LITHIUM PERCHLORATE TRIHYDRATE

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# 2,3-Dimethyl-4a,9a-diaza-1,4,4a,9,9a,10-hexahydroanthracene-9,10-dione*

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**Abstract.** $C_{14}H_{14}O_2N_2$, monoclinic, $P2_1/c$, $a = 12.216$ (2), $b = 12.627$ (2), $c = 7.890$ (1) Å, $\beta = 101.62$ (1)°, $Z = 4$, $D_x = 1.34$ g cm$^{-3}$, Cu $K\alpha$ radiation. $R = 0.056$ for 1888 observed reflexions. The molecule can be described as a 'strip' twisted about 15° from end to end.

**Introduction.** The determination of the crystal structure of the title compound is a continuation of a study on tetracycline analogues mentioned in a previous work (Foces-Foces, Cano & García-Blanco, 1977).

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\* Crystal and Molecular Structure of Diazapolycyclic Compounds. III.

A crystal 0.40 × 0.20 × 0.40 mm was used, and the lattice parameters and intensities were measured on a Philips PW 1100 diffractometer, with graphite-monochromated Cu $K\alpha$ radiation ($\omega/2\theta$ scan mode); no decomposition was observed. Of the 2036 reflexions recorded with $\theta < 65°$, 1888 had $I > 2\sigma(I)$ and were classified as observed. Lorentz and polarization factors were applied but no absorption correction was made ($\mu = 7.5$ cm$^{-1}$). The structure was solved with *MULTAN* 74 (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and refined by full-matrix least-squares methods. All H atoms were located in a difference Fourier map. A mixed refinement converged to $R = 0.056$ and $R_w = 0.068$. The reflexions were weighted as follows: $w = K/(f|F_o|)^2 f(S)$ where $f(F_o) = 0.25 + 0.05|F_o|$ and

Table 1. *Final positional parameters* (×10⁴, *for* H ×10³) *with their standard deviations and bond distances* (Å) *for the hydrogen atoms*

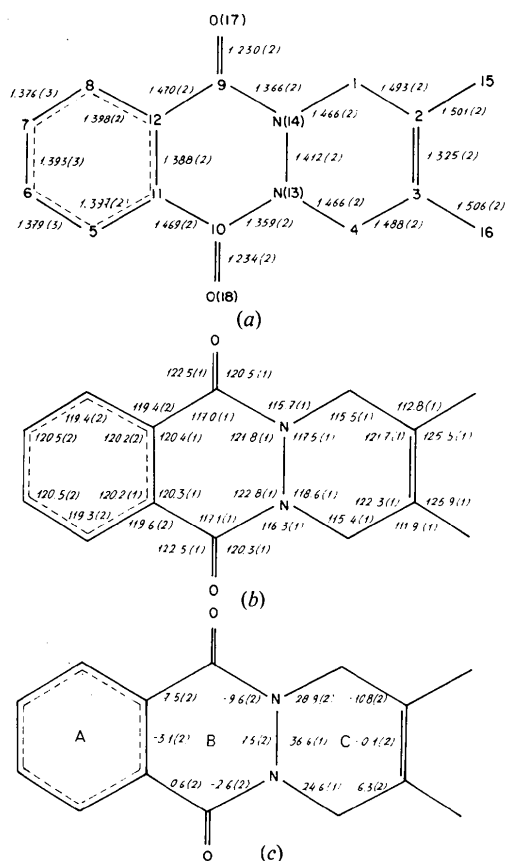|       | $x$       | $y$       | $z$       |         | $x$     | $y$     | $z$     | Bond length |
|-------|-----------|-----------|-----------|---------|---------|---------|---------|-------------|
| C(1)  | 5525 (1)  | 5309 (1)  | 2526 (2)  | H(1a)   | 555 (2) | 458 (2) | 273 (3) | 0.94 (2)    |
| C(2)  | 6618 (1)  | 5751 (1)  | 3442 (2)  | H(1b)   | 541 (2) | 543 (2) | 132 (3) | 0.95 (2)    |
| C(3)  | 6675 (1)  | 6566 (1)  | 4513 (2)  | H(4a)   | 571 (2) | 780 (2) | 456 (3) | 0.93 (2)    |
| C(4)  | 5659 (1)  | 7097 (1)  | 4876 (2)  | H(4b)   | 562 (2) | 699 (2) | 611 (3) | 1.00 (2)    |
| C(5)  | 1604 (1)  | 7329 (2)  | 3486 (2)  | H(5)    | 164 (2) | 800 (2) | 397 (3) | 0.93 (3)    |
| C(6)  | 583 (2)   | 6853 (2)  | 2886 (3)  | H(6)    | −8 (2)  | 721 (2) | 293 (4) | 0.93 (3)    |
| C(7)  | 523 (2)   | 5856 (2)  | 2115 (3)  | H(7)    | −20 (2) | 556 (2) | 167 (3) | 0.96 (2)    |
| C(8)  | 1483 (1)  | 5326 (2)  | 1960 (2)  | H(8)    | 144 (2) | 464 (2) | 147 (4) | 0.95 (3)    |
| C(9)  | 3546 (1)  | 5245 (1)  | 2374 (2)  | H(15a)  | 754 (2) | 515 (2) | 186 (4) | 0.87 (3)    |
| C(10) | 3671 (1)  | 7292 (1)  | 3989 (2)  | H(15b)  | 761 (3) | 450 (3) | 331 (4) | 0.91 (3)    |
| C(11) | 2580 (1)  | 6799 (1)  | 3319 (2)  | H(15c)  | 832 (2) | 545 (2) | 352 (4) | 0.95 (3)    |
| C(12) | 2521 (1)  | 5799 (1)  | 2577 (2)  | H(16a)  | 785 (2) | 777 (2) | 507 (4) | 0.95 (3)    |
| N(13) | 4595 (1)  | 6732 (1)  | 3831 (2)  | H(16b)  | 842 (2) | 669 (2) | 544 (3) | 0.98 (3)    |
| N(14) | 4539 (1)  | 5701 (1)  | 3125 (2)  | H(16c)  | 774 (2) | 706 (2) | 673 (4) | 0.99 (3)    |
| C(15) | 7602 (1)  | 5189 (2)  | 2983 (2)  |         |         |         |         |             |
| C(16) | 7731 (1)  | 7079 (2)  | 5477 (2)  |         |         |         |         |             |
| O(17) | 3528 (1)  | 4410 (1)  | 1565 (1)  |         |         |         |         |             |
| O(18) | 3766 (1)  | 8161 (1)  | 4720 (2)  |         |         |         |         |             |

Fig. 1. Intramolecular geometry. (a) Bond lengths (Å), (b) bond angles (°) and (c) endocyclic torsion angles (°).



Fig. 2. (a) Orthonormal reference for the 'strip' description and (b) the $\varphi$ angles.

$f(S) = 0.57 + 18.03S - 36.28S^2$ when $S < 0.4$, and $f(S) = 14.12 - 55.89S + 57.85S^2$ otherwise ($S = \sin\theta/\lambda$, $K = 0.61$). A final difference synthesis contained random fluctuations within $\pm0.44$ e Å$^{-3}$.

Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Final atomic parameters are given in Table 1.* Bond distances, angles and torsion angles are given in Fig. 1.

**Discussion.** The conformational parameters for N—C($sp^2$) are $\chi_N$(17·6; 25·7°), $\chi_C$(2·3; 0·3°) and $\tau$(5·1; 3·1°) for N(13) and N(14) respectively (Winkler & Dunitz, 1971). These atoms show mean bond angles of 119·2 and 118·3° respectively.

The differences in angles around N(14) and C(2) prevent a possible binary axis, as compared by half-normal probability plots (Abrahams & Keve, 1971). When, in the same way, this compound is compared

Table 2. *The conformational parameters describing the three-ring 'strip'*

| Atomic planes | $\tau$ | $\theta$ | $\varphi$ | $\kappa$ |
|---|---|---|---|---|
| $\pi_1$ (5, 6, 7, 8, 11, 12) | 86° | 5° | −11° | 1° |
| $\pi_2$ (9, 10, 11, 12) | 85 | 4 ↑ | −11 ↑ | 1 |
| $\pi_3$ (9, 10, 13, 14) | 86 | 5 ~ 15° | 33 ~ 180° | −3 |
| $\pi_4$ (1, 4, 13, 14) | 97 | 7 ↓ | 194 ↓ | 2 |
| $\pi_5$ (1, 2, 3, 4) | 100 | 10— | 175— | −1 |

with a previously reported three-ring epoxide system (Foces-Foces, Cano & Garcia-Blanco, 1977), it is found that the only differences are due to the distances involving the epoxide group and the different conformation of ring C. The conformational parameters for the pyridazine ring (Cano, Foces Foces & Garcia Blanco, 1977) are $\tau_m = -17.9°$, $q = 18.5°$, $\Sigma_0 = 344.2°$, $\delta = 328.9°$, $\alpha_1 = 336.6°$, and $\alpha_2 = 7.7°$, which show a slightly distorted half-chair conformation at the C(2) -C(3) bond. It can be seen from the values of the torsion angles that the binary axis is approximately maintained in ring C, while in ring B it passes through the C(9)—N(14) bond.

As far as the planarity is concerned we have divided the three rings into five parts, four half-rings plus the aromatic ring, $\pi_5$ to $\pi_1$, and so, to a first approxi-

mation, the molecule can be considered as a strip twisted somewhere between N(13)–C(4) and N(14)–C(1). Quantitatively the situation can be described with an orthonormal reference frame $\{T,N,B\}$, as is shown in Fig. 2. Then each plane can be described by its normal, giving the alternative parameters $(\tau,\theta)(\varphi,\kappa)$. The results are given in Table 2.

A short intermolecular contact of 2·47 Å has been found between O(17) and H(4a) [O(17)···C(4) = 3·356 (2) Å and O(17)···H(4a)–C(4) = 159 (2)°].

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# Berylliumtellurat Be4TeO7

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**Abstract.** Powder samples of $Be_4TeO_7$ have been obtained by solid-state reaction of BeO with $TeO_2$ in the presence of air at 690–800°C. X-ray and neutron powder diffraction data indicate a face-centred cubic lattice with $a = 7\cdot5770$ (4) Å, $Z = 4$, $D_c = 4\cdot208$ (2), $D_x = 4\cdot18$ (2) g cm$^{-3}$. A structure model, space group $F\bar{4}3m$, containing regular octahedral $TeO_6$ groups $(d_{Te-O} = 1\cdot919$ Å), tetrahedrally coordinated Be atoms $(d_{Be-O} = 1\cdot614$ and $1\cdot636$ Å), and O atoms, which are tetrahedrally coordinated by Be, has been refined by neutron diffraction profile analysis to $R = 0\cdot042$ on intensity values. In this model, $TeO_6$ and $Be_4O$ groups are arranged like the Zn and S atoms in the sphalerite structure.

**Einleitung.** Zur Präparation von $Be_4TeO_7$ wurden Mischungen von Berylliumoxid, reinst (Merck), und $TeO_2$ (99,7%, Th. Schuchardt), die einen $TeO_2$-Überschuss von ca 10% aufwiesen, fein verrieben und

3d in einer Platinschale in einem langsamen Luftstrom bei 690–700°C erhitzt. Danach wurde neu verrieben und 10–14d bei 800°C erhitzt. Das erhaltene gelblichweisse Pulver zersetzt sich bei Temperaturen über ca 850°C, wobei BeO zurückbleibt. Versuche zur Darstellung von Einkristallen blieben bisher erfolglos. BeO und $TeO_2$ setzen sich ausreichend schnell erst nahe 700°C um, wobei $TeO_2$ schon merklich flüchtig ist. Alle untersuchten Proben enthielten – hauptsächlich im Neutronenbeugungsdiagramm erkennbar – noch geringe Mengen der Ausgangssubstanzen. Daher erlaubten die Analysenwerte keinen Schluss auf die Zusammensetzung der Verbindung, die sich endgültig erst aus der Auswertung der Beugungsdaten ergab.

Röntgen-Pulveraufnahmen wurden mit einem Siemens-Pulverdiffraktometer bzw. mit einer Huber-Zählrohr-Guinierkamera (mit Ni gefilterte bzw. an Quarzmonochromatisierte Cu-$K\alpha$-Strahlung, $\lambda = 1{,}54051$ Å) ausgeführt. Die Gitterkonstante wurde