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(2*R*,5*S*)-2-Trichloromethyl-3-oxa-1-azabicyclo-[3.3.0]octane-4,8-dione

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Key indicators

Single-crystal X-ray study
T = 190 K
Mean σ(C–C) = 0.002 Å
R factor = 0.027
wR factor = 0.064
Data-to-parameter ratio = 21.0

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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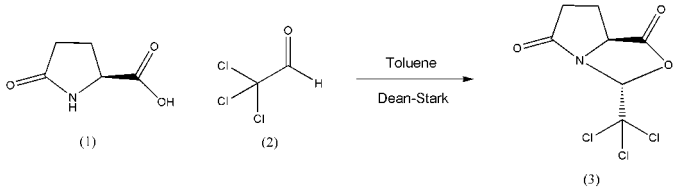
The crystal structure of the title bicyclic oxazolidindione, $C_7H_6Cl_3NO_3$, confirmed the absolute stereochemistry as 2*R*,5*S*.

Comment

Natural α-amino acids are at the core of many natural products (Ikota, 1992). (*S*)-pyroglutamic acid, in particular, forms the core of many excitatory amino acids, such as kaitocephalin (Watanabe *et al.*, 2002) and kainic acid (Oppolzer & Thirring, 1982). Synthetic routes to these classes of compounds require stereochemical control at various positions around a pyrrolidine ring. Seebach's method of the so-called self-reproduction of chirality involves a dual protection of the amine and carboxylic acid of (*S*)-proline with pivaldehyde to give a bicyclic system (Seebach *et al.*, 1983). The analogous protection of (*S*)-pyroglutamic acid was found to be unfavourable for pivaldehyde, the resulting product being particularly unstable (Dikshit *et al.*, 1995). However, analogous protection of (*S*)-pyroglutamic acid with chloral (Amedjkouh & Ahlberg, 2002) provided the title compound, (3) (shown in Fig. 1 and Table 1), as an air-stable crystalline solid.



(1) (2) (3)

Experimental

The title compound was prepared by the method described by Amedjkouh & Ahlberg (2002). Slow recrystallization from ethyl acetate gave colourless needle-like crystals. These tend to fracture when cut and therefore a large crystal was used. The multi-scan technique was used to correct for changes in the illuminated volume.

Crystal data

$C_7H_6Cl_3NO_3$
M_r = 258.49
Orthorhombic, $P2_12_12_1$
a = 6.0480 (1) Å
b = 10.1735 (3) Å
c = 15.5791 (4) Å
V = 958.57 (4) Å^3
Z = 4
D_x = 1.791 Mg m^{-3}
Mo *Kα* radiation
Cell parameters from 1485 reflections
θ = 5–30°
μ = 0.93 mm^{-1}
T = 190 K
Needle, colourless
0.80 × 0.20 × 0.20 mm

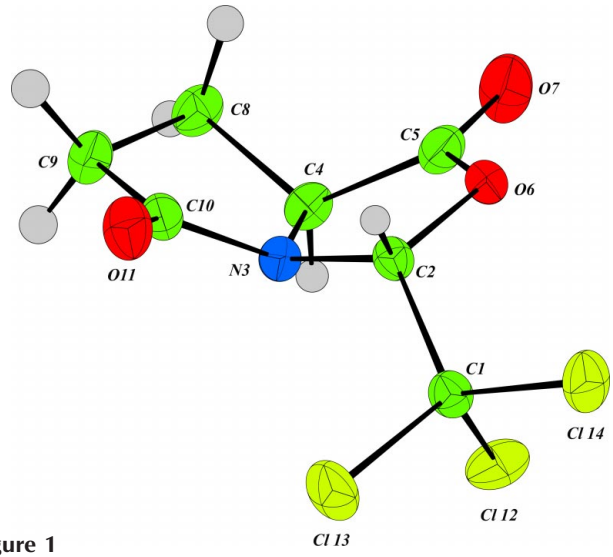


Figure 1
The molecular structure of (3), with displacement ellipsoids drawn at the 50% probability level. H-atom radii are arbitrary. Cl atoms are displayed in bright green.

Data collection

Nonius KappaCCD diffractometer	R_{int} = 0.028
ω scans	θ_{max} = 30.0°
Absorption correction: none	$h = -8 \rightarrow 8$
2708 measured reflections	$k = -14 \rightarrow 14$
2692 independent reflections	$l = -21 \rightarrow 21$
2545 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F^2) + 0.02 + 0.49p]$ where $p = [\max(F_o^2,0) + 2F_c^2]/3$
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$(\Delta/\sigma)_{max}$ = 0.001
$wR(F^2)$ = 0.064	$\Delta\rho_{max}$ = 0.27 e Å^{-3}
S = 0.95	$\Delta\rho_{min}$ = −0.26 e Å^{-3}
2692 reflections	Absolute structure: Flack (1983), 1104 Friedel pairs
128 parameters	Flack parameter = 0.01 (6)
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—C2	1.544 (2)	C4—C5	1.516 (2)
C1—Cl12	1.7638 (16)	C4—C8	1.547 (2)
C1—Cl13	1.7617 (17)	C5—O6	1.366 (2)
C1—Cl14	1.7716 (16)	C5—O7	1.191 (2)
C2—N3	1.4352 (19)	C8—C9	1.531 (3)
C2—O6	1.4314 (19)	C9—C10	1.514 (3)
N3—C4	1.457 (2)	C10—O11	1.207 (2)
N3—C10	1.391 (2)		
C2—C1—Cl12	110.98 (10)	N3—C4—C5	102.17 (14)
C2—C1—Cl13	108.10 (11)	N3—C4—C8	105.25 (13)
Cl12—C1—Cl13	110.16 (9)	C5—C4—C8	117.03 (14)
C2—C1—Cl14	108.76 (11)	C4—C5—O6	109.41 (14)
Cl12—C1—Cl14	109.83 (9)	C4—C5—O7	129.11 (18)
Cl13—C1—Cl14	108.97 (8)	O6—C5—O7	121.44 (16)
C1—C2—N3	113.21 (12)	C2—O6—C5	110.71 (12)
C1—C2—O6	108.34 (12)	C4—C8—C9	102.61 (13)
N3—C2—O6	105.82 (11)	C8—C9—C10	104.36 (14)
C2—N3—C4	110.58 (12)	C9—C10—N3	106.57 (14)
C2—N3—C10	121.30 (13)	C9—C10—O11	129.32 (16)
C4—N3—C10	112.34 (13)	N3—C10—O11	124.11 (16)

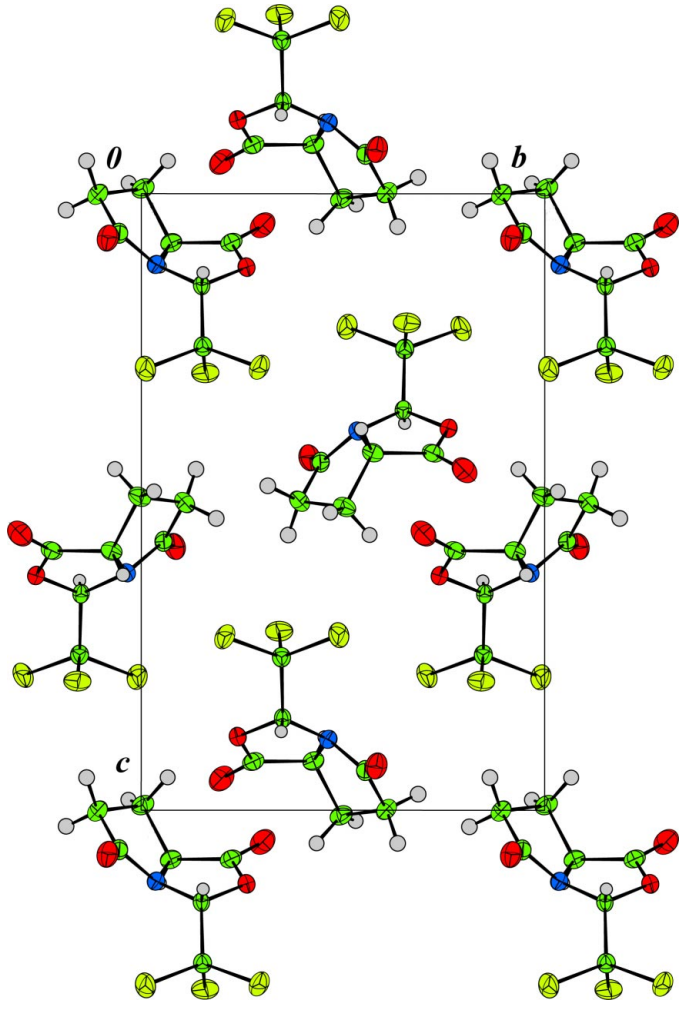


Figure 2
Packing diagram of (3), viewed down the *a* axis.

The H atoms were all seen in a difference map but those attached to C atoms were repositioned geometrically. The H atoms were initially refined with soft restraints on the bonds to regularize their geometry [bond lengths to accepted values, angles either set by symmetry or to accepted values, and U_{iso}(H) dependent on the adjacent bonded atom], after which they were refined with riding constraints only. C—H = 0.93–0.98 Å and U_{iso}(H) = 1.2U_{eq}(C).

Data collection: *COLLECT* (Nonius, 1997–2001); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *CAMERON* (Watkin *et al.*, 1996); software used to prepare material for publication: *CRYSTALS*.

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