

Humulene Triepoxide

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Abstract. C₁₅H₂₄O₃, $M_r = 252.34$. Monoclinic, space \bar{A} , $\beta = 121.11(5)^\circ$ from diffractometer measurements group $C2/c$, $a = 36.98(4)$, $b = 8.72(4)$, $c = 21.25(2)$ (Mo $K\alpha$ radiation); $V = 5867.5 \text{ \AA}^3$, $Z = 16$, $F(000) =$

Table 1. Fractional atomic coordinates and thermal parameters ($\times 10^4$)The expression for the anisotropic temperature factor is $T = \exp[-2\pi^2(h^2 a^{*2} U_{11} + \dots + 2klb^* c^* U_{21})]$.

	<i>x</i>	<i>y</i>	<i>z</i>	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
O(1A)	3672 (2)	4661 (7)	1563 (3)	815 (37)	1003 (49)	679 (35)	-142 (35)	586 (32)	94 (35)
O(2A)	4426 (2)	489 (7)	1280 (4)	1080 (49)	602 (47)	1399 (56)	363 (42)	798 (45)	324 (40)
O(3A)	4618 (2)	6206 (7)	213 (3)	971 (43)	639 (43)	917 (42)	120 (35)	708 (37)	76 (34)
O(1B)	6347 (1)	7136 (6)	1009 (2)	551 (30)	678 (40)	377 (27)	-22 (28)	236 (24)	66 (28)
O(2B)	7461 (1)	3951 (6)	2838 (3)	590 (32)	563 (41)	643 (34)	-127 (30)	239 (27)	222 (29)
O(3B)	6899 (1)	8680 (6)	3978 (2)	657 (32)	634 (39)	472 (29)	-63 (29)	355 (26)	50 (29)

	Molecule A				Molecule B			
	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}	<i>x</i>	<i>y</i>	<i>z</i>	U_{iso}
C(1)	3988 (2)	5129 (10)	1394 (4)	593 (22)	6542 (2)	7742 (8)	1753 (3)	408 (18)
C(2)	3718 (2)	3844 (9)	1007 (4)	525 (20)	6438 (2)	6123 (9)	1616 (4)	415 (18)
C(3)	3885 (2)	2255 (10)	1239 (4)	650 (23)	6797 (2)	5022 (8)	1791 (4)	478 (19)
C(4)	4148 (2)	1736 (9)	908 (4)	557 (22)	7060 (2)	4652 (8)	2602 (4)	430 (19)
C(5)	4605 (2)	1952 (10)	1308 (4)	569 (21)	7449 (2)	5487 (9)	3083 (3)	397 (18)
C(6)	4869 (2)	2104 (9)	952 (4)	527 (20)	7642 (2)	5744 (8)	3908 (3)	403 (18)
C(7)	4959 (2)	3791 (10)	916 (4)	607 (22)	7518 (2)	7369 (8)	4012 (3)	431 (18)
C(8)	4573 (2)	4787 (9)	519 (4)	517 (20)	7048 (2)	7606 (8)	3634 (3)	381 (18)
C(9)	4524 (2)	6275 (9)	795 (4)	542 (21)	6824 (2)	9007 (9)	3259 (4)	431 (18)
C(10)	4082 (2)	6851 (10)	514 (4)	653 (23)	6364 (2)	8907 (9)	2664 (4)	518 (20)
C(11)	3944 (3)	6731 (10)	1088 (4)	709 (25)	6282 (2)	8942 (10)	1873 (4)	598 (22)
C(12)	3340 (3)	3999 (10)	244 (4)	684 (24)	6062 (2)	5410 (10)	1596 (4)	618 (22)
C(13)	4870 (3)	6964 (12)	1502 (5)	927 (30)	7057 (3)	10424 (11)	3236 (5)	753 (26)
C(14)	4667 (2)	1360 (10)	190 (4)	646 (24)	7503 (2)	4529 (9)	4254 (4)	545 (21)
C(15)	5299 (3)	1283 (11)	1453 (5)	851 (28)	8124 (2)	5673 (9)	4260 (4)	576 (21)

Hydrogen atom parameters. E.s.d.'s are approximately 25, 100 and 40 in *x*, *y* and *z* respectively.

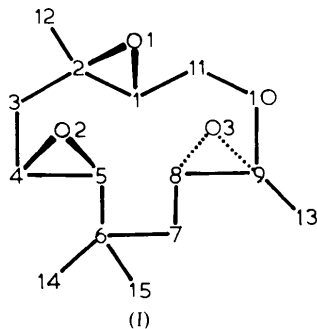
	Molecule A			Molecule B			Molecule A			Molecule B			
	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>	<i>x</i>	<i>y</i>	<i>z</i>	
H(1)	4276	4893	1773	6854	8107	1977	H(13)	3285	5048	106	5978	4570	1282
H(2)	3664	1551	1027	6969	5628	1633	H(14)	3132	3230	175	6166	4805	2169
H(3)	4106	2239	1728	6646	3943	1581	H(15)	3455	3662	-199	5844	6152	1487
H(4)	3965	1708	308	6889	4189	2826	H(16)	4854	6354	1852	7092	10307	2810
H(5)	4740	2622	1820	7541	6265	2836	H(17)	4907	7583	1359	7337	10264	3673
H(6)	5068	4174	675	7639	8087	3766	H(18)	5191	6486	1606	6884	11086	3200
H(7)	5128	4148	1411	7631	7556	4580	H(19)	4616	367	215	7197	4491	4126
H(8)	4314	4197	222	6865	6537	3504	H(20)	4402	1992	-220	7560	3533	4178
H(9)	3865	6206	41	6278	7731	2772	H(21)	4847	1588	-152	7639	4601	4842
H(10)	4083	7963	385	6189	9653	2733	H(22)	5427	1663	1948	8221	4550	4206
H(11)	3634	6964	798	6391	10052	1802	H(23)	5456	1527	1185	8211	6343	3964
H(12)	4137	7311	1492	5999	8576	1603	H(24)	5229	-25	1418	8256	5849	4789

Isotropic temperature factors ($U_{iso} \times 10^4$)

	Molecule A	Molecule B
H(1)–(12)	684	496
H(13)–(24)	954	723

2208, $\mu = 0.44 \text{ cm}^{-1}$, $D_c = 1.16 \text{ g cm}^{-3}$. The crystals contain two molecules per asymmetric unit, with identical conformations.

Introduction. The title compound (I) was recrystallized as needles, m.p. 125°C .



Systematic absences (from precession photographs) hkl for $h + k$ odd, $h0l$ for l odd indicated space group $C2/c$. Data were collected for $h0-6l$ with $\theta_{\max} = 22.5^\circ$ on a Stoe STADI-2 two-circle diffractometer (graphite-monochromated Mo $K\alpha$ radiation). There were 2719 unique data, of which 1760 with $I > 3\sigma(I)$ were used in subsequent calculations. Lorentz and polarization corrections (but none for extinction or absorption) were applied and the data scaled by a Wilson plot. The structure was solved by direct phasing methods with the *SHELX-76* program (Sheldrick, 1976). Complex neutral atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). H atoms were located from difference maps and given isotropic temperature factors which were allowed to refine. Only O atoms were refined anisotropically. Full-matrix least-squares refinement (unit weights) con-

Table 2. *Molecular geometry*

(a) Bond lengths (\AA). E.s.d.'s are all $1 \times 10^{-3} \text{\AA}$.

	A	B		A	B
C(1)-O(1)	1.45	1.46	C(6)-C(7)	1.52	1.54
-C(2)	1.44	1.45	-C(14)	1.53	1.52
C(11)	1.51	1.53	-C(15)	1.56	1.54
C(2)-O(1)	1.46	1.45	C(7)-C(8)	1.50	1.51
-C(3)	1.49	1.52	C(8)-O(3)	1.48	1.46
-C(12)	1.50	1.51	C(9)	1.47	1.46
C(3)-C(4)	1.53	1.51	C(9)-O(3)	1.47	1.43
C(4)-O(2)	1.42	1.43	-C(10)	1.51	1.51
-C(5)	1.46	1.46	-C(13)	1.51	1.52
C(5)-O(2)	1.43	1.45	C(10)-C(11)	1.55	1.55
-C(6)	1.52	1.53			

(b) Bond angles ($^\circ$). E.s.d.'s are $5-8 \times 10^{-1} (^\circ)$.

	A	B		A	B
C(2)-C(1)-C(11)	125.8	125.1	C(5)-C(6)-C(7)	108.9	107.6
-O(1)	60.8	59.9	-C(14)	113.6	112.3
C(11)-O(1)	116.3	115.4	-C(15)	108.2	106.8
C(1)-C(2)-C(3)	119.1	117.3	C(7)-C(14)	110.8	111.8
C(1)-C(12)	121.9	123.4	-C(15)	107.9	108.8
-O(1)	59.9	60.2	C(14)-C(15)	107.2	109.4
C(3)-C(12)	115.8	115.1	C(6)-C(7)-C(8)	114.8	113.4
O(1)	112.8	112.0	C(7)-C(8)-C(9)	124.2	124.4
C(12)-O(1)	113.3	116.0	-O(3)	116.7	116.8
C(2)-C(3)-C(4)	112.0	112.3	C(9)-O(3)	59.4	58.8
C(3)-C(4)-C(5)	120.8	120.8	C(8)-C(9)-C(10)	117.8	119.0
O(2)	114.7	115.4	-C(13)	122.2	121.7
C(5)-O(2)	59.2	60.1	-O(3)	59.4	60.5
C(4)-C(5)-C(6)	124.8	125.6	C(10)-C(13)	116.8	115.7
-O(2)	59.1	59.2	-O(3)	111.4	113.3
C(6)-O(2)	117.6	117.7	C(13)-O(3)	114.4	113.2
			C(9)-C(10)-C(11)	113.4	114.3
			C(10)-C(11)-C(1)	113.1	112.4
			C(1)-O(1)-C(2)	59.3	60.0
			C(4)-O(2)-C(5)	61.7	60.8
			C(8)-O(3)-C(9)	61.2	60.7

(c) Ring torsion angles ($^\circ$). The values found in humulene diepoxide by Cradwick, Cradwick & Sim (1973) are given for comparison (CCS).

	A	B	CCS		A	B	CCS
C(11)-C(1)-C(2)-C(3)	-156	-158	-153	C(6)-C(7)-C(8)-C(9)	-134	-142	-123
C(1)-C(2)-C(3)-C(4)	82	80	73	C(7)-C(8)-C(9)-C(10)	157	156	157
C(2)-C(3)-C(4)-C(5)	-93	-96	-89	C(8)-C(9)-C(10)-C(11)	-106	-101	-109
C(3)-C(4)-C(5)-C(6)	154	153	169	C(9)-C(10)-C(11)-C(1)	55	53	52
C(4)-C(5)-C(6)-C(7)	-99	-99	-106	C(10)-C(11)-C(1)-C(2)	74	80	80
C(5)-C(6)-C(7)-C(8)	56	60	47				

verged at $R = 0.075$ for 1760 observed reflexions ($R = \Sigma ||F_o| - |F_c|| / \Sigma |F_o|$). In the final cycle all shifts in parameters were less than their standard deviations. Atomic parameters are given in Table 1 and the molecular geometry in Table 2.*

Discussion. As part of a study of sesquiterpenoid transformations the crystal structure of (I) was undertaken to check the stereochemistry of the products from epoxidation of humulene (Parker, Roberts & Mitra, 1977). The observed structure (Fig. 1) is consistent with NMR data and has a very similar conformation to the related 1,2–8,9 diepoxide (Cradwick, Cradwick & Sim, 1973) from which it can be synthesized. The two molecules of (I) in the asymmetric unit have identical conformations and differ only slightly from the diepoxide [see Table 2(c)] and from the silver nitrate adduct of humulene (McPhail & Sim, 1966). This constancy of conformation supports the suggestion of Cradwick, Cradwick & Sim (1973) that humulene, although a liquid at room temperature, has a preferred conformation.

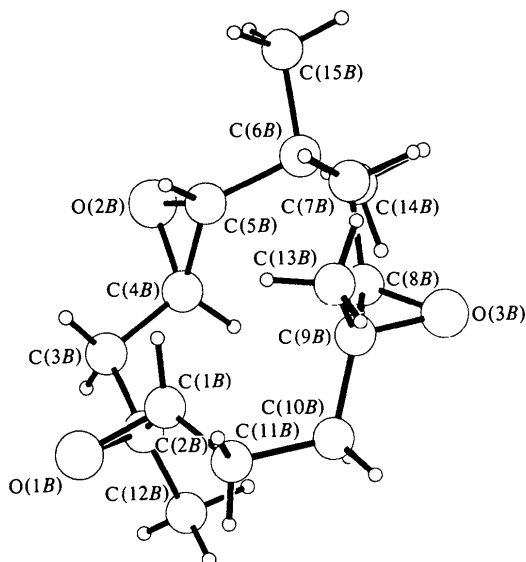


Fig. 1. General view of the molecule.

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32936 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

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A Neutron Diffraction Study of Lanthanum Magnesium Nitrate $\text{La}_2\text{Mg}_3(\text{NO}_3)_{12} \cdot 24\text{H}_2\text{O}$

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Abstract. $\text{La}_2\text{Mg}_3(\text{NO}_3)_{12} \cdot 24\text{H}_2\text{O}$, trigonal, $R\bar{3} (C_{3i}^2)$, $a = 13.172(3) \text{ \AA}$, $\alpha = 49.29^\circ$, 23°C , FW 1527.18, $Z = 1$, $V = 1207 \text{ \AA}^3$, $D_c = 2.101 \text{ g cm}^{-3}$ [hexagonal, $a = 10.989(2)$, $c = 34.63(1) \text{ \AA}$, $Z = 3$, $V = 3621(2) \text{ \AA}^3$]. The non-hydrogen atom positions were similar to

those found in the corresponding cerium double salt whereas some differences were found with the H positions. A hydrogen-bonding scheme is proposed in which seven H atoms form single bonds and one forms a weak bifurcated bond.

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Introduction. Lanthanum magnesium nitrate hydrate (LMN) is of great importance as a proton spin-polarized target in both nuclear physics and polarized