C4—N1 bond, while in (I-11) it bisects the N1—C1 bond. Asymmetry parameters (Duax & Norton 1975) are, respectively, ΔC_2(C4—N1) = 0·76 for (I-6), 1·2 for (I-10) and ΔC_2(C1—N1) = 0·65 for (I-11). It follows that, from the viewpoint of the structure, the molecules of compounds (I-10) and (I-11) differ only in the symmetry of the five-membered rings. The bond lengths and angles in the three structures have expected values.

In conclusion, it follows from our studies that for the presence of anticonvulsant properties the pyridyl ring must be anticlinal with respect to the succinimide moiety. This position can be achieved by the *ortho* substitution of a methyl group into the pyridyl ring. This, in turn, facilitates the formation of hydrogen bonds with a receptor. The presence of a Cl substituent in the phenyl ring influences for instance the reduction of (I-11) toxicity, but in principle does not change the conformation of the molecule.

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Structure of *N,N,N′,N′*-Tetrakis(2-fluoro-2,2-dinitroethyl)oxamide by the Consistent Electron Density Approach

BY T. NARAYANA BHAT*

Laboratory of Molecular Biology, National Institutes of Health, Bethesda, Maryland 20892, *USA*

AND HERMAN L. AMMON

Department of Chemistry and Biochemistry and Center for Advanced Research in Biotechnology, University of Maryland, College Park, Maryland 20742, *USA*

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Abstract. $C_{10}H_8F_4N_{10}O_{18}$, $M_r = 632·22$, monoclinic, *Pc*, $a = 7·8885$ (6), $b = 6·7787$ (4), $c = 21·595$ (2) Å, $\beta = 108·21$ (1)°, $V = 1096$ (1) Å^3, $Z = 2$, $D_x = 1·914$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 18·7$ cm^{-1}, $F(000) = 636$, $T = 293$ K, 1791 unique data, 1733 with $I > 3\sigma(I)$, $R = 0·047$. The structure was solved by the consistent electron density approach (CEDA), in which a small starting set of random phases was refined and expanded by the application of restraints to the electron density. A refinable preliminary structure was obtained by fitting a model to a 3 Å map, calculated with 27 reflections (all data with $d > 3$ Å, and $|F_o| > 170$) whose phases had been determined by the CEDA. The molecule has an extended, open conformation; the two pairs of fluorodinitroethyl substituents located across the molecular center from each other show *i* and C_2 pseudo-symmetry respectively.

Introduction. The fluorodinitromethyl group is of interest as a substituent in energetic materials such as explosives and propellants. It has been called an 'explosophore', a term that has a meaning similar to that of 'chromophore' for appropriate functionality. At the University of Maryland we have been examin-

*Present address: Département d'immunologie, Institut Pasteur, 75724 Paris CEDEX 15, France.

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ing the crystal structures of a number of high density, poly-nitro group containing organic compounds as a preliminary step in an investigation of the relationships between structure and crystal density in energetic substances. This paper describes the structure solution of (I) [X_2N—C(=O)—C(=O)—NX_2, $X = CH_2$—$CF(NO_2)_2$] by the consistent electron density approach (CEDA) (Bhat, 1984, 1985).

Experimental. Compound (I) was obtained from Dr M. Sitzman, Naval Surface Weapons Center, Silver Spring, Maryland. Crystals were grown from chloroform/hexane solution. A 0·07 × 0·2 × 0·5 mm colorless, needle-like crystal was used for a preliminary photographic survey and all diffractometer measurements. Picker FACS-I diffractometer, Cu $K\alpha$ radiation, graphite monochromator. Cell parameters from 12 reflections centered manually at 2θ. Data collection with a 2θ–θ scan, 2θ scan speed $2°$ min^{-1}, 2θ scan width $(1{\cdot}4 + 0{\cdot}3\tan\theta)°$, 20 s backgrounds; h, k, l range of 0 to −9, 0 to −7, −25 to −23; four standards measured every 100 reflections, average and maximum intensity deviations of 0·3 and 1·1%, 2212 reflections measured to $2\theta_{max} = 127°$, 1791 unique data excluding systematic absences, 1733 with $I > 3\sigma(I)$.

In the CEDA (Bhat, 1984, 1985) the electron density map obtained from a starting set of phases is modified (Hope & Gassman, 1968; reviews *e.g.* Tulinsky, 1985; Wang, 1985; Podjarny, Bhat & Zwick, 1987), and converted into an OMIT map (Bhat, 1988; Bhat & Cohen, 1984), which itself is modified, and the overall procedure of modified map → OMIT map → modified map → OMIT map is iterated several times. When the procedure has converged for data at some resolution, the final OMIT map can be examined and perhaps interpreted by the fitting of a molecular model, or the procedure can be continued with the inclusion of additional reflections. If the electron density map is not interpretable, the calculations may also be continued with a different type of modification function for the electron density.

The application of the CEDA to (I) is described below.

(*a*) Initial random phase set. Seventeen strongest reflections (all with $|F_o| > 490$ and with no limit on resolution) were selected. (Inadvertently the $\bar{1}27$, $|F_o| = 590$ was omitted and the 113, $|F_o| = 350$, and 018, $|F_o| = 425$ were included in this set.) This is the starting set H to which random phases were assigned. Reflection phase set H was used to calculate the initial electron density map in step (*b*).

(*b*) Calculation of an electron density map. A map (map_1) was calculated with 28, 26 and 76 grid divisions (*ca* 0·25 Å spacing along the *a*, *b* and *c* unit-cell directions).

(*c*) Electron density modification. The electron density values in map_1 below −1·25 root-mean-square (r.m.s) were set to −1·25 r.m.s., and a value of 1·25 r.m.s. was added to all the electron density values and then the values were squared ($map_1 \rightarrow map_2$).

(*d*) Calculation of an OMIT map. An OMIT map (map_3) was formed (Bhat, 1988) from map_2 with the following values for the dimensions of the electron density boxes. The dimensions of the 'phased volume' boxes were 8 × 6 × 16 grid divisions along the three unit-cell directions. These dimensions, if required, were changed by the computer program at the unit-cell edges. Each phased volume was surrounded with a 'neutral volume' made up of a four grid division extension in each direction.

(*e*) OMIT map → density modification → OMIT map cycle. This cycle was performed by the use of map_3 as map_1 in the density modification step (*c*). Steps (*c*) and (*d*) were repeated for several (*e.g.* 50) cycles. The r.m.s. threshold values for restricting the negative density in step (*c*), the dimensions of the 'phased' and 'netural' volumes in step (*d*) and the reflections (set H) used in the OMIT map calculations were unchanged during all the step-(*c*)-to-(*d*) iterations.

(*f*) Phase extension to include smaller $|F_o|$. Phase extension was carried out by the inverse Fourier transform of an OMIT map [map_3, step (*d*)] followed by the addition of a new set of reflection/phases (H') to set H. This expanded reflection set ($H + H'$) was used to calculate map_1 in step (*b*), and steps (*c*) through (*e*) were repeated. The H' reflection set consisted of all of the amplitudes above some threshold. Overall, step (*f*) was repeated 12 times to obtain phases for a total of 147 reflections (this set contained all the reflections with $|F_o| > 170$, with no limit on resolution).*

(*g*) Use of a molecular mask as a restraint. A 3 Å electron density map (map_4) was used as a mask for map_3. Electron density in map_3 was set to −1·25 r.m.s. if either the map_3 density < −1·25 r.m.s. or the map_4 density < −1·0 r.m.s.

(*h*) Steps (*c*) and (*d*) were completed using map_3 as map_1.

(*i*) Steps (*g*) and (*h*) were repeated 20 times.

(*j*) Model building and structure solution. A map was calculated from all the (27) reflections within 3 Å and $|F_o| > 170$, and a model was fitted with the graphics program *FRODO* (Jones, 1978; Pflugrath Saper & Quiocho, 1984). This model was refined by the least-squares procedure with the *SHELX* system (Sheldrick, 1976) with restraints on bond distances.

* See depostion footnote.

The electron density map calculated from all the 147 reflections was not interpretable.

The final structure refinement was by full-matrix least squares, $\sum[(|F_o|-|F_c|)/\sigma(|F_o|)]^2$, reflections with $I<3\sigma(I)$ omitted; atomic scattering factors were obtained from *International Tables for X-ray Crystallography* (1974*a*) and included dispersion corrections (*International Tables for X-ray Crystallography*, 1974*b*); anisotropic temperature factors for C, N, O and F; H positions calculated from the geometry of the C atom skeleton and refined with isotropic terms; isotropic secondary extinction refined, value used = 0·14 (3) × 10^{-5}; maximum shift/e.s.d. in final least-squares cycle of 0·04; minimum and maximum density in final difference map of −0·27 and 0·71 e Å^{-3}; final *R*, *wR* and *S* of 0·047, 0·060, 3·77. $w = 1/\sigma^2(F_o)$. These final calculations were carried out with the *TEXSAN* system (Molecular Structure Corporation, 1986) on a Digital Equipment Corporation MicroVAX II computer. Atomic coordinates and thermal parameters are given in Table 1.*

Table 1. *Fractional coordinates and equivalent isotropic temperature factors* (Å^2), *with e.s.d.'s* (*in parentheses*) *for* (I)

	x	*y*	*z*	B_{eq}
F1	−0·5952	0·5750 (5)	−0·0824	4·2 (1)
F2	−0·3455 (6)	0·2958 (4)	−0·1187 (3)	4·2 (1)
F3	0·0549 (7)	0·6492 (7)	0·1743 (3)	6·0 (2)
F4	−0·1721 (7)	1·2908 (5)	0·0877 (3)	4·8 (2)
O1	−0·0444 (7)	0·5820 (5)	0·0088 (2)	3·5 (1)
O2	−0·4079 (7)	0·8228 (7)	0·0195 (3)	4·8 (2)
O3	−0·8484 (9)	0·694 (1)	−0·2121 (3)	7·5 (3)
O4	−0·596 (1)	0·580 (1)	−0·2147 (3)	7·5 (3)
O5	−0·7079 (8)	1·0482 (7)	−0·1302 (3)	6·3 (2)
O6	−0·8120 (8)	0·842 (1)	−0·0731 (3)	6·1 (2)
O7	−0·091 (1)	0·3625 (8)	−0·2111 (3)	7·5 (3)
O8	−0·176 (1)	0·0884 (7)	−0·1837 (3)	6·3 (2)
O9	0·1066 (8)	0·3061 (7)	−0·0584 (3)	4·9 (2)
O10	−0·094 (1)	0·1451 (7)	−0·0299 (3)	5·3 (2)
O11	0·3289 (8)	0·668 (1)	0·0831 (4)	7·3 (3)
O12	0·225 (1)	0·4203 (6)	0·1320 (3)	6·0 (2)
O13	0·271 (1)	0·976 (1)	0·2239 (3)	10·0 (4)
O14	0·4666 (9)	0·778 (1)	0·2119 (4)	7·4 (3)
O15	−0·317 (1)	1·4764 (7)	0·1617 (3)	6·8 (3)
O16	−0·298 (1)	1·2110 (9)	0·2162 (3)	7·5 (3)
O17	−0·479 (1)	1·252 (1)	0·0101 (3)	8·1 (3)
O18	−0·5770 (8)	1·1186 (8)	0·0826 (3)	6·1 (2)
N1	−0·2622 (7)	0·6865 (6)	−0·0807 (3)	2·6 (1)
N2	−0·1285 (7)	0·8904 (6)	0·0833 (3)	2·7 (1)
N3	−0·7186 (8)	0·8893 (8)	−0·1062 (3)	4·3 (2)
N4	−0·689 (1)	0·6627 (9)	−0·1889 (3)	4·6 (2)
N5	−0·1512 (9)	0·2642 (7)	−0·1781 (3)	3·8 (2)
N6	−0·0423 (8)	0·2617 (7)	−0·0631 (3)	3·4 (2)
N7	0·249 (1)	0·588 (1)	0·1154 (4)	5·6 (3)
N8	0·317 (1)	0·8442 (8)	0·1979 (3)	4·5 (2)
N9	−0·297 (1)	1·2993 (8)	0·1697 (3)	4·2 (2)
N10	−0·4640 (9)	1·1874 (8)	0·0627 (3)	4·4 (2)
C1	−0·1737 (8)	0·6862 (7)	−0·0150 (3)	2·6 (2)
C2	−0·2470 (8)	0·8090 (7)	0·0299 (3)	2·8 (2)
C3	−0·4096 (8)	0·8205 (7)	−0·1111 (3)	2·9 (2)
C4	−0·5932 (8)	0·7324 (8)	−0·1183 (3)	3·0 (2)
C5	−0·1747 (8)	0·5837 (7)	−0·1215 (3)	2·7 (2)
C6	−0·1874 (8)	0·3606 (7)	−0·1192 (3)	2·7 (2)
C7	0·0644 (8)	0·9067 (7)	0·0940 (3)	2·7 (2)
C8	0·167 (1)	0·7501 (8)	0·1419 (3)	4·3 (2)
C9	−0·2003 (9)	0·9783 (8)	0·1320 (3)	3·0 (2)
C10	−0·2737 (9)	1·1840 (8)	0·1126 (3)	3·2 (2)

Discussion. The structure was solved from a model built, using stereochemical information of the expected chemical compound, on to a 3 Å electron density map calculated with the strongest reflections ($|F_o|>170$) and CEDA phases. Fig. 1 shows this 3 Å map along with the map calculated from the final phases from the refined molecule. A comparison of the CEDA derived phases and the final phases is complicated by the possible origin differences. This was addressed by moving the final refined model in the *X,Z* plane to a position which minimized the difference between the two phase sets. The phase difference was 67°; which has been subsequently improved to 47°. These phases were obtained without the use of an atomic model.

The 3 Å set of 27 reflections phased by the CEDA had seven reflections in common with the starting set of 17 (set *H*, step *a*). The remaining 20 reflections were introduced during the 12 phase extensions. During the phase extension and improvement cycles, the phases of these seven common reflections changed by a mean of 50° (range of 6–90°).

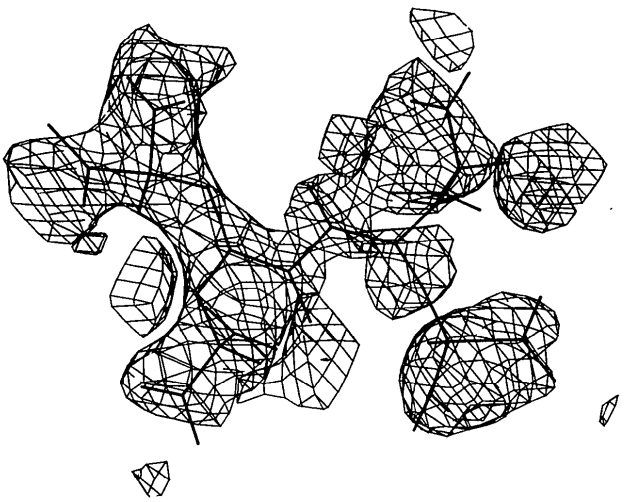
An *ORTEP* (Johnson, 1971) drawing of the molecule is shown in Fig. 2. and bond lengths and angles are listed in Table 2. The view in Fig. 2, which is perpendicular to the six-atom central oxamide plane, shows that the four fluorodinitroethyl substituents are arranged in two symmetry-related patterns about the center of the molecule. The two more-or-less vertical substituents show approximate C_2 symmetry through the center of the molecule while the horizontal groups show approximate inversion symmetry. The oxamide central C—C bond is twisted by about 40°; the O1—C1—C2—O2 and N1—C1—C2—N2 dihedral angles are 138·5 (6) and 146·7 (5)°, respectively. This twist together with the *s-trans* oxamide geometry places all four CH_2 groups on the same side of the oxamide plane. The C1—N1 and C2—N2 bonds are twisted by about 8 and 11°, respectively. All of the sp^2-hybridized C and N atoms are planar within experimental error.

Bond lengths and angles and the fluorodinitroethyl group conformations are similar to those previously observed in compounds containing this substituent, such as 1,4-difluoro-1,1,4,4-tetranitro-2,3-dinitrooxybutane (Ammon & Bhattacharjee, 1982), bis(2-fluoro-2,2-dinitroethyl)nitramine (Ammon, Bhattacharjee, Hall & Skelton, 1983) and 1,2-bis-(fluorodinitromethyl)-1,3-dioxolan-2-one (Ammon & Bhattacharjee, 1984). Several of the bond lengths and angles associated with the C8-linked substituent show atypical values. One finds, for example,

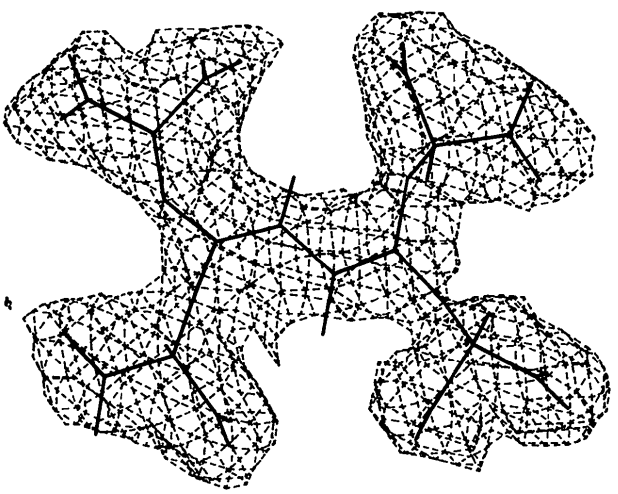
* Lists of reflections used in the CED calculations, structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52162 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C8—F3 = 1·460 (8), C8—N7 = 1·475 (9) Å and O11—N7—O12 = 138·0 (7)°, all of which are substantially different from the corresponding units in other sections of (I) and in other molecules. It should be noted that the largest peak in the difference electron density map is associated with this region of the molecule. The intermolecular contacts are ordinary and there are no close approaches that might rationalize the discrepancy.

It should be noted that our preliminary attempts to determine the structure of (I) by *MULTAN* (Main *et.al.*, 1980) and *MITHRIL* (Gilmore, 1983) were unsuccessful and that this avenue of attack was then discontinued to 'save' the structural problem for the



(*a*)



(*b*)

Fig. 1. The electron density map calculated from the 27 strongest reflections out to 3 Å: (*a*) with the phases determined by the CEDA; (*b*) with the phases calculated from the final refined model. Molecular models are also shown. Contour levels are adjusted so that the two figures display approximately the same number of vectors.

Table 2. *Bond distances* (Å) *and angles* (°), *with e.s.d.'s* (*in parentheses*), *for* (I)

F1—C4	1·321 (6)	O18—N10	1·199 (8)
F2—C6	1·325 (6)	N1—C1	1·371 (6)
F3—C8	1·460 (8)	N1—C5	1·455 (6)
F4—C10	1·312 (6)	N1—C3	1·460 (6)
O1—C1	1·215 (6)	N2—C2	1·355 (7)
O2—C2	1·221 (7)	N2—C9	1·467 (6)
O3—N4	1·220 (8)	N2—C7	1·470 (6)
O4—N4	1·192 (8)	N3—C4	1·529 (7)
O5—N3	1·210 (8)	N4—C4	1·548 (7)
O6—N3	1·219 (7)	N5—C6	1·533 (6)
O7—N5	1·178 (7)	N6—C6	1·537 (7)
O8—N5	1·208 (7)	N7—C8	1·475 (9)
O9—N6	1·187 (7)	N8—C8	1·541 (8)
O10—N6	1·219 (7)	N9—C10	1·521 (7)
O11—N7	1·207 (9)	N10—C10	1·551 (7)
O12—N7	1·223 (8)	C1—C2	1·523 (7)
O13—N8	1·174 (8)	C3—C4	1·529 (7)
O14—N8	1·207 (8)	C5—C6	1·517 (6)
O15—N9	1·216 (8)	C7—C8	1·527 (7)
O16—N9	1·169 (7)	C9—C10	1·518 (7)
O17—N10	1·190 (8)		

C1—N1—C5	116·2 (4)	N1—C1—C2	119·1 (4)
C1—N1—C3	122·8 (4)	O2—C2—N2	121·7 (5)
C5—N1—C3	119·2 (4)	O2—C2—C1	120·3 (5)
C2—N2—C9	117·2 (4)	N2—C2—C1	117·9 (4)
C2—N2—C7	124·6 (4)	N1—C3—C4	113·5 (4)
C9—N2—C7	118·0 (4)	F1—C4—C3	116·1 (4)
O5—N3—O6	128·6 (6)	F1—C4—N3	109·6 (4)
O5—N3—C4	114·2 (5)	F1—C4—N4	103·8 (4)
O6—N3—C4	117·2 (5)	C3—C4—N3	110·7 (4)
O4—N4—O3	126·6 (6)	C3—C4—N4	111·9 (4)
O4—N4—C4	115·1 (5)	N3—C4—N4	103·8 (4)
O3—N4—C4	118·3 (6)	N1—C5—C6	114·2 (4)
O7—N5—O8	125·5 (5)	F2—C6—C5	113·7 (4)
O7—N5—C6	118·3 (5)	F2—C6—N5	106·5 (4)
O8—N5—C6	116·0 (5)	F2—C6—N6	108·6 (4)
O9—N6—O10	128·3 (5)	C5—C6—N5	111·6 (4)
O9—N6—C6	115·4 (5)	C5—C6—N6	114·8 (4)
O10—N6—C6	116·2 (5)	N5—C6—N6	100·5 (4)
O11—N7—O12	138·0 (7)	N2—C7—C8	111·0 (4)
O11—N7—C8	104·9 (6)	F3—C8—N7	103·9 (5)
O12—N7—C8	117·1 (7)	F3—C8—C7	112·1 (5)
O13—N8—O14	126·0 (6)	F3—C8—N8	104·4 (5)
O13—N8—C8	114·4 (6)	N7—C8—C7	117·4 (5)
O14—N8—C8	119·6 (5)	N7—C8—N8	107·0 (5)
O16—N9—O15	126·4 (5)	C7—C8—N8	111·1 (5)
O16—N9—C10	117·8 (5)	N2—C9—C10	112·0 (4)
O15—N9—C10	115·8 (5)	F4—C10—C9	113·0 (4)
O17—N10—O18	128·8 (7)	F4—C10—N9	107·1 (4)
O17—N10—C10	116·9 (6)	F4—C10—N10	106·9 (4)
O18—N10—C10	114·3 (5)	C9—C10—N9	112·4 (4)
O1—C1—N1	121·8 (4)	C9—C10—N10	114·1 (4)
O1—C1—C2	118·9 (4)	N9—C10—N10	102·6 (4)

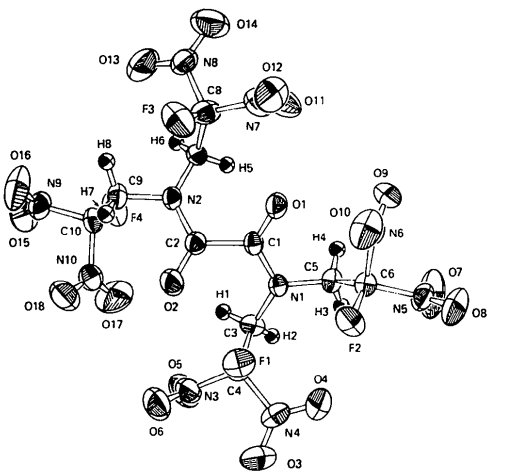


Fig. 2. *ORTEP* drawing normal to the oxamide plane. The C, O, N and F atoms are displayed as 50% probability ellipsoids and the H atoms as spheres with $B = 1·5$ Å^2.

CEDA. Following the completion of the structure refinement, the reflection data were given to Dr G. M. Sheldrick who solved the structure with some difficulty with the *SHELX* system (Sheldrick, 1985). He has suggested that the difficulties in solving the structure with *MULTAN* and *MITHRIL* were probably associated with the molecular pseudo-symmetry.

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Structure of 5-Hydroxy-5-phenyl-7-azatricyclo[7.4.0.0^{2,7}]trideca-2,9(1),10,12-tetraen-8-one by the Consistent Electron Density Approach

By T. Narayana Bhat*

Laboratory of Molecular Biology, National Institutes of Health, Bethesda, Maryland 20892, *USA*

Herman L. Ammon

Department of Chemistry and Biochemistry and Centre for Advanced Research in Biotechnology, University of Maryland, College Park, Maryland 20742, *USA*

and Paul H. Mazzocchi and Mazuaki Oda

Department of Chemistry and Biochemistry, University of Maryland, College Park, Maryland 20742, *USA*

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Abstract. $C_{18}H_{15}NO_2$, $M_r = 277{\cdot}3$, monoclinic, $P2_1/n$, $a = 7{\cdot}408$ (2), $b = 22{\cdot}311$ (7), $c = 8{\cdot}613$ (2) Å, $\beta = 103{\cdot}53$ (3)°, $V = 1384$ (1) Å^3, $Z = 4$, $D_x = 1{\cdot}33$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}94$ cm^{-1}, $F(000) = 594$, $T = 293$ K, 2229 unique diffractometer data, 963 with $I > 3\sigma(I)$, $R = 0{\cdot}050$. Structure solution was by the consistent electron density approach

* Present address: Département d'immunologie, Institut Pasteur, 75724 Paris CEDEX 15, France.

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