Table 3. *Hydrogen-bond geometry* (Å, °)

	Donor···Acceptor	Donor—H···Acceptor	H···Acceptor
O1*B*···O2*A*	2.631 (3)	–	–
O1*A*···O2*B*	2.638 (3)	–	–
N*A*—HN*A*···O3*A*'	2.952 (4)	154	2.17
N*B*—HN*B*···O3*B*ⁱ	3.034 (4)	163	2.19

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

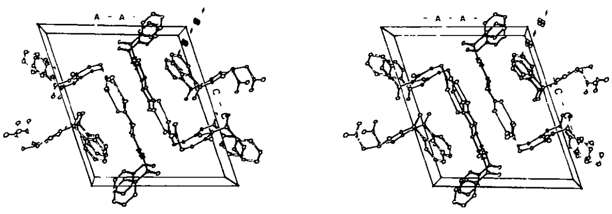

Fig. 2. A stereo pair (Johnson, 1976) seen along the *b* axis illustrating the molecular packing. The molecules are drawn as in Fig. 1 and the hydrogen bonds are shown as thin lines.

two C—O bonds in the carboxylic acid for two of the dimers where the H atoms were not located, 2,2-dibromo-1-methylcyclopropanecarboxylic acid (Rømming & Sydnes, 1987) and *trans*-dichlorobis-(*trans*,*trans*-dibenzylideneacetone)dioxouranium(IV) acetic acid solvate (Alcock, de Meester & Kemp, 1979), providing evidence for a disordered carboxylic acid dimer at low temperature in these two structures.

The CSD search showed that the present structure represents one of the few exceptions from the general observation that the hydrogen-bonded carboxylic acid dimers tend to be ordered at low temperature. This could indicate that disorder in (*R*)-*N*(1-phenylethyl)succinamic acid is of static origin.

Another interesting aspect of this molecule is that, though the two independent molecules are involved in identical hydrogen-bond patterns, they adopt different conformations. The non-bonded interactions between the chiral moieties will also influence the crystal packing and may contribute to the conformational differences.

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Structures of 9-Ethyl-3-nitrocarbazole and 1-(9-Carbazolyl)-3-(3-nitro-9-carbazolyl)propane

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Abstract. 9-Ethyl-3-nitrocarbazole, $C_{14}H_{12}N_2O_2$, $M_r = 240.26$, triclinic, $P\bar{1}$, $a = 10.572$ (4), $b = 12.910$ (5), $c = 9.547$ (4) Å, $\alpha = 94.85$ (3), $\beta = 111.62$ (3), $\gamma = 75.40$ (4)°, $V = 1172$ (1) Å^3, $Z = 4$, $D_m = 1.35$, $D_x = 1.361$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.869$ cm^{-1}, $F(000) = 504$, $T = 302$ K, $R = 0.061$, wR

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Table 1. *Positional parameters and their e.s.d.'s for the non*-H *atoms of* (I)

$$B_{eq} = (1/3)\sum_i\sum_j B_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	*y*	*z*	B_{eq}(Å^2)
O1	−0.2953 (3)	0.3598 (2)	0.1100 (3)	7.73 (9)
O2	−0.2162 (3)	0.3954 (3)	0.3474 (3)	8.93 (9)
N3	−0.2035 (3)	0.3795 (3)	0.2243 (4)	6.34 (9)
N9	0.2888 (3)	0.3996 (2)	0.1634 (3)	4.99 (8)
C1	0.1541 (4)	0.4205 (3)	0.3350 (4)	5.4 (1)
C2	0.0300 (4)	0.4129 (3)	0.3399 (4)	5.4 (1)
C3	−0.0731 (4)	0.3868 (3)	0.2112 (4)	4.80 (9)
C4	−0.0554 (3)	0.3660 (3)	0.0754 (4)	4.43 (8)
C4*A*	0.0701 (3)	0.3729 (2)	0.0677 (3)	4.20 (8)
C4*B*	0.1269 (3)	0.3540 (3)	−0.0493 (4)	4.29 (8)
C5	0.0776 (4)	0.3229 (3)	−0.1993 (4)	5.06 (9)
C6	0.1624 (4)	0.3099 (3)	−0.2827 (4)	5.8 (1)
C7	0.2932 (4)	0.3302 (3)	−0.2194 (4)	6.3 (1)
C8	0.3476 (4)	0.3608 (3)	−0.0706 (4)	5.6 (1)
C8*A*	0.2620 (3)	0.3719 (3)	0.0134 (4)	4.81 (9)
C9*A*	0.1741 (3)	0.4000 (3)	0.1980 (4)	4.60 (9)
C10	0.4201 (4)	0.4207 (3)	0.2689 (4)	6.0 (1)
C11	0.4157 (4)	0.5383 (4)	0.2824 (5)	7.6 (1)
O1′	−0.0123 (3)	0.1142 (2)	0.1666 (3)	6.99 (8)
O2′	0.0457 (2)	0.1330 (2)	0.4064 (3)	6.98 (8)
N3′	0.0734 (3)	0.1242 (2)	0.2917 (3)	5.38 (8)
N9′	0.6107 (3)	0.1359 (2)	0.3597 (3)	4.91 (7)
C1′	0.3736 (3)	0.1241 (3)	0.1804 (4)	4.77 (9)
C2′	0.2437 (3)	0.1213 (3)	0.1732 (4)	4.79 (9)
C3′	0.2139 (3)	0.1267 (3)	0.3041 (4)	4.27 (8)
C4′	0.3106 (3)	0.1347 (3)	0.4445 (3)	4.15 (8)
C4*A*′	0.4423 (3)	0.1382 (2)	0.4552 (3)	4.13 (8)
C4*B*′	0.5691 (3)	0.1448 (3)	0.5789 (3)	4.16 (8)
C5′	0.6060 (3)	0.1503 (3)	0.7344 (4)	4.78 (9)
C6′	0.7413 (4)	0.1524 (3)	0.8234 (4)	5.6 (1)
C7′	0.8396 (4)	0.1488 (3)	0.7580 (4)	5.9 (1)
C8′	0.8078 (4)	0.1435 (3)	0.6045 (4)	5.4 (1)
C8*A*′	0.6706 (3)	0.1425 (3)	0.5161 (4)	4.44 (9)
C9*A*′	0.4744 (3)	0.1325 (3)	0.3221 (4)	4.32 (8)
C10′	0.6842 (4)	0.1243 (3)	0.2547 (4)	5.6 (1)
C11′	0.7403 (4)	0.0087 (4)	0.2239 (4)	7.1 (1)

Table 2. *Bond distances* (Å) *and bond angles* (°) *for* (I)

O1—N3	1.222 (4)	O1′—N3′	1.224 (3)
O2—N3	1.226 (5)	O2′—N3′	1.224 (5)
N3—C3	1.456 (6)	N3′—C3′	1.454 (5)
N9—C8*A*	1.387 (4)	N9′—C8*A*′	1.396 (4)
N9—C9*A*	1.369 (5)	N9′—C9*A*′	1.361 (4)
N9—C10	1.459 (4)	N9′—C10′	1.456 (5)
C1—C2	1.358 (6)	C1′—C2′	1.359 (5)
C1—C9*A*	1.394 (5)	C1′—C9*A*′	1.396 (4)
C2—C3	1.392 (4)	C2′—C3′	1.389 (5)
C3—C4	1.372 (5)	C3′—C4′	1.369 (4)
C4—C4*A*	1.381 (5)	C4′—C4*A*′	1.371 (5)
C4*A*—C4*B*	1.429 (5)	C4*A*′—C4*B*′	1.440 (4)
C4*A*—C9*A*	1.411 (4)	C4*A*′—C9*A*′	1.423 (5)
C4*B*—C5	1.393 (4)	C4*B*′—C5′	1.390 (5)
C4*B*—C8*A*	1.402 (5)	C4*B*′—C8*A*′	1.403 (6)
C5—C6	1.376 (6)	C5′—C6′	1.374 (5)
C6—C7	1.374 (6)	C6′—C7′	1.387 (7)
C7—C8	1.386 (5)	C7′—C8′	1.377 (6)
C8—C8*A*	1.390 (6)	C8′—C8*A*′	1.386 (4)
C10—C11	1.503 (6)	C10′—C11′	1.508 (6)

O1—N3—O2	123.9 (4)	O1′—N3′—O2′	122.8 (3)
O1—N3—C3	117.5 (3)	O1′—N3′—C3′	118.5 (3)
O2—N3—C3	118.6 (3)	O2′—N3′—C3′	118.6 (3)
C8*A*—N9—C9*A*	108.9 (3)	C8*A*′—N9′—C9*A*′	108.6 (3)
C8*A*—N9—C10	125.6 (3)	C8*A*′—N9′—C10′	125.9 (3)
C9*A*—N9—C10	125.4 (3)	C9*A*′—N9′—C10′	125.3 (3)
C2—C1—C9*A*	117.5 (3)	C2′—C1′—C9*A*′	118.1 (4)
C1—C2—C3	120.6 (4)	C1′—C2′—C3′	120.2 (3)
N3—C3—C2	117.8 (4)	N3′—C3′—C2′	118.6 (3)
N3—C3—C4	119.4 (3)	N3′—C3′—C4′	118.3 (3)
C2—C3—C4	122.9 (4)	C2′—C3′—C4′	123.1 (3)
C3—C4—C4*A*	117.7 (3)	C3′—C4′—C4*A*′	118.0 (3)
C4—C4*A*—C4*B*	133.5 (3)	C4′—C4*A*′—C4*B*′	134.1 (3)
C4—C4*A*—C9*A*	119.3 (3)	C4′—C4*A*′—C9*A*′	119.6 (3)
C4*B*—C4*A*—C9*A*	107.1 (3)	C4*B*′—C4*A*′—C9*A*′	106.3 (3)
C4*A*—C4*B*—C5	134.3 (3)	C4*A*′—C4*B*′—C5′	134.2 (4)
C4*A*—C4*B*—C8*A*	106.5 (3)	C4*A*′—C4*B*′—C8*A*′	106.6 (3)
C5—C4*B*—C8*A*	119.2 (4)	C5′—C4*B*′—C8*A*′	119.2 (3)
C4*B*—C5—C6	119.5 (3)	C4*B*′—C5′—C6′	119.4 (4)
C5—C6—C7	120.1 (3)	C5′—C6′—C7′	120.0 (4)
C6—C7—C8	122.7 (4)	C6′—C7′—C8′	122.6 (3)
C7—C8—C8*A*	116.7 (4)	C7′—C8′—C8*A*′	116.7 (4)
N9—C8*A*—C4*B*	108.8 (3)	N9′—C8*A*′—C4*B*′	109.1 (3)
N9—C8*A*—C8	129.4 (3)	N9′—C8*A*′—C8′	128.8 (4)
C4*B*—C8*A*—C8	121.8 (3)	C4*B*′—C8*A*′—C8′	122.1 (3)
N9—C9*A*—C1	129.4 (3)	N9′—C9*A*′—C1′	129.6 (4)
N9—C9*A*—C4*A*	108.5 (3)	N9′—C9*A*′—C4*A*′	109.4 (3)
C1—C9*A*—C4*A*	122.0 (4)	C1′—C9*A*′—C4*A*′	121.1 (3)
N9—C10—C11	112.1 (3)	N9′—C10′—C11′	112.5 (4)

= 0.077 for 2316 independent reflections with $I \geq 3\sigma(I)$ and 326 variables. 1-(9-Carbazolyl)-3-(3-nitro-9-carbazolyl)propane, $C_{27}H_{21}N_3O_2$, $M_r = 419.49$, triclinic, $P\bar{1}$, $a = 13.1193$ (3), $b = 8.8488$ (2), $c = 9.3648$ (4) Å, $\alpha = 100.35$ (3), $\beta = 103.16$ (2), $\gamma = 92.61$ (2)°, $V = 1037$ (1) Å^3, $Z = 2$, $D_m = 1.34$, $D_x = 1.343$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.805$ cm^{-1}, $F(000) = 440$, $T = 302$ K, $R = 0.052$, $wR = 0.073$ for 2668 independent reflections with $I \geq 3\sigma(I)$ and 290 variables. Bond distances and angles are consistent with those in related structures. Distortions introduced by the presence of the nitro substituents are slight. The nitrated carbazole rings of both structures associate intimately in pairs, probably owing to intermolecular dipole–dipole interactions which are stronger between nitrocarbazole rings than between their unsubstituted counterparts.

Introduction. Our interest in the mechanism of electrophilic aromatic substitution has prompted us to examine several reactions which can be shown to proceed with an initial electron-transfer step. The photochemical nitration of aromatic compounds by tetranitromethane is one example of such a process (Masnovi, Krafcik, Baker & Towns, 1990). As part of the product analysis, we have undertaken single-crystal X-ray structure determinations of two products formed by this type of reaction: 9-ethyl-3-nitrocarbazole (I), derived from nitration of a monochromophoric species, and 1-(9-carbazolyl)-3-(3-nitro-9-carbazolyl)propane (II), derived from a bichromophoric system.

Experimental. Compound (I) was prepared by photochemical nitration of 9-ethylcarbazole with tetranitromethane (Iles & Ledwith, 1969). Purification was accomplished by column chromatography on silica gel, eluting fractionally with petroleum ether and dichloromethane. Compound (II) (Masnovi, Krafcik, Baker & Towns, 1990) was prepared under identical conditions from 1,3-di(9-carbazolyl)propane (Johnson, 1974).

An Enraf–Nonius CAD-4 X-ray diffractometer equipped with a graphite monochromator was used for data collection. Cell parameters were determined from accurately measured goniometer settings for 25 reflections, $5.4 < \theta < 11.7°$ for (I) and $8.1 < \theta < 13.7°$ for (II). No conditions were observed for limiting possible reflections. Three intensity controls were measured at 1 h intervals and showed no decay over

42 h exposure for (I) and 37 h exposure for (II). The structures were solved in $P\bar{1}$ by direct methods. The H atoms were located or generated in chemically reasonable positions. $F_{\rm obs}$ were corrected for Lorentz and polarization effects, and empirical absorption corrections were applied. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Full-matrix least-squares refinements were performed with Enraf–Nonius (1983) *SDP-Plus* on a PDP-11 minicomputer; function minimized $\sum w(|F_o| - |F_c|)^2$ with $w = 1.0/\sigma(F_o)^2$.

9-Ethyl-3-nitrocarbazole. Crystals of (I) suitable for X-ray diffraction were grown by slow evaporation from a concentrated solution in dichloromethane/hexane (crystal dimensions 0.42 × 0.48 × 0.49 mm); $2.0 \leq \theta \leq 25.0°$. Of 4139 unique reflections collected with ω–2θ scans over index ranges $-12 \leq h \leq 12$, $-15 \leq k \leq 15$, $0 \leq l \leq 11$, 2316 reflections were considered observed $[I > 3\sigma(I)]$. An empirical absorption correction ranging from 0.974 to 1.000 was applied on the basis of ψ scans for 12 reflections with χ near 90°, measured at 10° intervals from 0 to 360°. The final refinement parameters for 326 variables are $R = 0.061$, $wR = 0.077$; $w = 1/\sigma^2(F_o)$, $S = 1.72$; $(\Delta/\sigma)_{\rm max} < 0.01$. The largest peak in the resulting difference Fourier map was 0.16 e Å^{-3} and the deepest hole was -0.32 e Å^{-3}.

Final positional parameters and equivalent isotropic displacement parameters are listed in Table 1.* Important bond lengths and angles are given in Table 2. The conformation of the molecule is illustrated by the *ORTEP* (Johnson, 1976) drawing in Fig. 1(*a*), which also defines the atom-labeling scheme. The intermolecular interactions between carbazole rings are shown in Fig. 2(*a*).

1-(9-Carbazolyl)-3-(3-nitro-9-carbazolyl)propane. Crystals of (II) suitable for X-ray diffraction were grown by slow evaporation from a concentrated solution in chloroform/cyclohexane (crystal dimensions 0.39 × 0.31 × 0.51 mm); $2.0 \leq \theta \leq 25.0°$. Of 3647 unique reflections collected with ω–2θ scans over index ranges $-15 \leq h \leq 15$, $-10 \leq k \leq 10$, $0 \leq l \leq 11$, 2668 reflections were considered observed $[I > 3\sigma(I)]$. An empirical absorption correction ranging from 0.940 to 1.000 was applied on the basis

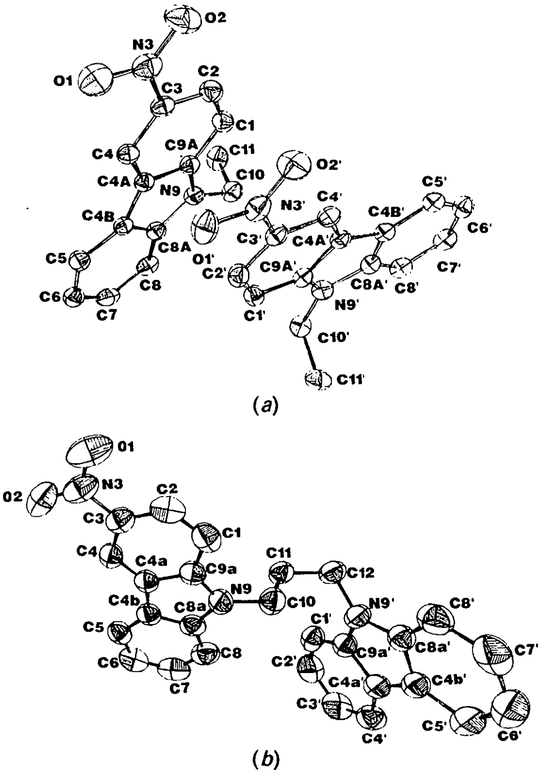

Fig. 1. (a) *ORTEP* drawing of 9-ethyl-3-nitrocarbazole (I) (thermal ellipsoids are drawn at the 20% probability level). (b) *ORTEP* drawing of 1-(9-carbazolyl)-3-(3-nitro-9-carbazolyl)-propane (II) (thermal ellipsoids are drawn at the 50% probability level).

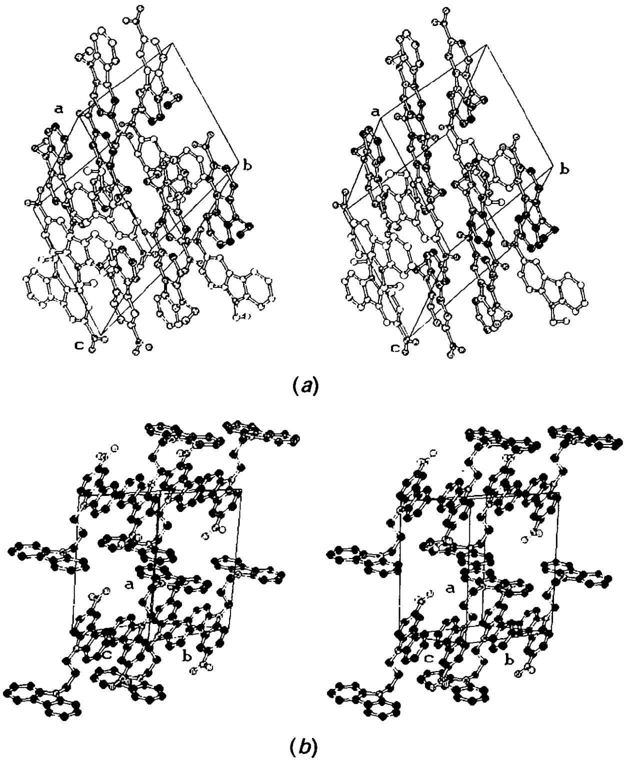

Fig. 2. Packing diagrams of (*a*) (I) and (*b*) (II).

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55356 (30 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0584]

Table 3. *Positional parameters and their e.s.d.'s for the non*-H *atoms of* (II)

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

	x	y	z	B_{eq}(Å^2)
O1	−0.3165 (2)	−0.1974 (2)	0.2088 (2)	7.90 (6)
O2	−0.2677 (2)	−0.2122 (3)	0.0016 (2)	9.37 (7)
N3	−0.2600 (2)	−0.1548 (2)	0.1325 (2)	6.39 (6)
N9	0.0579 (1)	0.3067 (2)	0.4190 (2)	4.77 (4)
C1	−0.0354 (2)	0.1354 (3)	0.1783 (3)	5.46 (6)
C2	−0.1164 (2)	0.0217 (3)	0.1153 (3)	5.63 (6)
C3	−0.1776 (2)	−0.0292 (3)	0.2031 (3)	4.96 (6)
C4	−0.1628 (2)	0.0306 (2)	0.3529 (3)	4.48 (5)
C4a	−0.0817 (2)	0.1456 (2)	0.4183 (2)	4.15 (5)
C4b	−0.0408 (2)	0.2313 (2)	0.5686 (2)	4.33 (5)
C5	−0.0693 (2)	0.2338 (3)	0.7030 (3)	5.22 (6)
C6	−0.0099 (2)	0.3313 (3)	0.8287 (3)	6.28 (7)
C7	0.0757 (2)	0.4253 (3)	0.8235 (3)	6.62 (7)
C8	0.1055 (2)	0.4257 (3)	0.6916 (3)	5.73 (6)
C8a	0.0456 (2)	0.3282 (2)	0.5650 (3)	4.65 (5)
C9a	−0.0179 (2)	0.1956 (2)	0.3306 (2)	4.46 (5)
N9′	0.3568 (1)	0.5277 (2)	0.4295 (2)	4.61 (4)
C1′	0.3126 (2)	0.6256 (3)	0.1901 (3)	5.29 (6)
C2′	0.3068 (2)	0.7614 (3)	0.1336 (3)	5.90 (6)
C3′	0.3318 (2)	0.9048 (3)	0.2263 (3)	5.96 (6)
C4′	0.3633 (2)	0.9203 (3)	0.3793 (3)	5.15 (6)
C4a′	0.3686 (2)	0.7870 (2)	0.4403 (2)	4.22 (5)
C4b′	0.3974 (2)	0.7597 (2)	0.5901 (3)	4.36 (5)
C5′	0.4303 (2)	0.8559 (3)	0.7306 (3)	5.29 (6)
C6′	0.4546 (2)	0.7906 (3)	0.8547 (3)	6.36 (7)
C7′	0.4459 (2)	0.6311 (3)	0.8416 (3)	6.58 (7)
C8′	0.4146 (2)	0.5323 (3)	0.7061 (3)	5.61 (6)
C8a′	0.3891 (2)	0.5984 (2)	0.5785 (3)	4.47 (5)
C9a′	0.3437 (2)	0.6411 (2)	0.3440 (2)	4.28 (5)
C10	0.1446 (2)	0.3763 (3)	0.3746 (3)	5.33 (6)
C11	0.2417 (2)	0.2903 (2)	0.4103 (3)	4.91 (5)
C12	0.3373 (2)	0.3616 (2)	0.3726 (3)	5.12 (6)

Table 4. *Bond distances* (Å) *and bond angles* (°) *for* (II)

N9—C8a	1.395 (3)	N9′—C8a′	1.385 (3)
N9—C9a	1.375 (2)	N9′—C9a′	1.388 (3)
N9—C10	1.448 (3)	N9′—C12	1.464 (3)
C1—C2	1.377 (3)	C1′—C2′	1.399 (4)
C1—C9a	1.393 (3)	C1′—C9a′	1.387 (3)
C2—C3	1.390 (4)	C2′—C3′	1.383 (3)
C3—C4	1.375 (3)	C3′—C4′	1.379 (4)
C4—C4a	1.390 (3)	C4′—C4a′	1.401 (3)
C4a—C4b	1.444 (3)	C4a′—C4b′	1.437 (3)
C4a—C9a	1.410 (3)	C4a′—C9a′	1.416 (3)
C4b—C5	1.391 (4)	C4b′—C5′	1.398 (3)
C4b—C8a	1.403 (3)	C4b′—C8a′	1.413 (3)
C5—C6	1.378 (3)	C5′—C6′	1.372 (4)
C6—C7	1.385 (4)	C6′—C7′	1.395 (4)
C7—C8	1.379 (4)	C7′—C8′	1.371 (3)
C8—C8a	1.386 (3)	C8′—C8a′	1.405 (4)
O1—N3	1.232 (3)	C10—C11	1.520 (3)
O2—N3	1.221 (3)	C11—C12	1.521 (4)
N3—C3	1.465 (3)		

C8a—N9—C9a	108.5 (2)	C8a′—N9′—C9a′	108.4 (2)
C8a—N9—C10	124.7 (2)	C8a′—N9′—C12	125.6 (2)
C9a—N9—C10	126.3 (2)	C9a′—N9′—C12	126.0 (2)
C2—C1—C9a	117.9 (2)	C2′—C1′—C9a′	116.9 (2)
C1—C2—C3	120.0 (2)	C1′—C2′—C3′	121.8 (2)
C2—C3—C4	123.0 (2)	C2′—C3′—C4′	121.4 (2)
C3—C4—C4a	117.8 (2)	C3′—C4′—C4a′	118.5 (2)
C4—C4a—C4b	133.9 (2)	C4′—C4a′—C4b′	133.7 (2)
C4—C4a—C9a	119.5 (2)	C4′—C4a′—C9a′	119.4 (2)
C4b—C4a—C9a	106.6 (2)	C4b′—C4a′—C9a′	106.8 (2)
C4a—C4b—C5	133.9 (2)	C4a′—C4b′—C5′	133.7 (2)
C4a—C4b—C8a	106.6 (2)	C4a′—C4b′—C8a′	106.5 (2)
C5—C4b—C8a	119.4 (2)	C5′—C4b′—C8a′	119.7 (2)
C4b—C5—C6	118.1 (2)	C4b′—C5′—C6′	118.8 (2)
C5—C6—C7	121.7 (3)	C5′—C6′—C7′	120.8 (2)
C6—C7—C8	121.4 (2)	C6′—C7′—C8′	122.4 (3)
C7—C8—C8a	117.0 (2)	C7′—C8′—C8a′	117.1 (2)
N9—C8a—C4b	108.9 (2)	N9′—C8a′—C4b′	109.3 (2)
N9—C8a—C8	128.7 (2)	N9′—C8a′—C8′	129.5 (2)
C4b—C8a—C8	122.4 (2)	C4b′—C8a′—C8′	121.1 (2)
N9—C9a—C1	129.0 (2)	N9′—C9a′—C1′	129.2 (2)
N9—C9a—C4a	109.2 (2)	N9′—C9a′—C4a′	108.9 (2)
C1—C9a—C4a	121.8 (2)	C1′—C9a′—C4a′	121.9 (2)
O1—N3—O2	123.9 (2)	N9′—C12—C11	113.1 (2)
O1—N3—C3	118.3 (2)	N9—C10—C11	110.9 (2)
O2—N3—C3	117.8 (2)	C10—C11—C12	113.7 (2)
N3—C3—C2	118.1 (2)		
N3—C3—C4	119.0 (2)		

of ψ scans for eight reflections with χ near 90°, measured at 10° intervals from 0 to 360°. The final refinement parameters for 290 variables are $R = 0.052$, $wR = 0.073$; $w = 1/\sigma^2(F_o)$, $S = 2.52$; $(\Delta/\sigma)_{\max} < 0.01$. The largest peak in the resulting difference Fourier map was 0.17 e Å^{-3} and the deepest hole was −0.26 e Å^{-3}.

Final positional parameters and equivalent isotropic displacement parameters are listed in Table 3. Important bond lengths and angles are given in Table 4. The conformation of the molecule is illustrated by the *ORTEP* (Johnson, 1976) drawing in Fig. 1(*b*), which also defines the atom-labeling scheme. The intermolecular interactions are shown in Fig. 2(*b*).

Discussion. Both (I) and (II) crystallize in the space group $P\bar{1}$ and the asymmetric units for each contain two substituted carbazole rings. The two molecules in the asymmetric unit of (I) are not equivalent owing to differences in packing. For example, the closest intermolecular contacts between methyl groups are 3.97 Å for C11···C11 and 5.34 Å for C11′···C11′. The conformation of the propylene chain of (II) is *gauche,anti* and resembles the conformation of the parent 1,3-di(9-carbazolyl)propane (Baker, Chen, Krafcik & Masnovi, 1991). However, the nitro group slightly distorts the ring to which it is attached. The interior angles about the C atoms bearing the nitro groups [C3 and C3′ of (I) and C3 of (II)] are all 123°, compared with 120.0–121.7° for the related unnitrated C atoms [C6 and C6′ of (I); C6, C3′ and C6′ of (II)]. Bond distances are also slightly affected. For example, the N9—C9*A*, N9′—C9*A*′ and N9—C9a distances (1.361–1.375 Å) are about 0.025 Å shorter than the N9—C8*A*, N9′—C8*A*′ and N9—C8a distances (1.387–1.396 Å); the corresponding lengths in the unnitrated ring of (II) (N9′—C8a′ and N9′—C9a′) are intermediate (about 1.386 Å). The O atoms of the nitro groups and the methyl C atoms of the ethyl groups have somewhat higher temperature factors than the other atoms, probably owing to the greater thermal motion of these terminal atoms.

Inspection of the packing diagrams (Fig. 2) reveals that each carbazole ring in the structures of (I) and (II) associates in a shifted parallel planar orientation with a centrosymmetrically related carbazole ring of a neighboring molecule. The dipoles of each such centrosymmetrically related pair are thus antiparallel. For (I), the distances between the centers of gravity of the unprimed and primed pairs of nitrocarbazole rings are 3.83 and 3.86 Å, respectively. The nitrocarbazole ring of (II) also associates closely with the nitrocarbazole ring of a neighboring molecule (4.00 Å between centers). Interactions which occur between the π systems of two neighboring unnitrated carbazole rings of (II) are much less extensive (5.13 Å between centers). This can be ascribed to dipolar effects which will be stronger for nitrocarbazole than for carbazole moieties.

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{2-[*p*-(*o*-Butoxybenzamido)benzoyloxy]ethyl}diethylmethylammonium Iodide

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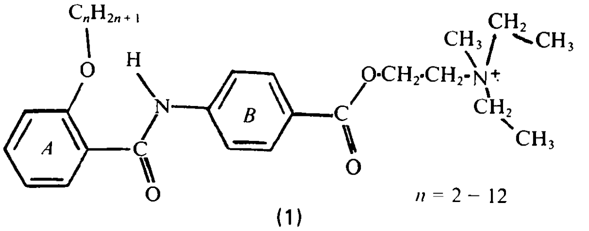
Abstract. $C_{25}H_{35}N_2O_4^+.I^-$, $M_r = 554.47$, orthorhombic, $Pbca$, $a = 13.331\,(3)$, $b = 13.797\,(5)$, $c = 28.179\,(6)$ Å, $V = 5183\,(2)$ Å^3, $Z = 8$, $D_x = 1.42$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 12.51$ cm^{-1}, $F(000) = 2272$, $T = 298$ K, $R = 0.065$ for 1734 unique observed reflections. A comparison of the title compound with other compounds belonging to the same homologous series is offered. It appears that van der Waals interactions are responsible for the differences in conformation found in the solid state of this series of compounds.

Introduction. Compounds of the series {2-[*p*-(*o*-alkyloxybenzamido)benzoyloxy]ethyl}diethylmethylammonium halide (1) show smooth-muscle relaxant activity which is strongly influenced by the length of the alkyloxy side chain and its position on the aromatic ring (Ghelardoni, Pestellini, Pisanti & Volterra, 1973; Maggi, Grimaldi, Volterra & Meli, 1983; Maggi, Manzini & Meli, 1983). This pharmacological effect increases when the alkyloxy chain is *ortho* to the amide group and reaches a maximum with the octyloxy ($n = 8$) derivative. This octyloxy derivative is almost completely devoid of central, ocular and cardiovascular atropine-like side effects when administered in doses that produce spasmolytic effects comparable with those of *N*-butylscopolammonium bromide.



(1)

The solid-state conformations of the propoxy ($n = 3$), octyloxy and decyloxy ($n = 10$) derivatives, as previously determined by X-ray structural analysis, show differences which may explain their different biological behaviour (Dapporto & Sega, 1986, 1987). To obtain information on the conformational features of these compounds in polar solution, NMR investigations have been carried out in dimethyl sulfoxide (DMSO) (Sega, Ghelardoni, Pestellini, Pogliani & Valensin, 1984; Valensin, Pogliani, Ghelardoni, Pestellini & Sega, 1984; Sega, Gaggelli & Valensin, 1985; Valensin, Gaggelli, Lepri & Sega, 1986). These studies have shown that the conformational freedom of the alkyloxy side chain changes

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