Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 3,5-Di-*p*-toluoyl-1,2-dideoxy-β-1-(imidazol-1-yl)-D-ribofuranose

**Nicole Düpre,[a] Wei-Zheng Shen,[a] Pablo J. Sanz Miguel[b] and Jens Müller[a]***

[a]Anorganische Chemie, Technische Universität Dortmund, Otto-Hahn-Strasse 6, 44227 Dortmund, Germany, and [b]Departamento de Quimica Inorganica, Universidad Autonoma de Madrid, 28049 Madrid, Spain
Correspondence e-mail: mueller@muellerlab.org

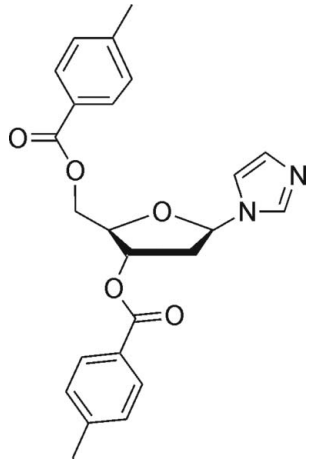
Received 2 November 2007; accepted 7 November 2007

Key indicators: single-crystal X-ray study; $T$ = 103 K; mean $\sigma$(C–C) = 0.006 Å; $R$ factor = 0.060; $wR$ factor = 0.137; data-to-parameter ratio = 9.5.

The title compound, $C_{24}H_{24}N_2O_5$, was obtained as a reaction intermediate in the synthesis of the artificial imidazole nucleoside, which is a highly useful synthetic nucleoside for the formation of metal-ion-mediated base pairs. Compared to the free nucleoside, both OH groups of the sugar moiety are protected by *p*-toluoyl groups. The sugar is in its C3′-*endo* conformation.

## Related literature

The structures of the analogous 1,2,4-triazole and tetrazole nucleosides have been reported recently (Müller *et al.*, 2005). These nucleosides have been used in metal-ion-mediated base pairs (Böhme *et al.*, 2007). For related literature, see: Polonius & Müller (2007); Müller *et al.* (2007); Allen *et al.* (1987); Farrugia (1997, 1999).



## Experimental

### Crystal data

$C_{24}H_{24}N_2O_5$
$M_r$ = 420.45
Monoclinic, $P2_1$
$a$ = 14.436 (3) Å
$b$ = 5.3850 (11) Å
$c$ = 15.061 (3) Å
$\beta$ = 116.25 (3)°
$V$ = 1050.1 (5) Å$^3$
$Z$ = 2
Mo $K\alpha$ radiation
$\mu$ = 0.09 mm$^{-1}$
$T$ = 103 (2) K
0.1 × 0.1 × 0.1 mm

### Data collection

Bruker–Nonius KappaCCD diffractometer
Absorption correction: none
12891 measured reflections
2647 independent reflections
1726 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.097

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.060
$wR(F^2)$ = 0.137
$S$ = 1.01
2647 reflections
280 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.34 e Å$^{-3}$

Data collection: *KappaCCD* (Nonius, 1998); cell refinement: *DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO* and *SCALEPACK*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

This work was supported by the Deutsche Forschungsgemeinschaft, the Department of Chemistry at the University of Dortmund and the Fonds der Chemischen Industrie.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH2551).

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 doi:10.1107/S1600536807056760 © 2007 International Union of Crystallography 

# supplementary materials

*Acta Cryst.* (2007). E**63**, o4693 [ **doi:10.1107/S1600536807056760** ]

# 3,5-Di-*p*-toluoyl-1,2-dideoxy-$\beta$-1-(imidazol-1-yl)-D-ribofuranose

**N. Düpre, W.-Z. Shen, P. J. Sanz Miguel and J. Müller**

## Comment

In the course of our studies regarding the use of artificial azole nucleosides (azole = imidazole, 1,2,4-triazole, tetrazole) in metal-ion mediated base pairs (Böhme *et al.*, 2007; Müller *et al.*, 2007; Polonius & Müller, 2007; Müller *et al.*, 2005) we have isolated the title compound (Figure 1) as a reaction intermediate in the synthesis of imidazole nucleoside. It crystallizes in the same space group and with very similar unit-cell dimensions as the related triazole nucleoside (Müller *et al.*, 2005). All bond lengths and angles are within the normal range (Allen *et al.*, 1987). The artificial nucleobase is connected to the sugar moiety *via* an N-glycosidic bond. It is oriented *anti* with respect to the sugar. The deoxyribose adopts a C3'-*endo* conformation (phase angle of pseudorotation = 7.7 (5)°). The title compound forms long columns along the crystallographic *b* axis (Fig. 2). However, there is no π stacking between neighboring toluoyl rings that are related by the symmetry operator (*x*, *y* + 1, *z*). Instead, neighboring rings that are related by the symmetry operator (1 − *x*, *y* + 1/2, 3 − *z*) are oriented at an angle of 62.7 (2)° with respect to each other.

## Experimental

The title compound was prepared as reported previously (Müller *et al.*, 2005). Single crystals suitable for X-ray measurement were obtained by crystallization from dichloromethane and cyclohexane with triethylamine at room temperature. A satisfactory elemental analysis was obtained: Calcd. for $C_{24}H_{24}N_2O_5$: C 68.6, H 5.8, N 6.7; found: C 68.2, H 6.2, N 6.6.

## Refinement

In the absence of significant anomalous dispersion effects, a total of 1277 Friedel pairs have been merged during the final refinement.

Hydrogen atoms were included in the refinement at calculated positions with C—H = 0.95–1.00 Å and $U_{iso}$(H) = 1.2$U_{eq}$(C) or 1.5$U_{eq}$(C) for methyl groups.

**Figures**

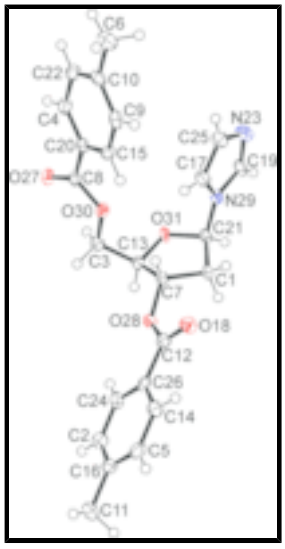



Fig. 1. View of the title compound showing displacement ellipsoids at the 50% probability level.

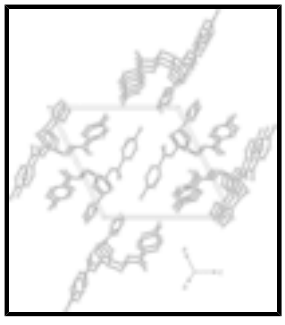


Fig. 2. Packing of the title compound with a view along the *b* axis.

**3,5-Di-*p*-toluoyl-1,2-dideoxy-β-1-(imidazol-1-yl)-*D*-ribofuranose**

*Crystal data*

| | |
|---|---|
| $C_{24}H_{24}N_2O_5$ | $Z = 2$ |
| $M_r = 420.45$ | $F_{000} = 444$ |
| Monoclinic, $P2_1$ | $D_x = 1.33$ Mg m$^{-3}$ |
| Hall symbol: P 2yb | Mo $K\alpha$ radiation<br>$\lambda = 0.71073$ Å |
| $a = 14.436$ (3) Å | $\theta = 3$–27.5º |
| $b = 5.3850$ (11) Å | $\mu = 0.09$ mm$^{-1}$ |
| $c = 15.061$ (3) Å | $T = 103$ (2) K |
| $\beta = 116.25$ (3)º | Cubes, colorless |
| $V = 1050.1$ (5) Å$^3$ | 0.1 × 0.1 × 0.1 mm |

*Data collection*

| | |
|---|---|
| Bruker–Nonius Kappa CCD diffractometer | $R_{int} = 0.097$ |
| profile fitled scans | $\theta_{max} = 27.5$º |

| | |
|---|---|
| Absorption correction: none | $\theta_{min}$ = 3.0º |
| 12891 measured reflections | $h$ = −18→18 |
| 2647 independent reflections | $k$ = −7→5 |
| 1726 reflections with $I > 2\sigma(I)$ | $l$ = −19→19 |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | 1 restraint |
| Least-squares matrix: full | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.060$ | $w = 1/[\sigma^2(F_o^2) + (0.0694P)^2 + 0.2112P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $wR(F^2) = 0.137$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S$ = 1.01 | $\Delta\rho_{max}$ = 0.28 e Å$^{-3}$ |
| 2647 reflections | $\Delta\rho_{min}$ = −0.34 e Å$^{-3}$ |
| 280 parameters | Extinction correction: none |

*Special details*

**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.

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O30 | 0.3603 (2) | 0.8395 (6) | 0.96544 (19) | 0.0222 (7) |
| N29 | 0.1239 (2) | 1.0452 (6) | 0.9936 (2) | 0.0200 (8) |
| O28 | 0.44002 (19) | 0.7940 (6) | 1.24729 (19) | 0.0221 (7) |
| O27 | 0.4201 (2) | 0.9804 (6) | 0.8587 (2) | 0.0259 (7) |
| C26 | 0.5335 (3) | 0.5234 (8) | 1.3793 (3) | 0.0200 (9) |
| C25 | 0.0239 (3) | 0.8334 (9) | 0.8628 (3) | 0.0268 (10) |
| H25A | −0.0014 | 0.7064 | 0.8141 | 0.032* |
| C24 | 0.6206 (3) | 0.6745 (9) | 1.4091 (3) | 0.0267 (11) |
| H24A | 0.6179 | 0.822 | 1.3734 | 0.032* |
| N23 | −0.0292 (3) | 1.0479 (8) | 0.8608 (3) | 0.0303 (9) |
| C22 | 0.1929 (3) | 0.5296 (8) | 0.6379 (3) | 0.0242 (10) |
| H22A | 0.1737 | 0.5516 | 0.5694 | 0.029* |
| C21 | 0.2101 (3) | 1.1388 (8) | 1.0832 (3) | 0.0205 (10) |
| H21A | 0.1866 | 1.2855 | 1.1085 | 0.025* |
| C20 | 0.2901 (3) | 0.6638 (8) | 0.8066 (3) | 0.0196 (9) |
| C19 | 0.0339 (3) | 1.1715 (9) | 0.9398 (3) | 0.0249 (10) |
| H19A | 0.0187 | 1.3304 | 0.9575 | 0.03* |
| O18 | 0.3571 (2) | 0.4591 (6) | 1.2656 (2) | 0.0262 (7) |
| C17 | 0.1172 (3) | 0.8278 (9) | 0.9437 (3) | 0.0256 (10) |
| H17A | 0.1675 | 0.6995 | 0.962 | 0.031* |
| C16 | 0.7183 (3) | 0.3957 (9) | 1.5446 (3) | 0.0283 (12) |

| | | | | |
|---|---|---|---|---|
| C15 | 0.2466 (3) | 0.4697 (8) | 0.8370 (3) | 0.0214 (9) |
| H15A | 0.2654 | 0.4478 | 0.9054 | 0.026* |
| C14 | 0.5391 (3) | 0.3097 (9) | 1.4337 (3) | 0.0257 (10) |
| H14A | 0.48 | 0.2064 | 1.4147 | 0.031* |
| C13 | 0.3831 (3) | 1.0697 (8) | 1.1102 (3) | 0.0194 (9) |
| H13A | 0.4338 | 1.1726 | 1.1657 | 0.023* |
| C12 | 0.4342 (3) | 0.5834 (8) | 1.2935 (3) | 0.0209 (10) |
| C11 | 0.8179 (3) | 0.3260 (12) | 1.6335 (3) | 0.0429 (14) |
| H11A | 0.8709 | 0.4508 | 1.6431 | 0.064* |
| H11B | 0.8412 | 0.1632 | 1.6223 | 0.064* |
| H11C | 0.8059 | 0.3188 | 1.6927 | 0.064* |
| C10 | 0.1487 (3) | 0.3368 (9) | 0.6677 (3) | 0.0245 (10) |
| C9 | 0.1769 (3) | 0.3097 (9) | 0.7689 (3) | 0.0261 (10) |
| H9A | 0.1477 | 0.1794 | 0.7907 | 0.031* |
| C8 | 0.3638 (3) | 0.8416 (9) | 0.8767 (3) | 0.0220 (9) |
| C7 | 0.3513 (3) | 0.8494 (8) | 1.1542 (3) | 0.0180 (9) |
| H7A | 0.3321 | 0.7032 | 1.1087 | 0.022* |
| C6 | 0.0724 (3) | 0.1619 (9) | 0.5931 (3) | 0.0337 (12) |
| H6A | 0.0619 | 0.2086 | 0.5265 | 0.051* |
| H6B | 0.0064 | 0.1713 | 0.5969 | 0.051* |
| H6C | 0.0991 | −0.0082 | 0.6075 | 0.051* |
| C5 | 0.6310 (3) | 0.2462 (9) | 1.5159 (3) | 0.0299 (11) |
| H5A | 0.634 | 0.1001 | 1.5524 | 0.036* |
| C4 | 0.2640 (3) | 0.6893 (9) | 0.7056 (3) | 0.0266 (10) |
| H4A | 0.295 | 0.8159 | 0.6838 | 0.032* |
| C3 | 0.4296 (3) | 1.0075 (9) | 1.0408 (3) | 0.0234 (10) |
| H3A | 0.4392 | 1.1609 | 1.0096 | 0.028* |
| H3B | 0.4979 | 0.9279 | 1.0779 | 0.028* |
| C2 | 0.7116 (3) | 0.6093 (10) | 1.4912 (3) | 0.0315 (12) |
| H2A | 0.7703 | 0.714 | 1.511 | 0.038* |
| C1 | 0.2577 (3) | 0.9476 (9) | 1.1657 (3) | 0.0199 (9) |
| H1A | 0.2082 | 0.8122 | 1.1576 | 0.024* |
| H1B | 0.2792 | 1.0256 | 1.2314 | 0.024* |
| O31 | 0.2904 (2) | 1.2143 (5) | 1.05768 (19) | 0.0208 (7) |

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

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O30 | 0.0189 (14) | 0.0291 (16) | 0.0216 (15) | −0.0078 (14) | 0.0116 (12) | −0.0057 (14) |
| N29 | 0.0149 (17) | 0.0197 (19) | 0.0251 (18) | −0.0006 (16) | 0.0087 (15) | −0.0016 (16) |
| O28 | 0.0160 (14) | 0.0252 (16) | 0.0221 (15) | 0.0017 (13) | 0.0057 (12) | 0.0027 (13) |
| O27 | 0.0230 (15) | 0.0327 (19) | 0.0266 (16) | −0.0017 (15) | 0.0153 (13) | −0.0005 (15) |
| C26 | 0.022 (2) | 0.022 (2) | 0.020 (2) | 0.0020 (19) | 0.0131 (18) | −0.0048 (18) |
| C25 | 0.023 (2) | 0.027 (2) | 0.029 (2) | −0.003 (2) | 0.010 (2) | −0.001 (2) |
| C24 | 0.021 (2) | 0.032 (3) | 0.029 (2) | 0.001 (2) | 0.014 (2) | −0.001 (2) |
| N23 | 0.0244 (19) | 0.033 (2) | 0.027 (2) | −0.0011 (19) | 0.0055 (17) | 0.0014 (19) |
| C22 | 0.026 (2) | 0.025 (3) | 0.022 (2) | 0.005 (2) | 0.0110 (19) | −0.001 (2) |
| C21 | 0.014 (2) | 0.023 (2) | 0.027 (2) | −0.0015 (19) | 0.0100 (18) | 0.001 (2) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C20 | 0.016 (2) | 0.023 (2) | 0.022 (2) | 0.006 (2) | 0.0095 (18) | 0.000 (2) |
| C19 | 0.017 (2) | 0.026 (2) | 0.033 (2) | 0.007 (2) | 0.012 (2) | 0.006 (2) |
| O18 | 0.0247 (16) | 0.0233 (16) | 0.0275 (16) | −0.0036 (15) | 0.0087 (13) | 0.0007 (15) |
| C17 | 0.020 (2) | 0.028 (2) | 0.027 (2) | 0.001 (2) | 0.009 (2) | −0.001 (2) |
| C16 | 0.025 (2) | 0.043 (3) | 0.019 (2) | 0.011 (2) | 0.010 (2) | 0.001 (2) |
| C15 | 0.022 (2) | 0.026 (2) | 0.018 (2) | 0.004 (2) | 0.0107 (18) | 0.005 (2) |
| C14 | 0.026 (2) | 0.026 (2) | 0.026 (2) | 0.004 (2) | 0.012 (2) | 0.001 (2) |
| C13 | 0.016 (2) | 0.021 (2) | 0.021 (2) | 0.0005 (18) | 0.0076 (17) | −0.0008 (19) |
| C12 | 0.025 (2) | 0.018 (2) | 0.021 (2) | 0.006 (2) | 0.0122 (19) | 0.002 (2) |
| C11 | 0.026 (2) | 0.066 (4) | 0.034 (3) | 0.019 (3) | 0.012 (2) | 0.010 (3) |
| C10 | 0.017 (2) | 0.025 (2) | 0.028 (2) | 0.007 (2) | 0.0070 (19) | 0.001 (2) |
| C9 | 0.021 (2) | 0.026 (2) | 0.035 (2) | 0.005 (2) | 0.016 (2) | 0.000 (2) |
| C8 | 0.018 (2) | 0.028 (2) | 0.024 (2) | 0.006 (2) | 0.0132 (19) | 0.002 (2) |
| C7 | 0.0150 (19) | 0.023 (2) | 0.017 (2) | −0.0027 (19) | 0.0075 (17) | 0.0003 (19) |
| C6 | 0.024 (2) | 0.033 (3) | 0.036 (3) | 0.001 (2) | 0.006 (2) | −0.003 (2) |
| C5 | 0.034 (3) | 0.035 (3) | 0.025 (2) | 0.009 (2) | 0.017 (2) | 0.010 (2) |
| C4 | 0.024 (2) | 0.033 (3) | 0.028 (2) | 0.003 (2) | 0.016 (2) | 0.002 (2) |
| C3 | 0.018 (2) | 0.029 (3) | 0.025 (2) | −0.004 (2) | 0.0101 (18) | −0.001 (2) |
| C2 | 0.024 (2) | 0.044 (3) | 0.025 (2) | −0.002 (2) | 0.010 (2) | 0.000 (2) |
| C1 | 0.0130 (19) | 0.026 (2) | 0.023 (2) | −0.0015 (19) | 0.0096 (17) | 0.000 (2) |
| O31 | 0.0145 (14) | 0.0247 (16) | 0.0266 (15) | 0.0009 (13) | 0.0123 (12) | 0.0029 (13) |

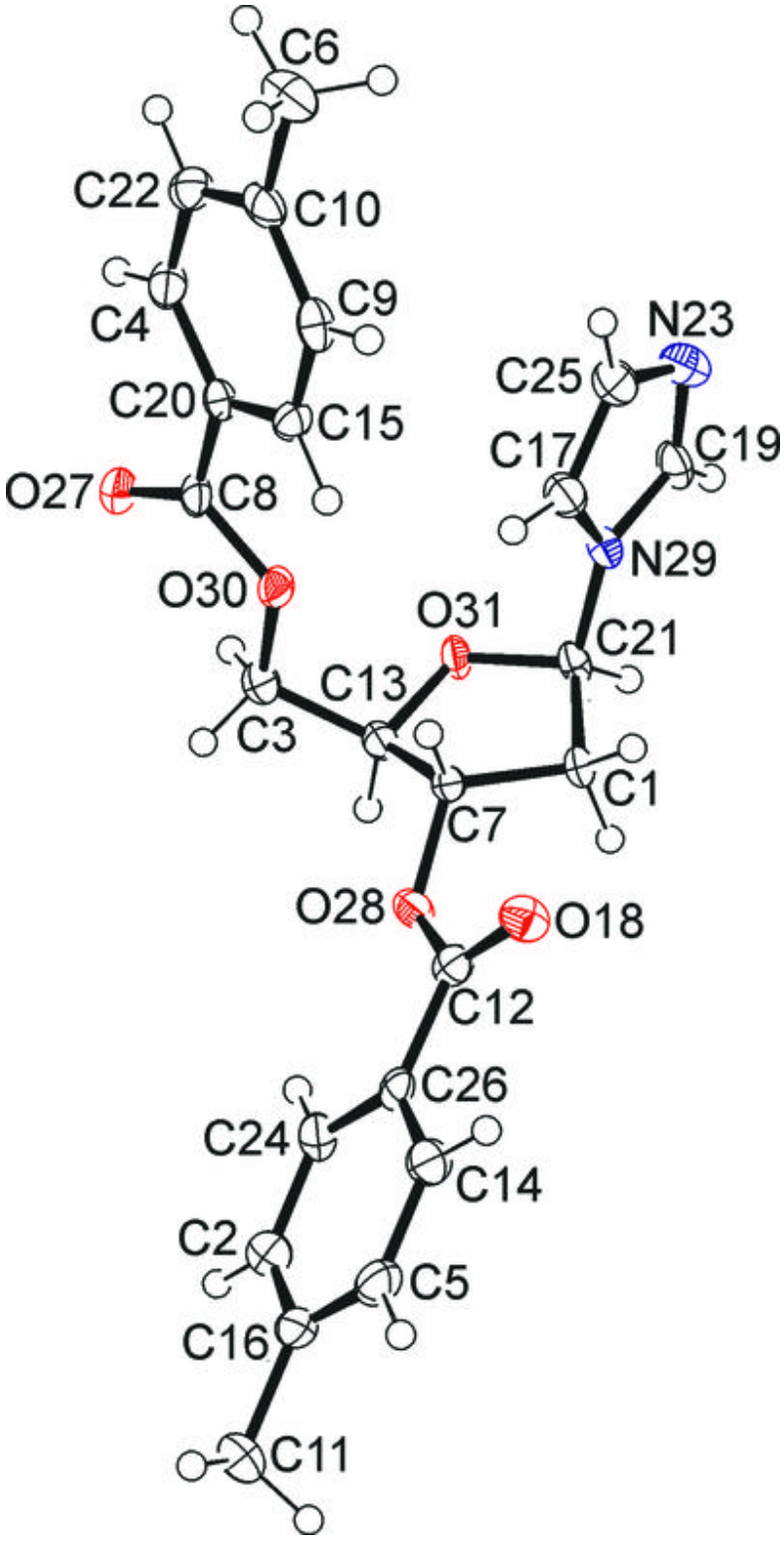
*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O30—C8 | 1.360 (5) | C16—C2 | 1.382 (6) |
| O30—C3 | 1.450 (5) | C16—C5 | 1.393 (7) |
| N29—C19 | 1.370 (5) | C16—C11 | 1.516 (6) |
| N29—C17 | 1.371 (6) | C15—C9 | 1.377 (6) |
| N29—C21 | 1.462 (5) | C15—H15A | 0.95 |
| O28—C12 | 1.352 (5) | C14—C5 | 1.400 (6) |
| O28—C7 | 1.451 (4) | C14—H14A | 0.95 |
| O27—C8 | 1.220 (5) | C13—O31 | 1.445 (5) |
| C26—C14 | 1.394 (6) | C13—C3 | 1.509 (5) |
| C26—C24 | 1.395 (6) | C13—C7 | 1.525 (6) |
| C26—C12 | 1.480 (5) | C13—H13A | 1 |
| C25—C17 | 1.360 (6) | C11—H11A | 0.98 |
| C25—N23 | 1.379 (6) | C11—H11B | 0.98 |
| C25—H25A | 0.95 | C11—H11C | 0.98 |
| C24—C2 | 1.393 (6) | C10—C9 | 1.400 (6) |
| C24—H24A | 0.95 | C10—C6 | 1.506 (6) |
| N23—C19 | 1.315 (6) | C9—H9A | 0.95 |
| C22—C4 | 1.381 (6) | C7—C1 | 1.530 (6) |
| C22—C10 | 1.393 (6) | C7—H7A | 1 |
| C22—H22A | 0.95 | C6—H6A | 0.98 |
| C21—O31 | 1.432 (5) | C6—H6B | 0.98 |
| C21—C1 | 1.523 (6) | C6—H6C | 0.98 |
| C21—H21A | 1 | C5—H5A | 0.95 |
| C20—C15 | 1.396 (6) | C4—H4A | 0.95 |
| C20—C4 | 1.404 (5) | C3—H3A | 0.99 |

| | | | |
|---|---|---|---|
| C20—C8 | 1.473 (6) | C3—H3B | 0.99 |
| C19—H19A | 0.95 | C2—H2A | 0.95 |
| O18—C12 | 1.205 (5) | C1—H1A | 0.99 |
| C17—H17A | 0.95 | C1—H1B | 0.99 |
| C8—O30—C3 | 116.8 (3) | O28—C12—C26 | 112.1 (4) |
| C19—N29—C17 | 106.3 (3) | C16—C11—H11A | 109.5 |
| C19—N29—C21 | 124.9 (4) | C16—C11—H11B | 109.5 |
| C17—N29—C21 | 128.7 (3) | H11A—C11—H11B | 109.5 |
| C12—O28—C7 | 116.1 (3) | C16—C11—H11C | 109.5 |
| C14—C26—C24 | 118.9 (4) | H11A—C11—H11C | 109.5 |
| C14—C26—C12 | 118.3 (4) | H11B—C11—H11C | 109.5 |
| C24—C26—C12 | 122.7 (4) | C22—C10—C9 | 118.3 (4) |
| C17—C25—N23 | 111.0 (4) | C22—C10—C6 | 121.0 (4) |
| C17—C25—H25A | 124.5 | C9—C10—C6 | 120.7 (4) |
| N23—C25—H25A | 124.5 | C15—C9—C10 | 120.7 (4) |
| C2—C24—C26 | 120.0 (4) | C15—C9—H9A | 119.6 |
| C2—C24—H24A | 120 | C10—C9—H9A | 119.6 |
| C26—C24—H24A | 120 | O27—C8—O30 | 122.8 (4) |
| C19—N23—C25 | 104.4 (4) | O27—C8—C20 | 125.5 (4) |
| C4—C22—C10 | 121.5 (4) | O30—C8—C20 | 111.7 (4) |
| C4—C22—H22A | 119.2 | O28—C7—C13 | 105.9 (3) |
| C10—C22—H22A | 119.2 | O28—C7—C1 | 114.1 (3) |
| O31—C21—N29 | 108.0 (3) | C13—C7—C1 | 103.3 (3) |
| O31—C21—C1 | 106.5 (3) | O28—C7—H7A | 111 |
| N29—C21—C1 | 114.4 (4) | C13—C7—H7A | 111 |
| O31—C21—H21A | 109.3 | C1—C7—H7A | 111 |
| N29—C21—H21A | 109.3 | C10—C6—H6A | 109.5 |
| C1—C21—H21A | 109.3 | C10—C6—H6B | 109.5 |
| C15—C20—C4 | 119.0 (4) | H6A—C6—H6B | 109.5 |
| C15—C20—C8 | 122.6 (4) | C10—C6—H6C | 109.5 |
| C4—C20—C8 | 118.4 (4) | H6A—C6—H6C | 109.5 |
| N23—C19—N29 | 112.3 (4) | H6B—C6—H6C | 109.5 |
| N23—C19—H19A | 123.8 | C16—C5—C14 | 120.5 (4) |
| N29—C19—H19A | 123.8 | C16—C5—H5A | 119.8 |
| C25—C17—N29 | 105.9 (4) | C14—C5—H5A | 119.8 |
| C25—C17—H17A | 127.1 | C22—C4—C20 | 119.7 (4) |
| N29—C17—H17A | 127.1 | C22—C4—H4A | 120.2 |
| C2—C16—C5 | 118.6 (4) | C20—C4—H4A | 120.2 |
| C2—C16—C11 | 121.0 (5) | O30—C3—C13 | 108.4 (3) |
| C5—C16—C11 | 120.3 (4) | O30—C3—H3A | 110 |
| C9—C15—C20 | 120.7 (4) | C13—C3—H3A | 110 |
| C9—C15—H15A | 119.6 | O30—C3—H3B | 110 |
| C20—C15—H15A | 119.6 | C13—C3—H3B | 110 |
| C26—C14—C5 | 120.4 (4) | H3A—C3—H3B | 108.4 |
| C26—C14—H14A | 119.8 | C16—C2—C24 | 121.5 (5) |
| C5—C14—H14A | 119.8 | C16—C2—H2A | 119.2 |
| O31—C13—C3 | 109.2 (3) | C24—C2—H2A | 119.2 |
| O31—C13—C7 | 105.8 (3) | C21—C1—C7 | 104.1 (3) |
| C3—C13—C7 | 116.1 (4) | C21—C1—H1A | 110.9 |

| | | | |
|---|---|---|---|
| O31—C13—H13A | 108.5 | C7—C1—H1A | 110.9 |
| C3—C13—H13A | 108.5 | C21—C1—H1B | 110.9 |
| C7—C13—H13A | 108.5 | C7—C1—H1B | 110.9 |
| O18—C12—O28 | 123.0 (4) | H1A—C1—H1B | 109 |
| O18—C12—C26 | 124.9 (4) | C21—O31—C13 | 111.3 (3) |
| C12—O28—C7—C1 | 75.4 (5) | C22—C4—C20—C8 | −178.2 (4) |
| C12—O28—C7—C13 | −171.7 (3) | C22—C4—C20—C15 | 2.7 (7) |
| C7—O28—C12—O18 | −7.0 (6) | C20—C4—C22—C10 | −2.3 (7) |
| C7—O28—C12—C26 | 171.5 (3) | C14—C5—C16—C2 | 1.0 (7) |
| C8—O30—C3—C13 | 153.6 (4) | C14—C5—C16—C11 | −179.8 (4) |
| C3—O30—C8—O27 | −1.7 (6) | C16—C5—C14—C26 | 0.0 (7) |
| C3—O30—C8—C20 | 180.0 (4) | O28—C7—C13—O31 | −146.7 (3) |
| C21—O31—C13—C3 | 139.0 (4) | O28—C7—C13—C3 | 92.0 (4) |
| C21—O31—C13—C7 | 13.4 (4) | C1—C7—C13—O31 | −26.5 (4) |
| C13—O31—C21—N29 | −117.7 (3) | C1—C7—C13—C3 | −147.8 (4) |
| C13—O31—C21—C1 | 5.6 (4) | O30—C8—C20—C15 | −17.9 (6) |
| C19—N23—C25—C17 | 1.3 (5) | O27—C8—C20—C15 | 163.9 (5) |
| C25—N23—C19—N29 | −1.3 (5) | O30—C8—C20—C4 | 163.0 (4) |
| C19—N29—C17—C25 | 0.0 (5) | O27—C8—C20—C4 | −15.2 (7) |
| C19—N29—C21—O31 | −104.2 (4) | C15—C9—C10—C6 | −180.0 (4) |
| C19—N29—C21—C1 | 137.4 (4) | C10—C9—C15—C20 | 0.5 (7) |
| C21—N29—C19—N23 | 177.9 (4) | C15—C9—C10—C22 | 0.0 (7) |
| C21—N29—C17—C25 | −176.9 (4) | C6—C10—C22—C4 | −179.1 (4) |
| C17—N29—C19—N23 | 0.9 (5) | C9—C10—C22—C4 | 1.0 (7) |
| C17—N29—C21—O31 | 72.1 (5) | O18—C12—C26—C24 | −179.6 (4) |
| C17—N29—C21—C1 | −46.3 (6) | O28—C12—C26—C14 | −179.6 (4) |
| C21—C1—C7—C13 | 29.4 (4) | O18—C12—C26—C14 | −1.1 (7) |
| C7—C1—C21—O31 | −22.1 (4) | O28—C12—C26—C24 | 1.9 (6) |
| C21—C1—C7—O28 | 143.8 (4) | C5—C14—C26—C12 | −179.5 (4) |
| C7—C1—C21—N29 | 97.1 (4) | C5—C14—C26—C24 | −0.9 (7) |
| C16—C2—C24—C26 | 0.2 (7) | C9—C15—C20—C8 | 179.1 (4) |
| C24—C2—C16—C11 | 179.7 (4) | C9—C15—C20—C4 | −1.8 (7) |
| C24—C2—C16—C5 | −1.1 (7) | N29—C17—C25—N23 | −0.8 (5) |
| O30—C3—C13—O31 | −67.2 (4) | C2—C24—C26—C14 | 0.8 (7) |
| O30—C3—C13—C7 | 52.4 (5) | C2—C24—C26—C12 | 179.3 (4) |

**Fig. 1**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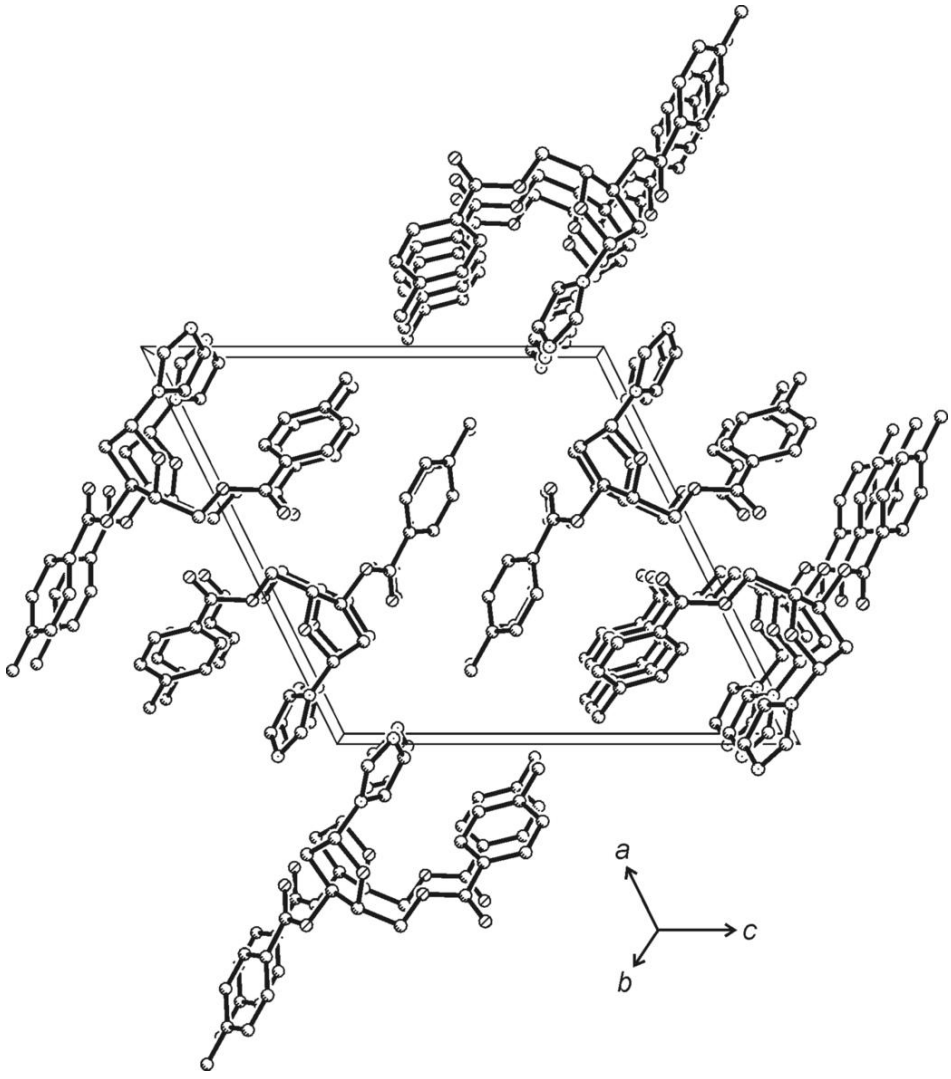
**Fig. 2**