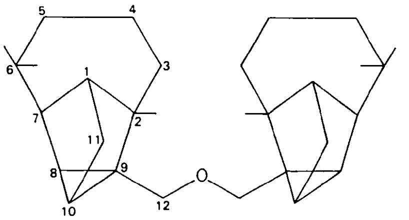

Fig. 1. Chemical formula of the title molecule, with atom numbering.

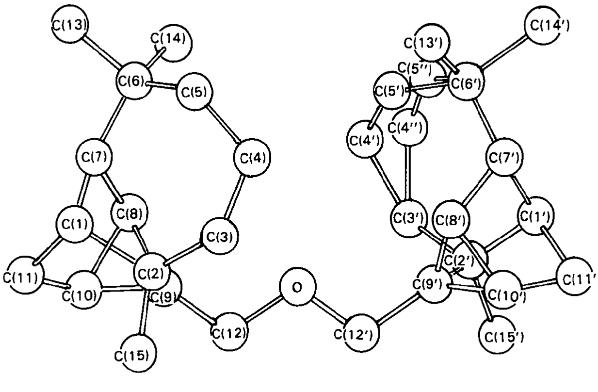

Fig. 2. Perspective view of the molecule.

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Structure of Dichloro(*p*-methoxyphenyl)(8-oxo-7-oxabicyclo[4.3.0]non-5-yl)-tellurium(IV)–Chloroform (1/0·27)

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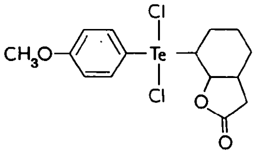
Abstract. $[TeCl_2(CH_3OC_6H_4)(C_8H_{11}O_2)].0{\cdot}27CHCl_3$, $M_r = 447{\cdot}0$, hexagonal, $P6_5$, $a = 15{\cdot}061\,(3)$, $c = 14{\cdot}409\,(3)$ Å, $Z = 6$, $V = 2831\,(1)$ Å^3, $D_x = 1{\cdot}68$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 1{\cdot}99$ mm^{-1}, $F(000) = 1402$, $T = 296\,(1)$ K, final $R = 0{\cdot}0471$ for 2493 observed reflections. The central Te atom is bonded to the *p*-methoxyphenyl group, to the cyclohexane moiety of the *cis*-fused γ-butyrolactone and to the two Cl atoms in a pseudo-trigonal-bipyramidal arrangement with axial halogens and one equatorial position vacant. Bond lengths are Te—Cl(1) = 2·525 (4), Te—Cl(2) = 2·499 (3), Te—C(ph) = 2·111 (8) and Te—C(lactone) = 2·184 (9) Å. If an intermolecular, secondary Te···O bond of 3·29 Å, *trans* to Te—C(lactone), is taken into consideration, the structure may be regarded as pseudo-octahedral. This weak bond ties the molecules together in spirals around the sixfold screw axes. Inside the resulting tunnels, disordered $CHCl_3$ molecules are trapped.

Introduction. The tellurium compound (I) is one of several aryltellurium dichloride lactones made by the reaction between γ–δ unsaturated carboxylic acids and $ArTeCl_3$ (Comasseto & Petragnani, 1983). Since the mechanism of the reaction is assumed to be the same as that found for halo- and selenolactonization, a *cis*-fused lactone should be the result. This is supported by ^1H NMR data (Comasseto & Petragnani, 1983). The purpose of this work is to find corroborative evidence for this.



(I)

Experimental. The preparation of the title compound was reported by Comasseto & Petragnani (1983). The compound was recrystallized from a mixture of $CHCl_3$ and petroleum ether (30–60) and yielded very small, colorless crystals. A subsequent recrystallization from pure $CHCl_3$ gave large needle-like prisms suitable for X-ray analysis.

The X-ray data were collected with an Enraf–Nonius CAD-4 diffractometer by the Molecular Structure Corp., College Station, Texas. A crystal with dimensions 0·40 × 0·25 × 0·25 mm was used with graphite-monochromatized Mo $K\alpha$ radiation. 25 reflections ($4 < \theta < 12°$) were used to determine cell parameters. Systematic absences: $00l$, all reflections absent except for $l = 6n$: space group $P6_1$ or $P6_5$. Intensities of 3133 unique reflections with $2\theta < 60°$ ($0 \leq h \leq 17$, $0 \leq k \leq 18$, $0 \leq l \leq 20$) were measured using the ω–2θ scan technique. 481 reflections had $I < 0{\cdot}5\sigma(I)$, an additional 157 had $F < \sigma(F)$, and these were omitted from calculations. In addition (010) and (100) were excluded since $|F_o| \ll |F_c|$, and, with their low 2θ values, they may have only partially hit the counter opening. No significant decline was found in the intensities of three standard reflections. Corrections were applied for Lorentz and polarization effects and for absorption (ψ scan, transmission coefficient, min. 0·954, max. 0·991). Programs used came from *SHELX*76 (Sheldrick, 1976) modified for a PDP 1144 computer. The structure was solved by Patterson and Fourier methods in space group $P6_1$, and refined by successive least-squares iterations. Positions of H atoms were calculated for C—H = 1·08 Å, and they were assigned the same isotropic temperature factors as the C atoms to which they belong. After introducing anisotropic temperature factors for the non-H atoms, refinement of them was carried out in both possible space groups: $R = 0{\cdot}066$ for $P6_5$, slightly better than for $P6_1$. Some bond lengths and angles were unreasonable in both models, and difference maps revealed several maxima in hexagonal channels along the sixfold screw axes. It had been assumed that the material investigated was the pure tellurium compound, but because of the unusual appearance of the difference maps, chemical analysis of the X-ray sample that had been recrystallized from $CHCl_3$ was performed by Galbraith Laboratories (Knoxville, Tennessee). The results were: C, 38·03; H, 3·71; Cl, 20·96%, compared with C, 40·51; H, 4·08; Cl, 15·94% expected for the original compound. It was then assumed that $CHCl_3$ molecules had been trapped in the crystal lattice, and from the analysis that there was 0·27 mol $CHCl_3$ per mol of tellurium.

When partial Cl and C atoms were put in positions corresponding to the four highest peaks in the difference map, the structure of the complex molecule improved significantly with subsequent refinement in both space groups. However, the $CHCl_3$ molecules were obviously disordered. In refinement, they stayed close to the sixfold screw axis. Since the translation along this axis ($\frac{1}{6}c$) is only 2·4 Å, the calculated separation between Cl atoms in adjacent equivalent positions is less than the intramolecular Cl···Cl separation, 2·8 Å. Because only 27% of the equivalent sites need be occupied, this short contact can be rationalized if $CHCl_3$ molecules do not occupy adjacent sites. After much trial and error, the $CHCl_3$ occupancies were fixed at 0·41, 0·25 and 0·16 for Cl(3), Cl(4), and Cl(5), respectively (ave. 0·27), and 0·27 for C(16). The main factors used in selecting the occupancies were: (1) how many Cl···Cl contacts near 2·8 Å were made by each Cl, (2) the results of refinements where the occupancies were systematically varied from cycle to cycle while their values were required to average 0·27 and remain constant during refinements, and (3) peak sizes in difference Fourier maps. The atoms were not required to belong to a single molecule.

Refinements were terminated when all parameter shifts were less than $0{\cdot}04\sigma$ in both space groups. In the final refinements, the H-atom parameters were kept fixed. The quantity minimized was $\sum w(|F_o| - |F_c|)^2$; $w = 1/\sigma^2(F)$, $\sigma(I) = [\sigma^2 + (0{\cdot}05I)^2]^{1/2}$, with σ calculated from counting statistics. $R = 0{\cdot}047$, $wR = 0{\cdot}050$, $S = 1{\cdot}34$ for $P6_5$; $R = 0{\cdot}050$ for $P6_1$. The difference between refinements in $P6_5$ and $P6_1$ is not significant at the 5% level (Hamilton, 1965), but since this difference persisted in all our models, including those without any $CHCl_3$, space group $P6_5$ was chosen to be the most likely one. (The best R value, 0·045, was obtained by refining both occupancies and temperature factors for $CHCl_3$, although not simultaneously. The extreme values for both types of parameters after very slow convergence led us to discard this solution, and the disorder of the $CHCl_3$ molecules was not resolved.) A final difference map ranged from +0·6 to −0·6 e Å^{-3}. The highest peaks were found near $CHCl_3$.

Scattering factors used were from Cromer & Liberman (1970), Cromer & Mann (1968) and Stewart, Davidson & Simpson (1965).

Discussion. Fig. 1 shows the structure of (I). Final parameters are given in Table 1, and interatomic distances and angles are listed in Table 2.*

The lactone ligand. For the bicyclic lactone, the expected *cis* ring fusion is found [interplanar angle C(9), C(10), C(12), C(13)/C(12), C(13), O(2), C(15) = 62·3°]. The γ-butyrolactone ring has the envelope conformation (Bryan & Smith, 1975; Danishefsky, Hirama, Fritch & Clardy, 1979). The flap atom, C(12), is *ca* 0·5 Å above the plane of the four other ring atoms. Bond lengths involving oxygen atoms in the lactone are C(13)–O(2) = 1·477 (9), O(2)–C(14) = 1·386 (6), C(14)–O(3) = 1·181 (12) Å. These values agree well with those found in other cyclic lactones and acid anhydrides (Bryan & Smith, 1975; Aravamudhan, Haeberlein, Irngartinger & Krieger, 1979; Brisse & Sygusch, 1975; Murray-Rust, Murray-Rust & Newton, 1979). The ring angles are normal and a plane through all ring atoms is within 0·22 Å of these atoms plus O(3).

The cyclohexane ring is fused to the lactone ring at C(12) and C(13). It has a chair configuration with C(9) 0·70 Å above and C(12) 0·55 Å below the plane through C(8), C(10), C(11) and C(13). This plane forms an angle of 74·3° with that through the non-flap atoms of the lactone ring. There is some flattening of the cyclohexane ring as seen by comparison of the average C—C—C ring angle of 112·3° with the 111·3° found in cyclohexane itself (Davis & Hassel, 1963; Buys & Geise, 1970). However, substituents are found to have a flattening effect (Altona & Sundaralingam, 1970). The large Te atom is equatorial, as is expected for large substituents.

Tellurium coordination. The central Te atom is essentially 4-coordinate and bonded to two Cl atoms,

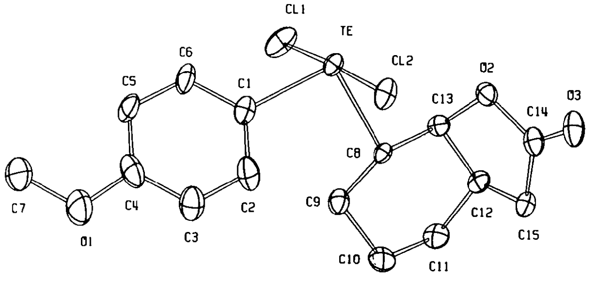


Fig. 1. *ORTEP* drawing of [$TeCl_2(CH_3OC_6H_4)(C_8H_{11}O_2)$] (Johnson, 1965).

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å²) *for non-hydrogen atoms with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}
Te compound				
Te	0·50375 (4)	0·44001 (4)	0·00000	0·0425 (2)
Cl(1)	0·5059 (2)	0·3143 (2)	0·1147 (2)	0·077 (2)
Cl(2)	0·4911 (2)	0·5607 (2)	–0·1109 (2)	0·063 (1)
O(1)	0·2476 (7)	0·0920 (8)	–0·2849 (8)	0·118 (6)
O(2)	0·4914 (4)	0·6081 (5)	0·1352 (4)	0·051 (3)
O(3)	0·4708 (7)	0·7416 (5)	0·1098 (6)	0·079 (5)
C(1)	0·4200 (8)	0·3188 (7)	–0·0940 (6)	0·055 (4)
C(2)	0·3339 (10)	0·3137 (7)	–0·1407 (8)	0·078 (6)
C(3)	0·2790 (11)	0·2358 (10)	–0·2025 (8)	0·085 (7)
C(4)	0·3103 (10)	0·1657 (8)	–0·2204 (8)	0·082 (6)
C(5)	0·3972 (8)	0·1741 (8)	–0·1800 (10)	0·073 (6)
C(6)	0·4503 (9)	0·2498 (8)	–0·1155 (8)	0·070 (5)
C(7)	0·2822 (12)	0·0264 (13)	–0·3158 (12)	0·131 (10)
C(8)	0·3789 (6)	0·4367 (6)	0·0799 (5)	0·036 (3)
C(9)	0·2897 (7)	0·3306 (7)	0·1018 (7)	0·054 (4)
C(10)	0·2103 (7)	0·3438 (8)	0·1568 (9)	0·069 (5)
C(11)	0·2515 (7)	0·4029 (8)	0·2438 (7)	0·058 (4)
C(12)	0·3472 (6)	0·5057 (7)	0·2294 (6)	0·045 (4)
C(13)	0·4262 (6)	0·5009 (6)	0·1660 (6)	0·044 (3)
C(14)	0·4359 (8)	0·6574 (7)	0·1388 (6)	0·053 (4)
C(15)	0·3344 (7)	0·5905 (6)	0·1831 (6)	0·052 (4)
Chloroform				
Cl(3)	0·1077 (12)	0·0259 (12)	0·1276 (11)	0·166 (5)
Cl(4)	–0·0388 (26)	–0·0066 (32)	–0·0925 (40)	0·251 (16)
Cl(5)	–0·0278 (19)	0·0738 (19)	0·1409 (17)	0·101 (7)
C(16)	0·0075 (35)	0·0072 (35)	–0·0283 (29)	0·074 (11)

Table 2. *Interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Te–Cl(1)	2·525 (4)	Te–Cl(2)	2·499 (3)
Te–C(1)	2·111 (9)	Te–C(8)	2·184 (9)
C(1)–C(2)	1·429 (19)	C(2)–C(3)	1·371 (15)
C(3)–C(4)	1·379 (25)	C(4)–C(5)	1·380 (20)
C(5)–C(6)	1·375 (16)	C(6)–C(1)	1·362 (20)
C(4)–O(1)	1·392 (14)	O(1)–C(7)	1·399 (26)
C(8)–C(9)	1·521 (10)	C(9)–C(10)	1·528 (17)
C(10)–C(11)	1·481 (15)	C(11)–C(12)	1·513 (11)
C(12)–C(13)	1·531 (14)	C(13)–C(8)	1·513 (11)
C(13)–O(2)	1·477 (9)	O(2)–C(14)	1·368 (16)
C(14)–C(15)	1·489 (12)	C(14)–O(3)	1·181 (12)
Te···O(2)	3·273 (7)	Te···O(3′)	3·292 (9)
Cl(1)–Te–Cl(2)	176·7 (1)	Cl(1)–Te–C(1)	89·8 (3)
Cl(1)–Te–C(8)	88·4 (2)	Cl(2)–Te–C(1)	89·4 (3)
Cl(2)–Te–C(8)	88·6 (2)	C(1)–Te–C(8)	100·6 (3)
Te–C(1)–C(2)	118·8 (8)	Te–C(1)–C(6)	121·6 (8)
C(2)–C(1)–C(6)	119·5 (9)	C(1)–C(2)–C(3)	119·7 (13)
C(2)–C(3)–C(4)	119·0 (14)	C(3)–C(4)–C(5)	121·5 (10)
C(4)–C(5)–C(6)	119·5 (13)	C(5)–C(6)–C(1)	120·6 (12)
C(3)–C(4)–O(1)	112·4 (13)	C(5)–C(4)–O(1)	126·0 (14)
C(4)–O(1)–C(7)	115·7 (12)	Te–C(8)–C(9)	115·5 (6)
Te–C(8)–C(13)	107·0 (5)	C(9)–C(8)–C(13)	112·8 (6)
C(8)–C(9)–C(10)	108·0 (8)	C(9)–C(10)–C(11)	113·4 (8)
C(10)–C(11)–C(12)	113·4 (7)	C(11)–C(12)–C(13)	113·5 (8)
C(12)–C(13)–C(8)	113·0 (6)	C(11)–C(12)–C(15)	117·3 (8)
C(13)–C(12)–C(15)	101·8 (6)	C(12)–C(13)–O(2)	103·6 (7)
C(8)–C(13)–O(2)	107·2 (6)	C(13)–O(2)–C(14)	109·2 (6)
O(2)–C(14)–C(15)	110·4 (8)	C(12)–C(15)–C(14)	103·6 (9)
O(2)–C(14)–O(3)	120·4 (9)	C(15)–C(14)–O(3)	129·3 (12)
O(3′)···Te–Cl(1)	82·5 (1)	O(3′)···Te–Cl(2)	100·8 (1)
O(3′)···Te–C(1)	95·1 (3)	O(3′)···Te–C(8)	161·8 (2)
O(2)···Te–C(1)	144·2 (3)		

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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, best planes and shortest intermolecular distances have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43726 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

the *p*-methoxyphenyl group and the cyclohexane part of the lactone. A lone pair of electrons on Te^{IV} possibly occupies the vacant equatorial position of a trigonal bipyramid in accordance with the VSEPR theory (Gillespie, 1970), while two C atoms are equatorial and two Cl atoms are axial. Within the coordination sphere, Te—Cl(1) = 2·525 (4), Te—Cl(2) = 2·499 (3), Te—C(1) = 2·111 (8) and Te—C(8) = 2·184 (9) Å. The Te—Cl bond lengths are close to those found in other $TeCl_2R_2$ compounds, 2·47–2·55 Å (Alcock & Harrison, 1982; Ziolo & Troup, 1983; Cameron, Amero & Cordes, 1980; Chadha & Drake, 1984; Jones & Hamor, 1984). Most Te^{IV}—C(aryl) bonds lie in the range 2·12 ±0·02 Å (Chadha & Miller, 1982) in good agreement with the value found for Te—C(1). Te^{IV}—C(alkyl) bonds have similar lengths, but are usually longer, in agreement with the 0·03 Å greater radius of $C(sp^3)$ compared with $C(sp^2)$ (Bastiansen & Trætteberg, 1962). Although 2·18 Å is in the upper range for such a bond, several Te—C(alkyl) bonds have recently been found in the range 2·19 ±0·05 Å (Cameron, Amero & Cordes, 1980; Cameron, Amero, Chan & Cordes, 1980; Dewan & Silver, 1977; Jones & Hamor, 1984; Blom, Haaland & Seip, 1983; Singh, McWhinnie, Hamor & Jones, 1984). *Trans* to the long Te—C(8) bond, an intermolecular Te···O(3′) contact distance of 3·292 (9) Å is present, just below an upper range of 3·3–3·4 Å found by Alcock, Harrison & Howes (1984) for weak bonds. These contacts seem to couple the complex molecules into helices around the sixfold screw axes, and indicate that the Te atom has some tendency toward distorted pseudo-octahedral coordination. This weak bond may also have a lengthening effect on the Te—C(8) bond *trans* to it. The intramolecular distance, Te—O(2), is 3·273 (9) Å, comparable to the weak secondary bond and roughly $\frac{1}{3}$ Å shorter than the sum of the van der Waals radii of Te and O (Pauling, 1960). It is 0·76 Å below the C(1)C(8)Te plane [O(3′) is 0·52 Å above], but the C(8)—Te—O(2) angle is only 47·3°.

Alternatively, a linear three-center four-electron bond description may be used to describe the geometry around Te (Foss, 1962, 1967; Mangion, Zingaro & Meyers, 1975; Husebye, 1983). This description accounts well for an average Te—Cl bond length of 2·512 Å, which is 0·15 Å longer than the sum of the respective covalent radii, 2·36 Å (Pauling, 1960).

The Cl(1)—Te—Cl(2) angle is 176·7 (1)° while C(1)—Te—C(8) is 100·6 (4)°, close to the values of 176·4 (2) and 101·0 (6)° found for the corresponding angles in $TeCl_2RR'$, where R is a substituted cyclohexyl group and R' is tolyl (Cameron, Amero & Cordes, 1980). The C—Te—C angle is significantly larger than that found in Me_2Te, which is 94 (2)° (Blom, Haaland & Seip, 1983). This effect may be due to the greater repulsion between the larger carbon-containing ligands in (I) relative to Me_2Te.

Average C—C bond lengths in the phenyl and cyclohexyl groups are 1·383 and 1·515 Å, respectively. The average C—C—C angle in the phenyl group is 120·0°.

Solvent molecules. Disordered guest molecules have been found in hexagonal channels in other compounds (Flippen, Karle & Karle, 1970; Hardy, McKendrick, MacNicol & Wilson, 1979).

The minimum distances between (I) and the $CHCl_3$ molecules are O(1)···Cl(5) = 3·174 and C(7)···Cl(5) = 3·401 Å. Corresponding van der Waals contacts are 3·20 and 3·80 Å (Pauling, 1960). Thus C(7)···Cl(5) seems very short, but Bondi's (1964) van der Waals radii correspond to a C···Cl contact of only 3·45 Å. These two short distances are possibly reflected in the small occupancy found for Cl(5). There are no other especially short contacts between (I) and $CHCl_3$.

Space groups $P6_1$ *and* $P6_5$. The structure of (I) refined in $P6_1$ differed from that in $P6_5$ mainly in the Te—Cl bond lengths and in the bond lengths of the *p*-methoxyphenyl group, although the average bond distances are nearly the same in both space groups. One bond length seems more reasonable in $P6_1$, 2·167 Å for Te—C(8), compared with 2·184 Å found in $P6_5$. In the *p*-methoxyphenyl group, however, distances compare much better with the conventional model in $P6_5$ than in $P6_1$. Since Te—C distances vary much more than aromatic C—C distances, the bond-length differences seem to favor $P6_5$, as do the R values quoted previously.

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Structure of 10-(2,6-Dichlorophenyl)-8-oxa-9-aza[5.3.3]propell-9-en-2-one* (DCPOP)

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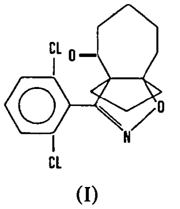
Abstract. $C_{17}H_{17}Cl_2NO_2$, $M_r = 338\cdot2$, monoclinic, $P2_1/c$, $a = 6\cdot8940\ (8)$, $b = 10\cdot941\ (1)$, $c = 20\cdot431\ (6)$ Å, $\beta = 95\cdot59\ (2)°$, $V = 1533\cdot73$ Å^3, $Z = 4$, $D_m = 1\cdot454$, $D_x = 1\cdot464$ Mg m^{-3}, m.p. = 408–410 K, $\lambda(\text{Mo}\ K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot4274$ mm^{-1}, $F(000) = 704$, $T = 298$ K, final $R = 0\cdot083$ for 1172 unique reflections. The mean plane of the cycloheptanone ring nearly bisects the angle formed at the common C—C bond, of length 1·55(1) Å, by the cyclopentane and dihydroisoxazole rings. The plane of the dichlorophenyl ring is almost perpendicular [84·8 (3)°] to the plane of the latter. The cycloheptanone ring shows a chair conformation.

Introduction. Propellanes are tricyclic compounds in which three rings are fused together at a common conjoining carbon–carbon bond. In these compounds it is of special importance to know the type of bonding between the two quarternary bridge-head C atoms and the resulting characteristics of the conjoining bond. In large-ring propellanes, as in our case, the hybridization of these C atoms may be sp^3 or sp^2 (Herr, 1977). The title compound was isolated during systematic preparative work on new heterocyclic propellanes using 1,3-dipolar cycloaddition of nitrile oxides to bicyclo[5.3.0]dec-1(7)-en-2-one (Malamidou-Xenikaki, Coutouli-Argyropoulou & Alexandrou, 1985). The spectroscopic and theoretical data favoured the structure (I). However, to determine the structure unambiguously an X-ray analysis was considered necessary.



(I)

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[*] 10-(2,6-Dichlorophenyl)-8-oxa-9-azatricyclo[5.3.3.$0^{1,7}$]tridec-9-en-2-one.

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