2,4,6-Trinitrophenyl 4-methylbenzoate

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In the title compound, $C_{14}H_9N_3O_8$, the benzene rings form a dihedral angle of 69.02 (5)°. The central ester group is rotated by 25.86 (9)° relative to the *p*-tolyl group. In the crystal, the molecules are linked by C—H···O interactions into helical chains along [010].

Related literature

For optical, pharmacological and crystalline properties of picric acid, see: Khan *et al.* (2010); Zaderenko *et al.* (1997). For picric acid derivatives, see: Bertolasi *et al.* (2011). For bond-length data, see: Allen *et al.* (1987). For similar structures, see: Moreno-Fuquen *et al.* (2012); Bibi *et al.* (2009); Shibakami *et al.* (1994); Shibakami & Sekiya (1995); For hydrogen bonding, see: Nardelli (1995) and for supramolecular aggregation behaviour of isomers, see: Glidewell *et al.* (2005).
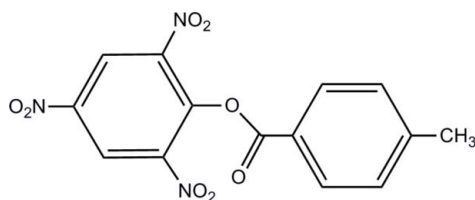
Experimental

Crystal data

C_{14}H_9N_3O_8
M_r = 347.24
Monoclinic, $P2_1/c$
a = 7.6126 (2) Å
b = 8.2124 (2) Å
c = 23.9893 (7) Å
β = 94.448 (1)°

V = 1495.24 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 295 K
0.27 × 0.22 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
5494 measured reflections
3044 independent reflections

2296 reflections with $I > 2\sigma(I)$
R_{int} = 0.017

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.123
S = 1.02
3044 reflections
227 parameters

1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.18 e Å^{-3}
$\Delta\rho_{min}$ = −0.24 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O8^i	0.93	2.50	3.4286 (19)	175

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

Data collection: *COLLECT* (Nonius, 2000); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* (Otwinowski & Minor, 1997) and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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.

Bertolasi, V., Gilli, P. & Gilli, G. (2011). *Cryst. Growth Des.* **11**, 2724–2735.

Bibi, U., Siddiqi, H. M., Bolte, M. & Akhter, Z. (2009). *Acta Cryst.* E**65**, o3038.

Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.

Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.

Glidewell, C., Low, J. N., Skakle, J. M. S., Wardell, S. M. S. V. & Wardell, J. L. (2005). *Acta Cryst.* B**61**, 227–237.

Khan, I. M., Ahmad, A. & Oves, M. (2010). *Spectrochim. Acta Part A*, **77**, 1059–1064.

Macrae, C. F., Edgington, P. R., McCabe, P., Pidcock, E., Shields, G. P., Taylor, R., Towler, M. & van de Streek, J. (2006). *J. Appl. Cryst.* **39**, 453–457.

Moreno-Fuquen, R., Mosquera, F., Ellena, J. & Tenorio, J. C. (2012). *Acta Cryst.* E**68**, o2187.

Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.

Nonius (2000). *COLLECT*. Nonius BV, Delft, The Netherlands.

Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.

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

Shibakami, M. & Sekiya, A. (1995). *Acta Cryst.* C**51**, 326–330.

Shibakami, M., Tamura, M., Arimura, T., Kurosawa, S. & Sekiya, A. (1994). *Acta Cryst.* C**50**, 592–594.

Zaderenko, P., Gil, M. S., López, P., Ballesteros, P., Fonseca, I. & Albert, A. (1997). *Acta Cryst.* B**53**, 961–967.

supporting information

2,4,6-Trinitrophenyl 4-methylbenzoate

Rodolfo Moreno-Fuquen, Fabricio Mosquera, Javier Ellena, Juan C. Tenorio and Rodrigo S. Corrêa

S1. Comment

The title compound, $C_{14}H_9O_8N_3$ [2,4,6-trinitrophenyl 4-methylbenzoate] (I), belongs to a group of molecules known as nitro aryl benzoates. Recently, we have been investigating the synthesis, properties and main features of these nitro-aromatic compounds, in particular - the derivatives of picric acid (trinitrophenol - TNP) (Bertolasi *et al.*, 2011). These compounds have never been synthesized before and there is no any prior entries in CCSD related to such esters, except 2,4,6-trinitrophenyl 3-methylbenzoate (TNP3MeBA), an isomer of the title compound, that we recently published (Moreno-Fuquen *et al.*, 2012). Our special interest in TNP is attributable to the optical, pharmacological and solid-state properties of its molecular adducts (Khan *et al.*, 2010); Zaderenko *et al.*, 1997) and the absence of any information of the picric acid as a part of aryl esters that probably can show similar or more interesting properties. In order to find out the molecular conformation of (I) and its supramolecular behavior, the title compound was synthesized. The molecular structure of (I) is presented on Fig. 1 with the numbering scheme similar to that for TNP-3MeBA in order to simplify structural comparisons. In the title structure, the phenolic C1—O7 bond length is significantly shortened [1.3715 (17) Å] and the benzoic C7—O7 bond length is significantly elongated [1.3898 (17) Å] as compared to other phenylbenzoates systems [1.422 (5) Å and 1.354 (5) Å respectively (Shibakami *et al.*, 1994) and 1.415 (2) Å and 1.351 (2) Å respectively (Shibakami & Sekiya, 1995)]. However, these parameters are pretty close to the values presented in other crystal systems which contain similar substituents with respect to this structure, more specifically when there is nitro substituent in any of the benzene rings at the benzoate [for C1—O7= 1.395 (2) Å and for C7—O7= 1.376 (3) Å (Bibi *et al.*, 2009) and 1.3676 (17) Å and 1.3820 (18) Å respectively (Moreno-Fuquen *et al.*, 2012)]. Other bond lengths and bond angles of (I) agree with the literature values (Allen *et al.*, 1987). The benzene rings of (I) form a dihedral angle of 69.02 (5)°, compared with values of 87.48 (5)° in TNP3MeBA. The central ester moiety forms an angle of 25.86 (9)° with the methylbenzene ring to which it is attached. This value is similar to the corresponding angle in its structural isomer TNP3MeBA - 19.42 (7)°. The nitro groups form dihedral angles with the adjacent benzene ring of 30.57 (11)°, 14.75 (16)° and 7.37 (17)° for O1—N1—O2, O3—N2—O4 and O5—N3—O6, respectively. The molecules are packed forming weak interactions C—H···O in one-dimensional helical chains which grow along [010] (see Fig. 2). The C3 atom of the phenyl ring at (*x*,*y*,*z*) acts as a hydrogen-bond donor to carbonyl atom O8 at (-*x* + 1,+*y* + 1/2,-*z* + 1/2) (see Table 1; Nardelli, 1995). We have found that (I) and its isomer TNP3MeBA show a marked similarity in terms of spatial group, unit-cell parameters and finally the intermolecular interactions given the supramolecular aggregation of these isomers. This behavior is very uncommon in compounds showing constitutional isomerism, since small changes in the molecular structures of the isomers usually lead to large changes in molecular aggregation of the structures (Glidewell *et al.*, 2005).

S2. Experimental

The reagents and solvents for the synthesis were obtained from the Aldrich Chemical Co., and were used without additional purification. The title molecule was obtained through a two-step reaction. First the 4-methylbenzoic acid (0.25 g, 1.836 mmol) was refluxed in an excess of thionyl chloride (10 ml) during an hour. Then thionyl chloride was distilled off under reduced pressure to purify the 4-methylbenzoyl chloride obtained as a pale yellow traslucent liquid. The same reaction flask was rearranged and a solution of picric acid (0.42 g, 1.835 mmol) in acetonitrile was added dropwise with constant stirring. The reaction mixture was let to reflux for about an hour. A pale yellow solid was obtained after leaving the solvent to evaporate. The solid was washed with distilled water and cold methanol to eliminate impurities. Crystals of good quality and suitable for single-crystal X-ray diffraction were grown from acetonitrile. IR spectra were recorded on a FT—IR SHIMADZU IR-Affinity-1 spectrophotometer. Pale Yellow crystals; yield 38%; m.p 417 (1) K. IR (KBr) 3112.86 cm^{-1}, 3086.07 cm^{-1} (aromatic C—H); 2962.07 cm^{-1}, 2915.70 cm^{-1} (methyl C—H); 1754.68 cm^{-1} (ester C=O); 1609.43 cm^{-1} (C=C); 1549.55 cm^{-1}, 1341.69 cm^{-1} (–NO2); 1224.07 cm^{-1} (C(=O)—O).

S3. Refinement

All the H-atoms attached to C atoms were positioned at geometrically idealized positions and treated as riding with C—H= 0.93 Å (aromatic) and 0.96 Å (methyl) with U_{iso}(H) = 1.2 U_{eq} (aromatic) and U_{iso}(H) = 1.5 U_{eq} (methyl). The positions of H atoms of the methyl group were rotationally optimized
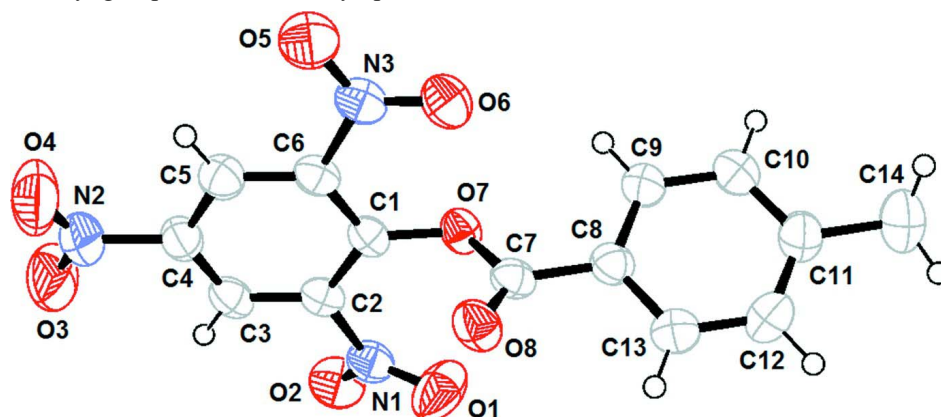
Figure 1

Molecular conformation and atom numbering scheme of the title compound with displacement ellipsoids drawn at 50% probability level. H atoms are shown as spheres of arbitrary radius.
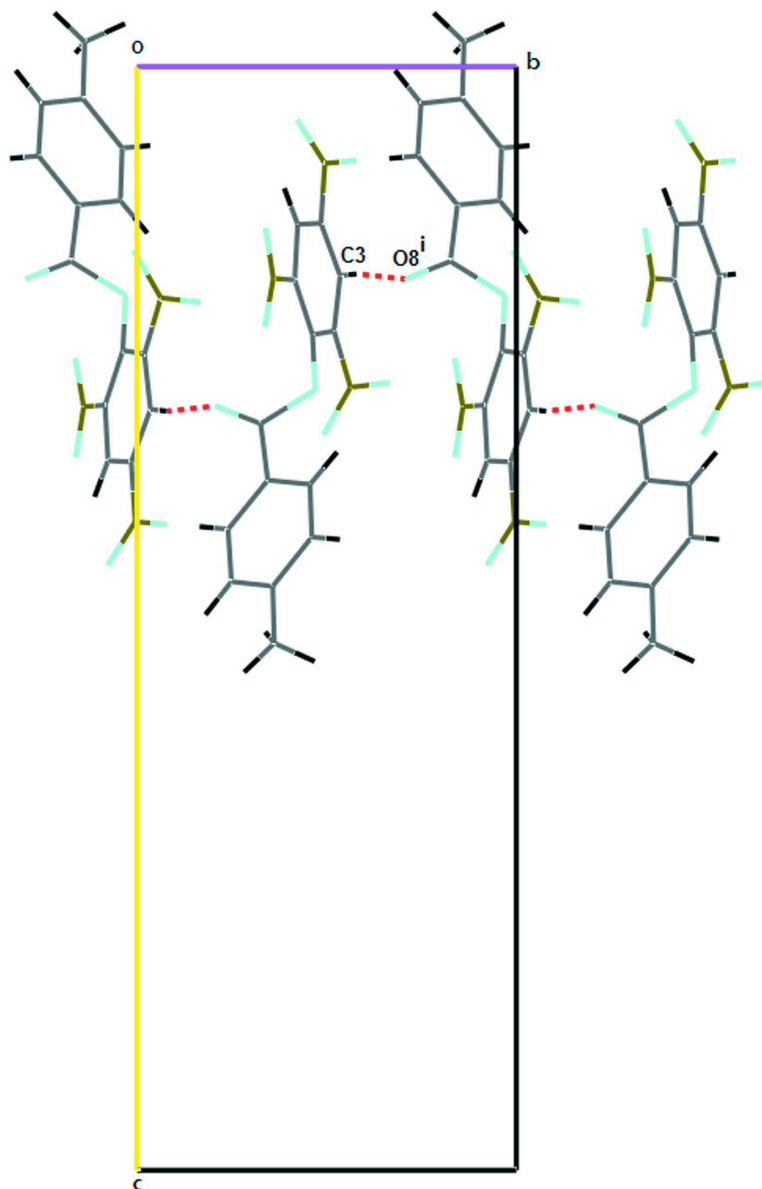
Figure 2

Part of the crystal structure of (I) showing the formation of helical chains along [010]. Symmetry code: (i) -x+1,+y+1/2,-z+1/2.

2,4,6-Trinitrophenyl 4-methylbenzoate

Crystal data

$C_{14}H_9N_3O_8$	$V = 1495.24 (7)$ Å^3
$M_r = 347.24$	$Z = 4$
Monoclinic, $P2_1/c$	$F(000) = 712$
Hall symbol: -P 2ybc	$D_x = 1.543$ Mg m^{-3}
$a = 7.6126 (2)$ Å	Melting point: 417(1) K
$b = 8.2124 (2)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$c = 23.9893 (7)$ Å	Cell parameters from 3076 reflections
$\beta = 94.448 (1)°$	$\theta = 3.0–26.4°$

$\mu = 0.13$ mm^{-1}

$T = 295$ K

Block, pale-yellow

$0.27 \times 0.22 \times 0.18$ mm

Data collection

Nonius KappaCCD diffractometer	2296 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	$R_{int} = 0.017$
Graphite monochromator	$\theta_{max} = 26.4°$, $\theta_{min} = 3.0°$
CCD rotation images, thick slices scans	$h = -9 \rightarrow 9$
5494 measured reflections	$k = -9 \rightarrow 10$
3044 independent reflections	$l = -29 \rightarrow 29$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.043$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.123$	
$S = 1.02$	H-atom parameters constrained
3044 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0698P)^2 + 0.224P]$
227 parameters	where $P = (F_o^2 + 2F_c^2)/3$
1 restraint	$(\Delta/\sigma)_{max} < 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.18$ e Å^{-3}
	$\Delta\rho_{min} = -0.24$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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}
O1	0.4307 (2)	0.4914 (2)	0.33344 (6)	0.0840 (5)
O2	0.56533 (16)	0.66653 (15)	0.28637 (6)	0.0633 (4)
O3	0.5292 (2)	0.5772 (3)	0.08518 (7)	0.1011 (6)
O4	0.3254 (2)	0.4222 (3)	0.04835 (7)	0.1007 (6)
O5	−0.18224 (17)	0.33867 (18)	0.14637 (6)	0.0732 (4)
O6	−0.17197 (15)	0.33696 (15)	0.23563 (5)	0.0595 (3)
O7	0.09683 (13)	0.46979 (12)	0.29293 (4)	0.0407 (3)
O8	0.16920 (15)	0.20662 (13)	0.30759 (5)	0.0525 (3)
N1	0.45379 (16)	0.56271 (17)	0.29052 (6)	0.0469 (3)
N2	0.3987 (2)	0.4953 (2)	0.08753 (7)	0.0664 (4)
N3	−0.10536 (17)	0.35918 (15)	0.19191 (6)	0.0460 (3)
C1	0.17239 (18)	0.46265 (16)	0.24291 (6)	0.0362 (3)
C2	0.34413 (18)	0.51938 (17)	0.23953 (6)	0.0386 (3)
C3	0.41940 (19)	0.53543 (18)	0.18945 (7)	0.0433 (4)
H3	0.5318	0.5787	0.1878	0.052*

C4	0.3213 (2)	0.48477 (19)	0.14189 (7)	0.0462 (4)
C5	0.1537 (2)	0.42336 (19)	0.14288 (7)	0.0457 (4)
H5	0.0918	0.3878	0.1102	0.055*
C6	0.07943 (18)	0.41560 (17)	0.19332 (6)	0.0393 (3)
C7	0.09911 (18)	0.32603 (18)	0.32354 (7)	0.0396 (4)
C8	0.00660 (19)	0.34449 (18)	0.37471 (7)	0.0420 (4)
C9	−0.1286 (2)	0.4560 (2)	0.37848 (7)	0.0516 (4)
H9	−0.1580	0.5273	0.3491	0.062*
C10	−0.2198 (2)	0.4607 (2)	0.42623 (8)	0.0618 (5)
H10	−0.3118	0.5344	0.4283	0.074*
C11	−0.1768 (2)	0.3578 (3)	0.47078 (7)	0.0595 (5)
C12	−0.0394 (2)	0.2490 (2)	0.46659 (7)	0.0617 (5)
H12	−0.0074	0.1803	0.4965	0.074*
C13	0.0511 (2)	0.2402 (2)	0.41905 (7)	0.0537 (4)
H13	0.1414	0.1648	0.4167	0.064*
C14	−0.2789 (3)	0.3625 (4)	0.52240 (9)	0.0880 (7)
H14A	−0.2015	0.3370	0.5547	0.132*
H14B	−0.3727	0.2841	0.5187	0.132*
H14C	−0.3274	0.4693	0.5265	0.132*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0738 (9)	0.1154 (12)	0.0599 (8)	−0.0385 (9)	−0.0135 (7)	0.0239 (8)
O2	0.0497 (7)	0.0579 (8)	0.0813 (9)	−0.0212 (6)	−0.0008 (6)	−0.0053 (6)
O3	0.0687 (10)	0.1650 (17)	0.0728 (10)	−0.0334 (11)	0.0253 (8)	0.0139 (11)
O4	0.0954 (12)	0.1528 (17)	0.0567 (9)	−0.0247 (12)	0.0243 (8)	−0.0252 (10)
O5	0.0516 (7)	0.0986 (11)	0.0677 (9)	−0.0220 (7)	−0.0064 (6)	−0.0147 (7)
O6	0.0435 (6)	0.0694 (8)	0.0667 (8)	−0.0163 (6)	0.0113 (6)	0.0084 (6)
O7	0.0426 (6)	0.0342 (5)	0.0468 (6)	−0.0005 (4)	0.0123 (5)	0.0012 (4)
O8	0.0546 (7)	0.0402 (6)	0.0648 (8)	0.0080 (5)	0.0179 (6)	0.0051 (5)
N1	0.0364 (7)	0.0469 (8)	0.0571 (8)	−0.0044 (6)	0.0021 (5)	−0.0011 (6)
N2	0.0529 (9)	0.0924 (12)	0.0560 (10)	−0.0001 (9)	0.0169 (8)	0.0064 (9)
N3	0.0384 (7)	0.0389 (7)	0.0608 (9)	−0.0058 (5)	0.0028 (7)	−0.0017 (6)
C1	0.0359 (7)	0.0275 (7)	0.0461 (8)	0.0019 (5)	0.0083 (6)	0.0008 (6)
C2	0.0350 (7)	0.0306 (7)	0.0504 (8)	0.0001 (6)	0.0036 (6)	0.0003 (6)
C3	0.0338 (7)	0.0396 (8)	0.0576 (10)	−0.0003 (6)	0.0103 (7)	0.0051 (7)
C4	0.0440 (8)	0.0482 (9)	0.0478 (9)	0.0046 (7)	0.0117 (7)	0.0044 (7)
C5	0.0443 (8)	0.0447 (8)	0.0481 (9)	0.0007 (7)	0.0036 (7)	−0.0014 (7)
C6	0.0334 (7)	0.0331 (7)	0.0515 (9)	−0.0006 (6)	0.0038 (6)	0.0007 (6)
C7	0.0318 (7)	0.0368 (8)	0.0503 (9)	−0.0017 (6)	0.0035 (6)	0.0033 (7)
C8	0.0362 (7)	0.0431 (8)	0.0469 (9)	−0.0026 (6)	0.0037 (6)	0.0027 (7)
C9	0.0464 (9)	0.0600 (10)	0.0492 (10)	0.0113 (8)	0.0083 (7)	0.0104 (8)
C10	0.0504 (10)	0.0798 (13)	0.0566 (11)	0.0186 (9)	0.0125 (8)	0.0066 (9)
C11	0.0529 (10)	0.0821 (13)	0.0440 (10)	−0.0031 (9)	0.0074 (8)	0.0034 (9)
C12	0.0652 (11)	0.0722 (12)	0.0472 (10)	0.0042 (10)	0.0009 (8)	0.0161 (9)
C13	0.0493 (9)	0.0575 (10)	0.0542 (10)	0.0091 (8)	0.0031 (8)	0.0082 (8)
C14	0.0818 (15)	0.130 (2)	0.0552 (13)	0.0046 (14)	0.0247 (11)	0.0093 (13)

Geometric parameters (Å, º)

O1—N1	1.2089 (18)	C5—C6	1.376 (2)
O2—N1	1.2128 (17)	C5—H5	0.9300
O3—N2	1.204 (2)	C7—C8	1.470 (2)
O4—N2	1.214 (2)	C8—C9	1.386 (2)
O5—N3	1.2106 (18)	C8—C13	1.387 (2)
O6—N3	1.2135 (17)	C9—C10	1.385 (2)
O7—C1	1.3715 (17)	C9—H9	0.9300
O7—C7	1.3898 (17)	C10—C11	1.382 (3)
O8—C7	1.1936 (18)	C10—H10	0.9300
N1—C2	1.470 (2)	C11—C12	1.385 (3)
N2—C4	1.474 (2)	C11—C14	1.513 (2)
N3—C6	1.4790 (19)	C12—C13	1.379 (2)
C1—C6	1.391 (2)	C12—H12	0.9300
C1—C2	1.396 (2)	C13—H13	0.9300
C2—C3	1.377 (2)	C14—H14A	0.9600
C3—C4	1.378 (2)	C14—H14B	0.9600
C3—H3	0.9300	C14—H14C	0.9600
C4—C5	1.374 (2)		
C1—O7—C7	115.91 (11)	O8—C7—O7	121.07 (13)
O1—N1—O2	124.00 (14)	O8—C7—C8	127.42 (14)
O1—N1—C2	118.73 (13)	O7—C7—C8	111.50 (12)
O2—N1—C2	117.24 (13)	C9—C8—C13	119.77 (15)
O3—N2—O4	124.83 (17)	C9—C8—C7	121.94 (14)
O3—N2—C4	117.50 (17)	C13—C8—C7	118.19 (14)
O4—N2—C4	117.66 (16)	C10—C9—C8	119.65 (15)
O5—N3—O6	123.63 (13)	C10—C9—H9	120.2
O5—N3—C6	117.16 (13)	C8—C9—H9	120.2
O6—N3—C6	119.20 (13)	C11—C10—C9	121.23 (16)
O7—C1—C6	122.69 (12)	C11—C10—H10	119.4
O7—C1—C2	119.66 (13)	C9—C10—H10	119.4
C6—C1—C2	117.35 (13)	C10—C11—C12	118.26 (16)
C3—C2—C1	122.52 (14)	C10—C11—C14	120.74 (18)
C3—C2—N1	117.06 (13)	C12—C11—C14	120.99 (18)
C1—C2—N1	120.41 (13)	C13—C12—C11	121.48 (16)
C2—C3—C4	117.26 (14)	C13—C12—H12	119.3
C2—C3—H3	121.4	C11—C12—H12	119.3
C4—C3—H3	121.4	C12—C13—C8	119.59 (16)
C5—C4—C3	122.72 (15)	C12—C13—H13	120.2
C5—C4—N2	118.18 (15)	C8—C13—H13	120.2
C3—C4—N2	119.10 (15)	C11—C14—H14A	109.5
C4—C5—C6	118.54 (15)	C11—C14—H14B	109.5
C4—C5—H5	120.7	H14A—C14—H14B	109.5
C6—C5—H5	120.7	C11—C14—H14C	109.5
C5—C6—C1	121.51 (13)	H14A—C14—H14C	109.5
C5—C6—N3	116.71 (13)	H14B—C14—H14C	109.5

C1—C6—N3	121.76 (13)		
C7—O7—C1—C6	87.32 (16)	C2—C1—C6—C5	0.5 (2)
C7—O7—C1—C2	−99.17 (15)	O7—C1—C6—N3	−4.2 (2)
O7—C1—C2—C3	−171.34 (12)	C2—C1—C6—N3	−177.83 (12)
C6—C1—C2—C3	2.5 (2)	O5—N3—C6—C5	−6.7 (2)
O7—C1—C2—N1	9.53 (19)	O6—N3—C6—C5	173.77 (13)
C6—C1—C2—N1	−176.61 (12)	O5—N3—C6—C1	171.70 (14)
O1—N1—C2—C3	−148.89 (16)	O6—N3—C6—C1	−7.9 (2)
O2—N1—C2—C3	29.57 (19)	C1—O7—C7—O8	2.8 (2)
O1—N1—C2—C1	30.3 (2)	C1—O7—C7—C8	−176.87 (12)
O2—N1—C2—C1	−151.26 (14)	O8—C7—C8—C9	−151.99 (17)
C1—C2—C3—C4	−3.3 (2)	O7—C7—C8—C9	27.6 (2)
N1—C2—C3—C4	175.80 (13)	O8—C7—C8—C13	24.5 (2)
C2—C3—C4—C5	1.3 (2)	O7—C7—C8—C13	−155.96 (14)
C2—C3—C4—N2	−178.25 (13)	C13—C8—C9—C10	−0.9 (3)
O3—N2—C4—C5	165.82 (18)	C7—C8—C9—C10	175.47 (16)
O4—N2—C4—C5	−13.5 (3)	C8—C9—C10—C11	1.1 (3)
O3—N2—C4—C3	−14.6 (3)	C9—C10—C11—C12	0.0 (3)
O4—N2—C4—C3	166.10 (18)	C9—C10—C11—C14	−179.2 (2)
C3—C4—C5—C6	1.5 (2)	C10—C11—C12—C13	−1.2 (3)
N2—C4—C5—C6	−178.95 (14)	C14—C11—C12—C13	178.0 (2)
C4—C5—C6—C1	−2.4 (2)	C11—C12—C13—C8	1.3 (3)
C4—C5—C6—N3	175.99 (13)	C9—C8—C13—C12	−0.2 (3)
O7—C1—C6—C5	174.10 (13)	C7—C8—C13—C12	−176.76 (16)

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C3—H3···O8[i]	0.93	2.50	3.4286 (19)	175

Symmetry code: (i) −x+1, y+1/2, −z+1/2.