Migration of the Methoxy Group in an Organosilicon Cation. Reactions of (Bromodiphenylsilyl)(methoxydimethylsilyl)bis(trimethylsilyl)methane with Silver Borofluoride and Silver Chloride. Crystal Structure of (Chlorodiphenylsilyl)(methoxydimethylsilyl)bis(trimethylsilyl)methane

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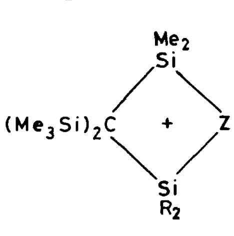
The reaction of the bromide $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2Br)$ (**1**) with $AgBF_4$ in Et_2O is found to give a 4:1 mixture of the rearranged fluoride $(Me_3Si)_2C(SiMe_2F)(SiPh_2OMe)$ and the unrearranged $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2F)$. The 1,3 Si–Si migration of the OMe group is interpreted in terms of rate-determining formation of an MeO-bridged cation formed by abstraction of Br^- by Ag^+ with anchimeric assistance by the OMe group. Surprisingly the reaction of (**1**) with AgCl, although anchimerically assisted, gives only the unrearranged chloride $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2Cl)$ (**2**), possibly because the reaction takes place on the surface of the insoluble AgCl. The crystal structure of (**2**) shows that the MeO group is well placed to carry out a backside nucleophilic attack on the silicon of the $SiPh_2Cl$ group to provide anchimeric assistance to the departure of Cl^-.

Compounds of the type $(Me_3Si)_3CSiR_2I$ (*e.g.* R = Et or Ph) are known to react with Ag^I salts AgY or $AgBF_4$ to give the rearranged products $(Me_3Si)_2C(SiR_2Me)(SiMe_2Y)$ (Y = Y or F), either exclusively (*e.g.* R = Ph) or (*e.g.* R = Et) along with the unrearranged products $(Me_3Si)_3C(SiR_2Y)$ (Y = Y or F).[1] It is thought that the methyl-bridged cations of type (**I**; Z = Me) are initially formed, and are then attacked by the nucleophile Y^- at either the α- or γ-silicon centre, attack at the least sterically hindered centre usually being favoured.[1,2] Analogy with 1,2-bridging in carbocations and with bridging between Al centres, and also calculations on model ions,[3] suggested that the OMe (or OH) group should bridge much more effectively than the Me group in cations of the type (**I**), and thus that corresponding migration of this group should occur. We show below that migration of the group does, indeed, take place in the reaction of $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2Br)$ (**1**), with $AgBF_4$, but, surprisingly, not in that with AgCl. (The outcome of the reaction with $AgBF_4$ has been briefly mentioned previously in a preliminary communication.[4]) The crystal structure of the product from the reaction with AgCl, $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2Cl)$ (**2**), has been determined, and shows that the OMe group is well placed to provide anchimeric assistance to the departure of Cl^-. Such anchimeric assistance is believed to be responsible for the fact that $(Me_3Si)_2C(SiMe_2OMe)(SiMe_2Cl)$ reacts with MeOH > 10^6 times as readily as $(Me_3Si)_3C(SiMe_2Cl)$.[3,4]



(**I**)

$(Me_3Si)_2C(SiMe_2OMe)(SiPh_2Br)$

(**1**)

$(Me_3Si)_2C(SiMe_2OMe)(SiPh_2Cl)$

(**2**)

Results and Discussion

Compound (**1**) was obtained by the bromination of $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2H)$, which was itself made from $(Me_3Si)_2C(SiMe_2OMe)Li$ (*cf.* ref. 5) and Ph_2SiHCl.

A mixture of (**1**) in Et_2O and $AgBF_4$ was stirred at room temperature for 5 min and the solvent was then evaporated off under reduced pressure, the residue extracted with pentane, and the extract filtered and evaporated. The ^{19}F n.m.r. spectrum showed two peaks, a septet at δ −138.1 (*J* 8 Hz) and a singlet at −161.3 p.p.m. in *ca.* 4:1 integration ratio, and these were attributed respectively to the rearranged product $(Me_3Si)_2C(SiMe_2F)(SiPh_2OMe)$ and unrearranged $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2F.)$ [An authentic sample of the latter was available, produced by methanolysis of $(Me_3Si)_2C(SiMe_2I)(SiPh_2F)$.] The ^1H n.m.r. spectrum of the mixture was also consistent with the presence of these two products in 4:1 ratio; *e.g.* the integral for the doublet at δ 0.22 attributable to $SiMe_2F$ was about four times as great as that of the singlet at δ 0.11 attributable to $Si\mathit{Me}_2OMe$, and there were two OMe peaks, in *ca.* 4:1 ratio. Sublimation left the mixture unchanged, and the analysis and mass spectrum of the sublimed material were consistent with either isomer.

When a solution of (**1**) in toluene was stirred with AgCl for 5 min at room temperature and the mixture then worked up as before, the product appeared from its ^1H n.m.r. spectrum to be a single compound, either unrearranged $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2Cl)$ (**2**) or rearranged $(Me_3Si)_2C(SiMe_2Cl)(SiPh_2OMe)$, but an unambiguous choice could not be made between these from spectroscopic data alone (though the chemical shift for the $SiMe_2$ protons is markedly closer to that expected for $SiMe_2Cl$ than for $SiMe_2OMe$). Recrystallization from pentane gave crystals suitable for an *X*-ray diffraction study, which revealed that the product was the unrearranged species (**2**) (see Figure). (Because of the slight possibility that the crystal used might have been unrepresentative, and have come from a small amount of the minor product, several crystals were examined, and were found to be identical with that used.)

The absence of rearrangement in the reaction with AgCl was completely unexpected, but we should note that this is the first time this silver salt has been used in reactions with compounds of the type $(Me_3Si)_2C(SiMe_2Z)(SiR_2X)$ or related species. It is possible that with this highly insoluble salt the reaction takes place wholly at the surface of the solid (whereas it is likely that with $AgBF_4$ it takes place in solution), and that the transfer of an adjacent Cl^- ion to the α-Si from which the Br^- has been abstracted occurs before the cation can diffuse away from the surface or become reoriented. It will be of interest to see whether AgCl behaves differently from the commonly used silver salts in reactions with other $(Me_3Si)_2C(SiMe_2Z)(SiR_2X)$ compounds,

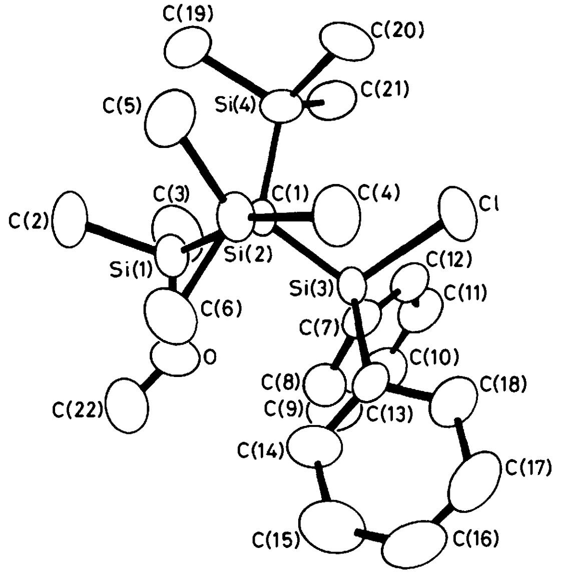
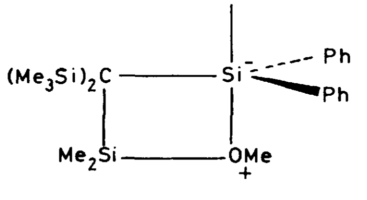


Figure. Structure of (Me$_3$Si)$_2$C(SiMe$_2$OMe)(SiPh$_2$Cl) (**2**) showing the atom numbering used

and if so whether any other silver salts can be found which show a similarly unusual behaviour.

It must be emphasized that what might seem at first sight an alternative possibility for the absence of rearrangement in the reaction of (**1**) with AgCl, namely that a bridged cation (**I**; Z = OMe, R = Ph) is not formed in this case, can be ruled out with some confidence, because without powerful anchimeric assistance by the OMe group to the leaving of Br$^-$, which results in formation of the bridged cation, there would be no reaction, since the bromide (Me$_3$Si)$_3$CSiMe$_2$Br does not react at all with AgCl under the conditions used.

The Structure of (**2**) *and its Significance for the Mechanism of Reactions of Compounds* (Me$_3$Si)$_2$C(SiMe$_2$OMe)(SiR$_2$X).—The most interesting feature of the structure of (**2**) in the context of the present discussion concerns the relative orientation of the MeOMe$_2$Si and ClPh$_2$Si groups, since it is reasonable to assume that the conformation adopted in the solid will also be dominant, or at least make an important contribution, in solution. It has been noted earlier that the γ-OMe group can provide powerful nucleophilic assistance to the departure of Cl$^-$, to form a cyclic cation of type (**I**; Z = OMe), and it can be seen from the Figure that the OMe group in (**2**) is almost ideally placed to give such assistance, the lone pairs side of the oxygen lying close to the side of Si(3) away from the Si–Cl bond. In fact the O–Si(3) distance is only 3.2 Å, compared with the value of 3.6 Å for the sum of the van der Waals radii, and the O–Si(3)–Cl angle is 168°, so that little movement is required to reach a transition state resembling the (probably hypothetical) five-co-ordinate species (**II**). Furthermore, because there is such steric restriction to molecular motions in (**2**), the entropy loss would be only a small part of that normally associated with going from an open to a rigid cyclic structure.



(**II**)

Table 1. Intramolecular distances (Å) and angles (°) with estimated standard deviations in parentheses

Bond lengths

Cl–Si(3)	2.089(2)	Si(1)–O	1.657(3)
Si(1)–C(1)	1.892(4)	Si(1)–C(2)	1.871(5)
Si(1)–C(3)	1.850(5)	Si(2)–C(1)	1.935(4)
Si(2)–C(4)	1.851(4)	Si(2)–C(5)	1.871(5)
Si(2)–C(6)	1.878(5)	Si(3)–C(1)	1.896(4)
Si(3)–C(7)	1.889(4)	Si(3)–C(13)	1.884(4)
Si(4)–C(1)	1.931(4)	Si(4)–C(19)	1.880(5)
Si(4)–C(20)	1.867(5)	Si(4)–C(21)	1.856(5)
O–C(22)	1.302(7)	C(7)–C(8)	1.374(6)
C(7)–C(12)	1.390(6)	C(8)–C(9)	1.383(7)
C(9)–C(10)	1.360(7)	C(10)–C(11)	1.360(7)
C(11)–C(12)	1.382(6)	C(13)–C(14)	1.367(6)
C(13)–C(18)	1.403(6)	C(14)–C(15)	1.389(7)
C(15)–C(16)	1.338(7)	C(16)–C(17)	1.354(7)
C(17)–C(18)	1.374(7)		

Bond angles

O–Si(1)–C(1)	103.9(2)	O–Si(1)–C(2)	108.0(2)
O–Si(1)–C(3)	107.8(2)	C(1)–Si(1)–C(2)	115.4(2)
C(1)–Si(1)–C(3)	115.1(2)	C(2)–Si(1)–C(3)	106.3(2)
C(1)–Si(2)–C(4)	112.8(2)	C(1)–Si(2)–C(5)	113.0(2)
C(1)–Si(2)–C(6)	113.6(2)	C(4)–Si(2)–C(5)	106.0(2)
C(4)–Si(2)–C(6)	106.2(2)	C(5)–Si(2)–C(6)	104.5(2)
Cl–Si(3)–C(1)	107.4(1)	Cl–Si(3)–C(7)	103.2(1)
Cl–Si(3)–C(13)	103.5(1)	C(1)–Si(3)–C(7)	117.3(2)
C(1)–Si(3)–C(13)	119.0(2)	C(7)–Si(3)–C(13)	104.6(2)
C(1)–Si(4)–C(19)	111.9(2)	C(1)–Si(4)–C(20)	113.9(2)
C(1)–Si(4)–C(21)	114.4(2)	C(19)–Si(4)–C(20)	105.1(2)
C(19)–Si(4)–C(21)	104.3(2)	C(20)–Si(4)–C(21)	106.3(2)
Si(1)–O–C(22)	125.5(3)	Si(1)–C(1)–Si(2)	109.8(2)
Si(1)–C(1)–Si(3)	110.0(2)	Si(1)–C(1)–Si(4)	108.4(2)
Si(2)–C(1)–Si(3)	109.4(2)	Si(2)–C(1)–Si(4)	108.5(2)
Si(3)–C(1)–Si(4)	110.7(2)	Si(3)–C(7)–C(8)	120.8(3)
Si(3)–C(7)–C(12)	122.5(3)	C(8)–C(7)–C(12)	116.5(4)
C(7)–C(8)–C(9)	122.3(4)	C(8)–C(9)–C(10)	119.3(5)
C(9)–C(10)–C(11)	120.6(4)	C(10)–C(11)–C(12)	119.6(4)
C(7)–C(12)–C(11)	121.7(4)	Si(3)–C(13)–C(14)	124.3(3)
Si(3)–C(13)–C(18)	119.5(3)	C(14)–C(13)–C(18)	116.0(4)
C(13)–C(14)–C(15)	122.1(4)	C(14)–C(15)–C(16)	120.2(5)
C(15)–C(16)–C(17)	120.0(5)	C(16)–C(17)–C(18)	120.4(5)
C(13)–C(18)–C(17)	121.2(4)		

It is tempting to attribute the relative orientation of the OMe and Cl substituents in (**2**) to incipient operation of electronic factors which would favour the diaxial configuration of the O and Cl atoms in a five-co-ordinate intermediate of type (**II**), but it is likely that it is, in fact, simply the arrangement which minimizes the steric crowding. A silicon atom can accommodate the approach of the lone pairs side of the oxygen atom more readily than that of an Me group, and, we suggest, tolerates the closeness of lone pairs rather than interacting with one of them in a bonding fashion. (It is, noteworthy, however, that Ebsworth and his colleagues have, on reasonable grounds, interpreted intermolecular Si - - - O contacts of 3.1—3.3 Å in terms of weak but directionally specific secondary bonding, and have drawn a parallel between O–SiH$_3$ - - - O and O–H - - - O bonding.[6])

The close approach of the oxygen to the adjacent silicon is reflected in the small C(1)–Si(2)–O angle [103.9(2)°], which is in marked contrast with the associated C(1)–Si(2)–Me angle [mean 115.3(2)°]. There is a similar effect in the C(1)–SiPh$_2$Cl system, the C(1)–Si(3)–Cl angle [107.4(1)°] being much smaller than the C(1)–Si–Ph angles [mean 118(1)°], and the Cl–Si(4) distance only 3.4 Å.

Other features of the structure (see Table 1) are as follows. (*a*) The C(1)–$SiMe_3$ bonds [mean 1.933(3)°] are significantly longer than the C(1)–$SiMe_2OMe$ and C(1)–$SiPh_2Cl$ bonds [1.892(4) and 1.896(4)°, respectively]. (*b*) Within the $(Me_3Si)_2C$ system the Si–Me bonds [mean 1.867(10) Å] are significantly shorter than the Me_3Si–C bonds [mean 1.933(3) Å] and the strain is mainly relieved by opening of the C(1)–Si–Me angles [mean 113.3(9)°] and corresponding closing of the Me–Si–Me angles [mean 105.4(9)°]; both of these effects have been noted previously for related compounds.[7] (*c*) There is a marked closing of the Ph–Si–Ph angle, to 104.6(2)°.

Experimental

Spectra.—^1H N.m.r. spectra were determined at 90 or (for the analysis of the mixture of isomeric fluorides) 360 MHz with solutions in CCl_4 containing CH_2Cl_2 as lock and reference. ^{19}F N.m.r. spectra were measured with a Bruker WP80SY spectrometer operating at 75.4 MHz; solutions were in CCl_4–$CDCl_3$ with $CFCl_3$ in $CDCl_3$ as external reference.

Mass spectra (electron impact) were recorded at 70 eV.

Preparation of $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2X)$ (X = H *or* Br).—(*a*) A 1.6M-solution of Bu^nLi in hexane (5.3 cm^3; 8.50 mmol of BuLi) cooled to −80 °C, was added dropwise to a stirred solution of $(Me_3Si)_2C(SiMe_2OMe)Cl$[5] (2.40 g, 8.50 mmol) in THF (30 cm^3) at −100 °C. The mixture was stirred at −100 °C for a further 1 h then allowed to warm to −78 °C, and a solution of Ph_2SiHCl (1.86 g, 8.50 mmol) in THF (10 cm^3) was added dropwise with stirring. The mixture was allowed to warm gradually to room temperature and the solvent was evaporated under reduced pressure. The residue was extracted with pentane, the extract was filtered then evaporated, and the residual solid was recrystallized from pentane to give *(diphenylsilyl)(methoxydimethylsilyl)bis(trimethylsilyl)methane* (1.50 g, 41%), m.p. 153 °C; δ_H 0.13 (6 H, s, $SiMe_2$), 0.15 (18 H, s, $SiMe_3$), 3.63 (3 H, s, SiOMe), 4.9 (1 H, s, SiH), and 7.13—8.03 (10 H, m, Ph).

(*b*) A solution of Br_2 (1.20 mmol) in CCl_4 (1.20 cm^3) was added dropwise with stirring to a solution of $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2H)$ (0.50 g, 1.16 mmol) in CCl_4 (5 cm^3). The solvent was evaporated under reduced pressure and the residue recrystallized from pentane to give *(bromodiphenylsilyl)(methoxydimethylsilyl)bis(trimethylsilyl)methane* (0.50 g, 85%), m.p. 171 °C (Found: C, 51.9; H, 7.5. $C_{22}H_{37}BrOSi_4$ requires C, 51.9; H, 7.3%); δ_H 0.09 (6 H, s, $SiMe_2$), 0.30 (18 H, s, $SiMe_3$), 3.51 (3 H, s, SiOMe), and 7.1—8.25 (10 H, m, $SiPh_2$); *m/z* 495 (40%, $[M - Me]^+$), 429 (50, $[M - Br]^+$), 417 (45, $[M - PhH - Me]^+$), 381 (100), 341 (85), 73 (40), and 59 (10). The isotope patterns for Br-containing ions were as expected.

Reaction of (**1**) *with* $AgBF_4$.—A mixture of (**1**) (0.50 g, 0.98 mmol), $AgBF_4$ (0.50 g, 2.56 mmol), and anhydrous Et_2O (10 cm^3) was stirred at room temperature for 5 min then evaporated under reduced pressure. The residue was extracted with pentane, and the extract was filtered then evaporated. The ^1H n.m.r. spectrum of the residue was assigned in terms of a *ca.* 4:1 mixture of the rearranged fluoride $(Me_3Si)_2C(SiMe_2F)(SiPh_2OMe)$ (R) [δ_H 0.18 (s, $SiMe_3$), 0.22 (d, $SiMe_2F$), 3.60 (s, OMe), and 7.2—8.0 (m, Ph)] and unrearranged fluoride $(Me_3Si)_2C(SiMe_2OMe)(SiPh_2F)$ (U) [δ_H 0.11 (s, $Si\mathit{Me}_2OMe$), 0.18 (s, $SiMe_3$), 3.65 (s, OMe), and 7.2—8.0 (m, Ph)]; in particular, the integrals for the $SiMe_2F$ and $Si\mathit{Me}_2OMe$ peaks were in *ca.* 4:1 ratio, as were those for the two OMe peaks. The ^{19}F n.m.r. spectrum showed two peaks in *ca.* 4:1 integration ratio, at −138.1 (m, *J* 8 Hz) and −161.3 (s), attributed respectively to $SiMe_2F$ in (R) and $SiPh_2F$ in (U). [The ^1H and ^{19}F n.m.r. spectra of the unrearranged fluoride (U) were available.] The mixture was sublimed (110 °C at 0.2 Torr) to give a solid, m.p. 115—116 °C, which was still a *ca.* 4:1 mixture of the two fluorides (Found: C, 58.7; H, 8.2. $C_{22}H_{37}FOSi_4$ requires C, 58.9; H, 8.3%); g.l.c.–mass spectrometry gave only one peak, with *m/z* 433 (100, $[M - Me]^+$), 371 (10, $[M - SiMe_2F]^+$), 355 (40, $[M - PhH - Me]^+$), 329 (30), 175 (40), 73 (80), and 59 (20).

Table 2. Fractional atomic co-ordinates (× 10^4) with estimated standard deviations in parentheses

Atom	*x*	*y*	*z*
Cl	4 840(1)	1 821(1)	558(1)
Si(1)	9 656(1)	1 856(1)	1 490(1)
Si(2)	6 773(1)	2 167(1)	2 136(0)
Si(3)	6 818(1)	2 549(1)	823(0)
Si(4)	7 166(1)	135(1)	1 323(1)
O	9 909(3)	3 202(2)	1 391(1)
C(1)	7 608(4)	1 689(3)	1 436(2)
C(2)	10 542(5)	1 469(5)	2 192(2)
C(3)	10 659(5)	1 102(4)	945(2)
C(4)	4 812(5)	2 504(5)	2 047(2)
C(5)	6 924(6)	1 096(5)	2 711(2)
C(6)	7 675(5)	3 433(4)	2 456(2)
C(7)	7 868(4)	2 587(3)	150(2)
C(8)	8 972(5)	3 341(4)	87(2)
C(9)	9 684(5)	3 452(4)	−416(2)
C(10)	9 298(5)	2 786(4)	−864(2)
C(11)	8 204(5)	2 034(4)	−824(2)
C(12)	7 504(5)	1 927(4)	−318(2)
C(13)	6 315(4)	4 041(3)	954(2)
C(14)	7 244(5)	4 813(4)	1 191(2)
C(15)	6 863(5)	5 925(4)	1 236(3)
C(16)	5 564(6)	6 277(4)	1 031(2)
C(17)	4 601(5)	5 545(4)	793(2)
C(18)	4 955(5)	4 441(4)	752(2)
C(19)	8 395(5)	−782(4)	1 776(2)
C(20)	5 278(5)	−257(4)	1 508(2)
C(21)	7 400(6)	−369(4)	588(2)
C(22)	11 168(6)	3 698(5)	1 384(3)

Reaction of (**1**) *with* AgCl.—A mixture of (**1**) (0.25 g, 0.49 mmol), freshly made but carefully dried AgCl (0.70 g, 4.90 mmol), and toluene (5 cm^3) was stirred at room temperature for 10 min then evaporated under reduced pressure. The residue was extracted with pentane, the extract was filtered and evaporated, and the solid residue was sublimed (125 °C at 0.2 Torr) to give *(chlorodiphenylsilyl)(methoxydimethylsilyl)bis(trimethylsilyl)methane* (0.19 g, 83%), m.p. 155 °C (Found: C, 56.9; H, 7.8. $C_{22}H_{37}ClOSi_4$ requires C, 56.9; H, 8.0%); δ_H 0.10 (6 H, s, $SiMe_2$), 0.30 (18 H, s, $SiMe_3$), 3.58 (3 H, s, OMe), and 7.20—8.20 (10 H, m, Ph_2); *m/z* 449 (50%, $[M - Me]^+$), 371 (20, $[M - PhH - Me]^+$), 341 (35, $[M - Me_3SiCl - Me]^+$), 247 (15, $[M - SiPh_2Cl]^+$), 89 (10), 73 (100), and 59 (15). (The Cl-containing ions showed the expected isotope pattern.)

A sample was recrystallized from pentane to give crystals suitable for the *X*-ray diffraction study.

Crystal Structure Determination.—*Crystal data.* $C_{22}H_{37}ClOSi_4$, $M = 465.4$, monoclinic, $a = 9.188(6)$, $b = 12.034(1)$, $c = 23.551(2)$ Å, $\beta = 92.38(7)°$, $U = 2\,603.9$ Å^3, $Z = 4$, $D_c = 1.18$ g cm^3, $F(000) = 1\,000$. Mo-K_α radiation, $\lambda = 0.710\,69$ Å, $\mu = 3.4$ cm^{-1}, space group $P2_1/c$.

Data were measured on an Enraf–Nonius CAD4 diffractometer with a crystal of dimensions *ca.* 0.25 × 0.2 × 0.25 mm. Preliminary cell dimensions were derived by use of the SEARCH and INDEX routines of the CAD4, and final values calculated from the setting angles for 16 reflections with $\theta \approx 14°$. Intensities for $h \pm k \pm l$ reflections with $2 < \theta < 23°$ were measured by a θ—2θ scan with a scan width $\Delta\theta =$

$(0.8 \pm 0.35 \tan \theta)^\circ$ and monochromated Mo-K_α radiation. The scan rate for each reflection was determined by a rapid prescan at 10° min^{-1} in θ, when any reflections with $I < \sigma(I)$ was coded as unobserved. The remaining reflections were measured at such a speed as to give a minimum value of $\sigma(I)/I$ of 0.05 subject to a maximum scan time of 60 s. Two standard reflections monitored every hour showed no significant variation. After correction for Lorentz-polarization (Lp) effects but not absorption, any equivalent data were averaged and 2 329 reflections with $|F^2| > \sigma |F^2|$ were used in the structure refinement. The values of $\sigma(F^2)$ were taken as $[\sigma^2(I) + (0.02I)^2]^{\frac{1}{2}}/\text{Lp}$.

The structure was solved by direct method using the MULTAN program.[8] Refinement of non-hydrogen atoms with anisotropic thermal parameters was by full matrix least squares. Hydrogen atoms were located on a difference map and included in the refinement with fixed co-ordinates and isotropic temperature factors of $B = 6.0$ Å^2. Refinement converged at $R = 0.044$, $R' = 0.046$ where the weighting scheme was $\omega = 1/\sigma^2(F)$. A final difference map was featureless.

The structure solution and refinement were carried out on a PDP11/34 computer with the Enraf–Nonius structure determination package. Scattering factors for neutral atoms were from reference 9. Non-hydrogen atom co-ordinates are listed in Table 2.

Acknowledgements

We thank the S.E.R.C. for support, Dr. A. G. Avent for n.m.r. spectra, Mr. A. M. Greenway for mass spectra, and the Iraqi Ministry of Higher Education for the award of scholarships to S. T. N. and N. H. B.

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Received 8th September 1986; *Paper* 6/1794