Crystal structure of (phenyl) (piperidino) (dicyclohexylamino)-phoshiniminocyclotrithiazene dichloromethane hemisolvate, ((C6H5)N(C6H11)2)(C5H10)PN(S3N3))2(CH2Cl2)

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Inorganic Crystal Structures

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Crystal structure of (phenyl) (piperidino) (dicyclohexylamino)-phoshinimi-nocyclotrithiazene dichloromethane hemisolvate, $((C_6H_5)N(C_6H_{11})_2)(C_5H_{10})PN(S_3N_3))_2(CH_2Cl_2)$

Janaswamy Srinivas, G. Sreenivasa Murthy

Indian Insitute of Technology, Department of Physics, Madras 600 036, India

T. Mohan and M. N. Sudheedra Rao

Indian Insitute of Technology, Department of Chemistry, Madras 600 036, India

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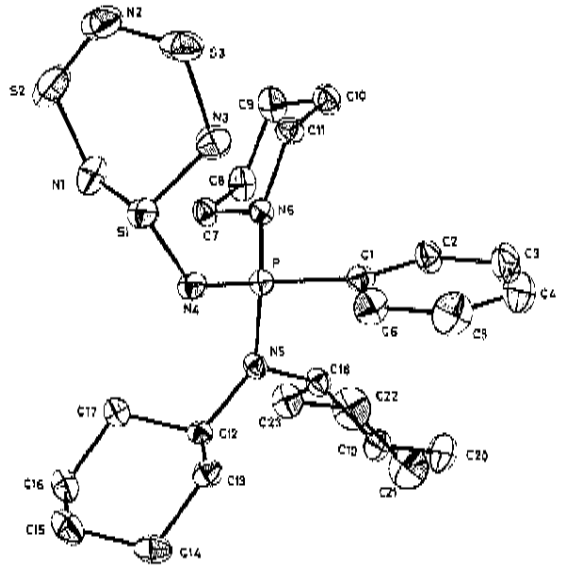

Table 1. Parameters used for the X-ray data collection

Crystal:	red, rectangular parallelepiped, size 0.3 x 0.5 x 0.5 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	4.33 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	ω/2θ
$T_{measurement}$:	300 K
$2\theta_{max}$:	60°
$N(hkl)_{unique}$:	3267
Criterion for I_o:	$I_o > 3\ \sigma(I_o)$
$N(param)_{refined}$:	312
Program:	SHELX-76

Source of material: The title compound was prepared from the reaction of S_4N_4 and the corresponding phosphine $(C_5H_6)(N(C_6H_{11})_2)(C_5H_{10}N)P$ in acetonritile and recrystallized from CH_2Cl_2/CH_3CN (1:2) at room temperatuer (see ref. 1).

The S–N bond lengths in the corresponding ring fall into three groups: (i) 1.659(3) Å, 1656(3) Å; (ii) 1.611(3) Å, 1.608(4) Å, 1.601(4) Å; (iii) 1.562(3) Å. In the S_3N_3 ring the angle at S(1), 106.8(1)°, is the smallest while 124.1(2)° at N(2) is the largest. S(1) deviates by 0.689 Å from N(1)–S(2)–N(2)–S(3)–N(3) mean plane. The S_3N_3 ring folds towards a chair conformation. The angle Cl–C(24)–Cl in the solvent molecule is 109.7(6)°.

$C_{47}H_{76}Cl_2N_{12}P_2S_6$, monoclinic, $I2_11$ (No. 15), a =20.9925(3) Å, b =9.4116(1) Å, c =29.2757(3) Å, β =99.01(2)°, V =5712.7(1) $Å^3$, Z =8, $R(F)$ =0.038, $R_w(F)$ =0.040.

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

Atom	Site	x	y	z	U_{iso}
H(1)	8*f*	0.0246	0.3851	−0.1602	0.0228
H(2)	8*f*	0.0652	0.3640	−0.1112	0.0793
H(3)	8*f*	0.0283	0.6314	−0.1527	0.0461
H(4)	8*f*	0.0999	0.5717	−0.1435	0.0625
H(5)	8*f*	0.0837	0.7343	−0.0884	0.0850
H(6)	8*f*	0.1068	0.5972	−0.0622	0.0590
H(7)	8*f*	0.0141	0.6790	−0.0301	0.0553
H(8)	8*f*	−0.0244	0.6943	−0.0784	0.0636
H(9)	8*f*	0.0155	0.4288	−0.0407	0.0373
H(10)	8*f*	−0.0544	0.4827	−0.0475	0.0460
H(11)	8*f*	−0.0913	0.5241	−0.2786	0.0844
H(12)	8*f*	−0.1332	0.3239	−0.3100	0.0922
H(13)	8*f*	−0.0676	0.5178	−0.1948	0.0459
H(14)	8*f*	−0.1506	0.1093	−0.2684	0.0957
H(15)	8*f*	−0.1231	0.1119	−0.1893	0.0684
H(16)	8*f*	−0.2309	0.3821	−0.0838	0.0617
H(17)	8*f*	−0.2456	0.2073	−0.1400	0.0384
H(18)	8*f*	−0.1922	0.1072	−0.1066	0.0703
H(19)	8*f*	−0.3134	0.1913	−0.0842	0.0657
H(20)	8*f*	−0.2962	0.0470	−0.0953	0.0684
H(21)	8*f*	−0.2848	0.0629	−0.0139	0.0705
H(22)	8*f*	−0.2201	0.0168	−0.0299	0.0848
H(23)	8*f*	−0.2518	0.2966	0.0044	0.0744
H(24)	8*f*	−0.2052	0.2002	−0.0221	0.0415
H(25)	8*f*	−0.1487	0.3692	−0.0190	0.0449
H(26)	8*f*	−0.1356	0.2235	−0.0326	0.0414
H(27)	8*f*	−0.1338	0.5638	−0.1441	0.0489
H(28)	8*f*	−0.1429	0.6561	−0.0702	0.0561
H(29)	8*f*	−0.2178	0.6102	−0.0754	0.1017
H(30)	8*f*	−0.1998	0.8460	−0.0875	0.0848
H(31)	8*f*	−0.1580	0.8197	−0.1226	0.0785
H(32)	8*f*	−0.2608	0.8684	−0.1733	0.0970
H(33)	8*f*	−0.2899	0.7486	−0.1437	0.0837
H(34)	8*f*	−0.2062	0.6991	−0.2038	0.0649
H(35)	8*f*	−0.2796	0.6554	−0.2163	0.0985
H(36)	8*f*	−0.2646	0.4847	−0.1539	0.0580
H(37)	8*f*	−0.2253	0.4524	−0.1876	0.0677
H(38)	8*f*	−0.0040	0.0041	−0.2776	0.1054

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}
P	8*f*	−0.0852	0.3183(1)	−0.1240	0.0346(4)	0.0278(4)	0.0340(4)	0.0001(4)	0.0054(3)	−0.0017(4)
S(1)	8*f*	−0.0222	0.0629(1)	−0.0900	0.0429(5)	0.0300(4)	0.0658(6)	0.0058(4)	0.0097(4)	0.0072(4)
S(2)	8*f*	0.0965(1)	0.0333(1)	−0.1207	0.0521(6)	0.0564(6)	0.119(1)	0.0117(5)	0.0315(6)	0.0120(6)
S(3)	8*f*	0.0911(1)	0.0906(1)	−0.0277	0.0734(8)	0.0917(9)	0.0930(9)	0.0061(7)	−0.0254(7)	0.0224(7)
N(1)	8*f*	0.0219(1)	0.0800(3)	−0.1320(1)	0.057(2)	0.053(2)	0.064(2)	0.013(2)	0.014(2)	−0.001(2)
N(2)	8*f*	0.1285(2)	0.0436(3)	−0.0689(1)	0.051(2)	0.059(2)	0.121(3)	0.006(2)	0.005(2)	0.025(2)
N(3)	8*f*	0.0180(2)	0.1420(3)	−0.0438(1)	0.074(2)	0.059(2)	0.054(2)	0.015(2)	−0.005(2)	0.005(5)
N(4)	8*f*	−0.0824(1)	0.1594(2)	−0.1094(1)	0.037(1)	0.029(1)	0.057(2)	0.006(1)	0.007(1)	0.005(1)
N(5)	8*f*	−0.1499(1)	0.3892(2)	−0.1084(1)	0.038(1)	0.026(1)	0.036(1)	0.001(1)	0.011(1)	0.004(1)
N(6)	8*f*	−0.0258(1)	0.4297(3)	−0.1078(1)	0.037(1)	0.039(1)	0.034(1)	−0.006(1)	0.007(1)	−0.005(1)
C(1)	8*f*	−0.0954(1)	0.3155(3)	−0.1861(1)	0.035(2)	0.042(2)	0.035(2)	0.000(2)	0.009(1)	−0.008(2)
C(2)	8*f*	−0.0852(2)	0.4334(4)	−0.2124(1)	0.056(2)	0.058(2)	0.033(2)	−0.006(2)	0.008(2)	−0.006(2)
C(3)	8*f*	−0.1003(2)	0.4315(5)	−0.2598(1)	0.082(3)	0.086(3)	0.036(2)	−0.013(2)	0.007(2)	−0.003(2)
C(4)	8*f*	−0.1263(2)	0.3080(6)	−0.2813(1)	0.081(3)	0.122(4)	0.035(2)	−0.007(3)	0.003(5)	−0.011(3)
C(5)	8*f*	−0.1353(2)	0.1926(5)	−0.2566(1)	0.085(3)	0.079(3)	0.057(3)	−0.022(3)	−0.001(2)	−0.025(2)
C(6)	8*f*	−0.1202(2)	0.1937(4)	−0.2089(1)	0.061(2)	0.055(2)	0.052(2)	−0.005(2)	0.002(2)	−0.010(2)
C(7)	8*f*	0.0345(1)	0.4280(3)	−0.1275(1)	0.038(2)	0.049(2)	0.041(2)	−0.007(2)	0.008(1)	−0.007(2)
C(8)	8*f*	0.0597(2)	0.5777(4)	−0.1306(1)	0.052(2)	0.058(2)	0.043(2)	−0.016(2)	0.011(2)	0.003(2)
C(9)	8*f*	0.0692(2)	0.6481(4)	−0.0840(1)	0.070(2)	0.046(2)	0.051(2)	−0.024(2)	0.006(2)	−0.001(2)
C(10)	8*f*	0.0079(2)	0.6432(3)	−0.0628(1)	0.073(3)	0.041(2)	0.044(2)	−0.007(2)	0.006(2)	−0.007(2)
C(11)	8*f*	−0.0156(2)	0.4920(3)	−0.0613(1)	0.050(2)	0.045(2)	0.030(2)	−0.008(2)	0.006(2)	−0.003(2)
C(12)	8*f*	−0.1969(1)	0.3157(3)	−0.0839(1)	0.032(2)	0.029(2)	0.039(2)	0.001(1)	0.009(1)	0.002(1)
C(13)	8*f*	−0.1701(1)	0.2839(3)	−0.0339(1)	0.042(2)	0.040(2)	0.040(2)	−0.003(1)	0.009(1)	−0.000(1)
C(14)	8*f*	−0.2223(2)	0.2255(3)	−0.0089(1)	0.064(2)	0.046(2)	0.046(2)	−0.001(2)	0.019(2)	0.008(2)
C(15)	8*f*	−0.2537(2)	0.0959(4)	−0.0330(1)	0.065(2)	0.047(2)	0.068(2)	−0.004(2)	0.029(2)	0.010(2)
C(16)	8*f*	−0.2801(2)	0.1272(3)	−0.0828(1)	0.040(2)	0.037(2)	0.082(2)	−0.007(2)	0.014(2)	−0.007(2)
C(17)	8*f*	−0.2286(1)	0.1854(3)	−0.1086(1)	0.039(2)	0.041(2)	0.045(2)	−0.005(2)	−0.001(2)	−0.002(2)
C(18)	8*f*	−0.1699(1)	0.5307(3)	−0.1285(1)	0.046(2)	0.029(2)	0.040(2)	0.005(1)	0.011(2)	0.007(1)
C(19)	8*f*	−0.1814(2)	0.6390(3)	−0.0922(1)	0.069(2)	0.032(2)	0.062(2)	0.009(2)	0.017(2)	−0.003(2)
C(20)	8*f*	−0.1958(2)	0.7842(4)	−0.1147(2)	0.107(4)	0.033(2)	0.102(4)	0.015(2)	0.023(3)	0.002(2)
C(21)	8*f*	−0.2522(2)	0.7794(5)	−0.1523(2)	0.101(4)	0.058(3)	0.122(4)	0.038(3)	0.027(3)	0.036(3)
C(22)	8*f*	−0.2428(2)	0.6681(5)	−0.1885(1)	0.080(3)	0.069(3)	0.074(3)	0.016(2)	0.002(2)	0.035(2)
C(23)	8*f*	−0.2275(2)	0.5225(4)	−0.1667(1)	0.052(2)	0.050(2)	0.052(2)	0.008(2)	0.001(2)	0.011(2)
C(24)	4*e*	0	0.0673(8)	1/4	0.120(6)	0.069(5)	0.156(7)	0	−0.008(6)	0
Cl	8*f*	0.0668(1)	0.1726(2)	0.2487(1)	0.142(2)	0.098(1)	0.309(3)	−0.024(1)	0.018(2)	−0.018(2)

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