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Key indicators

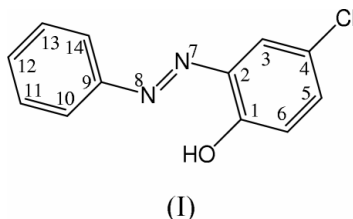
Powder X-ray study
T = 295 K
Mean $\sigma(\text{C}-\text{C}) = 0.008 \text{ \AA}$
R factor = 0.027
wR factor = 0.036For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

4-Chloro-2-(phenyldiazenyl)phenol from powder data: a form stable at room temperature

The low-temperature form of the title compound, $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}$, (I), was obtained from an ethanol solution of (I) at room temperature. The molecule of (I) is almost planar; the dihedral angle formed by the planes of the phenyl rings is $5.0 (4)^\circ$. Translationally related molecules form stacks with an interplanar separation of $3.32 (1) \text{ \AA}$.

Comment

The hydroxy group of the title compound, (I), forms an intramolecular hydrogen bond with the azo linkage [$\text{O}1 \cdots \text{N}8 = 2.622 (12) \text{ \AA}$], thus precluding the formation of strong intermolecular hydrogen bonds.



The molecules form stacks along [010]; the angle between the normal to the molecular plane and the stack axis is $44.0 (1)^\circ$. Neighbouring molecules within the stack overlap each other in essentially the same manner as in the high-temperature form prepared from the melt (Yatsenko *et al.*, 2001), but the two polymorphs differ in the way in which adjacent stacks are arranged with respect to each other. In the room-temperature form, neighbouring molecules belonging to different stacks are crossed (Fig. 1), whereas in the high-temperature form they are arranged side-by-side. Two relatively short intermolecular contacts are present in this structure: the side-by-side contact $\text{H}5 \cdots \text{H}5(1-x, -1-y, -z)$ (2.12 \AA), and the $3.03 (1) \text{ \AA}$ contact between the phenolic-O atoms. However, this contact is not a hydrogen bond since the $\text{O}1-\text{H}1 \cdots \text{O}1(\frac{1}{2}-x, y-\frac{1}{2}, -\frac{1}{2}-z)$ angle is 110° .

Experimental

Compound (I) was prepared by the reaction of phenyldiazonium chloride with *p*-chlorophenol according to the established procedure of Socha & Vecera (1969) and recrystallized from ethanol.

Crystal data

 $\text{C}_{12}\text{H}_9\text{ClN}_2\text{O}$
 $M_r = 232.66$
Monoclinic, $P2_1/n$
 $a = 19.706 (6) \text{ \AA}$
 $b = 4.614 (2) \text{ \AA}$
 $c = 12.308 (4) \text{ \AA}$
 $\beta = 104.70 (3)^\circ$
 $V = 1082.5 (7) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.428 \text{ Mg m}^{-3}$ Cu $K\alpha$ radiation
Cell parameters from 26 reflections
 $\theta = 3.8\text{--}15.6^\circ$
 $\mu = 2.95 \text{ mm}^{-1}$
 $T = 295 (2) \text{ K}$
Yellow
Specimen shape: flat sheet
 $25 \times 25 \times 2.0 \text{ mm}$
Particle morphology: needle

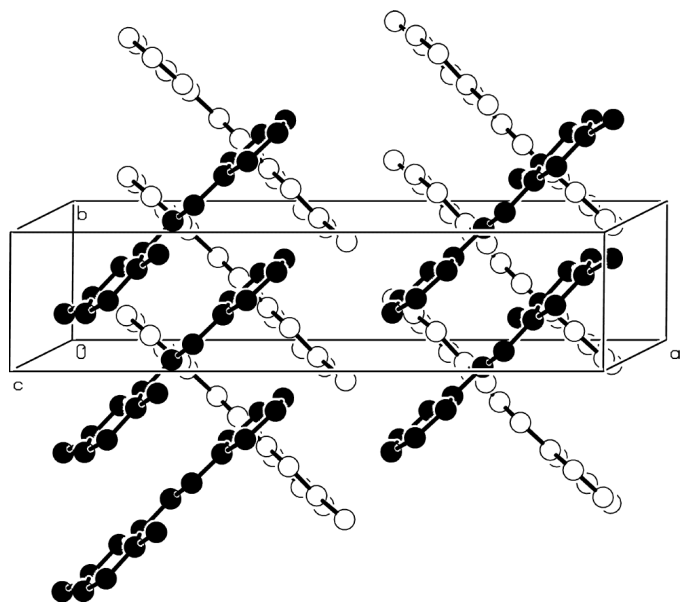


Figure 1
Stacking arrangement adopted by (I).

Data collection

DRON-3M diffractometer
(Burevestnik, Russia)
Specimen mounting: pressed as a
thin layer in the specimen holder
Specimen mounted in reflection
mode

Absorption correction: none
 $h = 0 \rightarrow 14$
 $k = 0 \rightarrow 3$
 $l = -9 \rightarrow 9$
 $2\theta_{\min} = 5.0$, $2\theta_{\max} = 70.0^\circ$
Increment in $2\theta = 0.02^\circ$

Refinement

$R_p = 0.027$
 $R_{wp} = 0.036$
 $R_{exp} = 0.033$
 $S = 1.09$
 $2\theta_{\min} = 7.0$, $2\theta_{\max} = 70.0^\circ$
Wavelength of incident radiation:
1.5418 Å
Excluded region(s): 5.00–6.98°

Profile function: split-type pseudo-
Voigt
115 parameters
H-atom parameters not refined
 $(\Delta/\sigma)_{\max} = 0.034$
Preferred orientation correction:
Spherical harmonics (Ahtee *et al.*,
1989)

The monoclinic cell dimensions were determined with *TREOR90* (Werner *et al.*, 1985) and refined to $M_{20} = 20$ and $F = 46$ (0.0072,71) using the first 26 peak positions. The initial molecular model was built with *MOPAC7.0* (Stewart, 1993) on the *PM3* level (Stewart, 1989). The position and orientation of the molecule were determined using the grid-search procedure (Chernyshev & Schenk, 1998). The X-ray diffraction profile and the difference between the measured and calculated profiles after Rietveld refinement are shown in Fig. 2, final $R_B = 0.062$. The Cl atom was refined anisotropically; C, N and O atoms were refined isotropically and were gathered together into groups with a common U_{iso} parameter for each group. H atoms were

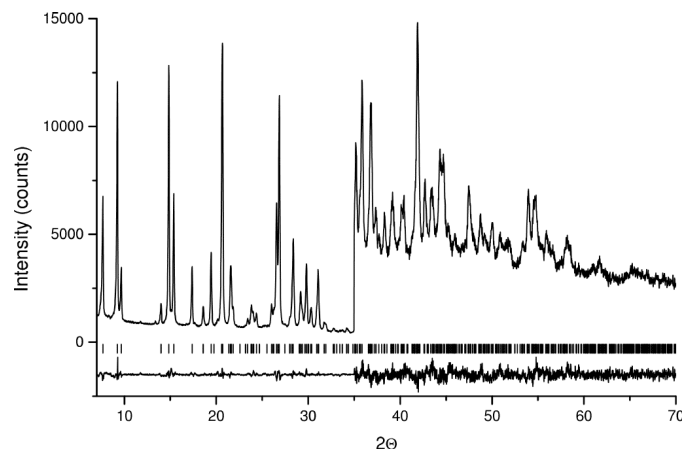


Figure 2

The Rietveld plot showing the observed and difference profiles. The reflection positions are shown above the difference profile. The high-angle area is magnified by a factor of ten.

placed in geometrically calculated positions and their isotropic displacement parameters were fixed at $B = 6.0 \text{ \AA}^2$. The planarity of the phenyl groups was restrained. The anisotropy of diffraction-line broadening was approximated by a quartic form in hkl (Popa, 1998). The standard uncertainties obtained from the Rietveld refinement were corrected for the serial correlation effects (Bérar & Lelann, 1991).

Data collection: local program; cell refinement: *LSPAID* (Visser, 1986); program(s) used to solve structure: *MRIA* (Zlokazov & Chernyshev, 1992); program(s) used to refine structure: *MRIA*; molecular graphics: *PLUTON* (Spek, 1992); software used to prepare material for publication: *PARST* (Nardelli, 1983).

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