Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis(*N*,*N*′-di-2-furylethane-1,2-diamine-κ^2N,N')bis(perchlorato-κO)copper(II)

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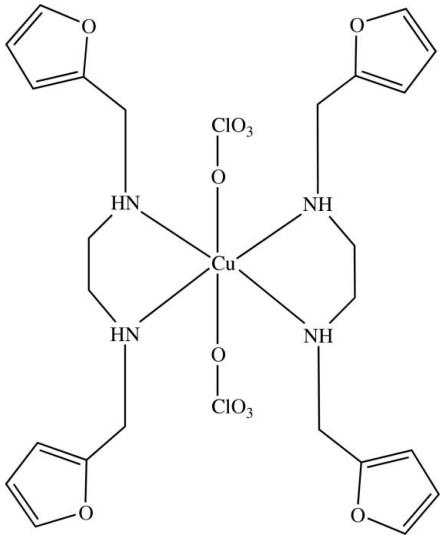
Received 16 April 2007; accepted 20 April 2007

Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.006 Å; R factor = 0.050; wR factor = 0.106; data-to-parameter ratio = 14.2.

In the title complex, $[Cu(ClO_4)_2(C_{12}H_{16}N_2O_2)_2]$, the six-coordinate Cu atom lies on an inversion center with four amine N atoms in the equatorial plane, with Cu—N distances of 2.049 (3) and 2.055 (3) Å, and two perchlorate O atoms in axial positions, with Cu—O distances of 2.580 (2) Å.

Related literature

For related literature, see: Akitsu & Einaga (2003); Hori *et al.* (2001); Karunakaran & Kandaswamy (1994); McCollum *et al.* (1994); Rameau (1938); Sun *et al.* (2001).



Experimental

Crystal data

$[Cu(ClO_4)_2(C_{12}H_{16}N_2O_2)_2]$
M_r = 702.98
Monoclinic, $P2_1/n$
a = 10.0062 (7) Å
b = 9.6093 (7) Å
c = 15.1933 (11) Å
β = 103.371 (1)°
V = 1421.27 (18) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.03 mm^{-1}
T = 291 (2) K
0.30 × 0.24 × 0.22 mm

Data collection

Bruker SMART APEX CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.70, T_{max} = 0.80
8032 measured reflections
2786 independent reflections
2068 reflections with $I > 2\sigma(I)$
R_{int} = 0.045

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.106
S = 1.03
2786 reflections
196 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.43 e Å^{-3}
$\Delta\rho_{min}$ = −0.75 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···O11	0.91	2.18	3.021 (4)	154

Data collection: *SMART* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank the National Science Foundation of China (No. 20271039).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2227).

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 doi:10.1107/S1600536807019885

supplementary materials

Acta Cryst. (2007). E**63**, m1545 [**doi:10.1107/S1600536807019885**]

Bis(*N*,*N*'-di-2-furylethane-1,2-diamine-κ^2*N*,*N*')bis(perchlorato-κ*O*)copper(II)

X.-H. Wang, H.-P. Zhang, J.-D. Hu, Z.-Q. Pan, Y.-Z. Li and H. Zhou

Comment

The synthesis of phenol-based macrocyclic ligands having dissimilar coordination environments and their functional bimetallic complexes has lead to increasing attention to their potentially unique properties (Karunakaran *et al.*, 1994; Hori *et al.*, 2001; McCollum *et al.*, 1994;). The compartmental ligand synthesized by the precursor diamine N, *N*'-bis(2-furyl)-1,2-diaminoethane has been reported (Sun *et al.*, 2001). However, the crystal structures of Cu(II) complex with perchlorate anions participating in coordination of the precursor compound have not been reported. In this paper, we reported the structure of the title complex Bis[N, *N*'-bis(2-furyl)-1,2-diaminoethane-κ*N*,κ*N*'] bis(perchlorato-κ*O*)copper(II), (I).

In the structure of (I) the copper atom is six coordinate with four N atoms and two O atoms (Fig. 1). The equatorial positions completed by four nitrogen atoms from two diamines in which the distance of Cu—N are 2.049 (3) and 2.055 (3) Å. The Cu(II) atom lies on a crystallographic inversion centre at the centre of the *N*(amine)$_4$ plane. Two perchlorate O atoms occupy the axial positions with the Cu—O distance of 2.580 (2) Å. Intramolecular N—H···O hydrogen bonds play an important role in stabilizing the axial coordination of the perchlorate anions.

Experimental

N, *N*'-bis(2-furyl)-1,2-diaminoethane was prepared using a variant of the method suggested by Rameau (1938). The title complex was synthesized by the following procedure: a solution of N, *N*'-bis(2-furyl)-1,2-diaminoethane (2 mmol) in 15 ml of absolute methanol was added dropwise a methanol solution (10 ml) of $Cu(OAc)_2H_2O$ (1 mmol). The mixture was stirred at ambient temperature for about 3 h and then a dark blue solution appeared. A methanol solution (5 ml) of $NaClO_4H_2O$ (2 mmol) was added to the mixture and the stirring was continued for 2 h. Blue block crystals of the title complex suitable for X-ray diffraction precipitated in about a month.

Refinement

All H atoms were placed in calculated positions, with C—H distances 0.93, 0.97 Å, and N—H distances 0.91 Å, and included in the refinement in the riding-model approximation, with U_{iso}(H) = 1.2–1.5 U_{eq}(C/N). Data collection: *SMART*(Bruker, 2000); cellrefinement: *SAINT*(Bruker, 2000); data reduction: *SAINT*; program(*s*) used to solve structure: *SHELXTL*(Bruker, 2000); program(*s*) used to refine structure: *SHELXTL SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

Figures

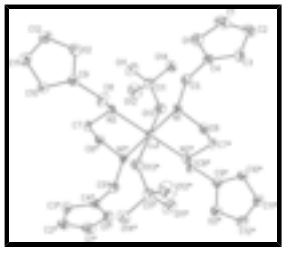


Fig. 1. A view of the title complex cation, showing the labeling of the non-H atoms and 30% probability ellipsoids. H atoms have been omitted. Atoms marked with an asterisk(*) are at the symmetry-generated position (Symmetry code for primed atoms: $-x$, $1-y$, $2-z$).

Bis(*N*,*N*'-di-2-furylethane-1,2-diamine-κ^2*N*,*N*')bis(perchlorato-κ*O*)copper(II)

Crystal data

$[Cu(ClO_4)_2(C_{12}H_{16}N_2O_2)_2]$	F_{000} = 726
M_r = 702.98	D_x = 1.643 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 3385 reflections
a = 10.0062 (7) Å	θ = 2.1–25,56º
b = 9.6093 (7) Å	μ = 1.03 mm^{-1}
c = 15.1933 (11) Å	T = 291 (2) K
β = 103.3710 (10)º	Block, blue
V = 1421.27 (18) Å^3	0.30 × 0.24 × 0.22 mm
Z = 2	

Data collection

Bruker SMART APEX CCD diffractometer	2786 independent reflections
Radiation source: sealed tube	2068 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.045
T = 291(2) K	θ_{max} = 26.0º
φ and ω scans	θ_{min} = 2.2º
Absorption correction: multi-scan (SADABS; Bruker, 2000)	h = −12→12
T_{min} = 0.7, T_{max} = 0.8	k = −11→11
8032 measured reflections	l = −12→18

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained
$wR(F^2)$ = 0.106	$w = 1/[\sigma^2(F_o^2) + (0.05P)^2 + 0.22P]$ where $P = (F_o^2 + 2F_c^2)/3$

S = 1.03	$(\Delta/\sigma)_{max} < 0.001$
2786 reflections	$\Delta\rho_{max}$ = 0.43 e Å^{-3}
196 parameters	$\Delta\rho_{min}$ = −0.75 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.2589 (5)	0.0200 (5)	0.8115 (3)	0.0586 (12)
H1	0.3444	−0.0139	0.8077	0.070*
C2	0.1440 (5)	−0.0538 (4)	0.7913 (3)	0.0525 (10)
H2	0.1340	−0.1453	0.7709	0.063*
C3	0.0384 (4)	0.0363 (4)	0.8075 (3)	0.0457 (9)
H3	−0.0542	0.0152	0.8001	0.055*
C4	0.1012 (3)	0.1590 (4)	0.8358 (2)	0.0341 (7)
C5	0.0510 (4)	0.2937 (4)	0.8638 (2)	0.0373 (8)
H5A	−0.0471	0.2999	0.8381	0.045*
H5B	0.0947	0.3686	0.8381	0.045*
C6	0.0145 (3)	0.2074 (3)	1.0105 (2)	0.0324 (7)
H6A	0.0573	0.2077	1.0746	0.039*
H6B	0.0287	0.1166	0.9862	0.039*
C7	0.1361 (3)	0.7639 (3)	1.0038 (2)	0.0359 (8)
H7A	0.1777	0.8306	0.9703	0.043*
H7B	0.1804	0.7723	1.0675	0.043*
C8	0.2997 (4)	0.5723 (4)	1.0038 (3)	0.0457 (9)
H8A	0.3193	0.5606	1.0690	0.055*
H8B	0.3080	0.4817	0.9774	0.055*
C9	0.4052 (4)	0.6668 (4)	0.9813 (2)	0.0386 (8)
C10	0.4827 (4)	0.7663 (5)	1.0263 (3)	0.0518 (11)
H10	0.4842	0.7954	1.0849	0.062*
C11	0.5631 (4)	0.8205 (5)	0.9692 (3)	0.0517 (10)
H11	0.6276	0.8917	0.9830	0.062*
C12	0.5297 (4)	0.7519 (5)	0.8937 (3)	0.0537 (10)
H12	0.5671	0.7664	0.8439	0.064*
Cl1	−0.13795 (9)	0.60868 (10)	0.76242 (6)	0.0420 (2)

Cu1	0.0000	0.5000	1.0000	0.02706 (16)
N1	0.0763 (3)	0.3160 (3)	0.96415 (17)	0.0272 (6)
H1A	0.1686	0.3153	0.9874	0.033*
N2	0.1541 (3)	0.6213 (3)	0.97196 (19)	0.0292 (6)
H2A	0.1380	0.6263	0.9106	0.035*
O1	0.2368 (3)	0.1501 (3)	0.8381 (2)	0.0499 (7)
O2	0.4316 (3)	0.6552 (3)	0.89835 (19)	0.0560 (7)
O11	0.0075 (3)	0.6289 (3)	0.7745 (2)	0.0543 (7)
O12	−0.2058 (3)	0.7378 (3)	0.7460 (2)	0.0690 (9)
O13	−0.1621 (3)	0.5492 (3)	0.84465 (16)	0.0443 (6)
O14	−0.1861 (3)	0.5187 (3)	0.68809 (19)	0.0562 (7)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.071 (3)	0.055 (3)	0.055 (3)	0.021 (2)	0.027 (2)	−0.003 (2)
C2	0.085 (3)	0.035 (2)	0.043 (2)	0.010 (2)	0.026 (2)	0.0018 (18)
C3	0.055 (2)	0.034 (2)	0.050 (2)	−0.0114 (16)	0.0143 (18)	−0.0025 (16)
C4	0.0398 (19)	0.0319 (18)	0.0322 (18)	0.0031 (14)	0.0118 (15)	0.0042 (14)
C5	0.050 (2)	0.0312 (18)	0.0314 (18)	0.0001 (15)	0.0101 (15)	0.0062 (14)
C6	0.0460 (19)	0.0212 (15)	0.0304 (18)	0.0043 (14)	0.0096 (15)	0.0031 (13)
C7	0.0460 (19)	0.0264 (17)	0.039 (2)	−0.0131 (14)	0.0182 (16)	−0.0034 (14)
C8	0.0340 (18)	0.041 (2)	0.062 (3)	−0.0051 (16)	0.0112 (17)	0.0145 (19)
C9	0.0384 (18)	0.047 (2)	0.0314 (18)	−0.0067 (16)	0.0104 (15)	−0.0036 (15)
C10	0.056 (2)	0.061 (3)	0.045 (2)	−0.023 (2)	0.0239 (19)	−0.021 (2)
C11	0.054 (2)	0.052 (3)	0.050 (3)	−0.0142 (19)	0.015 (2)	−0.002 (2)
C12	0.053 (2)	0.069 (3)	0.043 (2)	−0.012 (2)	0.0191 (19)	0.003 (2)
Cl1	0.0446 (5)	0.0399 (5)	0.0386 (5)	0.0043 (4)	0.0037 (4)	0.0034 (4)
Cu1	0.0272 (3)	0.0256 (3)	0.0288 (3)	−0.0034 (2)	0.0074 (2)	−0.0020 (2)
N1	0.0300 (13)	0.0224 (14)	0.0292 (14)	0.0006 (10)	0.0066 (11)	0.0003 (11)
N2	0.0326 (14)	0.0235 (14)	0.0304 (14)	−0.0060 (11)	0.0050 (11)	−0.0001 (11)
O1	0.0443 (14)	0.0449 (16)	0.0651 (18)	−0.0031 (12)	0.0221 (13)	−0.0113 (13)
O2	0.0613 (17)	0.0652 (19)	0.0438 (16)	−0.0189 (14)	0.0167 (13)	−0.0143 (14)
O11	0.0411 (14)	0.069 (2)	0.0525 (17)	−0.0067 (13)	0.0108 (12)	0.0176 (14)
O12	0.078 (2)	0.052 (2)	0.070 (2)	0.0331 (16)	0.0033 (17)	0.0144 (16)
O13	0.0485 (14)	0.0503 (15)	0.0328 (13)	−0.0055 (12)	0.0068 (11)	0.0001 (11)
O14	0.0743 (18)	0.0513 (18)	0.0442 (16)	−0.0127 (14)	0.0162 (14)	−0.0109 (13)

Geometric parameters (Å, °)

C1—C2	1.325 (6)	C8—H8A	0.9700
C1—O1	1.348 (5)	C8—H8B	0.9700
C1—H1	0.9300	C9—C10	1.319 (5)
C2—C3	1.430 (6)	C9—O2	1.351 (4)
C2—H2	0.9300	C10—C11	1.413 (5)
C3—C4	1.359 (5)	C10—H10	0.9300
C3—H3	0.9300	C11—C12	1.298 (6)
C4—O1	1.351 (4)	C11—H11	0.9300
C4—C5	1.486 (5)	C12—O2	1.365 (5)

C5—N1	1.502 (4)	C12—H12	0.9300
C5—H5A	0.9700	Cl1—O12	1.408 (3)
C5—H5B	0.9700	Cl1—O14	1.415 (3)
C6—N1	1.472 (4)	Cl1—O11	1.437 (3)
C6—C7^{i}	1.497 (5)	Cl1—O13	1.444 (3)
C6—H6A	0.9700	Cu1—N1^{i}	2.049 (3)
C6—H6B	0.9700	Cu1—N1	2.049 (3)
C7—N2	1.478 (4)	Cu1—N2	2.055 (3)
C7—C6^{i}	1.497 (5)	Cu1—N2^{i}	2.055 (3)
C7—H7A	0.9700	Cu1—O13	2.580 (2)
C7—H7B	0.9700	N1—H1A	0.9100
C8—C9	1.491 (5)	N2—H2A	0.9099
C8—N2	1.500 (4)		
C2—C1—O1	111.9 (4)	C9—C10—H10	126.3
C2—C1—H1	124.1	C11—C10—H10	126.3
O1—C1—H1	124.1	C12—C11—C10	106.7 (4)
C1—C2—C3	105.8 (4)	C12—C11—H11	126.6
C1—C2—H2	127.1	C10—C11—H11	126.6
C3—C2—H2	127.1	C11—C12—O2	110.0 (4)
C4—C3—C2	105.9 (4)	C11—C12—H12	125.0
C4—C3—H3	127.0	O2—C12—H12	125.0
C2—C3—H3	127.0	O12—Cl1—O14	109.6 (2)
O1—C4—C3	109.9 (3)	O12—Cl1—O11	109.6 (2)
O1—C4—C5	116.9 (3)	O14—Cl1—O11	109.43 (19)
C3—C4—C5	133.2 (3)	O12—Cl1—O13	109.36 (19)
C4—C5—N1	115.2 (3)	O14—Cl1—O13	110.76 (17)
C4—C5—H5A	108.5	O11—Cl1—O13	108.06 (16)
N1—C5—H5A	108.5	N1^{i}—Cu1—N1	180.000 (1)
C4—C5—H5B	108.5	N1^{i}—Cu1—N2	84.79 (10)
N1—C5—H5B	108.5	N1—Cu1—N2	95.21 (10)
H5A—C5—H5B	107.5	N1^{i}—Cu1—N2^{i}	95.21 (10)
N1—C6—C7^{i}	108.7 (3)	N1—Cu1—N2^{i}	84.79 (10)
N1—C6—H6A	110.0	N2—Cu1—N2^{i}	180.0
C7^{i}—C6—H6A	110.0	N1^{i}—Cu1—O13	83.74 (10)
N1—C6—H6B	110.0	N1—Cu1—O13	96.26 (10)
C7^{i}—C6—H6B	110.0	N2—Cu1—O13	92.84 (10)
H6A—C6—H6B	108.3	N2^{i}—Cu1—O13	87.16 (10)
N2—C7—C6^{i}	108.6 (3)	C6—N1—C5	113.2 (3)
N2—C7—H7A	110.0	C6—N1—Cu1	105.13 (19)
C6^{i}—C7—H7A	110.0	C5—N1—Cu1	114.0 (2)
N2—C7—H7B	110.0	C6—N1—H1A	108.1
C6^{i}—C7—H7B	110.0	C5—N1—H1A	108.1
H7A—C7—H7B	108.4	Cu1—N1—H1A	108.1
C9—C8—N2	114.9 (3)	C7—N2—C8	111.6 (3)
C9—C8—H8A	108.5	C7—N2—Cu1	107.67 (19)
N2—C8—H8A	108.5	C8—N2—Cu1	118.3 (2)

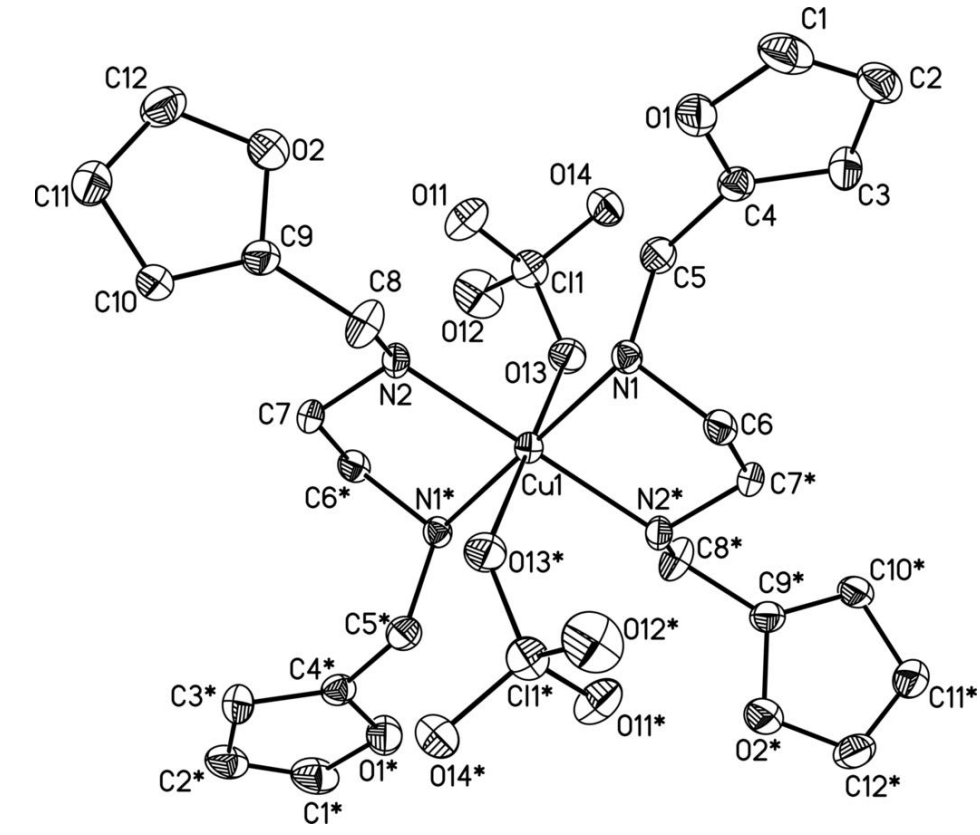
C9—C8—H8B	108.5	C7—N2—H2A	106.3
N2—C8—H8B	108.5	C8—N2—H2A	106.0
H8A—C8—H8B	107.5	Cu1—N2—H2A	106.2
C10—C9—O2	109.0 (3)	C1—O1—C4	106.5 (3)
C10—C9—C8	133.2 (4)	C9—O2—C12	106.9 (3)
O2—C9—C8	117.8 (3)	Cl1—O13—Cu1	131.97 (15)
C9—C10—C11	107.4 (3)		

Symmetry codes: (i) −*x*, −*y*+1, −*z*+2.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2A···O11	0.91	2.18	3.021 (4)	154

Fig. 1


C1
C2
O1
C3
C4
C5
N1
C6
C7*
N2*
C8*
C9*
C10*
C11*
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C12*
C12
O2
C11
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C10
C8
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O13*
O12*
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O11*
O14*