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Key indicators

Single-crystal X-ray study
 $T = 170$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.037
 wR factor = 0.094
Data-to-parameter ratio = 16.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

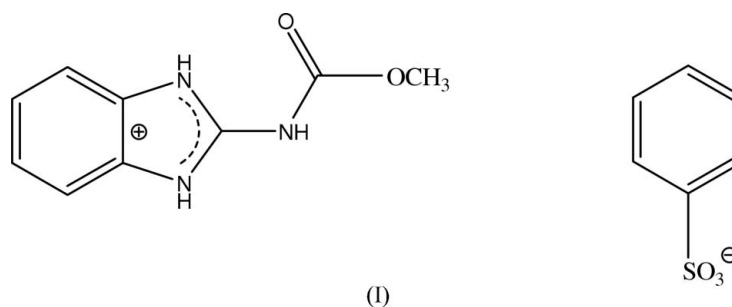
The besylate salt of carbendazim: 2-(methoxy-carbonylamino)benzimidazolium benzenesulfonate

In the title compound, $\text{C}_9\text{H}_{10}\text{N}_3\text{O}_2^+ \cdot \text{C}_6\text{H}_5\text{O}_3\text{S}^-$, the asymmetric unit comprises a carbendazim cation and a besylate anion. Three intermolecular $\text{N}-\text{H} \cdots \text{O}$ hydrogen bonds stabilize the structure.

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Comment

Carbendazim is used as an antifungal agent and more recently was discovered to have antitumor activity (Ni *et al.*, 2002). The compound is an ampholyte, with a melting point of 593 K, a basic pK_a of 4.48 and an acidic pK_a of 10.80. Salt formation can potentially increase the aqueous solubility of the poorly soluble neutral carbendazim and the structure of the besylate (benzenesulfonate) salt, (I), of carbendazim (m.p. 484 K) is reported here (Fig. 1).



The benzene and five-membered rings of carbendazim form a planar structure, with an r.m.s. deviation of 0.0153 Å from the plane through atoms C6/N7/C8–C13/N14. The methyl

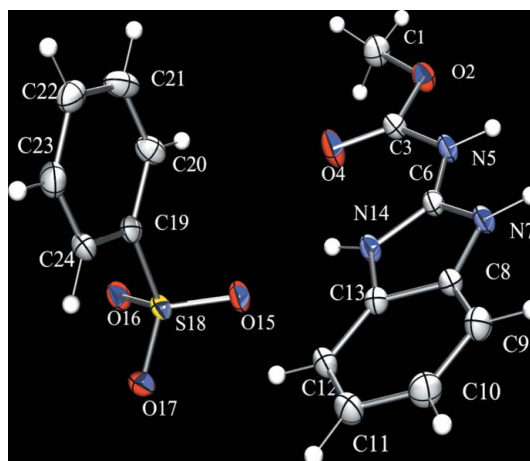


Figure 1
The asymmetric unit of (I), with displacement ellipsoids drawn at the 50% probability level.

carbamate substituent is similarly planar (r.m.s. deviation of 0.0045 Å from the plane through O2/C3/O4/N5/C6), with a dihedral angle of 11.80 (7)° between the planes.

Intermolecular N—H···O hydrogen bonding between the carbendazim and besylate ions stabilizes the structure, forming alternating ribbons along the *b* axis (Fig. 2).

Experimental

A 0.1M solution of benzenesulfonic acid was prepared in water and heated to 353 K. The solution was then supersaturated with an equimolar amount of carbendazim. The mixture was stirred continuously for thirty minutes, then filtered with a 0.45 μ syringe filter. The filtrate was allowed to cool at ambient temperature and crystallization occurred over a five-day period. Crystals were then stored in solution until required.

Crystal data

$C_9H_{10}N_3O_2^+ \cdot C_6H_5O_3S^-$	$V = 765.01 (8) \text{ \AA}^3$
$M_r = 349.36$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.517 \text{ Mg m}^{-3}$
$a = 9.0068 (5) \text{ \AA}$	Mo $K\alpha$ radiation
$b = 9.3944 (5) \text{ \AA}$	$\mu = 0.24 \text{ mm}^{-1}$
$c = 9.6643 (6) \text{ \AA}$	$T = 170 (2) \text{ K}$
$\alpha = 87.9448 (12)^\circ$	Rod, colorless
$\beta = 75.7679 (12)^\circ$	$0.39 \times 0.35 \times 0.18 \text{ mm}$
$\gamma = 74.9244 (12)^\circ$	

Data collection

Bruker SMART 1K CCD diffractometer	9724 measured reflections
ω scans	3619 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	2821 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.909, T_{\max} = 0.957$	$R_{\text{int}} = 0.056$
	$\theta_{\max} = 27.9^\circ$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.037$	$w = 1/[\sigma^2(F_o^2) + (0.0479P)^2]$
$wR(F^2) = 0.094$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.97$	$(\Delta/\sigma)_{\max} < 0.001$
3619 reflections	$\Delta\rho_{\max} = 0.37 \text{ e \AA}^{-3}$
217 parameters	$\Delta\rho_{\min} = -0.36 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N14—H14A···O15	0.88	2.01	2.7971 (16)	148
N5—H5A···O16 ⁱ	0.88	1.91	2.7716 (16)	167
N7—H7A···O17 ⁱ	0.88	1.91	2.7894 (16)	171

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

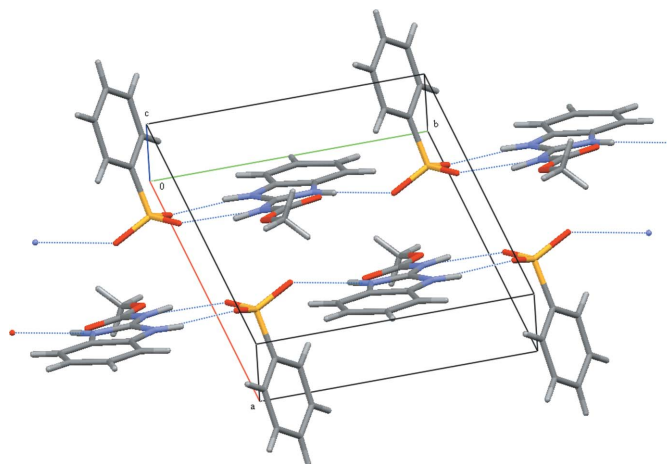


Figure 2

Packing of (I), showing the intermolecular hydrogen bonding (dashed lines) between the carbendazim cations and besylate anions.

H atoms were positioned geometrically, with N—H = 0.88 Å and C—H = 0.95 and 0.98 Å for aromatic and methyl H atoms, respectively. They were constrained to ride on their parent atoms, with $U_{\text{iso}}(\text{H}) = xU_{\text{eq}}(\text{C,N})$, where $x = 1.5$ for methyl and 1.2 for all other H atoms.

Data collection: SMART (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SHELXTL (Bruker, 2001); program(s) used to refine structure: SHELXTL; molecular graphics: ORTEP-3 for Windows (Farrugia, 1997) and MERCURY (Version 1.4; Macrae *et al.*, 2006); software used to prepare material for publication: SHELXTL and local programs.

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