

C(81)'-C(81)-O(80) and C(81)-C(81)'-O(80)'. The presence of the solvent molecule did not significantly affect the refinement of the crystal structure.

References

- ALDOSHI, S. M., D'YACHENKO, O. A., TKACHEV, V. V. & ATOVMIAN, L. O. (1981). *Koord. Khim.* **7**, 287-296.
- BRODERSEN, V. K. & HUMMEL, H. U. (1982). *Z. Anorg. Allg. Chem.* **491**, 34-38.
- DOBLER, M. (1981). *Ionophores and Their Structures*. New York: John Wiley.
- HILGENFELD, R. & SAENGER, W. (1981). *Angew. Chem. Int. Ed. Engl.* **20**, 1045-1046.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- IZATT, R. M., EATOUGH, D. J. & CHRISTENSEN, J. J. (1973). *Thermodynamics of Cation-Macrocyclic Compound Interaction*. In *Structure and Bonding*, No. 16, New York: Springer-Verlag.
- JEFFREY, J. W. & ROSE, K. M. (1968). *Acta Cryst.* **B24**, 653-662.
- KAWASAKI, Y. & MATSUURA, Y. (1984). *Chem. Lett.* pp. 155-158.
- LARSON, S. B. & DALLEY, N. K. (1982). *Acta Cryst.* **B38**, 1309-1312.
- MOK, K. F. (1988). *Inorg. Chim. Acta*, **142** (1), 27-28.
- MOK, K. F., MCKEE, V. & ROBINSON, W. T. (1987). *Acta Cryst.* **C43**, 2093-2096.
- NAGANO, O. (1979). *Acta Cryst.* **B35**, 465-467.
- POLADYAN, V. E., ERMAKOV, A. A., SOKOLOVA, N. P., AVLESOVICH, L. M., PAKHOL'CHUK, S. F. & MUKELO, K. A. (1984). *Zh. Neorg. Khim.* **29**, 1192-1196.
- SHELDRIK, G. M. (1984). *SHELXTL*. Revision 4-1. Nicolet XRD Corporation, Madison, Wisconsin, USA.
- SHELDRIK, G. M. (1985). *SHELXTL*. Revision 5-1 (Eclipse 32K Version). Nicolet XRD Corporation, Madison, Wisconsin, USA.
- UDUPA, M. R. & KREBS, B. (1980). *Inorg. Chim. Acta*, **42**, 37-41.

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Structure of Tetrakis{[1-(phenyltriazene-1,3-diyl)-2-(phenyltriazenyl)benzene]copper(I)}

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Abstract. [$\{C_6H_5NNNC_6H_4NNN(H)C_6H_5\}Cu$]₄, $M_r = 1515.60$, orthorhombic, *Pbcn*, $a = 20.552$ (2), $b = 18.996$ (3), $c = 17.140$ (3) Å, $V = 6691.4$ Å³, $Z = 4$, $D_x = 1.504$ g cm⁻³, $\lambda(Cu K\alpha) = 1.54184$ Å, $\mu = 19.028$ cm⁻¹, $F(000) = 3104$, $T = 293$ K. The final R value is 0.058 for 2319 significant [$I > 3\sigma(I)$] reflections. In the tetrameric complex of site symmetry 2 each Cu atom of the distorted Cu₄ rhombus is bonded to two *ortho* N atoms and to one N atom of the bridging N1, N3 function of the ligands. The Cu...Cu distances are 2.650 (2) and 2.684 (2) Å, respectively.

Introduction. Triazeno ligands in metal complexes are monodentate, (N1,N3)-chelating and bridging (Moore & Robinson, 1986); (N1,N1)- and (N1,N2)-bridges and coordination *via* N2 or an η^3 -structure similar to the η^3 -allyl complexes have not yet been observed. We synthesized a new bis(triazene) ligand with both N₃

units in one molecule to extend the possibilities of coordination and to force close contacts between metal atoms.

Experimental. The reaction of 1,2-bisphenyltriazeno-benzene with Cu(NH₃)₄Cl in ether/methanol yields dark red crystals of the title compound. The parameters for intensity-data collection, structure solution and refinement are listed in Table 1. The final atomic coordinates are given in Table 2, selected bond distances and angles in Table 3.* Figs. 1 and 2 show the numbering scheme and a stereoview, respectively.

* Lists of structure factors, anisotropic thermal parameters, planes, further distances and angles, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51373 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Data collection and structure refinement parameters

Crystal shape, dimensions (mm)	Prismatic, 0.10 × 0.20 × 0.28
Diffractometer used	CAD-4, Enraf-Nonius
Method of intensity measurement	$\theta/2\theta$
Number and θ range ($^\circ$) of reflections for lattice parameters	25; 11–24
Absorption coefficient μ (cm^{-1})	19.028
Method used for absorption correction	DIFABS (Walker & Stuart, 1983)
Minimum absorption correction	0.654
Maximum absorption correction	1.171
Average absorption correction	0.981
Maximum value of $(\sin\theta/\lambda)$ reached in intensity measurement (\AA^{-1})	0.588
Range of h, k and l	0–24, 0–22, 0–20
Standard reflections	371; 480
Interval, std. reflections measured	2h, no intensity variation
Total number of reflections measured; θ range ($^\circ$)	6271; 65 (587 systematic absences included)
Number of observed reflections	2319 [$I > 3\sigma(I)$]
Methods used to solve structure	Patterson, subsequent difference Fourier
Refinement	Cu, N atomic positions with anisotropic thermal parameters, C isotropic
Use of F or F^2 in LS refinement	F
Method of locating hydrogens	Not found in ΔF map H's of phenyl groups calculated and only included in structure factor calculation
Weighting scheme	$1/\sigma^2$
Parameters refined	272
Value of R	0.058
Value of wR	0.055
Ratio of max. LS shift to e.s.d. (Δ/σ)	0.017
Max. height in final ΔF map ($e \text{\AA}^{-3}$)	0.32
Error in an observation of unit weight	3.564
Secondary-extinction coefficient	$4.9267(1) \times 10^{-8}$ (Zachariassen, 1963)
Sources of atomic scattering factors	International Tables for X-ray Crystallography (1974)
Computer used	DEC MicroVAX II
Programs used	VAXSDP, version 3.0 (1986) (Frenz, 1980)

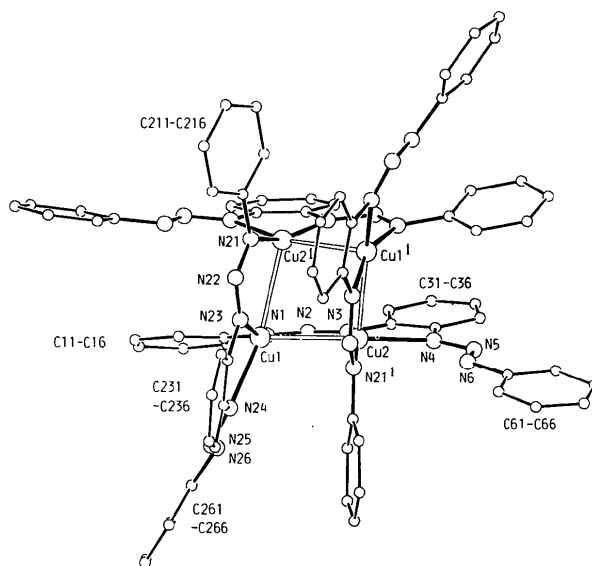


Fig. 1. Plot of the molecule showing the numbering scheme.

Table 2. Positional parameters, equivalent isotropic thermal parameters $U_{eq}(\text{\AA}^2)$ for the Cu and N atoms and isotropic thermal parameters U_{iso} for the C atoms

	x	y	z	U_{eq}/U_{iso}
Cu1	0.4896 (1)	0.1859 (1)	0.3639 (1)	0.0743 (8)
Cu2	0.4195 (1)	0.2230 (1)	0.2408 (1)	0.0774 (8)
N1	0.4840 (3)	0.2851 (3)	0.3923 (4)	0.067 (4)
N2	0.4577 (3)	0.3347 (4)	0.3508 (4)	0.066 (5)
N3	0.4309 (3)	0.3157 (4)	0.2868 (4)	0.063 (4)
N4	0.3629 (4)	0.2861 (4)	0.1608 (4)	0.084 (5)
N5	0.3254 (4)	0.2694 (4)	0.1051 (5)	0.088 (6)
N6	0.3186 (4)	0.2005 (4)	0.0977 (5)	0.089 (5)
N21	0.6067 (3)	0.1249 (4)	0.2648 (4)	0.069 (5)
N22	0.5757 (3)	0.0750 (4)	0.3015 (4)	0.065 (4)
N23	0.5257 (3)	0.0943 (4)	0.3402 (4)	0.067 (5)
N24	0.4325 (3)	0.1185 (4)	0.4451 (4)	0.079 (5)
N25	0.4027 (4)	0.1255 (4)	0.5078 (5)	0.088 (6)
N26	0.3944 (4)	0.1922 (4)	0.5285 (5)	0.098 (6)
C11	0.5098 (4)	0.3092 (4)	0.4644 (5)	0.073 (6)
C12	0.4982 (6)	0.3772 (5)	0.4923 (6)	0.098 (6)
C13	0.5231 (6)	0.3956 (6)	0.5656 (7)	0.118 (7)
C14	0.5576 (6)	0.3484 (6)	0.6078 (7)	0.124 (7)
C15	0.5692 (5)	0.2827 (6)	0.5812 (7)	0.109 (7)
C16	0.5438 (5)	0.2623 (5)	0.5087 (6)	0.086 (6)
C31	0.4082 (4)	0.3738 (4)	0.2409 (5)	0.070 (5)
C32	0.3740 (4)	0.3579 (5)	0.1754 (5)	0.077 (6)
C33	0.3519 (5)	0.4121 (6)	0.1248 (6)	0.103 (6)
C34	0.3681 (5)	0.4800 (6)	0.1457 (7)	0.111 (7)
C35	0.4015 (5)	0.4957 (6)	0.2115 (6)	0.098 (6)
C36	0.4223 (4)	0.4435 (4)	0.2597 (6)	0.079 (5)
C61	0.2766 (4)	0.1720 (5)	0.0421 (6)	0.082 (5)
C62	0.2468 (6)	0.2122 (6)	−0.0142 (6)	0.106 (6)
C63	0.2043 (6)	0.1801 (6)	−0.0676 (7)	0.122 (7)
C64	0.1931 (6)	0.1115 (6)	−0.0634 (7)	0.115 (6)
C65	0.2240 (6)	0.0708 (6)	−0.0081 (7)	0.124 (7)
C66	0.2658 (5)	0.1027 (6)	0.0438 (6)	0.100 (6)
C211	0.6637 (4)	0.1007 (5)	0.2255 (5)	0.072 (5)
C212	0.6901 (5)	0.0360 (5)	0.2401 (6)	0.098 (6)
C213	0.7476 (6)	0.0158 (6)	0.2025 (7)	0.119 (7)
C214	0.7772 (5)	0.0599 (6)	0.1514 (6)	0.106 (6)
C215	0.7522 (6)	0.1224 (6)	0.1350 (7)	0.115 (7)
C216	0.6938 (5)	0.1439 (6)	0.1745 (6)	0.096 (6)
C231	0.4915 (4)	0.0354 (4)	0.3706 (5)	0.071 (5)
C232	0.4417 (4)	0.0487 (5)	0.4240 (5)	0.075 (5)
C233	0.4029 (5)	−0.0054 (6)	0.4527 (6)	0.090 (6)
C234	0.4156 (5)	−0.0731 (6)	0.4290 (6)	0.104 (6)
C235	0.4658 (5)	−0.0870 (5)	0.3775 (6)	0.096 (6)
C236	0.5042 (5)	−0.0337 (5)	0.3475 (5)	0.085 (6)
C261	0.3692 (5)	0.2057 (5)	0.6038 (6)	0.092 (6)
C262	0.3425 (6)	0.1538 (6)	0.6513 (7)	0.119 (7)
C263	0.3171 (6)	0.1701 (7)	0.7249 (8)	0.138 (7)
C264	0.3174 (6)	0.2372 (6)	0.7480 (8)	0.134 (7)
C265	0.3399 (6)	0.2893 (7)	0.7017 (8)	0.137 (8)
C266	0.3665 (6)	0.2730 (6)	0.6301 (7)	0.122 (7)

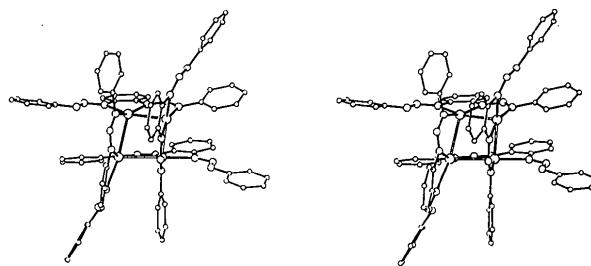


Fig. 2. Stereoscopic view of the title complex.

Discussion. Each ligand is N1,N3-bridging with one of its N₃ units also chelating with the *ortho* N atoms (N3, N4). The N₃ units bridge the Cu atoms alternately above and below the resulting distorted Cu₄ rhombus. Similar tetrameric units are reported from 1,3-dimethyltriazenocopper(I) (O'Connor, Janusonis &

Corey, 1968) and 1,3-bis(4-trifluoromethylphenyl)triazenocopper(I) (Hartmann & Strähle, 1988). In the title compound the dihedral angle between the planes (Cu1, Cu2, Cu2') and (Cu2, Cu2', Cu1') is 140.5 (1) $^\circ$. Compared with 152.2 $^\circ$ observed in 1,3-bis(4-trifluoromethylphenyl)triazenocopper(I), the larger deviation

Table 3. Selected distances (Å) and angles (°) with *e.s.d.*'s

Symmetry operator: (i) 1-x, y, 0.5-z.

Cu1...Cu2	2.650 (2)	Cu1...Cu1 ¹	3.927 (2)
Cu1...Cu2 ¹	2.684 (2)	Cu2...Cu2 ¹	3.324 (2)
Cu1-N1	1.950 (6)	Cu2-N21 ¹	1.942 (7)
Cu1-N23	1.934 (7)	Cu2-N3	1.945 (7)
Cu1-N24	2.225 (7)	Cu2-N4	2.161 (8)
N1-N2	1.298 (9)	N21-N22	1.304 (9)
N2-N3	1.280 (9)	N22-N23	1.276 (9)
N4-N5	1.27 (2)	N24-N25	1.24 (2)
N5-N6	1.32 (1)	N25-N26	1.33 (1)
Cu2-Cu1-Cu2 ¹	77.08 (5)	Cu1-Cu2-Cu1 ¹	94.82 (6)
Cu2-Cu1-N1	84.9 (2)	Cu1-Cu2-N21 ¹	86.2 (2)
Cu2-Cu1-N23	106.3 (2)	Cu1-Cu2-N3	81.5 (2)
Cu2-Cu1-N24	111.4 (2)	Cu1-Cu2-N4	160.9 (2)
Cu2-Cu1-N1	87.4 (2)	Cu1 ¹ -Cu2-N21 ¹	84.7 (2)
Cu2 ¹ -Cu1-N23	80.1 (2)	Cu1 ¹ -Cu2-N3	115.2 (2)
Cu2 ¹ -Cu1-N24	159.4 (2)	Cu1 ¹ -Cu2-N4	95.6 (2)
N1-Cu1-N23	160.8 (3)	N21 ¹ -Cu2-N3	157.3 (3)
N1-Cu1-N24	111.6 (3)	N21 ¹ -Cu2-N4	110.6 (3)
N23-Cu1-N24	79.4 (3)	N3-Cu2-N4	79.6 (3)
Cu1-N1-N2	126.1 (5)	Cu2-N21-N22	125.9 (5)
Cu1-N1-C11	120.5 (5)	Cu2-N21-C211	120.9 (5)
N2-N1-C11	113.5 (6)	N22-N21-C211	113.3 (7)
N1-N2-N3	116.4 (7)	N21-N22-N23	115.7 (7)
Cu2-N3-N2	131.0 (5)	Cu1-N23-N22	132.6 (5)
Cu2-N3-C31	115.8 (5)	Cu1-N23-C231	116.2 (5)
N2-N3-C31	113.2 (7)	N22-N23-C231	111.2 (7)
Cu2-N4-N5	131.8 (6)	Cu1-N24-N25	137.6 (6)
Cu2-N4-C32	109.7 (6)	Cu1-N24-C232	108.4 (5)
N5-N4-C32	118.4 (8)	N25-N24-C232	113.2 (7)
N4-N5-N6	112.5 (8)	N24-N25-N26	113.4 (8)
N5-N6-C61	121.08 (8)	N25-N26-C261	117.6 (8)

from planarity might be caused by the bridging and chelating ligand C₆H₅NNNC₆H₄NNN(H)C₆H₅. Obviously the influence of the ligands leads to angles Cu...Cu...Cu of 77.08 (5) and 94.82 (6)°, whereas in the above mentioned compound the Cu₄ unit is almost rectangular with angles of 88.30 (1)°. Of note are the

Cu...Cu distances with values of 2.650 (2) and 2.684 (2) Å, which may be interpreted as weak *d*¹⁰-*d*¹⁰ interactions (Mehrotra & Hoffmann, 1978). The distances Cu-N(azenediyl) range from 1.934 (7) to 1.950 (6) Å, Cu-N(azanyl) from 2.161 (8) to 2.225 (7) Å. In [(F₃CC₆H₄NNNC₆H₄CF₃)Cu]₄ the *N*-Lewis base causes shorter Cu-N distances [1.882 (7) Å] and the Cu...Cu distances decrease to 2.579 (1) Å.

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References

- FRENZ, B. A. (1978). *The Enraf-Nonius CAD-4 SDP - A Real-Time System for Concurrent X-ray Data Collection and Crystal Structure Determination*. In *Computing in Crystallography*, edited by H. SCHENK, R. OLTJOF-HAZEKAMP, H. VAN KONINGSVELD & G. C. BASSI, pp. 64-71.
- HARTMANN, E. & STRÄHLE, J. (1988). *Z. Naturforsch.* In the press.
- International Tables for X-ray Crystallography* (1974). Vol. IV, Table 2.2A. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- MEHROTRA, P. K. & HOFFMANN, R. (1978). *Inorg. Chem.* **17**, 2187-2190.
- MOORE, D. S. & ROBINSON, S. D. (1986). *Adv. Inorg. Chem. Radiochem.* **30**, 1-68.
- O'CONNOR, J. E., JANUSONIS, G. A. & COREY, E. R. (1968). *J. Chem. Soc. Chem. Commun.* pp. 445-446.
- WALKER, N. & STUART, D. (1983). *Acta Cryst.* **A39**, 158-166.
- ZACHARIASEN, W. H. (1963). *Acta Cryst.* **16**, 1139-1144.

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The Structure of Selenium(IV) Oxo Pinacolate (4,4,5,5-Tetramethyl-1,3,2λ⁴-dioxaselenole 2-Oxide)

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Abstract. C₆H₁₂O₃Se, *M*_r = 211.1, monoclinic, *P*2₁/*n*, *a* = 6.7584 (5), *b* = 12.1616 (7), *c* = 10.6218 (11) Å, β = 98.290 (8)°, *U* = 863.9 Å³, *Z* = 4, *D*_x = 1.623, *D*_m(floatation) = 1.62 Mg m⁻³, *F*(000) = 424, λ(Cu Kα) = 1.5418 Å, μ = 5.636 mm⁻¹, *T* = 295 (2) K, *R* = 0.048 for 1621 observed reflections. The Se atom is in a tetrahedral environment surrounded by three O atoms and a stereochemically active lone pair. The Se atom

further interacts with an O atom from each of two adjacent molecules [Se...O 2.885 (3) and 2.932 (3) Å] to give a linear polymer which lies along the *a* axis.

Introduction. As part of our investigation of the stereochemistry and reactivity of hypervalent Group 16 complexes, we have synthesized the title compound (I), (C₆H₁₂O₂)Se(O), in order to assess its Lewis base properties. In principle, (C₆H₁₂O₂)Se(O) could act as a Lewis base by coordination from either the O or the Se atoms. We have subsequently determined the crystal

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