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PHOTOCHROMIC DIHETARYLETHENES.

8*. A NOVEL ROUTE TO THE SYNTHESIS OF 3,4-BIS(2,5-DIMETHYL-3-THIENYL)FURAN-2,5-DIONE AS A POTENTIAL PHOTOCHROME*[2]

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There is proposed, and in the case of 2,5-dimethylthiophene carried out, a novel route to the synthesis of 3,4-dithienylfuran-2,5-dione type photochromes. This is done in two stages, the first being a Friedel–Crafts reaction of the starting thiophene with the dichloride of squaric acid and the second is a Baeyer–Villiger oxidation of the 3,4-bis(2,5-dimethyl-3-thienyl)cyclobutenedione to give the target 3,4-bis(2,5-dimethyl-3-thienyl)furan-2,5-dione.

Keywords: 3,4-bis(2,5-dimethyl-3-thienyl)furan-2,5-dione, 3,4-bis(2,5-dimethyl-3-thienyl)cyclobutenedione, 3,4-dithienylfuran-2,5-diones, 1,2-dithienylethenes, squaric acid dichloride, Friedel–Crafts reaction with thiophenes, Baeyer–Villiger oxidation, photochromism.

In the last decade there has been a rapid expansion in the chemistry of thermally irreversible photochromes based on dithienylethenes [2-6]. For the overwhelming majority of cases, the thienyl rings are separated by hexafluorocyclopentene or maleic anhydride. Synthesis of compounds of the first type is achieved in moderate yields by the reaction of lithium salts of thiophene with octafluorocyclopentene [5]. Preparation of the furandione derivatives is a difficult task. There are two basic routes to their synthesis described in the literature.

The first route (a four stage synthesis) includes the preparation of the nitrile of thienylacetic acid, subsequent reaction using a coupling-dimerization reaction to the corresponding 1,2-dicyanoethene, and cyclization of the latter to the dithienyl derivative of maleic anhydride [7] in an overall yield of 4-6%. The second route (reported in the review [8]) consists of 5 stages, among which the key step is the synthesis of thienylacetic and the acid chloride of thienylglyoxylic acid followed by their subsequent reaction. This gives the maleic anhydride derivative but reference to the original work is absent and the yields of the intermediate and final products are not given.

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* For Communication 7 see [1].

*[2] Dedicated to the 100th year anniversary of the birth of Ya. L. Gol'dfarb (1901-1985).

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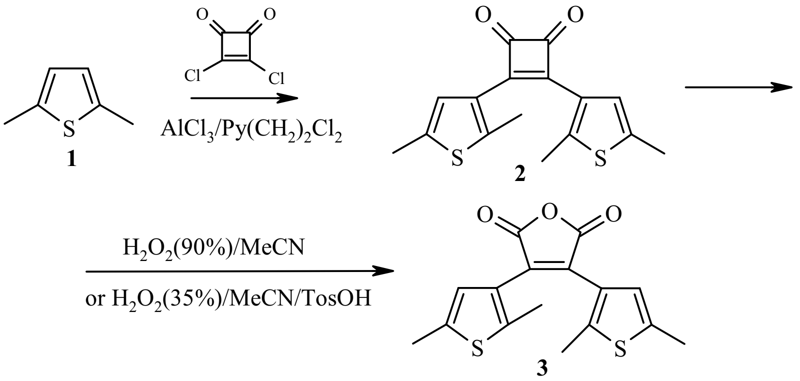
In this publication we describe the synthesis and the study of the physicochemical properties of novel photochromic compounds for the case of 2,5-dimethylthiophene [**1**] in terms of the work carried out in our laboratories in recent year [1, 6, 9-14]. We propose a novel route which, as presented here, may become a convenient general method for the construction of dithienylmaleic anhydrides. Thus acylation of compound **1** with squaric acid dichloride and subsequent Baeyer-Villiger oxidation of the 3,4-bis(2,5-dimethyl-3-thienyl)cyclobutenedione (**2**) gave the target maleic anhydride derivative 3,4-bis(2,5-dimethyl-3-thienyl)furan-2,5-dione (**3**).



It has previously been shown [15] that the substituted cyclobutene-1,2-dione **2** can be obtained in one stage and in 63% yield *via* a modified Friedel–Crafts reaction by treating the dimethylthiophene **1** with 3,4-dichlorobutene-1,2-dione in the presence of aluminium chloride and pyridine. The structure of the diketone **2** is in agreement with its mass spectrum and ^{1}H NMR spectrum and the composition with the results of elemental analysis. Since in the patent [16] where compound **2** was reported for the first time the melting point was not given and the ^{1}H NMR spectrum chemical shifts differed significantly from those observed by us, the structure of the substituted cyclobutenedione **2** we have prepared was confirmed with the aid of X-ray structural analysis.

Figure 1 shows the structure of molecule **2** in the "open" form **A** and in Table 1 the most significant of its geometric parameters are given. All of the bond lengths and valence angles do not deviate from the limits of standard values [17] and do not warrant comment. Attention is drawn to the conformation of the molecule.

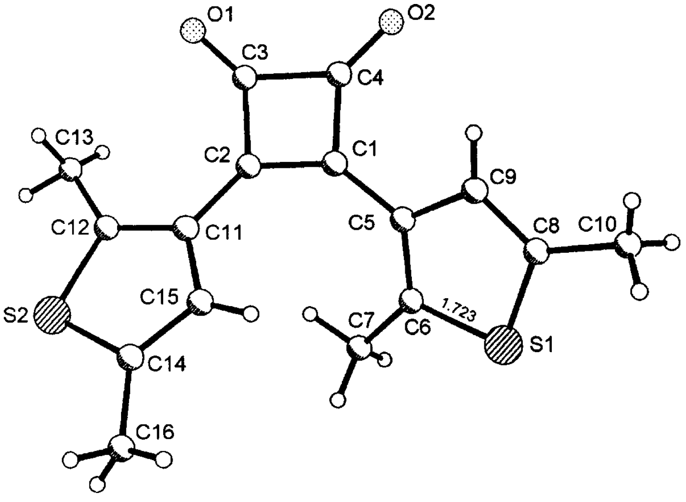


Fig. 1. Molecular structure of compound **2**.

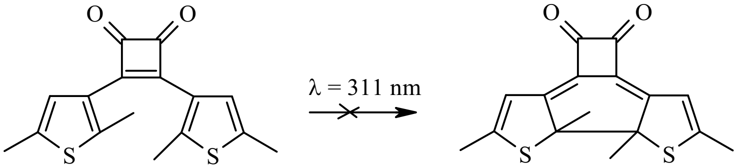
TABLE 1. Bond Lengths (d) and Valence Angles (ω) in Compound **2**

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
S(2)–C(12)	1.712(2)	C(1)–C(4)	1.489(2)	C(8)–C(9)	1.346(2)
S(2)–C(14)	1.734(2)	C(2)–C(11)	1.443(2)	C(8)–C(10)	1.499(3)
S(1)–C(6)	1.722(2)	C(2)–C(3)	1.502(2)	C(11)–C(12)	1.382(2)
S(1)–C(8)	1.730(2)	C(3)–C(4)	1.542(3)	C(11)–C(15)	1.435(2)
O(1)–C(3)	1.201(2)	C(5)–C(6)	1.375(2)	C(12)–C(13)	1.501(2)
O(2)–C(4)	1.198(2)	C(5)–C(9)	1.440(2)	C(14)–C(15)	1.348(2)
C(1)–C(2)	1.387(2)	C(6)–C(7)	1.495(2)	C(14)–C(16)	1.495(2)
C(1)–C(5)	1.450(2)				

Angle	ω, deg.	Angle	ω, deg.
C(12)–S(2)–C(14)	93.55(8)	C(5)–C(6)–C(7)	130.3(2)
C(6)–S(1)–C(8)	93.30(8)	C(5)–C(6)–S(1)	110.16(12)
C(2)–C(1)–C(5)	138.0(2)	C(7)–C(6)–S(1)	119.37(13)
C(2)–C(1)–C(4)	93.66(14)	C(9)–C(8)–C(10)	128.3(2)
C(5)–C(1)–C(4)	128.3(2)	C(9)–C(8)–S(1)	110.20(13)
C(1)–C(2)–C(11)	133.30(14)	C(10)–C(8)–S(1)	121.6(2)
C(1)–C(2)–C(3)	92.24(14)	C(8)–C(9)–C(5)	113.9(2)
C(11)–C(2)–C(3)	134.11(14)	C(12)–C(11)–C(15)	112.33(14)
O(1)–C(3)–C(2)	136.5(2)	C(12)–C(11)–C(2)	125.64(14)
O(1)–C(3)–C(4)	136.3(2)	C(15)–C(11)–C(2)	122.01(13)
C(2)–C(3)–C(4)	87.16(13)	C(11)–C(12)–C(13)	130.7(2)
O(2)–C(4)–C(1)	136.6(2)	C(11)–C(12)–S(2)	110.13(12)
O(2)–C(4)–C(3)	136.4(2)	C(13)–C(12)–S(2)	119.12(14)
C(1)–C(4)–C(3)	86.88(13)	C(15)–C(14)–C(16)	128.4(2)
C(6)–C(5)–C(9)	112.36(14)	C(15)–C(14)–S(2)	109.89(12)
C(6)–C(5)–C(1)	127.5(2)	C(16)–C(14)–S(2)	121.61(14)
C(9)–C(5)–C(1)	120.09(14)	C(14)–C(15)–C(11)	114.08(14)

The methyl substituents in positions 2 of thiophene rings T_1 and T_2 are differently orientated with respect to the double bond of the squaric acid fragment (S): for T_1 the C_1–C_2–C_{11}–C_{12} torsional angle is -164.34° (*trans* orientation of the methyl groups) and for T_2 the corresponding angle for C_2–C_1–C_5–C_6 is 40.19° (*cis* orientation of the methyl groups). This results in a significantly smaller angle of rotation of the T_1 fragment relative to the plane of the four membered S ring (22.00°) when compared with the angle of rotation T_2/S (36.78°). It was noted that both angles and also the dihedral angle between the T_1 and T_2 fragments (47.89°) are the smallest of all of the analogous angles found previously for the dithienylperfluorocyclopentenes [12]. However, with the trans/cis orientation of the substituents in the 2 positions the distance between the potential reaction centers C_6 and C_{12} (5.03 Å) proves to be much larger that the normal van der Waals C···C contact (3.55 Å). In other words, in the crystalline state there are no steric grounds for changing compound **2** to the "closed" form **B**. Evidently, this also explains the difficulty we observed in cyclization in solutions.

Although it was reported in the patent [16] that compound **2** is converted to form **B** by UV irradiation in chloroform, our efforts to establish it for compound **2** by fixed irradiation with light of wavelength 313 nm or with the use of pulsed photolysis at 254 nm proved unsuccessful. Irradiation of compound **2** in hexane and chloroform led only to a progressive destruction of the material and the identification of the decomposition products proved almost impossible. Irradiation with UV light in ethanol gives a mixture of derivatives of maleic, malonic, and fumaric acids, the characterization of which is the subject of a subsequent investigation.



2A **2B**

It is known that Baeyer–Villiger oxidation of α-diketones gives carboxylic acid anhydrides. Moreover, it has been found [18] that 3,4-diphenylcyclobutenedione is readily oxidized to diphenylmaleic anhydride using 30-35% hydrogen peroxide in carbon tetrachloride. However, the use of this method for the thiophene analog **2** did not give the desired results; the yields of the 3,4-bis(2,5-dimethyl-3-thienyl)furan-2,5-dione (**3**) not exceeding 5-10%. A large excess of hydrogen peroxide and even refluxing for 72 h also did not led to an increase in the yield of the target product. In this connection we have used 90% hydrogen peroxide for the oxidation. For a safe process the oxidation was carried out at room temperature and without stirring. Acetonitrile was used as solvent since it dissolved both the hydrogen peroxide and the starting compound. Using these conditions for 5-6 h the target product **3** was prepared in 95% yield. Further investigation showed that this product is obtained in high yield (89%) if 35% hydrogen peroxide is used in the presence of a catalytic amount of p-toluenesulfonic acid. The structure of compound **3** was proved using ^{1}H NMR and mass spectroscopic data and by X-ray structural analysis.

In the crystal the molecule of **3** has C_2 symmetry. The molecular axis passes through atom O_1 and the middle of the double bond in the furan-2,5-dione fragment (Fr), corresponding to a second order crystallographic axis. The structure of the molecule is shown in Figure 2. Bond lengths and valence angles (Table 2) are in good agreement with the corresponding values for molecule **2** but the conformations of molecules **2** and **3** differ markedly. The dihedral angles between the T/Fr fragments in molecule **3** are significantly larger (47.0°) than the analogous dihedral angles (T/S) in molecule **2** but, in contrast to the latter, in structure **3** both methyl substituents in position 2 of the thienyl fragments are *cis* orientated relative to the double bond of fragment Fr. Moreover, the distance between the potential reaction centers C_6 and $C_{6'}$ is decreased to a normal van der Waals contact for C···C (3.55 Å). Hence there are no factors in the crystal of structure **3** which are unfavorable for cyclization to form **B**.

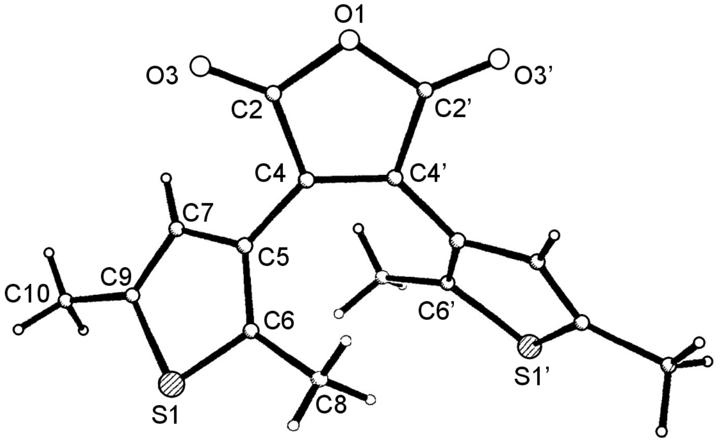


Fig. 2. Molecular structure of compound **3**.

TABLE 2. Bond Lengths (d) and Valence Angles (ω) in Compound **3**

Bond	*d*, Å	Bond	*d*, Å	Bond	*d*, Å
S(1)–C(6)	1.721(2)	C(2)–C(4)	1.492(2)	C(5)–C(7)	1.435(2)
S(1)–C(9)	1.730(2)	C(4)–C(4*)	1.349(3)	C(6)–C(8)	1.494(2)
O(1)–C(2)	1.389(2)	C(4)–C(5)	1.458(2)	C(7)–C(9)	1.353(2)
O(1)–C(2*)	1.389(2)	C(5)–C(6)	1.375(2)	C(9)–C(10)	1.501(2)
C(2)–O(3)	1.187(2)				

Angle	ω, deg.	Angle	ω, deg.
C(6)–S(1)–C(9)	93.28(8)	C(6)–C(5)–C(4)	124.45(13)
C(2)–O(1)–C(2*)	108.1(2)	C(7)–C(5)–C(4)	122.84(13)
O(3)–C(2)–O(1)	120.94(14)	C(5)–C(6)–C(8)	129.68(14)
O(3)–C(2)–C(4)	130.6(2)	C(5)–C(6)–S(1)	110.12(11)
O(1)–C(2)–C(4)	108.45(12)	C(8)–C(6)–S(1)	120.17(12)
C(4*)–C(4)–C(5)	131.80(8)	C(9)–C(7)–C(5)	113.65(14)
C(4*)–C(4)–C(2)	107.52(8)	C(7)–C(9)–C(10)	128.9(2)
C(5)–C(4)–C(2)	120.67(12)	C(7)–C(9)–S(1)	110.20(12)
C(6)–C(5)–C(7)	112.71(13)	C(10)–C(9)–S(1)	120.9(2)

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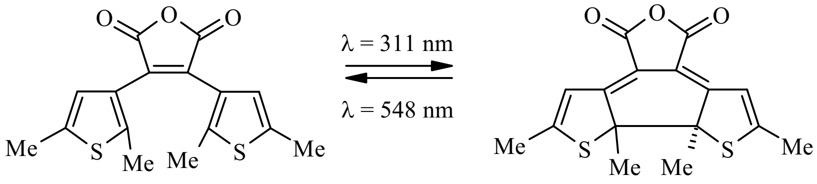
* Atomic coordinates were obtained from the basic coordinates using the transformation $-x + 1, y, -z + 5/2$.

As was expected, compound **3A** is converted to the closed form **3B** under the action of UV irradiation (λ = 313 nm) but the action of longer wavelength light (548 nm) causes it to return to form **3A**.



3A **3B**

Possibly one of the reasons for the discrepancy between our data about the absence of photochromic properties in compound **2** and with that presented in the patent [16] is its slow conversion to compound **3** in air. We have found that diketone **2** in the crystalline state undergoes a 6% conversion to anhydride **3** over 9 months (the content of the oxidized product being determined by the relative integrated intensities in the ^{1}H NMR spectroscopic signals for the starting compound and its oxidation product).

Hence we have developed a novel and convenient two stage synthesis of the 3,4-bis(2,5-dimethyl-3-thienyl)furan-2,5-dione **3**. It should be noted that the shortening of the number of stages leads to an increase in the overall yield of the final product by 4-5 times to 48-50%.

EXPERIMENTAL

^{1}H NMR spectra were recorded on a Bruker AC-200 radio frequency spectrometer using $CDCl_3$ solvent and EI mass spectra on a Kratos (70 eV) instrument with direct input of the sample into the ion source. The melting points were measured on a Boetius microscope stage and are not corrected.

Anhydrous dichloroethane was prepared by distillation over P_2O_5. Pyridine (99.9%) was supplied by the Aldrich Chemical Company. Column chromatography was carried out using neutral Al_2O_3 Brockmann II activity grade. The synthesis of 3,4-dichlorobutene-1,2-dione was performed as described in the method [19], however anhydrous dichloroethane was used instead of benzene and the refluxing time was shortened from 6 to 3 h.

3,4-Di(2,5-dimethyl-3-thienyl)cyclobutene-1,2-dione (2). To a stirred suspension of aluminium chloride (30.0 g, 0.225 mol) in anhydrous dichloroethane (50 ml) at a temperature of -20 to -25°C there were successively added 2,5-dimethylthiophene (22.4 g, 0.2 mol), and dropwise, a solution of 3,4-dichlorobutene-1,2-dione (18.12 g, 0.12 mol) in anhydrous dichloroethane (40 ml), and anhydrous pyridine (7.9 g, 0.1 mol). The reaction mixture was stirred at the same temperature for 30 min, allowed to reach room temperature, held for a further hour, poured onto ice, and the organic layer separated. The aqueous phase was extracted with dichloromethane (2 × 50 ml) and the extracts and organic layer were then combined, washed with aqueous sodium bicarbonate solution, twice with water, and dried over magnesium sulfate. After distillation of solvent the residue was chromatographed on alumina using 60-80°C petroleum ether–ethyl acetate (6:1) as eluent. Yellow crystalline compound **2** was separated in 63% yield; mp 130-131°C (benzene–heptane). ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 6.88 (2H, s, H_{arom}); 2.50 (6H, s, CH_3); 2.42 (6H, s, CH_3). Mass spectrum, *m/z* (I_{rel}, %): 304 (5) $[M+2]^+$, 303 (8) $[M+1]^+$, 302 (40) M^+, 247 (13) $[M\text{-}C_2O_2+1]^+$, 246 (22) $[M\text{-}C_2O_2]^+$, 245 (100) $[M\text{-}C_2O_2\text{-}H]^+$, 231 (19) $[M\text{-}C_2O_2\text{-}CH_3]^+$, 230 (7), 211 (6), 198 (16), 197 (10), 185 (8), 184 (5), 171 (11), 152 (5), 122 (7), 114 (13), 110 (7), 91 (5), 58 (17), 45 (7), 44 (12), 43 (63). Found, %: C 63.53; H 4.66; S 20.97. $C_{16}H_{14}O_2S_2$. Calculated, %: C 63.55; H 4.67; S 21.20.

3,4-Bis(2,5-dimethyl-3-thienyl)furan-2,5-dione (3). A. Diketone **2** (0.3 g, 0.001 mol) was dissolved with heating in acetonitrile (10 ml) and 90% hydrogen peroxide (2 ml) was carefully added to the solution at 30-40°C. The reaction mixture was held at this temperature for 5-6 h and the precipitated crystals of product **3** were separated on a Schott filter, washed with water, dried, and recrystallized from acetonitrile. Yield 95%; mp 201-202°C. ^{1}H NMR spectrum ($CDCl_3$), δ, ppm: 6.76 (2H, s, H_{arom}); 2.42 (6H, s, CH_3); 1.91 (6H, s, CH_3). Mass spectrum, *m/z* (I_{rel}, %): 319 (8) $[M+1]^+$, 318 (39) M^+, 303 (28) $[M\text{-}CH_3]^+$, 302 (13), $[M\text{-}CH_3\text{-}H]^+$, 275 (18) $[M\text{-}CO_2+1]^+$, 274 (18) $[M\text{-}CO_2]^+$, 273 (79) $[M\text{-}CO_2\text{-}H]^+$, 272 (6), 258 (10), 248 (7), 247 (10) $[M\text{-}CO_2\text{-}CO\text{-}1]^+$, 246 (67) $[M\text{-}CO_2\text{-}CO]^+$, 245 (31) $[M\text{-}CO_2\text{-}CO\text{-}H]^+$, 273 (79) $[M\text{-}3CH_3]^+$, 272 (6), 258 (10) $[M\text{-}4CH_3]^+$, 248 (7), 247 (10), 246 (67), 245 (31), 244 (11), 231 (9), 230 (17), 229 (13), 219 (7), 218 (7), 217 (12), 216 (10), 214 (7), 213 (11), 212 (20), 211 (13), 204 (12), 203 (5), 199 (12), 198 (29), 197 (27), 195 (5), 187 (6), 186 (6), 185 (22), 184 (13), 179 (6), 178 (5), 171 (20), 165 (7), 152 (8), 135 (6), 134 (8), 114 (18), 110 (19), 90 (7), 69 (7), 59 (36), 58 (61), 57 (8), 45 (21), 44 (100), 43 (6), 40 (36). Found, %: C 60.24; H 4.82; S 19.75. $C_{16}H_{14}O_3S_2$. Calculated, %: C 60.36; H 4.43; S 20.14.

B. A mixture of dione **2** (0.3 g, 0.001 mol), hydrogen peroxide (30-35%, 2 ml), and *p*-toluenesulfonic acid (0.004 g, 0.023 mmol) were refluxed in acetonitrile (6 ml) until fully dissolved, the solution was held at 30-40°C for 15 h, and the precipitated crystals of the anhydride **3** were filtered off. Yield 89%; mp 198-199°C (acetonitrile).

Photochemical Reactions of Compounds 2 and 3. Irradiation of the samples in the fixed regime was carried out with a DRS-500 mercury lamp using light filters to separate the mercury lines in the spectrum (313 and 578 nm). The irradiation intensity of the mercury lamp was determined using a F_4 filter photocell which was calibrated using a ferrioxalate actinometer [20] for λ = 313 nm and an actinometer based on the Reinecke salt [21] for λ = 578 nm. Absorption spectra were recorded on a Shimadzu UV-2101PC spectrophotometer. For determining the quantum yield, a solution of the material was irradiated with λ = 313 nm light when carrying out the direct reaction and λ = 578 nm for the reverse reaction. The length of irradiation was gradually increased from 5 sec to 1-2 min (7-10 experimental points in all) and the absorption spectrum was recorded for each exposure of the irradiated solution.

In order to reveal the proposed cyclic form **B** of compound 2 we also used a pulsed photolytic assembly consisting of an IFP-2000 photolytic lamp, an Osram XB-150 spectroscopic lamp, and DMP-4 / FEU-38 monochromator. The signal at the photomultiplier was recorded using a Tektronix TDS-350 digital oscillograph combined with a computer. The decay time for the pulsed photolytic lamp light was 12 microseconds. The measurements were taken using a UFS-1 light filter and a cylindrical quartz cuvet of length 80 mm and diameter 15 mm.

X-ray Structural Investigation of Compounds 2 and 3. The intensity of the reflections was measured on a CAD-4 (compound **1**) automatic four-circle diffractometer and on a Siemens XSCANS (compound **2**) at room temperature (graphite monochromator, λMoKα radiation, $\theta/2\theta$ scanning). The structure was solved by a direct method and refined using F^2 full matrix least squares allowing for all independent reflections in the anisotropic approximation for non-hydrogen atoms. The hydrogen atoms were localized by difference synthesis of the electron density and then refined by least squares isotropically. In the calculations the SHELXTL PLUS (version 5.03^+) and AREN-90 program packages were used. The atomic coordinates, temperature factors, and geometrical parameters have been forwarded to the Cambridge structural data bank.

Compound 2. Colorless, transparent crystals of composition $C_{16}H_{14}O_2S_2$ were obtained from a solution in a mixture of ethyl acetate and hexane. Crystals are monoclinic. Unit cell parameters are: a = 8.617(2), b = 7.488(2), c = 22.997(5) Å; β = 97.57(3), V = 1471(5) Å^3; d_{calc} = 1.365 g/cm^3; mp 128-130°C. Space group $P2_1/n$, Z = 4. 3516 Independent reflections were measured. The difference factor for 2820 reflections with $I > 2\sigma(I)$ was R_1= 0.039. For all of the observations wR_2 = 0.121.

Compound 3. Orange crystals of composition $C_{16}H_{14}O_3S_2$ were obtained from a solution in chloroform and are monoclinic. Unit cell parameters: a = 14.592(5), b = 13.103(4), c = 9.095(2) Å; β = 119.56(2), V = 1512.8(7) Å^3; d_{calc} = 1.398 g/cm^3; mp = 203-204°C. Space group $C2/c$, Z = 4. 1639 Independent reflections were measured. The difference factor for 1513 reflections with $I > 2\sigma(I)$ was R_1 = 0.041. For all of the observations wR_2 = 0.108.

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