Crystal structure of 1,4-phenylenebis[(1-methylethylidene)-4,1-(2,6-dimethylphenylene)] phosphorous acid tetrakis(1,2,2,6,6-pentamethylpiperidin-4-yl ester), $C_{68}H_{112}N_4O_6P_2$

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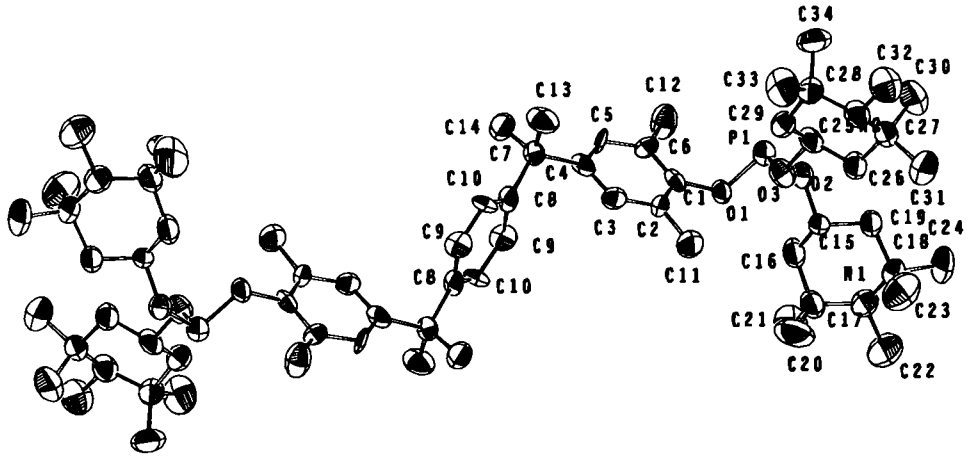

Source of material: The title compound was prepared from one equivalent 4,4'-[1,4-phenylenebis-(1-methylethylidene)]bis[2,6-dimethylphenol] and two equivalents diethylphosphoramidous acid bis(1,2,2,6,6-pentamethylpiperidin-4-yl ester) according to a modified method described in ref. 1. The reaction was carried out without catalyst in xylene (reflux). The product was obtained in 34% yield. Single crystals (mp 451 K – 453 K) were grown from acetonitrile/toluene.

$C_{68}H_{112}N_4O_6P_2$, triclinic, $P\bar{1}$ (No. 2), a =10.621(1) Å, b =12.824(1) Å, c =14.910(1) Å, α =63.970(6)°, β =81.652(6)°, γ =72.615(8)°, V =1741.1 Å^3, Z =2, $R(F)$ =0.040, $R_w(F^2)$ =0.093.

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

Crystal:	colorless, prismatic, size 0.14 x 0.24 x 0.36 mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ:	1.12 cm^{-1}
Diffractometer:	Enraf-Nonius CAD4
Scan mode:	$\omega/2\theta$
$T_{measurement}$:	293 K
$2\theta_{max}$:	34°
$N(hkl)_{unique}$:	1622
Criterion for I_o:	$I_o > 2\,\sigma(I_o)$
$N(param)_{refined}$:	375
Programs:	SHELXS-86, SHELXL-93

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

Atom	Site	x	y	z	U_{iso}
H(3)	2*i*	–0.0440(6)	0.5969(6)	0.7455(7)	0.057
H(5)	2*i*	0.0951(8)	0.8728(7)	0.6940(8)	0.054
H(9)	2*i*	0.170(1)	0.556(1)	0.8959(8)	0.060
H(10)	2*i*	0.2078(8)	0.371(1)	1.028(1)	0.058
H(11A)	2*i*	0.1101(8)	0.520(2)	0.567(3)	0.110
H(11B)	2*i*	–0.032(3)	0.5998(7)	0.528(2)	0.110
H(11C)	2*i*	–0.012(3)	0.506(2)	0.640(1)	0.110
H(12A)	2*i*	0.229(3)	0.951(3)	0.550(1)	0.110
H(12B)	2*i*	0.157(2)	0.973(2)	0.456(2)	0.110
H(12C)	2*i*	0.289(2)	0.8705(8)	0.492(2)	0.110
H(13A)	2*i*	–0.2034(7)	0.884(2)	0.759(2)	0.120
H(13B)	2*i*	–0.236(1)	0.818(3)	0.872(1)	0.120
H(13C)	2*i*	–0.236(1)	0.760(2)	0.799(3)	0.120
H(14A)	2*i*	–0.007(3)	0.8968(8)	0.820(2)	0.114
H(14B)	2*i*	0.1069(8)	0.781(2)	0.873(3)	0.114
H(14C)	2*i*	–0.023(3)	0.810(3)	0.932(1)	0.114
H(15)	2*i*	0.2029(7)	0.6474(6)	0.3060(5)	0.052
H(16A)	2*i*	0.3783(7)	0.6210(7)	0.3973(5)	0.077
H(16B)	2*i*	0.4504(7)	0.6897(7)	0.2990(5)	0.077
H(19A)	2*i*	0.3440(6)	0.7763(6)	0.1383(5)	0.066
H(19B)	2*i*	0.2045(6)	0.7608(6)	0.1338(5)	0.066
H(20A)	2*i*	0.412(5)	0.398(4)	0.4564(6)	0.192
H(20B)	2*i*	0.421(4)	0.354(2)	0.372(3)	0.192
H(20C)	2*i*	0.298(1)	0.455(2)	0.379(3)	0.192
H(21A)	2*i*	0.642(2)	0.396(1)	0.374(4)	0.195
H(21B)	2*i*	0.603(1)	0.495(5)	0.414(3)	0.195

Table 2. (Continued)

Atom	Site	x	y	z	U_{iso}
H(21C)	2i	0.657(2)	0.525(4)	0.304(2)	0.195
H(22A)	2i	0.568(5)	0.4545(9)	0.129(1)	0.182
H(22B)	2i	0.486(2)	0.379(2)	0.216(4)	0.182
H(22C)	2i	0.619(3)	0.391(3)	0.239(3)	0.182
H(23A)	2i	0.174(2)	0.593(2)	0.124(3)	0.166
H(23B)	2i	0.218(4)	0.513(4)	0.235(1)	0.166
H(23C)	2i	0.285(2)	0.472(3)	0.151(4)	0.166
H(24A)	2i	0.331(2)	0.720(3)	0.0008(7)	0.170
H(24B)	2i	0.410(5)	0.588(2)	0.020(1)	0.170
H(24C)	2i	0.478(3)	0.673(4)	0.0331(8)	0.170
H(25)	2i	−0.1118(7)	0.9298(6)	0.2457(5)	0.065
H(26A)	2i	−0.1470(7)	0.7132(6)	0.2614(5)	0.073
H(26B)	2i	−0.0594(7)	0.7886(6)	0.1797(5)	0.073
H(29A)	2i	−0.2368(7)	0.8972(6)	0.3919(5)	0.067
H(29B)	2i	−0.2590(7)	0.7822(6)	0.3912(5)	0.067

Table 2. (Continued)

Atom	Site	x	y	z	U_{iso}
H(30A)	2i	−0.302(1)	1.027(2)	0.026(2)	0.132
H(30B)	2i	−0.155(3)	0.9522(6)	0.025(2)	0.132
H(30C)	2i	−0.197(4)	1.023(2)	0.0922(5)	0.132
H(31A)	2i	−0.1924(8)	0.746(3)	0.087(3)	0.143
H(31B)	2i	−0.333(4)	0.8316(9)	0.051(2)	0.143
H(31C)	2i	−0.316(4)	0.723(3)	0.157(1)	0.143
H(32A)	2i	−0.496(2)	1.036(1)	0.113(2)	0.131
H(32B)	2i	−0.5725(7)	0.952(3)	0.1978(5)	0.131
H(32C)	2i	−0.501(2)	0.915(2)	0.112(2)	0.131
H(33A)	2i	−0.5684(8)	0.974(3)	0.327(1)	0.144
H(33B)	2i	−0.477(2)	0.929(4)	0.417(2)	0.144
H(33C)	2i	−0.495(3)	0.837(2)	0.380(3)	0.144
H(34A)	2i	−0.379(4)	1.096(1)	0.2963(5)	0.125
H(34B)	2i	−0.453(2)	1.1187(7)	0.203(3)	0.125
H(34C)	2i	−0.298(2)	1.0848(9)	0.203(3)	0.125

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(1)	2i	0.0936(2)	0.8404(2)	0.3558(1)	0.063(2)	0.053(1)	0.043(2)	−0.003(1)	−0.004(1)	−0.021(1)
O(1)	2i	0.1630(4)	0.7357(3)	0.4609(3)	0.049(3)	0.052(3)	0.029(3)	0.003(2)	0.000(3)	−0.016(3)
O(2)	2i	0.2095(4)	0.8135(4)	0.2804(3)	0.061(3)	0.054(4)	0.038(3)	−0.013(3)	0.014(3)	−0.018(3)
O(3)	2i	−0.0125(4)	0.7738(4)	0.3518(3)	0.053(3)	0.067(3)	0.053(3)	−0.020(3)	−0.011(3)	−0.017(3)
N(1)	2i	0.4705(6)	0.5385(5)	0.2142(5)	0.060(5)	0.072(5)	0.061(5)	0.007(4)	0.009(4)	−0.036(4)
N(2)	2i	−0.3750(6)	0.8902(4)	0.2135(4)	0.051(5)	0.064(4)	0.054(4)	−0.007(3)	−0.011(4)	−0.021(4)
C(1)	2i	0.1108(7)	0.7422(9)	0.5510(6)	0.027(5)	0.042(6)	0.024(6)	0.000(4)	0.002(4)	−0.001(6)
C(2)	2i	0.0443(8)	0.6568(7)	0.6118(8)	0.057(5)	0.043(6)	0.033(6)	−0.005(5)	−0.007(4)	−0.023(6)
C(3)	2i	−0.0014(6)	0.6553(6)	0.7037(7)	0.061(5)	0.036(6)	0.048(8)	−0.019(4)	0.003(5)	−0.016(5)
C(4)	2i	0.0130(7)	0.7369(8)	0.7369(6)	0.041(5)	0.036(6)	0.030(6)	−0.012(4)	0.015(5)	0.002(6)
C(5)	2i	0.0810(8)	0.8186(7)	0.6733(8)	0.062(5)	0.058(6)	0.029(5)	−0.006(5)	0.001(4)	−0.036(5)
C(6)	2i	0.1299(6)	0.8247(7)	0.5798(7)	0.048(5)	0.036(6)	0.051(8)	−0.016(4)	0.001(5)	−0.025(5)
C(7)	2i	−0.0478(7)	0.7424(7)	0.8341(6)	0.063(6)	0.043(6)	0.025(5)	−0.020(5)	0.021(5)	−0.009(5)
C(8)	2i	−0.024(2)	0.6160(7)	0.9175(6)	0.057(7)	0.049(8)	0.025(6)	−0.03(1)	0.019(8)	−0.014(5)
C(9)	2i	0.099(1)	0.534(1)	0.9379(8)	0.025(8)	0.061(8)	0.059(9)	−0.009(6)	0.015(4)	−0.026(9)
C(10)	2i	0.1227(8)	0.422(1)	1.018(1)	0.055(8)	0.024(8)	0.043(5)	0.005(6)	−0.015(8)	−0.001(6)
C(11)	2i	0.0260(6)	0.5619(6)	0.5842(4)	0.115(7)	0.054(5)	0.057(5)	−0.029(5)	0.003(4)	−0.024(5)
C(12)	2i	0.2084(7)	0.9128(6)	0.5134(5)	0.086(6)	0.094(6)	0.059(5)	−0.050(5)	0.020(4)	−0.039(5)
C(13)	2i	−0.1945(7)	0.8071(6)	0.8141(5)	0.066(6)	0.064(5)	0.064(5)	0.018(5)	0.011(4)	−0.013(4)
C(14)	2i	0.0130(7)	0.8142(5)	0.8680(5)	0.142(7)	0.048(5)	0.045(5)	−0.046(5)	0.023(4)	−0.020(4)
C(15)	2i	0.2645(7)	0.6977(6)	0.2758(5)	0.047(5)	0.039(5)	0.037(6)	0.005(4)	−0.007(4)	−0.017(4)
C(16)	2i	0.3929(7)	0.6363(7)	0.3274(5)	0.066(6)	0.076(6)	0.039(5)	−0.004(5)	−0.005(4)	−0.022(4)
C(17)	2i	0.4621(8)	0.5179(8)	0.3200(6)	0.076(7)	0.073(7)	0.040(6)	0.015(6)	−0.003(5)	−0.018(5)
C(18)	2i	0.3527(8)	0.6087(7)	0.1510(5)	0.069(6)	0.063(6)	0.047(6)	−0.008(5)	0.005(5)	−0.031(5)
C(19)	2i	0.2882(6)	0.7215(6)	0.1679(5)	0.057(5)	0.062(6)	0.041(6)	−0.005(4)	−0.005(4)	−0.023(4)
C(20)	2i	0.3917(9)	0.4223(7)	0.3881(6)	0.177(9)	0.054(6)	0.093(7)	−0.010(6)	0.032(7)	0.000(5)
C(21)	2i	0.6042(8)	0.4800(7)	0.3564(6)	0.070(7)	0.169(9)	0.109(7)	0.057(6)	−0.043(6)	−0.065(6)
C(22)	2i	0.5417(8)	0.4315(8)	0.1984(6)	0.119(7)	0.115(7)	0.109(7)	0.024(6)	0.007(6)	−0.067(6)
C(23)	2i	0.2474(8)	0.5403(7)	0.1666(6)	0.117(7)	0.119(7)	0.139(8)	−0.050(6)	−0.007(6)	−0.079(6)
C(24)	2i	0.3970(8)	0.6513(7)	0.0410(5)	0.156(8)	0.122(7)	0.049(6)	−0.003(6)	0.009(5)	−0.049(5)
C(25)	2i	−0.1259(7)	0.8500(6)	0.2868(5)	0.043(6)	0.058(5)	0.046(5)	0.004(4)	−0.020(5)	−0.013(5)
C(26)	2i	−0.1388(7)	0.7942(6)	0.2205(5)	0.054(6)	0.071(5)	0.047(5)	0.005(5)	−0.007(5)	−0.029(5)
C(27)	2i	−0.2571(8)	0.8656(6)	0.1533(6)	0.059(6)	0.055(6)	0.043(5)	−0.005(5)	−0.006(6)	−0.019(5)
C(28)	2i	−0.3717(7)	0.9386(7)	0.2875(6)	0.038(6)	0.067(6)	0.057(5)	0.003(5)	−0.004(5)	−0.032(5)
C(29)	2i	−0.2476(7)	0.8618(6)	0.3489(5)	0.052(5)	0.061(5)	0.052(5)	−0.007(5)	0.008(5)	−0.029(4)
C(30)	2i	−0.2248(7)	0.9775(6)	0.0660(5)	0.105(7)	0.089(6)	0.055(5)	−0.032(5)	−0.005(5)	−0.012(5)
C(31)	2i	−0.2764(7)	0.7839(6)	0.1076(5)	0.119(7)	0.100(6)	0.083(6)	−0.020(5)	−0.012(5)	−0.056(6)
C(32)	2i	−0.4969(7)	0.9541(6)	0.1538(5)	0.067(6)	0.104(6)	0.090(6)	−0.016(5)	−0.025(5)	−0.036(5)
C(33)	2i	−0.4887(7)	0.9176(6)	0.3594(5)	0.061(6)	0.131(7)	0.081(6)	−0.016(5)	0.024(5)	−0.045(5)
C(34)	2i	−0.3757(6)	1.0719(6)	0.2433(5)	0.087(6)	0.052(6)	0.111(6)	0.007(4)	−0.030(5)	−0.043(5)

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