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# 9-Methyl-9β,10α-oestr-4-en-3-one-17β-ol *p*-Bromobenzoate

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$C_{26}H_{25}O_3Br$, monoclinic, space group $P2_1$; $a=16{\cdot}84$ (2), $b=13{\cdot}26$ (1), $c=10{\cdot}18$ (1) Å, $\beta=95{\cdot}43$ (5)°; $Z=4$, $M=465{\cdot}39$, $D_x=1{\cdot}37$, $D_m=1{\cdot}36$ g cm$^{-3}$. The conformation of the unsaturated 4-en-3-one *A* ring is midway between a sofa and a half-chair. The conformations of the two unrelated molecules in the asymmetric unit do not differ significantly.

## Introduction

The total synthesis of 17β-hydroxy-9-methyl-9β,10α-oestr-4-en-3-one (Bull & Tuinman, 1972, 1973) is part of a study of the properties of skeletally modified steroids through the synthesis of steroids possessing the 9-methyl-19-nor-9β,10α-skeleton (Bull, Floor & Tuinman, 1975). The structure determination of the title compound was undertaken in order to confirm the structure assigned to this steroid.

The lattice parameters and intensities were measured on a Philips PW 1100 four-circle diffractometer in the ω–2θ mode with graphite-monochromated Mo Kα radiation and a crystal 0·2 × 0·2 × 0·2 mm. The systematic absences $k=2n+1$ for $0k0$ indicated space group $P2_1$. Data were collected at a scan rate of 0·02° s$^{-1}$ and a constant scan width of 0·8° in the range $\theta=3$ to 22°. The background was counted for half the total scanning time on each side of a reflexion. 2915 independent reflexions were measured of which 711 were considered unobserved with $I<1{\cdot}65\sigma$. No absorption corrections were applied ($\mu=19{\cdot}5$ cm$^{-1}$).

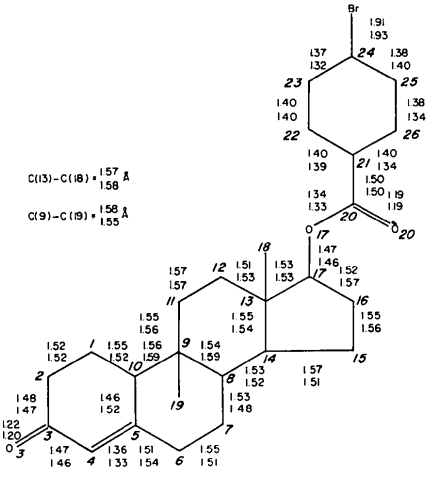


Fig. 1. Nomenclature and interatomic distances. Values for molecules *A* and *B* are given above and below respectively.

The structure was solved by conventional Patterson and Fourier techniques; H atoms, except those of the methyl groups, were located from a difference synthesis. Refinement was by least squares. In the final cycles non-hydrogen atoms were given anisotropic temperature factors and H atoms a constant isotropic temperature factor equal to the overall value estimated from a Wilson plot. The function minimized was $\sum w(\Delta F)^2$ with $1/\sigma_F^2$ weights. $R=\sum\Delta F/\sum F_o$ and $R_w=[\sum w(\Delta F)^2/\sum wF_o^2]^{1/2}$ converged to 0·055 and 0·061 respectively.

All calculations were done with the X-RAY system of crystallographic programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1972). The scattering factors of Stewart, Davidson & Simpson (1965) were used for H; those for all other atoms were generated from the analytical expressions of Cromer & Mann (1968). Final atomic parameters are given in Tables 1 and 2.*

* A list of structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 31797 (18 pp., 1 microfiche). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

## Discussion

Valency angles and interatomic distances among non-hydrogen atoms are given in Table 3 and Fig. 1. Fig. 1 also illustrates the structural formula. The standard deviations of the bond lengths range from 0·015 to 0·030 Å and from 1·0 to 1·9° for the angles. C–H lengths vary between 0·76 and 1·22 Å with an average value of 1·04 Å.

The molecular conformation is illustrated by the

Table 1. *Fractional coordinates* ($\times 10^4$) *and thermal parameters* ($Å^2 \times 10^3$), *with e.s.d.'s, of the non-hydrogen atoms*

Thermal parameters are of the form $T=\exp[-2\pi^2(a^{*2}h^2U_{11}+\ldots+2b^*c^*klU_{23})]$. The first and second values given for each parameter are for molecules *A* and *B* respectively. The *y* coordinate of Br(*A*) is arbitrary and constant to fix the origin.

| | *x* | *y* | *z* | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|---|---|---|---|---|---|---|---|---|---|
| Br | −890 (1) | 5000 | 8766 (1) | 48 (1) | 65 (1) | 55 (1) | 16 (1) | 4 (1) | 1 (1) |
| | 5786 (1) | 2797 (2) | 2965 (2) | 58 (1) | 123 (2) | 74 (11) | −7 (1) | 26 (1) | 32 (1) |
| O(3) | 8592 (6) | 2218 (13) | 11182 (11) | 40 (6) | 187 (15) | 76 (8) | −17 (8) | −16 (6) | 63 (9) |
| | −3617 (7) | −381 (10) | 4678 (14) | 53 (7) | 73 (10) | 126 (11) | −3 (7) | −2 (7) | −8 (8) |
| O(17) | 2708 (5) | 2637 (7) | 9475 (8) | 33 (5) | 39 (6) | 49 (5) | −1 (4) | 18 (4) | 10 (5) |
| | 2189 (5) | 1175 (8) | 4621 (9) | 39 (5) | 63 (7) | 46 (6) | 10 (5) | 15 (5) | −17 (6) |
| O(20) | 2198 (6) | 1954 (9) | 11233 (9) | 44 (6) | 107 (10) | 51 (6) | 18 (6) | 16 (5) | 33 (6) |
| | 2671 (7) | −315 (10) | 4094 (18) | 51 (8) | 69 (10) | 210 (18) | −8 (7) | 39 (10) | −57 (10) |
| C(1) | 6893 (8) | 3243 (11) | 9242 (13) | 48 (8) | 40 (9) | 52 (9) | −12 (7) | −9 (7) | −21 (8) |
| | −2030 (9) | 1291 (17) | 4080 (16) | 45 (9) | 123 (18) | 47 (10) | −19 (11) | 1 (8) | 31 (11) |
| C(2) | 7487 (10) | 3219 (17) | 10456 (15) | 51 (11) | 112 (18) | 43 | −26 (11) | 13 (8) | −18 (11) |
| | −2582 (10) | 421 (18) | 3649 (16) | 48 (10) | 127 (21) | 56 (10) | 0 (12) | 14 (8) | 8 (12) |
| C(3) | 7925 (9) | 2254 (18) | 10609 (15) | 27 (9) | 130 (19) | 50 (10) | −105 (11) | −1 (7) | 41 (11) |
| | −2955 (8) | −34 (15) | 4757 (16) | 33 (8) | 59 (11) | 94 (12) | −9 (10) | 2 (8) | −25 (12) |
| C(4) | 7498 (9) | 1347 (14) | 10110 (18) | 39 (10) | 58 (13) | 79 (13) | −7 (9) | 3 (9) | 40 (10) |
| | −2425 (9) | −102 (16) | 5978 (15) | 69 (10) | 73 (14) | 56 (10) | 0 (11) | 36 (8) | −17 (11) |
| C(5) | 6730 (8) | 1403 (11) | 9567 (14) | 29 (8) | 38 (9) | 58 (10) | 11 (7) | 10 (7) | 23 (8) |
| | −1698 (8) | 301 (12) | 6147 (13) | 36 (8) | 59 (13) | 43 (8) | 4 (8) | 3 (7) | 6 (8) |
| C(6) | 6291 (10) | 417 (13) | 9366 (20) | 47 (10) | 53 (12) | 105 (15) | −1 (9) | 1 (10) | 26 (11) |
| | −1131 (9) | 29 (18) | 7370 (14) | 58 (10) | 94 (14) | 46 (9) | 27 (12) | 23 (8) | 37 (12) |
| C(7) | 5730 (9) | 389 (11) | 8068 (21) | 34 (9) | 19 (9) | 150 (18) | 1 (7) | 53 (11) | −19 (10) |
| | −661 (10) | 926 (16) | 7930 (13) | 68 (12) | 99 (16) | 21 (8) | −7 (12) | 17 (8) | 6 (9) |
| C(8) | 5199 (8) | 1327 (12) | 7900 (14) | 31 (8) | 50 (10) | 53 (9) | −10 (8) | 14 (7) | −21 (8) |
| | −254 (8) | 1464 (13) | 6907 (13) | 27 (8) | 71 (12) | 32 (8) | −3 (8) | −3 (6) | 3 (8) |
| C(9) | 5715 (7) | 2292 (11) | 7982 (12) | 19 (7) | 51 (9) | 36 (8) | −9 (7) | 18 (6) | −4 (7) |
| | −872 (9) | 1878 (13) | 5757 (15) | 38 (9) | 62 (12) | 59 (10) | 2 (8) | 23 (8) | −15 (9) |
| C(10) | 6301 (7) | 2346 (11) | 9268 (12) | 16 (6) | 46 (9) | 37 (8) | −5 (6) | 1 (6) | −4 (7) |
| | −1368 (8) | 953 (12) | 5104 (13) | 33 (8) | 61 (11) | 35 (8) | −2 (8) | 9 (6) | −8 (8) |
| C(11) | 5168 (7) | 3234 (10) | 7889 (13) | 23 (7) | 30 (8) | 46 (8) | 6 (6) | 13 (6) | 17 (7) |
| | −427 (8) | 2387 (13) | 4661 (14) | 40 (9) | 59 (12) | 51 (9) | 23 (8) | 9 (7) | 17 (9) |
| C(12) | 4483 (8) | 3231 (9) | 8829 (13) | 44 (8) | 12 (7) | 47 (8) | 5 (6) | −6 (7) | −12 (6) |
| | 292 (8) | 1772 (12) | 4203 (13) | 29 (8) | 70 (11) | 34 (8) | −9 (8) | 9 (6) | 15 (8) |
| C(13) | 3998 (7) | 2272 (10) | 8681 (12) | 19 (7) | 26 (7) | 36 (7) | 0 (6) | 4 (6) | −2 (6) |
| | 879 (8) | 1475 (11) | 5379 (12) | 37 (8) | 40 (9) | 31 (8) | −2 (7) | −1 (6) | −10 (7) |
| C(14) | 4597 (7) | 1386 (10) | 8937 (14) | 25 (7) | 26 (8) | 49 (9) | −3 (6) | −1 (6) | 9 (7) |
| | 415 (8) | 876 (11) | 6352 (12) | 33 (7) | 49 (10) | 31 (2) | −4 (7) | 10 (6) | 4 (7) |
| C(15) | 4027 (9) | 458 (12) | 9040 (15) | 50 (9) | 47 (10) | 61 (10) | −1 (8) | 14 (8) | 21 (8) |
| | 1041 (8) | 405 (13) | 7316 (13) | 29 (8) | 74 (13) | 40 (8) | 2 (8) | −12 (6) | 18 (8) |
| C(16) | 3321 (8) | 927 (12) | 9706 (15) | 29 (8) | 57 (11) | 65 (10) | 0 (8) | 23 (7) | 5 (9) |
| | 1722 (8) | 134 (15) | 6435 (15) | 32 (7) | 71 (12) | 62 (9) | −5 (9) | −2 (7) | −10 (11) |
| C(17) | 3452 (7) | 2065 (10) | 9765 (13) | 24 (7) | 28 (9) | 46 (8) | 3 (6) | −2 (6) | −5 (7) |
| | 1495 (8) | 680 (13) | 5086 (15) | 29 (8) | 58 (11) | 53 (10) | 4 (8) | 7 (7) | −7 (9) |
| C(18) | 3512 (7) | 2237 (12) | 7283 (13) | 19 (7) | 82 (12) | 31 (8) | 2 (8) | −13 (6) | −5 (8) |
| | 1301 (8) | 2436 (11) | 6041 (15) | 44 (8) | 25 (9) | 72 (11) | 0 (7) | 0 (8) | −13 (8) |
| C(19) | 6193 (9) | 2316 (14) | 6722 (14) | 55 (10) | 76 (13) | 41 (9) | −15 (10) | 15 (7) | −4 (9) |
| | −1425 (9) | 2674 (14) | 6314 (16) | 47 (9) | 46 (10) | 80 (11) | 13 (9) | 25 (8) | −1 (10) |
| C(20) | 2143 (8) | 2505 (11) | 10305 (13) | 48 (9) | 40 (10) | 40 (8) | −9 (8) | 8 (7) | −6 (7) |
| | 2742 (8) | 578 (12) | 4177 (16) | 32 (9) | 36 (9) | 77 (12) | −14 (7) | 22 (8) | −27 (9) |
| C(21) | 1409 (7) | 3096 (11) | 9851 (11) | 25 (7) | 44 (9) | 17 (6) | −2 (7) | −4 (5) | 0 (6) |
| | 3459 (7) | 1168 (13) | 3859 (14) | 17 (7) | 74 (12) | 49 (9) | 15 (8) | 6 (6) | −29 (9) |
| C(22) | 701 (8) | 2868 (11) | 10411 (13) | 45 (8) | 35 (9) | 39 (8) | −4 (8) | −4 (6) | 7 (7) |
| | 4143 (10) | 606 (14) | 3711 (19) | 63 (11) | 57 (11) | 91 (14) | −7 (10) | 31 (10) | −23 (11) |
| C(23) | 6 (9) | 3420 (13) | 10070 (16) | 31 (7) | 63 (11) | 54 (9) | −3 (8) | 14 (7) | 12 (9) |
| | 4839 (10) | 1093 (16) | 3386 (17) | 44 (10) | 95 (16) | 68 (12) | 26 (10) | 11 (9) | −13 (11) |
| C(24) | 46 (8) | 4209 (11) | 9204 (13) | 38 (8) | 47 (10) | 32 (8) | 9 (7) | −1 (7) | −12 (7) |
| | 4838 (9) | 2089 (14) | 3379 (14) | 57 (10) | 71 (14) | 30 (8) | 7 (9) | 4 (7) | −12 (9) |
| C(25) | 743 (9) | 4464 (11) | 8674 (15) | 59 (11) | 29 (8) | 49 (9) | 3 (8) | 13 (8) | 12 (8) |
| | 4163 (9) | 2674 (12) | 3562 (15) | 64 (11) | 37 (10) | 65 (11) | 5 (9) | 31 (9) | 21 (9) |
| C(26) | 1430 (8) | 3921 (12) | 8996 (13) | 37 (9) | 47 (10) | 41 (9) | −5 (8) | 6 (7) | 1 (8) |
| | 3502 (8) | 2175 (11) | 3811 (13) | 46 (8) | 38 (9) | 40 (8) | 5 (7) | 19 (7) | 7 (7) |

Table 2. *Fractional coordinates* ($\times 10^3$), *with e.s.d.'s, of the hydrogen atoms*

| | Molecule *A* | | | Molecule *B* | | |
|---|---|---|---|---|---|---|
| | *x* | *y* | *z* | *x* | *y* | *z* |
| H(1*A*) | 718 (7) | 311 (11) | 830 (13) | −232 (8) | 165 (11) | 437 (13) |
| H(1*B*) | 665 (8) | 394 (11) | 926 (13) | −170 (8) | 147 (11) | 333 (13) |
| H(2*A*) | 777 (8) | 385 (11) | 1043 (13) | −303 (8) | 78 (11) | 299 (13) |
| H(2*B*) | 716 (8) | 332 (11) | 1130 (13) | −230 (7) | 5 (12) | 325 (12) |
| H(4) | 776 (8) | 62 (11) | 1013 (13) | −266 (8) | −40 (11) | 670 (13) |
| H(6*A*) | 678 (8) | −20 (11) | 939 (13) | −145 (8) | −40 (11) | 795 (13) |
| H(6*B*) | 592 (8) | 30 (11) | 1025 (13) | −75 (8) | −55 (11) | 711 (13) |
| H(7*A*) | 617 (8) | 17 (10) | 731 (12) | −107 (8) | 136 (11) | 849 (13) |
| H(7*B*) | 547 (8) | −10 (11) | 812 (12) | −29 (8) | 85 (11) | 879 (13) |
| H(8) | 494 (8) | 124 (11) | 693 (13) | 8 (8) | 214 (11) | 736 (13) |
| H(10) | 603 (8) | 240 (11) | 1008 (13) | −101 (8) | 62 (11) | 458 (13) |
| H(11*A*) | 546 (8) | 381 (11) | 777 (13) | −78 (7) | 268 (12) | 391 (12) |
| H(11*B*) | 497 (8) | 328 (12) | 707 (13) | −23 (7) | 281 (12) | 536 (12) |
| H(12*A*) | 475 (7) | 323 (11) | 985 (13) | 9 (8) | 94 (11) | 365 (13) |
| H(12*B*) | 423 (8) | 386 (11) | 859 (13) | 71 (8) | 228 (11) | 372 (13) |
| H(14) | 491 (8) | 156 (11) | 997 (13) | 10 (8) | 29 (10) | 582 (12) |
| H(15*A*) | 379 (7) | 39 (11) | 800 (12) | 124 (8) | 92 (11) | 812 (13) |
| H(15*B*) | 434 (7) | −7 (12) | 954 (12) | 93 (7) | −29 (11) | 802 (12) |
| H(16*A*) | 280 (8) | 55 (11) | 915 (13) | 225 (8) | 39 (10) | 695 (13) |
| H(16*B*) | 336 (8) | 57 (11) | 1067 (14) | 179 (8) | −68 (11) | 633 (13) |
| H(17) | 375 (8) | 224 (11) | 1082 (13) | 126 (7) | 23 (12) | 421 (12) |
| H(22) | 77 (8) | 232 (11) | 1096 (12) | 403 (8) | −10 (11) | 378 (12) |
| H(23) | −49 (7) | 309 (11) | 1055 (12) | 539 (8) | 56 (11) | 325 (13) |
| H(25) | 80 (7) | 514 (12) | 816 (12) | 422 (8) | 333 (10) | 360 (13) |
| H(26) | 190 (8) | 413 (11) | 867 (13) | 290 (8) | 261 (11) | 417 (13) |

stereo drawings in Figs. 2 and 3 of one of the molecules and of the contents of the unit cell, drawn with 50% probability ellipsoids. The 9$\beta$,10$\alpha$ (*retro*) configuration is confirmed. The two symmetrically unrelated molecules differ very little and the small differences can best be seen from the torsion angles listed in Table 4. The conformation of the *A* ring is midway between a half-chair and a sofa. The *B* and *C* rings have slightly flattened chair conformations and the *D* ring is a half-chair slightly distorted towards a 13$\beta$-envelope.

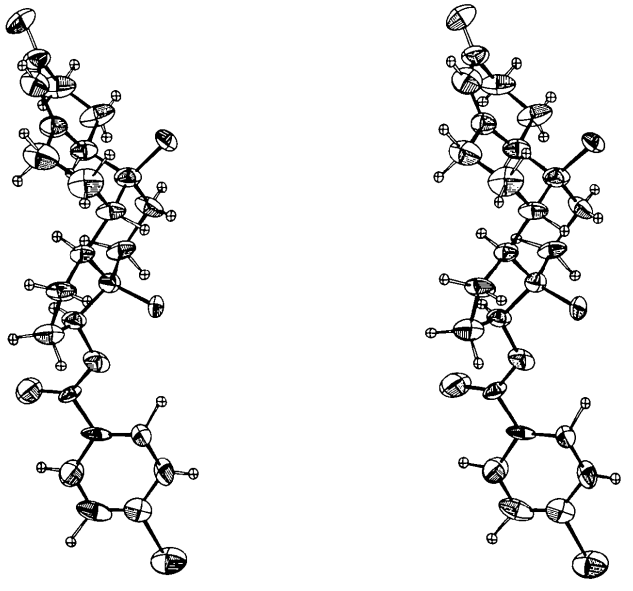

Fig. 2. 50% probability ellipsoids for 9-methyl-9$\beta$,10$\alpha$-oestr-4-en-3-one-17$\beta$-ol *p*-bromobenzoate (molecule *A*).

Table 3. *Bond angles* (°)

| | Molecules *A* | *B* | | Molecules *A* | *B* |
|---|---|---|---|---|---|
| C(10)–C(1)—C(2) | 110 | 111 | C(12)–C(13)–C(18) | 110 | 111 |
| C(1)—C(2)—C(3) | 113 | 113 | C(14)–C(13)–C(17) | 100 | 100 |
| C(2)—C(3)—C(4) | 117 | 114 | C(14)–C(13)–C(18) | 114 | 112 |
| C(2)—C(3)—O(3) | 121 | 124 | C(17)–C(13)–C(18) | 111 | 111 |
| O(3)—C(3)—C(4) | 122 | 121 | C(8)—C(14)–C(13) | 113 | 114 |
| C(3)—C(4)—C(5) | 121 | 124 | C(8)—C(14)–C(15) | 117 | 118 |
| C(4)—C(5)—C(6) | 116 | 120 | C(13)–C(14)–C(15) | 102 | 105 |
| C(4)—C(5)—C(10) | 124 | 122 | C(14)–C(15)–C(16) | 102 | 103 |
| C(10)–C(5)—C(6) | 119 | 117 | C(15)–C(16)–C(17) | 107 | 105 |
| C(5)—C(6)—C(7) | 113 | 113 | C(16)–C(17)–C(13) | 104 | 105 |
| C(6)—C(7)—C(8) | 112 | 112 | C(16)–C(17)–O(17) | 113 | 111 |
| C(7)—C(8)—C(9) | 110 | 112 | C(13)–C(17)–O(17) | 109 | 109 |
| C(7)—C(8)—C(14) | 112 | 115 | C(17)–O(17)–C(20) | 117 | 117 |
| C(14)–C(8)—C(9) | 109 | 111 | O(17)–C(20)–O(20) | 125 | 123 |
| C(8)—C(9)—C(10) | 113 | 109 | O(17)–C(20)–C(21) | 111 | 111 |
| C(8)—C(9)—C(11) | 110 | 111 | O(20)–C(20)–C(21) | 124 | 126 |
| C(8)—C(9)—C(19) | 107 | 110 | C(20)–C(21)–C(22) | 118 | 116 |
| C(10)–C(9)—C(11) | 110 | 108 | C(20)–C(21)–C(26) | 122 | 125 |
| C(10)–C(9)—C(19) | 111 | 112 | C(22)–C(21)–C(26) | 120 | 119 |
| C(11)–C(9)—C(19) | 106 | 108 | C(21)–C(22)–C(23) | 121 | 120 |
| C(1)—C(10)–C(9) | 112 | 112 | C(22)–C(23)–C(24) | 118 | 117 |
| C(1)—C(10)–C(5) | 111 | 111 | C(23)–C(24)–C(25) | 122 | 124 |
| C(5)—C(10)–C(9) | 113 | 111 | C(23)–C(24)–Br | 119 | 119 |
| C(9)—C(11)–C(12) | 115 | 115 | C(25)–C(24)–Br | 119 | 117 |
| C(11)–C(12)–C(13) | 111 | 111 | C(24)–C(25)–C(26) | 120 | 117 |
| C(12)–C(13)–C(14) | 106 | 108 | C(25)–C(26)–C(21) | 119 | 123 |
| C(12)–C(13)–C(17) | 116 | 115 | | | |

Table 4. *Torsion angles* (°) *in the steroid skeleton*

| *i* | *j* | *k* | *l* | $\varphi_A$ | $\varphi_B$ |
|---|---|---|---|---|---|
| C(10) | C(1) | C(2) | C(3) | 56 | 59 |
| C(1) | C(2) | C(3) | C(4) | −31 | −38 |
| C(2) | C(3) | C(4) | C(5) | −2 | 7 |
| C(3) | C(4) | C(5) | C(10) | 9 | 5 |

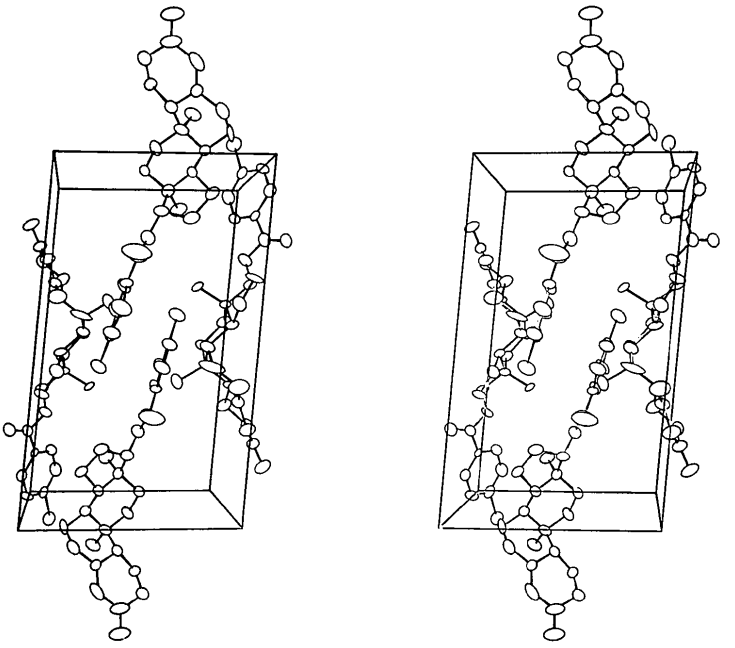

Fig. 3. Contents of the unit cell viewed down [010]. The *a* axis is down and the *c* axis horizontal.

Table 4 (*cont.*)

| *i* | *j* | *k* | *l* | $\varphi_A$ | $\varphi_B$ |
|---|---|---|---|---|---|
| C(4) | C(5) | C(10) | C(1) | 17 | 15 |
| C(5) | C(10) | C(1) | C(2) | −48 | −45 |
| C(10) | C(5) | C(6) | C(7) | 42 | 45 |
| C(5) | C(6) | C(7) | C(8) | −49 | −52 |
| C(6) | C(7) | C(8) | C(9) | 56 | 60 |
| C(7) | C(8) | C(9) | C(10) | −54 | −59 |
| C(8) | C(9) | C(10) | C(5) | 46 | 50 |
| C(9) | C(10) | C(5) | C(6) | −41 | −45 |
| C(14) | C(8) | C(9) | C(11) | −53 | −47 |
| C(8) | C(9) | C(11) | C(12) | 49 | 47 |
| C(9) | C(11) | C(12) | C(13) | −52 | −53 |
| C(11) | C(12) | C(13) | C(14) | 56 | 57 |
| C(12) | C(13) | C(14) | C(8) | −64 | −61 |
| C(13) | C(14) | C(8) | C(9) | 64 | 57 |
| C(17) | C(13) | C(14) | C(15) | 48 | 47 |
| C(13) | C(14) | C(15) | C(16) | −35 | −36 |
| C(14) | C(15) | C(16) | C(17) | 8 | 11 |
| C(15) | C(16) | C(17) | C(13) | 22 | 17 |
| C(16) | C(17) | C(13) | C(14) | −44 | −39 |
| C(16) | C(17) | O(17) | C(20) | 62 | 72 |
| C(17) | O(17) | C(20) | O(20) | −1 | 3 |

There are no hydrogen bonds and the closest approach not involving H atoms is 3·53 Å.

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