

common to all reported nitramine structures, the greatest deviation of C from the NNO_2 plane (0.536 Å) being found in cyclotrimethylene-trinitramine (Choi & Prince, 1972). In the present compound both nitroxyethyl groups are almost planar with only the amine C atoms C(3), C(2) appreciably deviating (0.118 and 0.187 Å). The intermolecular contact, 2.90 Å, between O(7) and N(1) of *b*-related molecules is less than the sum of the van der Waals radii (3.25 Å) across the π bonding electrons (Bondi, 1964); similar interactions, most probably polar in nature, have been found in other nitramine structures (Cobbledick & Small, 1973). An attempt was made to interpret the U_{ij} values in terms of rigid-body motion of the molecule, using the program *MGTLS* (Schomaker & Trueblood, 1968); the results suggested that the assumption of rigidity was likely to be invalid.

We thank the Science Research Council for the award of a research studentship to one of us (JH).

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The Structure of Erythromycin A Cyclic Carbonate

BY A. HEMPEL*

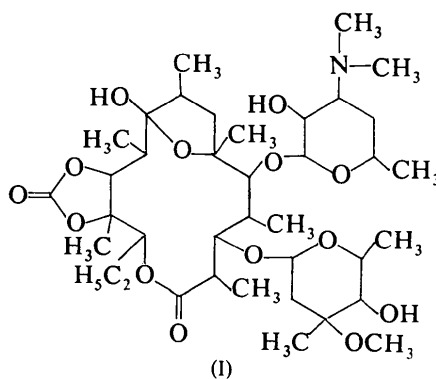
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Abstract. $\text{C}_{38}\text{H}_{65}\text{NO}_{14}$, FW 759, $P2_12_12_1$, $a = 17.08$ (2), $b = 22.02$ (2), $c = 10.84$ (1) Å, $Z = 4$, $V = 4075$ Å³, $D_m = 1.23$, $D_c = 1.24$ g cm⁻³, $F(000) = 1648$. 3117 reflections were measured on a Hilger & Watts automatic diffractometer with Mo $K\alpha$ radiation. The structure was refined by a full-matrix least-squares method to a final *R* index of 0.080. The structure appears to be in the 6,9-hemiacetal form and the absolute configuration at C(9) is *R*.

Introduction. The first successful attempt to obtain an erythromycin modification product of higher biological activity than that of the parent antibiotic gave erythromycin A cyclic carbonate (I) (Murphy, Stephens & Conine, 1968; Bojarska-Dahlig & Slawinski, 1972). As was stated earlier (Hempel, Bogucka-Ledochowska, Dauter, Borowski & Kosturkiewicz, 1975) the knowledge of the complete three-dimensional structure of the compound is particularly important when discussing the relation between molecular structure and biological activity and when formulating new derivatives. This analysis was undertaken to establish the absolute

configuration at C(9) and also to confirm the position of the carbonate moiety. Some doubts were removed about the possible formation of 6,9-hemiacetal and 9-keto forms.



The sample of erythromycin carbonate was kindly supplied by Professor H. Bojarska-Dahlig from The Institute of Pharmaceutical Industry (Warsaw, Poland). Crystals were grown from methanol–ethyl acetate–water solution as colourless prisms elongated in the *c* direction. Dimensions of the crystal selected were 0.4 × 0.5 × 0.5 mm. Precession photographs

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Table 1. *Intensity-measurement data*

Scan method	$2\theta/\omega$
Scan range	0.5°
$2\theta_{\max}$	46°
Number of unique reflexions	3215

Table 3. *Coordinates for the hydrogen atoms ($\times 10^4$)*

	<i>x</i>	<i>y</i>	<i>z</i>
H(20)	-9796 (5)	-3066 (4)	-6140 (7)
H(30)	-8746 (4)	-3768 (3)	-7471 (7)
H(40)	-9771 (4)	-3141 (4)	-8348 (8)
H(50)	-9231 (4)	-4197 (4)	-9413 (7)
H(71)	-9265 (5)	-2680 (4)	-11266 (8)
H(72)	-9598 (5)	-2606 (4)	-10044 (8)
H(80)	-7954 (6)	-2314 (4)	-10927 (8)
H(100)	-7107 (5)	-2038 (4)	-8573 (8)
H(110)	-7405 (5)	-3209 (4)	-7250 (8)
H(130)	-7408 (5)	-3217 (4)	-4907 (7)
H(141)	-8481 (12)	-2593 (10)	-3364 (12)
H(142)	-8365 (12)	-1923 (10)	-4236 (12)
H(151)	-7062 (16)	-2616 (11)	-3030 (21)
H(152)	-7824 (16)	-2090 (11)	-2775 (21)
H(153)	-7069 (16)	-1917 (11)	-3827 (21)
H(161)	-9898 (6)	-4319 (5)	-4959 (9)
H(162)	-10654 (6)	-3975 (5)	-5818 (9)
H(163)	-10364 (6)	-3661 (5)	-4391 (9)
H(171)	-10919 (5)	-4100 (5)	-8390 (9)
H(172)	-10948 (5)	-3354 (5)	-8979 (9)
H(173)	-10931 (5)	-3476 (5)	-7370 (9)
H(181)	-8748 (6)	-3691 (4)	-11985 (9)
H(182)	-8216 (6)	-4103 (4)	-10871 (9)
H(183)	-7859 (6)	-3400 (4)	-11409 (9)
H(191)	-8760 (8)	-1507 (4)	-11069 (11)
H(192)	-7984 (8)	-1425 (4)	-10009 (11)
H(193)	-8958 (8)	-1455 (4)	-9475 (11)
H(201)	-6758 (6)	-2592 (5)	-10480 (10)
H(202)	-6929 (6)	-3271 (5)	-9660 (10)
H(203)	-6115 (6)	-2813 (5)	-9288 (10)
H(211)	-8053 (8)	-1734 (4)	-6714 (9)
H(212)	-7058 (8)	-1575 (4)	-7000 (9)
H(213)	-7445 (8)	-1480 (4)	-5503 (9)
H(310)	-9223 (4)	-6161 (3)	-9745 (7)
H(350)	-12043 (3)	-4349 (3)	-11462 (6)
H(360)	-10938 (5)	-4084 (4)	-12424 (9)
H(370)	-11335 (5)	-5256 (4)	-10994 (8)
H(381)	-10357 (5)	-5101 (4)	-13315 (8)
H(382)	-10793 (5)	-5809 (4)	-12449 (8)
H(390)	-9977 (5)	-5474 (4)	-10869 (9)
H(400)	-10448 (5)	-4561 (3)	-9890 (8)
H(411)	-12573 (7)	-5651 (5)	-11323 (13)
H(412)	-12186 (7)	-5940 (5)	-12713 (13)
H(413)	-13047 (7)	-5498 (5)	-12735 (13)
H(421)	-11681 (7)	-4497 (5)	-14237 (9)
H(422)	-12612 (7)	-4832 (5)	-14064 (9)
H(423)	-11776 (7)	-5295 (5)	-14228 (9)
H(430)	-8565 (3)	-2509 (2)	-7170 (5)
H(440)	-8863 (5)	-4679 (4)	-5922 (8)
H(451)	-8959 (6)	-5714 (4)	-6048 (9)
H(452)	-9795 (6)	-5507 (4)	-6036 (9)
H(470)	-8131 (6)	-6018 (4)	-7795 (9)
H(480)	-8852 (5)	-4987 (4)	-9210 (9)
H(491)	-7128 (6)	-5359 (6)	-8768 (14)
H(492)	-7391 (6)	-4660 (6)	-9448 (14)
H(493)	-7658 (6)	-5349 (6)	-10164 (14)
H(501)	-9668 (8)	-6827 (4)	-7953 (14)
H(502)	-9916 (8)	-6537 (4)	-6487 (14)
H(503)	-8933 (8)	-6715 (4)	-6832 (14)
H(511)	-11063 (6)	-5513 (7)	-8865 (13)
H(512)	-10734 (6)	-5345 (7)	-7361 (13)
H(513)	-10922 (6)	-6103 (7)	-7789 (13)
H(521)	-9130 (6)	-5306 (6)	-13386 (12)
H(522)	-9220 (6)	-6002 (6)	-12594 (12)
H(523)	-8611 (6)	-5425 (6)	-12000 (12)

Table 2. *Coordinates for the non-hydrogen atoms ($\times 10^4$)*

	<i>x</i>	<i>y</i>	<i>z</i>
C(1)	-8846 (5)	-3403 (3)	-5316 (7)
C(2)	-9591 (5)	-3522 (4)	-6022 (7)
C(3)	-9397 (4)	-3830 (3)	-7297 (7)
C(4)	-9836 (4)	-3541 (4)	-8367 (8)
C(5)	-9493 (4)	-3752 (4)	-9593 (7)
C(6)	-8841 (5)	-3335 (4)	-10130 (7)
C(7)	-9112 (5)	-2710 (4)	-10507 (8)
C(8)	-8398 (6)	-2311 (4)	-10217 (8)
C(9)	-8137 (5)	-2596 (4)	-8996 (8)
C(10)	-7257 (5)	-2514 (4)	-8623 (8)
C(11)	-7103 (5)	-2802 (4)	-7339 (8)
C(12)	-7236 (6)	-2408 (4)	-6144 (8)
C(13)	-7713 (5)	-2773 (4)	-5190 (7)
C(14)	-8080 (12)	-2333 (10)	-3925 (12)
C(15)	-7429 (16)	-2276 (11)	-3456 (21)
C(16)	-10163 (6)	-3892 (5)	-5212 (9)
C(17)	-10722 (5)	-3640 (5)	-8251 (9)
C(18)	-8379 (6)	-3654 (4)	-11179 (9)
C(19)	-8553 (8)	-1625 (4)	-10158 (11)
C(20)	-6718 (6)	-2810 (5)	-9589 (10)
C(21)	-7469 (8)	-1751 (4)	-6333 (9)
C(22)	-5932 (8)	-2736 (6)	-6323 (13)
O(23)	-5277 (5)	-2832 (5)	-5984 (9)
O(24)	-6291 (4)	-2965 (3)	-7279 (7)
O(25)	-6434 (4)	-2403 (3)	-5656 (7)
O(26)	-8462 (3)	-2915 (2)	-5790 (5)
O(27)	-8611 (4)	-3687 (3)	-4449 (5)
O(28)	-8282 (3)	-3228 (2)	-9123 (5)
O(29)	-9560 (3)	-4469 (2)	-7291 (5)
O(30)	-8289 (3)	-4849 (2)	-7501 (6)
O(31)	-8621 (4)	-6161 (3)	-9476 (7)
O(32)	-9922 (3)	-5679 (2)	-8501 (6)
O(33)	-10075 (3)	-3786 (2)	-10569 (5)
O(34)	-9711 (3)	-4657 (2)	-11525 (5)
O(35)	-11675 (3)	-4080 (3)	-10910 (6)
C(36)	-11053 (5)	-4359 (4)	-11586 (9)
C(37)	-11264 (5)	-5014 (4)	-11898 (8)
C(38)	-10619 (5)	-5331 (4)	-12560 (8)
C(39)	-9861 (5)	-5281 (4)	-11826 (9)
C(40)	-10316 (5)	-4377 (3)	-10811 (8)
C(41)	-12486 (7)	-5561 (5)	-12293 (13)
C(42)	-11998 (7)	-4875 (5)	-13846 (9)
O(43)	-8646 (3)	-2344 (2)	-8067 (5)
C(44)	-8975 (5)	-4828 (4)	-6753 (8)
C(45)	-9293 (6)	-5466 (4)	-6506 (9)
C(46)	-9329 (5)	-5888 (4)	-7628 (9)
C(47)	-8584 (6)	-5825 (4)	-8355 (9)
C(48)	-8371 (5)	-5188 (4)	-8656 (9)
C(49)	-7570 (6)	-5110 (5)	-9296 (11)
C(50)	-9479 (7)	-6552 (4)	-7205 (11)
C(51)	-10714 (5)	-5660 (5)	-8086 (12)
C(52)	-9146 (6)	-5521 (5)	-12507 (10)
N(53)	-12059 (4)	-4997 (4)	-12516 (8)

taken with Cu $K\alpha$ radiation showed $P2_12_1$ symmetry. Details of the data collection on a Hilger & Watts four-circle diffractometer are summarized in Table 1. Cell dimensions were determined by least-squares fit of 23 high-angle reflexions. The usual Lorentz and polarization corrections were applied.

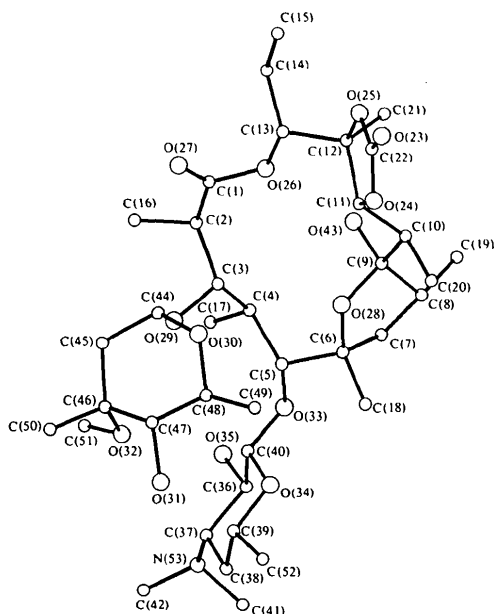


Fig. 1. The molecule of erythromycin A cyclic carbonate and atom numbering.

Table 4. Bond lengths (Å) with standard deviations in parentheses

C(1)–C(2)	1.51 (2)	C(1)–O(26)	1.36 (2)
C(1)–O(27)	1.20 (2)	C(2)–C(3)	1.57 (2)
C(2)–C(16)	1.55 (2)	C(3)–C(4)	1.52 (2)
C(3)–O(29)	1.44 (2)	C(4)–C(5)	1.52 (2)
C(4)–C(17)	1.54 (2)	C(5)–C(6)	1.56 (2)
C(5)–O(33)	1.45 (2)	C(6)–C(7)	1.51 (2)
C(6)–C(18)	1.55 (2)	C(6)–O(28)	1.47 (2)
C(7)–C(8)	1.54 (2)	C(8)–C(9)	1.53 (2)
C(8)–C(19)	1.54 (2)	C(9)–C(10)	1.57 (2)
C(9)–O(28)	1.42 (2)	C(9)–O(43)	1.44 (2)
C(10)–C(11)	1.55 (2)	C(10)–C(20)	1.54 (2)
C(11)–C(12)	1.58 (2)	C(11)–O(24)	1.44 (2)
C(12)–C(13)	1.54 (2)	C(12)–C(21)	1.51 (2)
C(12)–O(25)	1.47 (2)	C(13)–C(14)	1.79 (4)
C(13)–O(26)	1.47 (2)	C(14)–C(15)	1.23 (4)
C(22)–O(23)	1.20 (2)	C(22)–O(24)	1.31 (2)
C(22)–O(25)	1.34 (2)	O(29)–C(44)	1.41 (2)
O(30)–C(44)	1.43 (2)	O(30)–C(48)	1.46 (2)
O(31)–C(47)	1.42 (2)	O(32)–C(51)	1.43 (2)
O(33)–C(40)	1.39 (2)	O(34)–C(39)	1.44 (2)
O(34)–C(40)	1.43 (2)	O(35)–C(36)	1.43 (2)
C(36)–C(37)	1.53 (2)	C(36)–C(40)	1.51 (2)
C(37)–C(38)	1.49 (2)	C(37)–N(53)	1.52 (2)
C(38)–C(39)	1.52 (2)	C(39)–C(52)	1.52 (2)
C(41)–N(53)	1.46 (2)	C(42)–N(53)	1.47 (2)
C(44)–C(45)	1.53 (2)	C(45)–C(46)	1.53 (2)
C(46)–C(47)	1.50 (2)	C(46)–C(50)	1.55 (2)
C(47)–C(48)	1.49 (2)	C(48)–C(49)	1.54 (2)

The structure was solved by direct methods with the *MULTAN* package of programs (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). Structure amplitudes were normalized using a non-analytical K curve. The E map showed 47 out of 53 atoms of the molecule. The refinement of the structure was accomplished using *SHELX 76* (Sheldrick, 1976). All non-methyl H atoms were found from a difference Fourier synthesis. The coordinates of the methyl H atoms were calculated to fit the geometry of the molecule. During the refinement procedure methyl residues were treated as rigid groups. The remaining H atoms were allowed to 'ride' on corresponding C and O atoms while positional and thermal parameters for all non-hydrogen atoms were refined. Non-hydrogen atoms were allowed

Table 5. Bond angles (°) with standard deviations in parentheses

C(2)–C(1)–O(26)	111 (1)	C(2)–C(1)–O(27)	126 (1)
O(26)–C(1)–O(27)	123 (1)	C(1)–C(2)–C(3)	110 (1)
C(1)–C(2)–C(16)	110 (1)	C(3)–C(2)–C(16)	114 (1)
C(2)–C(3)–C(4)	113 (1)	C(2)–C(3)–O(29)	112 (1)
C(4)–C(3)–O(29)	108 (1)	C(3)–C(4)–C(5)	110 (1)
C(3)–C(4)–C(17)	111 (1)	C(5)–C(4)–C(17)	114 (1)
C(4)–C(5)–C(6)	115 (1)	C(4)–C(5)–O(33)	113 (1)
C(6)–C(5)–O(33)	104 (1)	C(5)–C(6)–C(7)	115 (1)
C(5)–C(6)–C(18)	112 (1)	C(7)–C(6)–C(18)	112 (1)
C(5)–C(6)–O(28)	106 (1)	C(7)–C(6)–O(28)	105 (1)
C(18)–C(6)–O(28)	107 (1)	C(6)–C(7)–C(8)	103 (1)
C(7)–C(8)–C(9)	100 (1)	C(7)–C(8)–C(19)	116 (1)
C(9)–C(8)–C(19)	115 (1)	C(8)–C(9)–C(10)	117 (1)
C(8)–C(9)–C(28)	106 (1)	C(10)–C(9)–O(28)	108 (1)
C(8)–C(9)–O(43)	106 (1)	C(10)–C(9)–O(43)	111 (1)
O(28)–C(9)–O(43)	110 (1)	C(9)–C(10)–C(11)	110 (1)
C(9)–C(10)–C(20)	111 (1)	C(11)–C(10)–C(20)	110 (1)
C(10)–C(11)–C(12)	119 (1)	C(10)–C(11)–O(24)	108 (1)
C(12)–C(11)–O(24)	104 (1)	C(11)–C(12)–C(13)	110 (1)
C(11)–C(12)–C(21)	117 (1)	C(13)–C(12)–C(21)	117 (1)
C(11)–C(12)–O(25)	100 (1)	C(13)–C(12)–O(25)	105 (1)
C(21)–C(12)–O(25)	107 (1)	C(12)–C(13)–C(14)	115 (3)
C(12)–C(13)–O(26)	106 (1)	C(14)–C(13)–O(26)	99 (2)
C(13)–C(14)–C(15)	93 (3)	O(23)–C(22)–O(24)	128 (1)
O(23)–C(22)–O(25)	122 (1)	O(24)–C(22)–O(25)	110 (1)
C(11)–O(24)–C(22)	113 (1)	C(12)–O(25)–C(22)	114 (1)
C(1)–O(26)–C(13)	115 (1)	C(6)–O(28)–C(9)	110 (1)
C(3)–O(29)–C(44)	114 (1)	C(44)–O(30)–C(48)	115 (1)
C(46)–O(32)–C(51)	118 (1)	C(39)–O(34)–C(40)	114 (1)
O(35)–C(36)–C(37)	110 (1)	O(35)–C(36)–C(40)	110 (1)
C(37)–C(36)–C(40)	107 (1)	C(36)–C(37)–C(38)	112 (1)
C(36)–C(37)–N(53)	107 (1)	C(38)–C(37)–N(53)	118 (1)
C(37)–C(38)–C(39)	110 (1)	O(34)–C(39)–C(38)	110 (1)
O(34)–C(39)–C(52)	108 (1)	C(38)–C(39)–C(52)	114 (1)
O(33)–C(40)–O(34)	107 (1)	O(33)–C(40)–C(36)	109 (1)
O(34)–C(40)–C(36)	108 (1)	O(29)–C(44)–O(30)	112 (1)
O(29)–C(44)–C(45)	109 (1)	O(30)–C(44)–C(45)	111 (1)
C(44)–C(45)–C(46)	116 (1)	O(32)–C(46)–C(45)	111 (1)
O(32)–C(46)–C(47)	103 (1)	C(45)–C(46)–C(47)	109 (1)
C(47)–C(46)–C(50)	112 (1)	C(45)–C(46)–C(50)	110 (1)
O(31)–C(47)–C(48)	108 (1)	O(31)–C(47)–C(46)	111 (1)
O(30)–C(48)–C(47)	109 (1)	C(46)–C(47)–C(48)	114 (1)
C(47)–C(48)–C(49)	115 (1)	O(30)–C(48)–C(49)	104 (1)
C(37)–N(53)–C(42)	112 (1)	C(37)–N(53)–C(41)	111 (1)
		C(41)–N(53)–C(42)	111 (1)

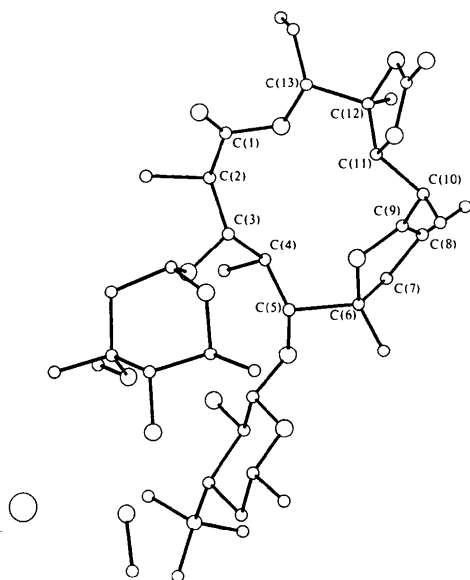


Fig. 2. The molecule of anhydroerythromycin A cyclic carbonate *N*-methyl iodide (Hempel *et al.*, 1975).

Table 6. *Intramolecular hydrogen-bond geometry*

O(31)···O(32)	2.68 Å	O(32)···H(310)	2.09 Å
O(31)—H(310)	1.07	C(47)—O(31)—H(310)	106°
O(35)···N(53)	2.75	N(53)···H(350)	1.83 Å
O(35)—H(350)	1.05	C(36)—O(35)—H(350)	85°
O(43)···O(26)	2.79	O(26)···H(430)	1.75 Å
O(43)—H(430)	1.05	C(9)—O(43)—H(430)	116°

anisotropic temperature factors and for H atoms a single isotropic temperature factor was refined. 3117 structure amplitudes were used throughout the refinement procedure.* The function minimized was $\sum |F_o - k|F_c||^2$. The final *R* value obtained was 0.080 where $R = \sum |F_o - k|F_c| / \sum |F_o|$. All the calculations were performed with the University of York DEC-10 computer. The positional parameters are given in Tables 2 and 3.

Discussion. The molecule and atom numbering are shown in Fig. 1. Bond distances and angles are presented in Tables 4 and 5 respectively. The results of the X-ray study indicate that the molecule occurs in a 6,9-hemiacetal form. The new asymmetric centre at

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33766 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C(9) has an *R* configuration. The configuration of the remaining asymmetric centres is the same as reported previously (Harris, McGeachin & Mills, 1965). In this structure C(14) and C(15) ethyl-group C atoms have unusually large thermal parameters. The temperature factors for these two atoms are, in order U_{11} , U_{22} , U_{33} , U_{23} , U_{13} , U_{12} (all $\times 10^4$): 3327 (244), 2654 (207), 564 (71), 447 (99), -885 (115), -2515 (203) for C(14) and 4224 (350), 1679 (253), 2412 (250), 1254 (216), 2543 (266), 1548 (268) for C(15). The final difference electron-density distribution revealed no peaks higher than 0.31 e \AA^{-3} . A possible explanation is that the ethyl group is disordered as it has the possibility of free rotation. To illustrate the differences between the conformation of the previously reported structure of an erythromycin carbonate derivative (Hempel, Bogucka-Ledochowska, Dauter, Borowski & Kosturkiewicz, 1975) and this one, similar drawings of both molecules are shown in Figs. 1 and 2. It can easily be seen that the ethyl group takes totally different conformations in the two structures, whose conformations are otherwise very similar. This is not surprising as there are no intermolecular forces in the crystal other than van der Waals. The intramolecular hydrogen-bond geometry is summarized in Table 6.

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