# Crystal structure of (Z,Z)-3,8-bis[1-(difluoroboryloxy)ethylidene]decane-2,9-dione, $C_{10}H_{14}O_2(C_2H_3OBF_2)_2$

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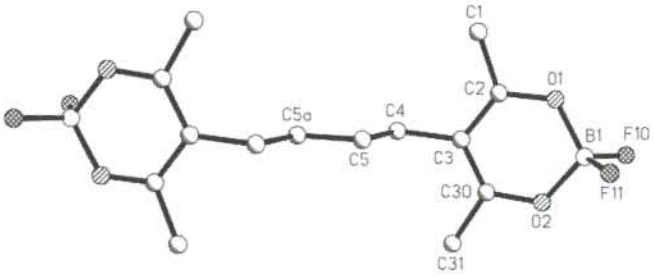


## Abstract

$C_{14}H_{20}B_2F_4O_4$, monoclinic, $P12_1/n1$ (No. 14), $a$ = 7.691(1) Å, $b$ = 11.482(2) Å, $c$ = 9.997(1) Å, β = 106.22(1)°, $V$ = 847.7 Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.093, $wR(F)$ = 0.095, $T$ = 293 K.

## Source of material

The title compound was prepared, according to the general method by Mao and Hauser [1], by conversion of decane-2,9-dione into the enol acetate with a large excess of acetic anhydride (= solvent) in the presence of *p*-toluenesulfonic acid, followed by the addition of the complex consisting of boron trifluoride and two moles of acetic acid, and stirring of the mixture for 48 h at 293 K – 298 K. Workup with ice-water gave a light-brown powder (93%). Recrystallization from formic acid afforded colourless needles, mp 538 K – 542 K [2]. The precursor of the title compound, decane-2,9-dione, was obtained in 83% yield, according to the general method by Reynolds and Hauser [3], from octanedioic chloride and the magnesium enolate of diethyl malonate [2].

**Table 1.** Data collection and handling.

| | |
|---|---|
| Crystal: | colourless lath, size 0.55 × 0.25 × 0.15 mm |
| Wavelength: | Mo $K_\alpha$ radiation (0.71073 Å) |
| μ: | 1.20 cm$^{-1}$ |
| Diffractometer, scan mode: | Siemens P4, ω |
| $2\theta_{max}$: | 55° |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$: | 2185, 1935 |
| Criterion for $F_{obs}$, $N(hkl)_{gt}$: | $F_{obs} > 3\ \sigma(F_{obs})$, 1115 |
| $N(param)_{refined}$: | 109 |
| Program: | SHELXTL-plus [4] |

**Table 2.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{iso}$ |
|---|---|---|---|---|---|
| H(1A) | 4*e* | –0.4306(8) | 0.3013(5) | 1.0320(6) | 0.08 |
| H(1B) | 4*e* | –0.3439(8) | 0.4212(5) | 1.0116(6) | 0.08 |
| H(1C) | 4*e* | –0.2631(8) | 0.3486(5) | 1.1476(6) | 0.08 |
| H(4A) | 4*e* | –0.5072(7) | 0.1775(5) | 0.8953(5) | 0.08 |
| H(4B) | 4*e* | –0.4520(7) | 0.0785(5) | 0.8071(5) | 0.08 |
| H(5A) | 4*e* | –0.38607 | 0.07364 | 1.09911 | 0.08 |
| H(5B) | 4*e* | –0.31492 | –0.02162 | 1.01745 | 0.08 |
| H(31A) | 4*e* | –0.2580(8) | –0.0082(5) | 0.7615(5) | 0.08 |
| H(31B) | 4*e* | –0.0496(8) | –0.0336(5) | 0.8132(5) | 0.08 |
| H(31C) | 4*e* | –0.1292(8) | 0.0388(5) | 0.6768(5) | 0.08 |

**Table 3.** Atomic coordinates and displacement parameters (in Å$^2$).

| Atom | Site | $x$ | $y$ | $z$ | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|---|
| O(1) | 4*e* | –0.0397(5) | 0.3349(3) | 0.9996(4) | 0.056(2) | 0.058(2) | 0.084(2) | –0.013(2) | 0.025(2) | –0.011(2) |
| C(1) | 4*e* | –0.3193(8) | 0.3440(5) | 1.0491(6) | 0.074(4) | 0.065(4) | 0.087(4) | –0.004(3) | 0.039(3) | –0.006(3) |
| B(1) | 4*e* | 0.1069(8) | 0.2862(6) | 0.9502(7) | 0.053(4) | 0.071(4) | 0.068(4) | –0.011(3) | 0.019(3) | –0.005(3) |
| O(2) | 4*e* | 0.0469(5) | 0.1801(3) | 0.8649(3) | 0.056(2) | 0.067(2) | 0.070(2) | –0.010(2) | 0.024(2) | –0.014(2) |
| C(2) | 4*e* | –0.1940(7) | 0.2836(4) | 0.9829(5) | 0.055(3) | 0.053(3) | 0.059(3) | 0.001(2) | 0.023(2) | 0.004(2) |
| C(3) | 4*e* | –0.2397(6) | 0.1793(4) | 0.9079(4) | 0.058(3) | 0.048(3) | 0.048(3) | –0.007(2) | 0.019(2) | 0.003(2) |
| C(4) | 4*e* | –0.4177(7) | 0.1195(5) | 0.8942(5) | 0.055(3) | 0.062(3) | 0.062(3) | –0.010(3) | 0.014(2) | –0.000(3) |
| C(5) | 4*e* | –0.4132(7) | 0.0333(5) | 1.0088(5) | 0.060(3) | 0.057(3) | 0.075(3) | –0.009(3) | 0.022(3) | 0.008(3) |
| F(11) | 4*e* | 0.2428(5) | 0.2507(4) | 1.0649(4) | 0.079(3) | 0.135(3) | 0.105(3) | 0.014(3) | 0.003(2) | –0.044(3) |
| F(10) | 4*e* | 0.1682(6) | 0.3632(3) | 0.8748(4) | 0.158(4) | 0.084(3) | 0.145(4) | –0.045(3) | 0.098(3) | –0.013(2) |
| C(30) | 4*e* | –0.1118(7) | 0.1328(4) | 0.8509(5) | 0.063(3) | 0.051(3) | 0.052(3) | –0.005(3) | 0.015(2) | 0.006(2) |
| C(31) | 4*e* | –0.1397(8) | 0.0225(5) | 0.7684(5) | 0.091(4) | 0.065(3) | 0.079(4) | –0.005(3) | 0.030(4) | –0.015(3) |

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## References

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