reflections, showed a variation of 0·2%. No absorption corrections. Lorentz and polarization corrections. 1884 unique reflections measured. 979 observed reflections with $I > 3 \cdot 0\sigma(I)$. Direct methods (*MULTAN*82; Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) used for structure determination. H atoms located by difference Fourier synthesis. Anisotropic full-matrix least-squares refinement (on F) for non-H atoms, isotropic for H atoms. $\sum w(|F_o| - |F_c|)^2$ minimized. $R = 0.039$, $wR = 0.040$, max. $\Delta/\sigma = 0.89$. Max. peak height in the final difference Fourier map 0.34 e Å^{-3}, $S = 0.94$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). Enraf–Nonius *SDP* (Frenz, 1984).

Atomic parameters are given in Table 1,* bond distances, bond angles, and relevant torsion angles are presented in Table 2. Atomic numbering is shown in Fig. 1 and molecular packing in Fig. 2.

Related literature. The title compound is the first reported structure in the systematic study of the conformational preferences of methoxy derivatives of flavone, isoflavone and flavanone which is now being undertaken in our laboratory. Probably because of the difficulty in growing good quality crystals only a few structure determinations have been made. In 3',5,5',6-tetramethoxyflavone the dihedral angle between the

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares-planes data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51595 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 2. Molecular packing diagram.

phenyl ring and the benzopyrone portion of the molecule is 28° (Ting & Watson, 1972), whereas in 4',5,7-trihydroxyisoflavone the reported dihedral angle is 53·2° (Breton, Precigoux, Courseille & Hospital, 1975). In 5,7,4'-trimethoxyflavanone the value for the dihedral angle is 70·8° (Mariezcurrena, 1978).

References

BRETON, P. M., PRECIGOUX, G., COURSEILLE, C. & HOSPITAL, M. (1975). *Acta Cryst.* B31, 921–923.
FRENZ, B. A. (1984). *Structure Determination Package.* College Station, Texas, USA, and Enraf–Nonius, Delft, The Netherlands.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.
MARIEZCURRENA, R. A. (1978). *Acta Cryst.* B34, 2322–2324.
TING, H. & WATSON, W. H. (1972). *Acta Cryst.* B28, 1046–1051.

4,N-Dimethyl-2-nitrosoaniline

BY R. G. PRITCHARD, G. S. HEATON AND I. M. EL-NAHHAL

Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England

Abstract. $C_8H_{10}N_2O$, $M_r = 150.18$, triclinic, $P\bar{1}$, $a = 8.636$ (3), $b = 8.631$ (3), $c = 11.101$ (2) Å, $\alpha = 92.57$ (2), $\beta = 94.12$ (2), $\gamma = 105.02$ (2)°, $V = 795$ (1) Å^3, $Z = 4$, $D_x = 1.25$ Mg m^{-3}, $F(000) = 320$, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.05$ mm^{-1}, $T = 293$ K, $R = 0.043$ for 2170 observed reflexions [$F \geq 3\sigma(F)$]. The crystal contains two crystallographically distinct but similarly conformed molecules. Both are planar and the configuration facilitates weak intramolecular 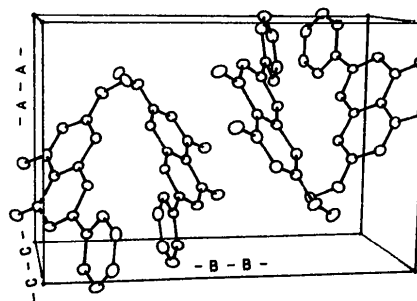hydrogen bonding between the nitroso oxygen and amine hydrogen atom [N···O 2·619 (5), 2·592 (5); O···H 2·01 (4), 1·97 (4) Å; N—H···O 128 (1), 128 (1)°]. The considerable 3,5-diene character within the rings and short extra-annular C—N bonds indicate a dipole, whose positive and negative poles are focused at the amine and nitrosyl substituents respectively.

Experimental. Green crystals suitable for X-ray work were prepared by nitrosating 2-acetoxymercurio-4,N-dimethylaniline with sodium nitrite in concentrated

sulfuric acid at 275 K followed by recrystallization from petroleum ether (b.p. 313–333 K).

Crystal dimensions $0.4 \times 0.4 \times 0.4$ mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo $K\alpha$ radiation, unit-cell dimensions from setting angles of 25 accurately centred reflexions $(6.2 \le \theta \le 10.2°)$, ω–2θ scan mode, ω scan width $0.80 + 0.35\tan\theta(°)$ and scan speed ranging from 0.4 to $5°$ min^{-1} according to the intensity gathered in a pre-scan, $0 \le h \le 10$, $-10 \le k \le 10$, $-13 \le l \le 13$, $0 \le \theta \le 25°$, 2866 reflexions measured, 2469 unique $(R_{int} = 0.007)$, 2170 observed $[F \ge 3\sigma(F)]$, maximum fluctuation in intensity standards $(\overline{3}11, 131, 10\overline{3})$ measured every 3 h was 1.5%, Lp corrections applied but absorption ignored. MULTAN80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve the phase problem. All non-hydrogen atoms were found in Fourier maps, hydrogens from ΔF maps. The structure was refined by blocked matrix least squares based on F using SHELX76 (Sheldrick, 1976), final $R = 0.043$, $wR = 0.054$, $w = 2.0697/[\sigma^2(F) + 0.0001F^2]$, anisotropic vibrational parameters for heavier atoms, isotropic for H. Maximum fluctuation in final ΔF map in range -0.13 to $+0.16$ e Å^{-3}, maximum Δ/σ 0.065 [z, H(6)]. Scattering factors from International Tables for X-ray Crystallography (1974), computation carried out on the joint CDC7600/Amdahl 470 system of the University of Manchester Regional Computing Centre. Literature survey from the Cambridge Structural Database was performed using the Crystal Structure Search and Retrieval interactive system (CSSR, 1984). Fractional atomic coordinates and equivalent isotropic vibrational parameters for non-hydrogen atoms are presented in Table 1* and selected bond lengths and angles in Table 2. The title molecule, including atomic labelling, is displayed in Fig. 1.

Related literature. Further details of the method of preparation have been reported by Belyanov & Petrova (1977). Structural data for several C-nitroso structures have been compared by Talberg (1977), who also identified a relationship between C—N and N=O bond lengths, to which the title compound adheres. Polarization in the title molecule is comparable to that in 5-methoxy-2-nitrosophenol (Bartindale, Crowder & Morley, 1959).

Table 1. *Fractional atomic coordinates* $(\times 10^4)$ *and equivalent isotropic vibrational parameters* $(Å^2 \times 10^4)$ *for non-hydrogen atoms*

	x	y	z	U_{eq}†
C(1)	6599 (2)	4651 (2)	6197 (1)	585 (4)
C(2)	6187 (2)	3160 (2)	5495 (1)	621 (4)
C(3)	4638 (2)	2116 (2)	5488 (1)	683 (4)
C(4)	3490 (2)	2445 (2)	6134 (1)	681 (4)
C(5)	3908 (2)	3923 (2)	6841 (2)	743 (5)
C(6)	5383 (2)	4980 (2)	6874 (1)	692 (4)
N(1)	8042 (2)	5676 (2)	6238 (1)	716 (4)
C(7)	8517 (3)	7215 (3)	6930 (2)	847 (5)
N(2)	7182 (2)	2583 (2)	4793 (1)	866 (4)
O(1)	8579 (2)	3467 (2)	4752 (1)	1069 (4)
C(8)	1843 (3)	1328 (3)	6141 (2)	934 (6)
C(11)	6345 (2)	9330 (2)	8849 (1)	573 (4)
C(12)	6131 (2)	8097 (2)	9687 (1)	619 (4)
C(13)	4596 (2)	7044 (2)	9765 (2)	730 (5)
C(14)	3293 (2)	7128 (2)	9055 (2)	749 (5)
C(15)	3529 (3)	8323 (2)	8237 (2)	788 (5)
C(16)	4968 (2)	9379 (2)	8126 (2)	723 (4)
N(11)	7766 (2)	10356 (2)	8754 (1)	668 (4)
C(17)	8050 (3)	11610 (3)	7917 (2)	871 (6)
N(12)	7301 (2)	7806 (2)	10452 (1)	890 (4)
O(11)	8691 (2)	8711 (2)	10427 (1)	1053 (4)
C(18)	1657 (3)	6005 (3)	9138 (3)	1084 (8)

† $U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}\mathbf{a}_i.\mathbf{a}_j a_i^* a_j^*.$

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

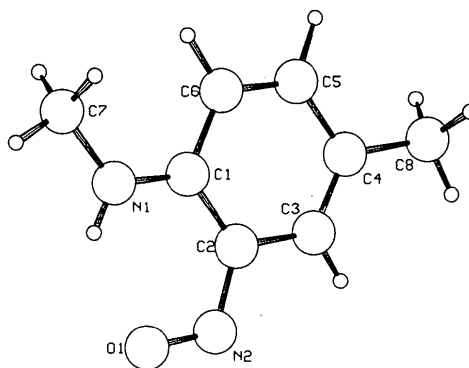|	Molecule A	Molecule B
C(1)–C(2)	1.422 (2)	1.429 (2)
C(1)–C(6)	1.412 (3)	1.398 (3)
C(1)–N(1)	1.325 (2)	1.329 (2)
C(2)–C(3)	1.405 (2)	1.411 (2)
C(2)–N(2)	1.371 (3)	1.354 (3)
C(3)–C(4)	1.344 (3)	1.346 (3)
C(4)–C(5)	1.415 (2)	1.390 (3)
C(4)–C(8)	1.498 (3)	1.504 (3)
C(5)–C(6)	1.358 (2)	1.354 (3)
N(1)–C(7)	1.450 (3)	1.443 (3)
N(2)–O(1)	1.254 (2)	1.254 (2)
N(1)–C(1)–C(2)	122.6 (2)	122.2 (2)
N(2)–C(2)–C(1)	126.1 (2)	125.8 (1)
N(2)–C(2)–C(3)	113.6 (1)	114.0 (2)
C(7)–N(1)–C(1)	124.7 (2)	124.4 (2)
O(1)–N(2)–C(2)	116.8 (2)	116.8 (2)

Fig. 1. The title molecule including atomic labelling scheme produced using PLUTO (Motherwell & Clegg, 1978). Labels for the second molecule in the asymmetric unit have been incremented by 10.

References

BARTINDALE, G. W. R., CROWDER, M. M. & MORLEY, K. A. (1959). *Acta Cryst.* **12**, 111–115.

BELYANOV, E. YU & PETROVA, S. V. (1977). *Zh. Org. Khim.* **13**, 1220–1222.

CSSR (1984). *Crystal Structure Search and Retrieval Instruction Manual.* SERC Daresbury Laboratory, Warrington, England.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO.* Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

SHELDRICK, G. M. (1976). *SHELX76.* Program for crystal structure determination. Univ. of Cambridge, England.

TALBERG, H. J. (1977). *Acta Chem. Scand. Ser. A,* **31**, 37–46.

1,8-Diphosphatricyclo[6.2.2.0²,⁷]dodeca-2(7),3,5-triene

BY MARTIN NIEGER, ERNST EGERT AND GEORGE M. SHELDRICK

Institut für Anorganische Chemie der Universität, Tammannstraße 4, D-3400 Göttingen, Federal Republic of Germany

Abstract. $C_{10}H_{12}P_2$, $M_r = 194\cdot2$, monoclinic, $P2_1/c$, $a = 10\cdot904$ (2), $b = 6\cdot345$ (1), $c = 14\cdot445$ (2) Å, $\beta = 93\cdot71$ (1)°, $V = 997\cdot3$ Å³, $Z = 4$, $D_x = 1\cdot293$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0\cdot71069$ Å, $\mu = 0\cdot37$ mm⁻¹, $F(000) = 408$, $T = 298$ K, $R = 0\cdot038$ 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) $1\cdot840$ (2) and P–C(sp^2) (mean) $1\cdot826$ (2) Å.

Experimental. Crystal size $0\cdot8 \times 0\cdot4 \times 0\cdot2$ 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 $\pm2\theta$ 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 ($R_{int} = 0\cdot035$), of which 1558 with $F > 3\sigma(F)$ were used for all calculations (*SHELXTL,* Sheldrick, 1985), $h\pm12$, 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 $R = 0\cdot038$, $wR = 0\cdot050$; 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 [$d(\text{C–H}) = 0\cdot96$ Å, H–C–H = $109\cdot5°$, $U(\text{H}) = 1\cdot2U_{eq}(\text{C})$]. 109 parameters refined, $S = 1\cdot53$, weighting scheme $w^{-1} = \sigma^2(F) + 0\cdot0005F^2$, which led to a featureless analysis of variance in terms of $\sin\theta$ and F_o, max. $\Delta/\sigma=0\cdot005$, max. and min. heights in final $\Delta\rho$ map $0\cdot26$ and $-0\cdot25$ e Å⁻³, respectively.

Table 1. *Atomic coordinates (× 10⁵ for P, × 10⁴ for rest) and equivalent isotropic displacement parameters (Å² × 10⁴ for P, Å² × 10³ 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)