component only were allowed anisotropic thermal motion. The long molecular axes of the ordered molecules of compound (3) lie along the z axis, while those of the disordered molecules are disposed approximately orthogonal to this direction, lying parallel to [110]. This non-crystallographic relationship, combined with the disorder, presumably results in a lower lattice energy than any arrangement with one molecule per asymmetric unit. For compound (3), H atoms were initially placed at calculated positions, with $U_{iso} = 1.5U_{eq}$ (for methyl groups) or $1.2U_{eq}$ (for others) of their parent atom. The H atoms of the minor disorder component (primed molecule) were not included.

For both compounds, data collection: *DIF*4 (Stoe & Cie, 1992*a*); cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*) for (3); *X-RED* (Stoe & Cie, 1995) for (4). Program(s) used to solve structures: *SHELXTL/PC* (Sheldrick, 1995) for (3); *SIR*92 (Altomare *et al.*, 1994) for (4). For both compounds, program(s) used to refine structures: *SHELXL*93 (Sheldrick, 1993); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXL*93.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and least-squares-planes data for compounds (3) and (4), and displacement ellipsoid plots of compounds (1), (2) and the disordered molecule of (3), have been deposited with the IUCr (Reference: AB1382). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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4-Methyl-1,2,4-triazole and 1-Methyltetrazole

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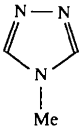
Abstract

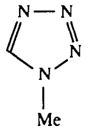
The crystal structures of 4-methyl-4*H*-1,2,4-triazole, $C_3H_5N_3$, and 1-methyl-1*H*-tetrazole, $C_2H_4N_4$, are composed of layers of planar molecules with partially delocalized π systems. Differences in the bond lengths in these two closely related ring systems are ascribed to differing π-electron polarization effects, which are analysed with reference to *ab initio* calculations performed using a triple-zeta + polarization basis set at both the SCF and MP2 levels of theory.

Comment

The 1,2,4-triazole and tetrazole ring systems are typical planar 6π-electron partially aromatic systems. They possess an extensive chemistry (Temple, 1981; Benson, 1967) and we have in the past investigated the nature of the predominant tautomer of the parent molecules (1*H*-1,2,4-triazole and 2*H*-tetrazole) under gas-phase conditions by comparison of the UV–photoelectron spectra with those of the selectively prepared methyl derivatives (Palmer, Simpson & Wheeler, 1981). After prolonged storage at room temperature, two of the methylated compounds formed crystals suitable for structure determination by X-ray crystallography and their structures are reported here. Other than the parent compounds, which are heavily hydrogen bonded (Goldstein, Ladell & Abowitz, 1969; van der Putten, Heijdenrijk & Schenk, 1974), 4-methyl-1,2,4-triazole, (I), and 1-methyltetrazole, (II), are the simplest derivatives of their respective classes yet to be characterized structurally.



(I)



(II)

The crystal structures of both compounds (I) and (II) consist of planar molecules occupying crystallographic mirror planes (Figs. 1 and 2; see also Table 3). Compound (I) does not deviate significantly from C_{2v} symmetry, with N1—C5 and N2—C3 bond lengths markedly shorter [average 1.305 (3) Å] than either N4—C3/N4—C5 [average 1.347 (3) Å] or N1—N2 [1.395 (2) Å]. Similarly, in compound (II), which is related to (I) by the isovalent replacement of HC by :N, N2—N3 and N4—C5 are the shortest bonds in the system; this is consistent with the classical canonical form *A* (Fig. 3) being the dominant contributor to the bonding in both molecules. There are, however, differences between the lengths of related bonds in compounds (I) and (II) [*e.g.* N1—N2 is 1.395 (2) Å in (I), but N3—N4 is 1.360 (2) Å in (II)]. We have performed a series of *ab initio* calculations with the aim of explaining these effects.

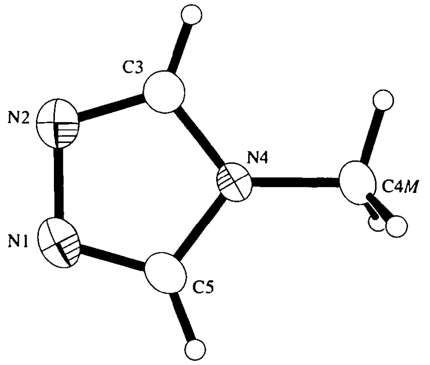


Fig. 1. The molecular structure of (I) with the atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces.

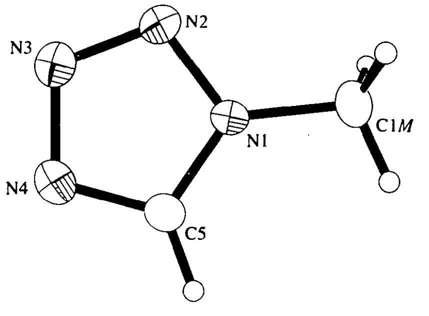
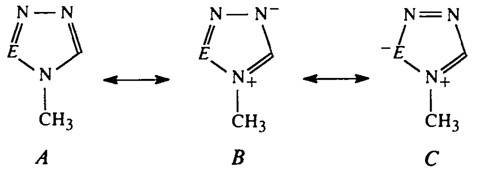


Fig. 2. The molecular structure of (II) with the atom-numbering scheme. Displacement ellipsoids enclose 50% probability surfaces.



(I) *E* = CH

(II) *E* = N

Fig. 3. Contributing valence-bond structures in (I) and (II).

The equilibrium structures and corresponding electron distributions were obtained by *ab initio* Hartree–Fock SCF and MP2 calculations using a large Gaussian orbital basis of triple-zeta valence + polarization quality (TZVP) (Dunning, 1971); we have shown that this basis set and the two methodologies give good agreement with various spectroscopic studies of the gas-phase structures of small molecules (Palmer, 1990; Palmer & Guest, 1992). A comparison of the experimentally determined structures with the corresponding theoretical (gas phase) equilibrium structures is shown in Table 3. The MP2 calculations reproduce all the bond lengths to within 0.02 Å and these are all longer than the corresponding SCF ones, a direct effect of the electron correlation introducing an element of antibonding character in each case. The calculated N1—N2 bond length in compound (I) is unique in being substantially too short by comparison with the crystallographic result and it is probable that the MP2 level of theory imperfectly corrects for correlation effects in this bond. In all other cases where significant differences arise, the calculated bond lengths are too long [by up to 0.02 Å for N1—C5 in (I)]. The bond angles are reproduced remarkably well. We note here that apparent disparities in simple comparisons of this type between X-ray and *ab initio* results might be as much due to packing or librational effects as to deficiencies in the theory.

The atomic populations (Mulliken analysis) for both molecules show that the total density is a rather insensitive tool for investigation, beyond showing that the total populations follow the electronegativity of the elements $N > C > H$; when this is dissected into σ and π components, and separated into bond contributions (Fig. 4), the effects are more selective. Since the individual σ and π components are not separable in the MP2 cases, the following analysis is based on the SCF data; the same effects are expected in the MP2 calculations, but all the bond dipoles are likely to be smaller. The results of these analyses quantitatively reproduce the σ–π donation properties that would be anticipated; the CH_3 groups, for example, in both compounds (I) and (II) are very weak π acceptors, but much stronger σ donors (0.22 e in each N—CH_3 bond). These calculations also provide a textbook example of the differing properties of N atoms in heterocycles contributing either one or two electrons to the cyclic π system. The substituted N atom [N4 in (I) and N1 in

(II)] is both a strong σ acceptor and a strong π donor, in a classic illustration of the push–pull effect. In contrast, the β-N atoms [N1 and N2 in (I), and N3 and N4 in (II)] are actually rather weak σ donors, but much more powerful π acceptors.

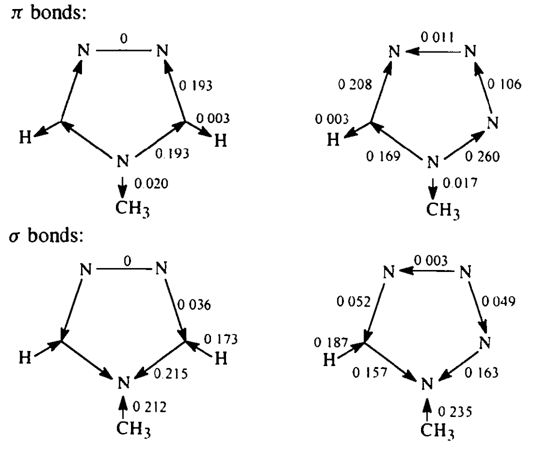


Fig. 4. Decomposition of the σ- and π-electron donation in (I) and (II).

While the σ-acceptor/π-donor properties of the substituted N atoms roughly balance in compounds (I) and (II), there is a powerful π-electron polarization towards the β-N atoms. This polarization is weakest in the N2—N3 bond of (II) (0.11 e compared with 0.20 e elsewhere) and the influence that this has on the bond lengths is conveniently examined with reference to valence-bond theory (Fig. 3). Both theory and experiment show that the classical non-polar form *A* is dominant in both (I) and (II). Form *B* most closely represents the π-electron polarization towards the β-N atoms, while a contribution from form *C* attenuates the π donation from *E* to β-N. It is this last form that is responsible for the weaker polarization in the N2—N3 bond in (II) and, since this introduces π character into the N3—N4 bond, explains the shorter length of this bond compared with the formally equivalent N1—N2 bond in (I).

The π polarization in these ring systems induces very high dipole moments on these molecules [calculated to be 6.53 and 6.27 D for (I) and (II), respectively, a trend consistent with the analysis presented above]. The revised experimental dipole moment for (II) is 5.8 D (Lumbroso, Liegois, Pappalardo & Grassi, 1982), about 8% lower than in the present study. While there are no data for (I), experimental dipole moments have been determined for both 1- and 2-ethyltetrazole as 5.46 and 2.65 D, respectively (Kaufman, Ernsberger & McEwan, 1956). We calculate values for these to be (SCF/MP2) 6.30/6.28 and 3.07/3.14 D, respectively. These are in reasonable agreement with the experimental values, giving us confidence in the high dipole moments calculated here for (I) and (II). For both 1-methyl- and 1-ethyltetrazole, the dipole moment is closely aligned with the N1—N3 vector.

These high dipole moments naturally influence the packing adopted by compounds (I) and (II) in the solid state. They both pack in layers perpendicular to the crystallographic *b* axis, with an interlayer spacing of *b*/2 [3.23 in (I) and 3.16 Å in (II)]. These values fall into the range associated with weak 'graphitic' interactions commonly observed between planar aromatic ring systems (the layer spacing in graphite itself is 3.35 Å). The molecules form an infinite two-dimensional network in (I); rows (*A* in Fig. 5) are formed along the **c** direction by simple lattice repetition, while a second row (*B*) is related to the first by a glide operation. This pattern is then repeated by lattice translation in **a** to form a *head-to-tail* herringbone arrangement of the type frequently observed in the crystal structures of highly polar but weakly hydrogen-bonding molecules. The in-layer packing in (II) can be analysed in a similar way, except that the *B* rows are related to the *A* rows by a glide perpendicular to that which relates the rows in (I) (Fig. 6). This leads to the disruption of the infinite two-

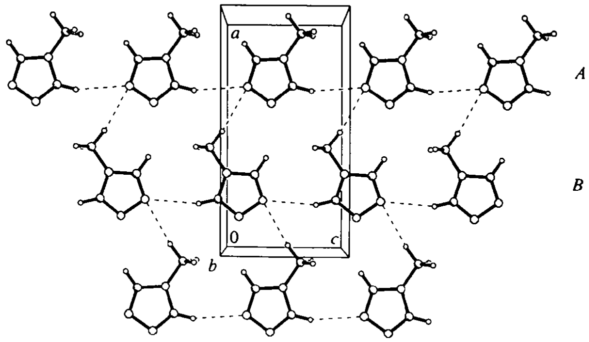


Fig. 5. Packing in the layers formed in the crystal structure of (I).

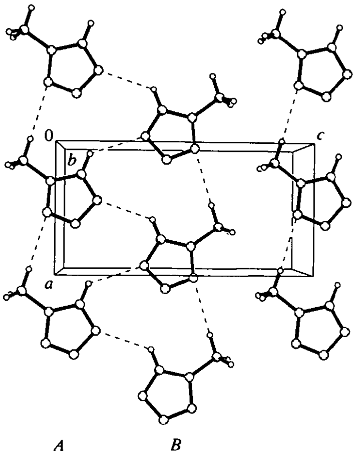


Fig. 6. Packing in the layers formed in the crystal structure of (II).

dimensional network observed in (I) and the formation of bimolecular ribbons of molecules in a *head-to-head* herringbone arrangement.

Experimental

The title compounds were originally synthesized from 1*H*-1,2,4-triazole and 2*H*-tetrazole. Under basic conditions, the corresponding anion is obtained by H-atom loss (Temple, 1981). In the case of the triazole, acetylation, methylation and deacetylation (Olofson & Kendall, 1970) gives compound (I) exclusively. In the case of the tetrazole, direct methylation of the anion yields a mixture, separable by distillation, of the 1-methyl and 2-methyl isomers. ^{13}C NMR [20 MHz, δ (p.p.m.)]: 142.1 ($C_{\rm ring}$), 29.4 (CH_3), for (I); 142.9 ($C_{\rm ring}$), 33.2 (CH_3), for (II). ^{15}N NMR [36.5 MHz, referenced to external $MeNO_2$, δ (p.p.m.)]: 63.1, 218.1 (NCH_3), for (I); −11.2, 10.7, 51.3, 152.3 (NCH_3), for (II). Crystals of compounds (I) and (II) formed on standing under ambient conditions for 15 years. The reproducibility of this method has not been confirmed.

Compound (I)

Crystal data

$C_3H_5N_3$
$M_r = 83.10$
Orthorhombic
Pnma
$a = 10.933\,(2)$ Å
$b = 6.4597\,(14)$ Å
$c = 5.6849\,(9)$ Å
$V = 401.49\,(13)$ Å^3
$Z = 4$
$D_x = 1.375$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 36 reflections
$\theta = 20$–$22°$
$\mu = 0.789$ mm^{-1}
$T = 150.0\,(2)$ K
Block
0.39 × 0.39 × 0.31 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer with Cryostream cooler (Cosier & Glazer, 1986)
ω–2θ scans
Absorption correction: none
833 measured reflections
326 independent reflections
315 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0508$
$\theta_{\rm max} = 59.91°$
$h = -12 \rightarrow 12$
$k = -7 \rightarrow 7$
$l = -6 \rightarrow 6$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0352$
$wR(F^2) = 0.0904$
$S = 1.124$
326 reflections
51 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0469P)^2 + 0.1142P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = 0.001$

$\Delta\rho_{\rm max} = 0.137$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.187$ e Å^{-3}
Extinction correction: *SHELXTL/PC* (Sheldrick, 1995)
Extinction coefficient: 0.021 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (I)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.6428 (2)	1/4	0.3876 (3)	0.0368 (6)
N2	0.71478 (15)	1/4	0.1850 (3)	0.0384 (6)
C3	0.8286 (2)	1/4	0.2567 (4)	0.0329 (6)
N4	0.83535 (12)	1/4	0.4929 (3)	0.0268 (6)
C4*M*	0.9454 (2)	1/4	0.6395 (3)	0.0335 (7)
C5	0.7179 (2)	1/4	0.5649 (4)	0.0330 (6)

Compound (II)

Crystal data

$C_2H_4N_4$
$M_r = 84.09$
Orthorhombic
Pnma
$a = 5.578\,(2)$ Å
$b = 6.324\,(2)$ Å
$c = 10.799\,(6)$ Å
$V = 380.9\,(3)$ Å^3
$Z = 4$
$D_x = 1.466$ Mg m^{-3}
D_m not measured

Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 32 reflections
$\theta = 20$–$22°$
$\mu = 0.918$ mm^{-1}
$T = 150.0\,(2)$ K
Needle
0.51 × 0.19 × 0.16 mm
Colourless

Data collection

Stoe Stadi-4 diffractometer with Cryostream cooler (Cosier & Glazer, 1986)
ω–2θ scans
Absorption correction: none
1774 measured reflections
314 independent reflections
303 observed reflections [$I > 2\sigma(I)$]

$R_{\rm int} = 0.0498$
$\theta_{\rm max} = 60.14°$
$h = -6 \rightarrow 6$
$k = -7 \rightarrow 7$
$l = -12 \rightarrow 12$
3 standard reflections
frequency: 60 min
intensity decay: none

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0353$
$wR(F^2) = 0.0825$
$S = 1.138$
314 reflections
48 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0494P)^2 + 0.0791P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.199$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.163$ e Å^{-3}
Extinction correction: *SHELXTL/PC* (Sheldrick, 1995)
Extinction coefficient: 0.018 (3)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

Table 2. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2) for (II)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.7935 (2)	1/4	0.53002 (11)	0.0222 (5)
C1*M*	0.6392 (4)	1/4	0.6387 (2)	0.0310 (6)
N2	1.0334 (3)	1/4	0.54185 (12)	0.0262 (5)
N3	1.1194 (2)	1/4	0.43009 (14)	0.0296 (5)
N4	0.9395 (3)	1/4	0.34509 (13)	0.0303 (6)
C5	0.7399 (3)	1/4	0.4099 (2)	0.0251 (5)

Table 3. *Comparison of observed (X-ray) and calculated molecular geometry parameters (Å, °) for (I) and (II)*

(I) was constrained in the molecular-orbital calculations to C_{2v} symmetry and (II) to C_s symmetry.

(I)	X-ray	SCF	MP2	(II)	X-ray	SCF	MP2
N4—C4*M*	1.463 (2)	1.4481	1.4537	N1—C1*M*	1.455 (2)	1.4480	1.4524
N4—C3	1.345 (3)	1.3537	1.3624	N1—N2	1.344 (2)	1.3234	1.3411
C3—N2	1.309 (3)	1.2802	1.3199	N2—N3	1.299 (2)	1.2512	1.3180
N2—N1	1.395 (2)	1.3605	1.3783	N3—N4	1.360 (2)	1.3356	1.3555
N1—C5	1.300 (3)	1.2802	1.3199	C5—N4	1.315 (2)	1.2898	1.3220
C5—N4	1.348 (2)	1.3537	1.3624	C5—N1	1.331 (2)	1.3273	1.3465
C4*M*—N4—C5	127.6 (2)	128.33	127.98	C1*M*—N1—C5	130.78 (15)	131.25	130.73
C4*M*—N4—C3	127.9 (2)	128.33	127.98	C1*M*—N1—N2	120.81 (13)	121.56	120.86
C3—N4—C5	104.5 (2)	103.34	104.03	N2—N1—C5	108.42 (14)	107.91	108.41
N2—C3—N4	111.3 (2)	110.99	111.04	N3—N2—N1	106.23 (12)	107.33	106.21
N1—N2—C3	106.2 (2)	107.34	106.94	N4—N3—N2	110.78 (14)	111.19	110.71
C5—N1—N2	106.5 (2)	107.34	106.94	C5—N4—N3	105.39 (14)	105.48	105.71
N4—C5—N1	111.5 (2)	110.99	111.04	N1—C5—N4	109.2 (2)	108.71	108.96

All quantum-mechanical calculations were performed with the *GAMESS-UK* package (Dupuis, Spangler & Wendoloski, 1980; Guest, Kendrick, van Lenthe, Schoeffel & Sherwood, 1995); the MP2 total energies for compounds (I) and (II) were −280.95197 and −296.95764 a.u., respectively (1 a.u. = 2626 kJ mol^{-1}).

For both compounds, data collection: *DIF*4 (Stoe & Cie, 1990*a*); cell refinement: *DIF*4; data reduction: *REDU*4 (Stoe & Cie, 1990*b*); program(s) used to solve structures: *SIR*92 (Altomare *et al.*, 1994); program(s) used to refine structures: *SHELXTL/PC* (Sheldrick, 1995); molecular graphics: *SHELXTL/PC*; software used to prepare material for publication: *SHELXTL/PC*.

The authors thank the EPSRC for funding.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: CF1129). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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17β-Isopropylsparteine and 17β-Isopropyllupanine Perchlorates

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Abstract

In the sparteine derivative, (1), $C_{18}H_{34}N_2^{2+}.2ClO_4^-$ {sparteine = [7*S*-(7α,7aα,14α,14aβ)]-dodecahydro-7,14-methano-2*H*,6*H*-dipyrido[1,2-*a*:1′,2′-*e*][1,5]diazocine}, the quinolizidine skeletons (*A*/*B* and *C*/*D* systems) have a *trans*/*trans* configuration with chair, chair, distorted boat and chair conformations for the *A*, *B*, *C* and