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Key indicators

Single-crystal X-ray study
 $T = 198\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.002\text{ \AA}$
Disorder in main residue
 R factor = 0.036
 wR factor = 0.110
Data-to-parameter ratio = 12.0

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

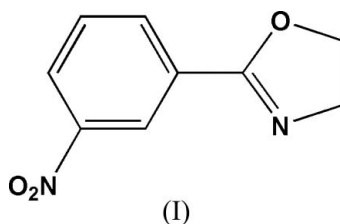
2-(3-Nitrophenyl)-2-oxazoline

The title compound, $\text{C}_9\text{H}_8\text{N}_2\text{O}_3$, was obtained by a zinc chloride-promoted addition and ring-closure reaction involving 3-nitrobenzonitrile and 2-ethanolamine. The planar oxazoline and aromatic rings of the molecule are almost coplanar, with a dihedral angle of $8.79(5)^\circ$ between them. The oxazoline ring is disordered over two positions, rendering the oxygen and nitrogen positions indistinguishable.

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Comment

2-Oxazolines represent an important class of heterocyclic compounds which have found application in a number of areas of chemical endeavour. A non-exhaustive list of these uses would include asymmetric organic synthesis (Meyers, 1978, 2005), polymerization chemistry (Aoi & Okada, 1996; Culbertson, 2002), Lewis acid catalysis (Cross *et al.*, 2006; Eisnor *et al.*, 2006; Gossage *et al.*, 2004; McManus & Guiry, 2004; Pfaltz, 1999) and coordination chemistry (Barclay *et al.*, 2003; Decken *et al.*, 2006; Gómez *et al.*, 1999; Hoyeyda *et al.*, 1992). Despite the widespread use of oxazolines, relatively few metal-free species have been examined in the solid-state by X-ray diffraction methods (*e.g.* Asano & Doi, 2004; Doi *et al.*, 2001, 2002, 2003; Gzella & Rozwadowska, 2000; Langer *et al.*, 2005, 2006; Ruiz-Valero *et al.*, 1984). In this report, we detail the structural characterization of a 2-aryl-oxazoline devoid of organic substituents (Poindexter, 1983) on oxazoline ring positions 4 and 5, *viz.* 2-(3'-nitrophenyl)-2-oxazoline (I). The title compound is formed *via* a metal-halide (ZnCl_2)-mediated coupling, with concomitant loss of ammonia, of 3-nitrobenzonitrile and ethanolamine. This is followed by *in situ* dehydrative intramolecular ring closure at reflux temperature in chlorobenzene (Button *et al.*, 2002; Witte & Seelinger, 1974), a situation that leads to the formation of (I).



The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are collected in Table 1. The bond lengths and angles are typical for a 2-aryl-2-oxazoline (Gzella & Rozwadowska, 2000; Langer *et al.*, 2005, 2006; Ruiz-Valero *et al.*, 1984). Of note is the virtual planarity of the molecule. The maximum deviation from planarity is observed for O1 [0.2283 (9) Å]. The torsion angle between the aromatic

and heterocyclic ring systems measures $8.79(5)^\circ$ while the angle between the plane of the NO_2 group and the aromatic ring is $8.04(9)^\circ$.

The oxazoline ring is disordered *via* a 180° rotation of the C3–C7 bond. Independent refinement of the O8, N8, O11, N11 atom positions resulted in convergence of O8 with N8 and O11 with N11. Consequently, the observed bond lengths of C7–N8/O8 [1.304 (1) Å] and C7–O11/N11 [1.304 (1) Å] fall in between values usually observed for C–O [1.36 (1) Å] and C=N bonds [1.264 (5) Å] (MOGUL; Version 1.1; Bruno *et al.*, 2004) in aryl oxazoline rings.

The packing of compound (I) in the solid state involves stacking of the planar molecules in columns in a head-to-tail fashion that places the NO_2 groups and oxazoline rings on top of one another.

Experimental

A mixture of 3-nitrobenzonitrile (10.4 g, 0.070 mol) and 2-aminoethanol (7.13 g, 0.117 mol) was placed in a round-bottomed flask and dissolved in 85 ml of chlorobenzene. The flask was subsequently charged with 1.0 g (10 mol %) of anhydrous ZnCl_2 . The resulting suspension was heated at reflux temperature for a period of 48 h, during which time a light-pink coloration was noted in the reaction vessel. The flask was cooled to room temperature and all volatile components were removed *via* rotary evaporation. The residue was then extracted with 50 ml of distilled water and 50 ml of dichloromethane and the two extracts were combined together. The two layers were then separated and the inorganic portion was further extracted with CH_2Cl_2 (2×30 ml). The organic fractions were then combined and dried over Na_2SO_4 and filtered, and the solvent was removed *in vacuo*. The crude product was purified by flash silica gel (230–400 mesh) chromatography using $\text{CH}_2\text{Cl}_2/\text{MeOH}$ (93:3 *v/v*, $R_f = 0.441$) as eluant; this resulted in the isolation of a light-yellow powder (9.74 g, 77%) following solvent evaporation. M.p. 394 K (lit. 391–392 K; Leffler & Adams, 1937). Crystals suitable for X-ray diffraction were grown by recrystallization of (I) from an Et_2O solution.

Crystal data

$\text{C}_9\text{H}_8\text{N}_2\text{O}_3$	$Z = 4$
$M_r = 192.17$	$D_x = 1.497 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 6.0910(5) \text{ \AA}$	$\mu = 0.12 \text{ mm}^{-1}$
$b = 19.7825(18) \text{ \AA}$	$T = 198(1) \text{ K}$
$c = 7.1119(7) \text{ \AA}$	Plate, colorless
$\beta = 95.584(2)^\circ$	$0.48 \times 0.23 \times 0.05 \text{ mm}$
$V = 852.88(13) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	5794 measured reflections
φ and ω scans	1902 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1997b)	1466 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.872$, $T_{\max} = 0.994$	$R_{\text{int}} = 0.027$
	$\theta_{\text{max}} = 27.5^\circ$

Refinement

Refinement on F^2	All H-atom parameters refined
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0634P)^2]$
$wR(F^2) = 0.110$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.13$	$(\Delta/\sigma)_{\text{max}} < 0.001$
1902 reflections	$\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
159 parameters	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

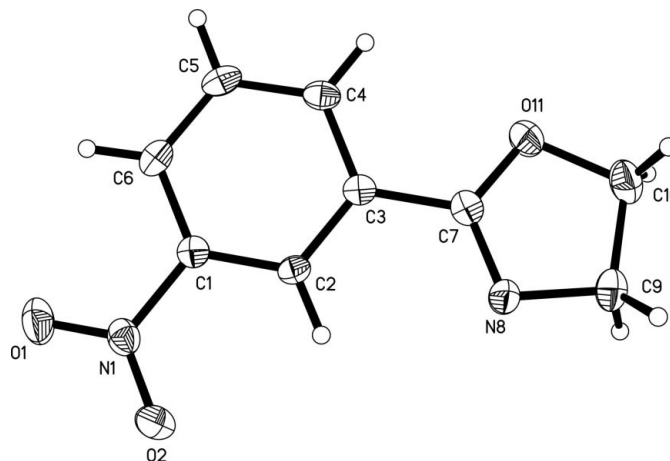


Figure 1

A view of the molecular structure of compound (I) with displacement parameters at the 30% probability level.

Table 1

Selected geometric parameters (Å, °).

C7–N8	1.3048 (15)	C9–C10	1.508 (2)
C7–O11	1.3049 (15)	C10–O11	1.4632 (18)
N8–C9	1.4607 (17)		
N8–C7–O11	118.84 (11)	O11–C10–C9	104.88 (12)
C7–N8–C9	106.14 (10)	C7–O11–C10	105.56 (11)
N8–C9–C10	104.16 (11)		

The oxazoline ring is disordered and the N8 and O11 positions were not completely resolved. Refinement using a disorder model was unsuccessful as the disordered atoms converged into one position. Identical atom positions and displacement parameters were used in the refinement for the pairs N8/O8 and O11/N11, with site occupancies fixed at 0.5. H atoms were found in Fourier difference maps and refined using isotropic displacement parameters [C–H = $0.925(19)$ – $0.991(14) \text{ \AA}$].

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 2006); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 2000); software used to prepare material for publication: SHELXTL.

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