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Key indicators

Single-crystal X-ray study

$T = 123\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$

R factor = 0.045

wR factor = 0.119

Data-to-parameter ratio = 7.7

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

N-(3-Nitrophenyl)maleimide

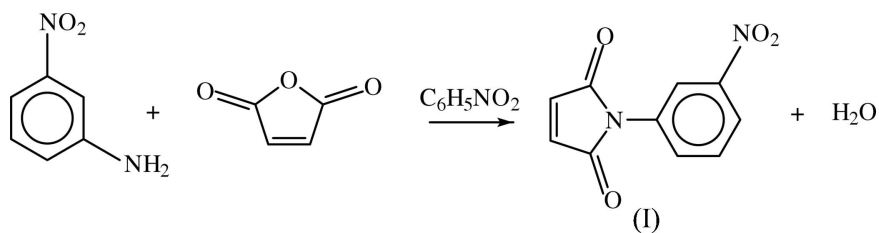
The title compound, $\text{C}_{10}\text{H}_6\text{N}_2\text{O}_4$, belongs to a series of *N*-arylmaleimides, which can be used as photoinitiators for free-radical polymerization. The dihedral angles between the planes of the benzene and imide rings are $56.2(1)$ and $52.9(1)^\circ$ in the two independent molecules in the asymmetric unit.

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Comment

There is considerable activity related to the use of *N*-substituted maleimides as photoionizers for free-radical polymerization, where the maleimide can produce the initiating radical species (Pyriadi & Nabeel, 1988; Andersson *et al.*, 1996; Hoyle *et al.*, 1999). In continuing the structural studies on *N*-substituted maleimide systems, to study the behaviour of $\text{C}_{\text{aryl}}-\text{N}$ distance and imide/benzene interplanar angle, the crystal structure determination of *m*-nitrophenylmaleimide, $\text{C}_{10}\text{H}_6\text{N}_2\text{O}_4$, (I), was undertaken. The reactivity of *N*-aromatic maleimides in photopolymerization processes as a function of the angle between the maleimide and benzene rings has been analysed (Miller *et al.*, 2000). The *p*-nitrophenylmaleimide (*p*-NPM) system has been reported by our research group (Moreno-Fuquen *et al.*, 2003). This structure has a close analogy to the title compound and it has been used as a model for comparison.



A perspective view of the two independent molecules in the asymmetric unit of the title compound, showing the atomic numbering scheme, is given in Fig. 1. Focusing on the $\text{N}-\text{C}_{\text{aryl}}$ bond length, in the title compound the $\text{N}2-\text{C}5$ and $\text{N}4-\text{C}15$ distances are $1.424(4)$ and $1.421(4)\text{ \AA}$, respectively. These values are close to the $\text{N}-\text{C}_{\text{aryl}}$ bond length for *p*-nitrophenylmaleimide (Moreno-Fuquen *et al.*, 2003) and are slightly smaller than the average value reported for nine *N*-arylmaleimide derivatives (Miller *et al.*, 2000). The benzene ring mean plane is rotated $56.2(1)$ and $52.9(1)^\circ$ with respect to the imide ring mean plane. These values are dictated probably by the weak hydrogen bond between an O atom of

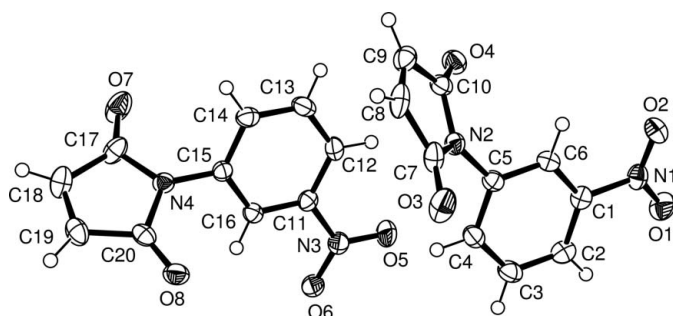


Figure 1

The asymmetric unit of the title compound with the atomic labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as spheres of arbitrary radii.

the maleimide group and a C atom of the benzene ring. The rotation is smaller in the case of *p*-NPM, which has an angle of 42.98 (5)°. This is consistent with the literature values, where other maleimides with bulky *ortho* substituents show angles of rotation greater than 80°. Other bond lengths and internal geometrical parameters of the title compound (Table 1) are similar to those in *p*-NPM. There are no significant intermolecular hydrogen bonds in the structure.

Experimental

Reagents and solvents for the synthesis were obtained from Aldrich Chemical Co., and were used without additional purification. The title compound was prepared by taking equimolar quantities of *m*-nitroaniline and maleic anhydride in nitrobenzene and refluxing at 513 K for 3 h. The reaction product was filtered and washed with hexane and then it was dissolved in a mixture of ethyl acetate–hexane (15% hexane) in order to purify it by column chromatography. The solid was crystallized from chloroform, giving pale-yellow prisms with a melting point of 395 (1) K.

Crystal data

$C_{10}H_6N_2O_4$	$Z = 8$
$M_r = 218.17$	$D_x = 1.541 \text{ Mg m}^{-3}$
Orthorhombic, $Pna2_1$	Mo $K\alpha$ radiation
$a = 18.9815 (6) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$b = 6.6643 (2) \text{ \AA}$	$T = 123 (2) \text{ K}$
$c = 14.8702 (4) \text{ \AA}$	Prism, pale yellow
$V = 1881.06 (10) \text{ \AA}^3$	$0.40 \times 0.25 \times 0.07 \text{ mm}$

Data collection

Enraf–Nonius CAD-4 diffractometer	1693 reflections with $I > 2\sigma(I)$
$\omega/2\theta$ scans	$R_{\text{int}} = 0.032$
Absorption correction: none	$\theta_{\text{max}} = 27.5^\circ$
4072 measured reflections	2 standard reflections
2238 independent reflections	frequency: 150 min
	intensity decay: 0.1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.045$
 $wR(F^2) = 0.119$
 $S = 1.05$
 2238 reflections
 289 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0754P)^2 + 0.0433P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.39 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.27 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (Å, °).

N2—C10	1.393 (4)	C4—C5	1.394 (5)
N2—C7	1.404 (4)	C5—C6	1.392 (4)
N2—C5	1.424 (4)	C8—C9	1.318 (6)
N4—C17	1.400 (5)	C14—C15	1.383 (4)
N4—C20	1.403 (4)	C15—C16	1.386 (5)
N4—C15	1.421 (4)	C18—C19	1.329 (6)
C10—N2—C5	124.8 (3)	C20—N4—C15	124.8 (3)
C7—N2—C5	124.2 (3)	C2—C1—N1	118.8 (3)
C17—N4—C15	124.8 (3)	C16—C11—N3	118.4 (3)
O1—N1—C1—C2	−6.9 (5)	O5—N3—C11—C16	175.7 (3)
C7—N2—C5—C6	116.7 (4)	C20—N4—C15—C16	−47.8 (5)

In the absence of significant anomalous scattering, Friedel pairs were merged. H atoms were located in electron-density difference maps and subsequently treated as riding atoms, with C—H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *CAD-4 SDP* (Frenz, 1978); program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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References

- Andersson, H., Gedde, U. W. & Hult, A. (1996). *Macromolecules*, **29**, 1649–1654.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Frenz, B. A. (1978). *Computing in Crystallography*, edited by H. Schenk, R. Olthof-Hazekamp, H. van Koningsveld & G. C. Bassi, pp. 64–71. Delft: University Press.
- Hoyle, C. E., Viswanathan, K., Clark, S. C., Miller, C. W., Nguyen, C., Jonsson, S. & Shao, L. (1999). *Macromolecules*, **32**, 2793–2795.
- Miller, C. W., Hoyle, C. E., Valente, E. J., Zubkowski, J. D. & Jonsson, E. S. (2000). *J. Chem. Crystallogr.* **30**, 563–571.
- Moreno-Fuquen, R., Valencia, H., Abonia, R., Kennedy, A. R. & Graham, D. (2003). *Acta Cryst. E59*, o1717–o1718.
- Pyriadi, T. M. & Nabeel, E. (1988). *J. Macromol. Sci. Chem. A*, **25**, 1683–1688.
- Sheldrick, G. M. (1990). *Acta Cryst. A46*, 467–473.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.