0039 A          Wavelength=0.71073

Cell:          a=8.9476(8)       b=10.2758(9)     c=12.1482(11)
               alpha=98.043(4)   beta=95.114(4)   gamma=110.648(4)
Temperature:   150 K
```

| | Calculated | Reported |
|---|---|---|
| Volume | 1023.30(16) | 1023.30(16) |
| Space group | P -1 | P -1 |
| Hall group | -P 1 | -P 1 |
| Moiety formula | C20 H28 N2 O S2 | C20 H28 N2 O S2 |
| Sum formula | C20 H28 N2 O S2 | C20 H28 N2 O S2 |
| Mr | 376.56 | 376.56 |
| Dx,g cm-3 | 1.222 | 1.222 |
| Z | 2 | 2 |
| Mu (mm-1) | 0.270 | 0.270 |
| F000 | 404.0 | 404.0 |
| F000’ | 404.62 | |
| h,k,lmax | 10,12,14 | 10,12,14 |
| Nref | 3608 | 3573 |
| Tmin,Tmax | 0.949,0.987 | 0.690,0.990 |
| Tmin’ | 0.864 | |

```
Correction method= MULTI-SCAN

Data completeness= 0.990          Theta(max)= 25.000

R(reflections)= 0.0512( 3242)     wR2(reflections)= 0.1467( 3573)

S = 1.059                 Npar= 228
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The following ALERTS were generated. Each ALERT has the format
**test-name_ALERT_alert-type_alert-level**.
Click on the hyperlinks for more details of the test.

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## Alert level C

```
DIFMX01_ALERT_2_C  The maximum difference density is > 0.1*ZMAX*0.75
           _refine_diff_density_max given =      1.379
           Test value =      1.200
DIFMX02_ALERT_1_C  The maximum difference density is > 0.1*ZMAX*0.75
           The relevant atom site should be identified.
PLAT094_ALERT_2_C Ratio of Maximum / Minimum Residual Density ....       3.28 Report
PLAT097_ALERT_2_C Large Reported Max.  (Positive) Residual Density       1.38 eA-3
```

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## Alert level G

```
PLAT005_ALERT_5_G No _iucr_refine_instructions_details  in the CIF     Please Do !
PLAT007_ALERT_5_G Number of Unrefined Donor-H Atoms ..............          1 Report
PLAT154_ALERT_1_G The su's on the Cell Angles are Equal ..........    0.00400 Degree
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #          1 Do !
           N1  -C1  -C3  -C8   -104.00  9.00   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #          2 Do !
           N1  -C1  -C3  -C4     74.00  9.00   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #          7 Do !
           N2  -C2  -C4  -C5    -28.00 12.00   1.555   1.555   1.555   1.555
PLAT710_ALERT_4_G Delete 1-2-3 or 2-3-4 Linear Torsion Angle ... #          8 Do !
           N2  -C2  -C4  -C3    151.00 12.00   1.555   1.555   1.555   1.555
```

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0 **ALERT level A** = Most likely a serious problem - resolve or explain
0 **ALERT level B** = A potentially serious problem, consider carefully
4 **ALERT level C** = Check. Ensure it is not caused by an omission or oversight
7 **ALERT level G** = General information/check it is not something unexpected

2 ALERT type 1 CIF construction/syntax error, inconsistent or missing data
3 ALERT type 2 Indicator that the structure model may be wrong or deficient
0 ALERT type 3 Indicator that the structure quality may be low
4 ALERT type 4 Improvement, methodology, query or suggestion
2 ALERT type 5 Informative message, check

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It is advisable to attempt to resolve as many as possible of the alerts in all categories. Often the minor alerts point to easily fixed oversights, errors and omissions in your CIF or refinement strategy, so attention to these fine details can be worthwhile. In order to resolve some of the more serious problems it may be necessary to carry out additional measurements or structure refinements. However, the purpose of your study may justify the reported deviations and the more serious of these should normally be commented upon in the discussion or experimental section of a paper or in the "special_details" fields of the CIF. checkCIF was carefully designed to identify outliers and unusual parameters, but every test has its limitations and alerts that are not important in a particular case may appear. Conversely, the absence of alerts does not guarantee there are no aspects of the results needing attention. It is up to the individual to critically assess their own results and, if necessary, seek expert advice.

**Publication of your CIF in IUCr journals**

A basic structural check has been run on your CIF. These basic checks will be run on all CIFs submitted for publication in IUCr journals (*Acta Crystallographica, Journal of Applied Crystallography, Journal of Synchrotron Radiation*); however, if you intend to submit to *Acta Crystallographica Section C* or *E*, you should make sure that full publication checks are run on the final version of your CIF prior to submission.

**Publication of your CIF in other journals**

Please refer to the *Notes for Authors* of the relevant journal for any special instructions relating to CIF submission.

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**PLATON version of 20/08/2014; check.def file version of 18/08/2014**

**Datablock I** - ellipsoid plot