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Tetrakis(3,5-diphenyl-4-amino-4*H*-1,2,4-triazole-κN^1)zinc(II) bis(perchlorate)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.008 Å
R factor = 0.055
wR factor = 0.149
Data-to-parameter ratio = 13.2

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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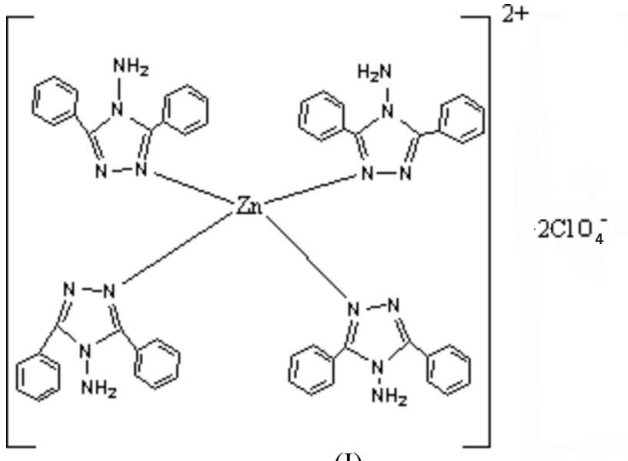
In the title compound, $[Zn(C_{14}H_{12}N_4)_4](ClO_4)_2$, the Zn atom is coordinated tetrahedrally by four monodentate 3,5-diphenyl-4-amino-4*H*-1,2,4-triazole ligands.

Comment

Substituted 1,2,4-triazoles have been actively studied because they combine the coordinating properties of pyrazoles and imidazoles, and frequently act as bridging ligands between metal centres. Their complexes have interesting structures and magnetic properties (Groeneveld *et al.*, 1982; Liu *et al.*, 2003; Haasnoot, 2000).



(I)

The title compound, $[Zn(L)_4](ClO_4)_2$, (I), (Fig. 1), contains the ligand *L* = 3,5-diphenyl-4-amino-1,2,4-triazole (Ikemi *et al.*, 2002). To the best of my knowledge, this is the first crystal structure containing this species coordinated to a metal cation.

Compound (I) contains one Zn^{II} ion, four crystallographically independent ligand molecules and two perchlorate anions (Fig. 1), which is consistent with the elemental analysis results. The Zn^{II} centre is four-coordinated by four N atoms of four ligand molecules. The Zn—N bond lengths in (I) (Table 1) are significantly shorter than those in the six-coordinate complex $[Zn(cltrz)_6](ClO_4)_2\cdot H_2O$ [cltrz = 4-(*p*-chlorophenyl)-1,2,4-triazole; Yi *et al.*, 2003], which range from 2.132 (4) to 2.221 (4) Å. The triazole rings of the ligand in (I) are planar, while the phenyl rings make dihedral angles of 23.9 (3)° (C1 phenyl ring), 41.5 (2)° (C9), 44.0 (2)° (C15), 28.0 (3)° (C23), 53.6 (3)° (C29), 40.3 (3)° (C37), 25.3 (3)° (C43) and 42.8 (3)° (C51), with respect to their bonded triazole ring mean planes (Fig. 1).

The packing of (I) is shown in Fig. 2. The perchlorate ions are surrouned by the *L* ligands. N—H···O hydrogen bonds (Table 2) help to stabilize the crystal packing. The fact that (I)

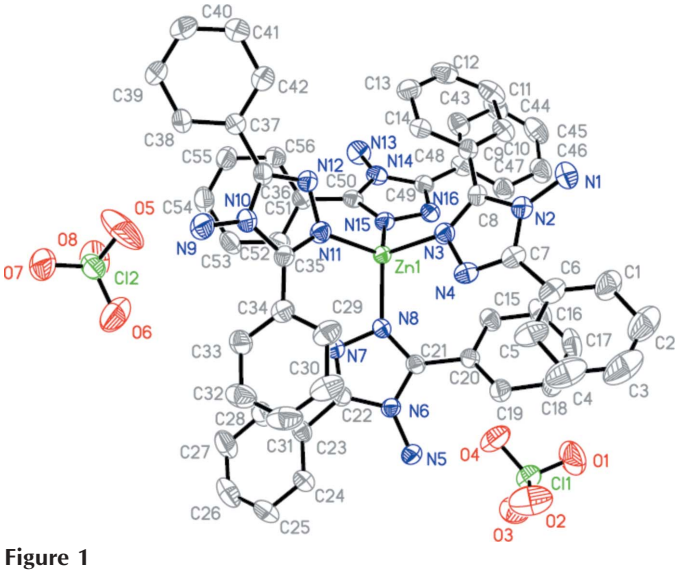


Figure 1
A view of (I), showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

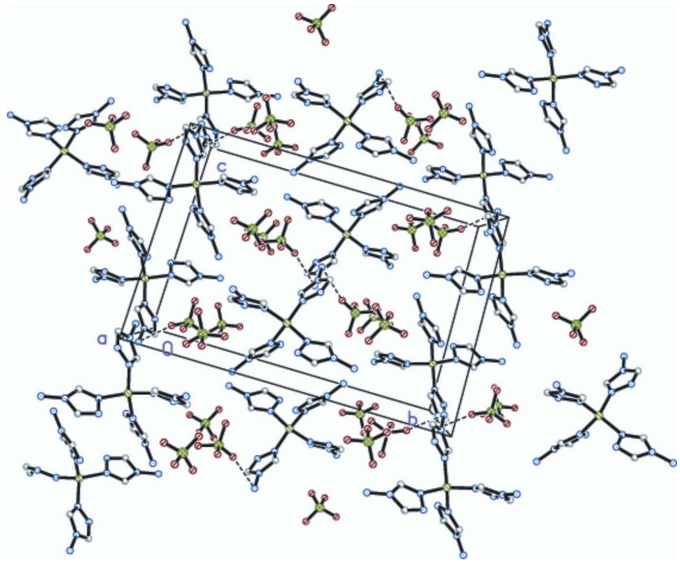


Figure 2
The packing of (I), viewed along the *a* axis. The phenyl rings and H atoms have been omitted for clarity.

does not form an extended polymeric structure may be due to the steric bulk of the phenyl groups of the ligands.

Experimental

Ligand *L* was prepared according to the previously reported method of Liljegren & Potts (2003). A methanol solution (15 ml) of *L* (0.7 mmol) was slowly added to an aqueous solution (10 ml) of $Zn(ClO_4)_2·6H_2O$ (0.7 mmol). The mixture was stirred for 30 min, refluxed for 3 h and then stirred for 20 h at room temperature, and the resulting precipitate was filtered off. After 20 d, colourless block crystals of (I) suitable for X-ray diffraction were obtained from the filtrate. Analysis, calculated for $C_{56}H_{48}Cl_2N_{16}O_8Zn$: C 55.62, H 4.00, N 18.53%; found: C 55.66, H 4.03, N 18.55%.

Crystal data

$[Zn(C_{14}H_{12}N_4)_4](ClO_4)_2$
M_r = 1209.37
Monoclinic, $P2_1/c$
a = 20.566 (5) Å
b = 20.174 (5) Å
c = 14.243 (3) Å
β = 108.614 (3)°
V = 5600 (2) Å^3
Z = 4
D_x = 1.434 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 1922 reflections
θ = 2.3–17.4°
μ = 0.61 mm^{-1}
T = 293 (2) K
Block, colourless
0.26 × 0.22 × 0.14 mm

Data collection

Bruker APEX-II CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.859, T_{max} = 0.920
29708 measured reflections
9901 independent reflections
5141 reflections with $I > 2\sigma(I)$
R_{int} = 0.091
θ_{max} = 25.0°
$h = -24 \rightarrow 24$
$k = -24 \rightarrow 12$
$l = -16 \rightarrow 16$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.149
S = 1.00
9901 reflections
748 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0302P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.28 e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.257 (15)

Table 1
Selected interatomic distances (Å).

Zn1—N11	2.004 (4)	Zn1—N3	2.005 (4)
Zn1—N8	2.004 (3)	Zn1—N15	2.018 (3)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···O1^{i}	0.90	2.36	3.153 (6)	148
N5—H5A···O2^{ii}	0.90	2.07	2.932 (6)	159
N5—H5B···O4	0.91	2.17	3.067 (6)	174
N9—H9A···O5	0.90	2.14	3.009 (8)	161
N13—H13A···O8^{iii}	0.90	2.43	3.132 (7)	136
N13—H13B···O7^{iv}	0.90	2.41	3.266 (7)	159

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $-x+1, -y+1, -z+1$; (iii) $-x, -y+1, -z+1$; (iv) $-x, y+\frac{1}{2}, -z+\frac{3}{2}$.

N-bound H atoms were located in difference Fourier maps, relocated in idealized positions (N—H = 0.90 and 0.91 Å), and refined as riding [U_{iso}(H) = 1.2U_{eq}(N)]. C-bound H atoms were placed in calculated positions (C—H = 0.93–0.96 Å) and refined as riding, with the constraint U_{iso}(H) = 1.2U_{eq}(carrier) applied.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT* (Bruker, 1998); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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References

Bruker (1998). *SMART*, *SAINT* and *SHELXTL*. Bruker AXS, Madison, Wisconsin, USA.

Groeneveld, L. R., Vos, G., Verschoor, G. C. & Reedijk, J. (1982). *J. Chem. Soc. Chem. Commun.* pp. 620–624.
Haasnoot, J. G. (2000). *Coord. Chem. Rev.* **200–202**, 131–185.
Ikemi, Y., Hayashi, N., Kakehi, A. & Matsumoto, K (2002). *Heterocycl. Commun.* **8**, 439–442.
Liljegren, D. R. & Potts, K. T. (2003). *J. Chem. Soc.* pp. 518–522.
Liu, J. C., Guo, G. C., Huang, J. S. & You, X. Z. (2003). *Inorg. Chem.* **42**, 235–243.
Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Yi, L., Zhu, L. N., Ding, B., Cheng, P., Liao, D. Z., Yan, S. P. & Jiang, Z. H. (2003). *Inorg. Chem. Commun.* **6**, 1209–1212.