0·547 (5) and 0·549 (5) Å in NONCO and $NONCH_2$, respectively.

There are appreciable distortions in the $C_6H_5{-}C{=}C{-}X{-}C{=}C{-}C_6H_5$ fragments of the molecules, limiting π-electron interactions. The $C_6H_5{-}C{=}C$ torsion angles range from 11·3 (4), 40·2 (3)° in $NONCH_2$ to 25·6 (2), 29·2 (2)° in NONCO. The C=C's are approximately planar in both molecules. There is substantial distortion in the C=C–C=O fragments in NONCO, the central C–C bonds being twisted by 29·7 (3) and 55·0 (2)°. The torsional angles in these molecules suggests that conjugation between a substituent linked *via* the exocyclic double bond, and the π electrons in the nine-membered rings, will be minimal. Additionally, the π-electron system in the biphenyl fragment is effectively isolated by the large torsion angles (64–79°) in the bonds connecting this fragment to the $C_6H_5{-}C{=}C{-}X{-}C{=}C{-}C_6H_5$ region. It would seem that the double benzene annelation has stabilized the basic cyclononatetraene structure by effectively removing two of the C=C's from the ring, thereby converting it to a $C{=}C{-}X{-}C{=}C$ structure. An example of the instability of the (I*a*) type of structure may be found in nonafulvene (I*a*, $X = CH_2$), which has a half-life of only 60 min in hexane solution at room temperature (Neuenschwander & Frey, 1975).

The crystal packing is ordinary.

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Structures of (*E*,*E*)-2,3-Butanedione Bis(4′-nitrophenylhydrazone) (I), $C_{16}H_{16}N_6O_4$, as the Bis(*N*,*N*-dimethylformamide) Adduct (III), $C_{16}H_{16}N_6O_4.2C_3H_7NO$, and (*E*)-2,3-Butanedione 4′-Nitrophenylhydrazone (II), $C_{10}H_{11}N_3O_3$

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Abstract. (III): $M_r = 502{\cdot}2$, $P\bar{1}$, $a = 11{\cdot}656\,(8)$, $b = 6{\cdot}888\,(6)$, $c = 8{\cdot}574\,(7)$ Å, $\alpha = 85{\cdot}4\,(1)$, $\beta = 69{\cdot}3\,(1)$, $\gamma = 94{\cdot}4\,(1)°$, $V = 637{\cdot}6$ Å^3, $Z = 1$, $D_m = 1{\cdot}31\,(1)$, $D_x = 1{\cdot}31$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 1{\cdot}05$ cm^{-1}, $F(000) = 266$, $T = 298$ K, $R = 0{\cdot}086$ for 707 independent reflections. The molecule takes up the *E*,*E* conformation and contains a crystallographic centre of symmetry. There is a strong intermolecular hydrogen bond between each solvent *N*,*N*-dimethylformamide molecule and an NH group. (II): $M_r = 221{\cdot}1$, $P2_1/c$, $a = 12{\cdot}474\,(8)$, $b = 9{\cdot}220\,(7)$, $c = 10{\cdot}049\,(7)$ Å, $\beta = 112{\cdot}1\,(1)°$, $V = 1070{\cdot}6$ Å^3, $Z = 4$, $D_m = 1{\cdot}37\,(1)$ g cm^{-3}, $D_x = 1{\cdot}37$ g cm^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 1{\cdot}05$ cm^{-1}, $F(000) = 464$, $T = 298$ K, $R = 0{\cdot}066$ for 1023 independent reflections. Unusually for such molecules there is no intramolecular hydrogen bonding between the NH group and the carbonyl oxygen as has consistently been the case in all previously related structures. Instead there is an infinite chain of intermolecular hydrogen bonds.

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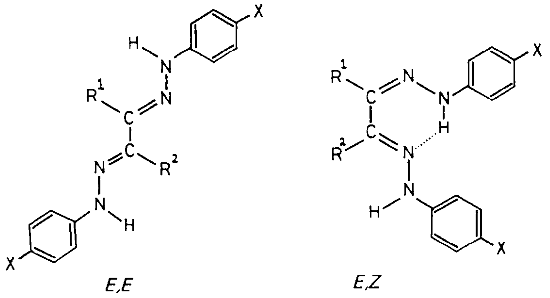
Introduction. In a recent paper Butler & Cunningham (1980) discuss the isomeric structures of a series of *para*-substituted phenylosazones of various glyoxals and 1,2-diketones. Whereas examples of both the *E,E* (predominant) and *E,Z* forms were confirmed by spectroscopic data, only the bis(phenylhydrazones) ($R^1 = R^2$) showed retention of structure *E,E* in solution and in the solid state. Another point of interest concerning the symmetrical derivatives was that each gave yellow crystals on direct treatment with hexamethylphosphoramide (HMPA). These were viewed as either the *E,Z* isomer stabilized by HMPA of crystallization or the *E,E* isomer as an adduct with two HMPA molecules with perhaps a slight preference towards the former.

Having an interest in the spectral and structural chemistry of phenylhydrazone derivatives, especially the implications arising from intramolecular hydrogen bonding (Vickery, Willey & Drew, 1981; Drew, Vickery & Willey, 1981), we were intrigued by these observations and decided to carry out an X-ray crystallographic study. Compound (I) with $R^1 = R^2 = CH_3$, $X = NO_2$ was selected as being the most suitable model.



$R^1 = R^2$ and $R^1 \neq R^2$ for R = H, Me, Ph, $(CH_2)_4$;
$X = NO_2$, Br
This work: $R^1 = R^2 = CH_3$; $X = NO_2$ for (I)

Experimental. Reaction of 2,3-butanedione and *p*-nitrophenylhydrazine following the Vogel (1978) recipe gave a bright red solid which was assumed to be the bis(*p*-nitrophenylhydrazone) derivative (I). However, a variable m.p. 498–505 K and certain spectral discrepancies suggested otherwise. Since the material was crystalline, we decided to carry out an X-ray structure determination; quite fortuitously the crystal selected turned out to be the single condensation product 2,3-butanedione 4′-nitrophenylhydrazone (II) (see Fig. 1). Rather than a chromatographic separation of the mixture – the bis derivative (I) was assumed to have been formed in some degree – the reaction was repeated, this time with a threefold excess of *p*-nitrophenylhydrazine and under more forcing conditions. The deep-red solid thus obtained was confirmed by microanalysis as the expected double condensation product 2,3-butanedione bis(4′-nitrophenylhydrazone) (I), m.p. 583–585 K, literature: 584–585 K (Butler & Cunningham, 1980).

Attempts to recrystallize (I) from the common organic solvents were disappointing but success came with strong polar/donor solvents, *e.g.* Me_2SO or $HCONMe_2$ (DMF). Specifically, the deep red crystalline solid (III) was isolated from concentrated DMF solutions and was shown to contain (microanalysis) two molecules of coordinated solvent [m.p. 606–607 K; ν(NH) 3250 cm^{-1} (mull); δ_{NH} 10·55 ($HCONMe_2$, 220 mHz)]. The formation of a yellow solid over a 24 h period from a concentrated solution of (I) in HMPA as solvent was observed but in our hands this was neither crystalline nor isolable.

On the evidence that the two DMF solvent molecules in (III) are bound by hydrogen bonding to the NH centres (see *Discussion* and Fig. 3), it would appear that the yellow crystals previously noted (Butler & Cunningham, 1980) were the analogous bis(HMPA) adducts but with (*a*) a much weaker hydrogen-bonding N—H····O=P interaction and (*b*) unfavourable steric requirements.

D_m for (II) and (III) by flotation, 0·2 × 0·2 × 0·5 mm (II), 0·3 × 0·3 × 0·4 mm (III), Stoe STADI2, cell dimensions from high-angle axial reflections, variable-width ω scan, background counts 20s, scan rate 0·033 °s^{-1}, scan width 1·5 + sin μ/tan θ, maximum 2θ 50° (II and III), systematic absences for (II) $h0l$, $h+l = 2n+l$, $0k0$, $k=2n+1$, absorption and extinction corrections not applied, standard reflections measured every 30 min for each layer, no significant intensity variation, 1899 (II) and 2206 (III) reflections measured, 1023 (II), 707 (III) with $I > 3\sigma(I)$; (II) solved with *MULTAN* 80 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1980); (III) more difficult, correct orientation from *MULTAN* 80 in $P1$, converted to $P\bar{1}$; (II) and (III) full-matrix least squares (*SHELX* 76, Sheldrick, 1976), weighting scheme chosen to give equivalent values of $w\Delta^2$ over ranges of F_o and $\sin\theta/\lambda$, $w = 1/[\sigma^2(F) + 0{\cdot}003F^2]$, $\sigma(F)$ from counting statistics, O, C, N anisotropic, H bonded to C placed in tetrahedral or trigonal positions, thermal parameters refined but those on the same atom were constrained to be equivalent; H on N(7) allowed to refine independently, H on methyl groups refined as rigid groups with a common thermal parameter, scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELX* 76 on the CDC 7600 at the University of Manchester Computer Centre; (II): $R = 0{\cdot}066$, R_w 0·077; (III): $R = 0{\cdot}086$, $R_w = 0{\cdot}101$.*

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* Lists of structure factors, anisotropic thermal parameters, H-atom positions and least-squares planes' calculations have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38245 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Discussion. Coordinates are listed in Tables 1 and 2, details of interatomic distances and angles in Table 3.

The structure of the mono-*p*-nitrophenylhydrazone derivative (II) is shown in Fig. 1 using the atomic numbering scheme adopted by us in previous phenylhydrazone structures (Drew, Vickery & Willey, 1981). It is remarkable that the *E* isomer is formed preferentially over the *Z* isomer since the latter would almost certainly feature an NH···O=C intramolecular hydrogen bond. In all our previous phenylhydrazone structures (Vickery *et al.*, 1981) (and there are now over a dozen of them), such has been the case and it is difficult to see why (II) should prove to be the exception. Instead of the intramolecular hydrogen bond, an intermolecular chain of hydrogen bonds is formed with N(7)···O(21) ($\frac{1}{2}+x$, $\frac{1}{2}-y$, $\frac{1}{2}+z$) 3·028 (5) Å. (The arrangement is shown in Fig. 2 in the *z* projection.) The N–H···O angle is 163 (4)°.* These values can be compared with (mean) N···O 2·65 Å and (mean) N–H···O 127° for a typical intramolecular hydrogen bond. The H(7)···O(21) distance is 2·25 (4) Å [as

Table 1. *Atomic coordinates* (× 10⁴) *for* (II) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}(Å^2 × 10^3)†
C(1)	6204 (3)	7171 (4)	10684 (4)	73 (5)
C(2)	7045 (4)	6202 (5)	10679 (4)	75 (5)
C(3)	6817 (4)	5308 (5)	9518 (4)	77 (5)
C(4)	5754 (3)	5335 (4)	8391 (4)	73 (4)
C(5)	4931 (4)	6330 (5)	8411 (5)	78 (5)
C(6)	5162 (4)	7233 (5)	9551 (5)	89 (6)
N(1)	6427 (3)	8081 (4)	11942 (4)	84 (5)
O(1)	7305 (3)	7893 (4)	12998 (4)	89 (5)
O(2)	5716 (3)	8995 (4)	11912 (4)	118 (6)
N(7)	5524 (3)	4354 (4)	7275 (4)	76 (4)
N(8)	4471 (3)	4350 (3)	6209 (3)	71 (4)
C(9)	4221 (3)	3366 (4)	5221 (4)	78 (5)
C(20)	3025 (4)	3459 (5)	4147 (4)	78 (5)
O(21)	2667 (3)	2535 (3)	3207 (3)	85 (4)
C(10)	5012 (5)	2195 (5)	5119 (6)	97 (6)
C(22)	2249 (4)	4667 (6)	4227 (5)	101 (6)

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

Table 2. *Atomic coordinates* (×10⁴) *for* (III) *with e.s.d.'s in parentheses*

	x	y	z	U_{eq}(Å^2 × 10^3)†
C(1)	6935 (9)	8435 (13)	2237 (11)	65 (13)
C(2)	7484 (8)	7270 (14)	1015 (12)	72 (13)
C(3)	6823 (8)	5500 (13)	932 (12)	68 (12)
C(4)	5611 (8)	5035 (12)	2003 (11)	62 (12)
C(5)	5090 (9)	6266 (13)	3237 (11)	71 (12)
C(6)	5739 (8)	7926 (14)	3371 (11)	62 (12)
N(7)	4894 (7)	3330 (10)	1942 (9)	63 (10)
N(8)	5383 (7)	2156 (11)	708 (8)	65 (10)
C(9)	4689 (9)	583 (14)	689 (11)	62 (12)
C(10)	3425 (9)	−38 (13)	1910 (12)	74 (13)
N(1)	7619 (10)	10231 (12)	2366 (13)	84 (15)
O(1)	7142 (8)	11203 (11)	3511 (10)	102 (13)
O(2)	8646 (9)	10729 (12)	1289 (11)	107 (13)
N(11)	9399 (8)	6280 (13)	3561 (10)	73 (12)
C(12)	10058 (13)	8162 (22)	3692 (18)	113 (24)
C(13)	10009 (12)	5160 (20)	2186 (14)	121 (20)
C(14)	8283 (11)	5670 (16)	4602 (14)	82 (16)
O(15)	7678 (6)	6493 (11)	5783 (9)	103 (12)

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

Table 3. *Molecular dimensions* (Å, °)

(II)

C(1)–C(2)	1·380 (6)	N(1)–O(1)	1·214 (4)
C(1)–C(6)	1·367 (6)	N(1)–O(2)	1·215 (5)
C(1)–N(1)	1·449 (5)	N(7)–N(8)	1·344 (5)
C(2)–C(3)	1·363 (6)	N(8)–C(9)	1·289 (5)
C(4)–C(3)	1·379 (5)	C(9)–C(20)	1·476 (6)
C(4)–C(5)	1·382 (6)	C(9)–C(10)	1·492 (6)
C(4)–N(7)	1·380 (5)	C(20)–O(21)	1·222 (5)
C(5)–C(6)	1·352 (6)	C(20)–C(22)	1·497 (6)

C(2)–C(1)–C(6)	120·8 (4)	C(1)–N(1)–O(1)	119·1 (3)
C(2)–C(1)–N(1)	118·8 (3)	C(1)–N(1)–O(2)	118·8 (3)
C(6)–C(1)–N(1)	120·4 (4)	O(1)–N(1)–O(2)	122·1 (4)
C(1)–C(2)–C(3)	118·6 (4)	C(4)–N(7)–N(8)	119·1 (3)
C(2)–C(3)–C(4)	120·9 (4)	N(7)–N(8)–C(9)	119·4 (3)
C(3)–C(4)–C(5)	119·3 (4)	N(8)–C(9)–C(20)	113·7 (3)
C(3)–C(4)–N(7)	119·5 (3)	N(8)–C(9)–C(10)	125·9 (3)
C(5)–C(4)–N(7)	121·2 (3)	C(20)–C(9)–C(10)	120·3 (3)
C(1)–C(6)–C(5)	120·4 (4)	C(9)–C(20)–O(21)	119·7 (4)
C(4)–C(5)–C(6)	119·9 (4)	C(9)–C(20)–C(22)	120·0 (3)

(III)

C(1)–C(2)	1·375 (11)	N(8)–C(9)	1·306 (10)
C(1)–C(6)	1·386 (11)	C(9)–C(10)	1·480 (11)
C(1)–N(1)	1·453 (11)	C(9)–C(9^{i})	1·483 (15)
C(2)–C(3)	1·412 (12)	N(1)–O(1)	1·223 (11)
C(3)–C(4)	1·381 (11)	N(1)–O(2)	1·227 (10)
C(4)–C(5)	1·406 (10)	N(11)–C(12)	1·488 (14)
C(4)–N(7)	1·403 (10)	N(11)–C(13)	1·458 (12)
C(5)–C(6)	1·357 (11)	N(11)–C(14)	1·304 (11)
N(7)–N(8)	1·371 (3)	C(14)–O(15)	1·221 (10)

C(2)–C(1)–C(6)	121·5 (8)	N(7)–N(8)–C(9)	117·4 (6)
C(2)–C(1)–N(1)	119·7 (9)	N(8)–C(9)–C(9^{i})	112·9 (7)
C(6)–C(1)–N(1)	118·8 (8)	C(9^{i})–C(9)–C(10)	122·2 (7)
C(1)–C(2)–C(3)	119·2 (8)	N(8)–C(9)–C(10)	124·9 (7)
C(2)–C(3)–C(4)	119·5 (8)	C(1)–N(1)–O(1)	119·1 (10)
C(3)–C(4)–C(5)	119·1 (8)	C(1)–N(1)–O(2)	118·1 (9)
C(3)–C(4)–N(7)	122·1 (7)	O(1)–N(1)–O(2)	122·8 (9)
C(5)–C(4)–N(7)	118·8 (8)	C(12)–N(11)–C(13)	118·4 (9)
C(4)–C(5)–C(6)	121·6 (9)	C(12)–N(11)–C(14)	121·3 (9)
C(1)–C(6)–C(5)	118·9 (8)	C(13)–N(11)–C(14)	120·1 (9)
C(4)–N(7)–N(8)	119·0 (7)	N(11)–C(14)–O(15)	125·9 (10)

Symmetry code: (i) $1-x,-y,-z$.

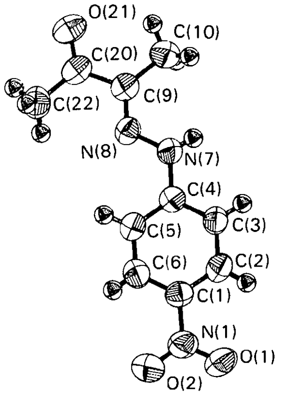

Fig. 1. The structure of (II). 50% probability ellipsoids are shown. For clarity hydrogen atoms are given a fixed radius.

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* Note that hydrogen atoms were not refined and therefore such dimensions are approximations only.

compared with 1·92 Å (mean) observed in the intermolecular-hydrogen-bond situation]. In previous work we have established that the dimensions of the phenylhydrazine skeleton are very dependent upon the presence of intramolecular hydrogen bonding between a carbonyl oxygen and N(7). With such a bond, the C(4)—N(7), N(7)—N(8), N(8)—C(9) bond lengths are 1·400, 1·307, 1·313 Å and without such a bond values are 1·36, 1·39, 1·28 Å. (These are average values taken from 18 and 9 structures respectively.) In (II), the bond lengths are 1·380 (5), 1·344 (5), and 1·289 (5) Å respectively, dimensions which are almost exactly intermediate between the two extremes. This suggests, as indeed do the dimensions of the hydrogen bond (above), that the intermolecular-hydrogen-bond linkage is considerably weaker than that of the more common intramolecular one. The dimensions of the molecule are otherwise as expected. It is interesting that the N(8)—C(9)—C(20) angle is considerably less than the N(8)—C(9)—C(10) angle, 113·7 (3) *vs* 125·9 (3)°. This must be a consequence of the requirement to keep H(7) away from C(10). Such a difference has been observed in structures with intramolecular C=O···HN hydrogen bonds and has been ascribed to the need to maximize the N—H···O angle. In the light of the structure of (II), however, it would appear to be a common feature whatever groups are bonded to C(10). As can be seen from Fig. 1, the hydrogen atoms on C(10) are positioned in such a way as to maximize their distance from H(7). The phenyl ring and atoms N(7), N(8) are essentially coplanar, but the three carbon atoms C(9), C(10), C(20) are 0·32 (2), 0·42 (2), 0·44 (2) Å respectively from this plane. As a general observation these atoms conspicuously lie outside the plane of the phenyl ring when there is no intramolecular hydrogen bonding present (Vickery *et al.*, 1981); this is certainly the case here where the deviations are among the largest found. Presumably the position of O(21) is governed to some extent by the requirements of the intermolecular hydrogen bond. Indeed it would seem likely that the position of O(21) is particularly sensitive to such packing effects. Whether the presence of an *ortho*-nitro group on the aromatic ring of (II) to assist intramolecular-hydrogen-bond formation, possibly involving a bifurcated H environment, would negate the formation of these intermolecular hydrogen bonds and lead to the *Z* isomer instead is a point under current investigation.

The structure of the *E,E* dimer (III) is shown in Fig. 3. In this *E,E* dimer form there is clearly no possibility of intramolecular hydrogen bonds. The molecule contains a crystallographically imposed centre of symmetry. The unit cell therefore contains one dimer and two solvent molecules. The packing diagram of the unit cell in the *y* projection is shown in Fig. 4. Each solvent molecule is hydrogen bonded to an NH group. The dimensions of the bond are N···O 2·96 (1) Å, N—H···O 155·5°, H···O 2·07 (5) Å, which means that

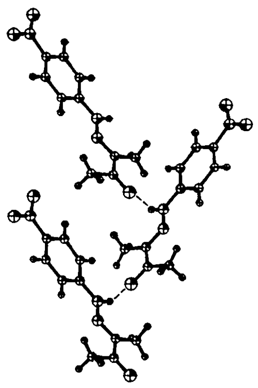

Fig. 2. Packing diagram for (II) showing the intermolecular hydrogen bond.

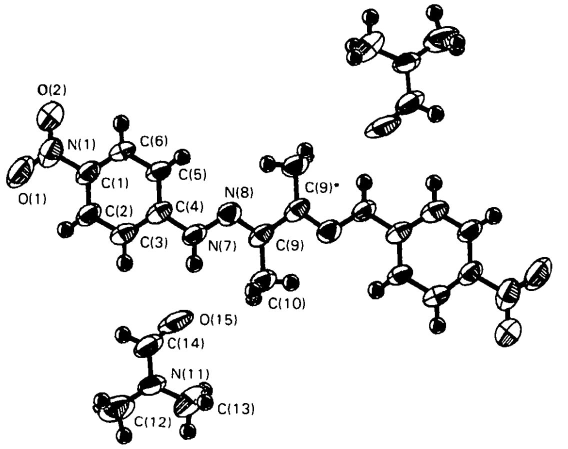

Fig. 3. The structure of (III). 50% probability ellipsoids are shown. For clarity hydrogen atoms are given a fixed radius.

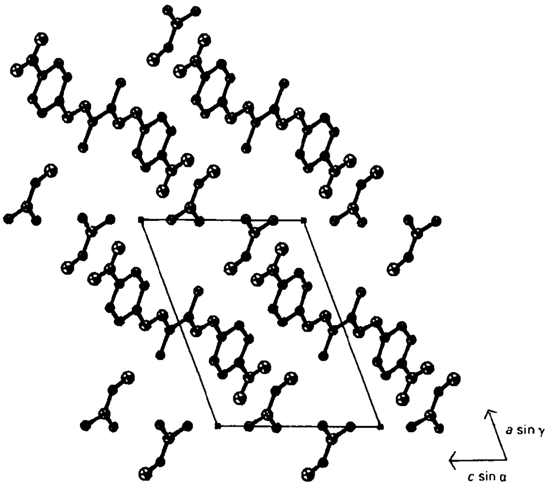

Fig. 4. Packing diagram for (III) in the **b** projection.

it is rather stronger than the intermolecular hydrogen bond in (II). The dimensions of C(4)—N(7), N(7)—N(8), N(8)—C(9), *viz* 1·403 (10), 1·371 (8), 1·306 (10) Å, respectively, are similar (within experimental error) to those observed in (II). This is not unexpected as the N···O distance is considerably longer than the mean value observed in the intramolecularly hydrogen-bonded compounds.

Though there are precedents for intermolecular hydrogen bonds in phenylhydrazone derivatives involving substituted nitro groups, *e.g. para* substitution (Menczel, 1969) and *meta* substitution (Menczel, Samay & Simon, 1972), the presence of DMF solvent strongly bound to each NH centre precludes this type of interaction in the present case. As in the monomer (II) there is a considerable difference between the angles at C(9), *viz* N(8)—C(9)—C(10), N(8)—C(9)—$C9^{i}$) are 124·9 (3), 112·9 (7)°, respectively, which must again be a reflection of the repulsions involving H(7). The whole dimer is closely coplanar with atom deviations less than 0·10 (1) Å. This includes the DMF solvent and indeed the dimer and the solvent planes intersect at only 3·1 (1)°.

Clearly the solvent molecule has been positioned in such a way so as to form strong hydrogen bonds. [The N···O distance of 2·96 (2) Å can be compared to a mean intramolecular hydrogen bond of 2·65 Å.] The packing diagram (Fig. 4) illustrates the way in which planes of dimers and solvent are formed. It may well be the case that the size and geometry of the DMF ligand are particularly critical to the formation of the bis adduct structure. In particular we note that two solvent molecules fit squarely in between the *p*-nitro groups of adjacent molecules. HMPA is far more bulky and will clearly not fit easily into these regular planes.

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Structural Studies of Pyrimidine Cyclonucleoside Derivatives. VII. Structure of 6,5′-Anhydro-6-hydroxy-1-β-D-ribofuranosylcytosine (6,5′-*O*-cyclo-C), $C_9H_{11}N_3O_5$

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Abstract. $M_r = 241·2$, orthorhombic, $P2_12_12_1$, $a = 7·047$ (1), $b = 11·292$ (1), $c = 12·403$ (1) Å, $V = 987·1$ (2) Å^3, $Z = 4$, $D_m = 1·614$ (1), $D_x = 1·623$ Mg m^{-3}, μ(Cu $K\alpha$) = 1·17 mm^{-1}, $F(000) = 504$, $T = 293$ K, $R = 0·053$ for 974 independent reflexions. The glycosidic torsion angle is in the *anti* range, $\chi_{CN} = 64·9$ (6)°, and the ribose-ring conformation is C(4′)-*endo*.

Introduction. As a series of structural studies on pyrimidine cyclonucleosides, we have reported the structures of 2,2′-cyclonucleosides (parts II, III and V: Yamagata, Suzuki, Fujii, Fujiwara & Tomita, 1979; Yamagata, Koshibe, Tokuoka, Fujii, Fujiwara, Kanai & Tomita, 1979; Yamagata, Yoshimura, Fujii, Fujiwara, Tomita & Ueda, 1980), of a 2,5′-cyclonucleoside (part IV: Yamagata, Fujii, Fujiwara,

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