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X-RAY CRYSTAL, AND MOLECULAR, STRUCTURE OF 1,2,3,4-TETRA-*O*-ACETYL-5,6-DIDEOXY-6-*C*-NITRO-5-(PHENYLPHOSPHINYL)-L-IDO-PYRANOSE: A CORRECTION OF OUR PREVIOUS WORK THEREON

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ABSTRACT

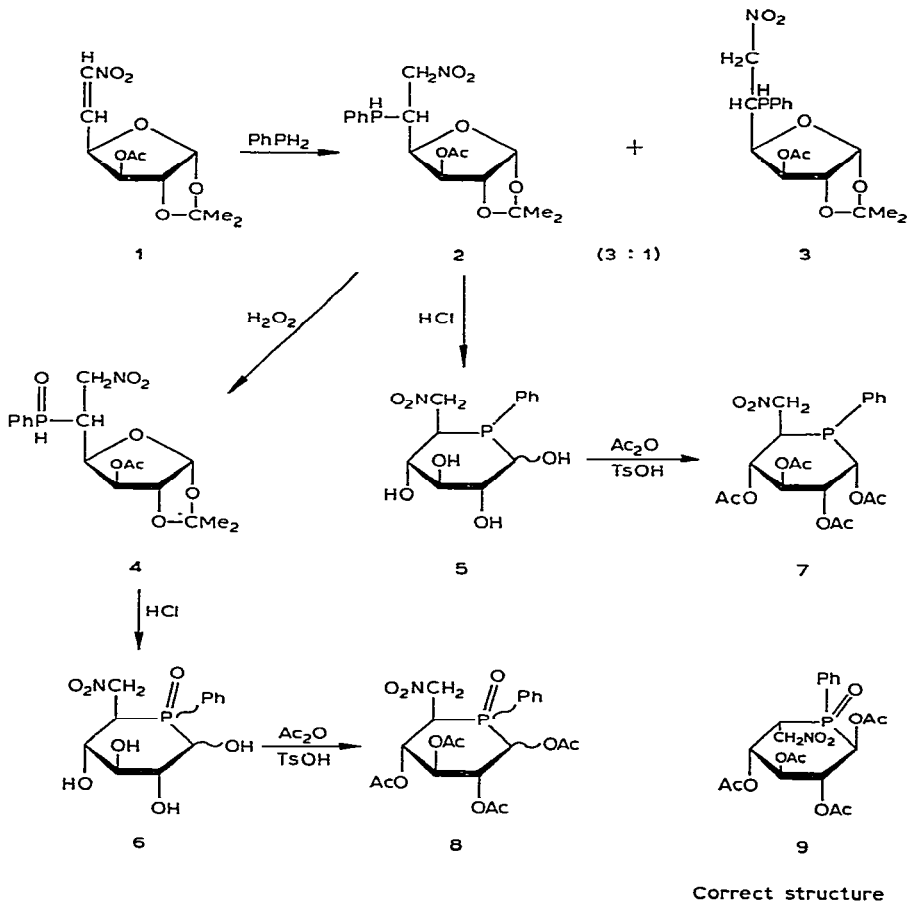
X-Ray crystallographic analysis was performed on the compound to which had been assigned the structure of 1,2,3,4-tetra-*O*-acetyl-5,6-dideoxy-6-*C*-nitro-5-(phenylphosphinyl)-D-glucopyranose. The results showed that the compound has the L-*ido* configuration, the pyranoid ring is in the $^4C_1$(L) conformation, the acetoxyl groups at C-1 and C-5 and the phenyl ring on P are linked axially, and the acetoxyl groups at C-2, C-3, and C-4 are linked equatorially.

INTRODUCTION

In a previous paper[1], we reported the synthesis, by the following processes, of compounds to which were assigned the structures of 1,2,3,4-tetra-*O*-acetyl-5,6-dideoxy-6-*C*-nitro-5-(phenylphosphono)-D-glucopyranose (**7**) and 1,2,3,4-tetra-*O*-acetyl-5,6-dideoxy-6-*C*-nitro-5-(phenylphosphinyl)-D-glucopyranose (**8**).

The configuration of **2** was considered to be D-*gluco* from a study of its Cotton effects[2] $\{[\theta]_{299\,nm}$ −12,460 (trough) (*c* 0.2, methanol)$\}$, and the conformation of **7** to be $^4C_1$(D) from a study of its p.m.r. spectrum, although the situation at the phosphorus atom was unknown. The conformation of **8** was not determined, because its p.m.r. spectrum was not well resolved, even at 100 MHz.

A precise, X-ray crystallographic analysis of any sugar having phosphorus in the hemiacetal ring has not hitherto been reported; a precise, X-ray crystallographic analysis of **8** would be of value, not only from the viewpoint of X-ray crystallography, but also from that of molecular biology.



RESULTS AND DISCUSSION

As Fig. 1 shows[3], compound **8**, to which had been assigned the structure of 1,2,3,4-tetra-*O*-acetyl-5,6-dideoxy-6-*C*-nitro-5-(phenylphosphinyl)-D-glucopyranose, actually has the L-*ido* configuration (**9**). The pyranoid ring is in the $^4C_1$(L) conformation, the substituents at C-1 and C-5, and the phenyl ring at P-5, are linked axially, and those at C-2, C-3, and C-4, equatorially.

The atom-numbering scheme and the average bond-lengths are given in Fig. 2. All bond-lengths, valence angles, and a choice of torsion angles are listed in Tables I–IV. There is no significant difference in the structural results for the two independent molecules.

The geometry of the pyranoid ring is that of an almost ideal chair, as the Cremer–Pople puckering[4,5] parameters [Q = 0.061 (0.059, mol. 2) nm, θ = 3.7 (3.8)°, φ = 338.7 (329.2)°] and the ring-torsion angles show.

The acetyl groups have the usual orientation, *i.e.*, the C=O (carbonyl) bond

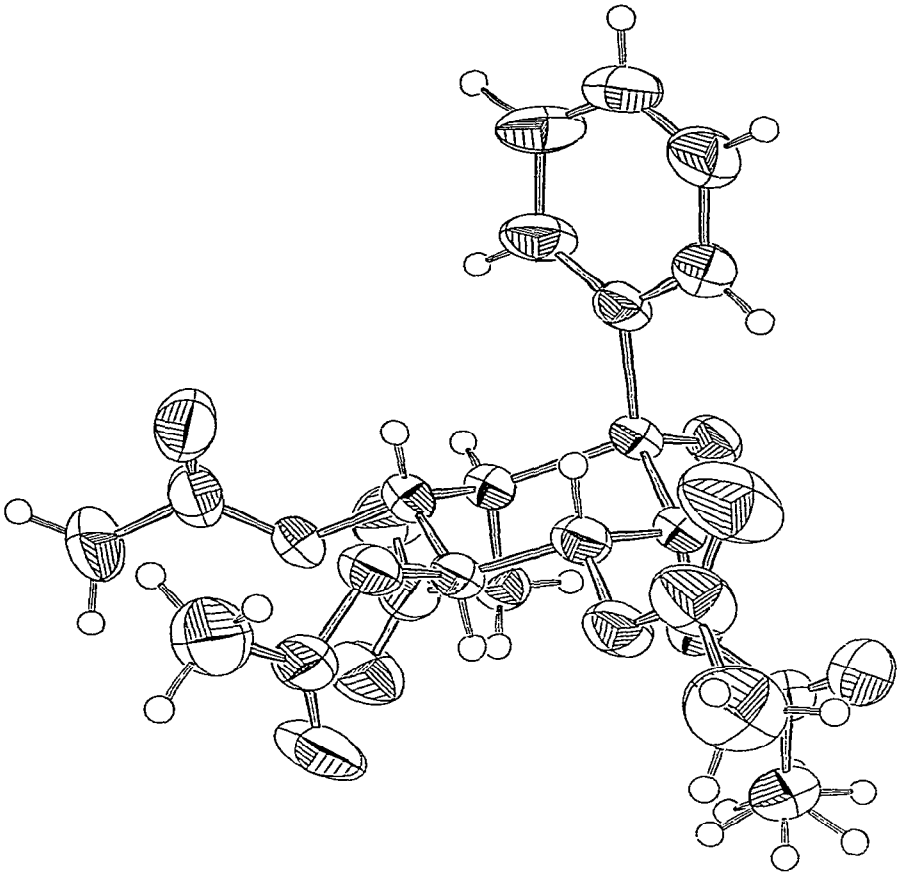


Fig. 1. ORTEP[2] representation of a molecular model of 1,2,3,4-tetra-*O*-acetyl-5,6-dideoxy-6-*C*-nitro-5-(phenylphosphinyl)-L-idopyranose (**9**).

TABLE I

CRYSTAL DATA FOR 1,2,3,4-TETRA-*O*-ACETYL-5,6-DIDEOXY-6-*C*-NITRO-5-(PHENYLPHOSPHINYL)-L-IDOPYRANOSE (**9**) (ESD VALUES IN PARENTHESES)

Formula	$C_{20}H_{24}NO_{11}P$
Lattice constants	
a (nm)	1.1691 (3)
b (nm)	1.0693 (3)
c (nm)	1.1985 (4)
α (degrees)	102.98 (2)
β (degrees)	111.68 (3)
γ (degrees)	61.89 (2)
Cell volume (nm^3)	1.2257
Unit cell contents, *Z*	2 (+ 1 MeOH)
Linear absorption	
Coefficient μ (CuKα) (cm^{-1})	15.65
Total number of reflections	4109
Unobserved ($I < 2\,\sigma\,(I)$)	21
X-Ray density (mg × m^{-3})	1.350

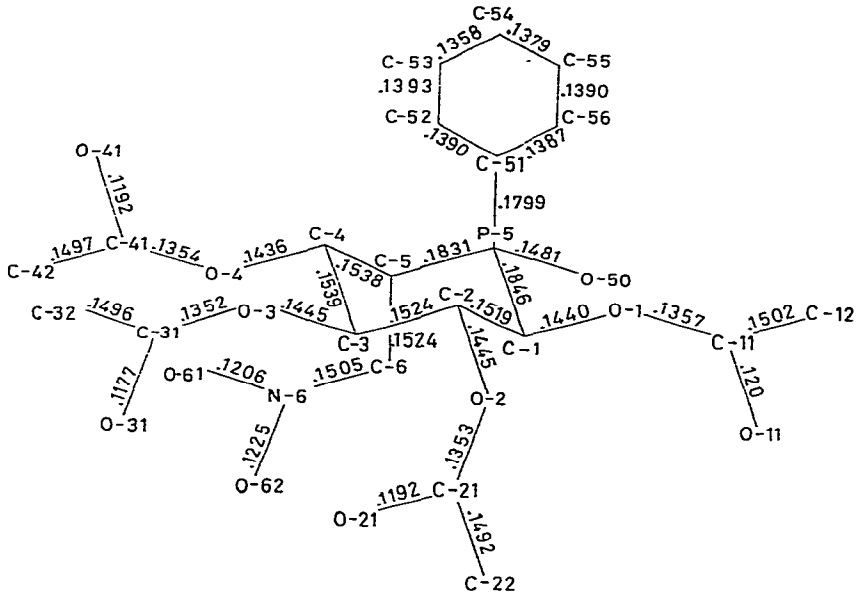


Fig. 2. Atom-numbering scheme and bond lengths (averaged over both independent molecules).

TABLE II

BOND LENGTHS (nm) IN MOLECULES 1 AND 2 OF **9** (ESD VALUES IN PARENTHESES)

Bond	*Molecule 1*	*Molecule 2*	*Bond*	*Molecule 1*	*Molecule 2*
O-1′–C-1′	0.137 (2)		C-1–O-1	0.1438 (5)	0.1441 (5)
C-1–C-2	0.1530 (6)	0.1507 (6)	C-1–P-5	0.1841 (4)	0.1850 (5)
O-1–C-11	0.1351 (8)	0.1363 (6)	C-11–O-11	0.1209 (6)	0.119 (1)
C-11–C-12	0.1505 (8)	0.1498 (9)	C-2–O-2	0.1449 (6)	0.1440 (5)
C-2–C-3	0.1524 (6)	0.1524 (7)	O-2–C-21	0.1345 (7)	0.1360 (8)
C-21–O-21	0.1192 (7)	0.1192 (6)	C-21–C-22	0.149 (1)	0.1493 (8)
C-3–O-3	0.1446 (5)	0.1444 (5)	C-3–C-4	0.1541 (7)	0.1536 (7)
O-3–C-31	0.1341 (5)	0.1362 (5)	C-31–O-31	0.1180 (6)	0.1174 (5)
C-31–C-32	0.1493 (9)	0.1499 (7)	C-4–O-4	0.1446 (5)	0.1426 (6)
C-4–C-5	0.1539 (6)	0.1536 (6)	O-4–C-41	0.1345 (8)	0.1362 (6)
C-41–O-41	0.1201 (6)	0.1183 (8)	C-41–C-42	0.1504 (7)	0.149 (1)
C-5–C-6	0.1531 (6)	0.1517 (7)	C-5–P-5	0.1825 (4)	0.1837 (5)
C-6–N-6	0.1487 (7)	0.1522 (9)	N-6–O-61	0.1222 (5)	0.1189 (6)
N-6–O-62	0.1231 (8)	0.1219 (7)	P-5–O-50	0.1479 (3)	0.1482 (3)
P-5–C-51	0.1798 (4)	0.1799 (4)	C-51–C-52	0.1396 (9)	0.1383 (6)
C-51–C-56	0.1394 (6)	0.1380 (8)	C-52–C-53	0.1397 (8)	0.1389 (7)
C-53–C-54	0.1356 (9)	0.136 (1)	C-54–C-55	0.137 (1)	0.1387 (8)
C-55–C-56	0.1397 (8)	0.1382 (7)			

TABLE III

BOND ANGLES IN MOLECULES 1 AND 2 OF 9 (ESD VALUES IN PARENTHESES)

Bond	*Angle (degrees)*		*Bond*	*Angle (degrees)*	
	Molecule 1	*Molecule 2*		*Molecule 1*	*Molecule 2*
O-1–C-1–C-2	110.2 (3)	109.4 (3)	O-1–C-1–P-5	104.4 (2)	104.1 (3)
C-2–C-1–P-5	111.7 (4)	113.9 (3)	C-1–O-1–C-11	117.8 (3)	117.3 (4)
O-1–C-11–O-11	123.2 (5)	122.8 (4)	O-1–C-11–C-12	109.9 (5)	110.0 (6)
O-11–C-11–C-12	126.9 (7)	127.2 (5)	C-1–C-2–O-2	107.9 (4)	105.9 (3)
C-1–C-2–C-3	114.8 (3)	114.2 (4)	O-2–C-2–C-3	104.7 (3)	108.9 (3)
C-2–O-2–C-21	117.2 (3)	118.6 (3)	O-2–C-21–O-21	123.4 (6)	122.8 (6)
O-2–C-21–C-22	110.2 (5)	110.9 (5)	O-21–C-21–C-22	126.2 (6)	126.3 (7)
C-2–C-3–O-3	105.3 (3)	107.2 (4)	C-2–C-3–C-4	113.3 (3)	113.0 (3)
O-3–C-3–C-4	105.8 (4)	104.4 (3)	C-3–O-3–C-31	119.2 (3)	117.7 (3)
O-3–C-31–O-31	123.3 (5)	123.1 (4)	O-3–C-31–C-32	109.4 (4)	109.7 (4)
O-31–C-31–C-32	127.2 (4)	127.1 (4)	C-3–C-4–O-4	107.9 (3)	105.1 (3)
C-3–C-4–C-5	113.8 (4)	116.5 (3)	O-4–C-4–C-5	106.6 (3)	108.8 (5)
C-4–O-4–C-41	118.1 (3)	118.1 (4)	O-4–C-41–O-41	124.5 (5)	121.9 (7)
O-4–C-41–C-42	111.1 (5)	109.8 (5)	O-41–C-41–C-42	124.5 (7)	128.3 (6)
C-4–C-5–C-6	114.6 (3)	114.2 (3)	C-4–C-5–P-5	111.4 (2)	109.7 (4)
C-6–C-5–P-5	109.6 (4)	109.6 (3)	C-5–C-6–N-6	111.2 (5)	112.5 (4)
C-6–N-6–O-61	116.9 (5)	120.9 (5)	C-6–N-6–O-62	117.2 (4)	114.0 (5)
O-61–N-6–O-62	125.7 (5)	125.0 (7)	C-1–P-5–C-5	101.6 (2)	101.6 (2)
C-1–P-5–O-50	114.4 (2)	110.9 (2)	C-1–P-5–C-51	105.9 (2)	105.2 (2)
C-5–P-5–O-50	111.5 (2)	114.6 (2)	C-5–P-5–C-51	107.9 (2)	107.4 (2)
O-50–P-5–C-51	114.5 (2)	115.8 (2)	P-5–C-51–C-52	119.7 (3)	122.2 (4)
P-5–C-51–C-56	119.9 (4)	118.3 (3)	C-52–C-51–C-56	120.0 (4)	119.3 (4)
C-51–C-52–C-53	119.1 (5)	119.8 (6)	C-52–C-53–C-54	120.6 (7)	120.7 (5)
C-53–C-54–C-55	120.8 (5)	119.8 (5)	C-54–C-55–C-56	120.7 (5)	119.7 (6)
C-51–C-56–C-55	118.7 (6)	120.7 (5)			

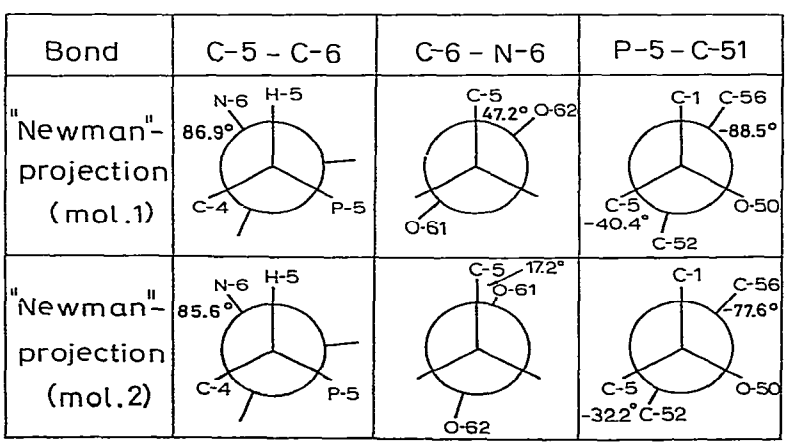


Fig. 3. "Newman" projections along the C-5–C-6, C-6–N-6, and P-5–C-51 bonds.

TABLE IV

CHOICE OF TORSION ANGLES IN MOLECULES 1 AND 2 OF **9** (ESD VALUES IN PARENTHESES)

Bond	*Angle (degrees)*	
	Molecule 1	*Molecule 2*
C-1–C-2–C-3–C-4	−59.4 (5)	−57.3 (4)
C-2–C-3–C-4–C-5	60.1 (4)	60.4 (5)
C-3–C-4–C-5–P-5	−58.5 (3)	−58.1 (4)
C-4–C-5–P-5–C-1	50.7 (4)	48.2 (3)
C-5–P-5–C-1–C-2	−49.7 (3)	−49.2 (4)
P-5–C-1–C-2–C-3	56.8 (4)	55.7 (4)
O-1–C-1–P-5–C-5	69.3 (3)	69.9 (3)
C-6–C-5–P-5–C-1	−77.2 (3)	−77.9 (4)
O-50–P-5–C-1–C-2	−170.0 (2)	−171.4 (3)
C-51–P-5–C-1–C-2	62.9 (3)	62.7 (4)
O-11–C-11–O-1–C-1	−1.0 (6)	−0.4 (7)
O-21–C-21–O-2–C-2	0.1 (9)	9.8 (8)
O-31–C-31–O-3–C-3	3.1 (9)	8.6 (8)
O-41–C-41–O-4–C-4	−6.4 (7)	−4.0 (1)
N-6–C-6–C-5–P-5	−147.0 (3)	−151.0 (3)
N-6–C-6–C-5–C-4	86.9 (5)	85.6 (5)
O-61–N-6–C-6–C-5	−135.9 (5)	17.2 (7)
O-62–N-6–C-6–C-5	47.2 (6)	−164.5 (5)
C-52–C-51–P-5–C-5	−40.4 (4)	−32.2 (4)
C-56–C-51–P-5–C-5	146.7 (3)	152.9 (4)

is almost *syn* parallel to the corresponding C–H bond at the pyranoid ring [see the torsional angles O-il–C-il–O-i–C-i, where i = 1–4 (Table IV), which do not differ greatly from zero].

The steric situation around the bonds C-5–C-6, C-6–N-6, and P-5–C-51 is illustrated in the "Newman" projections in Fig. 3.

From the results of X-ray crystallographic analysis, the correct structure of **8** is 1,2,3,4-tetra-*O*-acetyl-5,6-dideoxy-6-*C*-nitro-5-[(*R*)-phenylphosphinyl]-α-L-ido-pyranose (**9**). Consequently, compounds "**2**, **4**, **5**, **6**, and **7**" must be L-*ido* derivatives, and compound "**3**" is actually the D-*gluco* derivative **2**.

EXPERIMENTAL

Only a few crystals of **8** (**9** correctly) were available, of which, from optical inspection, only two seemed to be single crystals. However, both specimens were relatively large (0.7–1.0 mm), and, to avoid the risk of damage, no attempt was made to cut either crystal to a smaller size. The X-ray measurements were made with a crystal measuring 0.95 × 0.75 × 0.25 mm, and the drawback of exposing parts of the crystal to the outer, less-homogeneous regions of the primary beam was taken into account.

X-Ray measurements were conducted on a DEC PDP 15/40 controlled Stoe, four-circle diffractometer with Ni-filtered CuKα radiation (λ = 154.18 pm). The relevant crystal-data are given in Table I.

As no intensity symmetry (except for Friedel symmetry) was observed, a triclinic lattice was present. Chemical aspects required the existence of a polar space group, so P1 was chosen, although two formula-units were found to be in the unit cell, and the E-value statistics (MULTAN[6], NORMAL subprogram) showed almost centrosymmetric averages. Nevertheless, phase determination (with MULTAN[6]) and refinement (XRAY 76 program system[7], CDC Cyber 175 computer) ran successfully in the space group P1.

This structure is another example of an almost centrosymmetric, carbohydrate crystal structure, the first representative of which has recently been discussed in detail[8]. In the course of the refinement, one molecule of methanol per unit cell was located from a difference-Fourier synthesis. The hydrogen atoms of the pyranoid and phenyl rings could also be unambiguously located from difference syntheses. However, the methyl hydrogen atoms of the acetyl groups could only be identified after positions calculated theoretically were compared to the difference-density maxima. For the acetoxyl group on C-1, the methyl hydrogen atoms were found to occupy staggered positions relative to the carbonyl oxygen atom and to O-1, with an almost 50% probability for both independent molecules. This property of acetyl groups has been found several times[9,10]. The hydrogen atoms of the solvent molecule could not be located.

In the final stage, refinement was made with anisotropic temperature-factors for all non-hydrogen atoms. Isotropic temperature-factors were given to the hydrogen atoms. The methyl hydrogen atoms were left unrefined. The intensity data were corrected for absorption, and for the anomalous scattering of phosphorus. A weighting scheme was used which made $w\Delta F$ independent from $|F|$. After convergence of all parameters, a final R value of 5% was obtained. Fractional coordinates of all atoms are given in Table V*.

ACKNOWLEDGMENT

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*A complete atom list, with the temperature parameters included, and the list of observed and calculated structure factors can be obtained on request from Elsevier Scientific Publishing Company, BBA Data Deposition, P.O. Box 1527, Amsterdam, The Netherlands. Reference should be made to No. BBA/DD/000/Carbohydr. Res., 84 (1980) 25–33.

TABLE V

FRACTIONAL COORDINATES OF 9 (ESD VALUES IN PARENTHESES)

Atom		*Molecule 1*			*Molecule 2*		
		X	*Y*	*Z*	*X*	*Y*	*Z*
O	1′	0.9666 (9)	0.7605 (9)	0.958 (1)			
C	1′	0.848 (1)	0.831 (1)	0.987 (1)			
C	101	0.3392 (4)	0.1944 (4)	−0.1343 (3)	0.8064 (4)	0.3218 (4)	0.1769 (3)
H	101	0.441 (5)	0.149 (5)	−0.118 (4)	0.832 (5)	0.331 (5)	0.104 (4)
O	101	0.2623 (3)	0.3278 (3)	−0.1903 (2)	0.8924 (3)	0.1864 (3)	0.2296 (2)
C	111	0.3223 (5)	0.3686 (6)	−0.2471 (4)	0.9969 (5)	0.0942 (5)	0.1853 (5)
O	111	0.4371 (4)	0.2900 (5)	−0.2515 (4)	1.0182 (5)	0.1211 (5)	0.1059 (5)
C	112	0.2263 (6)	0.5054 (7)	−0.3001 (5)	1.0774 (7)	−0.0386 (7)	0.2524 (7)
H	1121	0.141 (0)	0.503 (0)	−0.359 (0)	1.114 (0)	−0.017 (0)	0.340 (0)
H	1122	0.203 (0)	0.581 (0)	−0.234 (0)	1.016 (0)	−0.086 (0)	0.241 (0)
H	1123	0.271 (0)	0.534 (0)	−0.343 (0)	1.155 (0)	−0.107 (0)	0.220 (0)
H	1124	0.276 (0)	0.569 (0)	−0.262 (0)	1.074 (0)	−0.118 (0)	0.189 (0)
H	1125	0.218 (0)	0.491 (0)	−0.388 (0)	1.173 (0)	−0.049 (0)	0.287 (0)
H	1126	0.131 (0)	0.569 (0)	−0.297 (0)	1.059 (0)	−0.062 (0)	0.319 (0)
C	102	0.2970 (4)	0.0813 (4)	−0.2162 (3)	0.8209 (4)	0.4384 (4)	0.2704 (3)
H	102	0.353 (5)	−0.008 (6)	−0.176 (5)	0.768 (5)	0.533 (6)	0.221 (5)
O	102	0.3184 (3)	0.0708 (3)	−0.3302 (3)	0.9627 (3)	0.4093 (3)	0.3089 (3)
C	121	0.4423 (6)	−0.0198 (7)	−0.3405 (5)	0.9962 (5)	0.5141 (6)	0.3058 (5)
O	121	0.5333 (5)	−0.0906 (6)	−0.2615 (5)	0.9140 (4)	0.6338 (5)	0.2876 (5)
C	122	0.4508 (8)	−0.007 (1)	−0.4579 (7)	1.4584 (7)	0.4584 (7)	0.3310 (7)
H	1221	0.525 (0)	0.025 (0)	0.439 (0)	1.159 (0)	0.480 (0)	0.259 (0)
H	1222	0.487 (0)	−0.110 (0)	−0.494 (0)	1.178 (0)	0.517 (0)	0.403 (0)
H	1223	0.377 (0)	0.048 (0)	−0.526 (0)	1.218 (0)	0.358 (0)	0.347 (0)
C	103	0.1466 (4)	0.1165 (4)	−0.2486 (3)	0.7805 (4)	0.4458 (4)	0.3798 (3)
H	103	0.102 (4)	0.196 (4)	−0.296 (3)	0.849 (4)	0.364 (4)	0.427 (4)
O	103	0.1325 (3)	−0.0087 (3)	−0.3155 (2)	0.7810 (3)	0.5739 (3)	0.4512 (2)
C	131	0.0450 (5)	0.0029 (5)	−0.4272 (4)	0.8625 (4)	0.5608 (5)	0.5677 (4)
O	131	−0.0183 (5)	0.1099 (4)	−0.4767 (4)	0.9423 (4)	0.4508 (4)	0.6082 (3)
C	132	0.0438 (8)	−0.1381 (7)	−0.4755 (6)	0.8356 (6)	0.7052 (6)	0.6307 (5)
H	1321	0.137 (0)	−0.211 (0)	−0.478 (0)	0.807 (0)	0.706 (0)	0.699 (0)
H	1322	0.014 (0)	−0.171 (0)	−0.424 (0)	0.925 (0)	0.711 (0)	0.661 (0)
H	1323	−0.021 (0)	−0.131 (0)	−0.560 (0)	0.768 (0)	0.801 (0)	0.595 (0)
C	104	0.1000 (4)	0.1390 (4)	−0.1381 (3)	0.6334 (4)	0.4647 (4)	0.3470 (3)
H	104	0.149 (4)	0.049 (5)	−0.109 (4)	0.579 (4)	0.550 (4)	0.316 (4)
O	104	−0.0444 (3)	0.1762 (3)	−0.1805 (3)	0.6133 (3)	0.4719 (3)	0.4590 (3)
C	141	−0.0844 (5)	0.0763 (5)	−0.1828 (4)	0.4939 (5)	0.5723 (7)	0.4771 (5)
O	141	−0.0084 (4)	−0.0455 (4)	−0.1616 (4)	0.4110 (6)	0.6567 (7)	0.4054 (5)
C	142	−0.2371 (5)	0.1367 (7)	−0.2173 (6)	0.4911 (7)	0.559 (1)	0.5975 (6)
H	1421	−0.270 (0)	0.217 (0)	−0.159 (0)	0.500 (0)	0.460 (0)	0.600 (0)
H	1422	−0.278 (0)	0.176 (0)	−0.300 (0)	0.572 (0)	0.568 (0)	0.663 (0)
H	1423	−0.268 (0)	0.064 (0)	−0.220 (0)	0.406 (0)	0.631 (0)	0.612 (0)
C	105	0.1190 (4)	0.2611 (4)	−0.0490 (3)	0.5989 (4)	0.3469 (4)	0.2637 (3)
H	105	0.098 (4)	0.267 (4)	0.013 (4)	0.505 (4)	0.367 (4)	0.246 (4)
C	106	0.0288 (4)	0.4091 (4)	−0.0975 (4)	0.6736 (5)	0.2038 (5)	0.3183 (4)
H	1061	0.070 (7)	0.468 (7)	−0.053 (7)	0.677 (6)	0.114 (6)	0.250 (6)
H	1062	0.018 (6)	0.396 (6)	−0.187 (6)	0.756 (6)	0.189 (5)	0.362 (5)
N	106	−0.1063 (4)	0.4711 (4)	−0.0776 (4)	0.6008 (5)	0.1892 (5)	0.3935 (4)
O	161	−0.2063 (4)	0.5298 (5)	−0.1613 (5)	0.4847 (4)	0.2670 (5)	0.3838 (4)

TABLE V *(continued)*

Atom		*Molecule 1*			*Molecule 2*		
		X	*Y*	*Z*	*X*	*Y*	*Z*
O	162	−0.1072 (4)	0.4643 (4)	0.0233 (4)	0.6711 (6)	0.0909 (6)	0.4596 (5)
P	105	0.2969 (0)	0.2323 (0)	0.0068 (0)	0.6336 (1)	0.3285 (1)	0.1219 (1)
O	150	0.3131 (3)	0.3572 (3)	0.0806 (3)	0.6319 (3)	0.1996 (3)	0.0448 (3)
C	151	0.3956 (4)	0.0694 (4)	0.0825 (3)	0.5241 (4)	0.4944 (4)	0.0513 (3)
C	152	0.3445 (6)	0.0372 (6)	0.1553 (4)	0.3960 (4)	0.5797 (5)	0.0634 (4)
H	152	0.255 (6)	0.076 (6)	0.142 (5)	0.350 (6)	0.533 (6)	0.103 (5)
C	153	0.4303 (7)	−0.0773 (6)	0.2285 (5)	0.3107 (5)	0.7001 (5)	−0.0018 (5)
H	153	0.384 (8)	−0.105 (8)	0.275 (7)	0.217 (7)	0.773 (7)	0.017 (6)
C	154	0.5618 (6)	−0.1556 (5)	0.2298 (4)	0.3525 (5)	0.7363 (5)	−0.0768 (4)
H	154	0.618 (6)	−0.236 (6)	0.272 (5)	0.293 (9)	0.818 (9)	−0.142 (8)
C	155	0.6111 (5)	−0.1286 (6)	0.1553 (6)	0.4822 (5)	0.6533 (5)	−0.0872 (4)
H	155	0.704 (7)	−0.186 (7)	0.156 (6)	0.509 (8)	0.686 (9)	−0.143 (8)
C	156	0.5285 (5)	−0.0165 (5)	0.0792 (5)	0.5661 (5)	0.5315 (5)	−0.0248 (4)
H	156	0.560 (5)	0.003 (5)	0.024 (4)	0.653 (5)	0.469 (5)	−0.040 (4)

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