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 54, 2001

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: ajc@publish.csiro.au

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

www.publish.csiro.au/journals/ajc

10.1071/CH01183_AC

Accessory Publication: *Aust. J. Chem.*, 2001, **54**(11), 683-684.

```
data_global

_audit_creation_method            'SHELXL-97 plus manual editing'

_publ_contact_author_name         'Andrew D.Bond'
_publ_contact_author_address
;
 Department of Chemistry
 University of Cambridge
 Lensfield Road
 Cambridge
 CB2 1EW
 UK
;
_publ_contact_author_email        adb29@cam.ac.uk
_publ_contact_author_fax          '(01223) 336362'
_publ_contact_author_phone        '(01223) 336352'
_publ_requested_journal           'Aust. J. Chem.'

data_I

_chemical_name_systematic
;
 1,3,3-trimethyl-2-oxabicyclo(2.2.2)octane
;
_chemical_name_common             '1,8-Cineole; Eucalyptol; Cajuputol'
_chemical_melting_point           274
_chemical_formula_moiety          'C10 H18 O'
_chemical_formula_sum             'C10 H18 O'
_chemical_formula_weight          154.24

loop_
 _atom_type_symbol
 _atom_type_description
 _atom_type_scat_dispersion_real
 _atom_type_scat_dispersion_imag
 _atom_type_scat_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'
 'O'  'O'   0.0106   0.0060
 'International Tables Vol C Tables 4.2.6.8 and 6.1.1.4'

_symmetry_cell_setting            monoclinic
_symmetry_space_group_name_H-M    P21/m

loop_
 _symmetry_equiv_pos_as_xyz
 'x, y, z'
 '-x, y+1/2, -z'
 '-x, -y, -z'
 'x, -y-1/2, z'

_cell_length_a                    6.0488(3)
_cell_length_b                    10.7165(9)
```

```
_cell_length_c                    7.8433(7)
_cell_angle_alpha                 90.00
_cell_angle_beta                  110.553(5)
_cell_angle_gamma                 90.00
_cell_volume                      476.06(6)
_cell_formula_units_Z             2
_cell_measurement_temperature     265(2)
_cell_measurement_reflns_used     2096
_cell_measurement_theta_min       1.00
_cell_measurement_theta_max       27.48

_exptl_crystal_description        cylinder
_exptl_crystal_colour             colourless
_exptl_crystal_size_length        0.35
_exptl_crystal_size_rad           0.15
_exptl_crystal_density_meas       ?
_exptl_crystal_density_diffrn     1.076
_exptl_crystal_density_method     'not measured'
_exptl_crystal_F_000              172
_exptl_absorpt_coefficient_mu     0.067
_exptl_absorpt_correction_type    none
_exptl_absorpt_correction_T_min   ?
_exptl_absorpt_correction_T_max   ?
_exptl_absorpt_process_details    ?

_exptl_special_details
;
 Grown in-situ at 268K in a 0.3 mm Lindemann capillary tube.
;

_diffrn_ambient_temperature       265(2)
_diffrn_radiation_wavelength      0.71073
_diffrn_radiation_type            MoK\a
_diffrn_radiation_source          'fine-focus sealed tube'
_diffrn_measurement_device_type   'Nonius Kappa CCD'
_diffrn_measurement_method        'Thin slice \w and \f scans'
_diffrn_standards_number          0
_diffrn_reflns_number             2063
_diffrn_reflns_av_R_equivalents   0.0330
_diffrn_reflns_av_sigmaI/netI     0.0435
_diffrn_reflns_limit_h_min        -7
_diffrn_reflns_limit_h_max        7
_diffrn_reflns_limit_k_min        -13
_diffrn_reflns_limit_k_max        13
_diffrn_reflns_limit_l_min        -10
_diffrn_reflns_limit_l_max        10
_diffrn_reflns_theta_min          3.60
_diffrn_reflns_theta_max          27.62
_diffrn_measured_fraction_theta_max    0.966
_diffrn_reflns_theta_full              27.62
_diffrn_measured_fraction_theta_full   0.966
_reflns_number_total              1123
_reflns_number_gt                 688
_reflns_threshold_expression      >2sigma(I)

_computing_data_collection        'Collect (Nonius B.V. 1998)'
_computing_cell_refinement        'HKL Scalepack (Otwinowski & Minor 1997)'
_computing_data_reduction
;
 HKL Denzo and Scalepack (Otwinowski & Minor 1997)
;
```

```
_computing_structure_solution     'SIR-92 (Altomare et al. 1994)'
_computing_structure_refinement   'SHELXL-97 (Sheldrick 1997)'
_computing_publication_material   'SHELXL-97 (Sheldrick 1997)'

_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^ > 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.
;

_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^)+(0.0510P)^2^+0.0855P] 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
_refine_ls_extinction_coef        0.42(5)
_refine_ls_extinction_expression
 'Fc^*^=kFc[1+0.001xFc^2^\l^3^/sin(2\q)]^-1/4^'
_refine_ls_number_reflns          1123
_refine_ls_number_parameters      70
_refine_ls_number_restraints      0
_refine_ls_R_factor_all           0.0870
_refine_ls_R_factor_gt            0.0466
_refine_ls_wR_factor_ref          0.1369
_refine_ls_wR_factor_gt           0.1076
_refine_ls_goodness_of_fit_ref    1.029
_refine_ls_restrained_S_all       1.029
_refine_ls_shift/su_max           0.000
_refine_ls_shift/su_mean          0.000
_refine_diff_density_max          0.143
_refine_diff_density_min          -0.132
_refine_diff_density_rms          0.028

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
C1 C 0.3909(4) 0.2500 1.0137(3) 0.0482(6) Uani 1 2 d S . .
C2 C 0.2421(3) 0.36495(16) 1.0071(2) 0.0639(6) Uani 1 1 d . . .
H2A H 0.3347 0.4393 1.0100 0.091(4) Uiso 1 1 calc R . .
H2B H 0.1939 0.3663 1.1127 0.091(4) Uiso 1 1 calc R . .
```

```
C3 C 0.0248(3) 0.36431(17) 0.8343(3) 0.0673(6) Uani 1 1 d . . .
H3A H 0.0204 0.4396 0.7646 0.082(4) Uiso 1 1 calc R . .
H3B H -0.1172 0.3613 0.8649 0.082(4) Uiso 1 1 calc R . .
C4 C 0.0375(4) 0.2500 0.7229(3) 0.0594(7) Uani 1 2 d S . .
H4 H -0.0967 0.2500 0.6077 0.073(7) Uiso 1 2 calc SR . .
C7 C 0.6189(5) 0.2500 1.1743(4) 0.0790(9) Uani 1 2 d S . .
H7A H 0.715(4) 0.1741(19) 1.169(3) 0.099(5) Uiso 1 1 d . . .
H7C H 0.592(5) 0.2500 1.288(4) 0.099(5) Uiso 1 2 d S . .
C8 C 0.2693(3) 0.2500 0.6844(3) 0.0517(6) Uani 1 2 d S . .
C9 C 0.2983(3) 0.1350(2) 0.5818(2) 0.0801(7) Uani 1 1 d . . .
H9A H 0.2939 0.0617 0.6510 0.108(4) Uiso 1 1 calc R . .
H9B H 0.4469 0.1388 0.5635 0.108(4) Uiso 1 1 calc R . .
H9C H 0.1724 0.1315 0.4659 0.108(4) Uiso 1 1 calc R . .
O11 O 0.4618(2) 0.2500 0.85718(18) 0.0566(5) Uani 1 2 d S . .

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
C1 0.0518(11) 0.0582(14) 0.0374(12) 0.000 0.0192(9) 0.000
C2 0.0736(11) 0.0552(11) 0.0685(12) -0.0096(9) 0.0319(10) 0.0001(8)
C3 0.0594(9) 0.0686(12) 0.0799(13) 0.0097(10) 0.0321(9) 0.0159(8)
C4 0.0420(11) 0.0758(17) 0.0531(14) 0.000 0.0078(10) 0.000
C7 0.0718(17) 0.112(3) 0.0424(15) 0.000 0.0071(14) 0.000
C8 0.0476(11) 0.0698(15) 0.0337(12) 0.000 0.0092(9) 0.000
C9 0.0841(13) 0.1007(16) 0.0573(12) -0.0200(11) 0.0269(10) -0.0013(11)
O11 0.0396(8) 0.0924(13) 0.0382(9) 0.000 0.0141(6) 0.000

_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
C1 O11 1.435(2) . N
C1 C7 1.507(3) . N
C1 C2 1.516(2) 4_565 N
C1 C2 1.516(2) . N
C2 C3 1.521(3) . N
C2 H2A 0.9700 . N
C2 H2B 0.9700 . N
C3 C4 1.522(2) . N
C3 H3A 0.9700 . N
C3 H3B 0.9700 . N
C4 C3 1.522(2) 4_565 N
C4 C8 1.534(3) . N
C4 H4 0.9800 . N
```

```
C7 H7A 1.01(2) . N
C7 H7C 0.97(3) . N
C8 O11 1.444(2) . N
C8 C9 1.516(2) . N
C8 C9 1.516(2) 4_565 N
C9 H9A 0.9600 . N
C9 H9B 0.9600 . N
C9 H9C 0.9600 . N

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
O11 C1 C7 104.73(18) . . N
O11 C1 C2 108.84(12) . 4_565 N
C7 C1 C2 112.76(13) . 4_565 N
O11 C1 C2 108.84(12) . . N
C7 C1 C2 112.76(13) . . N
C2 C1 C2 108.72(18) 4_565 . N
C1 C2 C3 110.10(15) . . N
C1 C2 H2A 109.6 . . N
C3 C2 H2A 109.6 . . N
C1 C2 H2B 109.6 . . N
C3 C2 H2B 109.6 . . N
H2A C2 H2B 108.2 . . N
C2 C3 C4 108.26(14) . . N
C2 C3 H3A 110.0 . . N
C4 C3 H3A 110.0 . . N
C2 C3 H3B 110.0 . . N
C4 C3 H3B 110.0 . . N
H3A C3 H3B 108.4 . . N
C3 C4 C3 107.2(2) . 4_565 N
C3 C4 C8 110.28(12) . . N
C3 C4 C8 110.28(12) 4_565 . N
C3 C4 H4 109.7 . . N
C3 C4 H4 109.7 4_565 . N
C8 C4 H4 109.7 . . N
C1 C7 H7A 109.6(12) . . N
C1 C7 H7C 111.8(19) . . N
H7A C7 H7C 109.3(17) . . N
O11 C8 C9 107.02(12) . . N
O11 C8 C9 107.02(12) . 4_565 N
C9 C8 C9 108.8(2) . 4_565 N
O11 C8 C4 107.87(17) . . N
C9 C8 C4 112.87(12) . . N
C9 C8 C4 112.87(12) 4_565 . N
C8 C9 H9A 109.5 . . N
C8 C9 H9B 109.5 . . N
H9A C9 H9B 109.5 . . N
C8 C9 H9C 109.5 . . N
H9A C9 H9C 109.5 . . N
H9B C9 H9C 109.5 . . N
C1 O11 C8 114.70(15) . . N

loop_
 _geom_torsion_atom_site_label_1
 _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
O11 C1 C2 C3 59.67(18) . . . . N
C7 C1 C2 C3 175.43(17) . . . . N
C2 C1 C2 C3 -58.8(2) 4_565 . . . N
C1 C2 C3 C4 -2.4(2) . . . . N
C2 C3 C4 C3 63.9(2) . . . 4_565 N
C2 C3 C4 C8 -56.1(2) . . . . N
C3 C4 C8 O11 59.08(13) . . . . N
C3 C4 C8 O11 -59.08(13) 4_565 . . . N
C3 C4 C8 C9 177.11(15) . . . . N
C3 C4 C8 C9 58.9(2) 4_565 . . . N
C3 C4 C8 C9 -58.9(2) . . . 4_565 N
C3 C4 C8 C9 -177.11(15) 4_565 . . 4_565 N
C7 C1 O11 C8 180.000(1) . . . . N
C2 C1 O11 C8 59.18(12) 4_565 . . . N
C2 C1 O11 C8 -59.18(12) . . . . N
C9 C8 O11 C1 -121.72(12) . . . . N
C9 C8 O11 C1 121.72(12) 4_565 . . . N
C4 C8 O11 C1 0.0 . . . . N
```