is manifested more in bending of the aryl ring as opposed to distortion of C—S—C bond angles than is the case in related paracyclophanes.

We would like to thank Jane Bae for technical assistance in the preparation and crystal growth of compound (3). Acknowledgment is made to the donors of the Petroleum Research Fund, administered by the American Chemical Society, for support of this research. CJC would like to thank Professor Gene B. Carpenter of Brown University for helpful discussions regarding the space group assignment and Professor Paul G. Williard of Brown University for gracious assistance with data collection.

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Structure of 1-Amino-5-benzoyl-4-phenyl-1H-pyrimidine-2-thione

BY MEHMET AKKURT,* AYHAN GÜLDESTE AND HÜSEYİN SOYLU†

Department of Physics, Faculty of Arts and Sciences, Erciyes University, TR-38039 Kayseri, Turkey

AND BEHZAT ALTURAL AND EMİN SARIPINAR

Department of Chemistry, Faculty of Arts and Sciences, Erciyes University, TR-38039 Kayseri, Turkey

Abstract. $C_{17}H_{13}N_3OS$, $M_r = 307.38$, monoclinic, $P2_1/n$, $a = 9.712 (3)$, $b = 15.072 (3)$, $c = 10.713 (3)$ Å, $\beta = 107.65 (3)°$, $V = 1494.34$ Å^3, $Z = 4$, $D_m = 1.372$, $D_x = 1.366$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0.71069$ Å, $\mu = 2.11$ cm^{-1}, $F(000) = 640$, $T = 295$ K, final $R(F) = 0.0417$ for 2608 unique reflections. The pyrimidine ring is almost planar, the angle between the planes formed by the ring atoms N1—C6—C5 and C2—N3—C4 being 0.74°.

Introduction. Thiopyrimidines possess effective antibacterial, antifungal, antiviral, insecticidal and

* Author for correspondence.

† Present address: Department of Physics, Faculty of Arts and Sciences, Eastern Mediterranean University, G. Magusa, North Cyprus.

miticidal properties (Cheng, 1969; McNair-Scott, Ulbricht, Rogers, Chu & Rose, 1959; Sankyo Co. Ltd & Ube Industries Ltd, 1984; Ziegler, Eder, Belegratis & Prewedorakis, 1967; Akçamur, Altural, Sarıpınar, Kollenz, Kappe, Peters, Peters & von Schnering, 1988; Özbey, Kendi, Akçamur, Yildirim, Elerman & Soylu, 1991).

The furan-2,3-dione, (1), can easily be made from dibenzoylmethane and oxalyl dichloride (Akçamur, Penn, Ziegler, Sterk, Kollenz, Kappe, Peters, Peters & von Schnering, 1986) and with the thiosemicarbazones (2) it gives a number of 1,4,5-substituted $1H$-pyrimidine-2-thiones, (3) and (4), in moderate yields (30–60%) (see scheme). We have carried out an X-ray analysis of (5), a hydrolysis product of (3), in order to confirm the structure deduced from the IR and NMR spectra.
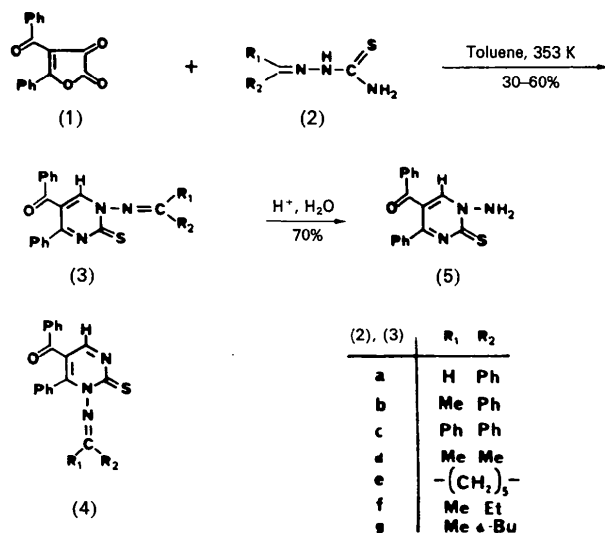

Table 1. *Atomic coordinates and equivalent isotropic temperature factors* $(\mathring{A}^2 \times 10^4)$ *with e.s.d.'s in parentheses*

Equivalent isotropic U is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N1	0.1210 (2)	0.5836 (1)	0.5118 (2)	330 (1)
C2	0.1508 (3)	0.5397 (2)	0.4088 (3)	345 (1)
N3	0.0947 (2)	0.5754 (2)	0.2877 (2)	347 (1)
C4	0.0159 (3)	0.6487 (2)	0.2683 (2)	300 (1)
C5	−0.0193 (3)	0.6921 (2)	0.3718 (2)	303 (1)
C6	0.0377 (3)	0.6555 (2)	0.4947 (3)	341 (1)
N10	0.1715 (3)	0.5472 (2)	0.6393 (2)	447 (1)
S20	0.2542 (1)	0.4490 (1)	0.4377 (1)	511 (1)
C41	−0.0305 (3)	0.6820 (2)	0.1306 (2)	339 (1)
C42	−0.0194 (3)	0.7705 (2)	0.1017 (3)	437 (2)
C43	−0.0603 (4)	0.7991 (2)	−0.0286 (3)	542 (2)
C44	−0.1124 (4)	0.7379 (3)	−0.1281 (3)	562 (2)
C45	−0.1222 (4)	0.6500 (3)	−0.1002 (3)	510 (2)
C46	−0.0801 (3)	0.6206 (2)	0.0297 (3)	394 (2)
O50	−0.2270 (2)	0.7751 (1)	0.2612 (2)	461 (1)
C50	−0.1212 (3)	0.7678 (2)	0.3578 (2)	338 (1)
C51	−0.0936 (3)	0.8318 (2)	0.4681 (2)	322 (1)
C52	0.0478 (3)	0.8555 (2)	0.5379 (3)	455 (2)
C53	0.0709 (4)	0.9151 (3)	0.6397 (4)	608 (2)
C54	−0.0421 (4)	0.9503 (2)	0.6742 (3)	591 (3)
C55	−0.1829 (4)	0.9272 (2)	0.6050 (3)	553 (2)
C56	−0.2081 (3)	0.8690 (2)	0.5014 (3)	429 (2)

Table 2. *Bond distances* $(\mathring{A})$ *and angles* (°) *with e.s.d.'s in parentheses*

N1—C2	1.390 (3)	C45—C46	1.399 (4)
N1—C6	1.332 (3)	C46—C41	1.393 (4)
N1—N10	1.414 (3)	C5—C6	1.379 (3)
C2—N3	1.357 (3)	C5—C50	1.489 (4)
C2—S20	1.669 (3)	C50—O50	1.222 (3)
N3—C4	1.324 (3)	C50—C51	1.486 (4)
C4—C5	1.415 (4)	C51—C52	1.396 (4)
C4—C41	1.492 (3)	C52—C53	1.378 (4)
C41—C42	1.381 (4)	C53—C54	1.367 (5)
C42—C43	1.398 (4)	C54—C55	1.387 (5)
C43—C44	1.384 (5)	C55—C56	1.378 (4)
C44—C45	1.367 (5)	C56—C51	1.384 (4)

C6—N1—N10	117.9 (2)	C44—C45—C46	120.4 (3)
N10—N1—C2	119.1 (2)	C45—C46—C41	119.3 (3)
C6—N1—C2	122.8 (2)	C4—C5—C50	125.9 (2)
N1—C2—S20	120.1 (2)	C50—C5—C6	117.7 (2)
S20—C2—N3	123.1 (2)	C4—C5—C6	116.2 (2)
N1—C2—N3	116.8 (2)	C5—C6—N1	120.5 (3)
C2—N3—C4	121.7 (2)	C5—C50—O50	121.1 (2)
N3—C4—C41	115.0 (2)	O50—C50—C51	121.2 (2)
C41—C4—C5	123.2 (2)	C5—C50—C51	117.6 (2)
N3—C4—C5	121.8 (2)	C50—C51—C52	120.2 (2)
C4—C41—C42	121.7 (2)	C50—C51—C56	120.1 (2)
C4—C41—C46	118.2 (2)	C52—C51—C56	119.7 (3)
C42—C41—C46	120.0 (3)	C51—C52—C53	119.2 (3)
C41—C42—C43	120.2 (3)	C52—C53—C54	121.1 (3)
C42—C43—C44	119.4 (3)	C53—C54—C55	120.0 (3)
C43—C44—C45	120.7 (3)	C54—C55—C56	119.7 (3)
		C55—C56—C51	120.3 (3)

Experimental. The compound was synthesized by the method of Akçamur *et al.* (1988). Colourless prismatic-shaped transparent crystals were recrystallized from acetic acid by slow evaporation at room temperature for a week. Density was measured by flotation in carbon tetrachloride and toluene. For X-ray examination and data collection, a suitable crystal of approximate dimensions $0.35 \times 0.50 \times 0.70$ mm was mounted in a glass capillary. Intensity data were collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation.

Unit-cell parameters were obtained by least-squares treatment of the angular settings of 25 reflections lying in a 2θ range 15–21°. Intensity data were collected in ω–2θ scan mode with $6 \leq 2\theta \leq 56°$, $-12 \leq h \leq 12, 0 \leq k \leq 19, 0 \leq l \leq 14$. Three standard reflections were measured every 4000 s of exposure time: no intensity variation. $R_{int} = 0.023$. A total of 3915 reflections were collected of which 2608 unique

reflections with $I \geq \sigma(I)$ were considered observed. Data were corrected for Lorentz–polarization effects and absorption (North, Phillips & Mathews, 1968) (empirical from ψ scans of four close-to-axial reflections), transmission factors: 96.81–99.95%. The structure was solved by direct methods (*SHELXS*86; Sheldrick, 1986) and refined by full-matrix least

squares on F (*SHELX*76; Sheldrick, 1976) with anisotropic thermal parameters for non-H atoms. H atoms were obtained from a ΔF map and refined with a common isotropic thermal parameter ($U =$ 0.066 Å^2). 238 parameters were refined. $R = wR =$ 0.0417, $w = 1$. In the last cycle $(\Delta/\sigma)_{max} = 0.343$. A final difference Fourier map revealed residual electron density between -0.61 and 0.35 e Å^{-3}.

Computer programs used: Enraf–Nonius *SDP* (B. A. Frenz & Associates Inc., 1985) (for data reduction on a PDP11/44 computer), *SHELX*76 (Sheldrick, 1976), *SHELXS*86 (Sheldrick, 1986), *ORTEP*II (Johnson, 1976), *HXDUA* (Aytaç, Soylu & Ülkü, 1973). Scattering factors for neutral atoms and f', f'' from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Discussion. Final positional and thermal parameters are presented in Table 1 and bond distances and angles in Table 2.* These values are comparable with those found for similar compounds, especially those in the (3c) form (Akçamur et al., 1988). A view of the molecule and the numbering scheme are shown in Fig. 1.

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* Tables of anisotropic thermal parameters, positional and thermal parameters for the H atoms, bond distances and angles involving the H atoms, and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54545 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU0155]

The C2—S20 bond distance of 1.669 (3) Å is a little longer than 1.61 Å, the distance expected for a C—S double bond (Pauling, 1960) and the N1—N10 bond distance of 1.414 (3) Å is unexceptional (cf. 1.434 (4) Å, Akçamur et al., 1988). In the pyrimidine ring the angle between the planes formed by the atoms N1—C6—C5 and C2—N3—C4 is 0.74°. This angle appears sensitive to the type of substituents present on N10 since in the related (3c) compound it is 5.3° (Akçamur et al., 1988) and 7.5° in 5-benzoyl-1-[4-(dimethylamino)phenylmethyleneamino]-4-phenyl-1H-pyrimidine-2-thione (Sarıpınar, 1990; Akkurt, Hiller, Sarıpınar, Akçamur & Soylu, 1992).

The X-ray structural determination of (5) confirms the formula suggested on the basis of NMR and IR spectroscopy.
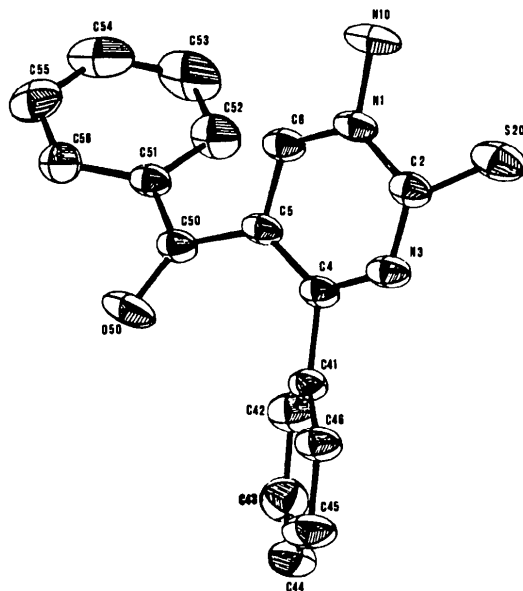
Fig. 1. An *ORTEP* (Johnson, 1976) drawing of the title molecule with the atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

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