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7-Amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid as the dimethylformamide and water monosolvates at 293 K

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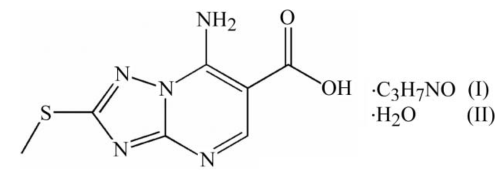
The molecular structure of 7-amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid is reported in two crystal environments, *viz*. as the dimethylformamide (DMF) monosolvate, $C_7H_7N_5O_2S \cdot C_3H_7NO$, (I), and as the monohydrate, $C_7H_7N_5O_2S \cdot H_2O$, (II), both at 293 (2) K. The triazolo[1,5-*a*]pyrimidine molecule is of interest with respect to the possible biological activity of its coordination compounds. While the DMF solvate exhibits a layered structural arrangement through N···O hydrogen-bonding interactions, the monohydrate displays a network of intermolecular O···O and N···O hydrogen bonds assisted by cocrystallized water molecules and weak π–π stacking interactions, leading to a different three-dimensional supramolecular architecture. Based on results from topological analyses of the electron-density distribution in X—H···O (X = O, N and C) regions, hydrogen-bonding energies have been estimated from structural information only, enabling the characterization of hydrogen-bond graph energies.

Comment

Given the similarity between the skeleton of 7-amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid and the purine ring, 1,2,4-triazolo[1,5-*a*]pyrimidines can be considered as model systems for various coordination compounds that exist in nature (Salas *et al.*, 1999). In addition, 1,2,4-triazolo[1,5-*a*]pyrimidines are known to possess a variety of biological properties (Ram, 1988), antitumour activity being one of the most important. 7-Amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid is an amino acid with carboxylic acid and amine groups in positions 7 and 6, respectively, and an S—CH_3 group in position 2 (Figs. 1 and 2 for numbering scheme). The presence of all these groups makes the molecule a more versatile ligand than the triazolopyrimidines already known in the literature (Salas *et al.*, 1999; Haasnoot, 2000). Here, the 7-amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid molecule crystallizes with either dimethylformamide (DMF) [(I)] or water [(II)] molecules, in the monoclinic system (space group $P2_1/c$). Crystal structure determinations were carried out for (I) and (II) at room temperature. In both cases, the asymmetric unit contains one 1,2,4-triazolo[1,5-*a*]pyrimidine molecule and one solvent molecule.



The molecular structures of (I) and (II) are shown in Figs. 1 and 2, respectively. It is known that the association of triazole and pyrimidine rings involves a loss of aromaticity in the latter

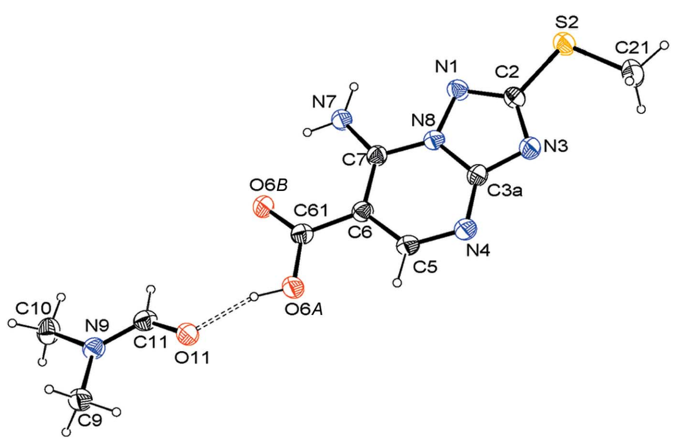


Figure 1
The asymmetric unit of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The double-dashed line indicates a hydrogen bond.

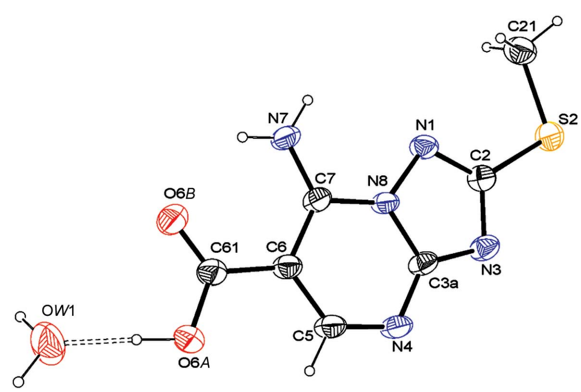


Figure 2
The asymmetric unit of (II), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The double-dashed line indicates a hydrogen bond.

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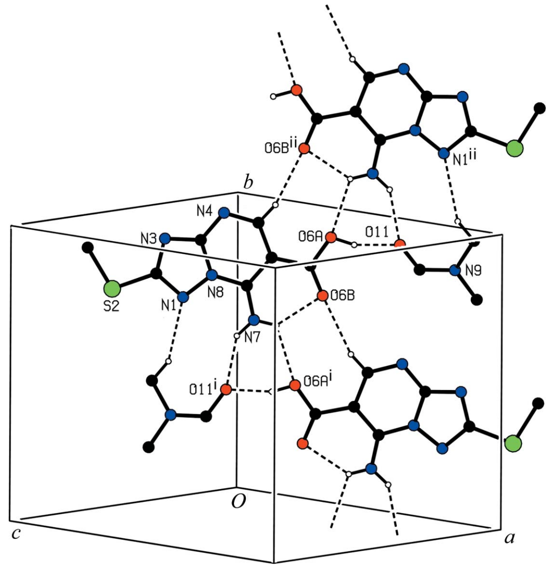


Figure 3
A partial packing view, showing the hydrogen-bond network of (I). Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen-bonding interactions have been omitted for clarity. [Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 1, y + \frac{1}{2}, -z + \frac{1}{2}$.]

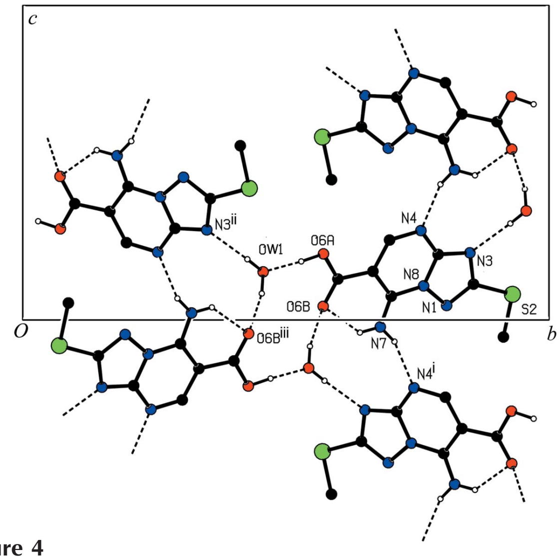


Figure 4
A partial packing view of (II) along the crystallographic *a* axis. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen-bonding interactions have been omitted for clarity. [Symmetry codes: (i) $x - 1, -y + \frac{3}{2}, z - \frac{1}{2}$; (ii) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (iii) $-x + 1, -y + 1, -z$.]

(Boulanger *et al.*, 1988; Surdykowski *et al.*, 1999). This is indeed observed in (I) and (II), as indicated by the increase in the bond lengths C6—C5 [1.404 (3) and 1.3992 (19) Å, respectively, in (I) and (II)] and N8—C3a [1.376 (3) and 1.3782 (17) Å] with respect to the characteristic distances observed in nonconjugated aryl-substituted pyrimidine compounds (1.377 and 1.333 Å, respectively; Surdykowski *et al.*, 1999). In both 1,2,4-triazolo[1,5-*a*]pyrimidine molecules, the carboxyl group adopts a synplanar conformation (Leiserowitz, 1976), as shown by the lengths of the C═O [1.227 (3) and 1.2209 (18) Å] and C—O(H) [1.322 (3) and 1.3174 (18) Å] bonds and the O═C—O bond angle [122.6 (2) and 122.79 (15)°]. The thiolic CH_3 and the NH_2 group are parallel in both molecular structures. However, while in (I) they point opposite to each other, in (II) they point in the same direction, as shown by the C21—S2—C2—N1 torsion angles of 177.1 (2) and 1.2 (2)° in (I) and (II), respectively. Except for the orientation of the thiolic CH_3 group, the molecular structures of the 1,2,4-triazolo[1,5-*a*]pyrimidine molecules are almost equivalent within both crystal environments as far as angles and bond distances are concerned. In addition, the molecule exhibits a planar conformation in both cases [r.m.s. deviations = 0.0315 and 0.0202 Å for (I) and (II), respectively, when all non-H atoms are used in the calculation]. It is known that the presence of substituents in a triazolopyrimidine ring does not lead to radical changes in bond distances (Lokaj *et al.*, 2006). However, we can make some observations (see Table 5). When the pyridine is substituted in the *meta* position by electron-donating groups, the C6—C7 bond length is shorter than that of a typical C—C aromatic bond. This also happens if an electron donor or acceptor is in the *ortho* position. The introduction of a group in position 2 does not change the electronic distribution in the pyridine. In our case, we have two substituents in the *ortho* position, namely the amino group and the carboxyl. It follows that the bond distances C5—C6 and C6—C7 are close to each other, owing to the synergy of the two groups.

The different orientations of the $-SCH_3$ group found in the crystal structures of (I) and (II) together with distinct hydrogen-bonding networks (see Tables 2 and 4) formed upon the influence of the cocrystallized solvent molecules, lead to strongly different supramolecular architectures. Thus, in (I), each amino group is involved in three N···O hydrogen bonds (Table 2): one intramolecular hydrogen bond with the carbonyl group (N7···O6*B*) and two intermolecular bonds, one with a carboxyl group of a neighbouring molecule (N7···O6*A*^{i}) and one with the DMF carbonyl group (N7···O11^{i}). The DMF molecule also forms hydrogen bonds with an adjacent carboxyl group (O6*A*···O11). These interactions lead to a two-dimensional hydrogen-bonding pattern, as displayed in Fig. 3. Short contacts in the structure are also generated by C5^{i}···O6 interactions [3.340 (2) Å] within the layer, and by C9···π(C5) [3.467 (2) Å], S···π(C7) [3.488 (2) Å] and π(C5)···π(C5) [3.326 (3) Å] interactions between layers. In (II), atoms N3 and N4 are involved in hydrogen bonds with one water molecule (O*W*1···N3^{ii}) and one neighbouring amino group (N7···N4^{i}) (Table 4). The water molecule further participates in a hydrogen bond with a carboxyl O atom to provide the three-dimensional structure (Fig. 4). The backbone hydrogen-bonding network is completed with an intramolecular N7···O6*B* interaction (Table 4). Additional short lattice contacts are assisted by S···S [3.6125 (5) Å] and π–π stacking [3.381 (2) Å between the mean-square planes of two neighbouring molecules] interactions, which form columns of parallel molecules stacked in a head-to-head arrangement along the crystallographic *a* axis. The π–π interactions involve the triazole ring of one molecule and the pyrimidine ring of a neighbouring molecule related by the ($x - 1, y, z$) symmetry element, with a separation of 3.5150 (9) Å between the corresponding ring centroids; the angle between the ring planes is as small as 0.88 (5)°.

The hydrogen-bond patterns observed in both crystal structures can be described using graph-set theory (Etter, 1990). In addition, based on topological analyses of the electron-density distribution characterized experimentally for 83 X—H···O (X = C, N and O) hydrogen-bonding interactions (Espinosa *et al.*, 1998), graph and molecular binding energies involving H···O interactions can be estimated from the H···O distances. Indeed, supported by previous results on ice (Espinosa & Molins, 2000), the energy associated with each interaction can be estimated from the function $E_i \simeq 25000\exp(-3.6d_{H\cdots O})$, where E_i and $d_{H\cdots O}$ are in kJ mol^{-1} and Å, respectively. As the H-atom positions have here been derived from X-ray data, they are expected to be systematically too close to their bound atom, therefore leading to an overestimation of the H···O distance with a concomitant underestimation of the corresponding E_i value. Thus, to calculate E_i we have shifted the H-atom positions along their bond direction according to the mean X—H bond lengths observed from neutron diffraction analyses: (O═Csp^2—)O—H = 1.018 Å, (C_{ar}—)N—H_2 = 1.011 Å, C_{ar}—H = 1.083 Å, (Z—)Csp^3—H_3 = 1.077 Å (Allen & Bruno, 2010) and OW—H = 0.97 Å (Espinosa *et al.*, 1996).

In (I), the two-dimensional arrangement of molecules, which is parallel to the (101) plane, involves graph sets $R_3^2(6)$ (after normalization of the H-atom positions, the actual hydrogen-bond H···O distances are 1.59, 1.97 and 2.29 Å), $R_3^2(7)$ (1.96, 2.29 and 2.29 Å), $R_2^2(10)$ (1.97 and 2.46 Å) and $S(6)$ (1.96 Å) (Fig. 3). The $R_3^2(6)$ motif shares one interaction with $R_2^2(7)$ and another with $R_2^2(10)$. Within a graph, the addition of the interaction energies corresponding to the hydrogen bonds building the motif leads to the total interaction energy of the pattern embedded in the crystal structure ($E_{graph} = \Sigma_j E_{ij}$). For $R_3^2(6)$, $R_2^2(7)$ and $S(6)$, the estimated E_{graph} values are 109.0, 34.7 and 21.6 kJ mol^{-1}, respectively. The hydrogen-bond energy component of the total molecular binding energy of 7-amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid within the crystal structure of (I) is roughly 116 kJ mol^{-1}, this result being obtained by adding the contributions of the four main intermolecular H···O hydrogen-bond interactions less than 2.45 Å (1.59, 1.97, 2.29 and 2.29 Å).

In the case of (II), $S(6)$, $R_4^3(10)$ and $R_4^4(12)$ graphs are exhibited (Fig. 4). The last of these involves four hydrogen-bond interactions (two symmetry-equivalent interactions corresponding to each hydrogen-bond distance H···O = 1.57 and 1.90 Å, after replacement of H atoms) and links asymmetric units that lie in parallel planes. Here, the estimated E_{graph} value is 229 kJ mol^{-1}. The intramolecular hydrogen-bond interaction is shared by the $S(6)$ and $R_4^3(10)$ graphs. The hydrogen-bond distance (H···O = 1.96 Å, after normalization of the H-atom position) is equivalent to that found in (I), therefore leading to the same estimated $S(6)$ graph energy (21.6 kJ mol^{-1}). The main hydrogen-bond interactions contributing to the molecular binding energy of 7-amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid within (II) belong to the $R_4^3(10)$ and $R_4^4(12)$ graphs and involve H···O (1.57 and 1.90 Å) and H···N (1.94 and 1.98 Å) contacts, the first two leading to an energetic contribution of roughly 115 kJ mol^{-1}.

Even though the method used here to derive E_i is expected to give approximate values only, it can be used to extract tendencies. For molecules involving X—H···O (X = C, N and O) hydrogen-bonding interactions, the dependence of E_i on H···O quickly enables the characterization of graph energies and the estimation of hydrogen-bond contributions to molecular binding energies from structural information only. In particular, the method could be applied to classify graphs, or higher hierarchical patterns formed by the addition of several graphs, exhibiting the same number of hydrogen bonds.

Experimental

7-Amino-2-methylsulfanyl-1,2,4-triazolo[1,5-*a*]pyrimidine-6-carboxylic acid was a Maybridge (UK) product sold by C. Erba. Crystals of (I) were obtained by slow evaporation of a DMF/HCOOH (1:1 *v*/*v*) solution at 277 K, while crystals of (II) were grown by the liquid–liquid diffusion method in dimethyl sulfoxide (DMSO)/water at room temperature. In both cases, the crystals were colourless blocks. Good quality crystals of dimensions 0.25 × 0.25 × 0.10 mm [for (I)] and 0.28 × 0.16 × 0.05 mm [for (II)] were used for the X-ray diffraction experiments. IR spectra (Nujol mulls, CsI windows) were recorded with a Perkin–Elmer Spectrum One FT–IR spectrometer. ^{1}H and ^{13}C NMR spectra, in DMSO-d_6, were recorded with a Bruker AC 300 Avance instrument operating at 300 and 75 MHz, respectively. On the basis of spectroscopic studies on anthranilic acid (Samsonowicz *et al.*, 2005), we report the following IR assignments (ν, cm^{-1}): 3498 [*s*, $\nu_{as}(NH_2)$], 3333 [*s*, $\nu_{sy}(NH_2)$], 1698 [*vs*, ν(CO)], 1336 [*vs*, ν(C—NH_2)], 1256 [*vs*, ν(C—OH)]. NMR assignments were tried by COSY, HMBC, HSCQ and DEPT two-dimensional spectra. ^{1}H NMR (ligand skeleton, DMSO-d_6): δ 8.65, 8.96 (*d*, 2H, –NH_2), 8.81 (*s*, 1H, arom. CH), 2.73 (*s*, 3H, S—CH_3); ^{13}C NMR (ligand skeleton, DMSO-d_6): δ 168.27 (COOH), 168.03 (C2), 94.67 (C3*a*), 156.75 (C5), 157.63 (C6), 149.71 (C7).

Compound (I)

Crystal data

$C_7H_7N_5O_2S\cdot C_3H_7NO$	V = 1336.52 (12) Å^3
M_r = 298.33	Z = 4
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
a = 12.1365 (6) Å	μ = 0.26 mm^{-1}
b = 11.2180 (7) Å	T = 293 K
c = 10.3559 (5) Å	0.25 × 0.25 × 0.10 mm
β = 108.570 (3)°	

Data collection

Nonius KappaCCD area-detector diffractometer	3385 independent reflections
24197 measured reflections	2605 reflections with $I > 2\sigma(I)$
	R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054	184 parameters
$wR(F^2)$ = 0.120	H-atom parameters constrained
S = 1.16	$\Delta\rho_{max}$ = 0.31 e Å^{-3}
3385 reflections	$\Delta\rho_{min}$ = −0.31 e Å^{-3}

Table 1
Selected bond lengths (Å) for (I).

N1—C2	1.330 (3)	N4—C3a	1.350 (3)
N1—N8	1.370 (2)	N8—C7	1.360 (3)
N3—C3a	1.337 (3)	N8—C3a	1.376 (3)
N3—C2	1.357 (3)	C5—C6	1.404 (3)
N4—C5	1.333 (3)	C6—C7	1.408 (3)

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

D—H···A	D—H	H···A	D···A	D—H···A
O6A—H6A···O11	0.91	1.71	2.584 (2)	162
N7—H7A···O11^{i}	0.88	2.11	2.870 (2)	145
N7—H7B···O6B	0.86	2.06	2.718 (2)	132
N7—H7B···O6A^{i}	0.86	2.36	2.899 (2)	121
C5—H5···O6B^{ii}	0.93	2.44	3.340 (3)	163
C9—H9B···N1^{ii}	0.96	2.59	3.462 (3)	152

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

Table 3
Selected bond lengths (Å) for (II).

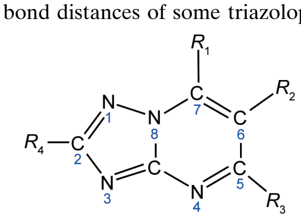
N1—C2	1.3255 (19)	N4—C3a	1.3442 (19)
N1—N8	1.3773 (17)	N8—C7	1.3619 (19)
N3—C3a	1.323 (2)	N8—C3a	1.3782 (17)
N3—C2	1.3624 (18)	C5—C6	1.3992 (19)
N4—C5	1.319 (2)	C6—C7	1.415 (2)

Table 4
Hydrogen-bond geometry (Å, °) for (II).

D—H···A	D—H	H···A	D···A	D—H···A
O6A—H6A···OW1	0.97	1.62	2.5880 (19)	176
N7—H7A···N4^{i}	0.89	2.06	2.9032 (18)	159
N7—H7B···O6B	0.85	2.08	2.7117 (19)	131
OW1—HW1···N3^{ii}	0.88	2.06	2.9371 (18)	173
OW1—HW2···O6B^{iii}	0.95	1.92	2.849 (2)	167

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

Table 5
C5–C6 and C6–C7 bond distances of some triazolopyrimidines.



R_1	R_2	R_3	R_4	C5—C6	C6—C7	Reference
–NH_2	–COOH	–H	–SCH_3	1.404 (3)	1.408 (3)	(I)
–NH_2	–COOH	–H	–SCH_3	1.3992 (19)	1.415 (2)	(II)
–Me	–H	–Me	–H	1.408–1.414	1.349–1.351	(*a*)
–Ph	–H	–Ph	–H	1.427	1.370	(*b*)
–Me	–H	–Me	–NHPh	1.400	1.355	(*c*)
–Me	–H	–Ph	–SCH_2Ph	1.396	1.374	(*d*)
–Me	–H	–NHPh	–SCH_3	1.400	1.377	(*e*)
–Me	–CN	–H	–H	1.414	1.372	(*f*)
–NH_2	–CN	–Ph	–H	1.416	1.393	(*g*)
–NHPh	–NO_2	–CH_3	–H	1.414	1.400	(*h*)

References: (*a*) Odabaşoğlu & Büyükgüngör (2006); (*b*) Surdykowski *et al.* (1999); (*c*) Vas'kevich *et al.* (2006); (*d*) Kleschick & Bordner (1989); (*e*) Britsun *et al.* (2006); (*f*) Lokaj *et al.* (2006); (*g*) Wendt *et al.* (2007); (*h*) Rusinov *et al.* (2007).

Compound (II)

Crystal data

$C_7H_7N_5O_2S{\cdot}H_2O$
M_r = 243.26
Monoclinic, $P2_1/c$
a = 4.0134 (3) Å
b = 20.3287 (12) Å
c = 12.6897 (7) Å
β = 107.039 (6)°
V = 989.87 (11) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.33 mm^{-1}
T = 293 K
0.28 × 0.16 × 0.05 mm

Data collection

Oxford Supernova CCD area-detector diffractometer
6449 measured reflections
2242 independent reflections
1816 reflections with $I > 2\sigma(I)$
R_{int} = 0.026

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.108
S = 1.11
2242 reflections
146 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.39 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

H atoms were located from difference Fourier syntheses and fixed (N—H and O—H) or placed in calculated positions using a riding model (C—H = 0.93–0.96 Å), and with $U_{iso}(H) = kU_{eq}$(parent atom), where k = 1.5 for Csp^3—H and O—H bonds, and 1.2 for the rest.

Data collection: *COLLECT* (Nonius, 1998) for (I); *CrysAlis CCD* (Oxford Diffraction, 2004) for (II). Cell refinement: *COLLECT* for (I); *CrysAlis RED* (Oxford Diffraction, 2004) for (II). Data reduction: *HKL* (Otwinowski & Minor, 1997) for (I); *CrysAlis RED* for (II). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993) for (I); *SIR2004* (Burla *et al.*, 2005) for (II). For both compounds, program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996), *ORTEP-3 for Windows* (Farrugia, 1997) and *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DN3145). Services for accessing these data are described at the back of the journal.

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