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1,8-Diphosphatricyclo[6.2.2.0^{2,7}]dodeca-2(7),3,5-triene

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Abstract. $C_{10}H_{12}P_2$, $M_r = 194 \cdot 2$, monoclinic, $P2_1/c$, $a = 10 \cdot 904$ (2), $b = 6 \cdot 345$ (1), $c = 14 \cdot 445$ (2) Å, $\beta = 93 \cdot 71$ (1)°, $V = 997 \cdot 3$ Å^3, $Z = 4$, $D_x = 1 \cdot 293$ Mg m^{-3}, λ(Mo $K\alpha$) $= 0 \cdot 71069$ Å, $\mu = 0 \cdot 37$ mm^{-1}, $F(000) = 408$, $T = 298$ K, $R = 0 \cdot 038$ for 1553 observed reflections. The fixed ring system forces the four methylene groups to adopt completely eclipsed conformations; the molecule possesses non-crystallographic $mm2$ symmetry, with P—C(sp^3) (mean) $1 \cdot 840$ (2) and P—C(sp^2) (mean) $1 \cdot 826$ (2) Å.

Experimental. Crystal size $0 \cdot 8 \times 0 \cdot 4 \times 0 \cdot 2$ mm. Stoe–Siemens four-circle diffractometer, monochromated Mo $K\alpha$ radiation, profile-fitting mode involving variable scan width and speed (Clegg, 1981). Cell constants refined from $\pm 2\theta$ values of 54 reflections in the range 20–25°. 3757 reflections measured ($2\theta_{max}$ 50°, octants $\pm h \pm k + l$). Three check reflections with no significant intensity variation. 1743 unique reflections ($R_{int} = 0 \cdot 035$), of which 1558 with $F > 3\sigma(F)$ were used for all calculations (SHELXTL, Sheldrick, 1985), $h \pm 12$, k 0–7, l 0–17. Absorption and extinction corrections were not necessary, but five low-angle reflections had to be ignored during refinement. Structure solution by multisolution direct methods. Refinement on F to $R = 0 \cdot 038$, $wR = 0 \cdot 050$; all non-H atoms anisotropic, H atoms were all found in a difference electron density synthesis, but were refined using a riding model and idealized geometry [d(C—H) = 0 \cdot 96 Å, H—C—H = $109 \cdot 5$°, U(H) = $1 \cdot 2U_{eq}$(C)]. 109 parameters refined, $S = 1 \cdot 53$, weighting scheme $w^{-1} = \sigma^2(F) + 0 \cdot 0005F^2$, which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma = 0 \cdot 005$, max. and min. heights in final $\Delta\rho$ map $0 \cdot 26$ and $-0 \cdot 25$ e Å^{-3}, respectively.

Table 1. *Atomic coordinates* ($\times 10^5$ *for* P, $\times 10^4$ *for rest) and equivalent isotropic displacement parameters* (Å$^2 \times 10^4$ *for* P, Å$^2 \times 10^3$ *for rest*)

	x	y	z	U_{eq}*
P(1)	39316 (4)	28372 (9)	55221 (4)	482 (2)
P(2)	10206 (4)	21280 (9)	57263 (4)	491 (2)
C(1)	3167 (2)	4091 (3)	6467 (1)	41 (1)
C(2)	1907 (2)	3800 (3)	6550 (1)	41 (1)
C(3)	1343 (2)	4808 (4)	7267 (1)	51 (1)
C(4)	2015 (2)	6078 (4)	7894 (1)	59 (1)
C(5)	3250 (2)	6343 (4)	7815 (1)	61 (1)
C(6)	3828 (2)	5363 (4)	7110 (1)	51 (1)
C(11)	3386 (2)	122 (3)	5653 (2)	62 (1)
C(12)	2017 (2)	−218 (4)	5764 (2)	62 (1)
C(21)	2867 (2)	3650 (4)	4547 (1)	60 (1)
C(22)	1496 (2)	3284 (4)	4636 (1)	58 (1)

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and bond angles* (°)

P(1)–C(1)	1·827 (2)	P(1)–C(11)	1·836 (2)
P(1)–C(21)	1·840 (2)	P(2)–C(2)	1·824 (2)
P(2)–C(12)	1·841 (2)	P(2)–C(22)	1·842 (2)
C(1)–C(2)	1·400 (2)	C(1)–C(6)	1·395 (3)
C(2)–C(3)	1·394 (3)	C(3)–C(4)	1·385 (3)
C(4)–C(5)	1·370 (3)	C(5)–C(6)	1·380 (3)
C(11)–C(12)	1·528 (3)	C(21)–C(22)	1·527 (3)
C(1)–P(1)–C(11)	99·5 (1)	C(1)–P(1)–C(21)	98·9 (1)
C(11)–P(1)–C(21)	98·6 (1)	C(2)–P(2)–C(12)	99·4 (1)
C(2)–P(2)–C(22)	99·2 (1)	C(12)–P(2)–C(22)	98·6 (1)
P(1)–C(1)–C(2)	120·3 (1)	P(1)–C(1)–C(6)	120·5 (1)
C(2)–C(1)–C(6)	119·1 (2)	P(2)–C(2)–C(1)	120·1 (1)
P(2)–C(2)–C(3)	120·8 (1)	C(1)–C(2)–C(3)	119·1 (2)
C(2)–C(3)–C(4)	120·9 (2)	C(3)–C(4)–C(5)	119·9 (2)
C(4)–C(5)–C(6)	120·3 (2)	C(1)–C(6)–C(5)	120·7 (2)
P(1)–C(11)–C(12)	117·9 (2)	P(2)–C(12)–C(11)	117·5 (2)
P(1)–C(21)–C(22)	117·7 (1)	P(2)–C(22)–C(21)	117·6 (1)

Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Atomic parameters are given in Table 1, bond distances and angles in Table 2.* Fig. 1 shows a thermal-ellipsoid plot with the atom numbering.

Related literature. For the preparation and charac- terization of the compound see Issleib, Leissring & Schmidt (1986).
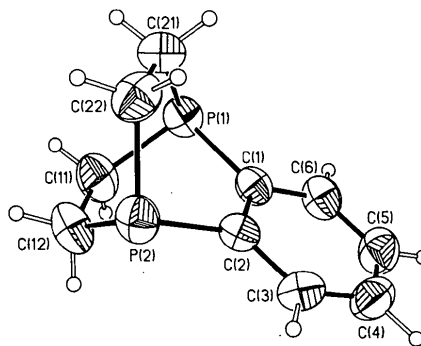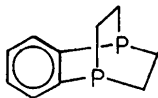
* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51597 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 1. A 50% thermal-ellipsoid plot with atom numbering.

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Structure of *N*-Tritylglycine Methylamide

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Abstract. $C_{22}H_{22}N_2O$, $M_r = 330.43$, orthorhombic, $P2_12_12_1$, $a = 8.936$ (2), $b = 13.752$ (2), $c = 14.611$ (2) Å, $V = 1795.5$ (4) Å^3, $Z = 4$, $D_x = 1.22$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.82$ cm^{-1}, $F(000) = 704$, room temperature, $R = 0.043$ for 953 unique observed reflections. The amide bond adopts the *trans* conformation [O(23)—C(22)—N(24)—H(24) = 169.5 (8)°]. The structure is stabilized by means of intermolecular hydrogen bonding [N(24)—H(24)$\cdots$ O(23^i) = 1.91 (8) Å, (i) = x, y, $1+z$]. Other bond lengths and angles are normal.

Experimental. Prismatic crystal $0.26 \times 0.25 \times 0.22$ mm. Enraf–Nonius CAD-4 diffractometer, data collection using ω–2θ scans, lattice parameters from 25 reflections in range $7 < \theta < 10°$. 5293 measured reflec- tions in index range h 0–10, k 0–17, $l \pm 17$ up to $2\theta_{max} = 56°$, 2483 unique reflections, mean dis- crepancy on I 1.1% (on averaging 5105 reflections); θ scan width $(0.7 + 0.3\tan\theta)°$, scan rate 1.03– 5.49° min^{-1}, max. scan time 60 s, aperture $(2.4 + 0.9\tan\theta)$ mm; reference reflections (222, 230, 006) every 2 h, intensity decrease 1.2%, orientation-control