C(2)—C(3)	1.499 (9)	C(11)—C(18)	1.52 (1)
C(3)—C(4)	1.370 (8)	C(12)—C(13)	1.51 (1)
C(3)—C(8)	1.38 (1)	C(13)—C(14)	1.54 (1)
C(4)—C(5)	1.37 (1)	C(14)—C(15)	1.539 (9)
C(5)—C(6)	1.36 (1)	C(15)—C(16)	1.52 (1)
C(6)—C(7)	1.37 (1)	C(15)—C(17)	1.52 (1)

C(1)—O(2)—C(9)	115.6 (5)	O(2)—C(9)—C(14)	106.6 (6)
O(1)—C(1)—O(2)	124.5 (7)	O(2)—C(9)—C(10)	109.9 (6)
O(2)—C(1)—C(2)	110.5 (6)	C(10)—C(9)—C(14)	112.8 (6)
O(1)—C(1)—C(2)	125.0 (6)	C(9)—C(10)—C(11)	110.8 (6)
Br(1)—C(2)—C(1)	104.1 (4)	C(10)—C(11)—C(18)	111.8 (7)
C(1)—C(2)—C(3)	116.0 (5)	C(10)—C(11)—C(12)	110.1 (7)
Br(1)—C(2)—C(3)	110.9 (5)	C(12)—C(11)—C(18)	113.1 (7)
C(2)—C(3)—C(8)	122.4 (6)	C(11)—C(12)—C(13)	112.0 (6)
C(2)—C(3)—C(4)	119.8 (6)	C(12)—C(13)—C(14)	112.3 (6)
C(4)—C(3)—C(8)	117.9 (6)	C(9)—C(14)—C(13)	107.1 (6)
C(3)—C(4)—C(5)	122.2 (6)	C(13)—C(14)—C(15)	113.3 (6)
C(4)—C(5)—C(6)	119.5 (7)	C(9)—C(14)—C(15)	113.8 (6)
C(5)—C(6)—C(7)	120.2 (7)	C(14)—C(15)—C(17)	114.2 (6)
C(6)—C(7)—C(8)	120.3 (7)	C(14)—C(15)—C(16)	112.6 (6)
C(3)—C(8)—C(7)	120.0 (7)	C(16)—C(15)—C(17)	110.5 (6)

O(1)—C(1)—C(2)—C(3)	16.4 (10)
C(10)—C(9)—C(14)—C(13)	57.9 (8)
C(14)—C(9)—C(10)—C(11)	−59.6 (8)
C(9)—C(10)—C(11)—C(12)	55.4 (8)
C(10)—C(11)—C(12)—C(13)	−54.3 (9)
C(11)—C(12)—C(13)—C(14)	55.8 (9)
C(12)—C(13)—C(14)—C(9)	−55.7 (8)

Refinement was based on full-matrix least-squares methods with anisotropic displacement parameters for non-H atoms and one common isotropic displacement parameter [0.060 (4) Å^2] for the H atoms, which were placed in calculated positions. Unit weights led to featureless analysis of variance in terms of $\sin\theta$ and $(F/F_{\max})^{1/2}$. An η refinement [the data set contained the pairs (h −1 l), (h 1 l)] (Rogers, 1981) gave η = 0.97 (10) and confirms the proposed chirality.

Data collection: Nicolet *R3m/V* diffractometer software. Cell refinement: Nicolet *R3m/V* diffractometer software. Data reduction: Nicolet *R3m/V* diffractometer software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus* and *SHELX76* (Sheldrick, 1976). Software used to prepare material for publication: *PARST* (Nardelli, 1983), *PLATON* (Spek, 1982) and *MISSYM* (Le Page, 1987) .

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1107). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Bis(2,4-di-*tert*-butylphenyl) Pentaerythritol Diphosphite

JAMES P. BARREN, GAROLD L. BRYANT† AND
MARY F. GARBAUSKAS

*General Electric Research and Development,
Schenectady, NY 12301, USA*

JAMES A. MAHOOD

*General Electric Plastics, Parkersburg, WV 12301,
USA*

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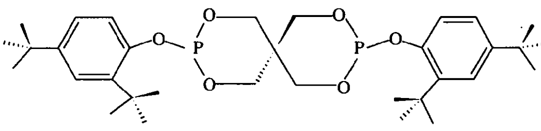
Abstract

The title compound, 3,9-bis(2,4-di-*tert*-butylphenoxy)-2,4,8,10-tetraoxa-3,9-diphosphaspiro[5.5]undecane, (1), $C_{33}H_{50}O_6P_2$, has three different conformers in the unit cell. The structure of each is very similar to the others, differing principally in the rotational conformations of the aryl *tert*-butyl groups. The remaining portion of the structure comprises two six-membered rings, each in a chair conformation and sharing a common spiro atom. Bond angles and bond lengths in the spiro rings are very similar to those of the related molecule 3,9-dimethoxy-2,4,8,10-tetraoxa-3,9-diphosphaspiro[5.5]undecane [Carpenter, Jacobson & Verkade (1979). *Phosphorus Sulfur*, **6**, 475–479].

Comment

The solution of the structure of the title compound, (I), indicated the presence of three conformers in the unit cell, differing principally in the rotational conformations of the aryl *tert*-butyl groups. Fig. 1 shows the molecule with the numbering scheme employed.



(I)

On comparison with the structure of 3,9-dimethoxy-2,4,8,10-tetraoxa-3,9-diphosphaspiro[5.5]undecane (Carpenter, Jacobson & Verkade, 1979), (II), minor differences are observed in the P—O—Me and P—O—aryl bond angles. The six P—O—aryl angles in (I) [123.02 (14), 123.2 (2), 122.3 (2), 122.2 (2), 122.2 (2) and 123.8 (2)°] are larger than the P—O—Me an-

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† Present address: The Upjohn Company, Physical and Analytical Chemistry Research, Kalamazoo, MI 49001, USA.

gle in (II) [117.8 (5)°], presumably to accommodate the more sterically demanding aryl ring. The longer P—O bond lengths for the six P—O—aryl bonds [1.654 (2), 1.652 (2), 1.651 (2), 1.659 (2), 1.656 (2) and 1.657 (2) Å] compared with those in the P—O—Me bonds [1.624 (5) Å] are also attributed to the added steric requirements of the aryl ring.

The unusually large cell prompted an independent determination of the cell parameters. The results reported here were confirmed by the Molecular Structure Corporation (The Woodlands, Texas, USA). For additional information on related structures and chemistry see Carpenter, Jacobson & Verkade (1979), White, Bertrand, McEwen & Verkade (1970), Goghova, Karias & Durmis (1989), and Bentrude & Hargis (1970).

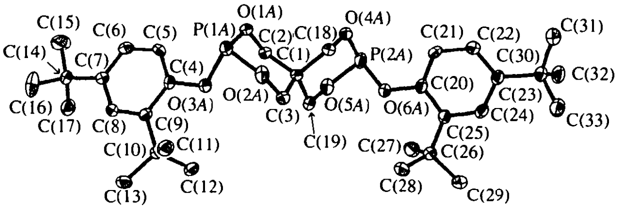

Fig. 1. Displacement ellipsoid plot (50% probability) of one of the molecules with the atomic numbering scheme employed. The different molecules are distinguished by the suffix letter *A* for molecule *A*, *B* for molecule *B*, and *C* for molecule *C*.

Experimental

Compound (I) was obtained by crystallization of a commercial sample (GE Specialty Chemicals, Parkersburg, USA) of 0.480 g of 3,9-bis(2,4-di-*tert*-butylphenoxy)-2,4,8,10-tetraoxo-3,9-diphosphaspiro[5.5]undecane from 1.57 g of dichloromethane and 2.0 g of *n*-heptane. (I) was dissolved, the solution filtered and placed in a screw-capped vial with the cap loosely in place. The solvents were allowed to evaporate slowly in a glove box under nitrogen at room temperature (approximately 298 K). Crystals were collected by decanting the mother liquor two days later. The crystal was sealed in a glass capillary because of its moisture sensitivity.

Crystal data

$C_{33}H_{50}O_6P_2$
$M_r = 604.7$
Monoclinic
$C2/c$
$a = 78.98\ (4)$ Å
$b = 5.836\ (3)$ Å
$c = 43.68\ (2)$ Å
$\beta = 96.89\ (3)°$
$V = 19987\ (16)$ Å^3
$Z = 24$
$D_x = 1.206$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 33 reflections
$\theta = 4.1–44.3°$
$\mu = 1.256$ mm^{-1}
$T = 170$ K
Flat needle
0.62 × 0.28 × 0.20 mm
Clear

Data collection

Siemens *R3m/V* diffractometer
Wyckoff scans
Absorption correction: none
28 386 measured reflections
11 299 independent reflections
9141 observed reflections $[I > 2\sigma(I)]$

$R_{\text{int}} = 0.0285$
$\theta_{\max} = 55°$
$h = 0 \rightarrow 49$
$k = 0 \rightarrow 6$
$l = -46 \rightarrow 46$
3 standard reflections monitored every 50 reflections
intensity decay: 5%

Refinement

Refinement on F^2
$R(F) = 0.0423$
$wR(F^2) = 0.1099$
$S = 1.185$
11 299 reflections
1295 parameters
$w = 1/[\sigma^2(F_o^2) + (0.0718P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} = -0.004$
$\Delta\rho_{\max} = 0.303$ e Å^{-3}
$\Delta\rho_{\min} = -0.340$ e Å^{-3}

Extinction correction: least-squares refinement of χ in $(1 + 0.001\chi F_c^2\lambda^3/\sin 2\theta)^{-1/4}$
Extinction coefficient: 0.000051 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$$U_{\text{eq}} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
P(1*A*)	0.0234 (1)	0.0041 (1)	0.0689 (1)	0.032 (1)
O(1*A*)	0.0123 (1)	−0.1805 (3)	0.0473 (1)	0.031 (1)
O(2*A*)	0.0359 (1)	0.1007 (3)	0.0451 (1)	0.033 (1)
O(3*A*)	0.0380 (1)	−0.1630 (3)	0.0878 (1)	0.030 (1)
C(1*A*)	0.0329 (1)	−0.2337 (5)	0.0098 (1)	0.024 (1)
C(2*A*)	0.0213 (1)	−0.3479 (5)	0.0309 (1)	0.027 (1)
C(3*A*)	0.0448 (1)	−0.0606 (5)	0.0278 (1)	0.030 (1)
C(4*A*)	0.0355 (1)	−0.2784 (5)	0.1148 (1)	0.027 (1)
C(5*A*)	0.0198 (1)	−0.3734 (5)	0.1186 (1)	0.033 (1)
C(6*A*)	0.0176 (1)	−0.4944 (5)	0.1450 (1)	0.034 (1)
C(7*A*)	0.0310 (1)	−0.5279 (5)	0.1681 (1)	0.025 (1)
C(8*A*)	0.0468 (1)	−0.4313 (5)	0.1635 (1)	0.023 (1)
C(9*A*)	0.0497 (1)	−0.3065 (5)	0.1373 (1)	0.023 (1)
C(10*A*)	0.0674 (1)	−0.2090 (5)	0.1332 (1)	0.026 (1)
C(11*A*)	0.0745 (1)	−0.3296 (5)	0.1060 (1)	0.030 (1)
C(12*A*)	0.0668 (1)	0.0498 (5)	0.1278 (1)	0.034 (1)
C(13*A*)	0.0802 (1)	−0.2523 (6)	0.1622 (1)	0.033 (1)
C(14*A*)	0.0295 (1)	−0.6616 (5)	0.1978 (1)	0.028 (1)
C(15*A*)	0.0419 (1)	−0.8648 (5)	0.2000 (1)	0.038 (1)
C(16*A*)	0.0341 (1)	−0.5043 (6)	0.2257 (1)	0.051 (1)
C(17*A*)	0.0118 (1)	−0.7593 (7)	0.1990 (1)	0.058 (1)
P(2*A*)	0.0238 (1)	−0.4681 (1)	−0.0538 (1)	0.033 (1)
O(4*A*)	0.0133 (1)	−0.2716 (3)	−0.0381 (1)	0.030 (1)
O(5*A*)	0.0353 (1)	−0.5726 (3)	−0.0236 (1)	0.034 (1)
O(6*A*)	0.0395 (1)	−0.3177 (3)	−0.0656 (1)	0.031 (1)
C(18*A*)	0.0226 (1)	−0.1102 (5)	−0.0172 (1)	0.027 (1)
C(19*A*)	0.0444 (1)	−0.4165 (5)	−0.0019 (1)	0.030 (1)
C(20*A*)	0.0384 (1)	−0.2139 (5)	−0.0945 (1)	0.028 (1)
C(21*A*)	0.0241 (1)	−0.0914 (5)	−0.1057 (1)	0.034 (1)
C(22*A*)	0.0233 (1)	0.0201 (5)	−0.1340 (1)	0.033 (1)
C(23*A*)	0.0368 (1)	0.0100 (5)	−0.1514 (1)	0.026 (1)
C(24*A*)	0.0511 (1)	−0.1184 (5)	−0.1394 (1)	0.026 (1)
C(25*A*)	0.0527 (1)	−0.2296 (5)	−0.1110 (1)	0.024 (1)
C(26*A*)	0.0691 (1)	−0.3546 (5)	−0.0979 (1)	0.027 (1)
C(27*A*)	0.0767 (1)	−0.2407 (5)	−0.0676 (1)	0.035 (1)
C(28*A*)	0.0656 (1)	−0.6085 (5)	−0.0921 (1)	0.038 (1)
C(29*A*)	0.0828 (1)	−0.3423 (6)	−0.1201 (1)	0.038 (1)
C(30*A*)	0.0366 (1)	0.1280 (5)	−0.1829 (1)	0.028 (1)
C(31*A*)	0.0529 (1)	0.2678 (6)	−0.1846 (1)	0.039 (1)
C(32*A*)	0.0351 (1)	−0.0568 (5)	−0.2081 (1)	0.038 (1)
C(33*A*)	0.0215 (1)	0.2941 (5)	−0.1893 (1)	0.040 (1)
P(1*B*)	0.1435 (1)	0.1344 (2)	0.2171 (1)	0.040 (1)
O(1*B*)	0.1534 (1)	0.3188 (3)	0.1980 (1)	0.035 (1)
O(2*B*)	0.1320 (1)	0.0051 (3)	0.1890 (1)	0.040 (1)
O(3*B*)	0.1278 (1)	0.2914 (3)	0.2275 (1)	0.035 (1)
C(1*B*)	0.1333 (1)	0.3112 (5)	0.1503 (1)	0.026 (1)
C(2*B*)	0.1435 (1)	0.4592 (5)	0.1749 (1)	0.030 (1)

C(3*B*)	0.1223 (1)	0.1400 (5)	0.1654 (1)	0.035 (1)	O(3*C*)	0.2042 (1)	0.7963 (3)	0.2509 (1)	0.036 (1)
C(4*B*)	0.1287 (1)	0.3956 (5)	0.2564 (1)	0.030 (1)	C(1*C*)	0.1991 (1)	0.8173 (5)	0.1708 (1)	0.027 (1)
C(5*B*)	0.1429 (1)	0.5175 (5)	0.2679 (1)	0.036 (1)	C(2*C*)	0.1877 (1)	0.9462 (5)	0.1906 (1)	0.031 (1)
C(6*B*)	0.1438 (1)	0.6291 (5)	0.2961 (1)	0.033 (1)	C(3*C*)	0.2117 (1)	0.6669 (5)	0.1914 (1)	0.035 (1)
C(7*B*)	0.1302 (1)	0.6176 (5)	0.3134 (1)	0.028 (1)	C(4*C*)	0.2017 (1)	0.8999 (5)	0.2789 (1)	0.030 (1)
C(8*B*)	0.1159 (1)	0.4902 (5)	0.3011 (1)	0.026 (1)	C(5*C*)	0.1867 (1)	1.0112 (5)	0.2820 (1)	0.036 (1)
C(9*B*)	0.1144 (1)	0.3806 (5)	0.2727 (1)	0.025 (1)	C(6*C*)	0.1843 (1)	1.1226 (5)	0.3091 (1)	0.035 (1)
C(10*B*)	0.0979 (1)	0.2578 (5)	0.2592 (1)	0.029 (1)	C(7*C*)	0.1972 (1)	1.1269 (5)	0.3339 (1)	0.026 (1)
C(11*B*)	0.0906 (1)	0.3784 (6)	0.2291 (1)	0.039 (1)	C(8*C*)	0.2123 (1)	1.0103 (4)	0.3298 (1)	0.024 (1)
C(12*B*)	0.1010 (1)	0.0049 (5)	0.2531 (1)	0.040 (1)	C(9*C*)	0.2152 (1)	0.8973 (5)	0.3029 (1)	0.025 (1)
C(13*B*)	0.0841 (1)	0.2694 (6)	0.2812 (1)	0.040 (1)	C(10*C*)	0.2325 (1)	0.7802 (5)	0.2996 (1)	0.027 (1)
C(14*B*)	0.1302 (1)	0.7341 (5)	0.3449 (1)	0.027 (1)	C(11*C*)	0.2411 (1)	0.8985 (5)	0.2740 (1)	0.037 (1)
C(15*B*)	0.1139 (1)	0.8780 (5)	0.3457 (1)	0.036 (1)	C(12*C*)	0.2301 (1)	0.5250 (5)	0.2921 (1)	0.038 (1)
C(16*B*)	0.1310 (1)	0.5502 (5)	0.3700 (1)	0.035 (1)	C(13*C*)	0.2448 (1)	0.7997 (6)	0.3294 (1)	0.038 (1)
C(17*B*)	0.1454 (1)	0.8974 (5)	0.3520 (1)	0.038 (1)	C(14*C*)	0.1955 (1)	1.2473 (5)	0.3644 (1)	0.027 (1)
P(2*B*)	0.1426 (1)	0.5065 (1)	0.0885 (1)	0.037 (1)	C(15*C*)	0.2100 (1)	1.4217 (5)	0.3719 (1)	0.036 (1)
O(4*B*)	0.1536 (1)	0.3353 (3)	0.1121 (1)	0.034 (1)	C(16*C*)	0.1964 (1)	1.0682 (6)	0.3903 (1)	0.044 (1)
O(5*B*)	0.1298 (1)	0.6174 (3)	0.1108 (1)	0.038 (1)	C(17*C*)	0.1787 (1)	1.3784 (6)	0.3637 (1)	0.043 (1)
O(6*B*)	0.1281 (1)	0.3305 (3)	0.0707 (1)	0.033 (1)	P(2*C*)	0.1888 (1)	0.9755 (1)	0.1033 (1)	0.036 (1)
C(18*B*)	0.1447 (1)	0.1815 (5)	0.1305 (1)	0.030 (1)	O(4*C*)	0.1790 (1)	0.7974 (3)	0.1233 (1)	0.034 (1)
C(19*B*)	0.1211 (1)	0.4667 (5)	0.1300 (1)	0.034 (1)	O(5*C*)	0.2001 (1)	1.1156 (3)	0.1305 (1)	0.039 (1)
C(20*B*)	0.1308 (1)	0.2121 (5)	0.0439 (1)	0.028 (1)	O(6*C*)	0.2048 (1)	0.8145 (3)	0.0944 (1)	0.033 (1)
C(21*B*)	0.1467 (1)	0.1225 (5)	0.0405 (1)	0.033 (1)	C(18*C*)	0.1890 (1)	0.6635 (5)	0.1469 (1)	0.030 (1)
C(22*B*)	0.1494 (1)	0.0042 (5)	0.0141 (1)	0.033 (1)	C(19*C*)	0.2098 (1)	0.9906 (5)	0.1552 (1)	0.033 (1)
C(23*B*)	0.1361 (1)	−0.0314 (5)	−0.0095 (1)	0.025 (1)	C(20*C*)	0.2043 (1)	0.6891 (5)	0.0672 (1)	0.029 (1)
C(24*B*)	0.1202 (1)	0.0567 (5)	−0.0049 (1)	0.024 (1)	C(21*C*)	0.1890 (1)	0.5991 (5)	0.0531 (1)	0.035 (1)
C(25*B*)	0.1168 (1)	0.1783 (5)	0.0212 (1)	0.024 (1)	C(22*C*)	0.1887 (1)	0.4754 (5)	0.0260 (1)	0.033 (1)
C(26*B*)	0.0989 (1)	0.2711 (5)	0.0247 (1)	0.027 (1)	C(23*C*)	0.2035 (1)	0.4386 (5)	0.0124 (1)	0.028 (1)
C(27*B*)	0.0921 (1)	0.1600 (5)	0.0530 (1)	0.034 (1)	C(24*C*)	0.2186 (1)	0.5276 (5)	0.0277 (1)	0.026 (1)
C(28*B*)	0.0991 (1)	0.5321 (5)	0.0284 (1)	0.039 (1)	C(25*C*)	0.2196 (1)	0.6502 (5)	0.0551 (1)	0.025 (1)
C(29*B*)	0.0861 (1)	0.2120 (5)	−0.0036 (1)	0.034 (1)	C(26*C*)	0.2368 (1)	0.7373 (5)	0.0713 (1)	0.028 (1)
C(30*B*)	0.1387 (1)	−0.1683 (5)	−0.0385 (1)	0.028 (1)	C(27*C*)	0.2403 (1)	0.6283 (5)	0.1036 (1)	0.035 (1)
C(31*B*)	0.1257 (1)	−0.1019 (8)	−0.0657 (1)	0.061 (1)	C(28*C*)	0.2369 (1)	1.0003 (5)	0.0740 (1)	0.042 (1)
C(32*B*)	0.1372 (1)	−0.4196 (6)	−0.0320 (1)	0.090 (2)	C(29*C*)	0.2517 (1)	0.6698 (6)	0.0535 (1)	0.039 (1)
C(33*B*)	0.1560 (1)	−0.1157 (9)	−0.0490 (1)	0.078 (1)	C(30*C*)	0.2032 (1)	0.2981 (5)	−0.0174 (1)	0.030 (1)
P(1*C*)	0.1900 (1)	0.6195 (2)	0.2327 (1)	0.040 (1)	C(31*C*)	0.2196 (1)	0.3272 (7)	−0.0325 (1)	0.058 (1)
O(1*C*)	0.1789 (1)	0.7926 (4)	0.2092 (1)	0.037 (1)	C(32*C*)	0.2010 (1)	0.0458 (5)	−0.0098 (1)	0.048 (1)
O(2*C*)	0.2030 (1)	0.5133 (3)	0.2103 (1)	0.039 (1)	C(33*C*)	0.1885 (1)	0.3757 (6)	−0.0411 (1)	0.043 (1)

Table 2. *Comparison of selected bond lengths* (Å) *and angles* (°) *for three conformers of* 3,9-*bis*(2,4-*di-tert-butylphenoxy*)-2,4,8,10-*tetraoxa*-3,9-*diphosphaspiro*[5.5]*undecane* (I) *and* 3,9-*dimethoxy*-2,4,8,10-*tetraoxa*-3,9-*diphosphaspiro*[5.5]*undecane** (II)

(II)		(I)	Molecule *A*	Molecule *B*	Molecule *C*
P—O1	1.616 (5)	P1—O1	1.618 (2)	1.617 (2)	1.620 (2)
		P2—O4	1.616 (2)	1.612 (2)	1.615 (2)
P—O2	1.611 (5)	P1—O2	1.619 (2)	1.624 (2)	1.624 (2)
		P2—O5	1.626 (2)	1.620 (2)	1.620 (2)
P—O3	1.624 (5)	P1—O3	1.654 (2)	1.651 (2)	1.656 (2)
		P2—O6	1.652 (2)	1.659 (2)	1.657 (2)
O1—P—O2	101.9 (3)	O1—P1—O2	100.77 (9)	100.48 (10)	100.84 (10)
		O4—P—O5	100.79 (9)	100.96 (9)	100.72 (10)
O1—P—O3	102.1 (3)	O1—P1—O3	101.23 (10)	101.37 (11)	101.52 (11)
		O4—P2—O6	101.5 (10)	101.75 (11)	100.98 (10)
O2—P—O3	98.2 (3)	O2—P1—O3	94.67 (9)	95.36 (10)	95.05 (10)
		O5—P2—O6	94.93 (9)	94.89 (10)	95.23 (10)
P—O1—C1	119.6 (4)	P1—O1—C2	118.32 (14)	118.8 (2)	119.1 (2)
		P2—O4—C18	118.60 (14)	118.90 (14)	118.4 (2)
P—O2—C2	120.2 (4)	P1—O2—C3	119.0 (2)	119.2 (2)	119.0 (2)
		P2—O5—C19	118.8 (2)	118.8 (2)	119.3 (2)
P—O3—C4	117.8 (5)	P1—O3—C4	123.02 (14)	122.3 (2)	122.2 (2)
		P2—O6—C20	123.2 (2)	122.2 (2)	123.8 (2)
O1—C1—C3	111.9 (5)	O1—C2—C1	111.6 (2)	111.4 (2)	112.1 (2)
		O4—C18—C1	111.2 (2)	111.6 (2)	111.4 (2)
O2—C2—C3	111.6 (4)	O2—C3—C1	112.8 (2)	112.8 (2)	111.7 (2)
		O5—C19—C1	112.8 (2)	112.6 (2)	113.1 (2)
C1—C3—C2	109.1 (4)	C2—C1—C3	110.9 (2)	110.1 (2)	109.5 (2)
		C18—C1—C19	110.5 (2)	110.1 (2)	111.1 (2)
		C18—C1—C3	108.7 (2)	109.1 (2)	108.3 (2)
		C2—C1—C19	108.8 (2)	108.4 (2)	108.8 (2)
		C2—C1—C18	112.0 (2)	112.2 (2)	112.1 (2)
		C3—C1—C19	105.8 (2)	106.8 (2)	106.6 (2)

* Carpenter, Jacobson & Verkade (1979).

H atoms were placed in idealized positions, but allowed to ride on the parent C atom. Methyl groups were allowed to rotate. Isotropic displacement parameters were refined for all H atoms.

The structure was solved using the *SHELXTL-Plus* program package (Sheldrick, 1988) on a MicroVAX II computer and refined using *SHELXL*93 (Sheldrick, 1993).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: AS1091). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-Nitro-*N*-phenyl-4-(phenylamino)-benzenesulfonamide

Marek L. Główka, Zbysława Olubek and Andrzej Olczak

Institute of General & Ecological Chemistry, Technical University of Łódź, Żwirki 36, 90-924 Łódź, Poland

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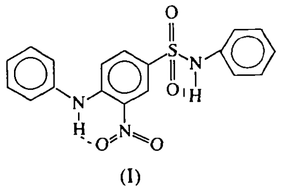
Abstract

The structure of the title compound, $C_{18}H_{15}N_3O_4S$, displays the characteristic features of benzenesulfonamide derivatives. The *N*-phenyl rings are twisted by 48.3 (1) and 77.8 (1)° with respect to the benzene ring of the phenylsulfonamide group. In the crystal, the molecules are linked to form dimers *via* N—H···O hydrogen bonds between sulfonamide groups. The amine H atom participates in an intramolecular hydrogen bond with the neighbouring nitro substituent.

Comment

The title compound, (I), is a well known commercial dye, Dispersol Yellow CT (CI Disperse Yellow 42-10338, ICI, Manchester, England), and may be considered as a derivative of either phenyl benzenesulfonamide or diphenylamine.



(I)

Structures of both systems are well known (Cambridge Structural Database, 1994) and their geometries are similar to that observed for the title compound. However, the structure of the title compound has three characteristic features. Firstly, the endocyclic bond angle C(3)—C(4)—C(5) of 115.9 (2)° is rather small; this is caused by the presence of an electron-withdrawing nitro group at the *ortho* position. This observation is in agreement with the values of 111.4, 112.2 and 113.0° found in all *N*-picrylanilides and their analogues (Gridunova, Shklover, Struchkov, Il'ina, Mikhalev & Potapov, 1989; Gridunova, Petrov, Struchkov, Il'ina & Mikhalev, 1990). Secondly, the S^{VI}—C(aryl) distance of 1.745 (2) Å found in this study is shorter than the average value of 1.76 Å for 120 arylsulfonamides (Kálmán, Czugler & Argay, 1981), although about 25 other benzenesulfonamide structures, mostly substituted by electron-donating groups at *ortho* and *para* positions, with similar S^{VI}—C(aryl) distances are known (Cambridge Structural Database, 1994). Finally, one N—C(aryl) bond, N(4)—C(4) of 1.347 (3) Å, is shortened relative to the other [N(4)—C(41) 1.438 (3) Å], which can be attributed to the strong electron-withdrawing substituents at the *ortho* and *para* positions of the benzene ring of the nitrobenzenesulfonamide system. Similar differences have been observed in all *N*-picrylanilides (Gridunova *et al.*, 1989, 1990).

There are two types of hydrogen bond in the title compound: an intermolecular N(11)—H···O sulfonamide–sulfonamide hydrogen bond which links the molecules into dimers and an intramolecular N(4)—H···O amine–nitro hydrogen bond. The latter maintains the coplanarity of the phenyl ring and the nitro group. There are several 2-nitrophenylamine structures in the literature, all showing similar intramolecular hydrogen-bonding schemes (Cambridge Structural Database, 1994). The other phenyl ring in the diphenylamine

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