other non-H atoms. H atoms were included in fixed positions in structure factor calculations. Refinement was considered complete when the shift/e.s.d. ratio was less than 0·02; 94 parameters varied. Final residuals $R = 0·044$, $wR = 0·049$; goodness of fit 1·004; quantity minimized $\Sigma w(F_o - F_c)^2$, where $w = 1$. Highest peak in final electron density map was 3·4 (2) e Å^{-3} at a distance 1·01 Å from Ir atom and ρ_{min} was $-1·3$ (2) e Å^{-3}. Analytical forms of scattering factors for neutral atoms used (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Tables 2.2B and 2.3.1.); all non-H-atom scattering factors corrected for real and imaginary components of anomalous dispersion. Inspection of $w\Delta^2$ as a function of $\sin\theta/\lambda$, F_o and values of h, k, l showed no unusual features or trends. Positional parameters are listed in Table 1, and bond distances and angles are listed in Table 2.† Programs used were from the *NRC VAX Crystal Structure System* (Gabe, Le Page, Charland, Lee & White, 1989) including their version of *ORTEP* (Johnson, 1965), and were run on a MicroVAX II computer. Fig. 1. shows the cation with the atom-labelling scheme.

Related literature. The structures of the related compounds [Cp*Ir(PMe₃)₂Cl][PF₆] (Kaner, Kouvetakis & Mayorga, 1986) and [Cp*Ir(CO)₂(CH₂Cl)]-[Re₂(CO)₆(μ-Cl)$_x$(μ-Br$_{3-x}$)] (Einstein, Glavina, Pomeroy & Willis, 1986) have been reported.

† Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54100 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
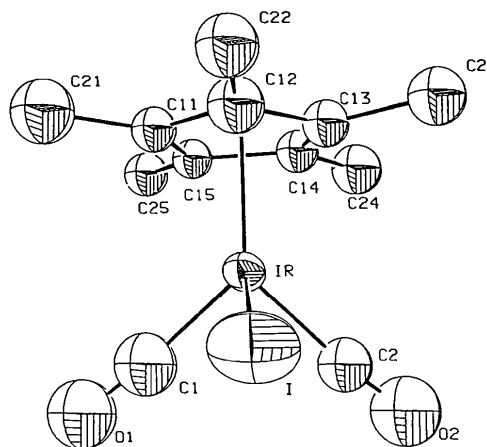

Fig. 1. View of the cation [Cp*Ir(CO)₂I]⁺ with atom labelling.

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Structure of N-Ethyl-m-fluorophenylsuccinimide

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Abstract. 1-Ethyl-3-(3-fluorophenyl)-2,5-pyrrolidine-dione, $C_{12}H_{12}FNO_2$, $M_r = 221·23$, monoclinic, $P2_1/c$, $a = 6·623$ (1), $b = 10·810$ (2), $c = 15·297$ (2) Å, $\beta = 96·82$ (1)°, $V = 1087·6$ (3) Å^3, $Z = 4$, $D_x = 1·351$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, $\mu = 0·84$ mm^{-1}, $F(000) = 464$, room temperature, final $R = 0·052$ for 1577 observed reflections (of 1969 unique data). The succinimide fragment of the molecule is non-planar and has an envelope conformation [the deviation of C1 from the plane N1–C4–C2–C3 is $-0·096$ (2) Å and the dihedral angle between planes C4–C1–C2 and N1–C4–C2–C3 is 5·8 (2)°]. The phenyl ring is planar. The angle between the best planes of the five-membered ring and the phenyl ring is 87·1 (1)°.

Experimental. The title compound recrystallized from ethanol and gave colourless crystals. Crystal

dimensions $0.18 \times 0.20 \times 0.25$ mm. All data were obtained from a KM-4 diffractometer. Final lattice parameters from least-squares refinement of 25 reflections ($10 < \theta < 40°$); no absorption correction applied; $\theta < 80°$ (h: $-8/8$, k: $0/12$, l: $0/19$); $\omega-1.8\theta$ scan technique; Cu $K\alpha$ radiation at room temperature; intensities of three standard reflections monitored every 50 reflections showed no significant fluctuations; 1969 unique reflections measured; 1577 observed reflections with $I > 3\sigma(I)$.

Structure was solved by direct methods (*SHELXS*86; Sheldrick, 1985) with $R(E) = 0.24$. E map provided positions for all non-H atoms. All H atoms (except H103 which was taken from a $\Delta\rho$ map) were located in calculated positions and refined in the riding model; isotropic thermal parameters for H atoms were kept as 1.5 times the temperature factors for their parent C atoms. Refinement by full-matrix least squares with anisotropic temperature factors for all non-H atoms converged to $R = 0.052$ {with $w = 1/[\sigma(F)^2 + 0.002F^2]$ to $wR = 0.068$} with the empirical extinction correction coefficient $g = 0.025$; for all parameters $\Delta/\sigma < 0.005$. The minimum and maximum peaks in the final $\Delta\rho$ map were -0.22 and 0.38 e Å^{-3}; atomic scattering factors from *SHELXTL-PC* (Sheldrick, 1989). All calculations were performed using *SHELXTL-PC* system and *CSU* programs (Vicković, 1988) on the PC computer. The molecule is shown in Fig. 1, coordinates are given in Table 1, and bond distances and angles are listed in Table 2.*

Related literature. The aim of this work is to collect information for further examinations of structure–activity relationships, since the title compound is one of a series of compounds with potential anticonvulsant properties. All compounds considered have been the subject of pharmacological screening

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* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53923 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
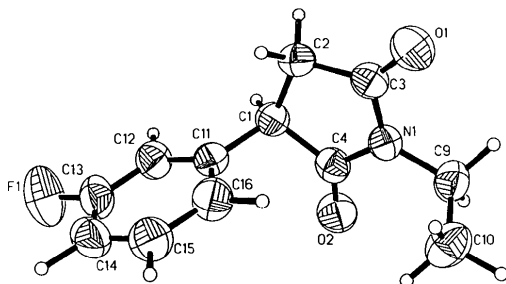

Fig. 1. Numbering scheme and conformation of N-ethyl-m-fluoro-phenylsuccinimide.

Table 1. *Atomic coordinates ($\times 10^4$) and equivalent isotropic displacement coefficients (Å$^2 \times 10^3$)*

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
N(1)	2362 (3)	413 (2)	6590 (1)	48 (1)
C(4)	457 (3)	79 (2)	6776 (2)	50 (1)
C(1)	61 (3)	692 (2)	7632 (2)	49 (1)
C(2)	1926 (4)	1537 (3)	7861 (2)	55 (1)
C(3)	3329 (3)	1252 (2)	7186 (2)	51 (1)
O(1)	5001 (3)	1655 (2)	7140 (2)	75 (1)
O(2)	-667 (3)	-608 (2)	6324 (1)	71 (1)
C(11)	-195 (3)	-281 (2)	8322 (1)	48 (1)
C(12)	-1916 (4)	-311 (3)	8751 (2)	57 (1)
C(13)	-2071 (4)	-1204 (3)	9384 (2)	67 (1)
C(14)	-604 (5)	-2071 (3)	9614 (2)	70 (1)
C(15)	1105 (5)	-2038 (3)	9182 (2)	72 (1)
C(16)	1325 (4)	-1164 (3)	8543 (2)	62 (1)
F(1)	-3756 (3)	-1220 (2)	9797 (2)	111 (1)
C(9)	3322 (4)	-105 (3)	5862 (2)	60 (1)
C(10)	4232 (5)	-1340 (3)	6086 (2)	73 (1)

Table 2. *Bond lengths (Å) and bond angles (°)*

N(1)—C(4)	1.374 (3)	N(1)—C(3)	1.388 (3)
N(1)—C(9)	1.459 (3)	C(4)—C(1)	1.517 (3)
C(4)—O(2)	1.209 (3)	C(1)—C(2)	1.543 (3)
C(1)—C(11)	1.515 (3)	C(2)—C(3)	1.500 (4)
C(3)—O(1)	1.200 (3)	C(11)—C(12)	1.382 (4)
C(11)—C(16)	1.399 (4)	C(12)—C(13)	1.379 (4)
C(13)—C(14)	1.366 (4)	C(13)—F(1)	1.347 (4)
C(14)—C(15)	1.377 (5)	C(15)—C(16)	1.379 (4)
C(9)—C(10)	1.488 (4)		
C(4)—N(1)—C(3)	113.5 (2)	C(4)—N(1)—C(9)	123.5 (2)
C(3)—N(1)—C(9)	122.9 (2)	N(1)—C(4)—C(1)	108.7 (2)
N(1)—C(4)—O(2)	124.1 (2)	C(1)—C(4)—O(2)	127.2 (2)
C(4)—C(1)—C(2)	103.8 (2)	C(4)—C(1)—C(11)	110.1 (2)
C(2)—C(1)—C(11)	113.9 (2)	C(1)—C(2)—C(3)	105.5 (2)
N(1)—C(3)—C(2)	108.1 (2)	N(1)—C(3)—O(1)	123.7 (2)
C(2)—C(3)—O(1)	128.1 (2)	C(1)—C(11)—C(12)	121.0 (2)
C(1)—C(11)—C(16)	120.4 (2)	C(12)—C(11)—C(16)	118.7 (2)
C(11)—C(12)—C(13)	118.9 (2)	C(12)—C(13)—C(14)	123.4 (3)
C(12)—C(13)—F(1)	118.3 (3)	C(14)—C(13)—F(1)	118.3 (3)
C(13)—C(14)—C(15)	117.3 (3)	C(14)—C(15)—C(16)	121.3 (3)
C(11)—C(16)—C(15)	120.3 (3)	N(1)—C(9)—C(10)	111.7 (2)

(Łucka-Sobstal, Zejc & Obniska, 1977; Lange, Rump, Ilczuk, Łapiszewicz, Rabsztyn & Walczyna, 1977; Lange, Rump, Gałecka, Ilczuk, Lechowska-Postek, Rabsztyn, Szymańska & Walczyna, 1977; Łapiszewicz, Lange, Rump & Walczyna, 1978; Chmielewska, 1983, 1984; Zejc & Obniska, 1984; Zejc, Obniska, Chojnacka-Wójcik, Tatarczyńska & Wiczyńska, 1987). Some have been subjected to X-ray structure analysis (Kwiatkowski, Karolak-Wojciechowska, Obniska & Zejc, 1990; Kwiatkowski & Karolak-Wojciechowska, 1990).

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Structure of Di(4,4',5,5'-tetramethyl-1,3-dithia-1',3'-diselenafulvalene) Tetrafluoroborate: (TMDTSDF)₂BF₄

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Abstract. $(C_{10}H_{12}S_2Se_2)_2BF_4$, $M_r = 795.31$, triclinic, $P\bar{1}$, $a = 7.208$ (16), $b = 7.571$ (5), $c = 13.068$ (4) Å, $\alpha = 85.65$ (4), $\beta = 86.88$ (7), $\gamma = 70.46$ (8)°, $V = 669.9$ Å³, $Z = 1$, $D_x = 1.971$ g cm⁻³, $F(000) = 385$, $\lambda(Mo\ K\alpha) = 0.71073$ Å, $\mu = 57.59$ cm⁻¹, $T = 293$ K, $R = 0.044$ based on 1206 observed reflections with $I \geq 3\sigma(I)$. The F atoms of the tetrahedral BF_4^- unit are disordered as already observed in such an anion. However, the B atom is located at the origin of the unit cell, in contrast with its distribution on both sides of the origin in $(TMTCF)_2BF_4$, $C = S$ and Se. The heteroatoms (S and Se) also present a statistical disorder. The bond distances (specially C—S/Se: 1.80–1.85 Å), the interplanar separation (3.59 and 3.62 Å) between adjacent organic molecules and the intermolecular heteroatom contacts are midway between those observed in $(TMTTF)_2BF_4$ and $(TMTSF)_2BF_4$.

Experimental. The hybrid molecule TMDTDSF has been prepared using the procedure developed in the literature (Lerstrup, Johannsen & Jorgensen, 1988). Single crystals of $(TMDTDSF)_2BF_4$ are obtained by electro-oxidation on a platinum electrode of TMDTDSF $(10^{-3}M)$ in tetrahydrofuran solution

containing $(Bu_4N)BF_4$ $(0.1M)$ as supporting electrolyte. A black crystal with approximate dimensions $0.4 \times 0.1 \times 0.1$ mm was selected for data collection on an Enraf–Nonius CAD-4 diffractometer equipped with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.71073$ Å). ω–2θ scan. Cell dimensions were obtained by least-squares refinement from 25 centered reflections ($\theta \leq 50°$). Three standard reflections measured every hour: no fluctuations in intensities. 2566 reflections were recorded in the range $2 \leq 2\theta \leq 50°$ with $0 \leq h \leq 8$, $-8 \leq k \leq 8$, $-15 \leq l \leq 15$. Lorentz–polarization correction. Absorption correction was performed by using the *DIFABS* procedure (Walker & Stuart, 1983). Min. and max. correction factors were 0.803 and 1.520. The structure was solved by direct methods and a successive Fourier difference synthesis. 1206 unique reflections with $I \geq 3\sigma(I)$, after averaging of the symmetry related reflections ($R_{int} = 0.030$), were used for the refinements. H atoms from computed positions (C—H = 1 Å, $B_{eq} = 4$ Å²). Full-matrix least squares (on F) anisotropic (β_{ij}) refinement. Atoms of the disordered BF_4 unit were refined isotropically with an occupancy of 0.5 for the F atoms: H atoms were not refined. Secondary-extinction refinement, $g = -5.2618 \times 10^{-8}$. The convergence was obtained by the introduction of a disordered atom TS (see Table

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