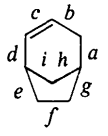
Table 5. *Torsion angles* (°), *with standard deviations of* 1°, *in OZOBR, OZONO, and BCO*



Bonds involved	OZOBR	OZONO	BCO
a–b–c	2	6	−6
a–h–i	74	−67	−68
b–c–d	1	−1	4
c–d–e	−79	73	72
c–d–i	33	−39	−38
d–i–h	−70	75	69
d–e–f	79	−91	−92
e–f–g	15	−1	5
f–g–a	−100	89	81
f–g–h	13	−25	−32
g–a–b	69	−81	−71
g–h–i	−36	42	48
h–a–b	−40	29	40
h–i–e	46	−42	−43
i–e–f	−36	26	24

Table 6. *Ring-puckering coordinates*

Ring	q_2 (Å)	q_3 (Å)	φ_2 (°)	φ_3 (°)	θ (°)
Cyclopentyl	0·412		199·1		
Cyclohexyl	0·484	0·358	355·1		53·5
Cycloheptyl	1·063	0·374	339·3	61·1	

An envelope conformation of the six-atom ring is indicated. An ideal envelope conformation for an isometric six-atom ring has puckering coordinates $\varphi_2 = 0°$ and $\tan\theta = 2^{1/2}$. The geometry of the seven-atom ring may be described in terms of two planar residues. One residue contains the atoms C(1), C(10), C(11), C(5) and C(4), while the other consists of C(1), O(1), O(2) and C(4). These residues intersect to form an angle of 106·0°. The ring-puckering coordinates for the five-atom ring are intermediate between the values expected for the envelope form and those expected for the half-boat form. The magnitudes of the positional shifts for atoms O(1) and O(2) required for the ring to assume the envelope form are less than 0·15 Å; thus the conformational difference between OZOBR and OZONO may be due to the different packing environments within their crystals.

No intermolecular contact distances are shorter than the sum of the respective van der Waals radii of the atoms (Pauling, 1960).

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2-Phenylquinazoline 1,3-Dioxide

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Abstract. $C_{14}H_{10}N_2O_2$, $M_r = 238·2$, orthorhombic, $Pca2_1$, $a = 12·868$ (1), $b = 5·3709$ (5), $c = 15·784$ (1) Å (Cu $K\alpha$, $\lambda = 1·5418$ Å), $Z = 4$, $D_m = 1·44$, $D_c = 1·45$ Mg m^{-3}; $R = 0·030$ for 683 diffractometer data. The N—O bonds and the phenyl ring disturb the electronic structure of the parent quinazoline.

0567-7408/79/092276-03$01.00

Introduction. Quinazoline *N*-monooxides undergo interesting photochemical rearrangements (Rees, Somanathan, Storr & Woolhouse, 1975; Field & Sternbach, 1968). The title compound (I) was prepared to investigate the photochemistry of a quinazoline *N,N'*-dioxide. The analysis was undertaken to characterize

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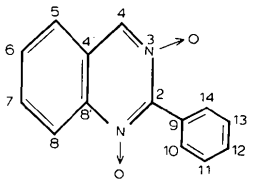
the compound structurally and to investigate the effects of the *N*-oxide substitution on the aromatic system.



(I)

The crystals were pale-yellow rectangular prisms. The systematic absences $0kl$, $l \neq 2n$ and $h0l$, $h \neq 2n$ are consistent with space groups $Pca2_1$ and $Pcam$. Subsequent solution of the structure showed $Pca2_1$ to be correct. Intensities and cell dimensions were obtained from a crystal 0·19 × 0·20 × 0·23 mm. Cell dimensions were determined by least squares from the parameters to 12 reflections centred on a Hilger & Watts automated diffractometer. Data were collected to a maximum of $\theta = 57{\cdot}3°$ (Ni-filtered Cu $K\alpha$ radiation, θ–2θ scan, scan width = 0·60°, scan time = 60 s). Three standard reflections measured periodically showed only random fluctuations of ±1·5%.

Intensities were obtained for 803 reflections of which 683 were considered ($I > 3\sigma_I$). The structure was solved by direct methods and refined by full-matrix least squares. Initially, an experimental ($w = 4F_o^2/\sigma_I^2$) weighting scheme was used; however, this proved to be biased, so an empirical scheme [$w = 1/(10{\cdot}0 + F_o + 0{\cdot}018F_o^2 + 0{\cdot}0008F_o^2)$] was used. The non-hydrogen atoms were refined anisotropically. Only the positional parameters of the H atoms were refined ($U_H = 0{\cdot}05$ Å^2). At convergence (all shifts $<0{\cdot}1\sigma$), $R = 0{\cdot}030$, $R_w = 0{\cdot}037$. A final difference synthesis showed no unusual features.

Table 1. *Atomic coordinates* (×10^4) *for the non-hydrogen atoms in 2-phenylquinazoline* 1,3*-dioxide*

E.s.d.'s are in parentheses.

	x	*y*	*z*
O(1)	8022 (2)	8264 (4)	2947 (2)
O(3)	9532 (2)	1569 (5)	1637 (2)
N(1)	8803 (2)	6793 (5)	2848 (2)
N(3)	9613 (2)	3305 (5)	2201 (2)
C(2)	8781 (3)	4984 (6)	2261 (2)
C(4)	10442 (3)	3530 (7)	2694 (3)
C(4′)	10495 (3)	5449 (6)	3295 (2)
C(5)	11359 (3)	5754 (8)	3848 (3)
C(6)	11361 (3)	7624 (8)	4426 (3)
C(7)	10538 (3)	9301 (8)	4482 (3)
C(8)	9688 (3)	9092 (7)	3958 (3)
C(8′)	9678 (3)	7128 (6)	3373 (2)
C(9)	7903 (2)	4809 (6)	1673 (3)
C(10)	7754 (3)	6750 (7)	1098 (3)
C(11)	6918 (3)	6641 (7)	548 (3)
C(12)	6230 (3)	4703 (8)	578 (3)
C(13)	6372 (3)	2774 (7)	1150 (3)
C(14)	7224 (3)	2817 (7)	1693 (3)

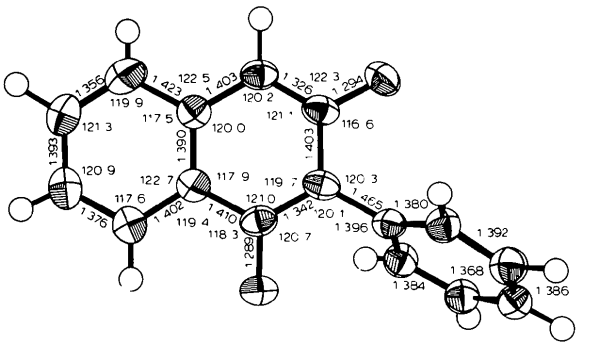
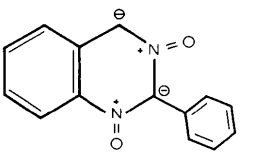


Fig. 1. A view of the molecule showing bond lengths (Å) and angles (°). E.s.d.'s for non-hydrogen bond lengths are in the range 0·004–0·006 Å, and for the bond angles given are in the range 0·3–0·4°. Thermal ellipsoids are drawn at the 50% probability level.

The coordinates for the non-hydrogen atoms are given in Table 1,* the numbering scheme being the same as in (I). Bond lengths and angles are shown in Fig. 1.

Discussion. The crystal structure consists of discrete molecules. At 1·292 Å, the average N–O length is slightly shorter than those in similar bicyclic aromatic systems [*e.g.* 1·333 (3) Å, Desiderato, Terry, Freeman & Levy, 1971; 1·310 (2) Å, Golič, Kaučič, Stanovnik & Tišler, 1975], about the same as pyridine *N*-oxides (Shiro, Yamakawa & Kubota, 1977) and longer than those in non-aromatic systems [*e.g.* 1·258 (8) Å, Keana, Norton, Morello, van Engen & Clardy, 1978]. All of the C–N bonds are longer than those of quinazoline (Huiszoon, 1976). These facts suggest a greater contribution to the resonance structure by canonical forms such as (II). This is in agreement with the findings of Shiro, Yamakawa & Kubota (1977) who investigated a series of substituted pyridine *N*-oxides.



(II)

The quinazoline fragment is planar within experimental error; the angle between this plane and that of the pendant phenyl ring is 66·3°. The bond angles show no unusual features. There are no intermolecular

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and phenyl-ring bond angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 34516 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

close contacts, the molecular packing being due to van der Waals forces only.

We thank Dr A. D. Woolhouse for supplying the crystals.

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Oxalate de *N*-(Diméthylammonio-3 propyl) Amino-2 Dichloro-4,4′ Diphénylsulfure*

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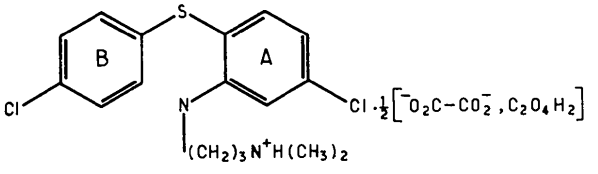
(*Reçu le* 15 *décembre* 1978, *accepté le* 4 *juin* 1979)

Abstract. $(C_{17}H_{21}Cl_2N_2S)_2^+(C_2O_4)^{2-}.C_2H_2O_4$, triclinic, $P\bar{1}$, $Z = 1$, $a = 8{\cdot}732\,(4)$, $b = 13{\cdot}334\,(5)$, $c = 11{\cdot}245\,(5)$ Å, $\alpha = 64{\cdot}95\,(8)$, $\beta = 68{\cdot}45\,(8)$, $\gamma = 89{\cdot}97\,(8)°$, $R = 0{\cdot}061$ for 3340 observed reflexions. The angle between the two ring planes is 99·0 (8)°. The aminoaliphatic chain lies approximately in the *A*-ring plane.

Introduction. La formule développée du composé étudié est:



Ce composé, comme tous les homologues étudiés précédemment, a été synthétisé au Laboratorie de Pharmacie Chimique, UER de Pharmacie, Lille, France (Professeur M. Debaert); dans la nomenclature de ces composés il est référencé CB30. Rappelons que ces molécules peuvent être considérées comme des phénothiazines dont l'hétérocycle central serait ouvert. On retrouve ici essentiellement le motif de la chlorpromazine mais un deuxième atome de chlore a été substitué sur le cycle *B*. Le cristal de CB30 utilisé est une plaquette de 0,3 × 0,3 × 0,1 mm obtenue par évaporation lente d'une solution à chaud dans un mélange eau–méthanol. Les angles θ, χ et φ de 14 réflexions, mesurés au diffractomètre Siemens, ont permis d'obtenir les paramètres de la maille cristalline (affinement par moindres carrés). La structure a été résolue au moyen du programme *SIR* (Busetta, 1979), à partir de 223 facteurs normalisés avec $E > 1{,}5$. La molécule de CB30 est apparue sans ambiguïté sur les cartes de Fourier (associée à quelques pics parasites) tandis que l'acide oxalique et l'anion oxalate, tous deux en position particulière, sur un centre de symétrie, pouvaient être définis de multiples façons. Les atomes d'hydrogène ont tous été placés à partir des cartes de Fourier à l'exception de la molécule d'acide oxalique. Le Tableau 1 fournit l'ensemble des coordonnées atomiques et facteurs isotropes d'agitation thermique.† Il n'a pas été utilisé de schéma de pondération.

Discussion. Les distances interatomiques et les angles de liaison sont représentés sur la Fig. 1. L'écart-type est en moyenne de 0,008 Å (0,007 Å pour les distances S–C et S–Cl) et 0,4° pour les angles. La liaison S–C est parfaitement symétrique ce qui n'est pas habituel

* Dérivés de l'Amino-2 Diphénylsulfure. V.

† Les listes des facteurs de structure et des facteurs d'agitation thermique anisotrope ont été déposées au dépôt d'archives de la British Library Lending Division (Supplementary Publication No. SUP 34453: 26 pp.). On peut en obtenir des copies en s'adressant à: The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, Angleterre.

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