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2-[*N*-(2,3-Dimethylphenyl)carbamoyl]benzenesulfonamide and the 3,4- and 2,6-dimethylphenyl analogues

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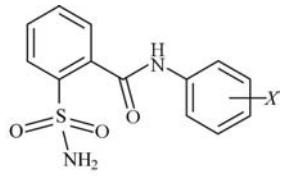
The structures of 2-[(2,3-dimethylphenyl)carbamoyl]benzenesulfonamide, 2-[(3,4-dimethylphenyl)carbamoyl]benzenesulfonamide and 2-[(2,6-dimethylphenyl)carbamoyl]benzenesulfonamide, all $C_{15}H_{16}N_2O_3S$, are stabilized by extensive intra- and intermolecular hydrogen bonds. In all three structures, the sulfonamide and carbamoyl groups are involved in hydrogen bonding. In the 2,3-dimethyl and 2,6-dimethyl derivatives, dimeric units and chains of molecules are formed parallel to the *c* axis. In the 3,4-dimethyl derivative, the hydrogen bonding creates tetrameric units, resulting in macrocyclic $R_4^4(22)$ rings that form sheets in the *ab* plane. The three analogues are closely related to the fenamate class of nonsteroidal anti-inflammatory drugs.

Comment

Saccharin derivatives have always been of interest because of their diverse applications (Marta *et al.*, 2003; Culf *et al.*, 1997). Their open-ring benzenesulfonamide derivatives have shown cyclooxygenase-2 (COX-2) inhibitory action and act as analgesic and anti-inflammatory agents (Eatedal *et al.*, 2002). Various biologically important saccharin skeletons and their *N*-alkyl derivatives have been efficiently prepared (Xu *et al.*, 2006) by chromium oxide-catalyzed oxidation of *N*-alkyl-2-methylarenesulfonamides in acetonitrile as well as by the already developed methodology utilizing irradiation from tungsten and mercury lamps (Masashi *et al.*, 1999) for a similar type of conversion. In continuation of our research on 1,2-benzothiazine 1,1-dioxide and saccharin derivatives (Siddiqui, Ahmad, Khan & Siddiqui, 2007; Siddiqui, Ahmad, Khan *et al.*, 2008; Siddiqui, Ahmad, Siddiqui *et al.*, 2007; Siddiqui, Ahmad, Tariq *et al.*, 2008), we now report the syntheses and crystal structures of the title compounds, 2-[(*X*-dimethylphenyl)-carbamoyl]benzenesulfonamide [*X* = 2,3- for (I), 3,4- for (II) and 2,6- for (III)]. The three dimethylphenyl-substituted analogues are closely related to the fenamate class of nonsteroidal anti-inflammatory drugs and are expected to exhibit very potent biological activities since sulfonamide and carbamoyl functions exist in the same nucleus simultaneously.



(I) *X* = 2,3-dimethyl
(II) *X* = 3,4-dimethyl
(III) *X* = 2,6-dimethyl

The molecular structure of (I) is presented in Fig. 1. The mean planes of the benzene rings (C1–C6 and C8–C13) are inclined at 8.19 (8)° with respect to one another, while the carbamoyl group (O3/N2/C7) is inclined at 55.43 (13) and 48.73 (13)°, respectively, to these rings. Atoms S1 and C7 lie 0.062 (3) and 0.066 (4) Å from the mean plane of the C1–C6 ring, on opposite sides, indicating a significant strain on this portion of the molecule. The structure contains two distinct patterns of hydrogen bonds, involving intermolecular N—H···O interactions (Fig. 2). The sulfonamide groups are hydrogen bonded *via* atoms N1 and O1, forming dimers about inversion centers at $(0, \frac{1}{2}, 0)$ and $(0, 0, \frac{1}{2})$ along the *b* and *c* axes. The eight-membered rings thus formed may be described in graph-set notation as $R_2^2(8)$ (Bernstein *et al.*, 1994). The carbamoyl groups are also involved in hydrogen bonds, involving atoms O3 and N2, resulting in chains of molecules running parallel to the *c* axis and affording stability to the structure. In addition, there is a rather weak nonclassical intermolecular hydrogen bond (C4—H4···O1^{iii}; details of the hydrogen-bonding geometry are provided in Table 1). The structure is further stabilized by three additional intramolecular interactions, *viz.* N1—H1N···O3, C2—H2···O1 and C14—H14*D*···N2, resulting in seven-, five- and five-membered rings representing *S*(7), *S*(5) and *S*(5) motifs, respectively (Bernstein *et al.*, 1994).

The molecular structure of (II) is presented in Fig. 3. The mean planes of the benzene rings (C1–C6 and C8–C13) are

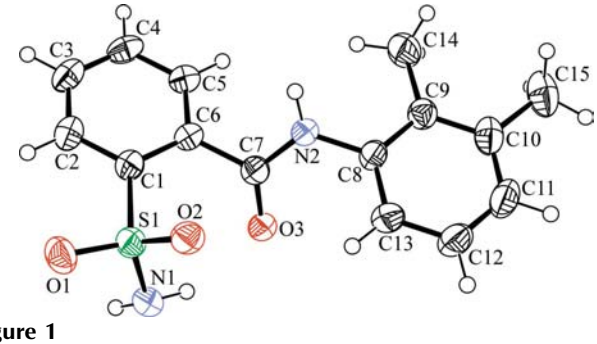


Figure 1
An *ORTEP-3* (Farrugia, 1997) drawing of (I), with displacement ellipsoids plotted at the 50% probability level.

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inclined at 40.55 (8)° with respect to one another, while the carbamoyl group (O1/N2/C7) is inclined at 59.30 (13) and 26.05 (18)°, respectively, to these rings. A comparison of mean-plane angles shows that the conformations of (I) and (II) are significantly different from one another. Molecules of (II) related by translational symmetry along the *a* and *b* axes form a cluster of four molecules *via* N1—H1N···O1^{i} and N2—H3N···O3^{ii} hydrogen bonds (details of the hydrogen-bonding geometry are provided in Table 2), resulting in a macrocyclic ring (Fig. 4) that may be described in graph-set notation as $R_4^4(22)$ (Bernstein *et al.*, 1994). These hydrogen bonds result in the formation of sheets that are extended in the *ab* plane. The structure is further stabilized by three additional intramolecular interactions, *viz.* N1—H2N···O3, C13—H13···O3 and C2—H2···O1, resulting in seven-, six- and five-membered rings representing *S*(7), *S*(6) and *S*(5) motifs, respectively (Bernstein *et al.*, 1994) (Fig. 3 and Table 2).

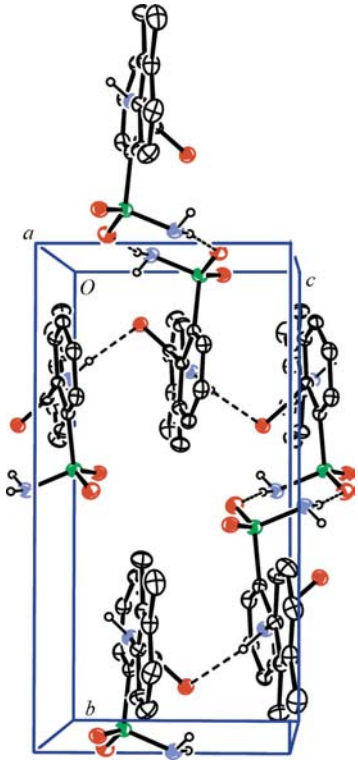

Figure 2
Intermolecular interactions (dashed lines) in the unit cell of (I), showing eight-membered rings formed by sulfonamide groups and chains formed by carbamoyl groups running parallel to the *c* axis.

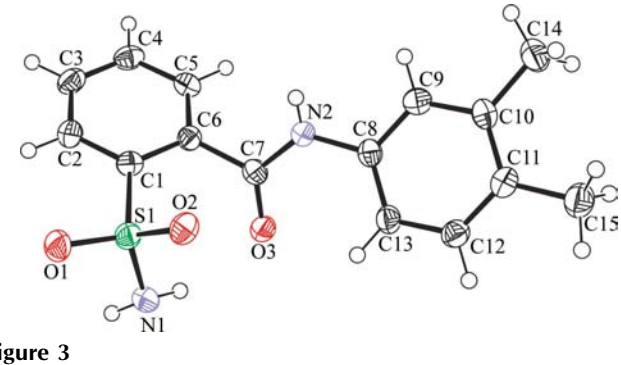

Figure 3
An *ORTEP-3* (Farrugia, 1997) drawing of (II), with displacement ellipsoids plotted at the 50% probability level.

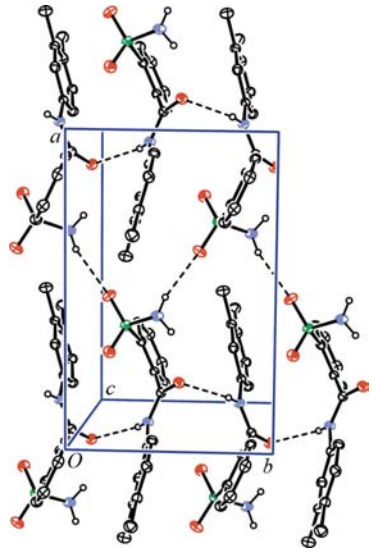

Figure 4
Intermolecular interactions (dashed lines) in the unit cell of (II), showing macrocyclic rings formed by clusters of four molecules; these clusters are further extended into sheets in the *ab* plane.

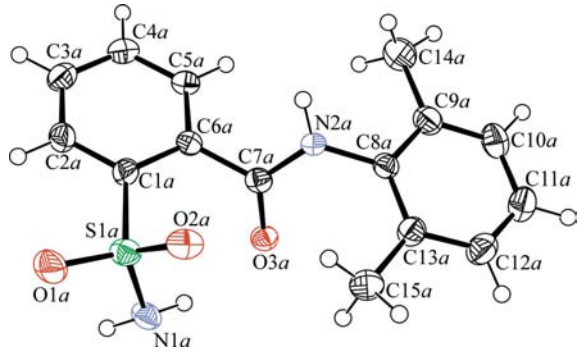

Figure 5
An *ORTEP-3* (Farrugia, 1997) drawing of molecule *A* of (III), with displacement ellipsoids plotted at the 50% probability level.

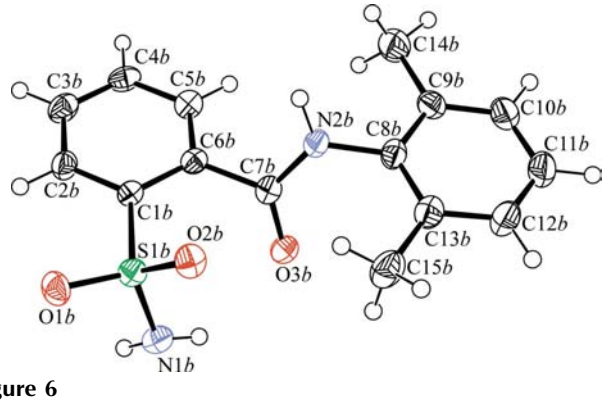

Figure 6
An *ORTEP-3* (Farrugia, 1997) drawing of molecule *B* of (III), with displacement ellipsoids plotted at the 50% probability level.

The asymmtric unit of (III) is composed of two independent molecules (hereafter called *A* and *B*), depicted in Figs. 5 and 6, respectively. In molecule *A*, the mean planes of the benzene rings (C1*a*–C6*a* and C8*a*–C13*a*) are inclined at 6.53 (9)° with respect to one another, while the carbamoyl group (O3*a*/N2*a*/C7*a*) is inclined at 60.34 (16) and 57.20 (17)°, respectively, to these rings. The corresponding mean-plane angles in molecule *B* are 3.11 (10), 61.17 (16) and 59.10 (17)°, respectively. The conformations of both molecules of (III) are more closely related to the conformation of (I). The sulfonamide groups of the two molecules in (III) are hydrogen bonded to form dimeric units; the eight-membered rings thus formed represent $R_2^2(8)$ motifs (Bernstein *et al.*, 1994). The carbamoyl groups of molecules *A* and *B* are hydrogen bonded to form chains of molecules running parallel to the *a* axis (Fig. 7). The two molecules contain an identical pair of intramolecular interactions, *viz.* N1*a*/*b*—H···O3*a*/*b* and C2*a*/*b*—H2*a*/*b*···O1*a*/*b*, resulting in seven- and five-membered rings representing *S*(7) and *S*(5) motifs, respectively (Bernstein *et al.*, 1994). However, the intramolecular interactions involving hydrogen bonding to atom C15 show markedly different patterns in the two molecules; in molecule *A*, atom C15 is bonded to O3*a*, while in molecule *B*, it is bonded to N2*b*, resulting in *S*(7) and *S*(5) motifs, respectively.

The molecular dimensions in all three structures are in agreement with the corresponding dimensions reported for similar structures (Clark *et al.*, 2003; Vyas *et al.*, 2003; Singh *et al.*, 2004; Bocelli *et al.*, 1995; Sutton & Cody, 1989; Furuya *et al.*, 1989; Siddiqui, Ahmad, Khan *et al.*, 2008; Siddiqui, Ahmad, Tariq *et al.*, 2008), with S═O, S—N, S—C, N2—C7, N2—C8 and C═O distances lying in very close ranges of 1.430 (2)–1.438 (2), 1.598 (3)–1.614 (3), 1.773 (3)–1.781 (3), 1.331 (4)–1.338 (4), 1.429 (4)–1.440 (4) and 1.236 (3)–1.241 (3) Å, respectively. The C—N—C angle at N2 in (II) is significantly larger than the corresponding angles in (I), (III*A*) and (III*B*). The remaining angles lie within narrow ranges in all three structures.

In all molecules, the conformation about the S—N bond is in agreement with the conformation of a handful of structures containing an *o*-C-substituted benzenesulfonamide fragment [Cambridge Structural Database (CSD), Version 5.29; Allen, 2002]. The N1 atoms in all these structures are tetrahedral, the sum of angles around this atom being in the range 334–340°. The H atoms bonded to atom N1, and atoms O1 and O2 bonded to S1, are staggered, as observed in chlorophenyl analogues of the title compounds (Siddiqui, Ahmad, Khan *et al.*, 2008) and the compound with CSD refcode COYVER (Foresti *et al.*, 1985). Several structures have been reported wherein the H and O atoms of the sulfonamide unit are eclipsed, *e.g.* CSD refcodes ENIROI (Vyas *et al.*, 2003), GUFQED01 (Clark *et al.*, 2003) and ZZZULS01 (Tremayne *et al.*, 2002). Classical work on the three-dimensional orientation of sulfonamides has been reported by several groups of investigators (*e.g.* Bordner *et al.*, 1984, 1989; Beddoes *et al.*, 1986; Street *et al.*, 1987; Luger *et al.*, 1996; Helliwell *et al.*, 1997; Bhatt *et al.*, 2005).

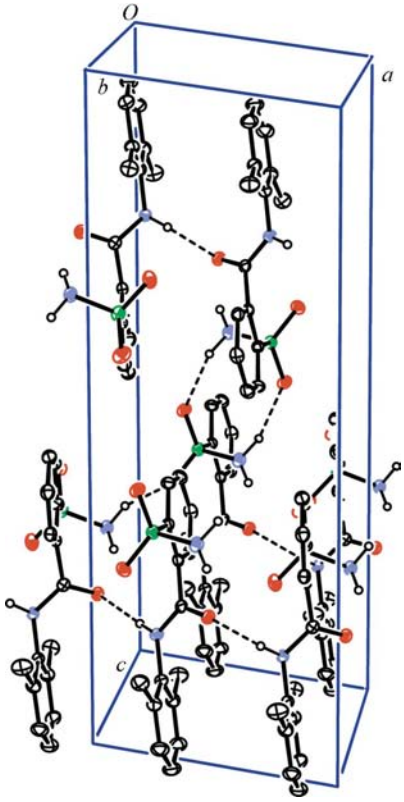

Figure 7
Intermolecular interactions (dashed lines) in the unit cell of (III), showing eight-membered rings formed by sulfonamide groups and chains formed by carbamoyl groups running parallel to the *a* axis.

Experimental

Suspensions of saccharin (1.0 g, 5.46 mmol) and dimethylaniline (5 ml in the case of 2,3- and 2,6-dimethylaniline, and 0.5 g in the case of 3,4-dimethylaniline) in xylene (25 ml) were stirred at room temperature for 1.5 h and then heated at 373 K for 2–7 h. The reaction mixtures were subsequently cooled to room temperature, filtered and dried to obtain colorless solid products. The products were crystallized from MeOH/CH_3CN (1:3) solutions by slow evaporation at 313 K.

For (I): m.p. 463–465 K. IR (neat, ν_{max}, cm^{-1}): NH_2 3415, 3325; CO 1650; SO_2 1343, 1150; ^{1}H NMR (300 MHz, methanol-d_4): δ 2.30 (*s*, 3H, CH_3), 2.35 (*s*, 3H, CH_3), 7.12–7.34 (*m*, 3H, C_6H_3), 7.63–8.10 (*m*, 4H, C_6H_4); ^{13}C NMR: δ 169.7, 142.2, 138.8, 137.3, 135.4, 134.3, 132.2, 131.7, 130.5, 129.5, 124.2, 120.5, 21.4, 20.8. LRMS (ES^+): *m*/*z*: 304.09 [M^+] (39.7%).

For (II): m.p. 443–444 K. IR (neat, ν_{max}, cm^{-1}): NH_2 3425, 3365; CO 1705; SO_2 1354, 1167; ^{1}H NMR (300 MHz, methanol-d_4): δ 2.30 (*s*, 3H, CH_3), 2.35 (*s*, 3H, CH_3), 7.12–7.32 (*m*, 2H, C_6H_3), 7.53–7.70 (*m*, 4H, C_6H_4), 8. 20 (*m*, 1H, C_6H_3); ^{13}C NMR: δ 171.8, 144.2, 140.9, 139.2, 137.4, 136.3, 134.2, 133.7, 132.5, 131.6, 126.3, 122.5, 23.5, 22.9. LRMS (ES^+): *m*/*z*: 304.09 [M^+] (25.1%).

For (III): m.p. 496–497 K. IR (neat, ν_{max}, cm^{-1}): NH_2 3423, 3345; CO 1715; SO_2 1345, 1150; ^{1}H NMR (300 MHz, methanol-d_4): δ 2.30 (*s*, 3H, CH_3), 2.42 (*s*, 3H, CH_3), 7.26–7.52 (*m*, 3H, C_6H_3), 7.68–8.10 (*m*, 4H, C_6H_4); ^{13}C NMR: δ 168.7, 141.2, 139.7, 139.2, 137.4, 136.3, 134.2, 133.7, 132.5, 130.4, 125.3, 121.1, 22.3, 21.1. LRMS (ES^+): *m*/*z*: 304.09 [M^+] (21.9%).

Compound (I)

Crystal data

$C_{15}H_{16}N_2O_3S$
M_r = 304.36
Monoclinic, $P2_1/c$
a = 12.842 (7) Å
b = 15.153 (9) Å
c = 7.572 (4) Å
β = 91.16 (4)°
V = 1473.2 (14) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.23 mm^{-1}
T = 173 (2) K
0.40 × 0.04 × 0.02 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.913, T_{max} = 0.995
9490 measured reflections
2583 independent reflections
1926 reflections with $I > 2\sigma(I)$
R_{int} = 0.059

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.043
$wR(F^2)$ = 0.114
S = 1.09
2583 reflections
200 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.26 e Å^{-3}
$\Delta\rho_{min}$ = −0.52 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H3N···O3^{i}	0.87 (3)	2.08 (3)	2.941 (3)	170 (2)
N1—H2N···O1^{ii}	0.84 (3)	2.13 (3)	2.963 (3)	171 (3)
N1—H1N···O3	0.92 (3)	2.04 (3)	2.850 (3)	146 (2)
C4—H4···O1^{iii}	0.95	2.53	3.268 (3)	134

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

Compound (II)

Crystal data

$C_{15}H_{16}N_2O_3S$
M_r = 304.36
Monoclinic, $P2_1/c$
a = 11.597 (9) Å
b = 7.450 (3) Å
c = 16.324 (13) Å
β = 97.29 (3)°
V = 1399.0 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 173 (2) K
0.20 × 0.10 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.953, T_{max} = 0.986
4440 measured reflections
2423 independent reflections
1767 reflections with $I > 2\sigma(I)$
R_{int} = 0.048

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.154
S = 1.05
2423 reflections
201 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.31 e Å^{-3}
$\Delta\rho_{min}$ = −0.41 e Å^{-3}

Table 2
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···O1^{i}	0.85 (4)	2.35 (4)	3.131 (4)	153 (3)
N2—H3N···O3^{ii}	0.76 (3)	2.32 (3)	3.022 (3)	155 (3)
N1—H2N···O3	0.85 (4)	2.14 (4)	2.888 (4)	146 (3)

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

Table 3
Hydrogen-bond geometry (Å, °) for (III).

D—H···A	D—H	H···A	D···A	D—H···A
N1a—H1Na···O2b^{i}	0.91 (3)	2.17 (3)	2.891 (4)	135 (3)
N2a—H3Na···O3b^{ii}	0.90 (3)	2.08 (3)	2.971 (3)	174 (3)
N1b—H1Nb···O1a^{iii}	0.81 (3)	2.58 (3)	2.996 (3)	114 (3)
N1b—H2Nb···O1b^{ii}	0.95 (3)	2.04 (3)	2.941 (4)	157 (3)
N2b—H3Nb···O3a^{iv}	0.90 (3)	2.03 (3)	2.921 (4)	170 (3)
N1a—H2Na···O3a	0.91 (3)	2.04 (3)	2.863 (4)	151 (3)
N1b—H1Nb···O3b	0.81 (3)	2.33 (4)	2.993 (4)	139 (3)

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

Compound (III)

Crystal data

$C_{15}H_{16}N_2O_3S$
M_r = 304.36
Triclinic, $P\overline{1}$
a = 7.968 (3) Å
b = 8.509 (5) Å
c = 21.510 (11) Å
α = 84.30 (2)°
β = 82.46 (3)°
γ = 88.66 (3)°
V = 1438.5 (12) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.24 mm^{-1}
T = 173 (2) K
0.12 × 0.07 × 0.06 mm

Data collection

Nonius KappaCCD diffractometer
Absorption correction: multi-scan (*SORTAV*; Blessing, 1997)
T_{min} = 0.972, T_{max} = 0.986
9182 measured reflections
5044 independent reflections
3197 reflections with $I > 2\sigma(I)$
R_{int} = 0.062

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.134
S = 1.02
5044 reflections
401 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

For the three title structures, H atoms bonded to C atoms were included in the refinements at geometrically idealized positions with aromatic and methyl C—H distances of 0.95 and 0.98 Å, respectively, and U_{iso}(H) values of 1.2 times U_{eq} of the atoms to which they were bonded; the H atoms bonded to atom C14 in (I) were equally disordered over six sites. H atoms bonded to N atoms were allowed to refine with U_{iso}(H) values of 1.2 times U_{eq} of the N atoms. The final difference maps were free of chemically significant features.

For all three compounds, data collection: *COLLECT* (Hooft, 1998); cell refinement: *DENZO* (Otwinowski & Minor, 1997); data reduction: *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SAPI91* (Fan, 1991); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: HJ3076). Services for accessing these data are described at the back of the journal.

References

Allen, F. H. (2002). *Acta Cryst.* B**58**, 380–388.
Beddoes, R. L., Dalton, L., Joule, J. A., Mills, O. S., Street, J. D. & Watt, C. I. F. (1986). *J. Chem. Soc. Perkin Trans. 2*, pp. 787–797.
Bernstein, J., Etter, M. C. & Leiserowitz, L. (1994). *Structure Correlation*, Vol. 2, edited by H.-B. Bürgi & J. D. Dunitz, pp. 431–507. New York: VCH.

Bhatt, P. M., Ravindra, N. V., Banerjee, R. & Desiraju, R. (2005). *Chem. Commun.* pp. 1073–1075.
Blessing, R. H. (1997). *J. Appl. Cryst.* **30**, 421–426.
Bocelli, G., Cantoni, A., Simonov, Y. A., Fonari, M. S. & Ganin, E. V. (1995). *J. Inclusion Phenom. Mol. Recognit. Chem.* **20**, 105–114.
Bordner, J., Hammen, P. D. & Whipple, E. B. (1989). *J. Am. Chem. Soc.* **111**, 6572–6578.
Bordner, J., Richards, J. A., Weeks, P. & Whipple, E. B. (1984). *Acta Cryst.* C**40**, 989–990.
Clark, J. C., McLaughlin, M. L. & Fronczek, F. R. (2003). *Acta Cryst.* E**59**, o2005–o2006.
Culf, A. S., Gerig, J. T. & Willias, P. G. (1997). *J. Biomol. NMR*, **10**, 293–299.
Eatedal, H. A., Osama, I. E., Shaker, Y. & Sameh, M. E. (2002). *Monatsh. Chem.* **133**, 255–266.
Fan, H.-F. (1991). *SAPI91*. Rigaku Corporation, Tokyo, Japan.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Foresti, E., Riva di Sanseverino, L. & Sabatino, P. (1985). *Acta Cryst.* C**41**, 240–243.
Furuya, T., Fujita, S. & Fujikura, T. (1989). *Anal Sci.* **5**, 489–490.
Helliwell, M., Zhao, Y. & Joule, J. A. (1997). *Acta Cryst.* C**53**, 884–886.
Hooft, R. (1998). *COLLECT*. Nonius BV, Delft, The Netherlands.
Luger, P., Daneck, K., Engel, W., Trummlitz, G. & Wagner, K. (1996). *Eur. J. Pharm. Sci.* **4**, 175–187.
Marta, G. A., Gloria, A., Joaquin, B., Marguerite, P. & Bernard, M. (2003). *J. Biol. Inorg. Chem.* **8**, 644–652.
Masashi, K., Hideo, T., Kentaro, Y. & Masataka, Y. (1999). *Tetrahedron*, **55**, 14885–14900.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr & R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Siddiqui, W. A., Ahmad, S., Khan, I. U. & Siddiqui, H. L. (2007). *Synth. Commun.* **37**, 767–773.
Siddiqui, W. A., Ahmad, S., Khan, I. U., Siddiqui, H. L. & Parvez, M. (2008). *Acta Cryst.* C**64**, o286–o289.
Siddiqui, W. A., Ahmad, S., Siddiqui, H. L., Tariq, M. I. & Parvez, M. (2007). *Acta Cryst.* E**63**, o4117.
Siddiqui, W. A., Ahmad, S., Tariq, M. I., Siddiqui, H. L. & Parvez, M. (2008). *Acta Cryst.* C**64**, o4–o6.
Singh, S. K., Reddy, M. S., Shivaramakrishna, S., Kavitha, D., Vasudev, R., Babu, J. M., Sivalakshmidevi, A. & Rao, Y. K. (2004). *Tetrahedron Lett.* **45**, 7679–7682.
Street, J. D., Harris, M., Bishop, D. I., Heatley, F., Beddoes, R. L., Mills, O. S. & Joule, J. A. (1987). *J. Chem. Soc. Perkin Trans. 1*, pp. 1599–1606.
Sutton, P. A. & Cody, V. (1989). *Acta Cryst.* C**45**, 757–760.
Tremayne, M., Seaton, C. C. & Glidewell, C. (2002). *Acta Cryst.* B**58**, 823–834.
Vyas, K., Sivalakshmidevi, A., Singh, S. K., Koteswar Rao, Y. & Om Reddy, G. (2003). *Acta Cryst.* E**59**, o1731–o1732.
Xu, L., Shu, H., Liu, Y., Zhang, S. & Trudell, M. (2006). *Tetrahedron*, **62**, 7902–7910.