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Key indicators

Powder X-ray study

$T = 295$ K

Mean $\sigma(C-C) = 0.014$ Å

R factor = 0.032

w R factor = 0.042

For details of how these key indicators were automatically derived from the article, see

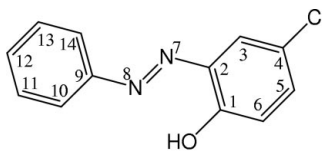
<http://journals.iucr.org/e>.

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

The high-temperature form of the title compound, $C_{12}H_9ClN_2O$, (I), was obtained from the melt. The molecule of (I) is almost planar; the dihedral angle formed by the planes of the phenyl rings is 8.0 (5)°. Translationally related molecules form stacks with an interplanar separation of 3.41 (1) Å.

Comment

The unit cell volume of this form is 2.8% larger than that of the room-temperature form, prepared by crystallization of (I) from an ethanol solution (Yatsenko *et al.*, 2001). Thus the conclusion can be drawn that the room-temperature polymorph is the stable form, whereas the high-temperature form is metastable.



(I)

The hydroxy group forms an intermolecular hydrogen bond with the azo linkage [$O1 \cdots N8 = 2.59$ (2) Å].

The molecules form stacks along $[001]$; the angle between the normal to the molecular plane and the stack axis is 41.4 (1)°. Neighbouring molecules belonging to different stacks are arranged side-by-side (Fig. 1). There are two interstack contacts shorter than the sum of van der Waals radii (Bondi, 1964), *i.e.* the herring-bone-type contact $C5 \cdots H12(\frac{1}{2}-x, y-\frac{1}{2}, \frac{1}{2}-z)$ (2.82 Å) and $O1 \cdots H10(1-x, 1-y, -z)$, which can be considered as a weak hydrogen bond [$H10 \cdots O1$ 2.42 , $C10 \cdots O1$ 3.33 (1) Å and $C10-H10 \cdots O1$ 151°].

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. The sample used for X-ray diffraction study was prepared from the melt (m.p. 384 K).

Crystal data

$C_{12}H_9ClN_2O$

$M_r = 232.66$

Monoclinic, $P2_1/n$

$a = 12.384$ (4) Å

$b = 19.767$ (6) Å

$c = 4.554$ (2) Å

$\beta = 93.27$ (3)°

$V = 1113.0$ (7) Å³

$Z = 4$

$D_x = 1.389$ Mg m⁻³

Cu $K\alpha$ radiation

Cell parameters from 22 reflections

$\theta = 4.5$ – 17.1°

$\mu = 2.87$ mm⁻¹

$T = 295$ (2) K

Yellow

Specimen shape: flat sheet

$25 \times 25 \times 2.0$ mm

Particle morphology: irregular block

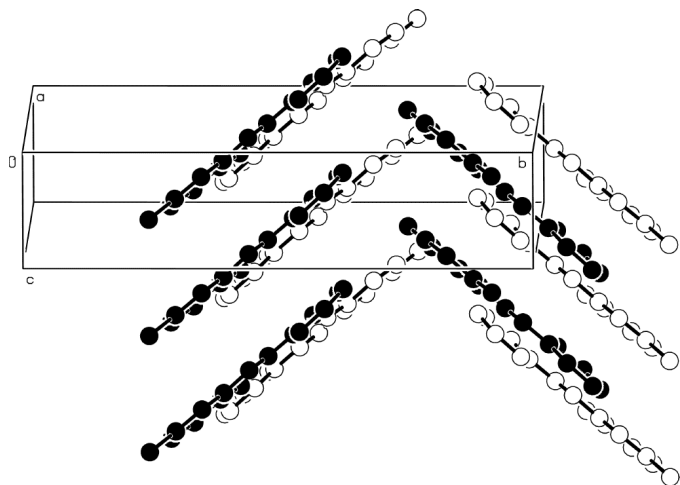


Figure 1
Stacking arrangement adopted by (I).

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
 $h = 0 \rightarrow 9$
 $k = 0 \rightarrow 14$
 $l = -3 \rightarrow 3$
 $2\theta_{\min} = 7.0$, $2\theta_{\max} = 70.0^\circ$
Increment in $2\theta = 0.02^\circ$

Refinement

$R_p = 0.032$
 $R_{wp} = 0.042$
 $R_{exp} = 0.025$
 $S = 1.71$
 $2\theta_{\min} = 7.8$, $2\theta_{\max} = 70.0^\circ$
Wavelength of incident radiation:
1.5418 Å
Excluded region(s): $7.00\text{--}7.78^\circ$

Profile function: split-type pseudo-
Voigt
115 parameters
H-atom parameters not refined
 $(\Delta/\sigma)_{\max} = 0.040$
Preferred orientation correction:
spherical harmonics (Ahtee *et al.*,
1989)

The monoclinic cell dimensions were determined with *TREOR90* (Werner *et al.*, 1985) and refined to $M_{20} = 32$ and $F = 48$ (0.0067,69) using the first 22 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 was determined using the grid-search procedure (Chernyshev & Schenk, 1998). The X-ray diffraction profile and the difference between the measured and calculated profiles after the Rietveld refinement are shown in Fig. 2, final $R_B = 0.087$. 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 placed in geometrically calculated positions and their isotropic displacement parameters were fixed at $B = 9.0 \text{ \AA}^2$. The planarity of

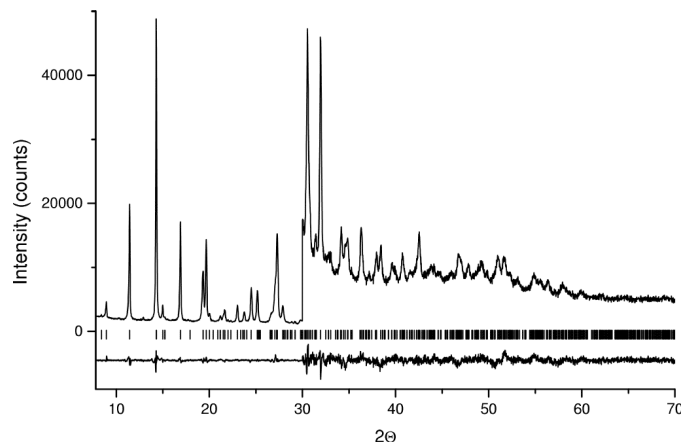


Figure 2
The Rietveld plot showing the observed and difference profiles. The reflection positions are shown above the difference profile. The high-angle area is magnified by a factor of ten.

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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