

The pK_a value for (E)-3-fluoro-4-PAM was found by potentiometric titration to be 8.2. Therefore, this compound meets the empirical pK_a requirement for rapid reactivation of phosphonylated acetylcholinesterase. Since the corresponding pK_a value of the nonfluorinated analog (4-PAM) is 8.6 (Ginsburg & Wilson, 1957; Poziomek, Kramer, Mosher & Michel, 1961), substitution of fluorine for hydrogen at the 3-position of the pyridine ring decreases the pK_a by 0.4 pK_a units, an effect expected from the electron-withdrawing properties of the fluorine atom. Preliminary studies indicate that the fluorine-substituted compound has an increased rate of dephosphorylation of enzyme relative to the unsubstituted parent compound, but less than that of 2-PAM (in spite of the similarities in pK_a values). Thus, except for the fact that the fluorine substitution causes more stringent coplanarity between the oxime group and the pyridine ring, the effect of fluorine is confined to lowering the pK_a value.

This research was supported by grants CA-10925, CA-06927, RR-05539 and CA-22780 from the National Institutes of Health, US Public Health Service, BC-242 from the American Cancer Society, the US Army Medical Research and Development Command (DAMD 17-81-C-1160) and by an appropriation from the Commonwealth of Pennsylvania.

EK is the recipient of a Research Career Award of the US Public Health Service.

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Acta Cryst. (1985). **C41**, 788–791

Structure of 6,6,10,10-Tetranitropentacyclo[5.3.0.0^{2,5}.0^{3,9}.0^{4,8}]decane, C₁₀H₈N₄O₈

BY CLIFFORD GEORGE, RICHARD GILARDI AND JUDITH L. FLIPPEN-ANDERSON

Laboratory for the Structure of Matter, Naval Research Laboratory, Washington, DC 20375, USA

CHANG S. CHOI*

Energetic Materials Division, US Army AMCCOM, Dover, NJ 07801, USA

AND ALAN P. MARCHAND AND D. SIVAKUMAR REDDY

Department of Chemistry, North Texas State University, Box 5068, Denton, TX 76203, USA

(Received 26 July 1984; accepted 6 December 1984)

Abstract. $M_r = 312.20$, monoclinic, Pc , $a = 7.761$ (1), $b = 11.500$ (2), $c = 14.277$ (2) Å, $\beta = 108.63$ (2)°, $V = 1207.5$ (3) Å³, $Z = 4$, $D_x = 1.717$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.113$ mm⁻¹, $F(000) =$

640, $T = 295$ K. Final $R = 0.041$, $wR = 0.042$, for 2457 independent observed reflections. The carbon skeleton (bishomocubane) consists of four puckered five-membered rings and two four-membered rings which are bent about their diagonals by approximately 20°. The two molecules in the asymmetric unit are nearly identical, with the primary difference being the orientations of the NO₂ groups.

* Mailing address: National Bureau of Standards, Reactor Division, Gaithersburg, MD 20899, USA.

Introduction. The title compound, a tetranitrobishomocubane (TNBHC), was prepared as part of a program to synthesize strained energetic compounds (Marchand & Reddy, 1984). The structure (Ammon, Zhang, Choi, Sandus, Marchand & Suri, 1985) and synthesis of a trinitrobishomocubane have been reported previously (Marchand & Suri, 1984). The structure of this compound was investigated as part of a continuing study of the structural parameters of energetic substituents in polycyclic nitro compounds. The structural parameters provide a basis to evaluate and modify the semi-empirical parameters used for computational modelling of proposed but not yet synthesized compounds.

Experimental. Clear, $0.40 \times 0.25 \times 0.60$ mm crystal, recrystallized from ethyl acetate-hexane, m.p. 428–429 K. Automated Nicolet R3m diffractometer with incident-beam graphite monochromator, $\lambda = 0.71069$ Å (Mo K α). 24 centered reflections within $20^\circ < 2\theta < 30^\circ$ used for determining lattice parameters. Lorentz and polarization but not absorption corrections applied, $(\sin\theta/\lambda)_{\max} = 0.65$ Å $^{-1}$, range of hkl : $-10 \leq h \leq 4$, $0 \leq k \leq 14$, $-18 \leq l \leq 18$; standards 300, 064, 006 monitored every 60 reflections with random variation of 2% over data collection; θ - 2θ mode, scan width 2° , scan rate a function of count rate (4° min^{-1} minimum, $30^\circ \text{ min}^{-1}$ maximum). 4330 reflections measured, 2790 unique, $R_{\text{int}} = 0.007$, 2457 observed $F_o > 3\sigma(F_o)$. Structure solved by direct methods (Karle & Karle, 1966) with use of partial-structure recycling (Karle, 1968) from 5- and 6-atom fragments from the two independent molecules in the asymmetric unit. The least-squares refinement used program *SHELXTL* (Sheldrick, 1980). H atoms located in difference maps. $\sum w(|F_o| - |F_c|)^2$ minimized where $w = 1/[\sigma^2(|F_o|) + g(F_o)^2]$ and $g(F_o)^2$ is included to account for random instrumental error (g estimated to be 0.0003). 461 parameters refined: atom coordinates, anisotropic temperature factors for all non-H atoms, isotropic temperature factors for H, a maximum of 103 parameters refined each least-squares cycle with subset of coordinates fixed each cycle. $R = 0.041$, $wR = 0.042$, $S = 1.43$, $(\Delta/\sigma)_{\max} = 0.09$. Final difference Fourier excursions 0.22 and $-0.20 e \text{ Å}^{-3}$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Discussion. Table 1 lists the refined coordinates and the equivalent isotropic U_{eq} values;* bond distances and angles are given in Table 2.

* Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39943 (18 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Final coordinates and equivalent isotropic thermal parameters

$$U_{\text{eq}} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i a_j$$

Molecule 1	x	y	z	$U_{\text{eq}}(\text{Å}^2)$
C(1)	0.1148 (4)	0.4797 (2)	0.3476 (2)	0.036 (1)
C(2)	0.1176 (3)	0.5748 (2)	0.4896 (2)	0.032 (1)
C(3)	0.2990 (4)	0.6148 (2)	0.4763 (2)	0.030 (1)
C(4)	0.3123 (4)	0.5276 (2)	0.3954 (2)	0.035 (1)
C(5)	0.3684 (4)	0.4103 (3)	0.4533 (2)	0.043 (1)
C(6)	0.1626 (4)	0.3836 (3)	0.4256 (2)	0.043 (1)
C(7)	0.1678 (4)	0.4477 (2)	0.5229 (2)	0.039 (1)
C(8)	0.3805 (4)	0.4436 (2)	0.5597 (2)	0.040 (1)
C(9)	-0.0015 (4)	0.5642 (2)	0.3805 (2)	0.035 (1)
C(10)	0.4430 (4)	0.5682 (2)	0.5685 (2)	0.035 (1)
N(1)	-0.0258 (3)	0.6800 (2)	0.3265 (2)	0.042 (1)
N(2)	-0.1939 (3)	0.5203 (3)	0.3642 (2)	0.046 (1)
N(3)	0.6338 (3)	0.5813 (2)	0.5632 (2)	0.042 (1)
N(4)	0.4479 (3)	0.6297 (2)	0.6641 (2)	0.040 (1)
O(1)	0.0098 (4)	0.6828 (2)	0.2504 (2)	0.069 (1)
O(2)	-0.0748 (4)	0.7610 (2)	0.3636 (2)	0.073 (1)
O(3)	-0.2077 (4)	0.4379 (3)	0.4124 (2)	0.074 (1)
O(4)	-0.3179 (3)	0.5671 (3)	0.3020 (2)	0.078 (1)
O(5)	0.6628 (3)	0.6561 (2)	0.5113 (2)	0.060 (1)
O(6)	0.7473 (3)	0.5135 (2)	0.6116 (2)	0.069 (1)
O(7)	0.5613 (4)	0.7035 (3)	0.6954 (2)	0.069 (1)
O(8)	0.3336 (3)	0.6006 (2)	0.6998 (2)	0.059 (1)
Molecule 2				
C(1)'	-0.2923 (4)	-0.1079 (3)	-0.0476 (2)	0.043 (1)
C(2)'	-0.3216 (4)	0.0286 (2)	0.0631 (2)	0.033 (1)
C(3)'	-0.1545 (4)	0.0739 (2)	0.0368 (2)	0.033 (1)
C(4)'	-0.1128 (4)	-0.0350 (2)	-0.0177 (2)	0.039 (1)
C(5)'	-0.0234 (4)	-0.1223 (3)	0.0689 (2)	0.045 (1)
C(6)'	-0.2176 (4)	-0.1699 (3)	0.0520 (2)	0.044 (1)
C(7)'	-0.2341 (4)	-0.0769 (3)	0.1291 (2)	0.038 (1)
C(8)'	-0.0239 (4)	-0.0556 (3)	0.1619 (2)	0.040 (1)
C(9)'	-0.4352 (4)	-0.0252 (2)	-0.0348 (2)	0.039 (1)
C(10)'	-0.0044 (4)	0.0693 (3)	0.1359 (2)	0.033 (1)
N(1)'	-0.5026 (3)	0.0656 (2)	-0.1153 (2)	0.051 (1)
N(2)'	-0.6031 (3)	-0.0907 (2)	-0.0309 (2)	0.048 (1)
N(3)'	0.1843 (3)	0.0968 (2)	0.1286 (2)	0.043 (1)
N(4)'	-0.0337 (4)	0.1585 (2)	0.2072 (2)	0.043 (1)
O(1)'	-0.4198 (3)	0.0764 (2)	-0.1726 (2)	0.088 (1)
O(2)'	-0.6321 (4)	0.1217 (2)	-0.1135 (2)	0.087 (1)
O(3)'	-0.6086 (4)	-0.1219 (4)	0.0474 (2)	0.091 (7)
O(4)'	-0.7102 (4)	-0.1182 (3)	-0.1064 (2)	0.108 (1)
O(5)'	0.1927 (4)	0.1421 (3)	0.0560 (2)	0.074 (1)
O(6)'	0.3106 (5)	0.0659 (3)	0.1958 (2)	0.094 (2)
O(7)'	0.0075 (5)	0.2573 (3)	0.1966 (2)	0.075 (1)
O(8)'	-0.1045 (4)	0.1262 (3)	0.2665 (2)	0.064 (1)

TNBHC crystallizes in the space group *Pc* with the two molecules in the asymmetric unit having similar conformations. In Fig. 1, prepared with *ORTEP* (Johnson, 1965), the two molecules are numbered similarly with the primed molecule corresponding to molecule 2 in Table 1. The primary difference between molecules 1 and 2 is the orientations of the NO $_2$ groups; corresponding C–C–N–O and O–N–C–N torsion angles have values that differ from one molecule to the other by amounts ranging from 8 to 30°. The relative orientation of adjacent NO $_2$ groups is such that O(3) and O(8) are *trans* to N(1) and N(3). Dihedral angles between C–NO $_2$ least-squares planes with a common C atom are 102.1 and 111.9° at C(9), and 92.3 and 89.5° at C(10), respectively, for molecules 1 and 2.

The carbon cage is made up of four puckered five-membered rings and two puckered four-membered rings which are bent such that the dihedral angle between planes C(1)–C(4)–C(6) and C(5)–C(4)–C(6), and planes C(6)–C(5)–C(7) and C(8)–C(5)–

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

Molecule 1		Molecule 2			
C(1)–C(4)	1.565 (4)	1.564 (4)	C(8)–C(10)	1.505 (4)	1.504 (4)
C(1)–C(6)	1.529 (4)	1.529 (4)	C(9)–N(1)	1.520 (4)	1.517 (4)
C(1)–C(9)	1.501 (4)	1.516 (4)	C(9)–N(2)	1.522 (4)	1.521 (4)
C(2)–C(3)	1.549 (4)	1.552 (4)	C(10)–N(3)	1.514 (4)	1.534 (4)
C(2)–C(7)	1.549 (4)	1.554 (4)	C(10)–N(4)	1.526 (4)	1.513 (4)
C(2)–C(9)	1.541 (3)	1.524 (4)	N(1)–O(1)	1.204 (4)	1.196 (5)
C(3)–C(4)	1.558 (4)	1.561 (4)	N(1)–O(2)	1.193 (4)	1.202 (5)
C(3)–C(10)	1.527 (3)	1.519 (3)	N(2)–O(3)	1.196 (4)	1.188 (5)
C(4)–C(5)	1.569 (4)	1.571 (4)	N(2)–O(4)	1.208 (3)	1.175 (4)
C(5)–C(6)	1.549 (5)	1.547 (4)	N(3)–O(5)	1.203 (4)	1.181 (4)
C(5)–C(8)	1.541 (5)	1.535 (5)	N(3)–O(6)	1.214 (4)	1.187 (4)
C(6)–C(7)	1.562 (4)	1.569 (4)	N(4)–O(7)	1.202 (4)	1.202 (4)
C(7)–C(8)	1.565 (4)	1.566 (4)	N(4)–O(8)	1.204 (4)	1.206 (4)
C(4)–C(1)–C(6)	87.7 (2)	87.5 (2)	C(1)–C(9)–C(2)	98.1 (2)	97.7 (2)
C(4)–C(1)–C(9)	103.8 (2)	104.6 (2)	C(1)–C(9)–N(1)	113.4 (3)	115.4 (3)
C(6)–C(1)–C(9)	105.6 (3)	104.8 (3)	C(1)–C(9)–N(2)	113.3 (2)	111.2 (2)
C(3)–C(2)–C(7)	99.7 (2)	99.9 (2)	C(2)–C(9)–N(1)	112.3 (2)	112.0 (2)
C(3)–C(2)–C(9)	100.0 (2)	101.4 (2)	C(2)–C(9)–N(2)	115.1 (2)	114.8 (3)
C(7)–C(2)–C(9)	104.2 (2)	104.6 (2)	N(1)–C(9)–N(2)	104.9 (2)	106.0 (2)
C(2)–C(3)–C(4)	100.2 (2)	100.0 (2)	C(3)–C(10)–C(8)	97.8 (2)	99.0 (2)
C(2)–C(3)–C(10)	103.5 (2)	102.3 (2)	C(3)–C(10)–N(3)	112.4 (2)	113.2 (2)
C(4)–C(3)–C(10)	102.1 (2)	102.5 (2)	C(3)–C(10)–N(4)	114.2 (2)	111.6 (2)
C(1)–C(4)–C(3)	105.2 (2)	104.9 (2)	C(8)–C(10)–N(3)	112.7 (2)	112.8 (2)
C(1)–C(4)–C(5)	89.8 (2)	90.2 (2)	C(8)–C(10)–N(4)	115.5 (3)	115.7 (2)
C(3)–C(4)–C(5)	103.7 (2)	103.4 (2)	N(3)–C(10)–N(4)	104.6 (2)	105.0 (2)
C(4)–C(5)–C(6)	86.9 (2)	86.6 (2)	O(1)–N(1)–C(9)	117.1 (3)	117.3 (3)
C(4)–C(5)–C(8)	103.0 (2)	103.8 (2)	O(2)–N(1)–C(9)	117.7 (3)	116.2 (3)
C(6)–C(5)–C(8)	91.9 (2)	92.3 (2)	O(1)–N(1)–O(2)	125.3 (3)	126.5 (3)
C(1)–C(6)–C(5)	91.9 (2)	92.4 (2)	O(3)–N(2)–C(9)	116.0 (2)	118.2 (2)
C(1)–C(6)–C(7)	103.8 (2)	104.1 (2)	O(4)–N(2)–C(9)	118.5 (3)	117.7 (3)
C(5)–C(6)–C(7)	87.0 (2)	86.7 (2)	O(3)–N(2)–O(4)	125.4 (3)	123.6 (3)
C(2)–C(7)–C(6)	103.9 (2)	102.9 (2)	O(5)–N(3)–C(10)	118.7 (2)	118.2 (2)
C(2)–C(7)–C(8)	105.5 (2)	105.7 (2)	O(6)–N(3)–C(10)	116.8 (3)	116.3 (3)
C(6)–C(7)–C(8)	90.5 (2)	90.3 (2)	O(5)–N(3)–O(6)	124.5 (3)	125.4 (3)
C(5)–C(8)–C(7)	87.2 (2)	87.3 (2)	O(7)–N(4)–C(10)	117.8 (3)	116.9 (3)
C(5)–C(8)–C(10)	103.6 (2)	103.6 (2)	O(8)–N(4)–C(10)	116.1 (2)	117.6 (2)
C(7)–C(8)–C(10)	106.1 (2)	104.7 (2)	O(7)–N(4)–O(8)	126.1 (3)	125.3 (3)

C(7) averages 19.5 (6)°. Interior C–C–C angles for the four-membered rings average 89.2 (2.2)° and the interior C–C–C angles of the five-membered rings average 102.7 (2.4)°.

C–C bonds to the bridging dinitro carbons average 1.517 (13) Å while the remaining C–C cage bonds average 1.554 (13) Å. In trinitrobishomocubane (Ammon *et al.*, 1985) the corresponding averages are 1.513 and 1.550 Å, while in cubane (Fleischer, 1964) the average C–C distance is 1.551 Å. For those geometric parameters which are equivalent, trinitrobishomocubane and tetranitrobishomocubane are nearly isostructural. C–NO₂ distances average 1.521 Å and N–O distances average 1.197 Å. The C–NO₂ bond distances are typical of alicyclic nitro compounds with two electron-withdrawing groups bound to the same carbon, *e.g.* in 2,2-dinitroadamantane where C–N = 1.555 (5) and 1.560 (5) Å (George & Gilardi, 1983).

There are no unusual intermolecular contacts, and no hydrogen bonds. The nearest contacts are O(2')...H(3) = 2.488 (33) Å, O(6)...H(3') = 2.673 (34) Å, and O(7')...H(1) = 2.636 (34) Å. The molecular packing and relative NO₂ group orientation may be seen in

* Errors quoted for averaged quantities are the (sample variance)^{1/2}. Individual distances and angles are determined more accurately.

Fig. 2. The molecules pack with the oxygens of molecule 1 opposing those of molecule 2. Nearest O...O approaches are in the range of 2.84 to 3.26 Å for each of the O atoms in the asymmetric unit.

Work at the Naval Research Laboratory was supported in part by the Office of Naval Research under contract N0014-84-WR-24053.

Work at North Texas State University was supported under contracts DAAK10-83-M-1031 and DAAK10-83-M1943 from the United States Army Armanent, Munitions and Chemical Command, Dover, NJ, and by grants from the Air Force Office of Scientific Research (Grant No. AFOSR-84-0085), The Robert A. Welch Foundation (Grant No. B-963), and The North Texas State University Faculty Research Committee.

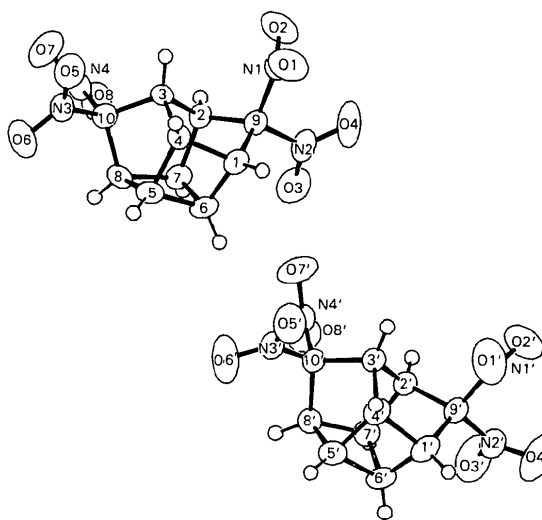


Fig. 1. ORTEP drawing of the two molecules in the asymmetric unit with C atoms numbered 1 through 10. The molecules differ primarily by small differences in the nitro-group orientation. Thermal ellipsoids enclose 50% probability.

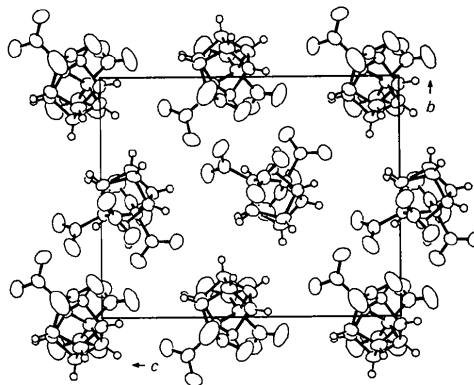


Fig. 2. Molecular packing viewed normal to the *bc* plane.

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Acta Cryst. (1985). C41, 791–793

Acétoxy-4 *tert*-Butyl-2 Cyclohexène-3 Carboxylate-1 de Méthyle-*cis*, C₁₄H₂₂O₄

PAR ROBERT VIANI

Département de Physique, Faculté des Sciences, Université Nationale de Côte d'Ivoire, 04 BP 322, Abidjan 04, Côte d'Ivoire

JACQUES LAPASSET

Laboratoire de Minéralogie–Cristallographie (CNRS–LA 233), Université de Montpellier II, place E. Bataillon, 34060 Montpellier CEDEX, France

ET JEAN-PIERRE AYCARD

Laboratoire de Chimie Organique Structurale, Université de Provence, Centre de Saint-Jérôme, 13397 Marseille CEDEX 13, France

(Reçu le 4 janvier 1983, accepté le 2 novembre 1984)

Abstract. $M_r = 254.33$, monoclinic, $P2_1/c$, $a = 8.576$ (3), $b = 7.834$ (3), $c = 22.291$ (7) Å, $\beta = 110.34$ (5)°, $V = 1404$ (3) Å³, $Z = 4$, $D_m = 1.19$ (2), $D_x = 1.203$ Mg m⁻³, $\lambda(\text{Cu } K\alpha) = 1.5418$ Å, $\mu = 0.719$ mm⁻¹, $F(000) = 552$, $T = 293$ K, final $R = 0.059$ for 1777 observed reflections. The ring conformation is intermediate between half-chair and sofa. The conformation of the methoxycarbonyl group averages its interactions with the vicinal methyls of the *tert*-butyl group. Bond lengths and angles are normal.

Introduction. Pour le composé étudié ici, les paramètres de résonance magnétique nucléaire (Lafrance, Aycard & Bodot, 1977) ont mis en évidence, en solution, une hétérogénéité conformationnelle où le conformère 'normal' comportant un groupe *tert*-butyle pseudo-équatorial est seulement présent à 60%. Ce résultat se distingue de celui obtenu pour l'acétoxy-1 *tert*-butyl-3 cyclohexèncarbonitrile-4-*cis*, conformationnellement homogène en solution, dont la structure cristalline et moléculaire a été étudiée (Viani & Lapasset, 1978) dans le cadre de cette série de mémoires (Viani, Lapasset, Aycard, Lafrance & Bodot, 1978; Viani & Lapasset, 1978, 1981; Cossu, Viani & Lapasset, 1981; Viani, Cossu & Lapasset, 1981; Viani, Lapasset & Aycard,

1984). Le présent mémoire permet, en particulier, d'établir une comparaison entre les deux structures.

Partie expérimentale. Le composé a été obtenu par réaction de Diels–Alder (Lafrance, Aycard & Bodot, 1977); cristal incolore parallélépipédique (0,32 × 0,20 × 0,15 mm); D_m mesuré par flottation. Diffractomètre Enraf–Nonius CAD-4. Réflexions enregistrées en balayage $\theta/2\theta$ ($\theta_{\max} = 65^\circ$). 12 réflexions pour déterminer les valeurs des paramètres de la maille. Pas de correction d'absorption. $(\sin \theta/\lambda)_{\max} = 0,588$ Å⁻¹. Pas de variation systématique de la réflexion de référence mesurée toutes les 49 mesures; 2383 réflexions indépendantes enregistrées; 1778 conservées [$I > 3\sigma(I)$]. h 0–10, k 0–9, l –26–23. Détermination par méthodes directes (*MULTAN*; Main, Woolfson, Lessinger, Germain & Declercq, 1974). Affinement par moindres carrés (*ORFLS*; Busing, Martin & Levy, 1962); quantité minimisée: $\sum w|F_o| - |F_c|$, w déterminé par la méthode de Hughes (1941): $w = 1/(0,8 - 0,0003F + 0,001F^2)$ si $F_o \geq 18$, $w = 1/0,8$ si $F_o \leq 18$. Hydrogènes localisés par Fourier-différence. Agitation thermique anisotrope pour les atomes lourds. Hydrogènes affectés du $B_{\text{éq}}$ des atomes porteurs. Pas d'affinement des paramètres