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Triamterene*

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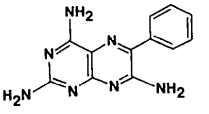
Abstract. 2,4,7-Triamino-6-phenylpteridine, $C_{12}H_{11}N_7$, $M_r = 253{\cdot}28$, triclinic, $P\bar{1}$, $a = 7{\cdot}440$ (1), $b = 10{\cdot}164$ (1), $c = 16{\cdot}666$ (2) Å, $\alpha = 77{\cdot}43$ (1), $\beta = 88{\cdot}75$ (1), $\gamma = 88{\cdot}56$ (1)°, $V = 1229{\cdot}5$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 6{\cdot}59$ cm^{-1}, $F(000) = 528$, $T = 291$ (2) K, final $R = 0{\cdot}090$ for 3186 observed reflections. The two independent molecules are similar in geometry, with the bonds between phenyl and pteridine rings twisted by 31·1 and 33·4° respectively. Molecules are linked into ribbons by hydrogen bonds between both H atoms of the 2-amino groups and N(1) and N(3) of adjacent rings. The ribbons are connected by paired N(7)—H···N(8) hydrogen bonds around centers of symmetry; pteridine rings are also stacked.

Introduction. Triamterene (I) is a valuable potassium-sparing diuretic and also a modest inhibitor of dihydrofolate reductase (DHFR). It contains the same 2,4-diaminopteridine moiety as active DHFR inhibitors such as methotrexate, but the 6-phenyl and 7-amino substituents are evidently not conducive to maximum antifolate activity. The present study was undertaken with the intention of comparing the molecular geometry of triamterene with that of the parent compound 2,4-diaminopteridine (Schwalbe & Williams, 1986*a*) and other antifolates and diuretics.



(I)

Experimental. Platelets grown by slow evaporation from dimethyl sulfoxide with a small quantity of acetone added near the end. Specimen 0·27 × 0·24 × 0·03 mm. Unit-cell dimensions by least-squares analysis of setting angles of 25 reflections, $10{\cdot}7 \le \theta \le 59{\cdot}0°$, graphite-monochromated Cu $K\alpha$ radiation, Enraf–Nonius CAD-4 diffractometer. Data collection by ω–2θ scans, ω-scan rate 1·2 to 3·4° min^{-1}, ω-scan

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range $1{\cdot}50° + 0{\cdot}14°\tan\theta$, background counts taken over the first and final sixths of the scan range. Two intensity and orientation monitor reflections every 1 h and 100 reflections respectively; *ca* 1% decomposition and small movement corrected by reorientation. Lorentz–polarization correction assuming an ideally mosaic monochromator. No correction for absorption. 4808 reflections with $1 \le \theta \le 78°$, $0 \le h \le 9$, $-12 \le k \le 12$, $-20 \le l \le 20$, 4251 unique, $R_{int} = 0{\cdot}036$, 3186 considered observed with $F_o > 3\sigma$, where σ is based on counting statistics and $0{\cdot}02 \times$ (net count) for experimental instability.

Structure solution by *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), least-squares refinement with *SHELX*76 (Sheldrick, 1976), phenyl H atoms inserted in calculated positions, all other H atoms located in successive difference Fourier syntheses. Four peaks in reasonable locations for H atoms repeatedly occurred near each 4-amino group and were eventually accepted with occupancy factors of 0·5, thereby representing twofold positional disorder of these groups, which do not participate in any strong intermolecular interactions (see below). Final refinement on F magnitudes, weighted by $1/\sigma^2$, based on stored scattering factors (Cromer & Mann, 1968; Stewart, Davidson & Simpson, 1965), adjusting positional parameters and anisotropic thermal parameters for non-H atoms and one common isotropic temperature factor for all H atoms, $R = 0{\cdot}090$, $wR = 0{\cdot}084$, $(\Delta/\sigma)_{max} = 0{\cdot}12$, $\Delta\rho_{max} = 0{\cdot}50$, $\Delta\rho_{min} = -0{\cdot}42$ e Å^{-3}. The relatively high R value of 0·090 is attributable to the preponderance of weak diffraction maxima resulting from the thinness of the crystal and the occurrence of H-atom disorder and considerable thermal motion in some regions of the molecule.

Discussion. The molecules with their numbering scheme are shown in Fig. 1. Final atomic coordinates are given in Table 1, bond distances and angles in Table 2, and hydrogen-bond geometry in Table 3.*

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43686 (25 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

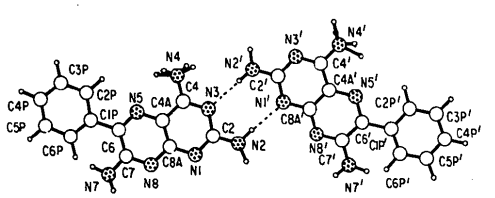

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) drawing of the two independent molecules of triamterene, shown with N atoms stippled, together with their numbering scheme.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* ($\times 10^3$) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}*(Å^2)
N(1)	−1632 (6)	1996 (5)	3614 (3)	38 (3)
C(2)	−102 (8)	2440 (6)	3239 (3)	36 (3)
N(2)	−94 (6)	2790 (5)	2409 (3)	44 (3)
N(3)	1513 (6)	2584 (5)	3596 (3)	39 (3)
C(4)	1516 (8)	2314 (6)	4407 (3)	39 (3)
N(4)	3047 (7)	2467 (6)	4794 (3)	52 (3)
C(4A)	−73 (8)	1894 (6)	4884 (3)	34 (3)
N(5)	−20 (7)	1637 (5)	5716 (3)	40 (3)
C(6)	−1493 (8)	1192 (6)	6126 (3)	37 (3)
C(7)	−3033 (8)	900 (6)	5691 (3)	40 (3)
N(7)	−4527 (7)	314 (5)	6075 (3)	48 (3)
N(8)	−3095 (6)	1185 (5)	4869 (3)	41 (3)
C(8A)	−1599 (8)	1711 (6)	4450 (3)	37 (3)
C(1*P*)	−1425 (9)	939 (6)	7050 (3)	44 (3)
C(2*P*)	237 (9)	535 (6)	7410 (3)	47 (4)
C(3*P*)	347 (13)	311 (8)	8275 (4)	66 (6)
C(4*P*)	−1119 (14)	482 (8)	8750 (4)	82 (6)
C(5*P*)	−2710 (14)	905 (9)	8384 (4)	82 (6)
C(6*P*)	−2903 (10)	1118 (7)	7535 (4)	60 (5)
N(1′)	3416 (6)	3044 (5)	1407 (2)	38 (3)
C(2′)	4987 (8)	2626 (5)	1782 (3)	34 (3)
N(2′)	4929 (6)	2298 (5)	2603 (3)	43 (3)
N(3′)	6601 (7)	2480 (5)	1420 (3)	43 (3)
C(4′)	6644 (8)	2700 (6)	613 (3)	38 (3)
N(4′)	8208 (7)	2524 (6)	217 (3)	47 (3)
C(4A′)	5087 (9)	3147 (6)	138 (3)	40 (3)
N(5′)	5174 (7)	3346 (5)	−691 (3)	39 (3)
C(6′)	3678 (8)	3767 (5)	−1104 (3)	32 (3)
C(7′)	2103 (8)	4068 (6)	−661 (3)	42 (3)
N(7′)	617 (7)	4649 (5)	−1068 (3)	47 (3)
N(8′)	2000 (6)	3828 (5)	148 (3)	40 (3)
C(8A′)	3494 (8)	3319 (6)	562 (3)	35 (3)
C(1*P*′)	3817 (9)	3971 (6)	−2027 (3)	42 (3)
C(2*P*′)	5433 (9)	4414 (7)	−2421 (4)	50 (4)
C(3*P*′)	5621 (12)	4580 (8)	−3263 (4)	71 (5)
C(4*P*′)	4194 (14)	4315 (9)	−3711 (4)	84 (6)
C(5*P*′)	2628 (12)	3847 (9)	−3327 (4)	78 (6)
C(6*P*′)	2409 (11)	3677 (7)	−2489 (4)	61 (5)

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

The two independent molecules are approximately related by the pseudosymmetry operation $\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}-z$. Application of the relation to generate primed atoms from unprimed works best for N(1) with no discrepancy along any crystallographic axis exceeding 0·04 Å, and worst for C(5*P*) with discrepancies up to 0·25 Å. Thus the molecular packing mimics the monoclinic space group $P2_1/n$ with the a axis unique. This pseudo monoclinic symmetry is reflected in the cell parameters ($\beta \simeq \gamma \simeq 90°$). The $h00$ reflections with h odd and $0kl$ with $k+l$ odd are generally weak. Unprimed and primed molecules are fairly similar in geometry, the major variation in bond distances of 0·024–0·026 Å (3σ) occurring around C(2). The mean difference between corresponding bond lengths is 0·011 Å; no differences are significant if the e.s.d.'s are underestimated by a factor of about 1·4 (Taylor & Kennard, 1986).

In a series of protonated diamino-substituted nitrogen heterocycles it was found that the exocyclic C—NH_2 distances decrease as the number of ring N atoms increases (Schwalbe & Williams, 1986*b*). Similarly, the rather short average exocyclic C—N distance in 2,4-diaminopteridine of 1·335 (3) Å (Schwalbe & Williams, 1986*a*) can be attributed to the

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

	Unprimed	Primed		Unprimed	Primed		Unprimed	Primed
C(2)–N(1)	1·327 (6)	1·352 (6)	N(5)–C(4A)	1·354 (6)	1·352 (7)	C(8A)–N(8)	1·358 (6)	1·352 (6)
C(8A)–N(1)	1·360 (6)	1·375 (6)	C(8A)–C(4A)	1·398 (7)	1·393 (8)	C(2*P*)–C(1*P*)	1·399 (8)	1·395 (8)
N(2)–C(2)	1·351 (6)	1·337 (6)	C(6)–N(5)	1·316 (7)	1·332 (6)	C(6*P*)–C(1*P*)	1·382 (8)	1·390 (8)
N(3)–C(2)	1·377 (6)	1·351 (7)	C(7)–C(6)	1·441 (7)	1·434 (7)	C(3*P*)–C(2*P*)	1·414 (8)	1·380 (8)
C(4)–N(3)	1·320 (7)	1·315 (7)	C(1*P*)–C(6)	1·507 (7)	1·509 (7)	C(4*P*)–C(3*P*)	1·364 (11)	1·376 (11)
N(4)–C(4)	1·350 (7)	1·352 (7)	N(7)–C(7)	1·355 (6)	1·363 (7)	C(5*P*)–C(4*P*)	1·361 (11)	1·362 (11)
C(4A)–C(4)	1·431 (7)	1·426 (7)	N(8)–C(7)	1·338 (6)	1·316 (6)	C(6*P*)–C(5*P*)	1·394 (9)	1·376 (9)
C(8A)–N(1)–C(2)	115·3 (5)	115·2 (5)	C(8A)–C(4A)–N(5)	123·4 (5)	122·4 (6)	N(8)–C(8A)–N(1)	118·1 (5)	118·2 (5)
N(2)–C(2)–N(1)	117·3 (5)	116·2 (5)	C(6)–N(5)–C(4A)	117·5 (5)	117·5 (5)	N(8)–C(8A)–C(4A)	119·4 (5)	120·4 (5)
N(3)–C(2)–N(1)	127·7 (5)	127·4 (5)	C(7)–C(6)–N(5)	119·8 (5)	119·1 (5)	C(2*P*)–C(1*P*)–C(6)	116·9 (6)	118·8 (6)
N(3)–C(2)–N(2)	115·0 (5)	116·4 (5)	C(1*P*)–C(6)–N(5)	116·8 (5)	116·2 (5)	C(6*P*)–C(1*P*)–C(6)	122·8 (6)	121·9 (6)
C(4)–N(3)–C(2)	116·1 (5)	116·8 (5)	C(1*P*)–C(6)–C(7)	123·3 (5)	124·6 (5)	C(6*P*)–C(1*P*)–C(2*P*)	120·3 (6)	119·3 (6)
N(4)–C(4)–N(3)	119·0 (5)	119·6 (5)	N(7)–C(7)–C(6)	123·0 (5)	120·7 (5)	C(3*P*)–C(2*P*)–C(1*P*)	118·1 (7)	120·3 (7)
C(4A)–C(4)–N(3)	121·6 (6)	121·7 (6)	N(8)–C(7)–C(6)	122·3 (5)	123·1 (5)	C(4*P*)–C(3*P*)–C(2*P*)	121·3 (8)	119·4 (8)
C(4A)–C(4)–N(4)	119·3 (5)	118·7 (5)	N(8)–C(7)–N(7)	114·7 (6)	116·1 (6)	C(5*P*)–C(4*P*)–C(3*P*)	119·4 (7)	120·5 (7)
N(5)–C(4A)–C(4)	119·9 (5)	120·1 (6)	C(8A)–N(8)–C(7)	117·3 (5)	117·0 (5)	C(6*P*)–C(5*P*)–C(4*P*)	121·7 (8)	121·0 (8)
C(8A)–C(4A)–C(4)	116·6 (5)	117·5 (5)	C(4A)–C(8A)–N(1)	122·4 (5)	121·3 (5)	C(5*P*)–C(6*P*)–C(1*P*)	119·2 (7)	119·4 (7)

Table 3. *Contacts related to hydrogen bonds in the packing of triamterene molecules*

	X···*Y* (Å)	*X*–H···*Y* (°)
N(2)–H(21)···N(3^{i})	3·051 (7)	170
N(2)–H(22)···N(1$'$)	3·055 (6)	160
N(4)–H(41)···N(8^{ii})	3·116 (7)	121
N(4)–H(43)···N(7^{ii})	3·247 (7)	131
N(7)–H(71)···N(2$'^{iii}$)	3·075 (6)	138
N(7)–H(72)···N(8^{iv})	3·038 (6)	169
N(2$'$)–H(21$'$)···N(3)	3·044 (7)	156
N(2$'$)–H(22$'$)···N(1^{ii})	3·067 (6)	165
N(4$'$)–H(41$'$)···N(8$''^{ii}$)	3·136 (7)	126
N(4$'$)–H(43$'$)···N(7$''^{ii}$)	3·237 (7)	113
N(7$'$)–H(71$'$)···N(2^{v})	3·062 (7)	122
N(7$'$)–H(72$'$)···N(8$''^{v}$)	3·048 (7)	164

Symmetry code: (i) $-1+x, y, z$; (ii) $1+x, y, z$; (iii) $-x, -y, 1-z$; (iv) $-1-x, -y, 1-z$; (v) $-x, 1-y, -z$.

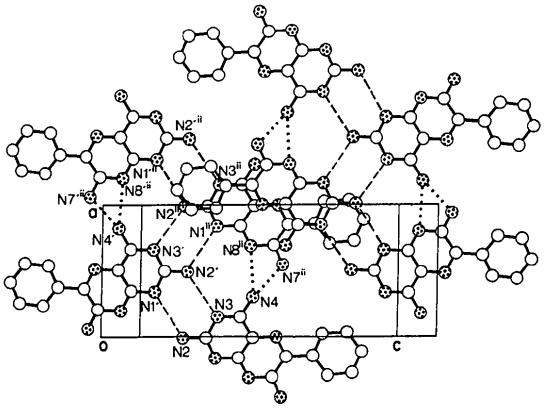

Fig. 2. View of the contents of two unit cells in projection down **b***. The hydrogen bonds from N(2) and N(2′) are shown as dashed lines, while the possible interactions involving disordered H atoms on N(4) and N(4′) are dotted. Symmetry codes follow those in Table 3. Additional hydrogen bonds (not shown) from N(7) and N(7′) provide links in the third dimension.

demand for electrons of four ring N atoms eliciting $C{=}NH_2^+$ resonance structure contributions for just two amino groups. In triamterene the demand is shared among three amino groups, there is less double-bond character in each exocyclic C–N bond, and the average distance increases to 1·351 (8) Å.

As expected (Chatar Singh, 1965), bond angles are less than 120° at the ring N atoms, which are all unprotonated. Steric hindrance is expected between the 6-phenyl and 7-amino substituents. Indeed, the C(6)–C(7) distances in the triamterene molecules are 0·052 (7σ) and 0·045 Å (6σ) longer than that observed in 2,4-diaminopteridine. Interference is relieved by opening the C(7)–C(6)–C(1*P*), C(6)–C(7)–N(7) and C(7′)–C(6′)–C(1*P*′) angles well above 120°, and by twists of −31·1 (4) and −33·4 (4)° respectively between phenyl and pyrazine rings as measured by the N(5)–C(6)–C(1*P*)–C(2*P*) and N(5′)–C(6′)–C(1*P*′)–C(2*P*′) torsion angles. Even so, the C(1*P*)···N(7), C(6*P*)···N(7), C(1*P*′)···N(7′) and C(6*P*′)···N(7′) contacts of 3·008 (7), 3·014 (7), 2·988 (7) and 3·030 (7) Å, respectively, are shorter than the sum of van der Waals radii (Bondi, 1964). The occurrence of such a twist was predicted by Osdene, Russell & Rane (1967) and discussed in relation to possible antifolate activity. It is less than the 67° observed in the antifolate pyrimethamine hydrobromide (Phillips & Bryan, 1969), where the phenyl substituent abuts an ethyl group as well as an amino group.

Molecules are linked by an intricate and unusual network of hydrogen bonds (Table 3 and Fig. 2), in which the 2-amino group is very heavily involved but the 4-amino group plays little part. The usual mode of association of diaminopyridine antifolates is by pairing around a center or pseudo-center of inversion (Schwalbe & Cody, 1983) with N–H···N interactions between N(2) or N(4) amino groups and N(1) or N(3) ring N atoms respectively. This type of interaction does indeed link adjacent N(7) amino groups with N(8) ring N atoms in the pyrazine portion of triamterene. However, the pyrimidine moieties form ribbons in which the N(2) amino group directs one of its protons toward a nearby N(1′) and the other toward N(3′) of a molecule related by translation along −**a**. The N(2′) amino group interacts with N(3), as well as with N(1) of an adjacent molecule generated by the **a** translation.

Both 2-amino groups not only donate both of their protons in hydrogen bonds but also appear to act as proton acceptors for additional hydrogen bonds: the N(7′)···N(2), H(71′)···N(2), N(7)···N(2′) and H(71)···N(2′) contacts are all short. The sum of bond angles at N(2) and N(2′) is 10–13° less than 360°, suggesting some pyramidalization. Pyramidalized amino groups participating in hydrogen bonds as proton acceptors as well as donors have been previously observed in similar triamino compounds: 2,4,6-triamino-5-chloroquinazoline (Rogan & Williams, 1980) and 2,4,6-triaminopyrimidine (Schwalbe & Williams, 1982). The remaining proton donor and acceptor sites at N(4) and N(5) do not hydrogen bond together; instead the N(4) and N(4′) amino groups with H atoms in one set of alternative positions H(41), H(43), H(41′) and H(43′) enter into weak hydrogen-bond-like interactions with N(8), N(7), N(8′) and N(7′) respectively. The alternative coherent set of H-atom positions H(42), H(44), H(42′) and H(44′) does not show this degree of H···N interactions.

Pteridine rings of like kind stack around centers of inversion (Fig. 2). Somewhat surprisingly, the closest contacts are between C atoms with similar substituents: thus C(4)···C(7) and C(4′)···C(7′) are 3·465 (7) and 3·455 (7) Å, respectively. Unprimed phenyl rings are roughly parallel to primed pteridine rings [3·2 (1)° angle between phenyl and pyrazine least-squares planes] and *vice versa* [6·2 (1)° angle].

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Structure of (6,7)-Benzo-2,3,4α,4aβ,8aβ-pentamethyl-4aβ,5,8,8aβ-tetrahydro-1-naphthoquin-4β-ol*

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Abstract. $C_{19}H_{24}O_2$, $M_r = 284{\cdot}40$, monoclinic, $P2_1/c$, $a = 12{\cdot}2068$ (7), $b = 7{\cdot}6627$ (5), $c = 17{\cdot}3623$ (15) Å, $\beta = 105{\cdot}290$ (6)°, $V = 1566{\cdot}5$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}206$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}6$ cm^{-1}, $F(000) = 616$, $T = 295$ K, $R = 0{\cdot}069$ for 1454 observed reflections. The conformation of the molecule is twisted such that the bridgehead methyl groups are staggered with a torsion angle of 64·9°. Bond lengths and angles are close to normal values. Despite the *syn* hydroxyl group, the bulkier methyl causes the molecule to adopt a conformation typical of OH-*anti* derivatives. Molecules in the crystal are linked by hydrogen bonds between symmetry-related molecules with

* IUPAC name: 4β-hydroxy-2,3,4α,4aβ,9aβ-pentamethyl-*cis*-4aβ,9,9aβ,10-tetrahydro-1(4*H*)-anthracenone.

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