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endo–endo Diels–Alder Diadduct of 5,5-Dimethoxy-1,2,3,4-tetrachloro-1,3-cyclopentadiene with 1,5-Cyclooctadiene, (1), and the Dechlorinated Diels–Alder Diadduct, (2)

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**Abstract.** 1,6,7,8,9,14,15,16-Octachloro-17,17,18,18-tetramethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (1), $C_{22}H_{24}Cl_8O_4$, $M_r = 636\cdot1$, triclinic, $P\bar{1}$, $a = 7\cdot3110$ (7), $b = 7\cdot9195$ (5), $c = 12\cdot2113$ (10) Å, $\alpha = 84\cdot490$ (7), $\beta = 86\cdot064$ (7), $\gamma = 71\cdot490$ (8)°, $V = 666\cdot8$ (1) Å^3, $Z = 1$, $D_x = 1\cdot584$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 81\cdot7$ cm^{-1}, $F(000) = 324$, $T = 296$ K, $R = 0\cdot043$ for 2259 observations with $I > 3\sigma(I)$ (of 2659 unique data). 17,17,18,18-Tetramethoxypentacyclo[12.2.1.1^{6,9}.0^{2,13}.0^{5,10}]octadeca-7,15-diene (2), $C_{22}H_{32}O_4$, $M_r = 360\cdot5$, triclinic, $P\bar{1}$, $a = 6\cdot5121$ (7), $b = 8\cdot5391$ (8), $c = 9\cdot4741$ (4) Å, $\alpha = 67\cdot293$ (6), $\beta = 76\cdot580$ (6), $\gamma = 88\cdot202$ (8)°, $V = 471\cdot7$ (1) Å^3, $Z = 1$, $D_x = 1\cdot269$ g cm^{-3}, λ(Cu $K\alpha$) = 1·54184 Å, $\mu = 6\cdot45$ cm^{-1}, $F(000) = 196$, $T = 296$ K, $R = 0\cdot036$ for 1815 observations with $I > 3\sigma(I)$ (of 1942 unique data). The *endo–endo* isomer (1) of the four possible Diels–Alder diadducts is preferred at lower temperature. Both molecules lie on inversion centers, thus the cyclooctane ring in both structures adopts the chair conformation. The near-zero torsion angles of the chair occur at the ring-fusion bonds, and have magnitudes of 1·8 (4)° for the chlorinated compound (1) and 0·89 (13)° for the dechlorinated compound (2). The C=C bond has a length of 1·324 (3) in (1) and 1·329 (1) Å in (2).

Experimental. The title compound (1) was prepared by heating a 4:1 mixture of 5,5-dimethoxy-1,2,3,4-tetrachloro-1,3-cyclopentadiene with 1,5-cyclooctadiene to 453 K and extracting the white solid with pyridine (Akhtar, Fray & Yarrow, 1968). Crystals of the *endo–endo* isomer (1), grown by slow cooling of xylenes, m.p. 629–631 K (dec.), were suit-

able; a clear colorless crystal with dimensions 0·10 × 0·20 × 0·22 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $30 > \theta > 11°$. The ω–2θ scans were designed for $I = 25\sigma(I)$, subject to maximum scan time = 120 s, scan rates varied in the range 0·72–3·30° min^{-1}. One hemisphere of data having 2 $< \theta < 75°$, $0 \le h \le 9$, $-9 \le k \le 9$, $-15 \le l \le 15$ was measured and corrected for background, Lorentz and polarization effects, decay, and absorption. Absorption corrections were based on ψ scans, with minimum relative transmission coefficient 0·574. Three standard reflections (300, 020, 003) were measured every 10 000 s, exhibiting 19, 53, and 20% intensity decline, respectively. An anisotropic decay correction was applied. The structure was solved by direct methods, refined by full-matrix least squares based upon F, using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0\cdot02F_o^2)^2]^{-1}$ using the *Enraf–Nonius Structure Determination Package* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). C-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF and were refined with isotropic thermal parameters. Final $R = 0\cdot043$ for 2259 observed data (0·059 for all 2659 data), $wR = 0\cdot059$, and $S = 3\cdot303$ for 203 variables. Maximum shift 0·15σ in the final cycle, max. residual density 0·30, min. $-0\cdot48$ e Å^{-3}, and extinction coefficient $g = 8\cdot4$ (6) × 10^{-6} where the factor $(1 + gI_c)^{-1}$ was applied to F_c. A structural diagram of compound (1) is shown below while the fractional coordinates are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering for title molecule (1),

and Fig. 2 shows the unit cell. Bond distances, angles, and selected torsion angles for (1) are presented in Table 2.*

(1)
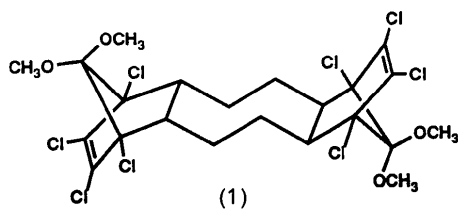
The title compound (2) was prepared by heating (1) with sodium metal, at reflux, in tetrahydrofuran and *tert*-butyl alcohol for 36 h, followed by extraction with dichloromethane (Gassman & Marshall, 1968). Crystals of the *endo–endo* isomer (2), grown by slow cooling and partial evaporation of a 1:1:1 mixture of xylenes:toluene:hexane, m.p. 512–515 K, were suitable; a clear colorless crystal with dimensions 0·18 × 0·32 × 0·50 mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer
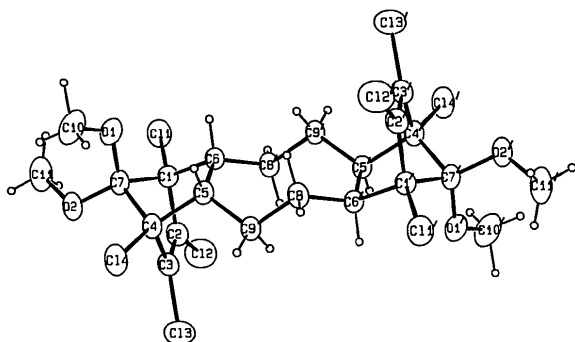

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule (1), representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.
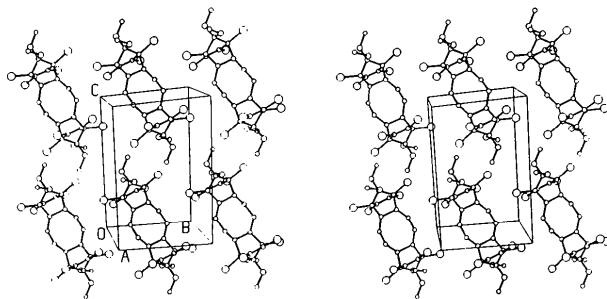

Fig. 2. Stereoview of the unit cell of (1).

Table 1. *Coordinates and equivalent isotropic thermal parameters for* (1)

$$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
Cl(1)	-0·2803 (1)	0·67907 (9)	0·33858 (5)	4·41 (2)
Cl(2)	-0·5855 (1)	0·5674 (1)	0·19640 (8)	5·23 (2)
Cl(3)	-0·3811 (1)	0·1432 (1)	0·12159 (7)	4·79 (2)
Cl(4)	0·0488 (1)	-0·02223 (9)	0·22287 (6)	4·45 (2)
O(1)	0·0928 (3)	0·3358 (3)	0·3496 (2)	4·06 (4)
O(2)	-0·1662 (3)	0·2300 (3)	0·4057 (2)	4·35 (5)
C(1)	-0·1994 (4)	0·4906 (3)	0·2605 (2)	2·94 (5)
C(2)	-0·3564 (4)	0·4315 (3)	0·2167 (2)	3·19 (5)
C(3)	-0·2766 (4)	0·2662 (3)	0·1880 (2)	3·17 (5)
C(4)	-0·0641 (4)	0·2117 (3)	0·2129 (2)	3·00 (5)
C(5)	0·0369 (3)	0·3176 (3)	0·1292 (2)	2·70 (5)
C(6)	-0·0622 (4)	0·5156 (3)	0·1609 (2)	2·71 (5)
C(7)	-0·0762 (4)	0·3108 (4)	0·3196 (2)	3·34 (6)
C(8)	0·1651 (4)	0·3469 (3)	-0·0710 (2)	2·95 (5)
C(9)	0·0280 (4)	0·2790 (3)	0·0100 (2)	2·90 (5)
C(10)	0·2422 (5)	0·1821 (5)	0·3918 (3)	5·63 (9)
C(11)	-0·2015 (6)	0·3106 (5)	0·5074 (3)	5·98 (9)

Table 2. *Bond distances* (Å), *angles* (°), *and selected torsion angles* (°) *for* (1)

Cl(1)—C(1)	1·768 (2)	C(1)—C(7)	1·561 (3)
Cl(2)—C(2)	1·699 (2)	C(2)—C(3)	1·324 (3)
Cl(3)—C(3)	1·703 (3)	C(3)—C(4)	1·519 (4)
Cl(4)—C(4)	1·769 (2)	C(4)—C(5)	1·555 (4)
O(1)—C(7)	1·390 (4)	C(4)—C(7)	1·568 (4)
O(1)—C(10)	1·432 (4)	C(5)—C(6)	1·579 (3)
O(2)—C(7)	1·411 (3)	C(5)—C(9)	1·525 (3)
O(2)—C(11)	1·421 (4)	C(6)—C(8)	1·523 (3)
C(1)—C(2)	1·515 (4)	C(8)—C(9)	1·542 (4)
C(1)—C(6)	1·561 (3)		

C(7)—O(1)—C(10)	117·7 (3)	C(3)—C(4)—C(5)	108·8 (2)
C(7)—O(2)—C(11)	116·7 (3)	C(3)—C(4)—C(7)	100·3 (2)
Cl(1)—C(1)—C(2)	115·6 (2)	C(5)—C(4)—C(7)	101·4 (2)
Cl(1)—C(1)—C(6)	112·6 (2)	C(4)—C(5)—C(6)	101·9 (2)
Cl(1)—C(1)—C(7)	117·4 (2)	C(4)—C(5)—C(9)	113·3 (2)
C(2)—C(1)—C(6)	107·5 (2)	C(6)—C(5)—C(9)	117·6 (2)
C(2)—C(1)—C(7)	100·2 (2)	C(1)—C(6)—C(5)	102·2 (2)
C(6)—C(1)—C(7)	101·9 (2)	C(1)—C(6)—C(8)	113·2 (2)
Cl(2)—C(2)—C(1)	124·2 (2)	C(5)—C(6)—C(8)	117·6 (2)
Cl(2)—C(2)—C(3)	127·8 (2)	O(1)—C(7)—O(2)	113·4 (2)
C(1)—C(2)—C(3)	107·7 (2)	O(1)—C(7)—C(1)	107·2 (2)
Cl(3)—C(3)—C(2)	127·5 (2)	O(1)—C(7)—C(4)	116·9 (2)
Cl(3)—C(3)—C(4)	125·1 (2)	O(2)—C(7)—C(1)	118·1 (2)
C(2)—C(3)—C(4)	107·1 (2)	O(2)—C(7)—C(4)	108·9 (2)
Cl(4)—C(4)—C(3)	113·3 (2)	C(1)—C(7)—C(4)	90·9 (2)
Cl(4)—C(4)—C(5)	114·6 (2)	C(6)—C(8)—C(9)	113·9 (2)
Cl(4)—C(4)—C(7)	117·0 (2)	C(5)—C(9)—C(8)	114·1 (2)

|			
C(10)—O(1)—C(7)—O(2)	56·5 (3)	C(11)—O(2)—C(7)—O(1)	52·2 (3)
C(7)—C(1)—C(2)—C(3)	-35·7 (3)	C(7)—C(1)—C(6)—C(5)	36·6 (2)
C(1)—C(2)—C(3)—C(4)	0·3 (3)	C(7)—C(4)—C(5)—C(6)	-39·5 (2)
C(4)—C(5)—C(6)—C(1)	1·8 (2)	C(4)—C(5)—C(6)—C(8)	-122·7 (2)
C(9)—C(5)—C(6)—C(8)	1·8 (3)	C(6)—C(5)—C(9)—C(8')	74·8 (3)
C(5)—C(6)—C(8)—C(9')	-78·3 (3)	C(5)—C(9)—C(8)—C(6')	-119·0 (2)

with Cu $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $36 > \theta > 25°$. The $\omega-2\theta$ scans were designed for $I = 25\sigma(I)$, subject to maximum scan time = 90 s, scan rates varied from 0·61–3·30° min^{-1}. A full sphere of data having $2 < \theta < 75°$, $-8 \le h \le 8$, $-10 \le k \le 10$, $-11 \le l \le 11$ was measured and corrected for background, Lorentz and polarization effects, and absorption. Absorption

Table 3. *Coordinates and equivalent isotropic thermal parameters for* (2)

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i{}^*a_j{}^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$B_{eq}(\text{Å}^2)$
O(1)	0·5754 (1)	0·16339 (9)	0·73955 (8)	3·44 (1)
O(2)	0·9369 (1)	0·18435 (8)	0·63884 (8)	3·34 (1)
C(3)	0·7861 (2)	0·1953 (1)	0·9057 (1)	2·94 (2)
C(2)	0·9828 (2)	0·3057 (1)	0·8715 (1)	3·37 (2)
C(3)	0·9836 (2)	0·4435 (1)	0·7425 (1)	3·25 (2)
C(4)	0·7896 (2)	0·4282 (1)	0·6854 (1)	2·78 (2)
C(5)	0·5942 (1)	0·4516 (1)	0·80569 (9)	2·65 (2)
C(6)	0·5923 (2)	0·2893 (1)	0·9597 (1)	2·73 (2)
C(7)	0·7710 (1)	0·2337 (1)	0·7352 (1)	2·81 (2)
C(8)	0·6005 (2)	0·3174 (1)	1·1084 (1)	3·07 (2)
C(9)	0·6074 (2)	0·6262 (1)	0·8134 (1)	2·93 (2)
C(10)	0·5261 (2)	0·2139 (2)	0·5904 (1)	4·59 (3)
C(11)	0·9727 (2)	0·0089 (1)	0·7020 (2)	4·66 (3)

corrections were based on ψ scans, with minimum relative transmission coefficient 0·948. Three standard reflections (500, 030, 003) showed only random fluctuation in intensity so no correction for decay was applied. Data from the two equivalent hemispheres were averaged, $R_{int} = 0·011$. Structure solution and refinement were identical to those for (1). Final $R = 0·036$ for 1815 observed data (0·038 for all 1942 unique data), $wR = 0·058$, and $S = 3·685$ for 183 variables. Maximum shift 0·11σ in the final cycle, max. residual density 0·21, min. $-0·13$ e Å^{-3}, and extinction coefficient $g = 3·01$ (13) $\times 10^{-5}$. A structural diagram of compound (2) is shown below while fractional coordinates are given in Table 3. Fig. 3 is a perspective drawing showing the atom numbering for title molecule (2), and Fig. 4 illustrates the unit cell. Bond distances, angles, and selected torsion angles for (2) are presented in Table 4.

CH₃O— OCH₃

(2)   OCH₃   OCH₃
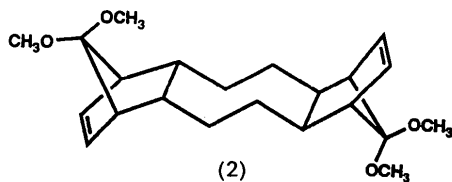
Related literature. Single-bond distances (Å) C(1)—C(6) and C(4)—C(5) of 1,2,4,5,6,7,8,9-octachloro-10,10-dimethoxytricyclo[5.2.1.0^{2,6}]deca-4,8-dien-3-one 1·572 (3) and 1·573 (3) (Galesic, Matijasic & Bruvo, 1985); 3,4,5,6-tetrachloro-12,12-dimethoxy-9,10-bis-(methoxycarbonyl)-*exo,endo*-pentacyclo[6.2.1.1^{3,6}.0^{2,7}]-dodeca-4,9-diene 1·550 (4) and 1·556 (3) (Battiste, Griggs, Sackett, Coxon & Steel, 1987); {9,10-η^2:11σ-3,4,5,6-tetrachloro-12,12-dimethoxy-9,10-bis(meth-oxycarbonyl)*exo,endo*-tetracyclo[6.2.1.1^{3,6}.0^{2,7}]do-deca-4,9-dien-11-yl}(η^5-cyclopentadienyl)nickel(II) 1·546 (3) and 1·550 (3) (Battiste *et al.*, 1987); 1,2,3,4,7,7-hexachloro-5,6-bis(chloromethyl)bicyclo-[2.2.1]hept-2-ene [Alodan(hoechst)®] 1·568 (7) and

1·549 (7) (Kennard, Smith & Palm, 1981); are similar to those in the title compounds (1) and (2): C(1)—C(6) and C(4)—C(5) bond distances of 1·561 (3) and 1·555 (4), and 1·566 (1) and 1·564 (1), respectively. The bond angles C(1)—C(7)—C(4) (1) [90·9 (2)°] and (2) [93·88 (8)°] of the title compounds are similar to the analogous bond angles of 1,2,4,5,6,7,8,9-octachloro-10,10-dimethoxytricyclo[5.2.1.0^{2,6}]deca-4,8-dien-3-one [91·6 (1)°]; 3,4,5,6-tetrachloro-12,12-dimethoxy-9,10-bis(methoxycarbonyl)-*exo,endo*-pentacyclo[6.2.1.1^{3,6}.0^{2,7}]-dodeca-4,9-diene and {9,10-η^2:11σ-3,4,5,6-tetrachloro-12,12-dimethoxy-9,10-bis-(methoxycarbonyl)-*exo,endo*-tetracyclo[6.2.1.1^{3,6}.0^{2,7}]-dodeca-4,9-dien-11-yl}(η^5-cyclopentadienyl)nickel(II) [90·9 (2)° and 90·7 (2)° respectively]; 1,2,3,4,7,7-hexachloro-5,6-bis(chloromethyl)bicyclo[2.2.1]hept-2-ene [Alodan(hoechst)®] [92·9 (4)°]. The C—C—C and O—C—O bridgehead angles of 90·9 (2)° for (1); 93·88 (8)° for (2) and 113·4 (2)° for (1); 109·94 (9)° for (2) respectively, as well as the O(1)—C(7)—O(2)—C(11) and O(2)—C(7)—O(1)—C(10) torsion angles of 52·23 (34)° and 56·46 (30)° for (1) and $-68·69$ (11)° and $-60·08$ (11)° for (2) respectively are in accord with literature data (Galesic, Matijasic & Bruvo, 1985; Van Hemelrijk & Lenstra, 1981; Van Hemelrijk, Lenstra & Geise, 1981, 1982). Intramolecular C(10)···C(11) distance 3·332 (5) Å for (1) is similar to that of (2), 3·510 (2) Å.
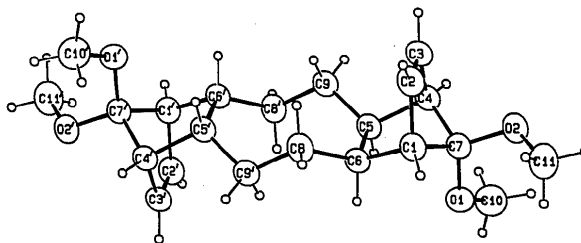
Fig. 3. *ORTEP* drawing (Johnson, 1965) of the molecule (2), representing heavy atoms as 50% probability ellipsoids and H atoms as circles of arbitrary radius.
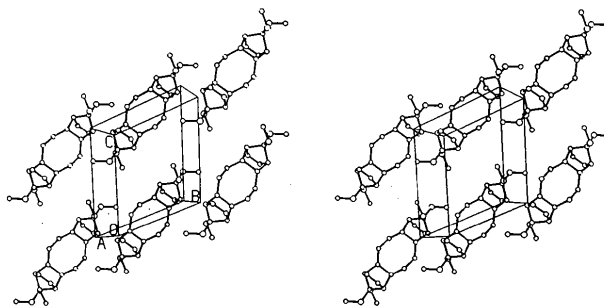

Fig. 4. Stereoview of the unit cell of (2).

Table 4. *Bond distances* (Å), *angles* (°), *and selected torsion angles* (°) *for* (2)

O(1)—C(7)	1·410 (1)	C(3)—C(4)	1·513 (2)
O(1)—C(10)	1·420 (1)	C(4)—C(5)	1·564 (1)
O(2)—C(7)	1·411 (1)	C(4)—C(7)	1·540 (1)
O(2)—C(11)	1·416 (1)	C(5)—C(6)	1·575 (1)
C(1)—C(2)	1·510 (1)	C(5)—C(9)	1·526 (1)
C(1)—C(6)	1·566 (1)	C(6)—C(8)	1·530 (2)
C(1)—C(7)	1·546 (1)	C(8)—C(9)	1·544 (1)
C(2)—C(3)	1·329 (1)		

C(7)—O(1)—C(10)	114·51 (7)	C(6)—C(5)—C(9)	118·16 (8)
C(7)—O(2)—C(11)	114·07 (7)	C(1)—C(6)—C(5)	102·81 (6)
C(2)—C(1)—C(6)	107·16 (9)	C(1)—C(6)—C(8)	112·43 (8)
C(2)—C(1)—C(7)	99·50 (7)	C(5)—C(6)—C(8)	117·59 (8)
C(6)—C(1)—C(7)	100·17 (7)	O(1)—C(7)—O(2)	109·94 (9)
C(1)—C(2)—C(3)	107·49 (9)	O(1)—C(7)—C(1)	108·94 (6)
C(2)—C(3)—C(4)	108·19 (8)	O(1)—C(7)—C(4)	116·30 (8)
C(3)—C(4)—C(5)	106·94 (9)	O(2)—C(7)—C(1)	117·44 (8)
C(3)—C(4)—C(7)	99·42 (7)	O(2)—C(7)—C(4)	109·81 (6)
C(5)—C(4)—C(7)	100·54 (6)	C(1)—C(7)—C(4)	93·88 (8)
C(4)—C(5)—C(6)	102·30 (7)	C(6)—C(8)—C(9)	114·39 (9)
C(4)—C(5)—C(9)	111·53 (7)	C(5)—C(9)—C(8)	115·83 (8)

C(10)—O(1)—C(7)—O(2)	−60·08 (11)	C(11)—O(2)—C(7)—O(1)	−68·69 (11)
C(7)—C(1)—C(2)—C(3)	34·32 (11)	C(7)—C(1)—C(6)—C(5)	−36·36 (9)
C(1)—C(2)—C(3)—C(4)	−0·81 (12)	C(7)—C(4)—C(5)—C(9)	164·32 (7)
C(4)—C(5)—C(6)—C(1)	−0·30 (9)	C(9)—C(5)—C(6)—C(8)	0·89 (13)
C(6)—C(5)—C(9)—C(8')	−77·93 (10)	C(6)—C(8)—C(9')—C(5')	−117·83 (9)
C(5)—C(6)—C(8)—C(9')	73·37 (11)		

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Redetermination of the Structure of Friedelin

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Abstract. $D:A$-Friedooleanan-3-one, $C_{30}H_{50}O$, $M_r = 426\cdot73$, orthorhombic, $P2_12_12_1$, $a = 6\cdot362$ (2), $b = 13\cdot923$ (2), $c = 28\cdot419$ (5) Å, $V = 2517$ (1) Å^3, $Z = 4$, $D_x = 1\cdot13$ g cm^{-3}, Cu $K\alpha$, $\lambda = 1\cdot5417$ Å, $\mu = 4\cdot92$ cm^{-1}, $F(000) = 952$, $T = 291$ K, $R = 0\cdot047$ for 3842 observed reflections. Friedelin was isolated for the first time from *Harungana madagascariensis* Lam. *ex* Poir (Clusiaceae). Recently, the crystal structure has been described [Mo, Winther & Scrimgeour (1989). *Acta Cryst.* B45, 261–270], but with relatively low precision ($R = 0\cdot133$ for 2201 reflections). Our data are of much better quality: mean e.s.d.'s of bond distances and angles are 0·003 Å and 0·2° here, com-

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