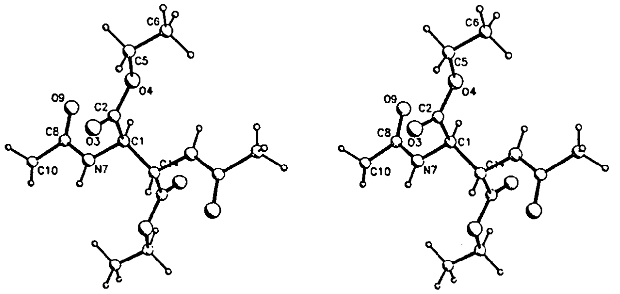

Fig. 1. Stereoscopic view of the molecule (program *PLUTO*, Motherwell & Clegg, 1978).

Table 1 gives atomic parameters,* Table 2 bond distances and angles. Fig. 1 shows a stereoscopic view of the molecule.

Related literature. This analysis is part of a study of the effects of *gem* electron-donor and -acceptor substitution on molecular deformation in dehydrodimers $(R_1R_2R_3)_2$. It was thought that the title compound could be a possible candidate for an electron deformation study but crystal quality was not sufficiently good. 1,1,2,2-Ethanetetracarbonitrile (Declercq, Tinant, Parfonry, Van Meerssche, Legrand & Lehmann, 1983), 2,3-bis(dimethylamino)-butanedinitrile (Parfonry, Declercq, Tinant, Van Meerssche & Schweiss, 1988), and 2,3-dimethyl-2,3-diphenyl-1,4-butanedinitrile (Tinant, Parfonry & Declercq, 1988) were chosen for deformation density analysis.

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* 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 52573 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1990). C46, 1148–1150

Structure of 4,4-Difluoro-1,3,5,7,8-pentamethyl-3a,4a-diaza-4-bora-*s*-indacene

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Abstract. $C_{14}H_{17}BF_2N_2$, $M_r = 262·1$, orthorhombic, *Pnma*, $a = 11·349$ (2), $b = 7·191$ (2), $c = 16·016$ (3) Å, $V = 1307·1$ (7) Å^3, $Z = 4$, $D_x = 1·332$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·927$ cm^{-1}, $F(000) = 552$, $T = 293$ K, final $R = 0·039$ for 597 reflections with $I > 3\sigma(I)$. The molecule is located on a crystallographic mirror plane passing through all of the C, N and B atoms. The C(5)—C(12) bond distance [1·512 (6) Å] is somewhat longer than the average of the other four C—CH_3 distances [1·495 (3) Å], suggesting a crowding effect. There are no unusual intermolecular distances.

Experimental. The title compound was synthesized following the procedure of Treibs & Kreuzer (1968) and recrystallized from ethyl acetate. An orange needle-shaped crystal with approximate dimensions 0·6 × 0·2 × 0·2 mm was glued to a glass fiber and mounted on an Enraf–Nonius CAD-4 diffractometer with graphite crystal monochromator. Unit-cell parameters were determined from least-squares refinement of the setting angles of 25 reflections with $18 < 2\theta < 40°$ using Mo $K\alpha$ radiation. Systematic absences, $0kl$: $k + l = 2n + 1$, $hk0$: $h = 2n + 1$, indicating either space group *Pnma* or $Pn2_1a$. *Pnma* was chosen on the basis of intensity statistics and confirmed by successful determination of the struc-

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Table 1. *Positional parameters with e.s.d.'s in parentheses*

	x	*y*	*z*	*B*(Å^2)
F1	0·1989 (2)	0·0930 (3)	0·6258 (1)	5·85 (5)
N1	0·2247 (3)	0·250	0·4948 (2)	3·21 (8)
N2	0·3825 (3)	0·250	0·6017 (2)	3·21 (8)
C1	0·1187 (4)	0·250	0·4586 (3)	3·7 (1)
C2	0·1340 (4)	0·250	0·3721 (3)	4·6 (1)
C3	0·2523 (4)	0·250	0·3542 (3)	3·9 (1)
C4	0·3114 (4)	0·250	0·4321 (3)	3·1 (1)
C5	0·4303 (4)	0·250	0·4538 (2)	2·91 (9)
C6	0·4662 (3)	0·250	0·5373 (3)	3·1 (1)
C7	0·5796 (4)	0·250	0·5756 (3)	3·5 (1)
C8	0·5602 (4)	0·250	0·6606 (3)	4·0 (1)
C9	0·4398 (4)	0·250	0·6750 (3)	3·8 (1)
C10	0·3800 (5)	0·250	0·7584 (3)	5·3 (1)
C11	0·6998 (4)	0·250	0·5376 (3)	4·8 (1)
C12	0·5246 (4)	0·250	0·3871 (3)	5·0 (1)
C13	0·3014 (5)	0·250	0·2673 (3)	5·4 (1)
C14	0·0055 (4)	0·250	0·5057 (3)	5·4 (1)
B1	0·2486 (5)	0·250	0·5887 (3)	3·6 (1)

Anisotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as: $(4/3)[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + (ab\cos\gamma)\beta(1,2) + (ac\cos\beta)\beta(1,3) + (bc\cos\alpha)\beta(2,3)]$.

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

F1—B1	1·394 (3)	C3—C4	1·417 (6)
N1—C1	1·335 (5)	C3—C13	1·499 (6)
N1—C4	1·406 (5)	C4—C5	1·394 (5)
N1—B1	1·528 (5)	C5—C6	1·398 (5)
N2—C6	1·401 (5)	C5—C12	1·512 (6)
N2—C9	1·342 (5)	C6—C7	1·427 (6)
N2—B1	1·535 (6)	C7—C8	1·380 (5)
C1—C2	1·398 (6)	C7—C11	1·494 (6)
C1—C14	1·489 (7)	C8—C9	1·385 (6)
C2—C3	1·373 (6)	C9—C10	1·498 (6)
C1—N1—C4	108·7 (3)	C4—C5—C12	120·6 (4)
C1—N1—B1	126·0 (3)	C6—C5—C12	118·0 (4)
C4—N1—B1	125·4 (3)	N2—C6—C5	120·5 (3)
C6—N2—C9	108·4 (3)	N2—C6—C7	107·2 (3)
C6—N2—B1	124·9 (3)	C5—C6—C7	132·4 (4)
C9—N2—B1	126·7 (3)	C6—C7—C8	106·3 (4)
N1—C1—C2	108·6 (4)	C6—C7—C11	130·5 (4)
N1—C1—C14	123·9 (4)	C8—C7—C11	123·3 (4)
C2—C1—C14	127·6 (4)	C7—C8—C9	108·8 (4)
C1—C2—C3	109·2 (4)	N2—C9—C8	109·4 (4)
C2—C3—C4	106·2 (4)	N2—C9—C10	124·1 (4)
C2—C3—C13	123·8 (4)	C8—C9—C10	126·5 (5)
C4—C3—C13	129·9 (4)	F1—B1—F1	108·2 (3)
N1—C4—C3	107·3 (3)	F1—B1—N1	110·3 (3)
N1—C4—C5	120·0 (4)	F1—B1—N2	110·1 (3)
C3—C4—C5	132·7 (4)	N1—B1—N2	108·0 (3)
C4—C5—C6	121·3 (3)		

ture. Intensities measured using $\omega/2\theta$ scans within the ranges $2 \le 2\theta \le 50°$, $0 \le h \le 8$, $0 \le k \le 13$, $0 \le l \le 19$. The intensities of three standard reflections measured at 2 h intervals showed a variation of less than 0·5% during data collection. Absorption as a function of ψ was measured and an empirical absorption correction applied, with relative transmission coefficients ranging from 0·999 to 0·873. Lorentz and polarization corrections were applied to a total of 1247 measured reflections of which 597 were considered observed $[I > 3\sigma(I)]$.

The structure was determined by direct methods using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980; modified by Frenz, 1982). Peaks corresponding to the 18 independent non-H atoms were located in the *E* map and the positions of the 12 independent H atoms were located in a subsequent difference Fourier summation. Refinement was by full-matrix least-squares minimization of $\sum w(|F_o| - |F_c|)^2$, where $w = 1/\sigma^2(F)$ and $\sigma^2(F^2) = \sigma^2_{cs} + (0{\cdot}04F^2)^2$. Positions and anisotropic thermal parameters were refined for non-H atoms, positions and isotropic thermal parameters for H atoms.* Final $R = 0{\cdot}039$, $wR = 0{\cdot}048$, $S = 1{\cdot}70$ and $(\Delta/\sigma)_{\max} = 0{\cdot}01$ for 154 variables. Maximum and minimum peaks in final difference Fourier synthesis 0·16 and $-0{\cdot}14$ e Å^{-3}, respectively. Atomic scattering factors and anomalous-dispersion corrections taken from *International Tables for X-ray Crystallography* (1974), and all computer programs from the *SDP* system (Frenz,

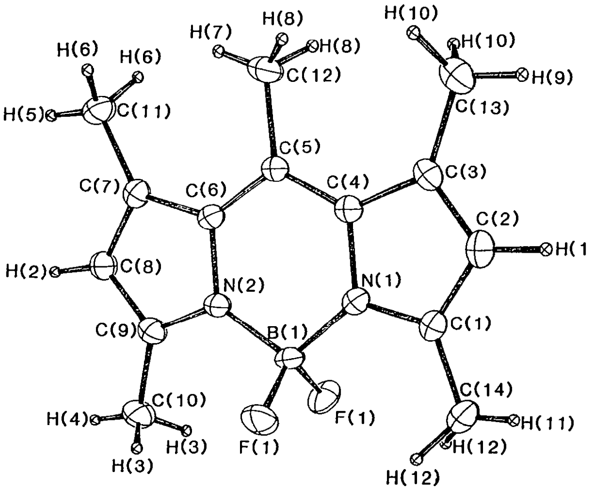

Fig. 1. *ORTEP* plot (Johnson, 1976) of the molecule showing the atomic numbering scheme.

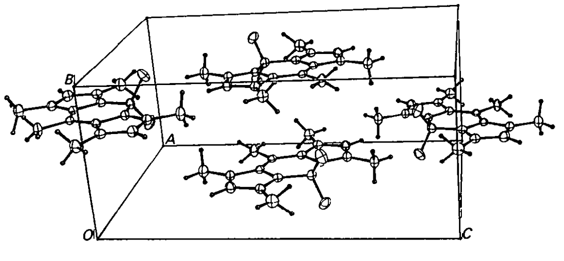

Fig. 2. Contents of the unit cell.

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* Lists of structure factors, anisotropic temperature factors, H-atom positions, and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52592 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1982). Table 1 shows atomic positional parameters and Table 2 the bond lengths and angles. Fig. 1 shows an *ORTEP* plot of the molecule and Fig. 2 shows the contents of the unit cell.

Related literature. Pyrromethene–BF_2 complexes are of interest because of their pronounced fluorescence (Vos de Wael, Pardoen, van Koeveringe & Lugtenburg, 1977). The structures of BF_2 complexes of 1,2,3,4-tetrahydro-1,10-phenanthroline (Klebe, Hensen & Fuess, 1983) and octaethyl-21*H*,24*H*-bilin-1,9-dione (Bonfiglio *et al.*, 1983), which forms a similar *s*-indacene ring system, have been reported.

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1-(1-Chlorovinyl)-2,7-dimethoxynaphthalene

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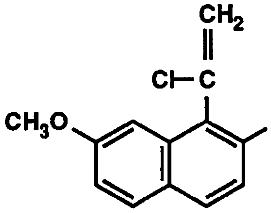
Abstract. $C_{14}H_{13}ClO_2$, $M_r = 248{\cdot}71$, monoclinic, $P2_1/n$, $a = 11{\cdot}291\,(1)$, $b = 7{\cdot}343\,(1)$, $c = 15{\cdot}223\,(2)$ Å, $\beta = 90{\cdot}899\,(8)°$, $V = 1262{\cdot}0\,(5)$ Å^3, $Z = 4$, $D_x = 1{\cdot}309$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1{\cdot}54184$ Å, $\mu = 26{\cdot}0$ cm^{-1}, $F(000) = 520$, $T = 299$ K, $R = 0{\cdot}041$ for 2405 observations (of 2516 unique data). The average deviation from planarity is 0·019 (2) Å with a maximum of 0·035 (1) Å for the fused rings. The dihedral angle between the naphthalene system and the chlorovinyl group is 101·93 (4)°. The methoxy group *ortho* to the chlorovinyl adopts a conformation with the methyl group *anti* to the neighboring α carbon of the ring, with a C—C—O—C torsion angle of −175·6 (2)°. The other methoxy group has the methyl *syn* to the neighboring α carbon, with a C—C—O—C torsion angle of 1·9 (3)°.

Experimental. The title compound (1) was prepared by the reaction of phosphorous trichloride and phosphorous pentachloride on 1-acetyl-2,7-dimethoxynaphthalene in benzene at room temperature (Buckle & Rockewell, 1985). Colorless irregular crystals of (1) were isolated by recrystallization from ether/hexane. Crystal size 0·68 × 0·60 × 0·52 mm, mounted on a glass fiber in random orientation on an Enraf–Nonius CAD-4 κ-axis diffractometer equipped with a graphite monochromator, $\lambda(\text{Cu }K\alpha) = 1{\cdot}54184$ Å. Cell dimensions from setting angles of 25 reflections having $25 < \theta < 29°$. Space group determined to be $P2_1/n$ from systematic absences $h0l$ with $h + 1$ odd, $0k0$ with k odd.



(1)

Data having $2 < 2\theta < 150°$, $0 \le h \le 14$, $0 \le k \le 9$, $-19 \le l \le 19$ were collected using ω–2θ scans designed for $I = 25\sigma(I)$, subject to max. scan time =

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