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Mackay, S., Gilmore, C. J.,Edwards, C., Stewart, N. & Shankland, K.
(1999). maXus  Computer Program for the Solution and Refinement of
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Crystal Structures. Nonius, The Netherlands, MacScience, Japan & The University of Glasgow.

Otwinowski, Z. and Minor, W, (1997). In Methods in Enzymology, 276, edited by C.W. Carter, Jr. & R.M. Sweet pp. 307-326, New York:Academic Press.

Altomare, A., Cascarano, G., Giacovazzo G., Guagliardi A., Burla M.C., Polidori, G. & Camalli, M. (1994) SIR92 - a program for automatic solution of crystal structures by direct methods. J. Appl. Cryst. (27), 435-435

Coppens, P. (1970). In Crystallographic Computing, edited by F.R. Ahmed, S.R.Hall & C.P.Huber, pp 255-270. Copenhagen: Munksgaard.

Larson, A.C. (1970), Crystallographic Computing, Ed Ahmed, F.R., Munksgaard, Copenhagen, 291-294

Carruthers, J.R. and Watkin, D.J. (1979), Acta Cryst, A35, 698-699

Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. (1996) CRYSTALS Issue 10. Chemical Crystallography Laboratory, OXFORD, UK.

Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996) CAMERON, Chemical Crystallography Laboratory, OXFORD, UK.
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Mackay, S., Gilmore, C. J.,Edwards, C., Stewart, N. & Shankland, K. (1999). maXus Computer Program for the Solution and Refinement of Crystal Structures. Nonius, The Netherlands, MacScience, Japan & The University of Glasgow.

Altomare, A., Cascarano, G., Giacovazzo G., Guagliardi A., Burla M.C., Polidori, G. & Camalli, M. (1994) SIR92 - a program for automatic solution of
crystal structures by direct methods. J. Appl. Cryst. (27), 435-435

Carruthers, J.R. and Watkin, D.J. (1979),
Acta Cryst, A35, 698-699

Blessing, R.H. (1995), Acta. Cryst. A51, 33-38.

Watkin, D.J., Prout, C.K. Carruthers, J.R. & Betteridge, P.W. (1996) CRYSTALS
Issue 10. Chemical Crystallography Laboratory, OXFORD, UK.

Watkin, D.J., Prout, C.K. & Pearce, L.J. (1996) CAMERON, Chemical Crystallography Laboratory, OXFORD, UK.
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O1 0.0424(11) 0.0611(13) 0.0408(11) 0.002(1) 0.0061(9) 0.007(1)
O2 0.0397(11) 0.0396(11) 0.0666(14) -0.012(1) -0.0002(9) 0.0059(9)
N1 0.0356(12) 0.0310(12) 0.0425(13) -0.003(1) 0.004(1) -0.0020(9)
N2 0.0387(12) 0.0323(12) 0.0422(13) -0.002(1) 0.002(1) 0.001(1)
C1 0.064(2) 0.0491(19) 0.082(3) 0.0045(18) 0.037(2) 0.0029(17)
C2 0.0554(18) 0.0472(18) 0.057(2) 0.0127(15) 0.0091(16) 0.0036(15)
C3 0.056(2) 0.053(2) 0.074(2) 0.0031(18) 0.0051(18) -0.0205(17)
C4 0.0438(15) 0.0310(14) 0.0512(17) -0.0041(12) 0.0117(13) 0.0031(12)
C5 0.0436(17) 0.0483(18) 0.067(2) 0.0014(16) 0.0057(15) 0.0135(14)
C6 0.062(2) 0.0326(15) 0.068(2) -0.0035(15) 0.0184(17) 0.0043(14)
C7 0.066(2) 0.0402(17) 0.061(2) -0.0029(15) 0.0235(18) 0.0037(15)
C8 0.0410(15) 0.0430(16) 0.0463(16) 0.0009(13) -0.0063(13) -0.0055(13)
C9 0.060(2) 0.056(2) 0.062(2) -0.0029(17) -0.0151(17) -0.0157(17)
C10 0.0543(19) 0.059(2) 0.0516(19) 0.0092(16) -0.0116(15) -0.0053(16)
C11 0.0417(17) 0.062(2) 0.065(2) 0.0061(17) -0.0046(15) 0.0080(15)
C12 0.066(2) 0.0434(17) 0.057(2) -0.0015(15) 0.0189(17) -0.0136(16)
C13 0.0497(18) 0.0345(16) 0.081(3) 0.0067(16) 0.0058(18) 0.0021(14)
C14 0.0462(17) 0.0424(17) 0.066(2) 0.0004(15) -0.0035(15) -0.0132(14)
C15 0.0413(16) 0.068(2) 0.0482(18) 0.0061(16) 0.0032(14) 0.0066(15)
C16 0.058(2) 0.091(3) 0.076(2) 0.031(2) 0.0046(19) 0.013(2)
C17 0.061(2) 0.152(5) 0.059(3) 0.039(3) -0.0007(19) -0.007(3)
C18 0.071(2) 0.073(2) 0.050(2) 0.0015(18) 0.0211(17) 0.0010(19)
C19 0.0523(19) 0.0451(18) 0.081(3) -0.0232(18) 0.0039(18) -0.0016(15)
C20 0.069(2) 0.063(2) 0.062(2) -0.0229(19) -0.0040(17) 0.0191(18)
C21 0.0552(19) 0.080(2) 0.0490(19) 0.0103(17) 0.0125(16) 0.0258(18)
C22 0.0388(16) 0.0415(16) 0.068(2) 0.0033(15) -0.0002(14) 0.0054(13)

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P1 . N1 . 1.581(2)    yes
P1 . N2 . 1.580(2)    yes
P1 . C4 . 1.876(3)    yes
P1 . C8 . 1.881(3)    yes
Si1 . N1 . 1.672(2)    yes
Si1 . C1 . 1.869(3)    yes
Si1 . C2 . 1.873(3)    yes
Si1 . C3 . 1.887(3)    yes
Si2 . N2 . 1.670(2)    yes
Si2 . C12 . 1.882(3)    yes
Si2 . C13 . 1.870(3)    yes
Si2 . C14 . 1.871(3)    yes
O1 . C15 . 1.436(4)    yes
O1 . C18 . 1.434(4)    yes
O2 . C19 . 1.450(4)    yes
O2 . C22 . 1.444(4)    yes
C1 . H11 . 1.000    no
C1 . H12 . 1.000    no
C1 . H13 . 1.000    no
C2 . H21 . 1.000    no
C2 . H22 . 1.000    no
C2 . H23 . 1.000    no
C3 . H31 . 1.000    no
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C3 . H32 . 1.000    no
C3 . H33 . 1.000    no
C4 . C5 . 1.537(4)    yes
C4 . C6 . 1.531(4)    yes
C4 . C7 . 1.530(4)    yes
C5 . H51 . 1.000    no
C5 . H52 . 1.000    no
C5 . H53 . 1.000    no
C6 . H61 . 1.000    no
C6 . H62 . 1.000    no
C6 . H63 . 1.000    no
C7 . H71 . 1.000    no
C7 . H72 . 1.000    no
C7 . H73 . 1.000    no
C8 . C9 . 1.538(4)    yes
C8 . C10 . 1.531(4)    yes
C8 . C11 . 1.532(4)    yes
C9 . H91 . 1.000    no
C9 . H92 . 1.000    no
C9 . H93 . 1.000    no
C10 . H101 . 1.000    no
C10 . H102 . 1.000    no
C10 . H103 . 1.000    no
C11 . H111 . 1.000    no
C11 . H112 . 1.000    no
C11 . H113 . 1.000    no
C12 . H121 . 1.000    no
C12 . H122 . 1.000    no
C12 . H123 . 1.000    no
C13 . H131 . 1.000    no
C13 . H132 . 1.000    no
C13 . H133 . 1.000    no
C14 . H141 . 1.000    no
C14 . H142 . 1.000    no
C14 . H143 . 1.000    no
C15 . C16 . 1.497(5)    yes
C15 . H151 . 1.000    no
C15 . H152 . 1.000    no
C16 . C17 . 1.504(6)    yes
C16 . H161 . 1.000    no
C16 . H162 . 1.000    no
C17 . C18 . 1.463(6)    yes
C17 . H171 . 1.000    no
C17 . H172 . 1.000    no
C18 . H181 . 1.000    no
C18 . H182 . 1.000    no
C19 . C20 . 1.479(5)    yes
C19 . H191 . 1.000    no
C19 . H192 . 1.000    no
C20 . C21 . 1.521(5)    yes
C20 . H201 . 1.000    no
C20 . H202 . 1.000    no
C21 . C22 . 1.502(5)    yes
C21 . H211 . 1.000    no
C21 . H212 . 1.000    no
C22 . H221 . 1.000    no
C22 . H222 . 1.000    no
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N1 . P1 . N2 . 109.00(12)    yes
N1 . P1 . C4 . 109.32(12)    yes
N2 . P1 . C4 . 108.93(12)    yes
N1 . P1 . C8 . 109.51(12)    yes
N2 . P1 . C8 . 109.56(12)    yes
C4 . P1 . C8 . 110.49(13)    yes
N1 . Si1 . C1 . 109.06(14)    yes
N1 . Si1 . C2 . 112.31(13)    yes
C1 . Si1 . C2 . 108.47(17)    yes
N1 . Si1 . C3 . 116.50(14)    yes
C1 . Si1 . C3 . 104.77(18)    yes
C2 . Si1 . C3 . 105.22(17)    yes
N2 . Si2 . C12 . 117.01(13)    yes
N2 . Si2 . C13 . 110.97(13)    yes
C12 . Si2 . C13 . 106.98(16)    yes
N2 . Si2 . C14 . 109.80(13)    yes
C12 . Si2 . C14 . 104.67(16)    yes
C13 . Si2 . C14 . 106.79(16)    yes
C15 . O1 . C18 . 106.8(2)    yes
C19 . O2 . C22 . 108.9(2)    yes
P1 . N1 . Si1 . 153.25(15)    yes
P1 . N2 . Si2 . 156.14(16)    yes
Si1 . C1 . H11 . 109.43(11)    no
Si1 . C1 . H12 . 109.58(13)    no
H11 . C1 . H12 . 109.476    no
Si1 . C1 . H13 . 109.40(13)    no
H11 . C1 . H13 . 109.476    no
H12 . C1 . H13 . 109.476    no
Si1 . C2 . H21 . 109.4(1)    no
Si1 . C2 . H22 . 109.64(11)    no
H21 . C2 . H22 . 109.475    no
Si1 . C2 . H23 . 109.40(11)    no
H21 . C2 . H23 . 109.475    no
H22 . C2 . H23 . 109.476    no
Si1 . C3 . H31 . 109.15(12)    no
Si1 . C3 . H32 . 109.59(12)    no
H31 . C3 . H32 . 109.476    no
Si1 . C3 . H33 . 109.66(12)    no
H31 . C3 . H33 . 109.475    no
H32 . C3 . H33 . 109.476    no
P1 . C4 . C5 . 104.87(19)    yes
P1 . C4 . C6 . 113.36(19)    yes
C5 . C4 . C6 . 107.7(2)    yes
P1 . C4 . C7 . 113.2(2)    yes
C5 . C4 . C7 . 108.3(2)    yes
C6 . C4 . C7 . 109.1(2)    yes
C4 . C5 . H51 . 109.06(15)    no
C4 . C5 . H52 . 110.14(16)    no
H51 . C5 . H52 . 109.476    no
C4 . C5 . H53 . 109.20(16)    no
H51 . C5 . H53 . 109.476    no
H52 . C5 . H53 . 109.476    no
C4 . C6 . H61 . 109.50(15)    no
C4 . C6 . H62 . 109.58(17)    no
H61 . C6 . H62 . 109.476    no
C4 . C6 . H63 . 109.32(17)    no
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H61 . C6 . H63 . 109.475    no
H62 . C6 . H63 . 109.476    no
C4 . C7 . H71 . 108.99(16)    no
C4 . C7 . H72 . 109.76(16)    no
H71 . C7 . H72 . 109.476    no
C4 . C7 . H73 . 109.65(17)    no
H71 . C7 . H73 . 109.475    no
H72 . C7 . H73 . 109.476    no
P1 . C8 . C9 . 113.7(2)    yes
P1 . C8 . C10 . 112.3(2)    yes
C9 . C8 . C10 . 108.5(3)    yes
P1 . C8 . C11 . 105.4(2)    yes
C9 . C8 . C11 . 109.0(3)    yes
C10 . C8 . C11 . 107.7(3)    yes
C8 . C9 . H91 . 109.90(17)    no
C8 . C9 . H92 . 109.17(18)    no
H91 . C9 . H92 . 109.476    no
C8 . C9 . H93 . 109.33(18)    no
H91 . C9 . H93 . 109.475    no
H92 . C9 . H93 . 109.476    no
C8 . C10 . H101 . 109.98(16)    no
C8 . C10 . H102 . 109.67(17)    no
H101 . C10 . H102 . 109.476    no
C8 . C10 . H103 . 108.74(18)    no
H101 . C10 . H103 . 109.475    no
H102 . C10 . H103 . 109.476    no
C8 . C11 . H111 . 109.97(16)    no
C8 . C11 . H112 . 108.87(17)    no
H111 . C11 . H112 . 109.476    no
C8 . C11 . H113 . 109.55(17)    no
H111 . C11 . H113 . 109.476    no
H112 . C11 . H113 . 109.475    no
Si2 . C12 . H121 . 109.5(1)    no
Si2 . C12 . H122 . 108.88(11)    no
H121 . C12 . H122 . 109.475    no
Si2 . C12 . H123 . 110.00(11)    no
H121 . C12 . H123 . 109.476    no
H122 . C12 . H123 . 109.477    no
Si2 . C13 . H131 . 109.3(1)    no
Si2 . C13 . H132 . 109.84(12)    no
H131 . C13 . H132 . 109.475    no
Si2 . C13 . H133 . 109.28(11)    no
H131 . C13 . H133 . 109.476    no
H132 . C13 . H133 . 109.476    no
Si2 . C14 . H141 . 109.8(1)    no
Si2 . C14 . H142 . 109.26(11)    no
H141 . C14 . H142 . 109.475    no
Si2 . C14 . H143 . 109.29(11)    no
H141 . C14 . H143 . 109.476    no
H142 . C14 . H143 . 109.475    no
O1 . C15 . C16 . 105.5(3)    yes
O1 . C15 . H151 . 109.93(17)    no
C16 . C15 . H151 . 110.2(2)    no
O1 . C15 . H152 . 110.69(16)    no
C16 . C15 . H152 . 111.0(2)    no
H151 . C15 . H152 . 109.466    no
C15 . C16 . C17 . 104.2(3)    yes
C15 . C16 . H161 . 111.1(2)    no
C17 . C16 . H161 . 110.1(2)    no
C15 . C16 . H162 . 110.3(2)    no
C17 . C16 . H162 . 111.5(3)    no
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H161 . C16 . H162 . 109.466 no
C16 . C17 . C18 . 106.2(3) yes
C16 . C17 . H171 . 110.9(3) no
C18 . C17 . H171 . 111.6(3) no
C16 . C17 . H172 . 109.5(3) no
C18 . C17 . H172 . 109.1(3) no
H171 . C17 . H172 . 109.467 no
O1 . C18 . C17 . 108.1(3) yes
O1 . C18 . H181 . 109.06(18) no
C17 . C18 . H181 . 108.8(3) no
O1 . C18 . H182 . 110.21(18) no
C17 . C18 . H182 . 111.2(2) no
H181 . C18 . H182 . 109.467 no
O2 . C19 . C20 . 106.6(3) yes
O2 . C19 . H191 . 110.64(19) no
C20 . C19 . H191 . 111.3(2) no
O2 . C19 . H192 . 109.21(17) no
C20 . C19 . H192 . 109.6(2) no
H191 . C19 . H192 . 109.466 no
C19 . C20 . C21 . 103.2(3) yes
C19 . C20 . H201 . 110.4(2) no
C21 . C20 . H201 . 110.81(18) no
C19 . C20 . H202 . 111.9(2) no
C21 . C20 . H202 . 110.9(2) no
H201 . C20 . H202 . 109.468 no
C20 . C21 . C22 . 101.7(3) yes
C20 . C21 . H211 . 111.2(2) no
C22 . C21 . H211 . 110.65(19) no
C20 . C21 . H212 . 111.25(19) no
C22 . C21 . H212 . 112.42(18) no
H211 . C21 . H212 . 109.466 no
O2 . C22 . C21 . 104.8(3) yes
O2 . C22 . H221 . 110.94(15) no
C21 . C22 . H221 . 110.83(19) no
O2 . C22 . H222 . 110.56(16) no
C21 . C22 . H222 . 110.15(19) no
H221 . C22 . H222 . 109.467 no