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Dr Alison Green
Australian Journal of Chemistry – an International Journal for Chemical Science
CSIRO PUBLISHING
PO Box 1139 (150 Oxford St)
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Telephone: +61 3 9662 7630
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    University of Sydney

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Vivienne P. Munk
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
S. Tsuey Cham
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
Ronald R. Fenton
;
  FIRST AUTHORS FOOTNOTES
;
```

 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
Rosalie K. Hocking
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
Trevor W. Hambley
;
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;
 Centre for Heavy Metals Research
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Molecular Structure Corporation. (1995). teXsan.
Single Crystal Structure Analysis Software. Version 1.7.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

de Meulenaer, J. & Tompa, H. (1965), Acta Cryst. 19, 1014.

Zachariasen, W. H. (1967). Acta Cryst. 23, 558-564.

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Sheldrick, G.M. (1985). In: "Crystallographic
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Vivienne P. Munk
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
S. Tsuey Cham
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
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;
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;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
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Rosalie K. Hocking
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
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Molecular Structure Corporation. (1995). teXsan.
Single Crystal Structure Analysis Software. Version 1.7.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

de Meulenaer, J. & Tompa, H. (1965), Acta Cryst. 19, 1014.

Sheldrick, G.M. (1985). In: "Crystallographic
Computing 3" (Eds G.M. Sheldrick, C. Kruger and
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;
  Cl 0     4 0.148 0.159
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Cl(1) 0.5678(4) 0.1798(4) -0.2044(4) 0.127(2) 1.000 . Uani d ?
O(1) 0.590(1) 0.050(1) -0.2272(10) 0.199(5) 1.000 . Uani d ?
O(2) 0.5811(10) 0.198(1) -0.0857(9) 0.179(5) 1.000 . Uani d ?
O(3) 0.4330(9) 0.2220(9) -0.2528(8) 0.170(4) 1.000 . Uani d ?
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N(1) 0.0743(8) -0.0142(7) -0.1428(6) 0.080(3) 1.000 . Uani d ?
```

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N(2) 0.1172(8) 0.1478(7) 0.0114(6) 0.080(3) 1.000 . Uani d ?
C(1) 0.009(1) 0.086(1) -0.2228(9) 0.106(5) 1.000 . Uani d ?
C(2) 0.065(1) 0.217(1) -0.186(1) 0.108(5) 1.000 . Uani d ?
C(3) 0.058(1) 0.255(1) -0.0636(9) 0.092(4) 1.000 . Uani d ?
C(4) 0.254(1) 0.099(1) -0.0183(9) 0.102(4) 1.000 . Uani d ?
C(5) 0.229(1) -0.001(1) -0.1152(8) 0.098(4) 1.000 . Uani d ?
C(6) 0.033(2) 0.060(1) -0.3435(10) 0.167(7) 1.000 . Uani d ?
C(7) 0.135(2) 0.375(2) -0.041(1) 0.088(7) 0.568  P Uiso d ?
C(7') -0.082(2) 0.285(2) -0.035(2) 0.069(8) 0.432  P Uiso d ?
H(1) 0.0538 -0.0961 -0.1742 0.099 1.000 . Uiso c ?
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H(4) 0.1604 0.2223 -0.1951 0.135 1.000 . Uiso c ?
H(5) 0.0135 0.2824 -0.2285 0.135 1.000 . Uiso c ?
H(6) 0.3068 0.1703 -0.0417 0.122 1.000 . Uiso c ?
H(7) 0.3024 0.0616 0.0476 0.122 1.000 . Uiso c ?
H(8) 0.2690 -0.0795 -0.0896 0.117 1.000 . Uiso c ?
H(9) 0.2704 0.0296 -0.1789 0.117 1.000 . Uiso c ?
H(10) -0.0055 -0.0247 -0.3657 0.198 1.000 . Uiso c ?
H(11) -0.0109 0.1210 -0.3943 0.198 1.000 . Uiso c ?
H(12) 0.1310 0.0564 -0.3512 0.198 1.000 . Uiso c ?
H(13) 0.2279 0.3683 -0.0580 0.104 0.620 . Uiso d ?
H(14) 0.0896 0.4458 -0.0835 0.104 0.620 . Uiso d ?
H(15) 0.1367 0.3995 0.0394 0.104 0.620 . Uiso d ?
H(16) -0.0828 0.3088 0.0425 0.076 0.380 . Uiso d ?
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 O(1)   0.23(1)     0.127(9)    0.24(1)     0.045(8)    0.017(9)    0.046(8)
 O(2)   0.182(9)    0.23(1)     0.126(7)    0.027(8)    0.041(6)    0.049(8)
 O(3)   0.122(7)    0.179(9)    0.207(10)   0.020(6)    0.004(6)    0.101(8)
 O(4)   0.110(6)    0.208(9)    0.138(7)    -0.024(6)   0.016(5)    0.072(7)
 N(1)   0.083(5)    0.066(5)    0.090(5)    -0.006(5)   0.009(4)    -0.003(5)
 N(2)   0.095(6)    0.073(6)    0.076(5)    0.000(5)    0.022(5)    -0.004(5)
 C(1)   0.128(10)   0.11(1)     0.078(8)    0.011(9)    0.013(7)    0.007(8)
 C(2)   0.112(9)    0.086(9)    0.12(1)     0.016(7)    -0.006(7)   0.039(8)
 C(3)   0.101(9)    0.084(8)    0.095(8)    0.008(7)    0.028(7)    0.005(7)
 C(4)   0.099(8)    0.098(9)    0.107(9)    -0.015(7)   0.005(7)    0.015(7)
 C(5)   0.104(8)    0.090(8)    0.104(8)    0.014(8)    0.034(6)    0.019(8)
 C(6)   0.24(2)     0.18(1)     0.088(9)    0.02(1)     0.030(9)    -0.005(9)
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 Ni(1) N(2) 1.900(7) . . yes
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 Cl(1) O(1) 1.40(1) . . yes
 Cl(1) O(2) 1.407(9) . . yes
 Cl(1) O(3) 1.424(7) . . yes
 Cl(1) O(4) 1.423(8) . . yes
 N(1) C(1) 1.50(1) . . yes
 N(1) C(5) 1.49(1) . . yes
 N(1) H(1) 0.94 . . no
 N(2) C(3) 1.50(1) . . yes
 N(2) C(4) 1.49(1) . . yes
 N(2) H(2) 0.95 . . no
 C(1) C(2) 1.50(2) . . yes
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 C(3) C(7) 1.46(2) . . yes
 C(3) C(7') 1.44(2) . . yes
 C(4) C(5) 1.54(1) . . yes
 C(4) H(6) 0.96 . . no
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```

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 C(5) H(8) 0.94 . . no
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 C(7) H(13) 0.94 . . no
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 N(1) Ni(1)  N(2) 98.7(3) 3 . . yes
 N(1) Ni(1)  N(2) 81.3(3) 3 . 3 yes
 N(2) Ni(1)  N(2) 180.00 . . 3 yes
 O(1) Cl(1)  O(2) 108.7(7) . . . yes
 O(1) Cl(1)  O(3) 111.5(7) . . . yes
 O(1) Cl(1)  O(4) 111.7(7) . . . yes
 O(2) Cl(1)  O(3) 109.9(6) . . . yes
 O(2) Cl(1)  O(4) 108.8(6) . . . yes
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 Ni(1) N(1)  C(1) 109.6(6) . . . yes
 Ni(1) N(1)  C(5) 104.9(5) . . . yes
 Ni(1) N(1)  H(1) 109.6 . . . no
 C(1) N(1)  C(5) 114.3(7) . . . yes
 C(1) N(1)  H(1) 109.1 . . . no
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 Ni(1) N(2)  C(3) 112.0(6) . . . yes
 Ni(1) N(2)  C(4) 103.8(6) . . . yes
 Ni(1) N(2)  H(2) 108.6 . . . no
 C(3) N(2)  C(4) 113.8(8) . . . yes
 C(3) N(2)  H(2) 108.3 . . . no
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 N(1) C(1)  C(6) 112(1) . . . yes
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 C(2) C(1)  C(6) 110(1) . . . yes
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 C(6) C(1)  H(3) 108.4 . . . no
 C(1) C(2)  C(3) 117.3(10) . . . yes
 C(1) C(2)  H(4) 110.5 . . . no
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```

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 N(2) C(3)  C(2) 108.8(8) . . . yes
 N(2) C(3)  C(7) 111.9(10) . . . yes
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 C(7) C(3)  C(7') 103(1) . . . yes
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Vivienne P. Munk
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
S. Tsuey Cham
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
Ronald R. Fenton
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
```

Rosalie K. Hocking
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
 School of Chemistry
 University of Sydney
;
Trevor W. Hambley
;
  FIRST AUTHORS FOOTNOTES
;
 Centre for Heavy Metals Research
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Molecular Structure Corporation. (1995). teXsan.
Single Crystal Structure Analysis Software. Version 1.7.
MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

de Meulenaer, J. & Tompa, H. (1965), Acta Cryst. 19, 1014.

Zachariasen, W. H. (1967). Acta Cryst. 23, 558-564.

Sheldrick, G.M. (1985). In: "Crystallographic
Computing 3" (Eds G.M. Sheldrick, C. Kruger and
R. Goddard) Oxford University Press, pp. 175-189.

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SHELXS86 (Sheldrick, 1985)
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analytical (de Meulenaer & Tompa, 1965)
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(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
;
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(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
;
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;
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;
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(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
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Cl(1) 0.0395(1) 0.60126(5) 0.2724(1) 0.0312(2) 1.000 . Uani d ?
N(1) 0.2385(4) 0.0460(2) 0.6290(4) 0.0244(8) 1.000 . Uani d ?
N(2) -0.0452(5) 0.1059(2) 0.3884(4) 0.0240(8) 1.000 . Uani d ?
C(1) 0.2085(5) 0.1227(2) 0.7376(5) 0.0258(9) 1.000 . Uani d ?
C(2) 0.1597(5) 0.1912(2) 0.6047(5) 0.0270(10) 1.000 . Uani d ?
C(3) -0.0520(5) 0.1793(2) 0.5102(5) 0.0257(9) 1.000 . Uani d ?
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C(6) 0.0390(6) 0.1132(2) 0.8806(6) 0.033(1) 1.000 . Uani d ?
```

```
H(1) 0.303(5) 0.010(2) 0.701(5) 0.04(1) 1.000 . Uiso d ?
H(2) 0.331(6) 0.057(2) 0.525(5) 0.05(1) 1.000 . Uiso d ?
H(3) 0.064(6) 0.106(2) 0.308(5) 0.05(1) 1.000 . Uiso d ?
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H(7) 0.267(5) 0.196(2) 0.509(5) 0.027(9) 1.000 . Uiso d ?
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H(11) -0.283(6) 0.111(2) 0.896(5) 0.05(1) 1.000 . Uiso d ?
H(12) -0.208(5) 0.054(2) 0.751(5) 0.026(9) 1.000 . Uiso d ?
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_atom_site_aniso_U_33
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 Cl(1)  0.0261(4)   0.0327(5)   0.0348(5)   -0.0016(3)  -0.0006(4)  -0.0037(4)
 N(1)   0.021(1)    0.020(1)    0.032(2)    0.002(1)    -0.003(1)   0.003(1)
 N(2)   0.023(2)    0.024(1)    0.025(2)    0.001(1)    -0.002(1)   0.000(1)
 C(1)   0.022(2)    0.025(2)    0.030(2)    -0.002(1)   -0.007(2)   -0.006(2)
 C(2)   0.025(2)    0.020(2)    0.036(2)    0.000(1)    -0.001(2)   -0.004(2)
 C(3)   0.027(2)    0.018(2)    0.033(2)    0.005(1)    -0.004(2)   0.000(2)
 C(4)   0.022(2)    0.039(2)    0.041(2)    0.005(2)    -0.002(2)   -0.014(2)
 C(5)   0.028(2)    0.041(2)    0.031(2)    -0.005(2)   0.007(2)    -0.009(2)
 C(6)   0.036(2)    0.034(2)    0.029(2)    -0.001(2)   -0.001(2)   -0.002(2)
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_refine_ls_wR_factor_all               0.0310
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 Ni(1) N(1) 1.932(3) . 3_556 yes
 Ni(1) N(2) 1.941(3) . . yes
 Ni(1) N(2) 1.941(3) . 3_556 yes
 N(1) C(1) 1.500(4) . . yes
 N(1) H(1) 0.89(4) . . no
 N(1) H(2) 0.98(4) . . no
 N(2) C(3) 1.496(4) . . yes
 N(2) H(3) 0.92(4) . . no
 N(2) H(4) 0.81(4) . . no
 C(1) C(2) 1.513(5) . . yes
 C(1) C(6) 1.515(5) . . yes
 C(1) H(5) 0.99(3) . . no
 C(2) C(3) 1.522(4) . . yes
 C(2) H(6) 0.89(4) . . no
 C(2) H(7) 0.98(3) . . no
 C(3) C(4) 1.503(5) . . yes
 C(3) H(8) 0.88(3) . . no
 C(4) C(5) 1.527(5) . . yes
 C(4) H(9) 0.93(3) . . no
 C(4) H(10) 0.90(4) . . no
 C(5) C(6) 1.531(5) . . yes
 C(5) H(11) 0.97(4) . . no
 C(5) H(12) 0.95(3) . . no
 C(6) H(13) 0.95(4) . . no
 C(6) H(14) 0.84(4) . . no
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 N(1) Ni(1)  N(1) 180.00 . . 3_556 yes
 N(1) Ni(1)  N(2) 87.9(1) . . . yes
 N(1) Ni(1)  N(2) 92.1(1) . . 3_556 yes
 N(1) Ni(1)  N(2) 92.1(1) 3_556 . . yes
 N(1) Ni(1)  N(2) 87.9(1) 3_556 . 3_556 yes
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```
 N(2) Ni(1)  N(2) 180.00 . . 3_556 yes
 Ni(1) N(1)  C(1) 118.4(2) . . . yes
 Ni(1) N(1)  H(1) 112(2) . . . no
 Ni(1) N(1)  H(2) 100(2) . . . no
 C(1) N(1)  H(1) 108(2) . . . no
 C(1) N(1)  H(2) 108(2) . . . no
 H(1) N(1)  H(2) 106(2) . . . no
 Ni(1) N(2)  C(3) 119.1(2) . . . yes
 Ni(1) N(2)  H(3) 99(2) . . . no
 Ni(1) N(2)  H(4) 110(2) . . . no
 C(3) N(2)  H(3) 113(2) . . . no
 C(3) N(2)  H(4) 108(2) . . . no
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 N(1) C(1)  H(5) 107(1) . . . no
 C(2) C(1)  C(6) 111.4(3) . . . yes
 C(2) C(1)  H(5) 110(1) . . . no
 C(6) C(1)  H(5) 107(1) . . . no
 C(1) C(2)  C(3) 111.4(3) . . . yes
 C(1) C(2)  H(6) 112(2) . . . no
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 C(3) C(2)  H(7) 108(1) . . . no
 H(6) C(2)  H(7) 109(2) . . . no
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 N(2) C(3)  C(4) 111.0(3) . . . yes
 N(2) C(3)  H(8) 108(2) . . . no
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 C(2) C(3)  H(8) 105(2) . . . no
 C(4) C(3)  H(8) 111(2) . . . no
 C(3) C(4)  C(5) 114.1(3) . . . yes
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 H(9) C(4)  H(10) 101(2) . . . no
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 C(6) C(5)  H(11) 109(2) . . . no
 C(6) C(5)  H(12) 110(1) . . . no
 H(11) C(5)  H(12) 102(2) . . . no
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 C(1) C(6)  H(13) 107(2) . . . no
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 C(5) C(6)  H(14) 104(2) . . . no
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```
 Cl(1)     N(1)      3.255(3)   . 4_454 no
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 Cl(1)     N(1)      3.372(3)   . 2 no
 Cl(1)     N(2)      3.452(3)   . 2 no
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 Ni(1) N(1) C(1) C(2) . 3_556 3_556 3_556 67.1(3) no
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 Ni(1) N(2) C(3) C(4) . . . . -59.0(3) no
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 C(2) C(3) C(4) C(5) . . . . -53.0(4) no
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