C36'	0.6074 (1)	0.4127 (3)	0.1227 (2)	0.017 (2)
C31	0.1126 (2)	0.1893 (3)	0.1538 (2)	0.026 (2)
C32	0.0782 (2)	0.3002 (3)	0.1595 (2)	0.027 (3)
C33	0.0476 (2)	0.3690 (3)	0.0992 (2)	0.032 (3)
C34	0.0514 (2)	0.3267 (3)	0.0331 (2)	0.032 (3)
C35	0.0858 (2)	0.2158 (3)	0.0274 (2)	0.027 (2)
C36	0.1165 (2)	0.1470 (3)	0.0877 (2)	0.021 (2)
C41	0.2912 (2)	−0.1000 (3)	0.1047 (2)	0.024 (2)
C42	0.3672 (2)	−0.0985 (3)	0.1279 (2)	0.025 (3)
C43	0.4033 (2)	0.0085 (3)	0.1561 (2)	0.029 (2)
C44	0.3636 (2)	0.1141 (3)	0.1610 (2)	0.025 (2)
C45	0.2876 (2)	0.1126 (3)	0.1378 (2)	0.021 (2)
C46	0.2514 (2)	0.0056 (3)	0.1096 (2)	0.021 (2)

Table 2. *Selected geometric parameters* (Å, °)

For molecule 2, each atom label should be appended by a prime.

	Molecule 1	Molecule 2
P1—C1	1.716 (5)	1.725 (4)
C1—C2	1.407 (8)	1.399 (8)
O1—C2	1.265 (7)	1.247 (7)
P1—C16	1.816 (5)	1.802 (5)
P1—C36	1.815 (4)	1.811 (4)
P1—C46	1.803 (4)	1.810 (4)
C2—C26	1.519 (7)	1.513 (7)
C36—P1—C46	107.9 (2)	105.3 (2)
C16—P1—C46	108.9 (2)	108.8 (2)
C16—P1—C36	105.2 (2)	107.4 (2)
C1—P1—C46	111.9 (3)	115.7 (3)
C1—P1—C36	115.7 (3)	115.2 (3)
C1—P1—C16	107.0 (3)	104.2 (3)
P1—C1—C2	115.3 (4)	123.0 (4)
O1—C2—C1	122.9 (5)	125.4 (5)
C1—C2—C26	119.8 (5)	116.1 (5)
O1—C2—C26	117.3 (5)	118.5 (5)

Table 3. *Comparison of some structural features* (Å, °) *of* $(C_6H_5)_3PC(X)COC_6H_5$ (X = H, Cl *and* I) *compounds*

	P—C	C=O	C—CO
$(C_6H_5)_3PCHCOC_6H_5$ (1)	1.716 (5)	1.265 (7)	1.407 (8)
$(C_6H_5)_3PCHCOC_6H_5$ (2)	1.725 (4)	1.247 (7)	1.399 (8)
$(C_6H_5)_3PC(Cl)COC_6H_5{}^a$	1.736 (14)	1.301 (19)	1.361 (20)
$(C_6H_5)_3PC(I)COC_6H_5{}^b$	1.71 (5)	1.28 (6)	1.35 (7)

References: (a) Stephens (1965b); (b) Stephens (1965a).

Initial data reduction was carried out using *SHELXTL* (Sheldrick, 1984). All the H atoms were included in the final cycle of refinement riding at distances of 1.08 Å with displacement parameters equal to $1.2U_{eq}$ of the parent non-ring atoms. The displacement parameters for ring H atoms were fixed at 0.05 Å^2. All phenyl rings were refined as rigid groups. The final refinement and the table of F_o and F_c were obtained using *SHELX76* (Sheldrick, 1976). Bond parameters and equations of planes were obtained by the use of *PARST* (Nardelli, 1983).

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Hexamethylenediammonium Bis(β-phenylacrylate) Dihydrate and Hexamethylenediammonium 3,3'-(1,4-Phenylene)diacrylate Monohydrate

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Abstract

The crystal structures of hexamethylenediammonium bis(β-phenylacrylate) dihydrate, $C_6H_{18}N_2^{2+}.2C_9H_7O_2^-.2H_2O$, (I), and hexamethylenediammonium phenylene-1,4-diacrylate monohydrate, $C_6H_{18}N_2^{2+}.C_{12}H_8O_4^{2-}.H_2O$, (II), have been studied. In (I) the anions related by $\bar{1}$ symmetry overlap each other, but the distance between the C=C double bonds, 5.165 (3) Å, is too long for photodimerization to occur. In (II) the anions and cations are stacked alternately, so no short contact is observed between the C=C double bonds of the anions.

Comment

As part of a study to design photoreactive crystals (Iwamoto & Kashino, 1990), the crystal structures of

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(I) and (II) were investigated in order to elucidate the effect of the functionality of carboxylic acids on molecular arrangement.

(I)   (II)
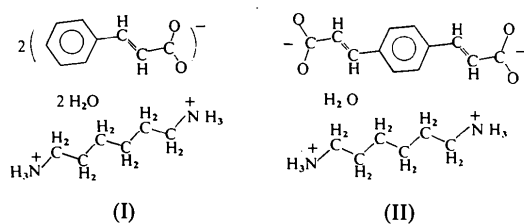
The cation in (I) has $\bar{1}$ symmetry in the crystals. However, the cation and anion in (II) have no symmetry as was also found for hexamethylene-diammonium terephthalate dihydrate (Moritani, Kashino & Haisa, 1990). The cations in (I) and (II) take *trans* zigzag conformations but in (II) the torsion angles N(1)—C(20)—C(21)—C(22)

[−174.3 (5)°] and C(21)—C(22)—C(23)—C(24) [172.5 (6)°] deviate significantly from 180°. The side chain in the anion of (I) lies nearly in the plane of the benzene ring as is also observed for the side chain attached to C(1) in (II) [C(2)—C(1)—C(7)—C(8) 170.5 (2) for (I) and 178.5 (6)° for (II); O(2)—C(9)—C(8)—C(7) 177.3 (2) for (I) and 171.3 (6)° for (II)]. However, the side chain attached to C(4) of (II) deviates significantly from the plane [C(5)—C(4)—C(10)—C(11) −165.1 (6) O(4)—C(12)—C(11)—C(10) 160.8 (6)°].

In both crystals three-dimensional networks are formed by hydrogen bonding through the terminal groups of the cations and anions and the water molecules (Table 3). In the crystals of (I), a salt of a diamine and a monocarboxylic acid, the anions and cations are arranged in the sequence anion–cation–anion–anion–cation–anion in the directions of the short and long molecular axes, and in the direction perpendicular to the molecular planes (Fig. 2a). The anions related by $\bar{1}$ symmetry are overlapped, with an interplanar distance of 3.519 (3) Å. On the other hand, in the crystals of (II), a salt of a diamine and a dicarboxylic acid, the anions and cations are arranged alternately (Fig. 2b) and overlap along the c axis as in hexamethylenediammonium terephthalate dihydrate. In the photoreactive crystals of cinnamic acid and its derivatives, the molecules which
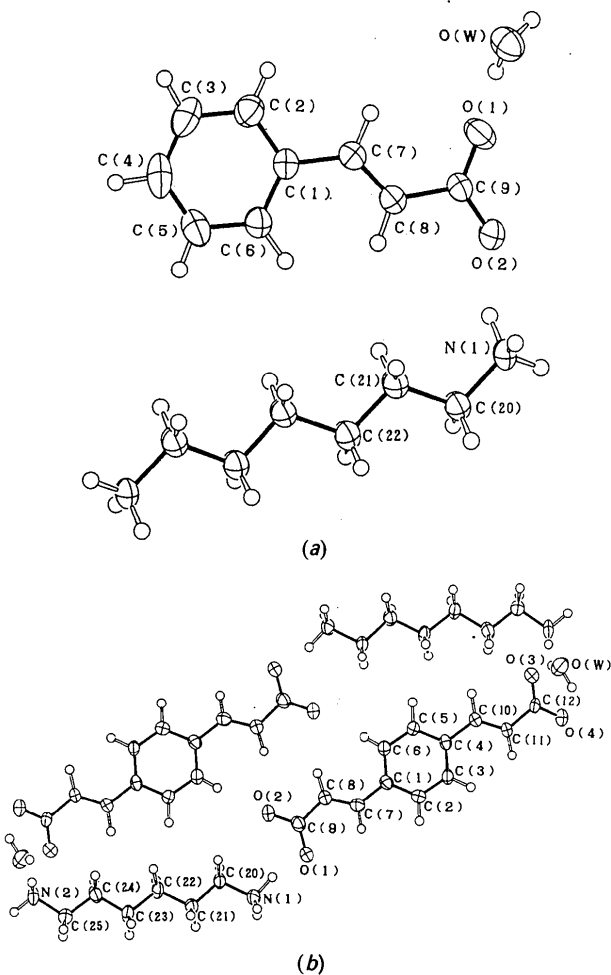

(a)

(b)

Fig. 1. The thermal ellipsoids (50% probability) with atomic numbering for (a) (I) and (b) (II) for non-H atoms. H atoms are represented as spheres equivalent to $B = 1.0$ Å^2.
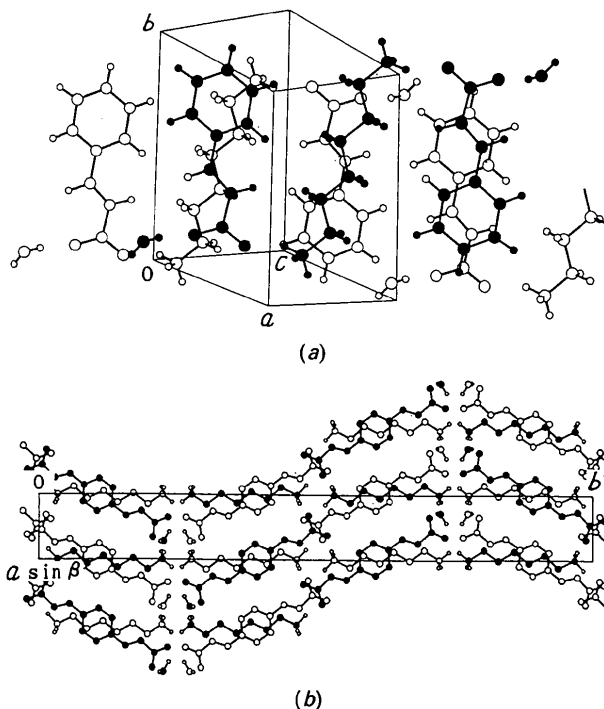

(a)

(b)

Fig. 2. The molecular arrangement in the crystals (a) viewed perpendicular to the molecular plane of the anion of (I) and (b) viewed down the c axis of (II).

react overlap with each other in order to satisfy the topochemical condition for the reaction (Bryan & Freyberg, 1975; Glusker, Zacharias & Carrell, 1975; Kashino, Oka & Haisa, 1989; Kanao, Kashino & Haisa, 1990), while in (II) such molecular overlap between the anions cannot be realized.

Experimental

Crystals of (I) were prepared by slow evaporation of an ethanol solution of hexamethylenediamine and β-phenylacrylic acid (cinnamic acid) in a molar ratio of 1:2. Crystals of (II) were prepared by slow evaporation of an aqueous solution of an equimolar mixture of hexamethylenediamine and phenylene-1,4-diacrylic acid. The acid was synthesized by the method described by Klein & Bergmann (1957). D_m was measured by flotation in aqueous KI. A crystal of (I) was sealed in a glass capillary for data collection.

Compound (I)

Crystal data

$C_6H_{18}N_2^{2+}.2C_9H_7O_2^-.2H_2O$
$M_r = 448.56$
Triclinic
$P\bar{1}$
$a = 8.098 (1)$ Å
$b = 9.929 (2)$ Å
$c = 7.515 (3)$ Å
$\alpha = 92.65 (2)°$
$\beta = 94.56 (2)°$
$\gamma = 96.37 (2)°$
$V = 597.6 (5)$ Å^3
$Z = 1$
$D_x = 1.246$ Mg m^{-3}
$D_m = 1.24$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 17.5–19.7°$
$\mu = 0.088$ mm^{-1}
$T = 298$ K
Plate, {010} developed
$0.30 \times 0.30 \times 0.10$ mm
Colorless

Data collection

Rigaku AFC-5R diffractometer
$\omega–2\theta$ scans [width (1.21 + 0.30tanθ)° in ω, rate 6° min^{-1} in ω]
Absorption correction: none
2288 measured reflections
2110 independent reflections
1802 observed reflections
  [$F > 2.0\sigma(F)$]

$R_{int} = 0.006$
$\theta_{max} = 25°$
$h = -9 \rightarrow 9$
$k = -11 \rightarrow 11$
$l = 0 \rightarrow 8$
3 standard reflections
  monitored every 97
  reflections
  intensity variation: 1%

Refinement

Refinement on F
$R = 0.038$
$wR = 0.041$
$S = 1.93$
1802 reflections
217 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F_o)$

$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.16$ e Å^{-3}
$\Delta\rho_{min} = -0.13$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Compound (II)

Crystal data
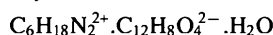
$C_6H_{18}N_2^{2+}.C_{12}H_8O_4^{2-}.H_2O$
$M_r = 352.43$
Monoclinic
$P2_1/c$
$a = 5.558 (2)$ Å
$b = 46.03 (2)$ Å
$c = 7.418 (3)$ Å
$\beta = 106.82 (4)°$
$V = 1816 (1)$ Å^3
$Z = 4$
$D_x = 1.289$ Mg m^{-3}
$D_m = 1.29$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 24 reflections
$\theta = 10.0–11.5°$
$\mu = 0.088$ mm^{-1}
$T = 295$ K
Plate, {010} developed
$0.30 \times 0.20 \times 0.10$ mm
Colorless

Data collection

Rigaku AFC-5R diffractometer
$\omega–2\theta$ scans [width (0.73 + 0.30tanθ)° in ω, rate 6° min^{-1} in ω]
Absorption correction: empirical
$T_{min} = 0.84$, $T_{max} = 1.00$
4690 measured reflections
4188 independent reflections
1545 observed reflections
  [$F > 2.0\sigma(F)$]

$R_{int} = 0.066$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 7$
$k = 0 \rightarrow 59$
$l = -9 \rightarrow 9$
3 standard reflections
  monitored every 97
  reflections
  intensity variation: 2%

Refinement

Refinement on F
$R = 0.060$
$wR = 0.045$
$S = 1.41$
1545 reflections
339 parameters
All H-atom parameters refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} = 0.01$

$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.27$ e Å^{-3}
Extinction correction: secondary
Extinction coefficient: 0.866×10^{-6}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Table 1. *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters* (Å^2)

B_{iso} for H atoms; $B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	B_{iso}/B_{eq}
(I)				
O(1)	0.1528 (2)	0.1338 (1)	0.1725 (2)	4.52 (6)
O(2)	0.3731 (1)	0.1268 (1)	0.3643 (2)	3.98 (5)
O(W)	-0.1918 (2)	0.0326 (2)	0.1108 (2)	4.34 (6)
N(1)	0.4635 (2)	0.0932 (1)	0.7299 (2)	3.23 (6)
C(1)	0.1344 (2)	0.5498 (2)	0.3441 (2)	2.90 (7)
C(2)	0.0283 (2)	0.6232 (2)	0.2440 (3)	3.75 (8)
C(3)	0.0199 (3)	0.7590 (2)	0.2865 (3)	4.6 (1)
C(4)	0.1158 (3)	0.8230 (2)	0.4315 (3)	4.7 (1)
C(5)	0.2192 (3)	0.7517 (2)	0.5342 (3)	4.36 (9)
C(6)	0.2290 (2)	0.6163 (2)	0.4902 (2)	3.57 (8)
C(7)	0.1438 (2)	0.4070 (2)	0.2916 (2)	3.07 (7)
C(8)	0.2515 (2)	0.3291 (2)	0.3563 (2)	3.23 (7)
C(9)	0.2575 (2)	0.1859 (2)	0.2922 (2)	2.91 (7)
C(20)	0.5437 (2)	0.2003 (2)	0.8652 (3)	3.37 (8)
C(21)	0.4562 (2)	0.3258 (2)	0.8661 (3)	3.38 (8)
C(22)	0.5393 (2)	0.4342 (2)	1.0016 (2)	3.17 (7)
H(OWA)	-0.176 (3)	-0.014 (2)	0.007 (3)	7.1 (6)

$C_6H_{18}N_2^{2+} \cdot 2C_9H_7O_2^- \cdot 2H_2O$ AND $C_6H_{18}N_2^{2+} \cdot C_{12}H_8O_4^{2-} \cdot H_2O$

H(OWB)	-0.097(3)	0.069(2)	0.139(3)	7.9(8)
H(N1A)	0.356(3)	0.055(2)	0.771(3)	5.3(5)
H(N1B)	0.437(2)	0.129(2)	0.614(3)	5.3(5)
H(N1C)	0.532(2)	0.023(2)	0.716(2)	4.1(4)

(II)

O(1)	0.3185(6)	0.01252(7)	0.7084(5)	3.4(2)
O(2)	0.6201(7)	-0.02086(7)	0.7914(6)	4.3(2)
O(3)	1.6614(7)	0.20555(7)	0.9160(6)	3.7(2)
O(4)	1.3530(7)	0.23373(7)	0.9484(6)	4.8(2)
O(W)	1.7254(8)	0.2259(1)	0.5599(7)	4.6(2)
N(1)	0.001(1)	-0.0334(1)	0.6372(8)	3.2(2)
N(2)	-0.002(1)	-0.2237(1)	0.7096(8)	3.7(2)
C(1)	0.860(1)	0.0811(1)	0.7991(7)	2.4(2)
C(2)	0.762(1)	0.1085(1)	0.7366(8)	2.6(3)
C(3)	0.907(1)	0.1331(1)	0.7677(8)	2.5(2)
C(4)	1.164(1)	0.1315(1)	0.8646(7)	2.1(2)
C(5)	1.263(1)	0.1042(1)	0.9267(8)	2.4(2)
C(6)	1.114(1)	0.0794(1)	0.8959(8)	2.8(3)
C(7)	0.690(1)	0.0562(1)	0.7656(8)	2.6(2)
C(8)	0.742(1)	0.0284(1)	0.8089(8)	3.1(3)
C(9)	0.545(1)	0.0051(1)	0.7661(8)	2.9(3)
C(10)	1.329(1)	0.1571(1)	0.8956(6)	2.5(2)
C(11)	1.257(1)	0.1847(1)	0.8810(8)	2.6(2)
C(12)	1.439(1)	0.2093(1)	0.9147(8)	2.9(2)
C(20)	0.119(1)	-0.0627(1)	0.689(1)	2.8(3)
C(21)	-0.072(1)	-0.0868(1)	0.631(1)	2.7(3)
C(22)	0.047(1)	-0.1159(1)	0.7028(9)	2.8(3)
C(23)	-0.129(1)	-0.1420(1)	0.647(1)	3.0(3)
C(24)	0.013(1)	-0.1702(1)	0.702(1)	3.5(3)
C(25)	-0.154(1)	-0.1964(1)	0.656(1)	3.5(3)
H(OWA)	1.73(1)	0.220(1)	0.671(8)	6(1)
H(OWB)	1.57(1)	0.236(2)	0.51(1)	11(1)
H(N1A)	-0.09(1)	-0.032(1)	0.515(8)	5(1)
H(N1B)	0.15(1)	-0.017(2)	0.67(1)	11(1)
H(N1C)	-0.13(1)	-0.029(1)	0.73(1)	10(1)
H(N2A)	0.11(1)	-0.222(2)	0.89(1)	15(1)
H(N2B)	0.10(1)	-0.226(1)	0.635(9)	7(1)
H(N2C)	-0.12(1)	-0.241(2)	0.68(1)	10(1)

Table 2. *Selected geometric parameters* (Å, °)

(I)

O(1)—C(9)	1.239(2)	C(4)—C(5)	1.370(3)
O(2)—C(9)	1.259(2)	C(5)—C(6)	1.382(3)
N(1)—C(20)	1.493(2)	C(7)—C(8)	1.309(2)
C(1)—C(2)	1.386(2)	C(8)—C(9)	1.487(2)
C(1)—C(6)	1.384(2)	C(20)—C(21)	1.500(2)
C(1)—C(7)	1.466(2)	C(21)—C(22)	1.509(2)
C(2)—C(3)	1.382(3)	C(22)—C(22i)	1.516(3)
C(3)—C(4)	1.374(3)		

O(1)—C(9)—O(2)	124.6(2)	C(2)—C(1)—C(7)	119.5(2)
O(1)—C(9)—C(8)	119.1(2)	C(2)—C(3)—C(4)	120.0(2)
O(2)—C(9)—C(8)	116.2(1)	C(3)—C(4)—C(5)	119.8(2)
N(1)—C(20)—C(21)	112.7(1)	C(4)—C(5)—C(6)	120.1(2)
C(1)—C(2)—C(3)	121.1(2)	C(6)—C(1)—C(7)	122.7(2)
C(1)—C(6)—C(5)	121.1(2)	C(7)—C(8)—C(9)	124.3(2)
C(1)—C(7)—C(8)	127.1(2)	C(20)—C(21)—C(22)	112.4(1)
C(2)—C(1)—C(6)	117.8(2)	C(21)—C(22)—C(22i)	113.8(2)

(II)

O(1)—C(9)	1.256(6)	C(4)—C(10)	1.467(6)
O(2)—C(9)	1.263(6)	C(5)—C(6)	1.388(7)
O(3)—C(12)	1.247(6)	C(7)—C(8)	1.333(7)
O(4)—C(12)	1.272(6)	C(8)—C(9)	1.496(7)
N(1)—C(20)	1.501(7)	C(10)—C(11)	1.326(6)
N(2)—C(25)	1.500(7)	C(11)—C(12)	1.490(7)
C(1)—C(2)	1.400(7)	C(20)—C(21)	1.510(7)
C(1)—C(6)	1.388(6)	C(21)—C(22)	1.518(7)
C(1)—C(7)	1.459(7)	C(22)—C(23)	1.532(7)
C(2)—C(3)	1.374(7)	C(23)—C(24)	1.509(8)
C(3)—C(4)	1.400(6)	C(24)—C(25)	1.502(8)
C(4)—C(5)	1.398(6)		

O(1)—C(9)—O(2)	124.2(5)	C(2)—C(3)—C(4)	120.4(5)
O(1)—C(9)—C(8)	118.6(5)	C(3)—C(4)—C(5)	117.5(5)
O(2)—C(9)—C(8)	117.2(5)	C(4)—C(5)—C(6)	121.7(5)
O(3)—C(12)—O(4)	123.3(5)	C(6)—C(1)—C(7)	124.0(5)
O(3)—C(12)—C(11)	121.1(5)	C(7)—C(8)—C(9)	122.7(5)
O(4)—C(12)—C(11)	115.6(5)	C(3)—C(4)—C(10)	122.4(5)
N(1)—C(20)—C(21)	111.6(5)	C(5)—C(4)—C(10)	120.1(5)
N(2)—C(25)—C(24)	110.4(5)	C(4)—C(10)—C(11)	126.7(5)
C(1)—C(2)—C(3)	122.3(5)	C(10)—C(11)—C(12)	123.0(5)
C(1)—C(6)—C(5)	120.7(5)	C(20)—C(21)—C(22)	110.2(5)
C(1)—C(7)—C(8)	128.8(5)	C(21)—C(22)—C(23)	114.6(5)
C(2)—C(1)—C(6)	117.4(5)	C(22)—C(23)—C(24)	111.1(5)
C(2)—C(1)—C(7)	118.6(5)	C(23)—C(24)—C(25)	113.0(5)

Symmetry code: (i) $1 - x, 1 - y, 2 - z$.

Table 3. *Hydrogen-bonding geometry* (Å, °)

D—H···A	H···A	D···A	D—H···A
(I)			
N(1)—H(N1B)···O(2)	1.91(2)	2.831(2)	158(2)
N(1)—H(N1C)···O(2i)	1.85(2)	2.769(2)	165(2)
N(1)—H(N1A)···O(Wii)	1.82(2)	2.789(2)	166(2)
O(W)—H(OWB)···O(1)	2.04(3)	2.853(2)	169(2)
O(W)—H(OWA)···O(1iii)	1.79(3)	2.694(2)	169(2)
(I)			
N(1)—H(N1B)···O(1)	1.62(7)	2.708(6)	166(5)
N(1)—H(N1A)···O(1ii)	1.99(5)	2.831(6)	155(5)
N(1)—H(N1C)···O(2iv)	1.65(7)	2.741(6)	162(5)
N(2)—H(N2A)···O(3v)	1.82(8)	2.991(7)	150(6)
N(2)—H(N2B)···O(Wvi)	1.97(6)	2.853(7)	169(6)
N(2)—H(N2C)···O(4vii)	1.80(7)	2.776(6)	163(6)
O(W)—H(OWA)···O(3)	2.07(6)	2.919(6)	167(6)
O(W)—H(OWB)···O(4viii)	1.83(7)	2.727(5)	160(7)

Symmetry codes: (i) $1-x, -y, 1-z$; (ii) $-x, -y, 1-z$; (iii) $-x, -y, -z$; (iv) $1 + x, y, z$; (v) $2 - x, -y, 2 - z$; (vi) $2 - x, -y, 1 - z$; (vii) $1 - x, \frac{1}{2} + y, \frac{3}{2} - z$; (viii) $x, \frac{1}{2} - y, \frac{1}{2} + z$.

The structures were solved by direct methods using *MITHRIL* (Gilmore, 1984) and *DIRDIF* (Beurskens, 1984). Refinement was carried out using full-matrix least-squares methods (*TEXSAN*; Molecular Structure Corporation, 1985). The ellipsoid plots were drawn using *ORTEP*II (Johnson, 1976). Computations were carried out on a VAX3100 computer at the X-ray Laboratory of Okayama University.

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D-Secoestrone Derivatives. III. 17-Fluoro-3-methoxy-16,17-secoestra-1,3,5(10)-triene-16-nitrile

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## Abstract

The asymmetric unit of the title compound, $C_{19}H_{24}FNO$, contains two molecules which differ in the orientation of the 16-cyano moieties. A discussion and comparison of the structure–activity relationships among the different halogeno derivatives of the title compound is given.

Comment

As a part of a broader project dealing with the synthesis and testing of novel potential antiestrogenic agents, we have previously reported a series of new 17-halogeno-16,17-secoestrone derivatives (Petrović, Pejanović, Miljković & Hranisavljević, 1990). By testing the residual estrogenic and antiestrogenic effect of these derivatives it was found that most of the compounds examined had lost their hormone activity completely, but at the same time expressed a considerable antagonistic effect, which was most pronounced in the case of the 17-bromo derivative. The 17-fluoro-16,17-secoestrone derivative (I) proved to

be an exception as it retained about 34% of the original hormonal activity and, furthermore, was found to act as a synergist in combination with estradiol.
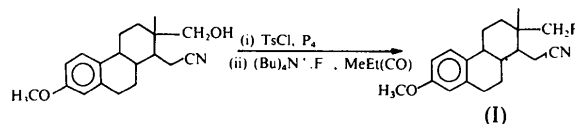
Keeping these facts in mind, we undertook the X-ray structure analysis of a number of 17-substituted seco compounds. On comparing the structural features of the 17-chloro, bromo and iodo derivatives (Stanković, Petrović, Miljković, Pejanović, Kovačević, Stefanović & Bruvo, 1992) and the 17-toluenesulfonyloxy compound (Stanković, Stefanović, Bruvo & Altomare, 1992) with those of the 17-fluoro derivative desribed here a high degree of similarity was noticed. Selected geometric parameters for the two independent molecules of the title compound found in the unit cell, together with the corresponding mean values for the chloro, bromo, and iodo derivatives and the mean values for the two independent molecules found in the unit cell of the toluenesulfonyloxy (Ts) derivative are given in Table 2. The most significant difference appears in the orientations of the F atoms in molecules 1 and 2 of the title compound compared to the rest of the halogeno derivatives. Taking C(13)—C(14)—C(15) as a reference plane, the C(18) methyl group and the C(16) nitrile function are in an *anti* orientation in molecule 1 (Fig. 2a) and in a *syn* orientation in molecule 2 (Fig. 2b). The *syn* orientation is observed in the 17-chloro, bromo and iodo derivatives (Stanković, Petrović, Miljković, Pejanović, Kovačević, Stefanović & Bruvo, 1992). The orientation of the 3-methoxy group, which is the same in
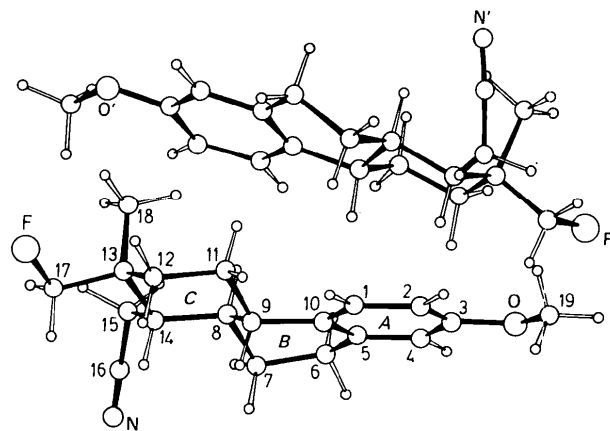

Fig. 1. A perspective view of the two symmetry independent molecules 1 and 2. The numbers correspond with C-atom labelling.