

The Crystal and Molecular Structure of 22,29,30-Trisnorhopane II, C₂₇H₄₆

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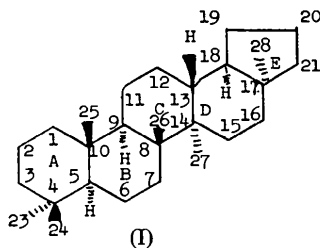
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22,29,30-Trisnorhopane II, C₂₇H₄₆, (triterpane *H*) is monoclinic, space group *P*2₁. The 1865 photographically recorded intensities were refined by block-diagonal least-squares calculations to a final *R* of 0.109. The structure is composed of four *trans*-fused chair rings, *A*, *B*, *C* and *D*, with a five-membered ring *E* *cis*-fused to ring *D*. A geminal dimethyl group is sited at C(4) and four other methyl groups at C(10), C(8), C(14) and C(17).

Introduction

As part of a programme on the constitution of crude petroleum several high molecular weight hydrocarbons have been extracted from a Nigerian crude oil with a view to providing information on the possible origin of petroleum. The preliminary separation of a number of pure pentacyclic compounds, initially labelled alphabetically, has been reported (Hills & Whitehead, 1966), and the structure of one, *E*, has been reported (Smith, 1970). This paper reports details of a second triterpane, *H*, which has been identified as 22,29,30-trisnorhopane II and has the formula (I).



Crystal data

Triterpane *H* crystallizes as thin monoclinic plates, the face of the plate being (001), with one edge parallel to *b*. Cell constants were obtained by extrapolation of successive orders of the principal rows from zero-layer Weissenberg photographs taken with Cu *K*α radiation ($\lambda = 1.5418 \text{ \AA}$).

Triterpane *H*: C₂₇H₄₆, *M* = 370.367 (*C* = 12), accurate *M* by mass spectrometry 370.362; m.p. 227.5–230.5 °C; *a* = 11.396 (5), *b* = 7.46 (1), *c* = 13.780 (6) Å, $\beta = 107.82 (6)^\circ$; *U* = 1115.3 Å³; space group *P*2₁, *Z* = 2, *D*_m = 1.100 (5), *D*_x = 1.103 g cm⁻³; $\mu(\text{Cu } K\alpha) = 4.5 \text{ cm}^{-1}$, *F*(000) = 416. Optical sign: positive. The density was measured by flotation in aqueous KI.

Experimental

A crystal of approximate dimensions 0.2 × 0.3 × 0.01 mm was used to record 1865 independent reflexions at room temperature by the multiple-film technique on

equi-inclination Weissenberg photographs about *b* for the layers *k* = 0 to 6 with Cu *K*α radiation. Intensities were measured by eye comparison with a calibrated scale, and after correction for the *L*_p factor the layers were put on an approximate common scale with *Ok**l* data collected about *a*. No absorption correction was made. Overall scale and temperature factors were obtained from a Wilson plot and the noncentrosymmetric space group confirmed by the *N*(*z*) test (Howells, Phillips & Rogers, 1950).

Structure determination

N.m.r. analysis indicated that compound *H* had six methyl groups whilst the accurate mass by mass spectrometry showed it to be C₂₇H₄₆. It therefore belonged to the family C_{*n*}H_{2*n*-8} and must be a trisnortriterpane, having lost an isopropyl group, with one ring, most likely *E*, being five-membered.

The unknown structure of *H* was solved along similar lines to that of *E* starting with trial structures using *Ok**l* and *h*0*l* data followed by model building in conjunction with the sharpened Patterson map. Three-dimensional trial structures quickly established the geometry of the *A*, *B* and *C* rings but many more attempts were required to find the *D* and *E* rings. Later, when computing ability had improved, the structure was re-solved with *MULTAN* (Germain, Main & Woolfson, 1971) in which 25 out of the 27 atoms were found in the first *E*₀ map.

Refinement

Isotropic refinement of carbon atoms alone was taken initially down to *R* = 0.18 with the Elliott 803 program system, and then on the University of London Atlas computer with the program system from NRC (Ahmed, Hall, Pippy & Huber, 1966). The block-diagonal approximation was used with an initial weighting scheme of the type $\sqrt{w} = 1$ if $F_o \leq F(\text{min})$ and $\sqrt{w} = F(\text{min})/F_o$ if $F_o > F(\text{min})$ with $F(\text{min}) = 10$. Scattering factors were taken from *International Tables for X-ray Crystallography*. As the molecule contains a large number of hydrogen atoms, which represent a substantial proportion of the total scattering power, their effect on the

refinement could not be ignored. Only some were visible in the F_o and difference maps and those hydrogen positions which were fixed by geometry were calculated on the basis of a C-H distance of 1.08 Å, whilst the approximate positions of the methyl hydrogen atoms were adjusted by computer program to satisfy threefold symmetry around their respective carbon atom. When the 28 hydrogen atoms with fixed positions were included, but not refined, the isotropic refinement of the carbon atoms alone converged at $R=0.14$. With the inclusion of the remaining 18 methyl hydrogen atoms, R fell to 0.134. All hydrogen atoms were given the same isotropic temperature factor as their associated carbon atoms. Subsequent attempts to refine the hydrogen coordinates failed. At this point, anisotropic refinement of the carbon atoms alone was started with a (9×9) matrix and the same weighting scheme with $F(\text{min})$ set to 4. The hydrogen-atom positions were recalculated after every three cycles and, throughout the refinement, layer scaling was carried out (Eichhorn, 1956). The 201 reflexion was omitted from the refinement as it appeared to be suffering from extinction. Refinement finally converged to $R=0.109$.

The final coordinates and thermal parameters for the carbon atoms are given in Table 1 and the calculated hydrogen-atom positions in Table 2. The calculated and observed structure factors are in Table 3.*

* Table 3 has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30693 (14 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. Fractional coordinates and thermal vibration parameters for the carbon atoms

The expression for the anisotropic temperature factor is $\exp[-(\beta_{11}h^2 + \beta_{22}k^2 + \beta_{33}l^2 + \beta_{23}kl + \beta_{13}hl + \beta_{12}hk)]$. Standard deviations in parentheses refer to the last decimal places. Values are $\times 10^4$.

	x/a	y/b	z/c	β_{11}	β_{22}	β_{33}	β_{23}	β_{13}	β_{12}
C(1)	2962 (5)	4249 (10)	2445 (4)	118 (5)	225 (16)	57 (3)	18 (11)	47 (6)	-28 (15)
C(2)	2705 (6)	3849 (11)	1304 (4)	146 (6)	280 (18)	55 (3)	16 (12)	58 (8)	-99 (17)
C(3)	2659 (6)	1891 (11)	1092 (4)	136 (6)	315 (19)	52 (3)	19 (12)	64 (7)	-31 (17)
C(4)	1758 (5)	816 (10)	1497 (4)	101 (5)	232 (15)	61 (3)	-15 (11)	44 (6)	-25 (14)
C(5)	2028 (4)	1345 (8)	2660 (4)	70 (3)	144 (12)	66 (3)	15 (10)	30 (5)	-6 (11)
C(6)	1294 (5)	265 (9)	3204 (4)	97 (4)	255 (17)	64 (3)	4 (12)	41 (6)	-51 (14)
C(7)	1900 (5)	436 (9)	4357 (4)	99 (4)	219 (15)	63 (3)	48 (11)	51 (6)	-31 (13)
C(8)	2005 (4)	2359 (9)	4744 (4)	82 (4)	187 (13)	65 (3)	5 (10)	37 (5)	-6 (12)
C(9)	2613 (4)	3503 (8)	4102 (4)	79 (4)	183 (14)	62 (3)	1 (10)	41 (5)	-1 (12)
C(10)	2049 (4)	3353 (9)	2898 (3)	85 (4)	196 (13)	49 (2)	13 (9)	32 (5)	-18 (12)
C(11)	2763 (5)	5422 (10)	4482 (4)	125 (5)	218 (16)	66 (3)	16 (12)	42 (7)	-94 (16)
C(12)	3558 (5)	5518 (11)	5595 (4)	123 (5)	260 (16)	58 (3)	14 (12)	58 (7)	-51 (16)
C(13)	3036 (4)	4360 (9)	6272 (4)	91 (4)	199 (14)	58 (3)	26 (10)	44 (6)	13 (12)
C(14)	2890 (4)	2395 (9)	5919 (4)	92 (4)	220 (14)	57 (3)	-3 (10)	48 (6)	-3 (13)
C(15)	2311 (6)	1406 (11)	6612 (4)	129 (5)	260 (17)	61 (3)	3 (13)	74 (7)	-13 (17)
C(16)	3110 (6)	1466 (12)	7739 (4)	158 (7)	309 (20)	66 (3)	46 (14)	82 (8)	-13 (20)
C(17)	3448 (5)	3377 (10)	8150 (4)	114 (5)	267 (16)	54 (3)	50 (12)	51 (6)	33 (15)
C(18)	3817 (4)	4635 (10)	7409 (4)	86 (4)	247 (15)	59 (3)	9 (11)	37 (6)	-5 (13)
C(19)	3626 (6)	6466 (11)	7806 (4)	161 (7)	235 (17)	63 (3)	-70 (13)	55 (8)	-24 (18)
C(20)	2620 (8)	6270 (13)	8294 (7)	176 (8)	258 (21)	136 (6)	-139 (19)	113 (12)	-0 (22)
C(21)	2352 (6)	4310 (12)	8330 (4)	128 (6)	328 (20)	64 (3)	13 (13)	85 (7)	43 (18)
C(23)	2013 (7)	-1113 (10)	1409 (4)	177 (7)	175 (15)	73 (3)	-22 (12)	76 (8)	4 (17)
C(24)	414 (6)	1132 (11)	844 (4)	132 (6)	257 (18)	61 (3)	-22 (12)	8 (7)	-36 (17)
C(25)	794 (5)	4311 (10)	2497 (4)	110 (5)	206 (16)	68 (3)	8 (12)	8 (7)	0 (14)
C(26)	702 (5)	2970 (10)	4694 (4)	92 (4)	223 (15)	80 (3)	-4 (12)	55 (6)	-9 (13)
C(27)	4165 (5)	1585 (10)	6015 (4)	97 (4)	266 (10)	59 (3)	31 (12)	33 (6)	61 (14)
C(28)	4510 (6)	3221 (12)	9149 (4)	155 (7)	337 (21)	57 (3)	35 (14)	45 (8)	63 (2)

Discussion of the structure

The ring structure of H is composed of four fused *trans*-chair six-membered rings A - D with a five-membered ring, E , *cis*-fused at C(17)-C(18). Methyl carbon atoms C(23)-C(27) are in expected positions but the sixth methyl atom C(28) is in a less common site which has an important bearing upon the origin of formation of the molecule.

The molecule is bent progressively from ring D to ring A as shown by the angles between the mean planes of these rings (Table 4) which are A - B 14.9°, B - C 10.2° and C - D 0.4°. Ring E is at an angle of 60.5° to ring D . The stereo view in Fig. 1 shows this, and it can also be seen in Fig. 2, which illustrates the splayed nature of the methyl groups C(24), C(25) and C(26). There would appear to be two possible reasons for this distortion: (a) the effect of the *cis* D/E ring junction and (b) a possible steric effect of the methyl hydrogen atoms associated with C(24), C(25) and C(26). A similar type of bending between the A and B rings is found in D (Smith, 1975), which possesses a ring structure identical with that of H . By contrast in the structure of E , the absence of a *cis* junction plus the modification to the A ring and the resultant dispersion of the positions of C(24) and C(25) leads to a regular molecular geometry.

The bond angles and bond distances are listed in Tables 5 and 6. The mean bond angle over 54 values is 110.4 (3.3)° so that all but two angles lie within 2σ . The two angles are those associated with the five-membered ring at C(17) and C(18). However, the mean C-C distance over 31 values is 1.532 (30) Å and all but

one distance lie inside 2σ . The odd distance C(8)–C(14) = 1.624 Å is just at the 3σ limit but it is thought that this is a significant departure from the average value

Table 2. Fractional coordinates ($\times 10^4$) and thermal parameters for the hydrogen atoms

The second and third digits of the hydrogen number refer to the bonded carbon atom. The positions of hydrogen atoms H(101) to H(221) are calculated positions based upon fixed geometry. Some of the positions of the methyl hydrogen atoms H(123)–H(328) were taken from an F_o map and with the remainder, their positions adjusted to give trigonal geometry around the methyl carbon atom. The isotropic temperature factor B is the temperature factor of the bonded carbon atom at the end of the last isotropic refinement cycle.

	x/a	y/b	z/c	B
H(101)	3894	3835	2850	4.539
H(201)	2958	5689	2545	4.539
H(102)	3413	4433	1027	5.290
H(202)	1836	4437	867	5.290
H(103)	3572	1334	1412	5.113
H(203)	2477	1649	300	5.113
H(105)	2966	905	3014	3.680
H(106)	1275	-1123	2981	4.492
H(206)	365	786	3004	4.492
H(107)	2813	-168	4562	4.476
H(207)	1399	-358	4751	4.476
H(109)	3544	3008	4271	3.877
H(111)	3185	6200	4017	4.747
H(211)	1871	5985	4398	4.747
H(112)	4486	5094	5660	4.974
H(212)	3611	6895	5846	4.974
H(113)	2124	4856	6197	3.995
H(115)	2135	40	6364	4.962
H(215)	1418	1999	6543	4.962
H(116)	3940	692	7822	5.769
H(216)	2634	776	8196	5.769
H(118)	4780	4445	7490	4.294
H(119)	4488	6861	8375	5.341
H(219)	3369	7387	7184	5.341
H(120)	2931	6803	9081	7.407
H(220)	1790	6965	7845	7.407
H(121)	2302	3942	9087	5.352
H(221)	1512	3976	7739	5.352
H(123)	2172	-1366	678	5.669
H(223)	1234	-1902	1446	5.669
H(323)	2824	-1501	2012	5.669
H(124)	-193	617	1254	5.457
H(224)	247	2531	699	5.457
H(324)	233	407	132	5.457
H(125)	630	4654	1692	4.926
H(225)	70	3438	2557	4.926
H(325)	796	5528	2915	4.926
H(126)	499	2549	5378	4.758
H(226)	38	2375	4036	4.758
H(326)	641	4410	4634	4.758
H(127)	4876	2494	6435	4.453
H(227)	4256	317	6393	4.453
H(327)	4241	1406	5254	4.453
H(128)	4484	4383	9616	6.060
H(228)	4420	2027	9552	6.060
H(329)	5386	3209	8988	6.060

Table 4. Equations to the mean planes of all five rings

Ring A	$-0.80768X + 0.35158Y - 0.47334Z + 2.048 = 0$
Ring B	$-0.91255X + 0.33383Y - 0.23626Z + 1.327 = 0$
Ring C	$-0.92896X + 0.36503Y - 0.06155Z + 0.293 = 0$
Ring D	$-0.92643X + 0.37159Y - 0.06036Z + 0.274 = 0$
Ring E	$-0.50271X + 0.06803Y - 0.86177Z + 9.410 = 0$

brought about in particular by the *cis* D/E ring junction. A very similar value, 1.622 Å, was obtained in *D* and both these values may be compared with the value 1.572 Å of *E*.

Table 5. Bond angles ($^\circ$)

Standard deviations are given in parentheses.

C(2)–C(1)–C(10)	112.9 (5)	C(5)–C(10)–C(25)	115.2 (5)
C(1)–C(2)–C(3)	112.1 (6)	C(9)–C(10)–C(25)	112.0 (4)
C(2)–C(3)–C(4)	115.9 (6)	C(9)–C(11)–C(12)	111.3 (5)
C(3)–C(4)–C(23)	106.8 (5)	C(11)–C(12)–C(13)	111.7 (5)
C(3)–C(4)–C(3)	108.4 (5)	C(12)–C(13)–C(14)	111.9 (5)
C(3)–C(4)–C(24)	111.6 (5)	C(12)–C(13)–C(18)	109.4 (5)
C(5)–C(4)–C(23)	110.0 (5)	C(14)–C(13)–C(18)	114.7 (5)
C(5)–C(4)–C(24)	113.2 (5)	C(8)–C(14)–C(13)	107.6 (4)
C(23)–C(4)–C(24)	106.9 (5)	C(8)–C(14)–C(15)	111.3 (5)
C(4)–C(5)–C(6)	114.0 (5)	C(8)–C(14)–C(27)	111.3 (5)
C(4)–C(5)–C(10)	116.2 (4)	C(13)–C(14)–C(15)	107.0 (5)
C(6)–C(5)–C(10)	112.9 (4)	C(13)–C(14)–C(27)	109.8 (5)
C(5)–C(6)–C(7)	109.1 (5)	C(15)–C(14)–C(27)	109.7 (5)
C(6)–C(7)–C(8)	114.0 (5)	C(14)–C(15)–C(16)	112.4 (6)
C(7)–C(8)–C(9)	108.8 (5)	C(15)–C(16)–C(17)	113.8 (6)
C(7)–C(8)–C(14)	108.9 (4)	C(16)–C(17)–C(18)	114.0 (5)
C(7)–C(8)–C(26)	107.0 (5)	C(16)–C(17)–C(21)	111.1 (6)
C(9)–C(8)–C(14)	108.2 (4)	C(16)–C(17)–C(28)	107.4 (6)
C(9)–C(8)–C(26)	114.1 (5)	C(18)–C(17)–C(21)	103.6 (5)
C(14)–C(8)–C(26)	109.8 (5)	C(18)–C(17)–C(28)	110.5 (5)
C(8)–C(9)–C(10)	116.9 (4)	C(22)–C(17)–C(28)	110.2 (6)
C(8)–C(9)–C(11)	110.2 (5)	C(13)–C(18)–C(17)	113.5 (5)
C(10)–C(9)–C(11)	113.4 (5)	C(13)–C(18)–C(19)	112.3 (5)
C(1)–C(10)–C(5)	108.2 (4)	C(17)–C(18)–C(19)	103.0 (5)
C(1)–C(10)–C(9)	106.7 (4)	C(18)–C(19)–C(20)	106.3 (6)
C(1)–C(10)–C(25)	109.0 (5)	C(19)–C(20)–C(21)	107.3 (7)
C(5)–C(10)–C(9)	105.4 (4)	C(17)–C(21)–C(20)	104.9 (6)

The effect of long-range steric strain has been discussed by Hall & Maslen (1965) for methyl metaleucate iodoacetate (MMI) and many of the features discussed are paralleled in the structures of *H* and *D*, but there are some points of difference. Whilst the steric effect

Table 6. Interatomic distances (Å)

Standard deviations in parentheses refer to the last two decimal places.

C(1)–C(2)	1.532 (7)	C(10)–C(25)	1.542 (8)
C(1)–C(10)	1.522 (8)	C(11)–C(12)	1.522 (8)
C(2)–C(3)	1.486 (12)	C(12)–C(13)	1.517 (9)
C(3)–C(4)	1.524 (9)	C(13)–C(14)	1.539 (9)
C(4)–C(5)	1.595 (7)	C(13)–C(18)	1.564 (7)
C(4)–C(23)	1.481 (11)	C(14)–C(15)	1.507 (9)
C(4)–C(24)	1.540 (9)	C(14)–C(27)	1.538 (8)
C(5)–C(6)	1.515 (8)	C(15)–C(16)	1.533 (8)
C(5)–C(10)	1.535 (9)	C(16)–C(17)	1.550 (12)
C(6)–C(7)	1.532 (7)	C(17)–C(18)	1.533 (9)
C(7)–C(8)	1.519 (9)	C(17)–C(21)	1.519 (10)
C(8)–C(9)	1.543 (8)	C(17)–C(28)	1.532 (8)
C(8)–C(14)	1.624 (7)	C(18)–C(19)	1.504 (11)
C(8)–C(26)	1.534 (8)	C(19)–C(20)	1.524 (12)
C(9)–C(10)	1.590 (6)	C(20)–C(21)	1.492 (13)
C(9)–C(11)	1.514 (9)		

of C(24), C(25) and C(26) undoubtedly contributes to the lengthening of C(8)–C(14), it seems that the strain produced by a five-membered *E* ring may be more important. For instance in triterpane *F* (Smith, Fowell &

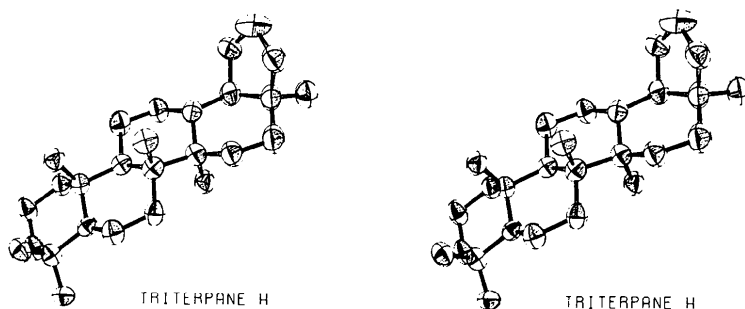


Fig. 1. Stereoscopic view of triterpane *H* drawn by *ORTEP* (Johnson, 1965). Vibration ellipsoids are scaled to 50% probability.

Melsom, 1970) the refined structure shows that the C(8)–C(14) bonds are 1.550 and 1.613 Å for the two molecules, mean 1.587 Å, which is not significantly different from 1.572 Å for *E*. In the latter, dispersion of the methyl groups C(24), C(25) and C(26) has occurred but both have identical arrangements for rings *C*, *D* and *E*, while *F* has ring arrangements of rings *A*, *B* and *C* identical with *H* and *D*.

Moreover the shortening of C(17)–C(22) and C(21)–C(22) in MMI [equivalent bonds are C(17)–C(21), C(20)–C(21) for *H* and *D*] is not evident and the increase in angles C(8)–C(9)–C(10) and C(14)–C(15)–C(16) is less marked. Also in *F*, *H* and *D*, a lower degree of distortion in the *A* and *B* rings is illustrated by the angles C(2)–C(3)–C(4) and C(4)–C(5)–C(10) which are respectively 4–5° and 3–4° less than those reported for MMI. However in all three the angle C(8)–C(9)–C(10) is in the range 115–117°, which although less than that reported for MMI, is in keeping with the repulsion of the C(25) methyl group by the other methyl groups C(24) and C(26) and is probably the prime cause of the bending between the mean planes of the *A* and *B* rings. Nevertheless, in none of the compounds is there a significant lengthening of C(4)–C(5) or C(5)–C(10) or a shortening of C(2)–C(3) or C(6)–C(7) noted in MMI. Neither can it be said that there is a significant increase of the bonds attached to the methyl groups C(24), C(25) and C(26).

It is felt therefore that some of the conclusions expressed by Hall & Maslen might be called into doubt and the results for MMI should be viewed with caution. The presence of large oxygenated groups at C(3), C(14) and C(17) plus the effect of a heavy atom, iodine, on the diffraction data could well produce the apparent extra molecular distortion. The geometry of pure hydrocarbon triterpanes has shown that these effects,

where they do occur, are less marked. As an example of the effect of absence of scattering power on calculated bond distances, the diffraction data for *E* were re-refined including the contribution of the 52 hydrogen atoms, whence the previously published lengths for C(2)–C(3) of 1.573, C(4)–C(23) of 1.545, C(17)–C(18) of 1.611 and C(18)–C(19) of 1.466 Å are now calculated as 1.505, 1.477, 1.567 and 1.533 Å respectively. The changes in the other bond distances are less substantial.

Triterpane *H* has been named as 22,29,30-trisnorhopane II although it could equally well be named as a derivative of lupane. However the derivation from lupane is considered to be energetically less likely since it would require the removal of the isopropyl group, followed by conversion of the less strained *trans*-fused *D/E* ring junction to a *cis*-fused arrangement and relocation of the C(28) methyl in the α position, all of which is contrary to the normal trend.

The alternative route makes use of the established conversion of derivatives of hopane, moretane, adianane, fernane and filicane from the ancient fern-like plants, under the severe acid isomerization conditions which are known to exist during crude oil maturation, to a hopene II system, where a double bond exists at C(13)–C(18).

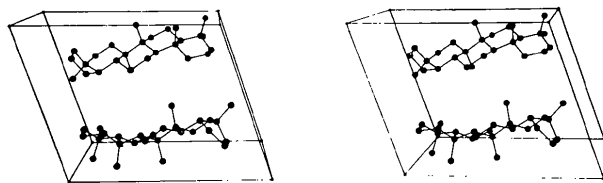
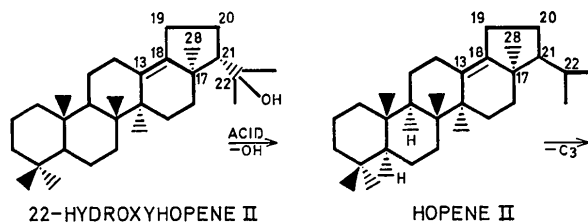
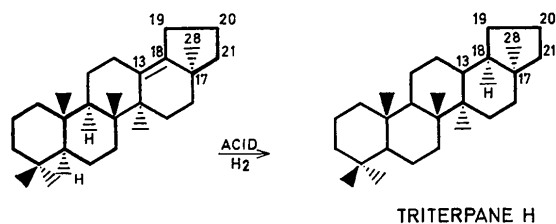


Fig. 2. Stereoscopic view showing packing in the unit cell, looking down *b* with *c* horizontal (Johnson, 1965).



Loss of the OH group leaves a migrating 21 (22) double bond blocked by the C(28) methyl at C(17) which could then be followed by loss of the isopropyl group by chemical or biological scission. Subsequent acid hydrogenation of the 13 (18) double bond from the now less hindered α face would produce the *D/E cis* junction and yield *H*. A more complete discussion of the origin and significance of triterpanes in crude petroleum is given by Whitehead (1973*a, b*).

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The Crystal and Molecular Structure of 29-Nor-17 α H-hopane, C₂₉H₅₀

By G. W. SMITH

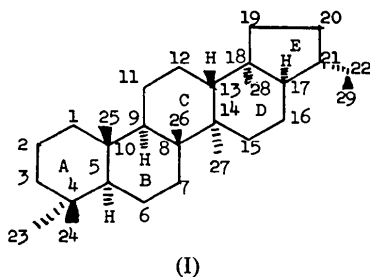
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The structure of 29-nor-17 α H-hopane (triterpane *D*) has been determined by direct methods from counter intensities. The compound is orthorhombic, space group *P*2₁2₁2₁, with *Z*=4, and the diffraction data were refined to *R*=0.120. The molecule is composed of four *trans*-fused six-membered chair rings, *A, B, C, D*, with a five-membered ring *E cis*-fused to ring *D*. A geminal dimethyl group is sited at C(4) with other methyl groups at C(10), C(8), C(14) and C(18) and an ethyl group at C(21).

Introduction

In two previous papers (Smith, 1970, 1975), the structures of the hitherto unknown triterpanes, *E* and *H* have been reported. A third unknown triterpane, *D*, another in a series of pentacyclic hydrocarbons extracted from a Nigerian crude petroleum (Hills & Whitehead, 1966), has been shown to be 29-nor-17 α H-hopane with formula (I).



N.m.r. evidence showed the presence of seven methyl groups and deductions had been made from mass spectrometry that compound *D* was likely to contain an ethyl group which would account for its relatively low melting point $\sim 180^\circ\text{C}$. This may be compared with the melting point of lupane $188\text{--}189^\circ\text{C}$ which possesses an isopropyl group, whereas the melting points of compounds *E* and *H*, neither of which contains rotatable groups are 260 and 233°C respectively.

Crystal data

Triterpane *D* crystallizes as orthorhombic laths or needles with the long axis parallel to *b*. Cell constants were obtained by extrapolation of successive orders of principal rows from zero-layer Weissenberg photographs taken with Cu *K* α radiation ($\lambda=1.5418 \text{ \AA}$). In addition there were sufficient high-angle reflexions on the *0kl* layer photograph to use the method of Farquhar & Lipson (1946) for better values of *b* and *c*.