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## 1,1-Dichloro-3,3,5,5-tetraphenyl- $1\lambda^4,2,6,3\lambda^5,5\lambda^5$ -selenadiazadiphosphorin

MICHAEL BOLTE

*Institut für Organische Chemie der Universität  
Frankfurt, Marie-Curie-Strasse 11, 60439 Frankfurt/  
Main, Germany*

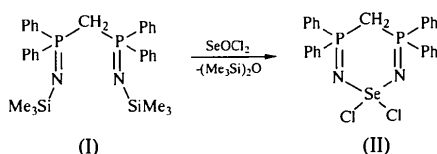
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### Abstract

The first X-ray structure of a six-membered ring containing the structural unit N—SeCl<sub>2</sub>—N is presented. The heterocycle in the title compound (as the trichloromethane solvate), C<sub>25</sub>H<sub>22</sub>Cl<sub>2</sub>N<sub>2</sub>P<sub>2</sub>Se·2CHCl<sub>3</sub>, adopts a boat conformation with the C and Se atoms deviating from the P<sub>2</sub>N<sub>2</sub> plane. The two Se—Cl bonds differ markedly. Disorder of one of the phenyl groups may be explained in terms of the crystal packing.

### Comment

The reaction leading to the title compound presents a new method for the synthesis of cyclic metallophosphazenes (Seseke, 1986). The reaction of (I) with WF<sub>6</sub> and WCl<sub>6</sub> yields analogous products which have been characterized by NMR spectroscopy. Compounds of type (I) with one, two and three methylene groups bridging the two P atoms have been investigated by Appel & Ruppert (1974), who showed the possibility of transforming (I) into cyclic products.



The title compound, (II), crystallizes with two chloroform molecules in the asymmetric unit. The heterocycle adopts a boat conformation with Se and C(1) deviating [by 0.32 (1) and 0.64 (1) Å, respectively] from the P<sub>2</sub>N<sub>2</sub> plane ( $\sigma = 0.023$  Å).

Taking into account the free electron pair, the Se coordination geometry can be described as trigonal bipyramidal with the lone pair and the two N atoms in the equatorial plane, while the Cl atoms occupy the axial positions. The Se—N bond lengths are shortened compared with an Se—N single bond. The two Se—Cl bonds differ by nearly 0.5 Å. Both P=N double bonds display typical values. The phenyl rings C11–C16 and C21–C26 are approximately parallel, the angle between them being 19.7 (6)°.

Inspection of the crystal packing reveals that the disordered phenyl ring is surrounded by other disordered phenyl rings and chloroform molecules, while the ring C11'–C16' lies in the vicinity of the nearly parallel aromatic rings which restrict its degrees of freedom. Therefore, it seems obvious that the crystal packing allows different orientations of one phenyl group while keeping the other in a more fixed position.

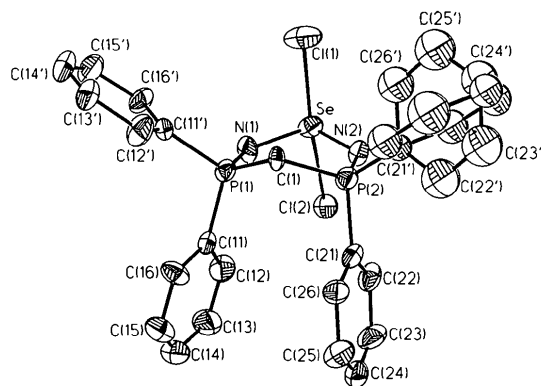


Fig. 1. View of C<sub>25</sub>H<sub>22</sub>Cl<sub>2</sub>N<sub>2</sub>P<sub>2</sub>Se·2CHCl<sub>3</sub> showing the labelling of the non-H atoms. Displacement ellipsoids are plotted at the 30% probability level.

### Experimental

#### Crystal data

C<sub>25</sub>H<sub>22</sub>Cl<sub>2</sub>N<sub>2</sub>P<sub>2</sub>Se·  
2CHCl<sub>3</sub>  
M<sub>r</sub> = 800.98  
Monoclinic  
P2<sub>1</sub>/n  
a = 10.785 (5) Å  
b = 15.709 (6) Å  
c = 20.884 (8) Å  
β = 100.89 (3)°  
V = 3474.5 (9) Å<sup>3</sup>  
Z = 4  
D<sub>x</sub> = 1.531 Mg m<sup>-3</sup>

Mo Kα radiation  
λ = 0.71069 Å  
Cell parameters from 40  
reflections  
θ = 10–12.5°  
μ = 1.810 mm<sup>-1</sup>  
T = 293 K  
Transparent block  
0.70 × 0.30 × 0.30 mm  
Yellow  
Crystal source: dimethyl-  
formamide/chloroform



H(secondary CH<sub>2</sub> group) 0.97 Å or C—H(aromatic) 0.93 Å. The disordered phenyl rings were refined as a rigid group applying the geometry of a regular hexagon with isotropic displacement parameters for the C atoms. The site occupancy factor of each of the two rings was refined as one additional variable by constraining the sum of the two values to 1.

Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993).

I thank Professor Dr H. W. Roesky, University of Göttingen, for providing the sample.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: SE1064). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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### 3-(4-Butoxycarbonylphenyloxycarbonyl)-xanthen-9-one

ROBERTO CENTORE, MARIA ROSARIA CIAJOLO  
AND ANGELA TUZI

*Dipartimento di Chimica, Università di Napoli  
'Federico II', Via Mezzocannone 4, 80134 Napoli,  
Italy*

HANS R. KRICHELDORF

*Institut für Technische und Makromolekulare  
Chemie, Universität Hamburg, Bundesstrasse 45,  
20146 Hamburg, Germany*

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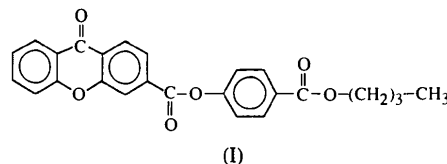
### Abstract

The title compound, butyl 4-[(9-oxoxanthen-3-yl)-carbonyloxy]benzoate, C<sub>25</sub>H<sub>20</sub>O<sub>6</sub>, has been obtained as a product of the intramolecular cyclization of 2-phenoxyterephthaloyl dichloride in the presence of butyl 4-hydroxybenzoate. The crystal and molecular structures

are reported. The xanthone group is planar to within 0.084(1) Å. Molecules in the crystal are packed with planes of xanthone groups parallel to one another.

### Comment

The title compound, (I), was obtained as a product (60% yield) of the reaction between 2-phenoxyterephthaloyl dichloride and butyl 4-hydroxybenzoate (mole ratio 2:1) in hot pyridine (2-phenoxyterephthaloyl dichloride was obtained by refluxing 2-phenoxyterephthalic acid with thionyl chloride and removing the excess thionyl chloride by distillation under reduced pressure; butyl 4-hydroxybenzoate was obtained by Fischer esterification of 4-hydroxybenzoic acid and *n*-butanol). The raw product, obtained after pouring the reaction mixture into an excess of ethanol, was purified by silica-gel chromatography, using chloroform as eluent, and finally recrystallized from chloroform–hexane solution (m.p. 403 K).



The proton NMR spectrum was not consistent with the structure of the expected product, *i.e.* bis(4-butoxycarbonylphenyl)-2-phenoxyterephthalate, indicating the formation of a different compound. This seemingly unusual behaviour is actually consistent with previous observations (Kricheldorf, Schwarz & Ruhser, 1988). It was shown, in fact, that intramolecular cyclization of 2-phenoxyterephthaloyl dichloride (*via* Friedel–Crafts acylation) may also occur under rather mild conditions giving xanthone-3-carboxylic acid chloride. In our case, the formation of the title compound may be accounted for by intramolecular cyclization of 2-phenoxyterephthaloyl dichloride followed by esterification with butyl 4-hydroxybenzoate. The X-ray analysis was undertaken for the definitive assignment of the molecular structure.

Bond lengths and angles in the molecule are all close to expected values. In particular, as far as the geometry of the xanthone moiety is concerned, the observed bond lengths and angles are in fair agreement with those previously reported for xanthone (Onuma, Iijima & Oonishi, 1990). A small, but probably statistically significant, spread of the bond lengths in the benzenoid rings of the xanthone group was observed, the shortest C—C distances being C17—C18 and C24—C25 [1.375(1) and 1.377(1) Å, respectively] and C13—C14 and C22—C23 [1.381(1) and 1.380(1) Å, respectively]. This is consistent with the central pyranoid ring having partial aromatic character (Lumbroso, Curé & Evers, 1986).