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*Australian Journal of Chemistry –  
an International Journal for Chemical Science*



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data\_jmwegs31 DNSAAM1.CIF COMPOUND (1B) Smith et al., *Aust. J. Chem.*, 2001.

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'O' 'O' 0.0492 0.0322  
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'-x, -y, -z'  
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'x+1/2, -y+1/2, z-1/2'

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goodness of fit S are based on F2, conventional R-factors R are based
on F, with F set to zero for negative F2. The threshold expression of
F2 > 2sigma(F2) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F2 are statistically about twice as large as those based on F, and R-
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'calc w=1/[\s2(Fo2)+(0.0737P)2+1.6951P] where P=(Fo2+2Fc2)/3'

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_atom_sites_solution_secondary difmap
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_atom_site_fract_z
_atom_site_U_iso_or_equiv
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# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

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O31	O	Uani	0.32999(15)	0.1682(5)	0.5232(3)	1.000	0.0245(10)
O32	O	Uani	0.42037(15)	0.0059(5)	0.6463(3)	1.000	0.0231(10)
O51	O	Uani	0.62609(15)	0.1992(5)	0.7022(3)	1.000	0.0298(10)
O52	O	Uani	0.62623(15)	0.2993(5)	0.5506(3)	1.000	0.0284(10)
O71	O	Uani	0.42052(15)	0.4937(5)	0.1948(3)	1.000	0.0228(10)
O72	O	Uani	0.33069(15)	0.3321(5)	0.1371(3)	1.000	0.0253(10)
N3	N	Uani	0.38802(17)	0.1172(5)	0.5613(3)	1.000	0.0166(11)
N5	N	Uani	0.59705(17)	0.2499(5)	0.5965(3)	1.000	0.0181(11)
C1	C	Uani	0.4203(2)	0.3091(6)	0.3364(3)	1.000	0.0141(12)
C2	C	Uani	0.3793(2)	0.2489(6)	0.3796(4)	1.000	0.0141(12)
C3	C	Uani	0.4202(2)	0.1899(6)	0.5042(4)	1.000	0.0158(12)
C4	C	Uani	0.4895(2)	0.1884(6)	0.5735(4)	1.000	0.0150(12)
C5	C	Uani	0.52386(18)	0.2500(6)	0.5238(4)	1.000	0.0142(11)
C6	C	Uani	0.4895(2)	0.3102(6)	0.4054(4)	1.000	0.0146(12)
C7	C	Uani	0.38779(16)	0.3837(6)	0.2144(3)	1.000	0.0043(10)
N1	N	Uani	0.74979(19)	0.4999(6)	0.6152(3)	1.000	0.0171(11)
H4	H	Uiso	0.516(2)	0.136(6)	0.656(4)	1.000	0.014(11)
H6	H	Uiso	0.5119(19)	0.343(6)	0.373(3)	1.000	0.007(11)
H1A	H	Uiso	0.720(2)	0.463(6)	0.544(4)	1.000	0.008(11)
H1B	H	Uiso	0.786(3)	0.560(9)	0.610(5)	1.000	0.053(18)
H1C	H	Uiso	0.772(4)	0.393(12)	0.681(7)	1.000	0.09(3)
H1D	H	Uiso	0.728(2)	0.579(8)	0.643(4)	1.000	0.026(13)

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

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O31	0.0167(15)	0.034(2)	0.0281(17)	0.0067(15)	0.0159(14)	0.0051(14)
O32	0.0228(17)	0.0270(19)	0.0191(15)	0.0073(15)	0.0115(14)	0.0001(15)
O51	0.0146(15)	0.043(2)	0.0222(17)	0.0096(16)	0.0045(13)	0.0059(16)
O52	0.0145(15)	0.044(2)	0.0295(17)	0.0009(17)	0.0140(14)	-0.0049(15)
O71	0.0220(16)	0.0260(19)	0.0245(16)	0.0052(15)	0.0155(14)	0.0041(14)
O72	0.0208(16)	0.036(2)	0.0152(15)	0.0046(14)	0.0078(13)	0.0017(15)
N3	0.0142(18)	0.020(2)	0.0163(17)	-0.0016(16)	0.0090(15)	-0.0011(16)
N5	0.0125(17)	0.018(2)	0.019(2)	-0.0021(16)	0.0059(16)	-0.0005(16)
C1	0.017(2)	0.013(2)	0.014(2)	0.0014(18)	0.0098(17)	-0.0008(18)
C2	0.014(2)	0.015(2)	0.015(2)	-0.0060(17)	0.0092(18)	-0.0040(18)

C3	0.019(2)	0.018(2)	0.017(2)	-0.0015(19)	0.0142(18)	0.0009(19)
C4	0.017(2)	0.015(2)	0.014(2)	0.0021(19)	0.0093(18)	0.0010(18)
C5	0.0092(18)	0.013(2)	0.018(2)	-0.0014(17)	0.0062(17)	-0.0004(17)
C6	0.017(2)	0.013(2)	0.020(2)	-0.0047(19)	0.0141(19)	-0.0027(18)
C7	0.0014(18)	0.0075(19)	0.0036(17)	-0.0002(15)	0.0012(15)	0.0014(15)
N1	0.0125(18)	0.024(2)	0.0150(19)	-0.0037(18)	0.0078(16)	0.0013(17)

#=====

\_geom\_special\_details

;

Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All esds are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

;

loop\_

\_geom\_bond\_atom\_site\_label\_1

\_geom\_bond\_atom\_site\_label\_2

\_geom\_bond\_distance

\_geom\_bond\_site\_symmetry\_1

\_geom\_bond\_site\_symmetry\_2

\_geom\_bond\_publ\_flag

O2	C2	1.255(6)	.	.	yes
O31	N3	1.233(6)	.	.	yes
O32	N3	1.231(5)	.	.	yes
O51	N5	1.235(5)	.	.	yes
O52	N5	1.215(6)	.	.	yes
O71	C7	1.227(6)	.	.	yes
O72	C7	1.219(5)	.	.	yes
N3	C3	1.453(7)	.	.	yes
N5	C5	1.451(7)	.	.	yes
N1	H1B	1.00(8)	.	.	no
N1	H1C	1.05(8)	.	.	no
N1	H1A	0.85(5)	.	.	no
N1	H1D	0.97(6)	.	.	no
C1	C2	1.457(7)	.	.	no
C1	C6	1.372(7)	.	.	no
C1	C7	1.463(5)	.	.	no
C2	C3	1.449(6)	.	.	no
C3	C4	1.374(8)	.	.	no
C4	C5	1.389(7)	.	.	no
C5	C6	1.387(6)	.	.	no
C4	H4	0.99(4)	.	.	no
C6	H6	0.89(5)	.	.	no

loop\_

\_geom\_angle\_atom\_site\_label\_1

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\_geom\_angle\_atom\_site\_label\_3

\_geom\_angle

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\_geom\_angle\_publ\_flag

O31	N3	O32	122.8(4)	.	.	.	yes
O31	N3	C3	119.3(4)	.	.	.	yes
O32	N3	C3	117.9(4)	.	.	.	yes
O51	N5	O52	123.8(4)	.	.	.	yes
O51	N5	C5	117.6(4)	.	.	.	yes
O52	N5	C5	118.6(4)	.	.	.	yes
H1A	N1	H1C	116(5)	.	.	.	no
H1A	N1	H1D	108(5)	.	.	.	no
H1B	N1	H1C	108(7)	.	.	.	no
H1B	N1	H1D	118(5)	.	.	.	no
H1C	N1	H1D	102(6)	.	.	.	no
H1A	N1	H1B	105(5)	.	.	.	no
C2	C1	C7	119.9(4)	.	.	.	no
C2	C1	C6	124.1(4)	.	.	.	no
C6	C1	C7	115.9(4)	.	.	.	no
C1	C2	C3	112.1(4)	.	.	.	no
O2	C2	C1	123.8(4)	.	.	.	yes
O2	C2	C3	124.2(5)	.	.	.	yes
N3	C3	C4	115.9(4)	.	.	.	yes
C2	C3	C4	124.2(5)	.	.	.	no
N3	C3	C2	119.9(5)	.	.	.	yes
C3	C4	C5	119.2(4)	.	.	.	no
C4	C5	C6	121.2(5)	.	.	.	no
N5	C5	C6	119.2(4)	.	.	.	yes
N5	C5	C4	119.6(4)	.	.	.	yes
C1	C6	C5	119.2(5)	.	.	.	no
O72	C7	C1	119.3(4)	.	.	.	yes
O71	C7	O72	123.2(4)	.	.	.	yes
O71	C7	C1	117.6(4)	.	.	.	yes
C3	C4	H4	122(3)	.	.	.	no
C5	C4	H4	118(3)	.	.	.	no
C5	C6	H6	121(3)	.	.	.	no
C1	C6	H6	120(2)	.	.	.	no

loop\_

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\_geom\_torsion\_publ\_flag

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O31	N3	C3	C4	156.8(4)	.	.	.	no
O32	N3	C3	C2	153.5(4)	.	.	.	no
O32	N3	C3	C4	-23.4(6)	.	.	.	no
O51	N5	C5	C4	-2.0(6)	.	.	.	no
O51	N5	C5	C6	178.6(4)	.	.	.	no
O52	N5	C5	C4	178.0(4)	.	.	.	no
O52	N5	C5	C6	-1.4(6)	.	.	.	no
C6	C1	C2	O2	179.2(4)	.	.	.	no
C6	C1	C2	C3	-0.1(6)	.	.	.	no
C7	C1	C2	O2	2.7(6)	.	.	.	no

C7	C1	C2	C3	-176.6(4)	.	.	.	.	no
C2	C1	C6	C5	0.4(7)	.	.	.	.	no
C7	C1	C6	C5	177.0(4)	.	.	.	.	no
C2	C1	C7	O71	154.1(4)	.	.	.	.	no
C2	C1	C7	O72	-27.2(6)	.	.	.	.	no
C6	C1	C7	O71	-22.7(6)	.	.	.	.	no
C6	C1	C7	O72	156.0(4)	.	.	.	.	no
O2	C2	C3	N3	3.3(6)	.	.	.	.	no
O2	C2	C3	C4	179.9(9)	.	.	.	.	no
C1	C2	C3	N3	-177.3(4)	.	.	.	.	no
C1	C2	C3	C4	-0.7(6)	.	.	.	.	no
N3	C3	C4	C5	178.0(4)	.	.	.	.	no
C2	C3	C4	C5	1.2(7)	.	.	.	.	no
C3	C4	C5	N5	179.7(4)	.	.	.	.	no
C3	C4	C5	C6	-0.9(6)	.	.	.	.	no
N5	C5	C6	C1	179.6(4)	.	.	.	.	no
C4	C5	C6	C1	0.1(7)	.	.	.	.	no

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N1	H1A	O72	0.85(5)	2.22(4)	3.046(5)	163(5)	2_655	yes
N1	H1B	O31	1.00(8)	2.11(7)	3.047(6)	156(5)	5_555	yes
N1	H1B	O51	1.00(8)	2.41(6)	2.933(6)	112(4)	6_656	yes
N1	H1C	O2	1.05(8)	1.81(8)	2.839(5)	168(7)	8_555	yes
N1	H1C	O72	1.05(8)	2.39(10)	2.924(6)	110(6)	8_555	yes
N1	H1D	O2	0.97(6)	1.87(6)	2.829(6)	174(5)	3_666	yes
N1	H1D	O31	0.97(6)	2.56(5)	2.910(5)	101(3)	3_666	yes
C4	H4	O32	0.99(4)	2.38(4)	3.373(6)	179(4)	2_656	yes
C6	H6	O71	0.89(5)	2.49(5)	3.373(7)	169(3)	2_655	yes

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data\_jmwwgs33 : DNSAEN.CIF COMPOUND 5. Smith et al., Aust. J. Chem., 2001.

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  'C' 'C' 0.0181 0.0091  
  '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' 0.0311 0.0180  
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'  
  'O' 'O' 0.0492 0.0322  
  'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'  
  
_symmetry_cell_setting          'Monoclinic '  
_symmetry_space_group_name_H-M  'P 21/c '  
  
loop_  
  _symmetry_equiv_pos_as_xyz  
  'x, y, z'  
  '-x, y+1/2, -z+1/2'  
  '-x, -y, -z'  
  'x, -y-1/2, z-1/2'  
  
_cell_length_a                  7.0290(10)  
_cell_length_b                  10.4069(9)  
_cell_length_c                  17.116(2)  
_cell_angle_alpha               90.00  
_cell_angle_beta                96.900(10)  
_cell_angle_gamma               90.00  
_cell_volume                     1243.0(3)  
_cell_formula_units_Z           4  
_cell_measurement_temperature    293(2)  
_cell_measurement_reflns_used    25  
_cell_measurement_theta_min      11  
_cell_measurement_theta_max      13  
  
_exptl_crystal_description      'plate '  
_exptl_crystal_colour           'yellow '
```

```

_exptl_crystal_size_max      ?
_exptl_crystal_size_mid     ?
_exptl_crystal_size_min     ?
_exptl_crystal_density_meas  ?
_exptl_crystal_density_diffn 1.636
_exptl_crystal_density_method 'not measured'
_exptl_crystal_F_000        640
_exptl_absorpt_coefficient_mu 1.269
_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 293(2)
_diffn_radiation_wavelength 1.54180
_diffn_radiation_type      'CuK\alpha '
_diffn_radiation_source    'fine-focus sealed tube'
_diffn_radiation_monochromator 'graphite '
_diffn_measurement_device_type 'Enraf-Nonius CAD-4 '
_diffn_measurement_method   '\w--2\q '
_diffn_detector_area_resol_mean ?
_diffn_standards_number     3
_diffn_standards_interval_count ?
_diffn_standards_interval_time 160
_diffn_standards_decay_%    ?
_diffn_reflns_number        2562
_diffn_reflns_av_R_equivalents 0.0171
_diffn_reflns_av_sigmaI/netI 0.0307
_diffn_reflns_limit_h_min    0
_diffn_reflns_limit_h_max    8
_diffn_reflns_limit_k_min    0
_diffn_reflns_limit_k_max    12
_diffn_reflns_limit_l_min    -20
_diffn_reflns_limit_l_max    20
_diffn_reflns_theta_min      4.98
_diffn_reflns_theta_max      69.95
_diffn_measured_fraction_theta_max 0.998
_diffn_reflns_theta_full     69.95
_diffn_measured_fraction_theta_full 0.998
_refine_diff_density_max     0.335
_refine_diff_density_min     -0.249
_refine_diff_density_rms     0.080

_reflns_number_total         2361
_reflns_number_gt            1899
_reflns_threshold_expression 'I >2\s (I)'

_computing_data_collection   ?
_computing_cell_refinement   ?
_computing_data_reduction    ?
_computing_structure_solution 'SHELXS97 (Sheldrick, 1997)'
_computing_structure_refinement 'SHELXL97 (Sheldrick, 1997)'

```

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_computing_molecular_graphics      'PLATON for Windows (Spek, 1999)
_computing_publication_material    ?
```

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```
;
Refinement of F2 against ALL reflections. The weighted R-factor wR and
goodness of fit S are based on F2, conventional R-factors R are based
on F, with F set to zero for negative F2. The threshold expression of
F2 > 2sigma(F2) is used only for calculating R-factors(gt) etc. and is
not relevant to the choice of reflections for refinement. R-factors based
on F2 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/[\s2(Fo2)+(0.0519P)2+0.3936P] where P=(Fo2+2Fc2)/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        'SHELXL97 '
_refine_ls_extinction_coef          0.0054(6)
_refine_ls_extinction_expression
'Fc**=kFc[1+0.001xFc2\l3/sin(2\q)]-1/4'
_refine_ls_number_reflns            2361
_refine_ls_number_parameters         247
_refine_ls_number_restraints         0
_refine_ls_R_factor_all              0.0541
_refine_ls_R_factor_gt              0.0400
_refine_ls_wR_factor_ref             0.1027
_refine_ls_wR_factor_gt             0.0944
_refine_ls_goodness_of_fit_ref       1.041
_refine_ls_restrained_S_all          1.041
_refine_ls_shift/su_max              0.008
_refine_ls_shift/su_mean             0.001
```

```
# ATOMIC COORDINATES AND THERMAL PARAMETERS
```

```
loop_
```

```
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_atom_site_thermal_displace_type
_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_occupancy
_atom_site_U_iso_or_equiv
O2      O      Uani      0.7076(2)  0.26610(13)  1.12208(8)   1.000  0.0400(5)
O31     O      Uani      0.7834(2)  0.45623(15)  1.23069(8)   1.000  0.0455(5)
O32     O      Uani      0.6233(3)  0.62335(15)  1.18757(9)   1.000  0.0489(5)
O51     O      Uani      0.8134(2)  0.77428(13)  0.94059(9)   1.000  0.0470(5)
O52     O      Uani      0.7874(3)  0.64110(17)  0.84390(9)   1.000  0.0668(7)
O71     O      Uani      0.8355(2)  0.18262(13)  0.89747(8)   1.000  0.0388(4)
O72     O      Uani      0.6857(2)  0.10673(13)  0.99342(8)   1.000  0.0412(5)
```

N3	N	Uani	0.7121(2)	0.52439(15)	1.17631(9)	1.000	0.0333(5)
N5	N	Uani	0.7898(3)	0.66432(16)	0.91398(10)	1.000	0.0356(5)
C1	C	Uani	0.7508(2)	0.33240(16)	0.99132(10)	1.000	0.0233(5)
C2	C	Uani	0.7291(3)	0.35432(17)	1.07361(10)	1.000	0.0251(5)
C3	C	Uani	0.7330(3)	0.48895(17)	1.09590(10)	1.000	0.0266(5)
C4	C	Uani	0.7490(3)	0.58802(18)	1.04465(11)	1.000	0.0290(6)
C5	C	Uani	0.7676(3)	0.56041(17)	0.96735(10)	1.000	0.0265(5)
C6	C	Uani	0.7690(3)	0.43341(18)	0.94131(10)	1.000	0.0255(5)
C7	C	Uani	0.7577(3)	0.19779(17)	0.95888(10)	1.000	0.0269(5)
N1	N	Uani	1.5285(3)	-0.05508(16)	0.86855(10)	1.000	0.0306(5)
N2	N	Uani	1.0911(3)	-0.00861(17)	0.86427(11)	1.000	0.0344(6)
C11	C	Uani	1.4072(3)	0.0348(2)	0.81722(12)	1.000	0.0346(6)
C21	C	Uani	1.2440(3)	0.08742(19)	0.85594(13)	1.000	0.0355(6)
O1	O	Uani	0.9090(3)	0.31403(16)	0.76360(11)	1.000	0.0528(6)
H4	H	Uiso	0.748(3)	0.669(2)	1.0620(12)	1.000	0.037(6)
H6	H	Uiso	0.780(3)	0.417(2)	0.8862(13)	1.000	0.032(5)
H11	H	Uiso	1.593(4)	-0.015(3)	0.9132(16)	1.000	0.061(8)
H12	H	Uiso	1.619(4)	-0.091(2)	0.8447(14)	1.000	0.047(7)
H13	H	Uiso	1.460(4)	-0.122(3)	0.8875(15)	1.000	0.055(7)
H21	H	Uiso	1.131(4)	-0.072(3)	0.9018(17)	1.000	0.072(9)
H22	H	Uiso	1.062(4)	-0.053(3)	0.8137(18)	1.000	0.065(8)
H23	H	Uiso	0.989(4)	0.037(3)	0.8774(14)	1.000	0.049(7)
H111	H	Uiso	1.491(4)	0.107(3)	0.8050(14)	1.000	0.052(7)
H112	H	Uiso	1.358(3)	-0.009(2)	0.7691(13)	1.000	0.038(6)
H211	H	Uiso	1.177(4)	0.156(3)	0.8232(16)	1.000	0.061(8)
H212	H	Uiso	1.287(3)	0.120(2)	0.9101(15)	1.000	0.044(6)
H1A	H	Uiso	0.882(5)	0.279(3)	0.811(2)	1.000	0.091(11)
H1B	H	Uiso	0.856(5)	0.265(4)	0.726(2)	1.000	0.103(13)

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

O2	0.0668(10)	0.0268(7)	0.0273(7)	-0.0003(6)	0.0098(7)	-0.0155(7)
O31	0.0677(11)	0.0401(8)	0.0279(7)	-0.0016(6)	0.0024(7)	-0.0036(8)
O32	0.0684(11)	0.0342(8)	0.0481(9)	-0.0127(7)	0.0235(8)	0.0041(8)
O51	0.0613(10)	0.0220(7)	0.0561(9)	0.0071(6)	0.0004(8)	0.0011(7)
O52	0.1207(17)	0.0457(10)	0.0325(8)	0.0111(7)	0.0031(9)	-0.0126(10)
O71	0.0582(9)	0.0279(7)	0.0335(7)	-0.0035(6)	0.0181(7)	0.0043(7)
O72	0.0657(10)	0.0248(7)	0.0345(7)	-0.0037(6)	0.0122(7)	-0.0130(7)
N3	0.0412(9)	0.0281(8)	0.0319(9)	-0.0080(7)	0.0100(7)	-0.0081(7)
N5	0.0430(10)	0.0258(8)	0.0366(9)	0.0070(7)	-0.0010(7)	0.0009(7)
C1	0.0218(8)	0.0220(8)	0.0260(8)	-0.0018(7)	0.0027(7)	-0.0009(7)
C2	0.0267(9)	0.0228(9)	0.0259(9)	-0.0019(7)	0.0040(7)	-0.0035(7)
C3	0.0294(9)	0.0259(9)	0.0251(9)	-0.0043(7)	0.0055(7)	-0.0023(7)
C4	0.0317(10)	0.0194(9)	0.0356(10)	-0.0044(7)	0.0032(8)	0.0013(7)
C5	0.0279(9)	0.0213(9)	0.0299(9)	0.0036(7)	0.0020(7)	0.0012(7)
C6	0.0267(9)	0.0259(9)	0.0236(8)	0.0004(7)	0.0023(7)	0.0014(7)
C7	0.0333(10)	0.0235(9)	0.0236(8)	-0.0013(7)	0.0020(7)	0.0010(7)
N1	0.0308(9)	0.0262(8)	0.0351(9)	0.0012(7)	0.0047(7)	-0.0003(7)
N2	0.0351(10)	0.0305(9)	0.0381(10)	0.0002(8)	0.0070(8)	0.0052(7)
C11	0.0370(11)	0.0329(10)	0.0340(10)	0.0069(9)	0.0042(9)	-0.0006(9)
C21	0.0395(11)	0.0255(9)	0.0412(11)	0.0004(9)	0.0034(9)	0.0040(8)

O1 0.0841(13) 0.0346(8) 0.0395(9) 0.0051(7) 0.0061(9) -0.0091(9)

#=====

\_geom\_special\_details

;

Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All esds are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

;

loop\_

\_geom\_bond\_atom\_site\_label\_1

\_geom\_bond\_atom\_site\_label\_2

\_geom\_bond\_distance

\_geom\_bond\_site\_symmetry\_1

\_geom\_bond\_site\_symmetry\_2

\_geom\_bond\_publ\_flag

O2	C2	1.259(2)	.	.	yes
O31	N3	1.228(2)	.	.	yes
O32	N3	1.231(2)	.	.	yes
O51	N5	1.235(2)	.	.	yes
O52	N5	1.222(2)	.	.	yes
O71	C7	1.252(2)	.	.	yes
O72	C7	1.255(2)	.	.	yes
O1	H1B	0.87(4)	.	.	no
O1	H1A	0.93(3)	.	.	no
N3	C3	1.449(2)	.	.	yes
N5	C5	1.436(2)	.	.	yes
N1	C11	1.481(3)	.	.	yes
N2	C21	1.487(3)	.	.	yes
N1	H13	0.93(3)	.	.	no
N1	H11	0.94(3)	.	.	no
N1	H12	0.88(3)	.	.	no
N2	H22	0.98(3)	.	.	no
N2	H23	0.91(3)	.	.	no
N2	H21	0.94(3)	.	.	no
C1	C7	1.510(2)	.	.	no
C1	C6	1.371(2)	.	.	no
C1	C2	1.453(2)	.	.	no
C2	C3	1.451(3)	.	.	no
C3	C4	1.367(3)	.	.	no
C4	C5	1.375(3)	.	.	no
C5	C6	1.395(3)	.	.	no
C4	H4	0.89(2)	.	.	no
C6	H6	0.97(2)	.	.	no
C11	C21	1.496(3)	.	.	no
C11	H111	0.99(3)	.	.	no
C11	H112	0.97(2)	.	.	no
C21	H212	1.00(2)	.	.	no
C21	H211	0.99(3)	.	.	no

loop\_

\_geom\_angle\_atom\_site\_label\_1

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<u>_geom_angle_atom_site_label_3</u>							
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<u>_geom_angle_site_symmetry_1</u>							
<u>_geom_angle_site_symmetry_2</u>							
<u>_geom_angle_site_symmetry_3</u>							
<u>_geom_angle_publ_flag</u>							
H1A	O1	H1B	108(3)	.	.	.	no
O31	N3	O32	122.19(16)	.	.	.	yes
O31	N3	C3	119.60(15)	.	.	.	yes
O32	N3	C3	118.20(15)	.	.	.	yes
O51	N5	O52	122.04(18)	.	.	.	yes
O51	N5	C5	118.88(16)	.	.	.	yes
O52	N5	C5	119.07(17)	.	.	.	yes
C11	N1	H11	112.9(19)	.	.	.	no
C11	N1	H12	113.0(15)	.	.	.	no
C11	N1	H13	113.3(17)	.	.	.	no
H11	N1	H12	105(2)	.	.	.	no
H11	N1	H13	106(2)	.	.	.	no
H12	N1	H13	106(2)	.	.	.	no
C21	N2	H23	105.9(19)	.	.	.	no
H21	N2	H22	107(3)	.	.	.	no
H21	N2	H23	112(2)	.	.	.	no
H22	N2	H23	112(2)	.	.	.	no
C21	N2	H22	108.0(17)	.	.	.	no
C21	N2	H21	112.2(18)	.	.	.	no
C6	C1	C7	118.18(15)	.	.	.	no
C2	C1	C7	120.93(15)	.	.	.	no
C2	C1	C6	120.89(16)	.	.	.	no
C1	C2	C3	113.92(15)	.	.	.	no
O2	C2	C3	122.06(16)	.	.	.	yes
O2	C2	C1	124.01(16)	.	.	.	yes
N3	C3	C4	116.29(16)	.	.	.	yes
C2	C3	C4	124.04(16)	.	.	.	no
N3	C3	C2	119.63(15)	.	.	.	yes
C3	C4	C5	118.97(17)	.	.	.	no
C4	C5	C6	120.69(16)	.	.	.	no
N5	C5	C4	118.99(16)	.	.	.	yes
N5	C5	C6	120.31(16)	.	.	.	yes
C1	C6	C5	121.47(16)	.	.	.	no
O71	C7	C1	117.54(16)	.	.	.	yes
O72	C7	C1	119.65(16)	.	.	.	yes
O71	C7	O72	122.81(17)	.	.	.	yes
C5	C4	H4	121.5(13)	.	.	.	no
C3	C4	H4	119.6(13)	.	.	.	no
C1	C6	H6	119.7(12)	.	.	.	no
C5	C6	H6	118.8(12)	.	.	.	no
N1	C11	C21	112.69(17)	.	.	.	yes
N2	C21	C11	113.45(17)	.	.	.	yes
N1	C11	H112	109.2(13)	.	.	.	no
C21	C11	H111	108.8(17)	.	.	.	no
N1	C11	H111	106.8(16)	.	.	.	no
H111	C11	H112	109.7(19)	.	.	.	no
C21	C11	H112	109.6(13)	.	.	.	no
N2	C21	H211	103.8(17)	.	.	.	no
N2	C21	H212	106.3(13)	.	.	.	no
C11	C21	H212	111.9(12)	.	.	.	no

H211	C21	H212	111(2)	.	.	.	no
C11	C21	H211	110.5(16)	.	.	.	no

loop\_

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_geom_torsion_atom_site_label_3								
_geom_torsion_atom_site_label_4								
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_geom_torsion_site_symmetry_3								
_geom_torsion_site_symmetry_4								
_geom_torsion_publ_flag								
O31	N3	C3	C2	36.3(3)	.	.	.	no
O31	N3	C3	C4	-145.90(19)	.	.	.	no
O32	N3	C3	C2	-144.1(2)	.	.	.	no
O32	N3	C3	C4	33.7(3)	.	.	.	no
O51	N5	C5	C4	8.1(3)	.	.	.	no
O51	N5	C5	C6	-170.5(2)	.	.	.	no
O52	N5	C5	C4	-172.9(2)	.	.	.	no
O52	N5	C5	C6	8.5(3)	.	.	.	no
C6	C1	C2	C3	-1.2(3)	.	.	.	no
C7	C1	C2	O2	-2.8(3)	.	.	.	no
C2	C1	C6	C5	0.1(3)	.	.	.	no
C7	C1	C6	C5	-178.79(18)	.	.	.	no
C7	C1	C2	C3	177.61(17)	.	.	.	no
C6	C1	C2	O2	178.39(19)	.	.	.	no
C6	C1	C7	O71	21.2(3)	.	.	.	no
C6	C1	C7	O72	-158.40(18)	.	.	.	no
C2	C1	C7	O71	-157.67(17)	.	.	.	no
C2	C1	C7	O72	22.7(3)	.	.	.	no
C1	C2	C3	N3	179.76(19)	.	.	.	no
O2	C2	C3	N3	0.1(3)	.	.	.	no
O2	C2	C3	C4	-177.5(2)	.	.	.	no
C1	C2	C3	C4	2.1(3)	.	.	.	no
N3	C3	C4	C5	-179.47(18)	.	.	.	no
C2	C3	C4	C5	-1.7(3)	.	.	.	no
C3	C4	C5	N5	-178.3(2)	.	.	.	no
C3	C4	C5	C6	0.4(3)	.	.	.	no
N5	C5	C6	C1	179.07(18)	.	.	.	no
C4	C5	C6	C1	0.4(3)	.	.	.	no
N1	C11	C21	N2	-71.8(2)	.	.	.	no

loop\_

_geom_hbond_atom_site_label_D								
_geom_hbond_atom_site_label_H								
_geom_hbond_atom_site_label_A								
_geom_hbond_distance_DH								
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O1	H1B	O31	0.87(4)	2.36(4)	2.981(2)	128(3)	4_554	yes
N1	H11	O72	0.94(3)	1.92(3)	2.839(2)	164(3)	1_655	yes
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N2	H21	O2	0.94(3)	2.38(3)	3.026(2)	126(2)	3_757	yes
N2	H21	O72	0.94(3)	2.11(3)	2.919(2)	143(3)	3_757	yes
N2	H22	O1	0.98(3)	1.94(3)	2.863(3)	155(3)	2_746	yes
N2	H22	O31	0.98(3)	2.49(3)	3.002(2)	112(2)	4_554	yes
N2	H23	O71	0.91(3)	1.92(3)	2.785(2)	159(3)	.	yes
C11	H112	O52	0.97(2)	2.60(2)	3.129(3)	114.7(15)	2_746	yes

# End of Crystallographic Information File



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Refinement of F2 against ALL reflections. The weighted R-factor wR and
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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.

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\_geom\_special\_details

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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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 C7 C1 C2 C3 -179.3(3) . . . . ?  
 O2 C2 C3 C4 179.7(3) . . . . ?  
 C1 C2 C3 C4 -0.3(4) . . . . ?  
 O2 C2 C3 N3 -1.3(5) . . . . ?  
 C1 C2 C3 N3 178.7(3) . . . . ?  
 O31 N3 C3 C4 -176.7(3) . . . . ?  
 O32 N3 C3 C4 2.7(4) . . . . ?  
 O31 N3 C3 C2 4.3(5) . . . . ?  
 O32 N3 C3 C2 -176.3(3) . . . . ?  
 C2 C3 C4 C5 -0.8(5) . . . . ?  
 N3 C3 C4 C5 -179.8(3) . . . . ?  
 C3 C4 C5 C6 1.5(4) . . . . ?  
 C3 C4 C5 N5 -176.2(3) . . . . ?  
 O51 N5 C5 C4 -10.2(4) . . . . ?  
 O52 N5 C5 C4 169.5(3) . . . . ?  
 O51 N5 C5 C6 171.9(3) . . . . ?  
 O52 N5 C5 C6 -8.3(4) . . . . ?  
 C2 C1 C6 C5 0.0(5) . . . . ?  
 C7 C1 C6 C5 179.9(3) . . . . ?  
 C4 C5 C6 C1 -1.1(5) . . . . ?  
 N5 C5 C6 C1 176.7(3) . . . . ?  
 C6 C1 C7 O71 -0.1(5) . . . . ?  
 C2 C1 C7 O71 179.9(3) . . . . ?  
 C6 C1 C7 O72 -179.5(3) . . . . ?  
 C2 C1 C7 O72 0.4(4) . . . . ?  
 C101 N31 C21 N11 -59.5(3) . . . . ?  
 C41 N31 C21 N11 59.7(3) . . . . ?  
 C81 N11 C21 N31 58.6(3) . . . . ?  
 C91 N11 C21 N31 -59.1(3) . . . . ?  
 C91 N51 C41 N31 60.8(3) . . . . ?  
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 C21 N31 C41 N51 -61.4(3) . . . . ?  
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 C21 N31 C101 N71 60.8(3) . . . . ?  
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\_refine\_diff\_density\_rms 0.063

data\_DNSAAM(2).CIF JMWGS38. COMPOUND (1A) Smith et al., Aust. J. Chem., 2001.

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_chemical_name_systematic
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;
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  '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
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  '-x, -y, -z'
  'x, -y-1/2, z-1/2'

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_cell_volume                   941.2(3)
_cell_formula_units_Z          4
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;
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_computing_structure_solution  ' SHELXS-97 (Sheldrick, 1997)'
_computing_structure_refinement ' SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics ' PLATON for Windows (Spek, 1999)'

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\_computing\_publication\_material ?

\_refine\_special\_details

;

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  $F^2 > 2\sigma(F^2)$  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.

;

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\_atom\_sites\_solution\_secondary difmap  
\_atom\_sites\_solution\_hydrogens geom  
\_refine\_ls\_hydrogen\_treatment reffall  
\_refine\_ls\_extinction\_method 'SHELXL97 (Sheldrick, 1997)'  
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\_refine\_ls\_extinction\_expression  
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\_refine\_ls\_wR\_factor\_ref 0.1059  
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loop\_

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\_atom\_site\_fract\_x  
\_atom\_site\_fract\_y  
\_atom\_site\_fract\_z  
\_atom\_site\_occupancy  
\_atom\_site\_U\_iso\_or\_equiv  
O2 O Uani 0.26664(19) 0.1644(2) 0.05878(8) 1.000 0.0410(5)  
O31 O Uani 0.4707(2) 0.3042(4) 0.17858(9) 1.000 0.0780(8)  
O32 O Uani 0.7298(3) 0.3930(3) 0.16063(9) 1.000 0.0692(7)  
O51 O Uani 0.9385(2) 0.4524(3) -0.09227(11) 1.000 0.0666(7)  
O52 O Uani 0.8184(2) 0.2906(3) -0.18697(9) 1.000 0.0542(6)  
O71 O Uani 0.20353(19) 0.0817(2) -0.17627(8) 1.000 0.0397(5)  
O72 O Uani 0.09644(18) 0.0568(2) -0.06087(9) 1.000 0.0441(5)  
N3 N Uani 0.5867(2) 0.3299(3) 0.13602(9) 1.000 0.0365(6)  
N5 N Uani 0.8210(2) 0.3529(3) -0.12027(10) 1.000 0.0377(6)  
C1 C Uani 0.3850(2) 0.1848(3) -0.06462(11) 1.000 0.0263(5)

C2	C	Uani	0.3964(3)	0.2116(3)	0.01939(11)	1.000	0.0284(6)
C3	C	Uani	0.5587(3)	0.2909(3)	0.05216(11)	1.000	0.0290(6)
C4	C	Uani	0.6956(3)	0.3376(3)	0.00695(11)	1.000	0.0313(6)
C5	C	Uani	0.6765(2)	0.3053(3)	-0.07267(11)	1.000	0.0291(6)
C6	C	Uani	0.5235(3)	0.2285(3)	-0.10874(11)	1.000	0.0290(6)
C7	C	Uani	0.2210(2)	0.1048(3)	-0.10518(11)	1.000	0.0295(6)
N1	N	Uani	1.1276(3)	0.1328(3)	0.20723(12)	1.000	0.0435(7)
H4	H	Uiso	0.803(3)	0.385(3)	0.0292(13)	1.000	0.046(6)
H6	H	Uiso	0.508(3)	0.205(3)	-0.1640(13)	1.000	0.033(5)
H72	H	Uiso	0.152(4)	0.096(5)	-0.002(2)	1.000	0.096(11)
H1A	H	Uiso	1.140(4)	0.226(5)	0.239(2)	1.000	0.081(10)
H1B	H	Uiso	1.192(6)	0.038(7)	0.236(3)	1.000	0.135(16)
H1C	H	Uiso	1.176(4)	0.157(4)	0.162(2)	1.000	0.080(10)
H1D	H	Uiso	1.010(4)	0.088(4)	0.1991(16)	1.000	0.067(8)

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

O2	0.0369(8)	0.0601(10)	0.0272(7)	0.0009(7)	0.0091(6)	-0.0113(7)
O31	0.0481(10)	0.156(2)	0.0311(9)	-0.0160(11)	0.0115(8)	-0.0216(12)
O32	0.0686(12)	0.1037(16)	0.0337(9)	-0.0028(10)	-0.0062(8)	-0.0459(12)
O51	0.0483(10)	0.0854(15)	0.0680(11)	-0.0185(10)	0.0170(8)	-0.0379(10)
O52	0.0425(9)	0.0876(13)	0.0343(9)	-0.0046(9)	0.0138(7)	-0.0117(9)
O71	0.0390(8)	0.0530(10)	0.0266(7)	-0.0013(7)	0.0001(6)	-0.0088(7)
O72	0.0306(8)	0.0691(12)	0.0328(8)	0.0042(7)	0.0032(6)	-0.0168(7)
N3	0.0399(10)	0.0428(10)	0.0264(9)	0.0012(8)	-0.0001(8)	-0.0011(8)
N5	0.0293(9)	0.0471(11)	0.0376(10)	0.0010(9)	0.0074(7)	-0.0064(8)
C1	0.0257(9)	0.0271(10)	0.0260(9)	0.0018(8)	0.0021(7)	0.0007(8)
C2	0.0304(10)	0.0275(10)	0.0277(10)	0.0033(8)	0.0056(8)	0.0013(8)
C3	0.0324(10)	0.0317(10)	0.0226(9)	0.0004(8)	0.0004(8)	0.0020(8)
C4	0.0287(11)	0.0309(11)	0.0336(11)	0.0003(9)	-0.0012(8)	-0.0020(9)
C5	0.0260(10)	0.0317(11)	0.0301(10)	0.0019(8)	0.0052(8)	-0.0005(8)
C6	0.0298(10)	0.0321(11)	0.0252(10)	0.0022(8)	0.0035(8)	0.0014(8)
C7	0.0279(10)	0.0318(10)	0.0287(10)	0.0040(8)	0.0023(8)	0.0005(8)
N1	0.0435(12)	0.0567(14)	0.0313(10)	-0.0070(10)	0.0092(9)	-0.0122(11)

#=====

\_geom\_special\_details

;

Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All esds are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

;

loop\_

\_geom\_bond\_atom\_site\_label\_1

\_geom\_bond\_atom\_site\_label\_2

\_geom\_bond\_distance

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<u>_geom_bond_publ_flag</u>							
O2	C2		1.280(3)	.	.		yes
O31	N3		1.197(2)	.	.		yes
O32	N3		1.219(3)	.	.		yes
O51	N5		1.216(3)	.	.		yes
O52	N5		1.225(2)	.	.		yes
O71	C7		1.221(2)	.	.		yes
O72	C7		1.302(2)	.	.		yes
O72	H72		1.10(3)	.	.		no
N3	C3		1.458(2)	.	.		yes
N5	C5		1.454(2)	.	.		yes
N1	H1B		0.96(5)	.	.		no
N1	H1C		0.90(3)	.	.		no
N1	H1A		0.87(4)	.	.		no
N1	H1D		0.95(3)	.	.		no
C1	C6		1.375(3)	.	.		no
C1	C7		1.490(2)	.	.		no
C1	C2		1.443(3)	.	.		no
C2	C3		1.430(3)	.	.		no
C3	C4		1.382(3)	.	.		no
C4	C5		1.375(3)	.	.		no
C5	C6		1.385(3)	.	.		no
C4	H4		0.93(2)	.	.		no
C6	H6		0.96(2)	.	.		no
loop_							
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<u>_geom_angle_publ_flag</u>							
C7	O72	H72	102.7(17)	.	.	.	no
O31	N3	O32	121.51(17)	.	.	.	yes
O32	N3	C3	117.88(17)	.	.	.	yes
O31	N3	C3	120.59(17)	.	.	.	yes
O51	N5	O52	123.04(18)	.	.	.	yes
O51	N5	C5	118.75(17)	.	.	.	yes
O52	N5	C5	118.21(17)	.	.	.	yes
H1A	N1	H1B	103(4)	.	.	.	no
H1A	N1	H1C	110(3)	.	.	.	no
H1B	N1	H1C	111(3)	.	.	.	no
H1B	N1	H1D	105(3)	.	.	.	no
H1C	N1	H1D	113(3)	.	.	.	no
H1A	N1	H1D	115(3)	.	.	.	no
C2	C1	C6	121.66(17)	.	.	.	no
C2	C1	C7	119.77(16)	.	.	.	no
C6	C1	C7	118.56(17)	.	.	.	no
C1	C2	C3	115.17(18)	.	.	.	no
O2	C2	C3	124.98(17)	.	.	.	yes
O2	C2	C1	119.85(18)	.	.	.	yes
N3	C3	C2	121.08(18)	.	.	.	yes
C2	C3	C4	122.61(18)	.	.	.	no

N3	C3	C4	116.30(18)	.	.	.	yes
C3	C4	C5	119.00(19)	.	.	.	no
C4	C5	C6	121.80(18)	.	.	.	no
N5	C5	C6	119.09(17)	.	.	.	yes
N5	C5	C4	119.10(17)	.	.	.	yes
C1	C6	C5	119.73(17)	.	.	.	no
O71	C7	O72	120.91(17)	.	.	.	yes
O71	C7	C1	122.39(16)	.	.	.	yes
O72	C7	C1	116.69(16)	.	.	.	yes
C3	C4	H4	122.0(14)	.	.	.	no
C5	C4	H4	119.0(14)	.	.	.	no
C5	C6	H6	122.9(14)	.	.	.	no
C1	C6	H6	117.3(14)	.	.	.	no

loop\_

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\_geom\_torsion\_atom\_site\_label\_3

\_geom\_torsion\_atom\_site\_label\_4

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\_geom\_torsion\_site\_symmetry\_3

\_geom\_torsion\_site\_symmetry\_4

\_geom\_torsion\_publ\_flag

O31	N3	C3	C4	175.4(2)	.	.	.	.	no
O32	N3	C3	C2	178.1(2)	.	.	.	.	no
O31	N3	C3	C2	-3.6(4)	.	.	.	.	no
O32	N3	C3	C4	-3.0(3)	.	.	.	.	no
O51	N5	C5	C6	165.3(2)	.	.	.	.	no
O51	N5	C5	C4	-14.7(3)	.	.	.	.	no
O52	N5	C5	C6	-14.6(3)	.	.	.	.	no
O52	N5	C5	C4	165.3(2)	.	.	.	.	no
C6	C1	C2	C3	1.7(3)	.	.	.	.	no
C6	C1	C2	O2	-178.0(2)	.	.	.	.	no
C2	C1	C6	C5	-2.0(3)	.	.	.	.	no
C7	C1	C2	O2	0.9(3)	.	.	.	.	no
C7	C1	C2	C3	-179.39(19)	.	.	.	.	no
C2	C1	C7	O72	-2.3(3)	.	.	.	.	no
C6	C1	C7	O71	-2.1(3)	.	.	.	.	no
C6	C1	C7	O72	176.65(19)	.	.	.	.	no
C7	C1	C6	C5	179.1(2)	.	.	.	.	no
C2	C1	C7	O71	178.9(2)	.	.	.	.	no
C1	C2	C3	N3	178.47(19)	.	.	.	.	no
O2	C2	C3	N3	-1.8(3)	.	.	.	.	no
O2	C2	C3	C4	179.3(2)	.	.	.	.	no
C1	C2	C3	C4	-0.4(3)	.	.	.	.	no
C2	C3	C4	C5	-0.6(3)	.	.	.	.	no
N3	C3	C4	C5	-179.6(2)	.	.	.	.	no
C3	C4	C5	N5	-179.5(2)	.	.	.	.	no
C3	C4	C5	C6	0.4(3)	.	.	.	.	no
C4	C5	C6	C1	0.8(3)	.	.	.	.	no
N5	C5	C6	C1	-179.2(2)	.	.	.	.	no

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

```

#
#D   H   A   D - H   H...A   D...A   D - H...A   symm(A)
#
N1   H1A   O71   0.87(4)   2.05(4)   2.911(3)   170(3)   4_655 yes
N1   H1B   O32   0.96(5)   2.10(5)   2.990(3)   153(4)   2_745 yes
N1   H1B   O52   0.96(5)   2.55(5)   3.151(3)   121(4)   3_755 yes
N1   H1C   O2    0.90(3)   1.94(3)   2.828(3)   169(3)   1_655 yes
N1   H1C   O31   0.90(3)   2.47(3)   2.955(3)   114(2)   1_655 yes
N1   H1D   O71   0.95(3)   2.05(3)   2.965(3)   162(3)   3_655 yes
O72  H72   O2    1.10(3)   1.39(3)   2.454(2)   162(3)   . yes
C4   H4    O51   0.93(2)   2.46(2)   3.389(3)   172.0(19) 3_765 yes
C6   H6    O71   0.96(2)   2.46(2)   2.807(3)   100.9(15) . yes
  
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# End of Crystallographic Information File



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data_DNSATEA.CIF      COMPOUND (3)

_audit_creation_method      SHELXL-97
_chemical_name_systematic
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?
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_chemical_name_common      'Triethylammonium 3,5-dinitrosalicylate '
_chemical_melting_point    '411.5-413.6'
_chemical_formula_moiety    'C6 H16 N^+^, C7 H3 N2 O7^-^'
_chemical_formula_sum      'C13 H19 N3 O7'
_chemical_formula_weight    329.31

loop_
_atom_type_symbol
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_atom_type_scatter_dispersion_imag
_atom_type_scatter_source
'C' 'C' 0.0181 0.0091
'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' 0.0311 0.0180
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

'O' 'O' 0.0492 0.0322
'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting      'Monoclinic '
_symmetry_space_group_name_H-M C2/c

loop_
_symmetry_equiv_pos_as_xyz
'x, y, z'
'-x, y, -z+1/2'
'x+1/2, y+1/2, z'
'-x+1/2, y+1/2, -z+1/2'
'-x, -y, -z'
'x, -y, z-1/2'
'-x+1/2, -y+1/2, -z'
'x+1/2, -y+1/2, z-1/2'
_cell_length_a              18.827(4)
_cell_length_b              11.678(2)
_cell_length_c              14.336(5)
_cell_angle_alpha           90.00
_cell_angle_beta            102.61(3)
_cell_angle_gamma           90.00
_cell_volume                 3075.9(14)
_cell_formula_units_Z       8
_cell_measurement_temperature 130.0(1)

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_cell_measurement_reflns_used	25
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_cell_measurement_theta_max	30
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_exptl_crystal_colour	'Yellow '
_exptl_crystal_size_max	0.50
_exptl_crystal_size_mid	0.40
_exptl_crystal_size_min	0.30
_exptl_crystal_density_meas	?
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_exptl_crystal_density_method	'not measured'
_exptl_crystal_F_000	1392
_exptl_absorpt_coefficient_mu	0.996
_exptl_absorpt_correction_type	None
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_exptl_special_details	
;	
?	
;	
_diffn_ambient_temperature	130.0(1)
_diffn_radiation_wavelength	1.54180
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_diffn_radiation_source	'fine-focus sealed tube'
_diffn_radiation_monochromator	Ni Filtered
_diffn_measurement_device_type	'Enraf-Nonius CAD-4 '
_diffn_measurement_method	?
_diffn_detector_area_resol_mean	?
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_diffn_standards_interval_count	?
_diffn_standards_interval_time	160
_diffn_standards_decay_%	3.0
_diffn_reflns_number	2975
_diffn_reflns_av_R_equivalents	0.0428
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_diffn_reflns_limit_h_min	0
_diffn_reflns_limit_h_max	22
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_diffn_reflns_limit_k_max	14
_diffn_reflns_limit_l_min	-17
_diffn_reflns_limit_l_max	15
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_reflns_number_gt	2644
_reflns_threshold_expression	'I >2\s(I) '
_computing_data_collection	?
_computing_cell_refinement	?
_computing_data_reduction	?
_computing_structure_solution	'SHELXS-97 (Sheldrick, 1997)'
_computing_structure_refinement	'SHELXL-97 (Sheldrick, 1997)'
_computing_molecular_graphics	'PLATON for Windows (Spek, 1999)'

\_computing\_publication\_material ?

\_refine\_special\_details

;  
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  $F^2 > 2\sigma(F^2)$  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)+(0.1147P)^2+3.9023P] 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 'SHELXL-97 (Sheldrick, 1997)'  
\_refine\_ls\_extinction\_coef 0.0014(2)  
\_refine\_ls\_extinction\_expression  
'Fc^k=kFc[1+0.001xFc^2^l^3^/sin(2\q)]^-1/4^'  
\_refine\_ls\_number\_reflns 2892  
\_refine\_ls\_number\_parameters 236  
\_refine\_ls\_number\_restraints 0  
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\_refine\_ls\_wR\_factor\_ref 0.1759  
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\_refine\_ls\_goodness\_of\_fit\_ref 1.071  
  
\_refine\_ls\_restrained\_S\_all 1.071  
\_refine\_ls\_shift/su\_max 0.865  
\_refine\_ls\_shift/su\_mean 0.032

loop\_

# 9. ATOMIC COORDINATES AND THERMAL PARAMETERS

loop\_

\_atom\_site\_label  
\_atom\_site\_type\_symbol  
\_atom\_site\_thermal\_displace\_type  
\_atom\_site\_fract\_x  
\_atom\_site\_fract\_y  
\_atom\_site\_fract\_z  
\_atom\_site\_occupancy  
\_atom\_site\_U\_iso\_or\_equiv  
O2 O Uani 0.08152(9) 0.60800(16) 0.19112(12) 1.000 0.0394(5)  
O31 O Uani 0.0277(2) 0.8165(3) 0.1135(7) 0.579(12) 0.070(2)  
O32 O Uani -0.0882(7) 0.8336(12) 0.0924(10) 0.579(12) 0.0397(19)  
O51 O Uani -0.23362(8) 0.52404(13) -0.04274(11) 1.000 0.0335(5)  
O52 O Uani -0.19475(8) 0.34956(13) -0.02647(12) 1.000 0.0367(5)  
O71 O Uani 0.05637(8) 0.26826(13) 0.13854(12) 1.000 0.0365(5)

O72	O	Uani	0.12481(8)	0.41256(17)	0.21045(12)	1.000	0.0395(5)
N3	N	Uani	-0.03113(13)	0.77321(18)	0.1130(2)	1.000	0.0507(8)
N5	N	Uani	-0.18563(9)	0.45333(14)	-0.01412(11)	1.000	0.0238(5)
C1	C	Uani	0.00570(10)	0.45450(16)	0.12371(13)	1.000	0.0206(5)
C2	C	Uani	0.01958(10)	0.57435(17)	0.14190(14)	1.000	0.0244(6)
C3	C	Uani	-0.03894(11)	0.64895(16)	0.10187(15)	1.000	0.0263(6)
C4	C	Uani	-0.10473(10)	0.61038(15)	0.05205(14)	1.000	0.0211(5)
C5	C	Uani	-0.11553(10)	0.49338(15)	0.03814(12)	1.000	0.0187(5)
C6	C	Uani	-0.06032(10)	0.41577(15)	0.07289(13)	1.000	0.0198(5)
C7	C	Uani	0.06607(11)	0.37153(19)	0.16009(15)	1.000	0.0286(6)
O31'	O	Uani	0.0223(3)	0.8015(4)	0.1898(7)	0.421(12)	0.062(2)
O32'	O	Uiso	-0.0769(11)	0.8316(16)	0.0850(13)	0.421(12)	0.062(4)
N1	N	Uani	-0.34928(9)	0.40692(13)	-0.30011(11)	1.000	0.0211(4)
C8	C	Uani	-0.27315(11)	0.36534(17)	-0.26359(15)	1.000	0.0275(6)
C9	C	Uani	-0.24560(13)	0.2922(2)	-0.33504(17)	1.000	0.0344(7)
C10	C	Uani	-0.37881(11)	0.46618(16)	-0.22308(14)	1.000	0.0268(6)
C11	C	Uani	-0.39710(13)	0.38249(19)	-0.15122(16)	1.000	0.0336(6)
C12	C	Uani	-0.35775(12)	0.48048(16)	-0.38840(14)	1.000	0.0278(6)
C13	C	Uani	-0.31491(15)	0.59116(19)	-0.37157(17)	1.000	0.0395(7)
H72	H	Uiso	0.115(3)	0.521(5)	0.206(4)	1.000	0.136(17)
H4	H	Uiso	-0.141900	0.661800	0.027700	1.000	0.0250
H6	H	Uiso	-0.068000	0.337800	0.061800	1.000	0.0240
H1	H	Uiso	-0.3791(14)	0.349(2)	-0.3177(18)	1.000	0.027(6)
H8A	H	Uiso	-0.271300	0.321200	-0.205800	1.000	0.0330
H8B	H	Uiso	-0.241200	0.430800	-0.247100	1.000	0.0330
H9A	H	Uiso	-0.196400	0.269000	-0.308400	1.000	0.0520
H9B	H	Uiso	-0.247000	0.335400	-0.392400	1.000	0.0520
H9C	H	Uiso	-0.275800	0.225500	-0.349800	1.000	0.0520
H10A	H	Uiso	-0.422300	0.508600	-0.252300	1.000	0.0320
H10B	H	Uiso	-0.343000	0.520500	-0.190200	1.000	0.0320
H11A	H	Uiso	-0.415400	0.423600	-0.103500	1.000	0.0500
H11B	H	Uiso	-0.354100	0.341100	-0.121400	1.000	0.0500
H11C	H	Uiso	-0.433400	0.329700	-0.183300	1.000	0.0500
H12A	H	Uiso	-0.408900	0.498500	-0.411400	1.000	0.0330
H12B	H	Uiso	-0.341900	0.437100	-0.437900	1.000	0.0330
H13A	H	Uiso	-0.323600	0.635000	-0.429600	1.000	0.0590
H13B	H	Uiso	-0.263900	0.574100	-0.352100	1.000	0.0590
H13C	H	Uiso	-0.330000	0.634500	-0.322400	1.000	0.0590

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

O2	0.0297(8)	0.0452(10)	0.0425(10)	-0.0138(7)	0.0060(7)	-0.0100(7)
O31	0.047(2)	0.0259(17)	0.134(7)	-0.007(2)	0.010(2)	-0.0201(15)
O32	0.053(3)	0.019(3)	0.052(4)	-0.007(2)	0.022(3)	0.005(2)
O51	0.0249(8)	0.0320(8)	0.0399(9)	0.0031(6)	-0.0012(6)	0.0067(6)
O52	0.0339(8)	0.0223(8)	0.0501(10)	-0.0107(7)	0.0010(7)	-0.0066(6)
O71	0.0324(8)	0.0260(8)	0.0516(10)	0.0161(7)	0.0104(7)	0.0112(6)
O72	0.0246(8)	0.0548(11)	0.0369(9)	0.0056(7)	0.0020(6)	0.0071(7)
N3	0.0450(13)	0.0177(10)	0.0923(18)	-0.0200(11)	0.0214(12)	-0.0089(10)
N5	0.0241(8)	0.0216(8)	0.0253(8)	-0.0022(6)	0.0045(6)	0.0008(6)
C1	0.0241(9)	0.0194(9)	0.0204(9)	0.0028(7)	0.0097(7)	0.0026(7)

C2	0.0231(10)	0.0257(10)	0.0268(10)	-0.0076(8)	0.0106(8)	-0.0059(8)
C3	0.0325(11)	0.0123(9)	0.0380(11)	-0.0067(8)	0.0162(9)	-0.0034(8)
C4	0.0252(9)	0.0135(8)	0.0270(10)	-0.0004(7)	0.0108(7)	0.0032(7)
C5	0.0226(9)	0.0149(8)	0.0199(9)	0.0001(6)	0.0077(7)	-0.0010(7)
C6	0.0249(9)	0.0133(8)	0.0233(9)	0.0019(7)	0.0101(7)	0.0002(7)
C7	0.0266(10)	0.0344(11)	0.0275(10)	0.0111(8)	0.0118(8)	0.0075(8)
O31'	0.061(3)	0.025(2)	0.088(6)	-0.018(3)	-0.009(3)	-0.013(2)
N1	0.0239(8)	0.0135(7)	0.0251(8)	-0.0020(6)	0.0035(6)	-0.0037(6)
C8	0.0271(10)	0.0215(9)	0.0317(11)	-0.0013(8)	0.0019(8)	-0.0010(8)
C9	0.0345(11)	0.0316(11)	0.0408(13)	0.0040(9)	0.0163(9)	0.0059(9)
C10	0.0328(10)	0.0181(9)	0.0304(10)	-0.0038(8)	0.0089(8)	-0.0009(8)
C11	0.0415(12)	0.0292(10)	0.0335(11)	-0.0029(9)	0.0157(9)	-0.0067(9)
C12	0.0375(11)	0.0187(9)	0.0251(10)	0.0013(7)	0.0021(8)	-0.0009(8)
C13	0.0602(15)	0.0245(11)	0.0340(12)	0.0040(9)	0.0107(10)	-0.0127(10)

\_geom\_special\_details

;

Bond distances, angles etc. have been calculated using the rounded fractional coordinates. All esds are estimated from the variances of the (full) variance-covariance matrix. The cell esds are taken into account in the estimation of distances, angles and torsion angles

;

loop\_

\_geom\_bond\_atom\_site\_label\_1

\_geom\_bond\_atom\_site\_label\_2

\_geom\_bond\_distance

\_geom\_bond\_site\_symmetry\_1

\_geom\_bond\_site\_symmetry\_2

\_geom\_bond\_publ\_flag

O2	C2	1.286(3)	.	.	yes
O31	N3	1.216(5)	.	.	yes
O31'	N3	1.360(9)	.	.	yes
O32	N3	1.265(14)	.	.	yes
O32'	N3	1.10(2)	.	.	yes
O51	N5	1.227(2)	.	.	yes
O52	N5	1.231(2)	.	.	yes
O71	C7	1.248(3)	.	.	yes
O72	C7	1.275(3)	.	.	yes
O2	H72	1.19(6)	.	.	no
O72	H72	1.28(6)	.	.	no
N3	C3	1.464(3)	.	.	yes
N5	C5	1.446(3)	.	.	yes
N1	C12	1.509(3)	.	.	yes
N1	C10	1.508(3)	.	.	yes
N1	C8	1.496(3)	.	.	yes
N1	H1	0.88(2)	.	.	no
C1	C6	1.373(3)	.	.	no
C1	C7	1.498(3)	.	.	no
C1	C2	1.437(3)	.	.	no
C2	C3	1.423(3)	.	.	no
C3	C4	1.364(3)	.	.	no
C4	C5	1.389(3)	.	.	no
C5	C6	1.387(3)	.	.	no
C4	H4	0.9303	.	.	no

C6	H6	0.9302	.	.	no
C8	C9	1.510(3)	.	.	no
C10	C11	1.513(3)	.	.	no
C12	C13	1.515(3)	.	.	no
C8	H8A	0.9699	.	.	no
C8	H8B	0.9695	.	.	no
C9	H9A	0.9606	.	.	no
C9	H9B	0.9602	.	.	no
C9	H9C	0.9603	.	.	no
C10	H10A	0.9703	.	.	no
C10	H10B	0.9697	.	.	no
C11	H11A	0.9592	.	.	no
C11	H11B	0.9595	.	.	no
C11	H11C	0.9599	.	.	no
C12	H12A	0.9704	.	.	no
C12	H12B	0.9704	.	.	no
C13	H13A	0.9600	.	.	no
C13	H13B	0.9607	.	.	no
C13	H13C	0.9598	.	.	no

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\_2

\_geom\_angle\_site\_symmetry\_3

\_geom\_angle\_publ\_flag

C2	O2	H72	103(3)	.	.	.	no
C7	O72	H72	104(3)	.	.	.	no
O31	N3	O32	119.7(7)	.	.	.	yes
O31'	N3	C3	111.6(3)	.	.	.	yes
O31	N3	C3	118.8(3)	.	.	.	yes
O32	N3	C3	117.9(7)	.	.	.	yes
O31'	N3	O32'	121.7(10)	.	.	.	yes
O32'	N3	C3	121.4(10)	.	.	.	yes
O51	N5	C5	118.55(16)	.	.	.	yes
O51	N5	O52	123.03(17)	.	.	.	yes
O52	N5	C5	118.41(16)	.	.	.	yes
C8	N1	C10	111.77(15)	.	.	.	yes
C8	N1	C12	113.34(16)	.	.	.	yes
C10	N1	C12	111.38(15)	.	.	.	yes
C10	N1	H1	104.4(17)	.	.	.	no
C12	N1	H1	104.7(16)	.	.	.	no
C8	N1	H1	110.7(17)	.	.	.	no
C6	C1	C7	120.13(17)	.	.	.	no
C2	C1	C7	118.21(17)	.	.	.	no
C2	C1	C6	121.66(17)	.	.	.	no
C1	C2	C3	115.39(17)	.	.	.	no
O2	C2	C3	124.31(19)	.	.	.	yes
O2	C2	C1	120.30(18)	.	.	.	yes
N3	C3	C2	120.8(2)	.	.	.	yes
N3	C3	C4	116.27(19)	.	.	.	yes
C2	C3	C4	122.92(18)	.	.	.	no
C3	C4	C5	119.18(18)	.	.	.	no
N5	C5	C4	118.75(17)	.	.	.	yes

C4	C5	C6	121.09(17)	.	.	.	no
N5	C5	C6	120.16(16)	.	.	.	yes
C1	C6	C5	119.73(17)	.	.	.	no
O71	C7	O72	124.4(2)	.	.	.	yes
O71	C7	C1	118.67(19)	.	.	.	yes
O72	C7	C1	116.9(2)	.	.	.	yes
C5	C4	H4	120.42	.	.	.	no
C3	C4	H4	120.40	.	.	.	no
C5	C6	H6	120.13	.	.	.	no
C1	C6	H6	120.14	.	.	.	no
N1	C8	C9	113.02(18)	.	.	.	yes
N1	C10	C11	112.15(16)	.	.	.	yes
N1	C12	C13	113.39(17)	.	.	.	yes
N1	C8	H8A	108.94	.	.	.	no
N1	C8	H8B	109.00	.	.	.	no
C9	C8	H8A	108.98	.	.	.	no
C9	C8	H8B	108.95	.	.	.	no
H8A	C8	H8B	107.82	.	.	.	no
C8	C9	H9A	109.52	.	.	.	no
C8	C9	H9B	109.54	.	.	.	no
C8	C9	H9C	109.50	.	.	.	no
H9A	C9	H9B	109.42	.	.	.	no
H9A	C9	H9C	109.39	.	.	.	no
H9B	C9	H9C	109.46	.	.	.	no
N1	C10	H10A	109.23	.	.	.	no
N1	C10	H10B	109.13	.	.	.	no
C11	C10	H10A	109.13	.	.	.	no
C11	C10	H10B	109.21	.	.	.	no
H10A	C10	H10B	107.91	.	.	.	no
C10	C11	H11A	109.44	.	.	.	no
C10	C11	H11B	109.48	.	.	.	no
C10	C11	H11C	109.43	.	.	.	no
H11A	C11	H11B	109.50	.	.	.	no
H11A	C11	H11C	109.52	.	.	.	no
H11B	C11	H11C	109.46	.	.	.	no
N1	C12	H12A	108.89	.	.	.	no
N1	C12	H12B	108.85	.	.	.	no
C13	C12	H12A	108.87	.	.	.	no
C13	C12	H12B	108.90	.	.	.	no
H12A	C12	H12B	107.79	.	.	.	no
C12	C13	H13A	109.45	.	.	.	no
C12	C13	H13B	109.46	.	.	.	no
C12	C13	H13C	109.53	.	.	.	no
H13A	C13	H13B	109.44	.	.	.	no
H13A	C13	H13C	109.48	.	.	.	no
H13B	C13	H13C	109.47	.	.	.	no

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\_geom\_torsion

\_geom\_torsion\_site\_symmetry\_1

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\_geom\_torsion\_site\_symmetry\_4

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O31	N3	C3	C2	34.6(6)	.	.	.	.	no
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O51	N5	C5	C6	178.99(18)	.	.	.	.	no
O51	N5	C5	C4	-1.4(3)	.	.	.	.	no
O52	N5	C5	C4	179.76(18)	.	.	.	.	no
O52	N5	C5	C6	0.2(3)	.	.	.	.	no
C12	N1	C8	C9	-60.8(2)	.	.	.	.	no
C12	N1	C10	C11	158.62(18)	.	.	.	.	no
C8	N1	C12	C13	-63.8(2)	.	.	.	.	no
C10	N1	C12	C13	63.3(2)	.	.	.	.	no
C10	N1	C8	C9	172.39(17)	.	.	.	.	no
C8	N1	C10	C11	-73.5(2)	.	.	.	.	no
C6	C1	C2	C3	-1.4(3)	.	.	.	.	no
C7	C1	C2	O2	-2.5(3)	.	.	.	.	no
C2	C1	C6	C5	-0.2(3)	.	.	.	.	no
C6	C1	C2	O2	178.80(19)	.	.	.	.	no
C7	C1	C6	C5	-178.95(18)	.	.	.	.	no
C2	C1	C7	O71	-174.69(19)	.	.	.	.	no
C2	C1	C7	O72	4.4(3)	.	.	.	.	no
C6	C1	C7	O71	4.1(3)	.	.	.	.	no
C7	C1	C2	C3	177.36(18)	.	.	.	.	no
C6	C1	C7	O72	-176.88(19)	.	.	.	.	no
C1	C2	C3	N3	-178.4(2)	.	.	.	.	no
C1	C2	C3	C4	2.0(3)	.	.	.	.	no
O2	C2	C3	C4	-178.2(2)	.	.	.	.	no
O2	C2	C3	N3	1.5(3)	.	.	.	.	no
C2	C3	C4	C5	-0.9(3)	.	.	.	.	no
N3	C3	C4	C5	179.42(19)	.	.	.	.	no
C3	C4	C5	C6	-0.9(3)	.	.	.	.	no
C3	C4	C5	N5	179.53(17)	.	.	.	.	no
C4	C5	C6	C1	1.4(3)	.	.	.	.	no
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loop\_

loop\_

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geom\_hbond\_angle\_DHA

geom\_hbond\_site\_symmetry\_A

geom\_hbond\_publ\_flag

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#D H A D - H H...A D...A D - H...A symm(A)

#

N1	H1	O71	0.88(2)	1.85(2)	2.726(2)	177(2)	8_454	yes
C4	H4	O32	0.9303	2.3448	2.674(14)	100.36	.	yes
C12	H12A	O31	0.9704	2.5309	3.209(5)	126.92	8_464	yes

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# End of Crystallographic Information File

data\_DNSAMA: 478gs67: COMPOUND (2) Smith et al, Aust. J. Chem., 2001.

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_computing_molecular_graphics ?

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\_computing\_publication\_material ?

\_refine\_special\_details

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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  $F^2 > 2\sigma(F^2)$  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.

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loop\_

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O31 O -0.15849(9) 0.8000(3) 0.0272(2) 0.0543(5) Uani 1 1 d . . .  
O32 O -0.13583(10) 0.7416(3) -0.2591(3) 0.0585(5) Uani 1 1 d . . .  
O51 O 0.09793(11) 0.9849(3) -0.4507(3) 0.0639(6) Uani 1 1 d . . .  
O52 O 0.19515(9) 0.9131(3) -0.2955(3) 0.0554(5) Uani 1 1 d . . .

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O71 O 0.17378(8) 0.6581(2) 0.3233(2) 0.0486(5) Uani 1 1 d . . .
O72 O 0.06313(9) 0.5968(3) 0.4283(2) 0.0494(5) Uani 1 1 d . . .
N1 N -0.19435(11) 0.6394(3) 0.3803(3) 0.0405(5) Uani 1 1 d . . .
N3 N -0.11567(9) 0.7700(2) -0.0999(2) 0.0352(4) Uani 1 1 d . . .
N5 N 0.12801(10) 0.9199(3) -0.3158(3) 0.0405(5) Uani 1 1 d . . .
C1 C 0.07092(10) 0.7225(3) 0.1283(3) 0.0300(4) Uani 1 1 d . . .
C2 C -0.00842(11) 0.7150(3) 0.1097(3) 0.0311(4) Uani 1 1 d . . .
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C4 C 0.00663(12) 0.8398(3) -0.2026(3) 0.0328(4) Uani 1 1 d . . .
C5 C 0.08196(11) 0.8473(3) -0.1726(3) 0.0327(5) Uani 1 1 d . . .
C6 C 0.11418(11) 0.7874(3) -0.0104(3) 0.0315(4) Uani 1 1 d . . .
C7 C 0.10715(11) 0.6571(3) 0.3006(3) 0.0353(5) Uani 1 1 d . . .
C8 C -0.22689(16) 0.5058(3) 0.2715(4) 0.0523(6) Uani 1 1 d . . .
H4 H -0.0155 0.8779 -0.3173 0.037 Uiso 1 1 d . . .
H6 H 0.1661 0.7905 0.0062 0.038 Uiso 1 1 d . . .
H11 H -0.149(2) 0.669(4) 0.345(5) 0.070(10) Uiso 1 1 d . . .
H12 H -0.1913(17) 0.616(4) 0.496(5) 0.063(9) Uiso 1 1 d . . .
H13 H -0.223(2) 0.723(5) 0.360(5) 0.067(10) Uiso 1 1 d . . .
H72 H 0.014(2) 0.608(5) 0.370(5) 0.090(12) Uiso 1 1 d . . .
H81 H -0.2651 0.5485 0.1909 0.061 Uiso 1 1 d . . .
H82 H -0.2481 0.4239 0.3498 0.061 Uiso 1 1 d . . .
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loop\_

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O32 0.0466(10) 0.0840(14) 0.0447(10) -0.0083(9) -0.0180(8) -0.0035(9)
O51 0.0630(12) 0.0823(15) 0.0464(10) 0.0331(10) 0.0088(8) 0.0056(10)
O52 0.0395(9) 0.0761(14) 0.0507(10) 0.0107(9) 0.0139(7) -0.0065(9)
O71 0.0317(8) 0.0679(12) 0.0460(9) 0.0134(8) -0.0091(7) -0.0002(7)
O72 0.0371(8) 0.0745(12) 0.0365(8) 0.0233(8) -0.0026(6) -0.0005(8)
N1 0.0309(10) 0.0584(13) 0.0322(10) -0.0039(9) 0.0002(7) -0.0020(9)
N3 0.0306(8) 0.0388(10) 0.0363(9) 0.0060(7) -0.0055(7) -0.0013(7)
N5 0.0426(10) 0.0446(11) 0.0344(9) 0.0074(8) 0.0104(7) -0.0004(8)
C1 0.0279(9) 0.0358(10) 0.0262(9) 0.0018(8) -0.0012(7) 0.0008(8)
C2 0.0285(9) 0.0358(11) 0.0291(9) 0.0034(8) 0.0005(7) 0.0000(8)
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C5 0.0354(10) 0.0356(10) 0.0273(9) 0.0023(8) 0.0066(7) 0.0002(8)
C6 0.0273(9) 0.0365(10) 0.0308(9) 0.0002(8) 0.0010(7) 0.0007(8)
C7 0.0315(10) 0.0422(12) 0.0323(10) 0.0051(9) -0.0027(8) 0.0010(8)
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\_geom\_special\_details

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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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N1 H13 0.87(4) . ?  
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C8 N1 H13 104(2) . . ?  
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 C5 C6 H6 121 . . ?  
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 O71 C7 C1 122.58(19) . . ?  
 O72 C7 C1 116.39(17) . . ?  
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 N1 C8 H82 111 . . ?  
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 \_geom\_torsion\_atom\_site\_label\_2  
 \_geom\_torsion\_atom\_site\_label\_3  
 \_geom\_torsion\_atom\_site\_label\_4  
 \_geom\_torsion  
 \_geom\_torsion\_site\_symmetry\_1  
 \_geom\_torsion\_site\_symmetry\_2  
 \_geom\_torsion\_site\_symmetry\_3  
 \_geom\_torsion\_site\_symmetry\_4  
 \_geom\_torsion\_publ\_flag

O31	N3	C3	C4	-142.5(2)	.	.	.	.	no
O32	N3	C3	C2	-143.9(2)	.	.	.	.	no
O31	N3	C3	C2	39.2(3)	.	.	.	.	no
O32	N3	C3	C4	34.4(3)	.	.	.	.	no
O51	N5	C5	C6	-173.8(2)	.	.	.	.	no
O51	N5	C5	C4	6.3(4)	.	.	.	.	no
O52	N5	C5	C6	6.2(4)	.	.	.	.	no
O52	N5	C5	C4	-173.8(2)	.	.	.	.	no
C6	C1	C7	O72	179.9(4)	.	.	.	.	no
C7	C1	C2	C3	-178.3(2)	.	.	.	.	no
C6	C1	C2	C3	1.1(3)	.	.	.	.	no
C7	C1	C2	O2	0.0(3)	.	.	.	.	no
C6	C1	C7	O71	-0.3(4)	.	.	.	.	no
C6	C1	C2	O2	179.4(2)	.	.	.	.	no
C2	C1	C7	O72	-0.7(3)	.	.	.	.	no
C2	C1	C6	C5	-0.1(4)	.	.	.	.	no
C2	C1	C7	O71	179.1(2)	.	.	.	.	no
C7	C1	C6	C5	179.3(2)	.	.	.	.	no
O2	C2	C3	C4	-178.6(2)	.	.	.	.	no

C1	C2	C3	N3	177.8(2)	.	.	.	.	no
O2	C2	C3	N3	-0.5(4)	.	.	.	.	no
C1	C2	C3	C4	-0.3(3)	.	.	.	.	no
C2	C3	C4	C5	-1.5(4)	.	.	.	.	no
N3	C3	C4	C5	-179.7(2)	.	.	.	.	no
C3	C4	C5	N5	-177.5(2)	.	.	.	.	no
C3	C4	C5	C6	2.6(4)	.	.	.	.	no
N5	C5	C6	C1	178.2(2)	.	.	.	.	no
C4	C5	C6	C1	-1.8(4)	.	.	.	.	no

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

#

#D H A D - H H...A D...A D - H...A symm(A)

#

N1	H11	O2	0.89(4)	1.99(4)	2.845(3)	160(3)	.	yes
N1	H11	O31	0.89(4)	2.52(4)	2.928(3)	108(3)	.	yes
N1	H12	O32	0.85(4)	2.26(3)	2.913(3)	133(3)	1_556	yes
N1	H12	O71	0.85(4)	2.58(3)	3.231(3)	133(3)	3_566	yes
N1	H12	O52	0.85(4)	2.57(3)	3.113(3)	123(3)	4_466	yes
N1	H13	O71	0.86(4)	2.12(4)	2.923(3)	155(4)	4_465	yes
O72	H72	O2	0.99(4)	1.52(4)	2.481(2)	163(4)	.	yes
C4	H4	O51	0.9647	2.4911	3.426(3)	163.29	3_574	yes

# End of Crystallographic Information File