

Related literature. The title compound was prepared from (+)-retronecine and 3,3-dimethylglutaric anhydride (Devlin, Robins & Sakdarat, 1982). Because the absolute configuration of (+)-retronecine is known (Bull, Culvenor & Dick, 1968; Warren, 1966), Fig. 1 and the coordinates (Table 1) correspond to the absolute configuration of the title compound. The geometry and conformation of the molecule (Table 3) are very close to those observed in the first pyrrolizidine alkaloid analogue studied, 1,2-didehydrocrotal-anine picrate (Stoekli-Evans & Robins, 1983). The latter was prepared from (+)-retronecine and glutaric anhydride (Devlin, Robins & Sakdarat, 1982). The ¹H NMR chemical shift difference [$\Delta\delta$] of the C(9) H atoms is quite different for the two compounds despite the same relative orientation of the C(9) H atoms with respect to bonds C(1)=C(2) and O(10)–C(11); see Table 3.

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trans-Decafluoroazobenzene–*trans*-Stilbene (1/1)

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Abstract. C₁₂F₁₀N₂·C₁₄H₁₂, $M_r = 542.4$, triclinic, $P\bar{1}$, $a = 6.167$ (1), $b = 7.365$ (1), $c = 12.394$ (2) Å, $\alpha = 98.39$ (1), $\beta = 92.01$ (1), $\gamma = 94.52$ (2)°, $V = 555$ (1) Å³, $D_m = 1.63$ (1), $D_x = 1.623$ Mg m⁻³, $Z = 1$, $\lambda(\text{Mo } K\alpha) = 0.7107$ Å, $\mu = 0.107$ mm⁻¹, $F(000) = 272$, $T = 293$ (2) K, $R = 0.040$ for 1041 reflections with $I \geq 2.5\sigma(I)$. The crystal structure of the title compound is comprised of alternate (almost parallel, 3.7°) layers of decafluoroazobenzene molecules and stilbene molecules with the individual molecules situated about crystallographic centres of inversion. The molecules are stacked along the b axis, forming columns, but are not eclipsed; the average interlayer spacing is 3.55 Å. The shortest F...H contact in the lattice is an inter-column contact between F(4) and H(7') (1+x, y, z–1) of 2.70 Å. Within the layers of decafluoroazobenzene, the closest intermolecular contact occurs between F(2) and F(2') (–x, –1–y, –z) of 3.04 Å; similarly, H(4)···H(6') (1+x, y, z) contacts of 2.95 Å are found within the layers of stilbene molecules.

Experimental. Orange crystals of the 1/1 molecular complex between decafluoroazobenzene and stilbene (*trans*-1,2-diphenylethene), m.p. 428–430 K, were originally obtained as the only isolable product from a reaction between the azo compound and Pt(η^2 -CHPh=CHPh)(PET₃)₂. The title compound was subsequently prepared directly from the two components in CCl₄ solution. Analysis: found C, 57.2; H, 2.2; N, 5.5; C₂₆H₁₂F₁₀N₂ calc. C, 57.6; H, 2.2; N, 5.2%. Crystals suitable for the X-ray analysis were obtained from the slow evaporation of a CCl₄ solution. Density measured in aq. ZnBr₂ solution. Enraf–Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; $\omega:2\theta$ scan technique. Cell parameters, on a needle 0.138 × 0.475 × 0.025 mm, from least-squares procedure on 25 reflections ($8 \leq \theta \leq 13^\circ$). Total of 4570 reflections ($1 \leq \theta \leq 27.5^\circ$) measured in the range $-8 \leq h \leq 8$, $-9 \leq k \leq 9$, $-16 \leq l \leq 9$. A decrease of approximately 8% was noted in the net intensities of

Table 1. Fractional atomic coordinates and $B_{eq}(\text{\AA}^2)$ values
$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq}
F(1)	-0.2149 (3)	-0.5483 (3)	-0.3150 (1)	4.85
F(2)	-0.1000 (3)	-0.4553 (3)	-0.1058 (2)	5.96
F(3)	0.2992 (3)	-0.2860 (3)	-0.0422 (2)	6.45
F(4)	0.5851 (3)	-0.2070 (3)	-0.1924 (2)	5.53
F(5)	0.4727 (3)	-0.2944 (3)	-0.4061 (2)	5.19
N(1)	0.0918 (4)	-0.4686 (4)	-0.4840 (2)	4.17
C(1)	0.0914 (5)	0.0384 (5)	0.5251 (3)	4.34
C(2)	0.1378 (5)	0.0822 (4)	0.6435 (3)	4.08
C(3)	0.3448 (6)	0.1602 (5)	0.6834 (3)	4.58
C(4)	0.3963 (6)	0.2015 (5)	0.7932 (3)	5.08
C(5)	0.2431 (7)	0.1669 (5)	0.8672 (4)	5.43
C(6)	0.0363 (6)	0.0908 (5)	0.8310 (3)	5.26
C(7)	-0.0136 (6)	0.0497 (5)	0.7208 (3)	4.60
C(8)	0.1262 (5)	-0.4249 (4)	-0.3692 (2)	3.43
C(9)	-0.0173 (5)	-0.4636 (4)	-0.2898 (3)	3.68
C(10)	0.0408 (5)	-0.4166 (5)	-0.1805 (3)	4.29
C(11)	0.2433 (5)	-0.3311 (5)	-0.1481 (3)	4.39
C(12)	0.3871 (5)	-0.2915 (4)	-0.2245 (3)	4.17
C(13)	0.3299 (5)	-0.3364 (4)	-0.3333 (3)	3.86
H(1)	0.2001 (61)	0.0755 (49)	0.4819 (29)	6.45
H(3)	0.4510 (65)	0.1780 (51)	0.6358 (30)	5.99
H(4)	0.5408 (66)	0.2515 (51)	0.8168 (31)	6.75
H(5)	0.2840 (58)	0.1906 (49)	0.9433 (33)	6.10
H(6)	-0.0769 (71)	0.0678 (56)	0.8817 (34)	8.40
H(7)	-0.1469 (55)	0.0023 (45)	0.6990 (25)	4.64

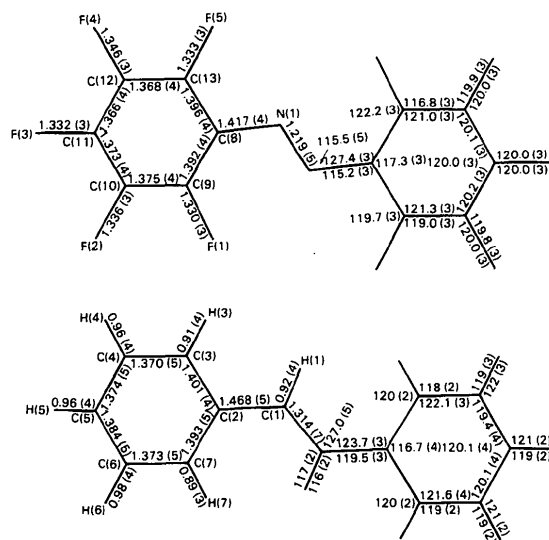
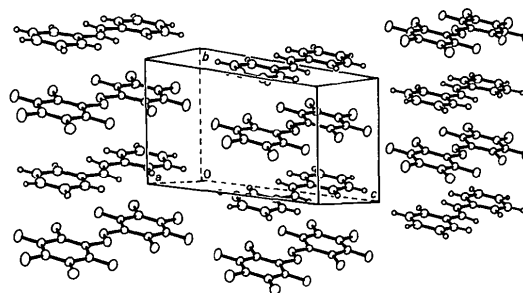
Fig. 1. Interatomic bond distances (\AA) and angles ($^\circ$) showing the numbering scheme employed. Note that both molecules are situated about crystallographic sites of symmetry, $\bar{1}$.

Fig. 2. The unit-cell contents for the molecular complex (Johnson, 1971).

three standards (025, 221, 302) monitored every 3600 s; data corrected for intensity variation but not for absorption. 2541 unique reflections ($R_{int} = 0.039$), 1041 satisfied $I \geq 2.5\sigma(I)$. Structure solved with the *MITHRIL* direct-methods routine (Gilmore, 1984), full-matrix least-squares refinement of 196 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms and H atoms refined isotropically. At convergence $R = 0.040$, $wR = 0.043$, $w = 6.15/[\sigma^2(F) + 0.0019F^2]$, $S = 2.2$, $(\Delta/\sigma)_{max} \leq 0.004$, $(\Delta\rho)_{max} = 0.16$, $(\Delta\rho)_{min} = -0.29 \text{ e \AA}^{-3}$; no extinction correction. Refinement performed with *SHELX76* (Sheldrick, 1976) on the University of Adelaide's VAX11/780 computer system. Atomic parameters are given in Table 1, bond distances and angles are shown, with the numbering scheme used, in Fig. 1 and a projection of the unit-cell contents is shown in Fig. 2.*

Related literature. The molecular complex is a conventional electron donor(stilbene)-acceptor(azobenzene) complex, and the structure resembles the stacking found in several polyfluoroaromatic/aromatic molecular complexes (Potenza & Mastropaolo, 1975; Dahl, 1981). The interlayer spacing of 3.55 \AA found in the title compound indicates that little charge transfer

* Lists of structure factors, thermal parameters, and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43971 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

occurs. Structural studies on the closely related *trans*-azobenzene/*trans*-stilbene system, $(C_{14}H_{12})_x(C_{12}H_{10}N_2)_{[1-x]}$ (for $x = 0.26, 0.56$ and 0.46), have been made (Bouwstra, Schouten, Kroon & Helmholdt, 1985); in contrast to their study, no evidence for configurational or substitutional disorder was observed in the present investigation.

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