coefficients make it difficult to estimate any error on these values.

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Multidisciplinary Crystal Structure Analysis of a Bis(cadinane)

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Abstract

4,9-Diisopropyl-2,6a,12-trimethylperhydrobenzo-[*de*]naphthacene, $C_{30}H_{52}$, $M_r = 412.75$, orthorhombic, $P2_12_12_1$, $a = 10.106 (1)$, $b = 17.831 (3)$, $c = 29.666 (5)$ Å, $V = 5346 (1)$ Å^3, $Z = 8$, $D_x = 1.026$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu(Mo\ K\alpha) = 0.05$ mm^{-1}, $F(000) = 1856$, room temperature, $R = 0.056$ for 2334 unique reflections. The crystal structure is pseudo $B22_12$ and the two independent molecules have identical conformations. The molecule consists of two cadinane units and represents a novel class of

saturated $C_{30}H_{52}$ pentacyclic isoprenoid hydrocarbons. The structure was solved by combining structural information obtained from NMR spectroscopy, mass spectrometry and Patterson synthesis, and then applying the *PATSEE* Patterson search procedure of E. Egert and G. M. Sheldrick using the devised models. NMR indicates 30 sp^3 C atoms, comprising two isopropyl and three methyl groups and a C_{21} pentacyclic skeleton. Mass spectrometry suggested that the molecule is composed of two sesquiterpane units and the Patterson function gives a pattern indicative of condensed cyclohexane rings. Molecular

mechanics was used to calculate the geometries for three possible skeletons, of which one was rejected on the basis of the calculated energy and the two remaining models were used for the Patterson search. Positioned fragments were used for tangent expansion and peak optimization, which gave the complete structure.

Introduction

In 1982 the occurrence was reported of relatively abundant amounts of three new pentacyclic C_{30} compounds (coded W, T and R) in some Far-Eastern crude oils (Grantham, Postuma & Baak, 1983). These authors showed that the compounds presumably originate from tertiary land-plant resin. Crystals of compound T were originally spontaneously formed in 20-year-old molecular-distillation fractions of saturated hydrocarbons of crude oil. The isolation of crystalline material from another crude oil, to obtain larger quantities of pure material for analysis, and a discussion concerning the significance of the discovery of this novel compound is reported elsewhere (Cox et al., 1986). This paper reports the crystal structure analysis, which was successful only after much effort, because of pseudo-symmetry and the previously unknown molecular structure.

Experimental

A crystal of dimensions $0.45 \times 0.30 \times 0.15$ mm originating from the old distillation fraction was used to collect 7114 independent reflections up to $\theta_{max} = 28°$, hkl (max. range 13, 23, 39), on an Enraf–Nonius CAD-4 diffractometer using graphite-monochromatized Mo $K\alpha$ radiation in the ω-scan mode with $\Delta\omega = [(0.90 + 0.35 \tan(\theta)]°$ and a maximum scan time of 120 s or until $\sigma(I)/I < 2\%$. The unit cell was determined by least-squares refinement of 25 reflections in the range of $16 < 2\theta < 34°$. Variations in control reflections $(0,6,-20, 0,14,0, 0,8,20)$ were less than 3%. Intensities of reflections with $h + l = 2n + 1$ were very weak, indicating a pseudo-B centering and only 2334 reflections with $I \geq 2.5\sigma(I)$ were considered observed and used for structure refinement. For the application of direct methods 2735 weak reflections with $I \leq 0$ were scaled up and set to $0.5\sigma(I)$.

Structure determination

Preliminary efforts to solve the crystal structure by direct methods failed. MULTAN80 (Main et al., 1980) runs gave only puckered chicken-wire patterns, which could not be interpreted. Likewise, direct methods using SHELXS84 (Sheldrick, 1984) did not give promising results. Several obviously correctly oriented fragments, composed of condensed cyclohexane rings, were used to apply the TRADIR trans-

lation function (e.g. Bruin Slot & Beurskens, 1984) and the DIRDIF program (e.g. Parthasarathi, Beurskens & Bruin Slot, 1983), but this failed too. It was felt that detailed information on the molecular structure was necessary and that under these conditions Patterson search methods would be more promising than direct methods. In the middle of 1984 a preliminary version of the PATSEE program became available and it was decided to make an effort to solve the structure using this program, which incorporates direct methods in position refinement during the translation search.

The $F * F$ Patterson map indicates several prominent pseudo-symmetries with $u = \frac{1}{2}$, $v = 0$, $w = \frac{1}{2}$ as the highest peak with a maximum density of 56% relative to the origin peak, implying that the crystal structure is pseudo $B22_12$ and that two independent, or even symmetry-related molecules are related by this pseudo-translation vector, without excluding the possibility that different parts of the molecules may be related by this vector. A renormalized $E * F$ Patterson map was generated by the SHELXS84 program (Sheldrick, 1984) and used for searches with the PATSEE program (Egert & Sheldrick, 1985). The effect of this sharpening and renormalization on the Patterson map is well illustrated by the splitting of the $u = \frac{1}{2}$, $v = 0$, $w = \frac{1}{2}$ peak in the renormalized $E * F$ map into two maxima at $u = 0.45$, $v = 0$, $w = \frac{1}{2}$ and $u = 0.55$, $v = 0$, $w = \frac{1}{2}$ with relative heights of 27%.

The intramolecular vectors indicate that the molecules consist of many condensed cyclohexane rings and that at least one of the independent molecules or parts of them are slightly rotated out of the $b - c$ plane with respect to the c axis, and that the molecules are orientated more or less parallel to the c axis. High densities in the vector map are spread out in the u direction due to severe overlap of the vectors.

In the preliminary stages search models were used consisting of up to four condensed cyclohexane rings, but this of course gave many translation solutions with comparable figures of merit (FOM's; see Egert & Sheldrick, 1985) and none of the positioned fragments could be expanded to the complete structure. It was therefore necessary to use a larger search model and this was achieved by combining the conditions set by NMR, mass spectrometry and the observed vector map.

NMR spectra (Cox et al., 1986) indicate the presence of two different isopropyl groups and ^{13}C NMR shows that all C atoms have sp^3 hybridization, the most probable distribution of the multiplicity of the 30 C atoms being 7 primary, 9 secondary, 13 tertiary and 1 quaternary. This implies that the molecule has a pentacyclic C_{21} skeleton with two isopropyl and three methyl substituents. Information concerning the relative positions of the substituents was obtained by 2D ^1H NMR at 500 MHz indicating the presence of

four $\geq$CH–CH$_2$–CH$\leq$ sequences with the CH$_2$ group in the 2-position of a 1,3-diequatorially substituted cyclohexane ring. At this stage it became clear that compound T was not a triterpane in the usual sense (*e.g.* Hills & Whitehead, 1970). Accurate mass spectrometry showed compound T to be C$_{30}$H$_{52}$ and comparison with the reference spectra for cadinane [Moshonas & Lund, 1970; (I): 4βH,10βH-cadinane] and eudesmane [Alexander, Kagi & Noble, 1983; (II): 4βH-eudesmane] indicated that the molecule is possibly composed of two of these or other related sesquiterpenoid hydrocarbons.

(I)   (II)
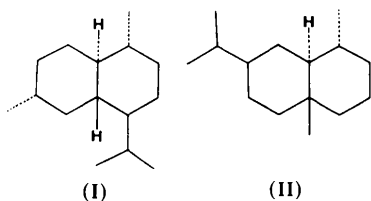
The pentacyclic C$_{21}$ skeleton can be obtained by joining two saturated sesquiterpene units in several ways. Connection of two units through a cyclopentane ring, by using one of the methyl groups, can be achieved in many different ways, but always results in an orientational difference between the units, which is in contradiction with the observed intramolecular vectors. *Trans* coupling through a cyclohexane ring can occur in three different ways in accordance with the observed intramolecular vectors (Fig. 1, models *A, B, C*). So far no mention has been made of the relative positions of the substituents. 2D ^{1}H NMR results are compatible with about ten different possibilities for these three models, but these were not considered mandatory for obtaining a suitable search model.

Patterson searches using the common parts of the three models gave many solutions with comparable FOM's, but none of them could be expanded to the complete structure.

Force-field calculations using the *MM*2 program (Allinger, 1977) were performed to generate the geometries of the three models and more accurate calculations including the H atoms resulted in comparable steric energies for models *A* and *B*, whereas the energy for model *C* was ~40 kJ mol^{-1} higher. It was therefore decided to concentrate on Patterson searches with *A* and *B* as search models. Rotation searches always gave an uncertainty of about 15° with respect to the orientation about the c axis, owing to broadening of the peaks in the u direction. Solutions with the best RFOM's* had the 2·5 Å (geminal C$\cdots$C) vector in the u–v plane more or less parallel to the v axis but this did not correspond with the observed maximum. However, solutions with some lower

RFOM's, which had this vector tilted out of the v – w plane, corresponded with the maximum in the Patterson map. Translation searches using the 'flat' oriented models almost always gave solutions with good CFOM's, but it was never possible to expand these structures because of their poor acentric information content.

Translation searches using two fragments with identical or different orientations failed too, as did translation searches with one fixed fragment and one free search fragment. Nevertheless, a translation search with 700 random positions using model *B*, with a tilted orientation and the fifth best RFOM, gave an outstanding CFOM and this solution was successfully expanded to the complete structure by using the tangent expansion and peak optimization (Sheldrick, 1982) of *SHELXS*84. It would also be interesting to apply the recently developed direct-method programs for superstructures (*e.g.* Cascarano, Giacovazzo & Luie, 1985) and to see whether these programs are able to solve the underlying phase problem without any structural information.

Structure refinement

Isotropic refinement of C atoms and riding H atoms was carried out with the *SHELX*76 (Sheldrick, 1976) program on a Cyber 855 computer of the University of Utrecht and converged at $R = 0·13$. Anisotropic refinement including the riding H atoms was not possible because of program limits and was therefore performed on an in-house DG-ECLIPSE s/230 minicomputer using a locally modified *SHELX*76 implementation. Final cycles of refinement on F with unit weights in three blocks of 180 parameters each and an overall scale factor converged only very slowly at $R = 0·056$ for 2334 observed reflections with $I \geq 2·5\sigma(I)$; $\Delta/\sigma_{mean} = 0·1$ (1) and $\Delta/\sigma_{max} = 2$. U_{11} parameters refined poorly and seven of these parameters had $\Delta/\sigma > 1·0$. H atoms were given an isotropic mean-square amplitude of 0·08 Å^2. Parameters of atoms related by the pseudo-B centering were refined in the same block, but no severe correlations ($r > 0·5$) were observed. The final residual electron density was $-0·16 < \rho < 0·21$ e Å^{-3}. The final atomic coordinates and the equivalent isotropic thermal parameters of

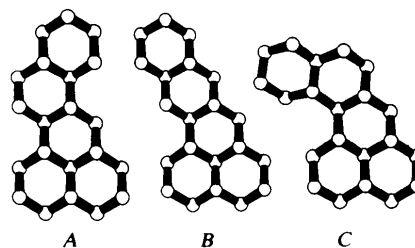*A*   *B*   *C*

Fig. 1. Pentacyclic C$_{21}$ search models based on NMR, mass spectrometry and Patterson-map information.

Table 1. *Positional and equivalent isotropic thermal parameters* ($Å^2$) *with e.s.d.'s in parentheses*

$$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$$

	Molecule (I)				Molecule (II)			
	x	y	z	U_{eq}	x	y	z	U_{eq}
C(1)	0·2237 (7)	0·4346 (4)	0·3493 (2)	0·048 (1)	0·1900 (7)	0·0660 (4)	0·1553 (2)	0·047 (1)
C(2)	0·2727 (7)	0·4302 (4)	0·3987 (2)	0·050 (2)	0·2114 (7)	0·0699 (4)	0·1040 (2)	0·053 (2)
C(3)	0·2703 (8)	0·5077 (4)	0·4205 (2)	0·062 (2)	0·1987 (8)	−0·0080 (4)	0·0823 (2)	0·061 (2)
C(4)	0·3429 (8)	0·5668 (4)	0·3936 (2)	0·059 (2)	0·2867 (7)	−0·0671 (4)	0·1050 (2)	0·059 (2)
C(5)	0·2845 (7)	0·5712 (4)	0·3461 (2)	0·056 (2)	0·2561 (8)	−0·0710 (4)	0·1552 (2)	0·059 (2)
C(6)	0·2903 (7)	0·4965 (4)	0·3213 (2)	0·048 (1)	0·2723 (7)	0·0050 (4)	0·1786 (2)	0·052 (2)
C(7)	0·2239 (7)	0·5006 (4)	0·2749 (2)	0·048 (1)	0·2343 (7)	0·0005 (4)	0·2294 (2)	0·055 (2)
C(8)	0·2788 (7)	0·5641 (4)	0·2447 (2)	0·057 (2)	0·3152 (7)	−0·0589 (4)	0·2548 (2)	0·058 (2)
C(9)	0·2063 (8)	0·5667 (4)	0·1986 (2)	0·058 (2)	0·2722 (8)	−0·0629 (4)	0·3047 (2)	0·059 (2)
C(10)	0·2574 (9)	0·6335 (4)	0·1706 (2)	0·079 (2)	0·3557 (9)	−0·1263 (4)	0·3290 (2)	0·082 (2)
C(11)	0·181 (1)	0·6341 (4)	0·1248 (2)	0·094 (2)	0·309 (1)	−0·1272 (5)	0·3788 (2)	0·091 (2)
C(12)	0·2046 (9)	0·5603 (4)	0·0998 (2)	0·083 (2)	0·3320 (9)	−0·0514 (4)	0·4016 (2)	0·088 (2)
C(13)	0·1520 (8)	0·4939 (4)	0·1274 (2)	0·069 (2)	0·2532 (8)	0·0100 (4)	0·3776 (2)	0·066 (2)
C(14)	0·2238 (8)	0·4914 (4)	0·1739 (2)	0·060 (2)	0·2932 (7)	0·0130 (4)	0·3277 (2)	0·056 (2)
C(15)	0·1679 (7)	0·4286 (4)	0·2036 (2)	0·059 (2)	0·2165 (7)	0·0732 (4)	0·3017 (2)	0·055 (2)
C(16)	0·2332 (7)	0·4260 (4)	0·2500 (2)	0·054 (2)	0·2518 (6)	0·0769 (4)	0·2522 (2)	0·045 (1)
C(17)	0·1681 (7)	0·3648 (4)	0·2786 (2)	0·058 (2)	0·1691 (7)	0·1359 (4)	0·2281 (2)	0·055 (2)
C(18)	0·2281 (7)	0·3560 (4)	0·3255 (2)	0·052 (2)	0·2007 (7)	0·1432 (4)	0·1773 (2)	0·052 (2)
C(19)	0·1411 (8)	0·3028 (4)	0·3528 (2)	0·067 (2)	0·981 (7)	0·1962 (4)	0·1557 (2)	0·063 (2)
C(20)	0·1847 (8)	0·2974 (4)	0·4022 (2)	0·064 (2)	0·1160 (8)	0·2024 (4)	0·1047 (2)	0·065 (2)
C(21)	0·1884 (7)	0·3731 (4)	0·4255 (2)	0·095 (2)	0·1143 (7)	0·1253 (4)	0·0818 (2)	0·055 (2)
C(22)	0·2272 (8)	0·3668 (5)	0·4761 (2)	0·068 (2)	0·1340 (7)	0·1333 (4)	0·0299 (2)	0·064 (2)
C(23)	0·1282 (9)	0·3159 (5)	0·5011 (3)	0·094 (2)	0·0277 (8)	0·1816 (5)	0·0083 (3)	0·089 (2)
C(24)	0·3700 (8)	0·3394 (5)	0·4827 (3)	0·092 (2)	0·2723 (8)	0·1614 (5)	0·0166 (3)	0·102 (2)
C(25)	0·3421 (8)	0·6435 (4)	0·4169 (2)	0·078 (2)	0·2707 (8)	−0·1437 (4)	0·0828 (2)	0·076 (2)
C(26)	0·229 (1)	0·7078 (5)	0·1937 (3)	0·112 (2)	0·333 (1)	−0·2017 (4)	0·3087 (3)	0·100 (2)
C(27)	0·1630 (8)	0·4188 (4)	0·1007 (2)	0·069 (2)	0·2668 (8)	0·0874 (4)	0·4023 (2)	0·076 (2)
C(28)	0·0784 (8)	0·4195 (5)	0·0578 (2)	0·095 (2)	0·212 (1)	0·0846 (5)	0·4498 (3)	0·114 (2)
C(29)	0·3015 (8)	0·3937 (5)	0·0913 (3)	0·105 (2)	0·4071 (9)	0·1189 (5)	0·4015 (3)	0·117 (2)
C(30)	0·3711 (7)	0·3247 (4)	0·3216 (2)	0·069 (2)	0·3432 (7)	0·1779 (4)	0·1729 (2)	0·067 (2)

the C atoms are given in Table 1. Geometrical computations were performed with the *EUCLID* package (Spek, 1982). Scattering factors were taken from Cromer & Mann (1968) for C atoms and from Stewart, Davidson & Simpson (1965) for H atoms.*

Discussion

The numbering system is the same in both independent molecules and is given in Fig. 2, which shows the molecular conformation of molecule (I), with arbitrary absolute configuration. Cleavage of bonds C(6)–C(7) and C(17)–C(18) gives two cadinane molecules and so compound T can be considered as a dimer of this sesquiterpane as was suggested by mass spectrometry experiments. All molecular features are consistent with NMR spectra, C(3), C(5), C(8) and C(15) being secondary C atoms in the 2-position of a 1,3-diequatorially substituted cyclohexane ring. The molecular structure of compound T represents a new class of saturated $C_{30}H_{52}$ hydrocarbons which do not obey the isoprene rule. It will be very interesting to see whether the two other isomers (compounds W and R) are also dimerization

products of cadinene or originate from one or two other sesquiterpenes and whether these compounds have the same pentacyclic skeleton (model B) or have skeletons according to models A or C. The mean bond lengths and bond angles involving C atoms are $1·54 (2)$ Å and $111 (2)°$ respectively, and have the same values for both molecules, the average absolute differences being $0·01$ Å and $0·8°$ respectively. The conformations of molecules (I) and (II) are identical as illustrated by the close agreement within 3σ of corresponding torsion angles. A least-squares fit of C(1) through C(21) gave a mean deviation of atomic positions of $0·06 (1)$ Å and including C(22) to C(30) resulted in a mean deviation of $0·09 (1)$ Å. The difference in conformation involves atoms C(10), C(11) and C(12) which deviate $0·17$, $0·19$ and $0·31$ Å respectively, and this may indicate a slight flexibility
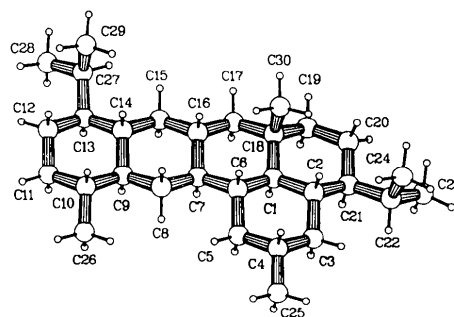
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* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates, bond lengths and angles, torsion angles, and several contour maps of sections through the Patterson function have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43261 (27 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Fig. 2. Molecular structure with atom numbering for the bis(cadinane) compound T.

in this region of the molecule. The conformation of the two isopropyl groups is given by the torsion angles C(12)-C(13)-C(27)-C(28) of $-62.9(8)$ and $-62\cdot1\,(9)°$, and C(20)-C(21)-C(22)-C(23) of $-58\cdot6(8)$ and $-58\cdot9\,(8)°$ for molecules (I) and (II) respectively. The molecules have a torsion in the C(12)-C(20) direction, as illustrated by the angles between the least-squares planes of the molecules and the C(21)-C(20), C(1)-C(18), C(7)-C(16), C(9)-C(14), C(11)-C(12) bonds of respectively $9\cdot4$, $12\cdot4$, $14\cdot3$, $17\cdot4$, $19\cdot8°$ for molecule (I) and $8\cdot7$, $12\cdot3$, $14\cdot6$, $15\cdot9$, $16\cdot6°$ for molecule (II). Energy minimization of the molecule with the $MM2$ program results in a close agreement between the two observed conformations and the minimized conformation. This is illustrated by the torsion in the C(12)-C(20) direction given by values of the previously defined angles of $12\cdot1$, $14\cdot6$, $14\cdot9$, $15\cdot2$ and $16\cdot5°$, respectively. The torsion angles C(12)-C(13)-C(27)-C(28) and C(20)-C(21)-C(22)-C(23) are $-56\cdot9$ and $-55\cdot7°$, respectively, and this conformation of the two isopropyl groups is a global minimum-energy conformation in vacuum as well; this was studied by rotating the isopropyl groups and successive energy minimizations.

The packing of the structure is illustrated in Fig. 3, which shows a stereoview down **a**. The two independent molecules are related by a pseudo-translation vector of $x = 0\cdot45$, $y = 0$, $z = 0\cdot50$. The crystal structure is therefore pseudo $B22_12$ and these molecules are slightly differently oriented as is given
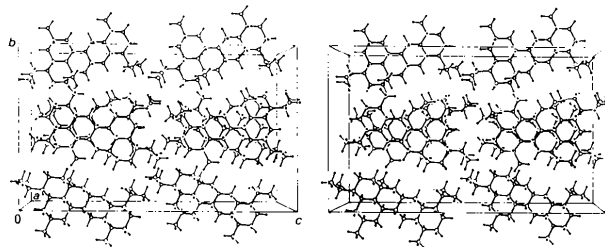
by the angle between the molecular mean planes of $11\cdot3°$. The orientation of the molecules is such that the screw axis parallel to **c** runs through the molecule parallel to the C(3)-C(13) vector. There are no H $\cdots$ H contacts less than $2\cdot0$ Å and no C $\cdots$ H contacts less than $3\cdot0$ Å. The shortest contact of $2\cdot03$ (1) Å is between the H atoms at C(13) of the independent molecules.

Fig. 3. Stereo packing diagram viewed down **a**.

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