$Q = 0.588$ (6) and $\theta = 126.6$ (6). Atomic scattering factors for C, H and O were those stored in *SHELXTL-Plus* which were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,* selected bond lengths, angles and torsion angles are given in Table 2. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1.

Related literature. Recently we observed (Khanapure, Crenshaw, Reddy & Biehl, 1988) that the reactions of diaryl-2-cyano-α-lithiomethanes with benzaldehydes are highly diastereoselective which is in accord with Cram's rule (Cram & Wilson, 1963). The lithio salts formed in these reactions undergo further intramolecular addition to nitrile to give isochroman-1-ones upon acidic work-up. The lithiation of alkylbenzonitriles and subsequent addition of the resulting anion to alkylbenzonitrile itself to give ketones is known (Kaiser & Petty, 1976). The one-pot high-yield synthesis of *cis*-3,4-diarylisochroman-1-one is of significance due to the presence of the 3-arylisochroman-1-one moiety in several natural products such as hydrangenol and phyllodulcin (Watanabe, Sahara, Furukawa, Billedeau & Snieckus, 1982). Several other methods involving *ortho*-directed metallation of benzamides and reaction of aldehydes with *ortho*-toluenes have also been used (Regan & Staunton, 1987) in the synthesis of 3,4-dialkylisochroman-1-ones.

This work was supported by the Robert A. Welch Foundation under grant N-118.

* Lists of anisotropic temperature factors, bond lengths, bond angles and torsion angles, H-atom parameters, and structure factors have been deposited with British Library Document Supply Centre as Supplementary Publication No. SUP 51800 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 3-Benzamido-6,8-dibenzoyl-1,3,5,6,8-pentaazabicyclo[3.2.2]nonane

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(*Received* 2 *December* 1988; *accepted* 18 *January* 1989)

Abstract. $C_{25}H_{23}N_6O_3$, $M_r = 455.50$, monoclinic, $P2_1/c$, $a = 11.325$ (3), $b = 18.161$ (4), $c = 11.952$ (5) Å, $\beta = 112.01$ (3)°, $V = 2280.3$ (10) Å^3, $Z = 4$, $D_x = 1.327$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 7.01$ cm^{-1}, $F(000) = 956$, $T = 295$ K, final $R = 0.072$, $wR = 0.061$ for 1269 independent reflections. The benzoyl-substituted ring nitrogens are near planar while the benzamido-substituted nitrogen is pyramidal. The bonds to the remaining unsubstituted ring nitrogens, N(1) and N(5), are eclipsed with respect to each other. A Newman projection down the N(1)–N(5) axis shows a maximum of 4° deviation for the three pairs of eclipsed bonds. Intermolecular hydrogen bonding occurs between the secondary amine and one of the benzoyl oxygens.

Experimental. A clear colorless 0.05 × 0.08 × 0.15 mm data crystal, crystallized from methanol. Synthesized by G. Kumar and J. Boyer of the University of Illinois at Chicago. Automated Nicolet *R3m* diffractometer with incident-beam monochromator, 20 centered reflections within $25 \le 2\theta \le 50°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{max} = 0.53$ Å^{-1}, range of *hkl*: $0 \le h \le 10$, $0 \le k \le 19$,

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

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

	x	y	z	U_{eq}
N(1)	1815 (6)	2572 (4)	2624 (6)	57 (4)
C(2)	1052 (8)	3193 (5)	2005 (7)	64 (5)
N(3)	−304 (7)	3062 (4)	1681 (5)	53 (3)
C(4)	−808 (7)	2427 (5)	889 (7)	56 (4)
N(5)	−121 (6)	1757 (4)	1372 (6)	51 (3)
N(6)	208 (7)	1691 (4)	2634 (6)	57 (4)
C(7)	1282 (8)	2167 (5)	3367 (7)	64 (5)
N(8)	2060 (8)	2074 (4)	1821 (6)	59 (4)
C(9)	977 (8)	1604 (4)	1075 (7)	61 (4)
N(10)	−1058 (7)	3690 (5)	1230 (6)	64 (4)
C(11)	−1392 (8)	4142 (6)	1972 (9)	58 (5)
O(11)	−1048 (5)	4015 (3)	3043 (5)	75 (3)
C(12)	−2235 (9)	4763 (5)	1356 (10)	59 (5)
C(13)	−2314 (9)	5055 (6)	258 (10)	75 (6)
C(14)	−3200 (13)	5604 (6)	−280 (11)	105 (7)
C(15)	−3978 (13)	5867 (6)	265 (14)	108 (8)
C(16)	−3880 (12)	5600 (7)	1345 (14)	105 (8)
C(17)	−2998 (11)	5060 (6)	1919 (9)	82 (6)
C(18)	−518 (9)	1306 (5)	3110 (9)	58 (5)
O(18)	−282 (5)	1370 (3)	4196 (5)	77 (3)
C(19)	−1514 (10)	820 (5)	2297 (8)	52 (5)
C(20)	−2670 (12)	811 (5)	2410 (7)	70 (5)
C(21)	−3622 (10)	328 (8)	1746 (11)	92 (7)
C(22)	−3374 (12)	−165 (6)	1007 (10)	90 (7)
C(23)	−2235 (13)	−168 (5)	875 (8)	77 (6)
C(24)	−1310 (9)	321 (6)	1519 (9)	65 (5)
C(25)	3175 (10)	2047 (5)	1646 (8)	61 (5)
O(25)	3220 (6)	1618 (3)	870 (5)	80 (3)
C(26)	4304 (9)	2500 (5)	2383 (8)	49 (4)
C(27)	5229 (11)	2527 (5)	1922 (9)	86 (6)
C(28)	6329 (11)	2931 (6)	2491 (11)	98 (7)
C(29)	6544 (9)	3284 (5)	3547 (9)	70 (5)
C(30)	5634 (10)	3244 (5)	4019 (8)	81 (5)
C(31)	4521 (9)	2859 (5)	3449 (9)	74 (5)

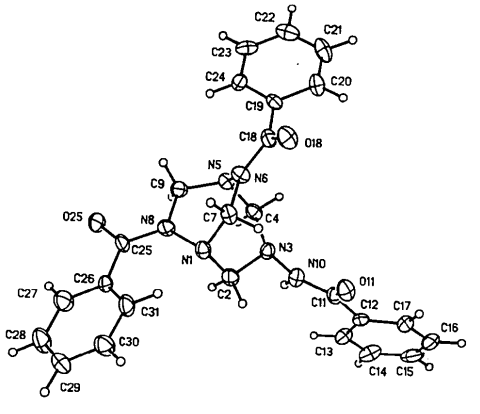

Fig. 1. Thermal ellipsoid plot of 3-benzamido-6,8-dibenzoyl-1,3,5,6,8-pentaazabicyclo[3.2.2]nonane with ellipsoids drawn at the 20% probability level.

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

N(1)–C(2)	1·445 (11)	N(1)–C(7)	1·448 (13)
N(1)–N(8)	1·419 (11)	C(2)–N(3)	1·457 (11)
N(3)–C(4)	1·466 (10)	N(3)–N(10)	1·405 (10)
C(4)–N(5)	1·443 (10)	N(5)–N(6)	1·416 (10)
N(5)–C(9)	1·442 (13)	N(6)–C(7)	1·482 (10)
N(6)–C(18)	1·356 (14)	N(8)–C(9)	1·486 (10)
N(8)–C(25)	1·356 (15)	N(10)–C(11)	1·363 (14)
C(11)–O(11)	1·214 (12)	C(11)–C(12)	1·482 (13)
C(12)–C(13)	1·388 (17)	C(12)–C(17)	1·388 (18)
C(13)–C(14)	1·389 (15)	C(14)–C(15)	1·364 (24)
C(15)–C(16)	1·344 (24)	C(16)–C(17)	1·384 (16)
C(18)–O(18)	1·229 (12)	C(18)–C(19)	1·475 (12)
C(19)–C(20)	1·366 (18)	C(19)–C(24)	1·379 (15)
C(20)–C(21)	1·385 (15)	C(21)–C(22)	1·359 (19)
C(22)–C(23)	1·357 (21)	C(23)–C(24)	1·369 (14)
C(25)–O(25)	1·227 (12)	C(25)–C(26)	1·498 (12)
C(26)–C(27)	1·354 (18)	C(26)–C(31)	1·369 (13)
C(27)–C(28)	1·384 (15)	C(28)–C(29)	1·354 (16)
C(29)–C(30)	1·350 (17)	C(30)–C(31)	1·377 (13)

C(2)–N(1)–C(7)	113·9 (7)	C(2)–N(1)–N(8)	112·5 (6)
C(7)–N(1)–N(8)	108·9 (7)	N(1)–C(2)–N(3)	112·1 (7)
C(2)–N(3)–C(4)	114·6 (7)	C(2)–N(3)–N(10)	113·4 (7)
C(4)–N(3)–N(10)	110·9 (5)	N(3)–C(4)–N(5)	112·6 (5)
C(4)–N(5)–N(6)	112·7 (7)	C(4)–N(5)–C(9)	116·7 (7)
N(6)–N(5)–C(9)	109·8 (6)	N(5)–N(6)–C(7)	114·2 (7)
N(5)–N(6)–C(18)	122·1 (6)	C(7)–N(6)–C(18)	123·0 (7)
N(1)–C(7)–N(6)	111·8 (7)	N(1)–N(8)–C(9)	116·4 (8)
N(1)–N(8)–C(25)	123·8 (7)	C(9)–N(8)–C(25)	119·6 (8)
N(5)–C(9)–N(8)	109·6 (7)	N(3)–N(10)–C(11)	121·1 (7)
N(10)–C(11)–O(11)	120·9 (8)	N(10)–C(11)–C(12)	114·8 (9)
O(11)–C(11)–C(12)	124·2 (10)	C(11)–C(12)–C(13)	124·2 (11)
C(11)–C(12)–C(17)	117·2 (10)	C(13)–C(12)–C(17)	118·6 (9)
C(12)–C(13)–C(14)	119·4 (12)	C(13)–C(14)–C(15)	121·0 (12)
C(14)–C(15)–C(16)	119·8 (12)	C(15)–C(16)–C(17)	121·1 (15)
C(12)–C(17)–C(16)	120·0 (12)	N(6)–C(18)–O(18)	117·9 (7)
N(6)–C(18)–C(19)	117·8 (9)	O(18)–C(18)–C(19)	124·2 (10)
C(18)–C(19)–C(20)	117·8 (9)	C(18)–C(19)–C(24)	123·9 (10)
C(20)–C(19)–C(24)	117·9 (8)	C(19)–C(20)–C(21)	121·3 (10)
C(20)–C(21)–C(22)	118·9 (12)	C(21)–C(22)–C(23)	121·0 (10)
C(22)–C(23)–C(24)	119·5 (11)	C(19)–C(24)–C(23)	121·3 (11)
N(8)–C(25)–O(25)	116·4 (8)	N(8)–C(25)–C(26)	121·9 (9)
O(25)–C(25)–C(26)	121·7 (10)	C(25)–C(26)–C(27)	113·7 (9)
C(25)–C(26)–C(31)	128·6 (10)	C(27)–C(26)–C(31)	117·7 (8)
C(26)–C(27)–C(28)	120·7 (10)	C(27)–C(28)–C(29)	121·5 (13)
C(28)–C(29)–C(30)	117·7 (9)	C(29)–C(30)–C(31)	121·4 (10)
C(26)–C(31)–C(30)	120·9 (11)		

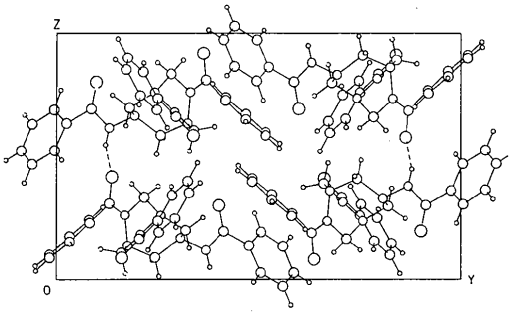

Fig. 2. Unit-cell contents viewed down the a axis. The hydrogen bonds are indicated by dashed lines.

$-12 \le l \le 11$. Standards (202, 040, 002) monitored every 60 reflections with random variation of 2·2% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K_{\alpha 1}) - 1{\cdot}0]$ to $[2\theta(K_{\alpha 2}) + 1{\cdot}0]°$, scan rate a function of count rate (2·0° min^{-1} minimum, 30·0° min^{-1} maximum), 2541 reflections measured, 2268 unique, $R_{int} = 0{\cdot}022$, 1269 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization but not for absorption effects. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$, $g = 0{\cdot}00050$. There were 310 parameters refined: atom coordinates, anisotropic thermal parameters for all non-H atoms, H atoms included using riding model, coordinate shifts of C atoms applied to bonded hydrogens, C—H = 0·96 Å. $U(H) = 1{\cdot}2U_{eq}(C)$, amine hydrogen refined isotropically. $(\Delta/\sigma)_{max} = -0{\cdot}19$, $R = 0{\cdot}072$, $wR = 0{\cdot}061$, $S = 1{\cdot}293$. The relatively high R value is due to the poor quality

and size of the crystal. Final difference Fourier excursions 0·24 and −0·22 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).* Atom numbering for Tables 1 and 2 follows that shown in Fig. 1; Fig. 2 shows the packing and hydrogen bonding. Hydrogen-bond parameters are H(10)···O(18)′ = 2·17 (7), N(10)···O(18)′ = 2·88 (1) Å, and ∠N–H···O′ = 145 (5)°.

Related literature. The title compound and a closely related pentaazabicyclo[4.2.1]nonane are products of a benzylhydrazine/formaldehyde/orthoformate condensation reaction which proceeds through the intermediate compound 1,3,5-tribenzamido-1,3,5-hexahydrotriazine. For the structure of the pentaazabicyclo-[4.2.1]nonane, see Gilardi (1987). For the structure of the intermediate, see George & Gilardi (1987).

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* Lists of structure factors, anisotropic thermal parameters and hydrogen coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51744 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of 3-Methoxy-2,4-dinitro-2,4-diazapentane

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(*Received* 2 *December* 1988; *accepted* 17 *January* 1989)

Abstract. $C_4H_{10}N_4O_5$, $M_r = 194{\cdot}15$, monoclinic, $P2_1/c$, $a = 7{\cdot}964$ (1), $b = 8{\cdot}320$ (1), $c = 14{\cdot}767$ (2) Å, $\beta = 119{\cdot}73$ (1)°, $V = 849{\cdot}7$ (2) Å^3, $Z = 4$, $D_x = 1{\cdot}518$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 1{\cdot}29$ cm^{-1}, $F(000) = 408$, $T = 295$ K, final $R = 0{\cdot}044$, $wR = 0{\cdot}059$ for 1606 independent reflections. In this compound the intramolecular contacts are primarily between first-row atoms of different electronegativity. The geometry about the amino nitrogens is very nearly planar. The angle the N—N vector makes with the three-atom plane through the amino nitrogen and the adjacent carbons in the pentane chain is 1·0° at the amino nitrogen *cis* to the methoxy carbon, and 8·5° at the amino nitrogen *trans* to the methoxy carbon.

Experimental. A clear colorless 0·35 × 0·41 × 0·65 mm data crystal was provided by H. Adolph of the Naval Surface Weapons Center. Automated Nicolet *R*3*m* diffractometer with incident-beam monochromator, 25 centered reflections within $25 \le 2\theta \le 35°$ used for determining lattice parameters. $[(\sin\theta)/\lambda]_{max} = 0{\cdot}65$ Å^{-1}, range of *hkl*: $0 \le h \le 10$, $0 \le k \le 10$, $-17 \le l \le 16$. Standards (008, 040, 700) monitored every 60 reflections with random variation of 5·1% over data collection, $\theta/2\theta$ mode, scan width $[2\theta(K_{\alpha 1}) - 1{\cdot}0]$ to $[2\theta(K_{\alpha 2}) + 1{\cdot}0]°$, scan rate a function of count rate (4·0° min^{-1} minimum, 30·0° min^{-1} maximum), 2347 reflections measured, 1955 unique, $R_{int} = 0{\cdot}017$, 1606 observed with $F_o > 3\sigma(F_o)$. Data corrected for Lorentz and polarization but not for absorption effects. Structure solved by direct methods. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/$

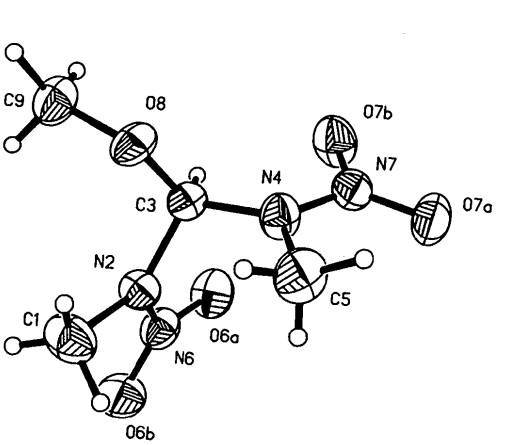

Fig. 1. Thermal ellipsoid plot of 3-methoxy-2,4-dinitro-2,4-diazapentane with ellipsoids drawn at the 20% probability level.

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