C(2)—C(3), C(6)—O(6)—C(1)—C(2)] fall within this category, the torsional-angle differences being the most pronounced.

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Structures of Two Conducting Salts of Pyrazinoethylenedithiotetrathiafulvalene (PEDTTTF): $(PEDTTTF)_2PF_6$ and $(PEDTTTF)_2BF_4.(CH_2Cl_2)_{0.5}$

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Abstract. (I): Bis{2-(dithiolo[4,5-*b*]dithiin-2-ylidene)-dithiolo[4,5-*b*]pyrazinium} hexafluorophosphate, $(C_{10}H_6N_2S_6)_2PF_6$, $M_r = 838{\cdot}07$, triclinic, $P\bar{1}$, $a = 5{\cdot}573\,(2)$, $b = 8{\cdot}173\,(1)$, $c = 16{\cdot}332\,(2)$ Å, $\alpha = 94{\cdot}70\,(1)$, $\beta = 89{\cdot}39\,(1)$, $\gamma = 75{\cdot}74\,(1)°$, $V = 718{\cdot}2\,(2)$ Å^3, $Z = 1$, $D_m = 1{\cdot}97$, $D_x = 1{\cdot}938$ Mg m^{-3}, Cu $K\bar{\alpha}$ ($\lambda = 1{\cdot}54178$ Å), $\mu = 9{\cdot}292$ mm^{-1}, $F(000) = 421{\cdot}0$, $T = 296\,(2)$ K. (II): Bis{2-(dithiolo[4,5-*b*]-dithiin-2-ylidene)dithiolo[4,5-*b*]pyrazinium} tetrafluoroborate dichloromethane solvate, $(C_{10}H_6N_2S_6)_2BF_4.(CH_2Cl_2)_{1/2}$. $M_r = 822{\cdot}38$, monoclinic, $C2/c$, $a = 34{\cdot}655\,(7)$, $b = 12{\cdot}122\,(3)$, $c = 14{\cdot}212\,(1)$ Å, $\beta = 97{\cdot}13\,(1)°$, $V = 5925\,(2)$ Å^3, $Z = 8$, $D_m = 1{\cdot}75$, $D_x = 1{\cdot}843$ Mg m^{-3}, Cu $K\bar{\alpha}$ ($\lambda = 1{\cdot}54178$ Å), $\mu = 9{\cdot}211$ mm^{-1}, $F(000) = 3312{\cdot}00$, $T = 296\,(2)$ K. Final R values 0·0393 (I) and 0·0396 (II) for 2626 (all reflections) (I) and 3570 observed reflections with $F_o \geq 5{\cdot}0\sigma(F_o)$ (II). The salts show metallic behavior down to 280 K (I) and 180 K (II). Below these temperatures they undergo metal-to-semiconductor (I) and metal-to-insulator (II) transition. Cations in both structures pack side by side, in a planar arrangement, through strong S···S and weak S···N contacts, the shortest of which are S···S = 3·456, S···N = 3·336 Å (I) and S···S = 3·467, S···N = 3·344 Å (II).

Introduction. Following a systematic study of the crystal structures of $(PEDTTTF)_2X$ salts (Terzis, Hountas & Papavassiliou, 1986; Psycharis, Hountas, Terzis & Papavassiliou, 1988; Terzis, Psycharis, Hountas & Papavassiliou, 1988) we present here two more salts, where $X = PF_6$ (I) and BF_4 (II). PEDTTTF (Fig. 1) is a modification of ET, and it

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was synthesized in the hope that some of its salts would afford the superconducting state. Some of the salts of ET [$(ET)_2X$ where $X = I_3$, IBr_2^-, AuI_2^-] are superconducting at very low temperatures (Ishiguro, 1988; Williams *et al.*, 1987) but none of the PEDTTTF salts studied so far afforded the superconducting state. The substitution of the two S atoms in one of the six-membered rings of ET by N atoms has caused a drastically different packing of this donor compared with the $(ET)_2X$ salts.

Experimental. Title compounds were prepared by electrocrystallization of the donor (Papavassiliou, Yiannopoulos & Zambounis, 1987) ($1\cdot55 \times 10^{-3}$ *M*) and Bu_4NPF_6 (I) or Bu_4NBF_4 (II) (5×10^{-3} *M*) in CH_2Cl_2, using platinum electrodes at 1 μA cm^{-2} and 295 K. Crystal dimensions $0\cdot1 \times 0\cdot5 \times 0\cdot3$ (I) and $0\cdot5 \times 0\cdot3 \times 0\cdot04$ (II) mm. Density measured by flotation. Syntex $P2_1$ diffractometer. θ–2θ scan, $2\theta < 136\cdot0°$ (I) and 120° (II), scan speed variable between 2·0 and 15·0° min^{-1}, scan range 2·2° (I) and 1·8° (II) (2θ) plus $\alpha_1 - \alpha_2$ separation, background counting 0·5 of scan time. Cell parameters from 15 independent reflections with $50 < 2\theta < 53°$. Data collected/unique/R_{int} (F), 4842/2636/0·0256 (I) and 4740/4407/0·0158 (II). Range of *hkl*: $0 \rightarrow 5$, $-9 \rightarrow 9$, $-19 \rightarrow 19$ (I) and $0 \rightarrow 38$, $0 \rightarrow 13$, $-15 \rightarrow 15$ (II). Three reflections monitored periodically showed $< 3\cdot0\%$ intensity fluctuation. Lp and numerical absorption correction applied, T_{max}/T_{min}: 0·4591/0·0866 (I) and

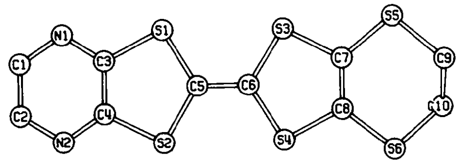

Fig. 1. Atom numbering for the PEDTTTF molecule. Substitution of the N atoms by S atoms gives ET.

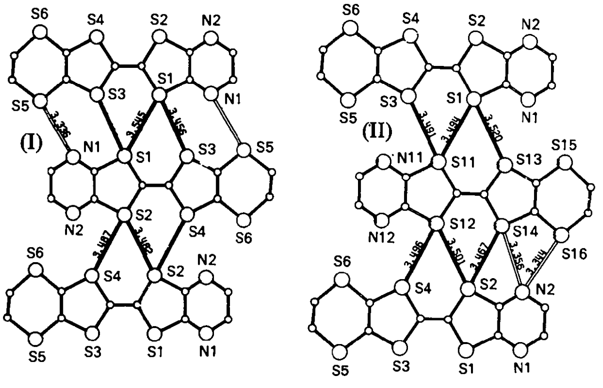

Fig. 2. Planar arrangement of donors with S···S and S···N contacts (Å).

Table 1. *Positional and equivalent isotropic thermal parameters* ($Å^2 \times 10^4$) *of the non*-H *atoms with e.s.d.'s in parentheses*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}
(I)				
C(1)	5134 (6)	5224 (4)	7633 (2)	390
C(2)	7510 (6)	4198 (4)	7599 (2)	407
N(1)	3584 (5)	5233 (3)	7010 (2)	349
N(2)	8421 (5)	3095 (3)	6946 (2)	364
C(3)	4486 (5)	4155 (4)	6372 (2)	300
C(4)	6865 (5)	3095 (4)	6343 (2)	296
S(1)	2693 (1)	3999 (1)	5507·6 (4)	318
S(2)	7752 (1)	1720·9 (9)	5450·5 (4)	313
C(5)	4981 (5)	2450 (3)	4956 (2)	285
C(6)	4614 (5)	1840 (3)	4177 (2)	289
S(3)	1879 (1)	2566 (1)	3674·9 (4)	319
S(4)	6855 (1)	289 (1)	3611·6 (5)	336
C(7)	2825 (5)	1286 (4)	2759 (2)	315
C(8)	5090 (5)	212 (4)	2739 (2)	317
S(5)	673 (2)	1619 (1)	1991·1 (5)	455
S(6)	6515 (2)	−82 (1)	1949·2 (5)	416
C(9*A*)	2570 (13)	597 (10)	1091 (6)	514
C(9*B*)	2101 (10)	−65 (8)	1211 (4)	484
C(10*A*)	3927 (12)	−1207 (9)	1248 (5)	455
C(10*B*)	4869 (10)	−372 (8)	1111 (3)	506
P	0	5000	0	545
F(1)	1554 (6)	4809 (5)	−826 (2)	1067
F(2)	2420 (5)	4209 (4)	462 (2)	946
F(3)	−382 (8)	3191 (4)	−210 (3)	1325
(II)				
C(1)	6204 (1)	3588 (4)	809 (3)	465
C(2)	6207 (1)	4722 (4)	851 (3)	438
N(1)	5884·0 (9)	2984 (3)	897 (3)	433
N(2)	5890·6 (9)	5304 (3)	988 (2)	389
C(3)	5574 (1)	3561 (3)	1036 (3)	334
C(4)	5579 (1)	4717 (3)	1072 (3)	331
S(1)	5137·0 (3)	2916·8 (8)	1196·1 (8)	420
S(2)	5141·9 (3)	5364·4 (8)	1263·4 (7)	365
C(5)	4882 (1)	4139 (3)	1305 (2)	317
C(6)	4495·9 (9)	4137 (3)	1399 (2)	317
S(3)	4228·2 (3)	2937·4 (8)	1439·1 (8)	435
S(4)	4234·2 (3)	5351·9 (8)	1480·9 (7)	369
C(7)	3792 (1)	3589 (3)	1595 (3)	390
C(8)	3794 (1)	4706 (3)	1595 (3)	357
S(5)	3409·2 (3)	2698·6 (9)	1721 (1)	582
S(6)	3406·7 (3)	5586·2 (9)	1703 (1)	567
C(9*A*)	3033 (2)	3633 (7)	2014 (6)	569
C(9*B*)	3010 (4)	3609 (13)	1318 (14)	675
C(10*A*)	3022 (3)	4680 (12)	1381 (7)	603
C(10*B*)	2975 (7)	4630 (20)	1690 (20)	741
C(11)	3590 (1)	−302 (4)	1504 (3)	442
C(12)	3591 (1)	−1434 (4)	1482 (3)	463
N(11)	3913·0 (9)	298 (3)	1457 (2)	407
N(12)	3913·2 (9)	−2024 (3)	1417 (2)	428
C(13)	4230 (1)	−283 (3)	1390 (3)	336
C(14)	4229 (1)	−1442 (3)	1368 (3)	349
S(11)	4677·7 (3)	368·7 (8)	1350·1 (7)	390
S(12)	4675·3 (3)	−2077·0 (8)	1295·1 (8)	429
C(15)	4941 (1)	−852 (3)	1271 (3)	331
C(16)	5324 (1)	−856 (3)	1192 (3)	336
S(13)	5599·5 (3)	334·7 (8)	1153·2 (7)	385
S(14)	5579·4 (3)	−2082·9 (8)	1098·0 (8)	412
C(17)	6042 (1)	−339 (3)	1099 (3)	355
C(18)	6032 (1)	−1465 (3)	1081 (3)	361
S(15)	6439·7 (3)	528·5 (9)	1083·6 (8)	494
S(16)	6415·6 (3)	−2377·1 (9)	1042·8 (9)	527
C(19*A*)	6822 (3)	−432 (9)	1236 (6)	503
C(19*B*)	6830 (5)	−429 (16)	814 (13)	755
C(20*A*)	6760 (2)	−1448 (6)	571 (5)	366
C(20*B*)	6832 (4)	−1527 (13)	1161 (13)	744
B	2454 (2)	655 (7)	1301 (5)	801
F(1*A*)	2124 (4)	117 (10)	1022 (10)	1293
F(2*A*)	2628 (3)	−179 (10)	1875 (7)	1250
F(3*A*)	2338 (4)	1538 (9)	1939 (9)	1828
F(4*A*)	2629 (3)	985 (17)	589 (6)	2024
F(1*B*)	2688 (3)	797 (15)	2035 (8)	1762
F(2*B*)	2099 (3)	612 (12)	1177 (11)	1323
F(3*B*)	2643 (4)	−64 (13)	745 (16)	2520
F(4*B*)	2549 (5)	1497 (11)	861 (15)	2343
C	2500	2500	5000	2957
Cl(1)	2833 (1)	2291 (5)	4612 (5)	1577
Cl(2)	2420 (2)	2760 (4)	6133 (5)	1686

Table 2. *Bond lengths* (Å) *and angles* (°) *of the cations with e.s.d.'s in parentheses*

For agreement in the numbering scheme with Table 1 add 10 to the atom numbers for cation (2).

		(II)	
	(I)	Cation (1)	Cation (2)
C(1)—C(2)	1·379 (4)	1·375 (6)	1·373 (6)
C(1)—N(1)	1·341 (4)	1·348 (5)	1·343 (5)
C(2)—N(2)	1·347 (4)	1·340 (5)	1·313 (5)
N(1)—C(3)	1·318 (4)	1·317 (5)	1·318 (5)
N(2)—C(4)	1·319 (4)	1·311 (5)	1·313 (5)
C(3)—C(4)	1·391 (4)	1·402 (5)	1·406 (5)
C(3)—S(1)	1·752 (3)	1·744 (4)	1·748 (4)
C(4)—S(2)	1·753 (3)	1·756 (4)	1·741 (4)
S(1)—C(5)	1·742 (3)	1·742 (4)	1·748 (4)
S(2)—C(5)	1·745 (3)	1·743 (4)	1·749 (4)
C(5)—C(6)	1·365 (4)	1·361 (5)	1·348 (5)
C(6)—S(3)	1·734 (3)	1·729 (4)	1·735 (4)
C(6)—S(4)	1·378 (3)	1·741 (4)	1·744 (4)
S(3)—C(7)	1·751 (3)	1·745 (4)	1·748 (4)
S(4)—C(8)	1·745 (3)	1·741 (4)	1·742 (4)
C(7)—C(8)	1·347 (4)	1·353 (6)	1·365 (5)
C(7)—S(5)	1·736 (3)	1·736 (4)	1·735 (4)
C(8)—S(6)	1·740 (3)	1·735 (4)	1·735 (4)
S(5)—C(9*A*)	1·819 (8)	1·814 (8)	1·76 (1)
S(6)—C(10*A*)	1·844 (7)	1·75 (1)	1·828 (7)
C(9*A*)—C(10*A*)	1·53 (1)	1·55 (2)	1·55 (1)
S(5)—C(9*B*)	1·817 (5)	1·81 (2)	1·86 (2)
S(6)—C(10*B*)	1·768 (6)	1·89 (3)	1·77 (1)
C(9*B*)—C(10*B*)	1·506 (8)	1·36 (3)	1·42 (3)
C(2)—C(1)—N(1)	122·6 (3)	122·8 (4)	122·3 (4)
C(1)—N(1)—C(3)	114·7 (3)	114·9 (3)	115·0 (3)
N(1)—C(3)—C(4)	122·7 (3)	122·0 (3)	122·4 (3)
C(3)—C(4)—N(2)	123·1 (3)	123·0 (3)	122·4 (3)
C(4)—N(2)—C(2)	114·4 (3)	115·3 (3)	115·1 (3)
N(2)—C(2)—C(1)	122·5 (3)	122·0 (4)	122·8 (4)
N(1)—C(3)—S(1)	120·6 (2)	121·3 (3)	120·8 (3)
N(2)—C(4)—S(2)	120·2 (2)	120·5 (3)	121·2 (3)
C(4)—C(3)—S(1)	116·7 (2)	116·7 (3)	116·8 (3)
C(3)—S(1)—C(5)	95·0 (1)	95·1 (2)	95·2 (2)
S(1)—C(5)—S(2)	116·7 (2)	116·8 (2)	116·0 (2)
C(5)—S(2)—C(4)	94·9 (1)	94·8 (2)	95.6 (2)
S(2)—C(4)—C(3)	116·7 (2)	116·5 (3)	116·4 (3)
S(1)—C(5)—C(6)	121·4 (2)	121·6 (3)	122·4 (3)
S(2)—C(5)—C(6)	121·9 (2)	121·6 (3)	121·6 (3)
C(5)—C(6)—S(3)	122·4 (2)	122·8 (3)	123·4 (3)
C(5)—C(6)—S(4)	122·6 (2)	122·1 (3)	121·7 (3)
S(3)—C(6)—S(4)	115·1 (2)	115·0 (2)	114·9 (2)
C(6)—S(3)—C(7)	95·5 (1)	95·7 (2)	95·8 (2)
S(3)—C(7)—C(8)	116·8 (2)	116·7 (3)	116·6 (3)
C(7)—C(8)—S(4)	117·0 (2)	117·0 (3)	116·8 (3)
C(8)—S(4)—C(6)	95·5 (1)	95·5 (2)	95·8 (2)
S(3)—C(7)—S(5)	114·1 (2)	114·6 (2)	114·8 (2)
S(4)—C(8)—S(6)	114·7 (2)	115·3 (2)	114·9 (2)
C(8)—C(7)—S(5)	129·1 (2)	128·7 (3)	128·6 (3)
C(7)—C(8)—S(6)	128·3 (2)	127·7 (3)	128·3 (3)
C(7)—S(5)—C(9*A*)	101·3 (3)	102·5 (3)	100·6 (4)
C(7)—S(5)—C(9*B*)	101·3 (2)	98·8 (5)	102·9 (6)
C(8)—S(6)—C(10*A*)	100·9 (2)	99·4 (4)	99·0 (3)
C(8)—S(6)—C(10*B*)	99·2 (2)	104·0 (8)	104·2 (5)
S(5)—C(9*A*)—C(10*A*)	109·9 (6)	110·1 (6)	114·0 (6)
S(6)—C(10*A*)—C(9*A*)	112·8 (5)	113·6 (6)	108·2 (6)
S(5)—C(9*B*)—C(10*B*)	114·7 (4)	122 (1)	119 (1)
S(6)—C(10*B*)—C(9*B*)	113·9 (4)	117 (2)	123 (1)

0·7002/0·1240 (II). Structures solved by direct methods using the *SHELX76* program (Sheldrick, 1976) (I) and integrated Patterson and direct methods using the *PATSEE* program (Egert, 1988) (II). Attempts to refine the structures in *P*1 (I) and *Cc* (II) were unsuccessful, leading to negative *U*'s and unreasonable bond distances. Refinement proceeded in *P*$\bar{1}$ (I) and *C*2/*c* (II) by blocked full-matrix least squares in which $\sum \Delta^2$ was minimized with *SHELX76* (Sheldrick, 1976). H [calculated for C(9), C(10), C(19) and C(20)] riding on C atoms at 1·08 Å. Other H's were located from difference maps and refined isotropically. Non-H atoms anisotropically. It was impossible to locate the H atoms of the dichloromethane (II). Unit weights were used since they gave a satisfactory analysis of variance. Number of refined parameters 226 (I) and 484 (II). Ten (I) and twelve (II) reflections showing strong extinction effects, were given zero weight during final refinement cycles. $wR = 0{\cdot}0391$ (I) and 0·0354 (II) for observed data. $R/wR = 0{\cdot}0493/0{\cdot}0423$ (II) for all data. $S = 0{\cdot}75$ (I) and 3·2 (II). $\Delta\rho_{\max}/\Delta\rho_{\min} = 0{\cdot}0533/-0{\cdot}306$ (I) and $0{\cdot}283/-0{\cdot}312$ (II) e Å^{-3}. $|\Delta/\sigma|_{\max} = 0{\cdot}127$ (I) and 0·484 (II) [U_{22} of F(4*A*) disordered]. Atomic scattering factors from *SHELX76*. The final atomic parameters of the non-H atoms are given in Table 1,* atom numbering in Fig. 2.

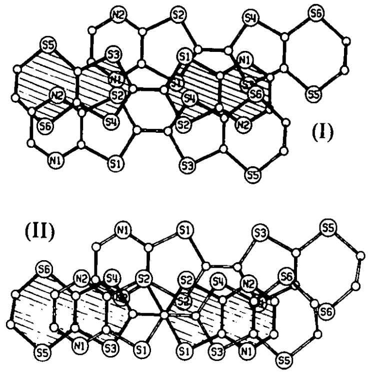

Fig. 3. Projection showing overlap of donors.

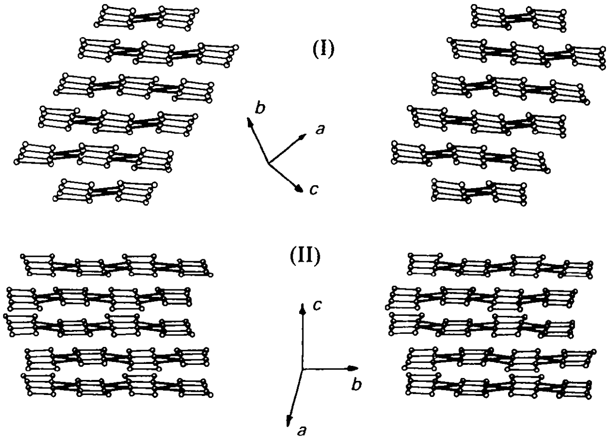

Fig. 4. Stereoviews of the donors. The molecules are simplified for clarity. Only S and N atoms are shown.

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* Lists of observed and calculated structure factors, anisotropic thermal parameters of the non-H atoms, and atomic and isotropic thermal parameters of the H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51861 (34 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. In both structures the ethylene groups are disordered. This disorder is evident in many ET-based structures (Leung *et al.*, 1986; Psycharis *et al.*, 1988; Terzis, Psycharis *et al.*, 1988). The difference maps showed two positions for the ethylene group, *A* and *B*. The site occupancies for these two positions were refined, keeping the sum of the two constant at one. Occupancies for *A* refined to 0·598 (3) (I) and 0·653 (2) and 0·601 (4) for the cations (1) and (2) of (II). Bond lengths and angles of the cations are given in Table 2. The anion in (II) is disordered as well. The difference map shows a plethora of peaks around the position of the boron. We accepted two models for BF_4^- with site occupancies equal to 0·5. At this stage a ΔF synthesis showed one weak and two strong peaks, the weaker one being at a center of symmetry. We assumed this to be a solvent molecule, CH_2Cl_2, disordered so that the average structure appears centrosymmetric. It was included in the refinement with site occupancy of 0·5. The bond distances and angles of the anion and the solvent molecule were not satisfactory and the *U*'s were very large.

In both structures the donors pack side by side in the plane of the molecule with strong S···S and weak S···N contacts (Fig. 2). Stacking of the donors is observed (Figs. 3 and 4), with weak intrastack S···S contacts between 3·64 and 3·80 Å. This mode of packing generates the 'sheet networks' (Fig. 4), of donor molecules that have been observed in all other salts of this donor that we have studied (Psycharis *et al.*, 1988; Terzis, Psycharis, *et al.*, 1988). This mode of packing apparently does not afford the superconducting state and these salts undergo a metal-to-semiconductor (I) (Terzis, Hountas *et al.*, 1988) and metal-to-insulator (II) (Papavasiliou, Underhill, Kaye, Geserich, Terzis & Yiannopoulos, 1987) transition at 280 K (I) and 180 K (II).

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Structure of a Tetraquinane Intermediate in the Synthesis of the Antibiotic Crinipellin A

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Abstract. 3αH,7αH-12α-Isopropyl-8β,11α-dimethyltetracyclo[6.6.0.0^{1,11}.0^{3,7}]tetradecane-6,9-dione, $C_{19}H_{28}O_2$, $M_r = 288·43$, monoclinic, $P2_1/n$, $a = 9·001$ (1), $b = 8·106$ (1), $c = 22·106$ (4) Å, $\beta = 96·14$ (1)°, $V = 1603·7$ (6) Å^3, $Z = 4$, $D_x = 1·195$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5·5$ cm^{-1}, $F(000) = 632$, $T = 295$ K, $R = 0·062$, $wR = 0·067$ for 2457 observed reflections, $I \geq 3·0\sigma(I)$. The ring skeleton consists of four fused five-membered rings. Rings *A* and *C* are in the envelope conformation while rings *B* and *D* are half chairs. The isopropyl group is in the 12α-orientation. Crystal packing is solely dictated by van der Waals forces.

Introduction. Polyquinanes, particularly those formed by linear or angular fusion of three five-

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