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#### Key indicators

Single-crystal X-ray study

$T = 100$  K

Mean  $\sigma(\text{C}-\text{C}) = 0.007$  Å

$R$  factor = 0.058

$wR$  factor = 0.186

Data-to-parameter ratio = 20.0

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

# An unexpected TTFAQ donor–fluorene acceptor reaction resulting in a novel salt: 2,6-dihexyloxy-9,10-bis(4,5-dimethyl-1,3-dithiol-2-ylidene)-anthracene bis(2,5,7-trinitro-4-bromo-9-cyanofluorenyl) dioxane trisolvate

Reaction of the electron acceptor 2,5,7-trinitro-4-bromo-9-dicyanomethylene-fluorene (1) and the electron donor 2,6-dihexyloxy-9,10-bis(4,5-dimethyl-1,3-dithiol-2-ylidene)-9,10-dihydroanthracene (2) in dioxane resulted in the formation of a novel type of salt with the previously unknown 2,5,7-trinitro-4-bromo-9-cyanofluorenyl anion ( $3^-$ ):  $(2)^{2+} \cdot (3^-)_2 \cdot 3\text{C}_4\text{H}_8\text{O}_2$ .

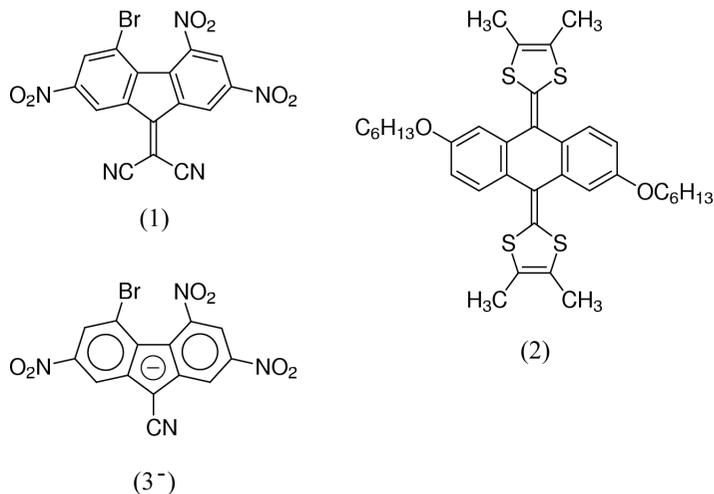
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## Comment

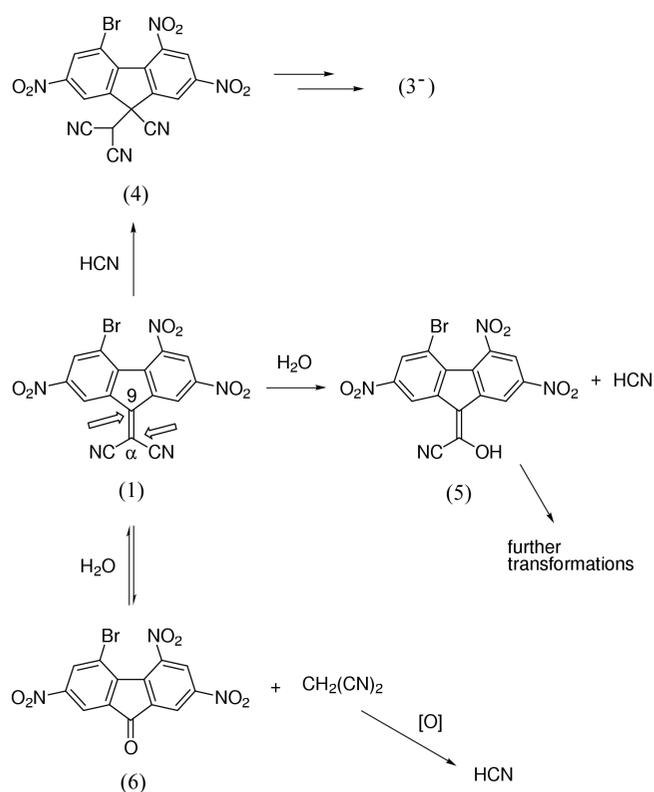
$\pi$ -Extended quinoid electron donors of the tetrathiafulvalene family (TTFAQ) are known to form stable dication salts (Bryce *et al.*, 1990, 2000; Triki *et al.*, 1993). In the course of our investigations on radical ion salts formed between electron acceptors of the fluorene series and TTFAQ (Perepichka *et al.*, 2002) we obtained a novel type of fluorene–TTFAQ salt [ $(3^-)_2 \cdot (2^{2+})$ ], containing the previously unknown 2,5,7-trinitro-4-bromo-9-cyanofluorenyl anion ( $3^-$ ). This salt was unexpectedly isolated from crystallization of a mixture of 2,5,7-trinitro-4-bromo-9-dicyanomethylene-fluorene (1) as an electron acceptor and 2,6-dihexyloxy-9,10-bis(4,5-dimethyl-1,3-dithiol-2-ylidene)-9,10-dihydroanthracene (2) as an electron donor, in dioxane.



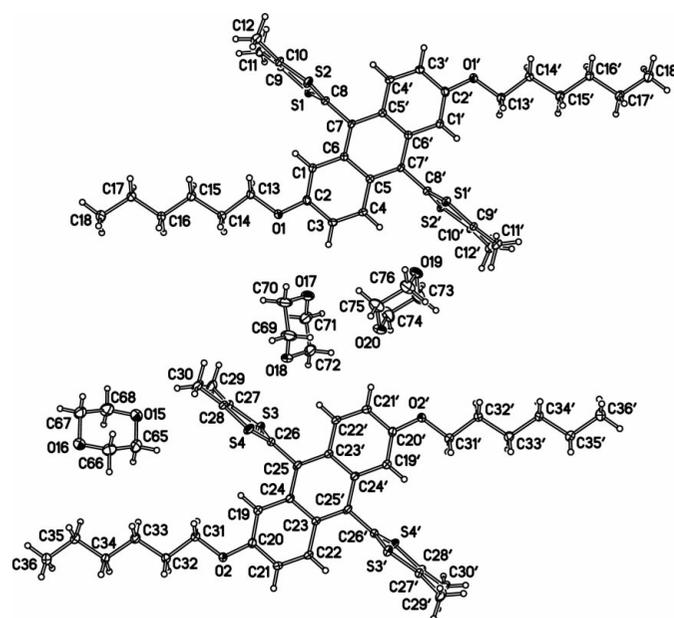
Recently we isolated the first crystalline complex for acceptor (1) (A) and the TTFAQ derivative (D) (Perepichka *et al.*, 2002), which has been shown to be a radical ion salt of the type  $2A^- \cdot D^{2+} \cdot 2\text{MeCN}$ , in contrast to charge-transfer complexes of fluorene acceptors with TTF donors, which are essentially neutral or have only partial charge transfer in the ground state (Perepichka *et al.*, 1998, 2000, 2001; Bryce *et al.*, 1999; Batsanov *et al.*, 2001; Kuz'mina *et al.*, 2002). Continuing these studies, attempts to obtain crystals of a radical salt using (1) as an acceptor and (2) as a donor in dioxane gave no solid during 1.5 months (although the greenish colour of the sol-

ution could indicate a charge transfer interaction between the donor and the acceptor). After leaving this solution to stand for 10 months, a few black crystals were isolated. X-ray analysis revealed that the crystals contained the anion ( $3^-$ ), instead of the expected anion radical ( $1^-$ ), and the composition of the salt is  $(2^{2+}) \cdot (3^-)_2 \cdot 3C_4H_8O_2$ .

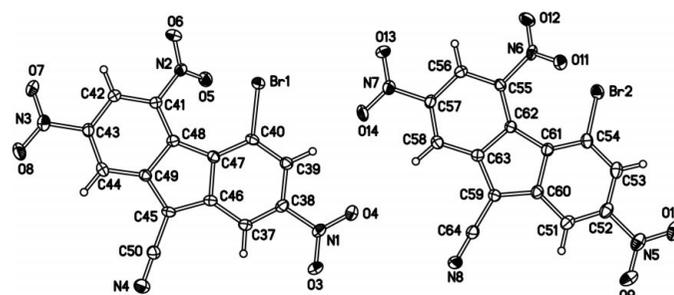
The origin of anion ( $3^-$ ) in the salt  $(2^{2+}) \cdot (3^-)_2$  is not clear, and we found no evidence for its presence in the starting sample of (1) [which was prepared from the corresponding 9-fluorenone precursor and malononitrile (Perepichka *et al.*, 2002)]. Some speculations to explain the formation of ( $3^-$ ) are as follows, with reference to the Scheme below.



Strong electron acceptors of the fluorenone and 9-dicyanomethylenefluorene series, in addition to charge-transfer interaction with electron donors (or a single electron transfer process in the case of strong donors) are known to undergo nucleophilic substitution reactions of the nitro and cyano groups by nucleophiles (Perepichka *et al.*, 2000; Semidetko *et al.*, 1987; Chetkina *et al.*, 1985; Andrievskii *et al.*, 1982). Generally, nucleophilic attack on activated 9-methylenefluorenes can occur at different sites (particularly, attack on  $sp^2$ -hybridized C-9 and C- $\alpha$  atoms), depending on the structures of the substrate and nucleophile, solvent properties, *etc.* (Hoz *et al.*, 1981, 1985; Hoz & Perach, 1982; Hoz & Speizman, 1983; Hoz, 1993; Perepichka *et al.*, 1996). Thus, the key reaction for the transformation of (1) into ( $3^-$ ) is nucleophilic addition of HCN (or  $CN^-$ ) to (1), resulting in compound (4), further oxidative transformation of which produces anion ( $3^-$ ).



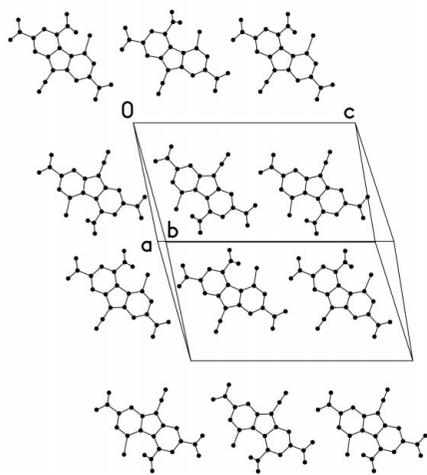
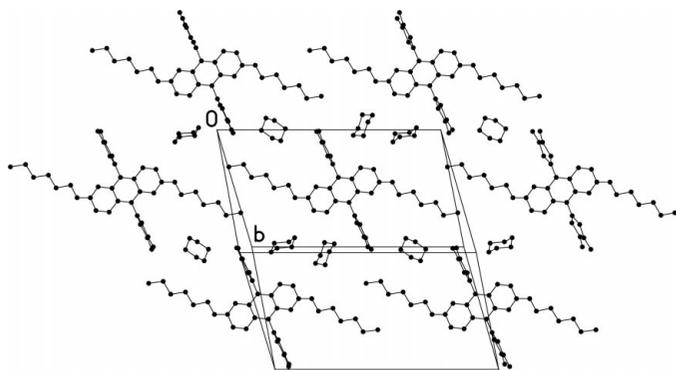
**Figure 1**  
Cations and solvent molecules in  $(2^{2+}) \cdot (3^-)_2 \cdot 3C_4H_8O_2$ . Atoms generated by inversion centres are primed. Atomic displacement ellipsoids are drawn at the 50% probability level.



**Figure 2**  
Anions in  $(2^{2+}) \cdot (3^-)_2 \cdot 3C_4H_8O_2$ . Atomic displacement ellipsoids are drawn at the 50% probability level.

Different reactions could be a source of HCN in the system: (i) nucleophilic attack of water on the C- $\alpha$  atom of compound (1), resulting in substitution of a CN group by an OH group (5); (ii) hydrolysis of (1) to fluorenone (6), after which malonodinitrile could generate HCN. Charge-transfer interaction between (1) and (2), resulting in reversible formation of ( $1^-$ ) and ( $2^{2+}$ ) in solution, could facilitate formation of ( $3^-$ ) through additional redox processes in the system (for example, expulsion of  $CN^-$  from ( $1^-$ ) or, alternatively, generation of  $CN^-$  by the reaction of ( $1^-$ ) with water). Consequently, although we have no clear mechanism for the transformation of (1) into ( $3^-$ ), two key reactions shown in the Scheme, which explain the generation of cyanide ion and its attack at position (9) of the activated fluorene system, have precedents in the literature.

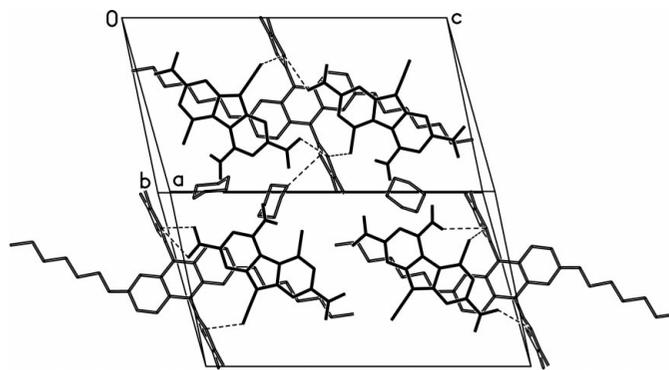
The asymmetric unit of  $(2^{2+}) \cdot (3^-)_2 \cdot 3C_4H_8O_2$  comprises two anions and three dioxane molecules in general positions, and



**Figure 3**  
Layers of dications (top) and anions (bottom), projected on the  $(1\bar{1}0)$  plane.

the halves of two cations, which are located at crystallographic inversion centres (Figs. 1 and 2). The cation is structurally similar to its analogues studied earlier (Bryce *et al.*, 1990, 2000; Christensen *et al.*, 2001; Jones *et al.*, 2001; Triki *et al.*, 1993): the central anthracene moiety is planar, and the dithiolium rings are also planar and nearly perpendicular to the former [interplanar angles  $88.2(1)$  and  $85.8(1)^\circ$  in the two independent cations]. Bond lengths in the anthracene moiety are essentially the same as in anthracene itself (Brock & Dunitz, 1990), indicating a neutral aromatic system. The dithiolium heterocycles in  $(2^+)$  have a similar geometry to the dication of tetrathiafulvalene (Shibaeva, 1984). Thus, each heterocycle carries a charge of  $+1$ , as can be deduced from charge/bond-distance correlations, well established by Reith *et al.* (1988) and Clemente & Marzotto (1996). In both cations, hexyl chains adopt almost ideal all-*trans* conformations and are roughly coplanar with the anthracene moieties. The deviations from the anthracene plane range from  $0.11$  to  $0.46$  Å for the atoms C13 to C18, and from  $0.12$  to  $0.26$  Å for C31 to C36.

The fluorene moieties of both  $(3^-)$  anions adopt a somewhat twisted conformation, similar to that of molecule (1) (Perepichka *et al.*, 2002), due to steric repulsion between the bromo- and nitro-substituents in positions 4 and 5. These



**Figure 4**  
Overlap between the adjacent layers; projection on the  $(1\bar{1}0)$  plane.

substituents are tilted out of the fluorene plane in opposite directions, and the adjacent C atoms follow their tilt. Thus the average deviation of the 13 fluorene C atoms from their mean plane is  $0.09$  and  $0.10$  Å in the two  $(3^-)$  anions, the strongest deviations being shown by C40 ( $0.16$  Å), C41 ( $-0.18$  Å), C54 ( $-0.18$  Å) and C55 ( $0.23$  Å). For the same reason, the twist of the nitro groups (compared to the eclipsed conformation) around the C41–N2 and C55–N6 bonds [ $38.8(5)^\circ$  and  $47.8(5)^\circ$ , respectively] is stronger than for other, sterically unencumbered, nitro groups, *viz.* C38–N1  $13.7(8)$ , C43–N3  $10.1(6)$ , C52–N5  $9(1)$ , C57–N7  $3.1(9)^\circ$ . In all previously studied structures containing molecules of (1), the bromo- and nitro-substituents were disordered between positions 4 and 5; no significant disorder is found here in the  $(3^-)$  anions (see *Experimental* section).

The twist does not disturb the aromaticity of the fluorene system of the  $(3^-)$  anions, the bond distances of which are close to those in the unsubstituted fluorene anion (Buchholz, Harms, Massa & Boche, 1989). In  $(3^-)$ , the cyano group actively participates in  $\pi$ -conjugation and hence in the negative charge delocalization, as indicated by the shortening of the C–CN bond [average  $1.414(6)$  Å] and lengthening of the C≡N bond [average  $1.153(6)$  Å] compared to the average values [ $1.443(8)$  and  $1.138(7)$  Å, respectively] for C(aryl)–C≡N moieties (Allen *et al.*, 1987). The central (five-membered) ring of  $(3^-)$  shows a less aromatic distribution of bond distances than in the fluorene anion. Although each of these differences individually is of marginal statistical significance, they are mutually consistent and suggest a substantial contribution of the C(flourene)=C=N $^-$  structure. In this structure,  $(3^-)$  can be compared to the 1,2,4-tricyanocyclopentadienide anion in the crystal structure of its ammonium salt (Buchholz, Harms, Marsch *et al.*, 1989).

The crystal packing of  $(2^+) \cdot (3^-)_2 \cdot 3C_4H_8O_2$  can be described as a succession of alternating layers of dications and anions (Fig. 3), the dioxane molecules occupying the gaps in and between the layers. Thus, no segregated stacks of similarly charged ions exist. On the other hand, the twisted conformation of the  $(2^+)$  dication, with the positively charged heterocycles oriented normally to the rest of the cation,

forbids an effective cation-anion stacking, such as is usual for charge-transfer complexes. As one can see from Fig. 4, the anion overlaps mainly with the side-chain of the dication. In fact, the packing is dominated by short contacts between the S atoms of the dications (*i.e.* the main centres of positive charge) and the (negatively charged) Br, nitro O and cyano N atoms of the anions. There are nine crystallographically independent S...O contacts shorter than the sum (3.39 Å) of their van der Waals radii (Rowland & Taylor, 1996). The shortest of these, S4...O14 at 3.075 (5) Å, falls within the range characteristic for weak donor-acceptor interactions (Kucsmán & Kapovits, 1985). Both cyano groups participate in short contacts with S atoms [S2...N4 3.111 (5) and S3...N8 2.982 (5) Å, *cf.* the sum of the van der Waals radii, 3.45 Å], as do both bromine atoms [S1...Br2 3.541 (1) and S4...Br1 3.447 (1) Å, *cf.* the sum of the van der Waals radii, 3.69 Å].

## Experimental

Acceptor (1) (Perepichka *et al.*, 2002) (1.4 mg, 3.2 mmol) and donor (2) (Bryce *et al.*, 2000) (1.0 mg, 1.6 mmol) were dissolved together in 1,4-dioxane (0.3 ml), with heating at 353 K for 5 min. The greenish solution was stored in a closed tube in the dark at 293 K. After 1.5 months, no further changes in colour and no precipitation were observed, whereas, after 10 months, a few black (or dark green) crystals were isolated from the bulk amorphous product.

### Crystal data

$C_{36}H_{44}O_2S_4^{2+} \cdot 2C_{14}H_4BrN_4O_6^{-} \cdot 3C_4H_8O_2$   
 $M_r = 1709.51$   
 Triclinic,  $P\bar{1}$   
 $a = 14.136$  (4) Å  
 $b = 14.244$  (4) Å  
 $c = 22.383$  (8) Å  
 $\alpha = 77.92$  (2)°  
 $\beta = 78.49$  (2)°  
 $\gamma = 60.26$  (2)°  
 $V = 3802$  (2) Å<sup>3</sup>

$Z = 2$   
 $D_x = 1.493$  Mg m<sup>-3</sup>  
 Mo  $K\alpha$  radiation  
 Cell parameters from 976 reflections  
 $\theta = 12.0$ – $22.8$ °  
 $\mu = 1.26$  mm<sup>-1</sup>  
 $T = 100$  (2) K  
 Block, black  
 $0.32 \times 0.28 \times 0.13$  mm

### Data collection

SMART 1K CCD area-detector diffractometer  
 $\omega$  scans  
 Absorption correction: by integration (*XPREP* in *SHELXTL*; Bruker, 1998)  
 $T_{min} = 0.695$ ,  $T_{max} = 0.894$   
 42220 measured reflections

19938 independent reflections  
 15653 reflections with  $I > 2\sigma(I)$   
 $R_{int} = 0.041$   
 $\theta_{max} = 29.0$ °  
 $h = -19 \rightarrow 19$   
 $k = -19 \rightarrow 18$   
 $l = -30 \rightarrow 30$

### Refinement

Refinement on  $F^2$   
 $R[F^2 > 2\sigma(F^2)] = 0.058$   
 $wR(F^2) = 0.186$   
 $S = 1.08$   
 19938 reflections  
 998 parameters  
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0951P)^2 + 6.2761P]$   
 where  $P = (F_o^2 + 2F_c^2)/3$   
 $(\Delta/\sigma)_{max} < 0.001$   
 $\Delta\rho_{max} = 1.49$  e Å<sup>-3</sup>  
 $\Delta\rho_{min} = -1.63$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å).

Br1—C40	1.893 (4)	C27—C28	1.355 (7)
Br2—C54	1.892 (4)	C37—C38	1.371 (6)
S1—C8	1.676 (4)	C37—C46	1.404 (6)
S1—C9	1.720 (4)	C38—C39	1.409 (6)
S2—C8	1.669 (4)	C39—C40	1.370 (6)
S2—C10	1.720 (4)	C40—C47	1.416 (5)
S3—C26	1.671 (4)	C41—C42	1.375 (6)
S3—C27	1.718 (4)	C41—C48	1.405 (5)
S4—C26	1.676 (4)	C42—C43	1.419 (6)
S4—C28	1.722 (4)	C43—C44	1.375 (6)
O1—C2	1.351 (5)	C44—C49	1.406 (6)
O2—C20	1.360 (5)	C45—C50	1.410 (6)
N4—C50	1.152 (6)	C45—C46	1.417 (6)
N8—C64	1.153 (6)	C45—C49	1.422 (5)
C1—C2	1.381 (5)	C46—C47	1.439 (5)
C1—C6	1.433 (5)	C47—C48	1.439 (6)
C2—C3	1.428 (5)	C48—C49	1.448 (5)
C3—C4	1.359 (5)	C51—C52	1.375 (6)
C4—C5	1.436 (5)	C51—C60	1.402 (6)
C5—C6	1.438 (5)	C52—C53	1.406 (7)
C6—C7	1.413 (5)	C53—C54	1.376 (6)
C7—C5 <sup>i</sup>	1.408 (5)	C54—C61	1.407 (6)
C7—C8	1.482 (5)	C55—C56	1.372 (6)
C9—C10	1.356 (7)	C55—C62	1.415 (6)
C19—C20	1.375 (5)	C56—C57	1.408 (6)
C19—C24	1.427 (5)	C57—C58	1.374 (6)
C20—C21	1.427 (5)	C58—C63	1.401 (6)
C21—C22	1.355 (6)	C59—C60	1.415 (6)
C22—C23	1.440 (5)	C59—C64	1.417 (6)
C23—C25 <sup>ii</sup>	1.401 (5)	C59—C63	1.428 (6)
C23—C24	1.437 (5)	C60—C61	1.449 (6)
C24—C25	1.411 (5)	C61—C62	1.434 (6)
C25—C23 <sup>ii</sup>	1.401 (5)	C62—C63	1.441 (6)
C25—C26	1.484 (5)		

Symmetry code: (i)  $1 - x, -y, -z$ ; (ii)  $-x, 1 - y, 1 - z$ .

The crystal was a pseudo-merohedral twin, with the twinning law  $0\bar{1}0/\bar{1}00/00\bar{1}$ , associated with the pseudo-monoclinic  $C$ -centred cell  $a = 24.539$ ,  $b = 14.245$ ,  $c = 22.382$  Å,  $\alpha = 90.61$ ,  $\beta = 103.65$ ,  $\gamma = 89.53$ °. The second crystal contribution was refined to 0.1026 (7). Without a twinning correction,  $R = 0.100$ .

The H atoms of the methyl groups were found by the circular Fourier method and refined in the rigid-body model ( $C-H$  0.978 Å); a riding model was used for other H atoms, with  $C(sp^2)-H$  0.948 and  $C(\text{methylene})-H$  0.988 Å.

The highest peak of residual electron density lies 2.66 Å from Br1 and in the close proximity of the adjacent nitro-group, and the largest hole is 0.45 Å from Br1. These features may indicate some minor disorder of the bromo and nitro-substituents between positions 4 and 5 of the fluorene, which could not be resolved.

Data collection: *SMART* (Bruker, 1999); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1990); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1998); software used to prepare material for publication: *SHELXTL*.

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## References

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

- Andrievskii, A. M., Grekhova, N. G., Andronova, N. A., Shifrina, R. R., Alexandrov, V. N. & Dyumaev, K. M. (1982). *Zh. Org. Khim.* **18**, 1961–1966 (*Chem. Abstr.* 1983, **98**, 106926v).
- Batsanov, A. S., Bryce, M. R., Chesney, A., Howard, J. A. K., John, D. E., Moore, A. J., Wood, C. L., Gershtenman, H., Becker, J. Y., Khodorkovsky, V. Y., Ellern, A., Bernstein, J., Perepichka, I. F., Rotello, V., Gray, M. & Cuello, A. O. (2001). *J. Mater. Chem.* **11**, 2181–2191.
- Brock, C. P. & Dunitz, J. D. (1990). *Acta Cryst.* **B46**, 795–806.
- Bruker (1999). *SMART* (Version 5.054) and *SAINT* (Version 6.02A). Bruker AXS, Madison, Wisconsin, USA.
- Bruker (1998). *SHELXTL*. Version 5.10. Bruker AXS, Madison, Wisconsin, USA.
- Bryce, M. R., Moore, A. J., Batsanov, A. S., Howard, J. A. K., Robertson, N. & Perepichka, I. F. (1999). *Supramolecular Engineering of Synthetic Metallic Materials: Conductors and Magnets*. NATO ASI Series, Vol. 518, edited by J. Veciana, C. Rovira and D. B. Amabilino, pp. 437–449. Dordrecht: Kluwer Academic Publishers.
- Bryce, M. R., Moore, A. J., Hasan, M., Ashwell, G. J., Fraser, A. T., Clegg, W., Hursthouse, M. B. & Karaulov, A. I. (1990). *Angew. Chem. Int. Ed. Engl.* **29**, 1450–1452.
- Bryce, M. R., Finn, T., Batsanov, A. S., Katakya, R., Howard, J. A. K. & Lyubchik, S. B. (2000). *Eur. J. Org. Chem.* pp. 1199–1205.
- Buchholz, S., Harms, K., Marsch, M., Massa, W. & Boche, G. (1989). *Angew. Chem. Int. Ed. Engl.* **28**, 72–73.
- Buchholz, S., Harms, K., Massa, W. & Boche, G. (1989). *Angew. Chem. Int. Ed. Engl.* **28**, 73–74.
- Chetkina, L. A., Povetyeva, Z. P., Bel'skii, V. K. & Bepalov, B. P. (1985). *Kristallografiya*, **30**, 910–914 (*Chem. Abstr.* 1985, **103**, 204122w).
- Christensen, C. A., Batsanov, A. S., Bryce, M. R. & Howard, J. A. K. (2001). *J. Org. Chem.* **66**, 3313–3320.
- Clemente, D. A. & Marzotto, A. (1996). *J. Mater. Chem.* **6**, 941–946.
- Hoz, S. (1993). *Acc. Chem. Res.* **26**, 69–74.
- Hoz, S., Speizman, D., Feit, B.-A., Djamal, G. & Melamed, U. (1981). *J. Org. Chem.* **46**, 450–452.
- Hoz, S. & Perach, S. S. (1982). *J. Org. Chem.* **47**, 4056–4059.
- Hoz, S. & Speizman, D. (1983). *J. Org. Chem.* **48**, 2904–2910.
- Hoz, S., Gross, Z. & Cohen, D. (1985). *J. Org. Chem.* **50**, 4056–4059.
- Jones, A. E., Christensen, C. A., Perepichka, D. F., Batsanov, A. S., Beeby, A., Low, P. J., Bryce, M. R. & Parker, A. W. (2001). *Chem. Eur. J.* **7**, 973–978.
- Kucsmán, A. & Kapovits, I. (1985). *Organic Sulfur Chemistry*, edited by F. Bernardi, I. G., Csizmadia and A. Manqini, pp. 191–245.
- Kuz'mina, L. G., Perepichka, I. F., Perepichka, D. F., Howard, J. A. K. & Bryce, M. R. (2002). *Cryst. Rep.*, **47**, 251–261.
- Perepichka, I. F., Popov, A. F., Orekhova, T. V., Bryce, M. R., Vdovichenko, A. N., Batsanov, A. S., Goldenberg, L. M., Howard, J. A. K., Sokolov, N. I. & Megson, J. L. (1996). *J. Chem. Soc. Perkin Trans. 2*, pp. 2453–2469.
- Perepichka, I. F., Kuz'mina, L. G., Perepichka, D. F., Bryce, M. R., Goldenberg, L. M., Popov, A. F. & Howard, J. A. K. (1998). *J. Org. Chem.* **63**, 6484–6493.
- Perepichka, I. F., Popov, A. F., Orekhova, T. V., Bryce, M. R., Andrievskii, A. M., Batsanov, A. S., Howard, J. A. K. & Sokolov, N. I. (2000). *J. Org. Chem.* **65**, 3053–3063.
- Perepichka, I. F., Perepichka, D. F., Lyubchik, S. B., Bryce, M. R., Batsanov, A. S. & Howard, J. A. K. (2001). *J. Chem. Soc. Perkin Trans. 2*, pp. 1546–1551.
- Perepichka, D. F., Bryce, M. R., Perepichka, I. F., Lyubchik, S. B., Godbert, N., Christensen, C. A., Batsanov, A. S., Levillain, E., McInnes, E. J. L. & Zhao, J. P. (2002). *J. Am. Chem. Soc.*. In the press.
- Reith, W., Polborn, K. & Amberger, E. (1988). *Angew. Chem. Int. Ed. Engl.* **27**, 699–700.
- Rowland, R. S. & Taylor, R. (1996). *J. Phys. Chem.* **100**, 7384–7391.
- Semidetko, O. V., Chetkina, L. A., Bel'skii, V. K., Mysyk, D. D., Perepichka, I. F., Andrievskii, A. M. (1987). *Zh. Obshch. Khim.* **57**, 415–420 (*J. Gen. Chem. USSR*, **57**, 360–364.)
- Sheldrick, G. M. (1990). *Acta Cryst.* **A46**, 467–473.
- Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
- Shibaeva, R. P. (1984). *Kristallografiya*, **29**, 480–483.
- Triki, S., Ouahab, L., Lorcy, D. & Robert, A. (1993). *Acta Cryst.* **C49**, 1189–1192.