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3,4-Dimethylphenyl benzoate

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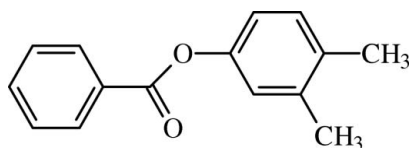
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Key indicators: single-crystal X-ray study; $T = 123$ K; mean $\sigma(C-C) = 0.002$ Å; R factor = 0.048; wR factor = 0.112; data-to-parameter ratio = 19.0.

In the title compound, $C_{15}H_{14}O_2$, the terminal rings form a dihedral angle of $52.39(4)^\circ$. The mean plane of the central ester group [r.m.s. deviation = 0.0488 Å] is twisted away from the benzene and phenyl rings by $60.10(4)$ and $8.67(9)^\circ$, respectively. In the crystal, molecules are linked by weak $C-H \cdots O$ hydrogen bonds, forming $C(6)$ chains which run along $[100]$.

Related literature

For similar structures, see: Gowda *et al.* (2008a,b). For hydrogen-bonding information, see: Nardelli (1995) and for hydrogen-bond motifs, see: Etter *et al.* (1990).



Experimental

Crystal data

$C_{15}H_{14}O_2$
 $M_r = 226.26$
 Triclinic, $P\bar{1}$
 $a = 6.0293(4)$ Å
 $b = 7.8506(3)$ Å

$c = 13.1163(9)$ Å
 $\alpha = 88.592(4)^\circ$
 $\beta = 77.020(5)^\circ$
 $\gamma = 77.680(4)^\circ$
 $V = 590.87(6)$ Å³

$Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹

$T = 123$ K
 $0.33 \times 0.25 \times 0.06$ mm

Data collection

Oxford Diffraction Xcalibur E diffractometer
 Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)
 $T_{\min} = 0.977$, $T_{\max} = 1.000$

5782 measured reflections
 2965 independent reflections
 2196 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.112$
 $S = 1.04$
 2965 reflections

156 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.28$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
$C2-H2 \cdots O8^i$	0.95	2.47	3.3710 (18)	157

Symmetry code: (i) $x - 1, y, z$.

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO*; data reduction: *CrysAlis PRO*; 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, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: TK5288).

References

- Etter, M. (1990). *Acc. Chem. Res.* **23**, 120–126.
 Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
 Gowda, B. T., Foro, S., Babitha, K. S. & Fuess, H. (2008a). *Acta Cryst.* **E64**, o844.
 Gowda, B. T., Tokarčík, M., Kožíšek, J., Babitha, K. S. & Fuess, H. (2008b). *Acta Cryst.* **E64**, o1280.
 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.
 Nardelli, M. (1995). *J. Appl. Cryst.* **28**, 659.
 Oxford Diffraction (2010). *CrysAlis PRO*. Oxford Diffraction Ltd, Yarnton, England.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2014). E70, o194 [doi:10.1107/S1600536814001299]

3,4-Dimethylphenyl benzoate

Rodolfo Moreno-Fuquen, Mauricio Rendón and Alan R. Kennedy

1. Introduction

2. Experimental

2.1. Synthesis and crystallization

The reagents and solvents for the synthesis were obtained from Aldrich Chemical Co., and were used without additional purification. The title molecule was synthesized using equimolar quantities of 3,4-dimethylphenol and benzoyl chloride. 3,4-Dimethylphenol (0.50 g, 4.10 mmol) was added to a solution of anhydrous aluminum chloride (0.40 g, 3.00 mmol) in anhydrous dichloromethane (25 mL). The resulting solution was cooled and benzoyl chloride (0.57 g) was added slowly at 0–5°. After complete addition, the mixture was left under stirring at room temperature for 0.5 h, and then it was heated (reflux) to 50° C for 1 h. The reaction mixture was poured onto ice (100 g). The crude product was isolated by extraction with dichloromethane, and it was separated. The solution was dried over Na₂SO₄ and it was evaporated at room temperature. The obtained amorphous product was dissolved in methanol and the solution was left to slow evaporation. Colourless crystals of good quality were obtained with M.pt = 322 (1) K.

2.2. Refinement

All H-atoms were positioned at geometrically idealized positions with C—H distance of 0.95–0.98 Å, and with $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}$ of the C-atoms to which they were bonded.

3. Results and discussion

In order to obtain more detailed information about the effect of substitution of the methyl groups on the structure of benzoate system, the structure determination of 3,4-dimethyl phenyl benzoate (I) has been carried out. Two very similar molecular structures, 2,3-dimethylphenyl benzoate, DMPB1 (Gowda *et al.*, 2008a), and 2,4-dimethylphenyl benzoate, DMPB2 (Gowda *et al.*, 2008b), were taken for comparison with the structure (I). The rings of (I), Fig. 1, form a dihedral angle of 52.39 (4)° while in DMPB1 and DMPB2 they form dihedral angles of 87.36 (6)° and 80.25 (5)°, respectively. The other bond lengths and bond angles of DMPB1 and DMPB2 are close to the title system. The central ester moiety (C1—O7—C7(O8)—C8) is twisted away from the dimethyl-substituted benzene and phenyl rings by 60.10 (4) and 8.67 (9)°, respectively. The crystal packing shows no classical hydrogen bonds and it is stabilized by weak C—H⋯O intermolecular hydrogen bonds, forming C(6) chains (Etter, 1990) along [100] (see Fig. 2). The C2 atom acts as hydrogen-bond donor to O1 atom at ($x-1$, $+y$, $+z$) (Nardelli, 1995); see Table 1.

Computing details

Data collection: *CrysAlis PRO* (Oxford Diffraction, 2010); cell refinement: *CrysAlis PRO* (Oxford Diffraction, 2010); data reduction: *CrysAlis PRO* (Oxford Diffraction, 2010); 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, 2012) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

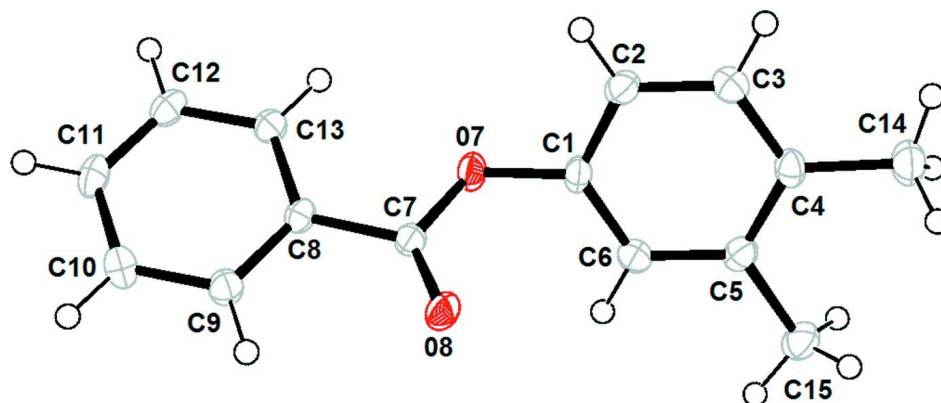


Figure 1

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

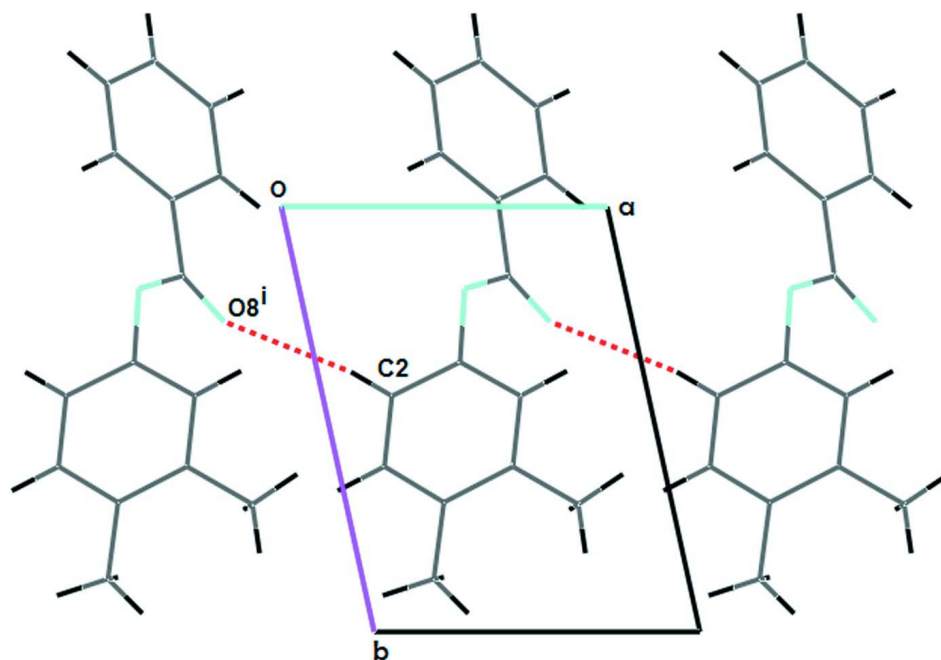


Figure 2

Part of the crystal structure of (I), showing the formation of chains which running along [100]. Symmetry code: (i) $x-1, +y, +z$.

3,4-Dimethylphenyl benzoate

Crystal data

$C_{15}H_{14}O_2$

$M_r = 226.26$

Triclinic, $P\bar{1}$

Hall symbol: $-P\ 1$

$a = 6.0293(4)$ Å

$b = 7.8506(3)$ Å

$c = 13.1163(9)$ Å

$\alpha = 88.592(4)^\circ$

$\beta = 77.020(5)^\circ$

$\gamma = 77.680(4)^\circ$

$V = 590.87 (6) \text{ \AA}^3$
 $Z = 2$
 $F(000) = 240$
 $D_x = 1.272 \text{ Mg m}^{-3}$
 Melting point: 322(1) K
 Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2013 reflections
 $\theta = 3.1\text{--}29.6^\circ$
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 123 \text{ K}$
 Plate, colourless
 $0.33 \times 0.25 \times 0.06 \text{ mm}$

Data collection

Oxford Diffraction Xcalibur E
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 ω scans
 Absorption correction: multi-scan
 (CrysAlis PRO; Oxford Diffraction, 2010)
 $T_{\min} = 0.977, T_{\max} = 1.000$

5782 measured reflections
 2965 independent reflections
 2196 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.021$
 $\theta_{\max} = 29.8^\circ, \theta_{\min} = 3.1^\circ$
 $h = -8 \rightarrow 8$
 $k = -10 \rightarrow 10$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.048$
 $wR(F^2) = 0.112$
 $S = 1.04$
 2965 reflections
 156 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0425P)^2 + 0.1253P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.28 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.24 \text{ e \AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
O7	0.50750 (18)	0.19314 (11)	0.74671 (8)	0.0239 (2)
O8	0.74671 (18)	0.27031 (12)	0.83880 (8)	0.0262 (3)
C1	0.4447 (2)	0.36566 (16)	0.71295 (11)	0.0202 (3)
C2	0.2126 (2)	0.44540 (17)	0.74124 (11)	0.0227 (3)
H2	0.1032	0.3888	0.7848	0.027*
C3	0.1425 (3)	0.61121 (18)	0.70426 (11)	0.0236 (3)
H3	-0.0171	0.6682	0.7231	0.028*
C4	0.3009 (3)	0.69544 (17)	0.64030 (11)	0.0218 (3)
C5	0.5366 (2)	0.61144 (17)	0.61281 (11)	0.0213 (3)
C6	0.6075 (2)	0.44503 (17)	0.64949 (11)	0.0211 (3)
H6	0.7665	0.3865	0.6310	0.025*

C7	0.6508 (2)	0.16320 (16)	0.81492 (10)	0.0184 (3)
C8	0.6695 (2)	-0.01620 (16)	0.85620 (10)	0.0182 (3)
C9	0.8327 (2)	-0.07177 (17)	0.91616 (11)	0.0226 (3)
H9	0.9322	0.0019	0.9267	0.027*
C10	0.8508 (3)	-0.23504 (18)	0.96080 (12)	0.0257 (3)
H10	0.9613	-0.2727	1.0025	0.031*
C11	0.7073 (3)	-0.34264 (17)	0.94426 (11)	0.0252 (3)
H11	0.7192	-0.4542	0.9748	0.030*
C12	0.5461 (3)	-0.28836 (17)	0.88330 (11)	0.0241 (3)
H12	0.4495	-0.3635	0.8716	0.029*
C13	0.5251 (2)	-0.12484 (16)	0.83937 (11)	0.0205 (3)
H13	0.4135	-0.0872	0.7982	0.025*
C14	0.2186 (3)	0.87556 (18)	0.60106 (13)	0.0314 (4)
H14A	0.0507	0.9147	0.6291	0.047*
H14B	0.2509	0.8716	0.5244	0.047*
H14C	0.3010	0.9570	0.6242	0.047*
C15	0.7122 (3)	0.6999 (2)	0.54453 (12)	0.0292 (3)
H15A	0.8692	0.6291	0.5400	0.044*
H15B	0.7010	0.8151	0.5748	0.044*
H15C	0.6807	0.7130	0.4743	0.044*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O7	0.0319 (6)	0.0159 (4)	0.0298 (6)	-0.0087 (4)	-0.0163 (5)	0.0063 (4)
O8	0.0332 (6)	0.0197 (5)	0.0327 (6)	-0.0133 (4)	-0.0150 (5)	0.0059 (4)
C1	0.0273 (8)	0.0157 (6)	0.0211 (7)	-0.0066 (5)	-0.0112 (6)	0.0045 (5)
C2	0.0236 (7)	0.0247 (7)	0.0220 (7)	-0.0100 (6)	-0.0057 (6)	0.0049 (6)
C3	0.0222 (7)	0.0248 (7)	0.0240 (7)	-0.0035 (6)	-0.0071 (6)	0.0014 (6)
C4	0.0283 (8)	0.0189 (6)	0.0206 (7)	-0.0050 (6)	-0.0111 (6)	0.0028 (5)
C5	0.0256 (8)	0.0232 (7)	0.0181 (7)	-0.0097 (6)	-0.0074 (6)	0.0033 (5)
C6	0.0204 (7)	0.0226 (7)	0.0207 (7)	-0.0041 (5)	-0.0058 (6)	0.0001 (5)
C7	0.0194 (7)	0.0174 (6)	0.0188 (7)	-0.0051 (5)	-0.0043 (5)	0.0016 (5)
C8	0.0216 (7)	0.0147 (6)	0.0175 (7)	-0.0042 (5)	-0.0025 (5)	-0.0001 (5)
C9	0.0261 (8)	0.0196 (6)	0.0249 (7)	-0.0067 (5)	-0.0097 (6)	0.0012 (6)
C10	0.0303 (8)	0.0225 (7)	0.0241 (7)	-0.0013 (6)	-0.0098 (6)	0.0030 (6)
C11	0.0322 (8)	0.0147 (6)	0.0251 (8)	-0.0034 (6)	-0.0012 (6)	0.0034 (5)
C12	0.0278 (8)	0.0183 (6)	0.0264 (8)	-0.0094 (6)	-0.0024 (6)	-0.0003 (6)
C13	0.0217 (7)	0.0182 (6)	0.0225 (7)	-0.0053 (5)	-0.0056 (6)	0.0002 (5)
C14	0.0376 (9)	0.0237 (7)	0.0361 (9)	-0.0062 (6)	-0.0161 (7)	0.0080 (6)
C15	0.0323 (9)	0.0324 (8)	0.0270 (8)	-0.0145 (7)	-0.0087 (7)	0.0101 (6)

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

O7—C7	1.3609 (16)	C8—C13	1.3950 (18)
O7—C1	1.4145 (15)	C9—C10	1.3892 (19)
O8—C7	1.2008 (16)	C9—H9	0.9500
C1—C2	1.3755 (19)	C10—C11	1.384 (2)
C1—C6	1.383 (2)	C10—H10	0.9500
C2—C3	1.3909 (19)	C11—C12	1.386 (2)

C2—H2	0.9500	C11—H11	0.9500
C3—C4	1.391 (2)	C12—C13	1.3867 (18)
C3—H3	0.9500	C12—H12	0.9500
C4—C5	1.402 (2)	C13—H13	0.9500
C4—C14	1.5133 (18)	C14—H14A	0.9800
C5—C6	1.3943 (19)	C14—H14B	0.9800
C5—C15	1.504 (2)	C14—H14C	0.9800
C6—H6	0.9500	C15—H15A	0.9800
C7—C8	1.4871 (17)	C15—H15B	0.9800
C8—C9	1.3872 (19)	C15—H15C	0.9800
C7—O7—C1	117.97 (10)	C8—C9—H9	120.0
C2—C1—C6	122.09 (13)	C10—C9—H9	120.0
C2—C1—O7	116.62 (12)	C11—C10—C9	119.75 (14)
C6—C1—O7	121.17 (12)	C11—C10—H10	120.1
C1—C2—C3	118.16 (13)	C9—C10—H10	120.1
C1—C2—H2	120.9	C10—C11—C12	120.31 (13)
C3—C2—H2	120.9	C10—C11—H11	119.8
C2—C3—C4	121.48 (14)	C12—C11—H11	119.8
C2—C3—H3	119.3	C11—C12—C13	120.32 (13)
C4—C3—H3	119.3	C11—C12—H12	119.8
C3—C4—C5	119.23 (12)	C13—C12—H12	119.8
C3—C4—C14	120.13 (13)	C12—C13—C8	119.38 (13)
C5—C4—C14	120.64 (13)	C12—C13—H13	120.3
C6—C5—C4	119.46 (13)	C8—C13—H13	120.3
C6—C5—C15	120.11 (13)	C4—C14—H14A	109.5
C4—C5—C15	120.43 (13)	C4—C14—H14B	109.5
C1—C6—C5	119.57 (13)	H14A—C14—H14B	109.5
C1—C6—H6	120.2	C4—C14—H14C	109.5
C5—C6—H6	120.2	H14A—C14—H14C	109.5
O8—C7—O7	123.50 (12)	H14B—C14—H14C	109.5
O8—C7—C8	125.00 (13)	C5—C15—H15A	109.5
O7—C7—C8	111.50 (11)	C5—C15—H15B	109.5
C9—C8—C13	120.17 (12)	H15A—C15—H15B	109.5
C9—C8—C7	117.48 (12)	C5—C15—H15C	109.5
C13—C8—C7	122.32 (12)	H15A—C15—H15C	109.5
C8—C9—C10	120.06 (13)	H15B—C15—H15C	109.5
C7—O7—C1—C2	-115.77 (14)	C1—O7—C7—O8	-8.5 (2)
C7—O7—C1—C6	67.97 (17)	C1—O7—C7—C8	170.72 (11)
C6—C1—C2—C3	-0.1 (2)	O8—C7—C8—C9	-8.7 (2)
O7—C1—C2—C3	-176.31 (12)	O7—C7—C8—C9	172.01 (12)
C1—C2—C3—C4	0.1 (2)	O8—C7—C8—C13	169.10 (14)
C2—C3—C4—C5	-0.2 (2)	O7—C7—C8—C13	-10.15 (18)
C2—C3—C4—C14	179.75 (13)	C13—C8—C9—C10	-0.8 (2)
C3—C4—C5—C6	0.4 (2)	C7—C8—C9—C10	177.06 (13)
C14—C4—C5—C6	-179.59 (13)	C8—C9—C10—C11	0.7 (2)
C3—C4—C5—C15	-179.47 (13)	C9—C10—C11—C12	0.1 (2)
C14—C4—C5—C15	0.6 (2)	C10—C11—C12—C13	-0.8 (2)

C2—C1—C6—C5	0.3 (2)	C11—C12—C13—C8	0.7 (2)
O7—C1—C6—C5	176.31 (12)	C9—C8—C13—C12	0.1 (2)
C4—C5—C6—C1	-0.4 (2)	C7—C8—C13—C12	-177.65 (12)
C15—C5—C6—C1	179.46 (13)		

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
C2—H2...O8 ⁱ	0.95	2.47	3.3710 (18)	157

Symmetry code: (i) $x-1, y, z$.