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Single Crystal X-Ray Diffraction Studies

Single crystals of compound **1**, suitable for X-ray diffraction, were obtained by slow evaporation of dichloromethane – methanol 97:3 solution. The crystals were introduced into perfluorinated oil and a suitable single crystal was carefully mounted on the top of a thin glass wire. Data collection was performed with an Oxford Xcalibur 3 diffractometer equipped with a Spellman generator (50 kV, 40 mA) and a Kappa CCD detector, operating with Mo-K_α radiation (λ = 0.71071 Å).

Data collection and data reduction were performed with the CrysAlisPro software.[a)] Absorption correction using the multiscan method[a)] was applied. The structures were solved with SHELXS-97,[b)] refined with SHELXL-97[c)] and finally checked using PLATON.[d)] Details for data collection and structure refinement are summarized in Table 1.

CCDC-2212310 contains supplementary crystallographic data for this compound. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre *via* www.ccdc.cam.ac.uk/data_request/cif.

Table 1. Details for X-ray data collection and structure refinement for compound **1**.

	1
Empirical formula	$C_{14}H_{22}NO_4P$
Formula mass	299.29
T[K]	123(2)
Crystal size [mm]	0.40 × 0.20 × 0.10
Crystal description	colorless block
Crystal system	triclinic
Space group	*P*-1
a [Å]	7.2778(2)
b [Å]	10.1592(4)
c [Å]	10.8460(4)
α [°]	91.681(3)
β [°]	102.741(3)
γ [°]	101.624(3)
V [Å^3]	763.84(5)
Z	2
$\rho_{calcd.}$ [g cm^{-3}]	1.301
μ [mm^{-1}]	0.192
F(000)	320
Θ range [°]	2.05 – 25.24
Index ranges	$-9 \leq h \leq 9$
	$-13 \leq k \leq 13$
	$-14 \leq l \leq 14$
Reflns. collected	13665
Reflns. obsd.	3188
Reflns. unique	3790 (R_{int} = 0.0306)
R_1, wR_2 (2σ data)	0.0390, 0.0998
R_1, wR_2 (all data)	0.0486, 0.1072
GOOF on F^2	1.026
Peak/hole [e Å^{-3}]	0.481 / -0.236

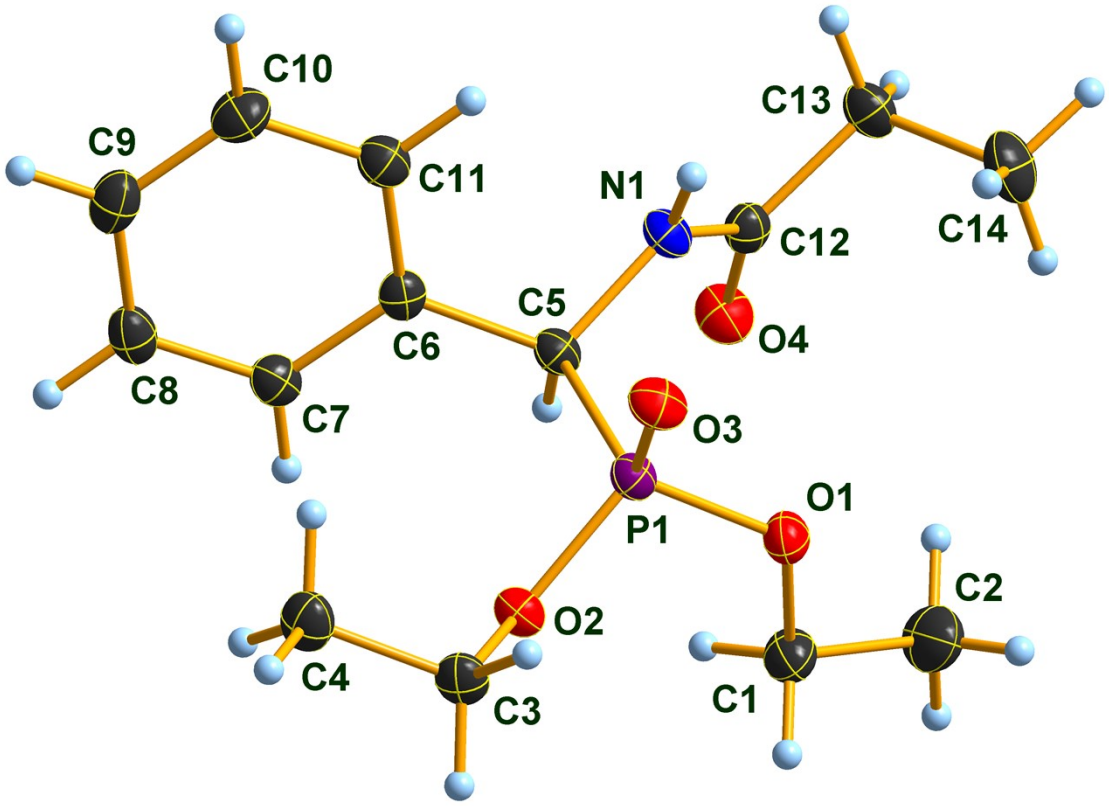

Figure 1. Molecular structure of compound **1** in the crystal. DIAMOND[e)] representation; thermal ellipsoids are drawn at 50 % probability level.

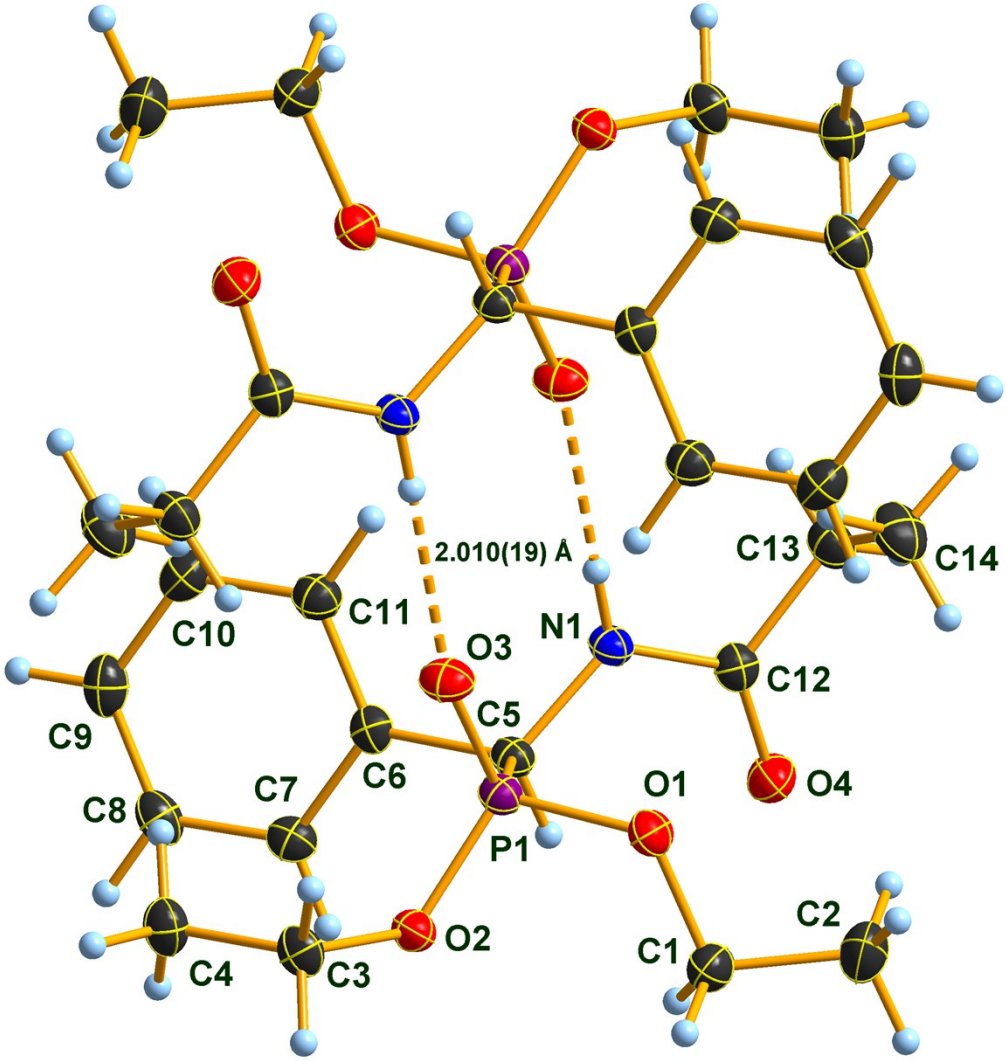

Figure 2. View of the hydrogen bonded dimers of compound **1** in the crystal. DIAMOND[e)] representation; thermal ellipsoids are drawn at 50 % probability level. Symmetry code for the non labeled molecule: -x, -y, 1-z.

Table 2. Selected bond lengths (Å) of compound **1**.

P1 – O3	1.467(1)	C3 – C4	1.493(2)
P1 – O2	1.570(1)	C1 – C2	1.499(2)
P1 – O1	1.574(1)	C11 – C10	1.391(2)
P1 – C5	1.822(1)	C10 – C9	1.390(2)
O2 – C3	1.466(2)	C13 – C14	1.525(2)
O1 – C1	1.459(2)	C6 – C11	1.394(2)
C5 – N1	1.455(2)	C6 – C7	1.395(2)
C5 – C6	1.518(2)	C8 – C9	1.387(2)
O4 – C12	1.228(2)	C8 – C7	1.393(2)

N1 – C12	1.356(2)	C12 – C13	1.512(2)

Table 3. Selected bond angles (°) of compound **1**.

O3 – P1 – O2	115.8(1)	O1 – C1 – C2	108.4(1)
O3 – P1 – O1	110.5(1)	C10 – C11 – C6	120.2(1)
O2 – P1 – O1	107.0(1)	C9 – C10 – C11	120.7(1)
O3 – P1 – C5	114.0(1)	C8 – C7 – C6	120.8(1)
O2 – P1 – C5	102.9(1)	C8 – C9 – C10	119.5(1)
O1 – P1 – C5	105.8(1)	C8 – C9 – H9	120.300
C3 – O2 – P1	122.2(1)	N1 – C12 – C13	114.2(1)
C1 – O1 – P1	121.6(1)	C12 – C13 – C14	110.8(1)
N1 – C5 – C6	114.5(1)	C11 – C6 – C7	118.9(1)
N1 – C5 – P1	107.9(1)	C11 – C6 – C5	121.6(1)
C6 – C5 – P1	110.9(1)	C7 – C6 – C5	119.5(1)
C12 – N1 – C5	121.5(1)	C9 – C8 – C7	120.0(1)
O4 – C12 – N1	122.9(1)	O2 – C3 – C4	110.2(1)
O4 – C12 – C13	122.9(1)		

Table 4. Selected torsion angles (°) of compound **1**.

O3 – P1 – O2 – C3	-10.7(1)	O4 – C12 – C13 – C14	-97.0(2)
O1 – P1 – O2 – C3	113.1(1)	N1 – C12 – C13 – C14	81.2(2)
C5 – P1 – O2 – C3	-135.7(1)	N1 – C5 – C6 – C11	-31.5(2)
O3 – P1 – O1 – C1	159.3(1)	P1 – C5 – C6 – C11	90.9(1)
O2 – P1 – O1 – C1	32.4(1)	N1 – C5 – C6 – C7	150.9(1)
C5 – P1 – O1 – C1	-76.9(1)	P1 – C5 – C6 – C7	-86.7(1)
O3 – P1 – C5 – N1	63.2(1)	P1 – O2 – C3 – C4	100.6(1)
O2 – P1 – C5 – N1	-170.6(1)	P1 – O1 – C1 – C2	153.6(1)
O1 – P1 – C5 – N1	-58.5(1)	C7 – C6 – C11 – C10	-0.2(2)
O3 – P1 – C5 – C6	-62.9(1)	C5 – C6 – C11 – C10	-177.8(1)
O2 – P1 – C5 – C6	63.3(1)	C6 – C11 – C10 – C9	0.9(2)
O1 – P1 – C5 – C6	175.4(1)	C9 – C8 – C7 – C6	0.9(2)
C6 – C5 – N1 – C12	-126.9(1)	C11 – C6 – C7 – C8	-0.7(2)
P1 – C5 – N1 – C12	109.0(1)	C5 – C6 – C7 – C8	176.9(1)
C5 – N1 – C12 – O4	5.5(2)	C7 – C8 – C9 – C10	-0.2(2)
C5 – N1 – C12 – C13	-172.7(1)	C11 – C10 – C9 – C8	-0.7(2)

References

a) Program package 'CrysAlisPro 1.171.40.82a (Rigaku OD, 2020)'.

b) Sheldrick, G. M. (1997) SHELXS-97: *Program for Crystal Structure Solution*, University of Göttingen, Germany.

c) Sheldrick, G. M. (1997) SHELXL-97: *Program for the Refinement of Crystal Structures*, University of Göttingen, Germany.

d) Spek, A. L. (1999) PLATON: *A Multipurpose Crystallographic Tool*, Utrecht University, Utrecht, The Netherlands.

e) DIAMOND, Crystal Impact GbR., Version 3.2i.