

## Supporting Information

# Cyclohexylcyanine: A new aliphatic phthalocyanine analogue

Roshinee Costa and Christopher J. Ziegler<sup>b</sup>

Department of Chemistry, University of Akron, Akron OH 44325-3601 e-mail:  
ziegler@uakron.edu

## Experimental Section

**General Considerations:** All reagents were purchased from Strem, Acros Organics or Sigma-Aldrich and used as received. Solution NMR spectroscopy was performed on Varian VXR 300 MHz and Varian 500MHz NMR instruments. Elemental analyses were carried out at the School of Chemical Sciences Microanalytical Laboratory at the University of Illinois at Urbana-Champaign. Mass Spectrometric analyses were carried out at Mass Spectrometry and Proteomics Facility at the Ohio State University in Columbus OH or The University of Akron.

**X-ray Crystallography.** X-ray intensity data were measured at 100 K (Bruker KYRO-FLEX) on a Bruker SMART APEX CCD-based X-ray diffractometer system equipped with a Mo-target X-ray tube ( $\lambda = 0.71073 \text{ \AA}$ ) operated at 2000 W power. The crystals were mounted on a cryoloop using Paratone N-Exxon oil and placed under a stream of nitrogen at 100 K. The detector was placed at a distance of 5.009 cm from the crystals.

The data were corrected for absorption with the SADABS program. The structures were refined using the Bruker SHELXTL Software Package (Version 6.1), and were solved using direct methods until the final anisotropic full-matrix, least squares refinement of  $F^2$  converged.<sup>1</sup> Diiminoisoindoline was synthesized as previously described in the literature.<sup>2</sup>

**Synthesis of cyclohexylecyanine (1):** 1.45g (9.9 mmol) of 1,3-diiminoisoindoline and 1.21 mL (9.9 mmol) 1, 3- cyclohexanediamine were added to 15 mL of warm butanol and the resultant solution was reflux for 20 h. Upon completion of the reaction, the light green product was collected by filtration. Then product was then washed with hot butanol followed by diethyl ether. Yield 2.8 g (53%). High Res. ESI MS (positive ion): 451.2596 M/z (M+H, calcd 451.2610, 3.1 ppm). CHN anal. calcd for  $C_{28}N_6H_{30} \cdot 2H_2O$  ( $C_{28}H_{34}N_6O_2$ ): C,69.11; H, 7.04; N, 17.27. Found: C, 70.16; H, 7.31; N, 17.06.  $^1H$  NMR ([D6]DMSO,  $\delta$ , temp = 130 °C):  $\delta$ = 0.92 (m, 2H), 1.60 (td,  $J_1 = 9.4$  Hz,  $J_2 = 11$  Hz, 2H), 1.99 (t,  $J = 9.6$  Hz, 2H), 4.26 (br, 1H), 7.40 (dd,  $J = 5.2$  Hz, 1H), 7.69 (br, 1H) ppm.  $^{13}C$  NMR ([D6]DMSO,  $\delta$ ): 13.78, 18.50, 23.27, 30.28, 33.88, 34.62, 41.28, 50.94, 51.76, 57.33, 58.19, 60.01, 120.13, 120.59, 129.05, 130.03, 135.14, 139.81, 164.66, 166.13 ppm. Figure S1 shows the variable temperature  $^1H$  NMR spectra for the aromatic region, and Figure S2 shows the high temperature COSY spectrum used for peak assignment.

**Synthesis of  $Ag(1)_2 \cdot Ag_2(1)_2py_2$ :** 50 mg of 1 (0.11 mmol) and 18 mg of  $AgNO_3$  (0.11 mmol) were refluxed in 4 mL of pyridine for 6 hr in the dark. After completion of the reaction, a light yellow product was collected, which was then recrystallized from DMF/methanol/pyridine to get crystals for suitable for X-ray crystallography. Yield

0.032 g (44%). ESI MS (positive ion): 559.19 M/z (M+H, calcd for  $\text{AgC}_{28}\text{H}_{34}\text{N}_6\text{O}_2 + \text{H}$   
559.46 g/mol). CHN anal. calcd for  $\text{C}_{84}\text{N}_{21}\text{H}_{90}\text{A}_3\text{O}_9$ : C, 54.20; H, 4.87; N, 15.80. Found:  
C, 54.30; H, 4.98; N, 15.49.  $^1\text{H}$  NMR ( $[\text{D}_6]\text{DMSO}$ ,  $\delta$ ): 0.92, 1.61, 2.01, 4.17, 4.73, 7.38,  
7.47, 7.78, 7.90, 8.21, 8.57, 9.30 ppm.

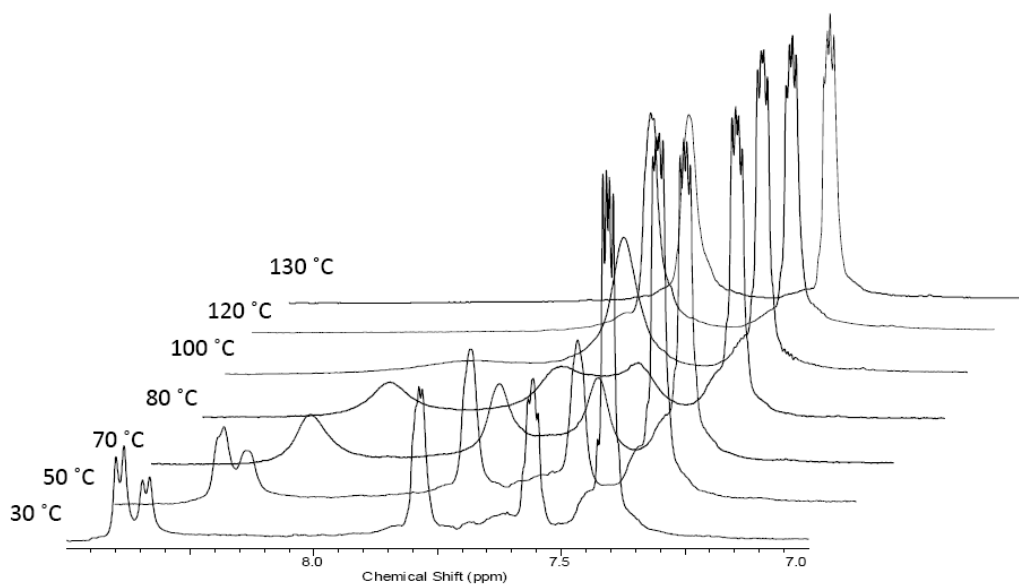


Figure S1: Variable temperature <sup>1</sup>H NMR of **1** in the aromatic region.

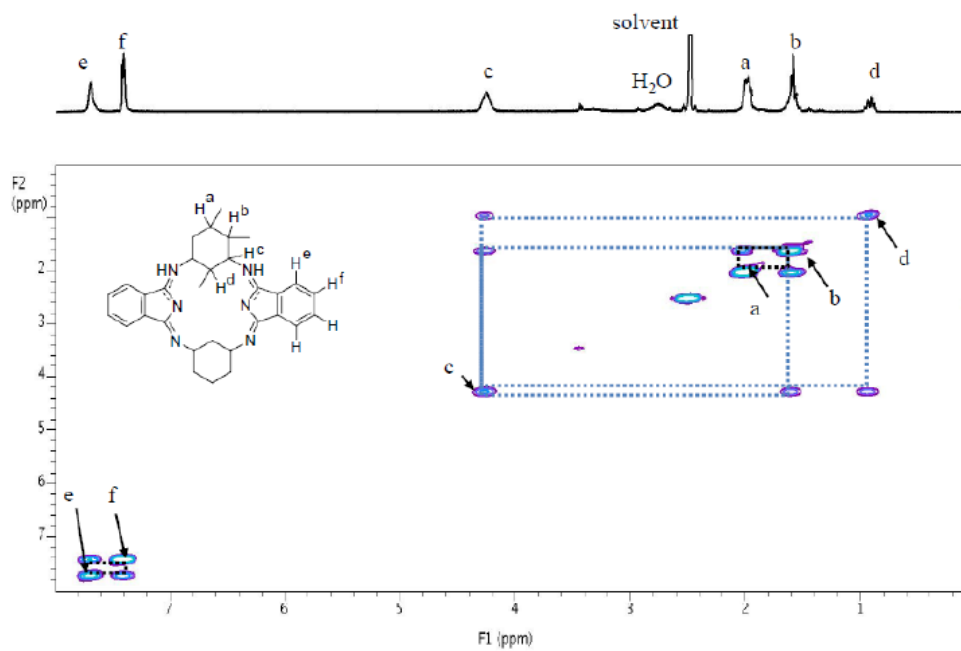


Figure S2:  $^1\text{H}$  COSY NMR spectrum with assignments of **1** at  $130^\circ\text{C}$ .

**References:**

- 1) Sheldrick, G. M. SHELXTL, Crystallographic Software Package, Version 6.10,  
Bruker-AXS: Madison, WI, 2000.
- 2) C. C. Leznoff, P. I. Svirskaya, B. Khouw, R. L. Cerny, P. Seymour, A. B. P. Lever, *J. Org. Chem.*, 1991, **56**, 82.