Sheldrick, G. M. (1985). *SHELXS86. Crystallographic Computing* 3, edited by G. M. Sheldrick, C. Krüger & R. Goddard, pp. 175–189. Oxford University Press.
Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.
Sundaralingam, M. & Jensen, L. H. (1965). *Acta Cryst.* **18**, 1053–1058.
Zachariasen, W. H. (1963). *Acta Cryst.* **16**, 1139–1144.
Zachariasen, W. H. (1968). *Acta Cryst.* A**24**, 212–216.

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9,10-Bis(1-pyridiniomethyl)anthracene Dichloride–Methanol–Water (1/2/1)

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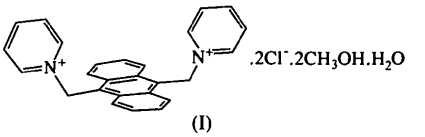
Abstract

The title compound, 9,10-bis(1-pyridiniomethyl)anthracene dichloride, crystallizes as the methanol/water solvate $C_{26}H_{22}N_2^{2+}.2Cl^-.2CH_3OH.H_2O$, with a one-dimensional network of hydrogen bonds. Two formula units make up the asymmetric unit. The pyridine rings are on the same side of the anthracene plane.

Comment

The structure of the title compound, (I), was determined as part of our continuing group interest in molecular-recognition phenomena (Moyer & Bonnesen, 1997) and as part of a growing interest in anion recognition (Seel, Galán & de Mendoza, 1995). The title compound represents a new topography for a large diffuse cation, one that may exhibit anion selectivity.



(I)

The asymmetric unit for the title compound contains two formula units and is shown in Fig. 1. All bond lengths and angles are in good agreement with standard values (Allen *et al.*, 1987). For example, the aromatic C—C bonds vary from 1.354 (3) to 1.440 (3) Å, with an average of 1.396 (3) Å. The anthracene units stack roughly along the *c* axis, with an interplanar distance of approximately 3.40 Å. One-dimensional chains are formed along the *a* axis by hydrogen bonding between two of the chloride ions (Cl1 and Cl2) and the two water solvate molecules (O5 and O6). Two of the methanol solvate molecules (containing O1 and O2) hydrogen bond to this chain at Cl1 and O6. The remaining two methanol solvate molecules (O3 and O4) hydrogen bond to Cl3 and Cl4, respectively.

The pyridyl groups are located on the same side of the anthracene ring, forming an electropositive cavity. Two methanol molecules fit head-to-tail into these cavities, with the oxygen lone pairs pointing into the

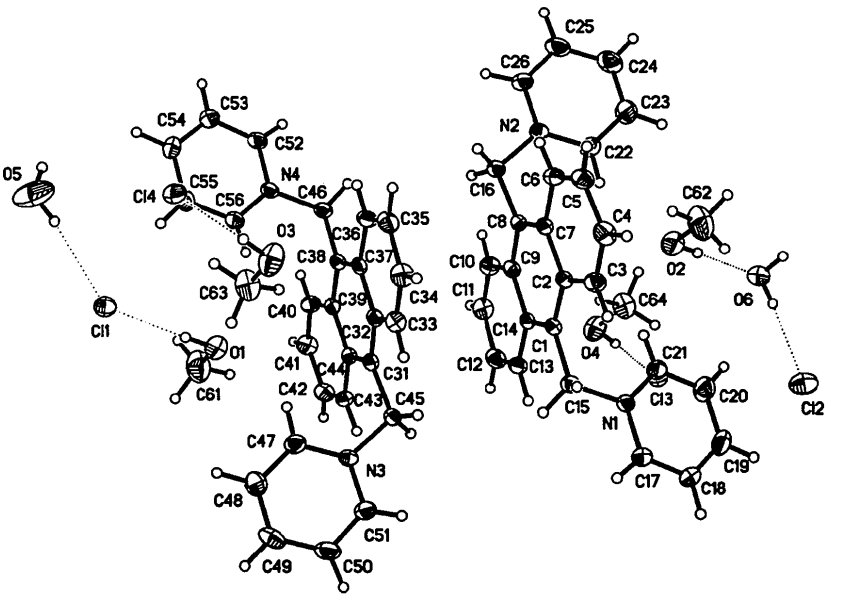


Fig. 1. The contents of the asymmetric unit shown with 50% probability displacement ellipsoids. H atoms are represented as open circles for clarity.

cavity. The O atom of one of the methanol molecules makes close contacts with H atoms on the pyridyl C atoms [H21···O2 2.44 (2), H22···O2 2.39 (3), H47···O1 2.35 (2) and H56···O1 2.52 (2) Å].

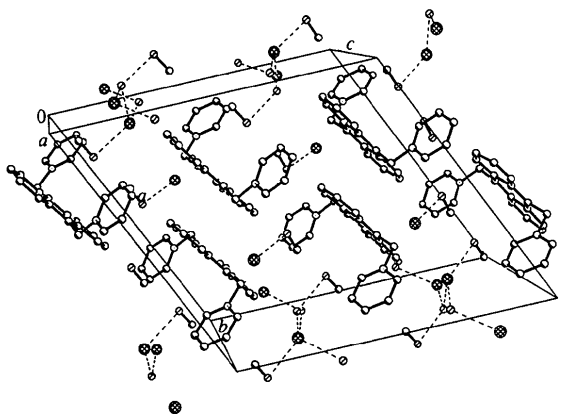


Fig. 2. A packing diagram for (I) viewed down the *a* axis. H atoms have been omitted for clarity.

Experimental

The title compound was prepared by heating 9,10-bis(chloromethyl)anthracene (1 part) with a large excess of pyridine (10 parts) at 363 K for 20 h. The resulting precipitate was filtered, washed with diethyl ether and recrystallized from MeOH/H_2O solution. X-ray quality crystals of (I) were prepared by slow evaporation from a wet methanol/2-propanol solution.

Crystal data

$C_{26}H_{22}N_2^{2+}.2Cl^-.2CH_4O.H_2O$
M_r = 515.46
Triclinic
$P\bar{1}$
a = 10.659 (5) Å
b = 15.827 (6) Å
c = 17.315 (3) Å
α = 65.22 (2)°
β = 81.60 (2)°
γ = 81.69 (3)°
V = 2612.3 (18) Å^3
Z = 4
D_x = 1.31 Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
λ = 0.71073 Å
Cell parameters from 25 reflections
θ = 10.3–13.5°
μ = 0.28 mm^{-1}
T = 163 (2) K
Block
0.31 × 0.27 × 0.21 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω scans
Absorption correction: empirical *via* ψ scans (Siemens, 1995)
T_{min} = 0.859, T_{max} = 0.943
9865 measured reflections
9182 independent reflections

6252 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.024
θ_{max} = 25°
$h = -6 \rightarrow 12$
$k = -18 \rightarrow 18$
$l = -20 \rightarrow 20$
3 standard reflections
frequency: 120 min
intensity decay: 5.7%

Refinement

Refinement on F^2
$R(F)$ = 0.038
$wR(F^2)$ = 0.101
S = 1.07
9182 reflections
887 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0632P)^2 + 0.5942P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.001$
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}
Extinction correction: none
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)*

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

	x	y	z	U_{eq}
Cl1	0.37372 (5)	0.07897 (4)	0.22591 (3)	0.02742 (14)
Cl2	0.11369 (5)	0.04533 (5)	0.77111 (4)	0.0376 (2)
O1	0.5477 (2)	0.16037 (12)	0.05305 (11)	0.0406 (4)
O2	0.4553 (2)	0.83533 (12)	0.44525 (12)	0.0389 (4)
C61	0.6211 (4)	0.0802 (2)	0.0491 (2)	0.0541 (8)
C62	0.3838 (4)	0.9136 (2)	0.4550 (2)	0.0531 (8)
Cl3	0.04399 (5)	0.37986 (4)	0.72629 (3)	0.02593 (14)
Cl4	0.05055 (5)	0.37836 (4)	0.22540 (3)	0.02818 (14)
O3	0.2588 (2)	0.41875 (12)	0.07697 (11)	0.0411 (5)
O4	0.7460 (2)	0.58756 (12)	0.42108 (11)	0.0356 (4)
C63	0.3360 (3)	0.3371 (2)	0.0792 (2)	0.0405 (6)
C64	0.6674 (3)	0.6696 (2)	0.4157 (2)	0.0372 (6)
O5	0.0881 (2)	0.0276 (2)	0.3015 (2)	0.0616 (7)
O6	0.5992 (2)	0.91553 (13)	0.28436 (11)	0.0333 (4)
N1	0.7437 (2)	0.80157 (11)	0.60966 (11)	0.0189 (4)
N2	0.1763 (2)	0.63446 (11)	0.56296 (10)	0.0189 (4)
N3	0.8151 (2)	0.36610 (11)	0.93424 (10)	0.0182 (4)
N4	0.2440 (2)	0.19615 (11)	0.89573 (10)	0.0192 (4)
C1	0.5586 (2)	0.70385 (14)	0.67409 (12)	0.0180 (4)
C2	0.4354 (2)	0.74994 (14)	0.67692 (12)	0.0177 (4)
C3	0.4117 (2)	0.83497 (15)	0.68968 (13)	0.0227 (5)
C4	0.2917 (2)	0.8763 (2)	0.69443 (14)	0.0257 (5)
C5	0.1864 (2)	0.8361 (2)	0.68753 (14)	0.0238 (5)
C6	0.2046 (2)	0.75636 (15)	0.67379 (13)	0.0208 (4)
C7	0.3294 (2)	0.71057 (13)	0.66713 (12)	0.0166 (4)
C8	0.3485 (2)	0.62934 (13)	0.65109 (12)	0.0165 (4)
C9	0.4725 (2)	0.58636 (14)	0.64333 (12)	0.0178 (4)
C10	0.4977 (2)	0.50640 (15)	0.62201 (14)	0.0234 (5)
C11	0.6182 (2)	0.4651 (2)	0.61743 (14)	0.0265 (5)
C12	0.7220 (2)	0.4988 (2)	0.63422 (14)	0.0254 (5)
C13	0.7032 (2)	0.57538 (15)	0.65285 (13)	0.0214 (4)
C14	0.5788 (2)	0.62283 (13)	0.65747 (11)	0.0167 (4)
C15	0.6687 (2)	0.74160 (15)	0.68999 (13)	0.0194 (4)
C16	0.2346 (2)	0.58591 (15)	0.64670 (13)	0.0196 (4)
C17	0.8500 (2)	0.8322 (2)	0.6187 (2)	0.0288 (5)
C18	0.9241 (2)	0.8848 (2)	0.5484 (2)	0.0376 (6)
C19	0.8910 (2)	0.9069 (2)	0.4674 (2)	0.0354 (6)
C20	0.7807 (3)	0.8768 (2)	0.4598 (2)	0.0358 (6)
C21	0.7080 (2)	0.8243 (2)	0.53155 (14)	0.0265 (5)
C22	0.2307 (2)	0.7020 (2)	0.49521 (14)	0.0256 (5)
C23	0.1738 (2)	0.7432 (2)	0.4200 (2)	0.0366 (6)
C24	0.0615 (2)	0.7138 (2)	0.4143 (2)	0.0372 (6)
C25	0.0071 (2)	0.6440 (2)	0.4849 (2)	0.0338 (6)
C26	0.0650 (2)	0.6053 (2)	0.5594 (2)	0.0271 (5)
C31	0.6423 (2)	0.36831 (13)	0.84708 (12)	0.0173 (4)
C32	0.5186 (2)	0.41055 (13)	0.85511 (12)	0.0175 (4)
C33	0.4930 (2)	0.49180 (15)	0.87426 (13)	0.0224 (5)
C34	0.3725 (2)	0.5331 (2)	0.87784 (14)	0.0265 (5)
C35	0.2690 (2)	0.4983 (2)	0.86237 (14)	0.0261 (5)
C36	0.2877 (2)	0.42037 (15)	0.84608 (13)	0.0221 (5)
C37	0.4125 (2)	0.37245 (13)	0.84226 (12)	0.0174 (4)
C38	0.4325 (2)	0.29003 (14)	0.82775 (12)	0.0183 (4)
C39	0.5566 (2)	0.24526 (14)	0.82418 (12)	0.0178 (4)
C40	0.5821 (2)	0.1600 (2)	0.81176 (14)	0.0243 (5)
C41	0.7022 (2)	0.1196 (2)	0.80655 (14)	0.0267 (5)

C42	0.8071 (2)	0.16091 (15)	0.81263 (14)	0.0238 (5)
C43	0.7877 (2)	0.24113 (15)	0.82536 (13)	0.0208 (4)
C44	0.6626 (2)	0.28606 (13)	0.83242 (12)	0.0170 (4)
C45	0.7560 (2)	0.41272 (14)	0.85072 (13)	0.0185 (4)
C46	0.3216 (2)	0.2510 (2)	0.81408 (13)	0.0202 (4)
C47	0.7606 (2)	0.2994 (2)	1.00306 (14)	0.0264 (5)
C48	0.8165 (2)	0.2615 (2)	1.07887 (15)	0.0352 (6)
C49	0.9276 (2)	0.2936 (2)	1.0837 (2)	0.0348 (6)
C50	0.9824 (2)	0.3611 (2)	1.0118 (2)	0.0331 (6)
C51	0.9257 (2)	0.3967 (2)	0.9371 (2)	0.0262 (5)
C52	0.1325 (2)	0.1711 (2)	0.88846 (15)	0.0259 (5)
C53	0.0562 (2)	0.1219 (2)	0.9596 (2)	0.0326 (6)
C54	0.0934 (2)	0.0977 (2)	1.03984 (15)	0.0316 (5)
C55	0.2088 (2)	0.1233 (2)	1.0460 (2)	0.0339 (6)
C56	0.2828 (2)	0.1724 (2)	0.97339 (14)	0.0269 (5)

Table 2. *Selected geometric parameters (Å)*

N1—C21	1.344 (3)	N3—C47	1.339 (3)
N1—C17	1.349 (3)	N3—C51	1.353 (3)
N1—C15	1.504 (3)	N3—C45	1.506 (3)
N2—C22	1.336 (3)	N4—C52	1.344 (3)
N2—C26	1.352 (3)	N4—C56	1.347 (3)
N2—C16	1.507 (3)	N4—C46	1.508 (3)

Table 3. *Hydrogen-bonding geometry (Å, °)*

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1···Cl1	0.92 (3)	2.23 (3)	3.145 (2)	173 (3)
O2—H2···O6	0.85 (3)	1.99 (3)	2.842 (3)	173 (3)
O3—H3A···Cl4	0.89 (3)	2.17 (3)	3.051 (2)	172 (3)
O4—H4A···Cl3^{i}	0.83 (2)	2.24 (2)	3.058 (2)	170 (3)
O5—H5A···Cl1	0.84 (4)	2.39 (4)	3.210 (3)	168 (3)
O5—H5B···Cl2^{ii}	0.87 (3)	2.38 (3)	3.204 (3)	159 (3)
O6—H6A···Cl2^{i}	0.83 (3)	2.32 (3)	3.140 (3)	174 (3)
O6—H6B···Cl1^{iii}	0.85 (3)	2.36 (3)	3.195 (3)	167 (3)

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

All H atoms were located in the difference maps and were refined isotropically. The C—H distances ranged from 0.87 (2) to 1.03 (3) Å and the O—H distances ranged from 0.83 (2) to 0.92 (3) Å. H-atom U_{iso} values ranged from 0.012 (5) to 0.124 (19) Å^2. Anisotropic displacement parameters were used for all non-H atoms.

Data collection and cell refinement were performed using *CAD-4/PC* (Enraf–Nonius, 1993) and data reduction was carried out with *XCAD*4 (Harms, 1995). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990) and refined using *SHELXL*93 (Sheldrick, 1993). *SHELXTL* (Siemens, 1995) was used for producing the molecular graphics and *PLATON* (Spek, 1990) was used for both the preparation of the CIF and the geometric analysis.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1271). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans.* 2, pp. S1–S19.

Enraf–Nonius (1993). *CAD-4/PC*. Version 1.2. Enraf–Nonius, Delft, The Netherlands.

Harms, K. (1995). *XCAD*4. *Program for the Lp Correction of Enraf–Nonius Four-Circle Diffractometer Data*. University of Marburg, Germany.

Moyer, B. A. & Bonnesen, P. V. (1997). *Physical Factors in Anion Separations*. In *The Supramolecular Chemistry of Anions*, edited by A. Bianchi, K. Bowman-James & E. Garcia-España. Weinheim, Germany: VCH.

Seel, C., Galán, A. & de Mendoza, J. (1995). *Top. Curr. Chem.* **175**, 102–132.

Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.

Sheldrick, G. M. (1993). *SHELXL*93. *Program for the Refinement of Crystal Structures*. University of Göttingen, Germany.

Siemens (1995). *SHELXTL*. Version 5.03/IRIX. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Acta Cryst. (1996). C**52**, 3093–3095

9,10-Bis(7-fluoro-2,5-dioxaheptyl)triptycene

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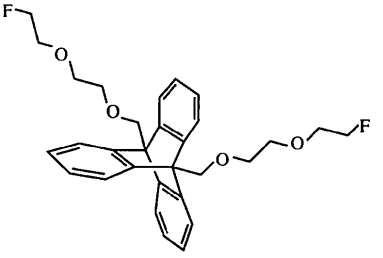
Abstract

The structure of the title compound, $C_{30}H_{32}F_2O_4$, exhibits C—H···π-arene hydrogen bonding.

Comment

In the course of our work on crown ether molecules incorporating the triptycene group (Gakh, Sachleben, Bryan & Moyer, 1995), we isolated the title compound. The interplanar angles between the arene rings (C3–C8 = *A*1, C9–C14 = *A*2 and C15–C20 = *A*3) on the triptycene are *A*1^*A*2 = 116.25 (7), *A*1^*A*3 = 121.74 (8) and *A*2^*A*3 = 121.68 (8)°.



Hydrogen-bond interactions involve the triptycene arene rings (Bakshi *et al.*, 1994; Gakh *et al.*, 1995). The space between rings *A*1 and *A*2 is occupied by