

rac-Methyl 4-azido-3-hydroxy-3-(2-nitro-phenyl)butanoate

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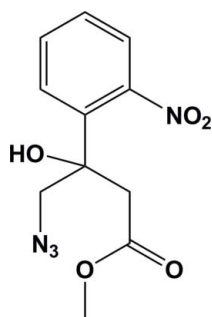
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Key indicators: single-crystal X-ray study; $T = 153$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å;
 R factor = 0.036; wR factor = 0.088; data-to-parameter ratio = 10.7.

In the title compound, $\text{C}_{11}\text{H}_{12}\text{N}_4\text{O}_5$, the mean plane through the nitro substituent on the benzene ring is inclined to the benzene mean plane by $85.8(2)^\circ$, which avoids steric interactions with the *ortho* substituents. The hydroxy group is involved in bifurcated hydrogen bonds. The first is an intramolecular $\text{O}-\text{H}\cdots\text{O}$ hydrogen bond, involving the ester carbonyl O atom, which gives rise to the formation of a boat-like hydrogen-bonded chelate ring. The second is an intermolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond involving the first N atom of the azide group of a symmetry-related molecule. In the crystal structure this leads to the formation of a polymer chain extending in the c -axis direction.

Related literature

For literature related to the antitumor properties of rhazinilam, see: Bonneau *et al.* (2007). For literature related to the synthesis and structure-activity relationships of rhazinilam analogues, see: Decor *et al.* (2006); Baudoin *et al.* (2002); Ghosez *et al.* (2001); Rubio & Bornmann (2001); Dupont *et al.* (2000, 1999); Alazard *et al.* (1996). For details of the Mukaiyama reaction, see: Mukaiyama *et al.* (1974). For literature related to the synthesis of pyrrolinone precursors, see: Vallat (2004); Vallat *et al.* (2009).



Experimental

Crystal data

$\text{C}_{11}\text{H}_{12}\text{N}_4\text{O}_5$	$V = 1272.4(2)$ Å ³
$M_r = 280.25$	$Z = 4$
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
$a = 9.4772(11)$ Å	$\mu = 0.12$ mm ⁻¹
$b = 14.0710(12)$ Å	$T = 153(2)$ K
$c = 10.1861(12)$ Å	$0.40 \times 0.30 \times 0.30$ mm
$\beta = 110.496(13)^\circ$	

Data collection

Stoe IPDS diffractometer	2451 independent reflections
Absorption correction: none	1587 reflections with $I > 2\sigma(I)$
8743 measured reflections	$R_{\text{int}} = 0.074$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$	230 parameters
$wR(F^2) = 0.088$	All H-atom parameters refined
$S = 0.87$	$\Delta\rho_{\text{max}} = 0.23$ e Å ⁻³
2451 reflections	$\Delta\rho_{\text{min}} = -0.21$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{O3}-\text{H3O}\cdots\text{O4}$	0.825 (19)	2.30 (2)	2.9439 (16)	135.5 (18)
$\text{O3}-\text{H3O}\cdots\text{N2}^i$	0.825 (19)	2.27 (2)	2.9193 (18)	135.6 (18)
$\text{C10}-\text{H10B}\cdots\text{O4}^{ii}$	0.95 (2)	2.557 (19)	3.350 (2)	141.0 (15)
$\text{C11}-\text{H11B}\cdots\text{O1}^{iii}$	0.95 (3)	2.57 (2)	3.268 (3)	130.9 (16)

Symmetry codes: (i) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (ii) $x - \frac{1}{2}, -y + \frac{1}{2}, z - \frac{1}{2}$; (iii) $x - \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *EXPOSE* in *IPDS Software* (Stoe & Cie, 2000); cell refinement: *CELL* in *IPDS Software*; data reduction: *INTEGRATE* in *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2749).

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supplementary materials

***rac*-Methyl 4-azido-3-hydroxy-3-(2-nitrophenyl)butanoate**

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Comment

Rhazinilam, a natural product, has been shown to possess antitumoral properties. It induces *in vitro* spiralization of microtubules [vinblastin effect] and inhibits the disassembly of these microtubules [paclitaxel effect] (Bonneau *et al.*, 2007). It has shown significant *in vitro* cytotoxicity towards various cancer cells, but it is not active *in vivo*. Several groups have been interested in synthesizing and studying the structure-activity relationship of rhazinilam analogues (Decor *et al.*, 2006; Baudoin *et al.*, 2002; Ghosez *et al.*, 2001; Rubia & Bornmann, 2001; Dupont *et al.*, 2000; Dupont *et al.*, 1999; Alazard *et al.*, 1996).

In the synthesis of Rhazinilam analogues developed in our group the Mukaiyama reaction, a versatile synthetic tool in organic chemistry, is a key step reaction (Mukaiyama *et al.*, 1974). In one of our retrosynthetic approaches (1-methoxyvinyl)trimethylsilane was used as a nucleophile, 2-azido-1-(2-nitrophenyl)ethanone as an electrophile and TiCl₄ as a Lewis acid, to synthesize the title hydroxyester, in high yield. This hydroxyester is a suitable precursor for the formation of the pyrrolinone required for the next step in the synthesis of Rhazinilam analogues (Vallat, 2004; Vallat *et al.*, 2009).

The molecular structure of the title compound is illustrated in Fig. 1. The bond distances and angles are normal. The mean plane through the nitro group is inclined to the benzene mean plane by 85.8 (2)°, so avoiding steric interactions with the *ortho* substituents. The hydroxyl group (O3) is involved in bifurcated hydrogen bonds (Table 1). The first is an intramolecular O—H···O hydrogen bond, involving the ester carbonyl O-atom (O4), and gives rise to the formation of a boat-like hydrogen bonded chelate ring. The second is an intermolecular O—H···N hydrogen bond involving the first N-atom (N2) of the azide group (Table 1). This leads to the formation of a polymer chain extending in the *c* direction. (Fig. 2). There are also two weak intermolecular C—H···O interactions involving atoms O1 and O4 and the hydrogen atoms of the butanoate moiety (Table 1).

Experimental

Under an atmosphere of Ar, (1-methoxyvinyl)trimethylsilane (1.06 g, 7.3 mmol) was dissolved in dry CH₂Cl₂ (15 ml) and the temperature lowered to 243K. 2-Azido-1-(2-nitrophenyl)ethanone (0.5 g, 2.4 mmol) dissolved in dry CH₂Cl₂ (6 ml) was added to the reaction mixture dropwise. A solution of TiCl₄ (0.13 ml, 1.2 mmol), freshly distilled over polyvinylpyridine, in dry CH₂Cl₂ (4 ml), was added slowly. The solution became immediately red and then dark red. The reaction mixture was stirred at 243K for 15 min and then at 258K for 30 min. The cold mixture was then poured into an aqueous solution of 2 N NaOH (2.4 ml) and extracted with chloroform. The combined organic layers were washed with brine, dried over MgSO₄ and concentrated under vacuum. Purification of the residue by flash chromatography (silica gel, CH₂Cl₂) followed by crystallization (ether/hexane) gave a white solid (Yield 76%). Colourless plate-like crystals, suitable for X-ray analysis, were obtained by slow evaporation of a solution in ether/hexane (v:v = 1:1)

Refinement

The H-atoms were located from difference Fourier maps and freely refined: O—H = 0.825 (19) Å, C—H = 0.91 (3) - 1.02 (2) Å.

Figures

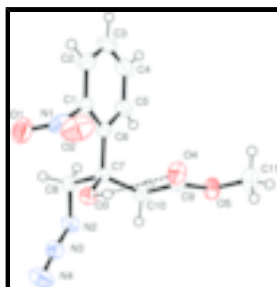


Fig. 1. Molecular structure of the title compound, showing the atom labelling scheme and the displacement ellipsoids drawn at the 50% probability level. The intramolecular O—H...O hydrogen bond is shown as a dashed line.

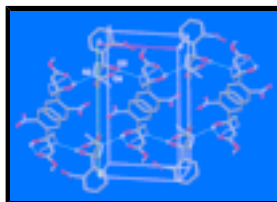


Fig. 2. A view along the *a* axis of the crystal packing of the title compound, showing the intra and intermolecular hydrogen bonds as dashed lines (see Table 1 for details).

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Crystal data

$C_{11}H_{12}N_4O_5$

$M_r = 280.25$

Monoclinic, $P2_1/n$

Hall symbol: -P 2yn

$a = 9.4772$ (11) Å

$b = 14.0710$ (12) Å

$c = 10.1861$ (12) Å

$\beta = 110.496$ (13)°

$V = 1272.4$ (2) Å³

$Z = 4$

$F_{000} = 584$

$D_x = 1.463$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 5300 reflections

$\theta = 2.6$ – 25.8 °

$\mu = 0.12$ mm⁻¹

$T = 153$ (2) K

Plate, colourless

$0.40 \times 0.30 \times 0.30$ mm

Data collection

Stoe IPDS
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 153$ (2) K

ϕ oscillation scans

1587 reflections with $I > 2\sigma(I)$

$R_{int} = 0.074$

$\theta_{max} = 25.9$ °

$\theta_{min} = 2.5$ °

$h = -11 \rightarrow 11$

Absorption correction: none $k = -17 \rightarrow 17$
 8743 measured reflections $l = -12 \rightarrow 12$
 2451 independent reflections

Refinement

Refinement on F^2 Hydrogen site location: difference Fourier map
 Least-squares matrix: full All H-atom parameters refined
 $R[F^2 > 2\sigma(F^2)] = 0.036$ $w = 1/[\sigma^2(F_o^2) + (0.0487P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $wR(F^2) = 0.088$ $(\Delta/\sigma)_{\max} < 0.001$
 $S = 0.87$ $\Delta\rho_{\max} = 0.23 \text{ e } \text{\AA}^{-3}$
 2451 reflections $\Delta\rho_{\min} = -0.21 \text{ e } \text{\AA}^{-3}$
 230 parameters Extinction correction: SHELXL97 (Sheldrick, 2008),
 $F_c^* = kF_c[1 + 0.001 \times F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Primary atom site location: structure-invariant direct methods Extinction coefficient: 0.0087 (18)
 Secondary atom site location: difference Fourier map

Special details

Geometry. Bond distances, angles *etc.* have been calculated using the rounded fractional coordinates. All su's are estimated from the variances of the (full) variance-covariance matrix. The cell e.s.d.'s are taken into account in the estimation of distances, angles and torsion angles

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	0.32528 (17)	-0.00702 (10)	0.18798 (17)	0.0599 (6)
O2	0.39794 (16)	0.06178 (11)	0.39169 (15)	0.0653 (5)
O3	0.20867 (11)	0.19457 (8)	0.17217 (12)	0.0272 (4)
O4	0.13982 (13)	0.26155 (8)	0.41688 (11)	0.0370 (4)
O5	-0.07815 (12)	0.34053 (9)	0.35528 (11)	0.0372 (4)
N1	0.30233 (17)	0.02628 (11)	0.28967 (16)	0.0424 (5)
N2	-0.03288 (14)	0.22705 (10)	-0.10334 (13)	0.0321 (4)
N3	0.08103 (17)	0.25861 (10)	-0.12165 (13)	0.0340 (5)
N4	0.17267 (19)	0.29547 (14)	-0.15016 (17)	0.0535 (6)
C1	0.1474 (2)	0.01792 (12)	0.29231 (16)	0.0330 (5)
C2	0.1235 (3)	-0.06299 (14)	0.35997 (18)	0.0472 (7)
C3	-0.0179 (3)	-0.07913 (17)	0.3639 (2)	0.0574 (9)
C4	-0.1334 (3)	-0.01579 (16)	0.3016 (2)	0.0524 (8)
C5	-0.1057 (2)	0.06436 (14)	0.23568 (19)	0.0391 (6)
C6	0.03622 (18)	0.08457 (11)	0.22890 (15)	0.0277 (5)

C7	0.05471 (16)	0.17238 (11)	0.14778 (15)	0.0253 (5)
C8	-0.01067 (19)	0.14495 (13)	-0.00855 (16)	0.0293 (5)
C9	0.02297 (16)	0.28584 (12)	0.32851 (16)	0.0263 (5)
C10	-0.02665 (18)	0.25954 (13)	0.17665 (17)	0.0293 (5)
C11	-0.0434 (3)	0.36959 (19)	0.4988 (2)	0.0489 (8)
H2	0.210 (2)	-0.1090 (17)	0.404 (2)	0.062 (6)*
H3	-0.039 (3)	-0.1301 (18)	0.409 (2)	0.071 (7)*
H3O	0.242 (2)	0.2153 (14)	0.253 (2)	0.043 (6)*
H4	-0.235 (3)	-0.0267 (16)	0.307 (2)	0.064 (6)*
H5	-0.183 (2)	0.1080 (15)	0.186 (2)	0.053 (6)*
H8A	0.0553 (19)	0.0951 (13)	-0.0298 (16)	0.032 (4)*
H8B	-0.113 (2)	0.1209 (12)	-0.0262 (17)	0.035 (4)*
H10A	-0.0016 (19)	0.3118 (13)	0.1284 (17)	0.037 (5)*
H10B	-0.133 (2)	0.2515 (14)	0.1442 (19)	0.047 (5)*
H11A	-0.035 (3)	0.315 (2)	0.560 (3)	0.084 (8)*
H11B	-0.128 (3)	0.4060 (17)	0.497 (2)	0.067 (7)*
H11C	0.044 (3)	0.4065 (17)	0.525 (2)	0.062 (6)*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0611 (9)	0.0464 (10)	0.0834 (11)	0.0058 (7)	0.0395 (8)	-0.0098 (8)
O2	0.0494 (8)	0.0595 (10)	0.0596 (9)	0.0016 (7)	-0.0151 (7)	0.0141 (8)
O3	0.0215 (6)	0.0308 (7)	0.0259 (6)	-0.0016 (5)	0.0039 (4)	-0.0006 (5)
O4	0.0298 (6)	0.0426 (8)	0.0307 (6)	0.0080 (5)	0.0006 (5)	-0.0054 (5)
O5	0.0262 (6)	0.0445 (8)	0.0401 (7)	0.0067 (5)	0.0108 (5)	-0.0081 (5)
N1	0.0440 (9)	0.0276 (9)	0.0482 (10)	0.0104 (7)	0.0069 (8)	0.0091 (7)
N2	0.0236 (7)	0.0413 (9)	0.0279 (7)	-0.0005 (6)	0.0048 (5)	0.0044 (6)
N3	0.0352 (8)	0.0377 (9)	0.0257 (7)	0.0053 (7)	0.0063 (6)	0.0049 (6)
N4	0.0431 (10)	0.0645 (12)	0.0571 (10)	-0.0023 (9)	0.0228 (8)	0.0169 (9)
C1	0.0452 (10)	0.0269 (10)	0.0264 (8)	-0.0012 (8)	0.0118 (7)	-0.0025 (7)
C2	0.0829 (15)	0.0269 (11)	0.0336 (10)	-0.0010 (11)	0.0226 (10)	0.0010 (8)
C3	0.109 (2)	0.0334 (13)	0.0435 (12)	-0.0247 (13)	0.0437 (13)	-0.0082 (9)
C4	0.0721 (15)	0.0475 (14)	0.0494 (12)	-0.0280 (12)	0.0360 (11)	-0.0173 (10)
C5	0.0432 (10)	0.0397 (11)	0.0370 (9)	-0.0133 (9)	0.0174 (8)	-0.0097 (9)
C6	0.0341 (9)	0.0255 (9)	0.0229 (7)	-0.0046 (7)	0.0091 (7)	-0.0049 (6)
C7	0.0201 (7)	0.0260 (9)	0.0269 (8)	-0.0015 (6)	0.0045 (6)	0.0003 (6)
C8	0.0272 (9)	0.0297 (10)	0.0272 (8)	-0.0021 (7)	0.0047 (7)	-0.0003 (7)
C9	0.0230 (8)	0.0223 (9)	0.0322 (8)	-0.0018 (7)	0.0080 (7)	0.0002 (7)
C10	0.0226 (8)	0.0299 (10)	0.0304 (9)	0.0017 (7)	0.0032 (7)	0.0012 (7)
C11	0.0456 (12)	0.0569 (15)	0.0476 (12)	0.0028 (11)	0.0207 (10)	-0.0157 (11)

Geometric parameters (\AA , $^\circ$)

O1—N1	1.224 (2)	C5—C6	1.400 (3)
O2—N1	1.221 (2)	C6—C7	1.530 (2)
O3—C7	1.426 (2)	C7—C10	1.531 (2)
O4—C9	1.206 (2)	C7—C8	1.542 (2)
O5—C9	1.330 (2)	C9—C10	1.497 (2)

O5—C11	1.441 (2)	C2—H2	1.02 (2)
O3—H3O	0.825 (19)	C3—H3	0.91 (2)
N1—C1	1.483 (3)	C4—H4	1.00 (3)
N2—C8	1.473 (2)	C5—H5	0.95 (2)
N2—N3	1.240 (2)	C8—H8A	1.012 (19)
N3—N4	1.133 (2)	C8—H8B	0.983 (19)
C1—C6	1.389 (2)	C10—H10A	0.959 (18)
C1—C2	1.390 (3)	C10—H10B	0.95 (2)
C2—C3	1.374 (4)	C11—H11A	0.98 (3)
C3—C4	1.382 (4)	C11—H11B	0.95 (3)
C4—C5	1.384 (3)	C11—H11C	0.93 (3)
C9—O5—C11	116.57 (15)	O4—C9—C10	125.11 (15)
C7—O3—H3O	105.2 (14)	C7—C10—C9	113.54 (14)
O1—N1—O2	125.32 (18)	C1—C2—H2	119.6 (12)
O2—N1—C1	117.49 (15)	C3—C2—H2	121.7 (12)
O1—N1—C1	117.11 (15)	C2—C3—H3	122.3 (18)
N3—N2—C8	116.57 (14)	C4—C3—H3	117.5 (18)
N2—N3—N4	171.07 (18)	C3—C4—H4	120.1 (13)
N1—C1—C6	122.19 (15)	C5—C4—H4	120.3 (13)
C2—C1—C6	123.7 (2)	C4—C5—H5	122.8 (13)
N1—C1—C2	114.10 (18)	C6—C5—H5	114.6 (12)
C1—C2—C3	118.7 (2)	N2—C8—H8A	111.1 (10)
C2—C3—C4	120.3 (2)	N2—C8—H8B	104.2 (10)
C3—C4—C5	119.6 (3)	C7—C8—H8A	109.8 (9)
C4—C5—C6	122.56 (19)	C7—C8—H8B	106.8 (10)
C1—C6—C7	125.77 (16)	H8A—C8—H8B	111.5 (15)
C5—C6—C7	118.97 (15)	C7—C10—H10A	106.5 (11)
C1—C6—C5	115.17 (16)	C7—C10—H10B	112.4 (12)
O3—C7—C6	112.72 (13)	C9—C10—H10A	107.2 (10)
O3—C7—C10	110.15 (13)	C9—C10—H10B	107.4 (11)
C6—C7—C8	105.96 (13)	H10A—C10—H10B	109.6 (16)
O3—C7—C8	104.56 (13)	O5—C11—H11A	111.3 (17)
C8—C7—C10	110.58 (13)	O5—C11—H11B	104.1 (12)
C6—C7—C10	112.49 (13)	O5—C11—H11C	108.2 (13)
N2—C8—C7	113.22 (14)	H11A—C11—H11B	109 (2)
O4—C9—O5	123.38 (14)	H11A—C11—H11C	113 (2)
O5—C9—C10	111.52 (14)	H11B—C11—H11C	111 (2)
C11—O5—C9—O4	-1.0 (3)	C4—C5—C6—C1	0.5 (3)
C11—O5—C9—C10	179.32 (17)	C4—C5—C6—C7	177.10 (16)
O1—N1—C1—C2	91.99 (19)	C1—C6—C7—O3	-15.3 (2)
O1—N1—C1—C6	-86.4 (2)	C1—C6—C7—C8	98.52 (18)
O2—N1—C1—C2	-84.7 (2)	C1—C6—C7—C10	-140.54 (16)
O2—N1—C1—C6	96.86 (19)	C5—C6—C7—O3	168.49 (14)
N3—N2—C8—C7	78.29 (18)	C5—C6—C7—C8	-77.74 (18)
N1—C1—C2—C3	-177.75 (16)	C5—C6—C7—C10	43.21 (19)
C6—C1—C2—C3	0.6 (3)	O3—C7—C8—N2	-73.41 (17)
N1—C1—C6—C5	177.43 (15)	C6—C7—C8—N2	167.29 (14)
N1—C1—C6—C7	1.1 (2)	C10—C7—C8—N2	45.12 (19)

C2—C1—C6—C5	-0.8 (2)	O3—C7—C10—C9	-69.34 (17)
C2—C1—C6—C7	-177.19 (15)	C6—C7—C10—C9	57.34 (19)
C1—C2—C3—C4	0.0 (3)	C8—C7—C10—C9	175.59 (14)
C2—C3—C4—C5	-0.3 (3)	O4—C9—C10—C7	19.8 (2)
C3—C4—C5—C6	0.1 (3)	O5—C9—C10—C7	-160.54 (14)

Hydrogen-bond geometry (\AA , $^\circ$)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O3—H3O \cdots O4	0.825 (19)	2.30 (2)	2.9439 (16)	135.5 (18)
O3—H3O \cdots N2 ⁱ	0.825 (19)	2.27 (2)	2.9193 (18)	135.6 (18)
C10—H10B \cdots O4 ⁱⁱ	0.95 (2)	2.557 (19)	3.350 (2)	141.0 (15)
C11—H11B \cdots O1 ⁱⁱⁱ	0.95 (3)	2.57 (2)	3.268 (3)	130.9 (16)

Symmetry codes: (i) $x+1/2, -y+1/2, z+1/2$; (ii) $x-1/2, -y+1/2, z-1/2$; (iii) $x-1/2, -y+1/2, z+1/2$.

Fig. 2

