

involved in hydrogen bonding with  $O(22)-H(22) = 0.92$  (8),  $O(22)\cdots O(23)$  ( $1-x$ ,  $0.5+y$ ,  $1.5-z$ ) =  $2.597$  (4),  $H(22)\cdots O(23) = 1.72$  (9) Å,  $\angle O(22)-H(22)\cdots O(23) = 160.5$  (5)°; and  $O(24)-H(24) = 1.05$  (5),  $O(24)\cdots O(21)$  ( $1-x$ ,  $0.5+y$ ,  $1.5-z$ ) =  $2.683$  (4),  $H(24)\cdots O(21) = 1.64$  (5) Å,  $O(24)-H(24)\cdots O(21) = 172.6$  (5)°. Haplociliatic acid is the C(10) epimer of the *cis*-clerodane cistodoic acid (Berti, Livi & Segnini, 1970).

We would like to acknowledge the financial support of The Robert A. Welch Foundation (P-074). This is FASTBIOS contribution No. 86.

*Acta Cryst.* (1983). C39, 1075–1076

### Structure of 3-(*o*-Carboxyphenyl)-1-phenyltriazeno 1-Oxide, $C_{13}H_{11}N_3O_3$

By S. B. SARKAR, MD. KHALIL, S. C. SAHA\* AND S. K. TALAPATRA†

*X-ray Laboratory, Department of Physics, Jadavpur University, Calcutta-700032, India*

(Received 7 February 1983; accepted 4 April 1983)

**Abstract.**  $M_r = 257.0$ , monoclinic,  $P2_1/n$ ,  $a = 8.006$  (3),  $b = 11.523$  (6),  $c = 13.937$  (10) Å,  $\beta = 102.09$  (4)°,  $V = 1257$  (1) Å<sup>3</sup>,  $Z = 4$ ,  $D_m = 1.25$  Mg m<sup>-3</sup> (aq. KI),  $D_x = 1.36$  Mg m<sup>-3</sup>,  $\lambda(Cu K\alpha) = 1.5418$  Å,  $\mu = 0.78$  mm<sup>-1</sup>,  $F(000) = 536$ , room temperature. Final value of  $R = 0.051$  for 1499 reflections. The structure establishes the *N*-oxide form of the triazene and reveals both intramolecular [N(3)–H(5)⋯O(3) =  $2.659$  (3) Å] and intermolecular [O(1)⋯H(4)–O(2) =  $2.643$  (2) Å] hydrogen bonds, the latter being responsible for the formation of dimers.

**Introduction.** This paper is part of a systematic study in progress on the metal complexes of the title compound. The compound is used as an analytical reagent and as a potential ligand for synthesis of complexes. IR studies show that the triazene should exist as the *N*-oxide instead of the *N*-hydroxy form (Majumdar & Saha, 1976). The present investigation confirms the *N*-oxide form.

**Experimental.** Light-yellow crystals (from ethanol),  $0.55 \times 0.32 \times 0.17$  mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Cu  $K\alpha$  radiation, least-squares cell parameters from 25 reflections taken from all octants in reciprocal space, scattering factors for non-hydrogen atoms from *International Tables for X-ray crystallography* (1962)

\* Department of Chemistry, Jadavpur University, Calcutta-700032, India.

† To whom correspondence should be addressed.

#### References

- ANDERSON, A. B., MCCRINDLE, R. & NAKAMURA, E. (1974). *J. Chem. Soc. Chem. Commun.* pp. 453–454.  
 BERTI, G., LIVI, O. & SEGNINI, E. (1970). *Tetrahedron Lett.* pp. 1401–1404.  
 BITTNER, M. L., ZABEL, V., SMITH, W. B. & WATSON, W. H. (1978). *Phytochemistry*, **17**, 1797.  
 GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.  
 HAMILTON, W. C. (1965). *Acta Cryst.* **18**, 502–510.  
 JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Oak Ridge, Tennessee.  
 STEWART, J. M., MACHIN, P. A., DICKINSON, C. W., AMMON, H. L., HECK, H. & FLACK, H. (1976). The XRAY76 system. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

and, for H atoms, from Stewart, Davidson & Simpson (1965),  $1858 \pm hkl$  with  $2\theta < 50^\circ$ , 1499 independent, 359 with  $I < 2\sigma(I)$ , Lp correction applied, absorption ignored; direct methods (*MULTAN78*, Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), non-hydrogen atoms anisotropic full-matrix, H atoms (from  $\Delta F$  synthesis and also calculated from known geometry around C atoms) isotropic: maximum and minimum heights in final difference Fourier map  $\pm 0.2e$  Å<sup>-3</sup>, maximum least-squares shift-to-error ratio 0.255 and average shift-to-error ratio 0.088, final  $R = 0.051$  with 216 parameters and  $R_w = 0.078$ ; block-diagonal approximation [ $\sum w(\Delta F)^2$  minimized] used with a weighting factor  $w = 1/\sigma^2(F)$ , with  $\sigma(F) = R_i |F_o|$  (Seal & Ray, 1981); Burroughs 6700 computer (Regional Computer Centre, Calcutta), modified versions of *MAMIE* and *BLOK* from the X-RAY ARC program system.

**Discussion.** The atomic coordinates are listed in Table 1 and interatomic distances and angles in Table 2.‡ The phenyl rings of the molecule are inclined to one another by  $13.3$  (1)°. All bond lengths and bond angles agree well with the values reported in the literature. The double-bond distance N(1)–N(2) is shorter than

‡ Lists of structure factors, anisotropic thermal parameters and least-squares-planes' data have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38507 (16 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates ( $\times 10^4$ ) and isotropic temperature factors ( $\text{\AA}^2$ )

$$B_{eq} = \frac{1}{3} \sum_i \sum_j \beta_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>B</i> <sub>eq</sub>
C(1)	42 (3)	909 (3)	1516 (3)	4.8
C(2)	426 (3)	1832 (3)	2162 (2)	4.9
C(3)	2100 (3)	2082 (3)	2557 (2)	4.3
C(4)	3439 (3)	1442 (2)	2312 (2)	3.4
C(5)	3028 (3)	518 (2)	1650 (2)	3.3
C(6)	1321 (3)	262 (3)	1254 (2)	4.1
C(7)	2963 (5)	-3971 (3)	-890 (3)	6.1
C(8)	3258 (4)	-3023 (3)	-267 (2)	5.1
C(9)	4899 (4)	-2560 (2)	-29 (2)	4.0
C(10)	6225 (4)	-3024 (3)	-377 (2)	5.2
C(11)	5878 (5)	-3991 (3)	-1006 (3)	6.3
C(12)	4282 (6)	-4445 (3)	-1254 (3)	6.6
C(13)	5226 (3)	1777 (2)	2730 (2)	3.7
N(1)	5221 (3)	-1559 (2)	612 (2)	3.6
N(2)	3909 (3)	-1020 (2)	780 (2)	3.7
N(3)	4336 (3)	-130 (2)	1382 (2)	3.7
O(1)	6755 (2)	-1231 (2)	988 (2)	4.6
O(2)	5361 (2)	2631 (2)	3373 (2)	5.2
O(3)	6466 (2)	1330 (2)	2505 (2)	5.0

Table 2. Bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ )

C(1)–C(2)	1.386 (5)	N(3)–N(2)	1.324 (3)
C(2)–C(3)	1.369 (4)	N(2)–N(1)	1.283 (3)
C(3)–C(4)	1.401 (4)	N(1)–C(9)	1.449 (3)
C(4)–C(5)	1.402 (4)	N(1)–O(1)	1.287 (3)
C(5)–C(6)	1.395 (4)	C(10)–C(11)	1.409 (5)
C(6)–C(1)	1.376 (4)	C(11)–C(12)	1.357 (6)
C(4)–C(13)	1.479 (4)	C(12)–C(7)	1.376 (6)
C(13)–O(3)	1.216 (3)	C(7)–C(8)	1.385 (5)
C(13)–O(2)	1.321 (4)	C(8)–C(9)	1.393 (4)
C(5)–N(3)	1.398 (3)	C(9)–C(10)	1.365 (4)
		C(1)–H(1)	1.010 (28)

C(2)–C(1)–C(6)	120.8 (3)	N(2)–N(1)–O(1)	122.1 (2)
C(1)–C(6)–C(5)	120.2 (3)	C(9)–N(1)–O(1)	121.0 (2)
C(6)–C(5)–C(4)	119.7 (2)	C(8)–C(9)–C(10)	122.1 (3)
C(4)–C(3)–C(2)	121.8 (3)	O(2)–H(4)–O(1)	158.5 (1)
C(3)–C(2)–C(1)	119.2 (3)	C(9)–C(10)–C(11)	117.6 (3)
C(5)–C(4)–C(3)	118.3 (2)	C(10)–C(11)–C(12)	121.0 (3)
C(5)–C(4)–C(13)	122.1 (2)	C(11)–C(12)–C(7)	120.5 (4)
C(3)–C(4)–C(13)	119.5 (2)	C(12)–C(7)–C(8)	120.1 (3)
C(4)–C(13)–O(2)	113.4 (2)	C(7)–C(8)–C(9)	118.6 (3)
C(4)–C(13)–O(3)	124.3 (2)	C(8)–C(9)–N(1)	119.2 (2)
C(5)–N(3)–N(2)	118.3 (2)	C(10)–C(9)–N(1)	118.7 (2)
N(2)–N(1)–C(9)	116.9 (2)	N(3)–H(5)–O(3)	127.0 (3)
C(4)–C(5)–N(3)	119.7 (3)	N(1)–N(2)–N(3)	112.2 (3)
C(6)–C(5)–N(3)	120.5 (3)		
O(2)–C(13)–O(3)	122.4 (3)		

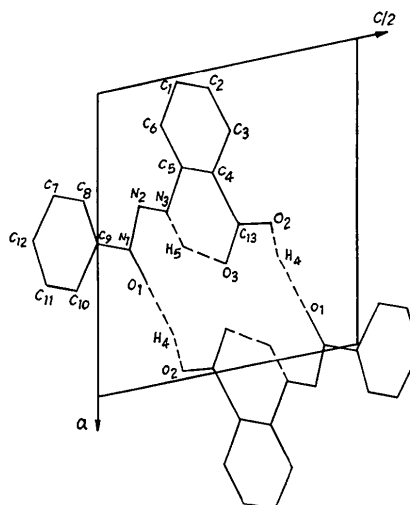


Fig. 1. Molecular projection of the title compound viewed down [010], showing the formation of dimers. Hydrogen bonds are indicated by broken lines.

N(2)–N(3) and C(9)–N(1) is longer than N(3)–C(5) possibly due to the linkage of a more electronegative oxygen to N(1) and a less electronegative hydrogen to N(3). This points to the *N*-oxide form of the triazene and the existence of the NH group which is responsible for the intramolecular H-bonding [N(3)–H(5)···O(3)]. The structure is characterized by the formation of dimers (Fig. 1), in which the molecules are linked by a pair of intermolecular [O(2)–H(4)···O(1)] hydrogen bonds.

The authors are indebted to Dr S. Ray of IACS for helpful discussions. Intensity measurements were carried out at RSIC (Bose Institute, Calcutta).

## References

- International Tables for X-ray Crystallography* (1962). Vol. III. Birmingham: Kynoch Press.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J. P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- MAJUMDAR, A. K. & SAHA, S. C. (1976). *J. Indian Chem. Soc.* **53**, 226.
- SEAL, A. & RAY, S. (1981). *Indian J. Phys.* **55A**, 414.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.