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N—CF_3 Bonds in a Carbamate and a Triazene

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Abstract. *tert*-Butyl *N*-trifluoromethylcarbamate, $C_6H_{10}F_3NO_2$, $M_r = 185{\cdot}146$, $Pbca$, $a = 11{\cdot}801$ (2), $b = 17{\cdot}030$ (4), $c = 9{\cdot}262$ (1) Å, $V = 1861{\cdot}3$ (6) Å^3, $Z = 8$, $D_x = 1{\cdot}321$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}13$ mm^{-1}, $F(000) = 768$, 296 K, $R = 0{\cdot}059$, 878 observed reflections. The carbamate possesses an unusually short N—CF_3 bond, 1·361 (6) Å, and a somewhat lengthened N—$C(O_2)$ bond, 1·369 (5) Å. The CNC(=O)OC section of the molecule is planar with the CC_3 and CF_3 groups staggered with respect to the C—O bond. Hydrogen bonding links the molecules into infinite chains. 3-Phenyl-1-trifluoromethyltriazene, $C_7H_6F_3N_3$, $M_r = 189{\cdot}143$, $P2_1/n$, $a = 9{\cdot}792$ (2), $b = 5{\cdot}240$ (1), $c = 16{\cdot}328$ (5) Å, $\beta = 95{\cdot}44$ (2)°, $V = 834{\cdot}0$ (6) Å^3, $Z = 4$, $D_x = 1{\cdot}506$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}13$ mm^{-1}, $F(000) = 384$, 295 K, $R = 0{\cdot}052$, 1172 observed reflections. A short N—CF_3 bond, 1·409 (3) Å, is found in the triazene, which exists as centrosymmetric dimers held together by weak hydrogen bonds. The CNNNC part of the molecule is planar and forms an angle of 160° with the plane of the phenyl ring. The CF_3 group is disordered.

Introduction. The N—CF_3 bond lengths in $N(CF_3)_{3-n}F_n$ ($n = 0,1,2$) compounds are known to increase as n increases (Oberhammer, Günther, Bürger, Heyder & Pawelke, 1982). This substitution effect has been attributed to a polar interaction between the net charges on the C and N atoms (Oberhammer, 1983). Since the N atom should be more negatively charged in $CF_3NHCO_2C(CH_3)_3$ (1) than in $N(CF_3)_3$ (2), we have studied the structure of (1) to see if its N—CF_3 bond is indeed shorter than that of (2).

The structure of $CF_3NNNH(C_6H_5)$ (3), which was previously formulated as $CF_3NHNNC_6H_5$ (Makarov, Yakubovich, Filatov, Englin & Nikiforova, 1968), is also reported. Comparison of the N—CF_3 bond in (3) with that of *trans*-CF_3NNCF_3 (4) (Bürger, Pawelke & Oberhammer, 1982) offers an additional test of the polar interaction model.

Experimental. Crystals, which were sealed in glass capillaries, were grown by sublimation from material prepared according to Lutz & Sundermeyer (1979) for (1) and Makarov *et al.* (1968) for (3). Crystal sizes are 0·30 × 0·35 × 0·61 mm for (1) and 0·24 × 0·28 × 0·78 mm for (3). Siemens AED 1 diffractometer employing Zr-filtered Mo $K\alpha$ radiation. The widths in ω of the ω–2θ step scans were $\Delta + 0{\cdot}34° \tan\theta$, with $\Delta = 1{\cdot}30°$ for (1) and 0·90° for (3), and 0·61 s per 0·02° step in ω. Peak intensity from middle two-thirds of scan range with one remeasurement if $2\sigma(I) < I < 25\sigma(I)$. Cell constants from 52 reflections, $6{\cdot}3 < \theta < 12{\cdot}3°$, for (1) and 39 reflections, $8{\cdot}2 < \theta < 13{\cdot}7°$, for (3). Numerical absorption corrections vary from 1·051 to 1·070 for (1) and 1·022 to 1·044 for (3). Max. $(\sin\theta)/\lambda = 0{\cdot}594$ Å^{-1}. One octant for (1) with $0 \le h \le 14$, $0 \le k \le 20$, $0 \le l \le 11$; four octants for (3) with $-12 \le h \le 12$, $0 \le k \le 6$, $-19 \le l \le 19$. Standards remeasured hourly and their fluctuations are $00\bar{6}$ (±2·4%), 220 (±8·1%), $\bar{2}20$ (±4·7%) for (1) and $00\bar{8}$ (±3·8%), 600 (±1·7%), 230 (±3·1%) for (3). 1889 reflections measured for (1), 3446 for (3). 1624 unique for (1) and 1462 for (3) ($R_{\rm int} = 0{\cdot}03$). Unobserved $[F_o < 4\sigma(F_o)]$ were 746 and 290 for (1) and (3) respectively. Structures were solved by multisolution direct methods and refined on F. Disorder of F atoms in (3) treated with F(i) anisotropic (occupancy α) and F(iA) isotropic (occupancy 1—α), $\alpha = 0{\cdot}855$ (5). All H atoms located in ΔF syntheses – 0·25 to 0·33 e Å^{-3} for (1) and 0·40 to 0·50 e Å^{-3} for (3). H(N) and H(N3) were refined isotropically. Phenyl and methyl H atoms

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Table 1. *Atom coordinates and equivalent isotropic temperature factors* (Å²) *for* $CF_3NHCO_2C(CH_3)_3$

$U_{eq} = \frac{1}{3}\sum_i U_{ii}$.

	x	y	z	U/U_{eq}
F(1)	0·3330 (3)	0·3660 (2)	0·1070 (3)	0·134 (2)
F(2)	0·2776 (3)	0·3935 (2)	0·3171 (3)	0·139 (2)
F(3)	0·1964 (3)	0·3052 (2)	0·1925 (5)	0·164 (2)
O(1)	0·3856 (3)	0·2020 (2)	0·0928 (3)	0·092 (1)
O(2)	0·4747 (2)	0·1769 (2)	0·3028 (2)	0·075 (1)
N	0·3674 (3)	0·2816 (2)	0·2874 (4)	0·082 (1)
C(1)	0·4083 (3)	0·2173 (2)	0·2158 (4)	0·069 (1)
C(2)	0·2962 (5)	0·3349 (3)	0·2260 (5)	0·093 (2)
C(3)	0·5305 (4)	0·1026 (2)	0·2577 (4)	0·079 (1)
C(4)	0·6059 (4)	0·1170 (4)	0·1292 (5)	0·116 (2)
C(5)	0·4393 (4)	0·0431 (3)	0·2294 (6)	0·110 (2)
C(6)	0·5988 (4)	0·0825 (3)	0·3895 (5)	0·105 (2)
H(N)	0·385 (4)	0·280 (3)	0·373 (6)	0·11 (2)

Table 2. *Atom coordinates and equivalent isotropic temperature factors* (Å²) *for* $CF_3NNNH(C_6H_5)$

$U_{eq} = \frac{1}{3}\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U/U_{eq}
F(1)	0·1509 (4)	−0·3556 (6)	−0·1563 (2)	0·134 (1)
F(1*A*)	0·274 (1)	0·004 (2)	−0·1668 (7)	0·052 (3)
F(2)	0·3290 (2)	−0·1530 (8)	−0·1255 (2)	0·142 (2)
F(2*A*)	0·106 (1)	−0·185 (3)	−0·1914 (7)	0·059 (4)
F(3)	0·1741 (5)	0·0115 (7)	−0·2011 (1)	0·159 (2)
F(3*A*)	0·265 (2)	−0·333 (3)	−0·1139 (7)	0·074 (4)
N(1)	0·1310 (2)	−0·0361 (4)	−0·0694 (1)	0·0540 (7)
N(2)	0·2050 (2)	0·1397 (4)	−0·0335 (1)	0·0528 (6)
N(3)	0·1497 (2)	0·2491 (4)	0·0271 (1)	0·0546 (7)
C(1)	0·1973 (3)	−0·1308 (5)	−0·1361 (2)	0·065 (1)
C(2)	0·2227 (2)	0·4398 (4)	0·0744 (1)	0·0495 (7)
C(3)	0·1512 (2)	0·6012 (5)	0·1212 (1)	0·0586 (9)
C(4)	0·2198 (3)	0·7895 (5)	0·1676 (1)	0·066 (1)
C(5)	0·3596 (3)	0·8165 (5)	0·1666 (2)	0·071 (1)
C(6)	0·4299 (3)	0·6536 (5)	0·1202 (2)	0·075 (1)
C(7)	0·3631 (2)	0·4626 (5)	0·0746 (1)	0·0662 (9)
H(N3)	0·063 (3)	0·200 (5)	0·040 (1)	0·081 (8)

were idealized (C—H 0·95 Å), allowed to ride on their C atoms with one U per C atom. For (1), the 116 parameters converged with $R = 0{\cdot}059$, $wR = 0{\cdot}081$, $S = 1{\cdot}97$, $w = [\sigma^2(F_o) + 0{\cdot}0009F_o^2]^{-1}$, $|\Delta/\sigma|_{\max} = 0{\cdot}02$, $(\Delta\rho)_{\max} = 0{\cdot}21$, $(\Delta\rho)_{\min} = -0{\cdot}24$ e Å^{-3}. Coordinates of nonidealized atoms in Table 1 with numbering according to Fig. 1(*a*). For (3), the 140 parameters converged with $R = 0{\cdot}052$, $wR = 0{\cdot}053$, $S = 4{\cdot}64$, $w = 1/\sigma^2(F_o)$, $|\Delta/\sigma|_{\max} = 0{\cdot}01$, $(\Delta\rho)_{\max} = 0{\cdot}24$, $(\Delta\rho)_{\min} = -0{\cdot}26$ e Å^{-3} – the largest features in final ΔF map near F atoms and no credible peak above background (0·11 e Å^{-3}) near N(1). Coordinates of nonidealized atoms in Table 2 with numbering according to Fig. 1(*b*).* Dispersion-corrected Hartree–Fock scattering factors (*International Tables for X-ray Crystallography*, 1974) used for all atoms except H, where the values of Stewart, Davidson & Simpson (1965) were used. For XCY_3 groups the tilt angle is the angle between the C—X vector and the normal to the Y_3 plane. Computer programs include *SHELX*76 (Sheldrick, 1976) and *ORTEP*II (Johnson, 1976).

Discussion. Selected distances and angles are given in Table 3 for (1). The molecular plane of the carbamate is defined by the atoms of the O(1), O(2), N, C(1), C(2), C(3) fragment, which are coplanar to within ±0·01 Å. The atoms C(6) and F(2) deviate from this plane by only 0·068 (5) and 0·090 (4) Å, respectively; therefore, both the CC_3 and CF_3 entities are essentially staggered with respect to the C(1)–O(1) bond. The CC_3 fragment is tilted so as to lengthen the O(1)···C(4) and O(1)···C(5) contacts, the tilt angle being 5·1 (5)°. Similarly, the somewhat smaller tilt of the CF_3 group [2·7 (6)°] lengthens the O(1)···F(1) and O(1)···F(3) contacts.

The N—CF_3 bond in (1) is 0·065 (8) Å shorter than that of (2) [1·426 (2) Å; Bürger, Niepel, Pawelke & Oberhammer (1979)]; indeed, it appears to be the shortest such bond studied to date.

A short N—C(2) bond in (1) might be accompanied by relatively long N—C(1) and N—H(N) distances compared to those of RNHCO$_2$C(CH$_3$)$_3$ compounds in which a nonfluorinated residue R is linked to the N atom by an sp^3-hybridized C atom [C(sp^3)]. (Unfortunately, the structure with $R = CH_3$ has apparently not been reported.) In the Cambridge Crystallographic File (September, 1986), 14 such structures were found

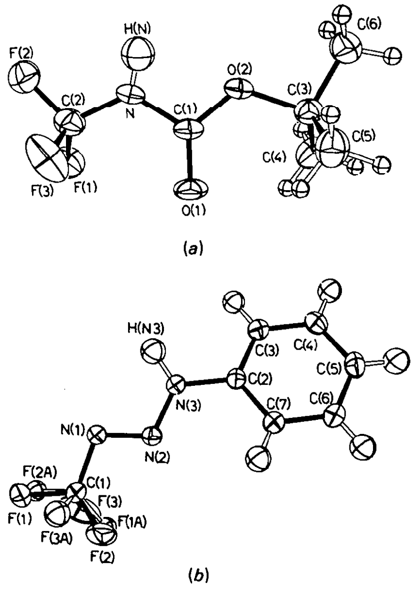

Fig. 1. (*a*) A perspective drawing of $CF_3NHCO_2C(CH_3)_3$ with 20% probability thermal ellipsoids except for the methyl H atoms. (*b*) A perspective drawing of $CF_3NNNH(C_6H_5)$ with 20% probability thermal ellipsoids.

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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 44506 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Selected distances* (Å) *and angles* (°) *in* $CF_3NHCO_2C(CH_3)_3$

C(1)–N	1·369 (5)	C(3)–C(4)	1·506 (6)
C(1)–O(1)	1·199 (4)	C(3)–C(5)	1·501 (6)
C(1)–O(2)	1·318 (5)	C(3)–C(6)	1·502 (6)
C(2)–F(1)	1·297 (5)	C(3)–O(2)	1·487 (5)
C(2)–F(2)	1·325 (6)	N–H(N)	0·82 (6)
C(2)–F(3)	1·318 (6)	N–O(1^i)	2·850 (4)
C(2)–N	1·361 (6)	H–O(1^i)	2·06 (6)

N–C(1)–O(1)	123·7 (4)	C(5)–C(3)–C(6)	111·9 (4)
N–C(1)–O(2)	109·3 (3)	O(2)–C(3)–C(4)	110·1 (4)
O(1)–C(1)–O(2)	127·0 (4)	O(2)–C(3)–C(5)	107·8 (4)
F(1)–C(2)–F(2)	106·8 (4)	O(2)–C(3)–C(6)	101·7 (3)
F(1)–C(2)–F(3)	104·8 (5)	C(1)–N–C(2)	123·3 (4)
F(2)–C(2)–F(3)	106·9 (5)	C(1)–N–H(N)	110 (4)
N–C(2)–F(1)	114·9 (5)	C(2)–N–H(N)	126 (4)
N–C(2)–F(2)	109·8 (4)	C(1)–O(2)–C(3)	122·4 (3)
N–C(2)–F(3)	113·2 (4)	N–H(N)–O(1^i)	162 (4)
C(4)–C(3)–C(5)	113·3 (4)	C(1^i)–O(1^i)–H(N)	155 (4)
C(4)–C(3)–C(6)	111·3 (4)		

Symmetry code: (i) x, $0·5 - y$, $0·5 + z$.

Table 4. *Selected distances* (Å) *and angles* (°) *in* $CF_3NNNH(C_6H_5)$

C(1)–N(1)	1·409 (3)	C(1)–F(1)	1·295 (3)
N(1)–N(2)	1·279 (2)	C(1)–F(2)	1·290 (3)
N(2)–N(3)	1·306 (2)	C(1)–F(3)	1·299 (4)
N(3)–C(2)	1·414 (3)	C(1)–F(1*A*)	1·18 (1)
C(2)–C(3)	1·375 (3)	C(1)–F(2*A*)	1·24 (1)
C(3)–C(4)	1·380 (3)	C(1)–F(3*A*)	1·28 (1)
C(4)–C(5)	1·378 (4)	N(3)–H(N3)	0·94 (3)
C(5)–C(6)	1·369 (4)	N(3)–N(1^i)	3·104 (3)
C(6)–C(7)	1·376 (3)	N(1)–H($N3^i$)	2·17 (3)
C(2)–C(7)	1·380 (3)		

N(1)–C(1)–F(1)	110·0 (2)	N(2)–N(1)–H($N3^i$)	131 (2)
N(1)–C(1)–F(2*A*)	107·0 (5)	N(1)–N(2)–N(3)	114·0 (2)
N(1)–C(1)–F(2)	117·1 (2)	N(2)–N(3)–C(2)	119·9 (2)
N(1)–C(1)–F(1*A*)	118·6 (5)	N(2)–N(3)–H(N3)	120 (2)
N(1)–C(1)–F(3)	111·8 (2)	C(2)–N(3)–H(N3)	120 (2)
N(1)–C(1)–F(3*A*)	109·6 (5)	N(3)–C(2)–C(3)	118·7 (2)
F(1)–C(1)–F(2)	105·9 (3)	N(3)–C(2)–C(7)	121·0 (2)
F(1*A*)–C(1)–F(2*A*)	105·7 (8)	C(3)–C(2)–C(7)	120·3 (2)
F(1)–C(1)–F(3)	106·5 (3)	C(2)–C(3)–C(4)	119·9 (2)
F(2*A*)–C(1)–F(3*A*)	109·4 (8)	C(3)–C(4)–C(5)	120·0 (3)
F(2)–C(1)–F(3)	104·9 (3)	C(4)–C(5)–C(6)	119·6 (3)
F(1*A*)–C(1)–F(3*A*)	106·3 (8)	C(5)–C(6)–C(7)	121·0 (3)
C(1)–N(1)–N(2)	109·0 (2)	C(2)–C(7)–C(6)	119·1 (2)
C(1)–N(1)–H($N3^i$)	120 (2)	N(3)–H(N3)–N(1^i)	173 (2)

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

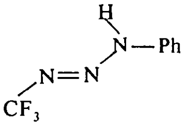
to have the same conformation as (1) and σ(C—C) $\leq 0·007$ Å, and average values were calculated for their N—C(sp^3) and N—C(O_2) distances. While the N—C(2) bond in (1) is much shorter [0·085 (11) Å] than the mean N—C(sp^3) distance [1·446 (9) Å], the N—C(1) bond in (1) is only marginally longer than the mean N—C(O_2) bond length [1·347 (10) Å]; therefore fluorination mainly affects the N—C(sp^3) bond. Small changes in the N—H(N) bond length cannot be detected by this study, and that a long N—H(N) bond should be associated with a strong N—H(N)···O(1^i) hydrogen bond is a matter of conjecture. While the latter interaction in (1) is strong, a comparable interaction has been reported in an *R*$NHCO_2C(CH_3)_3$ derivative (Capasso, Mattia, Mazzarella & Zagari, 1984).

In (1) the angle formed by the two C—F bonds straddling the C(1)—O(1) bond is 2·0 (7)° smaller than the mean of the other two F—C—F angles. The same type and degree [1·5 (5)°] of distortion was recently reported for a CF_3 group attached to a trigonally hybridized N atom in a 2-imidazolidinone derivate (Pawelke, Bürger, Brauer & Wilke, 1987). While these distortions are small, they are relevant to theoretical studies of the geometry of F_3CNX_2 species (Magnusson, 1986). These distortions may result from repulsive interactions between the N-atom lone pair and the C(2)—F(1) and C(2)—F(3) bonding electrons.

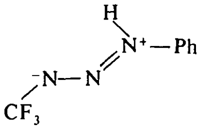
Selected distances and angles for (3) are given in Table 4. This compound contains a *W*-shaped C(1), N(1), N(2), N(3), C(2) fragment, which is planar to within 0·026 (3) Å. The normal to this plane forms a dihedral angle of 21·0 (4)° with the plane normal of the phenyl ring. As shown by the respective values of 38·4 (4) and −26 (1)° for the F(2)—C(1)—N(1)—N(2) and F(1*A*)—C(1)—N(1)—N(2) torsion angles, the disordered CF_3 groups assume asymmetrical orientations with respect to the N(1)—N(2) bond. For comparison, the eclipsed conformation was found by gas-phase electron diffraction for (4) (Bürger *et al.*, 1982). Both the higher- and lower-occupancy CF_3 groups are so tilted with respect to the C(1)—N(1) bond [4·2 (4) and 6 (1)° respectively] that the shortest F···N(2) contacts, F(2)···N(2) and F(1*A*)···N(2), are lengthened. A similar tilt was reported for (4), 4·9 (3)°.

No structure of a triazene with a saturated group on N(1) and an unsaturated group on the N(3) atom has been reported previously. Proof of the $CF_3NNNH(C_6H_5)$ formulation for (3) is given by the location and refinement of H(N3), but the weak N(3)—H(N3)···N(1^i) hydrogen bond shows how closely (3) is related to $CF_3NHNNC_6H_5$.

That the N(1)—N(2) bond is 0·027 (3) Å shorter than the N(2)—N(3) distance also confirms the proposed structure. Such small differences in N—N bond lengths are typical of triazenes (Randall, Schwalbe & Vaughan, 1984) and have been explained by resonance between valence structures analogous to (3*a*) and (3*b*) (Edwards, Sherfinski & Marsh, 1974). The importance of (3*b*) is also indicated by the N(1)—N(2) bond in (3) being 0·044 (4) Å longer than the N=N distance in (4) [1·235 (3) Å].



(3*a*)



(3*b*)

The N—CF_3 bond in (3) is 0·051 (4) Å shorter than that in (4), 1·460 (2) Å. Since the resonance structure

(3*b*) enhances the electron density on the N(1) atom with respect to that of an N atom in (4), the trend in these N—CF_3 bond lengths appears to support the polar interaction model.

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Acta Cryst. (1988). C**44**, 528–531

Acide Décahydro-1,2,3,4,5,6,7,8,9,10 Diméthyl-1,4a (Méthylène-1)éthano-7,8a Phénanthrènecarboxylique-1, Acide (—)-Kaurène-16 Oïque-19

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Abstract. Isolated from frond exudate of ferns of the genus *Notholaena* (*N. peninsularis* and *N. pallens*) collected in Mexico, $C_{20}H_{30}O_2$, $M_r = 302{\cdot}5$, orthorhombic, $P2_12_12_1$, $a = 24{\cdot}073$ (8), $b = 23{\cdot}781$ (8), $c = 12{\cdot}287$ (5) Å, $Z = 16$, $D_x = 1{\cdot}142$ (2) g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu = 10{\cdot}5$ cm^{-1}, $F(000) = 2656$, $T = 293$ K, $R = 0{\cdot}055$ for 5511 unweighted reflections. The four independent molecules have the (—)-kaurene conformation. The structure contains two dimers, each formed by two molecules which are not equivalent and bound by two hydrogen bonds between carboxylic groups.

Introduction. Un diterpène a été isolé récemment comme principal constituant de l'exudat de deux espèces de fougères tropicales originaires du Mexique, du genre *Notholaena* (*N. peninsularis* et *N. pallens*). Une étude spectroscopique (Wollenweber, Marx, Favre-Bonvin & Brassy, 1983) et une étude radiocristallographique ont été menées simultanément pour déterminer la nature et la conformation de ce diterpène décelé pour la première fois dans une fougère et dont la formule est: