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(3*S*,4*S*,4a*R*,10a*S*)-5-Acetyl-3,4,4a,5,6,7,10,10a-octahydro-8-methoxy-7-methyl-6-oxo-2*H*-pyrano[3,2-*g*]pteridine-3,4-diyl Diacetate

J. N. Low, G. Ferguson, L. M. Dolores, Q. M. Luisa, A. Sánchez, M. Nogueras and J. Cobo

Abstract

The structure and stereochemistry of the title compound, $C_{17}H_{22}N_4O_8$, are reported. The compound is formed by the reaction of 5,6-diamino-2-methoxy-3-methylpyrimidin-4(3*H*)-one and *L*-xylose. It is the enantiomer of the compound synthesized using D-xylose and reported previously [Low *et al.* (1995). Acta Cryst. C51, 2141–2143]. There are no significant differences (apart from the absolute configuration) between the structure report here and that of the enantiomer reported previously.

Experimental

5,6-Diamino-2-methoxy-3- methylpyrimidin-4(3*H*)-one and *L*-xylose phenylhydrazone were reacted under conditions described by Soyka *et al.* (1990). The product so obtained was then converted to its crystalline triactyl derivative.

Refinement

The title compound crystallized in the orthorhombic system; space group $P2_12_12_1$ from the systematic absences. During the refinement all H atoms were visible in difference maps and were treated as riding atoms (C—H 0.96 to 0.98, N—H 0.86 Å). The absolute configuration was not determined by the X-ray analysis but is established by the known absolute configuration of the starting *L*-xylose.

Computing details

Data collection: *CAD-4-PC* Software (Enraf-Nonius, 1992); cell refinement: *SET4* and *CELDIM* (Enraf-Nonius, 1992); data reduction: *DATRD2* in *NRCVAX96* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997b); program(s) used to refine structure: *NRCVAX96* and *SHELXL97* (Sheldrick, 1997a); molecular graphics: *NRCVAX96*, *ORTEPII* (Johnson, 1976) and *PLATON* (Spek, 1998); software used to prepare material for publication: *NRCVAX96*, *SHELXL97* and *WORDPERFECT* macro PREP8 (Ferguson, 1998).

(3*S*,4*S*,4a*R*,10a*s*)-5,10-Diacetyl-3,4,4a,5,6,7,10,10*a*-octahydro-8-methoxy-6-oxo –2*H*-pyrano[3,2 g]pteridin-3,4-diyl diacetate

Crystal data

$C_{17}H_{22}N_4O_8$	$V = 1981.4\ (5)$ Å^3
$M_r = 410.39$	$Z = 4$
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$
$a = 9.9115\ (13)$ Å	$\mu = 0.11$ mm^{-1}

b = 12.4531 (13) Å	T = 294 (1) K
c = 16.053 (3) Å	0.44 × 0.44 × 0.44 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	R_{int} = 0.027
Absorption correction: none	3 standard reflections
4728 measured reflections	every 120 min
4305 independent reflections	intensity decay: 1.6%
2511 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051	Constrained
$wR(F^2)$ = 0.124	$\Delta\rho_{max}$ = 0.32 e Å^{-3}
S = 0.95	$\Delta\rho_{min}$ = −0.22 e Å^{-3}
4305 reflections	Absolute structure: Flack (1983), 1853 Friedel pairs
267 parameters	Flack parameter: −0.3 (15)

Acknowledgements

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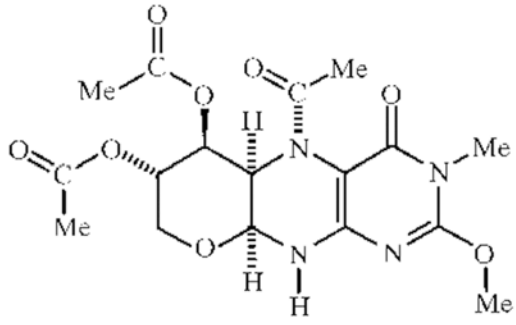
Scheme 1



supplementary materials

(3*S*,4S,4aR,10as)-5,10-Diacetyl-3,4,4a,5,6,7,10,10*a*-octahydro-8-methoxy-6-oxo −2*H*-pyrano[3,2 g]pteridin-3,4-diyl diacetate

Crystal data

$C_{17}H_{22}N_4O_8$	? #Insert any comments here.
$M_r = 410.39$	$D_x = 1.376$ Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation $\lambda = 0.7107$ Å
$a = 9.9115$ (13) Å	Cell parameters from 25 reflections
$b = 12.4531$ (13) Å	$\theta = 10.5$–17.9^o
$c = 16.053$ (3) Å	$\mu = 0.11$ mm^{-1}
$V = 1981.4$ (5) Å^3	$T = 294$ (1) K
$Z = 4$	Block, colorless
$F_{000} = 864$	0.44 × 0.44 × 0.44 mm

Data collection

Enraf-Nonius CAD-4 diffractometer	$R_{int} = 0.027$
Radiation source: X-ray tube	$\theta_{max} = 27.0^o$
Monochromator: graphite	$\theta_{min} = 2.1^o$
$T = 294$(1) K	$h = -12 \rightarrow 12$
θ/2θ scans	$k = 0 \rightarrow 15$
Absorption correction: none	$l = 0 \rightarrow 20$
4728 measured reflections	3 standard reflections
4305 independent reflections	every 120 min
2511 reflections with $I > 2\sigma(I)$	intensity decay: 1.6%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	Constrained
$R[F^2 > 2\sigma(F^2)] = 0.051$	$w = 1/[\sigma^2(F_o^2) + (0.0638P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.124$	$(\Delta/\sigma)_{max} = 0.025$
$S = 0.95$	$\Delta\rho_{max} = 0.32$ e Å^{-3}
4305 reflections	$\Delta\rho_{min} = -0.22$ e Å^{-3}
267 parameters	Extinction correction: none
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), 1853 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: −0.3 (15)

Special details

Experimental. ? #Insert any special details here.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.0373 (2)	0.85912 (16)	−0.04398 (11)	0.0475 (5)
O3	0.0537 (2)	0.65381 (19)	0.04076 (12)	0.0514 (6)
O4	−0.1195 (2)	0.64598 (16)	−0.15876 (11)	0.0445 (5)
O6	0.0652 (2)	0.58309 (16)	−0.36946 (12)	0.0513 (6)
O8	−0.2406 (2)	0.83777 (18)	−0.43581 (13)	0.0606 (6)
O31	−0.0928 (3)	0.5744 (3)	0.1214 (2)	0.1347 (17)
O41	−0.1778 (3)	0.4795 (2)	−0.1192 (2)	0.0826 (8)
O51	0.3619 (2)	0.6580 (2)	−0.18807 (14)	0.0656 (7)
N5	0.1475 (2)	0.69744 (19)	−0.22343 (13)	0.0366 (6)
N7	−0.0928 (3)	0.71097 (19)	−0.39914 (14)	0.0467 (6)
N9	−0.1232 (2)	0.86337 (18)	−0.31426 (14)	0.0389 (6)
N10	−0.0110 (2)	0.87356 (18)	−0.18793 (14)	0.0383 (6)
C2	−0.0767 (3)	0.7983 (3)	−0.01777 (18)	0.0507 (8)
C3	−0.0512 (3)	0.6788 (3)	−0.01900 (16)	0.0437 (7)
C4	−0.0036 (3)	0.6419 (2)	−0.10441 (16)	0.0382 (7)
C4A	0.1089 (3)	0.7147 (2)	−0.13650 (15)	0.0338 (6)
C5A	0.0503 (3)	0.7347 (2)	−0.28185 (16)	0.0361 (7)
C6	0.0139 (3)	0.6687 (2)	−0.35089 (16)	0.0386 (7)
C7	−0.1425 (4)	0.6447 (3)	−0.4689 (2)	0.0719 (11)
C8	−0.1502 (3)	0.8065 (2)	−0.37929 (18)	0.0438 (7)
C9A	−0.0264 (3)	0.8233 (2)	−0.26231 (16)	0.0341 (6)
C10A	0.0825 (3)	0.8347 (2)	−0.12522 (16)	0.0367 (7)
C31	0.0195 (3)	0.6022 (3)	0.1096 (2)	0.0577 (9)
C32	0.1323 (4)	0.5877 (4)	0.1685 (2)	0.0763 (12)
C41	−0.1994 (4)	0.5582 (3)	−0.1607 (2)	0.0568 (9)
C42	−0.3156 (4)	0.5758 (4)	−0.2187 (3)	0.0923 (14)
C51	0.2819 (3)	0.6858 (2)	−0.24141 (18)	0.0441 (7)
C52	0.3262 (3)	0.7115 (3)	−0.3285 (2)	0.0617 (10)
C81	−0.3088 (4)	0.9381 (3)	−0.4212 (2)	0.0701 (11)
H10	−0.0581	0.9300	−0.1776	0.046*
H2A	−0.1524	0.8144	−0.0540	0.061*
H2B	−0.1012	0.8198	0.0383	0.061*
H3	−0.1342	0.6406	−0.0040	0.052*
H4	0.0294	0.5678	−0.1008	0.046*
H4A	0.1889	0.6980	−0.1030	0.041*
H7A	−0.2393	0.6470	−0.4701	0.108*
H7B	−0.1132	0.5718	−0.4614	0.108*
H7C	−0.1074	0.6721	−0.5204	0.108*
H10A	0.1684	0.8722	−0.1337	0.044*
H32A	0.1020	0.5472	0.2158	0.114*
H32B	0.1641	0.6567	0.1866	0.114*
H32C	0.2043	0.5498	0.1413	0.114*
H42A	−0.3509	0.5076	−0.2362	0.138*
H42B	−0.2855	0.6154	−0.2665	0.138*
H42C	−0.3849	0.6155	−0.1905	0.138*

H52A	0.4223	0.7034	−0.3327	0.093*
H52B	0.3018	0.7841	−0.3417	0.093*
H52C	0.2829	0.6634	−0.3668	0.093*
H81A	−0.3725	0.9295	−0.3767	0.105*
H81B	−0.3554	0.9597	−0.4709	0.105*
H81C	−0.2439	0.9920	−0.4064	0.105*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0553 (13)	0.0539 (13)	0.0333 (10)	0.0004 (11)	−0.0035 (10)	−0.0085 (10)
O3	0.0380 (11)	0.0785 (15)	0.0378 (11)	−0.0062 (11)	−0.0008 (9)	0.0194 (11)
O4	0.0417 (12)	0.0497 (12)	0.0420 (11)	−0.0074 (10)	−0.0047 (9)	0.0019 (9)
O6	0.0773 (15)	0.0364 (12)	0.0401 (11)	0.0068 (12)	−0.0003 (11)	−0.0019 (9)
O8	0.0727 (15)	0.0571 (14)	0.0519 (13)	0.0160 (13)	−0.0286 (12)	−0.0046 (11)
O31	0.0537 (16)	0.236 (5)	0.115 (2)	−0.025 (2)	−0.0103 (17)	0.123 (3)
O41	0.0811 (18)	0.0541 (15)	0.113 (2)	−0.0190 (15)	0.0046 (18)	0.0060 (16)
O51	0.0433 (13)	0.105 (2)	0.0484 (12)	0.0230 (14)	0.0009 (11)	0.0077 (14)
N5	0.0353 (13)	0.0447 (14)	0.0298 (11)	0.0069 (11)	0.0002 (10)	0.0030 (11)
N7	0.0665 (17)	0.0421 (15)	0.0314 (12)	0.0019 (14)	−0.0095 (13)	−0.0026 (11)
N9	0.0429 (14)	0.0400 (13)	0.0339 (12)	0.0034 (12)	−0.0042 (12)	0.0018 (11)
N10	0.0440 (14)	0.0358 (12)	0.0352 (12)	0.0077 (11)	−0.0025 (11)	−0.0044 (11)
C2	0.0466 (18)	0.071 (2)	0.0346 (16)	0.0110 (18)	0.0006 (14)	−0.0022 (15)
C3	0.0298 (14)	0.068 (2)	0.0335 (14)	0.0018 (15)	−0.0019 (12)	0.0080 (14)
C4	0.0341 (15)	0.0444 (16)	0.0363 (14)	0.0006 (14)	−0.0038 (13)	0.0038 (13)
C4A	0.0329 (14)	0.0435 (16)	0.0249 (13)	0.0020 (13)	−0.0027 (12)	0.0021 (12)
C5A	0.0418 (17)	0.0375 (16)	0.0289 (14)	0.0030 (14)	0.0040 (13)	0.0025 (12)
C6	0.0532 (18)	0.0310 (15)	0.0314 (14)	−0.0015 (15)	−0.0009 (13)	0.0051 (12)
C7	0.105 (3)	0.059 (2)	0.0517 (19)	0.013 (2)	−0.038 (2)	−0.0153 (17)
C8	0.0517 (18)	0.0431 (18)	0.0366 (16)	0.0033 (15)	−0.0074 (15)	0.0075 (14)
C9A	0.0359 (15)	0.0367 (16)	0.0298 (14)	−0.0037 (14)	0.0012 (12)	0.0050 (12)
C10A	0.0390 (15)	0.0421 (16)	0.0291 (14)	−0.0013 (14)	−0.0001 (12)	0.0016 (12)
C31	0.0440 (19)	0.076 (2)	0.053 (2)	−0.0021 (18)	0.0059 (16)	0.0283 (18)
C32	0.068 (2)	0.109 (3)	0.052 (2)	−0.008 (2)	−0.018 (2)	0.034 (2)
C41	0.053 (2)	0.057 (2)	0.060 (2)	−0.0170 (19)	0.0080 (17)	−0.0123 (19)
C42	0.058 (2)	0.119 (4)	0.101 (3)	−0.025 (3)	−0.026 (2)	−0.021 (3)
C51	0.0422 (17)	0.0503 (19)	0.0398 (17)	0.0081 (16)	0.0022 (15)	0.0007 (14)
C52	0.055 (2)	0.077 (3)	0.053 (2)	0.0051 (19)	0.0154 (17)	0.0122 (19)
C81	0.067 (3)	0.068 (3)	0.075 (2)	0.017 (2)	−0.024 (2)	0.000 (2)

Geometric parameters (Å, °)

O1—C10A	1.412 (3)	N7—C6	1.412 (4)
O1—C2	1.425 (4)	N7—C7	1.476 (4)
O3—C31	1.323 (4)	N9—C8	1.289 (4)
O3—C3	1.449 (3)	N9—C9A	1.366 (3)
O4—C41	1.351 (4)	N10—C9A	1.357 (3)
O4—C4	1.443 (3)	N10—C10A	1.452 (3)
O6—C6	1.219 (3)	C2—C3	1.509 (4)

O8—C8	1.334 (3)	C3—C4	1.521 (4)
O8—C81	1.439 (4)	C4—C4A	1.527 (4)
O31—C31	1.181 (4)	C4A—C10A	1.529 (4)
O41—C41	1.205 (4)	C5A—C9A	1.376 (4)
O51—C51	1.217 (3)	C5A—C6	1.426 (4)
N5—C51	1.371 (4)	C31—C32	1.475 (4)
N5—C5A	1.422 (3)	C41—C42	1.496 (5)
N5—C4A	1.463 (3)	C51—C52	1.500 (4)
N7—C8	1.357 (4)		
C10A—O1—C2	114.2 (2)	C9A—C5A—C6	120.0 (3)
C31—O3—C3	118.3 (2)	N5—C5A—C6	119.7 (2)
C41—O4—C4	116.9 (2)	O6—C6—N7	120.3 (3)
C8—O8—C81	117.3 (2)	O6—C6—C5A	126.1 (3)
C51—N5—C5A	123.6 (2)	N7—C6—C5A	113.6 (2)
C51—N5—C4A	118.0 (2)	N9—C8—O8	122.0 (3)
C5A—N5—C4A	113.9 (2)	N9—C8—N7	125.8 (3)
C8—N7—C6	120.8 (2)	O8—C8—N7	112.2 (3)
C8—N7—C7	121.9 (3)	N10—C9A—N9	116.6 (2)
C6—N7—C7	117.3 (2)	N10—C9A—C5A	120.6 (2)
C8—N9—C9A	116.1 (2)	N9—C9A—C5A	122.8 (2)
C9A—N10—C10A	121.9 (2)	O1—C10A—N10	111.5 (2)
O1—C2—C3	112.8 (2)	O1—C10A—C4A	112.0 (2)
O3—C3—C2	108.9 (3)	N10—C10A—C4A	110.7 (2)
O3—C3—C4	108.0 (2)	O31—C31—O3	121.2 (3)
C2—C3—C4	111.2 (2)	O31—C31—C32	125.1 (3)
O4—C4—C3	106.7 (2)	O3—C31—C32	113.6 (3)
O4—C4—C4A	110.9 (2)	O41—C41—O4	122.8 (3)
C3—C4—C4A	110.6 (2)	O41—C41—C42	126.9 (4)
N5—C4A—C4	115.2 (2)	O4—C41—C42	110.3 (3)
N5—C4A—C10A	107.5 (2)	O51—C51—N5	121.0 (3)
C4—C4A—C10A	114.6 (2)	O51—C51—C52	121.7 (3)
C9A—C5A—N5	119.0 (2)	N5—C51—C52	117.3 (3)
C10A—O1—C2—C3	58.8 (3)	C9A—N9—C8—N7	1.2 (4)
C31—O3—C3—C2	109.0 (3)	C81—O8—C8—N9	−0.4 (4)
C31—O3—C3—C4	−130.1 (3)	C81—O8—C8—N7	179.8 (3)
O1—C2—C3—O3	63.8 (3)	C6—N7—C8—N9	−5.3 (5)
O1—C2—C3—C4	−55.1 (3)	C7—N7—C8—N9	171.7 (3)
C41—O4—C4—C3	−86.9 (3)	C6—N7—C8—O8	174.5 (3)
C41—O4—C4—C4A	152.7 (2)	C7—N7—C8—O8	−8.5 (4)
O3—C3—C4—O4	167.8 (2)	C10A—N10—C9A—N9	176.1 (2)
C2—C3—C4—O4	−72.8 (3)	C10A—N10—C9A—C5A	−2.0 (4)
O3—C3—C4—C4A	−71.6 (3)	C8—N9—C9A—N10	−170.6 (2)
C2—C3—C4—C4A	47.9 (3)	C8—N9—C9A—C5A	7.5 (4)
C51—N5—C4A—C4	−130.5 (3)	N5—C5A—C9A—N10	−1.0 (4)
C5A—N5—C4A—C4	72.4 (3)	C6—C5A—C9A—N10	166.1 (2)
C51—N5—C4A—C10A	100.5 (3)	N5—C5A—C9A—N9	−179.0 (2)
C5A—N5—C4A—C10A	−56.7 (3)	C6—C5A—C9A—N9	−11.9 (4)
O4—C4—C4A—N5	−52.4 (3)	C2—O1—C10A—N10	71.1 (3)

C3—C4—C4A—N5	−170.5 (2)	C2—O1—C10A—C4A	−53.5 (3)
O4—C4—C4A—C10A	73.1 (3)	C9A—N10—C10A—O1	−149.9 (2)
C3—C4—C4A—C10A	−45.0 (3)	C9A—N10—C10A—C4A	−24.5 (3)
C51—N5—C5A—C9A	−123.4 (3)	N5—C4A—C10A—O1	176.8 (2)
C4A—N5—C5A—C9A	32.3 (3)	C4—C4A—C10A—O1	47.4 (3)
C51—N5—C5A—C6	69.5 (4)	N5—C4A—C10A—N10	51.8 (3)
C4A—N5—C5A—C6	−134.8 (3)	C4—C4A—C10A—N10	−77.7 (3)
C8—N7—C6—O6	−179.6 (3)	C3—O3—C31—O31	2.1 (6)
C7—N7—C6—O6	3.2 (4)	C3—O3—C31—C32	−176.0 (3)
C8—N7—C6—C5A	0.8 (4)	C4—O4—C41—O41	0.5 (5)
C7—N7—C6—C5A	−176.4 (3)	C4—O4—C41—C42	179.1 (3)
C9A—C5A—C6—O6	−172.3 (3)	C5A—N5—C51—O51	176.8 (3)
N5—C5A—C6—O6	−5.4 (4)	C4A—N5—C51—O51	22.0 (4)
C9A—C5A—C6—N7	7.2 (4)	C5A—N5—C51—C52	−1.7 (5)
N5—C5A—C6—N7	174.1 (2)	C4A—N5—C51—C52	−156.5 (3)
C9A—N9—C8—O8	−178.6 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N10—H10···O6^{i}	0.86	2.05	2.819 (3)	148

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