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4-Nitrophenyl *N*-(2-isopropylthiazol-4-ylmethyl)-*N*-methylcarbamate

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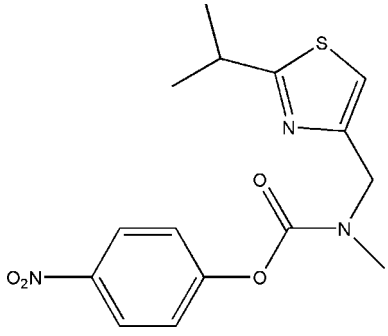
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.008 Å; R factor = 0.086; wR factor = 0.217; data-to-parameter ratio = 17.6.

In the title compound, $C_{15}H_{17}N_3O_4S$, the benzene and thiazole rings are oriented at a dihedral angle of 74.10 (3)°. In the crystal structure, intermolecular C—H···O hydrogen bonds are found.

Related literature

For related literature, see: Allen *et al.* (1987); Ishikawa *et al.* (1998); Riden & Hopkins (1961).



Experimental

Crystal data

$C_{15}H_{17}N_3O_4S$
M_r = 335.38
Orthorhombic, *Pbca*
a = 12.250 (3) Å
b = 10.876 (2) Å
c = 24.845 (5) Å
V = 3310.1 (12) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.22 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.10 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.937, T_{max} = 0.979
3281 measured reflections
3241 independent reflections
1335 reflections with $I > 2\sigma(I)$
R_{int} = 0.072
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.086
$wR(F^2)$ = 0.217
S = 1.04
3241 reflections
184 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C2—H2*C*···O4^{i}	0.96	2.58	3.493 (7)	159

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo,1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens,1996); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BQ2046).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Enraf–Nonius (1989). *CAD-4 Software.* Version 5.0. Enraf–Nonius, Delft, The Netherlands.
Harms, K. & Wocadlo, S. (1995). *XCAD4.* University of Marburg, Germany.
Ishikawa,T., Utoh, M., Sawada, N., Nishida, N., Fukase, Y., Sekiguchi, F. & Ishitsuka, H. (1998). *Biochem. Pharmacol.* **55**, 1091–1097..
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Riden, J. R. & Hopkins, T. R. (1961). *J. Agric. Food. Chem.* **9**, 47–48.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Siemens (1996). *SHELXTL*. Version 5.06. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.

supplementary materials

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4-Nitrophenyl *N*-(2-isopropylthiazol-4-ylmethyl)-*N*-methylcarbamate

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Comment

The title compound, $C_{15}H_{17}N_3O_4S$, is one of aromatic carbamates which are an important class of esters compounds and have widespread applications from pharmaceuticals (Ishikawa *et al.*, 1998) to agronomy (Riden & Hopkins, 1961). As part of our studies in this area, we report herein the synthesis and crystal structure of the title compound, (I).

In the molecule of (I) (Fig. 1), the ligand bond lengths and angles are within normal ranges (Allen *et al.*, 1987). Rings A (C4/N1/C5/C6/S) and B (C10—C15) are almost planar and they are oriented at a dihedral angle of 74.1°.

In the crystal structure, intermolecular C—H···O hydrogen bonds (Table 1) link the molecules (Fig.2), in which they seem to be effective in the stabilization of the structure.

Experimental

For the preparation of the title compound, (I), a solution of *N*-methyl-*N*-((2-isopropyl-4-thiazoyl)methyl)amine (3.7 g, 21.7 mmol) and excess *N*-methyl morpholine in methylene chloride (70 ml) was cooled to 273 K, and treated with 4-nitophenyl chloroformate (6.0 g, 30 mmol). After being stirred for 6 h, the reaction mixture was diluted with $CHCl_3$, washed successively with 1 N HCl, saturated aqueous $NaHCO_3$, and saturated bine, dried over $NaSO_4$, and concentrated *in vacuo*. The residue was purified by silica gel chromatography with 100% $CHCl_3$ to provide the title compound, (I) (yield: 6.5 g, 87%). Crystals of (I) suitable for *x*-ray analysis were obtained by slow evaporation of an ethanol solution.

Refinement

H atoms were positioned geometrically, with N—H = 0.86 Å (for NH) and C—H = 0.93, 0.98 and 0.96 Å for aromatic, methine and methyl H, and constrained to ride on their parent atoms, with $U_{iso}(H) = xU_{eq}(C,N)$, where $x = 1.5$ for methyl H, and $x = 1.2$ for all other H atoms.

Figures

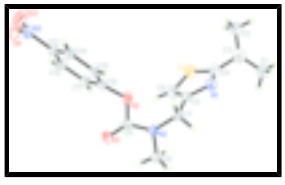

Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

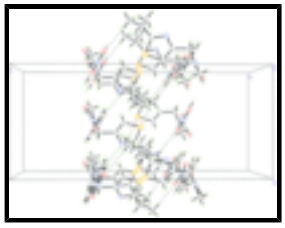

Fig. 2. A packing diagram for (I). Hydrogen bonds are shown as dashed lines.

4-Nitrophenyl *N*-(2-isopropylthiazol-4-ylmethyl)-*N*-methylcarbamate

Crystal data

$C_{15}H_{17}N_3O_4S$	$D_x = 1.346$ Mg m^{-3}
$M_r = 335.38$	Melting point: 330(2) K
Orthorhombic, *Pbca*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2ab	Cell parameters from 25 reflections
$a = 12.250$ (3) Å	θ = 9–12º
$b = 10.876$ (2) Å	μ = 0.22 mm^{-1}
$c = 24.845$ (5) Å	$T = 298$ (2) K
$V = 3310.1$ (12) Å^3	Block, colourless
$Z = 8$	0.30 × 0.20 × 0.10 mm
$F_{000} = 1408$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{int} = 0.072$
Radiation source: fine-focus sealed tube	$\theta_{max} = 26.0$º
Monochromator: graphite	$\theta_{min} = 1.6$º
$T = 298$(2) K	$h = 0 \rightarrow 15$
ω/2θ scans	$k = 0 \rightarrow 13$
Absorption correction: ψ scan (North *et al.*, 1968)	$l = 0 \rightarrow 30$
$T_{min} = 0.937$, $T_{max} = 0.979$	3 standard reflections
3281 measured reflections	every 200 reflections
3241 independent reflections	intensity decay: none
1335 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.086$	H-atom parameters constrained
$wR(F^2) = 0.217$	$w = 1/[\sigma^2(F_o^2) + (0.070P)^2 + 2.P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$

3241 reflections	$\Delta\rho_{max} = 0.28$ e Å^{-3}
184 parameters	$\Delta\rho_{min} = -0.39$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit S are based on F^2, conventional *R*-factors *R* are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > 2\text{sigma}(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on F, and R– factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.33658 (15)	0.04646 (16)	0.50156 (7)	0.089
N2	0.2887 (3)	−0.0166 (3)	0.68311 (16)	0.0430 (10)
O1	0.1531 (3)	−0.1308 (3)	0.71949 (15)	0.0610 (10)
C1	0.5582 (5)	0.2987 (6)	0.4986 (2)	0.084
H1A	0.5915	0.2283	0.5152	0.127*
H1B	0.5618	0.3675	0.5227	0.127*
H1C	0.5963	0.3182	0.4659	0.127*
O2	0.1198 (2)	0.0537 (3)	0.68088 (16)	0.0585 (10)
N3	−0.3347 (4)	0.0160 (5)	0.6666 (2)	0.0668 (14)
C2	0.3731 (4)	0.3175 (5)	0.4459 (2)	0.072
H2A	0.4102	0.3809	0.4262	0.108*
H2B	0.3087	0.3510	0.4623	0.108*
H2C	0.3529	0.2523	0.4218	0.108*
O3	−0.3851 (3)	0.1042 (4)	0.6861 (2)	0.1009 (16)
C3	0.4429 (5)	0.2710 (6)	0.4862 (3)	0.100 (2)
H3A	0.4661	0.2049	0.4619	0.120*
O4	−0.3738 (3)	−0.0692 (5)	0.6433 (2)	0.1038 (17)
C4	0.3950 (5)	0.1777 (6)	0.5238 (3)	0.0841 (19)
N1	0.3889 (4)	0.1941 (5)	0.5758 (2)	0.0771 (14)
C5	0.3383 (4)	0.0953 (4)	0.6001 (2)	0.0504 (13)
C6	0.3035 (5)	0.0073 (6)	0.5665 (2)	0.0785 (17)
H6A	0.2670	−0.0640	0.5767	0.094*
C7	0.3260 (4)	0.0983 (4)	0.6607 (2)	0.0491 (13)
H7A	0.3959	0.1191	0.6767	0.059*
H7B	0.2746	0.1625	0.6703	0.059*
C8	0.3718 (3)	−0.1137 (4)	0.6951 (2)	0.0547 (15)
H8A	0.3359	−0.1851	0.7094	0.082*
H8B	0.4231	−0.0830	0.7210	0.082*

H8C	0.4096	−0.1353	0.6626	0.082*
C9	0.1861 (4)	−0.0440 (5)	0.69588 (18)	0.0460 (12)
C10	0.0078 (3)	0.0370 (4)	0.6816 (2)	0.0448 (12)
C11	−0.0386 (4)	−0.0625 (4)	0.6552 (2)	0.0494 (12)
H11A	0.0058	−0.1238	0.6409	0.059*
C12	−0.1534 (4)	−0.0707 (5)	0.6500 (2)	0.0564 (13)
H12A	−0.1871	−0.1356	0.6322	0.068*
C13	−0.2133 (3)	0.0258 (5)	0.67368 (19)	0.0457 (12)
C14	−0.1691 (4)	0.1211 (4)	0.6979 (2)	0.0483 (12)
H14A	−0.2127	0.1839	0.7113	0.058*
C15	−0.0556 (3)	0.1265 (4)	0.70306 (18)	0.041
H15A	−0.0235	0.1918	0.7213	0.049*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.089	0.089	0.089	0.000	0.000	0.000
N2	0.035 (2)	0.035 (2)	0.059 (3)	0.0008 (17)	−0.0029 (19)	0.009 (2)
O1	0.059 (2)	0.044 (2)	0.080 (3)	−0.0118 (18)	−0.006 (2)	0.019 (2)
C1	0.084	0.084	0.084	0.000	0.000	0.000
O2	0.0345 (17)	0.0361 (19)	0.105 (3)	0.0031 (15)	0.0086 (19)	0.008 (2)
N3	0.041 (3)	0.083 (4)	0.076 (4)	−0.009 (3)	0.014 (3)	0.024 (3)
C2	0.072	0.072	0.072	0.000	0.000	0.000
O3	0.049 (2)	0.095 (3)	0.159 (5)	0.014 (2)	−0.002 (3)	−0.009 (3)
C3	0.091 (5)	0.102 (6)	0.106 (6)	−0.012 (4)	0.000 (5)	0.016 (5)
O4	0.052 (2)	0.129 (4)	0.131 (4)	−0.030 (3)	−0.001 (3)	−0.026 (4)
C4	0.067 (4)	0.099 (4)	0.087 (4)	−0.016 (3)	−0.010 (4)	0.024 (4)
N1	0.067 (3)	0.081 (3)	0.083 (3)	−0.019 (3)	−0.014 (3)	0.033 (3)
C5	0.040 (3)	0.037 (3)	0.074 (3)	0.005 (2)	−0.001 (3)	0.003 (2)
C6	0.082 (4)	0.073 (4)	0.080 (4)	−0.011 (3)	0.000 (3)	0.010 (3)
C7	0.041 (3)	0.034 (3)	0.072 (4)	−0.007 (2)	−0.001 (3)	0.001 (3)
C8	0.043 (3)	0.030 (3)	0.092 (4)	0.015 (2)	0.001 (3)	0.003 (3)
C9	0.048 (3)	0.053 (3)	0.037 (3)	0.011 (3)	0.002 (2)	0.010 (3)
C10	0.041 (2)	0.040 (3)	0.054 (3)	−0.006 (2)	0.009 (2)	0.015 (2)
C11	0.045 (3)	0.043 (3)	0.060 (3)	0.003 (2)	0.007 (2)	0.002 (2)
C12	0.050 (3)	0.061 (3)	0.058 (3)	0.000 (3)	−0.017 (3)	−0.004 (3)
C13	0.031 (2)	0.055 (3)	0.051 (3)	−0.005 (2)	−0.002 (2)	0.000 (2)
C14	0.040 (2)	0.038 (3)	0.067 (3)	0.000 (2)	0.008 (3)	0.002 (2)
C15	0.041	0.026	0.055	−0.003	0.013	0.003

Geometric parameters (Å, °)

S—C4	1.689 (7)	C4—N1	1.307 (7)
S—C6	1.716 (6)	N1—C5	1.379 (6)
N2—C9	1.330 (5)	C5—C6	1.341 (7)
N2—C7	1.442 (5)	C5—C7	1.513 (7)
N2—C8	1.496 (5)	C6—H6A	0.9300
O1—C9	1.183 (5)	C7—H7A	0.9700
C1—C3	1.477 (6)	C7—H7B	0.9700

C1—H1A	0.9600	C8—H8A	0.9600
C1—H1B	0.9600	C8—H8B	0.9600
C1—H1C	0.9600	C8—H8C	0.9600
O2—C10	1.384 (5)	C10—C15	1.355 (6)
O2—C9	1.388 (5)	C10—C11	1.387 (6)
N3—O4	1.193 (6)	C11—C12	1.415 (6)
N3—O3	1.239 (6)	C11—H11A	0.9300
N3—C13	1.502 (6)	C12—C13	1.409 (7)
C2—C3	1.411 (7)	C12—H12A	0.9300
C2—H2A	0.9600	C13—C14	1.314 (6)
C2—H2B	0.9600	C14—C15	1.398 (6)
C2—H2C	0.9600	C14—H14A	0.9300
C3—C4	1.498 (7)	C15—H15A	0.9300
C3—H3A	0.9800		
C4—S—C6	90.2 (3)	S—C6—H6A	125.3
C9—N2—C7	125.8 (4)	N2—C7—C5	113.4 (4)
C9—N2—C8	115.9 (4)	N2—C7—H7A	108.9
C7—N2—C8	118.3 (4)	C5—C7—H7A	108.9
C3—C1—H1A	109.5	N2—C7—H7B	108.9
C3—C1—H1B	109.5	C5—C7—H7B	108.9
H1A—C1—H1B	109.5	H7A—C7—H7B	107.7
C3—C1—H1C	109.5	N2—C8—H8A	109.5
H1A—C1—H1C	109.5	N2—C8—H8B	109.5
H1B—C1—H1C	109.5	H8A—C8—H8B	109.5
C10—O2—C9	118.4 (4)	N2—C8—H8C	109.5
O4—N3—O3	126.3 (5)	H8A—C8—H8C	109.5
O4—N3—C13	120.6 (5)	H8B—C8—H8C	109.5
O3—N3—C13	113.1 (5)	O1—C9—N2	128.3 (5)
C3—C2—H2A	109.5	O1—C9—O2	123.0 (4)
C3—C2—H2B	109.5	N2—C9—O2	108.5 (4)
H2A—C2—H2B	109.5	C15—C10—O2	118.6 (4)
C3—C2—H2C	109.5	C15—C10—C11	120.8 (4)
H2A—C2—H2C	109.5	O2—C10—C11	120.2 (4)
H2B—C2—H2C	109.5	C10—C11—C12	119.9 (5)
C2—C3—C1	130.9 (6)	C10—C11—H11A	120.0
C2—C3—C4	116.5 (5)	C12—C11—H11A	120.0
C1—C3—C4	112.6 (6)	C13—C12—C11	115.6 (5)
C2—C3—H3A	90.1	C13—C12—H12A	122.2
C1—C3—H3A	90.1	C11—C12—H12A	122.2
C4—C3—H3A	90.1	C14—C13—C12	124.3 (4)
N1—C4—C3	123.1 (6)	C14—C13—N3	121.2 (5)
N1—C4—S	114.4 (5)	C12—C13—N3	114.4 (5)
C3—C4—S	122.3 (5)	C13—C14—C15	119.0 (5)
C4—N1—C5	110.7 (5)	C13—C14—H14A	120.5
C6—C5—N1	115.2 (5)	C15—C14—H14A	120.5
C6—C5—C7	127.1 (5)	C10—C15—C14	120.3 (5)
N1—C5—C7	117.6 (5)	C10—C15—H15A	119.9
C5—C6—S	109.5 (5)	C14—C15—H15A	119.9
C5—C6—H6A	125.3		

C2—C3—C4—N1	−120.0 (7)	C8—N2—C9—O2	−178.4 (4)
C1—C3—C4—N1	59.8 (9)	C10—O2—C9—O1	−16.1 (7)
C2—C3—C4—S	55.9 (8)	C10—O2—C9—N2	168.8 (4)
C1—C3—C4—S	−124.4 (6)	C9—O2—C10—C15	135.6 (4)
C6—S—C4—N1	−1.3 (5)	C9—O2—C10—C11	−51.9 (6)
C6—S—C4—C3	−177.5 (6)	C15—C10—C11—C12	0.5 (7)
C3—C4—N1—C5	178.2 (5)	O2—C10—C11—C12	−171.8 (4)
S—C4—N1—C5	2.1 (7)	C10—C11—C12—C13	−0.8 (7)
C4—N1—C5—C6	−2.0 (7)	C11—C12—C13—C14	2.1 (8)
C4—N1—C5—C7	179.1 (5)	C11—C12—C13—N3	178.8 (4)
N1—C5—C6—S	1.0 (6)	O4—N3—C13—C14	178.0 (5)
C7—C5—C6—S	179.8 (4)	O3—N3—C13—C14	−0.6 (7)
C4—S—C6—C5	0.1 (5)	O4—N3—C13—C12	1.1 (7)
C9—N2—C7—C5	−97.3 (5)	O3—N3—C13—C12	−177.5 (5)
C8—N2—C7—C5	84.9 (5)	C12—C13—C14—C15	−2.9 (8)
C6—C5—C7—N2	10.8 (7)	N3—C13—C14—C15	−179.5 (4)
N1—C5—C7—N2	−170.4 (4)	O2—C10—C15—C14	171.1 (4)
C7—N2—C9—O1	−171.1 (5)	C11—C10—C15—C14	−1.3 (7)
C8—N2—C9—O1	6.8 (8)	C13—C14—C15—C10	2.5 (7)
C7—N2—C9—O2	3.7 (7)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C2—H2C···O4^{i}	0.96	2.58	3.493 (7)	159

Symmetry codes: (i) −*x*, −*y*, −*z*+1.

Fig. 1

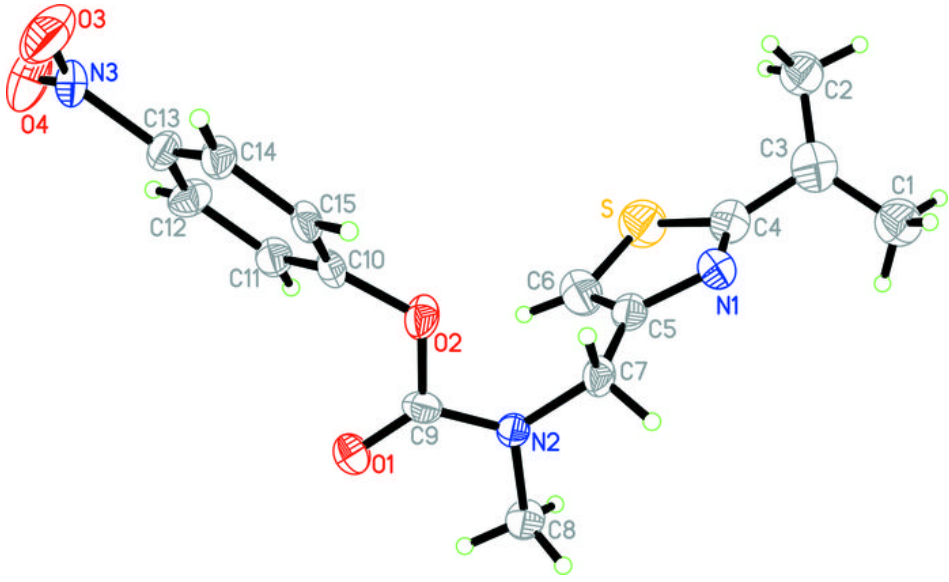
O3
N3
O4
C13
C14
C15
C12
C11
C10
O2
C9
O1
N2
C8
C7
C5
C6
S
N1
C4
C3
C2
C1

Fig. 2