

Benjamin J. Coe,* Emma C. Fitzgerald and James Raftery

 School of Chemistry, University of Manchester,
 Manchester M13 9PL, England

 Correspondence e-mail:
 b.coe@manchester.ac.uk

Key indicators

 Single-crystal X-ray study
 $T = 100$ K
 Mean $\sigma(\text{C}-\text{C}) = 0.003$ 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>.

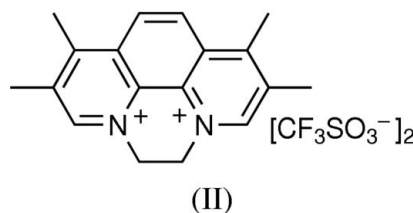
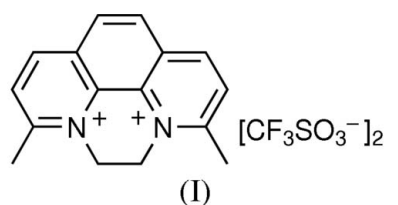
1,2,7,8-Tetramethyl-4,5-dihydro-3a,5a-diazapyrene ditriflate

 The title structure, $\text{C}_{18}\text{H}_{20}\text{N}_2^{2+} \cdot 2\text{CF}_3\text{O}_3\text{S}^-$, is the first to be reported for a diquaternized derivative of 3,4,7,8-tetramethyl-1,10-phenanthroline.

 Received 23 August 2006
 Accepted 1 September 2006

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.


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
 $\text{C}_{18}\text{H}_{20}\text{N}_2^{2+} \cdot 2\text{CF}_3\text{O}_3\text{S}^-$
 $M_r = 562.50$
 Monoclinic, $P2_1/c$
 $a = 12.882$ (1) Å
 $b = 8.152$ (1) Å
 $c = 22.585$ (1) Å
 $\beta = 104.284$ (1)°
 $V = 2298.4$ (2) Å³
 $Z = 4$
 $D_x = 1.626$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.32$ 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 $I > 2\sigma(I)$
 $R_{\text{int}} = 0.058$
 $\theta_{\text{max}} = 28.3^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.131$
 $S = 1.08$
 5467 reflections
 329 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0667P)^2 + 1.2422P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.81 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.67 \text{ e } \text{Å}^{-3}$

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 (CH₂) and 0.98 Å (CH₃); $U_{\text{iso}}(\text{H})$ values were fixed at $1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

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:

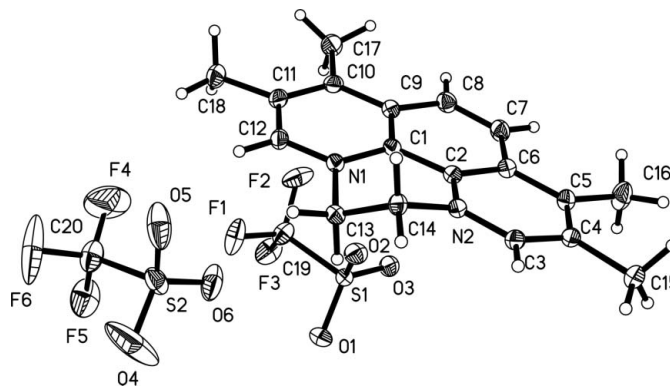


Figure 1

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

SHELXTL (Bruker, 2000); software used to prepare material for publication: *SHELXTL*.

The authors thank the EPSRC for funding (grant GR/R81459 and a PhD studentship).

References

Bruker (2000). *SHELXTL*. Version 6.10. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2001). *SMART*. Version 5.625. Bruker AXS Inc., Madison, Wisconsin, USA.
 Bruker (2003). *SAINT*. Version 6.45. Bruker AXS Inc., Madison, Wisconsin, USA.
 Burla, M. C., Caliendo, R., Camalli, M., Carrozzini, B., Cascarano, G. L., De Caro, L., Giacovazzo, C., Polidori, G. & Spagna, R. (2005). *J. Appl. Cryst.* **38**, 381–388.
 Coe, B. J., Curati, N. R. M. & Fitzgerald, E. C. (2006). *Synthesis*, pp. 146–150.
 Coe, B. J., Fitzgerald, E. C. & Raftery, J. (2006). *Acta Cryst. E* **62**, o4333–o4334.
 Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.