$w = 4F^2/[\sigma^2(I) + (0{\cdot}08F^2)^2]$, and also placed H atoms in fixed positions.} We also observe a large difference peak (1 e Å$^{-3}$) associated with the sulfonyl group: Allen *et al.* (1987) quote 0·33 e Å$^{-3}$. We are unable to explain this anomaly, but have noted similar effects in refinements that we considered unsatisfactory [see, for example, Jones, Sheldrick & Schmidt-Bäse (1987)].

In the light of this evidence we – tentatively – prefer to regard the anomalous C—O bond length in Tidwell's compound as an artefact of refinement. Further studies of related compounds are needed to confirm or disprove our opinion.

We thank the Fonds der Chemischen Industrie, and the Cambridge Philosophical Society, for support; Dr P. R. Raithby (Cambridge) for re-refining the data of Allen *et al.* (1987); and Professor T. T. Tidwell (Toronto) for supplying the data in advance of publication, and for friendly discussions.

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# Bond Length and Reactivity. Structures of the Triphenylmethyl Ethers of 1-Indanol and 5-Methoxy-1-indanol

BY PETER G. JONES* AND KAREN SCHMIDT-BÄSE

*Institut für Anorganische Chemie der Universität, Tammannstrasse* 4, *D*-3400 *Göttingen, Federal Republic of Germany*

AND ANTHONY J. KIRBY AND JANE K. PARKER

*University Chemical Laboratory, Cambridge CB*2 1*EW, England*

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**Abstract.** 1-Indanol triphenylmethyl ether (2) and its 5-methoxy derivative (3) are essentially isostructural, triclinic, space group $P\bar{1}$. For (2): $C_{28}H_{24}O$, $M_r = 376{\cdot}5$, $a = 8{\cdot}603\,(2)$, $b = 10{\cdot}047\,(2)$, $c = 12{\cdot}852\,(3)$ Å, $\alpha = 104{\cdot}25\,(2)$, $\beta = 104{\cdot}84\,(2)$, $\gamma = 94{\cdot}31\,(2)^\circ$, $V = 1029{\cdot}3$ Å$^3$, $Z = 2$, $D_x = 1{\cdot}22$ Mg m$^{-3}$, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}07$ mm$^{-1}$, $F(000) = 400$, $T = 293$ K, $R = 0{\cdot}051$ for 3567 unique observed reflections. For (3): $C_{29}H_{26}O_2$, $M_r = 406{\cdot}53$, $a = 8{\cdot}705\,(2)$, $b = 10{\cdot}195\,(2)$, $c = 13{\cdot}036\,(3)$ Å, $\alpha = 104{\cdot}82\,(2)$, $\beta = 100{\cdot}73\,(2)$, $\gamma = 93{\cdot}28\,(2)^\circ$, $V = 1092{\cdot}2$ Å$^3$, $Z = 2$, $D_x = 1{\cdot}24$ Mg m$^{-3}$, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}07$ mm$^{-1}$, $F(000) = 432$, $R = 0{\cdot}055$ for 2602 unique observed reflections. The introduction of the 5-methoxy substituent has very little effect on the structure. The C—OCPh$_3$ bond is lengthened by an insignificant 0·007 Å, from 1·432 (3) Å in (2) to 1·439 (3) Å in (3). This is consistent with our previous results, which show that a *p*-methoxy substituent has a negligible effect on the length of a benzylic C—O*X* bond.

**Introduction.** As part of our wider investigation of correlations between the length of a bond in the crystal and the rate at which it is broken in solution (Jones & Kirby, 1984), we have reported crystal structures for a series of derivatives (1) of 1-arylethanols (for full references see Edwards, Jones & Kirby, 1986*a*). In other systems, where the conformation is fixed, we have found a simple linear correlation between bond length and reactivity, but the 1-arylethanol derivatives show more complicated behaviour. As before, the C—O*X* bond is longer for better leaving groups O*X*, but this increase in length is associated with a systematic change in conformation: the longer the bond, the greater the dihedral angle *abd*, which increases from

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* Current address: Institut für Anorganische und Analytische Chemie der Technischen Universität, Hagenring 30, D-3300 Braunschweig, Federal Republic of Germany.

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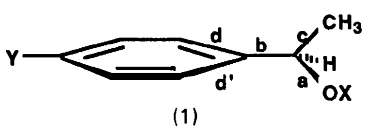
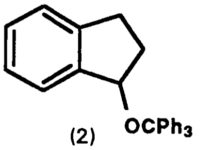
30 ± 15° in the generally preferred conformation (1) for these compounds towards the 90° expected to be optimal for C—O*X* cleavage (Edwards, Jones & Kirby, 1986*b*) as the O*X* group is made more electronegative.

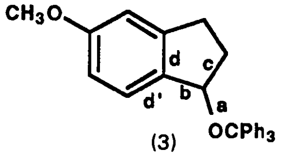


(1)

This increase in dihedral angle has the effect of further increasing the length of the C—O*X* bond, presumably by increasing the efficiency of $\pi$–$\sigma^*_{\text{C–O}X}$ orbital overlap. The effect is comparable in magnitude to that of changing the leaving group (O*X*), while substitution (varying *Y*) in the aromatic ring has a negligible effect on the length of the C—O*X* bond. It is in principle possible to separate the two major effects, and to set up a more sensitive test for the surprising apparent absence of a measurable *Y*-substituent effect, by choosing a system where the conformation is constant. We report results for two derivatives of 1-indanol, the triphenylmethyl ethers of the parent compound and of its 5-methoxy derivative [(2) and (3), respectively].



(2)



(3)

**Experimental.** The ethers were produced by alkylation of the 1-indanol in dichloromethane, in the presence of triethylamine and a catalytic amount of 4-dimethylaminopyridine. Diffusion of 60–80° petrol into solutions in dichloromethane gave (2) as colourless prisms, m.p. 430–432 K, and (3) as colourless blocks, m.p. 440–441 K.

Compound (2): a crystal 0·6 × 0·5 × 0·5 mm, cut from a larger prism, was mounted in a glass capillary and used to register 4735 profile-fitted intensities (Clegg, 1981) on a Stoe–Siemens four-circle diffractometer (monochromated Mo *K*α radiation, $2\theta_{\text{max}} = 55°$). Of 4727 unique reflections, 3567 with $F > 4\sigma(F)$ were used for all calculations (program system *SHELXTL*: Sheldrick, 1983). Cell constants were refined from $2\theta$ values of 89 reflections in the range 20–23°. No crystal decay was observed on monitoring three check reflections, and no corrections for absorption or extinction were carried out. Index ranges: *h* −11 to 11, *k* −13 to 13, *l* 0 to 16.

The structure was solved by routine direct methods and subjected to cascade least-squares refinement on *F* to $R = 0{\cdot}051$, $wR = 0{\cdot}058$. Non-H atoms were anisotropic, H atoms isotropic using a riding model. The weighting scheme was $w^{-1} = \sigma^2(F) + gF^2$, with $g = 0{\cdot}0002$. 262 parameters, $S = 2{\cdot}2$, max. $\Delta/\sigma = 0{\cdot}05$, max. $\Delta\rho$ variations within $\pm 0{\cdot}2$ e Å$^{-3}$. Atomic scattering factors from *SHELXTL*.

Table 1. *Atom coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å$^2 \times 10^3$)

| | *x* | *y* | *z* | $U_{\text{eq}}$* |
|---|---|---|---|---|
| (*a*) Compound (2) | | | | |
| C(1) | 6436 (2) | 2054 (1) | 1228 (1) | 39 (1) |
| C(2) | 7616 (2) | 1218 (2) | 1824 (1) | 50 (1) |
| C(3) | 7716 (2) | −17 (1) | 886 (1) | 48 (1) |
| C(3a) | 7440 (2) | 554 (1) | −114 (1) | 43 (1) |
| C(4) | 7826 (2) | 77 (2) | −1107 (1) | 55 (1) |
| C(5) | 7506 (2) | 818 (2) | −1896 (1) | 63 (1) |
| C(6) | 6801 (2) | 2019 (2) | −1699 (1) | 60 (1) |
| C(7) | 6397 (2) | 2489 (2) | −709 (1) | 49 (1) |
| C(7a) | 6718 (2) | 1753 (1) | 84 (1) | 39 (1) |
| O(1) | 6740 (1) | 3526 (1) | 1719 (1) | 40 (1) |
| C(10) | 5994 (2) | 4130 (1) | 2579 (1) | 37 (1) |
| C(11) | 4145 (2) | 4052 (1) | 2094 (1) | 40 (1) |
| C(12) | 3427 (2) | 3773 (1) | 956 (1) | 45 (1) |
| C(13) | 1763 (2) | 3733 (2) | 528 (1) | 55 (1) |
| C(14) | 797 (2) | 3986 (2) | 1232 (1) | 59 (1) |
| C(15) | 1491 (2) | 4301 (2) | 2368 (1) | 64 (1) |
| C(16) | 3153 (2) | 4336 (2) | 2798 (1) | 56 (1) |
| C(21) | 6453 (2) | 3429 (1) | 3527 (1) | 39 (1) |
| C(22) | 8003 (2) | 3765 (2) | 4262 (1) | 50 (1) |
| C(23) | 8510 (2) | 3060 (2) | 5057 (1) | 61 (1) |
| C(24) | 7475 (2) | 1998 (2) | 5126 (1) | 60 (1) |
| C(25) | 5944 (2) | 1630 (2) | 4391 (2) | 62 (1) |
| C(26) | 5427 (2) | 2342 (2) | 3602 (1) | 52 (1) |
| C(31) | 6745 (2) | 5670 (1) | 2962 (1) | 41 (1) |
| C(32) | 7530 (2) | 6237 (2) | 2325 (1) | 50 (1) |
| C(33) | 8190 (2) | 7630 (2) | 2669 (1) | 60 (1) |
| C(34) | 8079 (2) | 8478 (2) | 3642 (2) | 63 (1) |
| C(35) | 7292 (2) | 7936 (2) | 4283 (2) | 69 (1) |
| C(36) | 6631 (2) | 6546 (2) | 3950 (1) | 59 (1) |
| (*b*) Compound (3) | | | | |
| C(1) | 6363 (2) | 1990 (2) | 1077 (2) | 36 (1) |
| C(2) | 7493 (3) | 1136 (2) | 1606 (2) | 47 (1) |
| C(3) | 7674 (3) | −71 (2) | 679 (1) | 41 (1) |
| C(3a) | 7401 (2) | 498 (2) | −286 (2) | 35 (1) |
| C(4) | 7788 (2) | −6 (2) | −1291 (2) | 40 (1) |
| C(5) | 7463 (3) | 715 (2) | −2055 (2) | 44 (1) |
| C(6) | 6761 (3) | 1922 (2) | −1824 (2) | 55 (1) |
| C(7) | 6354 (3) | 2396 (2) | −836 (2) | 49 (1) |
| C(7a) | 6668 (2) | 1687 (2) | −58 (2) | 36 (1) |
| O(1) | 6665 (2) | 3440 (1) | 1564 (1) | 39 (1) |
| C(10) | 5925 (2) | 4058 (2) | 2449 (1) | 36 (1) |
| C(11) | 4132 (2) | 3968 (2) | 2060 (2) | 40 (1) |
| C(12) | 3480 (3) | 3816 (2) | 972 (2) | 48 (1) |
| C(13) | 1877 (3) | 3780 (2) | 615 (2) | 61 (1) |
| C(14) | 881 (3) | 3906 (2) | 1322 (2) | 65 (1) |
| C(15) | 1501 (3) | 4099 (2) | 2406 (2) | 65 (1) |
| C(16) | 3112 (3) | 4134 (2) | 2782 (2) | 52 (1) |
| C(21) | 6418 (2) | 3405 (2) | 3377 (2) | 38 (1) |
| C(22) | 7873 (3) | 3823 (2) | 4077 (2) | 54 (1) |
| C(23) | 8410 (3) | 3164 (3) | 4858 (2) | 70 (1) |
| C(24) | 7500 (4) | 2078 (3) | 4966 (2) | 68 (1) |
| C(25) | 6067 (3) | 1632 (2) | 4265 (2) | 60 (1) |
| C(26) | 5535 (3) | 2293 (2) | 3489 (2) | 48 (1) |
| C(31) | 6588 (2) | 5572 (2) | 2778 (2) | 36 (1) |
| C(32) | 7603 (3) | 6064 (2) | 2240 (2) | 58 (1) |
| C(33) | 8210 (3) | 7433 (2) | 2563 (2) | 72 (1) |
| C(34) | 7773 (3) | 8324 (2) | 3404 (2) | 61 (1) |
| C(35) | 6754 (3) | 7852 (2) | 3943 (2) | 56 (1) |
| C(36) | 6171 (3) | 6496 (2) | 3633 (2) | 50 (1) |
| O(5) | 7787 (2) | 327 (2) | −3080 (1) | 61 (1) |
| C(51) | 8773 (3) | −718 (2) | −3292 (2) | 57 (1) |

* Equivalent isotropic *U* calculated from anisotropic *U*.

Compound (3): as for (2), with the following differences. Crystal 0·7 × 0·3 × 0·3 mm, 4232 reflections to $2\theta_{\text{max}} = 50°$, 3280 unique ($R_{\text{int}} = 0{\cdot}025$), 2602 observed. Cell constants from 40 $2\theta$ values. Index ranges *h* −10 to 10, *k* −12 to 12, *l* 0 to 15. Refined to $R = 0{\cdot}055$, $wR = 0{\cdot}051$; $g = 0{\cdot}00025$, 283 param-

Table 2. *Molecular geometry for* (2) *and* (3) (*e.s.d.'s are in parentheses*)

| | (2) | (3) | | (2) | (3) |
|---|---|---|---|---|---|
| (*a*) Bond lengths (Å) | | | | | |
| C(1)–C(2) | 1·539 (3) | 1·534 (4) | C(1)–C(7a) | 1·513 (3) | 1·509 (4) |
| C(1)–O(1) | 1·432 (3) | 1·439 (3) | C(2)–C(3) | 1·528 (3) | 1·526 (4) |
| C(3)–C(3a) | 1·505 (3) | 1·502 (4) | C(3a)–C(4) | 1·381 (3) | 1·390 (4) |
| C(3a)–C(7a) | 1·394 (3) | 1·393 (4) | C(4)–C(5) | 1·384 (4) | 1·380 (4) |
| C(5)–C(6) | 1·388 (4) | 1·395 (4) | C(6)–C(7) | 1·385 (4) | 1·374 (4) |
| C(7)–C(7a) | 1·385 (3) | 1·386 (4) | C(10)–O(1) | 1·447 (3) | 1·447 (4) |
| C(10)–C(11) | 1·541 (3) | 1·538 (4) | C(10)–C(21) | 1·534 (3) | 1·531 (4) |
| C(10)–C(31) | 1·538 (3) | 1·539 (4) | C(11)–C(12) | 1·382 (3) | 1·391 (4) |
| C(11)–C(16) | 1·391 (4) | 1·397 (5) | C(12)–C(13) | 1·389 (3) | 1·381 (4) |
| C(13)–C(14) | 1·370 (4) | 1·367 (5) | C(14)–C(15) | 1·373 (4) | 1·374 (5) |
| C(15)–C(16) | 1·389 (3) | 1·392 (4) | C(21)–C(22) | 1·384 (3) | 1·388 (4) |
| C(21)–C(26) | 1·390 (3) | 1·388 (4) | C(22)–C(23) | 1·383 (4) | 1·383 (5) |
| C(23)–C(24) | 1·372 (4) | 1·376 (5) | C(24)–C(25) | 1·372 (3) | 1·377 (5) |
| C(25)–C(26) | 1·383 (4) | 1·379 (5) | C(31)–C(32) | 1·380 (4) | 1·373 (5) |
| C(31)–C(36) | 1·386 (3) | 1·384 (4) | C(32)–C(33) | 1·388 (3) | 1·392 (4) |
| C(33)–C(34) | 1·360 (4) | 1·363 (5) | C(34)–C(35) | 1·373 (4) | 1·366 (5) |
| C(35)–C(36) | 1·385 (3) | 1·375 (4) | O(5)–C(51) | | 1·411 (4) |
| C(5)–O(5) | | 1·380 (4) | | | |
| (*b*) Bond angles (°) | | | | | |
| C(2)–C(1)–C(7a) | 102·4 (2) | 102·4 (3) | C(2)–C(1)–O(1) | 115·6 (2) | 114·9 (2) |
| C(7a)–C(1)–O(1) | 108·6 (2) | 108·7 (3) | C(1)–C(2)–C(3) | 104·5 (2) | 105·4 (3) |
| C(2)–C(3)–C(3a) | 103·2 (2) | 102·9 (3) | C(3)–C(3a)–C(4) | 129·6 (2) | 128·6 (3) |
| C(3)–C(3a)–C(7a) | 109·9 (2) | 110·4 (3) | C(4)–C(3a)–C(7a) | 120·5 (3) | 121·0 (3) |
| C(3a)–C(4)–C(5) | 119·0 (3) | 118·6 (3) | C(4)–C(5)–C(6) | 120·8 (3) | 120·7 (3) |
| C(5)–C(6)–C(7) | 120·3 (3) | 120·3 (3) | C(6)–C(7)–C(7a) | 119·2 (3) | 120·0 (3) |
| C(1)–C(7a)–C(3a) | 110·0 (2) | 110·1 (3) | C(1)–C(7a)–C(7) | 129·7 (2) | 130·5 (3) |
| C(3a)–C(7a)–C(7) | 120·3 (2) | 119·4 (3) | C(1)–O(1)–C(10) | 119·5 (2) | 119·4 (3) |
| O(1)–C(10)–C(11) | 111·4 (2) | 111·0 (2) | O(1)–C(10)–C(21) | 108·7 (2) | 108·8 (3) |
| C(11)–C(10)–C(21) | 113·4 (2) | 113·7 (3) | O(1)–C(10)–C(31) | 103·5 (2) | 103·9 (3) |
| C(11)–C(10)–C(31) | 107·6 (2) | 108·0 (3) | C(21)–C(10)–C(31) | 111·8 (2) | 111·0 (2) |
| C(10)–C(11)–C(12) | 121·5 (2) | 120·4 (3) | C(10)–C(11)–C(16) | 120·7 (2) | 121·9 (3) |
| C(12)–C(11)–C(16) | 117·7 (2) | 117·4 (3) | C(11)–C(12)–C(13) | 121·1 (3) | 121·2 (3) |
| C(12)–C(13)–C(14) | 120·5 (3) | 121·0 (3) | C(13)–C(14)–C(15) | 119·4 (3) | 118·9 (3) |
| C(14)–C(15)–C(16) | 120·3 (3) | 121·0 (4) | C(11)–C(16)–C(15) | 121·0 (3) | 120·4 (3) |
| C(10)–C(21)–C(22) | 119·6 (2) | 120·2 (3) | C(10)–C(21)–C(26) | 122·2 (2) | 122·4 (3) |
| C(22)–C(21)–C(26) | 117·8 (2) | 117·1 (3) | C(21)–C(22)–C(23) | 121·2 (3) | 121·3 (3) |
| C(22)–C(23)–C(24) | 120·2 (3) | 120·5 (3) | C(23)–C(24)–C(25) | 119·5 (3) | 118·9 (4) |
| C(24)–C(25)–C(26) | 120·5 (3) | 120·3 (4) | C(21)–C(26)–C(25) | 120·8 (2) | 121·7 (3) |
| C(10)–C(31)–C(32) | 121·2 (2) | 121·9 (3) | C(10)–C(31)–C(36) | 121·2 (3) | 120·8 (3) |
| C(32)–C(31)–C(36) | 117·6 (2) | 117·4 (3) | C(31)–C(32)–C(33) | 121·0 (3) | 120·9 (3) |
| C(32)–C(33)–C(34) | 120·9 (3) | 120·6 (4) | C(33)–C(34)–C(35) | 118·9 (3) | 119·2 (3) |
| C(34)–C(35)–C(36) | 120·7 (3) | 120·2 (3) | C(31)–C(36)–C(35) | 120·9 (3) | 121·7 (3) |
| C(4)–C(5)–O(5) | | 124·4 (3) | C(6)–C(5)–O(5) | | 115·0 (3) |
| C(5)–O(5)–C(51) | | 117·7 (3) | | | |

eters, $S = 1\cdot6$, max. $\Delta/\sigma = 0\cdot04$, max. $\Delta\rho$ variations within $\pm 0\cdot2$ e Å$^{-3}$.

**Discussion.** Final atom coordinates are presented in Table 1* and derived parameters in Table 2. Plots of (2) and (3), showing the atom-numbering schemes, appear in Figs. 1 and 2. Compounds (2) and (3) are, except for the methoxy group, isostructural and thus closely similar.

The introduction of the 5-methoxy substituent affects the detailed structure of the triphenylmethyl ether extraordinarily little. The largest difference is in the C—O*X* bond length, which is 1·432 (3) Å in (2) and possibly slightly longer in (3), at 1·439 (3) Å. An increase is expected if the introduction of an electron-donating substituent does affect the length of this bond, but the difference is not significant. Our earlier results with 1-arylethanol derivatives (Edwards, Jones & Kirby, 1986*b*) also suggested that a *p*-methoxy substituent has a negligible effect on the length of a benzylic C—O*X* bond.

The comparison is, however, no longer complicated by a difference in conformation. The torsion angle *cbd* is identical within experimental error for the two compounds: 19·7 (2) and 18·7 (3)° in (2) and (3), respectively, so that the C—O*X* bond is indeed effectively conformationally fixed [the torsion angles *abd'* are −36·2 (3) and −38·4 (4)° respectively]. Even the conformations of the triphenylmethyl groups are closely similar, with just small differences in the tilt of the three phenyl rings. The $Ph_3C$—O bond length is 1·447 (3) Å in both compounds, in the range expected for an alkyl triphenylmethyl ether.

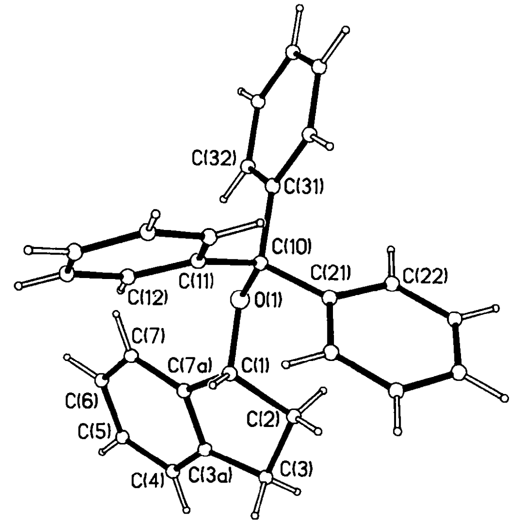


Fig. 1. Molecular structure of (2), showing the atom-numbering scheme.

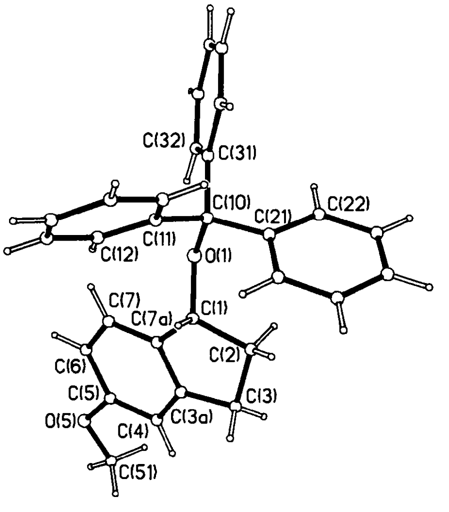


Fig. 2. Molecular structure of (3), showing the atom-numbering scheme.

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* Lists of H-atom coordinates and $U_{\text{iso}}$ values, structure factors, torsion angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51326 (58 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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# Bond Length and Reactivity.* Structure of 1-Indanol 4-Nitrobenzoate Ester

By Anthony J. Kirby, Jane K. Parker and Paul R. Raithby

*University Chemical Laboratory, Cambridge CB2 1EW, England*

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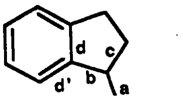
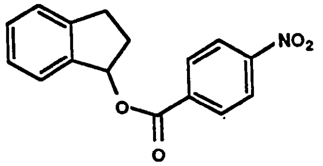
**Abstract.** 1-Indanol 4-nitrobenzoate, $C_{16}H_{13}NO_4$, $M_r = 283\cdot28$, triclinic, $P\bar{1}$, $a = 8\cdot116\,(1)$, $b = 12\cdot784\,(1)$, $c = 13\cdot954\,(2)$ Å, $\alpha = 100\cdot15\,(1)$, $\beta = 101\cdot03\,(1)$, $\gamma = 91\cdot66\,(1)°$, $V = 1395\cdot9\,(3)$ Å$^3$, $Z = 4$, $D_x = 1\cdot348$ Mg m$^{-3}$, $\lambda(\text{Cu}\,K\alpha) = 1\cdot5418$ Å, $\mu = 0\cdot77$ mm$^{-1}$, $\mu R = 0\cdot24$, $F(000) = 592$, $T = 290$ K, $R = 0\cdot058$ for 3196 observed reflections. The asymmetric unit contains two independent molecules with significant conformational differences, and matching differences in C—O(acyl) bond lengths. These are 1·470 (3) and 1·462 (3) Å for molecules (2*A*) and (2*B*), in which the C—O(acyl) bonds make torsion angles with the aromatic ring of 75·2 (3) and 54·7 (4)°, respectively. The C—O*X* bond lengths in three structures of indanol derivatives are consistently shorter than predicted for the corresponding open-chain (1-phenylethyl) compounds, by 0·009±1 Å.

**Introduction.** We have reported crystal structures for the triphenylmethyl ethers of 1-indanol (1) and its 5-methoxy derivative (Jones, Schmidt-Bäse, Kirby & Parker, 1989). The object was to find a system of fixed conformation, to allow us to separate the effects on benzylic C—O*X* bond lengths of varying conformation and varying leaving group (Edwards, Jones & Kirby, 1986). This first test was successful, in that the two ethers crystallized in almost identical conformations. For the particular leaving group ($OCPh_3$) this provides a sensitive test of the effect on the C—O*X* bond length of the substituent in the aromatic ring.

We report the structure of the *p*-nitrobenzoate ester (2) of 1-indanol, a compound with a much better leaving group. (Other derivatives were made, but no suitable crystals were obtained.)



(1)



(2)

**Experimental.** The ester (2) was prepared by acylation of the alcohol in dichloromethane containing triethylamine and a catalytic amount of 4-*N*,*N*-dimethylaminopyridine, using *p*-nitrobenzoyl chloride. Crystals, m.p. 349–350 K, were grown by diffusion of hexane into a solution in dichloromethane. Block-shaped single crystal 0·15 × 0·22 × 0·32 mm, Nicolet *R*3*mμ* diffractometer with graphite monochromator, Cu *Kα* radiation; unit-cell parameters refined from diffractometer angles of 25 centred reflections ($40 < 2\theta < 50°$); 4377 reflections with $5 < 2\theta < 120°$ (*hkl* range: *h* −10 to 10, *k* 0 to 15, *l* −16 to 16); no significant variations of three standard reflections; empirical absorption correction based on 375 azimuthal scan data and an ellipsoid model, transmission factors 0·34–0·77, 4022 unique reflections with $F > 3\sigma(F)$. Structure solved by centrosymmetric direct methods (*SHELXTL:SOLV*), blocked-cascade least-squares refinement on *F*, anisotropic thermal parameters for all non-H atoms; H atoms fixed to C with C—H 0·96 Å, and allowed to ride on the relevant C atom; each type of H assigned a common isotropic temperature factor; scattering factors from *International Tables for X-ray Crystallography* (1974); 381 parameters, $R = 0\cdot058$, $wR = 0\cdot065$, $w = [\sigma^2(F_o) +$

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* Previous paper in this series: Jones, Schmidt-Bäse, Kirby & Parker (1989).

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