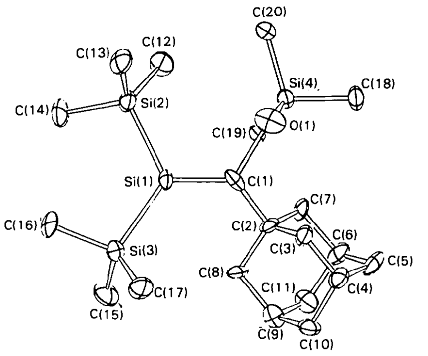

Fig. 1. *ORTEP* plot (Johnson, 1965) of the molecule with 50% probability thermal ellipsoids.

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Structure of the Complex of Dibenzotetrathiafulvalenium Tetrafluoroborate* with Ethanol

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Abstract. $2C_{14}H_8S_4.2C_{14}H_8S_4^{+}.2BF_4^{-}.C_2H_6O$, $M_r =$ 1437·51, triclinic, $P\bar{1}$, $a =$ 10·772 (5), $b =$ 27·309 (6), $c =$ 10·623 (3) Å, $\alpha =$ 85·13 (2), $\beta =$ 103·87 (4), $\gamma =$ 100·49 (4)°, $V =$ 2980·3 (18) Å^3, $Z = 2$, $D_m =$ 1·59, $D_x =$ 1·60 g cm^{-3}, Cu $K\alpha$, $\lambda =$ 1·5418 Å, $\mu =$ 57·64 cm^{-1}, $F(000) = 1464$, $T = 293$ K, $R =$ 0·132 for 7482 observed reflections. The crystal structure consists of two crystallographically independent stacks. Dibenzotetrathiafulvalene molecules, which are planar, are stacked to form columns along the *a* axis [mean interplanar distance 3·6 (1) Å]. The other dibenzotetrathiafulvalene molecule, ethanol and tetrafluoroborate anions stack together to form columns along the same *a* axis.

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* Alternative nomenclature: bis(2,2′-bi-1,3-benzodithiolylidenium) tetrafluoroborate.

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Table 1. *Positional parameters* ($\times 10^4$) *and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

$$B_{\text{eq}} = \tfrac{8}{3}\pi^2 \textstyle\sum_i \sum_j U_{ij} a^*_i a^*_j \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}(Å$^2\times 10^2$)
S(1)	−1217 (3)	8350 (1)	1551 (2)	568 (7)
C(2)	−1273 (8)	7845 (3)	2652 (8)	466 (25)
S(3)	−662 (2)	7981 (1)	4288 (2)	570 (7)
C(4)	294 (10)	8960 (4)	4995 (11)	659 (35)
C(5)	548 (11)	9464 (5)	4633 (12)	734 (40)
C(6)	316 (11)	9647 (4)	3366 (12)	748 (41)
C(7)	−238 (10)	9318 (4)	2363 (11)	664 (35)
C(8)	−537 (9)	8798 (4)	2705 (9)	544 (29)
C(9)	−296 (8)	8618 (4)	3998 (9)	559 (30)
S(10)	−1830 (3)	6854 (1)	3335 (2)	595 (7)
C(11)	−1757 (9)	7375 (4)	2269 (8)	523 (28)
S(12)	−2334 (3)	7251 (1)	622 (2)	571 (7)
C(13)	−3408 (10)	6297 (4)	−173 (11)	645 (34)
C(14)	−3737 (11)	5797 (4)	132 (13)	749 (42)
C(15)	−3492 (11)	5592 (4)	1388 (13)	764 (42)
C(16)	−2865 (11)	5911 (4)	2432 (12)	711 (38)
C(17)	−2568 (9)	6417 (4)	2140 (9)	551 (29)
C(18)	−2820 (9)	6618 (4)	853 (9)	570 (30)
S(19)	2680 (3)	7859 (1)	4553 (2)	593 (7)
C(20)	2057 (8)	7761 (3)	2923 (8)	474 (25)
S(21)	2138 (3)	8276 (1)	1898 (2)	625 (7)
C(22)	3096 (11)	9233 (4)	2895 (13)	740 (41)
C(23)	3616 (11)	9535 (5)	3899 (13)	783 (44)
C(24)	3870 (11)	9322 (5)	5195 (14)	800 (45)
C(25)	3603 (10)	8811 (4)	5451 (11)	659 (35)
C(26)	3056 (9)	8497 (4)	4390 (10)	577 (30)
C(27)	2825 (9)	8709 (4)	3103 (10)	590 (31)
S(28)	952 (2)	7155 (1)	894 (2)	620 (8)
C(29)	1571 (9)	7290 (4)	2529 (8)	571 (30)
S(30)	1551 (3)	6775 (1)	3588 (2)	617 (8)
C(31)	528 (12)	5817 (4)	2796 (12)	774 (42)
C(32)	−100 (12)	5493 (5)	1773 (14)	838 (46)
C(33)	−334 (11)	5684 (5)	525 (14)	839 (48)
C(34)	−79 (10)	6184 (5)	159 (11)	755 (41)
C(35)	543 (9)	6521 (4)	1163 (9)	613 (33)
C(36)	814 (9)	6339 (4)	2432 (9)	589 (31)
S(37)	5160 (2)	8115 (1)	1366 (2)	525 (6)
C(38)	5096 (8)	7617 (3)	2477 (8)	456 (24)
S(39)	5728 (2)	7776 (1)	4100 (2)	512 (6)
C(40)	6698 (9)	8758 (4)	4754 (10)	593 (31)
C(41)	6967 (10)	9256 (4)	4393 (10)	630 (34)
C(42)	6718 (11)	9420 (4)	3090 (12)	681 (37)
C(43)	6141 (10)	9086 (4)	2078 (11)	636 (33)
C(44)	5866 (8)	8577 (3)	2469 (8)	474 (25)
C(45)	6112 (8)	8407 (3)	3782 (8)	465 (25)
S(46)	3980 (2)	7001 (1)	541 (2)	523 (6)
C(47)	4643 (8)	7141 (3)	2158 (8)	431 (24)
S(48)	4558 (2)	6650 (1)	3269 (2)	537 (1)
C(49)	3689 (11)	5683 (4)	2476 (12)	730 (40)
C(50)	3131 (12)	5350 (4)	1466 (13)	776 (42)
C(51)	2897 (11)	5525 (4)	194 (12)	761 (41)
C(52)	3101 (10)	6032 (4)	−187 (11)	618 (33)
C(53)	3660 (8)	6369 (3)	819 (9)	520 (28)
C(54)	3925 (9)	6192 (3)	2143 (9)	502 (27)
S(55)	1821 (3)	2926 (1)	2936 (3)	646 (8)
C(56)	145 (8)	2703 (3)	2640 (9)	519 (27)
S(57)	−763 (3)	3129 (1)	3008 (3)	653 (8)
C(58)	347 (13)	4088 (4)	3739 (11)	733 (40)
C(59)	1491 (18)	4435 (4)	4053 (12)	941 (56)
C(60)	2730 (13)	4339 (4)	4020 (11)	743 (42)
C(61)	2889 (11)	3884 (4)	3674 (10)	660 (35)
C(62)	1783 (10)	3509 (4)	3365 (9)	545 (29)
C(63)	569 (12)	3617 (4)	3405 (9)	640 (35)
S(64)	544 (3)	1831 (1)	1904 (3)	617 (8)
C(65)	−347 (8)	2247 (3)	2240 (8)	478 (26)
S(66)	−2049 (3)	2032 (1)	1992 (3)	621 (8)
C(67)	−3090 (10)	1063 (4)	1152 (10)	647 (33)
C(68)	−2947 (12)	627 (4)	763 (11)	695 (39)
C(69)	−1729 (15)	533 (4)	752 (12)	823 (47)
C(70)	−582 (12)	878 (4)	1063 (10)	688 (36)
C(71)	−779 (10)	1347 (4)	1426 (9)	558 (29)
C(72)	−1990 (10)	1448 (3)	1489 (8)	527 (28)
B(73)	6846 (11)	9279 (4)	8579 (11)	503 (32)
F(74)	7906 (10)	9390 (4)	9466 (10)	1685 (51)
F(75)	6064 (9)	9594 (4)	8619 (11)	1384 (44)
F(76)	7096 (16)	9302 (5)	7458 (9)	2222 (77)
F(77)	6272 (15)	8853 (4)	8833 (16)	2168 (77)
B(78)	6763 (14)	4333 (5)	3707 (10)	552 (35)
F(79)	7717 (10)	4625 (4)	3517 (15)	1858 (64)
F(80)	6098 (14)	4575 (8)	4277 (16)	2763 (99)
F(81)	5857 (12)	4218 (5)	2700 (9)	1969 (65)
F(82)	7182 (18)	3998 (5)	4367 (21)	3076 (99)
C(83)	5344 (16)	7611 (7)	7667 (13)	1182 (69)
C(84)	4688 (14)	7136 (5)	7174 (14)	981 (53)
O(85)	5278 (18)	8007 (7)	7064 (16)	1270 (77)

Introduction. There has been considerable interest recently in organic conductors with highly anisotropic electrical, optical and magnetic properties. Tetrathiafulvalene–tetracyano-*p*-quinodimethane(TTF–TCNQ) is the one that has received the most detailed attention. After this, many compounds with fulvalene rings were synthesized to obtain radical salts with high electrical conductivity. Dibenzotetrathiafulvalene(DBTTF) is one of these compounds (Khidekel & Zhilyaeva, 1981) and the direct oxidation of DBTTF by halogens and metal halides was reported to yield radical salts such as $(DBTTF)_2I_3$ (Shibaeva, Rozenberg, Aldoshina, Lubovskaya & Khidekel, 1979), $(DBTTF)_8[SnCl_6]_3$ and $(DBTTF)_3[SnBr_6]$ (Shibaeva, Rozenberg & Lubovskaya, 1980), $(DBTTF)_2[Cu_2Cl_6]$ (Honda, Katayama, Tanaka & Tanaka, 1985*a*) and $(DBTTF)_2$-$[Cu_2Br_6]$ (Honda, Katayama, Tanaka & Tanaka, 1985*b*). The employment of tetrafluoroborate as an oxidant was reported to yield the ion-radical salt $(DBTTF)(BF_4)_{0.8}$ showing a high conductivity up to temperatures of about 180 K (Krivoshei, Babiyczuk, Guella, Golovkina, Mansia, Starodub & Chueva, 1978). In the present paper, we report the crystal structure of another complex $[(DBTTF)_4$-$(BF_4)_2.C_2H_5OH]$ of tetrafluoroborate and dibenzotetrathiafulvalene.

Experimental. Crystals of $(DBTTF)_4(BF_4)_2.C_2H_5OH$ prepared by recrystallization of $(DBTTF)(BF_4)_{0.8}$ in ethanol solution. The elemental analysis found experimentally was C: 48·32, H: 2·50, S: 35·7, B: 1·3, F: 10·45% and calculation gives C: 48·46, H: 2·66, S: 35·68, B: 1·50, F: 10·57, O: 1·11%. Dark-blue needle-like crystal 0·30 × 0·07 × 0·60 mm. D_m by flotation. Rigaku automated four-circle diffractometer. Cell dimensions from 20 selected reflections with $29 < \theta < 31°$. 9663 measured reflections, $2\theta_{\max} = 126°$, $-13 \le h \le 0$, $-32 \le k \le 32$, $-13 \le l \le 13$, $R_{\text{int}} = 0{\cdot}021$. Three standard reflections after every 100 reflections (decay less than 4%). Structure solved by the Monte-Carlo direct method (Furusaki, 1979) with the aid of *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) using 7482 non-zero unique reflections, and refined on F^2 by full-matrix least-squares program with analytical absorption correction (Katayama, Sakabe & Sakabe, 1972); transmission factors 0·03 to 0·67. On final difference Fourier synthesis no hydrogen atoms could be located satisfactorily. Non-H atoms refined anisotropically. F atoms in BF_4^- anion showed very high thermal motion and may be somewhat disordered, but it was not possible to identify discrete alternative sites for F atoms. These effects gave rather poor value of $R = 0{\cdot}132$ and made it impossible to improve refinement. In spite of the poor R value, we report the structure of $(C_{14}H_8S_4)_4(BF_4)_2.C_2H_5OH$ because it has an interesting stacked structure as is shown below. $S = [\sum w(|F_o|^2 - |F_c|^2)^2/(m-n)]^{1/2} = 4{\cdot}05$, $w = 1/\sigma^2(F^2)$, $(\Delta/\sigma)_{\max} = 0{\cdot}137$ for x of C(25), $\Delta\rho_{\max} = 0{\cdot}7$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). *ORTEP* (Johnson, 1965) employed for drawing molecular structure, and *PLUTO*78 (Motherwell, 1976) for crystal structure projection. All calculations made on a FACOM M-382 computer at the computation center of Nagoya University.

Discussion. Final positional parameters for the non-hydrogen atoms are given in Table 1.* The projection of the structure along the c axis is shown in Fig. 1. The structure consists of two crystallographically independent stacks. The DBTTF molecules (I, II and III) are stacked parallel to each other in columns along the a axis. The angles between the mean planes of these DBTFF and the a axis are almost equal and the mean interplanar spacing is 3·6 (1) Å. The shortest contact between adjacent DBTTF molecules is found between S(30) and S(48) with a distance of 3·42 (1) Å (see Table 2). Judging from the analysis of the polarized reflection spectra (Tanaka, 1985), the stacking sequence is the island type leading to the CT_1 and CT_2 absorption bands in the IR and near-IR (Tanaka, Tanaka, Kawai, Takabe & Maki, 1976). That is, the system of three DBTTF molecules (I, II and III) consists of two cations and one neutral molecule. The fourth DBTTF (IV) and ethanol and the BF_4^- anions form another column in the same direction. The molecular plane of DBTTF (IV) is almost perpendicular to the sheets of the other three DBTTF (I, II and III). In the column, DBTTF (IV) and another group consisting of one ethanol and two BF_4^- anions alternate. The arrangement of such mixed stacking does not belong to the known stacking types.

Fig. 2 shows the bond lengths and bond angles of DBTTF radicals (I; II and III) and neutral DBTTF (IV). These molecules are planar within experimental error. The length of the central C=C bond in molecules (I), (II) and (III) is slightly longer than that of a neutral DBTTF molecule [1·336 (2) Å] (Emge, Wiygul, Chappell, Bloch, Ferrais, Cowan & Kistenmacher, 1982) and

Table 2. *Selected intermolecular distances* (Å)

S(1)–S(21^{i})	3·58 (1)	C(22)–F(75^{v})	3·53 (2)
S(30)–S(48^{i})	3·42 (1)	S(19)–O(85^{i})	3·36 (2)
S(37)–S(1^{ii})	3·80 (1)	S(37)–F(77^{vi})	3·53 (2)
S(3)–S(55^{iii})	3·97 (1)	S(39)–O(85^{i})	3·42 (2)
C(7)–F(74^{iv})	3·26 (1)	C(41)–F(76^{i})	3·24 (2)

Symmetry code: (i) x, y, z; (ii) $1+x, y, z$; (iii) $-x, 1-y, 1-z$; (iv) $-1+x, y, -1+z$; (v) $1-x, 2-y, 1-z$; (vi) $x, y, -1+z$.

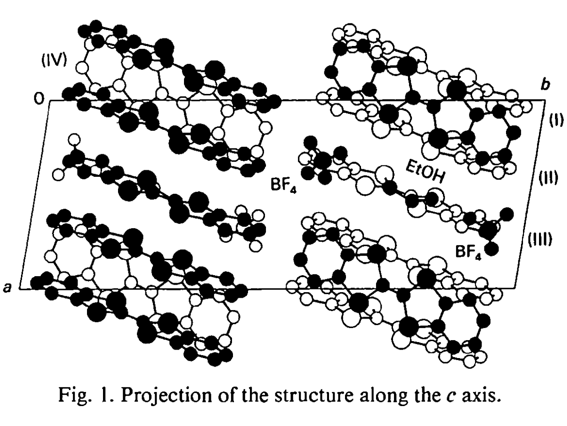

Fig. 1. Projection of the structure along the c axis.

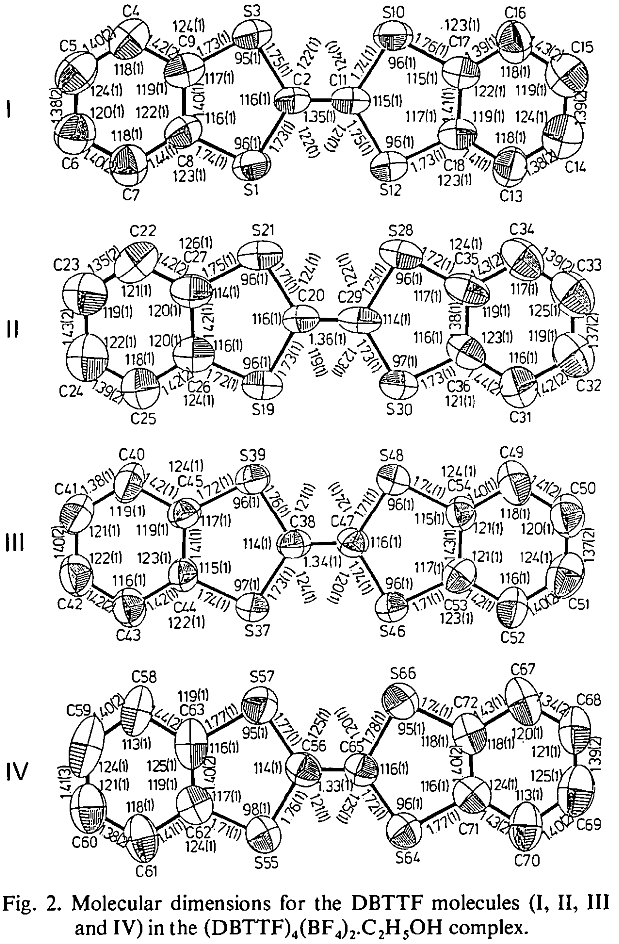

Fig. 2. Molecular dimensions for the DBTTF molecules (I, II, III and IV) in the $(DBTTF)_4(BF_4)_2.C_2H_5OH$ complex.

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* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, best-plane calculations and intermolecular distances have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42376 (51 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Interatomic distances* (Å) *and bond angles* (°) *of* BF_4^- *anions and* C_2H_5OH

B(73)–F(74)	1·30 (1)	B(78)–F(80)	1·33 (3)
B(73)–F(75)	1·32 (2)	B(78)–F(81)	1·28 (2)
B(73)–F(76)	1·28 (2)	B(78)–F(82)	1·19 (2)
B(73)–F(77)	1·25 (2)	C(83)–C(84)	1·44 (2)
B(78)–F(79)	1·23 (2)	C(83)–O(85)	1·21 (3)
F(74)–B(73)–F(75)	109 (1)	F(79)–B(78)–F(81)	116 (1)
F(74)–B(73)–F(76)	111 (1)	F(79)–B(78)–F(82)	105 (1)
F(74)–B(73)–F(77)	110 (1)	F(80)–B(78)–F(81)	97 (1)
F(75)–B(73)–F(76)	110 (1)	F(80)–B(78)–F(82)	113 (2)
F(75)–B(73)–F(77)	107 (1)	F(81)–B(78)–F(82)	117 (1)
F(76)–B(73)–F(77)	111 (1)	C(84)–C(83)–O(85)	125 (1)
F(79)–B(78)–F(80)	108 (1)		

is shorter than those of DBTTF cations, which are 1·404 (7) Å (Honda, Katayama, Tanaka & Tanaka, 1985*a*) and 1·397 (6) Å (Honda, Katayama, Tanaka & Tanaka, 1985*b*). For molecule (IV), the central C=C bond length is comparable with that of the neutral molecule. Table 3 shows the bond lengths and bond angles of the BF_4^- anions and ethanol.

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The Structures of Tetracyclo[4.4.0.0^{2,8}.0^{4,7}]dec-9-en-5-one 2,4-Dinitrophenylhydrazone, (2), 10-Bromotricyclo[5.2.1.0^{4,8}]dec-5-en-2-one, (3), and Tetracyclo[5.3.0.0^{2,5}.0^{4,8}]dec-9-en-3-yl 3,5-Dinitrobenzoate, (4)

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Abstract. $C_{16}H_{14}N_4O_4$, (2), $M_r = 326·3$, monoclinic, $P2_1/n$, $a = 6·745$ (5), $b = 24·915$ (8), $c = 8·911$ (8) Å, $\beta = 99·0$ (1)°, $V = 1480$ (2) Å^3, $Z = 4$, $D_x = 1·47$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 1·01$ cm^{-1}, $F(000) = 680$, $T = 290$ K, $R(F) = 0·080$ for 524 unique reflections $[I > 2·5\sigma(I)]$ and 118 variables. $C_{10}H_{11}BrO$, (3), $M_r = 227·1$, monoclinic, $P2_1/c$, $a = 11·088$ (3), $b = 6·649$ (2), $c = 12·667$ (3) Å, $\beta = 102·36$ (3)°, $V = 912·2$ (7) Å^3, $Z = 4$, $D_x = 1·65$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 44·08$ cm^{-1}, $F(000) = 456$, $T = 290$ K, $R(F) = 0·048$ for 987 unique reflections $[I > 3\sigma(I)]$ and 153 variables. $C_{17}H_{14}N_2O_6$, (4), $M_r = 342·3$, triclinic, $P\bar{1}$, $a = 6·428$ (2), $b = 9·912$ (3), $c = 12·455$ (3) Å, $\alpha = 94·74$ (3), $\beta = 92·64$ (3), $\gamma = 102·79$ (3)°, $V = 769·5$ (7) Å^3, $Z = 2$, $D_x = 1·48$ g cm^{-3}, Mo $K\alpha$ radiation, $\lambda = 0·71069$ Å, $\mu = 1·23$ cm^{-1}, $F(000) = 356$, $T = 290$ K, $R(F) = 0·032$ for 1562 unique reflections $[I > 3\sigma(I)]$ and 268 variables. An investigation was undertaken of possible orbital interaction between

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