

STENKAMP, R. E. & JENSEN, L. H. (1973). *Acta Cryst.* B29, 2871–2878.
 TERASHIMA, S., ACHIWA, K. & YAMADO, S. (1966). *Chem. Pharm. Bull.* 14, 1138.
 TURK, J., PANSE, G. T. & MARSHALL, G. R. (1975). *J. Org. Chem.* 40, 953–955.

WEI, C. H., DOHERTY, D. G. & EINSTEIN, J. R. (1972). *Acta Cryst.* B28, 907–915.

WILLIS, B. T. M. & PRYOR, A. W. (1975). *Thermal Vibrations in Crystallography*, pp. 101–102. Cambridge Univ. Press.

Acta Cryst. (1981). B37, 1788–1790

5(S)-Cyano-9-oxotricyclo[4.2.1.1^{4,7}]dec-2-en-8-yl Methanesulfonate*

BY K. A. NIRMALA† AND D. S. SAKE GOWDA

Department of Physics, Central College, Bangalore University, Bangalore-560001, India

AND WILLIAM H. WATSON

Department of Chemistry, Texas Christian University, Fort Worth, Texas 76129, USA

(Received 13 October 1980; accepted 30 March 1981)

Abstract. C₁₂H₁₃NO₄S, $M_r = 267.3$, orthorhombic, $Pca2_1$, $a = 10.159(5)$, $b = 8.656(3)$, $c = 13.950(5)$ Å, $Z = 4$, $D_m = 1.43$, $D_c = 1.45$ Mg m⁻³. The structure was solved by the Patterson search method and refined to $R = 0.054$ for 960 reflections. No hydrogen bonding is observed and the structure is held together by van der Waals interactions.

Introduction. The compound, supplied by Goverdhan Mehta (University of Hyderabad, Hyderabad, India), is formed in an unanticipated rearrangement pathway from pentacyclo[5.4.0.0^{2,6}.0^{3,10}.0^{5,9}]undecane-8,11-dione to new C₁₀ carbocyclic systems under Schmidt reaction conditions (Goverdhan Mehta *et al.*, 1977). Spectral data could not reveal the exact nature of the carbocyclic framework of this compound. The preparation, the initial survey of crystals, and the solution and the refinement of the structure have been carried out at Bangalore, India. The intensity data were collected at Texas Christian University, USA.

Preliminary data for the title compound were obtained from Weissenberg photographs taken about different crystallographic axes with Cu $K\alpha$ radiation. The systematic absences, $h0l: h = 2n + 1, 0kl: l = 2n + 1$, indicated the space group $Pca2_1$. The density of the crystal was determined by flotation using aqueous potassium iodide.

Intensity data were collected on a Syntex P2₁ diffractometer with the θ - 2θ scanning technique, using

* This paper was presented at the National Conference on Crystallography held at Calcutta (India) in 1980.

† Present address: Indian Institute of Science, Bangalore-560012, India.

a variable scan speed, Cu $K\alpha$ radiation and a graphite monochromator ($\lambda = 1.54178$ Å). The data were corrected for Lorentz and polarization effects, but no correction was made for absorption [$\mu(\text{Cu } K\alpha) = 2.37$ mm⁻¹]. Out of 1190 possible reflections with $2\theta \leq 140^\circ$, 960 were observed and the rest were treated as unobserved [$I < 3\sigma(I)$]. The data were placed on an approximately absolute scale using the Wilson plot. The overall isotropic thermal parameter was 4.73 Å².

Attempts were made to determine the structure using the heavy-atom method [*MULTAN* (Germain, Main & Woolfson, 1971) and *SHELX* (Sheldrick, 1976)]. Since the electron density maps showed mirror-image peaks and spurious peaks, the positions of the light atoms could not be derived. A sharpened Patterson function was computed using *SHELX*, the heavy-atom coordinates were calculated from the heavy-atom vectors (Buerger, 1959), and a search was made for the methanesulfonate fragment by collecting heavy-atom vectors within 2 Å of the origin. Some spurious peaks were eliminated by checking the S vectors in the Patterson map. With the methanesulfonate fragment in the difference map the remaining atoms were determined. Structure factor calculations based on these atoms gave $R = 0.27$.

Block-diagonal least-squares refinement of an overall scale factor, positional coordinates and isotropic temperature factors reduced R to 0.12. A difference Fourier map computed at this stage revealed the locations of the H atoms. Refinement of the positional and anisotropic thermal parameters of the non-hydrogen atoms, with the H atoms included for structure factor calculations only, resulted in a final R

of 0.054 for 960 observed data. The function minimized was $\sum w(|F_o| - k|F_c|)^2$ with $w = 1/\sigma^2(F_o)$. Final positional parameters are presented in Tables 1 and 2.* Scattering factors for non-hydrogen atoms were taken from Cromer & Waber (1965) and for H atoms from Stewart, Davidson & Simpson (1965).

Discussion. The structure and stereochemistry of the title compound are in agreement with one of the structures proposed by Goverdhan Mehta *et al.* (1977). The basic skeleton of the compound (Fig. 1) consists of

* Lists of structure factors, anisotropic thermal parameters and interatomic contacts have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36088 (7 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final positional ($\times 10^4$) and isotropic thermal ($\text{\AA}^2 \times 10^4$) parameters of the non-hydrogen atoms with standard deviations in parentheses

$$U_{\text{eq}} = (U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	U_{eq}
S	1020 (1)	597 (2)	4616 (2)	367 (8)
O(1)	-132 (7)	1537 (10)	4547 (7)	694 (9)
O(2)	993 (8)	-676 (8)	5297 (6)	555 (10)
O(3)	1358 (7)	45 (8)	3602 (6)	463 (13)
O(4)	-763 (6)	-2256 (7)	2053 (5)	518 (14)
C(1)	808 (8)	-2626 (10)	3333 (7)	377 (18)
C(2)	1278 (10)	-4349 (11)	3332 (7)	457 (18)
C(3)	2174 (10)	-4924 (12)	2759 (6)	487 (17)
C(4)	2972 (9)	-4139 (11)	2013 (7)	429 (16)
C(5)	2129 (8)	-3357 (9)	1230 (6)	391 (14)
C(6)	1572 (9)	-1884 (10)	1737 (6)	356 (13)
C(7)	2628 (9)	-1452 (10)	2488 (6)	378 (10)
C(8)	1940 (9)	-1526 (10)	3464 (7)	368 (15)
C(9)	359 (8)	-2270 (10)	2348 (7)	379 (14)
C(10)	3698 (9)	-2705 (13)	2410 (9)	529 (16)
C(11)	2370 (14)	1800 (13)	4935 (9)	617 (22)
C(12)	1176 (9)	-4351 (10)	790 (7)	434 (16)
N	480 (9)	-5154 (11)	440 (7)	648 (20)

Table 2. Positional ($\times 10^3$) and isotropic thermal ($\text{\AA}^2 \times 10^3$) parameters of the hydrogen atoms

Bonded to	x	y	z	U	
H(1)	C(11)	325 (13)	112 (14)	500 (10)	45 (21)
H(2)	C(11)	217 (12)	237 (13)	561 (11)	68 (20)
H(3)	C(11)	251 (13)	268 (13)	439 (10)	78 (20)
H(4)	C(8)	262 (12)	-189 (12)	404 (9)	66 (19)
H(5)	C(7)	303 (11)	-32 (13)	240 (10)	50 (19)
H(6)	C(1)	8 (12)	-244 (11)	389 (10)	49 (17)
H(7)	C(6)	138 (11)	-99 (12)	123 (9)	50 (18)
H(8)	C(10)	416 (10)	-293 (11)	309 (9)	50 (17)
H(9)	C(10)	442 (11)	-233 (12)	189 (9)	68 (18)
H(10)	C(4)	360 (10)	-507 (12)	177 (9)	37 (18)
H(11)	C(5)	265 (11)	-301 (12)	58 (10)	50 (17)
H(12)	C(2)	81 (11)	-512 (13)	383 (9)	74 (18)
H(13)	C(3)	236 (12)	-614 (11)	284 (9)	74 (18)

four bicyclo[4.2.1]nonane rings with six atoms in common; these can be further split into (i) two five-membered rings, (ii) two seven-membered rings and (iii) two eight-membered rings. Bond lengths and angles are given in Tables 3 and 4. No hydrogen bonding is observed and the structure is held together by van der Waals interactions.

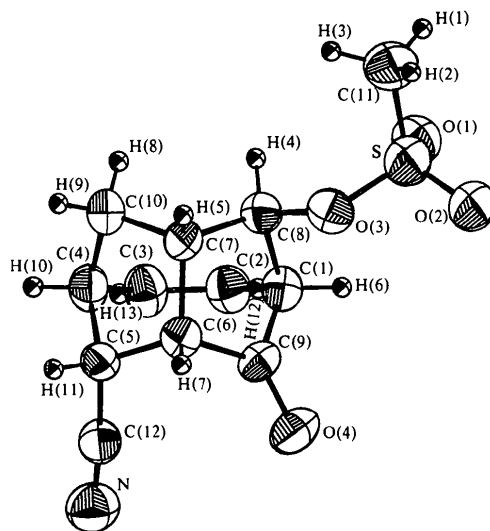


Fig. 1. The molecular structure and numbering of the atoms.

Table 3. Bond lengths (\AA) involving the non-hydrogen atoms

S—O(1)	1.430 (8)	C(2)—C(3)	1.309 (15)
S—O(2)	1.454 (9)	C(3)—C(4)	1.482 (15)
S—O(3)	1.529 (10)	C(4)—C(5)	1.542 (14)
S—C(11)	1.778 (14)	C(4)—C(10)	1.546 (15)
N—C(12)	1.104 (14)	C(5)—C(6)	1.563 (13)
O(3)—C(8)	1.496 (12)	C(5)—C(12)	1.432 (13)
O(4)—C(9)	1.212 (11)	C(6)—C(7)	1.544 (14)
C(1)—C(2)	1.566 (14)	C(6)—C(9)	1.533 (14)
C(1)—C(8)	1.502 (13)	C(7)—C(8)	1.529 (14)
C(1)—C(9)	1.483 (14)	C(7)—C(10)	1.539 (14)

Table 4. Bond angles ($^\circ$) involving the non-hydrogen atoms

O(1)—S—O(2)	117.3 (5)	C(4)—C(5)—C(12)	114.4 (8)
O(1)—S—O(3)	107.4 (5)	C(6)—C(5)—C(12)	115.9 (8)
O(1)—S—C(11)	108.3 (6)	C(5)—C(6)—C(7)	104.6 (7)
O(2)—S—O(3)	111.6 (5)	C(5)—C(6)—C(9)	111.3 (7)
O(2)—S—C(11)	107.1 (5)	C(7)—C(6)—C(9)	103.6 (7)
O(3)—S—C(11)	103.9 (5)	C(6)—C(7)—C(8)	105.8 (7)
S—O(3)—C(8)	119.4 (6)	C(6)—C(7)—C(10)	105.7 (8)
C(2)—C(1)—C(8)	111.7 (8)	C(8)—C(7)—C(10)	110.8 (8)
C(2)—C(1)—C(9)	106.6 (7)	O(3)—C(8)—C(1)	106.7 (7)
C(8)—C(1)—C(9)	102.2 (7)	O(3)—C(8)—C(7)	104.8 (7)
C(1)—C(2)—C(3)	125.2 (9)	C(1)—C(8)—C(7)	105.7 (8)
C(2)—C(3)—C(4)	129.2 (10)	O(4)—C(9)—C(1)	127.3 (9)
C(3)—C(4)—C(5)	113.1 (8)	O(4)—C(9)—C(6)	124.4 (9)
C(3)—C(4)—C(10)	112.2 (9)	C(1)—C(9)—C(6)	108.2 (8)
C(5)—C(4)—C(10)	99.5 (8)	C(4)—C(10)—C(7)	104.7 (8)
C(4)—C(5)—C(6)	103.8 (7)	N—C(12)—C(5)	177.2 (11)

We wish to thank Professor K. Venkatesan, Department of Organic Chemistry, Indian Institute of Science, Bangalore, for useful discussions and for suggesting the problem. We wish to thank Bangalore University for providing financial assistance.

References

- BUERGER, M. J. (1959). *Vector Space and Its Applications in Crystal Structure Investigations*. New York: John Wiley.
- CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104–109.
- GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* **A27**, 368–376.
- GOVERDHAN MEHTA, GHOSH, P., CHAUDHURI, B., SINGH, V. K., USHA, R., VARUGHESE, K. I. & VENKATESAN, K. (1977). *Tetrahedron Lett.* **47**, 4109–4112.
- SHELDRIK, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.
- STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Acta Cryst. (1981). **B37**, 1790–1793

Fenfluramine Hydrochloride, (\pm)-*N*-Ethyl-*m*-(trifluoromethyl)amphetamine Hydrochloride*

BY GARY L. GRUNEWALD† AND MARY W. CREESE

Department of Medicinal Chemistry, The University of Kansas, Lawrence, Kansas 66045, USA

AND MICHAEL W. EXTINE

Molecular Structure Corporation, PO Box DF, College Station, Texas 77840, USA

(Received 22 September 1980; accepted 30 March 1981)

Abstract. C₁₂H₁₇ClF₃N, $M_r = 267.72$, orthorhombic, *Pbca*, $Z = 8$, $a = 7.302(1)$, $b = 26.829(6)$, $c = 14.366(2)$ Å, $D_c = 1.264$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu(\text{Cu } K\alpha) = 2.57$ mm⁻¹, $R = 0.067$ for 705 observed reflections after anisotropic refinement of all non-H atoms. The solid-state conformations of the fenfluramine and amphetamine cations are the same.

Introduction. Single crystals of racemic fenfluramine hydrochloride were obtained by recrystallization from ethanol of a sample provided by A. H. Robins Co., Inc., Richmond, Virginia. A colourless prismatic crystal measuring 0.05 × 0.05 × 0.2 mm was used for the study. The symmetry was orthorhombic, and systematic absences were consistent with the space group *Pbca*. Intensity data were collected using an Enraf-Nonius CAD-4 diffractometer, a graphite monochromator, Cu $K\alpha$ radiation, and the θ - 2θ scanning technique. Intensities were measured for 2513 independent reflections with $2\theta \leq 120^\circ$. The intensity data were reduced to structure factors and corrected for Lorentz and polarization effects. No absorption or extinction corrections were necessary. The structure was determined by direct methods using *MULTAN*

(Germain, Main & Woolfson, 1971), and refined by a full-matrix least-squares procedure. Anisotropic thermal parameters were used for the non-H atoms; the H atoms were fixed in calculated positions (C–H = 0.95 Å), with $B_{\text{iso}} = 8.0$ Å², and not refined. The 705 reflections having $F_o^2 > 2\sigma(F_o^2)$ were used in the refinement. In the last cycle of refinement all parameter shifts were less than 0.36σ ; $R = \sum |F_o| - |F_c| / \sum |F_o| = 0.067$, and $R' = [\sum w(|F_o| - |F_c|)^2]^{1/2} = 0.76$. The e.s.d. of an observation of unit weight was 1.549.

The molecular structure and the atom-numbering system are shown in Fig. 1. The final atomic coordinates are given in Table 1, and the bond lengths and angles in Tables 2 and 3 respectively.‡ The Enraf-Nonius structure-determination package was used for all the calculations.

Discussion. Fenfluramine produces some, but not all, of the pharmacological effects of amphetamine and related phenethylamines. Like the latter compounds it is effective as an appetite depressant (Le Douarec &

* Amphetamine is α -methylphenethylamine.

† To whom correspondence should be addressed.

‡ Lists of structure factors and thermal parameters and Table 4 have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36092 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.