Efficient and Mild Microwave-Assisted Stepwise Functionalization of Naphthalenediimide with α-Amino Acids

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2006

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 HKL Denzo and Scalepack (Otwinowski & Minor 1997)
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 Refinement of F^2^ against ALL reflections.  The weighted R-factor wR and
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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
 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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O2 O 0.28978(16) 1.14537(18) 0.11171(11) 0.0256(3) Uani 1 1 d . . .
O3 O -0.75630(15) 1.41096(16) 0.42614(11) 0.0222(2) Uani 1 1 d . . .
O4 O -0.41955(17) 1.74795(16) 0.42687(11) 0.0234(2) Uani 1 1 d . . .
O1' O 0.07809(17) 0.96158(16) -0.10476(10) 0.0246(2) Uani 1 1 d . . .
O2' O 0.24187(18) 0.67580(17) -0.09467(11) 0.0273(3) Uani 1 1 d . . .
O1" O -0.67584(16) 1.48645(16) 0.64718(10) 0.0220(2) Uani 1 1 d . . .
O2" O -0.96528(16) 1.62916(16) 0.62053(11) 0.0233(2) Uani 1 1 d . . .
C1 C -0.3437(2) 1.03980(19) 0.23768(12) 0.0168(3) Uani 1 1 d . . .
H1 H -0.3572 0.9384 0.2167 0.020 Uiso 1 1 calc R . .
C2 C -0.18521(19) 1.06911(18) 0.20732(12) 0.0141(3) Uani 1 1 d . . .
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C3 C -0.16309(19) 1.21931(19) 0.23730(12) 0.0133(2) Uani 1 1 d . . .
C4 C -0.00191(19) 1.25159(19) 0.20574(12) 0.0145(3) Uani 1 1 d . . .
C5 C 0.0159(2) 1.3995(2) 0.23609(13) 0.0177(3) Uani 1 1 d . . .
H5 H 0.1241 1.4209 0.2144 0.021 Uiso 1 1 calc R . .
C6 C -0.03793(19) 0.94197(19) 0.14082(12) 0.0153(3) Uani 1 1 d . . .
C7 C 0.1471(2) 1.12636(19) 0.13912(12) 0.0160(3) Uani 1 1 d . . .
C8 C -0.48521(19) 1.16046(19) 0.29976(12) 0.0167(3) Uani 1 1 d . . .
H8 H -0.5943 1.1405 0.3206 0.020 Uiso 1 1 calc R . .
C9 C -0.46567(19) 1.30785(18) 0.33046(12) 0.0146(3) Uani 1 1 d . . .
C10 C -0.30508(19) 1.34027(18) 0.29991(11) 0.0131(2) Uani 1 1 d . . .
C11 C -0.28322(19) 1.49006(18) 0.33079(12) 0.0142(3) Uani 1 1 d . . .
C12 C -0.1250(2) 1.5191(2) 0.29892(13) 0.0171(3) Uani 1 1 d . . .
H12 H -0.1113 1.6205 0.3196 0.020 Uiso 1 1 calc R . .
C13 C -0.61401(19) 1.43127(19) 0.39748(12) 0.0154(3) Uani 1 1 d . . .
C14 C -0.43002(19) 1.61549(19) 0.39854(12) 0.0150(3) Uani 1 1 d . . .
C1' C 0.1923(2) 0.8167(2) -0.05835(13) 0.0178(3) Uani 1 1 d . . .
C2' C 0.26307(19) 0.85540(19) 0.04151(12) 0.0160(3) Uani 1 1 d . . .
H2' H 0.3511 0.9134 0.0117 0.019 Uiso 1 1 calc R . .
C3' C 0.3629(2) 0.6887(2) 0.11125(13) 0.0203(3) Uani 1 1 d . . .
H3'A H 0.3885 0.7222 0.1805 0.024 Uiso 1 1 calc R . .
H3'B H 0.2840 0.6196 0.1324 0.024 Uiso 1 1 calc R . .
C4' C 0.5365(2) 0.5749(2) 0.05188(12) 0.0178(3) Uani 1 1 d . . .
C5' C 0.6833(2) 0.6292(2) 0.03259(13) 0.0200(3) Uani 1 1 d . . .
H5' H 0.6736 0.7381 0.0564 0.024 Uiso 1 1 calc R . .
C6' C 0.8445(2) 0.5248(2) -0.02136(14) 0.0226(3) Uani 1 1 d . . .
H6' H 0.9438 0.5629 -0.0342 0.027 Uiso 1 1 calc R . .
C7' C 0.8596(2) 0.3655(2) -0.05625(14) 0.0239(3) Uani 1 1 d . . .
H7' H 0.9688 0.2947 -0.0933 0.029 Uiso 1 1 calc R . .
C8' C 0.7136(2) 0.3097(2) -0.03667(14) 0.0231(3) Uani 1 1 d . . .
H8' H 0.7237 0.2007 -0.0603 0.028 Uiso 1 1 calc R . .
C9' C 0.5528(2) 0.4140(2) 0.01761(14) 0.0213(3) Uani 1 1 d . . .
H9' H 0.4541 0.3752 0.0312 0.026 Uiso 1 1 calc R . .
C10' C 0.0097(3) 0.9364(3) -0.20209(17) 0.0363(4) Uani 1 1 d . . .
H10A H -0.0805 1.0463 -0.2271 0.054 Uiso 1 1 calc R . .
H10B H -0.0445 0.8465 -0.1838 0.054 Uiso 1 1 calc R . .
H10C H 0.1077 0.8994 -0.2614 0.054 Uiso 1 1 calc R . .
C1" C -0.8087(2) 1.60037(19) 0.59320(12) 0.0173(3) Uani 1 1 d . . .
C2" C -0.73422(19) 1.69877(19) 0.49608(12) 0.0158(3) Uani 1 1 d . . .
H2" H -0.6836 1.7738 0.5288 0.019 Uiso 1 1 calc R . .
C3" C -0.8786(2) 1.8206(2) 0.42452(13) 0.0193(3) Uani 1 1 d . . .
H3"A H -0.8202 1.8617 0.3556 0.023 Uiso 1 1 calc R . .
H3"B H -0.9473 1.7538 0.4030 0.023 Uiso 1 1 calc R . .
C4" C -1.0068(2) 1.97786(19) 0.48290(12) 0.0167(3) Uani 1 1 d . . .
C5" C -0.9503(2) 2.1121(2) 0.50094(13) 0.0197(3) Uani 1 1 d . . .
H5" H -0.8309 2.1045 0.4768 0.024 Uiso 1 1 calc R . .
C6" C -1.0674(2) 2.2579(2) 0.55413(14) 0.0224(3) Uani 1 1 d . . .
H6" H -1.0278 2.3490 0.5660 0.027 Uiso 1 1 calc R . .
C7" C -1.2418(2) 2.2692(2) 0.58955(14) 0.0227(3) Uani 1 1 d . . .
H7" H -1.3215 2.3674 0.6266 0.027 Uiso 1 1 calc R . .
C8" C -1.2998(2) 2.1364(2) 0.57076(14) 0.0215(3) Uani 1 1 d . . .
H8" H -1.4195 2.1448 0.5943 0.026 Uiso 1 1 calc R . .
C9" C -1.1827(2) 1.9910(2) 0.51745(13) 0.0200(3) Uani 1 1 d . . .
H9" H -1.2229 1.9008 0.5047 0.024 Uiso 1 1 calc R . .
C10" C -0.7304(3) 1.3847(2) 0.74185(15) 0.0280(3) Uani 1 1 d . . .
H10D H -0.6246 1.3052 0.7760 0.042 Uiso 1 1 calc R . .
H10E H -0.7935 1.3169 0.7168 0.042 Uiso 1 1 calc R . .
H10F H -0.8097 1.4635 0.7965 0.042 Uiso 1 1 calc R . .
N3 N -1.4736(4) 1.9749(3) 0.8260(2) 0.0569(7) Uani 1 1 d . . .
C15 C -1.3820(3) 1.6863(3) 0.72900(17) 0.0349(4) Uani 1 1 d . . .
H15A H -1.4213 1.7115 0.6543 0.052 Uiso 1 1 calc R . .
H15B H -1.4377 1.6086 0.7731 0.052 Uiso 1 1 calc R . .
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H15C H -1.2519 1.6297 0.7236 0.052 Uiso 1 1 calc R . .
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O1 0.0207(5) 0.0183(5) 0.0275(6) -0.0090(4) 0.0038(4) -0.0089(4)
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O3 0.0155(5) 0.0253(6) 0.0284(6) -0.0107(5) 0.0050(4) -0.0101(4)
O4 0.0233(6) 0.0194(5) 0.0308(6) -0.0117(4) 0.0059(4) -0.0114(4)
O1' 0.0284(6) 0.0209(5) 0.0181(5) -0.0033(4) -0.0066(4) 0.0011(5)
O2' 0.0272(6) 0.0212(5) 0.0304(6) -0.0118(5) -0.0083(5) 0.0003(5)
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O2" 0.0167(5) 0.0223(5) 0.0276(6) -0.0027(4) 0.0045(4) -0.0051(4)
C1 0.0161(6) 0.0168(6) 0.0193(7) -0.0052(5) 0.0010(5) -0.0078(5)
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C3 0.0122(6) 0.0145(6) 0.0134(6) -0.0018(5) -0.0006(4) -0.0050(5)
C4 0.0125(6) 0.0169(6) 0.0142(6) -0.0025(5) 0.0006(5) -0.0055(5)
C5 0.0157(6) 0.0201(7) 0.0194(7) -0.0044(5) 0.0017(5) -0.0092(5)
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C8' 0.0262(8) 0.0154(6) 0.0241(7) -0.0020(5) -0.0024(6) -0.0030(6)
C9' 0.0219(7) 0.0181(7) 0.0226(7) 0.0005(5) -0.0044(6) -0.0055(6)
C10' 0.0405(11) 0.0367(10) 0.0239(8) -0.0068(7) -0.0153(7) 0.0012(8)
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C8" 0.0156(6) 0.0223(7) 0.0218(7) 0.0018(6) -0.0001(5) -0.0026(6)
C9" 0.0174(6) 0.0182(7) 0.0236(7) 0.0012(5) -0.0024(5) -0.0059(5)
C10" 0.0362(9) 0.0226(7) 0.0207(7) 0.0013(6) 0.0020(6) -0.0079(7)
N3 0.0750(17) 0.0311(10) 0.0453(12) 0.0053(8) 0.0185(11) -0.0076(10)
C15 0.0278(9) 0.0461(11) 0.0288(9) -0.0039(8) -0.0001(7) -0.0114(8)
C16 0.0346(10) 0.0345(10) 0.0293(9) 0.0076(7) 0.0052(7) -0.0042(8)

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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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N2 C14 1.4020(18) . ?
N2 C2" 1.4753(18) . ?
O1 C6 1.2174(19) . ?
O2 C7 1.2153(19) . ?
O3 C13 1.2194(19) . ?
O4 C14 1.2197(19) . ?
O1' C1' 1.3344(19) . ?
O1' C10' 1.444(2) . ?
O2' C1' 1.2018(19) . ?
O1" C1" 1.3359(19) . ?
O1" C10" 1.456(2) . ?
O2" C1" 1.2050(19) . ?
C1 C2 1.3811(19) . ?
C1 C8 1.4086(19) . ?
C1 H1 0.9500 . ?
C2 C3 1.4108(18) . ?
C2 C6 1.4839(19) . ?
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C4 C5 1.378(2) . ?
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C5 C12 1.407(2) . ?
C5 H5 0.9500 . ?
C8 C9 1.3821(19) . ?
C8 H8 0.9500 . ?
C9 C10 1.4102(18) . ?
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C10 C11 1.4109(18) . ?
C11 C12 1.3801(19) . ?
C11 C14 1.4834(19) . ?
C12 H12 0.9500 . ?
C1' C2' 1.528(2) . ?
C2' C3' 1.538(2) . ?
C2' H2' 1.0000 . ?
C3' C4' 1.515(2) . ?
C3' H3'A 0.9900 . ?
C3' H3'B 0.9900 . ?
C4' C9' 1.395(2) . ?
C4' C5' 1.396(2) . ?
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C5' H5' 0.9500 . ?
C6' C7' 1.388(2) . ?
C6' H6' 0.9500 . ?
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C7' C8' 1.397(3) . ?
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C8' C9' 1.397(2) . ?
C8' H8' 0.9500 . ?
C9' H9' 0.9500 . ?
C10' H10A 0.9800 . ?
C10' H10B 0.9800 . ?
C10' H10C 0.9800 . ?
C1" C2" 1.531(2) . ?
C2" C3" 1.534(2) . ?
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C3" C4" 1.517(2) . ?
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C3" H3"B 0.9900 . ?
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C6" C7" 1.388(2) . ?
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C10" H10D 0.9800 . ?
C10" H10E 0.9800 . ?
C10" H10F 0.9800 . ?
N3 C16 1.151(3) . ?
C15 C16 1.451(3) . ?
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C13 N2 C14 124.98(12) . . ?
C13 N2 C2" 117.02(12) . . ?
C14 N2 C2" 117.98(12) . . ?
C1' O1' C10' 114.66(13) . . ?
C1" O1" C10" 115.30(13) . . ?
C2 C1 C8 119.88(13) . . ?
C2 C1 H1 120.1 . . ?
C8 C1 H1 120.1 . . ?
C1 C2 C3 120.82(13) . . ?
C1 C2 C6 119.32(12) . . ?
C3 C2 C6 119.86(12) . . ?
C2 C3 C10 119.30(11) . . ?
C2 C3 C4 121.32(12) . . ?
C10 C3 C4 119.38(11) . . ?
C5 C4 C3 120.07(13) . . ?
C5 C4 C7 120.15(13) . . ?
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C12 C5 H5 119.8 . . ?
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O1 C6 C2 122.86(13) . . ?
N1 C6 C2 117.05(12) . . ?
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O2 C7 C4 122.65(14) . . ?
N1 C7 C4 116.92(12) . . ?
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C9 C8 H8 119.9 . . ?
C1 C8 H8 119.9 . . ?
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C11 C12 C5 120.44(13) . . ?
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C5 C12 H12 119.8 . . ?
O3 C13 N2 119.89(13) . . ?
O3 C13 C9 122.87(13) . . ?
N2 C13 C9 117.24(12) . . ?
O4 C14 N2 120.40(13) . . ?
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N2 C14 C11 116.77(12) . . ?
O2' C1' O1' 124.24(15) . . ?
O2' C1' C2' 124.21(14) . . ?
O1' C1' C2' 111.38(13) . . ?
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N1 C2' C3' 111.85(12) . . ?
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C2' C3' H3'A 108.8 . . ?
C4' C3' H3'B 108.8 . . ?
C2' C3' H3'B 108.8 . . ?
H3'A C3' H3'B 107.7 . . ?
C9' C4' C5' 119.23(14) . . ?
C9' C4' C3' 120.67(14) . . ?
C5' C4' C3' 120.09(14) . . ?
C4' C5' C6' 120.61(15) . . ?
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C6' C7' H7' 120.1 . . ?
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C9' C8' H8' 119.9 . . ?
C7' C8' H8' 119.9 . . ?
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O1' C10' H10B 109.5 . . ?
H10A C10' H10B 109.5 . . ?
O1' C10' H10C 109.5 . . ?
H10A C10' H10C 109.5 . . ?
H10B C10' H10C 109.5 . . ?
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O2" C1" C2" 124.61(14) . . ?
O1" C1" C2" 110.19(12) . . ?
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N2 C2" C3" 111.80(12) . . ?
C1" C2" C3" 112.79(12) . . ?
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C1" C2" H2" 107.0 . . ?
C3" C2" H2" 107.0 . . ?
C4" C3" C2" 113.37(12) . . ?
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C2" C3" H3"B 108.9 . . ?
H3"A C3" H3"B 107.7 . . ?
C5" C4" C9" 119.29(14) . . ?
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C7" C8" H8" 119.9 . . ?
C9" C8" H8" 119.9 . . ?
C4" C9" C8" 120.14(14) . . ?
C4" C9" H9" 119.9 . . ?
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O1" C10" H10D 109.5 . . ?
O1" C10" H10E 109.5 . . ?
H10D C10" H10E 109.5 . . ?
O1" C10" H10F 109.5 . . ?
H10D C10" H10F 109.5 . . ?
H10E C10" H10F 109.5 . . ?
C16 C15 H15A 109.5 . . ?
C16 C15 H15B 109.5 . . ?
H15A C15 H15B 109.5 . . ?
C16 C15 H15C 109.5 . . ?
H15A C15 H15C 109.5 . . ?
H15B C15 H15C 109.5 . . ?
N3 C16 C15 178.8(3) . . ?

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C6 C2 C3 C10 179.46(12) . . . . ?
C1 C2 C3 C4 -179.38(14) . . . . ?
C6 C2 C3 C4 -0.4(2) . . . . ?
C2 C3 C4 C5 179.93(14) . . . . ?
C10 C3 C4 C5 0.1(2) . . . . ?
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C7 N1 C6 O1 -177.93(14) . . . . ?
C2' N1 C6 O1 1.4(2) . . . . ?
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C2' N1 C6 C2 -178.76(12) . . . . ?
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C10 C11 C12 C5 -0.4(2) . . . . ?
C14 C11 C12 C5 179.04(13) . . . . ?
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C14 N2 C13 O3 -178.28(14) . . . . ?
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C10 C9 C13 O3 179.70(14) . . . . ?
C8 C9 C13 N2 178.39(13) . . . . ?
C10 C9 C13 N2 -0.7(2) . . . . ?
C13 N2 C14 O4 177.63(15) . . . . ?
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C12 C11 C14 O4 1.3(2) . . . . ?
C10 C11 C14 O4 -179.30(14) . . . . ?
C12 C11 C14 N2 -179.26(13) . . . . ?
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C10' O1' C1' O2' 2.5(3) . . . . ?
C10' O1' C1' C2' 178.08(16) . . . . ?
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C6 N1 C2' C3' -75.84(17) . . . . ?
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O1' C1' C2' N1 41.94(17) . . . . ?
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N1 C2' C3' C4' -167.25(13) . . . . ?
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C14 N2 C2" C1" -133.52(13) . . . . ?
C13 N2 C2" C3" -78.44(17) . . . . ?
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N2 C2" C3" C4" -164.53(12) . . . . ?
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