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New Crystal Structures

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Organic and Metalorganic Crystal Structures

- Janaswamy Srinivas, G. Sreenivasa Murthy, T. Mohan and M. N. Sudheendra Rao*
Crystal structure of (phenyl) (piperidino) (dicyclohexylamino)-phoshiniminocyclotriazaizene dichloromethane hemisolvate, $((\text{C}_6\text{H}_5)\text{N}(\text{C}_6\text{H}_{11})_2)(\text{C}_5\text{H}_{10})\text{PN}(\text{S}_3\text{N}_3)_2(\text{CH}_2\text{Cl}_2)$ 323

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

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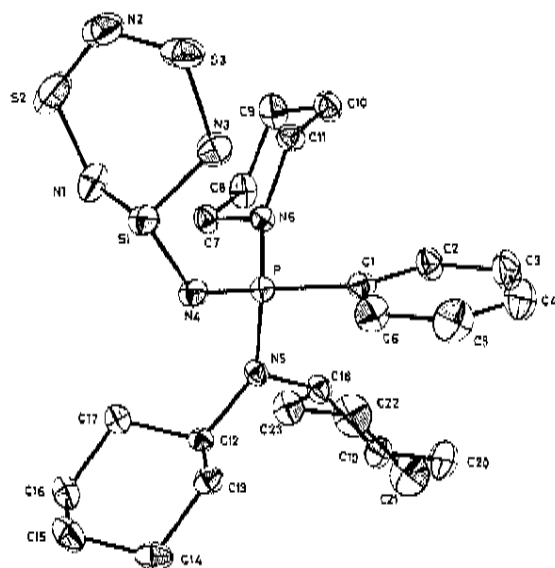


Table 1. Parameters used for the X-ray data collection

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:	$\omega/2\theta$
$T_{\text{measurement}}$:	300 K
$2\theta_{\text{max}}$:	60°
$N(hkl)_{\text{unique}}$:	3267
Criterion for I_{σ} :	$I_{\sigma} > 3 \sigma(I_{\sigma})$
$N(\text{param})_{\text{refined}}$:	312
Program:	SHELX-76

Source of material: The title compound was prepared from the reaction of S_4N_4 and the corresponding phosphine $(C_5H_9)N(C_6H_{11})_2(C_5H_{10})P$ in acetonitrile and recrystallized from CH_2Cl_2/CH_3CN (1:2) at room temperature (see ref. 1).

The S–N bond lengths in the corresponding ring fall into three groups: (i) 1.659(3) Å, 1.656(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)°.

$C_{27}H_{76}Cl_2N_{12}P_2S_6$, monoclinic, $I1211$ (No. 15), $a = 20.9925(3)$ Å, $b = 9.4116(1)$ Å, $c = 29.2757(3)$ Å, $\beta = 99.01(2)^\circ$, $V = 5712.7(1)$ Å³, $Z = 8$, $R(F) = 0.038$, $R_w(F) = 0.040$.

Table 2. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U_{iso}
H(1)	8f	0.0246	0.3851	-0.1602	0.0228
H(2)	8f	0.0652	0.3640	-0.1112	0.0793
H(3)	8f	0.0283	0.6314	-0.1527	0.0461
H(4)	8f	0.0999	0.5717	-0.1435	0.0625
H(5)	8f	0.0837	0.7343	-0.0884	0.0850
H(6)	8f	0.1068	0.5972	-0.0622	0.0590
H(7)	8f	0.0141	0.6790	-0.0301	0.0553
H(8)	8f	-0.0244	0.6943	-0.0784	0.0636
H(9)	8f	0.0155	0.4288	-0.0407	0.0373
H(10)	8f	-0.0544	0.4827	-0.0475	0.0460
H(11)	8f	-0.0913	0.5241	-0.2786	0.0344
H(12)	8f	-0.1332	0.3239	-0.3100	0.0922
H(13)	8f	-0.0676	0.5178	-0.1948	0.0459
H(14)	8f	-0.1506	0.1093	-0.2684	0.0957
H(15)	8f	-0.1231	0.1119	-0.1893	0.0684
H(16)	8f	-0.2309	0.3821	-0.0838	0.0617
H(17)	8f	-0.2456	0.2073	-0.1400	0.0384
H(18)	8f	-0.1922	0.1072	-0.1066	0.0703
H(19)	8f	-0.3134	0.1913	-0.0842	0.0657
H(20)	8f	-0.2962	0.0470	-0.0953	0.0684
H(21)	8f	-0.2848	0.0629	-0.0139	0.0705
H(22)	8f	-0.2201	0.0168	-0.0299	0.0848
H(23)	8f	-0.2518	0.2966	0.0044	0.0744
H(24)	8f	-0.2052	0.2002	-0.0221	0.0415
H(25)	8f	-0.1487	0.3692	-0.0190	0.0449
H(26)	8f	-0.1356	0.2235	-0.0326	0.0414
H(27)	8f	-0.1338	0.5638	-0.1441	0.0489
H(28)	8f	-0.1429	0.6561	-0.0702	0.0561
H(29)	8f	-0.2178	0.6102	-0.0734	0.1017
H(30)	8f	-0.1998	0.8460	-0.0875	0.0848
H(31)	8f	-0.1580	0.8197	-0.1226	0.0785
H(32)	8f	-0.2608	0.8684	-0.1733	0.0970
H(33)	8f	-0.2899	0.7486	-0.1437	0.0837
H(34)	8f	-0.2062	0.6991	-0.2038	0.0649
H(35)	8f	-0.2796	0.6554	-0.2163	0.0985
H(36)	8f	-0.2646	0.4347	-0.1539	0.0580
H(37)	8f	-0.2253	0.4524	-0.1876	0.0677
H(38)	8f	-0.0040	0.0041	-0.2776	0.1054

Table 3. Final atomic coordinates and displacement parameters (in Å²)

Atom	Site	x	y	z	U ₁₁	U ₂₂	U ₃₃	U ₁₂	U ₁₃	U ₂₃
P	8f	-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)	8f	-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)	8f	0.0965(1)	0.0333(1)	-0.1207	0.0521(6)	0.0364(6)	0.119(1)	0.0117(5)	0.0315(6)	0.0120(6)
S(3)	8f	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)	8f	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)	8f	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)	8f	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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-0.1202(2)	0.1937(4)	-0.2089(1)	0.061(2)	0.053(2)	0.052(2)	-0.005(2)	0.002(2)	-0.010(2)
C(7)	8f	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)	8f	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)	8f	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)	8f	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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	8f	-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)	4e	0	0.0673(8)	1/4	0.120(6)	0.069(5)	0.156(7)	0	-0.008(6)	0
Cl	8f	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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