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Bis(4-nitrophenyl) *N*,*N*-dimethylphosphoramidate

Khodayar Gholivand,[a] Azadeh Tadjarodi,[a]* Abbas Taeb,[b] Gholamhossein Garivani[a] and Seik Weng Ng[c]

[a]Department of Chemistry, Tarbiat Modarres University, PO Box 14155-4838, Tehran, Iran, [b]Faculty of Chemical Engineering, Iran University of Science and Technology, Narmak 16844, Tehran, Iran, and [c]Institute of Postgraduate Studies, University of Malaya, 50603 Kuala Lumpur, Malaysia

Correspondence e-mail: tajarodi@iust.ac.ir

Key indicators

Single-crystal X-ray study
T = 298 K
Mean σ(C–C) = 0.003 Å
R factor = 0.046
wR factor = 0.134
Data-to-parameter ratio = 17.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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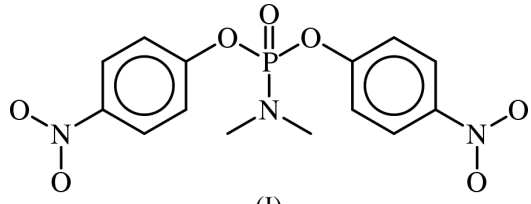
The P—N bond in the title compound, $C_{14}H_{14}N_3O_7P$, exhibits double-bond character that is attributed to the presence of the nitro substituents. Contrary to what is expected, the P—N bond in such a compound is not weak.

Comment

The title compound, (I) (Fig. 1), which acts as an inhibitor of blood coagulation (De Lange & Hemker, 1972), has been characterized by mass spectrometry (Meyer *et al.*, 1978). It possesses a nitro substituent in the *para* position of the aromatic ring; the presence of two nitrophenyl entities considerably shortens the P—N bond [1.6032 (17) Å], which is significantly shorter than the corresponding distance (1.77 Å) found in $NaHPO_3(NH_2)$, but is much longer than that (1.57 Å) found in $(C_6H_5)_3PN$ (Corbridge, 1995). On the basis of the P—N distance, the P—N bond in (I) is expected to display double-bond character. The double-bond character is also shown by the sum of the angles at the N atom of almost 360°.



(I)

Experimental

The title compound was synthesized from the reaction of sodium *p*-nitrophenoxide and *N*,*N*-dimethylaminophosphoryl dichloride. Crystals were obtained by recrystallization from chloroform–ethanol (1/1).

Crystal data

$C_{14}H_{14}N_3O_7P$
M_r = 367.25
Monoclinic, $P2_1/a$
a = 9.139 (2) Å
b = 11.083 (2) Å
c = 16.411 (3) Å
β = 95.838 (2)°
V = 1653.5 (5) Å^3
Z = 4
D_x = 1.475 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 12–18°
μ = 0.21 mm^{-1}
T = 298 (2) K
Irregular block, colorless
0.65 × 0.18 × 0.18 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scan (North *et al.*, 1968)
T_{min} = 0.898, T_{max} = 0.953
5105 measured reflections
4823 independent reflections
2557 reflections with $I > 2\sigma(I)$
R_{int} = 0.016
θ_{max} = 30.0°
$h = 0 \rightarrow 12$
$k = -15 \rightarrow 0$
$l = -23 \rightarrow 22$
3 standard reflections
frequency: 60 min
intensity decay: <1%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.134
S = 0.92
4823 reflections
282 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0759P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.21 e Å^{-3}
$\Delta\rho_{min}$ = −0.26 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

P1—O7	1.4547 (14)	P1—O4	1.5999 (14)
P1—O1	1.5845 (13)	P1—N3	1.6032 (17)
O7—P1—O1	115.81 (8)	O7—P1—N3	115.77 (8)
O7—P1—O4	113.65 (8)	O1—P1—N3	104.53 (8)
O1—P1—O4	98.49 (7)	O4—P1—N3	106.75 (9)

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1989); cell refinement: *CAD*-4 *EXPRESS*; data reduction: *NRCVAX* (Gabe *et al.*, 1989); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976); software used to prepare material for publication: *SHELXL*97.

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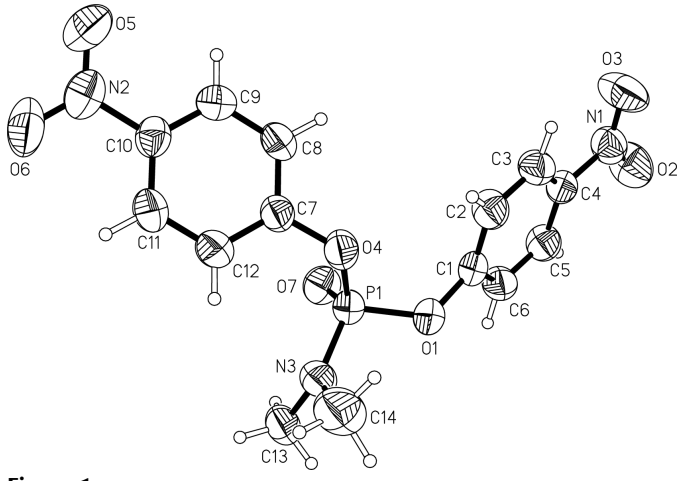

Figure 1
*ORTEP*II (Johnson, 1976) plot of the title compound at the 50% probability level. H atoms are drawn as spheres of arbitrary radii.

References

Corbridge, D. E. C. (1995). *Phosphorus, an Outline of its Chemistry, Biochemistry and Technology*. The Netherlands: Elsevier.
De Lange, J. A. & Hemker, H. C. (1972). *Fed. Eur. Biochem. Soc. Lett.* **24**, 265–268.
Enraf–Nonius (1989). *CAD*-4 *EXPRESS*. Enraf–Nonius, Delft, The Netherlands.
Gabe, E. J., Le Page, Y., Charland, J.-P., Lee, F. L. & White, P. S. (1989). *J. Appl. Cryst.* **22**, 384–387.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Meyer, H. J., Larsson, F. C. V., Lawesson, S. O. & Bowie, J. H. (1978). *Bull. Soc. Chim. Belg.* **87**, 517–523.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1997). *SHELXL*97 and *SHELXS*97. University of Göttingen, Germany.