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Reaction of 5,5-diphenyl-2-thiohydantoin with ethyl chloroacetate: Synthesis and crystal and molecular structure of 2,3,5,6-tetrahydroimidazo-[2,1-*b*]-thiazol-3,6-dione

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Abstract

The crystal and molecular structure of the title compound, obtained as a result of intramolecular cyclization of 5,5-diphenyl-2-carboxymethylmercaptohydantoin, has been determined from X-ray diffractometer data. The final R was 0.049 for 1869 reflections. The conformation of the molecule was found to approximate to the conformations of other similar compounds, but owing to the presence of a C=O group in the thiazol ring, important differences in conjugated bonds system were observed.

Introduction

In the course of our investigation concerning the structure–activity relationship of 5,5-diphenyl-2-thiohydantoin (**1**) derivatives, we reported that in the reaction of **1** with 1,2-dibromoethane, two isomeric products (**2** and **3**) were formed (Karolak-Wojciechowska *et al.*, 1985; Kieć-Kononowicz *et al.*, 1988). The bicyclic structures **4** and **5** enriched with reactive substituents were also obtained (Karolak-Wojciechowska and Kieć-Kononowicz, 1987).

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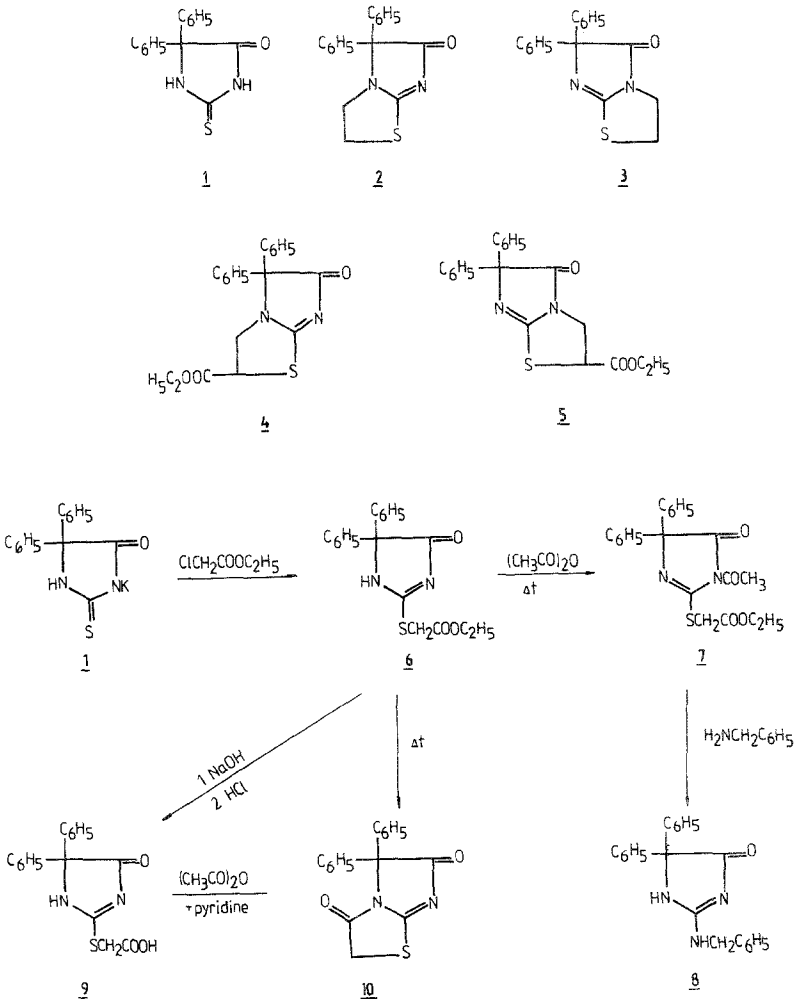
As a continuation of the research program we decided to extend our study to imidazothiazol-diones. Descriptions of the syntheses of such compounds among arylidene and arylazo derivatives of 2-thiohydantoin can be found in the literature (Shalaby *et al.*, 1976). Thus, we adopted the given reaction conditions for our purposes, and on this basis we attempted to synthesize imidazothiazol-diones (Scheme I). In this study we also performed an X-ray structure analysis of **10**.



Scheme I

Experimental

Melting points were determined on a Boetius hot-stage microscope and are uncorrected. Infrared spectra were measured with a Specord 75 IR (VEB Carl Zeiss, Jena) using KBr disks (1 : 300 mg KBr). ^1H NMR spectra were recorded on a Perkin–Elmer R 12 B spectrometer at 60 MHz and chemical shifts are given as parts per million from TMS. ^{13}C NMR spectra were recorded on a Bruker HX 90 spectrometer at 90 MHz using TMS as the internal standard. The mass spectra were obtained using a GCMS 2091 LKB mass spectrometer operating at an ionizing energy of 70 eV. The samples were introduced into the source via a direct inlet system. The tlc was performed with silica gel GF_{254} precoated tlc plates (5 × 10 cm; 0.25 mm) using chloroform : ethyl acetate (1 : 1) as a developing system.

*5,5-Diphenyl-2-carboethoxymethylmercaptohydantoin (**6**)*

The mixture of the potassium salt of 5,5-diphenyl-2-thiohydantoin (**1**) (3.06 g; 0.01 mol) and ethyl chloroacetate (1.22 g; 0.01 mol) in 20 ml of anhydrous ethanol was stirred at room temperature for 2 days. The precipitate was filtered, washed with water, and crystallized from ethanol to give 3.0 g of white solid (m.p., 137–139°C; yield, 85%; R_f = 0.80). *Anal.* Calc. for $C_{19}H_{18}N_2O_3S$ (354.4): C = 64.39; H = 5.11; N = 7.90. Found: C = 64.20; H = 5.02; N = 7.85.

*5,5-Diphenyl-2-carboxymethylmercaptohydantoin (**9**)*

The mixture of **6** (3.54 g; 0.01 mol) in 100 ml of a 2% aqueous solution of NaOH was kept at room temperature overnight. The clear solution was acidified with 2% HCl, and the formed precipitate was filtered and washed several times with water (m.p., 183–185°C); 3.06 g; yield, 94%; R_f = 0). *Anal.* Calc. for $C_{17}H_{14}N_2O_3S$ (326.4): C = 62.56; H = 4.32; N = 8.58. Found: C = 62.30; H = 4.21; N = 8.42.

Action of acetic anhydride on **6**

The suspension of **6** (3.54 g; 0.01 mol) in acetic anhydride (30 ml) was refluxed for 4 hr. The reaction mixture was allowed to cool and then poured into cold water. Chloroform was added and the reaction mixture was extracted three times with chloroform. The chloroform extracts were washed with a 1% water solution of NaOH, dried with Na_2SO_4, and evaporated. The residue was column chromatographed on silica gel (Merck, 200–300 mesh). The fractions obtained after elution with benzene : acetone (20 : 1.5) were evaporated to dry-

ness and crystallized from ethyl acetate : chloroform (1 : 1) to give 3.0 g (yield, 76%) of the white solid of **7**. (m.p., 68–70°C; R_f = 0.9). *Anal.* Calc. for $C_{21}H_{20}N_2O_4S$ (396.5): C = 63.61; H = 5.09; N = 7.06. Found: 63.59; H = 5.08; N = 7.01. MS (*m/e* rel. intensity): 396(36); 354(59); 325(14); 311(17); 309(19); 281(4); 267(6); 224(98); 207(62); 183(6); 149(6); 105(3); 78(100). ^{1}N NMR ($CDCl_3$): 1.26 (3H, t, J = 7.00, CH_3); 2.66 (3H, s, CH_3CO); 4.02 (2H, s, CH_2CO); 4.27 (2H, q, J = 7.20, CH_2-CH_3); 7.30–7.70 (10H, m, C_6H_5). ^{13}C NMR ($CDCl_3$): 14.03 (CH_3CH_2); 24.55 ($COCH_3$); 33.58 ($-SCH_2-$); 61.64 ($COOCH_2$); 79.00 [$C(Ph)_2$]; 126.43; 126.75; 127.68; 128.04; 128.19; 128.24; 128.48; 128.62; 128.68; 139.50 (aromatic carbons); 158.18 (COO); 168.52; 169.23 (C=O;C_4=O); 177.93 [(C=N)−S]. IR (cm^{-1}): 2968, 1742, 1714 (C=O), 1696 (C=O), estr., 1566 (C=N); 1552, 1428, 1354, 1286, 1272, 1140, 1030, 920, 808, 766, 756, 708, 668.

*5,5-Diphenyl-2,3,5,6-tetrahydroimidazo-[2,1b]-thiazol-3,6-dione (***10***)*

a. Compound **6** (3.54 g; 0.01 mol) was warmed at 150–160°C for 15 min. The melted product became dark red; it was cooled and dissolved in chloroform, washed several times with 1% NaOH, dried with Na_2SO_4, and evaporated. The residue column chromatographed with a benzene : acetone (20 : 1.5) solution, after evaporation of solvents crystallized from a $CHCl_3$: ethyl acetate (1 : 1) solution, gave 0.5 g (16%) of white solid (m.p., 201–202°C; R_f = 0.71). *Anal.* Calc. for $C_{17}H_{12}N_2O_2S$ (308.4): C = 66.21, H = 3.92, N = 9.08. Found: C = 65.98, H = 4.11, N = 9.02. MS (*m/e* rel. intensity): 308(100); 280(3); 266(32); 238(15); 234(11); 207(4); 165(12); 135(16); 103(11). ^{1}H NMR ($CDCl_3$): 4.13 (2H, s, CH_2) 7.38 (10H, s, C_6H_5). ^{13}C NMR ($CDCl_3$): 36.22 (CH_2); 77.60 [$C(Ph)_2$]; 128.04; 128.27; 128.62; 128.80; 129.04; 135.10 (aromatic carbons); 164.63 (C_3=O); 186.80; 187.00 [C=O and C(=N)S]. IR (cm^{-1}): 2964, 2916, 1718, 1703 (C=O); 1492 (C=N), 1344, 1256, 1130, 1002, 768, 756, 736.

b. The suspension of acid **9** (2.0g) in 2 ml of acetic anhydride and 3 ml of pyridine was left overnight at room temperature. The solid was diluted and the freshly precipitated solid was filtered, dissolved in $CHCl_3$, washed with a 1% NaOH solution, and dried over Na_2SO_4; after evaporation, it was recrystallized from a chloroform : ethyl acetate (1 : 1) solution to yield 1.6 g of **10** white crystals (52%).

Action of benzyloamine on **7**

The suspension of **7** (0.396 g; 1 mol) and (0.321 g; 1 mol) benzylamine in toluene (10 ml) was left at room temperature overnight. The solid obtained was filtered and crystallized from ethanol to give 0.290 g (85% yield) of white crystals of **8** (Kieć-Kononowicz and Kejc, 1980).

X-Ray structure determination of **10**

Crystal data: $C_{17}H_{12}O_2N_2S$, $M = 308.35$, monoclinic, $a = 10.518(1)$, $b = 8.550(1)$, $c = 16.586(1)$ Å, $\beta = 106.28(1)°$, $V_c = 1431.7(2)$ Å^3, $Z = 4$, $D_c = 1.430$ g · cm^{-3}, $F(000) = 640$, $\bar{\lambda}$ (Mo $K\alpha$) = 0.71069 Å, $\mu = 2.23$ cm^{-1}, space group $P2_1/c$.

Compound **10**, for X-ray analysis, was recrystallized from a mixture of chloroform and ethyl acetate (1 : 1). The crystal used in the analysis was cut from a long needle to the dimensions 0.3, 0.4, 0.4 mm. The preliminary information about the unit cell and space group was obtained photographically on a de Jonge–Bouman camera, with Cu $K\alpha$ radiation. The accurate cell dimensions were derived from diffractometer data. The intensity data were collected on a CAD-4 diffractometer using Mo $K\alpha$ radiation ($\omega - 2\theta$ scan mode). No intensity variations were observed during the experiment. The Lorentz and polarization corrections were applied to 2487 measured reflections, of which 1873 were assumed to be observed according to the criterion $F \leqslant 3\sigma(F)$.

The structure was solved by the direct method (MULTAN-80). The positions of all non-H atoms were obtained from the E map derived from the best solution (Abs.FOM. = 1.313). Then the structure was refined using F's by the standard full-matrix least-squares method and difference electron density synthesis (SHEL-X-76 system of programs for 400 atoms, IBM-PC-AT computer). All H atoms were located from the ΔF map after anisotropic refinement for non-H atoms. The isotropic temperature factors for H atoms were held at 1.5 times the respective values for the non-H atoms, and their positions were refined. The final $R = 0.0490$ ($R_w = 0.0423$, $w = 1/\sigma^2$) was calculated for 1869 reflections (4 reflections were omitted due to secondary extinction). At this stage the largest shift of esd's for non-H atoms was equal to 0.06.

The final positional and equivalent thermal parameters for non-H atoms are given in Table 1.

Results and discussion

Taking into account the given reaction conditions (Scheme I), we obtained a low melting substance, **7**, which during the reaction with benzylamine at room temperature, lost sulfur (**8**).

Based on the analyses of mass, ^{13}C NMR, ^{1}H NMR, and IR spectra, we stated that the product formed was not cyclic and was a result of *N*-acylation of 5,5-diphenyl-2-carboethoxymethylmercaptohydantoin, **6**. Thus, having in mind the synthesis of imidazathiazol-dione derivatives, we tried the reaction conditions described by Chaudhary *et al.* (1970). The acid **9** (obtained as a result of alkaline hydrolysis of ester **6**) was heated with acetic anhydride and pyridine.

Table 1. Positional parameters ($\times 10^4$) for the nonhydrogen atoms and U_{eq} ($\times 10^3$)

	x	y	z	U_{eq}
S	3450(1)	3269(1)	8729(1)	49(0)
O1	8191(2)	3878(3)	10266(1)	46(1)
O2	4778(2)	6176(3)	7363(1)	56(1)
N1	5989(2)	3210(3)	9734(2)	38(1)
N2	5553(2)	4552(3)	8504(1)	30(1)
C1	5128(3)	3616(3)	9050(2)	31(1)
C2	7169(3)	3922(3)	9714(2)	32(2)
C3	6982(2)	4843(3)	8863(2)	29(1)
C4	4597(3)	5183(4)	7836(2)	41(2)
C5	3277(3)	4458(5)	7802(2)	56(2)
C11	7267(2)	6576(3)	9057(2)	30(1)
C12	6311(3)	7555(3)	9219(2)	37(2)
C13	6597(3)	9096(4)	9437(2)	48(2)
C14	7846(3)	9689(4)	9498(2)	53(2)
C15	8793(3)	8727(4)	9337(2)	49(2)
C16	8516(3)	7166(3)	9125(2)	38(2)
C21	7803(3)	4101(3)	8333(2)	36(2)
C22	8996(3)	3383(4)	8700(2)	53(2)
C23	9725(4)	2702(5)	8210(3)	73(3)
C24	9258(5)	2764(6)	7349(3)	84(4)
C25	8102(5)	3487(6)	6981(3)	87(4)
C26	7363(4)	4161(5)	7465(2)	61(2)

Hydrogen atom positional parameters ($\times 10^3$) with isotropic temperature factors ($Å^2 \times 10^3$)

	x	y	z	U
H12	536(3)	724(4)	913(2)	55
H13	588(3)	972(4)	954(2)	70
H14	809(3)	1073(5)	967(2)	80
H15	972(3)	914(4)	937(2)	74
H16	921(3)	644(4)	902(2)	52
H22	937(3)	351(4)	931(2)	77
H23	1057(4)	240(5)	846(3)	101
H24	978(4)	205(5)	706(3)	122
H25	770(4)	327(6)	634(3)	125
H26	660(4)	464(5)	721(2)	87
H51	260(4)	529(5)	779(2)	81
H52	297(3)	371(5)	737(2)	81

Anisotropic temperature factors ($Å^2 \times 10^3$) in the form $\exp[-2\pi^2(U_{11}h^2a^{*2} + U_{22}k^2b^{*2} + U_{33}l^2c^{*2} + 2U_{23}klb^*c^* + 2U_{13}lhc^*a^* + 2U_{12}hka^*b^*)$

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S	37(0)	60(1)	53(0)	−9(0)	17(0)	−13(0)
O1	41(1)	60(1)	37(1)	13(1)	6(1)	7(1)
O2	62(1)	58(2)	42(1)	18(1)	3(1)	−3(1)
N1	41(1)	39(1)	39(1)	7(1)	18(1)	0(1)
N2	32(1)	29(1)	29(1)	0(1)	8(1)	−3(1)

Table 1. Continued

	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
C1	38(1)	25(1)	37(2)	−7(1)	18(1)	−2(1)
C2	37(2)	29(2)	34(1)	3(1)	15(1)	6(1)
C3	29(1)	31(2)	27(1)	2(1)	9(1)	−1(1)
C4	40(2)	44(2)	35(2)	−4(1)	3(1)	1(1)
C5	41(2)	67(3)	54(2)	−3(2)	1(2)	−5(2)
C11	35(1)	33(2)	21(1)	1(1)	6(1)	−2(1)
C12	42(2)	34(2)	40(2)	−1(1)	18(1)	2(1)
C13	59(2)	40(2)	47(2)	−8(2)	17(2)	5(2)
C14	65(2)	35(2)	53(2)	−10(2)	8(2)	−7(2)
C15	45(2)	45(2)	53(2)	−9(2)	5(2)	−11(2)
C16	34(1)	40(2)	36(2)	−2(1)	5(1)	−4(1)
C21	43(2)	29(2)	41(2)	−5(1)	22(1)	−6(1)
C22	44(2)	61(2)	62(2)	−3(2)	26(2)	6(2)
C23	58(2)	70(3)	109(4)	−8(3)	53(3)	7(2)
C24	104(4)	79(3)	100(4)	−22(3)	78(3)	2(3)
C25	117(4)	101(4)	62(3)	−17(3)	58(3)	7(3)
C26	76(2)	73(3)	43(2)	−3(2)	30(2)	9(2)

However, the only product we obtained was 5,5-diphenylhydantoin. Therefore, we adopted the following conditions: melting of ester **6** at 150–160°C and then reaction of **9** with acetic anhydride in the presence of pyridine (room temperature). The yield of **10** was satisfactory in the latter reaction. On the basis of our experience (Karolak-Wojciechowska *et al.*, 1985; Karolak-Wojciechowska and Kieć-Kononowicz, 1987; Kieć-Kononowicz *et al.*, 1988), the possibility of N_1 and N_3 cyclization should be taken into consideration, but in this particular case only one cyclic product (formula **10**) was separated.

The molecule of compound **10** was first described from its spectral features based on the spectral data for bicyclic derivatives obtained earlier (Karolak-Wojciechowska *et al.*, 1985; Karolak-Wojciechowska and Kieć-Kononowicz, 1987; Kieć-Kononowicz *et al.*, 1988). The MS, ^{13}C NMR, and IR properties suggested that the separated compound was a result of N_1 intramolecular cyclization, i.e., **10** is an example of a 1,2-substituted derivative of **1**. In accordance with that we have in MS the low-intensity M-CO (M-28) ion; also, in IR C=O and C=N absorption bands were in the ranges of 1718–1703 and 1492 cm^{-1} [Kieć-Kononowicz *et al.* (1988): 1710–1685 and 1490–1475 cm^{-1}]. In ^{13}C NMR the spectrum difference between chemical shifts of these carbon atoms was rather small at 0.20 ppm ($\Delta\sigma$ from 0.13 to 6.60 ppm).

The results of the X-ray analysis of **10** have also confirmed the above-suggested structure of the molecule. The molecular structure of **10**, along with the numbering system of the atoms, is shown in Fig. 1. Crystallographically found bond lengths and angles are given in Tables 2 and 3, respectively. An

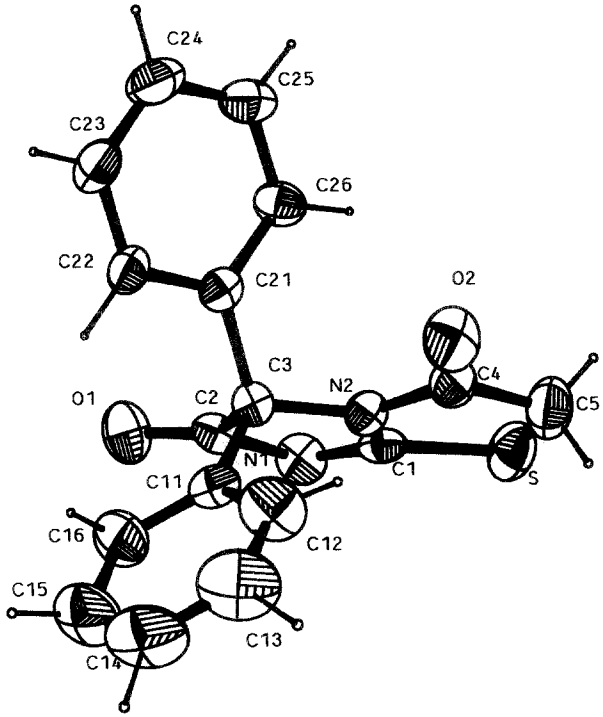


Fig. 1. Molecule of **10** with numbering of the atoms.

Table 2. Bond lengths (Å)

C1---S	1.720(3)	C5---S	1.809(4)
C2---O1	1.201(3)	C4---O2	1.207(4)
C1---N1	1.285(3)	C2---N1	1.391(3)
C1---N2	1.375(3)	C3---N2	1.474(3)
C4---N2	1.380(3)	C3---C2	1.580(3)
C11---C3	1.528(4)	C21---C3	1.532(4)
C5---C4	1.508(4)	C12---C11	1.391(4)
C16---C11	1.382(4)	C13---C12	1.377(4)
C14---C13	1.386(5)	C15---C14	1.375(5)
C16---C15	1.390(4)	C22---C21	1.376(4)
C26---C21	1.385(4)	C23---C22	1.393(5)
C24---C23	1.375(6)	C25---C24	1.348(6)
C26---C25	1.388(5)		
H51---C5	1.00(4)	H52---C5	0.95(4)
H12---C12	1.01(3)	H13---C13	0.98(3)
H14---C14	0.95(4)	H15---C15	1.03(3)
H16---C16	1.01(3)	H22---C22	0.98(4)
H23---C23	0.91(4)	H24---C24	1.03(5)
H25---C25	1.04(5)	H26---C26	0.89(4)

Table 3. Bond angles (deg)

C5—S—C1	91.0(1)	C2—N1—C1	105.9(2)
C3—N2—C1	108.4(2)	C4—N2—C1	117.2(2)
C4—N2—C3	133.6(2)	N1—C1—S	129.4(2)
N2—C1—S	113.1(2)	N2—C1—N1	117.3(2)
N1—C2—O1	125.6(3)	C3—C2—O1	123.9(2)
C3—C2—N1	110.5(2)	C2—C3—N2	97.8(2)
C11—C3—N2	111.5(2)	C11—C3—C2	109.1(2)
C21—C3—N2	111.9(2)	C21—C3—C2	110.9(2)
C21—C3—C11	114.4(2)	N2—C4—O2	125.8(3)
C5—C4—O2	125.2(3)	C5—C4—N2	109.0(3)
C4—C5—S	109.1(2)	C12—C11—C3	121.0(2)
C16—C11—C3	119.8(2)	C16—C11—C12	119.1(3)
C13—C12—C11	120.7(3)	C14—C13—C12	120.1(3)
C15—C14—C13	119.4(3)	C16—C15—C14	120.8(3)
C15—C16—C11	119.9(3)	C22—C21—C3	121.4(3)
C26—C21—C3	120.2(3)	C26—C21—C22	118.3(3)
C23—C22—C21	120.8(4)	C24—C23—C22	119.7(4)
C25—C24—C23	120.2(4)	C26—C25—C24	120.5(4)
C25—C26—C21	120.5(4)		

examination of Fig. 1 shows that the compound under discussion is a bicyclic derivative containing an imidazathiazol-dione moiety and two phenyl rings, and it is a result of N_1 intramolecular cyclization.

The selected torsion angles and the deviations of the atoms from the least-squares planes passing through both five-membered rings in the molecule of **10** are given in Tables 4 and 5, respectively. These values, necessary for deeper geometrical description, clearly show that the thiazol-dione ring is not flat and that it has an open-envelope conformation with the C(4) atom (from the C=O group) in a flap position. Both five-membered rings joined into an imidathiazol-dione moiety are inclined to each other at the angle of 5.9(1)°. It should be noted that in the molecule of **2** both these rings are flat and lie practically in this same plane (Karolak-Wojciechowska *et al.*, 1985).

Two phenyl rings present in the molecule are planar and inclined to each other at the angle of 78.0(1)°. The analogous angle in **2** is similar [79.4(4)°], but it decreases significantly in derivatives in which, in the place of the thiazole ring, there is a thiazine or thioazepine ring (Kieć-Kononowicz *et al.*, 1988).

Table 4. Torsion angles (deg) in the thiazole ring

C(1)—S—C(5)—C(4)	−4.4(3)
S—C(5)—C(4)—N(2)	7.5(4)
C(5)—C(4)—N(2)—C(1)	−8.1(5)
C(4)—N(2)—C(1)—S	4.9(4)
N(2)—C(1)—S—C(5)	0.1(3)

Table 5. Deviations (Å) of atoms from the least-squares planes

Plane 1		Plane 2	
C(1)	0.003(3)	C(1)	−0.020(3)
C(2)	−0.020(3)	C(4)	−0.059(3)
C(3)	0.018(3)	C(5)	0.048(4)
N(1)	0.009(2)	N(2)	0.015(2)
N(2)	−0.009(2)	S	−0.001(1)

Plane 1: $0.36212 * x - 0.81631 * Y - 0.44972 * Z = -8.57633$
Plane 2: $0.30089 * x - 0.79344 * Y - 0.52907 * Z = -9.69904$

All bond lengths and angles in the molecule of **10** have values in the expected range. However, there are important differences in bond lengths between the structure being described and the molecule of **2** (Karolak-Wojciechowska *et al.*, 1985). Owing to the presence of the second carbonyl group at the C(4) atom in the molecule, significant modifications in bond lengths within the conjugated bonds system have been observed. Thus, the N(1)—C(2), C(1)—S, and C(1)—N(2) bonds are longer, while N(1)—C(1) and N(2)—C(4) are shorter (the differences being larger than 3σ). More detailed information about the coupling bond system in **2** and **10** is given in Table 6. These differences have important consequences for pharmacological properties of **10**, namely, while **2** has some anticonvulsant properties, **10** is biologically inactive (Kieć-Kononowicz, 1988). The above observation is quite clear from the viewpoint of the mechanism of the drugs' action given by Wong *et al.* (1986). In this connection the differences in the conjugated bond system are equivalent to the differences in the charges of the atoms and with the possibilities of the H-bond formation, which play a significant role in the action of the drugs.

All intramolecular distances are shorter than the sum of the van der Waals radii.

Table 6. Bond lengths in the coupling part of molecules **2** and **10**

	2	**10**	Difference
C(2)=O(1)	1.210(4)	1.201(3)	
C(2)—N(1)	1.374(4)	1.391(3)	0.017
N(1)=C(1)	1.314(4)	1.285(3)	−0.029
C(1)—S	1.705(3)	1.720(3)	0.015
C(1)—N(2)	1.326(4)	1.375(3)	0.049
N(2)—C(4)	1.448(5)	1.380(3)	−0.068

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Structure factor data have been deposited with the British Library, Boston Spa, Wetherby, West Yorkshire, UK, as supplementary publication No. 60573 (13 pages).