

***p*-Nitrophenylmaleimide**

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**Key indicators**

Single-crystal X-ray study

*T* = 293 K

Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$

*R* factor = 0.030

w*R* factor = 0.084

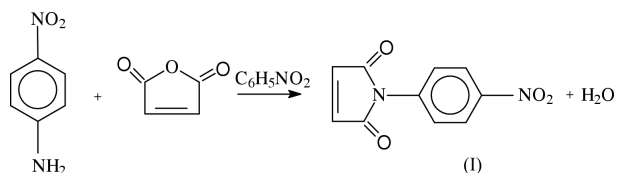
Data-to-parameter ratio = 10.8

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

The title compound,  $\text{C}_{10}\text{H}_6\text{N}_2\text{O}_4$ , belongs to a series of *N*-arylmaleimides. These *N*-substituted maleimides can be used as photo-initiators for free-radical polymerization. The molecule has crystallographic twofold rotation symmetry. The dihedral angle between the planes of the benzene and imide rings is  $42.98(5)^\circ$ .

**Comment**

Maleimide is a reactive vinyl monomer. It has been shown that the maleimide system participates in copolymerization reactions *via* a free-radical process (Teerenstra *et al.*, 2000). Monomers formed by *N*-aromatic maleimides are used in photopolymerization processes (Hoyle *et al.*, 1999). The title compound, (I), is of interest for its photochemical properties. Some *N*-arylmaleimide structures, for which the degree of rotation between the aryl and maleimide rings is analysed, have been reported in the literature (Miller *et al.*, 2000). One of the molecular structures reported, that of *N*-(4-methoxyphenyl)maleimide, has a close analogy to the title compound and is compared with it here.



A perspective view of the title molecule, showing the atomic numbering scheme, is given in Fig. 1. The molecule has crystallographic twofold rotation symmetry, and it shows a rotation between the benzene and maleimide planes of  $42.98(5)^\circ$ . This value is smaller than the value of  $63.7^\circ$  for *N*-(4-methoxyphenyl)maleimide (Miller *et al.*, 2000). Bond lengths and other internal geometrical parameters for the two compounds are similar. In the title compound, the formation of a weak intramolecular  $\text{O1}\cdots\text{H3}-\text{C3}$  hydrogen bond is observed [ $\text{O1}\cdots\text{H3} = 2.590 \text{ \AA}$ ,  $\text{O1}\cdots\text{C3} = 2.994(2) \text{ \AA}$  and  $\text{O1}\cdots\text{H3}-\text{C3} = 107^\circ$ ]; this prevents a larger rotation between the benzene and maleimide planes. There are no significant intermolecular hydrogen bonds.

**Experimental**

Reagents and solvents for the synthesis were purchased from Aldrich Chemical Co. and were used without additional purification.  $^1\text{H}$  NMR spectra were recorded on a Bruker 400 MHz instrument. Mass spectra were recorded on a Jeol AX505 spectrometer using chemical ionization (CI). The synthesis of the title compound was carried out by taking equimolar quantities of *p*-nitroaniline and maleic anhydride in nitrobenzene, and refluxing for 3 h. The product of this reaction

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was purified by column chromatography [40% yield; melting point 436 (1) K]. Single crystals suitable for X-ray analysis were obtained by slow evaporation using chloroform as solvent.

Another synthetic procedure gave a higher yield, starting with *N*-(4-nitrophenyl)maleamic acid (9.1 mmol) (easily obtained in almost quantitative yield as a green powder by heating equimolecular amounts of maleic anhydride and *p*-nitroaniline for 20 min in refluxing toluene) and sodium acetate (10.9 mmol). They were stirred in acetic anhydride (25 ml) at room temperature, giving the title compound as a pale-yellow powder (6.7 mmol) in 74% yield. It was crystallized from methanol, giving colourless plates with a melting point of 436 (1) K.  $R_f(A)$  0.38;  $^1\text{H NMR}$  (DMSO- $d_6$ ):  $\delta_H$  7.26 (2H, *s*, CH=CH), 7.70 (2H, *d*,  $J = 5.0$  Hz, Ar-H), 8.36 (2H, *d*,  $J = 5.1$  Hz, Ar-H);  $m/z$  (CI): 219.04058 [ $\text{C}_{10}\text{H}_6\text{N}_2\text{O}_4$  ( $M + \text{H}$ ) $^+$  < 2.5 p.p.m.]; Analysis calculated for  $\text{C}_{10}\text{H}_6\text{N}_2\text{O}_4$ : C 55.10, H 2.80, N 12.84%; found: C 54.80, H 2.67, N 12.63%.

#### Crystal data

$\text{C}_{10}\text{H}_6\text{N}_2\text{O}_4$	$D_x = 1.543 \text{ Mg m}^{-3}$
$M_r = 218.17$	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 25 reflections
$a = 10.535$ (2) Å	$\theta = 8\text{--}16^\circ$
$b = 11.624$ (3) Å	$\mu = 0.12 \text{ mm}^{-1}$
$c = 8.148$ (2) Å	$T = 293$ (2) K
$\beta = 109.69$ (3) $^\circ$	Prism, colourless
$V = 939.5$ (4) Å $^3$	$0.18 \times 0.15 \times 0.08 \text{ mm}$
$Z = 4$	

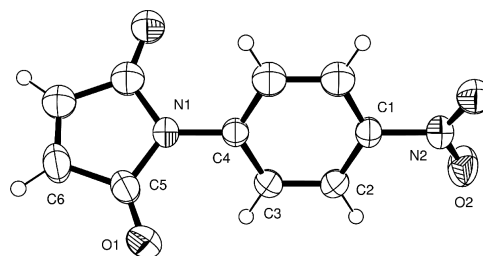
#### Data collection

Enraf-Nonius CAD-4 diffractometer	$\theta_{\text{max}} = 25.0^\circ$
$\omega/2\theta$ scans	$h = 0 \rightarrow 12$
Absorption correction: none	$k = -13 \rightarrow 0$
870 measured reflections	$l = -9 \rightarrow 9$
824 independent reflections	3 standard reflections
738 reflections with $I > 2\sigma(I)$	frequency: 120 min
$R_{\text{int}} = 0.017$	intensity decay: 1.2%

#### Refinement

Refinement on $F^2$	$w = 1/[\sigma^2(F_o^2) + (0.0429P)^2 + 0.4529P]$
$R[F^2 > 2\sigma(F^2)] = 0.030$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.085$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.05$	$\Delta\rho_{\text{max}} = 0.14 \text{ e \AA}^{-3}$
824 reflections	$\Delta\rho_{\text{min}} = -0.20 \text{ e \AA}^{-3}$
76 parameters	Extinction correction: <i>SHELXL97</i>
H-atom parameters constrained	Extinction coefficient: 0.0169 (18)

All H atoms were placed at geometrically idealized positions and were refined as riding; C–H = 0.93 Å and  $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ .



**Figure 1**

An *ORTEP-3* (Farrugia, 1997) plot of the title compound, with the atomic labelling scheme. Displacement ellipsoids are plotted at the 50% probability level. H atoms are shown as spheres of an arbitrary radius.

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*.

One of the authors (RMF) dedicates this work to the memory of Dr Johannes Rüdiger Lechat (Universidade de São Paulo, USP, Brazil). RMF and RA also acknowledge Universidad del Valle and Colciencias, Colombia, for partial support of this work.

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