

Table 2. Selected geometric parameters (Å, °)

C(1)—C(2)	1.386 (5)	C(1)—C(6)	1.390 (5)
C(1)—C(7)	1.493 (6)	C(2)—C(3)	1.383 (5)
C(3)—C(4)	1.384 (5)	C(3)—N(1)	1.464 (4)
C(4)—C(5)	1.382 (4)	C(4)—O(5)	1.384 (4)
C(5)—C(6)	1.371 (5)	C(5)—N(2)	1.481 (5)
N(1)—O(1)	1.217 (5)	N(1)—O(2)	1.212 (5)
N(2)—O(3)	1.206 (5)	N(2)—O(4)	1.210 (4)
O(5)—C(8)	1.398 (4)	C(8)—C(9)	1.385 (5)
C(8)—C(13)	1.372 (5)	C(9)—C(10)	1.366 (5)
C(10)—C(11)	1.391 (5)	C(11)—C(12)	1.390 (6)
C(11)—C(14)	1.484 (5)	C(12)—C(13)	1.393 (5)
C(14)—F(1)	1.304 (4)	C(14)—F(2)	1.327 (5)
C(14)—F(3)	1.324 (6)		
C(2)—C(1)—C(6)	116.9 (3)	C(2)—C(1)—C(7)	121.7 (3)
C(6)—C(1)—C(7)	121.4 (3)	C(1)—C(2)—C(3)	122.0 (3)
C(2)—C(3)—C(4)	121.3 (3)	C(2)—C(3)—N(1)	117.3 (3)
C(4)—C(3)—N(1)	121.4 (3)	C(3)—C(4)—C(5)	116.0 (3)
C(3)—C(4)—O(5)	125.4 (3)	C(5)—C(4)—O(5)	118.5 (3)
C(4)—C(5)—C(6)	123.5 (3)	C(4)—C(5)—N(2)	118.3 (3)
C(6)—C(5)—N(2)	118.2 (3)	C(1)—C(6)—C(5)	120.3 (3)
C(3)—N(1)—O(1)	117.5 (3)	C(3)—N(1)—O(2)	119.1 (3)
O(1)—N(1)—O(2)	123.3 (3)	C(5)—N(2)—O(3)	117.8 (3)
C(5)—N(2)—O(4)	117.3 (3)	O(3)—N(2)—O(4)	124.9 (3)
C(4)—O(5)—C(8)	118.7 (3)	O(5)—C(8)—C(9)	122.6 (3)
O(5)—C(8)—C(13)	116.0 (3)	C(9)—C(8)—C(13)	121.4 (3)
C(8)—C(9)—C(10)	119.9 (4)	C(9)—C(10)—C(11)	120.0 (4)
C(10)—C(11)—C(12)	119.8 (3)	C(10)—C(11)—C(14)	119.9 (4)
C(12)—C(11)—C(14)	120.3 (3)	C(11)—C(12)—C(13)	120.2 (4)
C(8)—C(13)—C(12)	118.8 (4)	C(11)—C(14)—F(1)	114.0 (4)
C(11)—C(14)—F(2)	112.8 (3)	F(1)—C(14)—F(2)	105.3 (4)
C(11)—C(14)—F(3)	113.5 (3)	F(1)—C(14)—F(3)	105.6 (3)
F(2)—C(14)—F(3)	104.8 (4)		
Dihedral angles			
Ring C(1)—C(6)/Ring C(8)—C(13)	80.2		
Ring C(1)—C(6)/N(1),O(1),O(2)	21.5		
Ring C(1)—C(6)/N(2),O(3),O(4)	60.3		
Ring C(1)—C(6)/N(2),O(3),O(4)	76.9		

All calculations were performed on a Data General Desktop Microclipse computer using *SHELXTL* (Sheldrick, 1985). The structure was solved by direct methods and Fourier techniques, and refined by blocked-cascade least-squares refinement (Sparks, 1961). A secondary-extinction correction was applied near the end of the refinement. Molecular graphics were obtained using *ORTEP* (Johnson, 1965).

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: CR1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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N,N-Diethyl-4-(2-nitroethenyl)phenylamine (DEANST), C₁₂H₆N₂O₂

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Abstract

The two independent molecules *A* and *B* in the unit cell of the title compound are nearly planar except for the methyl groups at the ends of the ethyl chains. In molecule *A*, these groups are on opposite sides of the plane. In molecule *B*, one of the ethyl branches is disordered and its methyl group is either on the same (occupancy 60%) or opposite (occupancy 40%) side of the plane as the methyl group of the other branch.

Comment

DEANST, (I), is a third-order non-linear optical material (Kurihara *et al.*, 1991) whose optical properties are being studied in our laboratory. The two independent molecules have nearly the same orientation, the axes along their lengths being approximately parallel to the [111] crystallographic axis. The mean planes through the rings of the two molecules make an angle of 60 (1)°. These structural results are in full agreement with the anisotropy of the third-harmonic susceptibility (Kurihara *et al.*, 1991).

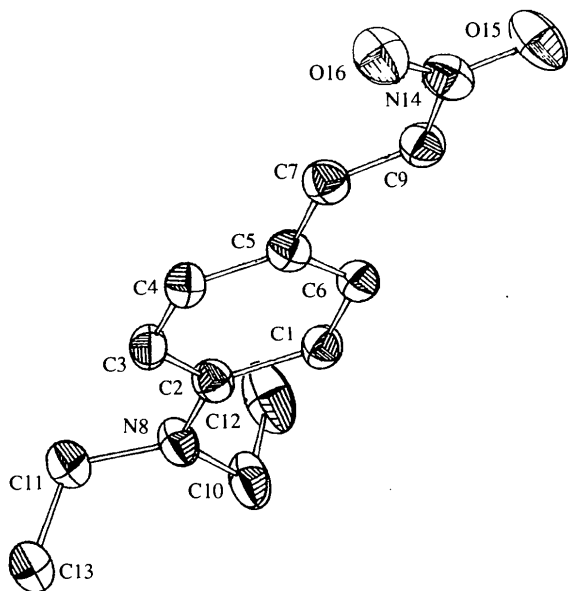
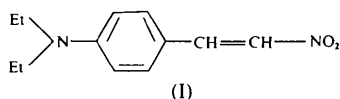


Fig. 1. Perspective view of molecule *A* showing the labelling of the atoms. Displacement ellipsoids are drawn at the 20% probability level.

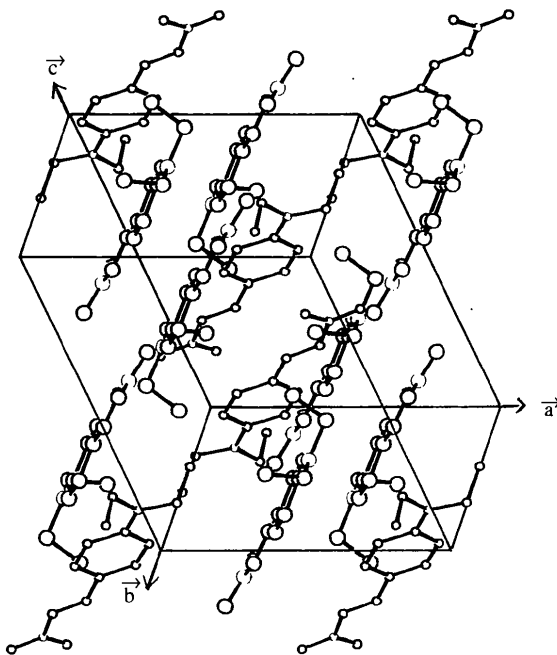


Fig. 2. Projection of the structure on the *ab* plane. Molecule *A* is represented by small circles, molecule *B* by larger circles.

Experimental

Crystal data

$C_{12}H_{16}N_2O_2$
 $M_r = 220$
 Triclinic
 $P\bar{1}$
 $a = 10.430 (7) \text{ \AA}$
 $b = 11.728 (7) \text{ \AA}$
 $c = 12.268 (9) \text{ \AA}$
 $\alpha = 108.31 (5)^\circ$
 $\beta = 98.34 (5)^\circ$
 $\gamma = 115.66 (5)^\circ$
 $V = 1212 (1) \text{ \AA}^3$
 $Z = 4$
 $D_x = 1.20 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation
 $\lambda = 0.7107 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 5\text{--}18^\circ$
 $\mu = 0.0827 \text{ mm}^{-1}$
 $T = 293 \text{ K}$
 Parallelepiped
 $0.3 \times 0.3 \times 0.2 \text{ mm}$
 Orange

Data collection

Nicolet *P3* diffractometer
 ω scans
 Absorption correction:
 none
 4448 measured reflections
 3195 independent reflections
 1589 observed reflections
 $[I > 2.5\sigma(I)]$
 $R_{int} = 0.04$

$\theta_{max} = 22.6^\circ$
 $h = 0 \rightarrow 11$
 $k = -12 \rightarrow 11$
 $l = -13 \rightarrow 12$
 2 standard reflections
 monitored every 100 reflections
 intensity decay: 6%

Refinement

Refinement on F
 $R = 0.089$
 $wR = 0.079$
 $S = 1.2$
 1589 reflections
 289 parameters
 H-atom parameters not refined
 Unit weights applied

$(\Delta/\sigma)_{max} = 0.5$
 $\Delta\rho_{max} = 0.3 \text{ e \AA}^{-3}$
 $\Delta\rho_{min} = -0.2 \text{ e \AA}^{-3}$
 Extinction correction: none
 Atomic scattering factors
 from *International Tables*
 for *X-ray Crystallography*
 (1974, Vol. IV)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (\AA^2)

$$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	U_{eq}
C(1A)	0.381 (1)	0.141 (1)	0.3422 (8)	0.070 (6)
C(2A)	0.229 (1)	0.098 (1)	0.3374 (9)	0.071 (6)
C(3A)	0.208 (1)	0.185 (1)	0.4343 (9)	0.076 (6)
C(4A)	0.326 (1)	0.304 (1)	0.5271 (9)	0.075 (6)
C(5A)	0.474 (1)	0.347 (1)	0.5294 (9)	0.071 (6)
C(6A)	0.500 (1)	0.264 (1)	0.4349 (9)	0.073 (6)
C(7A)	0.597 (1)	0.475 (1)	0.6288 (9)	0.080 (7)
N(8A)	0.1133 (8)	-0.0191 (9)	0.2432 (8)	0.082 (5)
C(10A)	0.138 (1)	-0.112 (1)	0.146 (1)	0.103 (7)
C(11A)	-0.042 (1)	-0.054 (1)	0.2261 (9)	0.090 (7)
C(12A)	0.169 (1)	-0.062 (1)	0.050 (1)	0.130 (9)
C(13A)	-0.122 (1)	-0.148 (1)	0.283 (1)	0.107 (8)
C(9A)	0.742 (1)	0.527 (1)	0.6439 (9)	0.081 (6)
N(14A)	0.854 (1)	0.656 (1)	0.7454 (9)	0.092 (6)
O(15A)	0.9838 (9)	0.7000 (8)	0.7487 (7)	0.128 (5)
O(16A)	0.8145 (9)	0.7182 (8)	0.8214 (8)	0.120 (6)
C(1B)	0.580 (1)	-0.047 (1)	0.1543 (8)	0.074 (6)
C(2B)	0.469 (1)	-0.172 (1)	0.1544 (9)	0.076 (6)
C(3B)	0.471 (1)	-0.176 (1)	0.268 (1)	0.079 (6)

C(4B)	0.578 (1)	-0.065 (1)	0.3746 (9)	0.078 (7)
C(5B)	0.686 (1)	0.058 (1)	0.3743 (9)	0.072 (6)
C(6B)	0.682 (1)	0.063 (1)	0.259 (1)	0.075 (6)
C(7B)	0.797 (1)	0.172 (1)	0.491 (1)	0.089 (7)
N(8B)	0.363 (1)	-0.2833 (9)	0.0471 (8)	0.096 (6)
C(10B)	0.359 (1)	-0.281 (1)	-0.072 (1)	0.099 (7)
C(12B)	0.465 (2)	-0.319 (1)	-0.122 (1)	0.128 (9)
C(9B)	0.908 (1)	0.287 (1)	0.503 (1)	0.100 (8)
N(14B)	1.014 (1)	0.393 (1)	0.624 (1)	0.113 (8)
O(15B)	1.126 (1)	0.493 (1)	0.6241 (8)	0.145 (7)
O(16B)	0.989 (1)	0.380 (1)	0.7152 (9)	0.143 (7)
C(11B)†	0.236 (2)	-0.421 (2)	0.041 (2)	0.130 (9)
C(13B)†	0.311 (3)	-0.503 (2)	0.047 (2)	0.121 (9)
C(21B)‡	0.288 (5)	-0.417 (4)	0.054 (3)	
C(23B)‡	0.123 (5)	-0.476 (4)	0.065 (4)	

† Occupancy 0.6.

‡ Occupancy 0.4.

Table 2. Selected geometric parameters (Å, °)

C(1A)—C(2A)	1.43 (2)	C(1B)—C(2B)	1.42 (1)
C(1A)—C(6A)	1.39 (1)	C(1B)—C(6B)	1.35 (1)
C(2A)—C(3A)	1.42 (2)	C(2B)—C(3B)	1.41 (2)
C(2A)—N(8A)	1.356 (9)	C(2B)—N(8B)	1.38 (1)
C(3A)—C(4A)	1.36 (1)	C(3B)—C(4B)	1.37 (1)
C(4A)—C(5A)	1.39 (2)	C(4B)—C(5B)	1.40 (2)
C(5A)—C(6A)	1.41 (2)	C(5B)—C(6B)	1.42 (2)
C(5A)—C(7A)	1.46 (1)	C(5B)—C(7B)	1.47 (1)
C(7A)—C(9A)	1.31 (2)	C(7B)—C(9B)	1.29 (2)
N(8A)—C(10A)	1.48 (2)	N(8B)—C(10B)	1.47 (2)
N(8A)—C(11A)	1.45 (1)	N(8B)—C(11B)	1.55 (2)
C(9A)—N(14A)	1.44 (1)	N(8B)—C(21B)	1.47 (1)
C(10A)—C(12A)	1.49 (2)	C(9B)—N(14B)	1.47 (1)
C(11A)—C(13A)	1.50 (2)	C(10B)—C(12B)	1.50 (2)
N(14A)—O(15A)	1.21 (1)	C(11B)—C(13B)	1.49 (4)
N(14A)—O(16A)	1.23 (2)	C(11B)—C(21B)	0.52 (2)
		C(21B)—C(23B)	1.59 (2)
		N(14B)—O(15B)	1.25 (2)
		N(14B)—O(16B)	1.22 (2)
C(2A)—C(1A)—C(6A)	121 (1)	C(2B)—C(1B)—C(6B)	121 (1)
C(1A)—C(2A)—C(3A)	116.4 (7)	C(1B)—C(2B)—C(3B)	117.2 (7)
C(1A)—C(2A)—N(8A)	120 (1)	C(1B)—C(2B)—N(8B)	121 (1)
C(3A)—C(2A)—N(8A)	124 (1)	C(3B)—C(2B)—N(8B)	121.8 (9)
C(2A)—C(3A)—C(4A)	122 (1)	C(2B)—C(3B)—C(4B)	122 (1)
C(3A)—C(4A)—C(5A)	121 (1)	C(3B)—C(4B)—C(5B)	121 (1)
C(4A)—C(5A)—C(6A)	118.6 (7)	C(4B)—C(5B)—C(6B)	117.2 (7)
C(4A)—C(5A)—C(7A)	120 (1)	C(4B)—C(5B)—C(7B)	118 (1)
C(6A)—C(5A)—C(7A)	122 (1)	C(6B)—C(5B)—C(7B)	124.7 (9)
C(1A)—C(6A)—C(5A)	121 (1)	C(1B)—C(6B)—C(5B)	122 (1)
C(5A)—C(7A)—C(9A)	126 (1)	C(5B)—C(7B)—C(9B)	124 (1)
C(2A)—N(8A)—C(10A)	122.5 (9)	C(2B)—N(8B)—C(10B)	122.4 (9)
C(2A)—N(8A)—C(11A)	121 (1)	C(2B)—N(8B)—C(11B)	123 (1)
C(9A)—N(14A)—O(15A)	117 (1)	C(2B)—N(8B)—C(21B)	114.9 (9)
C(9A)—N(14A)—O(16A)	120 (1)	C(9B)—N(14B)—O(15B)	114 (1)
C(10A)—N(8A)—C(11A)	115.9 (7)	C(9B)—N(14B)—O(16B)	121 (1)
N(8A)—C(10A)—C(12A)	112 (1)	C(10B)—N(8B)—C(11B)	114.1 (9)
N(8A)—C(11A)—C(13A)	114 (1)	C(10B)—N(8B)—C(21B)	119.6 (6)
C(7A)—C(9A)—N(14A)	121 (1)	N(8B)—C(10B)—C(12B)	113 (1)
O(15A)—N(14A)—O(16A)	123.4 (8)	N(8B)—C(11B)—C(13B)	104 (2)
		N(8B)—C(21B)—C(23B)	119 (4)
		C(7B)—C(9B)—N(14B)	120 (1)
		O(15B)—N(14B)—O(16B)	125.2 (9)

In the disordered branch, the pairs [C(11B), C(13B)] and [C(21B), C(23B)] were refined alternatively because of correlations. Isotropic displacement parameters for [C(21B), C(23B)] were not refined.

The structure was solved using *PATSEE* (*Xtal*; Hall & Stewart, 1990).

We thank G. Commandeur for the synthesis of the title compound.

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: PA1096). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1995). **C51**, 434–436

9-(4-Methyl-1-piperazinyl)-9,10-dihydro-9,10-ethanoanthracene Hydrochloride Dihydrate (Trazitilin)

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Abstract

The title compound, trazitilin, C₂₁H₂₄N₂.HCl.2H₂O, is an antagonist of histamine H1 receptors. Steric hindrance of the two components of the molecule (the piperazinyl radical and 9,10-dihydro-9,10-ethanoanthracene) causes a slight distortion of substituents and allows the structure very little flexibility.

Comment

As part of our conformational studies on antihistamine drugs, we investigated the X-ray structure of trazitilin. This compound as a whole is quite rigid and can be used therefore as a basis for the modeling of histamine H1 receptors (Brouant & Barbe, 1992).