

CSIRO Publishing

AUSTRALIAN JOURNAL OF
CHEMISTRY
AN INTERNATIONAL JOURNAL FOR CHEMICAL SCIENCE

publishing research papers from all fields of chemical science, including synthesis, structure, new materials, macromolecules, supramolecular chemistry, biological chemistry, nanotechnology, surface chemistry, and analytical techniques.

Volume 55, 2002
© CSIRO 2002

All enquiries and manuscripts should be directed to:

Dr Alison Green
*Australian Journal of Chemistry –
an International Journal for Chemical Science*



CSIRO PUBLISHING
PO Box 1139 (150 Oxford St)
Collingwood, Vic. 3066, Australia

Telephone: +61 3 9662 7630
Fax: +61 3 9662 7611
E-mail: publishing.ajc@csiro.au

Published by CSIRO PUBLISHING
for CSIRO and the Australian Academy of Science

www.publish.csiro.au/journals/ajc

10.1071/CH02065_AC

© CSIRO 2002

Accessory Publication: *Aust. J. Chem.*, 2002, 55(5), 311-313.

data_Mndca2H2O2Me4pyz2

```
_audit_creation_method          SHELXL-97
_chemical_name_systematic
;
?
;
_chemical_name_common           ?
_chemical_melting_point         ?
_chemical_formula_moiety        ?
_chemical_formula_sum
  'C20 H28 Mn N10 O2'
_chemical_formula_weight        495.46

loop_
  _atom_type_symbol
  _atom_type_description
  _atom_type_scatter_dispersion_real
  _atom_type_scatter_dispersion_imag
  _atom_type_scatter_source
  'C'  'C'    0.0033  0.0016
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'H'  'H'    0.0000  0.0000
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'N'  'N'    0.0061  0.0033
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'O'  'O'    0.0106  0.0060
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'
  'Mn' 'Mn'   0.3368  0.7283
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting          Triclinic
_symmetry_space_group_name_H-M  P-1

loop_
  _symmetry_equiv_pos_as_xyz
  'x, y, z'
  '-x, -y, -z'

_cell_length_a                  7.7131(4)
_cell_length_b                  9.1907(6)
_cell_length_c                  10.4207(6)
_cell_angle_alpha               66.339(3)
_cell_angle_beta               80.242(3)
_cell_angle_gamma              66.184(3)
_cell_volume                    618.97(6)
_cell_formula_units_Z           1
_cell_measurement_temperature   123(2)
_cell_measurement_reflns_used   ?
_cell_measurement_theta_min     ?
_cell_measurement_theta_max     ?

_exptl_crystal_description      plate
```

```

_exptl_crystal_colour          colourless
_exptl_crystal_size_max       0.375
_exptl_crystal_size_mid       0.125
_exptl_crystal_size_min       0.025
_exptl_crystal_density_meas   ?
_exptl_crystal_density_diffn  1.329
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000          259
_exptl_absorpt_coefficient_mu 0.570
_exptl_absorpt_correction_type none
_exptl_absorpt_correction_T_min ?
_exptl_absorpt_correction_T_max ?
_exptl_absorpt_process_details ?

_exptl_special_details
;
?
;

_diffn_ambient_temperature    123(2)
_diffn_radiation_wavelength    0.71070
_diffn_radiation_type          MoK\alpha
_diffn_radiation_source        'fine-focus sealed tube'
_diffn_radiation_monochromator graphite
_diffn_measurement_device_type 'Nonius KappaCCD'
_diffn_measurement_method      'phi scans'
_diffn_detector_area_resol_mean ?
_diffn_standards_number        ?
_diffn_standards_interval_count ?
_diffn_standards_interval_time ?
_diffn_standards_decay_%       ?
_diffn_reflns_number           9078
_diffn_reflns_av_R_equivalents 0.0482
_diffn_reflns_av_sigmaI/netI   0.0612
_diffn_reflns_limit_h_min      -10
_diffn_reflns_limit_h_max      9
_diffn_reflns_limit_k_min      -12
_diffn_reflns_limit_k_max      12
_diffn_reflns_limit_l_min      -13
_diffn_reflns_limit_l_max      13
_diffn_reflns_theta_min        2.61
_diffn_reflns_theta_max        28.38
_reflns_number_total            2940
_reflns_number_gt              2486
_reflns_threshold_expression    >2sigma(I)

_computing_data_collection     ?
_computing_cell_refinement     ?
_computing_data_reduction      ?
_computing_structure_solution   'SHELXS-97 (Sheldrick, 1990)'
_computing_structure_refinement 'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics   ?
_computing_publication_material ?

_refine_special_details
;
Refinement of F2 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.

;

```

_refine_ls_structure_factor_coef  Fsqd
_refine_ls_matrix_type            full
_refine_ls_weighting_scheme       calc
_refine_ls_weighting_details
'calc w=1/[\s^2*(Fo^2)+(0.0686P)^2+2.5037P] where P=(Fo^2+2Fc^2)/3'
_atom_sites_solution_primary      direct
_atom_sites_solution_secondary    difmap
_atom_sites_solution_hydrogens    geom
_refine_ls_hydrogen_treatment     mixed
_refine_ls_extinction_method      none
_refine_ls_extinction_coef        ?
_refine_ls_number_reflns          2940
_refine_ls_number_parameters       159
_refine_ls_number_restraints       0
_refine_ls_R_factor_all            0.0951
_refine_ls_R_factor_gt            0.0795
_refine_ls_wR_factor_ref          0.2187
_refine_ls_wR_factor_gt           0.2116
_refine_ls_goodness_of_fit_ref     1.206
_refine_ls_restrained_S_all        1.206
_refine_ls_shift/su_max            0.000
_refine_ls_shift/su_mean           0.000

```

loop_

```

_atom_site_label
_atom_site_type_symbol
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_adp_type
_atom_site_occupancy
_atom_site_symmetry_multiplicity
_atom_site_calc_flag
_atom_site_refinement_flags
_atom_site_disorder_assembly
_atom_site_disorder_group
Mn1 Mn -0.5000 0.5000 0.5000 0.0198(3) Uani 1 2 d S . .
O1 O -0.5680(5) 0.5199(6) 0.7037(4) 0.0355(9) Uani 1 1 d . . .
H1 H -0.497(13) 0.527(11) 0.751(9) 0.08(3) Uiso 1 1 d . . .
H2 H -0.669(10) 0.536(8) 0.739(7) 0.048(19) Uiso 1 1 d . . .
N1 N -0.3604(5) 0.6889(5) 0.4385(4) 0.0279(9) Uani 1 1 d . . .
C1 C -0.2437(6) 0.7467(5) 0.4122(5) 0.0231(9) Uani 1 1 d . . .
N2 N -0.1292(5) 0.8284(5) 0.3766(5) 0.0333(10) Uani 1 1 d . . .
C2 C 0.0516(6) 0.7471(5) 0.4052(5) 0.0231(9) Uani 1 1 d . . .
N3 N 0.2122(5) 0.6918(5) 0.4235(5) 0.0295(9) Uani 1 1 d . . .
N4 N -0.2928(5) 0.5426(5) 0.8311(4) 0.0275(8) Uani 1 1 d . . .
C3 C -0.2578(6) 0.6893(6) 0.7699(5) 0.0226(9) Uani 1 1 d . . .
C4 C -0.0733(6) 0.6850(6) 0.7602(5) 0.0221(9) Uani 1 1 d . . .

```

N5 N 0.0736(5) 0.5352(5) 0.8106(4) 0.0223(8) Uani 1 1 d . . .
 C5 C 0.0390(6) 0.3903(6) 0.8708(4) 0.0219(9) Uani 1 1 d . . .
 C6 C -0.1459(6) 0.3944(6) 0.8818(5) 0.0238(9) Uani 1 1 d . . .
 C7 C -0.4255(7) 0.8531(6) 0.7155(6) 0.0337(11) Uani 1 1 d . . .
 H7A H -0.5411 0.8280 0.7324 0.051 Uiso 1 1 calc R . .
 H7B H -0.4093 0.9100 0.6146 0.051 Uiso 1 1 calc R . .
 H7C H -0.4354 0.9282 0.7639 0.051 Uiso 1 1 calc R . .
 C8 C -0.0308(7) 0.8463(6) 0.6956(5) 0.0323(11) Uani 1 1 d . . .
 H8A H 0.1059 0.8169 0.6987 0.048 Uiso 1 1 calc R . .
 H8B H -0.0980 0.9211 0.7482 0.048 Uiso 1 1 calc R . .
 H8C H -0.0729 0.9056 0.5980 0.048 Uiso 1 1 calc R . .
 C9 C 0.2073(7) 0.2265(6) 0.9243(5) 0.0303(10) Uani 1 1 d . . .
 H9A H 0.3228 0.2520 0.9066 0.045 Uiso 1 1 calc R . .
 H9B H 0.2166 0.1515 0.8761 0.045 Uiso 1 1 calc R . .
 H9C H 0.1924 0.1693 1.0253 0.045 Uiso 1 1 calc R . .
 C10 C -0.1925(8) 0.2361(7) 0.9487(6) 0.0380(12) Uani 1 1 d . . .
 H10A H -0.3296 0.2678 0.9452 0.057 Uiso 1 1 calc R . .
 H10B H -0.1516 0.1786 1.0466 0.057 Uiso 1 1 calc R . .
 H10C H -0.1267 0.1588 0.8981 0.057 Uiso 1 1 calc R . .

loop_

_atom_site_aniso_label
 _atom_site_aniso_U_11
 _atom_site_aniso_U_22
 _atom_site_aniso_U_33
 _atom_site_aniso_U_23
 _atom_site_aniso_U_13
 _atom_site_aniso_U_12
 Mn1 0.0107(4) 0.0244(5) 0.0277(5) -0.0114(4) 0.0028(3) -0.0094(3)
 O1 0.0196(17) 0.066(3) 0.0336(19) -0.0275(19) 0.0098(15) -0.0231(18)
 N1 0.0201(18) 0.029(2) 0.038(2) -0.0125(17) -0.0002(16) -0.0116(16)
 C1 0.0164(19) 0.019(2) 0.030(2) -0.0080(17) -0.0020(16) -0.0024(16)
 N2 0.0177(18) 0.0198(19) 0.058(3) -0.0064(18) -0.0071(18) -0.0077(15)
 C2 0.019(2) 0.019(2) 0.033(2) -0.0098(18) 0.0013(17) -0.0088(17)
 N3 0.0179(18) 0.027(2) 0.044(2) -0.0131(18) -0.0004(16) -0.0090(16)
 N4 0.0195(18) 0.029(2) 0.038(2) -0.0138(17) -0.0075(16) -0.0077(16)
 C3 0.020(2) 0.022(2) 0.025(2) -0.0080(17) -0.0007(16) -0.0070(17)
 C4 0.022(2) 0.024(2) 0.023(2) -0.0110(17) 0.0036(16) -0.0116(17)
 N5 0.0187(17) 0.0280(19) 0.0235(18) -0.0113(15) 0.0027(14) -0.0111(15)
 C5 0.020(2) 0.025(2) 0.0181(19) -0.0072(17) 0.0012(15) -0.0084(17)
 C6 0.024(2) 0.025(2) 0.022(2) -0.0029(17) -0.0006(16) -0.0135(18)
 C7 0.026(2) 0.027(2) 0.038(3) -0.010(2) -0.004(2) -0.0002(19)
 C8 0.038(3) 0.029(2) 0.038(3) -0.013(2) 0.007(2) -0.021(2)
 C9 0.029(2) 0.026(2) 0.027(2) -0.0033(19) -0.0029(19) -0.0068(19)
 C10 0.038(3) 0.031(3) 0.041(3) 0.002(2) -0.003(2) -0.022(2)

_geom_special_details

;

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.

;

loop_

```

    _geom_bond_atom_site_label_1
    _geom_bond_atom_site_label_2
    _geom_bond_distance
    _geom_bond_site_symmetry_2
    _geom_bond_publ_flag
Mn1 O1 2.162(4) 2_466 ?
Mn1 O1 2.162(4) . ?
Mn1 N1 2.229(4) . ?
Mn1 N1 2.229(4) 2_466 ?
Mn1 N3 2.232(4) 1_455 ?
Mn1 N3 2.232(4) 2_566 ?
O1 H1 0.84(9) . ?
O1 H2 0.79(7) . ?
N1 C1 1.163(6) . ?
C1 N2 1.297(6) . ?
N2 C2 1.309(6) . ?
C2 N3 1.150(6) . ?
N3 Mn1 2.232(4) 1_655 ?
N4 C6 1.341(6) . ?
N4 C3 1.357(6) . ?
C3 C4 1.396(6) . ?
C3 C7 1.500(6) . ?
C4 N5 1.347(6) . ?
C4 C8 1.508(6) . ?
N5 C5 1.341(6) . ?
C5 C6 1.397(6) . ?
C5 C9 1.501(6) . ?
C6 C10 1.501(6) . ?

loop_
    _geom_angle_atom_site_label_1
    _geom_angle_atom_site_label_2
    _geom_angle_atom_site_label_3
    _geom_angle
    _geom_angle_site_symmetry_1
    _geom_angle_site_symmetry_3
    _geom_angle_publ_flag
O1 Mn1 O1 179.998(1) 2_466 . ?
O1 Mn1 N1 89.78(15) 2_466 . ?
O1 Mn1 N1 90.22(15) . . ?
O1 Mn1 N1 90.22(15) 2_466 2_466 ?
O1 Mn1 N1 89.78(15) . 2_466 ?
N1 Mn1 N1 179.999(1) . 2_466 ?
O1 Mn1 N3 89.69(15) 2_466 1_455 ?
O1 Mn1 N3 90.32(15) . 1_455 ?
N1 Mn1 N3 96.01(14) . 1_455 ?
N1 Mn1 N3 83.99(14) 2_466 1_455 ?
O1 Mn1 N3 90.32(15) 2_466 2_566 ?
O1 Mn1 N3 89.68(15) . 2_566 ?
N1 Mn1 N3 83.99(14) . 2_566 ?
N1 Mn1 N3 96.01(14) 2_466 2_566 ?
N3 Mn1 N3 179.998(2) 1_455 2_566 ?
H1 O1 H2 109(7) . . ?
H1 O1 Mn1 125(5) . . ?
H2 O1 Mn1 125(5) . . ?
C1 N1 Mn1 161.1(4) . . ?
N1 C1 N2 173.3(5) . . ?

```

C1 N2 C2 121.0(4) . . ?
 N3 C2 N2 173.0(5) . . ?
 C2 N3 Mn1 159.3(4) . 1_655 ?
 C6 N4 C3 118.3(4) . . ?
 N4 C3 C4 120.6(4) . . ?
 N4 C3 C7 117.0(4) . . ?
 C4 C3 C7 122.4(4) . . ?
 N5 C4 C3 120.7(4) . . ?
 N5 C4 C8 117.7(4) . . ?
 C3 C4 C8 121.7(4) . . ?
 C5 N5 C4 118.8(4) . . ?
 N5 C5 C6 120.7(4) . . ?
 N5 C5 C9 116.8(4) . . ?
 C6 C5 C9 122.5(4) . . ?
 N4 C6 C5 121.0(4) . . ?
 N4 C6 C10 116.1(4) . . ?
 C5 C6 C10 122.8(4) . . ?

loop_
 _geom_hbond_atom_site_label_D
 _geom_hbond_atom_site_label_H
 _geom_hbond_atom_site_label_A
 _geom_hbond_distance_DH
 _geom_hbond_distance_HA
 _geom_hbond_distance_DA
 _geom_hbond_angle_DHA
 _geom_hbond_site_symmetry_A
 _geom_hbond_publ_flag

 # Short contacts
 # =====
 #

#	D	H	A	D-H	H...A	D...A	D-H...A	symm	publ
#	-	-	-	----	-----	-----	-----	-----	-----
	O1	H1	N4	0.84(9)	1.98(10)	2.801(5)	169(8)	1_555	no
	O1	H2	N5	0.79(7)	2.00(7)	2.770(5)	166(7)	1_455	NO

_diffrn_measured_fraction_theta_max 0.950
 _diffrn_reflns_theta_full 27.50
 _diffrn_measured_fraction_theta_full 0.982
 _refine_diff_density_max 1.662
 _refine_diff_density_min -0.404
 _refine_diff_density_rms 0.117

_publ_contact_author
 ;
 Dr. Stuart R. Batten
 School of Chemistry
 PO Box 23
 Monash University 3800
 AUSTRALIA

_publ_contact_author_phone ' +61 3 9905 4606 '
 _publ_contact_author_fax ' +61 3 9905 4597 '
 _publ_contact_author_email '

Keith.S.Murray@sci.monash.edu.au '

loop_

_publ_author_name

_publ_author_address

'Kutasi, Anna M.'

;

School of Chemistry

PO Box 23

Monash University 3800

Australia

;

'Batten, Stuart R.'

;

School of Chemistry

PO Box 23

Monash University 3800

Australia

;

'Harris, Alexander R.'

;

School of Chemistry

PO Box 23

Monash University 3800

Australia

;

'Moubaraki, Boujema'

;

School of Chemistry

PO Box 23

Monash University 3800

Australia

;

'Murray, Keith S.'

;

School of Chemistry

PO Box 23

Monash University 3800

Australia

;

_publ_section_title

;

Structural and Magnetic Properties of the Coordination

Polymer $Mn(dca)_2(H_2O)_2 \cdot 2Me_4pyz$, dca =

Dicyanamide ($N(CN)_2^-$), Me_4pyz = Tetramethylpyrazine

;

#=END OF CIF