Table 1. *Fractional atomic coordinates* ($\times 10^5$ *for* Cu *and* $\times 10^4$ *for others) and equivalent isotropic temperature factors* ($\times 10^4$), *with their e.s.d.'s in parentheses*

$$U_{\text{eq}} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Cu	11686 (4)	28136 (3)	30514 (2)	282 (2)
N(1)	955 (3)	3009 (3)	1288 (2)	299 (7)
C(1)	2018 (4)	4081 (3)	908 (3)	389 (10)
C(2)	1676 (5)	4165 (4)	−335 (3)	477 (12)
C(3)	230 (5)	3123 (4)	−1201 (3)	473 (12)
C(4)	−857 (4)	2015 (3)	−819 (3)	391 (10)
C(5)	−450 (3)	1988 (3)	433 (2)	298 (8)
C(6)	−1485 (3)	824 (3)	954 (2)	298 (9)
C(7)	−2916 (4)	−378 (3)	233 (3)	378 (9)
C(8)	−3794 (4)	−1370 (4)	822 (3)	452 (10)
C(9)	−3201 (5)	−1183 (4)	2092 (3)	461 (11)
C(10)	−1748 (4)	31 (3)	2769 (3)	367 (9)
N(2)	−907 (3)	1021 (3)	2205 (2)	301 (7)
O(*W*1)	904 (3)	2672 (3)	4722 (2)	383 (7)
O(*W*2)	3311 (3)	1398 (2)	3028 (2)	417 (8)
O(*W*3)	3161 (3)	4630 (3)	3804 (2)	447 (8)
N(3)	−2765 (3)	4341 (3)	2809 (2)	482 (9)
O(1)	−1068 (3)	4605 (3)	3066 (2)	479 (9)
O(2)	−3765 (4)	3189 (3)	2086 (3)	646 (10)
O(3)	−3477 (3)	5291 (3)	3301 (2)	472 (8)
N(4)	−2951 (3)	1825 (3)	5680 (2)	398 (8)
O(4)	−2576 (3)	2769 (2)	5039 (2)	478 (7)
O(5)*	−2070 (7)	841 (8)	5754 (6)	636 (23)
O(5′)*	−2814 (29)	584 (26)	5372 (20)	545 (76)
O(6)	−3934 (5)	1995 (4)	6350 (4)	1076 (18)

* Site occupancies 0·8 for O(5) and 0·2 for O(5′).

Table 2. *Selected bond lengths* (Å) *and angles* (°) *involving non-*H *atoms, with their e.s.d.'s in parentheses*

Cu—N(1)	1·998 (2)	Cu—N(2)	1·983 (3)
Cu—O(*W*1)	1·963 (2)	Cu—O(*W*2)	2·322 (2)
Cu—O(*W*3)	1·949 (2)	Cu—O(1)	2·642 (2)
N(1)—C(1)	1·347 (4)	N(3)—O(1)	1·233 (3)
N(1)—C(5)	1·342 (4)	N(3)—O(2)	1·224 (4)
C(6)—N(2)	1·346 (3)	N(3)—O(3)	1·258 (3)
C(10)—N(2)	1·345 (4)	N(4)—O(4)	1·256 (3)
N(4)—O(5)	1·255 (7)	N(4)—O(5′)	1·189 (24)
N(4)—O(6)	1·202 (5)	O(5)—O(5′)	0·606 (24)

N(1)—Cu—N(2)	81·6 (1)	N(1)—Cu—O(*W*1)	169·2 (1)
N(1)—Cu—O(*W*2)	92·9 (1)	N(1)—Cu—O(*W*3)	95·5 (1)
N(1)—Cu—O(1)	85·4 (1)	N(2)—Cu—O(*W*1)	93·4 (1)
N(2)—Cu—O(*W*2)	91·5 (1)	N(2)—Cu—O(*W*3)	176·7 (1)
N(2)—Cu—O(1)	92·4 (1)	Cu—N(2)—C(6)	114·9 (2)
O(*W*1)—Cu—O(*W*2)	96·8 (1)	Cu—N(2)—C(10)	125·9 (2)
O(*W*1)—Cu—O(*W*3)	89·2 (1)	O(*W*1)—Cu—O(1)	85·3 (1)
O(*W*2)—Cu—O(*W*3)	90·3 (1)	O(*W*2)—Cu—O(1)	175·5 (1)
O(*W*3)—Cu—O(1)	85·7 (1)	Cu—N(1)—C(1)	126·3 (2)
Cu—N(1)—C(5)	114·7 (2)	Cu—O(1)—N(3)	131·8 (2)
O(1)—N(3)—O(2)	121·8 (3)	O(4)—N(4)—O(6)	120·2 (3)
O(1)—N(3)—O(3)	118·7 (2)	O(4)—N(4)—O(5)	118·7 (4)
O(2)—N(3)—O(3)	119·5 (3)	O(5)—N(4)—O(6)	120·2 (4)

References

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Nakai, H., Ooi, S. & Kuroya, H. (1977). *Bull. Chem. Soc. Jpn*, **50**, 531–532.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

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2-(Chlorodimethylstannyl)-4-methoxy-2′,4′-dinitroazobenzene

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Abstract. $C_{15}H_{15}ClN_4O_5Sn$, $M_r = 485{\cdot}45$, monoclinic, $P2_1/c$, $a = 9{\cdot}349\ (2)$, $b = 15{\cdot}330\ (3)$, $c = 13{\cdot}562\ (3)$ Å, $\beta = 106{\cdot}14\ (2)°$, $V = 1867{\cdot}1\ (7)$ Å^3, $Z = 4$, $D_x = 1{\cdot}727$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1{\cdot}55$ mm^{-1}, $F(000) = 960$, $T = 291\ (1)$ K, final $R = 0{\cdot}031$ for 3136 unique observed [$F \geq 4{\cdot}0\sigma(F)$] diffractometer data. Besides the three C atoms [Sn—C(mean) 2·127 (4) Å] and the Cl atom [Sn—Cl 2·433 (1) Å] bound to Sn there is a short intramolecular Sn···N contact [2·674 (2) Å] and a distorted trigonal bipyramid is formed around Sn with Cl and N in the apical [Cl—Sn—N 161·76 (6)°] and the C atoms in the equatorial [C—Sn—C 118·3 (2), 114·4 (1), 120·8 (1)°] positions. The structure consists of discrete molecular units.

Experimental. The crystals have been obtained from acetone/pentane 1:1, m.p. 434 K. A crystal of size 0·44 × 0·42 × 0·16 mm was used. Its quality was checked with optical polarizing microscopy. The crystal was mounted on a glass fibre. Intensity data were collected with $\omega/2\theta$ scans, variable scan speed 1·5–10·2° min^{-1} in θ, scan width 1·2° + dispersion, on a Nicolet *R*3*m*/*V* diffractometer with graphite-monochromated Mo $K\alpha$ radiation. Lattice parameters determined from symmetry-constrained least-squares fit of 36 reflections with $2\theta_{\max} = 38{\cdot}2°$. ω scans of low-order reflections along the three crystal axes showed acceptable mosaicity. Six standard reflections (400, 040, 002, $\bar{4}00$, $0\bar{4}0$, $00\bar{2}$) recorded every 300 reflections, only random deviations

Table 1. *Atomic coordinates and equivalent isotropic displacement parameters* ($Å^2 \times 10^4$)

$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j$.

	x	y	z	U_{eq}
Sn(1)	0·12429 (2)	0·16417 (1)	0·39051 (1)	354
Cl(1)	−0·02262 (9)	0·04150 (6)	0·30335 (7)	549
N(1)	0·4522 (3)	0·2618 (2)	0·4206 (2)	377
N(2)	0·3380 (3)	0·2849 (2)	0·4467 (2)	369
N(3)	0·2361 (4)	0·3527 (2)	0·6096 (2)	465
N(4)	0·2338 (3)	0·6456 (2)	0·4718 (3)	523
O(1)	0·5200 (3)	−0·0840 (2)	0·3293 (2)	523
O(2)	0·3306 (3)	0·2991 (2)	0·6496 (2)	647
O(3)	0·1189 (3)	0·3630 (2)	0·6328 (2)	687
O(4)	0·1823 (4)	0·6701 (2)	0·5404 (3)	753
O(5)	0·2612 (4)	0·6933 (2)	0·4084 (2)	729
C(1)	0·4581 (3)	0·1724 (2)	0·3978 (2)	353
C(2)	0·3355 (3)	0·1159 (2)	0·3809 (2)	347
C(3)	0·3535 (3)	0·0306 (2)	0·3575 (2)	364
C(4)	0·4925 (3)	0·0004 (2)	0·3520 (3)	389
C(5)	0·6131 (3)	0·0567 (3)	0·3678 (3)	469
C(6)	0·5954 (4)	0·1417 (2)	0·3899 (3)	447
C(7)	0·3265 (3)	0·3762 (2)	0·4570 (2)	360
C(8)	0·2634 (3)	0·4100 (2)	0·5315 (2)	366
C(9)	0·2317 (3)	0·4969 (2)	0·5370 (2)	415
C(10)	0·2676 (3)	0·5521 (2)	0·4670 (2)	420
C(11)	0·3296 (4)	0·5223 (2)	0·3925 (3)	466
C(12)	0·3573 (3)	0·4346 (2)	0·3873 (2)	421
C(13)	0·0177 (4)	0·2692 (3)	0·2965 (3)	529
C(14)	0·0862 (5)	0·1485 (3)	0·5359 (3)	572
C(15)	0·4001 (5)	−0·1460 (2)	0·3132 (4)	583

Table 2. *Bond distances* (Å), *bond angles* (°), *torsion angles* (°), *least-squares planes and dihedral angles* (°)

Sn(1)—Cl(1)	2·433 (1)	O(1)—C(4)	1·371 (4)
Sn(1)—C(2)	2·147 (3)	O(1)—C(15)	1·439 (5)
Sn(1)—C(13)	2·122 (4)	C(1)—C(2)	1·404 (4)
Sn(1)—C(14)	2·112 (5)	C(1)—C(6)	1·399 (5)
Sn(1)—N(2)	2·674 (2)	C(2)—C(3)	1·366 (5)
N(1)—N(2)	1·265 (5)	C(3)—C(4)	1·401 (4)
N(1)—C(1)	1·410 (4)	C(4)—C(5)	1·388 (5)
N(2)—C(7)	1·414 (4)	C(5)—C(6)	1·357 (5)
N(3)—O(2)	1·218 (4)	C(7)—C(8)	1·402 (5)
N(3)—O(3)	1·231 (5)	C(7)—C(12)	1·389 (5)
N(3)—C(8)	1·453 (4)	C(8)—C(9)	1·371 (4)
N(4)—O(4)	1·220 (6)	C(9)—C(10)	1·381 (5)
N(4)—O(5)	1·209 (5)	C(10)—C(11)	1·374 (5)
N(4)—C(10)	1·474 (4)	C(11)—C(12)	1·375 (5)

C(13)—Sn(1)—C(14)	118·3 (2)	Sn(1)—C(2)—C(1)	120·0 (2)
C(2)—Sn(1)—C(14)	114·4 (1)	C(1)—C(2)—C(3)	118·8 (3)
C(2)—Sn(1)—C(13)	120·8 (1)	Sn(1)—C(2)—C(3)	121·3 (2)
Cl(1)—Sn(1)—C(14)	98·4 (1)	C(2)—C(3)—C(4)	120·3 (3)
Cl(1)—Sn(1)—C(13)	100·8 (1)	O(1)—C(4)—C(3)	123·9 (3)
Cl(1)—Sn(1)—C(2)	96·2 (1)	C(3)—C(4)—C(5)	120·8 (3)
N(2)—Sn(1)—C(2)	68·2 (1)	O(1)—C(4)—C(5)	115·3 (3)
N(2)—Sn(1)—C(13)	80·5 (1)	C(4)—C(5)—C(6)	119·1 (3)
N(2)—Sn(1)—C(14)	96·8 (1)	C(1)—C(6)—C(5)	120·7 (3)
N(2)—Sn(1)—Cl(1)	161·76 (6)	N(2)—C(7)—C(12)	122·3 (3)
N(2)—N(1)—C(1)	114·9 (2)	N(2)—C(7)—C(8)	119·7 (3)
N(1)—N(2)—C(7)	113·4 (3)	C(8)—C(7)—C(12)	117·5 (3)
O(3)—N(3)—C(8)	117·2 (3)	N(3)—C(8)—C(7)	119·8 (3)
O(2)—N(3)—C(8)	118·7 (3)	C(7)—C(8)—C(9)	122·5 (3)
O(2)—N(3)—O(3)	124·1 (3)	N(3)—C(8)—C(9)	117·6 (3)
O(5)—N(4)—C(10)	117·8 (3)	C(8)—C(9)—C(10)	117·5 (3)
O(4)—N(4)—C(10)	117·9 (3)	N(4)—C(10)—C(9)	118·0 (3)
O(4)—N(4)—O(5)	124·3 (3)	C(9)—C(10)—C(11)	122·2 (3)
C(4)—O(1)—C(15)	118·1 (3)	N(4)—C(10)—C(11)	119·7 (3)
N(1)—C(1)—C(6)	115·9 (3)	C(10)—C(11)—C(12)	119·1 (3)
N(1)—C(1)—C(2)	123·8 (3)	C(7)—C(12)—C(11)	121·1 (3)
C(2)—C(1)—C(6)	120·3 (3)		

N(2)—N(1)—C(1)—C(2)	14·6 (4)	O(5)—N(4)—C(10)—C(11)	0·6 (5)
C(1)—N(1)—N(2)—C(7)	−172·8 (3)	C(15)—O(1)—C(4)—C(5)	179·3 (3)
N(1)—N(2)—C(7)—C(12)	42·3 (4)	N(1)—C(1)—C(2)—Sn(1)	−0·6 (4)
O(3)—N(3)—C(8)—C(9)	43·5 (4)	N(2)—C(7)—C(8)—N(3)	11·6 (4)

No.	Plane through atoms	Equation of the plane	χ^2
1	C(1),C(2),C(3) C(4),C(5),C(6)	$0·015x + 0·24y - 0·9745z = -4·412$ Å	21·0
2	C(7),C(8),C(9) C(7),C(8),C(9),C(10), C(11),C(12)	$-0·7723x - 0·139y - 0·620z = -5·516$ Å	27·8

Dihedral angle: 1,2 55·8 (1)°.

detected during 169·9 h of X-ray exposure; 9253 reflections with $2·0 \le 2\theta \le 50·0°$ (12 626 accessible), $-12 \le h \le 12$, $-19 \le k \le 0$, $-17 \le l \le 17$ plus $-12 \le h \le -3$, $-19 \le k \le 0$, $-17 \le l \le 17$. Data corrected for Lorentz–polarization and for absorption effects, the latter *via* ψ-scan data $0 \le \psi \le 360°$ of nine reflections with $80 \le \chi \le 90°$, the max./min. transmission factors were 1·00/0·76, and averaged ($R_{\rm int} = 0·036$) to 3287 unique reflections, 3136 of which had $F \ge 4·0\sigma(F)$. Six reflections omitted because of suspected extinction. Systematic absences ($h0l$) $l = 2n + 1$, ($0k0$) $k = 2n + 1$ conform to space group $P2_1/c$. Structure solved *via* Patterson function and $\Delta\rho$ maps. It was refined (on F) using full-matrix least squares with anisotropic displacement parameters for all non-H atoms and a fixed isotropic one ($U = 0·08$ Å^2) for the H atoms, which were placed in geometrically calculated positions (C—H 0·96 Å). 236 parameters refined. Weights $w = 1·0/[\sigma^2(F) + (0·0005F^2)]$ led to a featureless analysis of variance in terms of $\sin\theta$ and F_o. Refinement converged to $R = 0·031$, $wR = 0·035$, $S = 1·81$ $(\Delta/\sigma)_{\max} = 0·025$ (no extinction correction). Correctness of the space-group choice was checked by using *MISSYM* (Le Page, 1987). The largest peaks in final $\Delta\rho$ map were $\pm 0·7$ (4) e Å^{-3} (near Sn). Atomic scattering factors for neutral atoms and real and imaginary dispersion terms were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Programs used: *PARST* (Nardelli, 1983), *SHELX76* (Sheldrick, 1976), *SHELXTL-Plus* (Sheldrick, 1987), *PCK83* (Williams, 1984), *PLATON* (Spek, 1982), *MISSYM* (Le Page, 1987). The molecule and the numbering scheme are shown in Fig. 1 and a stereoscopic view of the unit-cell content in Fig. 2. Positional parameters and the equivalent values of the anisotropic displacement parameters for the non-H atoms are given in Table 1.* Bond lengths, bond angles, torsion angles, least-squares planes and dihedral angles are given in Table 2.

Related literature. The title compound has been obtained from 3-trimethylstannyl anisole and 2,4-

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54186 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

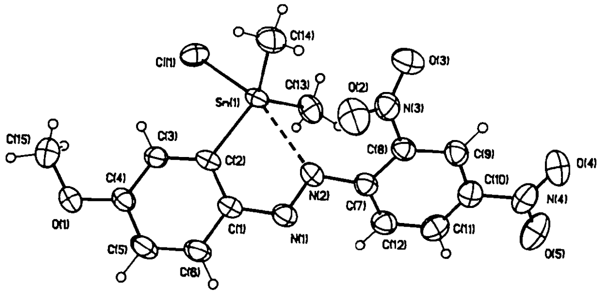

Fig. 1. General view (*SHELXTL-Plus*; Sheldrick, 1987) of the molecule, showing the atom-numbering scheme. Anisotropic ellipsoids represent 50% probability boundaries.

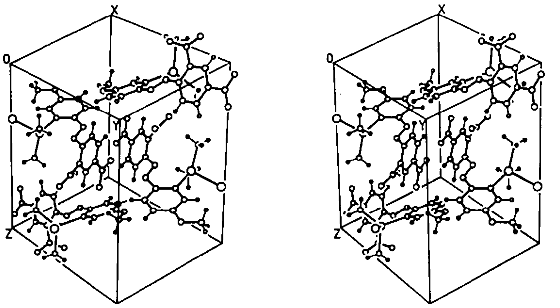

Fig. 2. Stereoscopic view (*SHELXTL-Plus*; Sheldrick, 1987) of part of the crystal structure with the boundaries of the unit cell.

dinitrobenzene tetrafluoroborate in acetonitrile at 296 K, and subsequent workup with column chromatography. Surprisingly, a tin methyl bond has been split instead of the tin phenyl bond, whereas the reactivity is normally the inverse (Al-Allaf, Kobs & Neumann, 1989). This is one of the first cases of complexation of a triorganotin halide by an azo compound forming intramolecular pentacoordination at the Sn atom (Brianso, Solans & Vicente, 1983; Vicente, Chicote, Carreno & del Carmen Ramirez-de-Arellano, 1989; Vicente, Chicote, del Carmen Ramirez-de-Arellano, Pelizzi & Vitali, 1990; Vicente, Chicote, del Carmen Ramirez-de-Arellano & Jones, 1990).

References

Al-Allaf, A. K., Kobs, U. & Neumann, W. P. (1989). *J. Organomet. Chem.* **373**, 29–35.

Brianso, J. L., Solans, X. & Vicente, J. (1983). *J. Chem. Soc. Dalton Trans.* pp. 168–171.

Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet *R3m/V* crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

Spek, A. L. (1982). *The EUCLID Package*. In *Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

Vicente, J., Chicote, M. T., del Carmen Ramirez-de-Arellano, M. & Jones, P. G. (1990). *J. Organomet. Chem.* **394**, 77–85.

Vicente, J., Chicote, M. T., del Carmen Ramirez-de-Arellano, M., Pelizzi, G. & Vitali, F. (1990). *J. Chem. Soc. Dalton Trans.* pp. 279–282.

Vicente, J., Chicote, M. T., Carreno, R. & del Carmen Ramirez-de-Arellano, M. (1989). *J. Organomet. Chem.* **368**, 263–268.

Williams, D. E. (1984). *PCK83. A Crystal and Molecular Packing Analysis Program*, Univ. of Louisville, QCPE Program No. 481. Department of Chemistry, Indiana Univ., Indiana, USA.

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Structure of Trichloro(η^5-methylcyclopentadienyl)titanium(IV)

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Abstract. [$TiCl_3(CH_3C_5H_4)$], $M_r = 233{\cdot}38$, triclinic, $P\bar{1}$, $a = 6{\cdot}862\,(1)$, $b = 6{\cdot}923\,(2)$, $c = 11{\cdot}215\,(2)$ Å, $\alpha = 82{\cdot}65\,(1)$, $\beta = 83{\cdot}27\,(1)$, $\gamma = 61{\cdot}30\,(1)$°, $V = 462\,(3)$ Å^3, $Z = 2$, $D_x = 1{\cdot}68$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 17{\cdot}2$ cm^{-1}, $F(000) = 232$, $T = 294\,(1)$ K, $R = 0{\cdot}036$ for 1568 unique reflections with $F_o^2 > 3\sigma(F_o^2)$. The five ring C atoms of the methylcyclopentadienyl ligand and the three Cl atoms form a piano-stool coordination about the Ti atom with a dihedral angle between the methylcyclopentadienyl plane and the Cl_3 plane of 2·7°. The Ti—Cl distances range from 2·2229 (9) to 2·228 (1) Å, the Ti—C* distance is 2·004 Å (C* = centroid of the cyclopentadienyl ring) and the Ti—C distances range from 2·301 (3) to 2·379 (3) Å. The torsion angle Cl(2)—Ti—C(5)—C(6), with a value of −0·9 (3)°, shows the

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