3-(2,3-Dimethoxyphenyl)-2,3-dihydro-1H-benzo[f]-chromen-1-one

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In the title compound, C_{21}H_{18}O_{4}, the central pyran ring is in an envelope conformation and the dihedral angle between the benzene ring and naphthalene ring system is 88.31 (1)°. The methoxy groups at the *ortho* and *meta* positions of the benzene ring are tilted to the ring with C—C—O—C torsion angles of 105.9 (4)° and 9.5 (5)°, respectively. In the crystal, pairwise C—H⋯O hydrogen bonds form R_{2}(14) inversion dimers, which are linked by another pair of C—H⋯O hydrogen bonds to form [210] chains in the crystal.

**Structure description**

Flavanones exhibit a wide range of biological properties, including antiviral (Shi et al., 2022), antifungal (Emami et al. 2013) and anticancer activities (Bailly, 2021; Zhao et al., 2019) as well as being used in the treatment of Alzheimer’s disease (Jin et al., 2021). In continuation of our research into flavanone derivatives (Sung, 2020), the title compound was synthesized and its crystal structure was determined.

The title compound, C_{21}H_{18}O_{4}, was prepared in a two-step reaction. A Claisen–Schmidt condensation reaction between 2,3-dimethoxy-benzaldehyde and 2-hydroxy-1-acetonaphthone gave the corresponding benzochalcone, which was then used for an intramolecular Michael addition reaction to provide the desired flavanone (Yong et al. 2014). The molecular structure of the title compound is shown in Fig. 1. The central pyran ring (C1/C2/C3/O2/C12/C21) has an envelope conformation with atom C3 as the flap. C3 is a stereogenic centre: in the arbitrarily chosen asymmetric unit, C3 has an S configuration, but crystal symmetry generates a racemic mixture. The hydrogen atom H3 attached to C3 forms a *trans* dihedral conformation with atom H2B of the C2 methylene group (H3—C3—C2—H2B = −179.1°) and a *gauche* conformation with the other H atom attached to C2 (H3—C3—C2—H2A = −60.8°). The methoxy group at the *meta* position of the benzene ring is twisted slightly from the ring [C9—C7—O4—C8 =
The molecular structure of the title compound, showing the atom-labeling scheme and displacement ellipsoids drawn at the 50% probability level.

Figure 1

The weak C⋯H⋯O hydrogen bonds forming R2(14) dimers as yellow lines. An additional pair of intermolecular hydrogen bonds (blue lines) link the dimers to form a chain.

Figure 2

Table 1

| D—H⋯A | D—H | H⋯A | D⋯A | D—H⋯A |
|--------|------|-----|------|--------|
| C13—H13⋯O2" | 0.95 | 2.52 | 3.454 (4) | 169 |
| C18—H18⋯O1" | 0.95 | 2.52 | 3.452 (4) | 166 |

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

9.5 (5)°]. However, the methoxy group at the ortho position is significantly distorted from the benzene ring due to steric hindrance with the pyran ring [C4—C5—O3—C6 = 105.9 (4)°]. The C12–C21 naphthalene ring system (rms. deviation = 0.036 Å) and benzene ring (C4/ C5/C7/ C11/C9/ C10; r.m.s. deviation = 0.003 Å) lie almost perpendicular to each other forming a dihedral angle of 88.31 (1)°. In the crystal, pairs of C18—H18⋯O1 hydrogen bonds form an inversion dimer with graph-set notation R2(14). The dimers are linked by another pair of C13—H13⋯O2 hydrogen bonds to form a [210] chain. (Table 1, Fig. 2).

Synthesis and crystallization

A solution of 2-hydroxy-1-acetonaphthone (186 mg, 1 mmol) and 2,3-dimethoxybenzaldehyde (166 mg, 1 mmol) was dissolved in ethanol (15 ml) and the temperature was adjusted to around 276–277 K in an ice bath. To the cooled reaction mixture was added 1.0 ml of 40% aqueous KOH solution, and the reaction mixture was poured into iced water to give a solid flavanone. Recrystallization from ethanol solution gave the crystals used in this X-ray diffraction study.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

Table 2

| Crystal data | Chemical formula | C21H18O4 |
|--------------|-----------------|----------|
| Mw (g/mol)   |                  | 334.35   |
| Crystal system, space group | Triclinic, P1 | |
| Temperature (K) | 200 | 8.3312 (14), 9.6506 (16), 11.797 (2) |
| a, b, c (Å) |                  | 94.261 (4), 107.335 (4), 112.326 (3) |
| α, β, γ (°) |                  | 81.84 (2) |
| μ (mm⁻¹) |                  | 2.09 |
| Radiation type | Mo Kα | |
| No. of reflections | 5181, 3202, 2136 | |
| Rint |                  | 0.020 |
| (sin θ/λ)max (Å⁻¹) |                  | 0.618 |

Refinement

R[F² > 2σ(F²)], wR(F²), S | 0.057, 0.204, 1.13 |
| No. of reflections | 3202 |
| No. of parameters | 228 |
| H-atoms treatment | H-atoms parameters constrained |
| Δρmax, Δρmin (e Å⁻³) | 0.30, −0.38 |

Computer programs: APEX2 and SAINT (Bruker, 2012), SHELXS and SHELXTL (Sheldrick, 2008), SHELXL2014 (Sheldrick, 2015) and pubICIF (Westrip, 2010).

Funding information

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full crystallographic data

*IUCrData* (2022). 7, x220885  [https://doi.org/10.1107/S2414314622008859]

3-(2,3-Dimethoxyphenyl)-2,3-dihydro-1*H*-benzo[f]chromen-1-one

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3-(2,3-Dimethoxyphenyl)-2,3-dihydro-1*H*-benzo[f]chromen-1-one

**Crystal data**

C₂₁H₁₈O₄

Mr = 334.35

Triclinic, *P*̅1

Hall symbol: -P 1

α = 8.3312 (14) Å

b = 9.6506 (16) Å

c = 11.797 (2) Å

α = 94.261 (4)°

β = 107.335 (4)°

γ = 112.326 (3)°

V = 818.4 (2) Å³

Z = 2

F(000) = 352

D*₀* = 1.357 Mg m⁻³

Mo *Kα* radiation, λ = 0.71073 Å

Cell parameters from 2013 reflections

θ = 2.7–25.9°

µ = 0.09 mm⁻¹

T = 200 K

Block, yellow

0.34 × 0.21 × 0.16 mm

**Data collection**

Bruker SMART CCD diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan

(SADABS; Krause et al., 2015)

T̃min = 0.969, T̃max = 0.985

5181 measured reflections

3202 independent reflections

2136 reflections with *I* > 2σ(*I*)

R̃int = 0.020

θ̃max = 26.1°, θ̃min = 2.3°

h = −10→9

k = −11→11

l = −14→13

**Refinement**

Refinement on *F*²

Least-squares matrix: full

*R*²([*F*² > 2σ(*F*²)]) = 0.057

w*R*²(*F*²) = 0.204

*S* = 1.13

3202 reflections

228 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/[σ*²(*F*ₐ²) + (0.0727*P*)² + 0.8943*P*]

where *P* = (*F*ₐ² + 2*F*₁*₂*)/3

(Δ/σ)max < 0.001

Δρ*max* = 0.30 e Å⁻³

Δρ*min* = −0.38 e Å⁻³

**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 $F^2 > 2\sigma(F^2)$ 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 ($\text{Å}^2$)**

| Atom | $x$     | $y$     | $z$     | $U_{iso}$/$U_{eq}$ |
|------|---------|---------|---------|---------------------|
| C1   | 0.5916 (4) | 0.4265 (4) | 0.1200 (3) | 0.0387 (7)        |
| O1   | 0.7393 (3) | 0.5280 (3) | 0.1255 (2) | 0.0534 (7)        |
| C2   | 0.4897 (5) | 0.4555 (4) | 0.1987 (3) | 0.0467 (8)        |
| H2A  | 0.5804   | 0.5215   | 0.2782   | 0.056*             |
| H2B  | 0.4162   | 0.5101   | 0.1594   | 0.056*             |
| C3   | 0.3641 (4) | 0.3088 (4) | 0.2179 (3) | 0.0404 (8)        |
| H3   | 0.4419   | 0.2569   | 0.2588   | 0.048*             |
| O2   | 0.2359 (3) | 0.2101 (2) | 0.10246 (19) | 0.0406 (6)        |
| C4   | 0.2501 (4) | 0.3214 (4) | 0.2935 (3) | 0.0387 (7)        |
| C5   | 0.2559 (4) | 0.2556 (3) | 0.3936 (3) | 0.0368 (7)        |
| O3   | 0.3634 (3) | 0.1743 (3) | 0.4229 (2) | 0.0470 (6)        |
| C6   | 0.5292 (5) | 0.2525 (5) | 0.5253 (4) | 0.0649 (11)       |
| H6A  | 0.6015   | 0.3530   | 0.5121   | 0.097*             |
| H6B  | 0.6025   | 0.1923   | 0.5364   | 0.097*             |
| H6C  | 0.4983   | 0.2664   | 0.5979   | 0.097*             |
| C7   | 0.1468 (4) | 0.2610 (4) | 0.4626 (3) | 0.0396 (7)        |
| C8   | 0.1617 (3) | 0.1893 (3) | 0.5578 (2) | 0.0501 (6)        |
| C9   | 0.0315 (5) | 0.1696 (5) | 0.6180 (4) | 0.0558 (10)       |
| H8A  | 0.0463   | 0.2702   | 0.6546   | 0.084*             |
| H8B  | 0.0537   | 0.1122   | 0.6816   | 0.084*             |
| H8C  | −0.0947  | 0.1128   | 0.5590   | 0.084*             |
| C9   | 0.0321 (5) | 0.3356 (4) | 0.4296 (3) | 0.0443 (8)        |
| H9   | −0.0429  | 0.3403   | 0.4753   | 0.053*             |
| C10  | 0.0275 (5) | 0.4029 (4) | 0.3300 (3) | 0.0474 (8)        |
| H10  | −0.0501  | 0.4549   | 0.3083   | 0.057*             |
| C11  | 0.1337 (5) | 0.3958 (4) | 0.2616 (3) | 0.0452 (8)        |
| H11  | 0.1277   | 0.4416   | 0.1928   | 0.054*             |
| C12  | 0.3185 (4) | 0.1820 (4) | 0.0248 (3) | 0.0366 (7)        |
| C13  | 0.2063 (4) | 0.0489 (4) | −0.0672 (3) | 0.0425 (8)       |
| H13  | 0.0818   | −0.0104  | −0.0740  | 0.051*             |
| C14  | 0.2764 (5) | 0.0057 (4) | −0.1459 (3) | 0.0427 (8)        |
| H14  | 0.1997   | −0.0838  | −0.2083  | 0.051*             |
| C15  | 0.4630 (4) | 0.0918 (4) | −0.1370 (3) | 0.0378 (7)        |
| C16  | 0.5352 (5) | 0.0391 (4) | −0.2161 (3) | 0.0428 (8)        |
| H16  | 0.4571   | −0.0510  | −0.2778  | 0.051*             |
| C17  | 0.7169 (5) | 0.1166 (4) | −0.2048 (3) | 0.0482 (9)        |
| H17  | 0.7653   | 0.0800   | −0.2577  | 0.058*             |
| C18  | 0.8306 (5) | 0.2500 (4) | −0.1150 (3) | 0.0474 (9)        |
| H18  | 0.9572   | 0.3027   | −0.1065  | 0.057*             |
C19 0.7633 (5) 0.3060 (4) −0.0389 (3) 0.0438 (8)  
H19 0.8427 0.3985 0.0200 0.053*  
C20 0.5757 (4) 0.2274 (4) −0.0469 (3) 0.0366 (7)  
C21 0.4989 (4) 0.2770 (3) 0.0347 (3) 0.0364 (7)  

### Atomic displacement parameters (Å²)

|        | \( U_{11} \)   | \( U_{22} \)   | \( U_{33} \)   | \( U_{12} \)   | \( U_{13} \)   | \( U_{23} \)   |
|--------|----------------|----------------|----------------|----------------|----------------|----------------|
| C1     | 0.0354 (17)    | 0.0354 (16)    | 0.0444 (18)    | 0.0114 (14)    | 0.0171 (14)    | 0.0101 (14)    |
| O1     | 0.0415 (14)    | 0.0415 (13)    | 0.0690 (17)    | 0.0044 (11)    | 0.0286 (13)    | 0.0005 (12)    |
| C2     | 0.0425 (19)    | 0.0418 (18)    | 0.053 (2)      | 0.0108 (15)    | 0.0244 (16)    | −0.0009 (16)   |
| C3     | 0.0327 (16)    | 0.0440 (18)    | 0.0390 (17)    | 0.0112 (14)    | 0.0130 (14)    | 0.0041 (14)    |
| O2     | 0.0309 (11)    | 0.0458 (12)    | 0.0404 (12)    | 0.0101 (10)    | 0.0165 (10)    | 0.0001 (10)    |
| C4     | 0.0335 (16)    | 0.0407 (17)    | 0.0353 (17)    | 0.0101 (14)    | 0.0120 (14)    | 0.0028 (14)    |
| C5     | 0.0330 (16)    | 0.0334 (16)    | 0.0428 (18)    | 0.0123 (13)    | 0.0157 (14)    | 0.0027 (14)    |
| O3     | 0.0464 (14)    | 0.0478 (13)    | 0.0530 (14)    | 0.0236 (11)    | 0.0217 (12)    | 0.0090 (11)    |
| C6     | 0.048 (2)      | 0.087 (3)      | 0.055 (2)      | 0.034 (2)      | 0.0076 (19)    | 0.006 (2)      |
| C7     | 0.0399 (18)    | 0.0389 (17)    | 0.0393 (17)    | 0.0124 (15)    | 0.0198 (15)    | 0.0042 (14)    |
| O4     | 0.0556 (15)    | 0.0598 (15)    | 0.0489 (14)    | 0.0277 (13)    | 0.0312 (12)    | 0.0186 (12)    |
| C8     | 0.061 (2)      | 0.061 (2)      | 0.056 (2)      | 0.023 (2)      | 0.039 (2)      | 0.0157 (19)    |
| C9     | 0.0443 (19)    | 0.0452 (18)    | 0.0471 (19)    | 0.0172 (16)    | 0.0246 (16)    | 0.0045 (15)    |
| C10    | 0.0438 (19)    | 0.050 (2)      | 0.053 (2)      | 0.0234 (17)    | 0.0196 (17)    | 0.0080 (17)    |
| C11    | 0.0451 (19)    | 0.0470 (19)    | 0.0431 (19)    | 0.0180 (16)    | 0.0163 (16)    | 0.0131 (15)    |
| C12    | 0.0325 (16)    | 0.0436 (17)    | 0.0360 (17)    | 0.0157 (14)    | 0.0158 (14)    | 0.0080 (14)    |
| C13    | 0.0292 (16)    | 0.0429 (18)    | 0.0457 (19)    | 0.0065 (14)    | 0.0136 (14)    | 0.0007 (15)    |
| C14    | 0.0402 (18)    | 0.0417 (18)    | 0.0411 (18)    | 0.0120 (15)    | 0.0161 (15)    | 0.0013 (14)    |
| C15    | 0.0361 (17)    | 0.0385 (17)    | 0.0371 (17)    | 0.0141 (14)    | 0.0127 (14)    | 0.0084 (14)    |
| C16    | 0.0433 (19)    | 0.0439 (18)    | 0.0452 (19)    | 0.0190 (15)    | 0.0208 (16)    | 0.0074 (15)    |
| C17    | 0.054 (2)      | 0.050 (2)      | 0.053 (2)      | 0.0255 (18)    | 0.0306 (18)    | 0.0131 (17)    |
| C18    | 0.0397 (18)    | 0.050 (2)      | 0.061 (2)      | 0.0178 (16)    | 0.0294 (17)    | 0.0189 (17)    |
| C19    | 0.0370 (18)    | 0.0438 (18)    | 0.051 (2)      | 0.0136 (15)    | 0.0213 (16)    | 0.0105 (15)    |
| C20    | 0.0343 (16)    | 0.0380 (16)    | 0.0404 (17)    | 0.0147 (14)    | 0.0165 (14)    | 0.0135 (14)    |
| C21    | 0.0315 (16)    | 0.0371 (16)    | 0.0396 (17)    | 0.0120 (13)    | 0.0143 (14)    | 0.0080 (14)    |

### Geometric parameters (Å, °)

|        | C1—O1  | 1.227 (4) | C8—H8C  | 0.9800  |
|--------|--------|-----------|---------|---------|
| C1—C21 | 1.471 (4) | C9—C10    | 1.381 (5) |         |
| C1—C22 | 1.507 (4) | C9—H9     | 0.9500  |         |
| C2—C3  | 1.488 (5) | C10—C11   | 1.379 (5) |         |
| C2—H2A | 0.9900  | C10—H10   | 0.9500  |         |
| C2—H2B | 0.9900  | C11—H11   | 0.9500  |         |
| C3—O2  | 1.439 (4) | C12—C21   | 1.394 (4) |         |
| C3—C4  | 1.513 (4) | C12—C13   | 1.406 (4) |         |
| C3—H3  | 1.0000  | C13—C14   | 1.352 (4) |         |
| O2—C12 | 1.366 (3) | C13—H13   | 0.9500  |         |
| C4—C5  | 1.379 (4) | C14—C15   | 1.423 (4) |         |
| C4—C11 | 1.396 (5) | C14—H14   | 0.9500  |         |
C5—O3 1.389 (4) C15—C20 1.410 (4)
C5—C7 1.400 (4) C15—C16 1.410 (4)
O3—C6 1.419 (4) C16—C17 1.369 (5)
C6—H6A 0.9800 C16—H16 0.9500
C6—H6B 0.9800 C17—C18 1.396 (5)
C6—H6C 0.9800 C17—H17 0.9500
C7—O4 1.362 (4) C18—C19 1.367 (5)
C7—C9 1.388 (5) C18—H18 0.9500
O4—C8 1.428 (4) C19—C20 1.424 (4)
C8—H8A 0.9800 C19—H19 0.9500
C8—H8B 0.9800 C20—C21 1.449 (4)
O1—C1—C2 119.8 (3) C10—C9—C7 119.7 (3)
O1—C1—C2 119.8 (3) C10—C9—H9 120.1
C21—C1—C2 116.4 (3) C11—C10—C9 121.1 (3)
C3—C2—C1 110.8 (3) C11—C10—H10 119.5
C3—C2—H2A 109.5 C12—C11—C4 120.0 (3)
C1—C2—H2A 109.5 C12—C11—H11 120.0
H2A—C2—H2B 108.1 C4—C13—C12 119.7 (3)
O2—C3—C2 109.8 (3) O2—C12—C21 123.3 (3)
O2—C3—C4 116.2 (3) O2—C12—C13 114.5 (3)
C2—C3—C4 107.0 (2) C21—C12—C13 122.3 (3)
C2—C3—H3 116.2 (3) C14—C13—C12 119.7 (3)
O2—C3—H3 107.8 C14—C13—H13 120.2
C4—C3—H3 107.8 C12—C13—H13 120.2
C12—O2—C3 114.2 (2) C13—C14—C15 121.3 (3)
C5—C4—C11 118.8 (3) C13—C14—H14 119.4
C5—C4—C3 120.3 (3) C15—C14—H14 119.4
C11—C4—C3 120.9 (3) C20—C15—C16 120.5 (3)
C4—C5—O3 119.6 (3) C20—C15—C14 119.6 (3)
C4—C5—C7 121.4 (3) C16—C15—C14 119.9 (3)
O3—C5—C7 118.9 (3) C17—C16—C15 120.6 (3)
C5—O3—C6 114.0 (3) C17—C16—H16 119.7
O3—C6—H6A 109.5 C15—C16—H16 119.7
O3—C6—H6B 109.5 C16—C17—C18 119.5 (3)
H6A—C6—H6B 109.5 C16—C17—H17 120.2
O3—C6—H6C 109.5 C18—C17—H17 120.2
H6A—C6—H6C 109.5 C19—C18—C17 121.2 (3)
H6B—C6—H6C 109.5 C19—C18—H18 119.4
O4—C7—C9 125.0 (3) C17—C18—H18 119.4
O4—C7—C5 116.0 (3) C18—C19—C20 120.8 (3)
C9—C7—C5 119.0 (3) C18—C19—H19 119.6
C7—O4—C8 117.8 (3) C20—C19—H19 119.6
O4—C8—H8A 109.5 C15—C20—C19 117.4 (3)
O4—C8—H8B 109.5 C15—C20—C21 119.2 (3)
H8A—C8—H8B 109.5 C19—C20—C21 123.4 (3)
O4—C8—H8C 109.5  C12—C21—C20 117.7 (3)
H8A—C8—H8C 109.5  C12—C21—C1 117.6 (3)
H8B—C8—H8C 109.5  C20—C21—C1 124.3 (3)

O1—C1—C2—C3  −156.5 (3)  C3—O2—C12—C13  158.7 (3)
C21—C1—C2—C3  26.8 (4)   C1—C2—C3—O2  −57.2 (4)
C1—C2—C3—C4  −178.8 (3)  C12—C13—C14—C15  0.6 (5)
C2—C3—O2—C12  55.5 (3)    C13—C14—C15—C20  −2.0 (5)
C4—C3—O2—C12  −177.5 (3)  C12—C13—C14—C16  176.4 (3)
O2—C3—C4—C5  111.0 (3)    C20—C15—C16—C17  1.6 (5)
C2—C3—C4—C5  −125.9 (3)  C14—C15—C16—C17  −176.8 (3)
O2—C3—C4—C11  −67.2 (4)  C15—C16—C17—C18  −8.9 (5)
C2—C3—C4—C11  55.9 (4)    C16—C17—C18—C20  1.7 (5)
C11—C4—C5—O3  176.6 (3)  C1—C2—C3—O2  −57.2 (4)
O2—C3—C4—C11  −177.7 (3)  C12—C13—C14—C15  0.6 (5)
C4—C5—C7—C9  −0.6 (5)    O1—C1—C21—C12  −170.1 (3)
O3—C5—C7—C9  −176.7 (3)  C2—C1—C21—C12  6.4 (4)
C9—C7—C9—C10  9.5 (5)    C3—O2—C12—C21  −21.5 (4)
C5—C7—C9—C10  −169.8 (3)  C2—C1—C21—C20  −179.1 (3)
C7—C9—C10—C11  0.7 (5)    C13—H13···O2ii  0.95  2.52  3.454 (4)  169
C9—C10—C11—C4  −0.8 (5)  C18—H18···O1iv  0.95  2.52  3.452 (4)  166
C5—C4—C11—C10  0.2 (5)    C19—C20—C21—C15  3.9 (4)
C3—C4—C11—C10  178.4 (3)  C15—C20—C21—C12  −173.9 (3)
C3—O2—C12—C21  −21.5 (4)  C1—C2—C3—O2  −57.2 (4)

Hydrogen-bond geometry (Å, °)

| D—H···A   | D—H | H···A | D···A  | D—H···A |
|-----------|------|------|--------|---------|
| C13—H13···O2ii | 0.95 | 2.52 | 3.454 (4) | 169     |
| C18—H18···O1iv  | 0.95 | 2.52 | 3.452 (4) | 166     |

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