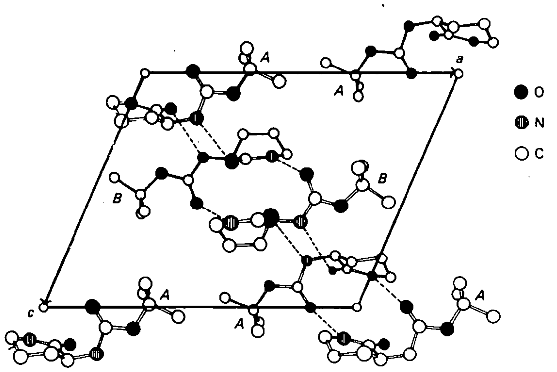

Fig. 2. Packing mode of molecules *A* and *B* of compound (1) viewed down the *b* axis. Intermolecular hydrogen bonds are shown as dashed lines.

molecule *B* are formed *via* (lactam)N—H···O=C-(urethane) intermolecular H bonds along the *y* direction (Fig. 2). The N(2)*A*···O(2)*A* ($2-x$, $y-\frac{1}{2}$, $2-z$) and N(2)*B*···O(2)*B* ($1-x$, $\frac{1}{2}+y$, $1-z$) separations are 2·990 (8) and 2·880 (9) Å, respectively (Ramakrishnan & Prasad, 1971; Taylor, Kennard & Versichel, 1984). These chains are interconnected by the formation of dimeric structures *via* (urethane)N—H···O=C-(lactam) intermolecular H bonds. The N(1)*B*···O(3)*A* ($1-x$, $\frac{1}{2}+y$, $1-z$) and O(3)*B*···N(1)*A* ($1-x$, $\frac{1}{2}+y$, $1-z$) separations are 2·913 (10) and 2·868 (9) Å, respectively.

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Bond Length and Reactivity: 1-Arylethyl Ethers and Esters. 7.* Structure of 1-[3,5-Bis(trifluoromethyl)phenyl]ethyl 4-Nitrobenzoate

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Abstract. $C_{17}H_{11}F_6NO_4$, $M_r = 407·3$, monoclinic, $C2/c$, $a = 15·002$ (1), $b = 7·709$ (1), $c = 30·546$ (3) Å, $\beta = 93·46$ (1)°, $V = 3526·1$ Å^3, $Z = 8$, $D_x = 1·53$ Mg m^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 1·3$ mm^{-1}, $F(000) = 1648$, $T = 293$ K, $R = 0·058$ for 2167 unique observed reflections. The CF_3 groups are disordered, with components of *ca* 0·9, 0·1; this led to slow convergence of refinement. The C—O bond length to

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* Part 6: Edwards, Jones & Kirby (1986*a*).

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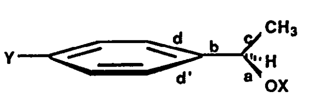
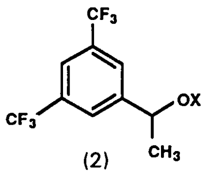
the 4-nitrobenzoate 'leaving group' is 1·461 (3) Å, which is consistent with our previously established correlations between the lengths of C—O bonds and the rates at which they are broken heterolytically in solution. The status of a published exception [Allen, Kwong-Chip, Mistry, Sawyer & Tidwell (1987). *J. Org. Chem.* **52**, 4164–4172] is considered: the apparent anomaly could be an artefact of their refinement.

Introduction. We have shown in several systems that there is a strong correlation between the lengths of C—O bonds and the rates at which they are broken heterolytically in solution – the longer the bond, the faster it breaks (Jones & Kirby, 1979, 1984). Recently we reported correlations for a series of 1-arylethanol derivatives (1) (Edwards, Jones & Kirby, 1986*b*). Here behaviour is more complicated, because the conformation about the ring—CO*X* bond *b* also varies. As a result the bond length–reactivity correlation is no longer linear, apparently because the length of the C—O*X* bond depends not only on the effective electronegativity of the O atom, but also on the dihedral angle *abd*. Nevertheless, the rule – the longer the bond, the faster it breaks – still applies.

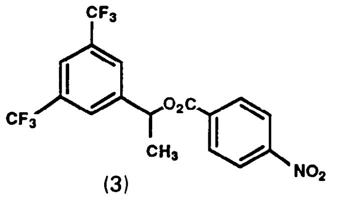


(1)



(2)

Tidwell and co-workers (Allen, Kwong-Chip, Mistry, Sawyer & Tidwell, 1987) recently reported the structure of a compound (2, $X = p$-toluenesulfonyl) which is a clear exception to this rule. This compound is solvolyzed more rapidly than, for example, Ph(CF$_3$)CHOTs, but has a substantially shorter C—O bond length [1·413 (5), compared with 1·453 (5) Å]. Since it is the very short C—O bond, rather than the reactivity, which is unexpected, we have examined a representative bis(trifluoromethyl)phenyl compound in our series (1). Of several derivatives of 1-[3,5-bis(trifluoromethyl)-phenyl]ethanol (2, X = H) prepared, only the 4-nitrobenzoate (3) gave suitable crystals.



(3)

Experimental. The ester (3) was prepared by acylating the lithium salt of the alcohol with *p*-nitrobenzoyl chloride, and gave colourless prisms, m.p. 354–356 K, on slow evaporation from a solution in hexane/dichloromethane.

A crystal 0·35 × 0·2 × 0·1 mm was mounted on a glass fibre. 5938 profile-fitted intensities (Clegg, 1981) were measured on a Stoe–Siemens four-circle diffractometer (monochromated Cu *K*α radiation, $2\theta_{\max} = 120°$, hemisphere of data). Merging equivalents gave 2634 unique reflections ($R_{\rm int} = 0{\cdot}034$; index ranges h −16 to 16, k 0 to 8, l 0 to 34), of which 2167 with $F > 4\sigma(F)$ were used for all calculations with program system *SHELX*76 (Sheldrick, 1976), locally modified by its author. Cell constants were refined from 2θ values of 42 reflections in the range 60–70°. Intensities were corrected for crystal decay (*ca* 5%, based on three check reflections), but not for absorption or extinction.

The structure was solved by routine direct methods and refined anisotropically on F to $R = 0{\cdot}058$, $wR = 0{\cdot}067$. H atoms were included using a riding model. The CF$_3$ groups showed disorder, and additional F atoms F(1*A*–6*A*) were introduced to take account of this; they were allotted a common isotropic temperature factor and refined subject to the constraint C—F = 1·32 Å, with a weight corresponding to a σ of 0·01 Å. The site-occupation factors were 0·118 (6) for F(1*A*–3*A*) and 0·063 (4) for F(4*A*–6*A*).

The weighting scheme was $w^{-1} = \sigma^2(F) + 0{\cdot}00035F^2$. 277 parameters; $S = 2{\cdot}3$; max. $\Delta/\sigma = 1{\cdot}0$ for a minor F component, 0·15 for non-F parameters; max. $|\Delta\rho|$ 0·25 e Å^{-3} near N(1). Despite several cycles of full-matrix refinement, the F-atom parameters converged slowly, as is clear from the above values.*

Discussion. Final atomic coordinates and derived parameters are presented in Tables 1 and 2, and a plot of (3) is shown in Fig. 1. The atom-numbering scheme in the benzylic system is the same as for previous compounds in this series.

Compound (3) crystallizes in an extended conformation [in contrast to Tidwell's tosylate (2, X = 4-CH$_3$C$_6$H$_4$SO$_2$—)] with the C-methyl group more closely perpendicular to the ring than the C—O*X* bond [torsion angles *cbd* and *abd* 109·8 (5) and 51·3 (5)°, respectively]. The C—O bond length is, within experimental error, that predicted from our published correlation equation (Edwards, Jones & Kirby, 1986*b*): $a = 1{\cdot}453 - 1{\cdot}48 \times 10^{-3}{\rm p}K_{{\rm HO}X} + 3{\cdot}3 \times 10^{-2}\cos^2\theta$ ($\theta = 90 - abd$), which predicts a value of 1·468, compared with the observed 1·461 (3) Å. Other parameters around the benzylic centre are also similar to those for related compounds of our series (1). We conclude that

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* Lists of H-atom coordinates and $U_{\rm iso}$ values, structure factors, torsion angles, and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51321 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* (Å^2 $\times 10^3$)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	4802 (2)	7390 (4)	1290 (1)	65 (1)
C(2)	4010 (2)	7961 (5)	996 (1)	85 (1)
C(3)	4149 (2)	6688 (4)	1961 (1)	65 (1)
O(1)	4512 (1)	6100 (2)	1602 (1)	61 (1)
O(2)	4054 (2)	8206 (3)	2037 (1)	99 (1)
C(11)	5516 (2)	6523 (3)	1040 (1)	57 (1)
C(12)	6323 (2)	7350 (4)	991 (1)	60 (1)
C(13)	6964 (2)	6589 (4)	746 (1)	60 (1)
C(14)	6816 (2)	4995 (4)	553 (1)	61 (1)
C(15)	6012 (2)	4160 (4)	600 (1)	58 (1)
C(16)	5367 (2)	4918 (4)	844 (1)	59 (1)
C(17)	7830 (2)	7504 (5)	706 (1)	82 (1)
F(1)	8333 (2)	6760 (5)	411 (1)	116 (1)
F(1*A*)	8119 (14)	7898 (37)	329 (4)	98 (5)
F(2)	7733 (2)	9109 (3)	577 (2)	135 (2)
F(2*A*)	7880 (15)	8909 (24)	972 (7)	98 (5)
F(3)	8329 (2)	7463 (6)	1073 (1)	136 (2)
F(3*A*)	8539 (13)	7043 (40)	943 (8)	98 (5)
C(18)	5816 (2)	2442 (5)	386 (1)	79 (1)
F(4)	5529 (2)	1284 (3)	664 (1)	117 (1)
F(4*A*)	4985 (11)	1967 (51)	409 (15)	98 (5)
F(5)	6507 (2)	1768 (4)	204 (1)	134 (1)
F(5*A*)	5959 (28)	2763 (48)	−30 (5)	98 (5)
F(6)	5160 (2)	2542 (3)	74 (1)	111 (1)
F(6*A*)	6474 (19)	1437 (42)	539 (13)	98 (5)
C(21)	3898 (2)	5259 (4)	2259 (1)	58 (1)
C(22)	3574 (2)	5704 (4)	2659 (1)	70 (1)
C(23)	3359 (2)	4441 (5)	2954 (1)	72 (1)
C(24)	3475 (2)	2739 (4)	2840 (1)	62 (1)
C(25)	3780 (2)	2239 (4)	2442 (1)	66 (1)
C(26)	3986 (2)	3526 (4)	2151 (1)	62 (1)
N(1)	3271 (2)	1370 (5)	3157 (1)	80 (1)
O(3)	2955 (2)	1810 (4)	3497 (1)	122 (1)
O(4)	3425 (2)	−126 (4)	3066 (1)	119 (1)

Table 2. *Bond lengths* (Å) *and bond angles* (°)

C(1)—C(2)	1·511 (4)	C(1)—O(1)	1·461 (3)
C(1)—C(11)	1·509 (4)	C(3)—O(1)	1·334 (3)
C(3)—O(2)	1·203 (4)	C(3)—C(21)	1·491 (4)
C(11)—C(12)	1·384 (4)	C(11)—C(16)	1·387 (4)
C(12)—C(13)	1·384 (4)	C(13)—C(14)	1·376 (4)
C(13)—C(17)	1·491 (4)	C(14)—C(15)	1·382 (4)
C(15)—C(16)	1·384 (4)	C(15)—C(18)	1·499 (4)
C(17)—F(1)	1·339 (4)	C(17)—F(1*A*)	1·291 (9)
C(17)—F(2)	1·304 (4)	C(17)—F(2*A*)	1·352 (9)
C(17)—F(3)	1·310 (5)	C(17)—F(3*A*)	1·299 (10)
C(18)—F(4)	1·323 (4)	C(18)—F(4*A*)	1·305 (10)
C(18)—F(5)	1·313 (4)	C(18)—F(5*A*)	1·323 (10)
C(18)—F(6)	1·330 (4)	C(18)—F(6*A*)	1·318 (10)
C(21)—C(22)	1·387 (4)	C(21)—C(26)	1·384 (4)
C(22)—C(23)	1·377 (4)	C(23)—C(24)	1·372 (4)
C(24)—C(25)	1·379 (4)	C(24)—N(1)	1·476 (4)
C(25)—C(26)	1·379 (4)	N(1)—O(3)	1·216 (4)
N(1)—O(4)	1·211 (4)		

O(1)—C(1)—C(2)	109·3 (2)	C(11)—C(1)—C(2)	112·6 (3)
C(11)—C(1)—O(1)	106·1 (2)	O(2)—C(3)—O(1)	123·2 (3)
C(21)—C(3)—O(1)	112·4 (3)	C(21)—C(3)—O(2)	124·3 (3)
C(3)—O(1)—C(1)	117·2 (2)	C(12)—C(11)—C(1)	120·1 (2)
C(16)—C(11)—C(1)	120·9 (2)	C(16)—C(11)—C(12)	118·8 (3)
C(13)—C(12)—C(11)	120·2 (3)	C(14)—C(13)—C(12)	120·8 (3)
C(17)—C(13)—C(12)	118·8 (3)	C(17)—C(13)—C(14)	120·4 (3)
C(15)—C(14)—C(13)	119·3 (3)	C(16)—C(15)—C(14)	120·2 (3)
C(18)—C(15)—C(14)	120·9 (3)	C(18)—C(15)—C(16)	118·9 (3)
C(15)—C(16)—C(11)	120·6 (3)	F(1)—C(17)—C(13)	112·4 (3)
F(2)—C(17)—C(13)	113·1 (3)	F(2)—C(17)—F(1)	105·1 (3)
F(3)—C(17)—C(13)	112·0 (3)	F(3)—C(17)—F(1)	104·5 (3)
F(3)—C(17)—F(2)	109·3 (4)	F(4)—C(18)—C(15)	112·2 (3)
F(5)—C(18)—C(15)	113·3 (3)	F(5)—C(18)—F(4)	107·5 (3)
F(6)—C(18)—C(15)	112·3 (3)	F(6)—C(18)—F(4)	104·1 (3)
F(6)—C(18)—F(5)	106·8 (3)	C(22)—C(21)—C(3)	118·0 (3)
C(26)—C(21)—C(3)	122·4 (3)	C(26)—C(21)—C(22)	119·5 (3)
C(23)—C(22)—C(21)	120·7 (3)	C(24)—C(23)—C(22)	118·2 (3)
C(25)—C(24)—C(23)	123·0 (3)	N(1)—C(24)—C(23)	118·9 (3)
N(1)—C(24)—C(25)	118·1 (3)	C(26)—C(25)—C(24)	117·8 (3)
C(25)—C(26)—C(21)	120·8 (3)	O(3)—N(1)—C(24)	117·8 (3)
O(4)—N(1)—C(24)	118·8 (3)	O(4)—N(1)—O(3)	123·4 (3)

there is no special electronic feature associated with the bis(trifluoromethyl)phenyl ring that can account for the extraordinarily short bond length observed by Tidwell and co-workers.

In the circumstances the next factor to examine is the reliability of X-ray results. In particular, it is important to consider when a 'rogue' result should be discounted. The presence of disorder (as in the CF_3 groups of the current structure and of Tidwell's compound) does not, *per se*, invalidate the structural results, as long as a sensible model can be refined. There can, however, be problems in refinement, notably that of slow convergence. The best guide to the precision of a structure is the e.s.d. of a typical bond length – assuming that it has been properly estimated. A comparison of the current structure with that of Allen *et al.* (1987) reveals the following significant features.

The current structure is disordered, and convergence poor, despite an apparently sensible model. The R values are reasonable ($R = 0{\cdot}058$, $wR = 0{\cdot}067$). The data/parameter ratio is 7·8, which is no more than satisfactory. There are no major unexplained features in the residual electron density.

Tidwell's compound is also disordered, but the disorder model appears successful. Satisfactory convergence was only obtained by blocking the refinement. The R values are poor, with $R = 0{\cdot}071$, $wR = 0{\cdot}102$, despite a higher threshold for unobserved reflections [$6\sigma(F)$ compared with our $4\sigma(F)$]. The weak point in their refinement, in our view, is the poor data/parameter ratio of 5·5; this is in the lower regions of the range usually considered acceptable. The bond length e.s.d.'s reported by Allen *et al.* (1987) are surprisingly low, bearing in mind the high R value and small number of data. We have subjected their structure to full-matrix refinement (program system *SHELX*76), using deposited structure factors, and obtain an e.s.d. of 0·008 Å (rather than 0·005 Å) for the C—O bond length under discussion, and mean e.s.d.'s of 0·010 Å for C—C bond lengths. The re-refinement produced no significant differences in geometry. The weighted R was 0·086, using the weighting scheme $w = 4{\cdot}34/[\sigma^2(F) + 0{\cdot}004|F^2|]$: H atoms were included using a riding model. {Allen *et al.* used a different weighting scheme,

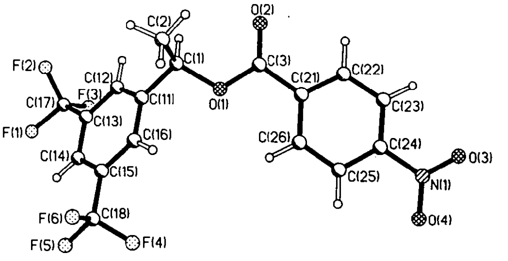

Fig. 1. Molecular struture of (3), showing the atom-numbering scheme.

$w = 4F^2/[\sigma^2(I) + (0{\cdot}08F^2)^2]$, 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

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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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