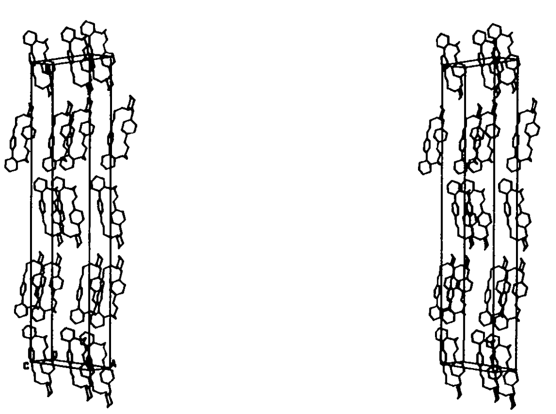

Fig. 3. Packing diagram; H atoms are omitted for clarity.

Fig. 3 shows how the molecules pack in the crystal. The shortest interatomic distances to adjacent different molecules are: no C···C or C···N below 3·5 Å; N···N 3·396 (3) Å, between N(2) and N(10) at $\frac{1}{2}+x, y, -\frac{1}{2}+z$; N···H 2·44 Å, between N(2) and H(10) at $\frac{1}{2}+x, y, -\frac{1}{2}+z$; C···H 2·86 Å, between C(5) and H(13) at $x, y-1+z$; and H···H 2·29 Å, between H(3*a*) and H(9*b*) at $\frac{1}{2}+x, y, -\frac{1}{2}+z$.

This work was supported by grant WPR III/6 from the Polish Academy of Sciences. The author is grateful to Dr Marek Pietraszkiewicz and Mr Rafał Gąsiorowski for the crystals used.

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Acta Cryst. (1990). C**46**, 101–103

2-(*p*-Methoxybenzoylmethylene)-3-phenyl-1,3-thiazolidine-4,5-dione, a Compound with a C—S···O Interaction

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(*Received* 16 *November* 1988; *accepted* 26 *April* 1989)

Abstract. $C_{18}H_{13}NO_4S$, M_r = 339·4, monoclinic, $P2_1/n$, a = 8·359 (1), b = 9·961 (2), c = 19·048 (3) Å, β = 93·749 (12)°, V = 1582·83 Å^3, Z = 4, D_m = 1·43, D_x = 1·42 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ = 1·897 mm^{-1}, F(000) = 704, T = 293 K, R = 0·0508 for 2390 unique observed reflections. The molecule has a short intramolecular S···O contact of 2·697 Å and a C—S···O angle of 164·8°.

Introduction. This study is supplementary to an earlier one (Ciechanowicz-Rutkowska, Grochowski & Stec, 1989) and concerns the structure of the Knoevenagel reaction substrate, crystallized successfully in a form of a *p*-methoxybenzoyl derivative, while the earlier study concerned the structure of the reaction product. This work was undertaken in order to assess the role of the benzoyl substituent in the course of the reaction with malonodinitrile which takes place for compound *A* but not for compound *B*, as shown schematically in Fig. 1.

Experimental. The title compound was obtained by Dr J. Eilmes (Żankowska-Jasinska & Eilmes, 1973) and crystallized from chlorobenzene at room tem-

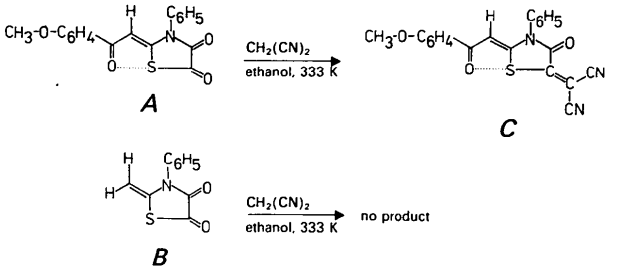

Fig. 1. The Knoevenagel reaction.

Table 1. *Data collection and refinement conditions*

Crystal size (mm)	0·15 × 0·35 × 0·50
Measuring D_m	By flotation
Diffractometer	Enraf–Nonius CAD-4 (graphite-monochromated Cu $K\alpha$ radiation)
Lattice parameter measurement	
θ range (°); number of reflections	$2 \le \theta \le 20$, 25
Intensity measurement	
θ range (°)	$1 \le \theta \le 78$
Indices range	$-10 \le h \le 10$, $0 \le k \le 12$, $0 \le l \le 23$
Scan width (°) and mode	$0·56 + 0·16\tan\theta$, $\omega/2\theta$
Intensity control reflections	110, 204, measured every hour
Changes in intensity	< 10%
Number of reflections measured	3916
Criterion for observed reflections	$\|F_o\| \ge 2\sigma(F)$
Number of observed unique reflections	2390
R_{int}	0·027
Corrections applied	Lorentz–polarization effects
Computer programs	*SHELX76* (Sheldrick, 1976)
Atomic scattering factors	As in *SHELX76*
Solution	Automatic centrosymmetric direct methods with *SHELX76*
Refinement method	Full-matrix least squares on $\|F_o\|$'s
Parameters refined	278
Non-H atoms	Positional and anisotropic thermal
H atoms	Positional and isotropic thermal*
Weighting scheme	$w = k[\sigma^2(F_o) + g(F_o)^2]^{-1}$ k converged to 1·0 and g to 0·00631
R, wR, S	0·051, 0·064, 1·095
Av., max. Δ/σ	0·01, 0·1
Max., min. height in final difference Fourier synthesis (e Å^{-3})	0·26, −0·39

*Initial positional parameters for all H atoms found from difference Fourier maps.

Table 2. *Fractional atomic coordinates* (× 10^4) *and equivalent isotropic thermal parameters* U_{eq} (Å^2 × 10^3) *with e.s.d.'s in parentheses*

$$U_{eq} = \tfrac{1}{3}(U_{11} + U_{22} + U_{33}).$$

	x	y	z	U_{eq}
N	−961 (2)	4080 (2)	6369 (1)	48 (1)
C3	−764 (3)	3634 (3)	5685 (1)	47 (1)
S	−2262 (1)	4266 (1)	5085 (0)	55 (1)
C5	−3221 (3)	5050 (3)	5765 (2)	68 (1)
O3	−4440 (3)	5687 (3)	5707 (1)	106 (1)
O2	−2605 (3)	5377 (3)	7007 (1)	86 (1)
C4	−2250 (3)	4868 (3)	6462 (1)	61 (1)
C13	221 (3)	3840 (3)	6944 (1)	45 (1)
C14	1587 (3)	4605 (3)	6988 (1)	57 (1)
C15	2719 (3)	4397 (3)	7548 (2)	65 (1)
C16	2425 (3)	3454 (3)	8057 (1)	64 (1)
C17	1043 (3)	2725 (3)	8011 (1)	65 (1)
C18	−89 (3)	2900 (3)	7448 (1)	58 (1)
C1	575 (3)	2381 (3)	4786 (1)	50 (1)
C2	385 (3)	2778 (3)	5515 (1)	51 (1)
C6	1623 (3)	1259 (3)	4627 (1)	50 (1)
O1	−138 (2)	3018 (2)	4302 (1)	65 (1)
C7	2234 (3)	336 (3)	5129 (1)	51 (1)
C8	3172 (3)	−719 (3)	4950 (1)	58 (1)
C9	3530 (3)	−888 (3)	4243 (1)	57 (1)
C10	2941 (3)	0 (3)	3739 (1)	64 (1)
C11	2005 (3)	1052 (3)	3929 (1)	61 (1)
C12	5020 (4)	−2099 (4)	3425 (2)	88 (1)
O4	4463 (3)	−1962 (2)	4114 (1)	78 (1)

perature in the form of yellow, flat needles by Dr B. Zaleska. The details of the X-ray measurements and the structure computation are given in Table 1.

Table 3. *Intramolecular distances* (Å) *and angles* (°), *and torsion angles* (°) *around the* S *atom, with e.s.d.'s in parentheses*

S—C3	1·756 (2)	C7—C8	1·368 (4)
S—C5	1·751 (3)	C8—C9	1·409 (4)
C5—C4	1·521 (4)	C9—C10	1·373 (4)
C5—O3	1·199 (4)	C10—C11	1·371 (4)
C4—O2	1·209 (4)	C11—C6	1·403 (4)
C4—N	1·354 (4)	C13—C14	1·371 (4)
N—C13	1·446 (3)	C14—C15	1·396 (4)
N—C3	1·396 (3)	C15—C16	1·383 (4)
C3—C2	1·340 (4)	C16—C17	1·362 (4)
C2—C1	1·464 (4)	C17—C18	1·394 (4)
C1—O1	1·239 (3)	C18—C13	1·379 (4)
C1—C6	1·464 (4)	C9—O4	1·355 (4)
C6—C7	1·398 (4)	O4—C12	1·429 (5)
S···O1	2·697 (2)	C5—S···O1	164·8 (1)
C5—S—C3	91·2 (1)	C1—C6—C11	118·9 (2)
S—C5—C4	110·4 (2)	C7—C6—C11	117·1 (2)
S—C5—O3	126·5 (3)	C6—C7—C8	121·7 (2)
O3—C5—C4	123·1 (3)	C7—C8—C9	119·4 (2)
O2—C4—C5	123·5 (3)	C8—C9—C10	120·2 (3)
O2—C4—N	126·7 (3)	C8—C9—O4	115·3 (2)
C5—C4—N	109·8 (2)	C10—C9—O4	124·5 (3)
C4—N—C3	116·7 (2)	C9—C10—C11	119·5 (3)
C4—N—C13	120·5 (2)	C10—C11—C6	122·1 (3)
C3—N—C13	122·5 (2)	C9—O4—C12	117·9 (3)
N—C3—C2	123·9 (2)	C16—C17—C18	120·9 (3)
N—C3—S	111·7 (2)	N—C13—C14	118·7 (2)
S—C3—C2	124·3 (2)	N—C13—C18	119·3 (2)
C3—C2—C1	121·7 (2)	C14—C13—C18	121·9 (2)
C2—C1—O1	119·4 (2)	C13—C14—C15	119·0 (3)
O1—C1—C6	120·2 (2)	C14—C15—C16	119·6 (3)
C2—C1—C6	120·3 (2)	C15—C16—C17	120·4 (2)
C1—C6—C7	123·9 (2)	C17—C18—C13	118·2 (2)
C2—C3—S—C5	173·8 (3)	C6—C1—C2—C3	−168·1 (5)
S—C3—C2—C1	5·6 (3)	O1—C1—C2—C3	13·1 (5)

Discussion. Positional and thermal parameters are listed in Table 2, intramolecular distances and angles, and torsion angles around the S atom, in Table 3.* The conformation of the molecule studied (*A*) is shown together with the atom numbering in Fig. 2. The molecular packing in the crystal unit cell is shown in Fig. 3. Figures were drawn on an IBM PC/AT with *ORTEP* (Johnson, 1971), within the *CRYSRULER* package (Rizzoli, Sangermano, Calestani & Andreetti, 1987). The S and O atoms of the methoxybenzoyl group, in close analogy to those of 2-[2-(benzoylmethylene)-4-oxo-3-phenyl-1,3-thiazolidin-5-ylidene]propanedinitrile, are in an *s-cis* conformation forming a 'single bond–no bond' interaction characterized by a short intramolecular S···O contact of 2·697 Å and a C—S···O angle of 164·8°. These values fit well into the ranges 2·55–2·73 Å and 154–174° quoted for a number of compounds of the same pattern (O bonded to a nucleophilic moiety —C—Ph— and C counteratom to an O atom) by Kálmán & Párkányi (1980). The environment of the S atom is nearly coplanar as shown by the torsion angles given in Table 3 and by the least-squares

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52192 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

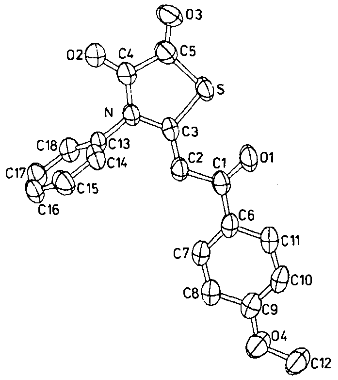

Fig. 2. The molecule with the atom-numbering scheme.

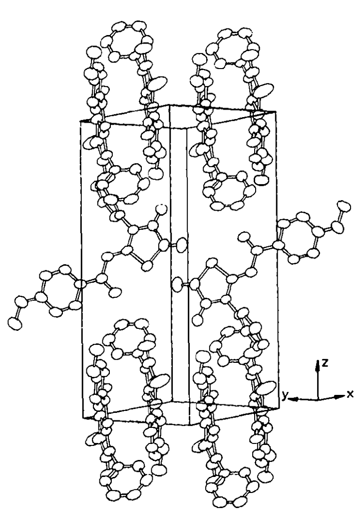

Fig. 3. Packing diagram.

Table 4. *The deviations* (Å) *of the atoms from the least-squares plane in the* (*carbonylmethylene*)*thiazolidine part of the molecule*

The equation of the plane is: $4{\cdot}874x + 8{\cdot}030y - 2{\cdot}663z = 1{\cdot}029$.

S	−0·060 (3)	C3	0·003 (4)
C5	−0·079 (4)	C2	−0·079 (4)
C4	0·063 (4)	C1	−0·111 (4)
N	0·082 (3)	O1	0·182 (3)

calculation (Table 4). The discussion of the close S···O contact and the quasi-linear C—S···O arrangement would be similar to that in our previous work quoted above. The fact that the O atom is replaced at C5 by the dicyanomethylidene group for compound *A* but not for compound *B*, can be explained by the existence of the electron-withdrawing forces due to S···O interaction.

The similarity of the structures of the Knoevenagel reaction substrate and product shows that the attractive forces due to sulfur–oxygen close contact can overcompensate the usual van der Waals repulsion between neighbouring nonbonded heteroatoms, thus controlling the conformation of compounds both in the solid state and, as it can be inferred, in solution.

This research was supported in part by the Polish Ministry of Science and Higher Education Project No. RP.II.13.2.14.

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Acta Cryst. (1990). **C46**, 103–106

Structure of 9,10-Dihydro-9,10-ethenoanthracene (Dibenzobarrelene)

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(*Received* 28 *February* 1989; *accepted* 9 *May* 1989)

Abstract. $C_{16}H_{12}$, $M_r = 204{\cdot}27$, monoclinic, $C2$, $a = 14{\cdot}467\,(1)$, $b = 8{\cdot}043\,(1)$, $c = 11{\cdot}734\,(1)$ Å, $\beta = 126{\cdot}16\,(1)^\circ$, $V = 1102{\cdot}2\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}231$ g cm^{-3}, Cu $K\alpha_1$, $\lambda = 1{\cdot}54056$ Å, $\mu = 4{\cdot}94$ cm^{-1}, $F(000) = 432$, $T = 295$ K, $R = 0{\cdot}034$ for 1106 unique observed reflexions with $I > 3\sigma(I)$. The molecule has approximate C_{2v} symmetry, with the three nearly planar sections at angles of about 120°. The isolated C=C double bond [1·316 (4) Å] is shorter than the equivalent bridge bond in the

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