Crystal structure of tris(morpholino)phosphoniminium triiodide, $(OC_4H_8N)_3PN^+H_2I_3^-$

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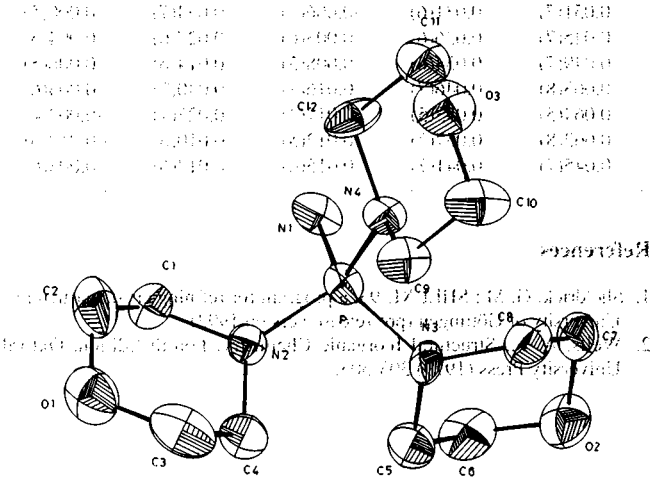

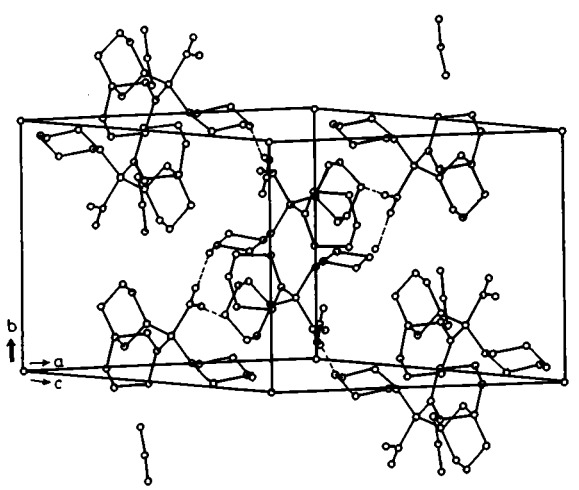

Source of material: The title compound was obtained from iodination reaction of $(OC_4H_8N)_3PN$-S_3N_3 with elemental iodine in acetonitrile medium and recrystallized from CH_3CN at room temperature.

The P–N distances (d(P–N(1)) = 1.601 Å, d(P–N(2)) = 1.609 Å, d(P–N(3)) = 1.643 Å, d(P–N(4)) = 1.620 Å) are considerably shorter than 1.77 Å expected for normal P(sp^3)–N(sp^2) bond, indicative of π-character in the bonds. The anion I_3^- is unsymmetrical and almost linear. The overall length of the anion (5.840 Å) is consistent with that observed in other triiodides (see ref. 2). N(1) takes part in two hydrogen bonds with O(2) and O(3) of two different fragments with N···O distances 2.89(1) Å and 2.98(1) Å, respectively.

$C_{12}H_{26}I_3N_4O_3P$, monoclinic, $P12_1/c1$ (No. 14), a = 13.35(1) Å, b = 10.74(1) Å, c = 16.15(1) Å, β = 110.97(7)°, V = 2162.2 Å^3, Z = 4, $R(F)$ = 0.045, $R_w(F^2)$ = 0.109.

Table 1. Parameters used for the X-ray data collection

Crystal:	brown plate, size 0.08 x 0.22 x 0.30 mm
Wavelength:	Mo $K\alpha$ radiation (0.71069 Å)
μ:	44.28 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω/2θ
$T_{measurement}$:	300 K
$2\theta_{max}$:	50°
N(hkl)$_{unique}$:	2164
Criterion for I_o:	$I_o > 3\,\sigma(I_o)$
N($param$)$_{refined}$:	312
Program:	SHELXL-93

Table 2. Final atomic coordinates and displacement parameters (in Å^2)

Atom	Site	x	y	z	U_{iso}
H(1A)	4e	0.097(7)	0.754(9)	0.095(6)	0.02(3)
H(1B)	4e	0.07(1)	0.65(1)	0.03(1)	0.10(5)
H(2A)	4e	−0.013(7)	0.685(9)	0.153(7)	0.03(3)
H(2B)	4e	−0.093(8)	0.713(9)	0.050(7)	0.05(3)
H(3A)	4e	0.022(6)	0.489(7)	0.200(6)	0.01(2)
H(3B)	4e	−0.02(1)	0.38(2)	0.152(9)	0.10(5)
H(4A)	4e	0.173(7)	0.427(8)	0.179(6)	0.03(3)
H(4B)	4e	0.139(9)	0.43(1)	0.085(8)	0.08(4)
H(5A)	4e	0.255(6)	0.867(7)	0.313(5)	0.00(2)
H(5B)	4e	0.15(1)	0.82(1)	0.324(8)	0.07(4)
H(6A)	4e	0.352(8)	0.836(9)	0.464(6)	0.03(3)
H(6B)	4e	0.250(9)	0.93(1)	0.454(8)	0.07(4)
H(7A)	4e	0.256(6)	0.562(8)	0.498(5)	0.02(2)
H(7B)	4e	0.38(1)	0.62(1)	0.497(9)	0.10(5)
H(8A)	4e	0.256(7)	0.521(9)	0.354(6)	0.03(3)
H(8B)	4e	0.161(9)	0.592(9)	0.345(7)	0.04(3)
H(9A)	4e	0.372(7)	0.374(9)	0.178(6)	0.04(3)
H(9B)	4e	0.305(6)	0.487(8)	0.101(6)	0.03(2)
H(10A)	4e	0.471(9)	0.57(1)	0.134(8)	0.06(4)
H(10B)	4e	0.48(1)	0.43(1)	0.12(1)	0.10(5)
H(11A)	4e	0.532(7)	0.647(9)	0.299(6)	0.03(2)
H(11B)	4e	0.612(9)	0.53(1)	0.355(7)	0.06(3)
H(12A)	4e	0.447(8)	0.43(1)	0.339(7)	0.06(3)
H(12B)	4e	0.433(9)	0.58(1)	0.353(8)	0.07(4)
HN(A′)	4e	0.339(8)	0.84(1)	0.202(7)	0.05(3)
HN(B′)	4e	0.285(8)	0.78(1)	0.115(8)	0.05(3)

Table 3. Final atomic coordinates and displacement parameters (in $Å^2$)

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
I(1)	4*e*	0.0957(1)	0.0845(1)	0.11567(8)	0.1226(9)	0.0784(7)	0.0879(8)	0.0253(6)	0.0340(7)	0.0106(6)
I(2)	4*e*	0.23285(7)	0.17512(7)	0.28671(6)	0.0721(5)	0.0512(4)	0.0836(6)	0.0190(4)	0.0511(5)	0.0285(4)
I(3)	4*e*	0.36865(9)	0.2622(1)	0.46551(7)	0.1035(8)	0.0747(7)	0.0832(7)	0.0048(6)	0.0238(6)	0.0269(6)
P	4*e*	0.2687(2)	0.6526(2)	0.2184(2)	0.032(1)	0.034(1)	0.024(1)	−0.001(1)	0.015(1)	−0.000(1)
N(2)	4*e*	0.2694(6)	0.6906(7)	0.3172(5)	0.043(5)	0.036(4)	0.018(4)	0.005(3)	0.017(3)	−0.002(3)
N(3)	4*e*	0.3518(6)	0.5369(7)	0.2375(5)	0.027(4)	0.044(5)	0.028(4)	0.015(4)	0.008(4)	−0.008(4)
N(4)	4*e*	0.2957(7)	0.7773(8)	0.1762(6)	0.052(6)	0.040(5)	0.028(5)	−0.014(4)	0.020(4)	0.000(4)
N(1)	4*e*	0.1589(6)	0.6019(7)	0.1458(5)	0.036(5)	0.032(4)	0.030(4)	−0.002(3)	0.011(4)	0.001(3)
C(1)	4*e*	0.0745(9)	0.685(1)	0.0857(8)	0.045(7)	0.039(7)	0.045(7)	0.008(6)	0.010(5)	0.004(6)
C(2)	4*e*	−0.029(1)	0.667(1)	0.100(1)	0.034(7)	0.09(1)	0.056(9)	0.001(7)	0.012(6)	−0.006(8)
O(1)	4*e*	−0.0631(6)	0.5441(8)	0.0914(6)	0.041(5)	0.076(6)	0.072(6)	−0.014(4)	0.025(4)	−0.014(5)
C(3)	4*e*	0.014(1)	0.465(1)	0.152(1)	0.08(1)	0.072(9)	0.051(8)	−0.028(8)	0.043(8)	−0.021(8)
C(4)	4*e*	0.1204(9)	0.473(1)	0.1400(8)	0.039(6)	0.038(6)	0.051(7)	−0.008(5)	0.017(6)	0.003(5)
C(5)	4*e*	0.238(1)	0.814(1)	0.3372(7)	0.08(1)	0.034(6)	0.038(7)	0.017(6)	0.033(6)	0.002(5)
C(6)	4*e*	0.284(1)	0.840(1)	0.4338(8)	0.070(9)	0.056(7)	0.037(7)	0.005(7)	0.022(7)	−0.011(6)
O(2)	4*e*	0.2524(7)	0.7452(8)	0.4824(5)	0.090(6)	0.073(6)	0.036(4)	0.009(5)	0.039(4)	−0.003(4)
C(7)	4*e*	0.284(1)	0.626(1)	0.4645(7)	0.09(1)	0.051(7)	0.031(6)	−0.006(7)	0.034(7)	0.000(5)
C(8)	4*e*	0.235(1)	0.597(1)	0.3673(7)	0.058(8)	0.045(7)	0.036(6)	−0.003(6)	0.027(6)	0.004(5)
C(9)	4*e*	0.3648(9)	0.464(1)	0.1641(7)	0.054(7)	0.049(7)	0.034(6)	0.008(5)	0.014(6)	−0.006(5)
C(10)	4*e*	0.469(1)	0.493(1)	0.1542(8)	0.064(8)	0.063(8)	0.046(7)	0.016(7)	0.040(7)	−0.004(6)
O(3)	4*e*	0.5572(6)	0.4729(7)	0.2338(5)	0.048(5)	0.064(5)	0.053(5)	0.017(4)	0.027(4)	−0.003(4)
C(11)	4*e*	0.5481(9)	0.551(1)	0.3004(8)	0.031(6)	0.062(8)	0.057(7)	0.012(5)	0.010(6)	−0.012(6)
C(12)	4*e*	0.4475(9)	0.525(1)	0.3189(8)	0.059(7)	0.045(7)	0.041(7)	0.015(6)	0.017(6)	0.003(6)

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References

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