The title compound, C₉H₁₀N₂O₅, crystallizes with two crystallographically independent molecules in the asymmetric unit. In the crystal structure, the nitropyridone rings are connected by weak C—H⋯O interactions, forming sheet-like arrays, which are in turn linked by C—H⋯π and π⋯π interactions between the nitropyridone rings on one side, and by C—H⋯O and van der Waals interactions between the ester groups on the other.

Comment

Pyridones can act as a P3 to P2 conformational restraint in the design of inhibitors of serine protease enzymes (Loughlin et al., 2004). This is often facilitated by the presence of a 3-amino group, which acts as a hydrogen-bonding site. Typically, 3-nitropyrid-2-ones (I) and (II) are used as synthetic precursors to 3-aminopyrid-2-ones (Breslin et al., 2003; Huang et al., 2003; Reiner et al., 2002; Warner et al., 1994). Compound (II) lacks the hydrogen-bonding site at the 3 position and solid-state structures for 3-nitropyridones (without C₄ to C₆ substituents) are restricted to a report of the complex of (I) with 2-amino-5-nitropyridine (Velikova et al., 1997). Here, we report the first solid-state structure of the title compound, (II). Compound (II) was prepared by N-alkylation of (I) with sodium hydride and ethyl bromoacetate, as reported elsewhere (Warner et al., 1994; Breslin et al., 2003).

The crystal structure of (II) contains two crystallographically independent molecules in the asymmetric unit (Figs. 1 and 2). Relevant bond lengths and angles are listed in Table 1 and are in accord with those reported for 3-nitro-2-pyridone (Velikova et al., 1997). The nitro groups are twisted slightly out of the plane of the pyridone ring, with torsion angles O32—N3—C3—C2 = 18.5 (4)° and O132—N13—C13—C12 = 22.4 (4)°.

In the crystal structure, the nitopyridone rings are connected by weak C—H⋯O interactions between the aromatic and aliphatic H atoms and the carbonyl and nitro group O atoms, forming sheet-like arrays in the bc plane. These sheets are in turn linked together by C—H⋯π and π⋯π interactions between the nitopyridone rings on one side, and by C—H⋯O and van der Waals interactions between the ester groups on the other (Fig. 2).
Spectroscopic analysis: $^1$H NMR (400 MHz, CDCl$_3$, 2006). 

**Experimental**

Sodium hydride (1.38 g, 57.8 mmol) was added in portions over a period of 30 min to compound (I) (6.75 g, 48.2 mmol) in dry tetrahydrofuran (100 ml). The resulting suspension was stirred for 30 min. Ethyl bromoacetate (5.86 ml, 53.0 mmol) was added dropwise over a period of 30 min. The resulting yellow suspension was heated to 328 K under nitrogen for 24 h. The red reaction mixture was filtered and the solid thoroughly washed with ethyl acetate. The filtrate was concentrated under reduced pressure and the resulting red oil purified by silica-gel column chromatography (ethyl acetate–dichloromethane gradient from 0–40% ethyl acetate, with 0.4% triethylamine). Pale-yellow crystals of compound (II) (m.p. 327–329 K; yield 10.7 g, 98%) were isolated by slow evaporation of an ethyl acetate solution of (II). Analysis, found: C 47.82, H 4.44, N 12.34%; calculated for C$_9$H$_{10}$N$_2$O$_5$: C 47.79, H 4.46, N 12.38%. Spectroscopic analysis: $^1$H NMR (400 MHz, CDCl$_3$, 6, p.p.m.): 1.293 (3H, t, $J = 7.2$ Hz, CH$_3$), 4.251 (2H, q, $J = 7.2$ Hz, CH$_2$), 4.769 (2H, s, NCH$_2$), 6.380 (1H, dd, $J = 7.6$ and 6.8 Hz, H5), 7.712 (1H, dd, $J = 6.8$ and 2.0 Hz, H6), 8.387 (1H, dd, $J = 7.6$ and 2.0 Hz, H4); $^{13}$C NMR (100 MHz, CDCl$_3$, $\delta$, p.p.m.): 14.3 (CH$_3$), 51.4 (NCH$_2$), 62.6 (CH$_2$), 103.8 (C5), 138.9 (C3), 139.7 (C4), 145.2 (C6), 154.5 (C2), 166.8 (CO); MS (ES$^+$): 248.8 (MNa$^+$, 60%) 226.8 (MH$^+$, 70%) 180.7 (MH$^+$ – NO$_2$, 100%).

**Crystal data**

C$_9$H$_{10}$N$_2$O$_5$  
$M_r = 226.19$  
Monoclinic. \( P2_1/c \)  
$\alpha = 20.175 (4)$ Å  
$\beta = 12.729 (4)$ Å  
$\gamma = 20.175 (4)$ Å  
$\beta = 90.00$°  
$V = 2104.2 (11)$ Å$^3$  

**Data collection**

Rigaku AFC-7R diffractometer  
$\omega/2$θ scans  
Absorption correction: none  
4183 measured reflections  
3689 independent reflections  
1988 reflections with $I > 2\sigma(I)$  

**Refinement**

Refinement on $F^2$  
$R[F^2 > 2\sigma(F^2)] = 0.045$  
$wR(F^2) = 0.143$  
$S = 1.03$  
3689 reflections  
289 parameters  
H-atom parameters constrained

**Table 1**

Selected geometric parameters (Å, °).  

|          | O2–C2   | O31–N3   | O32–N3   | O12–C12  | O18–C18  | O19–C19  | O19–C18  |
|----------|---------|----------|----------|----------|----------|----------|----------|
| C8–O9–C9| 118.6 (2) | 116.4 (2) | 125.5 (2) | 116.4 (2) | 118.6 (2) | 116.4 (2) | 118.6 (2) |
| C8–O9–C9| 116.4 (2) | 118.6 (2) | 116.4 (2) | 116.4 (2) | 118.6 (2) | 116.4 (2) | 118.6 (2) |
| C8–O9–C9| 118.6 (2) | 116.4 (2) | 118.6 (2) | 116.4 (2) | 118.6 (2) | 116.4 (2) | 118.6 (2) |

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Table 2
Hydrogen-bond geometry (Å, °).

| D—H—A  | D—H  | H···A  | D···A  | D—H—A  |
|---------|-------|--------|--------|--------|
| C4—H4  | 0.95  | 2.24   | 3.117  | (3) 154|
| C5—H5  | 0.95  | 2.64   | 3.457  | (4) 145|
| C16—H16 | 0.95 | 2.17   | 3.049  | (4) 153|
| C17—H17 | 0.95 | 2.67   | 3.536  | (4) 152|
| C9—H9  | 0.95  | 2.57   | 3.517  | (5) 173|

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

H atoms were constrained as riding atoms, with C—H = 0.95 Å, and with Uiso(H) = 1.2 Ueq(C).

Data collection: MSC/AFC7 Diffractometer Control Software (Molecular Structure Corporation, 1999); cell refinement: MSC/AFC7 Diffractometer Control Software; data reduction: TEXSAN for Windows (Molecular Structure Corporation, 2001); program(s) used to solve structure: TEXSAN for Windows; program(s) used to refine structure: TEXSAN for Windows and SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: TEXSAN for Windows and PLATON (Spek, 2003).

The authors acknowledge financial support of this work by Griffith University, by Eskitis Institute of Cell and Molecular Therapies, Griffith University, and by Natural Product Discovery, Griffith University.

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Figure 3
The crystal packing of (I), viewed down the b axis. Dashed lines indicate hydrogen bonds.