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Structure of 2,8-Dithia[9](9,10)anthracenophane

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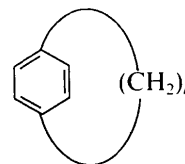
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Abstract. $C_{21}H_{22}S_2$, $M_r = 338.64$, monoclinic, $P2_1/n$, $a = 9.140$ (4), $b = 25.926$ (15), $c = 14.481$ (9) Å, $\beta = 93.55$ (4)°, $V = 3428.4$ Å³, $Z = 8$, $D_m = 1.29$, $D_x = 1.312$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.261$ mm⁻¹, $F(000) = 1440$, $T = 295$ K, $R = 0.0432$ for 3673 unique observed reflections. This represents the first crystal structure determination of an [n](9,10)anthracenophane. The two molecules in the asymmetric unit are nearly identical, have approximate C_2 symmetry and exhibit less-severe aryl ring distortion than predicted by molecular-mechanics calculations. The results of a comparison of selected angles in this structure with two other reported thiacyclophanes permits the strain in these molecules to be inferred.

Introduction. The current detailed picture of [n]paracyclophane (1) structure has evolved through more than 30 years of determined effort to prepare and characterize members of this family with shorter bridge lengths and correspondingly greater aryl ring distortion (Rosenfeld & Choe, 1983). That history is characterized by a wonderful interplay of computational chemistry and experimental structural probes, particularly NMR spectroscopy (Mitchell, 1983) and X-ray crystal structure determination

(Rosenfeld & Choe, 1983). This pattern, unfortunately, has not been repeated for benzologous systems though their structure and chemistry promise to be rich and decidedly different than the [n]paracyclophanes.

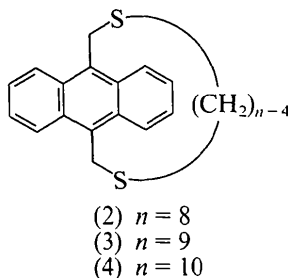


(1)

The smallest known [n](9,10)anthracenophane with a saturated aliphatic bridge has $n = 10$ (Vögtle & Koo Tze Mew, 1978). However, 3,5-diketo[8](9,10)anthracenophane (Wynberg & Helder, 1971; Rosenfeld & Sanford, 1987) and 2,7-dithia[8](9,10)anthracenophane (2) (Chung & Rosenfeld, 1983) have been reported and both undergo air oxidation on standing in solution at room temperature. This enhanced reactivity is presumably a result of bending of the central ring of the anthracene moiety and molecular-mechanics modeling suggests that (2)–(4) have central rings that are *more* distorted than the aryl rings of [n]paracyclophanes of corresponding n despite the substantially longer C—S bonds in the

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former series (Rosenfeld, Shedlow, Kirwin & Amaral, 1990). Firm evidence of geometric distortion is lacking since there are no [n](9,10)anthracenophane X-ray crystal structure determinations reported though a small number of more complex anthracenophanes have been described (Keehn, 1983; Toyoda, Kasai & Misumi, 1985; Toyoda & Misumi, 1978; Guinand, Marsau, Bouas, Castellan, Desvergne & Riffaud, 1986). For (2)–(4), the molecular-mechanics-derived minima have geometries with approximate C_2 symmetry but, again, experimental verification is unavailable. We now report the first X-ray crystal structure determination for an [n](9,10)anthracenophane, namely 2,8-dithia[9](9,10)-anthracenophane (3).



Experimental. Suitable pale-yellow crystals of (3) (Chung & Rosenfeld, 1983) were obtained by vapor diffusion using chloroform/pentane. The data crystal measured $0.30 \times 0.42 \times 0.50$ mm. Diffraction data were collected by $\theta/2\theta$ scans in air at ambient temperature on a Nicolet R3M/E four-circle autodiffractometer. Lattice parameters were determined by a least-squares fit of 25 machine-centered reflections between $25\text{--}35^\circ$ in 2θ .

4830 reflections were collected in the range of $3.6 \leq 2\theta \leq 45^\circ$, index range $0 \leq h \leq 9$, $0 \leq k \leq 27$, $0 \leq l \leq 15$, of which 4376 were unique and of those, 3673 had $I > 3\sigma(I)$ and were considered observed, $R_{\text{int}} = 0.028$. Three check reflections (006, 0,16,0, 0,14,1) were measured every 97 reflections. There was no significant deterioration of the check reflection intensities over the time of data collection. The data were corrected for Lorentz and polarization effects and a profile-analysis correction was applied. No correction for absorption was made.

The structure was solved by direct methods (MULTAN78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and refined (SHELX; Sheldrick, 1976) using block-diagonal least-squares methods. The initial E map revealed the positions of all four S atoms, attached C atoms, the intact anthracene of one molecule and 11 atoms of the second anthracene ring. Successive difference maps revealed the remaining C atoms and most of the H atoms. H atoms were placed at calculated positions (C—H = 1.08 \AA) and allowed to 'ride' on attached C

Table 1. Atomic coordinates and U_{eq} values ($\text{\AA}^2 \times 10^4$)

	x	y	z	U_{eq}
S1	0.61313 (8)	0.25711 (3)	0.14021 (5)	520 (3)
S2	0.00046 (8)	0.28269 (3)	0.35035 (5)	540 (3)
C1	0.57823 (28)	0.17891 (9)	0.33464 (18)	446 (9)
C2	0.60350 (32)	0.17849 (10)	0.42710 (19)	533 (11)
C3	0.48528 (33)	0.18252 (10)	0.48470 (19)	530 (11)
C4	0.34580 (30)	0.18760 (9)	0.44790 (16)	439 (10)
C5	-0.00197 (29)	0.19738 (10)	0.17013 (18)	481 (10)
C6	-0.02858 (33)	0.19393 (11)	0.07807 (19)	562 (11)
C7	0.08860 (34)	0.18748 (10)	0.02010 (19)	567 (13)
C8	0.22947 (32)	0.18423 (10)	0.05697 (17)	488 (12)
C9	0.40864 (26)	0.18520 (9)	0.19461 (16)	339 (8)
C10	0.16989 (26)	0.19713 (9)	0.30957 (16)	345 (9)
C11	0.14344 (26)	0.19335 (9)	0.21376 (16)	356 (9)
C12	0.26322 (27)	0.18683 (9)	0.15476 (16)	345 (9)
C13	0.43318 (25)	0.18325 (8)	0.29109 (16)	349 (9)
C14	0.31264 (26)	0.18878 (8)	0.34998 (15)	339 (8)
C15	0.53603 (28)	0.19160 (10)	0.13398 (17)	461 (10)
C16	0.04703 (28)	0.21418 (10)	0.36871 (17)	473 (10)
C17	0.45164 (29)	0.29796 (10)	0.13521 (17)	477 (10)
C18	0.40015 (28)	0.31250 (10)	0.22986 (17)	437 (10)
C19	0.25488 (30)	0.34172 (11)	0.22439 (17)	516 (9)
C20	0.20905 (31)	0.35889 (10)	0.32008 (18)	523 (11)
C21	0.17210 (29)	0.31496 (10)	0.38476 (16)	473 (10)
S1'	-0.08031 (8)	0.00720 (3)	0.85871 (5)	579 (3)
S2'	0.53889 (8)	0.03159 (3)	0.65249 (5)	549 (3)
C1'	-0.04413 (31)	-0.07262 (11)	0.66402 (20)	543 (12)
C2'	-0.06726 (35)	-0.07312 (12)	0.57190 (22)	657 (14)
C3'	0.04965 (37)	-0.06693 (12)	0.51388 (21)	638 (13)
C4'	0.18927 (33)	-0.06080 (10)	0.55165 (18)	511 (11)
C5'	0.53368 (28)	-0.05385 (10)	0.83130 (17)	451 (10)
C6'	0.55851 (32)	-0.05772 (11)	0.92355 (18)	527 (11)
C7'	0.44106 (33)	-0.06386 (10)	0.98055 (18)	530 (11)
C8'	0.30115 (30)	-0.06621 (10)	0.94309 (16)	465 (11)
C9'	0.12335 (26)	-0.06518 (9)	0.80431 (17)	377 (9)
C10'	0.36354 (27)	-0.05259 (9)	0.69097 (16)	353 (9)
C11'	0.38881 (26)	-0.05694 (9)	0.78721 (16)	346 (9)
C12'	0.26794 (26)	-0.06352 (9)	0.84535 (16)	351 (9)
C13'	0.10012 (27)	-0.06727 (9)	0.70821 (17)	384 (9)
C14'	0.22152 (28)	-0.06073 (9)	0.64965 (16)	395 (9)
C15'	-0.00559 (28)	-0.05883 (10)	0.86450 (19)	503 (11)
C16'	0.48775 (30)	-0.03609 (11)	0.63292 (17)	497 (11)
C17'	0.08287 (30)	0.04725 (11)	0.86457 (18)	505 (10)
C18'	0.13746 (28)	0.06171 (10)	0.77055 (17)	461 (10)
C19'	0.28297 (31)	0.09022 (11)	0.77780 (19)	525 (11)
C20'	0.33181 (33)	0.10863 (11)	0.68225 (19)	560 (11)
C21'	0.37044 (29)	0.06511 (11)	0.61676 (17)	488 (11)

atoms during refinement. Two common isotropic atomic displacement factors ($U = 0.085$ and 0.063 \AA^2) were assigned the aromatic and aliphatic H atoms, respectively. The function minimized was $\sum w(|F_o| - k|F_c|)^2$. C and S atoms were refined anisotropically. Convergence occurred at $R = 0.0432$, $wR = 0.0631$ with $S = 2.05$ and $w = 1.9649/[\sigma^2 F_o + 0.000823(F_o^2)]$, (σF_o from counting statistics), for 417 refined parameters. $(\Delta/\sigma)_{\text{max}} = 0.008$ [$S1(U_{22})$]; residual electron density in final difference map within $+0.17$ and -0.27 e \AA^{-3} , atomic scattering factors from SHELX76.

Geometrical calculations were performed with the program PARST (Nardelli, 1983). Final atomic coordinates are given in Table 1* and bond distances and angles are given in Table 2. All molecular-

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, selected torsion angles and least-squares-planes calculations have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54541 (21 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0299]

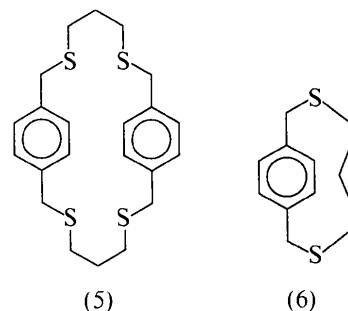
Table 2. Bond distances (Å) and bond angles (°)

S1—C15	1.8389 (28)	S1'—C15'	1.8431 (29)
S1—C17	1.8145 (28)	S1'—C17'	1.8150 (29)
S2—C16	1.8419 (29)	S2'—C16'	1.8334 (31)
S2—C21	1.8200 (28)	S2'—C21'	1.8151 (28)
C1—C2	1.3446 (39)	C1'—C2'	1.3377 (44)
C1—C13	1.4368 (34)	C1'—C13'	1.4362 (37)
C2—C3	1.4092 (43)	C2'—C3'	1.4088 (47)
C3—C4	1.3574 (40)	C3'—C4'	1.3662 (44)
C4—C14	1.4319 (33)	C4'—C14'	1.4314 (36)
C5—C6	1.3432 (38)	C5'—C6'	1.3448 (37)
C5—C11	1.4396 (35)	C5'—C11'	1.4362 (35)
C6—C7	1.4111 (43)	C6'—C7'	1.4031 (42)
C7—C8	1.3656 (42)	C7'—C8'	1.3592 (40)
C8—C12	1.4321 (35)	C8'—C12'	1.4308 (34)
C9—C12	1.4165 (34)	C9'—C12'	1.4157 (34)
C9—C13	1.4021 (34)	C9'—C13'	1.3957 (36)
C9—C15	1.5102 (36)	C9'—C15'	1.5175 (37)
C10—C11	1.3969 (34)	C10'—C11'	1.4030 (34)
C10—C14	1.4132 (33)	C10'—C14'	1.4112 (35)
C10—C16	1.5195 (36)	C10'—C16'	1.5155 (37)
C11—C12	1.4398 (35)	C11'—C12'	1.4400 (35)
C13—C14	1.4415 (34)	C13'—C14'	1.4476 (37)
C17—C18	1.5238 (37)	C17'—C18'	1.5256 (38)
C18—C19	1.5265 (38)	C18'—C19'	1.5195 (39)
C19—C20	1.5381 (38)	C19'—C20'	1.5550 (41)
C20—C21	1.5257 (37)	C20'—C21'	1.5292 (40)
C15—S1—C17	103.23 (19)	C15'—S1'—C17'	103.19 (20)
C16—S2—C21	102.49 (19)	C16'—S2'—C21'	102.15 (19)
C2—C1—C13	122.32 (33)	C2'—C1'—C13'	121.96 (35)
C1—C2—C3	119.87 (34)	C1'—C2'—C3'	120.98 (37)
C2—C3—C4	120.71 (33)	C2'—C3'—C4'	119.91 (35)
C3—C4—C14	121.80 (34)	C3'—C4'—C14'	121.80 (35)
C6—C5—C11	122.43 (33)	C6'—C5'—C11'	122.12 (33)
C5—C6—C7	120.06 (35)	C5'—C6'—C7'	120.31 (34)
C6—C7—C8	120.45 (33)	C6'—C7'—C8'	120.36 (32)
C7—C8—C12	121.59 (35)	C7'—C8'—C12'	121.96 (34)
C13—C9—C15	120.11 (30)	C13'—C9'—C15'	120.08 (31)
C12—C9—C15	119.84 (29)	C12'—C9'—C15'	119.57 (30)
C12—C9—C13	119.68 (31)	C12'—C9'—C13'	120.01 (32)
C14—C10—C16	120.45 (29)	C14'—C10'—C16'	120.53 (29)
C11—C10—C16	119.45 (30)	C11'—C10'—C16'	119.39 (30)
C11—C10—C14	119.94 (30)	C11'—C10'—C14'	119.98 (30)
C5—C11—C10	121.94 (32)	C5'—C11'—C10'	121.85 (32)
C10—C11—C12	120.43 (31)	C10'—C11'—C12'	120.35 (31)
C5—C11—C12	117.60 (29)	C5'—C11'—C12'	117.79 (29)
C9—C12—C11	119.38 (30)	C9'—C12'—C11'	119.22 (30)
C8—C12—C11	117.85 (31)	C8'—C12'—C11'	117.43 (31)
C8—C12—C9	122.73 (31)	C8'—C12'—C9'	123.31 (31)
C1—C13—C9	121.77 (31)	C1'—C13'—C9'	121.79 (33)
C9—C13—C14	120.33 (30)	C9'—C13'—C14'	120.31 (31)
C1—C13—C14	117.80 (29)	C1'—C13'—C14'	117.78 (30)
C10—C14—C13	119.35 (29)	C10'—C14'—C13'	119.17 (30)
C4—C14—C13	117.46 (30)	C4'—C14'—C13'	117.54 (31)
C4—C14—C10	123.14 (30)	C4'—C14'—C10'	123.24 (31)
S1—C15—C9	112.33 (23)	S1'—C15'—C9'	111.92 (23)
S2—C16—C10	111.80 (23)	S2'—C16'—C10'	112.15 (24)
S1—C17—C18	113.85 (25)	S1'—C17'—C18'	114.34 (26)
C17—C18—C19	113.09 (28)	C17'—C18'—C19'	113.02 (28)
C18—C19—C20	112.42 (29)	C18'—C19'—C20'	112.71 (29)
C19—C20—C21	114.84 (27)	C19'—C20'—C21'	114.54 (28)
S2—C21—C20	113.26 (25)	S2'—C21'—C20'	113.63 (26)

mechanics calculations were performed using the computer programs *PCMODEL* (*MMX88* force field incorporated) and *PCDISPLAY* (Serena Software, 1989).

Discussion. The two independent molecules of the asymmetric unit have approximate C_2 symmetry and are nearly identical. In fact, in a comparison of the two structures, the r.m.s.d. of all atoms is only 0.027 Å. A *PLUTO* (Motherwell & Clegg, 1978) drawing of the two molecules in the asymmetric unit in which a pseudosymmetric twofold screw axis is evident is shown in Fig. 1. As predicted by previous molecular-mechanics calculations (Rosenfeld, Shed-

low, Kirwin & Amaral, 1990), the anthracene ring in (3) deviates significantly from planarity with the least-squares planes of the outer six-membered rings, having dihedral angles of 3.7 (1) and 4.0 (1)° within each independent molecule. There are apparently no reported structures for dithia[9]paracyclophanes or dithia[9](1,4)naphthalenophanes that might offer comparisons of analogous bond lengths and angles in the bridge. However, Pfisterer & Ziegler (1983) have described a pair of thiacyclophanes, one an unstrained 2,6,15,19-tetrathia[7.7]paracyclophane (5) in which two 1,4-xylyl units are linked by a pair of 1,3-dithiaprolylene segments and the other a strained 2,6-dithia[7]paracyclophane (6) in which one 1,4-xylyl unit is spanned by a single 1,3-dithiaprolylene bridge. From a comparison of selected angles in these two compounds with the two molecules of the current study, a measure of strain in each compound may be inferred. The relevant angles are defined in Fig. 2 and listed in Table 3.



The angles α and β and the inferred strain of the two independent conformers of the title compound [(3) and (3')] lie nearly midway between the unstrained (5) and the significantly strained (6). The C—S—C bond angles in these substances also reveal an intermediate level of strain in (3) and (3') compared to (5) and (6) but are much closer to

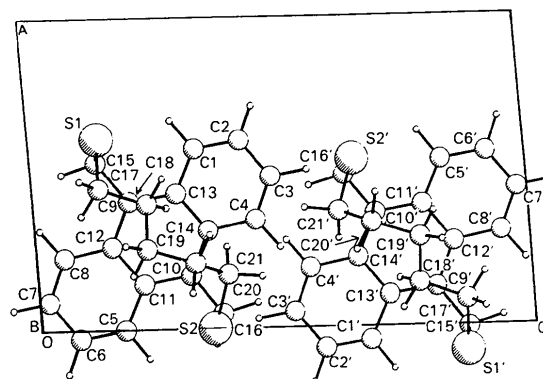


Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing of the two molecules of (3) in the asymmetric unit, showing the crystallographic numbering scheme.

Table 3. Comparison of analogous angles ($^{\circ}$) in compounds (3), (5) and (6)

Compound	α_1	α_2	β_1	β_2	C—S—C		(Average)
(5)	0.2 (3)	0.9 (3)	0.6 (3)	0.2 (3)	101.4 (2)	102.9 (2)	(102.2)
(6)	10.7 (4)	12.0 (4)	11.2 (4)	10.6 (5)	105.1 (3)	106.3 (3)	(105.7)
(3)	6.6 (2)	6.7 (2)	6.0 (3)	4.0 (3)	103.2 (2)	102.5 (2)	(102.9)
(3')	7.3 (3)	6.5 (2)	5.7 (3)	3.1 (3)	103.2 (2)	102.2 (2)	(102.7)

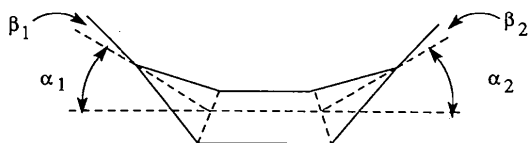
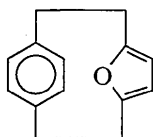


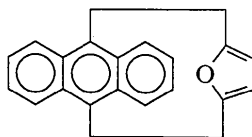
Fig. 2. Definition of the comparison angles listed in Table 3.

unstrained (5). The C—S—C—C torsion angles in (3) and (3') are all nearly *gauche* as expected from other studies (Desper, Powell & Gellman, 1990; Wolf, Hartman, Storey, Foxman & Cooper, 1987). It is interesting that (6) has a less-bent aryl ring and a more-distorted benzylic bond than does a carboxy derivative of the corresponding all-carbon [7]paracyclophane ($\alpha = 16.8$, $\beta = 6.8^{\circ}$) (Allinger, Walter & Newton, 1974).

Comparisons of paracyclophanes and (9,10)anthracenophanes suffer from a lack of information regarding the relative ease of bending benzene and anthracene rings. A useful calibration is, however, available by comparison of the data from X-ray crystallographic analysis of [2.2](2,5)furanoparacyclophane (7) and its anthracene analogue (8) (Keehn, 1983; Halvorson, Foxman, Keehn & Rosenfeld, 1983). Cyclophane (8) is, in fact, more puckered than (7) as demonstrated by the respective folding angles* of 18.4 and 15.7 $^{\circ}$. Our own molecular-mechanics calculations produce geometries in fairly close agreement with the crystal structures, but over-emphasize the aryl-ring distortion. The r.m.s.d. of the six atoms of the benzene ring plus the two benzylic C atoms in the molecular-mechanics structure and crystal structure of (7) is 0.058 Å. The analogous comparison for (8) is 0.053 Å.



(7)



(8)

* The folding angle is $180^{\circ} -$ (the dihedral angle between the least-squares planes defined by C atoms 1,2,3,4 and 1,4,5,6, respectively, of the benzene ring) for (7) and the analogous angle for (8).

The molecular-mechanics-derived geometry previously reported for (3) (Rosenfeld, Shedlow, Kirwin & Amaral, 1990) has an anthracene ring in which the r.m.s.d. of all atoms compared to the crystal structure is 0.084 Å. The significant difference is that the molecular-mechanics structure has a substantially more severe bend in the anthracene ring, 12.2 (α) vs 6.6 $^{\circ}$. The molecular-mechanics structure also deviates from the crystal structure in having a nearly undistorted benzylic bond (β). When the bridge atoms are included in the comparison, the r.m.s.d. is 0.387 Å owing to significant differences in some bridge dihedral angles. (A stereo depiction of the two overlapped structures is shown in Fig. 3.) This last result demonstrates that the reported molecular-mechanics-derived geometry does not represent a global minimum. Ethane-like staggering of bridge H atoms is possible in at least two bridge conformations that are of similar energy: the conformation in the crystal, after molecular-mechanics minimization, has a heat of formation that is only 0.1 kcal mol $^{-1}$ (0.42 kJ mol $^{-1}$) lower than that of the previously reported molecular-mechanics minimum. Molecular-mechanics minimization using the crystal structure coordinates leads to a structure with an essentially identical bridge conformation and an r.m.s.d. from the original coordinates for all atoms of 0.121 Å. The *MNDO* ionization potential of 8.18 e V* (Dewar & Thiel, 1977), is the same as that reported previously.

The X-ray crystal structure analysis of (3) confirms that the aryl rings in this compound are significantly bent and that the molecular symmetry is approximately C_2 . Both findings are in general agreement with previously reported molecular-mechanics calculations for (3) and related compounds although those calculations predict a more severely distorted anthracene ring than was observed experimentally. A comparison of the crystallographic data for (3) and two dithiaparacyclophanes suggests that strain in (3)

* A single SCF calculation was performed on this molecular-mechanics geometry using *MOPAC* (Stewart, 1983).

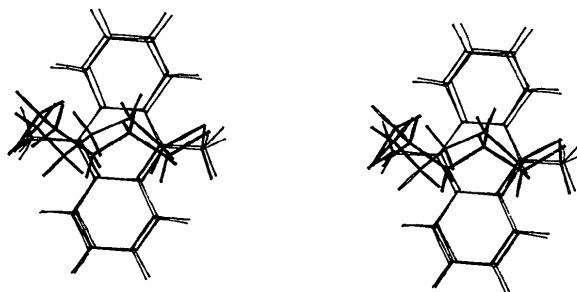


Fig. 3. Stereo representation of the crystal structure geometry of (3) superimposed on the geometry located independently by molecular-mechanics procedures.

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.

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

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Abstract. C₁₇H₁₃N₃OS, $M_r = 307.38$, monoclinic, $P2_1/n$, $a = 9.712$ (3), $b = 15.072$ (3), $c = 10.713$ (3) Å, $\beta = 107.65$ (3)°, $V = 1494.34$ Å³, $Z = 4$, $D_m = 1.372$, $D_x = 1.366$ g cm⁻³, $\text{Mo } K\alpha$, $\lambda = 0.71069$ 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°.

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Introduction. Thiopyrimidines possess effective anti-bacterial, antifungal, antiviral, insecticidal and