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(1 α ,2 β ,3 α ,6 α ,7 β ,8 α ,9 β ,13 β)-9,13-Di-bromo-11-methyl-14,15-dioxa-11-azapenta-cyclo[6.5.1.1^{3,6}.0^{2,7}.0^{9,13}]pentadec-4-ene-10,12-dione

RONALD N. WARRENER,^a L. MAKSIMOVIC^a AND COLIN H. L. KENNARD^b

^aCentre for Molecular Architecture, Central Queensland University, Rockhampton, Queensland 4702, Australia, and ^bDepartment of Chemistry, The University of Queensland, Brisbane, Queensland 4072, Australia. E-mail: c.kennard@mailbox.uq.edu.au

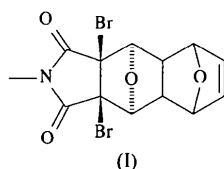
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Abstract

The crystal structure of the minor 2:1 isomer (C₁₃H₁₁Br₂NO₄) formed by the reaction of furan with *N*-methyl-3,4-dibromomaleimide is reported.

Comment

The reaction of furan with *N*-methyl-3,4-dibromomaleimide produces a single 1:1 adduct when conducted in neat furan (sealed vessel) at 443 K for 17 h in 40% yield together with unreacted maleimide (50%). Increasing the reaction time (3 days) leads to the formation of the 1:1 adduct plus two isomeric 2:1 adducts (ratio 3:1) (Warrener, Maksimovic & Butler, 1994) which have different stereochemistry of the terminal 7-oxanorbornane ring. It was not possible to obtain single crystals of the 1:1 adduct nor to determine its stereochemistry by spectroscopic means. The title 2:1 adduct [(I); minor



isomer, m.p. 516–521 K] not only provides interesting data in its own right, but also defines the stereochemistry of the succinimide ring. The stereochemical information obtained herein is critical to a theoretical study of the specificity of cycloaddition reactions of furan and cyclopentadiene with halo-substituted dienophiles (Warrener & Margetic, 1996).

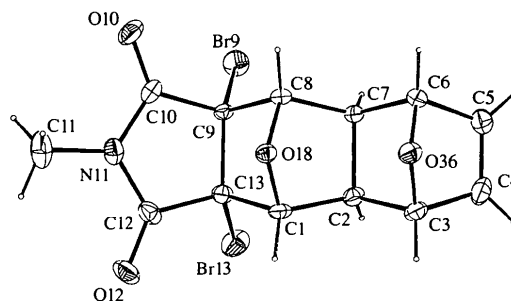


Fig. 1. Plot of the title compound with displacement ellipsoids plotted at the 20% probability level.

Experimental

The reaction of furan with *N*-methyl-3,4-dibromomaleimide yields a 1:1 adduct which on further heating produces two 2:1 adducts.

Crystal data

C₁₃H₁₁Br₂NO₄
M_r = 405.05
 Monoclinic
*P*2₁/*a*
a = 7.377 (1) Å
b = 18.664 (3) Å
c = 9.897 (2) Å
 β = 95.314 (8)°
V = 1356.8 (4) Å³
Z = 4
D_x = 1.983 Mg m⁻³
D_m not measured

Mo K α radiation
 λ = 0.71073 Å
 Cell parameters from 23 reflections
 θ = 6–14°
 μ = 5.986 mm⁻¹
T = 298 (2) K
 Needle
 0.23 × 0.15 × 0.11 mm
 Colourless

Data collection

CAD-4 MicroVAX-controlled diffractometer
 2 θ / ω scans
 Absorption correction: empirical ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.21, *T_{max}* = 0.52
 3412 measured reflections
 3412 independent reflections

1913 reflections with *I* > 2 σ (*I*)
 θ_{\max} = 29.9°
h = 0 → 8
k = 0 → 26
l = -13 → 13
 3 standard reflections every 250 reflections
 intensity decay: none

Refinement

Refinement on *F*²
R[*F*² > 2 σ (*F*²)] = 0.047
 wR (*F*²) = 0.110

$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 1.0814P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$S = 1.098$
3411 reflections
183 parameters
H atoms in calculated
positions, but not refined

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.623 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.958 \text{ e } \text{Å}^{-3}$
Extinction correction: none
Scattering factors from
*International Tables for
Crystallography* (Vol. C)

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Table 1. Selected geometric parameters (Å, °)

C1—O14	1.441 (5)	C8—O14	1.445 (5)
C1—C2	1.520 (7)	C8—C9	1.540 (6)
C1—C13	1.554 (6)	C9—C10	1.519 (6)
C2—C3	1.556 (6)	C9—C13	1.556 (6)
C2—C7	1.562 (6)	C9—Br9	1.925 (4)
C3—O15	1.439 (6)	C10—O10	1.207 (6)
C3—C4	1.505 (7)	C10—N11	1.366 (7)
C4—C5	1.317 (8)	N11—C12	1.372 (7)
C5—C6	1.518 (7)	N11—C11	1.468 (7)
C6—O15	1.437 (6)	C12—O12	1.209 (6)
C6—C7	1.559 (6)	C12—C13	1.506 (7)
C7—C8	1.521 (6)	C13—Br13	1.926 (4)
O14—C1—C2	104.6 (3)	O10—C10—N11	124.8 (5)
C4—C5—C6	105.4 (5)	O10—C10—C9	126.6 (5)
O15—C6—C5	101.7 (4)	N11—C10—C9	108.6 (4)
O15—C6—C7	102.2 (4)	C10—N11—C12	113.9 (4)
C5—C6—C7	104.9 (4)	C10—N11—C11	121.9 (5)
C8—C7—C6	115.5 (4)	C12—N11—C11	124.2 (5)
C8—C7—C2	101.8 (4)	O12—C12—N11	124.2 (5)
C6—C7—C2	100.5 (3)	O12—C12—C13	126.5 (5)
O14—C8—C7	104.4 (3)	N11—C12—C13	109.2 (4)
O14—C8—C9	99.0 (3)	C12—C13—C1	111.0 (4)
C7—C8—C9	110.6 (3)	C12—C13—C9	104.0 (4)
C10—C9—C8	110.2 (4)	C1—C13—C9	101.1 (3)
C10—C9—C13	104.3 (4)	C12—C13—Br13	107.5 (3)
C8—C9—C13	101.6 (3)	C1—C13—Br13	114.3 (3)
C10—C9—Br9	107.0 (3)	C9—C13—Br13	118.5 (3)
C8—C9—Br9	114.5 (3)	C1—O14—C8	97.1 (3)
C13—C9—Br9	118.7 (3)	C6—O15—C3	95.9 (3)

Data collection: *SDP-Plus* (Frenz, 1985). Cell refinement: *SDP-Plus*. Data reduction: *Xtal3.2 DIFDAT* (Hall, Flack & Stewart, 1992). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *PLATON96* (Spek, 1996). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: TA1163). Services for accessing these data are described at the back of the journal.

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(1*R*,2*R*)-4-Methylurazole- α -D-pyranosyl-2-deoxyriboside from Reaction of 4-Methylurazole with 2-Deoxy-D-ribose

CAL Y. MEYERS,^a YUQING HOU,^a VERA M. KOLB^b AND PAUL D. ROBINSON^c

^aDepartment of Chemistry and Biochemistry, Southern Illinois University 4409, Carbondale, IL 62901, USA,

^bDepartment of Chemistry, University of Wisconsin–Parkside, Kenosha, WI 53141-2000, USA, and ^cDepartment of Geology, Southern Illinois University 4324, Carbondale, IL 62901, USA. E-mail: robinson@geo.siu.edu

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Abstract

The crystalline nucleoside formed from an aqueous solution of 4-methylurazole and 2-deoxy-D-ribose was identified unequivocally by its X-ray structure as a single diastereomer, (1*R*,2*R*)-4-methylurazole- α -D-pyranosyl-2-deoxyriboside [IUPAC name: (1*R*,2*R*)-1-(2-deoxy- α -D-erythro-pentopyranosyl)-4-methyl-1,2,4-triazolidine-3,5-dione; C₈H₁₃N₃O₅]. In its urazole ring, the two hydrazidic N atoms are pyramidal and confer *R* chirality, the respective pyranosyl and H-atom substituents being substantially out of the ring plane and *trans* juxtaposed, while the imidic N atom is trigonal planar, its methyl substituent being essentially coplanar with the ring. The intermolecular hydrogen bonding involves pyranose–pyranose and pyranose–urazole interactions producing one-dimensional cross-linked double chains. From this study and our results from the preparation of three related nucleosides, we find that the crystalline nucleosides formed by direct coupling of these nucleobases with 2-deoxyribose are consistently diastereospecific.

Comment

Our studies of the crystalline structures of 1-methylurazole, 4-methylurazole and 1,2,4-trimethylurazole showed them to be racemic molecules (Meyers, Lutfi, Hou & Robinson, 1995, 1997; Meyers, Lutfi, Kolb & Robinson, 1994). While their imidic N4 atoms are trig-