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4,4′,6,6′-Tetrahydroseleno-2,2′-[(*E*,*E*)-cyclohexane-1,2-diylbis(nitrilomethylidyne)]diphenol

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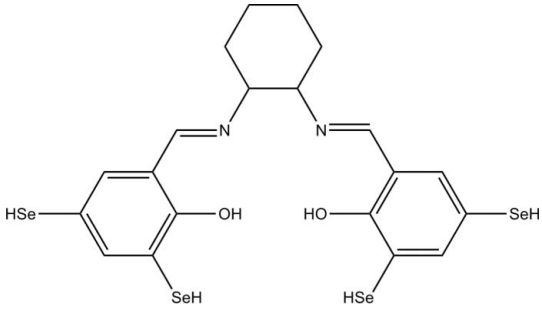
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.007 Å; R factor = 0.041; wR factor = 0.105; data-to-parameter ratio = 16.5.

In the title molecule, $C_{20}H_{22}N_2O_2Se_4$, the dihedral angle between the pendant aromatic rings is 67.1 (2)°. The conformation is stabilized by two intramolecular O—H···N hydrogen bonds.

Related literature

For background to the biological activity of Schiff base compounds, see: Shi *et al.* (2007). For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{20}H_{22}N_2O_2Se_4$
M_r = 638.24
Monoclinic, $P2_1/c$
a = 15.493 (2) Å
b = 9.2975 (15) Å
c = 16.353 (2) Å
β = 109.865 (5)°
V = 2215.4 (5) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 6.64 mm^{-1}
T = 296 K
0.32 × 0.28 × 0.24 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.225, T_{max} = 0.299
(expected range = 0.153–0.203)
11901 measured reflections
4196 independent reflections
2743 reflections with $I > 2\sigma(I)$
R_{int} = 0.048
3 standard reflections
every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.041
$wR(F^2)$ = 0.105
S = 1.06
4196 reflections
255 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O2—H2A···N2	0.82	1.85	2.575 (5)	147
O1—H1···N1	0.82	1.90	2.619 (5)	145

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

The project was supported by the Scientific Research Foundation for Returned Overseas Chinese Scholars, State Education Ministry, Educational Commission of Hubei Province (D20091703) and the Natural Science Foundation of Hubei Province (2008CDB038).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5041).

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4,4′,6,6′-Tetrahydroseleno-2,2′-[(*E*,*E*)-cyclohexane-1,2-diylbis(nitrilomethylidyne)]diphenol

Qiang Wang, Ru-Hua Zha, Ji-Wen Yuan and Qing-Fu Zeng

S1. Comment

There has been much research interest in Schiff base compounds due to their biological activities (Shi *et al.*, 2007). In this work, we report here the crystal structure of the title compound, (I). In (I), all bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). There are two intramolecular O—H···N hydrogen bonds in (I) (Table 1).

S2. Experimental

A mixture of 3,5-dihydroseleno-2-hydroxybenzaldehyde (564 mg, 2 mmol) and cyclohexane-1,2-diamine (114 mg, 1 mmol) in methanol (10 ml) was stirred for 2 h. After keeping the filtrate in air for 5 d, yellow blocks of (I) were formed.

S3. Refinement

The Se bound H atoms were located in a difference map and refined as riding in their as-found relative positions. The C-bound H atoms were positioned geometrically (C—H = 0.93 Å for the aromatic H atoms and C—H = 0.96 Å for the aliphatic H atoms) and were refined as riding, with U_{iso}(H) = 1.2U_{eq}(C).

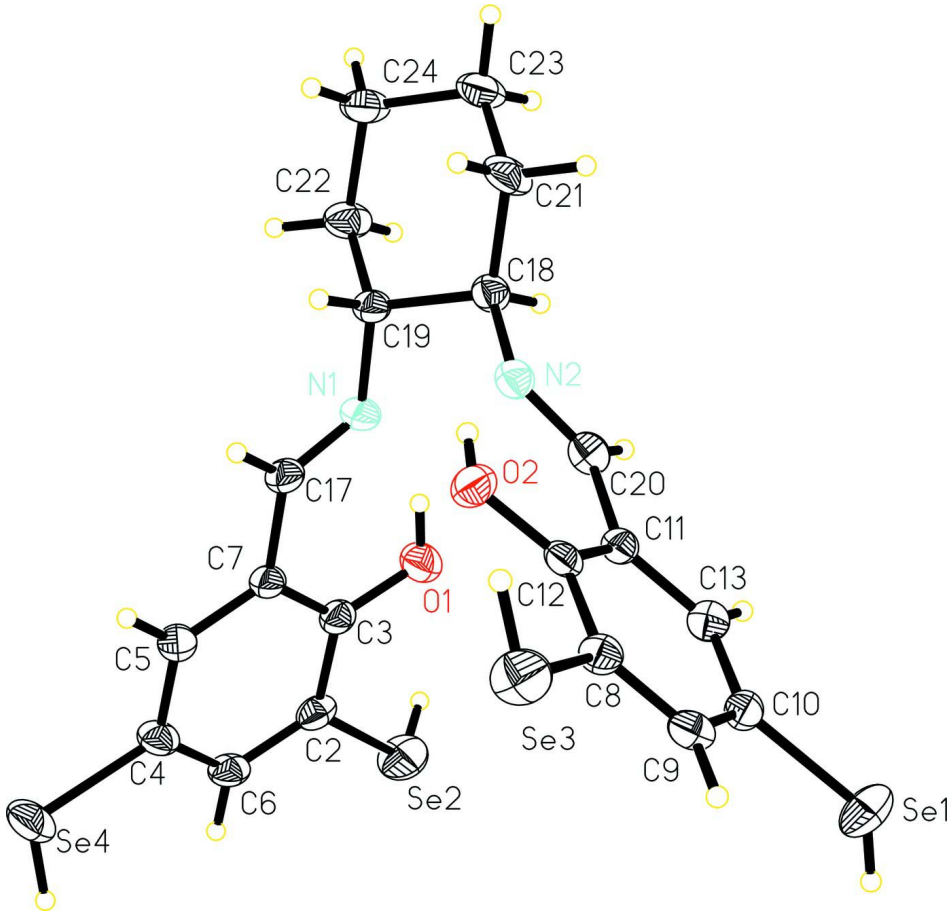

Figure 1

The molecular structure of (I) showing 30% probability displacement ellipsoids.

4,4′,6,6′-Tetrahydroseleno-2,2′-[(*E*,*E*)-cyclohexane-1,2- diylbis(nitrilomethylidyne)]diphenol

Crystal data

$C_{20}H_{22}N_2O_2Se_4$
$M_r = 638.24$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 15.493\ (2)$ Å
$b = 9.2975\ (15)$ Å
$c = 16.353\ (2)$ Å
$\beta = 109.865\ (5)°$
$V = 2215.4\ (5)$ Å^3
$Z = 4$
$F(000) = 1232$
$D_x = 1.914$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9–12°$
$\mu = 6.64$ mm^{-1}
$T = 296$ K
Block, yellow
0.32 × 0.28 × 0.24 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.225$, $T_{max} = 0.299$
11901 measured reflections
4196 independent reflections
2743 reflections with $I > 2\sigma(I)$
$R_{int} = 0.048$
$\theta_{max} = 25.7°$, $\theta_{min} = 1.4°$
$h = -18\rightarrow18$

$k = -9 \rightarrow 11$
$l = -19 \rightarrow 19$

3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.041$
$wR(F^2) = 0.105$
$S = 1.06$
4196 reflections
255 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.84$ e Å^{-3}
$\Delta\rho_{min} = -0.54$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Se1	1.08983 (4)	0.95743 (7)	1.27220 (3)	0.0629 (2)
Se2	0.64176 (5)	0.45492 (8)	1.21754 (4)	0.0694 (2)
Se3	0.87275 (4)	1.30994 (6)	1.00077 (4)	0.0618 (2)
Se4	0.38089 (4)	0.88004 (8)	1.06267 (4)	0.0683 (2)
O2	0.8152 (2)	1.0256 (4)	0.9129 (2)	0.0464 (9)
H2A	0.8043	0.9482	0.8874	0.070*
C20	0.8744 (3)	0.7422 (5)	0.9727 (3)	0.0397 (11)
H20	0.8937	0.6520	0.9965	0.048*
N2	0.8211 (2)	0.7512 (4)	0.8942 (2)	0.0400 (10)
N1	0.6651 (3)	0.5930 (4)	0.8980 (2)	0.0414 (10)
O1	0.6862 (2)	0.4896 (4)	1.0525 (2)	0.0544 (9)
H1	0.6915	0.4905	1.0043	0.082*
C8	0.9110 (3)	1.1261 (5)	1.0457 (3)	0.0373 (11)
C2	0.5888 (3)	0.5808 (5)	1.1248 (3)	0.0414 (12)
C17	0.6070 (3)	0.6798 (6)	0.9083 (3)	0.0412 (12)
H17	0.5811	0.7480	0.8654	0.049*
C9	0.9725 (3)	1.1108 (5)	1.1272 (3)	0.0399 (12)
H9	0.9948	1.1914	1.1616	0.048*
C10	1.0019 (3)	0.9748 (6)	1.1593 (3)	0.0388 (12)
C11	0.9058 (3)	0.8696 (5)	1.0263 (3)	0.0341 (11)
C3	0.6196 (3)	0.5804 (5)	1.0534 (3)	0.0384 (12)
C18	0.7924 (3)	0.6202 (5)	0.8436 (3)	0.0383 (11)

H18	0.8229	0.5374	0.8786	0.046*
C4	0.4792 (3)	0.7638 (6)	1.0590 (3)	0.0441 (13)
C5	0.5092 (3)	0.7665 (6)	0.9892 (3)	0.0448 (13)
H5	0.4822	0.8297	0.9435	0.054*
C6	0.5190 (3)	0.6701 (6)	1.1264 (3)	0.0431 (13)
H6	0.4983	0.6672	1.1735	0.052*
C7	0.5788 (3)	0.6770 (5)	0.9855 (3)	0.0384 (12)
C12	0.8760 (3)	1.0062 (5)	0.9929 (3)	0.0343 (11)
C19	0.6881 (3)	0.6039 (5)	0.8186 (3)	0.0381 (12)
H19	0.6579	0.6888	0.7856	0.046*
C13	0.9695 (3)	0.8555 (5)	1.1101 (3)	0.0391 (11)
H13	0.9899	0.7648	1.1322	0.047*
C21	0.8179 (3)	0.6275 (6)	0.7615 (3)	0.0564 (15)
H21A	0.7892	0.7109	0.7275	0.068*
H21B	0.8839	0.6378	0.7773	0.068*
C22	0.6558 (4)	0.4706 (6)	0.7630 (3)	0.0545 (15)
H22A	0.6815	0.3860	0.7974	0.065*
H22B	0.5895	0.4642	0.7454	0.065*
C23	0.7866 (3)	0.4919 (6)	0.7072 (4)	0.0566 (16)
H23A	0.8183	0.4091	0.7398	0.068*
H23B	0.8021	0.4990	0.6546	0.068*
C24	0.6834 (3)	0.4719 (6)	0.6833 (3)	0.0553 (15)
H24A	0.6517	0.5494	0.6451	0.066*
H24B	0.6652	0.3819	0.6521	0.066*
H2D	0.6487	0.3556	1.1959	0.066*
H3D	0.8639	1.2980	0.9337	0.066*
H1D	1.0891	0.9500	1.3302	0.066*
H4D	0.3812	0.8789	1.1309	0.066*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Se1	0.0686 (4)	0.0697 (5)	0.0371 (3)	−0.0124 (3)	0.0004 (3)	0.0052 (3)
Se2	0.0850 (5)	0.0807 (5)	0.0424 (3)	0.0074 (4)	0.0217 (3)	0.0183 (3)
Se3	0.0805 (4)	0.0374 (3)	0.0620 (4)	0.0104 (3)	0.0170 (3)	0.0004 (3)
Se4	0.0677 (4)	0.0887 (5)	0.0628 (4)	0.0210 (4)	0.0410 (3)	−0.0011 (3)
O2	0.049 (2)	0.046 (2)	0.0379 (19)	0.0044 (19)	0.0062 (17)	−0.0063 (16)
C20	0.043 (3)	0.031 (3)	0.048 (3)	0.002 (2)	0.019 (3)	0.002 (2)
N2	0.035 (2)	0.045 (3)	0.042 (2)	−0.003 (2)	0.0160 (19)	−0.005 (2)
N1	0.042 (2)	0.054 (3)	0.034 (2)	−0.001 (2)	0.0202 (19)	−0.003 (2)
O1	0.055 (2)	0.067 (3)	0.046 (2)	0.014 (2)	0.0226 (19)	0.0060 (19)
C8	0.042 (3)	0.037 (3)	0.036 (3)	0.000 (2)	0.017 (2)	0.002 (2)
C2	0.043 (3)	0.052 (3)	0.028 (2)	−0.012 (3)	0.011 (2)	−0.001 (2)
C17	0.040 (3)	0.057 (4)	0.028 (2)	−0.002 (3)	0.013 (2)	0.001 (2)
C9	0.046 (3)	0.035 (3)	0.047 (3)	−0.005 (3)	0.026 (3)	−0.006 (2)
C10	0.034 (3)	0.052 (3)	0.033 (3)	−0.003 (3)	0.013 (2)	0.001 (2)
C11	0.032 (2)	0.038 (3)	0.035 (3)	−0.003 (2)	0.014 (2)	−0.005 (2)
C3	0.034 (3)	0.048 (3)	0.033 (3)	−0.008 (2)	0.011 (2)	−0.006 (2)

C18	0.036 (3)	0.037 (3)	0.043 (3)	−0.002 (2)	0.015 (2)	−0.009 (2)
C4	0.044 (3)	0.055 (3)	0.042 (3)	−0.003 (3)	0.025 (2)	−0.005 (3)
C5	0.043 (3)	0.060 (4)	0.034 (3)	0.005 (3)	0.016 (2)	0.003 (2)
C6	0.045 (3)	0.057 (4)	0.033 (3)	−0.011 (3)	0.021 (2)	−0.006 (3)
C7	0.033 (3)	0.054 (3)	0.030 (2)	−0.003 (2)	0.013 (2)	−0.005 (2)
C12	0.033 (3)	0.040 (3)	0.036 (3)	0.005 (2)	0.019 (2)	−0.001 (2)
C19	0.036 (3)	0.052 (3)	0.030 (2)	−0.005 (2)	0.015 (2)	−0.004 (2)
C13	0.043 (3)	0.037 (3)	0.039 (3)	0.000 (3)	0.015 (2)	0.003 (2)
C21	0.047 (3)	0.073 (4)	0.065 (3)	−0.020 (3)	0.040 (3)	−0.023 (3)
C22	0.052 (3)	0.071 (4)	0.049 (3)	−0.023 (3)	0.029 (3)	−0.018 (3)
C23	0.056 (3)	0.070 (4)	0.055 (3)	−0.012 (3)	0.034 (3)	−0.027 (3)
C24	0.057 (3)	0.074 (4)	0.043 (3)	−0.018 (3)	0.028 (3)	−0.019 (3)

Geometric parameters (Å, º)

Se1—C10	1.893 (5)	C10—C13	1.362 (7)
Se1—H1D	0.9542	C11—C13	1.396 (6)
Se2—C2	1.869 (5)	C11—C12	1.398 (6)
Se2—H2D	1.0078	C3—C7	1.400 (6)
Se3—C8	1.875 (5)	C18—C21	1.524 (6)
Se3—H3D	1.0645	C18—C19	1.534 (6)
Se4—C4	1.885 (5)	C18—H18	0.9800
Se4—H4D	1.1136	C4—C5	1.372 (6)
O2—C12	1.340 (5)	C4—C6	1.376 (7)
O2—H2A	0.8200	C5—C7	1.380 (6)
C20—N2	1.271 (6)	C5—H5	0.9300
C20—C11	1.455 (7)	C6—H6	0.9300
C20—H20	0.9300	C19—C22	1.518 (7)
N2—C18	1.455 (6)	C19—H19	0.9800
N1—C17	1.262 (6)	C13—H13	0.9300
N1—C19	1.461 (5)	C21—C23	1.523 (7)
O1—C3	1.337 (6)	C21—H21A	0.9700
O1—H1	0.8200	C21—H21B	0.9700
C8—C9	1.357 (6)	C22—C24	1.503 (6)
C8—C12	1.402 (6)	C22—H22A	0.9700
C2—C6	1.371 (6)	C22—H22B	0.9700
C2—C3	1.404 (6)	C23—C24	1.524 (7)
C17—C7	1.470 (6)	C23—H23A	0.9700
C17—H17	0.9300	C23—H23B	0.9700
C9—C10	1.385 (7)	C24—H24A	0.9700
C9—H9	0.9300	C24—H24B	0.9700
C10—Se1—H1D	136.7	C7—C5—H5	119.4
C2—Se2—H2D	111.0	C2—C6—C4	120.7 (4)
C8—Se3—H3D	102.8	C2—C6—H6	119.7
C4—Se4—H4D	107.3	C4—C6—H6	119.7
C12—O2—H2A	109.5	C5—C7—C3	120.1 (4)
N2—C20—C11	121.6 (5)	C5—C7—C17	119.4 (4)

N2—C20—H20	119.2	C3—C7—C17	120.4 (4)
C11—C20—H20	119.2	O2—C12—C11	122.2 (4)
C20—N2—C18	119.2 (4)	O2—C12—C8	119.4 (4)
C17—N1—C19	118.3 (4)	C11—C12—C8	118.3 (4)
C3—O1—H1	109.5	N1—C19—C22	110.2 (4)
C9—C8—C12	121.1 (5)	N1—C19—C18	108.9 (4)
C9—C8—Se3	120.3 (4)	C22—C19—C18	110.1 (4)
C12—C8—Se3	118.6 (3)	N1—C19—H19	109.2
C6—C2—C3	120.7 (4)	C22—C19—H19	109.2
C6—C2—Se2	119.7 (3)	C18—C19—H19	109.2
C3—C2—Se2	119.6 (4)	C10—C13—C11	120.0 (5)
N1—C17—C7	122.5 (4)	C10—C13—H13	120.0
N1—C17—H17	118.8	C11—C13—H13	120.0
C7—C17—H17	118.8	C23—C21—C18	110.5 (4)
C8—C9—C10	119.9 (5)	C23—C21—H21A	109.5
C8—C9—H9	120.0	C18—C21—H21A	109.5
C10—C9—H9	120.0	C23—C21—H21B	109.5
C13—C10—C9	120.8 (4)	C18—C21—H21B	109.5
C13—C10—Se1	120.4 (4)	H21A—C21—H21B	108.1
C9—C10—Se1	118.8 (4)	C24—C22—C19	112.3 (4)
C13—C11—C12	119.9 (4)	C24—C22—H22A	109.1
C13—C11—C20	119.7 (5)	C19—C22—H22A	109.1
C12—C11—C20	120.4 (4)	C24—C22—H22B	109.1
O1—C3—C7	122.6 (4)	C19—C22—H22B	109.1
O1—C3—C2	119.4 (4)	H22A—C22—H22B	107.9
C7—C3—C2	118.0 (4)	C21—C23—C24	110.4 (4)
N2—C18—C21	110.1 (4)	C21—C23—H23A	109.6
N2—C18—C19	108.9 (4)	C24—C23—H23A	109.6
C21—C18—C19	109.6 (4)	C21—C23—H23B	109.6
N2—C18—H18	109.4	C24—C23—H23B	109.6
C21—C18—H18	109.4	H23A—C23—H23B	108.1
C19—C18—H18	109.4	C22—C24—C23	111.2 (4)
C5—C4—C6	119.5 (4)	C22—C24—H24A	109.4
C5—C4—Se4	121.5 (4)	C23—C24—H24A	109.4
C6—C4—Se4	119.0 (3)	C22—C24—H24B	109.4
C4—C5—C7	121.1 (5)	C23—C24—H24B	109.4
C4—C5—H5	119.4	H24A—C24—H24B	108.0

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···N2	0.82	1.85	2.575 (5)	147
O1—H1···N1	0.82	1.90	2.619 (5)	145