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# The new chiral ligand 3-ethoxy-4-[(1*R*,2S*S*)-(2-hydroxy-1,2-diphenylethyl)amino]-3-cyclobutene-1,2-dione

**Haibing Zhou,[a] Yi Yuan,[a] Albert S. C. Chan,[b] Teng-Kuei Yang[c] and Rugang Xie[a]***

[a]Institute of Bioorganic Chemistry, Sichuan University, Chengdu 610064, People's Republic of China, [b]Union Laboratory of Asymmetric Synthesis and Department of Applied Biology, and Chemical Technology, The Hong Kong Polytechnic University, Hong Kong, and [c]Department of Chemistry, Chung-Hsing University, Taichung, Taiwan
Correspondence e-mail: schemorg@mail.sc.cninfo.net

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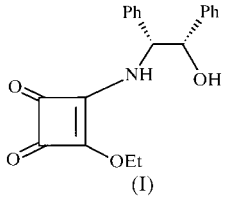
The asymmetric unit of $C_{20}H_{19}NO_4$ contains two molecules with slightly different conformations. In the crystal, the molecules are linked by O—H···O and N—H···O hydrogen bonds [O···O 2.764 (3) and 2.811 (3) Å; N···O 2.907 (3) and 2.968 (3) Å] to form a two-dimensional network.

## Comment

Squaric acid and its derivatives have long been known as aromatic ring systems having unique characteristics and wide application, but reports on the synthesis of chiral derivatives of squaric acid are few and their application in asymmetric catalytic reactions is totally unprecedented. We report herein for the first time the crystal structure of the new chiral ligand 3-ethoxy-4-[(1*R*,2S*S*)-(2-hydroxy-1,2-diphenylethyl)amino]-3-cyclobutene-1,2-dione, (I), which contains two independent molecules with slightly different conformations, they are linked by O—H···O and N—H···O hydrogen bonds (see Table 1) to form a two-dimensional network. This ligand may provide an effect chiral environment for coordination of the substrate in the reactions, such as the enantioselective catalytic borane reduction of prochiral ketones and addition of dialkylzinc to aldehydes *etc*, ascribed to the combining the aromatic squaric acid moiety of rigidity tetracarbon ring and



(I)

flexible ethoxy group with the chiral aminoalcohol, that proved to be the effective catalyst for the reactions mentioned above when the new ligand was used.

## Experimental

The title compound was prepared by reaction of 3,4-diethoxy-3-cyclobutene-1,2-dione and (1*S*,2*R*)-(±)-2-amino-1,2-diphenylethanol in the presence of triethylamine (molar ratio 1.1:1:1) in ethanol and recrystallized from it.

*Crystal data*

$C_{20}H_{19}NO_4$
$M_r$ = 337.36
Monoclinic, $P2_1$
$a$ = 10.758 (2) Å
$b$ = 15.879 (3) Å
$c$ = 11.040 (2) Å
$\beta$ = 112.740 (10)°
$V$ = 1739.3 (6) Å$^3$
$Z$ = 4
$D_x$ = 1.288 Mg m$^{-3}$
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta$ = 3.41–17.40°
$\mu$ = 0.090 mm$^{-1}$
$T$ = 296 (2) K
Flake, colourless
0.48 × 0.38 × 0.36 mm

*Data collection*

Bruker $P4$ diffractometer
$\omega$ scans
5817 measured reflections
4871 independent reflections
3018 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.016
$\theta_{max}$ = 28.50°
$h = -1 \rightarrow 14$
$k = -1 \rightarrow 21$
$l = -14 \rightarrow 14$
3 standard reflections every 97 reflections
intensity decay: 3.98%

*Refinement*

Refinement on $F^2$
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.073
$S$ = 0.846
4871 reflections
456 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0321P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.159 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.141 e Å$^{-3}$
Extinction correction: *SHELXL*97 (Sheldrick, 1997*a*)
Extinction coefficient: 0.0168 (8)

**Table 1**
Hydrogen-bonding geometry (Å, °).

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| O1—H1O···O3$^{i}$ | 0.82 | 2.01 | 2.811 (3) | 165 |
| O1′—H1′O···O3′$^{ii}$ | 0.82 | 1.95 | 2.764 (3) | 173 |
| N—H···O2′$^{iii}$ | 0.86 | 2.06 | 2.907 (3) | 169 |
| N'—H'···O2 | 0.86 | 2.15 | 2.968 (3) | 159 |

Symmetry codes: (i) $1-x, y-\frac{1}{2}, 1-z$; (ii) $-x, \frac{1}{2}+y, -z$; (iii) $1+x, y, 1+z$.

Data collection: *XSCANS* (Siemens, 1994); cell refinement: *XSCANS*; data reduction: *SHELXTL*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997*b*); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997*a*); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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