

Related literature. The U^{III} dimer, [U{C₃H₃(SiMe₃)₂-Cl]₂, has previously been prepared and structurally characterized (Blake, Lappert, Taylor, Atwood, Hunter & Zhang, 1986). The ability of isocyanides to act as good donor ligands toward uranium metal centers has been previously observed (Kanellakopoulos, Fischer, Dornberger & Baumgartner, 1970).

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Structure of (4*R*,5*R*)-4-(*D*-arabino-1,2,3,4-Tetraacetoxybutyl)-1,2-dimethyl-5-nitro-1-cyclohexene

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Abstract. C₂₀H₂₉NO₁₀, *M_r* = 443.45, triclinic, *P*1, *a* = 7.646 (2), *b* = 10.085 (3), *c* = 15.490 (4) Å, α = 78.73 (1), β = 88.31 (1), γ = 80.70 (1)°, *V* = 1156 (1) Å³, *Z* = 2, *D_m* = 1.242 (6), *D_x* = 1.274 (1) Mg m⁻³, λ(Mo *K*α) = 0.71069 Å, μ = 0.11 mm⁻¹, *F*(000) = 472, *T* = 297 K, *R* = 0.056 for 3306 unique observed reflections. The structure consists of two (CH₃)₂C₆H₆(NO₂)(CHOCOCH₃)₂CH₂-OCOCH₃ molecules, which are almost identical in their bond distances and angles but different in the conformation of their cyclohexene rings: Δ*C*₂(*C*1*A*,*C*2*A*) = 7.9 and Δ*C*₂(*C*1*B*,*C*2*B*) = 2.4° [Duax, Weeks & Rohrer (1976). *Top. Stereochem.* **9**, 271–273].

Experimental. The compound was prepared by Diels–Alder addition of 2,3-dimethyl-1,3-butadiene and *trans*-

3,4,5,6-tetra-*O*-acetyl-1-nitro-*D*-arabino-1-hexene-3,4,5,6-tetrol. The crystals were obtained by recrystallization from ethanol [m.p. 360.8 (3) K] and are optically active ([α]_D^{20.0°C} = 0 (1.6)° [chloroform, 16.7 g dm⁻³]) (Pacák, 1987). Colourless crystals were stable in air and to X-rays. Density was determined pycnometrically. A prismatic crystal of dimensions 0.2 × 0.3 × 0.5 mm was used for the measurements with an automatic Stoe four-circle diffractometer with graphite monochromator. The lattice parameters were obtained by least-squares fit of 44 reflections with 10 ≤ 2θ ≤ 20°. Intensity data were collected using ω–2θ scan; absorption ignored; max. (sinθ)/λ = 0.60 Å⁻¹; *h*–8→0, *k*–11→11, *l*–18→18; three standard reflections measured after 50 reflections with no significant variation; 4247 measured reflections, 3950 unique reflections, 3306 observed reflections with *I* ≥ 3σ(*I*). The structure was solved by direct methods;

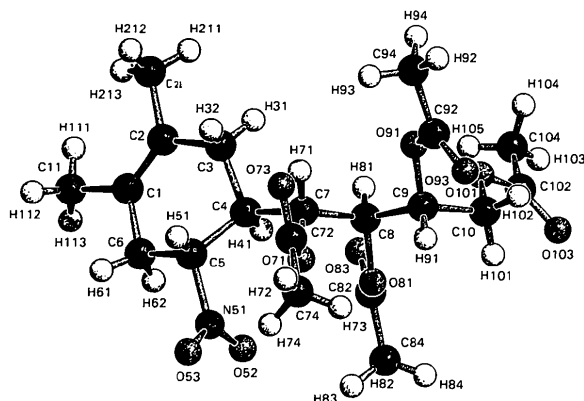
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Table 1. Atomic coordinates ($\times 10^4$) and equivalent isotropic thermal parameters ($\times 10^3$, \AA^2) of non-H atoms with e.s.d.'s in parentheses
$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C1A	-74 (12)	8445 (7)	1715 (4)	81 (3)
C11A	316 (15)	9324 (10)	842 (5)	157 (6)
C2A	-1653 (11)	8402 (6)	2061 (4)	72 (3)
C21A	-3371 (11)	9128 (8)	1615 (5)	107 (4)
C3A	-1900 (8)	7564 (6)	2962 (4)	63 (2)
C4A	-237 (8)	7173 (6)	3520 (4)	59 (2)
C5A	1241 (9)	6580 (7)	2988 (4)	69 (3)
N51A	3013 (8)	6155 (8)	3466 (4)	86 (3)
O52A	3491 (7)	6940 (6)	3880 (3)	96 (2)
O53A	3842 (8)	5064 (7)	3388 (4)	129 (3)
C6A	1598 (11)	7641 (8)	2162 (4)	92 (4)
C7A	-665 (7)	6273 (5)	4405 (3)	45 (2)
O71A	605 (5)	5034 (4)	4666 (2)	49 (1)
C72A	46 (10)	3850 (7)	4629 (5)	74 (3)
O73A	-1338 (8)	3799 (5)	4354 (5)	144 (3)
C74A	1404 (10)	2651 (6)	5005 (5)	90 (3)
C8A	-837 (7)	7048 (5)	5164 (3)	42 (2)
O81A	876 (5)	7285 (4)	5403 (2)	46 (1)
C82A	1218 (9)	8585 (6)	5236 (4)	56 (2)
O83A	234 (6)	9527 (4)	4847 (3)	78 (2)
C84A	2979 (8)	8635 (6)	5609 (5)	78 (3)
C9A	-1556 (7)	6264 (5)	6004 (3)	42 (2)
O91A	-3237 (5)	5967 (4)	5754 (2)	46 (1)
C92A	-3712 (9)	4770 (6)	6147 (4)	59 (2)
O93A	-2930 (7)	4048 (5)	6757 (3)	100 (2)
C94A	-5317 (8)	4504 (6)	5725 (4)	64 (3)
C10A	-1783 (8)	7035 (6)	6752 (3)	52 (2)
O101A	-2712 (5)	8380 (4)	6422 (2)	54 (1)
C102A	-2536 (11)	9360 (7)	6874 (4)	73 (3)
O103A	-1638 (8)	9134 (5)	7509 (4)	118 (3)
C104A	-3587 (12)	10693 (7)	6478 (5)	109 (4)
C1B	5436 (8)	1933 (6)	2904 (4)	58 (2)
C11B	5735 (10)	1042 (8)	3811 (4)	92 (3)
C2B	3944 (8)	2139 (6)	2454 (4)	52 (2)
C21B	2313 (8)	1528 (7)	2765 (4)	79 (3)
C3B	3733 (7)	3021 (6)	1559 (3)	57 (2)
C4B	5453 (7)	3351 (5)	1094 (3)	47 (2)
C5B	6619 (7)	3730 (6)	1767 (4)	55 (2)
N51B	8463 (7)	3967 (7)	1411 (4)	70 (2)
O52B	9271 (6)	3115 (6)	1020 (3)	90 (2)
O53B	8984 (7)	4980 (5)	1551 (3)	107 (3)
C6B	6996 (8)	2577 (6)	2571 (4)	66 (2)
C7B	5028 (7)	4486 (5)	272 (3)	42 (2)
O71B	6624 (5)	4968 (4)	-88 (2)	49 (1)
C72B	6732 (10)	6306 (7)	-101 (4)	68 (3)
O73B	5603 (7)	7073 (5)	176 (3)	90 (2)
C74B	8462 (9)	6614 (7)	-501 (5)	92 (3)
C8B	4135 (7)	3999 (5)	-458 (3)	43 (2)
O81B	5254 (5)	2799 (4)	-665 (2)	45 (1)
C82B	4558 (9)	1605 (6)	-551 (4)	54 (2)
O83B	3179 (6)	1470 (4)	-221 (3)	83 (2)
C84B	5837 (9)	531 (6)	-866 (4)	68 (3)
C9B	3940 (7)	5040 (5)	-1318 (3)	49 (2)
O91B	2899 (5)	6281 (4)	-1128 (2)	48 (1)
C92B	3297 (9)	7460 (7)	-1611 (4)	61 (3)
O93B	4432 (7)	7518 (5)	-2145 (3)	98 (2)
C94B	2136 (8)	8660 (6)	-1375 (4)	70 (3)
C10B	3129 (7)	4556 (6)	-2056 (3)	59 (2)
O101B	1481 (5)	4098 (4)	-1741 (2)	60 (2)
C102B	1117 (9)	2987 (7)	-2012 (5)	68 (3)
O103B	1996 (8)	2457 (6)	-2533 (4)	114 (3)
C104B	-486 (8)	2515 (7)	-1557 (4)	88 (3)

Table 2. Selected interatomic distances (\AA) and angles ($^\circ$)

Molecule A		Molecule B		Molecule A		Molecule B	
C1-C11	1.516 (10)	1.514 (8)	C7-C8	1.525 (8)	1.534 (8)		
C1-C2	1.310 (12)	1.321 (8)	C8-O81	1.440 (7)	1.450 (6)		
C2-C21	1.511 (11)	1.504 (9)	O81-C82	1.353 (8)	1.372 (7)		
C2-C3	1.508 (8)	1.491 (7)	C82-O83	1.185 (7)	1.174 (8)		
C3-C4	1.513 (8)	1.533 (8)	C82-C84	1.493 (10)	1.485 (8)		
C4-C5	1.495 (9)	1.532 (8)	C8-C9	1.522 (7)	1.521 (7)		
C5-N51	1.522 (9)	1.536 (8)	C9-O91	1.445 (7)	1.448 (6)		
N51-O52	1.216 (10)	1.224 (8)	O91-C92	1.342 (7)	1.349 (7)		
N51-O53	1.203 (10)	1.213 (9)	C92-O93	1.185 (7)	1.182 (8)		
C5-C6	1.548 (9)	1.528 (7)	C92-C94	1.492 (10)	1.480 (9)		
C6-C1	1.505 (11)	1.481 (9)	C9-C10	1.508 (8)	1.513 (8)		
C4-C7	1.547 (7)	1.540 (7)	C10-O101	1.430 (6)	1.450 (7)		
C7-O71	1.449 (6)	1.443 (6)	O101-C102	1.343 (9)	1.342 (9)		
O71-C72	1.343 (9)	1.361 (9)	C102-O103	1.181 (9)	1.187 (9)		
C72-O73	1.164 (10)	1.191 (9)	C102-C104	1.478 (10)	1.493 (10)		
C72-C74	1.494 (9)	1.497 (10)					
C1-C2-C3	121.2 (6)	121.8 (5)	O71-C7-C8	107.7 (4)	107.8 (4)		
C2-C3-C4	114.2 (6)	115.8 (4)	O71-C72-O73	123.0 (6)	123.6 (7)		
C3-C4-C5	108.7 (5)	107.7 (4)	O71-C72-C74	111.0 (6)	109.1 (6)		
C4-C5-C6	110.8 (5)	111.5 (5)	O73-C72-C74	126.0 (7)	127.3 (7)		
C5-C6-C1	113.1 (6)	114.3 (5)	C7-C8-C9	113.5 (5)	113.1 (5)		
C6-C1-C2	123.4 (6)	122.9 (5)	C7-C8-O81	110.4 (4)	108.8 (4)		
C11-C1-C2	125.0 (7)	124.3 (6)	C8-O81-C82	118.3 (4)	117.7 (4)		
C1-C2-C21	125.1 (6)	125.1 (5)	O81-C82-O83	123.5 (6)	123.7 (5)		
C21-C2-C3	113.7 (6)	113.1 (5)	O81-C82-C84	110.2 (5)	109.8 (5)		
C3-C4-C7	108.6 (5)	110.1 (4)	O83-C82-C84	126.4 (6)	126.4 (6)		
C7-C4-C5	117.0 (5)	113.5 (5)	C8-C9-C10	114.1 (5)	113.9 (5)		
C4-C5-N51	114.5 (5)	113.3 (5)	C8-O91	105.1 (4)	106.9 (4)		
C5-N51-O52	118.2 (6)	117.1 (6)	C9-O91-C92	117.6 (4)	115.5 (4)		
C5-N51-O53	116.5 (7)	116.7 (5)	O91-C92-O93	122.5 (7)	124.1 (6)		
O52-N51-O53	125.3 (6)	126.2 (6)	O91-C92-C94	111.5 (5)	111.0 (5)		
N51-C5-C6	104.9 (6)	104.4 (4)	O93-C92-C94	126.0 (6)	124.9 (6)		
C5-C6-C1	113.1 (6)	114.3 (5)	C9-C10-O101	108.0 (4)	108.2 (4)		
C6-C1-C11	111.6 (7)	112.8 (5)	C10-O101-C102	117.1 (5)	116.1 (5)		
C4-C7-C8	112.6 (4)	112.5 (4)	O101-C102-O103	121.9 (6)	123.2 (7)		
C4-C7-O71	114.3 (4)	110.7 (4)	O101-C102-C104	111.9 (6)	111.0 (6)		
C7-O71-C72	116.4 (5)	116.9 (4)	O103-C102-C104	126.3 (6)	125.8 (7)		

Fig. 1. A view of the molecule $C_{20}H_{29}NO_{10}$ (A) with atomic numbering.

F magnitudes were refined in full-matrix least-squares refinement in two blocks (one molecule fixed, another refined, in this way no explicit fixation of the origin was needed). H atoms were localized in calculated positions with C-H distance 0.96 \AA ; anisotropic thermal parameters of non-H atoms were refined; $R = 0.056$, $wR = 0.038$, $w = 1.76/\sigma^2(F)$; in the last refinement cycle $(\Delta/\sigma)_{\max} = -0.717$, for U_{22} of C(82A); $\Delta\rho_{\max}/\Delta\rho_{\min} = 0.22/-0.21 \text{ e \AA}^{-3}$; 595 parameters were refined. Siemens 7-882 computer was used with programs

SHELX76 (Sheldrick, 1976), *SHELXS86* (Sheldrick, 1986) and *SCHAKAL* (Keller, 1986). Atomic scattering factors were taken from *SHELX76*.*

* Lists of atomic coordinates and isotropic thermal parameters for H atoms, anisotropic thermal parameters for non-H atoms and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51664 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

The final atomic parameters are given in Table 1. Selected interatomic distances and angles are given in Table 2. Molecule *A* is depicted in Fig. 1. The greatest differences between the distances and angles of molecules *A* and *B* are 0.037 (4.4 σ) and 0.027 Å (2.8 σ) in C4—C5 and C72—O73, respectively, and 3.6 (9.0 σ) and 3.5° (7.0 σ) in C4—C7—O7 and C7—C4—C5, respectively. The shortest intermolecular contacts are O(103*A*)...H(94*B*ⁱ)—C(94*B*ⁱ) [2.54, 0.96 Å, 140°, (i) = *x*, *y*, 1+*z*], C(104*A*)—H(102*A*)...O(93*B*ⁱⁱ) [2.65, 0.96 Å, 128°, (ii) = 1+*x*, *y*, *z*−1] and O(93*A*)...H(104*B*ⁱ)—C(104*B*ⁱ) (2.67, 0.96 Å, 118°).

Related literature. The bond distances and angles in cyclohexene, acetoxybutyl and in the chain C4—C7—C8—C9—C10 are similar to those reported by Allen, Kennard, Watson, Brammer, Orpen & Taylor (1987), Gieren & Kokkinidis (1986) and Jiménez-Garay, López-Castro & Márquez (1976), respectively.

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Structure of *cyclo(-N-Hydroxyglycyl-L-alanyl-)*

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Abstract. C₅H₈N₂O₃, *M_r* = 144.1, orthorhombic, *P*2₁2₁2₁, *a* = 18.081 (3), *b* = 7.760 (1), *c* = 4.615 (1) Å, *V* = 647 (1) Å³, *Z* = 4, *D_x* = 1.478, *D_m*(flotation in toluene/carbon tetrachloride) = 1.468 (6) Mg m^{−3}, λ(Cu *Kα*) = 1.5418 Å, μ(Cu *Kα*) = 1.011 mm^{−1}, *F*(000) = 304.0, *T* = 296 K, *R* = 0.042, *wR* = 0.061, for 590 observations. The present diketopiperazine ring adopts an almost flat conformation. There are two kinds of hydrogen bonds: O₃—H...O₂($\frac{3}{2}$ −*x*, 1−*y*, $\frac{1}{2}$ +*z*) 2.599 (1) Å; and N₂—H...O₁(2−*x*, − $\frac{1}{2}$ +*y*, $\frac{3}{2}$ −*z*) 2.878 (1) Å.

Experimental. Colorless cubic crystals by slow evaporation of an aqueous methanol solution at room temperature. Crystal 0.3 × 0.3 × 0.05 mm. Rigaku-Denki RASA 5R-II automated diffractometer. Systematic absences *h*00 for *h* odd, 0*k*0 for *k* odd, 00*l* for *l* odd. Cell constants by least squares using 15 reflec-

tions with 40° ≤ 2θ(Cu) ≤ 80° measured on the diffractometer. Intensity data collected in ω−2θ scan mode. 603 independent reflections, 2θ ≤ 120.0°, 0 ≤ *h* ≤ 20, 0 ≤ *k* ≤ 8, 0 ≤ *l* ≤ 5. No systematic fluctuations in 11 $\bar{3}$, 732, 91 $\bar{2}$, monitored every 100 reflections.

Structure determined using *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Block-diagonal least-squares refinement (*HBL*SIV: Ashida, 1981). $\sum w(|F_o| - k|F_c|)^2$ minimized. Weighting scheme: $w = [\sigma^2(F_o) + 0.0590|F_o| + 0.0013|F_o|^2]^{-1}$. Subsequent difference Fourier maps revealed positions for all 8 H atoms; however, all H atoms were fixed at calculated positions (with C—H = 1.08 Å) with fixed isotropic temperature factors (*B* = 5.0 Å²). Final cycle of block-diagonal least squares gave *R* = 0.042, *wR* = 0.061, *S* = 0.69, using 590 observations with |*F_o*| > 2σ(*F_o*) and 92 variables. (Δ/σ)_{max} = 0.033. Final difference map contained no peak higher than 0.4 e Å^{−3}. Atomic scattering factors

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