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(E)-N-(3,3-Diphenylallylidene)-3-nitroanilineJoo Hwan Cha,^a Yong Koo Kang,^b Yong Seo Cho,^b
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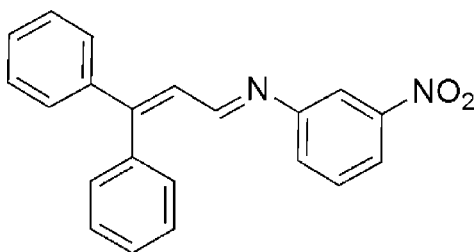
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.002$ Å; R factor = 0.043; wR factor = 0.136; data-to-parameter ratio = 17.1.

In the title compound, $\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_2$, the 3-nitrophenyl and two phenyl rings are twisted from the mean plane of the enimino fragment by 44.4 (1), 37.2 (1) and 74.1 (1)°, respectively. The crystal packing exhibits no classical intermolecular contacts.

Related literature

For the structure of (E)-N-(3,3-diphenylallylidene)-4-nitroaniline, see: Kang *et al.* (2012). For the crystal structures of other closely related compounds, see: Khalaji *et al.* (2008a,b).



Experimental

Crystal data

$\text{C}_{21}\text{H}_{16}\text{N}_2\text{O}_2$
 $M_r = 328.36$
 Monoclinic, $P2_1/n$
 $a = 5.8625$ (7) Å
 $b = 22.825$ (3) Å
 $c = 12.6370$ (17) Å
 $\beta = 94.772$ (4)°

$V = 1685.1$ (4) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 296$ K
 $0.40 \times 0.20 \times 0.20$ mm

Data collection

Rigaku R-Axis RAPID diffractometer
 Absorption correction: multi-scan (ABSCOR; Rigaku, 1995)
 $T_{\min} = 0.967$, $T_{\max} = 0.983$

16270 measured reflections
 3866 independent reflections
 2663 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.136$
 $S = 1.12$
 3866 reflections

226 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.18$ e Å⁻³
 $\Delta\rho_{\min} = -0.25$ e Å⁻³

Data collection: *RAPID-AUTO* (Rigaku, 2006); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2010); software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CV5343).

References

- Kang, Y. K., Cho, Y. S., Lee, J. K., Yu, B.-Y. & Cha, J. H. (2012). *Acta Cryst.* **E68**, o3031.
 Khalaji, A. D. & Harrison, W. T. A. (2008a). *Anal. Sci. X-Ray Struct. Anal. Online*, **24**, x3–x4.
 Khalaji, A. D., Welter, R., Amirnasr, M. & Barry, A. H. (2008b). *Anal. Sci. X-Ray Struct. Anal. Online*, **24**, x139–x140.
 Rigaku (1995). *ABSCOR*. Rigaku Corporation, Tokyo, Japan.
 Rigaku (2006). *RAPID-AUTO*. Rigaku Corporation, Tokyo, Japan.
 Rigaku (2010). *CrystalStructure*. Rigaku Corporation, Tokyo, Japan.
 Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2012). E68, o3030 [doi:10.1107/S1600536812040354]

(E)-N-(3,3-Diphenylallylidene)-3-nitroaniline

Joo Hwan Cha, Yong Koo Kang, Yong Seo Cho, Jae Kyun Lee and Jae Choon Woo

Comment

In the title molecule (Fig. 1), the dihedral angles between the mean planes of the central 3-nitrophenyl ring and phenyl rings C7–C12 and C1–C6 are 86.76 (8)° and 8.23 (3)°, respectively. The bond lengths and angles are unexceptional and correspond to those observed in the related compounds Khalaji *et al.* (2008*a,b*). The imine group displays a torsion angle C21–C16–N1–C15 of -45.19 (18)°. The nitro group is twisted at 1.1 (2)° from the attached benzene ring. The crystal packing exhibits no classical intermolecular contacts.

Experimental

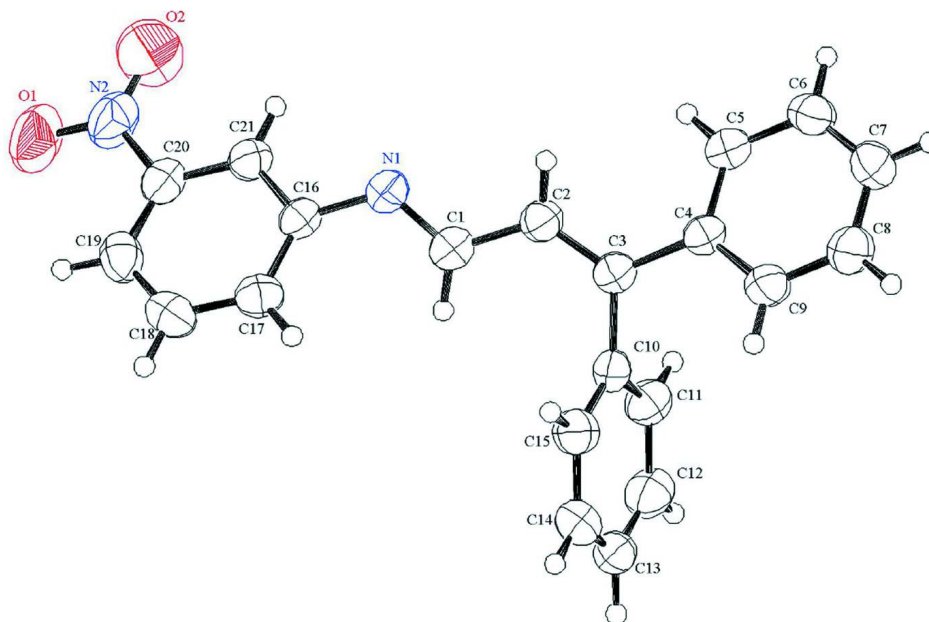
To a solution of 3-nitroaniline (4.0 mmol) in ethanol (10 mL) was treated with equimolar quantities of substituted 2-phenylcinnamaldehydes. The mixture was refluxed for 5 h, and the progress of the reaction was monitored by TLC. Upon completion, the solvent was removed under reduced pressure. The residue was purified by flash column chromatography to afford the title compound in 60% yield. Recrystallization from ethanol gave crystals suitable for X-ray analysis.

Refinement

All hydrogen atoms were positioned geometrically (C—H = 0.93 Å), and refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$.

Computing details

Data collection: *RAPID-AUTO* (Rigaku, 2006); cell refinement: *RAPID-AUTO* (Rigaku, 2006); data reduction: *RAPID-AUTO* (Rigaku, 2006); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2010); software used to prepare material for publication: *CrystalStructure* (Rigaku, 2010).


Figure 1

The molecular structure of the title compound showing the atomic numbering and 50% probability displacement ellipsoid.

(E)-N-(3,3-Diphenylallylidene)-3-nitroaniline
Crystal data

$C_{21}H_{16}N_2O_2$
 $M_r = 328.36$
 Monoclinic, $P2_1/n$
 Hall symbol: -P 2yn
 $a = 5.8625 (7) \text{ \AA}$
 $b = 22.825 (3) \text{ \AA}$
 $c = 12.6370 (17) \text{ \AA}$
 $\beta = 94.772 (4)^\circ$
 $V = 1685.1 (4) \text{ \AA}^3$
 $Z = 4$

$F(000) = 688$
 $D_x = 1.294 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation, $\lambda = 0.71075 \text{ \AA}$
 Cell parameters from 8104 reflections
 $\theta = 3.1\text{--}27.5^\circ$
 $\mu = 0.08 \text{ mm}^{-1}$
 $T = 296 \text{ K}$
 Block, colourless
 $0.40 \times 0.20 \times 0.20 \text{ mm}$

Data collection

Rigaku R-Axis RAPID
 diffractometer
 Radiation source: fine-focus sealed tube
 Graphite monochromator
 Detector resolution: $10.000 \text{ pixels mm}^{-1}$
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Rigaku, 1995)
 $T_{\min} = 0.967$, $T_{\max} = 0.983$

16270 measured reflections
 3866 independent reflections
 2663 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\max} = 27.5^\circ$, $\theta_{\min} = 3.1^\circ$
 $h = -7 \rightarrow 7$
 $k = -29 \rightarrow 29$
 $l = -16 \rightarrow 16$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.136$
 $S = 1.12$
 3866 reflections
 226 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
 $w = 1/[\sigma^2(F_o^2) + (0.075P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.18 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.25 \text{ e } \text{\AA}^{-3}$

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 $F^2 > 2\sigma(F^2)$ 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)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
O1	1.2806 (3)	0.79532 (9)	0.84554 (13)	0.1205 (6)
O2	1.5869 (3)	0.74640 (6)	0.83527 (11)	0.0942 (5)
N1	1.14274 (19)	0.90554 (5)	0.51096 (9)	0.0464 (3)
N2	1.4414 (3)	0.78014 (6)	0.79813 (11)	0.0690 (4)
C1	0.5605 (3)	1.01496 (6)	0.15119 (11)	0.0525 (4)
H1	0.6562	1.0034	0.0999	0.063*
C2	0.3920 (3)	1.05589 (7)	0.12595 (13)	0.0613 (4)
H2	0.3776	1.0724	0.0584	0.074*
C3	0.2448 (3)	1.07259 (7)	0.19989 (13)	0.0573 (4)
H3	0.1292	1.0996	0.1820	0.069*
C4	0.2700 (2)	1.04891 (6)	0.30091 (12)	0.0506 (3)
H4	0.1717	1.0602	0.3513	0.061*
C5	0.4406 (2)	1.00859 (6)	0.32705 (11)	0.0452 (3)
H5	0.4567	0.9931	0.3953	0.054*
C6	0.5893 (2)	0.99065 (5)	0.25294 (10)	0.0405 (3)
C7	1.0213 (2)	0.90953 (7)	0.14288 (12)	0.0557 (4)
H7	1.1302	0.9376	0.1645	0.067*
C8	1.0584 (3)	0.87204 (8)	0.05913 (13)	0.0639 (4)
H8	1.1913	0.8756	0.0243	0.077*
C9	0.9005 (3)	0.82994 (7)	0.02771 (12)	0.0604 (4)
H9	0.9265	0.8050	-0.0282	0.072*
C10	0.7044 (3)	0.82455 (6)	0.07869 (13)	0.0594 (4)
H10	0.5979	0.7957	0.0580	0.071*
C11	0.6648 (2)	0.86225 (6)	0.16137 (11)	0.0510 (3)
H11	0.5306	0.8587	0.1953	0.061*
C12	0.8224 (2)	0.90501 (6)	0.19400 (10)	0.0410 (3)

C13	0.7721 (2)	0.94683 (5)	0.28026 (10)	0.0398 (3)
C14	0.8838 (2)	0.94559 (6)	0.37731 (10)	0.0442 (3)
H14	0.8489	0.9749	0.4245	0.053*
C15	1.0533 (2)	0.90295 (6)	0.41509 (10)	0.0443 (3)
H15	1.0975	0.8737	0.3699	0.053*
C16	1.3099 (2)	0.86430 (5)	0.54794 (10)	0.0410 (3)
C17	1.2973 (2)	0.84251 (6)	0.65043 (10)	0.0463 (3)
H17	1.1783	0.8536	0.6904	0.056*
C18	1.4632 (2)	0.80448 (6)	0.69138 (11)	0.0482 (3)
C19	1.6476 (2)	0.78813 (6)	0.63736 (13)	0.0539 (4)
H19	1.7594	0.7630	0.6678	0.065*
C20	1.6606 (2)	0.81029 (6)	0.53643 (13)	0.0554 (4)
H20	1.7835	0.8002	0.4981	0.067*
C21	1.4928 (2)	0.84741 (6)	0.49177 (11)	0.0495 (3)
H21	1.5026	0.8613	0.4231	0.059*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.1312 (13)	0.1592 (16)	0.0769 (10)	0.0438 (12)	0.0438 (10)	0.0502 (10)
O2	0.1215 (11)	0.0826 (9)	0.0741 (9)	0.0176 (8)	-0.0187 (8)	0.0249 (7)
N1	0.0500 (6)	0.0493 (6)	0.0393 (6)	0.0027 (5)	0.0002 (5)	0.0003 (5)
N2	0.0866 (10)	0.0658 (9)	0.0528 (8)	0.0040 (7)	-0.0050 (8)	0.0130 (7)
C1	0.0609 (8)	0.0564 (8)	0.0408 (7)	0.0120 (7)	0.0083 (6)	0.0034 (6)
C2	0.0769 (10)	0.0594 (9)	0.0473 (8)	0.0162 (8)	0.0031 (7)	0.0086 (7)
C3	0.0589 (8)	0.0508 (8)	0.0614 (9)	0.0128 (7)	0.0000 (7)	-0.0032 (7)
C4	0.0492 (7)	0.0508 (8)	0.0528 (8)	0.0019 (6)	0.0092 (6)	-0.0097 (6)
C5	0.0489 (7)	0.0467 (7)	0.0402 (7)	-0.0042 (6)	0.0051 (6)	-0.0033 (6)
C6	0.0439 (6)	0.0399 (6)	0.0375 (6)	-0.0028 (5)	0.0025 (5)	-0.0023 (5)
C7	0.0442 (7)	0.0690 (9)	0.0536 (8)	-0.0002 (7)	0.0027 (6)	-0.0100 (7)
C8	0.0522 (8)	0.0849 (11)	0.0549 (9)	0.0185 (8)	0.0053 (7)	-0.0081 (8)
C9	0.0762 (10)	0.0566 (9)	0.0464 (8)	0.0275 (8)	-0.0068 (8)	-0.0081 (7)
C10	0.0752 (10)	0.0443 (8)	0.0564 (9)	0.0008 (7)	-0.0073 (8)	-0.0059 (6)
C11	0.0552 (8)	0.0493 (8)	0.0483 (8)	-0.0040 (6)	0.0020 (6)	-0.0015 (6)
C12	0.0420 (6)	0.0421 (7)	0.0380 (7)	0.0047 (5)	-0.0019 (5)	0.0020 (5)
C13	0.0409 (6)	0.0403 (6)	0.0386 (6)	-0.0033 (5)	0.0051 (5)	0.0012 (5)
C14	0.0474 (7)	0.0443 (7)	0.0405 (7)	-0.0004 (6)	0.0010 (6)	-0.0019 (5)
C15	0.0479 (7)	0.0448 (7)	0.0400 (7)	-0.0032 (6)	0.0020 (6)	-0.0004 (5)
C16	0.0439 (6)	0.0415 (6)	0.0370 (6)	-0.0037 (5)	-0.0001 (5)	-0.0026 (5)
C17	0.0477 (7)	0.0519 (7)	0.0395 (7)	0.0006 (6)	0.0053 (6)	-0.0013 (6)
C18	0.0559 (7)	0.0451 (7)	0.0425 (7)	-0.0035 (6)	-0.0034 (6)	0.0011 (6)
C19	0.0528 (8)	0.0434 (7)	0.0637 (9)	0.0046 (6)	-0.0056 (7)	-0.0020 (6)
C20	0.0485 (7)	0.0523 (8)	0.0667 (10)	0.0017 (7)	0.0123 (7)	-0.0082 (7)
C21	0.0544 (7)	0.0502 (7)	0.0450 (7)	-0.0032 (6)	0.0094 (6)	-0.0004 (6)

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

O1—N2	1.209 (2)	C9—C10	1.369 (2)
O2—N2	1.2144 (18)	C9—H9	0.9300
N1—C15	1.2808 (16)	C10—C11	1.388 (2)

N1—C16	1.4101 (16)	C10—H10	0.9300
N2—C18	1.4742 (19)	C11—C12	1.3831 (19)
C1—C2	1.378 (2)	C11—H11	0.9300
C1—C6	1.3977 (18)	C12—C13	1.4965 (18)
C1—H1	0.9300	C13—C14	1.3419 (18)
C2—C3	1.378 (2)	C14—C15	1.4435 (18)
C2—H2	0.9300	C14—H14	0.9300
C3—C4	1.383 (2)	C15—H15	0.9300
C3—H3	0.9300	C16—C21	1.3890 (19)
C4—C5	1.3792 (19)	C16—C17	1.3950 (18)
C4—H4	0.9300	C17—C18	1.3722 (19)
C5—C6	1.3930 (18)	C17—H17	0.9300
C5—H5	0.9300	C18—C19	1.377 (2)
C6—C13	1.4852 (17)	C19—C20	1.380 (2)
C7—C12	1.383 (2)	C19—H19	0.9300
C7—C8	1.392 (2)	C20—C21	1.383 (2)
C7—H7	0.9300	C20—H20	0.9300
C8—C9	1.370 (2)	C21—H21	0.9300
C8—H8	0.9300		
C15—N1—C16	120.07 (12)	C12—C11—C10	120.85 (14)
O1—N2—O2	122.82 (15)	C12—C11—H11	119.6
O1—N2—C18	118.43 (14)	C10—C11—H11	119.6
O2—N2—C18	118.75 (16)	C7—C12—C11	118.79 (12)
C2—C1—C6	120.77 (13)	C7—C12—C13	121.17 (12)
C2—C1—H1	119.6	C11—C12—C13	119.99 (11)
C6—C1—H1	119.6	C14—C13—C6	120.97 (12)
C3—C2—C1	120.55 (14)	C14—C13—C12	122.78 (11)
C3—C2—H2	119.7	C6—C13—C12	116.25 (10)
C1—C2—H2	119.7	C13—C14—C15	125.94 (12)
C2—C3—C4	119.55 (13)	C13—C14—H14	117.0
C2—C3—H3	120.2	C15—C14—H14	117.0
C4—C3—H3	120.2	N1—C15—C14	119.87 (13)
C5—C4—C3	120.11 (13)	N1—C15—H15	120.1
C5—C4—H4	119.9	C14—C15—H15	120.1
C3—C4—H4	119.9	C21—C16—C17	118.56 (12)
C4—C5—C6	121.14 (13)	C21—C16—N1	124.11 (12)
C4—C5—H5	119.4	C17—C16—N1	117.18 (12)
C6—C5—H5	119.4	C18—C17—C16	119.03 (13)
C5—C6—C1	117.87 (12)	C18—C17—H17	120.5
C5—C6—C13	121.42 (12)	C16—C17—H17	120.5
C1—C6—C13	120.71 (12)	C17—C18—C19	123.04 (13)
C12—C7—C8	120.03 (14)	C17—C18—N2	118.09 (14)
C12—C7—H7	120.0	C19—C18—N2	118.87 (13)
C8—C7—H7	120.0	C18—C19—C20	117.71 (13)
C9—C8—C7	120.46 (15)	C18—C19—H19	121.1
C9—C8—H8	119.8	C20—C19—H19	121.1
C7—C8—H8	119.8	C21—C20—C19	120.63 (14)
C10—C9—C8	120.01 (14)	C21—C20—H20	119.7

C10—C9—H9	120.0	C19—C20—H20	119.7
C8—C9—H9	120.0	C20—C21—C16	120.99 (13)
C9—C10—C11	119.85 (14)	C20—C21—H21	119.5
C9—C10—H10	120.1	C16—C21—H21	119.5
C11—C10—H10	120.1		
C6—C1—C2—C3	-1.6 (2)	C7—C12—C13—C6	107.60 (13)
C1—C2—C3—C4	1.4 (2)	C11—C12—C13—C6	-69.96 (15)
C2—C3—C4—C5	-0.4 (2)	C6—C13—C14—C15	175.77 (12)
C3—C4—C5—C6	-0.4 (2)	C12—C13—C14—C15	-4.3 (2)
C4—C5—C6—C1	0.18 (18)	C16—N1—C15—C14	-179.44 (11)
C4—C5—C6—C13	-179.42 (11)	C13—C14—C15—N1	-177.81 (13)
C2—C1—C6—C5	0.8 (2)	C15—N1—C16—C21	45.24 (18)
C2—C1—C6—C13	-179.58 (13)	C15—N1—C16—C17	-139.32 (13)
C12—C7—C8—C9	-1.0 (2)	C21—C16—C17—C18	-1.14 (18)
C7—C8—C9—C10	0.1 (2)	N1—C16—C17—C18	-176.83 (11)
C8—C9—C10—C11	0.7 (2)	C16—C17—C18—C19	2.3 (2)
C9—C10—C11—C12	-0.7 (2)	C16—C17—C18—N2	-177.55 (12)
C8—C7—C12—C11	1.0 (2)	O1—N2—C18—C17	-1.1 (2)
C8—C7—C12—C13	-176.60 (13)	O2—N2—C18—C17	179.95 (14)
C10—C11—C12—C7	-0.2 (2)	O1—N2—C18—C19	179.04 (17)
C10—C11—C12—C13	177.46 (12)	O2—N2—C18—C19	0.1 (2)
C5—C6—C13—C14	-36.10 (17)	C17—C18—C19—C20	-1.6 (2)
C1—C6—C13—C14	144.31 (14)	N2—C18—C19—C20	178.26 (12)
C5—C6—C13—C12	143.96 (12)	C18—C19—C20—C21	-0.3 (2)
C1—C6—C13—C12	-35.63 (16)	C19—C20—C21—C16	1.4 (2)
C7—C12—C13—C14	-72.33 (17)	C17—C16—C21—C20	-0.66 (19)
C11—C12—C13—C14	110.11 (14)	N1—C16—C21—C20	174.72 (11)