Table 1. *Atomic coordinates and equivalent isotropic temperature factors for the non-H atoms with e.s.d.'s in parentheses (atomic labeling as in Fig. 1)*

X refers to the equally distributed C and O atoms of the methanol molecule with an overall occupation factor of 0·5.

$U_{eq} = (U_1U_2U_3)^{1/3}$.

	x	*y*	*z*	U_{iso}
N(1)	−0·161 (1)	0·4265 (8)	0·1443 (3)	0·0832
C(1)	0·036 (1)	0·497 (1)	0·1517 (5)	0·0861
C(2)	0·1738 (9)	0·5019 (8)	0·2416 (5)	0·0720
C(3)	0·1121 (8)	0·4268 (7)	0·3315 (4)	0·0579
C(4)	−0·0931 (9)	0·3505 (8)	0·3244 (5)	0·0700
C(5)	−0·217 (1)	0·3550 (9)	0·2297 (6)	0·0783
C(6)	0·2469 (9)	0·4236 (8)	0·4294 (4)	0·0641
C(7)	0·4367 (9)	0·5047 (8)	0·4504 (4)	0·0607
X(8)	0·447 (2)	0·440 (1)	0·0216 (9)	0·099 (4)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

N(1)—C(1)	1·334 (8)	C(3)—C(6)	1·450 (7)
N(1)—C(5)	1·314 (8)	C(4)—C(5)	1·382 (9)
C(1)—C(2)	1·375 (8)	C(6)—C(7)	1·337 (7)
C(2)—C(3)	1·389 (7)	C(7)—C(7′)	1·44 (1)
C(3)—C(4)	1·397 (7)	C(8)—O(8)	1·29 (2)

C(5)—N(1)—C(1)	115·2 (6)	C(6)—C(3)—C(4)	119·5 (6)
C(2)—C(1)—N(1)	124·0 (6)	C(5)—C(4)—C(3)	118·0 (6)
C(3)—C(2)—C(1)	120·1 (6)	C(4)—C(5)—N(1)	126·2 (6)
C(4)—C(3)—C(2)	116·4 (5)	C(7)—C(6)—C(3)	126·2 (6)
C(6)—C(3)—C(2)	124·0 (5)	C(7)—C(7′)—C(6)	123·3 (7)

occupation of 0·5; the presence and the amount of this molecule were confirmed by NMR and chemical analysis. In order to take into account the equal distribution of C and O atoms at this position, this peak was refined with the nitrogen scattering factors. H atoms were placed in calculated positions (C—H 0·99 Å) after each refinement cycle, except those of the disordered atoms which were ignored. Full-matrix least-squares refinement (*CRYSTALS*; Watkin, Carruthers & Betteridge, 1985) on 78 parameters. Unit weights were used throughout. At the end of the refinement the maximum least-squares shift to e.s.d. was 0·01, the minimum and maximum

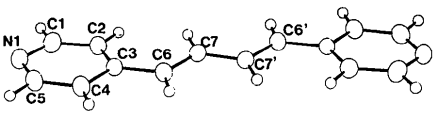


Fig. 1. Thermal ellipsoids drawn at the 30% probability level, excepting those of H which have $U_{iso} = 0·025$ Å^2 for clarity. The atoms with a superscript ′ are related to those without a superscript through an inversion center.

values of the residual electron density were −0·6 and 0·3 e Å^{-3}, respectively, and $R = 0·067$, $wR = 0·060$. Final positional parameters for the non-H atoms are listed in Table 1,* and bond lengths and bond angles in Table 2. An *ORTEP* (Johnson, 1976) drawing of the centrosymmetric molecule is shown in Fig. 1.

Related literature. The title compound has been synthesized in the course of a study of electron transfer through α,ω dipyridylpolyenes. The synthesis and spectral data have been described by Woitellier, Launay & Spangler (1989).

* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, all bond angles and details of selected least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52864 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1980). *MULTAN80. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

WATKIN, D. J., CARRUTHERS, J. R. & BETTERIDGE, P. W. (1985). *CRYSTALS. User Guide.* Chemical Crystallography Laboratory, Univ. of Oxford, England.

WOITELLIER, S., LAUNAY, J. P. & SPANGLER, C. W. (1989). *Inorg. Chem.* **28**, 758–762.

Acta Cryst. (1990). C**46**, 1567–1569

Structure of 3,3′,3″-Nitrilotripropionamide

BY F. EKKEHARDT HAHN,* MATTHIAS TAMM, ROMAN WEIMANN AND JOACHIM PICKARDT

Institut für Anorganische und Analytische Chemie, Technische Universität Berlin, Straße des 17. Juni 135, D-1000 Berlin 12, Federal Republic of Germany

(*Received* 26 *October* 1989; *accepted* 2 *March* 1990)

Abstract. $C_9H_{18}N_4O_3$, $M_r = 230·3$, rhombohedral, *R*3*c*, $a = 8·338$ (2) Å, $\alpha = 85·27$ (2)°, $V = 574·1$ (4) Å^3, $Z = 2$, D_m (by flotation) = 1·36 (1), $D_x = 1·332$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·09$ mm^{-1}, $F(000) = 248$, $T = 173$ (5) K, $R = 0·0198$ for 664 observed reflections. The central nitrogen

* To whom correspondence should be addressed.

0108-2701/90/081567-03$03.00

© 1990 International Union of Crystallography

atom resides on a special position with crystallographically imposed C_3 symmetry. The molecule has no unusual bond lengths or angles. Molecules are linked by N—H···O hydrogen bonds.

Experimental. Colourless prisms of the title compound were obtained from water. Crystal size 0·43 × 0·40 × 0·50 mm, Syntex $P2_1$ diffractometer, graphite monochromator. Cell constants from setting angles of 14 reflections with $10{\cdot}7 \le 2\theta \le 20{\cdot}2°$, ω–2θ scan with scan speed 1·54–29·30° min^{-1} in ω, scan width from 0·60° below $K\alpha_1$ peak to 0·60° above $K\alpha_2$ peak. Range of indices $-9 \le h \le 9$, $-9 \le k \le 9$, $-9 \le l \le 9$ ($2\theta_{max} = 50°$, 4027 data).

Reciprocal-lattice symmetry and systematic absences (hhl, $l = 2n$; hhh, $h = 2n$) are consistent with space group $R3c$; data corrected for Lorentz and polarization effects, no absorption correction, only statistical variation of three intensity standards; equivalent data merged in non-centrosymmetric point group $3m$ ($R_{int} = 0{\cdot}014$), leaving 672 unique reflections; 664 with $I \ge 3\sigma(I)$ used in refinement.

Structure solved by direct methods (*SHELXS*86; Sheldrick, 1986) and difference Fourier techniques (*SHELX*76; Sheldrick, 1976). Full-matrix least-squares refinement, minimized function $\sum w(|F_o| - |F_c|)^2$. Non-hydrogen atoms refined with anisotropic thermal parameters. All hydrogen atoms found in ΔF maps and included and refined with isotropic thermal parameters. The final refinement, based on 72 variables and 664 observations, resulted in $R = 0{\cdot}0198$; $wR = 0{\cdot}0203$; $w = 7{\cdot}55/\sigma^2(F)$, Δ/σ (max.) = 0·002, max. and min. heights in a final ΔF map 0·26 and −0·19 e Å^{-3}. Refinement of the inverse structure gave identical R and wR. Atomic scattering factors and real and imaginary dispersion terms were from *International Tables for X-ray Crystallography* (1974); other programs: *ORTEP*II (Johnson, 1976).

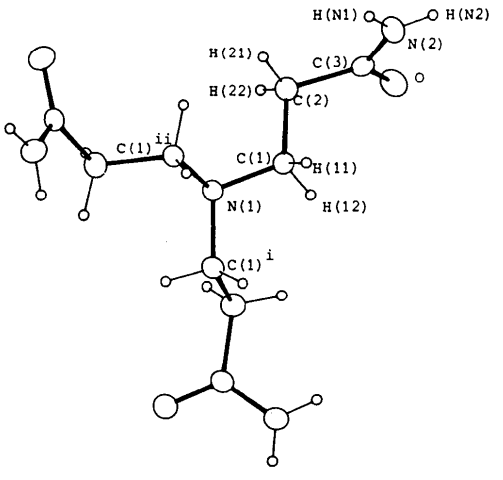


Fig. 1. A perspective view of one molecule of the title compound [symmetry code: (i) $z + 1$, x, $y - 1$; (ii) y, $z + 1$, $x - 1$].

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

	x	y	z	B_{eq}*
O	0·2182 (2)	0·7148 (2)	−0·0135 (2)	2·15
N(1)†	0·6787	0·6787	−0·3213	1·41
N(2)	0·3939 (2)	0·6423 (2)	0·1756 (2)	1·86
C(1)	0·5744 (2)	0·7302 (2)	−0·1814 (2)	1·48
C(2)	0·4773 (2)	0·5929 (2)	−0·1019 (2)	1·73
C(3)	0·3517 (2)	0·6548 (2)	0·0253 (2)	1·52
H(*N*1)‡	0·484 (2)	0·594 (2)	0·199 (2)	1·9
H(*N*2)	0·328 (2)	0·671 (2)	0·248 (2)	2·3
H(11)	0·636 (2)	0·773 (2)	−0·100 (2)	1·5
H(12)	0·497 (2)	0·817 (2)	−0·222 (2)	1·6
H(21)	0·551 (2)	0·509 (2)	−0·053 (2)	1·7
H(22)	0·429 (2)	0·548 (2)	−0·183 (2)	2·3

* Anistotropically refined atoms are given in the form of the isotropic equivalent thermal parameter defined as $B_{eq} = \frac{8}{3}\pi^2\{(aa^*)^2[U_{11} + U_{22} + U_{33} + 2\cos\alpha(U_{12} + U_{13} + U_{23})]\}$.

† N(1) resides on a special position on the threefold axis (Wyckoff letter *a*).

‡ Hydrogen positions were refined with isotropic thermal parameters.

Table 2. *Bond lengths* (Å) *and angles* (°), *and hydrogen-bond parameters* (Å *and* °)

N(1)—C(1)	1·468 (1)	N(2)—H(*N*1)	0·85 (2)
C(1)—C(2)	1·531 (1)	N(2)—H(*N*2)	0·82 (2)
C(2)—C(3)	1·519 (2)	C(1)—H(11)	0·99 (2)
C(3)—O	1·238 (2)	C(1)—H(12)	0·98 (1)
C(3)—N(2)	1·325 (2)	C(2)—H(21)	0·98 (2)
		C(2)—H(22)	0·93 (2)

C(1)—N(1)—C(1)^i	111·0 (1)	N(1)—C(1)—C(2)	111·6 (1)
C(1)—C(2)—C(3)	110·8 (1)	C(2)—C(3)—O	120·4 (1)
C(2)—C(3)—N(2)	116·5 (1)	N(2)—C(3)—O	123·1 (1)

A—H···B			H···B	A—H···B	A···B
N(2)	H(*N*1)	O^{iii}	2·09 (2)	170·2 (13)	2·933 (2)
N(2)	H(*N*2)	O^{iv}	2·18 (2)	162·3 (16)	2·870 (2)

Symmetry code: (i) $z + 1$, x, $y - 1$; (iii) $\frac{1}{2} + x$, $\frac{1}{2} + z$, $x - \frac{1}{2}$; (iv) $y - \frac{1}{2}$, $\frac{1}{2} + x$, $\frac{1}{2} + z$.

Table 1 lists fractional coordinates and equivalent isotropic thermal parameters. A list of bond distances and angles can be found in Table 2.* A perspective view of the title compound is shown in Fig. 1.

Related literature. The compound was first synthesized by Marsh (1963).

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52838 (6 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

JOHNSON, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

MARSH, J. T. (1963). *Tex. Rec.* **81**, 77–78.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELXS*86. Program for the solution of crystal structures. Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1990). C**46**, 1569–1570

Structure of a β-Caryophyllene Derivative

BY YOSHIHARU MATSUBARA, TOSHIKAZU UCHIDA AND TATSUSHI ONO

Department of Applied Chemistry, Kinki University, Kowakae, Higashi Osaka 577, Japan

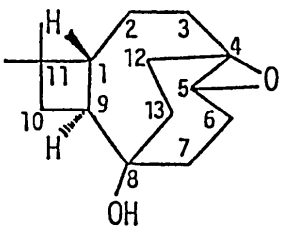
AND HIROZO KOYAMA*

Research Institute of Science and Technology, Kinki University, Kowakae, Higashi Osaka 577, Japan

(*Received* 20 *December* 1989; *accepted* 1 *February* 1990)

Abstract. $C_{15}H_{24}O_2$, $M_r = 236{\cdot}35$, trigonal, $P3_2$, $a = 13{\cdot}104$ (1), $c = 6{\cdot}756$ (1) Å, $V = 1004{\cdot}6$ (1) Å^3, $D_x = 1{\cdot}172$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 5{\cdot}982$ cm^{-1}, $F(000) = 390$, $T = 295$ K, $R = 0{\cdot}029$ for 1313 observed reflections. A novel sesquiterpenoid isolated from the oxidation of β-caryophyllene with lead tetraacetate is shown to be 5,5-dimethyl-12-oxatetracyclo[6.4.2.0^{1,11}.0^{4,7}]tetradecan-8-ol. The molecule consists of nine-, eight-, seven- and four-membered rings and the epoxide ring.

Experimental. Title compound (I): Crystal size 0·35 × 0·30 × 0·25 mm. Data were collected on the Rigaku AFC-5R four-circle diffractometer, monocromated Cu $K\alpha$ radiation. θ–2θ scans. Cell constants refined from 2θ values of 25 reflections in the range 15–25°.



(I)

1418 reflections measured, $(2\theta)_{max} = 140°$, range of indices $h = 0$ to 16, $k = -16$ to 16, $l = -8$ to 0, three check reflections monitored periodically showed no significant variation in intensity. 1330 unique reflections, of which 1313 with $F_o > 3\sigma(F_o)$ were used for all calculations. No absorption correction. The structure was solved by direct methods using *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). H atoms located by difference Fourier synthesis. Final full-matrix least-squares refinement was performed with all non-H atoms having anisotropic and H atoms having isotropic thermal parameters, with minimization of $\sum w(|F_o| - |F_c|)^2$. Final convergence at $R = 0{\cdot}029$, unit weights, $wR = 0{\cdot}029$, $S = 0{\cdot}33$ for 250 parameters, $(\Delta/\sigma)_{max} = 0{\cdot}02$. Final $(\Delta\rho)_{max} = 0{\cdot}3$ e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974).

Final atomic parameters are given in Table 1, bond distances and angles in Table 2.† Fig. 1 shows a thermal ellipsoid plot with the atomic numbering scheme.

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52652 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C(1)	0·6850 (2)	1·0259 (2)	0·86184	38 (1)
C(2)	0·7033 (3)	1·0925 (3)	1·0556 (7)	53 (1)
C(3)	0·6454 (3)	1·1697 (3)	1·0582 (7)	53 (1)
C(4)	0·5276 (2)	1·1099 (2)	0·9523 (6)	40 (1)
C(5)	0·5273 (2)	1·1364 (2)	0·7428 (6)	40 (1)
C(6)	0·4385 (3)	1·0471 (2)	0·6032 (6)	41 (1)
C(7)	0·4625 (2)	0·9450 (2)	0·5690 (6)	36 (1)
C(8)	0·4557 (2)	0·8740 (2)	0·7559 (6)	28·9 (9)
C(9)	0·5759 (2)	0·9006 (2)	0·8357 (6)	31 (1)
C(10)	0·6557 (3)	0·8709 (2)	0·7074 (7)	46 (1)
C(11)	0·7650 (2)	0·9737 (2)	0·8066 (7)	50 (1)
C(12)	0·4347 (2)	0·9951 (3)	1·0439 (6)	46 (1)
C(13)	0·3785 (2)	0·8827 (2)	0·9178 (6)	35 (1)
C(14)	0·8070 (3)	0·9330 (4)	0·9832 (9)	71 (2)
C(15)	0·8681 (3)	1·0515 (4)	0·673 (1)	78 (2)
O(16)	0·3963 (2)	0·7524 (1)	0·6879 (5)	36·9 (7)
O(17)	0·4867 (2)	1·1893 (2)	0·8879 (6)	54 (1)

* To whom correspondence should be addressed.

0108-2701/90/081569-02$03.00

© 1990 International Union of Crystallography