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2-(2,4-Dimethylphenyl)-1H-isoindole-1,3(2H)-dione

In the molecule of the title compound, $C_{16}H_{13}NO_2$, the N and C atoms of the phthalic anhydride and aniline units form a distorted trigonal–planar structure. The molecules are linked by intermolecular C—H···O hydrogen bonds, forming infinite chains along the [101] direction.

Comment

Phthalic anhydride (PA) is primarily used as a chemical intermediate in the production of plastics from vinyl chloride. PA is also known as 1,3-dioxophthalan, benzene-1,2-dicarboxylic acid anhydride and 1,3-phthalandione. It has another major use in the production of polyester resins and other minor uses in the production of alkyd resins used in paints and lacquers, certain dyes (such as anthraquinone, phthalein, rhodamine, phthalocyanine, fluorescein and xanthene dyes), insect repellents and urethane polyester polyols. It has also been used as a rubber scorch inhibitor and retarder (HSDB, 1995; NCI, 1979).

(I)
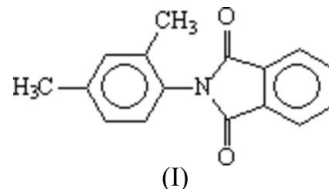
Symptoms in workers occupationally exposed to PA in the workplace over a long period of time affect the respiratory system, with increased incidence of rhinitis, asthma, etc. (Nielsen et al., 1988). Besides these toxic effects, PA has gained importance in the synthesis of carboxylic acids because of its high reactivity towards nucleophiles, e.g. aniline, alcohol and phenol.

We therefore used PA to synthesize 3-(2,4-dimethyl-phenylamido)benzoic acid and to unequivocally establish that the condensation of 2,4-dimethylaniline and PA gives the title compound, (I), after recrystallization from acetone. We report here the structure of (I).

The bond lengths and angles are within normal ranges (Allen et al., 1987) and comparable with the corresponding ones in 3-[(2,4,6-trichloroanilino)carbonyl]prop-2-enoic acid, (II) (Parvez et al., 2004).

The molecule of (I) (Fig. 1) has a distorted trigonal–planar structure with the N and C atoms of PA and aniline having slightly different N—C distances [N1—C1 = 1.407 (2) Å, N1—C2 = 1.398 (2) Å and N1—C9 = 1.433 (2) Å], which are longer than the corresponding distances in 3-[(2,4,6-trichloroanilino)carbonyl]prop-2-enoic acid, (II) [1.340 (2) and
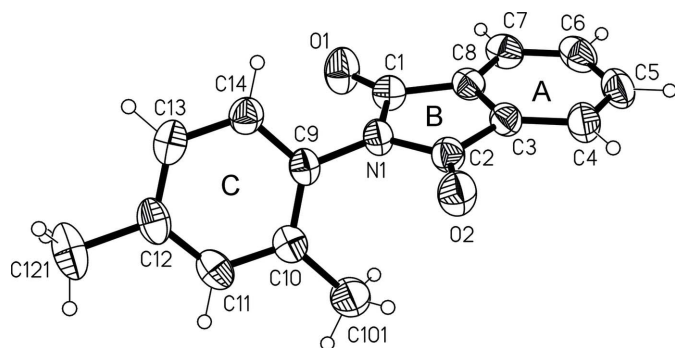
Figure 1
The structure of (I), showing 50% probability displacement ellipsoids and the atom-numbering scheme.
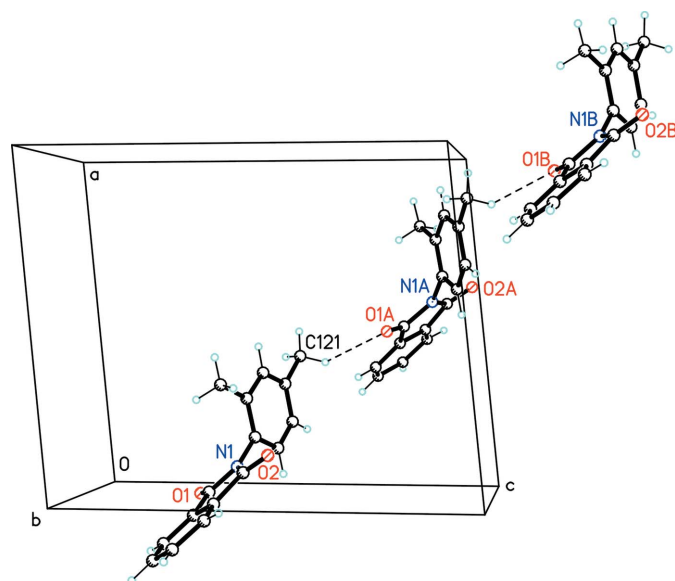

Figure 2
A partial packing diagram of (I). Hydrogen bonds are shown as dashed lines. Atoms marked with 'A' and 'B' are at the symmetry positions ($-\frac{3}{2} + x$, $-\frac{1}{2} - y$, $-\frac{3}{2} - z$) and ($1 + x, y, 1 + z$), respectively.

1.334 (3) Å] and this longer distance may be due to the presence of methyl groups in (I).

An examination of the deviations from the least-squares planes through the individual rings shows that each ring is planar. The isoindole ring system is nearly planar, with a dihedral angle A/B of 3.0 (1)° (rings are as defined in Fig. 1). Ring B has a local pseudo-mirror plane running through atom N1 and the mid-point of the C3—C8 bond, as evidenced by the torsion angles (Table 1). The orientation of benzene ring C with respect to the isoindole ring system may be described by the dihedral angle of 66.4 (1)°.

As can be seen from the packing diagram (Fig. 2), the molecules of (I) are linked by intermolecular C—H⋯O hydrogen bonds (Table 2), forming infinite chains along the [101] direction.

Experimental

A solution of phthalic anhydride (10 g, 67.5 mmol) in HOAc (300 ml) was added to a solution of 2,4-dimethylaniline (8.23 ml, 67.5 mmol) in

HOAc (150 ml) in a 500 ml round-bottomed flask and the mixture was stirred at room temperature overnight. The white precipitate was filtered off, washed with cold distilled water (200 ml) and air-dried. Compound (I) was obtained by recrystallization from acetone (yield 2.01 g, 90%, m.p. 421 K).

Crystal data

$C_{16}H_{13}NO_2$	$Z = 4$
$M_r = 251.27$	$D_x = 1.298$ Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 11.3169$ (4) Å	$\mu = 0.09$ mm^{-1}
$b = 8.4281$ (2) Å	$T = 233$ (2) K
$c = 13.5482$ (5) Å	Prism, colorless
$\beta = 95.757$ (2)°	$0.4 \times 0.3 \times 0.2$ mm
$V = 1285.71$ (7) Å^3	

Data collection

Nonius KappaCCD diffractometer	2219 independent reflections
φ and ω scans	1986 reflections with $I > 2\sigma(I)$
Absorption correction: none	$R_{int} = 0.020$
6524 measured reflections	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0478P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.038$	$+ 0.3326P]$
$wR(F^2) = 0.099$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.006$
2219 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
176 parameters	$\Delta\rho_{min} = -0.13$ e Å^{-3}
H-atom parameters constrained	Extinction correction: $SHELXL97$
	Extinction coefficient: 0.060 (6)

Table 1
Selected torsion angles (°).

C2—N1—C1—C8	3.01 (14)	C2—C3—C8—C1	−0.18 (15)
C1—N1—C2—C3	−3.10 (14)	N1—C1—C8—C3	−1.66 (15)
N1—C2—C3—C8	1.96 (14)		

Table 2
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C121—H12B⋯O1^i	0.97	2.54	3.339 (2)	140

Symmetry code: (i) $x + \frac{1}{2}, -y - \frac{1}{2}, z + \frac{1}{2}$.

H atoms were positioned geometrically, with C—H = 0.94 and 0.97 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C)$, where $x = 1.5$ for methyl and $x = 1.2$ for all other H atoms.

Data collection: COLLECT (Nonius, 1998); cell refinement: SCALEPACK (Otwinowski & Minor, 1997); data reduction: DENZO (Otwinowski & Minor, 1997) and SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXL97.

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References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

HSDB (1995). *Hazardous Subtances Data Bank*. National Library of Medicine, Bethesda, MD (TOMES CD-ROM Version). Denver, CO: Micromedex Inc. USA.

NCI (1979). *Bioassay of Phthalic Anhydride for Possible Carcinogenicity*, pp. 293–594. National Cancer Institute, Springfield, VA: NTIS.

Nielsen, J., Welinder, H., Schütz, A. & Skerfving, S. (1988). *J. Allergy Clin. Immunol.* **82**, 126–133.

Nonius (1998). *COLLECT*. Nonius BV Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp 307–326. New York: Academic Press.

Parvez, M., Shahzadi, S., Shahid, K. & Ali, S. (2004). *Acta Cryst.* E**60**, o2082–o2084.

Sheldrick, G. M. (1997a). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.

Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.10. Bruker AXS Inc., Madison, Wisconsin, USA.