

Proton transfer *versus* nontransfer in compounds of the diazo-dye precursor 4-(phenyldiazenyl)aniline (aniline yellow) with strong organic acids: the 5-sulfosalicylate and the dichroic benzenesulfonate salts, and the 1:2 adduct with 3,5-dinitrobenzoic acid

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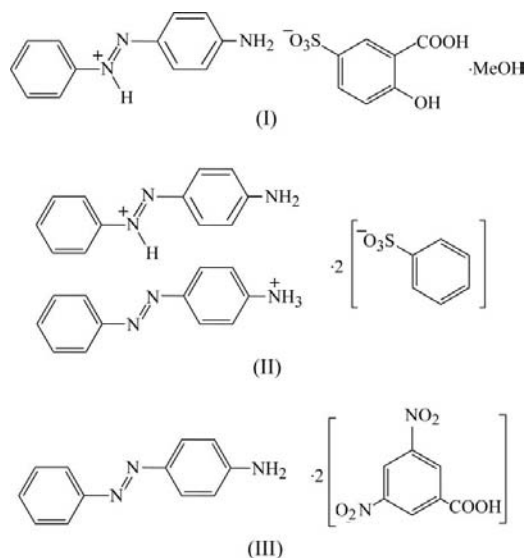
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The structures of two 1:1 proton-transfer red–black dye compounds formed by reaction of aniline yellow [4-(phenyldiazenyl)aniline] with 5-sulfosalicylic acid and benzenesulfonic acid, and a 1:2 nontransfer adduct compound with 3,5-dinitrobenzoic acid have been determined at either 130 or 200 K. The compounds are 2-(4-aminophenyl)-1-phenylhydrazin-1-ium 3-carboxy-4-hydroxybenzenesulfonate methanol solvate, $C_{12}H_{12}N_3^+ \cdot C_7H_5O_6S^- \cdot CH_3OH$, (I), 2-(4-amino-phenyl)-1-phenylhydrazin-1-ium 4-(phenyldiazenyl)anilinium bis(benzenesulfonate), $2C_{12}H_{12}N_3^+ \cdot 2C_6H_5O_3S^-$, (II), and 4-(phenyldiazenyl)aniline–3,5-dinitrobenzoic acid (1/2), $C_{12}H_{11}N_3 \cdot 2C_7H_4N_2O_6$, (III). In compound (I), the diazenyl rather than the aniline group of aniline yellow is protonated, and this group subsequently takes part in a primary hydrogen-bonding interaction with a sulfonate O-atom acceptor, producing overall a three-dimensional framework structure. A feature of the hydrogen bonding in (I) is a peripheral edge-on cation–anion association also involving aromatic C–H...O hydrogen bonds, giving a conjoint $R_2^1(6)R_2^1(7)R_1^1(4)$ motif. In the dichroic crystals of (II), one of the two aniline yellow species in the asymmetric unit is diazenyl-group protonated, while in the other the aniline group is protonated. Both of these groups form hydrogen bonds with sulfonate O-atom acceptors and these, together with other associations, give a one-dimensional chain structure. In compound (III), rather than proton transfer, there is preferential formation of a classic $R_2^2(8)$ cyclic head-to-head hydrogen-bonded carboxylic acid homodimer between the two 3,5-dinitrobenzoic acid molecules,

which, in association with the aniline yellow molecule that is disordered across a crystallographic inversion centre, results in an overall two-dimensional ribbon structure. This work has shown the correlation between structure and observed colour in crystalline aniline yellow compounds, illustrated graphically in the dichroic benzenesulfonate compound.

Comment

Aniline yellow [4-(phenyldiazenyl)aniline, *p*-aminoazobenzene, PAZAN] is a diazo-dye precursor (O'Neil, 2001) and in the structures of its proton-transfer compounds with organic acids [oxalic acid (Mahmoudkhani & Langer, 2001*b*), phenylphosphonic acid (two concomitant polymorphs; Mahmoudkhani & Langer, 2002*a*), butane-1,4-bisphosphonic acid (Mahmoudkhani & Langer, 2002*b*), and the isomeric 3- and 4-nitrophthalic and 5-nitroisophthalic acids (Smith *et al.*, 2008)], as might intuitively be expected, the aniline functional group is protonated. However, in the purple–black hydrochloride (Yatsenko *et al.*, 2000; Mahmoudkhani & Langer, 2001*a*) and the hydrochloride of the analogous 4-phenyldiazenyl-1,3-diaminobenzene (Moreiras *et al.*, 1981) the diazenyl group is protonated. Also, with phosphoric acid (Halasz *et al.*, 2007), the orange monohydrogen phosphate is an anilinium salt while the purple dihydrogen phosphate salt is diazenyl-group protonated. The dye methyl red {4-[(*N,N'*-dimethylanilino)diazenyl]benzene-2-carboxylic acid} also gives diazenyl-protonated salts and adducts with 2,5-dihydroxybenzoic acid (Benedict *et al.*, 2006) and the matrix assisted laser disruption ionization (MALDI) host 2,6-dihydroxybenzoic acid (Cohen *et al.*, 2007). It is of interest also that the azo dyes 4-[4-(*N,N'*-dimethylamino)phenyldiazenyl]benzenesulfonic acid (Burke *et al.*, 2004) and 4-[4-(*N,N'*-diethylamino)phenyldiazenyl]benzenesulfonic acid (Burke *et al.*, 2006) exist as sulfonate–diazenyl-group zwitterions.



This study was therefore aimed at characterizing crystalline products from the reaction of aniline yellow with strong organic acids, to enable the identification of the nature of the interaction involved, *i.e.* proton transfer *versus* nontransfer,

and its correlation with qualitative recognition through the observed colour of the crystals. Our general observation regarding the reaction of PAZAN with carboxylic acids has been that proton-transfer compounds are rarely formed (Smith *et al.*, 2008). This is in evidence in the general paucity of reported structures of PAZAN–aminium salts in the literature. We now have obtained suitable crystalline salts of aniline yellow with the strong organic acids, 5-sulfosalicylic acid (5-SSA), benzenesulfonic acid (BSA) and 3,5-dinitrobenzoic acid (3,5-DNBA) which, unlike the aminium salts, are intense red-black or deep red in colour, crystallizing from solutions with a significantly different colour. The structures of the two red-black compounds, the methanol solvate 2-(4-aminophenyl)-1-phenylhydrazin-1-ium 3-carboxy-4-hydroxybenzenesulfonate methanol solvate, (I), and anhydrous 2-(4-aminophenyl)-1-phenylhydrazin-1-ium 4-(phenyldiazenyl)anilinium bis(benzenesulfonate), (II), and the red crystal of the adduct compound 4-(phenyldiazenyl)aniline–3,5-dinitrobenzoic acid (1/2), (III), are reported here.

With both compounds (I) and (II), proton transfer has occurred while with (III) there is no transfer. However, the differences even between the structures of (I) and (II) are significant so the discussion considers each structure individually. In (I), the 1:1 methanol solvate compound of PAZAN with 5-SSA, the diazenyl rather than the amino group is protonated and gives a direct N11–H···O51A_{sulfonate} hydrogen bond, while a sulfonate O atom acts as an acceptor in an interaction with the methanol hydroxy group (O1B···O52A) (Fig. 1). Other lateral cation aromatic C–H···O_{sulfonate} hydrogen-bonding associations (Table 1) close

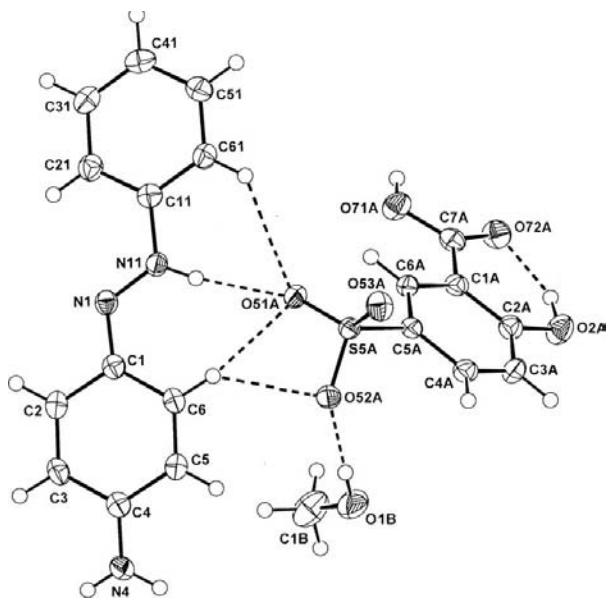


Figure 1

The molecular configuration and atom-numbering scheme for components of (I). Dashed lines indicate inter-species hydrogen bonds, including C–H···O associations which complete conjoint cyclic edge-on cation–anion interactions. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii.

conjoint cyclic ring systems [graph sets $R_2^1(6)R_2^1(7)R_1^2(4)$ (Etter *et al.*, 1990)], also shown in Fig. 1. The 5-sulfosalicylate anions form infinite head-to-tail homomolecular hydrogen-bonded chain structures through carboxylic acid proton donors and sulfonate O-atom acceptors, extending along the *a*-cell direction (Fig. 4). The result is a three-dimensional framework structure.

In the dichroic proton-transfer compound (II) with BSA, the unusual feature is the presence of both diazenyl- and amine-protonated PAZAN species (*A* and *B*, respectively) in the structure. The asymmetric unit in the triclinic unit cell comprises both of these cationic species as well as two benzenesulfonate anions (*C* and *D*), the *D* molecule having rotational disorder (80/20%) in the sulfonate group (Fig. 2). The diazenium-group H atom on N11A interacts directly with a sulfonate O atom (O11C), while all three anilinium H atoms on N4B similarly form hydrogen bonds with both *C*- and *D*-anion sulfonate O-atom acceptors (Table 2). These and other N–H···O interactions result in one-dimensional chain structures (Fig. 5). Within these chain structures, the *A*- and *B*-cation species are oriented approximately mutually perpendicularly in the unit cell, corresponding to the observed *circa* 90° red-to-black dichroism in the crystal. It should also be noted that there are solvent-accessible voids (41 Å³) in the structure of (II) (PLATON; Spek, 2009), centred at $(\frac{1}{2}, 0, 0)$ with the closest atom C5C being 3.28 (1) Å from the centre.

The formation of the 1:2 adduct molecule (III) rather than a proton-transfer compound from the 1:1 reaction of aniline yellow with 3,5-dinitrobenzoic acid is unexpected, considering the relative strength of the acid ($pK_a = 2.82$). However, 3,5-DNBA has been recognized as a useful adduct-forming synthon for crystal engineering (Etter & Frankenbach, 1989), with a number of 1:1 nontransfer adducts being reported,

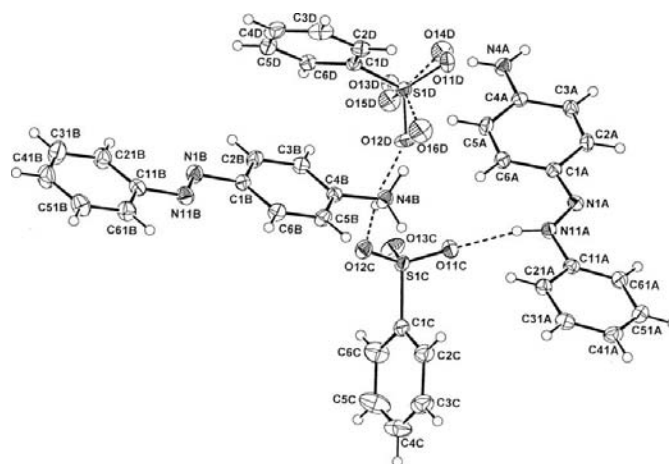


Figure 2

The molecular configuration and atom-numbering scheme for the two 4-(phenyldiazenyl)aniline cation species *A* and *B*, and the two benzenesulfonate anions *C* and *D*, in the asymmetric unit of (II). Isotropic O atoms of the rotationally disordered sulfonate group with site-occupancy factors of 0.20 (1) are O14D, O15D and O16D. Inter-species hydrogen bonds are shown as dashed lines. Displacement ellipsoids are drawn at the 40% probability level and H atoms are shown as small spheres of arbitrary radii.

centroid separation for ring C1–C6 to ring C1A–C6A (at x , $y - 1$, z) = 3.6973 (10) Å].

With the cation species in (I) and (II), as well as the PAZAN molecule in (III), the two phenyl rings are essentially coplanar, as has been found in other compounds of aniline yellow (Mahmoudkhani & Langer, 2001*a,b*, 2002*a,b*; Smith *et al.*, 2008). The torsion angles C2/6–C1–N1–N11 and C21/61–C11–N11–N1 are -177.08 (16) and 175.47 (16)°, respectively, for (I), 179.27 (14) and 165.81 (14)° for (IIA), and 179.45 (14) and -179.41 (15)° for (IIB), while the corresponding angles for (III) which are symmetry related are 167.00 (13)°.

The anion in (I) has conformational features similar to those found in other proton-transfer compounds of 5-sulfosalicylic acid (Smith *et al.*, 2005, 2005*a,b*, 2006). These include the presence of the intramolecular hydroxyl–carboxyl hydrogen bond [O··O = 2.624 (2) Å], which results in the essential coplanarity of the carboxylic acid group and the benzene ring [C2A–C1A–C71A–O71A = -173.86 (16)°]. Also, the common intramolecular C6A–H··O_{sulfonate} interaction [C··O = 2.888 (2) Å] is present. In (III), the 3,5-DNBA molecule is essentially planar [C2–C1–C11–O11 = 175.88 (13)°, C2–C3–N3–O32 = -176.79 (14)° and C4–C5–N5–O52 = 178.35 (14)°].

It has been shown in the structures of compounds (I) and (II) that the protonated PAZAN cation species act as acid–base indicators, undergoing a colour change from red [the anilinium ('benzenoid') form] to red–black (the 'quinoid' form) in much the same manner as the analogous azo-dye indicator methyl red (yellow to red). Compound (II) has fortuitously captured the dichroic red to red–black equilibrium state with the presence of both hybrid colour forms, the *circa* 90° colour dichroism being consistent with the orientation of the two protonated forms in the crystal. These forms are readily identified crystallographically, not only with the H atom location and the associated hydrogen bonding, but also convincingly in the comparative bond distances and angles in the aniline group and the diazene-group extension (Table 4). In (I) and molecule A in (II), these are consistent with the presence of the localized single–double bond 'quinoid' system in the aniline molecular moiety, as is also found in the hydrochloride (Mahmoudkhani & Langer, 2001*a*). The red anilinium-protonated examples [molecule B in (II) and the three isomeric PAZAN hydrogen nitrophthalates (Smith *et al.*, 2008)] are considered 'normal' for aminium-protonated compounds.

Experimental

Compounds (I)–(III) were synthesized by heating together for 10 min under reflux 4-(phenyldiazenyl)aniline (1 mmol) and, respectively, 3-carboxy-4-hydroxybenzenesulfonic acid (5-sulfosalicylic acid), benzenesulfonic acid or 3,5-dinitrobenzoic acid (1 mmol), in either methanol (50 ml) [for (I)] or ethanol–water (1:1, 50 ml) [for (II) and (III)]. Both (I) (m.p. 435–437 K) and (II) (m.p. >538 K) were obtained as red–black needle prisms, with (II) exhibiting red to red–black dichroism, and (III) (m.p. 433 K) was obtained as red needles after partial room-temperature evaporation of solvent.

Compound (I)

Crystal data

C₁₂H₁₂N₃⁺·C₇H₅O₆S[−]·CH₃OH
M_r = 447.46
 Monoclinic, *P*2₁/*c*
a = 8.7174 (7) Å
b = 20.1160 (16) Å
c = 11.8827 (9) Å
 β = 97.145 (2)°

V = 2067.6 (3) Å³
Z = 4
 Mo *K*α radiation
 μ = 0.21 mm^{−1}
T = 130 K
 0.45 × 0.25 × 0.20 mm

Data collection

Bruker SMART CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.91, *T_{max}* = 0.96

10840 measured reflections
 3640 independent reflections
 3102 reflections with *I* > 2σ(*I*)
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.101$
S = 1.05
 3640 reflections
 305 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.37 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e } \text{Å}^{-3}$

Compound (II)

Crystal data

2C₁₂H₁₂N₃⁺·2C₆H₅O₃S[−]
M_r = 710.82
 Triclinic, *P*1̄
a = 5.7601 (4) Å
b = 13.0794 (8) Å
c = 23.6556 (15) Å
 α = 77.937 (5)°
 β = 83.985 (5)°

$\gamma = 86.971$ (5)°
V = 1732.4 (2) Å³
Z = 2
 Mo *K*α radiation
 $\mu = 0.21 \text{ mm}^{-1}$
T = 200 K
 0.40 × 0.20 × 0.12 mm

Data collection

Oxford Gemini-S CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
T_{min} = 0.870, *T_{max}* = 0.980

22657 measured reflections
 7187 independent reflections
 5717 reflections with *I* > 2σ(*I*)
R_{int} = 0.028

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.036$
 $wR(F^2) = 0.094$
S = 1.06
 7187 reflections
 487 parameters

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\max} = 0.28 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.33 \text{ e } \text{Å}^{-3}$

Table 1
 Hydrogen-bond geometry (Å, °) for (I).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O2A–H2A···O72A	0.90 (3)	1.80 (3)	2.624 (2)	151 (2)
O71A–H71A···O52A ⁱ	0.90 (3)	1.74 (3)	2.6246 (19)	166 (2)
O1B–H1B···O52A	0.80 (2)	2.00 (2)	2.792 (2)	175 (2)
N11–H11···O51A	0.85 (2)	2.07 (2)	2.895 (2)	163.1 (17)
N4–H43···O53A ⁱⁱ	0.87 (2)	1.97 (2)	2.834 (2)	170.5 (17)
N4–H42···O1B ⁱⁱⁱ	0.88 (2)	1.98 (2)	2.845 (2)	173 (2)
C6–H6···O51A	0.95	2.52	3.421 (2)	158
C6–H6···O52A	0.95	2.41	3.184 (2)	138
C6A–H6A···O51A	0.95	2.49	2.888 (2)	105
C61–H61···O51A	0.95	2.51	3.260 (2)	136

Symmetry codes: (i) $x - 1, y, z$; (ii) $-x + 2, y - \frac{1}{2}, -z + \frac{3}{2}$; (iii) $-x + 2, -y + 1, -z + 1$.

Table 2

Hydrogen-bond geometry (Å, °) for (II).

D—H...A	D—H	H...A	D...A	D—H...A
N11A—H11A...O11C	0.873 (19)	1.921 (19)	2.7672 (18)	162.9 (17)
N4A—H42A...O11D ⁱ	0.84 (2)	2.18 (2)	2.967 (3)	155.7 (18)
N4A—H43A...O11D ⁱⁱ	0.92 (2)	2.05 (2)	2.962 (3)	177.2 (18)
N4B—H42B...O12C	0.82 (3)	2.14 (3)	2.820 (2)	141 (2)
N4B—H42B...O12D	0.82 (3)	2.43 (3)	3.001 (3)	128 (2)
N4B—H43B...O13C ⁱⁱⁱ	0.99 (2)	1.85 (2)	2.838 (2)	175 (2)
N4B—H44B...O13D ⁱⁱⁱ	0.99 (2)	1.93 (2)	2.890 (3)	165 (2)

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

Table 3

Hydrogen-bond geometry (Å, °) for (III).

D—H...A	D—H	H...A	D...A	D—H...A
O11—H11...O12 ⁱ	0.90 (2)	1.75 (2)	2.6426 (16)	175 (2)
N4A—H41A...O31 ⁱⁱ	0.84 (4)	2.31 (4)	3.042 (4)	147 (4)
N4A—H42A...O32	0.74 (5)	2.48 (4)	3.213 (4)	173 (4)

Symmetry codes: (i) $-x, -y, -z$; (ii) $-x + 1, -y + 2, -z$.

Table 4

Selected bond distances (Å) in the PAZAN species in (I), (II) (molecules A and B) and (III) compared with those in the hydrochloride (PAZAN·HCl) and the 1:1 PAZAN compounds with the three nitrophthalic acids, viz. PAZAN-3-NPA (with 3-nitrophthalic acid), PAZAN-4-NPA (with 4-nitrophthalic acid) and PAZAN-5-NPA (with 5-nitroisophthalic acid).

Bond	(I) [†]	(IIA) [†]	(IIB) [†]	(III) [†]
N1—N11	1.339 (2)	1.3103 (14)	1.2623 (17)	1.2625 (17)
N1—C1	1.339 (3)	1.356 (2)	1.442 (2)	1.429 (2)
N11—C11	1.407 (2)	1.426 (2)	1.436 (2)	1.429 (2)
N4—C4	1.318 (3)	1.366 (2)	1.476 (2)	1.352 (4)
C1—C2	1.426 (3)	1.436 (2)	1.403 (2)	1.398 (2)
C1—C6	1.431 (3)	1.437 (2)	1.401 (2)	1.391 (2)
C2—C3	1.358 (3)	1.361 (2)	1.395 (2)	1.376 (2)
C5—C6	1.351 (3)	1.365 (2)	1.385 (2)	1.388 (2)
C3—C4	1.430 (3)	1.442 (2)	1.390 (2)	1.395 (2)
C4—C5	1.431 (3)	1.429 (2)	1.398 (2)	1.396 (3)

Bond	PAZAN·HCl ^{††}	PAZAN-3-NPA ^{‡‡}	PAZAN-4-NPA ^{§§}	PAZAN-5-NPA ^{§§}
N1—N11	1.291 (3)	1.234 (4)	1.224 (4)	1.262 (6)
N1—C1	1.324 (3)	1.442 (5)	1.462 (4)	1.415 (6)
N11—C11	1.417 (3)	1.445 (5)	1.461 (4)	1.428 (7)
N4—C4	1.324 (3)	1.463 (2)	1.463 (4)	1.455 (6)
C1—C2	1.428 (3)	1.412 (6)	1.398 (4)	1.387 (6)
C1—C6	1.419 (3)	1.384 (7)	1.379 (4)	1.395 (6)
C2—C3	1.349 (3)	1.420 (5)	1.393 (4)	1.374 (7)
C5—C6	1.357 (3)	1.369 (4)	1.380 (4)	1.382 (7)
C3—C4	1.425 (3)	1.375 (2)	1.384 (4)	1.391 (6)
C4—C5	1.421 (3)	1.383 (2)	1.382 (4)	1.380 (7)

[†] This work. ^{††} Mahmoudkhani & Langer (2001a). ^{‡‡} § Smith *et al.* (2008).

Compound (III)

Crystal data

C₁₂H₁₁N₃·2C₇H₄N₂O₆

$M_r = 621.48$

Triclinic, $P\bar{1}$

$a = 8.2048$ (7) Å

$b = 8.8859$ (7) Å

$c = 11.5802$ (9) Å

$\alpha = 112.292$ (8)°

$\beta = 92.560$ (7)°

$\gamma = 114.072$ (8)°

$V = 693.09$ (13) Å³

$Z = 1$

Mo $K\alpha$ radiation

$\mu = 0.12$ mm⁻¹

$T = 200$ K

0.30 × 0.30 × 0.15 mm

Data collection

Oxford Gemini-S CCD area-detector diffractometer
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.930, T_{\max} = 0.980$

8021 measured reflections
2440 independent reflections
1989 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.085$

$S = 1.07$

2440 reflections

220 parameters

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\max} = 0.15$ e Å⁻³

$\Delta\rho_{\min} = -0.14$ e Å⁻³

H atoms potentially involved in hydrogen-bonding interactions were located by difference methods and their positional and isotropic displacement parameters were refined. Other H atoms were included at calculated positions, with C—H = 0.93 Å, and treated as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The sulfonate group of the *D* benzene-sulfonate anion in (II) is rotationally disordered, with refined occupancies of the major and minor component O atoms of 0.80 (1) for O11D/O12D/O13D and 0.20 (1) for O14D/O15D/O16D. The atoms of the minor component were refined isotropically.

Data collection: SMART (Bruker, 2000) for (I); *CrysAlis Pro* (Oxford Diffraction, 2009) for (II) and (III). Cell refinement: SMART for (I); *CrysAlis Pro* for (II) and (III). Data reduction: SAINT (Bruker, 1999) for (I); *CrysAlis Pro* for (II) and (III). Program(s) used to solve structure: SHELXS97 (Sheldrick, 2008) for (I); SIR92 (Altomare *et al.*, 1994) for (II); SHELXS97 within WinGX (Farrugia, 1999) for (III). Program(s) used to refine structure: SHELXL97 (Sheldrick, 2008) for (I); SHELXL97 within WinGX for (II) and (III). For all compounds, molecular graphics: PLATON (Spek, 2009); software used to prepare material for publication: PLATON.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GD3306). Services for accessing these data are described at the back of the journal.

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