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"Push–pull" 2-ferrocenyl-4-hydroxythiazoles: A novel method of the construction of the thiazole ring

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ABSTRACT

The title compounds were obtained by cyclization of ferrocenyl thioimidates in the presence of sodium ethoxide. They were transformed into the corresponding 4-acetoxy-derivatives in reaction with acetic anhydride/pyridine. The structure of 4-acetoxy-5-(ethoxycarbonyl)-2-ferrocenylthiazole was determined by X-ray diffraction. The EFISH measurements revealed significant second-order nonlinear properties of 4-acetoxy-2-ferrocenyl-5-(4-nitrophenyl)ferrocenylthiazole ($\mu\beta = 250 \times 10^{-48}$ esu at 1907 nm).

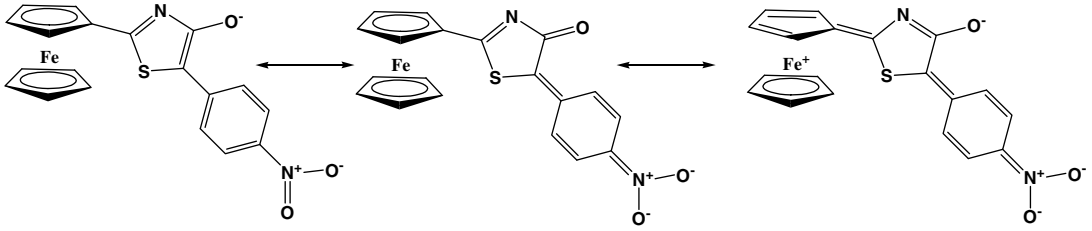
1. Introduction

Ferrocenyl thiazoles have attracted interest as electroactive ligands [1], selective ion-sensing [2] and biologically active compounds [3]. In our opinion they are also promising candidates for nonlinear optical (NLO) chromophores. Indeed, taking into account earlier results obtained for purely organic thiazole compounds [4], "push–pull" ferrocenyl thiazoles are expected to exhibit large second-order NLO properties. Continuing our research program devoted to ferrocene-based NLO chromophores [5] we became interested in synthesis and study of 2-ferrocenyl thiazoles having electron-accepting groups at C-4 or C-5. Here we report on the novel method of the synthesis of the thiazole ring, leading to 2-ferrocenyl-4-hydroxythiazoles with electron-withdrawing substituents at C-5.

2. Results and discussion

In our earlier paper [6] we described the *S*-alkylation of *N*-(ethoxycarbonyl)ferrocenecarbothioamide **1** yielding thioimidates **2a**–**2c** (Scheme 1). Now we have found that these compounds treated with excess of sodium ethoxide in ethanol at r.t. afford, after acidification, the 2-ferrocenyl-4-hydroxythiazoles **3a**–**3c** in >90% isolated yield.

In the case of reaction of **2a** and **2b** intense violet or red coloration of the reaction mixtures before acidification was observed, assignable to the strongly conjugated anions of **3a** and **3b** (e.g. **5**).



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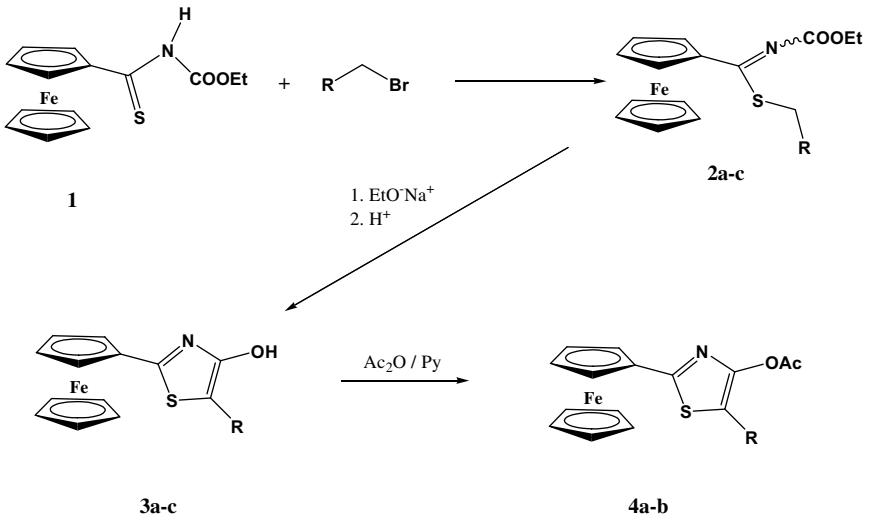


(a) R = C_6H_4-*p*- NO_2
(b) R = C_6H_4-*p*- CN
(c) R = COOEt

Scheme 1. Synthesis of ferrocenylthiazoles by cyclization of thioimidates **2a**–**2c**.

Compounds **3a** and **3b** are red solids insoluble in most organic solvents except pyridine. In their ^{13}C NMR spectra, measured in this solvent, the signals of the thiazolyl C-4 and C-2 appear at 160–165 ppm (in the spectrum of (^{15}N)-**3a** these signals are splitted into doublets with J (^{15}N·^{13}C) = 2–3 Hz. The signals of C-5 appear at ~104 ppm. The ^{15}N NMR spectrum of (^{15}N)-**3a** displays the signal of the thiazolyl nitrogen in the expected region [7], at −88 ppm (relative to external, neat CH_3NO_2).

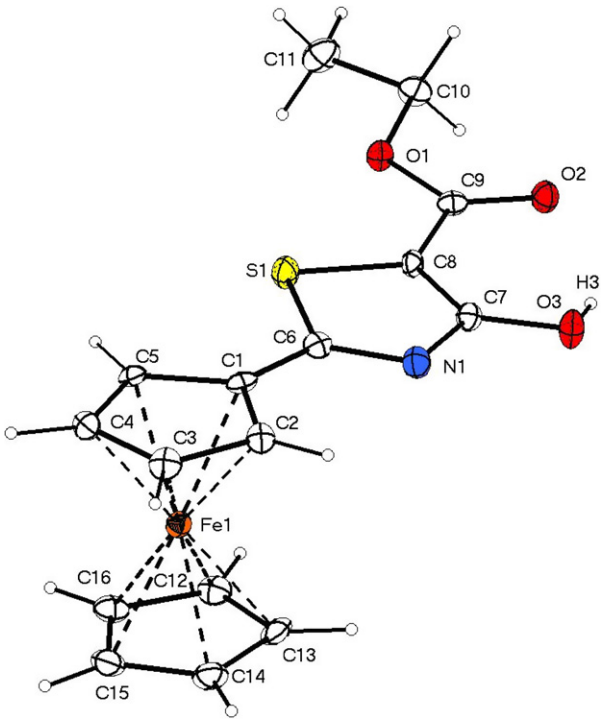


Fig. 1. Molecular view of **3c** with atom labeling scheme. Ellipsoids represent 50% probability.

Table 1
Selected geometrical parameters (Å, °) for **3c**

C1–C2	1.433(7)
C1–C5	1.438(7)
C2–C3	1.410(7)
C3–C4	1.416(7)
C4–C5	1.413(7)
C1–C6	1.445(7)
S1–C6	1.729(5)
S1–C8	1.727(5)
C7–C8	1.375(7)
N1–C7	1.368(6)
N1–C6	1.322(6)
C8–C9	1.439(7)
O1–C9	1.330(6)
O2–C9	1.234(6)
O3–C7	1.350(6)
C2–C1–C6	125.8(4)
C5–C1–C6	127.1(4)
C2–C1–C5	107.0(4)
C1–C6–S1	121.2(4)
N1–C6–C1	123.7(4)
N1–C6–S1	115.1(4)
C8–S1–C6	89.7(2)
C6–N1–C7	109.5(4)
O1–C9–C8	113.4(4)
O2–C9–C8	122.1(4)
O3–C7–N1	116.2(4)
C9–C8–S1	124.9(4)
C7–C8–S1	108.6(4)
S1–C8–C9–O2	177.6(4)
S1–C8–C9–O1	−2.3(6)
C7–C8–C9–O2	3.0(8)
C2–C1–C6–N1	3.0(8)
C2–C1–C6–S1	−176.4(4)

In the solid state molecules **3c** form centrosymmetric dimers with bifurcated intra- and intermolecular hydrogen bonds (Fig. 2 and Table 2).

The treatment of **3a**–**3b** with acetic anhydride in pyridine afforded acetates **4a**–**4b** which were much better soluble in organic solvents like dichloromethane, acetone or THF.

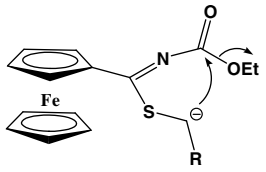
Compound **3c** is soluble in dichloromethane or chloroform. Its structure was confirmed by spectroscopic and analytical data as

well as by single crystal X-ray diffraction (Fig. 1, Table 1). The lengths of the C7–O3 and C7–C8 bonds (1.350(6) Å and 1.375(6) Å, respectively) are in accord with the 4-hydroxythiazole structure and exclude tautomeric structure of Δ^2-thiazolin-4-one. The hydroxythiazole form is the sole existing in solution of **3c** in $CDCl_3$ as indicated by the presence of the signal of the OH proton at ~10 ppm in the ^1H NMR spectrum of this compound. The thiazole ring is practically coplanar with the Cp ring (angle C2–C1–C6–S1 = −176.4(4)° and C5–C1–C6–S1 = 7.6(7)°) and the ester group (angle S1–C8–C9–O2 = 177.6(4)°, C7–C8–C9–O2 = 3.0(8)° and S1–C8–C9–O2 = −2.3(6)°).

In the solid state molecules **3c** form centrosymmetric dimers with bifurcated intra- and intermolecular hydrogen bonds (Fig. 2; Table 2).

In the literature there are only scarce data concerning 4-hydroxythiazoles. These compounds were synthesized mainly by reaction of thioamides with α-bromoesters or α-haloacid halides (Hantzsch synthesis) or α-mercaptoacids with nitriles [8]. Interestingly, some compounds of this class exhibited biological activity (inhibition of 5-lipoxygenase) [8b,8c].

Formation of **3a**–**3c** can be understood assuming that **2a**–**2c** are deprotonated by sodium ethoxide with formation of carboanions **6** stabilized by sulfur [9] and electron-accepting group R. This anion undergoes cyclization *via* nucleophilic attack on the COOEt group. The presence of a strong electron accepting group is necessary as indicated by the failure of cyclization of the compound **2** when R = C_6H_4–COOEt-(*p*). To our knowledge only one example of the similar type of cyclization, involving nucleophilic attack of a sulfur- and carbonyl group-stabilized carbanion on the nitrile group leading to the 4-aminothiazole ring has been reported [10].



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It is of interest to compare our cyclization method with the classical Hantzsch 4-hydroxythiazole synthesis [8,11]. In the latter method (RC(=S)NH_2 reacts with R′–CH(X)–COOEt and the cyclization occurs *via* formation of the C(5)–N bond. In our method RC(=S)NHCOOOEt reacts with R′CH_2X and the cyclization occurs *via* formation of the C(4)–C(5) bond. Therefore, these two methods can be considered as complementary.

Table 2
Hydrogen bonds in dimers of **3c**

D–H–A	Distance (Å)			Angle (°)
	D–H	H–A	D–A	D–H–A
O3–H3–O2	0.80(8)	2.19(8)	2.860(5)	142.(7)
O3–H3–O2′	0.80(8)	2.27(8)	2.874(5)	132.(7)

(i) 2−*x*, −*y*, 1−*z*.

Since ferrocenyl "push–pull" compounds are considered as efficient second-order NLO chromophores [12] we have studied electrochemistry, linear and nonlinear optical properties of **4a**. The data are gathered in Table 3 along with the data for (*E*)-Fc-CH=CH–C_6H_4–NO_2-(*p*) (**7**) taken from the literature [13], respectively.

Cyclic voltammetry of **4a** in CH_2Cl_2 reveals a reversible Fe(II)/Fe(III) ferrocenic couple with the redox potential shifted anodically with respect to that of **7**. This means that replacement of the ethylenic bond in the (*E*)-CH=CH–Ph–NO_2-(*p*) group by the acetoxythiazole nucleus brings about stronger electron withdrawal from the ferrocene moiety. The electronic absorption spectrum confirms the "push-pull" character of **4a**. Two bands observed in the visible region can be assign to π–π^* and MLCT transitions [13,14]. These bands show solvatochromic behavior similar to that reported for **7** (Table 4).

Compound **4a** is thermally stable up to 250 °C (TGA data) and displays slightly better transparency than **7**. Finally, the EFISH (electric field-induced second harmonic generation) measurements at 1907 nm gave the value of $\mu\beta$ higher than that reported for **7** (moreover, it should be noticed that the value reported for the latter compound is stronger dispersion-enhanced because of a shorter incident wavelength).

In conclusion, we have developed an efficient route to "push–pull" ferrocenyl thiazoles *via* unprecedented cyclization of deprotonated ferrocenyl thioimidates. Preliminary experiments confirmed potential of these compounds for the second-order nonlinear optics.

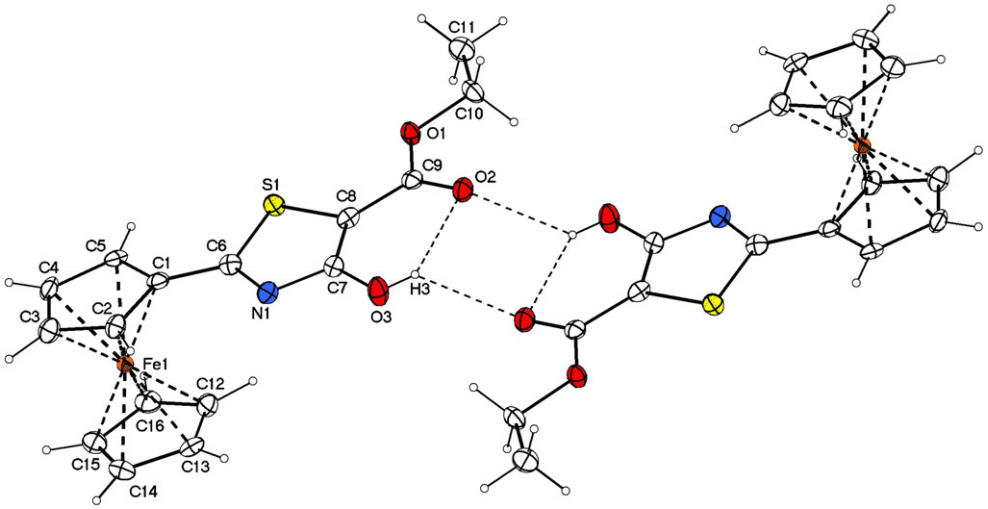

Fig. 2. Centrosymmetric dimers formed by **3c** in the solid state. Ellipsoids represent 50% probability.

Table 3
Electrochemical, linear and nonlinear optical properties of **4a** and **7**

Compound	CV[a]	Electronic transitions[b]		EFISH[c]
	$E_{1/2}$ (mV) (ΔE_p, mV)	($\pi-\pi^*$) λ_{max} (nm) (ε_{max}, M^{-1} cm^{-1})	(MLCT) λ_{max} (nm) (ε_{max}, M^{-1} cm^{-1})	$\mu\beta$ (10^{-48} esu)
4a	200 (90)	354 (54000)	498 (13200)	250
7	55 (100)[d]	363[d]	511[d]	140[e]

[a] In CH_2Cl_2 vs. ferrocene/ferrocenium.
[b] In $CHCl_3$.
[c] In $CHCl_3$ at 1907 nm.
[d] Data taken from [13a] (values of ε_{max} are not given).
[e] In $CHCl_3$ at 1064 nm. Value calculated using values of μ and β from Refs. [13a,13b], respectively.

Table 4
Solvatochromism of **4a** and **7**

Compound	$CHCl_3$	THF	MeOH
	λ_{max} (nm) (ε_{max}, M^{-1} cm^{-1})	λ_{max} (nm) (ε_{max}, $M^{-1}cm^{-1}$)	λ_{max} (nm) (ε_{max}, M^{-1} cm^{-1})
4a	354 (54000)	353 (52000)	351 (52600)
	498 (13200)	483 (13000)	488 (13000)
7	363[a]	360[a]	358[a]
	511[a]	503[a]	502[a]

[a] Data taken from Ref. [13a] (values of ε_{max} are not given).

3. Experimental

All reactions were carried out under argon. The solvents were purified according to standard procedures. Chromatographic separation was performed on Silica gel Merck 60 (230–400 mesh ASTM). Complexes **2a**–**2c** were synthesized as previously described [6]. Labeled (^{15}N)-**3a** was synthesized from (^{15}N)-**1**, obtained using (^{15}N)-ethoxycarbonylisothiocyanate (prepared from KS^{15}N and ClCOOEt) according to the published procedure [15]. NMR spectra were recorded in $CDCl_3$ on VARIAN GEMINI 200BB (200 MHz for ^{1}H) spectrometer and referenced to internal TMS. IR spectra were recorded on a FT-IR NEXUS (Thermo Nicolet) spectrometer.

3.1. Synthesis of *3a–3c*

Solution of **2a**–**2c** (0.25 mmol) in ethanol (5 ml) containing sodium ethoxide (0.5 mmol) was stirred 5 min at r.t. and acidified with diluted aqueous HCl. The red precipitate was filtered off, washed with cold aqueous ethanol and dried in vacuo.

Spectroscopic and analytical data: **3a.** ^{1}H NMR (200 MHz, C_5D_5N): δ 4.19 (s, 5H, Cp), 4.47 (s, 2H, Cp), 5.06 (s, 2H, Cp), 8.15 (d, J = 8.7 Hz, 2H, Ar), 8.30 (d, J = 8.5 Hz, 2H, Ar). ^{13}C NMR (50 MHz, C_5D_5N): δ 166.6(SCN), 162.8 (COH), 141.0 (Ar), 128.5 (Ar), 125.7 (Ar), 124.5 (Ar), 104.3 (CAr), 78.6 (Cp), 71.1 (Cp), 70.9 (Cp), 68.2 (Cp). IR (KBr, cm^{-1}): 3442 (OH), 1507, 1331 (NO_2), MS (EI): 406 (M^+), HRMS: Found: 406.007454, Calc. for $C_{19}H_{14}SFeN_2O_3$: 406.0074536. **3b**. ^{1}H NMR (200 MHz, C_5D_5N): δ 4.17 (s, 5H, Cp), 4.44 (s, 2H, Cp), 5.03 (s, 2H, Cp), 7.73 (d, J = 8.5 Hz, 2H, Ar), 8.07 (d, J = 8.5 Hz, 2H, Ar). ^{13}C NMR (50 MHz, C_5D_5N): δ 164.9 (SCN), 161.3 (COH), 138.1 (Ar), 132.2 (Ar), 125.6 (Ar), 119.4 (Ar), 107.1 (CN), 103.9 (CAr), 78.6 (Cp), 71.1 (Cp), 70.9 (Cp), 68.2 (Cp). Anal. Calc. for $C_{20}H_{14}SFeN_2O$: C, 62.19, H, 3.65, N, 7.25, S, 8.30. Found: C, 62.04, H, 3.75, N, 7.68, S, 8.19%. **3c** ^{1}H NMR (200 MHz, $CDCl_3$): δ 1.38 (t, J = 7.1 Hz, 3H, CH_2CH_3), 4.15 (s, 5H, Cp), 4.36 (q, J = 7.1 Hz, 2H, CH_2CH_3), 4.50 (t, J = 1.7 Hz, 2H, Cp), 4.92 (t, J = 1.7 Hz, 2H, Cp). ^{13}C NMR (50 MHz, $CDCl_3$): δ 173.4 (CO), 169.9 (SCN), 165.8 (COH), 94.0 (CCO), 71.3 (Cp), 70.6 (Cp), 69.8 (Cp), 68.3 (Cp), 61.4 (CH_2), 14.3 (CH_3) IR ($CHCl_3$, cm^{-1}): 3432 (OH), 1659 (CO). Anal. Calc. for $C_{16}H_{15}SFeNO_3$: C, 53.80, H, 4.23, N, 3.92, S, 8.98. Found: C, 53.67, H, 4.29, N, 3.72, S, 9.05%.

3.2. Synthesis of *4a–4b*

Acetic anhydride (24 l, 0.25 mmol) was added to a solution of **3a**–**3b** (0.25 mmol) in pyridine (200 l) and stirred at r.t. overnight. The reaction mixture was diluted by adding water (10 ml). The solution was extracted with dichloromethane and the organic extracts were dried ($MgSO_4$) and the solvent evaporated. The crude product was purified on silica gel column which was eluted with CH_2Cl_2.

Spectroscopic and analytical data: **4a**. ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.39 (s, 3H, CH_3), 4.21 (s, 5H, Cp), 4.51 (t, J = 1.9 Hz, 2H, Cp), 4.90 (t, J = 1.9 Hz, 2H, Cp), 7.72 (d, J = 8.9 Hz, 2H, Ar), 8.25 (d, J = 8.9 Hz, 2H, Ar). ^{13}C NMR (50 MHz, $CDCl_3$): δ 168.1 (CO), 146.5 (SCN), 136.8 (COC), 136.4 (Ar), 127.5 (Ar), 124.4 (Ar), 118.2 (Ar), 103.4 (CAr), 70.9 (Cp), 70.6 (Cp), 67.8 (Cp), 21.1 (CH_3); IR ($CHCl_3$, cm^{-1}): 1786 (CO), 1514, 1341 (NO_2). MS (EI): 448 (M^+), HRMS: Found: 448.018018, Calc. for $C_{21}H_{16}SFeN_2O_4$: 448.018079. **4b**. ^{1}H NMR (200 MHz, $CDCl_3$): δ 2.37 (s, 3H, CH_3), 4.19 (s, 5H, Cp), 4.49 (t, J = 1.7 Hz, 2H, Cp), 4.88 (t, J = 1.7 Hz, 2H, Cp), 7.65 (d, J = 8.5 Hz, 2H, Ar), 7.67 (d, J = 8.5 Hz, 2H, Ar). ^{13}C NMR (50 MHz, $CDCl_3$): δ 168.2 (CO), 166.8 (SCN), 135.1 (COC), 134.9 (Ar), 132.7 (Ar), 127.5 (Ar), 118.6 (Ar), 118.4 (CN), 110.9 (CAr), 70.8 (Cp), 70.5 (Cp), 68.9 (Cp), 67.8 (Cp), 21.0 (CH_3) IR ($CHCl_3$, cm^{-1}): 2227 (C≡N), 1782 (CO). Anal. Calc. for $C_{22}H_{16}SFeN_2O_2$: C, 61.70, H, 3.77, N, 6.54, S, 7.49. Found: C, 62.03, H, 3.69, N, 6.75, S, 7.31%.

4. X-ray diffraction measurements

The crystals of **3c** suitable for X-ray diffraction study was grown from layered dichloromethane–pentane. The crystal was mounted on a glass fiber and then flash-frozen to 100 K (Oxford Cryosystem-Cryostream Cooler). Preliminary examination and intensities data collections were carried out on a Kuma KM4CCD κ-axis diffractometer with graphite-monochromated Mo Kα radiation [16]. The data were corrected for Lorentz, polarization and absorption effects. The structures were solved by direct methods and refined by the full-matrix least-squares method on all F^2 data using the SHELXTL software [17]. Carbon bonded hydrogen atoms were included in calculated positions and refined in the riding mode using SHELXTL default

Table 5
The crystal and structure refinement data for **3c**

Formula	$C_{16}H_{15}FeNO_3S$
M	357.20
Crystal system	triclinic
Space group	$P\bar{1}$
a (Å)	7.822(5)
b (Å)	10.231(5)
c (Å)	10.602(5)
α	64.64(1)
β	76.94(1)
γ	71.88(1)
V (Å^{-3})	724.4(7)
Z	2
D_x (g cm^{-3})	1.638
μ (mm^{-1})	1.196
T (K)	100(2)
λ (Å)	0.71073
Index ranges	$-9 \leqslant h \leqslant 7$, $-12 \leqslant k \leqslant 11$, $-12 \leqslant l \leqslant 12$
Number of data collected	7939
Number of unique data	2547
R_{int}	0.041
Number of $I > 2\sigma(I)$ data	2516
Number of parameters	204
R_1 [$I > 2\sigma(I)$]	0.0719
wR_2 (all data)	0.1134
$\Delta\rho_{min}$ (e Å^{-3})	−0.681
$\Delta\rho_{max}$ (e Å^{-3})	0.408

parameters. Other hydrogen atoms were located in a difference map. The crystal and structure refinement data are gathered in Table 5.

5. EFISH measurements

The principle of EFISH technique is described elsewhere [18,19]. In order to avoid the reabsorption of the generated second harmonics the data were recorded using the 1907 nm, 10 ns incident laser pulses. The compound was dissolved in chloroform at various concentrations (0–5 mM). The centrosymmetry of the solution was broken by dipolar orientation of the chromophores with a high voltage pulse (~5 kV on 2 mm during 5 μs) Calibration of the cell was made by monitoring the second harmonic generation by a series of solutions of 2-methyl-4-nitroaniline ($\mu\beta = 71 \times 10^{-48}$ esu).

Supplementary material

CCDC 668118 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via http://www.ccdc.cam.ac.uk/data_request/cif.

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