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# 2,2,8-Trimethyltricyclo[6.2.2.0$^{1,6}$]dodec-5-ene-9,10-dicarboxylic Anhydride

BY PETER MURRAY-RUST AND JUDITH MURRAY-RUST

*Department of Chemistry, University of Stirling, Stirling FK9 4LA, Scotland*

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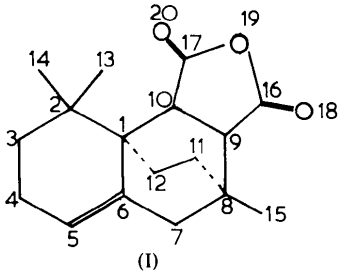
**Abstract.** $C_{17}H_{22}O_3$, $M_r$ = 274·4. Monoclinic, $P2_1/c$. $a$ = 9·44 (1), $b$ = 12·25 (1), $c$ = 13·10 (2) Å, $\beta$ = 108·02 (3)° from diffractometer measurements (Mo $K\bar{\alpha}$ radiation); $V$ = 1440·6 Å$^3$, $Z$ = 4, $F(000)$ = 592, $\mu$ = 0·49 cm$^{-1}$, $D_c$ = 1·26 g cm$^{-3}$. The compound contains a bicyclo[2.2.2] ring system in which torsion angles in the two-carbon bridges are about 14°.

**Introduction.** The title compound (I) was recrystallized from *n*-hexane.




(I)

Systematic absences (from precession photographs) *h*0*l*, *l* odd, and 0*k*0, *k* odd, indicated space group $P2_1/c$. Data were collected for 0–8*kl* with $\theta_{max}$ = 22·5° on a Stoe STADI-2 two-circle diffractometer (graphite-monochromated Mo $K\bar{\alpha}$ radiation). These gave 1579 data of which 1208 unique reflexions 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 system of crystallographic programs (Sheldrick, 1976), which was used for all calculations. Complex neutral atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974). Weighted full-matrix least-squares refinement (including isotropic H atoms) converged at $R$ = 0·048 for 1208 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.

Table 1. *Fractional atomic coordinates with e.s.d.'s given in parentheses* (*all* $\times 10^4$ *for* C *and* O, $\times 10^3$ *for* H)

| | *x* | *y* | *z* | | *x* | *y* | *z* |
|---|---|---|---|---|---|---|---|
| C(1) | 2437 (3) | 10764 (3) | 2807 (2) | H(1) | −22 (3) | 1011 (2) | 392 (2) |
| C(2) | 1217 (3) | 11013 (3) | 3339 (3) | H(2) | −5 (3) | 955 (2) | 291 (2) |
| C(3) | 554 (3) | 9947 (3) | 3597 (3) | H(3) | 124 (3) | 844 (3) | 439 (3) |
| C(4) | 1689 (4) | 9177 (3) | 4315 (3) | H(4) | 193 (4) | 940 (3) | 509 (3) |
| C(5) | 3042 (3) | 9097 (3) | 3983 (3) | H(5) | 369 (3) | 854 (3) | 426 (3) |
| C(6) | 3397 (3) | 9801 (3) | 3325 (2) | H(6) | 569 (3) | 995 (3) | 359 (3) |
| C(7) | 4793 (3) | 9697 (3) | 3013 (3) | H(7) | 510 (3) | 899 (3) | 299 (3) |
| C(8) | 4673 (3) | 10362 (3) | 2006 (3) | H(8) | 310 (3) | 1050 (3) | 53 (2) |
| C(9) | 3155 (3) | 10132 (3) | 1179 (2) | H(9) | 143 (2) | 1115 (2) | 123 (2) |
| C(10) | 1859 (3) | 10517 (3) | 1563 (2) | H(10) | 433 (3) | 1197 (3) | 165 (3) |
| C(11) | 4613 (4) | 11561 (3) | 2283 (3) | H(11) | 561 (3) | 1180 (2) | 270 (2) |
| C(12) | 3490 (4) | 11751 (3) | 2886 (3) | H(12) | 401 (3) | 1188 (3) | 361 (3) |
| C(13) | −24 (4) | 11737 (3) | 2634 (3) | H(13) | 281 (3) | 1240 (3) | 263 (3) |
| C(14) | 1905 (4) | 11632 (3) | 4417 (3) | H(14) | 39 (3) | 246 (3) | 243 (2) |
| C(15) | 5959 (4) | 10130 (4) | 1576 (3) | H(15) | −75 (3) | 1194 (3) | 305 (3) |
| C(16) | 2826 (4) | 8954 (3) | 903 (2) | H(16) | −61 (3) | 1137 (3) | 201 (3) |
| C(17) | 756 (4) | 9612 (3) | 1179 (2) | H(17) | 220 (3) | 1238 (3) | 427 (2) |
| O(18) | 3568 (3) | 8263 (2) | 678 (2) | H(18) | 287 (3) | 1118 (3) | 493 (3) |
| O(19) | 1410 (2) | 8701 (2) | 911 (2) | H(19) | 118 (3) | 1161 (3) | 485 (3) |
| O(20) | −556 (3) | 9594 (2) | 1039 (2) | H(20) | 591 (3) | 939 (3) | 129 (3) |
| | | | | H(21) | 693 (4) | 1031 (3) | 216 (3) |
| | | | | H(22) | 596 (4) | 1063 (3) | 99 (3) |

Table 2. *Bond distances* (Å) *and angles* (°)

For non-hydrogen atoms e.s.d.'s are about 0·005 Å for bonds and 0·3° for angles.

| | | | |
|---|---|---|---|
| C(1)–C(2) | 1·551 | C(8)–C(9) | 1·532 |
| –C(6) | 1·513 | –C(11) | 1·518 |
| –C(10) | 1·581 | –C(15) | 1·517 |
| –C(12) | 1·548 | C(9)–C(10) | 1·534 |
| C(2)–C(3) | 1·530 | –C(16) | 1·497 |
| –C(13) | 1·531 | C(10)–C(17) | 1·498 |
| –C(14) | 1·556 | C(11)–C(12) | 1·523 |
| C(3)–C(4) | 1·515 | C(16)–O(18) | 1·192 |
| C(4)–C(5) | 1·475 | –O(19) | 1·376 |
| C(5)–C(6) | 1·333 | C(17)–O(19) | 1·372 |
| C(6)–C(7) | 1·502 | –O(20) | 1·195 |
| C(7)–C(8) | 1·524 | Mean C–H | 0·99 (3) |
| | | | |
| C(2)–C(1)–C(6) | 112·1 | C(7)–C(8)–C(11) | 108·0 |
| –C(10) | 115·7 | –C(15) | 111·6 |
| –C(12) | 111·6 | C(9)–C(8)–C(11) | 104·5 |
| C(6)–C(1)–C(10) | 106·3 | –C(15) | 112·6 |
| –C(12) | 107·2 | C(11)–C(8)–C(15) | 111·3 |
| C(10)–C(1)–C(12) | 103·2 | C(8)–C(9)–C(10) | 112·2 |
| C(1)–C(2)–C(3) | 110·1 | –C(16) | 115·3 |
| –C(13) | 112·3 | C(10)–C(9)–C(16) | 104·4 |
| –C(14) | 110·1 | C(1)–C(10)–C(9) | 110·0 |
| C(3)–C(2)–C(13) | 109·8 | –C(17) | 118·1 |
| –C(14) | 107·5 | C(9)–C(10)–C(17) | 101·8 |
| C(13)–C(2)–C(14) | 107·0 | C(8)–C(11)–C(12) | 110·8 |
| C(2)–C(3)–C(4) | 114·1 | C(1)–C(12)–C(11) | 112·5 |
| C(3)–C(4)–C(5) | 111·7 | C(9)–C(16)–O(18) | 130·6 |
| C(4)–C(5)–C(6) | 124·1 | –O(19) | 110·1 |
| C(1)–C(6)–C(5) | 124·0 | O(18)–C(16)–O(19) | 119·3 |
| –C(7) | 113·4 | C(10)–C(17)–O(19) | 111·5 |
| C(5)–C(6)–C(7) | 122·6 | –O(20) | 130·2 |
| C(6)–C(7)–C(8) | 110·7 | O(19)–C(17)–O(20) | 118·2 |
| C(7) C(8)–C(9) | 108·5 | C(16)–O(19)–C(17) | 109·5 |

Positional parameters are given in Table 1 and bond distances and angles in Table 2.*

**Discussion.** The title compound (Fig. 1) had been produced as an intermediate in the synthesis of 2,2,8-trimethyltricyclo[6.2.2.$0^{1,6}$]dodec-5-ene (McDonald & Roberts, 1976) and the structure was undertaken to confirm that there is a double bond between C(5) and C(6). In general the C–C bond lengths appear somewhat short, probably as a result of librational motion of the molecule. Torsion angles in the two-carbon bridges of the [2.2.2] system (14·3, 17·4, 14·6°) are consistent with the shallow potential for twisting of this nucleus (Ermer & Dunitz, 1969).

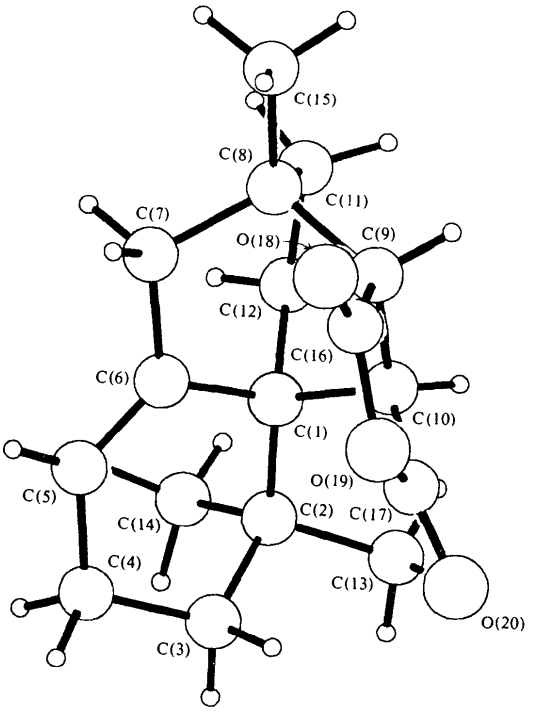



Fig. 1. General view of the molecule.

* Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32908 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

## References

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