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## Key indicators

Single-crystal X-ray study  
 $T = 296$  K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002$  Å  
 $R$  factor = 0.048  
 $wR$  factor = 0.130  
Data-to-parameter ratio = 19.2For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

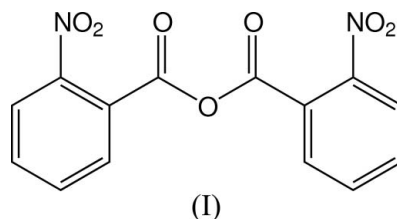
## o-Nitrobenzoic acid anhydride

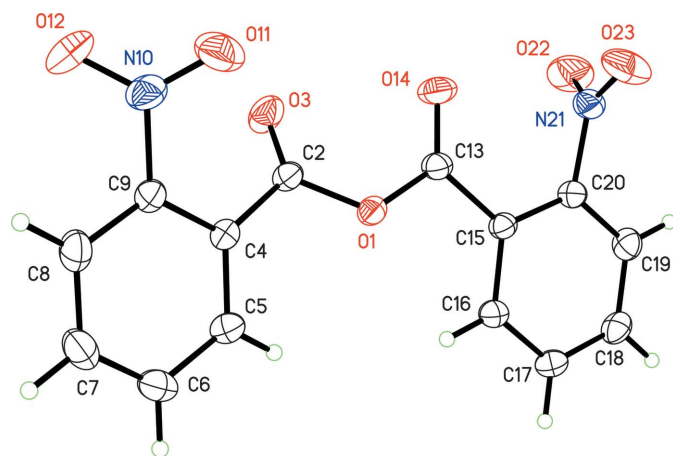
The title compound,  $\text{C}_{14}\text{H}_8\text{N}_2\text{O}_7$ , is a symmetrically substituted acyclic anhydride. The nitro groups are twisted with respect to the benzene rings; these are almost coplanar, with a dihedral angle of  $9.07$  ( $9$ )°.

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## Comment

The title molecule, (I), although being a symmetrically substituted anhydride, lies on a general position (Fig. 1). The conformation of the anhydride unit ( $\text{O}1/\text{C}2/\text{O}3/\text{C}13/\text{O}14$ ) may be defined by the pseudo-torsion angle  $\text{O}3=\text{C}2\cdots\text{C}13=\text{O}14$  of  $27.65$  ( $15$ )°. A search of the Cambridge Structural Database (CSD; Version 5.27, updated May 2006; Allen, 2002) indicates that acyclic anhydrides crystallize with almost unrestricted conformations. Observed  $|\text{O}=\text{C}\cdots\text{C}=\text{O}|$  angles range from *ca*  $6^\circ$  (Graham *et al.*, 1996) to  $95^\circ$  (Schubert *et al.*, 1988), contrasting with succinic and related cyclic anhydrides, for which observed absolute torsion angles fall in the range  $0$ – $25^\circ$ .The nitro groups in (I) are twisted with respect to their carrier benzene rings, with dihedral angles of  $45.16$  ( $13$ ) and  $79.68$  ( $12$ )° for the  $\text{N}10$ - and  $\text{N}21$ -nitro groups, respectively. Such a lack of delocalization of the nitrobenzene units is not uncommon and is, in the present case, dictated by the vicinity of the carbonyl groups. The arrangement of the  $\text{NO}_2$  and anhydride groups in (I) allows the benzene rings ( $\text{C}4$ – $\text{C}9$  and  $\text{C}15$ – $\text{C}20$ ) to be almost coplanar, with a dihedral angle of  $9.07$  ( $9$ )°.A totally different molecular structure had been described for the isomeric *m*-nitrobenzoic acid anhydride (Główka *et al.*, 1990). In that case, no hindering interactions occur for the nitro groups, which are nearly coplanar with their respective benzene rings (dihedral angles:  $10.5$  and  $14.8^\circ$ ), while the dihedral angle between the benzene rings is  $27.0^\circ$ . As a consequence, the anhydride group presents a  $|\text{O}=\text{C}\cdots\text{C}=\text{O}|$  torsion angle of  $40.9^\circ$ , significantly different from that observed in (I). A similar conformation was observed for another *meta*-substituted benzoic anhydride, namely 3-(dimethylamino)benzoic anhydride (two polymorphs have been reported; Lynch *et al.*, 2000).



**Figure 1**  
The structure of (I), with displacement ellipsoids drawn at the 30% probability level.

## Experimental

A solution of (*S*)-anthracen-9-ylmethyl-( $\alpha$ -phenylethyl)amine (4.51 g, 14.4 mmol) and Et<sub>3</sub>N (2.92 g, 28.8 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 ml) was added slowly to an ice-cooled solution of 2-nitrobenzoyl chloride (3.30 g, 16.3 mmol) in CH<sub>2</sub>Cl<sub>2</sub> (10 ml). The resulting mixture was allowed to warm to 298 K and stirred for 2 h. The solution was quenched with 25 ml of H<sub>2</sub>O, extracted with 3  $\times$  25 ml of CH<sub>2</sub>Cl<sub>2</sub>, and the combined organic phases were dried with magnesium sulfate. The solvent was then removed under reduced pressure. The crude product was purified by flash chromatography on deactivated silica gel (Et<sub>3</sub>N/SiO<sub>2</sub> = 2.5% v/v, hexanes–EtOAc, 80:20) to afford (*R<sub>a</sub>,S*)-2-nitro-*N*-anthracen-9-ylmethyl-*N*-( $\alpha$ -phenylethyl)benzamide (*R<sub>a</sub>* indicates axial chirality) (1.74 g, 5.72 mmol, 39.7% yield; Huelgas *et al.*, 2006) and (I) (0.773 g, 2.45 mmol, 30.0% yield). Compound (I) was recrystallized from hexane/CH<sub>2</sub>Cl<sub>2</sub> (5:1), affording colourless crystals (m.p. 405–406 K).

### Crystal data

C <sub>14</sub> H <sub>8</sub> N <sub>2</sub> O <sub>7</sub>	<i>Z</i> = 4
<i>M<sub>r</sub></i> = 316.22	<i>D<sub>x</sub></i> = 1.531 Mg m <sup>-3</sup>
Monoclinic, <i>P</i> 2 <sub>1</sub> / <i>c</i>	Mo <i>K</i> $\alpha$ radiation
<i>a</i> = 7.9365 (10) Å	$\mu$ = 0.13 mm <sup>-1</sup>
<i>b</i> = 16.1757 (14) Å	<i>T</i> = 296 (1) K
<i>c</i> = 11.0023 (8) Å	Prism, colourless
$\beta$ = 103.798 (7)°	0.60 $\times$ 0.44 $\times$ 0.28 mm
<i>V</i> = 1371.7 (2) Å <sup>3</sup>	

### Data collection

Siemens P4 diffractometer  
 $\omega$  scans  
Absorption correction: none  
7531 measured reflections  
3994 independent reflections  
2935 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.029$   
 $\theta_{\text{max}} = 30.0^\circ$   
3 standard reflections  
every 97 reflections  
intensity decay: 1%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.048$   
 $wR(F^2) = 0.130$   
*S* = 1.03  
3994 reflections  
208 parameters  
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0542P)^2 + 0.2914P]$   
where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} < 0.001$   
 $\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$

**Table 1**

Selected torsion angles (°).

C13–O1–C2–C4	–175.96 (11)	C2–O1–C13–C15	–154.97 (12)
O1–C2–C4–C5	–44.38 (16)	O1–C13–C15–C16	9.59 (19)
C4–C9–N10–O11	–44.7 (2)	C15–C20–N21–O23	80.84 (19)

H atoms were placed in idealized positions and refined as riding on their parent C atoms, with C–H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .

Data collection: XSCANS (Siemens, 1996); cell refinement: XSCANS; data reduction: XSCANS; program(s) used to solve structure: SHELXTL-Plus (Sheldrick, 1998); program(s) used to refine structure: SHELXTL-Plus; molecular graphics: SHELXTL-Plus; software used to prepare material for publication: SHELXTL-Plus.

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