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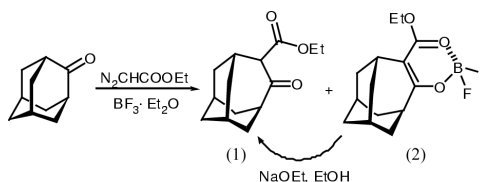
## Key indicators

Single-crystal X-ray study  
T = 293 K  
Mean  $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$   
R factor = 0.049  
wR factor = 0.170  
Data-to-parameter ratio = 32.7For details of how these key indicators were  
automatically derived from the article, see  
<http://journals.iucr.org/e>.Ethyl (*O*-*B*)-5-(difluoroboryloxy)tricyclo-  
[4.3.1.1<sup>3,8</sup>]undecane-4-carboxylate

The title compound,  $\text{C}_{14}\text{H}_{19}\text{BF}_2\text{O}_3$ , can be described as a resonance hybrid. The entering  $\text{BF}_2$  moiety interacts with the  $\text{C}=\text{O}$  (oxo) groups of the parent compound, 5-(ethoxycarbonyl)adamantan-4-one, generating two conjugated double bonds in the six-membered difluorodioxoborane ring. In spite of the conjugation between the multiple bonds, the tetrahedral configuration of the B atom gives rise to a non-planar conformation of the six-membered ring, which assumes a state intermediate between the boat and sofa forms.

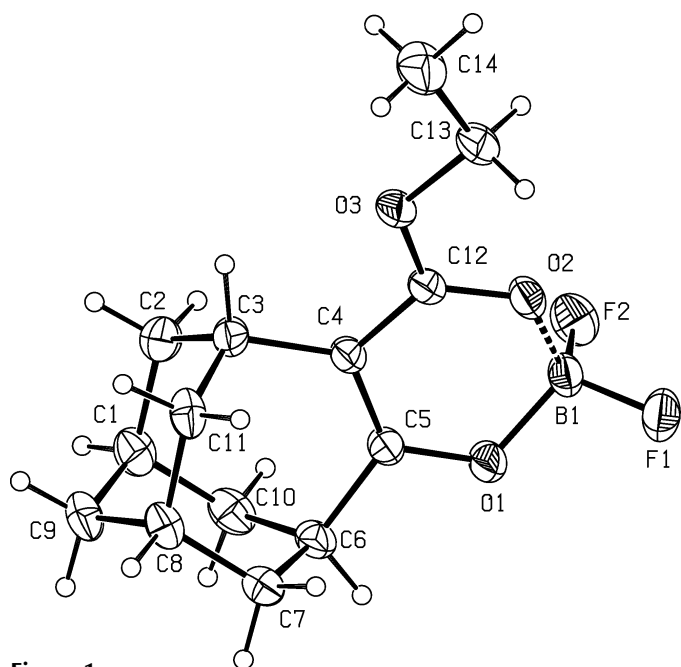
## Comment

For the synthesis of homoadamantane-fused pyridopyrimidinones (Cs. Gyarmati *et al.*, 2003), 5-(ethoxycarbonyl)homoadamantan-4-one, (1), or the title difluoroborate complex (2) of the latter was used as the key compound, the complex being prepared in the reaction of adamantan-2-one and ethyl diazoacetate in the presence of boron trifluoride diethyl etherate (see scheme).



We have found that (2) is much more stable than the boron complexes of other  $\beta$ -ketocarboxylates (Lin *et al.*, 1995; Mock & Hartman, 1970). In the present work, the structure of ethyl (*O*-*B*)-5-(difluoroboryloxy)tricyclo[4.3.1.1<sup>3,8</sup>]undecane-4-carboxylate, (2), has been established by X-ray diffraction. The structure determination of (2) confirms the ring closure between the oxo groups. Both B—O bonds [1.464 (1) and 1.510 (2)  $\text{\AA}$ ] are covalent and associated with C—O multiple bonds [1.312 (1) and 1.278 (1)  $\text{\AA}$ ]. In accordance with the 'diene'-type  $p\pi$ - $p\pi$  conjugation, the C—C bonds [1.411 (1) and 1.372 (1)  $\text{\AA}$ ] also have multiple-bond character. The exocyclic ethoxy group may account for the asymmetry of the C—C, C—O and O—B bond pairs. Presumably for similar reasons, in the majority of the 14 structures containing difluorodioxoborane rings that are archived in the Cambridge Structural Database (release of April 2003; Allen, 2002); namely CAMLUX10 (Jones *et al.*, 1990), CUQWEQ (Balasubramanian *et al.*, 2000), FIZGAW (Boeyens *et al.*, 1987), HAHHUT (Morris *et al.*, 1993), HATTAX (Stomberg *et al.*, 1994), KONWOZ (Stomberg & Lindquist, 1991), LIYHEG (Görlitz *et al.*, 1999), MIXXEW (Schiemenz & Näther, 2002) and NOLDOH (Dromzee *et al.*, 1997), the pairs of bond

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**Figure 1**

A perspective view of the title molecule, with displacement ellipsoids drawn at the 30% probability level. Only non-H atoms are labelled.

lengths also display differences. In contrast, in five structures these differences are within experimental error. In particular, VEJNUT [2,2-difluoro-4,6-dimethyl-5-(4'-nitrophenyl)-1,3,2-dioxaborinane; Emsley *et al.*, 1989] assumes almost perfect 'mirror' symmetry. The  $\text{BO}_2\text{F}_2$  moiety has a tetrahedral configuration, which results in a slightly puckered six-membered ring. Its conformation is intermediate between the boat and sofa (envelope) conformations, with the B atom as the flap.

## Experimental

The title complex, (2), was obtained as a by-product of the reaction between adamantan-2-one and ethyl diazoacetate in the presence of boron trifluoride diethyl etherate (Cs. Gyarmati *et al.*, 2003) (m.p. 408–410 K).

### Crystal data

$\text{C}_{14}\text{H}_{19}\text{BF}_2\text{O}_3$   
 $M_r = 284.10$   
 Triclinic,  $P\bar{1}$   
 $a = 7.169$  (1) Å  
 $b = 8.049$  (1) Å  
 $c = 11.858$  (1) Å  
 $\alpha = 89.06$  (1)°  
 $\beta = 83.36$  (1)°  
 $\gamma = 87.22$  (1)°  
 $V = 678.82$  (14) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.390$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 25 reflections  
 $\theta = 18.0$ – $18.9$ °  
 $\mu = 0.11$  mm<sup>-1</sup>  
 $T = 293$  (2) K  
 Prism, colourless  
 $0.50 \times 0.50 \times 0.45$  mm

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega$ – $\theta$  scans  
 Absorption correction:  $\psi$  scan (North *et al.*, 1968)  
 $T_{\min} = 0.936$ ,  $T_{\max} = 0.951$   
 6462 measured reflections  
 5947 independent reflections  
 3332 reflections with  $I > 2\sigma(I)$

$R_{\text{int}} = 0.011$   
 $\theta_{\text{max}} = 35.0$ °  
 $h = -11 \rightarrow 11$   
 $k = -6 \rightarrow 12$   
 $l = -19 \rightarrow 19$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: 2%

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.049$   
 $wR(F^2) = 0.170$   
 $S = 1.10$   
 5947 reflections  
 182 parameters

H-atom parameters constrained  
 $w = 1/[\sigma^2(F_o^2) + (0.0914P)^2]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{\text{max}} = 0.001$   
 $\Delta\rho_{\text{max}} = 0.30$  e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}} = -0.23$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å).

C1–C9	1.5205 (18)	C7–C8	1.5270 (17)
C1–C10	1.5275 (19)	C8–C9	1.5181 (17)
C1–C2	1.5295 (18)	C8–C11	1.5351 (16)
C2–C3	1.5321 (16)	C12–O2	1.2784 (12)
C3–C4	1.5154 (13)	C12–O3	1.3065 (12)
C3–C11	1.5295 (17)	C13–O3	1.4701 (13)
C4–C5	1.3723 (13)	C13–C14	1.4643 (19)
C4–C12	1.4111 (13)	O1–B1	1.4638 (14)
C5–O1	1.3118 (12)	O2–B1	1.5104 (15)
C5–C6	1.5014 (13)	B1–F2	1.3606 (16)
C6–C10	1.5370 (18)	B1–F1	1.3618 (15)
C6–C7	1.5387 (16)		

H atoms were placed geometrically in idealized positions, with C–H = 0.96 Å for methyl H atoms, 0.97 Å for methylene H atoms and 0.98 Å for all other H atoms.  $U_{\text{iso}}$  values were set equal to  $1.5U_{\text{eq}}$  of the carrier atom (for methyl H atoms) and  $1.3U_{\text{eq}}$  for other H atoms.

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1992); cell refinement: CAD-4 EXPRESS; data reduction: XCAD4 (Harms, 1996); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

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