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1,2,7,8-Tetramethyl-4,5-dihydro-3a,5a-diazapyrene ditriflate

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Key indicators

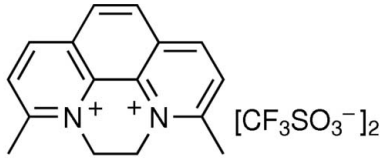
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.003 Å
R factor = 0.047
wR factor = 0.131
Data-to-parameter ratio = 16.6

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

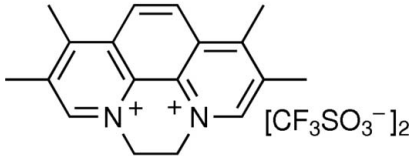
The title structure, $C_{18}H_{20}N_2^{2+}\cdot 2CF_3O_3S^-$, is the first to be reported for a diquaternized derivative of 3,4,7,8-tetramethyl-1,10-phenanthroline.

Comment

Relevant background information on this work and comments on the title structure, (II), together with that of the closely related salt 3,6-dimethyl-4,5-dihydro-3a,5a-diazapyrene ditriflate, (I), can be found in the preceding paper (Coe, Fitzgerald & Raftery, 2006). The molecular structure of (II) is shown in Fig. 1 and selected geometric parameters are given in Table 1.



(I)



(II)

Experimental

Salt (II) was synthesized as reported previously (Coe, Curati & Fitzgerald, 2006). Crystals suitable for single-crystal X-ray diffraction were obtained by slow diffusion of diethyl ether vapour into an acetone solution of (II) at 295 K.

Crystal data

$C_{18}H_{20}N_2^{2+}\cdot 2CF_3O_3S^-$
M_r = 562.50
Monoclinic, $P2_1/c$
a = 12.882 (1) Å
b = 8.152 (1) Å
c = 22.585 (1) Å
β = 104.284 (1)°
V = 2298.4 (2) Å^3
Z = 4
D_x = 1.626 Mg m^{-3}
Mo $K\alpha$ radiation
μ = 0.32 mm^{-1}
T = 100 (2) K
Block, white
0.45 × 0.30 × 0.20 mm

Data collection

Bruker SMART APEX CCD diffractometer
φ and ω scans
Absorption correction: none
19382 measured reflections
5467 independent reflections
4426 reflections with $I > 2\sigma(I)$
R_{int} = 0.058
θ_{max} = 28.3°

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 1.2422P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.047$	$(\Delta/\sigma)_{max} < 0.001$
$wR(F^2) = 0.131$	$\Delta\rho_{max} = 0.81$ e Å^{-3}
$S = 1.08$	$\Delta\rho_{min} = -0.67$ e Å^{-3}
5467 reflections	
329 parameters	
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—N1	1.364 (2)	C7—C8	1.355 (3)
C1—C9	1.404 (3)	C8—C9	1.434 (3)
C1—C2	1.433 (3)	C9—C10	1.427 (3)
C2—N2	1.369 (2)	C10—C11	1.388 (3)
C2—C6	1.399 (3)	C10—C17	1.501 (3)
C3—N2	1.330 (2)	C11—C12	1.395 (3)
C3—C4	1.393 (3)	C11—C18	1.503 (3)
C4—C5	1.385 (3)	C12—N1	1.329 (2)
C4—C15	1.505 (3)	C13—N1	1.483 (2)
C5—C6	1.430 (3)	C13—C14	1.504 (3)
C5—C16	1.501 (3)	C14—N2	1.479 (2)
C6—C7	1.435 (3)		
N1—C1—C2	119.87 (17)	N2—C14—C13	108.35 (15)
N2—C2—C1	119.65 (17)	C1—N1—C13	118.55 (15)
N1—C13—C14	108.40 (15)	C2—N2—C14	117.71 (15)
N1—C13—C14—N2	−58.83 (19)		

All H atoms were included in calculated positions, with C—H = 0.95 (CH), 0.99 (CH_2) and 0.98 Å (CH_3); U_{iso}(H) values were fixed at $1.2U_{eq}$(C) or $1.5U_{eq}$(methyl C).

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SIR2004* (Burla *et al.*, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

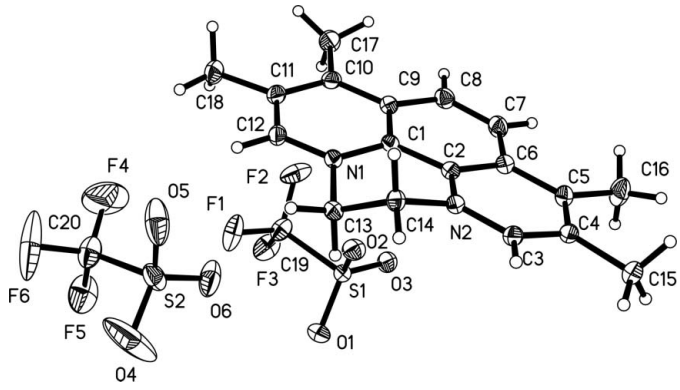


Figure 1
The asymmetric unit of (II), showing 50% probability displacement ellipsoids.

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