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3,5-Dinitrosalicylic acid-phenazine (1/1)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.059
wR factor = 0.132
Data-to-parameter ratio = 15.4

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

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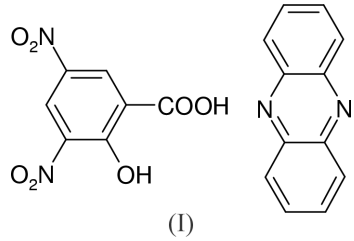
In the 1:1 molecular complex of 3,5-dinitrosalicylic acid and phenazine, $C_7H_4N_2O_7\cdot C_{12}H_8N_2$, the carboxylic acid group forms an O—H···N hydrogen bond to only one of the two heterocyclic N atoms. The structure is also stabilized extensively by C—H···O hydrogen bonds.

Comment

The carboxylic acid functionality is well known to form molecular complexes with compounds having strong acceptor groups, such as N and O (Palmore *et al.*, 1999). Recently, this concept was invoked in the cocrystallization experiment of simple alkane dicarboxylic acids with phenazine (Batchelor *et al.*, 2000), where both N atoms of the phenazine molecule are involved in O—H···N hydrogen bonds. Here, we have cocrystallized phenazine with 3,5-dinitrosalicylic acid.



(I)

The molecular geometry of the title compound, (I), is shown in Fig. 1 (*ORTEP*II; Johnson, 1976). The carboxylic acid group forms O—H···N hydrogen bonds on one side of the phenazine molecule, while the other N atom was found to be inactive. The emphasis of this crystal structure is on the structural attributes of C—H···O hydrogen bonding (Desiraju & Steiner, 1999). In the crystal structure, each 3,5-dinitrosalicylic acid molecule is connected to the phenazine molecule by C—H···O interactions and forms 2_1-screw-related ribbons parallel to [044] and [0$\bar{4}$4]. These two ribbons are connected by an O—H···N (1.76 Å and 161°) and an auxiliary C—H···O (2.78 Å and 143°) hydrogen bond to make a two-dimensional grid structure. These grids are further connected by C—H···O hydrogen bonds, forming another such grid. The packing diagram of the molecular complex is shown in Fig. 2. The occurrence of C—H···O hydrogen bonds follows from the presence of activated C—H groups in the constituent molecules.

Experimental

Yellow crystals of the 1:1 molecular complex of 3,5-dinitrosalicylic acid and phenazine were obtained when a 2:1 mixture of 3,5-dinitrosalicylic acid and phenazine was kept for crystallization in acetonitrile solvent.

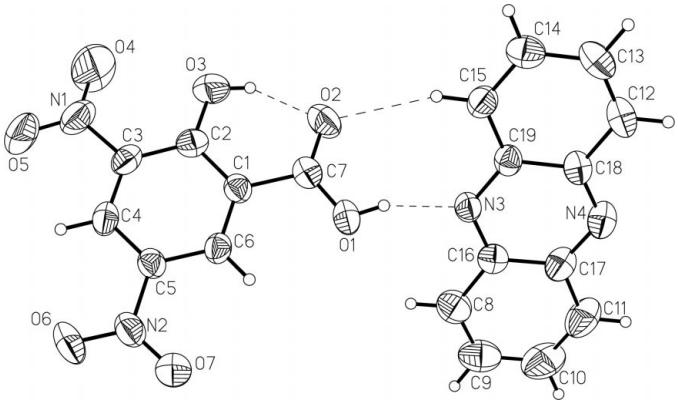


Figure 1
View of the title molecular complex, with the atom-numbering scheme. Displacement ellipsoids for non-H atoms are drawn at the 50% probability level.

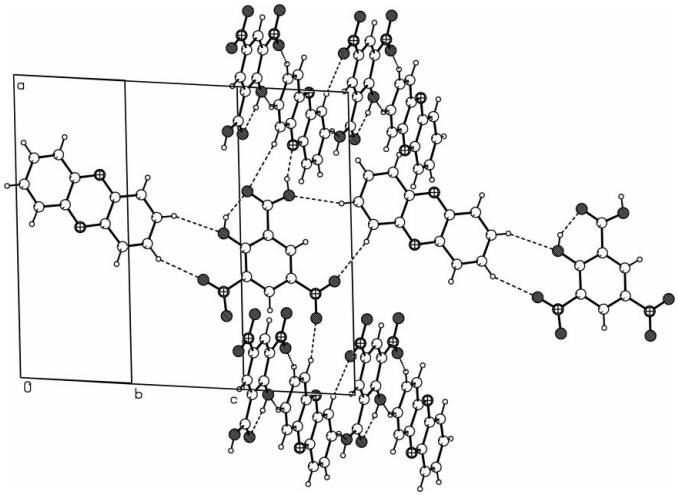


Figure 2
Packing diagram of the molecular complex, showing the C—H···O hydrogen-bonded ribbons elongated parallel to the [044] plane.

Crystal data

$C_7H_4N_2O_7\cdot C_{12}H_8N_2$
M_r = 408.33
Monoclinic, $P2_1/a$
a = 14.8002 (15) Å
b = 7.4029 (16) Å
c = 16.0091 (16) Å
β = 96.395 (8)°
V = 1743.1 (5) Å^3
Z = 4
D_x = 1.556 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 5–12°
μ = 0.12 mm^{-1}
T = 293 (2) K
Rhomb, yellow
0.36 × 0.34 × 0.26 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
w scans
Absorption correction: none
8396 measured reflections
4202 independent reflections
1930 reflections with $I > 2\sigma(I)$
R_{int} = 0.056
θ_{max} = 28.0°
$h = 0 \rightarrow 19$
$k = -9 \rightarrow 9$
$l = -21 \rightarrow 21$
3 standard reflections every 150 reflections
frequency: 90 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.059
$wR(F^2)$ = 0.132
S = 0.92
4202 reflections
273 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0573P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O1—H1A···N3	0.82	1.76	2.554 (3)	161
C10—H10A···O1^{i}	0.93	2.66	3.550 (4)	161
C11—H11A···O7^{i}	0.93	2.62	3.524 (3)	163
C13—H13A···O4^{ii}	0.93	2.49	3.332 (4)	151
C14—H14A···O3^{ii}	0.93	2.66	3.529 (3)	155
C15—H15A···O2	0.93	2.78	3.566 (3)	143

Symmetry codes: (i) $\frac{3}{2} - x, y - \frac{1}{2}, -z$; (ii) $\frac{3}{2} - x, \frac{1}{2} + y, 1 - z$.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *Software*; data reduction: *Xtal*3.5 (Hall *et al.*, 1995); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *PLUTON* (Spek, 2000); software used to prepare material for publication: *SHELXL*97.

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References

Batchelor, E., Klinowski, J. & Jones, W. (2000). *J. Mater. Chem.* **10**, 839–848.
Desiraju, G. R. & Steiner, T. (1999). *The Weak Hydrogen Bond in Structural Chemistry and Biology*. Oxford University Press.
Enraf–Nonius (1989). *CAD*-4 *Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Hall, S. R., Flack, H. D. & Stewart, J. M. (1995). Editors. *Xtal*3.5 *Reference Manual*. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Palmore, G. T. R., Luo, T. M., McBride-Wieser, M. T., Picciotto, E. T. & Reynoso-Paz, C. M. (1999). *Chem. Mater.* **11**, 3315–3328.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2000). *PLUTON*. Utrecht University, The Netherlands.