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1-Acetyl-3,4-dimethyl-6-phenylacetyl-3a,4,6,6a-tetrahydroimidazo[4,5-*d*]-imidazole-2,5(1*H*,3*H*)-dithione

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Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.049
wR factor = 0.135
Data-to-parameter ratio = 16.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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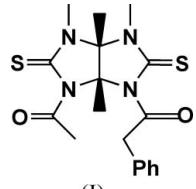
In the title compound, $C_{18}H_{22}N_4O_2S_2$, each of the five-membered rings of the glycoluril system has an envelope conformation. Weak intermolecular C—H···S hydrogen bonding helps to stabilize the crystal structure.

Comment

The title compound, (I), is a glycoluril derivative; these are used in industry as bleaching activators and explosives (Hofmann *et al.*, 1990). We report here the crystal structure of (I) to confirm the molecular structure.



(I)

The molecular structure of (I) is shown in Fig. 1. Each of the five-membered rings of the glycoluril system has an envelope conformation as indicated by the puckering parameters (Cremer & Pople, 1975) [q_2 = 0.463 (2) Å and φ_2 = 321.4 (2)°, and q_2 = 0.699 (3) Å and φ_2 = 324.2 (2)°]. Geometric parameters are normal.

Weak intermolecular C—H···S hydrogen bonding (Table 1) helps to stabilize the crystal structure.

Experimental

To a solution of 1-acetyl-3,4,7,8-tetramethyldithioglycoluril in dichloromethane (15 ml per 2 mmol) was added 1.5 equivalents of proton sponge. The mixture was stirred for 30 min. Phenylacetyl chloride (2 equiv.) was then added and stirring was continued for 1 h at room temperature. After quenching the reaction mixture with 5% HCl solution, the organic layer was washed with three portions of water. The resulting mixture was extracted three times with $CHCl_3$. The organic extracts were dried over anhydrous Na_2SO_4, filtered, and concentrated. The residue was subjected to flash chromatography on silica gel with MeOH/$CHCl_3$ (1:99) to yield (I). Single crystals of (I) were obtained by solvent evaporation of a chloroform solution.

Crystal data

$C_{18}H_{22}N_4O_2S_2$
M_r = 390.52
Monoclinic, $C2/c$
a = 25.161 (4) Å
b = 9.3806 (15) Å
c = 16.702 (3) Å
β = 109.370 (3)°
V = 3718.9 (10) Å^3
Z = 8
D_x = 1.395 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.31 mm^{-1}
T = 173 (2) K
Block, colorless
0.47 × 0.25 × 0.25 mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.869, T_{max} = 0.927
9828 measured reflections
4063 independent reflections
3033 reflections with $I > 2\sigma(I)$
R_{int} = 0.024
θ_{max} = 27.1°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.135
S = 1.05
4063 reflections
240 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0733P)^2 + 2.7496P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.54 e Å^{-3}
$\Delta\rho_{min}$ = −0.23 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C12—H12B···S2^{i}	0.99	2.99	3.957 (3)	166

Symmetry code: (i) $-x+1, -y+1, -z+1$.

H atoms were placed in calculated positions, with C—H = 0.98 (methyl), 0.95 (aromatic) and 0.99 Å (methylene). They were refined as riding with and U_{iso}(H) = xU_{eq}(carrier atom), where x = 1.5 for methyl H atoms and 1.2 for all others.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1999); software used to prepare material for publication: *SHELXTL*.

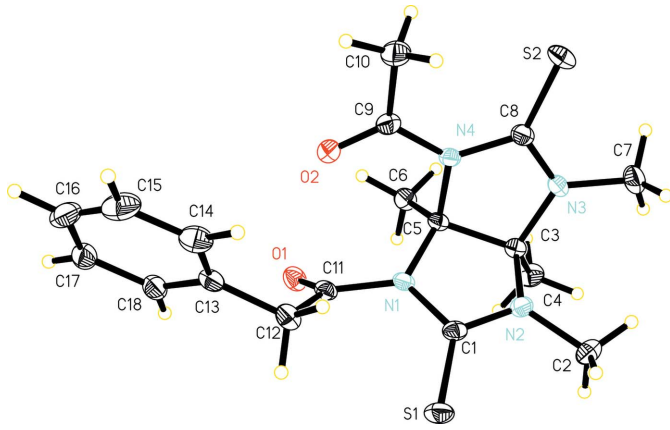


Figure 1
The molecular structure of (I), with 30% probability displacement ellipsoids (arbitrary spheres for H atoms).

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