

## Supporting Information for

### **Molecular design of novel non-planar heteropolycyclic fluorophores with bulky substituents: convenient synthesis and solid-state fluorescence characterization**

**Yousuke Ooyama, Shintaro Yoshikawa, Shigeru Watanabe and Katsuhira Yoshida\***

*Department of Material Science, Faculty of Science, Kochi University, Akebono-cho, Kochi 780-8520, Japan*

*E-mail: kyoshida@cc.kochi-u.ac.jp; Fax: +81-88-844-8359; Tel: +81-88-844-8296*

#### **Experimental Section:**

Melting points were measured with a Yanaco micro melting point apparatus MP-500D. IR spectra were recorded on a JASCO FT/IR-5300 spectrophotometer for samples in KBr pellet form. Absorption spectra were observed with a JASCO U-best30 spectrophotometer for solution and for a HITACHI the thin-film and fluorescence spectra were measured with a JASCO FP-777 spectrophotometer for both solution and the thin-film. Single-crystal X-ray diffraction was performed on Rigaku AFC7S diffractometer. Photoirradiation was carried out by using a UVP Model UVGL-25 as the light sources. The fluorescence quantum yields ( $\Phi$ ) were determined using 9,10-bisphenylethynylanthracene ( $\Phi = 0.84$ ,  $\lambda_{\text{ex}} = 440 \text{ nm}$ )<sup>1</sup> in benzene as the standard. The solid-fluorescence quantum yields ( $\Phi$ ) were determined by using a calibrated integrating sphere system ( $\lambda_{\text{ex}} = 325 \text{ nm}$ ). Elemental analyses were recorded on a Perkin Elmer 2400 II CHN analyzer. <sup>1</sup>H NMR spectra were recorded on a JNM-LA-400 (400 MHz) FT NMR spectrometer with tetramethylsilane (TMS) as an internal standard. Column chromatography was performed on silica gel (60N, spherical, neutral).

#### **Synthesis:**

**11a-Butyl-3-dibutylamino-6-phenyl-11aH-benzo[b]naphtho[2,3-d]furan-11-one 2a:** To a THF solution (200 ml) of benzofuranoquinol **1** (1.0 g, 2.20 mmol) in Ar atmosphere was added an ethereal solution of 1.6 M butyllithium (5.5 ml, 8.8 mmol) at  $-108 \text{ }^\circ\text{C}$  over 30 min. After stirring for 30 min at room temperature, the reaction was quenched with saturated  $\text{NH}_4\text{Cl}$  solution. The solvent was evaporated and the residue was extracted with  $\text{CH}_2\text{Cl}_2$ . The organic extract was washed with water. The organic extract was evaporated and the residue was chromatographed on silica gel ( $\text{CH}_2\text{Cl}_2$  as eluent) to give **2a** (0.75 g, yield 69%): mp  $141\text{-}143 \text{ }^\circ\text{C}$ ; IR (KBr)/ $\text{cm}^{-1}$  1692; <sup>1</sup>HNMR(acetone- $d_6$ )  $\delta = 0.77$  (t, 3H), 0.93 (t, 6H), 1.15-1.20 (m, 2H), 1.33-1.44 (m, 6H), 1.53-1.60 (m, 4H), 1.83-1.90 (m, 2H), 3.32 (m, 4H),

6.29 (d, 1H,  $J = 2.44$  Hz), 6.44 (dd, 1H,  $J = 2.44$  and 8.54 Hz), 7.08 (d, 1H,  $J = 7.81$  Hz), 7.33-7.37 (m, 1H), 7.42-7.44 (m, 5H), 7.49-7.57 (m, 2H), 7.90 (dd, 1H,  $J = 1.46$  and 7.56 Hz); elemental analysis calcd. For  $C_{34}H_{39}NO_2$ : C, 82.72; H, 7.96; N, 2.84; Found: C, 83.02; H, 8.26; N, 2.89.

**3-Dibutylamino-6,11a-diphenyl-11aH-benzo[b]naphtho[2,3-d]furan-11-one 2b:** To a THF solution (100ml) of benzofuranoquinol **1** (1.0 g, 2.20 mmol) in Ar atmosphere was added an ethereal solution of 1.8 M phenyllithium (4.9 ml, 8.8 mmol) at  $-108$  °C over 30 min. After stirring for 30 min at room temperature, the reaction was quenched with saturated  $NH_4Cl$  solution. The solvent was evaporated and the residue was extracted with  $CH_2Cl_2$ . The organic extract was washed with water. The organic extract was evaporated and the residue was chromatographed on silica gel ( $CH_2Cl_2$  as eluent) to give **2b** (0.84 g, yield 608%): mp 159-160 °C; IR (KBr)/ $cm^{-1}$  1693;  $^1H$ NMR(acetone- $d_6$ )  $\delta = 0.91$  (t, 6H), 1.28-1.38 (m, 4H), 1.48-1.56 (m, 4H), 3.22-3.34 (m, 4H), 6.25 (d, 1H,  $J = 2.44$  Hz), 6.41 (1H, dd,  $J = 2.44$  and 8.54 Hz), 7.05 (d, 1H,  $J = 7.56$  Hz), 7.19-7.33 (m, 4H), 7.45-7.51 (m, 2H), 7.55-7.59 (m, 4H), 7.67-7.71 (m, 3H), 7.76 (dd, 1H,  $J = 1.46$  and 7.56 Hz); elemental analysis calcd. For  $C_{36}H_{35}NO_2$ : C, 84.18; H, 6.87; N, 2.73; Found: C, 84.34; H, 6.92; N, 2.60.

**3-Dibutylamino-6-phenyl-11a-thiophen-2-yl-11aH-benzo[b]naphtho[2,3-d]furan-11-one 2c:** To a THF solution (100ml) of benzofuranoquinol **1** (1.0 g, 2.20 mmol) in Ar atmosphere was added an ethereal solution of 1.0 M 2-thienyllithium (8.8 ml, 8.8 mmol) at  $-108$  °C over 30 min. After stirring for 30 min at room temperature, the reaction was quenched with saturated  $NH_4Cl$  solution. The solvent was evaporated and the residue was extracted with  $CH_2Cl_2$ . The organic extract was washed with water. The organic extract was evaporated and the residue was chromatographed on silica gel ( $CH_2Cl_2$  as eluent) to give **2c** (0.77 g, yield 68%): mp 132-134 °C; IR (KBr)/ $cm^{-1}$  1699;  $^1H$ NMR(acetone- $d_6$ )  $\delta = 0.92$  (t, 6H), 1.30-1.39 (m, 4H), 1.51-1.58 (m, 4H), 3.31 (t, 4H), 6.27 (d, 1H,  $J = 2.44$  Hz), 6.46 (dd, 1H,  $J = 2.44$  and 8.54 Hz), 6.86-6.87 (m, 1H), 7.06-7.08 (m, 1H), 7.13-7.14 (m, 1H), 7.25-7.27 (m, 1H), 7.29-7.33 (m, 1H), 7.46-7.58 (m, 6H), 7.71 (d, 1H,  $J = 8.54$  Hz), 7.82 (dd, 1H,  $J = 0.98$  and 7.56 Hz); elemental analysis calcd. For  $C_{34}H_{33}NO_2S$ : C, 78.58; H, 6.40; N, 2.70; Found: C, 78.61; H, 6.39; N, 2.81.

**10-Butyl-7-dibutylamino-9b-phenyl-9bH-indeno[1,2-b]benzo[4,5-e]pyran-11-one 3a:** The dichloromethane solution (80 ml) of **2a** (0.1 g) was irradiated with 365 nm light. The solvent was evaporated and the residue was purified by silica gel column chromatography ( $CH_2Cl_2$  as eluent) and by recrystallization from a mixture solvent of dichloromethane and *n*-hexane to

give 0.099 g of **3a** in 99% yield as orange crystals: mp 120-122 °C; IR (KBr)/cm<sup>-1</sup> 1679; <sup>1</sup>HNMR(acetone-d<sub>6</sub>) δ = 0.95-1.00 (m, 9H), 1.35-1.51 (m, 6H), 1.58-1.65 (m, 4H), 2.04-2.09 (m, 2H), 3.19-3.23 (m, 2H), 3.39-3.43 (m, 4H), 6.37 (dd, 1H, *J* = 2.44 and 8.78 Hz), 6.53 (d, 1H, *J* = 2.44 Hz), 7.11-7.15 (m, 1H), 7.21-7.25 (m, 2H), 7.34 (d, 1H, *J* = 8.78 Hz), 7.48-7.56 (m, 3H), 7.60-7.63 (m, 1H), 7.71-7.77 (m, 2H); elemental analysis calcd. For C<sub>34</sub>H<sub>39</sub>NO<sub>2</sub>: C, 82.72; H, 7.96; N, 2.84; Found: C, 82.43; H, 8.25; N, 2.68.

**7-dibutylamino-9b,10-diphenyl-9bH-indeno[1,2-b]benzo[4,5-e]pyran-11-one 3b:** The dichloromethane solution (80 ml) of **2b** (0.1g) was irradiated with 365 nm light. The solvent was evaporated and the residue was purified by silica gel column chromatography (CH<sub>2</sub>Cl<sub>2</sub> as eluent) and by recrystallization from a mixture solvent of dichloromethane and *n*-hexane to give 0.098 g of **3b** in 98% yield as orange crystals: mp 157-159 °C; IR (KBr)/cm<sup>-1</sup> 1686; <sup>1</sup>HNMR(acetone-d<sub>6</sub>) δ = 0.96 (t, 6H), 1.36-1.45 (m, 4H), 1.59-1.66 (m, 4H), 3.40-3.44 (m, 4H), 6.30 (dd, 1H, *J* = 2.44 and 8.78 Hz), 6.61 (d, 1H, *J* = 2.44 Hz), 6.72 (d, 1H, *J* = 8.78 Hz), 7.16-7.20 (m, 1H), 7.28-7.31 (m, 2H), 7.47-7.50 (m, 5H), 7.62-7.68 (m, 3H), 7.73-7.78 (m, 3H); elemental analysis calcd. For C<sub>36</sub>H<sub>35</sub>NO<sub>2</sub>: C, 84.18; H, 6.87; N, 2.73; Found: C, 84.34; H, 6.92; N, 2.60.

**10-(2-thienyl)-7-dibutylamino-9b-phenyl-9bH-indeno[1,2-b]benzo[4,5-e]pyran-11-one 3c:** The dichloromethane solution (80 ml) of **2c** (0.1g) was irradiated with 365 nm light. The solvent was evaporated and the residue was purified by silica gel column chromatography (CH<sub>2</sub>Cl<sub>2</sub> as eluent) and by recrystallization from a mixture solvent of dichloromethane and *n*-hexane to give 0.099 g of **3c** in 99% yield as orange crystals: mp 129-131 °C; IR (KBr)/cm<sup>-1</sup> 1680; <sup>1</sup>HNMR(acetone-d<sub>6</sub>) δ = 0.98 (t, 6H), 1.36-1.46 (m, 4H), 1.60-1.67 (m, 4H), 3.43 (t, 4H), 6.35 (d, 1H, *J* = 2.68 and 8.78 Hz), 6.60 (d, 1H, *J* = 2.68 Hz), 7.11 (d, 1H, *J* = 9.21 Hz), 7.16-7.19 (m, 1H), 7.24-7.30 (m, 3H), 7.48-7.52 (m, 1H), 7.62-7.72 (m, 6H), 7.75-7.77 (m, 1H); elemental analysis calcd. For C<sub>34</sub>H<sub>33</sub>NO<sub>2</sub>S: C, 78.58; H, 6.40; N, 2.70; Found: C, 78.56; H, 6.49; N, 2.82.

#### X-ray crystal structure analyses:

X-ray crystal structure analyses of **2a-2c** and **3a-3c**: The data sets were collected at 23 ± 1 °C on a Rigaku AFC7S four-circle diffractometer by 2θ-ω scan technique, and using graphite-monochromated Mo-Kα (λ = 0.71069 Å) radiation at 50 kV and 30 mA. In all case, the data were corrected for Lorentz and polarization effects. A correction for secondary extinction was supplied. The reflection intensities were monitored by three standard reflections for every 150 reflections. An empirical absorption correction based on azimuthal

scans of several reflections was applied. All calculations were performed using the teXsan<sup>2</sup> crystallographic software package of Molecular Structure Corporation.

Crystal of **2a** was recrystallized from a mixture solvent of dichloromethane and *n*-hexane as yellow prism, air stable. The one selected had approximate dimensions 0.40×0.40×0.50 mm. The transmission factors ranged from 0.86 to 0.99. The crystal structure was solved by direct methods using SIR 92.<sup>3</sup> The structures were expanded using Fourier techniques.<sup>4</sup> The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were fixed geometrically and not refined.

Crystal data for **2a**: C<sub>34</sub>H<sub>39</sub>NO<sub>2</sub>, *M* = 493.69, monoclinic, *a* = 14.321(2), *b* = 11.583(3), *c* = 17.877(2) Å, β = 104.490(8)°, *U* = 2871.3(9) Å<sup>3</sup>, *T* = 296.2K, space group P2<sub>1</sub>/n (no.14), *Z* = 4, μ(Mo-K<sub>α</sub>) = 0.70 cm<sup>-1</sup>, 5552 reflections measured, 5047 unique (*R*<sub>int</sub> = 0.042) which were used in all calculations. The final *R* indices were *R*1 = 0.074, *wR* (*F*<sup>2</sup>) = 0.183 (all data).

Crystal of guest-free **2b** was recrystallized from a mixture solvent of dichloromethane and *n*-hexane as yellow prism, air stable. The one selected had approximate dimensions 0.70×0.10×0.40 mm. The transmission factors ranged from 0.96 to 1.00. The crystal structure was solved by direct methods using SIR 92.<sup>3</sup> The structures were expanded using Fourier techniques.<sup>4</sup> The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were fixed geometrically and not refined.

Crystal data for **2b**: C<sub>36</sub>H<sub>35</sub>NO<sub>2</sub>, *M* = 513.68, monoclinic, *a* = 9.081(2), *b* = 20.626(4), *c* = 15.600(3) Å, β = 98.69(2)°, *U* = 2888(1) Å<sup>3</sup>, *T* = 296.2K, space group P2<sub>1</sub>/c (no.14), *Z* = 4, μ(Mo-K<sub>α</sub>) = 0.72 cm<sup>-1</sup>, 5427 reflections measured, 5087 unique (*R*<sub>int</sub> = 0.059) which were used in all calculations. The final *R* indices were *R*1 = 0.055, *wR* (*F*<sup>2</sup>) = 0.174 (all data).

Crystal of **2c** was recrystallized from a mixture solvent of dichloromethane and *n*-hexane as yellow prism, air stable. The one selected had approximate dimensions 0.60×0.50×0.10 mm. The transmission factors ranged from 0.87 to 1.00. The crystal structure was solved by direct methods using SIR 92.<sup>3</sup> The structures were expanded using Fourier techniques.<sup>4</sup> The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were fixed geometrically and not refined.

Crystal data for **2c**: C<sub>34</sub>H<sub>33</sub>NO<sub>2</sub>S, *M* = 519.70, triclinic, *a* = 10.088(8), *b* = 16.317(6), *c* = 9.031(6) Å, α = 91.18(4)°, β = 97.28(6)°, γ = 72.73(4)°, *U* = 1407(1) Å<sup>3</sup>, *T* = 296.2K, space group P1- (no.2), *Z* = 2, μ(Mo-K<sub>α</sub>) = 1.46 cm<sup>-1</sup>, 6872 reflections measured, 6463 unique (*R*<sub>int</sub> = 0.084) which were used in all calculations. The final *R* indices were *R*1 = 0.094, *wR* (*F*<sup>2</sup>) = 0.194 (all data).

Crystal of **3a** was recrystallized from a mixture solvent of dichloromethane and *n*-hexane as orange prism, air stable. The one selected had approximate dimensions 0.20×0.20×0.40 mm. The transmission factors ranged from 0.97 to 1.00. The crystal structure was solved by direct methods using SIR 92.<sup>3</sup> The structures were expanded using Fourier techniques.<sup>4</sup> The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were fixed geometrically and not refined.

Crystal data for **3a**: C<sub>34</sub>H<sub>39</sub>NO<sub>2</sub>, *M* = 493.69, monoclinic, *a* = 9.471(5), *b* = 30.489(6), *c* = 10.734(5) Å, β = 114.03(3)°, *U* = 2830(2) Å<sup>3</sup>, *T* = 296.2K, space group P2<sub>1</sub>/a (no.14), *Z* = 4, μ(Mo-K<sub>α</sub>) = 0.71 cm<sup>-1</sup>, 5417 reflections measured, 4981 unique (*R*<sub>int</sub> = 0.050) which were used in all calculations. The final *R* indices were *R*1 = 0.053, *wR* (*F*<sup>2</sup>) = 0.117 (all data).

Crystal of **3b** was recrystallized from a mixture solvent of dichloromethane and *n*-hexane as orange prism, air stable. The one selected had approximate dimensions 0.20×0.10×0.45 mm. The transmission factors ranged from 0.95 to 1.00. The crystal structure was solved by direct methods using SAPI91.<sup>5</sup> The structures were expanded using Fourier techniques.<sup>4</sup> The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were fixed geometrically and not refined.

Crystal data for **3b**: C<sub>36</sub>H<sub>35</sub>NO<sub>2</sub>, *M* = 513.68, orthorhombic, *a* = 20.947(7), *b* = 31.759(7), *c* = 8.758(7) Å, *U* = 5826(4) Å<sup>3</sup>, *T* = 296.2K, space group Pbca (no.61), *Z* = 8, μ(Mo-K<sub>α</sub>) = 0.71 cm<sup>-1</sup>, 5385 reflections measured, 4786 unique (*R*<sub>int</sub> = 0.000) which were used in all calculations. The final *R* indices were *R*1 = 0.055, *wR* (*F*<sup>2</sup>) = 0.108 (all data).

Crystal of **3c** was recrystallized from a mixture solvent of dichloromethane and *n*-hexane as orange prism, air stable. The one selected had approximate dimensions 0.50×0.40×0.40 mm. The transmission factors ranged from 0.87 to 1.00. The crystal structure was solved by direct methods using SIR 92.<sup>3</sup> The structures were expanded using Fourier techniques.<sup>4</sup> The non-hydrogen atoms were refined anisotropically. Some hydrogen atoms were refined isotropically, the rest were fixed geometrically and not refined.

Crystal data for **3c**: C<sub>34</sub>H<sub>33</sub>NO<sub>2</sub>S, *M* = 519.70, triclinic, *a* = 11.009(2), *b* = 13.949(5), *c* = 9.752(2) Å, α = 97.99(2)°, β = 101.46(2)°, γ = 72.82(2)°, *U* = 1397.1(6) Å<sup>3</sup>, *T* = 296.2K, space group P1- (no.2), *Z* = 2, μ(Mo-K<sub>α</sub>) = 1.47 cm<sup>-1</sup>, 6801 reflections measured, 6426 unique (*R*<sub>int</sub> = 0.040) which were used in all calculations. The final *R* indices were *R*1 = 0.0793, *wR* (*F*<sup>2</sup>) = 0.184 (all data).

2. teXsan: Crystal Structure Analysis Package, Molecular Structure Corporation 1985 and 1992.
3. A. Altomare, M. C. Burla, M. Camalli, M. Cascarano, C. Giacovazzo, A. Guagliardi and G. Polidori, *J. Appl. Cryst.*, 1994, **27**, 435.
4. DIRDIF94. P. T. Beurskens, G. Admiraal, G. Beurskens, W. P. Bosman, R. de Gelder, R. Israel, and J. M. M. Smits, The DIRIF94 program system, Technical Report of the Crystallography Laboratory, University of Nijmegen, The Netherlands, 1994.
5. Fan Hai-Fu (1991). Structure Analysis Programs with Intelligent Control, Rigaku Corporation, Tokyo, Japan.

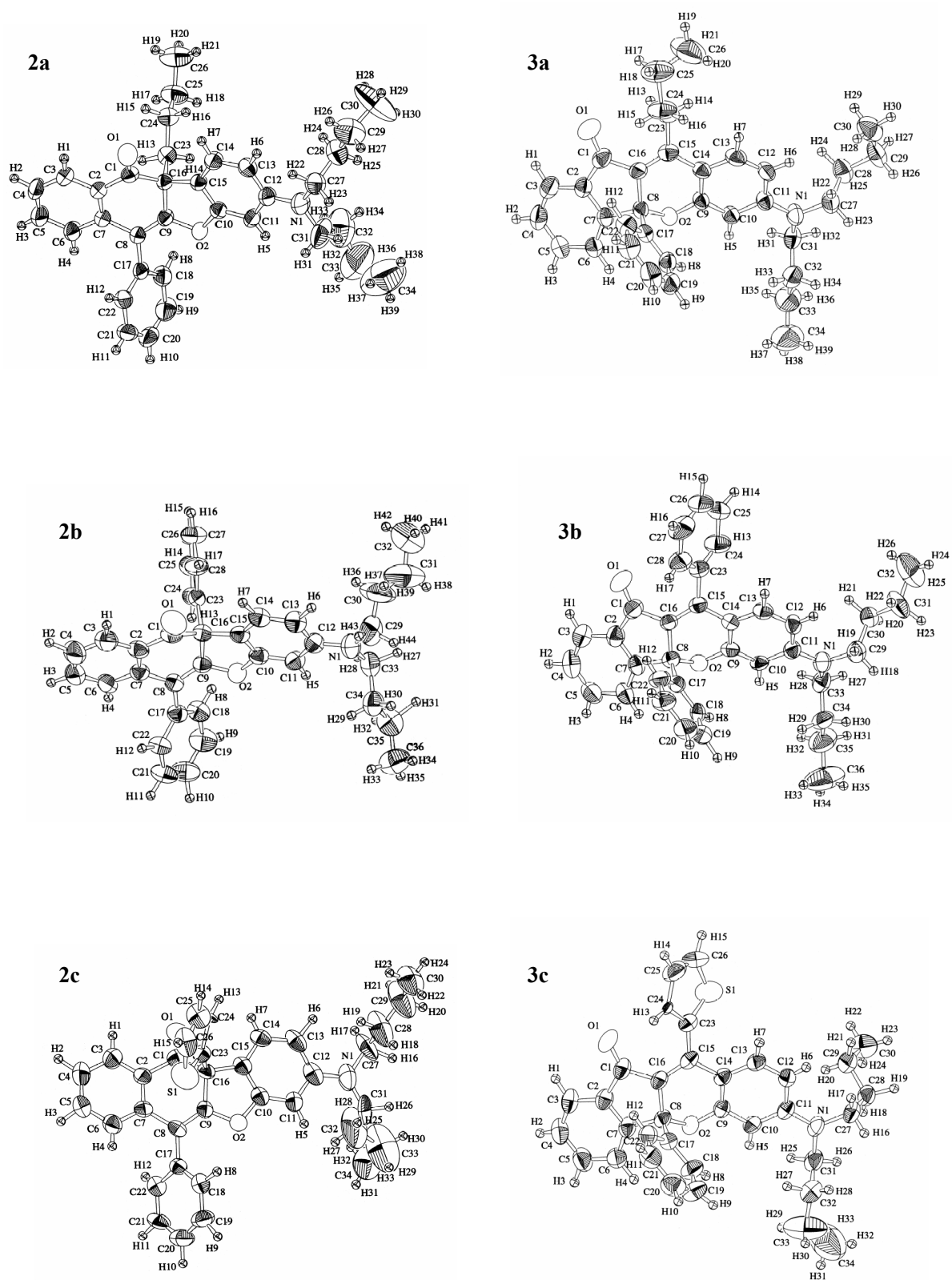


Figure S1 ORTEP diagrams including atom number: the compounds 2a-2c and 3a-3c.

**Table S1** Crystal data and structure refinement parameter for the compounds **2a-2c** and **3a-3c**.

Compound	<b>2a</b>	<b>3a</b>
Molecular formula	C <sub>34</sub> H <sub>39</sub> NO <sub>2</sub>	C <sub>34</sub> H <sub>39</sub> NO <sub>2</sub>
Formula weight	493.69	493.69
Number of reflection used for unit cell determination (2θ range/°)	25 (23.7-27.4)	25 (22.0-24.7)
Crystal System	monoclinic	monoclinic
Space group	P2 <sub>1</sub> /n	P2 <sub>1</sub> /a
a/Å	14.321(2)	9.471(5)
b/Å	11.583(3)	30.489(6)
c/Å	17.877(2)	10.734(5)
α/°		
β/°	104.490(8)	114.03(3)
γ/°		
V/Å <sup>3</sup>	2871.3(9)	2830(2)
Z	4	4
D <sub>c</sub> /g cm <sup>-3</sup>	1.142	1.158
F(000)	1064.00	1064.00
M(MoKα)/cm <sup>-1</sup>	0.70	0.71
Crystal dimensions/nm	0.40×0.40×0.50	0.20×0.20×0.40
Scan mode	ω-2θ	ω-2θ
Scan rate in ω/°min <sup>-1</sup>	8.0( up to 5 scans)	8.0( up to 5 scans)
Scan width/°	1.57 + 0.30 tanθ	0.79 + 0.30 tanθ
2θ max/°	50.0	50.0
Range of indices <i>h</i> ; <i>k</i> ; <i>l</i>	0, 17; 0, 13; -21, 20	-11, 0; 0, 36; -11, 12
Reflections collected (unique)	5047	4981
Reflection observed with I <sub>0</sub> >2σI <sub>0</sub>	2461	2071
Number of parameters	407	459
R	0.074	0.053
R <sub>w</sub>	0.183	0.117
w	(σ <sup>2</sup> F <sup>2</sup> ) <sup>-1</sup>	(σ <sup>2</sup> F <sup>2</sup> ) <sup>-1</sup>
S	1.75	1.10
Max. Shift/Error in final cycle	0.001	0.005
Max. peak in final diff. map/e Å <sup>-3</sup>	0.37	0.21
Min. peak in final diff. map/e Å <sup>-3</sup>	-0.32	-0.22



(Continued)

Compound	2b	3b
Molecular formula	C <sub>36</sub> H <sub>35</sub> NO <sub>2</sub>	C <sub>36</sub> H <sub>35</sub> NO <sub>2</sub>
Formula weight	513.68	513.68
Number of reflection used for unit cell determination (2θ range/°)	25 (22.2-25.5)	12 (22.1-23.9)
Crystal System	monoclinic	orthorhombic
Space group	P2 <sub>1</sub> /c	Pbca
a/Å	9.081(2)	20.947(7)
b/Å	20.626(4)	31.759(7)
c/Å	15.600(3)	8.758(7)
α/°		
β/°	98.69(2)	
γ/°		
V/Å <sup>3</sup>	2888(1)	5826(4)
Z	4	8
D <sub>c</sub> /g cm <sup>-3</sup>	1.181	1.171
F(000)	1096.00	2192.00
M(MoKα)/cm <sup>-1</sup>	0.72	0.71
Crystal dimensions/nm	0.70×0.10×0.40	0.20×0.10×0.45
Scan mode	ω-2θ	ω-2θ
Scan rate in ω/°min <sup>-1</sup>	4.0( up to 5 scans)	8.0( up to 5 scans)
Scan width/°	1.78 + 0.30 tanθ	0.79 + 0.30 tanθ
2θ max/°	50.0	50.0
Range of indices <i>h</i> ; <i>k</i> ; <i>l</i>	-10, 0; 0, 24; ±18	0, 22; 0, 37; -10, 0
Reflections collected (unique)	5087	4786
Reflection observed with I <sub>0</sub> >2σI <sub>0</sub>	1900	1284
Number of parameters	457	465
R	0.055	0.055
R <sub>w</sub>	0.174	0.108
w	(σ <sup>2</sup> F <sup>2</sup> ) <sup>-1</sup>	(σ <sup>2</sup> F <sup>2</sup> ) <sup>-1</sup>
S	1.21	1.00
Max. Shift/Error in final cycle	0.05	0.003
Max. peak in final diff. map/e Å <sup>-3</sup>	0.40	0.83
Min. peak in final diff. map/e Å <sup>-3</sup>	-0.21	-0.87

(Continued)

Compound	2c	3c
Molecular formula	C <sub>34</sub> H <sub>33</sub> NO <sub>2</sub> S	C <sub>34</sub> H <sub>33</sub> NO <sub>2</sub> S
Formula weight	519.70	519.70
Number of reflection used for unit cell determination (2θ range/°)	7 (22.5-24.5)	20 (25.1-34.0)
Crystal System	triclinic	triclinic
Space group	P1-	P1-
a/Å	10.088(8)	11.009(2)
b/Å	16.317(6)	13.949(5)
c/Å	9.031(6)	9.752(2)
α/°	91.18(4)	97.99(2)
β/°	97.28(6)	101.46(2)
γ/°	72.73(4)	72.82(2)
V/Å <sup>3</sup>	1407(1)	1397.1(6)
Z	2	2
D <sub>c</sub> /g cm <sup>-3</sup>	1.226	1.235
F(000)	552.00	552.00
M(MoKα)/cm <sup>-1</sup>	1.46	1.47
Crystal dimensions/nm	0.60×0.50×0.10	0.50×0.40×0.40
Scan mode	ω-2θ	ω-2θ
Scan rate in ω/°min <sup>-1</sup>	8.0( up to 5 scans)	8.0( up to 5 scans)
Scan width/°	1.68 + 0.30 tanθ	1.47 + 0.30 tanθ
2θ max/°	55.0	55.0
Range of indices <i>h</i> ; <i>k</i> ; <i>l</i>	±13; ±21; -11, 0	-14, 14; -18, 17; 0, 12
Reflections collected (unique)	6463	6426
Reflection observed with I <sub>0</sub> >2σI <sub>0</sub>	1656	2675
Number of parameters	392	432
R	0.094	0.079
R <sub>w</sub>	0.194	0.184
w	(σ <sup>2</sup> F <sup>2</sup> ) <sup>-1</sup>	(σ <sup>2</sup> F <sup>2</sup> ) <sup>-1</sup>
S	1.36	1.63
Max. Shift/Error in final cycle	0.03	0.002
Max. peak in final diff. map/e Å <sup>-3</sup>	1.14	0.54
Min. peak in final diff. map/e Å <sup>-3</sup>	-1.02	-0.43