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# Ethyl 4-(*tert*-butylamino)-3-nitrobenzoate

**Siti Marina Mohd. Maidin,[a] Aisyah Saad Abdul Rahim,[a]‡ Hasnah Osman,[b] Reza Kia[c] and Hoong-Kun Fun[c]***

[a]School of Pharmaceutical Sciences, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, [b]School of Chemical Sciences, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia, and [c]X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia
Correspondence e-mail: hkfun@usm.my

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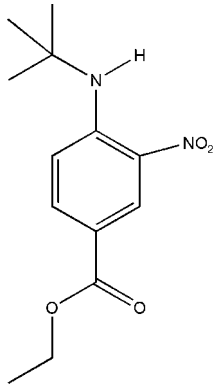
Key indicators: single-crystal X-ray study; $T$ = 100 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.047; $wR$ factor = 0.134; data-to-parameter ratio = 23.2.

In the title compound, $C_{13}H_{18}N_2O_4$, intramolecular N—H···O, N—H···N and C—H···O (× 3) hydrogen bonds generate $S$(6) and $S$(5) ring motifs. There are two crystallographically independent molecules (*A* and *B*) in the asymmetric unit. The nitro group is coplanar with the benzene ring, with O—N—C—C torsion angles of −0.33 (13) and 0.93 (14)° in molecules *A* and *B*, respectively. In the crystal structure, neighbouring molecules are linked together by intermolecular C—H···O hydrogen bonds. In addition, the crystal structure is stabilized by $\pi$–$\pi$ interactions with centroid–centroid distances ranging from 3.7853 (6) to 3.8625 (6) Å.

## Related literature

For literature on hydrogen-bond motifs, see: Bernstein *et al.* (1995). For values of bond lengths, see: Allen *et al.* (1987). For related literature, see, for example: Göker *et al.* (1998); Anderson (2005); Kakei *et al.* (1993).




## Experimental

### Crystal data

$C_{13}H_{18}N_2O_4$
$M_r$ = 266.29
Monoclinic, $P2_1/c$
$a$ = 16.0471 (5) Å
$b$ = 6.6417 (2) Å
$c$ = 30.0180 (9) Å
$\beta$ = 121.688 (2)°
$V$ = 2722.37 (14) Å$^3$
$Z$ = 8
Mo $K\alpha$ radiation
$\mu$ = 0.10 mm$^{-1}$
$T$ = 100.0 (1) K
0.51 × 0.43 × 0.17 mm

### Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
$T_{min}$ = 0.879, $T_{max}$ = 0.984
63326 measured reflections
8141 independent reflections
6368 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.033

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.133
$S$ = 1.04
8141 reflections
351 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.53 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.24 e Å$^{-3}$

**Table 1**
Selected centroid–centroid distances (Å).

*Cg*1 and *Cg*2 are the centroids of the C1*A*–C6*A* and C1*B*–C6*B* rings, respectively.

| | | | |
|---|---|---|---|
| *Cg*1···*Cg*2$^{i}$ | 3.7853 (6) | *Cg*1···*Cg*2$^{ii}$ | 3.8625 (6) |

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

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

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2*A*—H2*NA*···O4*A* | 0.86 | 1.93 | 2.6299 (15) | 138 |
| N2*A*—H2*NA*···N1*A* | 0.86 | 2.54 | 2.9361 (15) | 109 |
| N2*B*—H2*NB*···O4*B* | 0.87 | 1.95 | 2.6355 (15) | 134 |
| N2*B*—H2*NB*···N1*B* | 0.87 | 2.58 | 2.9419 (15) | 106 |
| C1*A*—H1*A*···O3*A* | 0.95 | 2.31 | 2.6522 (16) | 100 |
| C1*B*—H1*B*···O3*B* | 0.95 | 2.31 | 2.6498 (16) | 100 |
| C4*A*—H4*A*···O3*B*$^{iii}$ | 0.95 | 2.50 | 3.4124 (17) | 160 |
| C4*B*—H4*B*···O3*A*$^{iv}$ | 0.95 | 2.42 | 3.2566 (18) | 147 |
| C5*A*—H5*A*···O1*A* | 0.95 | 2.41 | 2.7326 (13) | 100 |
| C11*A*—H11*A*···O2*B*$^{v}$ | 0.98 | 2.55 | 3.4714 (17) | 157 |
| C11*B*—H11*F*···O2*A*$^{ii}$ | 0.98 | 2.52 | 3.4446 (17) | 158 |
| C13*B*—H13*D*···O2*A*$^{vi}$ | 0.98 | 2.60 | 3.5071 (17) | 154 |

Symmetry codes: (ii) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (iii) $-x+2, -y+1, -z+1$; (iv) $-x+1, -y+1, -z+1$; (v) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (vi) $x, -y+\frac{3}{2}, z-\frac{1}{2}$.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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‡ Additional correspondence author, e-mail: aisyah@usm.my.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: AT2594).

## References


Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.

Anderson, J. B. (2005). *Nat. Rev. Microbiol.* **3**, 547–556.

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.

Bruker (2005). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.

Göker, H., Tunçbilek, M., Ayhan, G. & Altanlar, N. (1998). *Farmaco*, **53**, 415–420.

Kakei, N., Ichinose, M., Tsukada, S., Tatematsu, M., Tezuka, N., Yahagi, N., Matsushima, M., Miki, K., Kurokawa, K., Takahashi, K. & Fukamachi, H. (1993). *Biochem. Biophys. Res. Commun.* **195**, 997–1004.

Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.

# supplementary materials

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# Ethyl 4-(*tert*-butylamino)-3-nitrobenzoate

**S. M. Mohd. Maidin, A. S. Abdul Rahim, H. Osman, R. Kia and H.-K. Fun**

## Comment

As a part of our ongoing studies on new nitro benzoic acid derivatives, we have synthesized the title compound (I) employing a modified protocol of previous procedure (Göker *et al.*, 1998). The nitro benzoic acid intermediates are a convenient starting material for the synthesis of heterocycles targeting important biological processes, *e.g.* antifungal (Fluconazole) (Anderson, 2005) and proton pump inhibitor (Omeprazole) (Kakei *et al.*, 1993). The crystal structure of the *tert*-butylamino functionalized nitro benzoic acid (I) has been determined, and herein, we present a full report on its crystal structure.

In the title compound (I) (Fig. 1), intramolecular N—H···O (*x* 2), N—H···N (*x* 2), and C—H···O (*x* 3) hydrogen bonds (Table 2) generate *S*(6) and *S*(5) ring motifs, respectively (Bernstein *et al.*, 1995). There are two crystallographically independent molecules, A and B in the asymmetric unit of the title compound. The nitro group is coplanar with the benzene ring with torsion angle of -0.33 (13) and 0.93 (14)° in the molecule A and B, respectively. In the crystal structure neighbouring molecules are linked together by intermolecular C—H···O hydrogen bonds (Table 1). In the crystal packing (Table 2 & Fig. 2), molecules are stacked down the *b* axis, being consolidated by π–π interactions with centroid to centroid distances ranging from 3.7853 (6) – 3.8625 (6) Å.

The crystal structure is stabilized by intramolecular N—H···O (*x* 2), N—H···N (*x* 2), C—H···O (*x* 3), and intermolecular C—H···O (*x* 5) hydrogen bonds and π–π interactions.

## Experimental

Ethyl 4-fluoro-3-nitrobenzoate (200 mg, 0.93 mmol) was dissolved in dry dichloromethane (10 ml). *N*, *N*-diisopropylethylamine (DIPEA) (0.20 ml, 1.12 mmol) was added to the stirred mixture. Then, *tert*-butylamine (0.11 ml, 1.03 mmol) was added dropwise using syringe and stirred at room temperature under $N_2$ overnight. After completion of the reaction, the mixture was washed with 10% $NaCO_3$ (10 ml). The aqueous layer was washed with dichloromethane (3 x 15 ml). The organic layers were collected and dried over $MgSO_4$ (anhydrous). The solvent was removed under reduced pressure to yield the crude product. Recrystallisation with hot hexane revealed the title compound (I) as bright yellow crystals.

## Refinement

The H-atoms attached to N2A and N2B were located from the difference Fourier map and refined as riding with the parent atom with an isotropic thermal parameter 1.2 times that of the parent atom. The rest of the hydrogen atoms were positioned geometrically [C—H = 0.95–98 Å] and refined using a riding model. A rotating-group model was used for the methyl groups. The highest peak is located 0.63 Å from C6B and the deepest hole is located 0.59 Å from N1A.

## Figures

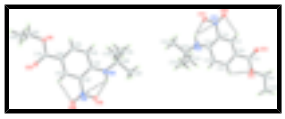

Fig. 1. The molecular structure of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering. Intramolecular hydrogen bonds are drawn as dashed lines.

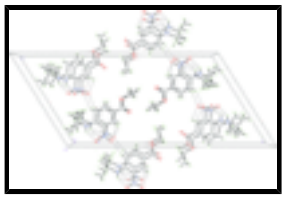

Fig. 2. The crystal packing of (I), showing stacking arrangement viewed down the *b*-axis. Intramolecular and intermolecular interactions are drawn as dashed lines.

## Ethyl 4-(*tert*-butylamino)-3-nitrobenzoate

*Crystal data*

| | |
|---|---|
| $C_{13}H_{18}N_2O_4$ | $F_{000}$ = 1136 |
| $M_r$ = 266.29 | $D_x$ = 1.299 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 9969 reflections |
| $a$ = 16.0471 (5) Å | θ = 2.5–30.1° |
| $b$ = 6.6417 (2) Å | μ = 0.10 mm$^{-1}$ |
| $c$ = 30.0180 (9) Å | $T$ = 100.0 (1) K |
| β = 121.688 (2)° | Plate, yellow |
| $V$ = 2722.37 (14) Å$^3$ | 0.51 × 0.43 × 0.17 mm |
| $Z$ = 8 | |

*Data collection*

| | |
|---|---|
| Bruker SMART APEXII CCD area-detector diffractometer | 8141 independent reflections |
| Radiation source: fine-focus sealed tube | 6368 reflections with $I > 2\sigma(I)$ |
| Monochromator: graphite | $R_{int}$ = 0.033 |
| $T$ = 100.0(1) K | $\theta_{max}$ = 30.3° |
| φ and ω scans | $\theta_{min}$ = 1.5° |
| Absorption correction: multi-scan (SADABS; Bruker, 2005) | $h$ = −21→22 |
| $T_{min}$ = 0.879, $T_{max}$ = 0.984 | $k$ = −9→9 |
| 63326 measured reflections | $l$ = −41→42 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)]$ = 0.047 | H-atom parameters constrained |

| | |
|---|---|
| $wR(F^2) = 0.133$ | $w = 1/[\sigma^2(F_o^2) + (0.0652P)^2 + 0.787P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.04$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 8141 reflections | $\Delta\rho_{max} = 0.53$ e Å$^{-3}$ |
| 351 parameters | $\Delta\rho_{min} = -0.24$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

*Special details*

**Experimental**. The low-temperature data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**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 (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1A | 1.01188 (6) | 0.50707 (13) | 0.90700 (3) | 0.02027 (18) |
| O2A | 0.87073 (6) | 0.53054 (14) | 0.90534 (3) | 0.02371 (19) |
| O3A | 0.57249 (6) | 0.54152 (13) | 0.73478 (3) | 0.02243 (18) |
| O4A | 0.56803 (6) | 0.54680 (13) | 0.66152 (3) | 0.02250 (18) |
| N1A | 0.61441 (6) | 0.54228 (13) | 0.71013 (4) | 0.01589 (18) |
| N2A | 0.73120 (6) | 0.54415 (13) | 0.66080 (3) | 0.01583 (18) |
| H2NA | 0.6682 | 0.5497 | 0.6444 | 0.019* |
| C1A | 0.76647 (7) | 0.53258 (15) | 0.79329 (4) | 0.0143 (2) |
| H1A | 0.7281 | 0.5335 | 0.8089 | 0.017* |
| C2A | 0.72036 (7) | 0.53705 (15) | 0.73898 (4) | 0.01349 (19) |
| C3A | 0.77441 (7) | 0.53716 (14) | 0.71324 (4) | 0.01347 (19) |
| C4A | 0.87825 (7) | 0.52932 (15) | 0.74731 (4) | 0.0151 (2) |
| H4A | 0.9180 | 0.5271 | 0.7325 | 0.018* |
| C5A | 0.92253 (7) | 0.52490 (15) | 0.80055 (4) | 0.0149 (2) |
| H5A | 0.9920 | 0.5204 | 0.8217 | 0.018* |
| C6A | 0.86747 (7) | 0.52682 (15) | 0.82473 (4) | 0.0142 (2) |
| C7A | 0.91408 (8) | 0.52224 (16) | 0.88226 (4) | 0.0159 (2) |
| C8A | 1.06291 (8) | 0.5008 (2) | 0.96363 (4) | 0.0252 (3) |
| H8A | 1.0412 | 0.3828 | 0.9751 | 0.030* |
| H8B | 1.0489 | 0.6242 | 0.9771 | 0.030* |
| C9A | 1.17025 (9) | 0.4861 (2) | 0.98412 (5) | 0.0347 (3) |
| H9A | 1.2069 | 0.4877 | 1.0225 | 0.052* |
| H9B | 1.1904 | 0.6006 | 0.9713 | 0.052* |
| H9C | 1.1837 | 0.3603 | 0.9720 | 0.052* |

| | | | | |
|---|---|---|---|---|
| C10A | 0.77667 (8) | 0.54745 (16) | 0.62878 (4) | 0.0163 (2) |
| C11A | 0.83246 (8) | 0.35165 (17) | 0.63510 (4) | 0.0203 (2) |
| H11A | 0.7877 | 0.2370 | 0.6254 | 0.030* |
| H11B | 0.8861 | 0.3373 | 0.6716 | 0.030* |
| H11C | 0.8593 | 0.3555 | 0.6124 | 0.030* |
| C12A | 0.68959 (9) | 0.56150 (19) | 0.57218 (4) | 0.0233 (2) |
| H12A | 0.6458 | 0.4465 | 0.5647 | 0.035* |
| H12B | 0.7136 | 0.5600 | 0.5481 | 0.035* |
| H12C | 0.6537 | 0.6869 | 0.5676 | 0.035* |
| C13A | 0.84047 (8) | 0.73502 (17) | 0.64034 (4) | 0.0195 (2) |
| H13A | 0.8927 | 0.7359 | 0.6774 | 0.029* |
| H13B | 0.8000 | 0.8560 | 0.6324 | 0.029* |
| H13C | 0.8695 | 0.7331 | 0.6186 | 0.029* |
| O1B | 0.49200 (5) | 0.43211 (13) | 0.10402 (3) | 0.02139 (18) |
| O2B | 0.63293 (6) | 0.45058 (13) | 0.10484 (3) | 0.02286 (18) |
| O3B | 0.93204 (6) | 0.47400 (13) | 0.27341 (3) | 0.02111 (18) |
| O4B | 0.93898 (6) | 0.47557 (13) | 0.34743 (3) | 0.02151 (18) |
| N1B | 0.89147 (6) | 0.47252 (13) | 0.29872 (4) | 0.01529 (18) |
| N2B | 0.77682 (6) | 0.47199 (14) | 0.34950 (3) | 0.01565 (18) |
| H2NB | 0.8407 | 0.4767 | 0.3671 | 0.019* |
| C1B | 0.73817 (7) | 0.46115 (15) | 0.21633 (4) | 0.01401 (19) |
| H1B | 0.7757 | 0.4608 | 0.2002 | 0.017* |
| C2B | 0.78566 (7) | 0.46722 (15) | 0.27069 (4) | 0.01293 (19) |
| C3B | 0.73273 (7) | 0.46737 (15) | 0.29702 (4) | 0.01340 (19) |
| C4B | 0.62889 (8) | 0.46322 (16) | 0.26366 (4) | 0.0157 (2) |
| H4B | 0.5900 | 0.4645 | 0.2790 | 0.019* |
| C5B | 0.58317 (7) | 0.45740 (15) | 0.21029 (4) | 0.0154 (2) |
| H5B | 0.5137 | 0.4545 | 0.1895 | 0.019* |
| C6B | 0.63721 (7) | 0.45564 (15) | 0.18553 (4) | 0.01401 (19) |
| C7B | 0.58976 (8) | 0.44669 (16) | 0.12800 (4) | 0.0168 (2) |
| C8B | 0.43816 (8) | 0.4179 (2) | 0.04737 (4) | 0.0260 (3) |
| H8C | 0.3751 | 0.3484 | 0.0350 | 0.031* |
| H8D | 0.4762 | 0.3370 | 0.0365 | 0.031* |
| C9B | 0.41883 (9) | 0.6226 (2) | 0.02271 (5) | 0.0302 (3) |
| H9D | 0.3804 | 0.6089 | −0.0155 | 0.045* |
| H9E | 0.4812 | 0.6888 | 0.0335 | 0.045* |
| H9F | 0.3822 | 0.7038 | 0.0340 | 0.045* |
| C10B | 0.73206 (8) | 0.46992 (16) | 0.38189 (4) | 0.0172 (2) |
| C11B | 0.67381 (8) | 0.27595 (17) | 0.37340 (4) | 0.0217 (2) |
| H11D | 0.6189 | 0.2698 | 0.3370 | 0.032* |
| H11E | 0.6487 | 0.2747 | 0.3969 | 0.032* |
| H11F | 0.7166 | 0.1593 | 0.3809 | 0.032* |
| C12B | 0.81999 (9) | 0.47351 (19) | 0.43837 (4) | 0.0246 (2) |
| H12D | 0.8582 | 0.5964 | 0.4441 | 0.037* |
| H12E | 0.8613 | 0.3555 | 0.4445 | 0.037* |
| H12F | 0.7966 | 0.4710 | 0.4627 | 0.037* |
| C13B | 0.66997 (8) | 0.65807 (18) | 0.37269 (5) | 0.0222 (2) |
| H13D | 0.7108 | 0.7784 | 0.3806 | 0.033* |
| H13E | 0.6433 | 0.6541 | 0.3955 | 0.033* |

| | | | | |
|---|---|---|---|---|
| H13F | 0.6160 | 0.6623 | 0.3360 | 0.033* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1A | 0.0132 (3) | 0.0327 (5) | 0.0134 (4) | 0.0004 (3) | 0.0059 (3) | −0.0014 (3) |
| O2A | 0.0181 (4) | 0.0365 (5) | 0.0190 (4) | 0.0000 (3) | 0.0114 (3) | −0.0013 (3) |
| O3A | 0.0154 (4) | 0.0298 (5) | 0.0260 (4) | 0.0006 (3) | 0.0135 (3) | 0.0003 (3) |
| O4A | 0.0136 (4) | 0.0309 (5) | 0.0186 (4) | 0.0002 (3) | 0.0055 (3) | −0.0002 (3) |
| N1A | 0.0126 (4) | 0.0142 (4) | 0.0205 (4) | −0.0004 (3) | 0.0084 (3) | −0.0004 (3) |
| N2A | 0.0120 (4) | 0.0192 (4) | 0.0151 (4) | 0.0004 (3) | 0.0062 (3) | 0.0003 (3) |
| C1A | 0.0149 (5) | 0.0111 (4) | 0.0189 (5) | −0.0006 (3) | 0.0104 (4) | −0.0004 (4) |
| C2A | 0.0102 (4) | 0.0119 (5) | 0.0175 (5) | 0.0002 (3) | 0.0068 (4) | 0.0002 (4) |
| C3A | 0.0135 (4) | 0.0099 (4) | 0.0168 (5) | −0.0006 (3) | 0.0078 (4) | −0.0003 (3) |
| C4A | 0.0127 (4) | 0.0155 (5) | 0.0182 (5) | 0.0003 (4) | 0.0088 (4) | 0.0005 (4) |
| C5A | 0.0124 (4) | 0.0145 (5) | 0.0175 (5) | 0.0003 (4) | 0.0076 (4) | 0.0006 (4) |
| C6A | 0.0147 (5) | 0.0131 (5) | 0.0148 (5) | −0.0004 (4) | 0.0077 (4) | −0.0005 (4) |
| C7A | 0.0140 (5) | 0.0160 (5) | 0.0170 (5) | −0.0009 (4) | 0.0075 (4) | −0.0013 (4) |
| C8A | 0.0188 (5) | 0.0421 (7) | 0.0122 (5) | 0.0004 (5) | 0.0065 (4) | −0.0021 (5) |
| C9A | 0.0184 (6) | 0.0630 (10) | 0.0180 (6) | 0.0030 (6) | 0.0063 (5) | −0.0027 (6) |
| C10A | 0.0161 (5) | 0.0183 (5) | 0.0154 (5) | −0.0005 (4) | 0.0088 (4) | 0.0001 (4) |
| C11A | 0.0208 (5) | 0.0181 (5) | 0.0240 (5) | −0.0002 (4) | 0.0131 (4) | −0.0028 (4) |
| C12A | 0.0212 (5) | 0.0303 (6) | 0.0157 (5) | −0.0009 (5) | 0.0078 (4) | −0.0005 (4) |
| C13A | 0.0205 (5) | 0.0190 (5) | 0.0203 (5) | −0.0011 (4) | 0.0116 (4) | 0.0018 (4) |
| O1B | 0.0133 (4) | 0.0343 (5) | 0.0138 (4) | −0.0022 (3) | 0.0053 (3) | 0.0013 (3) |
| O2B | 0.0181 (4) | 0.0339 (5) | 0.0178 (4) | 0.0012 (3) | 0.0102 (3) | −0.0004 (3) |
| O3B | 0.0150 (4) | 0.0280 (4) | 0.0238 (4) | 0.0003 (3) | 0.0127 (3) | 0.0009 (3) |
| O4B | 0.0136 (4) | 0.0302 (5) | 0.0164 (4) | 0.0002 (3) | 0.0049 (3) | 0.0004 (3) |
| N1B | 0.0135 (4) | 0.0134 (4) | 0.0186 (4) | 0.0001 (3) | 0.0081 (3) | 0.0002 (3) |
| N2B | 0.0129 (4) | 0.0195 (4) | 0.0143 (4) | 0.0001 (3) | 0.0070 (3) | 0.0001 (3) |
| C1B | 0.0144 (4) | 0.0125 (5) | 0.0168 (5) | 0.0004 (3) | 0.0093 (4) | 0.0006 (4) |
| C2B | 0.0108 (4) | 0.0113 (4) | 0.0166 (5) | 0.0003 (3) | 0.0072 (4) | 0.0007 (3) |
| C3B | 0.0147 (5) | 0.0101 (4) | 0.0151 (5) | 0.0004 (3) | 0.0077 (4) | 0.0010 (3) |
| C4B | 0.0137 (4) | 0.0173 (5) | 0.0185 (5) | 0.0004 (4) | 0.0100 (4) | 0.0005 (4) |
| C5B | 0.0121 (4) | 0.0151 (5) | 0.0185 (5) | 0.0000 (4) | 0.0076 (4) | 0.0007 (4) |
| C6B | 0.0135 (4) | 0.0133 (5) | 0.0148 (5) | 0.0000 (3) | 0.0071 (4) | 0.0009 (3) |
| C7B | 0.0152 (5) | 0.0170 (5) | 0.0163 (5) | 0.0001 (4) | 0.0070 (4) | 0.0007 (4) |
| C8B | 0.0176 (5) | 0.0410 (7) | 0.0148 (5) | −0.0037 (5) | 0.0055 (4) | −0.0017 (5) |
| C9B | 0.0236 (6) | 0.0469 (8) | 0.0197 (6) | 0.0022 (5) | 0.0111 (5) | 0.0074 (5) |
| C10B | 0.0194 (5) | 0.0187 (5) | 0.0158 (5) | 0.0009 (4) | 0.0107 (4) | 0.0007 (4) |
| C11B | 0.0255 (6) | 0.0209 (5) | 0.0226 (5) | −0.0013 (4) | 0.0154 (5) | 0.0024 (4) |
| C12B | 0.0265 (6) | 0.0306 (6) | 0.0152 (5) | 0.0009 (5) | 0.0099 (5) | 0.0008 (4) |
| C13B | 0.0242 (5) | 0.0205 (5) | 0.0251 (6) | 0.0019 (4) | 0.0151 (5) | −0.0022 (4) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1A—C7A | 1.3418 (13) | O1B—C7B | 1.3424 (13) |
| O1A—C8A | 1.4489 (13) | O1B—C8B | 1.4503 (13) |
| O2A—C7A | 1.2136 (13) | O2B—C7B | 1.2125 (13) |

| | | | |
|---|---|---|---|
| O3A—N1A | 1.2340 (12) | O3B—N1B | 1.2332 (12) |
| O4A—N1A | 1.2424 (12) | O4B—N1B | 1.2443 (12) |
| N1A—C2A | 1.4474 (13) | N1B—C2B | 1.4462 (13) |
| N2A—C3A | 1.3470 (13) | N2B—C3B | 1.3463 (13) |
| N2A—C10A | 1.4810 (13) | N2B—C10B | 1.4816 (13) |
| N2A—H2NA | 0.8617 | N2B—H2NB | 0.8733 |
| C1A—C6A | 1.3827 (14) | C1B—C6B | 1.3809 (14) |
| C1A—C2A | 1.3925 (14) | C1B—C2B | 1.3924 (14) |
| C1A—H1A | 0.9500 | C1B—H1B | 0.9500 |
| C2A—C3A | 1.4328 (14) | C2B—C3B | 1.4321 (14) |
| C3A—C4A | 1.4265 (14) | C3B—C4B | 1.4238 (14) |
| C4A—C5A | 1.3663 (14) | C4B—C5B | 1.3683 (14) |
| C4A—H4A | 0.9500 | C4B—H4B | 0.9500 |
| C5A—C6A | 1.4076 (14) | C5B—C6B | 1.4076 (14) |
| C5A—H5A | 0.9500 | C5B—H5B | 0.9500 |
| C6A—C7A | 1.4784 (14) | C6B—C7B | 1.4778 (14) |
| C8A—C9A | 1.4973 (17) | C8B—C9B | 1.5003 (19) |
| C8A—H8A | 0.9900 | C8B—H8C | 0.9900 |
| C8A—H8B | 0.9900 | C8B—H8D | 0.9900 |
| C9A—H9A | 0.9800 | C9B—H9D | 0.9800 |
| C9A—H9B | 0.9800 | C9B—H9E | 0.9800 |
| C9A—H9C | 0.9800 | C9B—H9F | 0.9800 |
| C10A—C13A | 1.5320 (15) | C10B—C13B | 1.5302 (15) |
| C10A—C11A | 1.5331 (15) | C10B—C11B | 1.5323 (15) |
| C10A—C12A | 1.5343 (15) | C10B—C12B | 1.5338 (15) |
| C11A—H11A | 0.9800 | C11B—H11D | 0.9800 |
| C11A—H11B | 0.9800 | C11B—H11E | 0.9800 |
| C11A—H11C | 0.9800 | C11B—H11F | 0.9800 |
| C12A—H12A | 0.9800 | C12B—H12D | 0.9800 |
| C12A—H12B | 0.9800 | C12B—H12E | 0.9800 |
| C12A—H12C | 0.9800 | C12B—H12F | 0.9800 |
| C13A—H13A | 0.9800 | C13B—H13D | 0.9800 |
| C13A—H13B | 0.9800 | C13B—H13E | 0.9800 |
| C13A—H13C | 0.9800 | C13B—H13F | 0.9800 |
| Cg1···Cg2$^{i}$ | 3.7853 (6) | Cg1···Cg2$^{ii}$ | 3.8625 (6) |
| C7A—O1A—C8A | 115.30 (9) | C7B—O1B—C8B | 116.24 (9) |
| O3A—N1A—O4A | 121.71 (9) | O3B—N1B—O4B | 121.87 (9) |
| O3A—N1A—C2A | 118.70 (9) | O3B—N1B—C2B | 118.70 (9) |
| O4A—N1A—C2A | 119.59 (9) | O4B—N1B—C2B | 119.43 (9) |
| C3A—N2A—C10A | 129.22 (9) | C3B—N2B—C10B | 129.04 (9) |
| C3A—N2A—H2NA | 113.4 | C3B—N2B—H2NB | 115.9 |
| C10A—N2A—H2NA | 117.3 | C10B—N2B—H2NB | 115.0 |
| C6A—C1A—C2A | 120.74 (9) | C6B—C1B—C2B | 120.85 (9) |
| C6A—C1A—H1A | 119.6 | C6B—C1B—H1B | 119.6 |
| C2A—C1A—H1A | 119.6 | C2B—C1B—H1B | 119.6 |
| C1A—C2A—C3A | 122.10 (9) | C1B—C2B—C3B | 121.91 (9) |
| C1A—C2A—N1A | 115.83 (9) | C1B—C2B—N1B | 115.79 (9) |
| C3A—C2A—N1A | 122.07 (9) | C3B—C2B—N1B | 122.30 (9) |

| | | | |
|---|---|---|---|
| N2A—C3A—C4A | 121.97 (9) | N2B—C3B—C4B | 121.65 (9) |
| N2A—C3A—C2A | 122.98 (9) | N2B—C3B—C2B | 123.11 (9) |
| C4A—C3A—C2A | 115.05 (9) | C4B—C3B—C2B | 115.23 (9) |
| C5A—C4A—C3A | 122.23 (10) | C5B—C4B—C3B | 122.25 (9) |
| C5A—C4A—H4A | 118.9 | C5B—C4B—H4B | 118.9 |
| C3A—C4A—H4A | 118.9 | C3B—C4B—H4B | 118.9 |
| C4A—C5A—C6A | 121.42 (9) | C4B—C5B—C6B | 121.22 (9) |
| C4A—C5A—H5A | 119.3 | C4B—C5B—H5B | 119.4 |
| C6A—C5A—H5A | 119.3 | C6B—C5B—H5B | 119.4 |
| C1A—C6A—C5A | 118.45 (9) | C1B—C6B—C5B | 118.54 (9) |
| C1A—C6A—C7A | 119.36 (9) | C1B—C6B—C7B | 119.12 (9) |
| C5A—C6A—C7A | 122.19 (9) | C5B—C6B—C7B | 122.34 (9) |
| O2A—C7A—O1A | 122.82 (10) | O2B—C7B—O1B | 123.56 (10) |
| O2A—C7A—C6A | 125.16 (10) | O2B—C7B—C6B | 124.77 (10) |
| O1A—C7A—C6A | 112.03 (9) | O1B—C7B—C6B | 111.67 (9) |
| O1A—C8A—C9A | 107.65 (9) | O1B—C8B—C9B | 111.18 (11) |
| O1A—C8A—H8A | 110.2 | O1B—C8B—H8C | 109.4 |
| C9A—C8A—H8A | 110.2 | C9B—C8B—H8C | 109.4 |
| O1A—C8A—H8B | 110.2 | O1B—C8B—H8D | 109.4 |
| C9A—C8A—H8B | 110.2 | C9B—C8B—H8D | 109.4 |
| H8A—C8A—H8B | 108.5 | H8C—C8B—H8D | 108.0 |
| C8A—C9A—H9A | 109.5 | C8B—C9B—H9D | 109.5 |
| C8A—C9A—H9B | 109.5 | C8B—C9B—H9E | 109.5 |
| H9A—C9A—H9B | 109.5 | H9D—C9B—H9E | 109.5 |
| C8A—C9A—H9C | 109.5 | C8B—C9B—H9F | 109.5 |
| H9A—C9A—H9C | 109.5 | H9D—C9B—H9F | 109.5 |
| H9B—C9A—H9C | 109.5 | H9E—C9B—H9F | 109.5 |
| N2A—C10A—C13A | 111.31 (9) | N2B—C10B—C13B | 111.59 (9) |
| N2A—C10A—C11A | 111.21 (9) | N2B—C10B—C11B | 111.24 (9) |
| C13A—C10A—C11A | 112.61 (9) | C13B—C10B—C11B | 111.97 (9) |
| N2A—C10A—C12A | 104.30 (8) | N2B—C10B—C12B | 104.10 (9) |
| C13A—C10A—C12A | 108.24 (9) | C13B—C10B—C12B | 108.70 (9) |
| C11A—C10A—C12A | 108.76 (9) | C11B—C10B—C12B | 108.88 (9) |
| C10A—C11A—H11A | 109.5 | C10B—C11B—H11D | 109.5 |
| C10A—C11A—H11B | 109.5 | C10B—C11B—H11E | 109.5 |
| H11A—C11A—H11B | 109.5 | H11D—C11B—H11E | 109.5 |
| C10A—C11A—H11C | 109.5 | C10B—C11B—H11F | 109.5 |
| H11A—C11A—H11C | 109.5 | H11D—C11B—H11F | 109.5 |
| H11B—C11A—H11C | 109.5 | H11E—C11B—H11F | 109.5 |
| C10A—C12A—H12A | 109.5 | C10B—C12B—H12D | 109.5 |
| C10A—C12A—H12B | 109.5 | C10B—C12B—H12E | 109.5 |
| H12A—C12A—H12B | 109.5 | H12D—C12B—H12E | 109.5 |
| C10A—C12A—H12C | 109.5 | C10B—C12B—H12F | 109.5 |
| H12A—C12A—H12C | 109.5 | H12D—C12B—H12F | 109.5 |
| H12B—C12A—H12C | 109.5 | H12E—C12B—H12F | 109.5 |
| C10A—C13A—H13A | 109.5 | C10B—C13B—H13D | 109.5 |
| C10A—C13A—H13B | 109.5 | C10B—C13B—H13E | 109.5 |
| H13A—C13A—H13B | 109.5 | H13D—C13B—H13E | 109.5 |
| C10A—C13A—H13C | 109.5 | C10B—C13B—H13F | 109.5 |

| | | | |
|---|---|---|---|
| H13A—C13A—H13C | 109.5 | H13D—C13B—H13F | 109.5 |
| H13B—C13A—H13C | 109.5 | H13E—C13B—H13F | 109.5 |

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

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2A—H2NA···O4A | 0.86 | 1.93 | 2.6299 (15) | 138 |
| N2A—H2NA···N1A | 0.86 | 2.54 | 2.9361 (15) | 109 |
| N2B—H2NB···O4B | 0.87 | 1.95 | 2.6355 (15) | 134 |
| N2B—H2NB···N1B | 0.87 | 2.58 | 2.9419 (15) | 106 |
| C1A—H1A···O3A | 0.95 | 2.31 | 2.6522 (16) | 100 |
| C1B—H1B···O3B | 0.95 | 2.31 | 2.6498 (16) | 100 |
| C4A—H4A···O3B$^{iii}$ | 0.95 | 2.50 | 3.4124 (17) | 160 |
| C4B—H4B···O3A$^{iv}$ | 0.95 | 2.42 | 3.2566 (18) | 147 |
| C5A—H5A···O1A | 0.95 | 2.41 | 2.7326 (13) | 100 |
| C11A—H11A···O2B$^{v}$ | 0.98 | 2.55 | 3.4714 (17) | 157 |
| C11B—H11F···O2A$^{ii}$ | 0.98 | 2.52 | 3.4446 (17) | 158 |
| C13B—H13D···O2A$^{vi}$ | 0.98 | 2.60 | 3.5071 (17) | 154 |

Symmetry codes: (iii) −*x*+2, −*y*+1, −*z*+1; (iv) −*x*+1, −*y*+1, −*z*+1; (v) *x*, −*y*+1/2, *z*+1/2; (ii) *x*, −*y*+1/2, *z*−1/2; (vi) *x*, −*y*+3/2, *z*−1/2.

**Fig. 1**

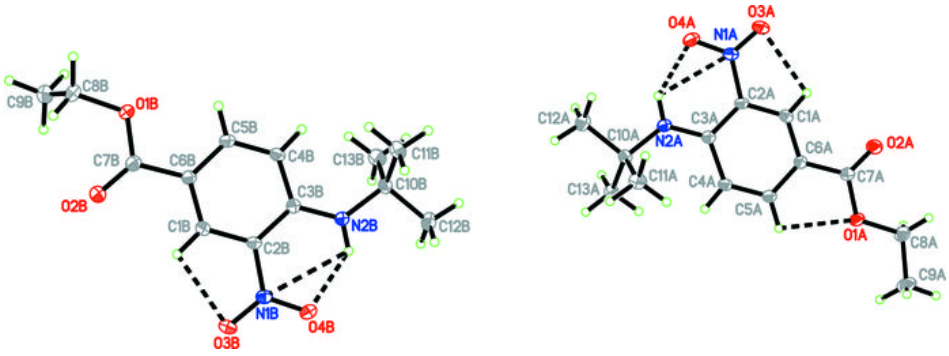
C8B
C9B
O1B
C5B
C7B
C6B
C4B
C13B
C11B
C10B
O2B
C3B
N2B
C12B
C1B
C2B
N1B
O4B
O3B
O4A
N1A
O3A
C2A
C12A
C1A
C10A
C3A
N2A
O2A
C6A
C13A
C11A
C4A
C7A
C5A
O1A
C8A
C9A

**Fig. 2**