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First Example of a Crystalline Urazole Nucleoside with an L-Configured Sugar. (1*S*,2*S*)-Urazole α-L-Pyranosyl-2-deoxyriboside

P. D. Robinson, V. M. Kolb, P. A. Colloton and C. Y. Meyers

Abstract

The reaction of *L*-2-deoxyribose with urazole led to the formation of a single crystalline α-pyranoside diastereomer, (1*S*,2S)-urazole α-*L*-pyranosyl-2-deoxyriboside [IUPAC name: 1-(2-deoxy-α- *L-erythro*-pentopyranosyl) 1-(S),2-(S),4-triazolidine-3,5-dione, $C_7H_{11}N_3O_5$], which is the enantiomer of 1*R*,2*R*)-urazole α- *D*-pyranosyl-2-deoxyriboside obtained in the analogous reaction with *D*-2-deoxyribose previously reported. The absolute configurations were ascertained from the known configurations of the 2-deoxyribose moieties. This is the first example of a crystalline urazole nucleoside with an *L*-configured sugar.

Experimental

To a stirred solution of urazole (101 mg; 1 mmol; Aldrich) in 2.5 ml of water, 2-deoxy-*L*-ribose (134 mg; 1 mmol; Sigma) was added. The slightly cloudy solution became clear when warmed in a 348 K bath for a few minutes. No crystallization was evident from this solution kept in a tightly stoppered flask at room temperature for six months. The flask was then unstoppered and left open overnight, which led to the formation of white crystals. The restoppered flask was set aside for three more weeks, when no further crystallization was noticeable. The crystals were isolated by filtration, air-dried and then dried *in vacuo* for three days: 80 mg, mp 463 K (brown spots), 468–469 K (dec., brown oil). The enantiomer melted at 475–476 K (corr., dec.); (Robinson, Meyers, Kolb & Colloton, 1996). X-ray diffraction identified the crystals unequivocally as (1*S*,2S)-urazole α-L– pyranosyl-2-deoxyriboside; the absolute configuration was ascertained from the known configuration of the *L*-2-deoxyribose substrate.

Refinement

The H2, H4, H7', and H8' atoms were refined isotropically. All other H atoms are riding.

Computing details

Data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation,1996); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN PROCESS* (Molecular Structure Corporation, 1995); program(s) used to solve structure: *TEXSAN SHELXS86* (Sheldrick, 1986); program(s) used to refine structure: *TEXSAN* LS and *SHELXL93* (Sheldrick, 1993); molecular graphics: *TEXSAN ORTEP* (Johnson, 1965); software used to prepare material for publication: *TEXSAN*, *SHELXL93*, and *PLATON* (Spek, 1990).

1-(2-deoxy-α-*L-erythro*-pentopyranosyl) 1-(S),2-(S), 4-triazolidine-3,5-dione

CIF access

Crystal data

$C_7H_{11}N_3O_5$	$V = 454.59\ (14)$ Å^3
$M_r = 217.19$	$Z = 2$
Monoclinic, $P2_1$	Mo $K\alpha$
$a = 8.5400\ (11)$ Å	$\mu = 0.14$ mm^{-1}
$b = 5.2249\ (14)$ Å	$T = 296$ K
$c = 10.2686\ (8)$ Å	0.51 × 0.34 × 0.12 mm
$\beta = 97.191\ (8)^o$	

Data collection

Rigaku AFC-5S diffractometer	$R_{int} = 0.013$
Absorption correction: none	3 standard reflections
1902 measured reflections	every 100 reflections
1805 independent reflections	intensity decay: 0.4%
1392 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.034$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.091$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
1805 reflections	Absolute structure: Ascertained from the known configuration of the L-2-deoxyribose substrate.
153 parameters	Flack parameter: −0.84 (121)
1 restraint	

Acknowledgements

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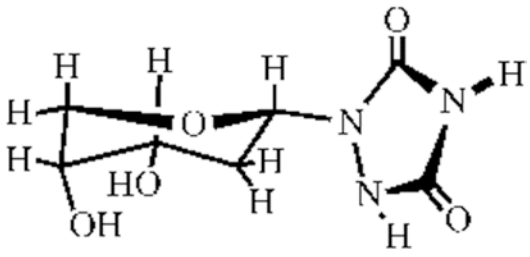
Scheme 1



supplementary materials

1-(2-deoxy-α-*L*-*erythro*-pentopyranosyl) 1-(S),2-(S), 4-triazolidine-3,5-dione

Crystal data

$C_7H_{11}N_3O_5$	$F_{000} = 228$
$M_r = 217.19$	$D_x = 1.587$ Mg m^{-3}
Monoclinic, $P2_1$	Mo $K\alpha$ radiation $\lambda = 0.71069$ Å
$a = 8.5400$ (11) Å	Cell parameters from 25 reflections
$b = 5.2249$ (14) Å	$\theta = 21.8$–24.8º
$c = 10.2686$ (8) Å	$\mu = 0.14$ mm^{-1}
$\beta = 97.191$ (8)º	$T = 296$ K
$V = 454.59$ (14) Å^3	Prism, colorless
$Z = 2$	0.51 × 0.34 × 0.12 mm

Data collection

Rigaku AFC-5S diffractometer	$R_{int} = 0.013$
Radiation source: X-ray tube	$\theta_{max} = 32.6$º
Monochromator: graphite	$\theta_{min} = 2.0$º
$T = 296$ K	$h = 0 \rightarrow 12$
ω (rate 3° min^{-1} in ω) scans	$k = 0 \rightarrow 7$
Absorption correction: none	$l = -15 \rightarrow 15$
1902 measured reflections	3 standard reflections
1805 independent reflections	every 100 reflections
1392 reflections with $I > 2\sigma(I)$	intensity decay: 0.4%

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H atoms treated by a mixture of independent and constrained refinement
$R[F^2 > 2\sigma(F^2)] = 0.034$	$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 0.0102P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.091$	$\Delta\rho_{max} = 0.26$ e Å^{-3}
$S = 1.04$	$\Delta\rho_{min} = -0.17$ e Å^{-3}
1805 reflections	Extinction correction: SHELXL, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
153 parameters	Extinction coefficient: 0.0128 (103)
1 restraint	Absolute structure: Ascertained from the known configuration of the L-2-deoxyribose substrate.
Primary atom site location: structure-invariant direct methods	Flack parameter: −0.84 (121)
Secondary atom site location: difference Fourier map	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
N1	0.7314 (2)	−0.0281 (3)	0.77402 (13)	0.0312 (3)
N2	0.7370 (2)	−0.2041 (4)	0.66960 (14)	0.0328 (3)
C3	0.8609 (2)	−0.3667 (4)	0.7033 (2)	0.0347 (4)
N4	0.9118 (2)	−0.3177 (4)	0.83454 (14)	0.0374 (4)
C5	0.8330 (2)	−0.1149 (4)	0.8794 (2)	0.0311 (3)
O6	0.8460 (2)	−0.0263 (4)	0.99043 (12)	0.0428 (4)
O7	0.9129 (2)	−0.5222 (4)	0.63293 (14)	0.0517 (4)
C1'	0.5826 (2)	0.0858 (3)	0.7927 (2)	0.0288 (3)
C2'	0.5048 (2)	0.2162 (4)	0.6695 (2)	0.0309 (3)
C3'	0.3503 (2)	0.3340 (3)	0.6993 (2)	0.0295 (3)
C4'	0.2452 (2)	0.1301 (4)	0.7488 (2)	0.0334 (3)
C5'	0.3364 (2)	−0.0070 (4)	0.8644 (2)	0.0364 (4)
O6'	0.48370 (14)	−0.1100 (2)	0.83395 (12)	0.0313 (3)
O7'	0.2752 (2)	0.4548 (3)	0.58403 (13)	0.0391 (3)
O8'	0.1948 (2)	−0.0540 (3)	0.65050 (15)	0.0428 (3)
H1'	0.6009 (2)	0.2138 (3)	0.8626 (2)	0.035*
H2'a	0.4846 (2)	0.0926 (4)	0.5990 (2)	0.037*
H2'b	0.5736 (2)	0.3482 (4)	0.6423 (2)	0.037*
H3'	0.3737 (2)	0.4639 (3)	0.7679 (2)	0.035*
H4'	0.1524 (2)	0.2125 (4)	0.7776 (2)	0.040*
H5'a	0.3571 (2)	0.1117 (4)	0.9372 (2)	0.044*
H5'b	0.2722 (2)	−0.1452 (4)	0.8920 (2)	0.044*
H2	0.729 (3)	−0.134 (6)	0.588 (2)	0.045 (7)*
H4	0.984 (3)	−0.399 (7)	0.884 (2)	0.044 (6)*
H7'	0.249 (4)	0.595 (9)	0.591 (3)	0.062 (9)*
H8'	0.140 (4)	0.006 (7)	0.590 (3)	0.063 (9)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0290 (6)	0.0371 (8)	0.0249 (6)	0.0008 (6)	−0.0069 (4)	−0.0018 (6)
N2	0.0325 (6)	0.0409 (8)	0.0227 (6)	0.0053 (6)	−0.0058 (5)	−0.0020 (6)
C3	0.0281 (7)	0.0446 (10)	0.0296 (7)	0.0032 (7)	−0.0035 (6)	0.0011 (8)
N4	0.0308 (6)	0.0502 (10)	0.0280 (7)	0.0083 (7)	−0.0091 (5)	0.0032 (7)
C5	0.0242 (6)	0.0401 (9)	0.0265 (7)	−0.0061 (6)	−0.0066 (5)	0.0023 (7)
O6	0.0376 (6)	0.0592 (9)	0.0277 (6)	−0.0056 (7)	−0.0109 (5)	−0.0062 (7)
O7	0.0454 (7)	0.0649 (11)	0.0425 (7)	0.0215 (8)	−0.0035 (6)	−0.0083 (8)
C1'	0.0315 (7)	0.0265 (7)	0.0262 (7)	−0.0007 (6)	−0.0051 (5)	0.0011 (6)
C2'	0.0329 (7)	0.0288 (7)	0.0294 (7)	0.0004 (6)	−0.0023 (6)	0.0059 (6)
C3'	0.0421 (8)	0.0196 (7)	0.0245 (6)	0.0053 (6)	−0.0049 (6)	−0.0013 (5)
C4'	0.0344 (7)	0.0290 (8)	0.0363 (8)	0.0073 (7)	0.0025 (6)	0.0014 (7)
C5'	0.0379 (8)	0.0404 (10)	0.0318 (7)	0.0064 (8)	0.0071 (6)	0.0061 (8)
O6'	0.0338 (6)	0.0272 (6)	0.0320 (6)	0.0022 (5)	0.0007 (4)	0.0062 (5)
O7'	0.0547 (8)	0.0316 (7)	0.0286 (6)	0.0171 (7)	−0.0040 (5)	0.0016 (5)

O8'	0.0472 (7)	0.0282 (6)	0.0472 (8)	0.0001 (6)	−0.0172 (6)	0.0023 (6)

Geometric parameters (Å, °)

O6'—C1'	1.424 (2)	N1—C1'	1.437 (2)
O6'—C5'	1.438 (2)	N2—C3	1.368 (2)
O6—C5	1.222 (2)	N4—C5	1.366 (3)
O7'—C3'	1.422 (2)	N4—C3	1.387 (2)
O7—C3	1.208 (3)	C1'—C2'	1.515 (2)
O8'—C4'	1.421 (2)	C2'—C3'	1.521 (2)
N1—C5	1.377 (2)	C3'—C4'	1.521 (3)
N1—N2	1.418 (2)	C4'—C5'	1.516 (2)
C1'—O6'—C5'	111.33 (14)	C4'—C3'—C2'	110.29 (14)
C5—N1—N2	107.7 (2)	O7—C3—N2	126.6 (2)
C5—N1—C1'	120.83 (14)	O7—C3—N4	127.7 (2)
N2—N1—C1'	118.89 (13)	N2—C3—N4	105.7 (2)
C3—N2—N1	108.03 (13)	O8'—C4'—C5'	108.4 (2)
C5—N4—C3	111.31 (15)	O8'—C4'—C3'	112.09 (14)
O6'—C1'—N1	108.03 (14)	C5'—C4'—C3'	109.0 (2)
O6'—C1'—C2'	110.92 (13)	O6—C5—N4	128.1 (2)
N1—C1'—C2'	112.21 (14)	O6—C5—N1	125.7 (2)
C1'—C2'—C3'	108.37 (13)	N4—C5—N1	106.20 (14)
O7'—C3'—C4'	111.41 (15)	O6'—C5'—C4'	112.55 (14)
O7'—C3'—C2'	108.93 (13)		
C5—N1—N2—C3	−11.0 (2)	C5—N4—C3—N2	−5.5 (2)
C1'—N1—N2—C3	−153.2 (2)	O7'—C3'—C4'—O8'	55.1 (2)
C5'—O6'—C1'—N1	175.56 (13)	C2'—C3'—C4'—O8'	−66.0 (2)
C5'—O6'—C1'—C2'	−61.1 (2)	O7'—C3'—C4'—C5'	175.20 (14)
C5—N1—C1'—O6'	−69.2 (2)	C2'—C3'—C4'—C5'	54.1 (2)
N2—N1—C1'—O6'	67.9 (2)	C3—N4—C5—O6	177.9 (2)
C5—N1—C1'—C2'	168.2 (2)	C3—N4—C5—N1	−1.2 (2)
N2—N1—C1'—C2'	−54.6 (2)	N2—N1—C5—O6	−171.8 (2)
O6'—C1'—C2'—C3'	60.0 (2)	C1'—N1—C5—O6	−30.5 (3)
N1—C1'—C2'—C3'	−179.09 (14)	N2—N1—C5—N4	7.4 (2)
C1'—C2'—C3'—O7'	−179.36 (14)	C1'—N1—C5—N4	148.7 (2)
C1'—C2'—C3'—C4'	−56.8 (2)	C1'—O6'—C5'—C4'	58.7 (2)
N1—N2—C3—O7	−170.9 (2)	O8'—C4'—C5'—O6'	67.6 (2)
N1—N2—C3—N4	9.9 (2)	C3'—C4'—C5'—O6'	−54.7 (2)
C5—N4—C3—O7	175.3 (2)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2···O7'^{i}	0.91 (2)	1.82 (2)	2.723 (2)	170 (3)
N4—H4···O6^{ii}	0.86 (3)	1.93 (3)	2.787 (2)	170 (3)
O7'—H7'···O8'^{iii}	0.77 (5)	2.01 (4)	2.764 (2)	168 (3)
O8'—H8'···O7^{iv}	0.79 (3)	2.28 (3)	2.945 (2)	142 (3)

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