C(41)	0.4084 (7)	0.1043 (2)	−0.1802 (2)	0.064 (2)
C(51)	0.1639 (7)	0.2116 (2)	−0.1918 (2)	0.060 (2)
C(52)	0.1524 (7)	0.2768 (2)	−0.1977 (2)	0.069 (2)
O(53)	0.0652 (4)	0.3017 (1)	−0.1433 (2)	0.069 (1)
S(11)	1.2310 (2)	0.2715 (1)	0.1148 (1)	0.073 (1)
C(11)	1.1388 (6)	0.2079 (2)	0.1220 (2)	0.066 (2)
N(11)	1.0801 (6)	0.1638 (2)	0.1270 (2)	0.092 (2)
S(12)	−0.0936 (2)	0.0538 (1)	−0.1516 (1)	0.082 (1)
C(12)	0.0439 (6)	0.0505 (2)	−0.0918 (2)	0.059 (2)
N(12)	0.1406 (6)	0.0477 (2)	−0.0512 (2)	0.101 (2)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and hydrogen-bond geometry* (Å, °)

N(1′)—C(2′)	1.354 (6)	N(3)—C(4)	1.388 (5)
C(2′)—N(3′)	1.303 (6)	C(4)—C(5)	1.345 (6)
N(3′)—C(4′)	1.361 (5)	C(5)—S(1)	1.731 (4)
C(4′)—C(5′)	1.423 (6)	C(4)—C(41)	1.493 (6)
C(5′)—C(6′)	1.351 (7)	C(5)—C(51)	1.505 (6)
C(6′)—N(1′)	1.347 (6)	C(51)—C(52)	1.513 (7)
C(2′)—C(21′)	1.485 (7)	C(52)—O(53)	1.420 (6)
C(4′)—N(41′)	1.325 (6)	S(11)—C(11)	1.651 (6)
C(5′)—C(35′)	1.496 (6)	C(11)—N(11)	1.128 (7)
C(35′)—N(3)	1.479 (6)	S(12)—C(12)	1.635 (5)
S(1)—C(2)	1.675 (5)	C(12)—N(12)	1.127 (7)
C(2)—N(3)	1.316 (5)		
N(1′)—C(2′)—N(3′)	122.1 (4)	C(35′)—N(3)—C(4)	121.0 (3)
C(2′)—N(3′)—C(4′)	118.7 (4)	S(1)—C(2)—N(3)	112.2 (3)
N(3′)—C(4′)—C(5′)	121.2 (4)	C(2)—N(3)—C(4)	114.1 (3)
C(4′)—C(5′)—C(6′)	116.6 (4)	N(3)—C(4)—C(5)	112.1 (3)
C(5′)—C(6′)—N(1′)	120.6 (4)	C(4)—C(5)—S(1)	110.3 (3)
C(6′)—N(1′)—C(2′)	120.7 (4)	C(5)—S(1)—C(2)	91.2 (2)
N(1′)—C(2′)—C(21′)	117.2 (4)	N(3)—C(4)—C(41)	120.3 (4)
N(3′)—C(2′)—C(21′)	120.7 (4)	C(5)—C(4)—C(41)	127.5 (4)
N(3′)—C(4′)—N(41′)	116.2 (4)	S(1)—C(5)—C(51)	122.2 (3)
C(5′)—C(4′)—N(41′)	122.6 (4)	C(4)—C(5)—C(51)	127.5 (4)
C(4′)—C(5′)—C(35′)	123.0 (4)	C(5)—C(51)—C(52)	114.1 (4)
C(6′)—C(5′)—C(35′)	120.0 (4)	C(51)—C(52)—O(53)	111.8 (4)
C(5′)—C(35′)—N(3)	114.8 (3)	S(11)—C(11)—N(11)	178.1 (5)
C(35′)—N(3)—C(2)	124.8 (3)	S(12)—C(12)—N(12)	178.7 (5)

D—H···*A*	*D*···*A*	H···*A*	*D*—H···*A*
N(1′)—H···N(11)	2.760 (6)	1.72	162
O(53)—H···S(11^{i})	3.223 (4)	2.23	166
N(41′)—H1···N(12)	2.834 (6)	1.76	165
N(41′)—H2···S(12^{ii})	3.447 (4)	2.62	134

Symmetry code: (i) $-\frac{3}{2}+x, \frac{1}{2}-y, -z$; (ii) $-x, -y, -z$.

The structure was solved by direct methods and refined by block-diagonal least squares on *F* with anisotropic thermal parameters for non-H atoms. All H atoms were located on a difference map and fixed with isotropic thermal parameters ($U = 0.06$ Å^2) in the final cycles of the refinement. All calculations were performed using the *SHELXTL* program system (Sheldrick, 1983).

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and intermolecular contacts have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55896 (24 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: OH1007]

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Non-Steroidal Anti-inflammatory Drugs. II. Structure of (*E*)-2,6-Di-*tert*-butyl-4-[2-(4-fluorophenyl)ethenyl]phenol

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Abstract

The central ethylenic double bond, C(7)═C(8) [1.296 (9) Å], is shorter than a normal C═C double bond. The two substituted phenyl rings are twisted by 15.5 (9) and 17.6 (9)°, respectively, out of the plane of the double bond. The dihedral angle between the mean planes of the two phenyl rings is 33.1 (1)°.

Comment

2,6-Di-*tert*-butylphenol derivatives appear to be a new class of non-steroidal anti-inflammatory drugs with antioxidant properties (Ikuta, Shirota, Kobayashi, Yamagishi, Yamada, Yamatsu & Katayama, 1987). The title compound has anti-inflammatory activity and is one of a series of related compounds prepared by Lazer, Wong, Possanza, Graham & Farina (1989). Their biological activity and molecular structure are being studied.

The bond length C(1)—F [1.367 (6) Å] is in good agreement with those in other fluorine-substituted phenyl rings (Taga, Yamamoto & Osaki, 1985; Kemmish & Hamor, 1990). However, the bond angle C(2)—C(1)—C(6) [122.3 (4)°] is significantly greater than 120°. This can be attributed to the strong σ-electron withdrawing properties of F, in agreement with the observation of Domenicano & Murray-Rust (1979). This enlargement of the angle is accompanied by shortening of the C—C bonds C(1)—C(2) [1.353 (8) Å] and C(1)—C(6) [1.361 (9) Å], compared to the central C—C bonds C(2)—C(3) [1.375 (7) Å] and C(6)—C(5) [1.398 (7) Å] of this ring (Colapietro,

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Domenicano, Marciante & Portalone, 1981; Briton & Gleason, 1977).

Some of the bond angles involving the *tert*-butyl groups are different from the standard values. Of note, C(12)—C(11)—C(19) and C(12)—C(13)—C(15) are 122.7 (4) and 121.5 (5)°, respectively, and C(16)—C(15)—C(17), C(20)—C(19)—C(22) and C(20)—C(19)—C(21) are 105.9 (4), 105.7 (5) and 106.3 (4)°, respectively. These deviations seem to reduce the intramolecular crowding of the *tert*-butyl groups (Burton, Le Page, Gabe & Ingold, 1980; Iimura, Sakurai, Ohno, Asahi & Isono, 1983).

The central ethylenic double bond [C(7)=C(8) = 1.296 (9) Å] is shorter than a normal C=C double bond [1.336 Å (Sutton, 1965)]. A similar shortening has been observed in the structures of (*E*)-1,2-bis(2-methylphenyl)ethene [C=C = 1.284 (4) Å (Tirado-Rives, Fronczek & Gandour, 1985) and (*E*)-2,6-di-*tert*-butyl-4-[2-(2-thienyl)ethenyl]phenol [C=C = 1.288 (7) Å (Ravikumar, 1992)]. It is noteworthy that in (*E*)-2,2′-dimethylstilbene (Ogawa, Suzuki, Sakurai, Kobayashi, Kira & Toriumi, 1988), the ethylenic C=C bond was found to be 1.280 (4) Å at 296 K and 1.325 (2) Å at 118 K.

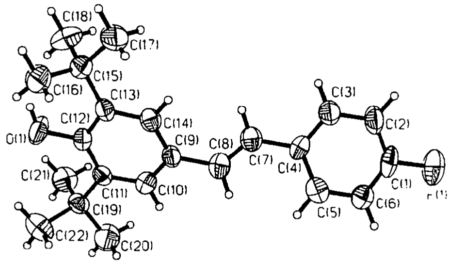

Fig. 1. Perspective view of the title compound showing the labelling of the non-H atoms. Thermal ellipsoids are shown at 50% probability levels except for H atoms which are drawn as small circles of arbitrary radii.

Experimental

Crystal data

$C_{22}H_{27}FO$
M_r = 326.4
Monoclinic
$P2_1/c$
a = 11.941 (1) Å
b = 18.423 (2) Å
c = 8.791 (1) Å
β = 90.49 (2)°
V = 1933.8 (3) Å^3
D_x = 1.12 Mg m^{-3}
D_m = 1.09 Mg m^{-3}
Density measured by flotation in aq. KI solution
Z = 4
Mo $K\alpha$ radiation
λ = 0.71063 Å
Cell parameters from 25 reflections
θ = 15–25°
μ = 0.069 mm^{-1}
T = 294 K
Cube
0.21 × 0.19 × 0.15 mm
Pale orange

Data collection

Siemens *R*3*m/V* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
2845 measured reflections
2543 independent reflections
1331 observed reflections [$I \geq 3\sigma(I)$]
$R_{\rm int}$ = 0.063
$\theta_{\rm max}$ = 22.5°
$h = -12 \rightarrow 12$
$k = 0 \rightarrow 19$
$l = 0 \rightarrow 9$
2 standard reflections monitored every 98 reflections intensity variation: 1%

Refinement

Refinement on F
Final R = 0.047
wR = 0.049
S = 1.02
1331 reflections
217 parameters
H atoms placed in calculated positions and allowed to ride on the parent C atoms
$w = [\sigma^2(F)+0.00076F^2]^{-1}$
$(\Delta/\sigma)_{\rm max}$ = 0.05
$\Delta\rho_{\rm max}$ = 0.20 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.21 e Å^{-3}
Extinction correction: none
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1990)

Data collection: Siemens *R*3*m/V* software. Cell refinement: Siemens *R*3*m/V* software. Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXTL-Plus*. Program(s) used for geometrical calculations including crystal packing: *PARST* (Nardelli, 1983). Molecular graphics: *SHELXTL-Plus*. Computer: VAX 3100 workstation.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^4)

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
F(1)	0.7003 (4)	0.3384 (3)	1.1848 (5)	113 (2)
O(1)	0.0795 (4)	0.4959 (3)	0.1403 (5)	68 (2)
C(1)	0.6386 (5)	0.3519 (4)	1.0561 (5)	67 (2)
C(2)	0.5873 (4)	0.2954 (4)	0.9861 (4)	63 (2)
C(3)	0.5252 (5)	0.3072 (4)	0.8561 (5)	54 (2)
C(4)	0.5124 (4)	0.3759 (4)	0.7963 (5)	47 (2)
C(5)	0.5692 (5)	0.4330 (4)	0.8691 (5)	59 (2)
C(6)	0.6320 (5)	0.4210 (4)	1.0017 (5)	66 (3)
C(7)	0.4401 (5)	0.3878 (4)	0.6600 (5)	50 (2)
C(8)	0.3995 (5)	0.4487 (4)	0.6109 (6)	54 (2)
C(9)	0.3181 (5)	0.4606 (3)	0.4833 (5)	43 (2)
C(10)	0.2590 (4)	0.5248 (3)	0.4756 (5)	46 (2)
C(11)	0.1768 (4)	0.5394 (3)	0.3642 (5)	39 (2)
C(12)	0.1596 (4)	0.4846 (3)	0.2545 (5)	41 (2)
C(13)	0.2175 (4)	0.4190 (3)	0.2554 (5)	44 (2)
C(14)	0.2970 (4)	0.4092 (3)	0.3722 (5)	45 (2)
C(15)	0.1944 (5)	0.3598 (3)	0.1339 (5)	54 (2)
C(16)	0.2197 (5)	0.3887 (4)	−0.0253 (5)	71 (3)
C(17)	0.2715 (6)	0.2936 (4)	0.1571 (6)	76 (3)
C(18)	0.0733 (5)	0.3339 (5)	0.1436 (6)	80 (3)
C(19)	0.1097 (5)	0.6105 (3)	0.3650 (5)	47 (2)
C(20)	0.1453 (5)	0.6594 (4)	0.4986 (5)	80 (3)
C(21)	−0.0148 (5)	0.5968 (4)	0.3834 (5)	66 (2)
C(22)	0.1320 (6)	0.6553 (4)	0.2196 (5)	77 (3)

Table 2. *Geometric parameters* (Å, °)

F(1)—C(1)	1.367 (6)	O(1)—C(12)	1.396 (6)
C(1)—C(2)	1.353 (8)	C(1)—C(6)	1.361 (9)
C(2)—C(3)	1.375 (7)	C(3)—C(4)	1.378 (9)
C(4)—C(5)	1.404 (9)	C(4)—C(7)	1.487 (7)
C(5)—C(6)	1.398 (7)	C(7)—C(8)	1.296 (9)
C(8)—C(9)	1.494 (7)	C(9)—C(10)	1.378 (8)
C(9)—C(14)	1.383 (7)	C(10)—C(11)	1.407 (7)

C(11)—C(12)	1.409 (7)	C(11)—C(19)	1.536 (8)
C(12)—C(13)	1.393 (8)	C(13)—C(14)	1.404 (7)
C(13)—C(15)	1.549 (8)	C(15)—C(16)	1.530 (7)
C(15)—C(17)	1.541 (9)	C(15)—C(18)	1.526 (9)
C(19)—C(20)	1.537 (8)	C(19)—C(21)	1.518 (8)
C(19)—C(22)	1.547 (7)		
F(1)—C(1)—C(6)	119.3 (5)	F(1)—C(1)—C(2)	118.3 (5)
C(2)—C(1)—C(6)	122.3 (4)	C(1)—C(2)—C(3)	119.7 (6)
C(2)—C(3)—C(4)	121.2 (5)	C(3)—C(4)—C(7)	120.2 (6)
C(3)—C(4)—C(5)	117.6 (5)	C(5)—C(4)—C(7)	122.2 (6)
C(4)—C(5)—C(6)	121.0 (6)	C(1)—C(6)—C(5)	118.1 (6)
C(4)—C(7)—C(8)	127.5 (5)	C(7)—C(8)—C(9)	128.1 (6)
C(8)—C(9)—C(14)	122.8 (5)	C(8)—C(9)—C(10)	119.6 (5)
C(10)—C(9)—C(14)	117.6 (5)	C(9)—C(10)—C(11)	123.5 (5)
C(10)—C(11)—C(19)	121.4 (4)	C(10)—C(11)—C(12)	115.8 (5)
C(12)—C(11)—C(19)	122.7 (4)	O(1)—C(12)—C(11)	118.7 (5)
C(11)—C(12)—C(13)	123.2 (4)	O(1)—C(12)—C(13)	118.0 (4)
C(12)—C(13)—C(15)	121.5 (5)	C(12)—C(13)—C(14)	116.6 (5)
C(14)—C(13)—C(15)	121.9 (5)	C(9)—C(14)—C(13)	123.2 (5)
C(13)—C(15)—C(18)	110.1 (4)	C(13)—C(15)—C(17)	111.3 (4)
C(13)—C(15)—C(16)	110.5 (5)	C(17)—C(15)—C(18)	108.1 (5)
C(16)—C(15)—C(18)	110.7 (4)	C(16)—C(15)—C(17)	105.9 (4)
C(11)—C(19)—C(22)	111.0 (4)	C(11)—C(19)—C(21)	111.7 (5)
C(11)—C(19)—C(20)	111.3 (4)	C(21)—C(19)—C(22)	110.7 (5)
C(20)—C(19)—C(22)	105.7 (5)	C(20)—C(19)—C(21)	106.3 (4)

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55920 (13 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HA1029]

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Structure of *N*-(2-Hydroxyethyl)ethylenediaminetriacetic Acid

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Abstract

The structure determination has shown that the title compound exists as a zwitterion in a *gauche* conformation about the central C—C bond. Both of the protonated N atoms participate in intramolecular hydrogen bonding. There are also two intermolecular hydrogen-bond interactions which link the molecules into chains extending parallel to **a**. One of these hydrogen bonds, acting between the carboxylic acid groups, is strong [O···O = 2.450 (2) Å] and close to symmetrical. The carboxyl groups assume the usual synplanar conformation. The results are compared with those obtained previously for closely related ethylenediaminetetraacetic acid.

Comment

This work is part of a more general study aiming at designing new metal-chelating agents. In this communication we report on the crystal structure of *N*-(hydroxyethyl)ethylenediaminetriacetic acid (H_3heedta) which is derived from the well known ligand ethylenediaminetetraacetic acid (H_4edta), by

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