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An omeprazole impurity

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Abstract

The title compound (I),9-methoxy-2,4-dimethyl-5-thioxo-5-hydro-pyrido[1',2':3,4] imidazo[1,2-*a*] benzimidazol-3-one is isolated during impurity profiling of Omeprazole. It is Crystallized from ethanol. The molecule is essentially planar. van der Waals interactions stabilize the molecules in the lattice.

Experimental

Crystals of Omeprazole impurity suitable for X-ray diffraction were grown from ethanol.

Computing details

Data collection: *MSC/AFC Diffractometer Control Software*; cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (MSC, 1992-1997); program(s) used to refine structure: *TEXSAN* (MSC, 1992-1997); software used to prepare material for publication: *TEXSAN* (MSC, 1992-1997).

8-methoxy-1,3-dimethyl-12-thioxopyrido[1',2':3,4]- imidazo[1,2-*a*][1,3]benzimidazol-2(12*H*)-one

Crystal data

$C_{16}H_{13}N_3O_2S$	V = 1441.5 (7) Å^3
M_r = 311.36	Z = 4
Monoclinic, $P2_1/c$	Cu $K\alpha$
a = 7.002 (2) Å	μ = 2.09 mm^{-1}
b = 18.442 (5) Å	T = 298.2 K
c = 11.443 (2) Å	1.00 × 0.20 × 0.10 mm
β = 102.72 (2)°	

Data collection

Rigaku AFC-7S diffractometer	2070 reflections with $I > 1.9\sigma(I)$
Absorption correction: empirical (using intensity measurements) (DIFABS; Walker & Stuart, 1983)	R_{int} = 0.079
T_{min} = 0.331, T_{max} = 0.815	3 standard reflections
2960 measured reflections	every 150 reflections
2722 independent reflections	intensity decay: 2.4%

CIF access

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.059$	200 parameters
$wR(F^2) = 0.094$	H-atom parameters not refined
$S = 1.93$	$\Delta\rho_{max} = 0.29$ e Å^{-3}
2070 reflections	$\Delta\rho_{min} = -0.23$ e Å^{-3}

Acknowledgements

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References

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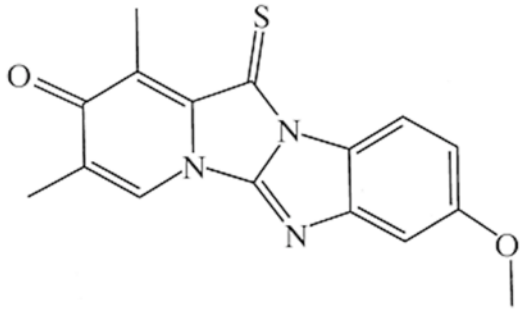
Scheme 1



supplementary materials

8-methoxy-1,3-dimethyl-12-thioxopyrido[1',2':3,4]- imidazo[1,2-*a*][1,3]benzimidazol-2(12*H*)-one

Crystal data

$C_{16}H_{13}N_3O_2S$	F_{000} = 648.00
M_r = 311.36	D_x = 1.435 Mg m^{-3}
Monoclinic, $P2_1/c$	Cu $K\alpha$ radiation λ = 1.5418 Å
a = 7.002 (2) Å	Cell parameters from 25 reflections
b = 18.442 (5) Å	θ = 20.1–26.8º
c = 11.443 (2) Å	μ = 2.09 mm^{-1}
β = 102.72 (2)º	T = 298.2 K
V = 1441.5 (7) Å^3	Needle, yellow
Z = 4	1.00 × 0.20 × 0.10 mm

Data collection

Rigaku AFC-7S diffractometer	R_{int} = 0.079
Radiation source: X-ray tube	θ_{max} = 70.1º
Monochromator: graphite	θ_{min} = 2.4º
T = 298.2 K	h = 0→8
ω–2θ scans	k = 0→21
Absorption correction: empirical (using intensity measurements) (DIFABS; Walker & Stuart, 1983)	l = −13→13
T_{min} = 0.331, T_{max} = 0.815	3 standard reflections
2960 measured reflections	every 150 reflections
2722 independent reflections	intensity decay: 2.4%
2070 reflections with $I > 1.9\sigma(I)$	

Refinement

Refinement on F	H-atom parameters not refined
Least-squares matrix: full	Weighting scheme based on measured s.u.'s $w = 1/[\sigma^2(F_o) + 0.0016\|F_o\|^2]$
$R[F^2 > 2\sigma(F^2)]$ = 0.059	$(\Delta/\sigma)_{max}$ = <0.001
$wR(F^2)$ = 0.094	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
S = 1.93	$\Delta\rho_{min}$ = −0.23 e Å^{-3}
2070 reflections	Extinction correction: Zachariasen (1967), equation 3, Acta Cryst. (1968), A24, p. 213.
200 parameters	Extinction coefficient: 8E-7 (8)

Special details

Experimental. The scan width was (1.47 + 0.14tanθ)° with an ω scan speed of 0° per minute (up to 7 scans to achieve I/σ(I) > 15). Stationary background counts were recorded at each end of the scan, and the scan time:background time ratio was 2:1.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
S(1)	0.2449 (1)	−0.09526 (4)	0.24173 (8)	0.0675 (3)
O(1)	0.2457 (3)	0.1992 (1)	0.4174 (2)	0.0704 (7)
O(2)	0.2642 (4)	−0.1182 (1)	−0.3956 (2)	0.0711 (8)
N(1)	0.2543 (3)	0.1004 (1)	0.1090 (2)	0.0435 (6)
N(2)	0.2633 (3)	0.0665 (1)	−0.0980 (2)	0.0460 (6)
N(3)	0.2458 (3)	−0.0143 (1)	0.0490 (2)	0.0435 (6)
C(1)	0.2488 (4)	0.0572 (1)	0.2083 (2)	0.0433 (7)
C(2)	0.2467 (4)	0.0885 (2)	0.3156 (2)	0.0496 (8)
C(3)	0.2480 (4)	0.1687 (2)	0.3217 (2)	0.0514 (8)
C(4)	0.2520 (4)	0.2095 (2)	0.2138 (3)	0.0510 (8)
C(5)	0.2564 (4)	0.1744 (2)	0.1112 (2)	0.0454 (7)
C(6)	0.2554 (4)	0.0562 (1)	0.0118 (2)	0.0417 (6)
C(7)	0.2587 (4)	−0.0049 (1)	−0.1424 (2)	0.0431 (6)
C(8)	0.2635 (4)	−0.0273 (2)	−0.2565 (2)	0.0500 (7)
C(9)	0.2567 (4)	−0.1014 (2)	−0.2799 (3)	0.0515 (8)
C(10)	0.2453 (4)	−0.1520 (2)	−0.1921 (3)	0.0547 (8)
C(11)	0.2411 (4)	−0.1296 (2)	−0.0762 (3)	0.0527 (8)
C(12)	0.2476 (4)	−0.0569 (1)	−0.0532 (2)	0.0425 (7)
C(13)	0.2462 (4)	−0.0200 (1)	0.1692 (2)	0.0461 (7)
C(14)	0.2422 (5)	0.0482 (2)	0.4280 (3)	0.067 (1)
C(15)	0.2510 (5)	0.2904 (2)	0.2200 (3)	0.067 (1)
C(16)	0.2431 (5)	−0.1922 (2)	−0.4314 (3)	0.075 (1)
H(1)	0.2569	0.1975	0.0440	0.055*
H(2)	0.2689	0.0066	−0.3155	0.054*
H(3)	0.2457	−0.2071	−0.2156	0.062*
H(4)	0.2329	−0.1644	−0.0220	0.064*
H(5)	0.2675	−0.0006	0.4276	0.088*
H(6)	0.3626	0.0630	0.4908	0.149*
H(7)	0.1447	0.0618	0.4585	0.119*
H(8)	0.3593	0.3102	0.2820	0.098*
H(9)	0.1413	0.3072	0.2504	0.122*
H(10)	0.2572	0.3161	0.1429	0.084*
H(11)	0.2516	−0.1921	−0.5181	0.123*
H(12)	0.1231	−0.2139	−0.4227	0.096*
H(13)	0.3652	−0.2247	−0.3844	0.109*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S(1)	0.0804 (7)	0.0579 (5)	0.0658 (5)	0.0030 (4)	0.0194 (4)	0.0164 (3)
O(1)	0.085 (2)	0.078 (2)	0.052 (1)	0.004 (1)	0.023 (1)	−0.012 (1)
O(2)	0.088 (2)	0.068 (1)	0.060 (1)	−0.005 (1)	0.022 (1)	−0.018 (1)
N(1)	0.039 (1)	0.048 (1)	0.044 (1)	−0.0017 (8)	0.0093 (9)	−0.0010 (8)
N(2)	0.046 (1)	0.047 (1)	0.044 (1)	−0.0026 (9)	0.009 (1)	−0.0001 (8)

N(3)	0.038 (1)	0.044 (1)	0.048 (1)	0.0006 (9)	0.0085 (9)	0.0029 (8)
C(1)	0.029 (1)	0.055 (1)	0.045 (1)	0.001 (1)	0.007 (1)	0.006 (1)
C(2)	0.035 (1)	0.067 (2)	0.045 (1)	0.005 (1)	0.007 (1)	0.002 (1)
C(3)	0.040 (1)	0.068 (2)	0.047 (1)	0.002 (1)	0.009 (1)	−0.005 (1)
C(4)	0.044 (2)	0.059 (2)	0.050 (1)	0.002 (1)	0.010 (1)	−0.006 (1)
C(5)	0.040 (1)	0.049 (1)	0.047 (1)	−0.002 (1)	0.008 (1)	0.000 (1)
C(6)	0.031 (1)	0.046 (1)	0.047 (1)	0.000 (1)	0.006 (1)	−0.002 (1)
C(7)	0.032 (1)	0.047 (1)	0.049 (1)	−0.001 (1)	0.006 (1)	0.001 (1)
C(8)	0.045 (2)	0.055 (1)	0.050 (1)	−0.001 (1)	0.010 (1)	−0.002 (1)
C(9)	0.040 (2)	0.060 (2)	0.052 (2)	−0.001 (1)	0.006 (1)	−0.011 (1)
C(10)	0.045 (2)	0.053 (1)	0.065 (2)	−0.002 (1)	0.008 (1)	−0.008 (1)
C(11)	0.045 (2)	0.050 (1)	0.060 (2)	−0.002 (1)	0.007 (1)	0.003 (1)
C(12)	0.030 (1)	0.046 (1)	0.048 (1)	−0.002 (1)	0.004 (1)	−0.001 (1)
C(13)	0.032 (1)	0.055 (1)	0.051 (1)	0.002 (1)	0.007 (1)	0.005 (1)
C(14)	0.074 (2)	0.079 (2)	0.049 (1)	0.011 (2)	0.017 (1)	0.010 (2)
C(15)	0.083 (2)	0.055 (2)	0.065 (2)	0.003 (2)	0.018 (2)	−0.011 (1)
C(16)	0.077 (2)	0.072 (2)	0.078 (2)	−0.009 (2)	0.020 (2)	−0.028 (2)

Geometric parameters (Å, °)

S(1)—C(13)	1.618 (3)	C(5)—H(1)	0.88
O(1)—C(3)	1.236 (3)	C(7)—C(8)	1.377 (3)
O(2)—C(9)	1.372 (3)	C(7)—C(12)	1.415 (3)
O(2)—C(16)	1.424 (4)	C(8)—C(9)	1.392 (4)
N(1)—C(1)	1.395 (3)	C(8)—H(2)	0.93
N(1)—C(5)	1.366 (4)	C(9)—C(10)	1.386 (4)
N(1)—C(6)	1.380 (3)	C(10)—C(11)	1.396 (4)
N(2)—C(6)	1.284 (3)	C(10)—H(3)	1.05
N(2)—C(7)	1.410 (3)	C(11)—C(12)	1.365 (4)
N(3)—C(6)	1.375 (3)	C(11)—H(4)	0.90
N(3)—C(12)	1.411 (3)	C(14)—H(5)	0.92
N(3)—C(13)	1.378 (3)	C(14)—H(6)	1.02
C(1)—C(2)	1.360 (4)	C(14)—H(7)	0.87
C(1)—C(13)	1.491 (4)	C(15)—H(8)	0.99
C(2)—C(3)	1.479 (4)	C(15)—H(9)	0.96
C(2)—C(14)	1.492 (4)	C(15)—H(10)	1.01
C(3)—C(4)	1.452 (4)	C(16)—H(11)	1.01
C(4)—C(5)	1.347 (4)	C(16)—H(12)	0.96
C(4)—C(15)	1.494 (4)	C(16)—H(13)	1.08
O(1)···C(5)^{i}	3.205 (3)	C(1)···C(8)^{iii}	3.382 (4)
O(1)···C(15)^{i}	3.460 (4)	C(1)···C(9)^{iii}	3.475 (4)
O(1)···C(16)^{ii}	3.464 (4)	C(1)···C(12)^{ii}	3.538 (3)
O(1)···C(16)^{iii}	3.550 (4)	C(1)···C(7)^{ii}	3.598 (4)
O(2)···C(2)^{iii}	3.389 (4)	C(2)···C(9)^{ii}	3.466 (4)
O(2)···C(3)^{iii}	3.461 (4)	C(2)···C(9)^{iii}	3.597 (4)
N(1)···C(11)^{ii}	3.448 (4)	C(3)···C(10)^{ii}	3.460 (4)
N(1)···C(12)^{ii}	3.523 (3)	C(3)···C(16)^{iii}	3.535 (4)

N(1)···C(9)^{iii}	3.549 (4)	C(4)···C(10)^{ii}	3.596 (4)
N(1)···C(10)^{iii}	3.553 (4)	C(5)···C(10)^{iii}	3.432 (4)
N(2)···C(15)^{iv}	3.351 (4)	C(5)···C(11)^{ii}	3.516 (4)
N(2)···C(12)^{iii}	3.486 (3)	C(6)···C(12)^{iii}	3.408 (3)
N(2)···N(3)^{iii}	3.494 (3)	C(6)···C(7)^{iii}	3.531 (4)
N(2)···C(13)^{ii}	3.584 (3)	C(7)···C(13)^{ii}	3.511 (4)
N(3)···C(7)^{iii}	3.417 (3)	C(7)···C(13)^{iii}	3.577 (4)
N(3)···N(3)^{ii}	3.419 (4)	C(8)···C(13)^{iii}	3.469 (4)
N(3)···C(6)^{ii}	3.510 (3)		
C(9)—O(2)—C(16)	118.1 (3)	O(2)—C(9)—C(10)	124.6 (3)
C(1)—N(1)—C(5)	123.9 (2)	C(8)—C(9)—C(10)	121.8 (3)
C(1)—N(1)—C(6)	109.0 (2)	C(9)—C(10)—C(11)	120.4 (3)
C(5)—N(1)—C(6)	127.0 (2)	C(9)—C(10)—H(3)	117.5
C(6)—N(2)—C(7)	102.2 (2)	C(11)—C(10)—H(3)	122.1
C(6)—N(3)—C(12)	105.0 (2)	C(10)—C(11)—C(12)	117.8 (3)
C(6)—N(3)—C(13)	113.1 (2)	C(10)—C(11)—H(4)	117.4
C(12)—N(3)—C(13)	141.8 (2)	C(12)—C(11)—H(4)	124.8
N(1)—C(1)—C(2)	120.0 (3)	N(3)—C(12)—C(7)	103.5 (2)
N(1)—C(1)—C(13)	107.5 (2)	N(3)—C(12)—C(11)	134.4 (2)
C(2)—C(1)—C(13)	132.5 (2)	C(7)—C(12)—C(11)	122.1 (2)
C(1)—C(2)—C(3)	117.7 (2)	S(1)—C(13)—N(3)	125.3 (2)
C(1)—C(2)—C(14)	124.9 (3)	S(1)—C(13)—C(1)	131.7 (2)
C(3)—C(2)—C(14)	117.3 (3)	N(3)—C(13)—C(1)	103.0 (2)
O(1)—C(3)—C(2)	119.7 (3)	C(2)—C(14)—H(5)	116.4
O(1)—C(3)—C(4)	121.6 (3)	C(2)—C(14)—H(6)	107.7
C(2)—C(3)—C(4)	118.7 (2)	C(2)—C(14)—H(7)	111.7
C(3)—C(4)—C(5)	120.0 (3)	H(5)—C(14)—H(6)	97.8
C(3)—C(4)—C(15)	118.5 (3)	H(5)—C(14)—H(7)	117.0
C(5)—C(4)—C(15)	121.5 (3)	H(6)—C(14)—H(7)	104.0
N(1)—C(5)—C(4)	119.6 (3)	C(4)—C(15)—H(8)	113.0
N(1)—C(5)—H(1)	118.1	C(4)—C(15)—H(9)	110.6
C(4)—C(5)—H(1)	122.3	C(4)—C(15)—H(10)	115.2
N(1)—C(6)—N(2)	135.3 (2)	H(8)—C(15)—H(9)	99.8
N(1)—C(6)—N(3)	107.4 (2)	H(8)—C(15)—H(10)	106.3
N(2)—C(6)—N(3)	117.3 (2)	H(9)—C(15)—H(10)	110.9
N(2)—C(7)—C(8)	128.2 (2)	O(2)—C(16)—H(11)	104.9
N(2)—C(7)—C(12)	111.9 (2)	O(2)—C(16)—H(12)	114.1
C(8)—C(7)—C(12)	119.9 (2)	O(2)—C(16)—H(13)	111.4
C(7)—C(8)—C(9)	118.0 (3)	H(11)—C(16)—H(12)	110.4
C(7)—C(8)—H(2)	120.1	H(11)—C(16)—H(13)	106.2
C(9)—C(8)—H(2)	121.9	H(12)—C(16)—H(13)	109.5
O(2)—C(9)—C(8)	113.6 (3)		
S(1)—C(13)—N(3)—C(6)	−177.4 (2)	N(3)—C(13)—C(1)—C(2)	178.6 (3)
S(1)—C(13)—N(3)—C(12)	−1.4 (5)	C(1)—N(1)—C(5)—C(4)	−0.3 (4)
S(1)—C(13)—C(1)—N(1)	178.4 (2)	C(1)—C(2)—C(3)—C(4)	−0.2 (4)
S(1)—C(13)—C(1)—C(2)	−1.7 (5)	C(1)—C(13)—N(3)—C(6)	2.3 (3)
O(1)—C(3)—C(2)—C(1)	179.9 (3)	C(1)—C(13)—N(3)—C(12)	178.4 (3)

O(1)—C(3)—C(2)—C(14)	0.1 (4)	C(2)—C(1)—N(1)—C(5)	−0.5 (4)
O(1)—C(3)—C(4)—C(5)	179.3 (3)	C(2)—C(1)—N(1)—C(6)	−180.0 (2)
O(1)—C(3)—C(4)—C(15)	−0.7 (4)	C(2)—C(3)—C(4)—C(5)	−0.6 (4)
O(2)—C(9)—C(8)—C(7)	−179.4 (2)	C(2)—C(3)—C(4)—C(15)	179.3 (3)
O(2)—C(9)—C(10)—C(11)	179.1 (3)	C(3)—C(2)—C(1)—C(13)	−179.2 (3)
N(1)—C(1)—C(2)—C(3)	0.7 (4)	C(4)—C(3)—C(2)—C(14)	−179.9 (3)
N(1)—C(1)—C(2)—C(14)	−179.6 (3)	C(4)—C(5)—N(1)—C(6)	179.0 (3)
N(1)—C(1)—C(13)—N(3)	−1.4 (3)	C(5)—N(1)—C(1)—C(13)	179.4 (2)
N(1)—C(5)—C(4)—C(3)	0.9 (4)	C(6)—N(1)—C(1)—C(13)	0.0 (3)
N(1)—C(5)—C(4)—C(15)	−179.0 (3)	C(6)—N(2)—C(7)—C(8)	−180.0 (3)
N(1)—C(6)—N(2)—C(7)	179.8 (3)	C(6)—N(2)—C(7)—C(12)	0.1 (3)
N(1)—C(6)—N(3)—C(12)	−179.9 (2)	C(6)—N(3)—C(12)—C(7)	0.1 (3)
N(1)—C(6)—N(3)—C(13)	−2.5 (3)	C(6)—N(3)—C(12)—C(11)	−179.9 (3)
N(2)—C(6)—N(1)—C(1)	−178.4 (3)	C(7)—C(8)—C(9)—C(10)	0.0 (4)
N(2)—C(6)—N(1)—C(5)	2.1 (5)	C(7)—C(12)—N(3)—C(13)	−176.2 (3)
N(2)—C(6)—N(3)—C(12)	0.0 (3)	C(7)—C(12)—C(11)—C(10)	−0.1 (4)
N(2)—C(6)—N(3)—C(13)	177.4 (2)	C(8)—C(7)—C(12)—C(11)	−0.1 (4)
N(2)—C(7)—C(8)—C(9)	−179.8 (3)	C(8)—C(9)—O(2)—C(16)	−175.3 (3)
N(2)—C(7)—C(12)—N(3)	−0.1 (3)	C(8)—C(9)—C(10)—C(11)	−0.3 (5)
N(2)—C(7)—C(12)—C(11)	179.9 (2)	C(9)—C(8)—C(7)—C(12)	0.1 (4)
N(3)—C(6)—N(1)—C(1)	1.4 (3)	C(9)—C(10)—C(11)—C(12)	0.3 (4)
N(3)—C(6)—N(1)—C(5)	−178.0 (3)	C(10)—C(9)—O(2)—C(16)	5.3 (5)
N(3)—C(6)—N(2)—C(7)	0.0 (3)	C(11)—C(12)—N(3)—C(13)	3.9 (5)
N(3)—C(12)—C(7)—C(8)	179.9 (2)	C(13)—C(1)—C(2)—C(14)	0.5 (5)
N(3)—C(12)—C(11)—C(10)	179.8 (3)		

Symmetry codes: (i) *x*, −*y*+1/2, *z*+1/2; (ii) −*x*, −*y*, −*z*; (iii) −*x*+1, −*y*, −*z*; (iv) *x*, −*y*+1/2, *z*−1/2.