Synthesis of α-Amino Acids via Asymmetric Phase Transfer-Catalyzed Alkylation of Achiral Nickel(II) Complexes of Glycine-Derived Schiff Bases

Belokon, Yuri N.; Bespalova, Natalia B.; Churkina, Tatiana D.; Císařová, Ivana; Ezernitskaya, Marina G.; Harutyunyan, Syuzanna; Hrdina, Radim; Kagan, Henri B.; Kočovský, Pavel; Kochetkov, Konstantin A.

Published in:
Journal of the American Chemical Society

Publication date:
2003

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Bruker (1998b) SMART. Bruker Molecular Analysis Research Tool, v. 5.059. Bruker AXS, Madison, Wisconsin, USA.

Sheldrick G.M. (1998a). SADABS v.2.01, Bruker/Siemens Area Detector Absorption Correction Program, Bruker AXS, Madison, Wisconsin, USA.

Sheldrick G.M. (1998b). SHELXTL v. 5.10, Structure Determination Software

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 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^ > 2sigma(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.
;

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 'calc w=1/[\s^2^(Fo^2^)+(0.0657P)^2^+0.0000P] where P=(Fo^2^+2Fc^2^)/3'
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O2 O 0.0245(3) 0.17384(10) 0.0785(2) 0.0355(6) Uani 1 1 d . A .
O3 O 0.3612(4) -0.09025(11) 0.5887(2) 0.0468(7) Uani 1 1 d . . .
N1 N 0.2779(6) -0.02191(14) 0.2844(3) 0.0684(12) Uani 1 1 d . . .
N2 N 0.2582(4) 0.00792(12) 0.5059(3) 0.0442(8) Uani 1 1 d . . .
N3 N 0.1588(4) 0.12644(12) 0.4084(3) 0.0311(7) Uani 1 1 d . . .
C1' C 0.2282(12) -0.0374(4) 0.1568(8) 0.0331(18) Uani 0.50 1 d P A 1
H1'A H 0.1584 -0.0101 0.0904 0.040 Uiso 0.50 1 calc PR A 1
C2' C 0.2834(13) -0.0951(4) 0.1266(9) 0.037(2) Uani 0.50 1 d P A 1
H2'A H 0.2580 -0.1065 0.0380 0.044 Uiso 0.50 1 calc PR A 1
C3' C 0.375(4) -0.1354(7) 0.225(3) 0.054(7) Uani 0.50 1 d P A 1
H3'A H 0.4324 -0.1708 0.2060 0.065 Uiso 0.50 1 calc PR A 1
C1" C 0.3369(13) -0.0250(4) 0.1715(8) 0.039(2) Uani 0.50 1 d P A 2
H1"A H 0.3464 0.0119 0.1268 0.047 Uiso 0.50 1 calc PR A 2
C2" C 0.3785(14) -0.0819(4) 0.1304(9) 0.041(2) Uani 0.50 1 d P A 2
H2"A H 0.3977 -0.0855 0.0497 0.050 Uiso 0.50 1 calc PR A 2
C3" C 0.392(4) -0.1350(11) 0.212(2) 0.061(8) Uani 0.50 1 d P A 2
H3"A H 0.4075 -0.1754 0.1824 0.074 Uiso 0.50 1 calc PR A 2
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C4 C 0.3810(5) -0.12496(17) 0.3423(4) 0.0414(10) Uani 1 1 d . . .
C5 C 0.3265(5) -0.06734(16) 0.3711(3) 0.0408(9) Uani 1 1 d . A .
C6 C 0.3187(5) -0.05134(15) 0.5027(3) 0.0338(8) Uani 1 1 d . A .
C7 C 0.2262(7) 0.03014(17) 0.6162(4) 0.0571(12) Uani 1 1 d . A .
C8 C 0.1587(5) 0.09181(15) 0.6191(3) 0.0395(9) Uani 1 1 d . . .
C9 C 0.1222(6) 0.11020(17) 0.7305(4) 0.0416(9) Uani 1 1 d . A .
C10" C 0.1048(18) 0.0727(9) 0.8221(17) 0.042(4) Uani 0.50 1 d P A 1
H10A H 0.0625 0.0881 0.8874 0.050 Uiso 0.50 1 calc PR A 1
C11" C 0.1488(12) 0.0121(4) 0.8202(7) 0.0350(18) Uani 0.50 1 d P A 1
H11A H 0.1494 -0.0148 0.8891 0.042 Uiso 0.50 1 calc PR A 1
C12" C 0.1935(12) -0.0103(3) 0.7153(8) 0.0318(19) Uani 0.50 1 d P A 1
H12A H 0.2034 -0.0539 0.7071 0.038 Uiso 0.50 1 calc PR A 1
C10' C 0.1843(17) 0.0739(8) 0.8474(17) 0.040(4) Uani 0.50 1 d P A 2
H10B H 0.1653 0.0877 0.9241 0.049 Uiso 0.50 1 calc PR A 2
C11' C 0.2754(13) 0.0166(4) 0.8475(8) 0.044(2) Uani 0.50 1 d P A 2
H11B H 0.3129 -0.0081 0.9243 0.052 Uiso 0.50 1 calc PR A 2
C12' C 0.3099(13) -0.0035(4) 0.7394(8) 0.038(2) Uani 0.50 1 d P A 2
H12B H 0.3855 -0.0381 0.7442 0.046 Uiso 0.50 1 calc PR A 2
C13 C 0.1327(4) 0.13796(14) 0.5168(3) 0.0293(8) Uani 1 1 d . A .
C14 C 0.0716(5) 0.20191(15) 0.5381(3) 0.0294(8) Uani 1 1 d . . .
C15 C -0.1085(5) 0.21682(17) 0.4829(3) 0.0342(8) Uani 1 1 d . A .
C16 C -0.1668(6) 0.27635(18) 0.4985(4) 0.0387(9) Uani 1 1 d . . .
C17 C -0.0467(6) 0.32039(17) 0.5708(4) 0.0396(9) Uani 1 1 d . A .
C18 C 0.1325(6) 0.30569(16) 0.6259(4) 0.0364(9) Uani 1 1 d . . .
C19 C 0.1913(6) 0.24673(15) 0.6094(3) 0.0331(9) Uani 1 1 d . A .
C20 C 0.1430(5) 0.17649(14) 0.3116(3) 0.0295(8) Uani 1 1 d . A .
C21 C 0.0944(5) 0.14495(15) 0.1791(3) 0.0310(8) Uani 1 1 d . . .
C22 C 0.3194(6) 0.21021(17) 0.3413(4) 0.0353(9) Uani 1 1 d . . .
H22C H 0.360(4) 0.2333(14) 0.425(3) 0.031(9) Uiso 1 1 d . . .
H22B H 0.417(5) 0.1805(17) 0.352(4) 0.049(11) Uiso 1 1 d . . .
H22A H 0.310(4) 0.2358(14) 0.277(3) 0.025(9) Uiso 1 1 d . . .
H9 H 0.079(4) 0.1522(17) 0.728(3) 0.040(10) Uiso 1 1 d . . .
H4 H 0.412(5) -0.1546(16) 0.396(3) 0.038(10) Uiso 1 1 d . . .
H15 H -0.183(5) 0.1883(15) 0.431(3) 0.032(10) Uiso 1 1 d . . .
H20 H 0.049(4) 0.2078(14) 0.310(3) 0.030(9) Uiso 1 1 d . . .
H11 H -0.276(4) 0.2852(14) 0.461(3) 0.019(9) Uiso 1 1 d . . .
H17 H -0.092(4) 0.3641(16) 0.582(3) 0.043(10) Uiso 1 1 d . . .
H19 H 0.304(4) 0.2384(14) 0.644(3) 0.024(10) Uiso 1 1 d . . .
H18 H 0.221(5) 0.3362(18) 0.680(4) 0.053(11) Uiso 1 1 d . . .

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O1 0.078(2) 0.0244(12) 0.0280(13) 0.0002(10) 0.0153(14) 0.0056(12)
O2 0.0405(16) 0.0369(13) 0.0276(13) 0.0065(11) 0.0102(12) 0.0067(11)
O3 0.0623(19) 0.0366(14) 0.0435(16) 0.0162(12) 0.0210(14) 0.0147(13)
N1 0.140(4) 0.0292(17) 0.0294(18) 0.0019(14) 0.021(2) 0.023(2)
N2 0.080(3) 0.0219(15) 0.0272(16) -0.0016(12) 0.0141(17) 0.0018(15)
N3 0.0418(19) 0.0246(14) 0.0245(15) 0.0024(11) 0.0085(14) 0.0009(12)
C1' 0.045(5) 0.025(4) 0.025(4) -0.004(3) 0.007(4) 0.006(4)
C2' 0.060(7) 0.025(4) 0.027(4) -0.006(3) 0.019(5) -0.012(4)
C3' 0.097(14) 0.008(6) 0.061(14) -0.003(6) 0.033(11) 0.015(6)
C1" 0.055(6) 0.022(4) 0.036(5) -0.002(3) 0.010(5) -0.006(4)
C2" 0.058(7) 0.032(5) 0.033(5) -0.001(4) 0.014(5) -0.001(5)
C3" 0.076(14) 0.067(13) 0.017(7) -0.016(6) -0.015(10) 0.019(9)
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C4 0.054(3) 0.028(2) 0.033(2) 0.0015(17) 0.002(2) 0.0019(18)
C5 0.053(3) 0.0289(18) 0.031(2) 0.0005(15) 0.0029(19) -0.0015(17)
C6 0.041(2) 0.0242(17) 0.0292(18) 0.0039(15) 0.0032(17) -0.0056(16)
C7 0.114(4) 0.0250(18) 0.032(2) 0.0026(16) 0.025(2) 0.002(2)
C8 0.059(3) 0.0285(17) 0.0296(19) -0.0010(15) 0.0141(19) -0.0068(17)
C9 0.064(3) 0.0265(19) 0.035(2) -0.0049(16) 0.018(2) -0.0088(18)
C10" 0.059(10) 0.037(5) 0.021(7) -0.013(5) 0.004(7) -0.017(8)
C11" 0.040(5) 0.038(4) 0.020(4) 0.009(3) 0.002(4) -0.005(4)
C12" 0.051(6) 0.018(3) 0.024(4) 0.002(3) 0.010(5) 0.001(4)
C10' 0.069(10) 0.025(4) 0.013(6) 0.000(4) -0.002(7) -0.018(8)
C11' 0.065(6) 0.037(4) 0.026(4) 0.003(3) 0.012(5) -0.001(5)
C12' 0.053(6) 0.030(4) 0.031(5) -0.001(3) 0.013(5) -0.003(4)
C13 0.035(2) 0.0275(17) 0.0229(17) -0.0013(13) 0.0066(16) -0.0054(15)
C14 0.038(2) 0.0304(17) 0.0213(17) 0.0029(14) 0.0118(17) 0.0011(15)
C15 0.040(2) 0.039(2) 0.0232(18) 0.0006(16) 0.0107(18) -0.0027(18)
C16 0.037(3) 0.052(2) 0.028(2) 0.0061(18) 0.014(2) 0.009(2)
C17 0.057(3) 0.034(2) 0.033(2) 0.0043(17) 0.023(2) 0.009(2)
C18 0.053(3) 0.0318(19) 0.0267(19) -0.0017(15) 0.0164(19) -0.0022(18)
C19 0.040(3) 0.0337(19) 0.0254(19) -0.0011(15) 0.0112(19) -0.0007(18)
C20 0.037(2) 0.0225(16) 0.0294(18) 0.0037(14) 0.0119(17) 0.0022(15)
C21 0.038(2) 0.0274(17) 0.0304(19) 0.0003(15) 0.0151(18) -0.0022(15)
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 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)
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Ni1 O1 1.857(2) . ?
Ni1 N2 1.874(3) . ?
Ni1 N1 1.881(3) . ?
O1 C21 1.299(4) . ?
O2 C21 1.217(4) . ?
O3 C6 1.217(4) . ?
N1 C5 1.323(4) . ?
N1 C1' 1.353(8) . ?
N1 C1" 1.469(9) . ?
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N3 C20 1.486(4) . ?
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C2' C3' 1.38(2) . ?
C3' C4 1.29(3) . ?
C1" C2" 1.386(11) . ?
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C3" C4 1.48(3) . ?
C4 C5 1.388(5) . ?
C5 C6 1.503(5) . ?
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C7 C8 1.439(5) . ?
C7 C12' 1.469(9) . ?
C7 C12" 1.483(8) . ?
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C8 C13 1.456(4) . ?
C9 C10" 1.33(2) . ?
C9 C10' 1.431(17) . ?
C10" C11" 1.35(2) . ?
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C10' C11' 1.43(2) . ?
C11' C12' 1.374(11) . ?
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C14 C19 1.392(5) . ?
C15 C16 1.396(5) . ?
C16 C17 1.385(5) . ?
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O1 Ni1 N1 90.68(12) . . ?
N2 Ni1 N1 86.09(13) . . ?
C21 O1 Ni1 115.2(2) . . ?
C5 N1 C1' 117.6(4) . . ?
C5 N1 C1" 118.0(4) . . ?
C1' N1 C1" 35.5(4) . . ?
C5 N1 Ni1 113.8(3) . . ?
C1' N1 Ni1 125.0(4) . . ?
C1" N1 Ni1 124.7(4) . . ?
C6 N2 C7 120.8(3) . . ?
C6 N2 Ni1 113.8(2) . . ?
C7 N2 Ni1 125.4(2) . . ?
C13 N3 C20 120.8(3) . . ?
C13 N3 Ni1 128.1(2) . . ?
C20 N3 Ni1 111.0(2) . . ?
N1 C1' C2' 117.4(7) . . ?
C3' C2' C1' 120.2(13) . . ?
C4 C3' C2' 119.6(18) . . ?
C2" C1" N1 119.7(7) . . ?
C1" C2" C3" 118.9(14) . . ?
C2" C3" C4 117.7(17) . . ?
C3' C4 C5 118.5(8) . . ?
C3' C4 C3" 6(3) . . ?
C5 C4 C3" 118.7(10) . . ?
N1 C5 C4 122.4(4) . . ?
N1 C5 C6 114.6(3) . . ?
C4 C5 C6 123.0(3) . . ?
O3 C6 N2 129.0(3) . . ?
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O3 C6 C5 119.2(3) . . ?
N2 C6 C5 111.8(3) . . ?
N2 C7 C8 121.1(3) . . ?
N2 C7 C12' 117.4(5) . . ?
C8 C7 C12' 119.3(4) . . ?
N2 C7 C12" 124.0(4) . . ?
C8 C7 C12" 111.4(4) . . ?
C12' C7 C12" 34.9(4) . . ?
C9 C8 C7 118.2(3) . . ?
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C10" C9 C10' 24.7(8) . . ?
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C11" C12" C7 123.7(6) . . ?
C9 C10' C11' 119.3(15) . . ?
C12' C11' C10' 121.6(10) . . ?
C11' C12' C7 118.1(8) . . ?
N3 C13 C8 123.1(3) . . ?
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C15 C14 C19 119.2(3) . . ?
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C18 C19 C14 120.7(4) . . ?
N3 C20 C22 110.7(3) . . ?
N3 C20 C21 106.5(2) . . ?
C22 C20 C21 109.1(3) . . ?
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Bruker (1998a) SAINTPlus Data Reduction and Correction Program v. 6.01, Bruker
AXS, Madison, Wisconsin, USA.

Bruker (1998b) SMART. Bruker Molecular Analysis Research Tool,
v. 5.059. Bruker AXS, Madison, Wisconsin, USA.

Sheldrick G.M. (1998a). SADABS v.2.01, Bruker/Siemens Area Detector
Absorption Correction Program, Bruker AXS, Madison, Wisconsin, USA.

Sheldrick G.M. (1998b). SHELXTL v. 5.10, Structure Determination Software
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 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^ > 2sigma(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
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C16 C 0.93460(14) 0.17267(14) 0.57133(11) 0.0478(5) Uani 1 1 d . . .
H16 H 0.8747 0.1931 0.5895 0.057 Uiso 1 1 calc R . .
C17 C 0.94104(13) 0.08856(14) 0.52926(11) 0.0434(4) Uani 1 1 d . . .
H17 H 0.8847 0.0552 0.5175 0.052 Uiso 1 1 calc R . .
C18 C 1.02933(12) 0.05495(12) 0.50524(9) 0.0357(4) Uani 1 1 d . . .
C19 C 1.11793(11) 0.10321(11) 0.52245(9) 0.0309(3) Uani 1 1 d . . .
C110 C 1.10843(13) 0.19227(12) 0.56077(9) 0.0361(4) Uani 1 1 d . . .
C111 C 0.99854(14) -0.11372(14) 0.48044(13) 0.0514(5) Uani 1 1 d . . .
C112 C 1.00962(17) -0.19056(17) 0.42173(17) 0.0735(8) Uani 1 1 d . . .
H11A H 1.0211 -0.2499 0.4467 0.110 Uiso 1 1 calc R . .
H11B H 1.0636 -0.1760 0.3891 0.110 Uiso 1 1 calc R . .
H11C H 0.9513 -0.1947 0.3919 0.110 Uiso 1 1 calc R . .
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O1 O 0.96137(13) -0.12884(10) 0.54246(10) 0.0716(5) Uani 1 1 d . . .
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C11' C 1.23668(11) -0.02581(11) 0.47267(9) 0.0309(3) Uani 1 1 d . . .
C12' C 1.25327(14) -0.10086(12) 0.52049(10) 0.0412(4) Uani 1 1 d . . .
C13' C 1.28042(17) -0.19063(13) 0.49160(11) 0.0525(5) Uani 1 1 d . . .
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C14' C 1.29136(15) -0.20477(13) 0.41602(12) 0.0499(5) Uani 1 1 d . . .
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C15' C 1.28652(15) -0.14255(15) 0.28424(11) 0.0498(5) Uani 1 1 d . . .
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C16' C 1.26913(16) -0.07056(16) 0.23540(11) 0.0537(5) Uani 1 1 d . . .
H16' H 1.2766 -0.0797 0.1833 0.064 Uiso 1 1 calc R . .
C17' C 1.23997(15) 0.01743(16) 0.26331(10) 0.0509(5) Uani 1 1 d . . .
H17' H 1.2273 0.0664 0.2294 0.061 Uiso 1 1 calc R . .
C18' C 1.22980(13) 0.03254(13) 0.33970(9) 0.0384(4) Uani 1 1 d . . .
H18' H 1.2107 0.0919 0.3570 0.046 Uiso 1 1 calc R . .
C19' C 1.24771(11) -0.04017(12) 0.39274(9) 0.0312(3) Uani 1 1 d . . .
C10' C 1.27512(13) -0.13088(13) 0.36407(10) 0.0392(4) Uani 1 1 d . . .
C21 C 0.80234(12) 0.03767(12) 0.25474(8) 0.0336(3) Uani 1 1 d . . .
C22 C 0.84684(15) 0.12335(15) 0.26730(11) 0.0493(5) Uani 1 1 d . . .
H22 H 0.8125 0.1704 0.2928 0.059 Uiso 1 1 calc R . .
C23 C 0.94181(16) 0.14242(19) 0.24320(13) 0.0641(6) Uani 1 1 d . . .
H23 H 0.9696 0.2012 0.2528 0.077 Uiso 1 1 calc R . .
C24 C 0.99279(15) 0.07582(19) 0.20618(13) 0.0631(6) Uani 1 1 d . . .
H24 H 1.0555 0.0894 0.1896 0.076 Uiso 1 1 calc R . .
C25 C 1.00548(17) -0.0841(2) 0.15069(14) 0.0702(7) Uani 1 1 d . . .
H25 H 1.0687 -0.0707 0.1352 0.084 Uiso 1 1 calc R . .
C26 C 0.9663(2) -0.1686(2) 0.13344(16) 0.0796(8) Uani 1 1 d . . .
H26 H 1.0014 -0.2119 0.1046 0.096 Uiso 1 1 calc R . .
C27 C 0.87237(19) -0.19141(16) 0.15891(14) 0.0674(6) Uani 1 1 d . . .
H27 H 0.8460 -0.2502 0.1472 0.081 Uiso 1 1 calc R . .
C28 C 0.81900(14) -0.12827(12) 0.20075(10) 0.0422(4) Uani 1 1 d . . .
C29 C 0.85563(12) -0.03583(12) 0.21672(9) 0.0359(4) Uani 1 1 d . . .
C210 C 0.95267(13) -0.01503(16) 0.19185(11) 0.0490(5) Uani 1 1 d . . .
C211 C 0.71329(16) -0.22135(12) 0.28435(11) 0.0464(4) Uani 1 1 d . . .
C212 C 0.61056(18) -0.23822(15) 0.30741(16) 0.0718(7) Uani 1 1 d . . .
H21A H 0.5952 -0.3040 0.3012 0.108 Uiso 1 1 calc R . .
H21B H 0.5681 -0.2009 0.2761 0.108 Uiso 1 1 calc R . .
H21C H 0.6022 -0.2207 0.3598 0.108 Uiso 1 1 calc R . .
C21' C 0.69756(11) 0.02886(11) 0.27652(9) 0.0312(3) Uani 1 1 d . . .
C22' C 0.62747(12) 0.04616(11) 0.22224(10) 0.0361(4) Uani 1 1 d . . .
C23' C 0.52746(13) 0.03792(13) 0.23968(12) 0.0447(4) Uani 1 1 d . . .
H23' H 0.4812 0.0483 0.2020 0.054 Uiso 1 1 calc R . .
C24' C 0.49889(13) 0.01488(13) 0.31119(12) 0.0491(5) Uani 1 1 d . . .
H24' H 0.4329 0.0112 0.3223 0.059 Uiso 1 1 calc R . .
C25' C 0.53865(17) -0.03205(14) 0.44328(12) 0.0546(5) Uani 1 1 d . . .
H25' H 0.4729 -0.0355 0.4554 0.066 Uiso 1 1 calc R . .
C26' C 0.60630(19) -0.05422(15) 0.49684(12) 0.0595(6) Uani 1 1 d . . .
H26' H 0.5865 -0.0728 0.5452 0.071 Uiso 1 1 calc R . .
C27' C 0.70563(18) -0.04924(14) 0.47972(11) 0.0544(5) Uani 1 1 d . . .
H27' H 0.7511 -0.0648 0.5168 0.065 Uiso 1 1 calc R . .
C28' C 0.73683(14) -0.02152(12) 0.40847(10) 0.0417(4) Uani 1 1 d . . .
H28' H 0.8030 -0.0186 0.3979 0.050 Uiso 1 1 calc R . .
C29' C 0.66853(12) 0.00239(11) 0.35147(9) 0.0335(3) Uani 1 1 d . . .
C20' C 0.56741(13) -0.00368(12) 0.36905(11) 0.0414(4) Uani 1 1 d . . .
N2 N 0.72633(13) -0.15798(10) 0.22759(9) 0.0432(4) Uani 1 1 d . . .
O2 O 0.78294(12) -0.26174(10) 0.31473(8) 0.0578(4) Uani 1 1 d . . .
O2' O 0.65783(10) 0.06703(10) 0.15028(7) 0.0456(3) Uani 1 1 d . . .
H1 H 1.0642(16) -0.0227(16) 0.4196(14) 0.057(6) Uiso 1 1 d . . .
H1' H 1.255(2) -0.140(2) 0.6236(16) 0.086(9) Uiso 1 1 d . . .
H2 H 0.6760(17) -0.1298(16) 0.2143(13) 0.053(6) Uiso 1 1 d . . .
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C17 0.0367(9) 0.0496(10) 0.0438(10) -0.0043(8) 0.0032(8) -0.0025(8)
C18 0.0393(9) 0.0373(9) 0.0304(8) -0.0045(7) 0.0030(7) -0.0025(7)
C19 0.0375(8) 0.0302(8) 0.0249(7) 0.0002(6) -0.0001(6) 0.0009(6)
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C111 0.0390(9) 0.0482(11) 0.0669(13) -0.0226(10) 0.0192(9) -0.0093(8)
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C26 0.0840(18) 0.0762(18) 0.0787(17) 0.0123(14) 0.0326(15) 0.0423(16)
C27 0.0864(17) 0.0444(11) 0.0713(15) 0.0058(11) 0.0170(13) 0.0217(12)
C28 0.0489(10) 0.0373(8) 0.0403(9) 0.0091(8) 0.0020(8) 0.0113(8)
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C29' 0.0401(8) 0.0264(7) 0.0340(8) -0.0063(6) 0.0057(7) -0.0024(7)
C20' 0.0442(9) 0.0316(8) 0.0484(10) -0.0052(8) 0.0148(8) -0.0001(7)
N2 0.0479(9) 0.0308(7) 0.0509(9) 0.0040(7) -0.0024(7) 0.0042(7)
O2 0.0808(10) 0.0486(8) 0.0441(7) 0.0138(6) 0.0038(7) 0.0108(8)
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 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.
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C112 H11C 0.9600 . ?
N1 H1 0.84(2) . ?
O1' C12' 1.353(2) . ?
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;
 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^ > 2sigma(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.
;

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 'calc w=1/[\s^2^(Fo^2^)+(0.0381P)^2^+0.2128P] where P=(Fo^2^+2Fc^2^)/3'
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_refine_ls_extinction_coef        ?
_refine_ls_abs_structure_details
 'Flack H D (1983), Acta Cryst. A39, 876-881'
_refine_ls_abs_structure_Flack    0.1(8)
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O1' O 0.78049(9) 0.57295(11) 0.48818(8) 0.0254(2) Uani 1 1 d . . .
O1 O 0.99651(8) 0.16545(13) 0.52696(8) 0.0293(2) Uani 1 1 d . . .
C1 C 0.57661(11) 0.39428(14) 0.48239(11) 0.0190(2) Uani 1 1 d . . .
C2 C 0.46221(12) 0.44554(16) 0.49214(12) 0.0253(3) Uani 1 1 d . . .
H2 H 0.4195 0.4969 0.4301 0.030 Uiso 1 1 calc R . .
C3 C 0.40756(13) 0.42329(17) 0.59205(13) 0.0293(3) Uani 1 1 d . . .
H3 H 0.3289 0.4560 0.5945 0.035 Uiso 1 1 calc R . .
C4 C 0.47008(13) 0.35376(16) 0.68517(13) 0.0280(3) Uani 1 1 d . . .
H4 H 0.4356 0.3444 0.7529 0.034 Uiso 1 1 calc R . .
C5 C 0.64770(13) 0.21654(17) 0.77719(11) 0.0268(3) Uani 1 1 d . . .
H5 H 0.6128 0.2107 0.8449 0.032 Uiso 1 1 calc R . .
C6 C 0.75562(13) 0.14981(16) 0.77206(11) 0.0280(3) Uani 1 1 d . . .
H6 H 0.7950 0.1001 0.8365 0.034 Uiso 1 1 calc R . .
C7 C 0.80840(12) 0.15552(16) 0.66945(11) 0.0247(3) Uani 1 1 d . . .
H7 H 0.8816 0.1073 0.6661 0.030 Uiso 1 1 calc R . .
C8 C 0.75323(11) 0.23138(14) 0.57416(10) 0.0189(2) Uani 1 1 d . . .
C9 C 0.64165(11) 0.30965(14) 0.57648(10) 0.0190(2) Uani 1 1 d . . .
C10 C 0.58721(12) 0.29533(15) 0.68045(11) 0.0231(3) Uani 1 1 d . . .
C11 C 0.91149(11) 0.18013(14) 0.44763(10) 0.0196(3) Uani 1 1 d . . .
C12 C 0.92925(11) 0.14986(14) 0.32215(10) 0.0182(2) Uani 1 1 d . . .
C13 C 0.96857(12) -0.01547(14) 0.31545(11) 0.0215(3) Uani 1 1 d . . .
H13A H 1.0403 -0.0333 0.3709 0.026 Uiso 1 1 calc R . .
H13B H 0.9048 -0.0804 0.3348 0.026 Uiso 1 1 calc R . .
C14 C 1.03048(12) 0.25216(15) 0.29009(12) 0.0243(3) Uani 1 1 d . . .
H14A H 1.0056 0.3559 0.2927 0.029 Uiso 1 1 calc R . .
H14B H 1.1029 0.2388 0.3455 0.029 Uiso 1 1 calc R . .
C15 C 0.81576(11) 0.17383(16) 0.23333(10) 0.0213(3) Uani 1 1 d . . .
H15A H 0.7511 0.1099 0.2523 0.026 Uiso 1 1 calc R . .
H15B H 0.7893 0.2769 0.2364 0.026 Uiso 1 1 calc R . .
C16 C 0.99532(13) -0.05115(16) 0.19305(11) 0.0271(3) Uani 1 1 d . . .
H16 H 1.0206 -0.1558 0.1897 0.033 Uiso 1 1 calc R . .
C17 C 1.09730(13) 0.05045(19) 0.16489(12) 0.0330(3) Uani 1 1 d . . .
H17A H 1.1171 0.0265 0.0887 0.040 Uiso 1 1 calc R . .
H17B H 1.1689 0.0345 0.2208 0.040 Uiso 1 1 calc R . .
C18 C 1.05761(13) 0.21392(18) 0.16818(12) 0.0308(3) Uani 1 1 d . . .
H18 H 1.1226 0.2787 0.1487 0.037 Uiso 1 1 calc R . .
C19 C 0.94359(14) 0.23923(19) 0.08076(12) 0.0334(3) Uani 1 1 d . . .
H19A H 0.9187 0.3430 0.0829 0.040 Uiso 1 1 calc R . .
H19B H 0.9605 0.2171 0.0032 0.040 Uiso 1 1 calc R . .
C20 C 0.84242(12) 0.13753(16) 0.11081(11) 0.0257(3) Uani 1 1 d . . .
H20 H 0.7694 0.1541 0.0553 0.031 Uiso 1 1 calc R . .
C21 C 0.88129(13) -0.02576(17) 0.10535(12) 0.0292(3) Uani 1 1 d . . .
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H21A H 0.8979 -0.0494 0.0280 0.035 Uiso 1 1 calc R . .
H21B H 0.8169 -0.0907 0.1232 0.035 Uiso 1 1 calc R . .
C1' C 0.62515(11) 0.44046(14) 0.37406(10) 0.0185(2) Uani 1 1 d . . .
C2' C 0.72444(11) 0.53466(14) 0.38144(11) 0.0200(3) Uani 1 1 d . . .
C3' C 0.76656(12) 0.59034(15) 0.28059(11) 0.0217(3) Uani 1 1 d . . .
H3' H 0.8351 0.6504 0.2872 0.026 Uiso 1 1 calc R . .
C4' C 0.70670(12) 0.55601(15) 0.17379(11) 0.0225(3) Uani 1 1 d . . .
H4' H 0.7341 0.5950 0.1083 0.027 Uiso 1 1 calc R . .
C5' C 0.54157(13) 0.42546(17) 0.05047(12) 0.0287(3) Uani 1 1 d . . .
H5' H 0.5672 0.4662 -0.0153 0.034 Uiso 1 1 calc R . .
C6' C 0.44436(14) 0.33100(18) 0.03906(13) 0.0341(3) Uani 1 1 d . . .
H6' H 0.4031 0.3095 -0.0339 0.041 Uiso 1 1 calc R . .
C7' C 0.40710(13) 0.26658(17) 0.13811(13) 0.0319(3) Uani 1 1 d . . .
H7' H 0.3426 0.1999 0.1298 0.038 Uiso 1 1 calc R . .
C8' C 0.46416(12) 0.30029(16) 0.24663(12) 0.0246(3) Uani 1 1 d . . .
H8' H 0.4378 0.2564 0.3109 0.030 Uiso 1 1 calc R . .
C9' C 0.56327(11) 0.40166(14) 0.26221(11) 0.0195(3) Uani 1 1 d . . .
C10' C 0.60363(12) 0.46201(14) 0.16088(11) 0.0212(3) Uani 1 1 d . . .
H1 H 0.7472(14) 0.2362(18) 0.4049(14) 0.025(4) Uiso 1 1 d . . .
H1' H 0.861(2) 0.615(3) 0.4818(18) 0.063(6) Uiso 1 1 d . . .

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O1' 0.0225(5) 0.0325(5) 0.0201(4) -0.0001(4) -0.0010(4) -0.0072(4)
O1 0.0200(5) 0.0447(6) 0.0220(5) -0.0064(4) -0.0011(4) 0.0073(4)
C1 0.0197(6) 0.0165(6) 0.0211(6) -0.0012(5) 0.0040(5) -0.0011(5)
C2 0.0245(7) 0.0238(6) 0.0283(7) 0.0018(5) 0.0060(5) 0.0035(5)
C3 0.0238(7) 0.0297(8) 0.0371(8) -0.0006(6) 0.0137(6) 0.0054(6)
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C5 0.0322(7) 0.0317(7) 0.0173(6) 0.0004(5) 0.0062(5) -0.0058(6)
C6 0.0316(7) 0.0318(8) 0.0196(6) 0.0056(6) 0.0004(5) -0.0014(6)
C7 0.0234(7) 0.0264(7) 0.0237(6) 0.0031(5) 0.0011(5) 0.0012(6)
C8 0.0193(6) 0.0196(6) 0.0179(5) 0.0002(5) 0.0031(5) -0.0036(5)
C9 0.0200(6) 0.0179(6) 0.0196(6) -0.0015(5) 0.0040(5) -0.0031(5)
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C11 0.0195(6) 0.0180(6) 0.0211(6) -0.0010(5) 0.0027(5) 0.0029(5)
C12 0.0173(6) 0.0185(6) 0.0190(6) -0.0008(5) 0.0033(4) 0.0022(5)
C13 0.0237(7) 0.0194(6) 0.0209(6) 0.0002(5) 0.0018(5) 0.0034(5)
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C16 0.0311(7) 0.0263(7) 0.0235(6) -0.0056(5) 0.0026(5) 0.0090(6)
C17 0.0259(7) 0.0496(9) 0.0246(7) -0.0035(6) 0.0078(5) 0.0075(7)
C18 0.0266(7) 0.0394(8) 0.0282(7) 0.0055(6) 0.0097(6) -0.0035(6)
C19 0.0365(8) 0.0414(9) 0.0236(7) 0.0101(6) 0.0087(6) 0.0026(7)
C20 0.0236(7) 0.0349(8) 0.0181(6) 0.0019(5) 0.0007(5) 0.0050(6)
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C2' 0.0193(6) 0.0195(6) 0.0208(6) 0.0005(5) 0.0010(5) 0.0010(5)
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C7' 0.0233(7) 0.0345(8) 0.0370(8) -0.0089(6) 0.0016(6) -0.0061(6)
C8' 0.0211(6) 0.0248(7) 0.0280(7) -0.0027(5) 0.0041(5) -0.0012(5)
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C9' 0.0171(6) 0.0179(6) 0.0230(6) -0.0004(5) 0.0012(5) 0.0033(5)
C10' 0.0199(6) 0.0207(6) 0.0223(6) 0.0007(5) 0.0007(5) 0.0041(5)

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 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.
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O1' H1' 0.98(2) . ?
O1 C11 1.2375(15) . ?
C1 C2 1.3774(18) . ?
C1 C9 1.4435(18) . ?
C1 C1' 1.5013(17) . ?
C2 C3 1.4036(19) . ?
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C3 C4 1.358(2) . ?
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C7 C8 1.3730(17) . ?
C7 H7 0.9300 . ?
C8 C9 1.4332(17) . ?
C9 C10 1.4367(17) . ?
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C17 C18 1.529(2) . ?
C17 H17A 0.9700 . ?
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C17 H17B 0.9700 . ?
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O1 C11 C12 120.60(11) . . ?
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H21A C21 H21B 108.3 . . ?
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O1' C2' C1' 118.23(11) . . ?
O1' C2' C3' 120.61(11) . . ?
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C6 C7 C8 C9 1.27(19) . . . . ?
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C2' C3' C4' C10' -1.5(2) . . . . ?
C10' C5' C6' C7' -1.4(2) . . . . ?
C5' C6' C7' C8' 2.1(2) . . . . ?
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
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 F^2^ > 2sigma(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
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C2 C 0.10327(17) 0.57887(16) 0.63638(10) 0.0283(3) Uani 1 1 d . . .
H2 H 0.1050 0.4825 0.6322 0.034 Uiso 1 1 calc R . .
C3 C -0.04767(19) 0.65042(18) 0.58434(11) 0.0344(3) Uani 1 1 d . . .
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C4 C -0.05303(19) 0.79032(18) 0.59040(11) 0.0340(3) Uani 1 1 d . . .
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C5 C 0.0789(2) 1.01276(17) 0.65292(12) 0.0385(4) Uani 1 1 d . . .
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C6 C 0.2151(2) 1.08913(17) 0.70469(13) 0.0437(4) Uani 1 1 d . . .
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C7 C 0.3732(2) 1.02382(16) 0.75400(13) 0.0373(4) Uani 1 1 d . . .
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C8 C 0.39148(18) 0.88193(14) 0.75257(10) 0.0258(3) Uani 1 1 d . . .
C9 C 0.24913(17) 0.79532(14) 0.69972(9) 0.0230(3) Uani 1 1 d . . .
C10 C 0.09189(18) 0.86653(16) 0.64741(10) 0.0296(3) Uani 1 1 d . . .
N1 N 0.55631(15) 0.82064(12) 0.79969(9) 0.0246(2) Uani 1 1 d . . .
O1 O 0.67043(15) 0.99034(11) 0.91183(8) 0.0359(3) Uani 1 1 d . . .
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C13 C 0.86439(18) 0.66090(14) 0.84686(11) 0.0274(3) Uani 1 1 d . . .
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H13B H 0.7702 0.5975 0.8501 0.041 Uiso 1 1 calc R . .
H13C H 0.9760 0.6158 0.8716 0.041 Uiso 1 1 calc R . .
C14 C 0.8472(2) 0.74311(16) 1.02264(11) 0.0336(3) Uani 1 1 d . . .
H14A H 0.7487 0.6823 1.0214 0.050 Uiso 1 1 calc R . .
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H14C H 0.9550 0.6955 1.0522 0.050 Uiso 1 1 calc R . .
C15 C 1.0111(2) 0.88132(17) 0.91482(13) 0.0367(3) Uani 1 1 d . . .
H15A H 1.1174 0.8274 0.9338 0.055 Uiso 1 1 calc R . .
H15B H 1.0127 0.9544 0.9637 0.055 Uiso 1 1 calc R . .
H15C H 1.0039 0.9207 0.8482 0.055 Uiso 1 1 calc R . .
O1' O 0.31974(13) 0.59998(11) 0.90372(7) 0.0278(2) Uani 1 1 d . . .
C1' C 0.39294(16) 0.54988(13) 0.74622(10) 0.0209(3) Uani 1 1 d . . .
C2' C 0.41306(16) 0.52184(13) 0.84944(10) 0.0217(3) Uani 1 1 d . . .
C3' C 0.52810(18) 0.41504(13) 0.89746(10) 0.0253(3) Uani 1 1 d . . .
H3' H 0.5395 0.3976 0.9668 0.030 Uiso 1 1 calc R . .
C4' C 0.62195(17) 0.33792(14) 0.84235(11) 0.0275(3) Uani 1 1 d . . .
H4' H 0.6951 0.2671 0.8744 0.033 Uiso 1 1 calc R . .
C5' C 0.71102(19) 0.28737(15) 0.67966(13) 0.0338(3) Uani 1 1 d . . .
H5' H 0.7876 0.2185 0.7117 0.041 Uiso 1 1 calc R . .
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C6' C 0.6977(2) 0.31317(17) 0.57820(13) 0.0368(4) Uani 1 1 d . . .
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C7' C 0.5811(2) 0.41632(17) 0.52894(12) 0.0341(3) Uani 1 1 d . . .
H7' H 0.5707 0.4325 0.4595 0.041 Uiso 1 1 calc R . .
C8' C 0.48243(18) 0.49329(15) 0.58221(10) 0.0279(3) Uani 1 1 d . . .
H8' H 0.4074 0.5619 0.5485 0.033 Uiso 1 1 calc R . .
C9' C 0.49286(17) 0.46999(13) 0.68792(10) 0.0231(3) Uani 1 1 d . . .
C10' C 0.60976(17) 0.36396(13) 0.73725(11) 0.0251(3) Uani 1 1 d . . .
H1 H 0.576(2) 0.744(2) 0.7802(12) 0.026(4) Uiso 1 1 d . . .
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C3 0.0243(7) 0.0509(10) 0.0266(7) 0.0014(6) 0.0025(5) -0.0016(6)
C4 0.0255(7) 0.0526(10) 0.0236(7) 0.0072(6) 0.0045(5) 0.0107(6)
C5 0.0435(9) 0.0394(9) 0.0324(8) 0.0107(7) 0.0078(6) 0.0199(7)
C6 0.0572(10) 0.0255(7) 0.0465(10) 0.0079(7) 0.0069(8) 0.0165(7)
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C8 0.0302(7) 0.0221(6) 0.0250(7) 0.0049(5) 0.0057(5) 0.0032(5)
C9 0.0247(6) 0.0262(7) 0.0192(6) 0.0051(5) 0.0075(5) 0.0060(5)
C10 0.0300(7) 0.0377(8) 0.0227(6) 0.0075(6) 0.0091(5) 0.0111(6)
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O1 0.0558(7) 0.0244(5) 0.0249(5) -0.0064(4) 0.0032(4) 0.0095(5)
C11 0.0330(7) 0.0193(6) 0.0178(6) 0.0003(5) 0.0093(5) -0.0005(5)
C12 0.0255(6) 0.0218(6) 0.0240(6) -0.0016(5) 0.0054(5) -0.0033(5)
C13 0.0242(6) 0.0245(7) 0.0338(7) -0.0066(5) 0.0070(5) 0.0001(5)
C14 0.0438(8) 0.0303(8) 0.0268(7) 0.0036(6) 0.0079(6) 0.0083(6)
C15 0.0347(7) 0.0311(7) 0.0438(8) -0.0067(7) 0.0071(6) -0.0118(6)
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C4' 0.0253(6) 0.0179(6) 0.0384(7) 0.0029(5) 0.0050(5) 0.0009(5)
C5' 0.0318(7) 0.0223(7) 0.0489(9) -0.0067(6) 0.0128(6) 0.0033(6)
C6' 0.0360(8) 0.0319(8) 0.0465(9) -0.0133(7) 0.0178(7) -0.0010(6)
C7' 0.0355(8) 0.0387(8) 0.0311(7) -0.0093(6) 0.0136(6) -0.0040(6)
C8' 0.0286(7) 0.0273(7) 0.0283(7) -0.0040(6) 0.0072(5) -0.0033(6)
C9' 0.0227(6) 0.0180(6) 0.0292(6) -0.0038(5) 0.0068(5) -0.0050(5)
C10' 0.0249(6) 0.0170(6) 0.0342(7) -0.0045(5) 0.0080(5) -0.0027(5)

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C2 H2 0.9300 . ?
C3 C4 1.351(2) . ?
C3 H3 0.9300 . ?
C4 C10 1.418(2) . ?
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C5 C6 1.348(3) . ?
C5 C10 1.415(2) . ?
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C6 H6 0.9300 . ?
C7 C8 1.375(2) . ?
C7 H7 0.9300 . ?
C8 N1 1.4242(17) . ?
C8 C9 1.440(2) . ?
C9 C10 1.4407(18) . ?
N1 C11 1.3516(18) . ?
N1 H1 0.812(19) . ?
O1 C11 1.2355(16) . ?
C11 C12 1.5292(18) . ?
C12 C13 1.5255(18) . ?
C12 C15 1.5324(19) . ?
C12 C14 1.5386(19) . ?
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C13 H13B 0.9600 . ?
C13 H13C 0.9600 . ?
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C14 H14C 0.9600 . ?
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C15 H15C 0.9600 . ?
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O1' H1' 0.86(2) . ?
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C4 C3 C2 119.50(15) . . ?
C4 C3 H3 120.3 . . ?
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C3 C4 H4 119.4 . . ?
C10 C4 H4 119.4 . . ?
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C6 C5 H5 119.6 . . ?
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C5 C6 C7 120.12(15) . . ?
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C8 C7 C6 121.31(15) . . ?
C8 C7 H7 119.3 . . ?
C6 C7 H7 119.3 . . ?
C7 C8 N1 119.42(13) . . ?
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N1 C8 C9 119.68(12) . . ?
C1 C9 C8 126.59(12) . . ?
C1 C9 C10 117.31(13) . . ?
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C4 C10 C9 120.19(13) . . ?
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C11 N1 H1 115.6(12) . . ?
C8 N1 H1 116.6(12) . . ?
O1 C11 N1 122.24(13) . . ?
O1 C11 C12 119.91(12) . . ?
N1 C11 C12 117.85(11) . . ?
C13 C12 C11 113.80(11) . . ?
C13 C12 C15 108.77(12) . . ?
C11 C12 C15 107.67(11) . . ?
C13 C12 C14 110.42(11) . . ?
C11 C12 C14 106.78(11) . . ?
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C12 C13 H13A 109.5 . . ?
C12 C13 H13B 109.5 . . ?
H13A C13 H13B 109.5 . . ?
C12 C13 H13C 109.5 . . ?
H13A C13 H13C 109.5 . . ?
H13B C13 H13C 109.5 . . ?
C12 C14 H14A 109.5 . . ?
C12 C14 H14B 109.5 . . ?
H14A C14 H14B 109.5 . . ?
C12 C14 H14C 109.5 . . ?
H14A C14 H14C 109.5 . . ?
H14B C14 H14C 109.5 . . ?
C12 C15 H15A 109.5 . . ?
C12 C15 H15B 109.5 . . ?
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H15A C15 H15C 109.5 . . ?
H15B C15 H15C 109.5 . . ?
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O1' C2' C3' 120.86(11) . . ?
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C2 C3 C4 C10 0.8(2) . . . . ?
C10 C5 C6 C7 0.2(3) . . . . ?
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C6 C7 C8 N1 177.30(14) . . . . ?
C6 C7 C8 C9 0.4(2) . . . . ?
C2 C1 C9 C8 -177.71(12) . . . . ?
C1' C1 C9 C8 4.1(2) . . . . ?
C2 C1 C9 C10 1.80(18) . . . . ?
C1' C1 C9 C10 -176.35(11) . . . . ?
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N1 C11 C12 C14 -110.90(13) . . . . ?
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C7' C8' C9' C1' 179.24(13) . . . . ?
C2' C1' C9' C8' -178.97(12) . . . . ?
C1 C1' C9' C8' 10.38(18) . . . . ?
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