Filippakis, S. E., Leiserowitz, L., Rabinovich, D. & Schmidt, G. M. J. (1972). *J. Chem. Soc. Perkin Trans.* 2, pp. 1750–1756.
Hall, S. R., Flack, H. D. & Stewart, J. M. (1992). Editors. *Xtal*3.2 *Reference Manual*. Universities of Western Australia, Australia, Geneva, Switzerland, and Maryland, USA.
He, Y., Shi, J. & Su, G. (1993). *Acta Cryst.* C**49**, 1432–1434.
Johnson, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
Keller, E. (1988). *SCHAKAL88. Fortran Program for the Graphic Representation of Molecular and Crystallographic Models*. University of Freiburg, Germany.
Kim, M. J., Mang, J. Y. & Suh, I. H. (1988). *J. Korean Phys. Soc.* **21**, 405.
Liangfeng, Z., Yonghua, L., Baoling, L., Biyao, L. & Nianhe, X. (1993). In *Aromatic Plants and Essential Constituents*. South China Institute of Botany, Chinese Academy of Sciences. Hai Feng Publishing Co., Hong Kong. (Chinese National Node for APINMAP.)
Wierda, D. A., Feng, T. L. & Barron, A. R. (1989). *Acta Cryst.* C**45**, 338–339.
Zachariasen, W. H. (1967). *Acta Cryst.* **23**, 558–564.

*Acta Cryst.* (1996). C**52**, 1257–1258

# 4-Hydroxyantipyrine

KALIYAMOORTHY PANNEERSELVAM, NARAYANAN JAYANTHI, ENRIQUE RUDIÑO-PIÑERA AND MANUEL SORIANO-GARCÍA*

*Instituto de Química,† Circuito Exterior, Ciudad Universitaria, Delegación Coyoacán, México DF 04510, México. E-mail: soriano@servidor.unam.mx*

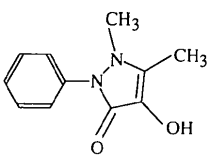
*(Received 26 June 1995; accepted 13 November 1995)*

## Abstract

The molecular structure of the title compound, 1,2-dihydro-4-hydroxy-1,5-dimethyl-2-phenyl-3*H*-pyrazol-3-one, $C_{11}H_{12}N_2O_2$, has been determined. Both rings are planar and make a dihedral angle of 42.5 (1)° with each other. In the crystal structure, the molecule is stabilized as a centrosymmetric hydrogen-bonded dimer. There is no conjugation between the phenyl and pyrazole rings.

## Comment

The crystal and molecular structure of the title compound, (I), has been investigated in order to determine the conformation and crystal packing, and also to confirm its stereochemistry.




(I)

† Contribution No. 1356 of the Instituto de Química, UNAM.

A view of (I) with the numbering scheme is shown in Fig. 1. Bond lengths within the molecule correspond with the average C—C distance for a phenyl ring [1.378 (3) Å] and the angles are normal. The molecule consists of a phenyl (C1–C6) and a pyrazole ring (C7–C9, N1, N2), which are planar within 0.007 and 0.026 Å, respectively, and form an interplanar angle of 42.5 (1)°.

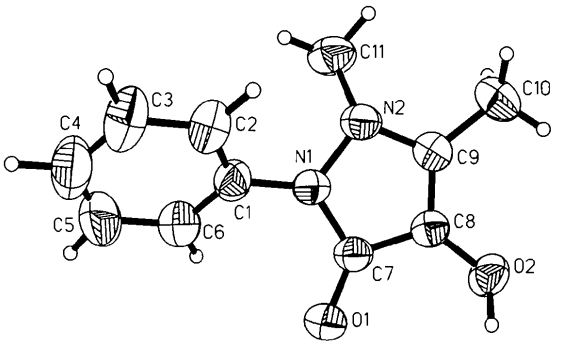



Fig. 1. The molecular structure of the title compound with the atom-labelling scheme and 50% probability displacement ellipsoids.

Fig. 2 shows a projection along the *c* axis. A hydrogen bond between the O2 hydroxy group and the carbonyl O1 atom of a neighbouring molecule constitutes the major intermolecular interaction and packing force. The two molecules are linked by a pair of O2—HO2···O1 hydrogen bonds across a crystallographic centre of inversion located at $(-x, -y, 1-z)$. The

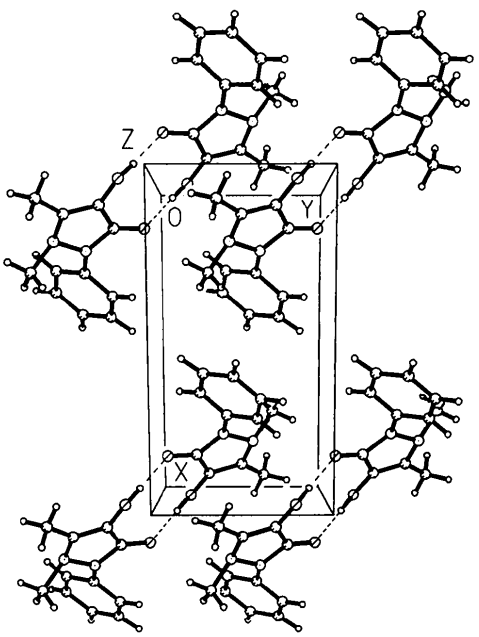



Fig. 2. A unit-cell drawing of the packing arrangement, with dashed lines indicating O—H···O intermolecular hydrogen-bonding interactions.

O2···O1 and HO2···O1 distances are 2.601 (2) and 1.66 (3) Å, respectively, and the O2—HO2···O1 angle is 161 (3)° (Allen, Kennard & Taylor, 1983). There are three intermolecular C—H···O non-bonded interactions in the range 3.40–3.58 Å (Desiraju, 1991).

## Experimental

The title compound was purchased from the Aldrich Chemical Co. Ltd and recrystallized from acetone at room temperature (m.p. 457–459 K).

*Crystal data*

$C_{11}H_{12}N_2O_2$
$M_r$ = 204.23
Monoclinic
$P2_1/n$
$a$ = 12.461 (6) Å
$b$ = 6.454 (4) Å
$c$ = 13.621 (5) Å
$\beta$ = 107.57 (1)°
$V$ = 1044.3 (9) Å$^3$
$Z$ = 4
$D_x$ = 1.299 Mg m$^{-3}$
$D_m$ = 1.300 Mg m$^{-3}$
$D_m$ measured by flotation in benzene/bromoform
Cu $K\alpha$ radiation
$\lambda$ = 1.54178 Å
Cell parameters from 25 reflections
$\theta$ = 10–25°
$\mu$ = 0.747 mm$^{-1}$
$T$ = 293 (2) K
Needle
0.35 × 0.20 × 0.20 mm
Yellow

*Data collection*

Siemens $P4$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
1930 measured reflections
1383 independent reflections
1253 observed reflections [$I > 2\sigma(I)$]
$R_{int}$ = 0.0452
$\theta_{max}$ = 56.74°
$h = -13 \rightarrow 12$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 14$
3 standard reflections monitored every 100 reflections
intensity decay: 3%

*Refinement*

Refinement on $F^2$
$R(F)$ = 0.0470
$wR(F^2)$ = 0.1226
$S$ = 1.073
1383 reflections
184 parameters
All H atoms refined isotropically
$w = 1/[\sigma^2(F_o^2) + (0.0786P)^2 + 0.2073P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.002
$\Delta\rho_{max}$ = 0.155 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.311 e Å$^{-3}$
Extinction correction: none
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å$^2$)*

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

| | $x$ | $y$ | $z$ | $U_{eq}$ |
|---|---|---|---|---|
| C1 | −0.2760 (2) | 0.4135 (3) | 0.5394 (1) | 0.0509 (5) |
| C2 | −0.2668 (2) | 0.5901 (4) | 0.5983 (2) | 0.0639 (6) |
| C3 | −0.3310 (2) | 0.6092 (5) | 0.6649 (2) | 0.0824 (8) |
| C4 | −0.4017 (2) | 0.4526 (6) | 0.6731 (2) | 0.0926 (9) |
| C5 | −0.4095 (2) | 0.2764 (6) | 0.6155 (3) | 0.0927 (9) |
| C6 | −0.3477 (2) | 0.2553 (4) | 0.5461 (2) | 0.0709 (7) |
| C7 | −0.1438 (2) | 0.2283 (3) | 0.4631 (1) | 0.0456 (5) |
| C8 | −0.0789 (1) | 0.3010 (3) | 0.3998 (1) | 0.0439 (5) |
| C9 | −0.1074 (2) | 0.4987 (3) | 0.3728 (1) | 0.0454 (5) |
| C10 | −0.0670 (2) | 0.6359 (4) | 0.3040 (2) | 0.0648 (6) |
| C11 | −0.2824 (2) | 0.6875 (4) | 0.3605 (2) | 0.0644 (6) |
| N1 | −0.2121 (1) | 0.3914 (2) | 0.4692 (1) | 0.0487 (5) |
| N2 | −0.1853 (1) | 0.5665 (2) | 0.4199 (1) | 0.0478 (5) |
| O1 | −0.1418 (1) | 0.0589 (2) | 0.5077 (1) | 0.0632 (5) |
| O2 | −0.0038 (1) | 0.1854 (2) | 0.3688 (1) | 0.0582 (5) |

Table 2. *Selected geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| C1—C6 | 1.377 (3) | C7—N1 | 1.372 (2) |
| C1—C2 | 1.379 (3) | C7—C8 | 1.428 (3) |
| C1—N1 | 1.424 (3) | C8—C9 | 1.346 (3) |
| C2—C3 | 1.384 (3) | C9—N2 | 1.387 (2) |
| C3—C4 | 1.367 (4) | C9—C10 | 1.483 (3) |
| C4—C5 | 1.368 (5) | C11—N2 | 1.462 (3) |
| C5—C6 | 1.395 (4) | N1—N2 | 1.406 (2) |
| C7—O1 | 1.247 (2) | | |
| C6—C1—C2 | 121.1 (2) | C9—C8—C7 | 109.0 (2) |
| C6—C1—N1 | 118.4 (2) | O2—C8—C7 | 125.1 (2) |
| C2—C1—N1 | 120.6 (2) | C8—C9—N2 | 109.7 (2) |
| C1—C2—C3 | 119.5 (2) | C8—C9—C10 | 128.8 (2) |
| C4—C3—C2 | 120.1 (3) | N2—C9—C10 | 121.5 (2) |
| C3—C4—C5 | 120.3 (2) | C7—N1—N2 | 110.3 (2) |
| C4—C5—C6 | 120.8 (3) | C7—N1—C1 | 126.9 (2) |
| C1—C6—C5 | 118.3 (3) | N2—N1—C1 | 120.1 (1) |
| O1—C7—N1 | 124.3 (2) | C9—N2—N1 | 105.6 (1) |
| O1—C7—C8 | 130.6 (2) | C9—N2—C11 | 119.4 (2) |
| N1—C7—C8 | 105.1 (2) | N1—N2—C11 | 114.7 (2) |
| C9—C8—O2 | 125.8 (2) | | |

Data collection: Siemens $P4$ diffractometer software. Cell refinement: *XSCANS* (Siemens, 1992). Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990*a*). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990*b*). Software used to prepare material for publication: *SHELXL*93.

KP acknowledges the CONACYT (Cátedra Patrimonial Nivel II) for fellowship assistance. The authors thank the Instituto de Biotecnología, UNAM, for data collection.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1148). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

## References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
Desiraju, G. R. (1991). *Acc. Chem. Res.* **24**, 290–296.
Sheldrick, G. M. (1990*a*). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1990*b*). *SHELXTL-Plus. Structure Determination Software Programs*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.
Siemens (1992). *XSCANS. X-ray Single Crystal Analysis System*. Version 2.0. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.