

Structure of a Protected C3–C10 Subunit of Erythromycin and its C8 Epimer

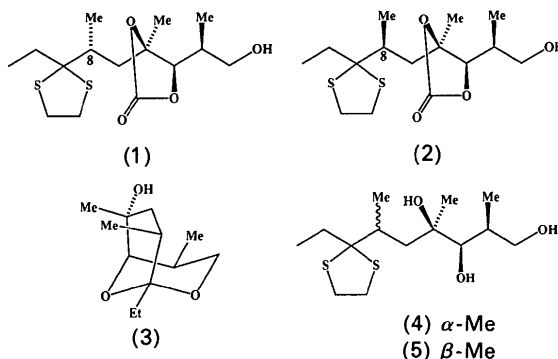
BY VINCENT M. LYNCH, WEN-CHERNG LEE, STEPHEN F. MARTIN AND BRIAN E. DAVIS

Department of Chemistry and Biochemistry, University of Texas at Austin, Austin, TX 78712, USA

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Abstract. (1) (2*S*,3*R*,4*R*,6*R*)-3,4-*O*-Carbonyl-7,7-dimethylenedithio-2,4,6-trimethylnonane-1,3,4-triol, C₁₅H₂₆O₄S₂, *M_r* = 334.49, triclinic, *P*1, *a* = 6.460 (2), *b* = 8.917 (3), *c* = 15.616 (5) Å, α = 83.60 (3), β = 83.41 (2), γ = 89.52 (2)°, *V* = 888.0 (5) Å³, *Z* = 2, *D_x* = 1.25 g cm⁻³, μ = 2.980 cm⁻¹, λ (Mo *K*α) = 0.7107 Å, *F*(000) = 360, *T* = 298 K, *R* = 0.0465 for 1832 reflections [*F_o* ≥ 4σ(*F_o*)]. (2) (2*S*,3*R*,4*R*,6*S*)-3,4-*O*-Carbonyl-7,7-dimethylenedithio-2,4,6-trimethylnonane-1,3,4-triol, C₁₅H₂₆O₄S₂, *M_r* = 334.49, monoclinic, *P*2₁, *a* = 8.1849 (8), *b* = 8.9456 (14), *c* = 12.0258 (14) Å, β = 100.878 (8)°, *V* = 864.7 (2) Å³, *Z* = 2, *D_x* = 1.28 g cm⁻³, μ = 3.060 cm⁻¹, λ (Mo *K*α) = 0.7107 Å, *F*(000) = 360, *T* = 298 K, *R* = 0.0569 for 2001 reflections [*F_o* ≥ 4σ(*F_o*)]. The two diastereomers differ in configuration at C6. For (1) there are two unique molecules in the unit cell. These two molecules differ in conformation by a rotation about the bond C7–C8. The molecules are hydrogen bonded into infinite chains along *b*. The hydroxyl O atom of molecule (2), O10', acts as both a donor and an acceptor in hydrogen-bonding interactions with the carbonyl O atom, O14, and the hydroxyl H atom, H10, of molecule (1). The relevant hydrogen-bonding parameters are: O10—H10···O10' (related by 1 + *x*, *y*, 1 + *z*), O···O 2.789 (15), H···O 1.946 (15) Å, O—H···O 170.9 (12)°; O10'—H10···O14 (related by *x*, *y* - 1, *z* - 1), O···O 2.789 (15), H···O 1.999 (15) Å, O—H···O 154.2 (13)°. For (2), molecules form hydrogen-bonded chains parallel to *a* with relevant parameters: O10—H10···O14 (related by *x*, *y* - 1, *z*), O···O 2.814 (8), H···O 2.10 (5) Å, O—H···O 154 (4)°. The conformation of the part of (1) and (2) from C1 to C5 is nearly identical for the three unique molecules.

Experimental. Compounds (1) and (2) were prepared from the inseparable mixture of corresponding dithioketals (4) and (5), which were formed upon the treatment of (3) (Martin, Pacofsky, Gist & Lee, 1989) with (CH₃)₃SiSCH₂CH₂SSi(CH₃)₃ and TiCl₄, by a straightforward sequence of reactions: (a) *tert*-butyldimethylsilyl chloride/dimethylaminopyridine, (b) 1,1'-carbonyldiimidazole, (c) tetramethylammonium fluoride.



In (1) and (2) the number 8 corresponds to the numbering employed in erythromycin.

The data crystal for (1) was a colorless needle of approximate dimensions 0.11 × 0.19 × 0.61 mm. The data crystal for (2) was a colorless block that was cut from a larger crystal and had approximate dimensions 0.28 × 0.31 × 0.52 mm. Crystals of both were obtained by slow evaporation from ethyl acetate. The data for both were collected at room temperature on a Nicolet *P*3 diffractometer using a graphite monochromator. Instrument and crystal stability were monitored by the remeasurement of check reflections throughout the course of data collection. A smoothed curve of the intensities of these check reflections was used to scale the data. The maximum variation in intensity of the check reflections did not exceed 3% for either (1) or (2). The data were also corrected for *L_p* effects but not for absorption. Data reduction and decay correction were performed using the Siemens *SHELXTL-Plus* software package (Sheldrick, 1989). Details of data collection and structure refinement are listed in Table 1.

The structures were solved by direct methods and refined by full-matrix least squares (Sheldrick, 1989) with anisotropic thermal parameters for the non-H atoms. The H-atom positions were calculated in idealized positions for (1) with an isotropic temperature factor refined for each H. The origin was defined by fixing the coordinates of C1. For (2), H-atom positions were obtained from a ΔF map except for those of the methyl C atom, C17, and methylene C atoms, C19 and C20, which were calculated in

Table 1. Data collection and structure refinement details

	(1)	(2)
Data collection		
Mode	ω scan	ω scan
Scan range	Symmetrically over 1.2° about $K\alpha_{1,2}$ maximum for (1) and (2)	
Background	Measured at ends of scan	
Scan rate (° min ⁻¹)	8–12	3–6
Decay correction factor range	0.972–1.00	0.999–1.02
2 θ range (°)	4.0–45.0	4.0–60
Standard reflections	022, 02 $\bar{2}$	1 $\bar{1}$ 7, 2 $\bar{4}$ 2, 216, 313
Reflections measured	2566	5377
Unique reflections	2316	2691
R _{int}	0.015	0.027
Range of h,k,l	0→11 -9→9 -16→16	-11→11 0→12 -16→16
Structure refinement		
Reflections used, $F > 4\sigma(F_o)$	1832	2001
Reflections omitted	474	690
No. of variables	428	265
R, wR	0.0465, 0.0470	0.0569, 0.0617
R _{int} , wR _{int}	0.0662, 0.0510	0.0733, 0.0644
Goodness of fit, S	1.303	1.879
Maximum $ \Delta F $	<0.1	<0.1
Minimum, maximum peaks in ΔF map (e Å ⁻³)	-0.20, 0.21	-0.49, 0.52

The lattice parameters were obtained from the least-squares refinement of 22 reflections with $13.5 < 2\theta < 16.6^\circ$ for (1) and of 36 reflections with $20.5 < 2\theta < 25.1^\circ$ for (2).

Table 2. Fractional coordinates and equivalent isotropic thermal parameters (Å²) for the non-H atoms of (1)

$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* A_{ij}$ where A_{ij} is the dot product of the i th and j th direct-space unit-cell vectors.

	x	y	z	U_{eq}
C1	1.2513	0.7140	0.7088	0.057 (4)
C2	1.099 (2)	0.8072 (13)	0.7621 (7)	0.049 (3)
C3	0.917 (2)	0.8649 (13)	0.7149 (7)	0.044 (3)
C4	0.965 (2)	0.9800 (13)	0.6325 (8)	0.039 (3)
C5	0.969 (2)	0.9118 (13)	0.5475 (7)	0.045 (3)
C6	0.764 (2)	0.8488 (13)	0.5250 (7)	0.044 (3)
C7	0.794 (2)	0.7126 (14)	0.4735 (8)	0.051 (3)
C8	0.934 (2)	0.7504 (15)	0.3879 (8)	0.069 (4)
C9	0.983 (3)	0.627 (2)	0.3289 (10)	0.114 (7)
O10	1.436 (2)	0.6829 (12)	0.7479 (7)	0.073 (3)
O11	1.017 (2)	0.716 (2)	0.8486 (8)	0.070 (4)
O12	0.781 (2)	0.9488 (12)	0.7711 (7)	0.054 (2)
O13	0.691 (2)	1.0590 (14)	0.7249 (8)	0.052 (4)
O14	0.553 (2)	1.1370 (12)	0.7520 (7)	0.078 (3)
O15	0.774 (2)	1.0734 (11)	0.6423 (7)	0.051 (2)
C16	1.147 (2)	1.0851 (14)	0.6336 (8)	0.064 (4)
C17	0.645 (2)	0.9785 (15)	0.4766 (9)	0.065 (4)
S18	0.9113 (14)	0.5573 (10)	0.5376 (6)	0.0722 (11)
C19	0.672 (3)	0.460 (2)	0.5765 (13)	0.140 (8)
C20	0.538 (3)	0.463 (2)	0.5081 (12)	0.127 (7)
S21	0.5381 (14)	0.6467 (10)	0.4516 (6)	0.0809 (11)
C1'	0.332 (2)	0.3137 (13)	-0.0916 (8)	0.054 (3)
C2'	0.166 (2)	0.1906 (13)	-0.0830 (7)	0.041 (3)
C3'	0.146 (2)	0.1050 (13)	0.0077 (7)	0.043 (3)
C4'	0.324 (2)	0.0028 (13)	0.0367 (7)	0.033 (3)
C5'	0.463 (2)	0.0770 (13)	0.0918 (8)	0.045 (3)
C6'	0.374 (2)	0.0911 (13)	0.1866 (7)	0.048 (3)
C7'	0.437 (2)	0.2440 (13)	0.2175 (8)	0.055 (4)
C8'	0.371 (2)	0.2526 (15)	0.3138 (8)	0.069 (5)
C9'	0.143 (2)	0.220 (2)	0.3454 (9)	0.089 (6)
O10'	0.367 (2)	0.3860 (12)	-0.1778 (7)	0.071 (3)
O11'	-0.039 (2)	0.2576 (15)	-0.1061 (9)	0.070 (4)
O12'	-0.026 (2)	-0.0011 (12)	0.0141 (7)	0.055 (2)
O13'	0.011 (2)	-0.1226 (14)	0.0682 (8)	0.058 (4)
O14'	-0.111 (2)	-0.2185 (12)	0.0932 (7)	0.092 (3)
O15'	0.205 (2)	-0.1201 (11)	0.0902 (7)	0.051 (2)
C16'	0.453 (2)	-0.0660 (14)	-0.0368 (8)	0.055 (3)
C17'	0.447 (2)	-0.0434 (14)	0.2450 (9)	0.080 (6)
S18'	0.3119 (14)	0.3982 (10)	0.1541 (6)	0.0551 (9)
C19'	0.524 (2)	0.527 (2)	0.1454 (12)	0.113 (5)
C20'	0.716 (3)	0.459 (2)	0.1345 (14)	0.146 (6)
S21'	0.7182 (14)	0.2764 (10)	0.1955 (6)	0.0808 (10)

Table 3. Fractional coordinates and equivalent isotropic thermal parameters (Å²) for the non-H atoms of (2)

$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* A_{ij}$ where A_{ij} is the dot product of the i th and j th direct-space unit-cell vectors.

	x	y	z	U_{eq}
C1	-0.2525 (6)	-0.1077	0.1176 (4)	0.0542 (14)
C2	-0.3152 (5)	0.0364 (7)	0.0557 (3)	0.0417 (11)
C3	-0.1821 (4)	0.1566 (6)	0.0703 (3)	0.0352 (11)
C4	-0.1095 (5)	0.2176 (7)	0.1886 (3)	0.0387 (11)
C5	0.0543 (5)	0.1429 (7)	0.2355 (3)	0.0446 (13)
C6	0.1443 (6)	0.1820 (7)	0.3571 (4)	0.0489 (14)
C7	0.2462 (5)	0.0511 (8)	0.4147 (3)	0.0485 (13)
C8	0.3515 (7)	0.0873 (10)	0.5326 (4)	0.071 (2)
C9	0.2521 (11)	0.1419 (12)	0.6180 (5)	0.089 (3)
O10	-0.1137 (4)	-0.1651 (7)	0.0775 (4)	0.0699 (11)
C11	-0.3758 (7)	0.0046 (8)	-0.0708 (4)	0.064 (2)
O12	-0.2521 (3)	0.2905 (6)	0.0105 (2)	0.0456 (9)
C13	-0.1763 (5)	0.4103 (7)	0.0626 (3)	0.0451 (13)
O14	-0.1968 (5)	0.5348 (6)	0.0242 (3)	0.0629 (12)
O15	-0.0801 (3)	0.3756 (6)	0.1604 (2)	0.0487 (9)
C16	-0.2328 (6)	0.2221 (8)	0.2682 (4)	0.053 (2)
C17	0.2474 (10)	0.3259 (9)	0.3561 (6)	0.109 (3)
S18	0.3977 (2)	-0.0218 (6)	0.33118 (13)	0.1040 (8)
C19	0.2816 (11)	-0.1890 (15)	0.2799 (8)	0.156 (5)
C20	0.2046 (12)	-0.2496 (12)	0.3721 (11)	0.149 (4)
S21	0.1049 (2)	-0.1030 (5)	0.42697 (11)	0.0717 (5)

idealized positions (C—H 0.96 Å) with U values fixed at $1.2 \times U_{eq}$ of the appropriate C atom (seven idealized H atoms). The isotropic temperature factors were refined for the other H atoms. The origin was defined along the 2₁ screw axis by fixing the y coordinate of C1. The function $\sum w(|F_o| - |F_c|)^2$ was minimized, where $w = 1/[\sigma(F_o)]^2$ and $\sigma(F_o) = 0.5kI^{-1/2}\{\sigma(I)^2 + (0.02I)^2\}^{1/2}$. The intensity, I , is given by $(I_{peak} - I_{background})$ (scan rate); where 0.02 is a factor to downweight intense reflections and to account for instrument instability, and k is the correction for Lp effects and decay. $\sigma(I)$ was estimated from counting statistics; $\sigma(I) = [(I_{peak} + I_{background})^{1/2}$ (scan rate)]. The absolute configuration for (1) and (2) could not be determined from the X-ray results but was assigned on the basis of internal comparison to the configuration at C2 and C3. The configuration of C2 and C3 were assigned via an asymmetric aldol reaction (Martin *et al.*, 1989). The scattering factors for the non-H atoms were taken from Cromer & Mann (1968), with the anomalous-dispersion corrections taken from the work of Cromer & Liberman (1970). The scattering factors for the H atoms were obtained from Stewart, Davidson & Simpson (1965). Values used to calculate the linear absorption coefficient are from *International Tables for X-ray Crystallography* (1974, Vol. IV, p. 55).^{*} Figures were generated using *SHELXTL-Plus* (Sheldrick, 1989). The positional

^{*} Lists of anisotropic thermal parameters, H-atom positional and thermal parameters, bond distances and angles involving the H atoms, torsion angles, structure-factor amplitudes and packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54838 (44 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0549]

Table 4. Bond lengths (Å) and angles (°) for (1) and (2)

			(1)		(1')		(2)	
1	2	3	1—2	1—2—3	1—2	1—2—3	1—2	1—2—3
C2	C1	O10	1.520 (11)	111.9 (6)	1.52 (2)	110.9 (11)	1.529 (6)	111.0 (4)
O10	C1		1.410 (11)		1.43 (2)		1.412 (7)	
C3	C2	C11	1.52 (2)	109.2 (9)	1.52 (2)	112.0 (9)	1.517 (7)	109.7 (4)
C3	C2	C1		113.3 (9)		110.4 (10)		111.9 (3)
C11	C2	C1	1.54 (2)	110.8 (9)	1.52 (2)	110.6 (10)	1.535 (6)	110.0 (5)
C4	C3	O12	1.56 (2)	102.6 (9)	1.54 (2)	102.1 (9)	1.536 (5)	102.2 (4)
C4	C3	C2		117.6 (9)		120.2 (9)		120.2 (4)
O12	C3	C2	1.43 (2)	109.1 (10)	1.46 (2)	107.8 (10)	1.458 (7)	108.6 (3)
C5	C4	O15	1.52 (2)	107.6 (10)	1.52 (2)	109.5 (9)	1.509 (6)	109.6 (4)
C5	C4	C16		112.8 (9)		111.0 (9)		115.2 (4)
C5	C4	C3		114.5 (9)		113.3 (10)		110.4 (4)
O15	C4	C16	1.49 (2)	106.1 (10)	1.468 (14)	107.3 (9)	1.483 (8)	106.0 (4)
O15	C4	C3		98.8 (8)		100.8 (8)		100.3 (3)
C16	C4	C3	1.52 (2)	115.2 (10)	1.52 (2)	114.2 (9)	1.516 (7)	114.0 (3)
C6	C5	C4	1.53 (2)	118.1 (9)	1.54 (2)	116.8 (9)	1.548 (6)	118.9 (4)
C7	C6	C17	1.53 (2)	112.0 (10)	1.57 (2)	110.8 (10)	1.526 (8)	113.0 (4)
C7	C6	C5		113.6 (10)		112.2 (9)		112.2 (5)
C17	C6	C5	1.56 (2)	108.8 (9)	1.53 (2)	109.1 (10)	1.540 (10)	110.2 (5)
C8	C7	S18	1.53 (2)	108.9 (9)	1.53 (2)	108.8 (9)	1.549 (6)	104.8 (3)
C8	C7	S21		109.7 (9)		108.8 (10)		109.6 (4)
C8	C7	C6		111.9 (10)		113.0 (9)		114.7 (5)
S18	C7	S21	1.825 (15)	107.1 (7)	1.840 (15)	106.9 (7)	1.855 (5)	106.1 (4)
S18	C7	C6		110.0 (9)		107.9 (9)		112.8 (4)
S21	C7	C6	1.85 (2)	109.1 (9)	1.83 (2)	111.3 (9)	1.823 (7)	108.4 (3)
C9	C8	C7	1.52 (2)	119.3 (11)	1.51 (2)	116.2 (12)	1.507 (10)	114.4 (5)
C13	O12	C3	1.32 (2)	110.2 (10)	1.34 (2)	109.5 (10)	1.334 (7)	108.9 (3)
O14	C13	O15	1.20 (2)	123.1 (12)	1.17 (2)	124.8 (12)	1.205 (8)	125.0 (5)
O14	C13	O12		125.7 (12)		124.0 (13)		122.9 (4)
O15	C13	O12	1.33 (2)	111.3 (11)	1.34 (2)	111.2 (11)	1.323 (5)	112.1 (5)
C4	O15	C13		109.9 (10)		109.8 (10)		109.4 (4)
C19	S18	C7	1.79 (2)	96.1 (8)	1.78 (2)	95.6 (8)	1.816 (13)	96.5 (4)
C20	C19	S18	1.44 (3)	110.4 (13)	1.38 (2)	113.1 (12)	1.48 (2)	108.5 (7)
S21	C20	C19	1.77 (2)	110.0 (12)	1.79 (2)	112.4 (12)	1.740 (12)	107.8 (8)
C7	S21	C20		99.1 (9)		98.9 (8)		100.9 (4)

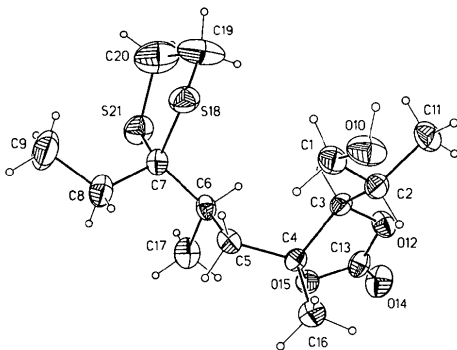


Fig. 1. View of molecule 1 of (1) showing the atom-labelling scheme. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size.

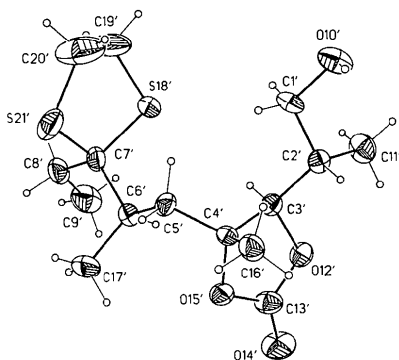


Fig. 2. View of molecule 2 of (1) showing the atom-labelling scheme. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size.

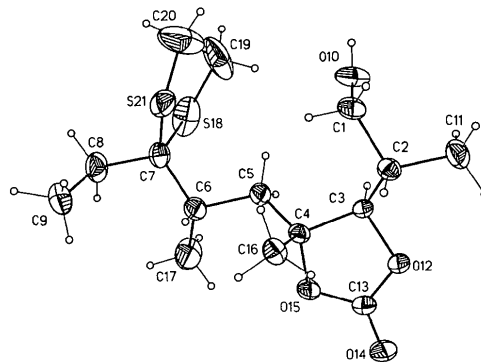


Fig. 3. View of (2) showing the atom-labelling scheme. The non-H atoms are scaled to the 30% probability level while the H atoms are drawn to an arbitrary size.

and thermal parameters for non-H atoms are listed in Tables 2 and 3 for (1) and (2), respectively. Both (1) and (2) use the same labelling scheme. Atoms from different molecules of (1) are distinguished by a prime symbol '. The bond lengths and angles for the non-H atoms are listed in Table 4 for (1) and (2). The atom-labelling scheme for (1) is shown in Figs. 1 and 2 and for (2) in Fig. 3. Other computer programs used in this work are listed in reference 11 of Gadoll & Davis (1982).

Related literature. The structures of (1) and (2) could not be established unequivocally by NMR techniques and, therefore, X-ray analysis of these crystalline substances was undertaken. The crystal

structures of some related substances have been reported (Lynch, Lee, Martin & Davis, 1991; Lynch, Pacofsky, Martin & Davis, 1989).

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N-(4,6-Diméthylpyridin-2-yl)(1-méthylindol-2-yl)carboxamide

PAR N. RODIER

Laboratoire de Chimie minérale, Faculté des Sciences pharmaceutiques et biologiques, 5 Rue J.-B. Clément, 92296 Châtenay-Malabry CEDEX, France

J.-M. CENSE

Laboratoire d'Informatique chimique, ENSCP, 11 Rue Pierre et Marie Curie, 75231 Paris CEDEX 05, France

ET J.-M. ROBERT ET G. LE BAUT

Laboratoire de Chimie thérapeutique, Faculté de Pharmacie, 1 Rue Gaston-Veill, 44035 Nantes CEDEX, France

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Abstract. N-(4,6-Diméthylpyridin-2-yl)(1-méthylindol-2-yl)carboxamide, C₁₇H₁₇N₃O, *M_r* = 279.3, orthorhombic, *P*2₁2₁2₁, *a* = 6.3847 (8), *b* = 10.234 (1), *c* = 22.251 (3) Å, *V* = 1453.9 (5) Å³, *Z* = 4, *D_x* = 1.276 Mg m⁻³, λ(Mo Kα) = 0.7107 Å, μ = 0.076 mm⁻¹, *F*(000) = 592, *T* = 294 (1) K, *R* = 0.048 for 1019 independent observed reflections. The whole molecule is approximately planar. The least-squares planes of the pyridyl ring and of the indolyl group make an angle of 2 (2)°. The intramolecular C(3)—H(3)···O(20) hydrogen bond [2.859 (5) Å, 118 (3)°] forms a pseudo-cycle and contributes to the planarity of the molecule. There is a delocalized orbital along the amide group. The title compound belongs to a family whose numerous members proved to have anti-inflammatory properties. Its crystal structure was solved in order to compare its molecular geometry with the geometries of active molecules.

Partie expérimentale. Cristaux obtenus par évaporation d'une solution dans l'éther isopropylique.

Cristal approximativement parallélépipédique: 0,11 × 0,16 × 0,33 mm. Dimensions de la maille déterminées avec 25 réflexions telles que 7,50 ≤ θ ≤ 12,84°. Diffractomètre Enraf-Nonius CAD-4. Balayage θ/2θ d'amplitude *s* = (0,80 + 0,35tgθ)°. 0,049 ≤ (sinθ)/λ ≤ 0,617 Å⁻¹, 0 ≤ *h* ≤ 7, 0 ≤ *k* ≤ 12, 0 ≤ *l* ≤ 27. Réflexions de contrôle de l'intensité: 134, 238 et 303. Variations non significatives de *I* au cours des mesures. 1671 réflexions indépendantes mesurées, 672 inobservées [*I* ≤ 1,5σ(*I*)]. Méthodes directes, programme MULTAN11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982). H des CH₃, série de Fourier des Δ*F*; autres H, positions calculées. Affinement basé sur les *F*, programme à matrice complète. Facteurs de diffusion des *International Tables for X-ray Crystallography* (1974, Tome IV, pp. 99, 149). Paramètres affinés: *x*, *y*, *z* et β_{*ij*} de C, N et O. Coordonnées des H non affinées à l'exception de celles de H(3): nombre insuffisant de réflexions. *R* = 0,048, *wR* = 0,046, *w* = 1/σ²(*F*), *S* = 1,36, (Δ/σ)_{max} = 0,01, Δρ_{max} = 0,18 (4), Δρ_{min} =