Supporting Information

Syntheses of Optically Active Trifluoro-norcoronamic Acids

Toshimasa Katagiri, Minoru Irie, and Kenji Uneyama*

Department of Applied Chemistry, Faculty of Engineering, Okayama University,

Okayama 700-8530, Japan

uneyamak@cc.okayama-u.ac.jp

Experimental Section

General. Unless otherwise stated, all non-aqueous reactions and distillations were carried out under atmosphere of dry nitrogen or dry argon. All commercially available reagents and solvents were employed without further purification. Dry THF was distilled from benzophenone-Na ketyl solution just prior to use.

Chromatography on silicagel was performed using a forced flow of the solvent system on E. Merck silica gel (Kieselgel 60, 230-400 mesh). HPLC analyses were performed using Shimadzu HPLC-10A system (l = 254 nm) connected to Daicel Chiralcel OJ column. GC analyses were performed using Shimadzu GC-12A system connected to GL-Science CP-Cyclodex-β-256M Capillary column. Melting points were obtained on Yanagimoto MP-S3 apparatus and are uncorrected.

^{1}H (200 MHz), ^{19}F (188 MHz), and ^{13}C (50.3 MHz) NMR spectra were recorded on Varian VXR-200 spectrometers. Chemical shifts are reported in δ ppm from tetramethylsilane (δ 0.0 ppm for ^{1}H and ^{13}C NMR) and C_6F_6 (δ 0.0 ppm for ^{19}F NMR). For quantitative analyses of yields, 1,3-bis(trifluoromethyl)benzene was used as an internal standard for ^{19}F NMR. Coupling constants (*J*) are reported in hertz. Optical rotation was measured in a cell with 50 mm length and 1 ml capacity using a Horiba High Sensitive Polarimeter SEPA-300. Elemental analyses were performed on Perkin Elmer series II CHNS/O Analyzer 2400. GC/MS was performed on a Hewlett-Packard HP5971A. Intensity measurements for X-ray crystallographic analyses were made on a Rigaku RAXIS-IV imaging plate area detector with graphite monochromated Mo-Kα radiation.

1-(cyanomethyl)-2,5-dimethylpyrrol

To a refluxed solution of KOH (0.35 g) in MeOH (2.8 ml)-H_2O (0.14 ml) was added dropwise a mixture of aminoacetonitrile sulfate (0.5040 g) with hexanedione (0.70 ml) dissolved in MeOH (2.8 ml). The mixture was refluxed for 12 hours. The reacting solution was cooled, then diluted by Et_2O, and washed with brine, sat. $NaHCO_3$ aq., and again brine. The solvent was removed from the mixture and crude product was distilled in vacuo using Kugelrohr distillation apparatus. The pure product, 1-(cyanomethyl)-2,5-dimethylpyrrol was obtained in 63% yield as a white solid.

M.p.: 74-77 °C; IR (KBr): 2250 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.29 (s, 6H), 4.64 (s, 2H), 5.84 (s, 2H); EI-MS (rel. int) 134 (100, M^+), 106 (60); Anal. Calcd. for $C_8H_{10}N_2$: C, 71.61; H, 7.51; N, 20.88. Found: C, 71.26; H, 7.39; N, 20.98. [Bruekelman, S. P.; Leach, S. E.; Meakins, G. D.; Tirel, M. D. *J. Chem. Soc., Perkin Trans I,* **1984**, 2801; Green, T. W.; Wuts, P. G. M. "Protective Groups in Organic Synthesis, 3rd ed", pp568]

5,5,5-trifluoro-4-hydroxy-2-(2,5-dimethyl-1-pyrrolyl)pentanenitrile (3a)

To a solution of 1-(cyanomethyl)-2,5-dimethylpyrrol (0.67 g, 5 mmol) in 15 ml THF, an 1.2 eq. amount of 40% NaHMDS / THF solution was added at -40 °C. The mixture was stirred at the temperature for a while, then added 2,3-epoxy-1,1,1-trifluoropropane **2** (0.56 g, 5 mmol) to the solution dropwise. After 3 hours reaction the mixture became brown. The reacting solution was quenched by NH_4Cl aq., extracted by Et_2O, and washed with brine. Then the crude product was submitted to column chromatography. The 5,5,5-trifluoro-4-hydroxy-2-(2,5-dimethyl-1-pyrrolyl)pentanenitrile (**3a**, 0.9 g), which was the 7:3 mixture of two diastereomers containing a small amount of AcOEt eluent, was obtained in 73% yield as a colorless oil.

IR (Neat): 2280 cm^{-1}; ^1H NMR ($CDCl_3$): δ 2.0-2.7 (m, 2H), 2.32 (s,6H), 3.44 (ddq, *J* = 11, 2, 6, 0.7H), 3.8 (br, 1H), 4.27 (ddq, *J* = 11, 2, 6, 0.3H), 5.34 (dd, *J* = 11, 4, 0.7H) 5.41 (dd, *J* = 11, 4, 0.3H) 5.82 (s,2H); ^{19}F NMR ($CDCl_3$): δ 81.9 (d, *J* = 6); EI-MS (rel. int) 246 (100, M^+), 134 (100), 119 (33), 94 (60).

The compound was rather unstable thus used for the next reaction without further purification.

2-trifluoromethyl-1-(2,5-dimethyl-1-pyrrolyl)cyclopropyl cyanide (4a)

To a solution of p-TsCl (0.40 g, 2.1 mmol) and NaH 60% in oil (0.31 g, 7.5 mmol) in THF (10 ml), solution of **3a** (0.47 g, 1.9 mmol) in THF (3 ml) was added dropwise at 0 °C. The mixture was stirred for 96h at 0 °C, then reacting solution was quenched by NH_4Cl aq., and extracted by Et_2O. The organic extracts were washed with brine, dried over anhydrous $MgSO_4$, and concentrated in vacuo. The crude 2-trifluoromethyl-1-

(2,5-dimethyl-1-pyrrolyl)cyclopropyl cyanide **4a** (0.35 g, 1.6 mmol) with one diastereomer of 75% ee was obtained in 82% yield. The compound was then submitted for the first recrystallization to eliminate the racemate and the second recrystallization to obtain the 0.25 g of colorless plates **4a** with >98% ee (70% from crude **4a**).

M.p.: 77-79 °C; IR (KBr): 2260 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 2.04 (dd(q), *J* = 7, 7, <1, 1H), 2.23 (dd, *J* = 7, 7, 1H), 2.34 (s, 6H), 2.55 (ddq, *J* = 10, 7, 6, 1H), 5.82(s, 2H); ^{19}F NMR ($CDCl_3$): δ 97.7 (d, *J* = 6); EI-MS (rel. int) 228 (100, M^+), 213 (27), 159 (64), 145 (27), 77 (27); Anal. Calcd. for $C_{11}H_{11}F_3N_2$: C, 57.89; H, 4.86; N, 12.27. Found: C, 57.76; H, 4.95; N, 12.33.

The compound was submitted for X-ray crystallographic analysis (tentative sample and file name is irie-4). The CIF file for the analysis are attached at the end of this supporting informations.

1-acetamido-2-trifluoromethylcyclopropyl cyanide (5)

A solution of **4a** (0.114 g, 0.5 mmol), periodic acid (1.283 g, 6.0 mmol), and ruthenium trichloride hydrate (0.0015 g, 1 μmol), in carbon tetrachloride (2 ml), acetonitrile (2 ml), and water (5 ml) was stirred in room temperature for 1 day. Ether was added (a deep black color appeared at this point), then stirred vigolously for 10 min. The organic phase was separated and was washed with brine, dried over anhydrous $MgSO_4$, and concentrated in vacuo. A column chromatography on silica gel gave 1-acetamido-2-trifluoromethylcyclopropyl cyanide **5** (0.072 g, 0.38 mmol) as a white solid in 71% yield.

M.p.: 93-96 °C; IR (KBr): 1670 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.74 (ddq, *J* = 10, 8, 1, 1H), 1.96 (dd, *J* = 7, 8, 1H), 2.03 (s, 3H), 2.14-2.35 (ddq, *J* = 10, 7, 7, 1H), 6.82 (br, 1H); ^{19}F NMR ($CDCl_3$): δ 97.2 (d, *J* = 7); EI-MS (rel. int) 192 (5, M^+), 150 (9), 43 (100),; Anal. Calcd. for $C_7H_7F_3N_2O$: C, 43.76; H, 3.67; N, 14.58. Found: C, 43.97; H, 3.78; N, 14.87.

trifluoro-norcoronamic acid hydrochloride (6)

A 0.072 g of nitrile **5** was allowed to react with 4 ml of conc. HCl solution at 60 °C for 24 hours. Water was evapolated in vacuo, and residue was submitted for column chromatography on silica gel with acetone eluent. A 0.054 g of trifluioro-norcoronamic acid hydrochloride monohydrate **6** was obtained as a white powder in 70 % yield.

M.p.: 200 °C (decomp.); $[\alpha]^{20}_D$ +13.6 (*c* 1.2, H_2O); IR (KBr): 3448, 3040, 1618 cm^{-1}; ^{1}H NMR (D_2O): δ 1.58 (ddq, *J* = 10, 8, 1, 1H), 1.90 (dd, *J* = 8, 7, 1H), 2.28 (ddq, *J* = 10, 7, 7, 1H); ^{19}F NMR ($CDCl_3$): δ 106.9 (d, *J* = 7); Anal. Calcd. for $C_5H_9ClF_3NO_3$: C, 26.86; H, 4.06; N, 6.26. Found: C, 27.08; H, 4.32; N,

6.47.

2-trifluoromethyl-1-(3,4-dimethoxyphenyl)cyclopropyl cyanide (4b)

To a solution of 3,4-dimethoxyphenylacetonitrile (8.9 g, 50 mmol) in 110 ml THF, an 1.2 eq. amount of n-BuLi solution was added dropwise at -78 °C. The mixture became green to orange, within 30 minutes, then added 2,3-epoxy-1,1,1-trifluoropropane **2** (5.6 g, 50 mmol) to the solution dropwise. After 22.5 hours the reaction mixture became black. The reacting solution was quenched by NH_4Cl aq., extracted by Et_2O, and washed with brine. Then the crude product was submitted for column chromatography. Crude 5,5,5-trifluoro-4-hydroxy-2-(3,4-dimethoxyphenyl)pentanenitrile (**3b**), was used for further reaction as it was.

Successive cyclization was undertaken as followings: To a solution of p-TsCl (9.5 g, 50 mmol) and NaH 60% in oil (8.0 g, 200 mmol) in THF (100 ml), solution of crude **3b** in THF (100 ml) was added dropwise at room temperature. The mixture was stirred for 24 hours at room temperature, then reacting solution was quenched by NH_4Cl aq., and extracted by Et_2O. The organic extracts were washed with brine, dried over anhydrous $MgSO_4$, and concentrated in vacuo. The crude product was distilled in vacuo to give slightly yellowish blocks, **4b** (8.25 g, 33.4 mmol) with 90% de mixture of two diastereomer of 75% ee was obtained in 67% yield from epoxide **2**.

Twice recrystallization of crude **4b** from benzene gave 2.7 g of colorless needles with >99% ee (33% from crude **4b**).

M.p.: 71-72 °C; IR (KBr): 2240 cm^{-1}; ^1H NMR ($CDCl_3$): δ 1.81 (ddq, *J* = 8, 7, <1, 1H), 2.02 (dd, *J* = 7, 6, 1H), 2.25 (ddq, *J* = 6, 6, 8, 1H), 3.88 (s, 3H), 3.91 (s, 3H), 6.81-6.88(m, 3H); ^{19}F NMR ($CDCl_3$): δ 97.4 (d, *J* = 6); EI-MS (rel. int) 271 (30, M^+), 240 (100), 69 (30); Anal. Calcd. for $C_{13}H_{12}F_3NO_2$: C, 57.57; H, 4.46; N, 5.16. Found: C, 57.17; H, 4.58; N, 5.52.

The compound was submitted to X-ray crystallographic analysis (tentative sample and file name is irie-3). The copy of X-ray-structure report, the FoFc table, and the CIF file for the analysis are attached at the end of this supporting informations.

2-trifluoromethyl-1-(3,4-dimethoxyphenyl)cyclopropylcarboxamide (7)

A solution of **4b** (2.1 g, 1.1 mmol), H_2O_2 (30%, 4 ml), KOH aq. (7 mol/l, 0.4 ml) in ethanol (5 ml) was refluxed for 12 hours, then the reacting solution was extracted by Et_2O. The organic layer was washed with brine, dried over anhydrous $MgSO_4$, and concentrated in vacuo. Recrystallization of crude product gave 1.8 g of

7 in 79% yield.

M.p. : 64-67 °C; IR (KBr): 3440, 3240, 1690 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.36 (m, 1H), 2.16-2.27 (m, 2H), 3.86 (s, 3H), 5.58 (br, 1H), 6.12 (br, 1H), 6.80-7.40 (m, 3H); ^{19}F NMR ($CDCl_3$): δ 100.8 (d, *J* = 6); EI-MS (rel. int) 289 (100, M^+), 274 (32), 245 (20), 230 (30), 214 (18), 44 (18); Anal. Calcd. for $C_{13}H_{14}F_3NO_3$: C, 53.98; H, 4.88; N, 4.84. Found: C, 54.11; H, 5.09; N, 4.77.

N-Boc Trifluoro-allo-norcoronamic acid (9)

A mixture of **7** (0.47 g, 1.6 mmol), $Pb(OAc)_4$ (1.01 g, 1.6 mmol), and t-BuOH (10 ml) was stirred at 40 °C for 24 hours. To the reacting solution, sat. $NaHCO_3$ aq. was added, then was extracted with Et_2O. Organic phase was dried over anhydrous $MgSO_4$, and concentrated in vacuo. The compound was rather unstable and was thus used for the next reaction without further purification. Crude **8** was purified by column chromatography just prior to the successive reaction.

A mixture of **8**, carbon tetrachloride (6 ml), acetonitrile (6 ml), water (15 ml), periodic acid (4.2 g, 20 mmol), and ruthenium trichloride hydrate (0.01 g, 0.02 mol) was stirred in room temperature for 20 hours. Ether was added (a deep black color appeared at this point), then stirred vigolously for 10 min. The organic phase was separated and was washed with brine, dried over anhydrous $MgSO_4$, and concentrated in vacuo. Recrystallization of crude product gave N-Boc trifluoro-allo-norcoronamic acid, **9** (white powder) in 30% yield from **7**.

M.p.: 158-159 °C; $[\alpha]^{20}_D$ -25.5 (*c* 3.2, $CDCl_3$); IR (KBr): 3370, 1700 cm^{-1}; ^{1}H NMR ($CDCl_3$): δ 1.45 (s, 9H), 1.81 (m, 1H), 2.00 (m, 1H), 2.52 (m, 1H), 5.15 (br, 1H); ^{19}F NMR ($CDCl_3$): δ 100.5 (d, *J* = 6); EI-MS (rel. int) 169 (17, M^+-Boc), 59 (33), 57 (100), 41 (27); Anal. Calcd. for $C_{10}H_{14}F_3NO_4$: C, 44.61; H, 5.24; N, 5.20. Found: C, 44.69; H, 5.05; N, 5.58.

CIF files

CIF file for 2-trifluoromethyl-1-(2,5-dimethyl-1-pyrrolyl)cyclopropyl cyanide (4a)

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_refine_ls_matrix_type full

_refine_ls_weighting_scheme 'w = 1/[\s^2^(Fo) + 0.00022|Fo|^2^]'

_refine_ls_hydrogen_treatment refU

_refine_ls_extinction_method none

_refine_ls_extinction_coef ?

_refine_ls_abs_structure_details ?

_refine_ls_abs_structure_Flack ?

_refine_ls_number_reflns 916

_refine_ls_number_parameters 156

_refine_ls_number_restraints 0

_refine_ls_number_constraints 0

_refine_ls_R_factor_all ?

_refine_ls_R_factor_obs 0.0776

_refine_ls_wR_factor_all ?

_refine_ls_wR_factor_obs 0.1070

_refine_ls_goodness_of_fit_all ?

_refine_ls_goodness_of_fit_obs 3.698

```
_refine_ls_shift/esd_max              0.0590
_refine_ls_shift/esd_mean             0.0020
_refine_diff_density_min              -0.22
_refine_diff_density_max              0.24
#--------------------------------------------------------------------------
_geom_special_details
;
 ?
;
loop_
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_geom_bond_atom_site_label_2
_geom_bond_distance
_geom_bond_site_symmetry_1
_geom_bond_site_symmetry_2
_geom_bond_publ_flag
     ? ?   ?     ? ? ?
# -- ENTER BONDS HERE, ONE PER LINE --
# e.g. C1 C2 1.541(1) . . yes
#--------------------------------------------------------------------------
loop_
_geom_angle_atom_site_label_1
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_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
# -- ENTER ANGLES HERE, ONE PER LINE --
```

```
# e.g. C1 C2 C3 109.4(3) . . . yes
      ? ? ?    ?    ? ? ? ?
#-------------------------------------------------------------------------------
loop_
_geom_contact_atom_site_label_1
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_geom_contact_site_symmetry_1
_geom_contact_site_symmetry_2
_geom_contact_publ_flag
      ? ?   ?     ? ? ?
# -- ENTER BONDS HERE, ONE PER LINE --
# e.g. O1 N1 1.541(1) . . yes
#-------------------------------------------------------------------------------
_publ_requested_journal               ' ENTER JOURNAL NAME HERE'

_publ_contact_author
;
   ENTER NAME
 ENTER ADDRESS
;
_publ_contact_letter
;
   ENTER TEXT OF LETTER
;

_publ_requested_coeditor_name         ?
_publ_contact_author_phone            ' ENTER PHONE NUMBER '
_publ_contact_author_fax              ' ENTER FAX NUMBER '
_publ_contact_author_email            ' ENTER EMAIL ADDRESS '
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;
  FIRST AUTHORS ADDRESS
;

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;
    ENTER EXPERIMENTAL SECTION
;

_publ_section_comment
;
    ENTER TEXT
;

_publ_section_references
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ENTER OTHER REFERENCES

Molecular Structure Corporation. (1995). teXsan.

Single Crystal Structure Analysis Software. Version 1.7.

MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

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CIF file for 2-trifluoromethyl-1-(3,4-dimethoxyphenyl)cyclopropyl cyanide (4b)

data_irie 4b

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_audit_creation_date 'Fri Mar 18 09:02:36 2000'

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_audit_update_record ?

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_computing_cell_refinement 'MSC/AFC Diffractometer Control'

_computing_data_reduction 'teXsan'

_computing_structure_solution ?

_computing_structure_refinement 'teXsan'

_computing_publication_material 'teXsan'

_computing_molecular_graphics ?

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_chemical_compound_source ?

_chemical_name_common ?

_chemical_formula_weight 271.24

_chemical_formula_analytical ?

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_chemical_formula_moiety 'C13 H12 F3 N O2 '

_chemical_formula_structural ?

_chemical_melting_point ?

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_cell_length_a 12.9184(5)

_cell_length_b 14.0537(5)

_cell_length_c 7.0743(2)

_cell_angle_alpha 90

_cell_angle_beta 90

_cell_angle_gamma 90

_cell_volume 1284.35(7)

_cell_formula_units_Z 4

_cell_measurement_temperature 288.2

_cell_measurement_reflns_used 0

_cell_measurement_theta_min 0.0

_cell_measurement_theta_max 0.0

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_symmetry_cell_setting orthorhombic

_symmetry_space_group_name_H-M 'P 21 21 21 '

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_symmetry_space_group_name_Hall      ?
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x,y,z
1/2-x,-y,1/2+z
1/2+x,1/2-y,-z
-x,1/2+y,1/2-z
_exptl_crystal_description           'needle'
_exptl_crystal_colour                'colorless'
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_exptl_crystal_size_mid              0.500
_exptl_crystal_size_min              0.500
_exptl_crystal_density_diffrn        1.403
_exptl_crystal_density_meas          'not measured'
_exptl_crystal_F_000                 560.00
_exptl_absorpt_coefficient_mu        0.123
_exptl_absorpt_correction_type       none
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;
 ?
;
#---------------------------------------------------------------------------
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_diffrn_radiation_monochromator graphite

_diffrn_radiation_detector 'Imaging Plate'

_diffrn_measurement_device 'Rigaku RAXISII'

_diffrn_measurement_method

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_diffrn_standards_interval_count 0

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_diffrn_standard_refln_index_l

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_reflns_number_total 1354

_reflns_number_observed 1343

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_diffrn_reflns_av_sigmaI/netI 0.045

_diffrn_reflns_limit_h_min 0

_diffrn_reflns_limit_h_max 16

_diffrn_reflns_limit_k_min 0

_diffrn_reflns_limit_k_max 18

_diffrn_reflns_limit_l_min 0

_diffrn_reflns_limit_l_max 9

_diffrn_reflns_theta_min 0.00

_diffrn_reflns_theta_max 0.00

_diffrn_reflns_reduction_process 'Lp corrections applied'

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_diffrn_orient_matrix_UB_12 0.00000

_diffrn_orient_matrix_UB_13 0.00000

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_diffrn_orient_matrix_UB_33          0.00000
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_atom_type_symbol
_atom_type_oxidation_number
_atom_type_number_in_cell
_atom_type_scat_dispersion_real
_atom_type_scat_dispersion_imag
_atom_type_scat_source
  C  0    52 0.003 0.002
;International Tables for Crystallography
(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
;
  H  0    48 0.000 0.000
;International Tables for Crystallography
(1992, Vol. C, Table 6.1.1.2)
;
  F  0    12 0.017 0.010
;International Tables for Crystallography
(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
;
  N  0     4 0.006 0.003
;International Tables for Crystallography
(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
;
```

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 O  0     8 0.011 0.006
;International Tables for Crystallography
(1992, Vol. C, Tables 4.2.6.8 and 6.1.1.1)
;
#---------------------------------------------------------------------------
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_atom_site_fract_x
_atom_site_fract_y
_atom_site_fract_z
_atom_site_U_iso_or_equiv
_atom_site_thermal_displace_type
_atom_site_calc_flag
_atom_site_calc_attached_atom
_atom_site_occupancy
_atom_site_refinement_flags
 F(4)   0.1938(2)   -0.1359(2)  -0.6621(7)  0.088(1)    Uani d . 1.00 .
 F(5)   0.1075(3)   -0.0971(3)  -0.4168(5)  0.092(1)    Uani d . 1.00 .
 F(6)   0.1857(2)   0.0094(2)   -0.5772(8)  0.105(1)    Uani d . 1.00 .
 O(2)   -0.4642(2)  -0.0855(2)  -0.8066(6)  0.0564(8)   Uani d . 1.00 .
 O(3)   -0.3476(2)  0.0613(2)   -0.8144(5)  0.0449(7)   Uani d . 1.00 .
 N(2)   0.0316(3)   -0.3076(3)  -0.7295(7)  0.063(1)    Uani d . 1.00 .
 C(2)   -0.1899(3)  -0.0339(2)  -0.8020(6)  0.0353(8)   Uani d . 1.00 .
 C(3)   -0.0292(3)  -0.1345(3)  -0.7871(6)  0.0403(9)   Uani d . 1.00 .
 C(4)   -0.2091(3)  -0.2040(3)  -0.7894(6)  0.0435(10)  Uani d . 1.00 .
 C(5)   -0.3609(3)  -0.1048(3)  -0.8023(6)  0.0414(9)   Uani d . 1.00 .
 C(6)   -0.2858(3)  0.1452(3)   -0.8063(7)  0.047(1)    Uani d . 1.00 .
 C(7)   -0.3165(3)  -0.1942(3)  -0.7946(7)  0.0462(10)  Uani d . 1.00 .
 C(9)   0.0068(3)   -0.2313(3)  -0.7541(6)  0.046(1)    Uani d . 1.00 .
 C(10)  0.0361(3)   -0.0535(3)  -0.7101(7)  0.048(1)    Uani d . 1.00 .
```

C(11) 0.0373(3) -0.0720(4) -0.9165(7) 0.056(1) Uani d . 1.00 .

C(12) 0.1296(3) -0.0704(4) -0.5917(10) 0.068(1) Uani d . 1.00 .

C(13) -0.5332(3) -0.1637(4) -0.7731(8) 0.063(1) Uani d . 1.00 .

C(14) -0.1453(3) -0.1251(2) -0.7928(5) 0.0362(8) Uani d . 1.00 .

C(15) -0.2971(3) -0.0237(2) -0.8075(6) 0.0354(8) Uani d . 1.00 .

H(1) -0.1487 0.0277 -0.8005 0.06(1) Uiso calc . 1.00 S

H(2) -0.1824 -0.2688 -0.7917 0.07(2) Uiso calc . 1.00 S

H(3) -0.3590 -0.2564 -0.7981 0.06(1) Uiso calc . 1.00 S

H(4) -0.0001 0.0011 -0.6609 0.05(1) Uiso calc . 1.00 S

H(5) 0.0032 -0.0218 -1.0018 0.06(1) Uiso calc . 1.00 S

H(6) 0.0924 -0.1016 -0.9695 0.09(2) Uiso calc . 1.00 S

H(7) -0.2424 0.1533 -0.9153 0.05(1) Uiso calc . 1.00 S

H(8) -0.2488 0.1522 -0.6920 0.06(1) Uiso calc . 1.00 S

H(9) -0.3373 0.1990 -0.8254 0.07(1) Uiso calc . 1.00 S

H(10) -0.5986 -0.1368 -0.7753 0.09(2) Uiso calc . 1.00 S

H(11) -0.5215 -0.2002 -0.6451 0.13(3) Uiso calc . 1.00 S

H(12) -0.5371 -0.2212 -0.8710 0.09(2) Uiso calc . 1.00 S

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_atom_site_aniso_U_22

_atom_site_aniso_U_33

_atom_site_aniso_U_12

_atom_site_aniso_U_13

_atom_site_aniso_U_23

F(4) 0.044(1) 0.086(2) 0.135(3) 0.028(1) -0.015(2) -0.017(2)

F(5) 0.081(2) 0.114(3) 0.082(2) 0.025(2) -0.028(2) 0.006(2)

F(6) 0.063(2) 0.074(2) 0.178(4) -0.012(2) -0.045(3) -0.007(3)

O(2) 0.031(1) 0.059(2) 0.079(2) -0.011(1) 0.000(1) 0.006(2)

O(3) 0.032(1) 0.039(1) 0.063(2) 0.006(1) -0.003(1) 0.000(1)

N(2) 0.063(2) 0.045(2) 0.082(3) 0.016(2) -0.004(2) -0.002(2)

C(2) 0.031(2) 0.032(2) 0.043(2) 0.001(1) 0.000(2) 0.003(2)

C(3) 0.040(2) 0.034(2) 0.047(2) 0.006(1) 0.002(2) 0.003(2)

C(4) 0.048(2) 0.031(2) 0.051(2) 0.001(2) 0.003(2) -0.001(2)

C(5) 0.034(2) 0.050(2) 0.041(2) -0.005(1) 0.000(2) 0.000(2)

C(6) 0.046(2) 0.035(2) 0.059(2) 0.003(2) 0.000(2) 0.001(2)

C(7) 0.047(2) 0.038(2) 0.054(2) -0.011(2) -0.002(2) 0.001(2)

C(9) 0.044(2) 0.043(2) 0.050(2) 0.010(2) 0.000(2) -0.006(2)

C(10) 0.036(2) 0.040(2) 0.069(3) 0.006(2) -0.005(2) -0.002(2)

C(11) 0.041(2) 0.062(3) 0.065(3) 0.006(2) 0.015(2) 0.016(2)

C(12) 0.042(2) 0.057(3) 0.105(4) 0.010(2) -0.013(3) -0.011(3)

C(13) 0.046(2) 0.072(3) 0.070(3) -0.026(2) 0.003(2) 0.005(3)

C(14) 0.038(2) 0.035(2) 0.035(2) 0.002(1) -0.001(2) -0.001(2)

C(15) 0.033(2) 0.037(2) 0.037(2) 0.002(1) 0.000(2) 0.002(2)

#---

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;

?

;

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;
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_geom_bond_site_symmetry_2
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# -- ENTER BONDS HERE, ONE PER LINE --
# e.g. C1 C2 1.541(1) . . yes
#--------------------------------------------------------------------------
loop_
_geom_angle_atom_site_label_1
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_geom_angle_atom_site_label_2
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_geom_angle
_geom_angle_site_symmetry_1
_geom_angle_site_symmetry_2
_geom_angle_site_symmetry_3
_geom_angle_publ_flag
# -- ENTER ANGLES HERE, ONE PER LINE --
# e.g. C1 C2 C3 109.4(3) . . . yes
     ? ? ?    ?    ? ? ?  ?
#---------------------------------------------------------------------------
loop_
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_geom_contact_site_symmetry_1
_geom_contact_site_symmetry_2
_geom_contact_publ_flag
     ? ?   ?     ? ?  ?
# -- ENTER BONDS HERE, ONE PER LINE --
# e.g. O1 N1 1.541(1) . . yes
#---------------------------------------------------------------------------
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_publ_contact_author
;
   ENTER NAME
 ENTER ADDRESS
;
_publ_contact_letter
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;
   ENTER TEXT OF LETTER
;

_publ_requested_coeditor_name   ?
_publ_contact_author_phone   ' ENTER PHONE NUMBER '
_publ_contact_author_fax   ' ENTER FAX NUMBER '
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ENTER OTHER REFERENCES

Molecular Structure Corporation. (1995). teXsan.

Single Crystal Structure Analysis Software. Version 1.7.

MSC, 3200 Research Forest Drive, The Woodlands, TX 77381, USA.

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