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# Methyl 3,4-dibromo-2-(triisopropylsilyl)-1*H*-pyrrole-1-carboxylate

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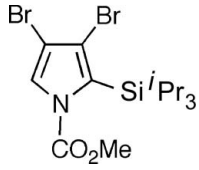
Key indicators: single-crystal X-ray study; $T$ = 200 K; mean $\sigma$(C–C) = 0.004 Å; $R$ factor = 0.024; $wR$ factor = 0.027; data-to-parameter ratio = 9.8.

The title compound, $C_{15}H_{25}Br_2NO_2Si$, represents a rare example of a C2-silylated pyrrole for which a single-crystal X-ray analysis has been completed. The molecule adopts an *s-transoid* conformation about the N—CO bond of the carbamate residue.

## Related literature

For related literature, see: Bray *et al.* (1990); Couzijn *et al.* (2004); Frenzel *et al.* (1996); König *et al.* (1995); Kang *et al.* (1999, 2000); Liu *et al.* (2000); Marsh (2004).




## Experimental

### *Crystal data*

$C_{15}H_{25}Br_2NO_2Si$
$M_r$ = 439.26
Monoclinic, $P2_1/c$
$a$ = 14.6807 (4) Å
$b$ = 7.3800 (2) Å
$c$ = 17.4291 (5) Å
$\beta$ = 92.8487 (15)°
$V$ = 1886.00 (9) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 4.37 mm$^{-1}$
$T$ = 200 K
0.36 × 0.10 × 0.07 mm

### *Data collection*

Nonius KappaCCD diffractometer
Absorption correction: integration [via Gaussian method (Coppens, 1970) implemented in *maXus* (Mackay *et al.*, 1999)]
$T_{min}$ = 0.447, $T_{max}$ = 0.762
41120 measured reflections
4336 independent reflections
2605 reflections with $I > 3\sigma(I)$
$R_{int}$ = 0.059

### *Refinement*

$R$ = 0.024
$wR$ = 0.027
$S$ = 1.15
2605 reflections
265 parameters
Only H-atom coordinates refined
$\Delta\rho_{max}$ = 0.33 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.49 e Å$^{-3}$

**Table 1**
Selected interatomic distances (Å).

| | | | |
|---|---|---|---|
| Br21···O7$^{i}$ | 3.105 (2) | C5···C18$^{ii}$ | 3.589 (5) |

Symmetry codes: (i) $x, -y+\frac{3}{2}, z+\frac{1}{2}$; (ii) $x, y+1, z$.

Data collection: *COLLECT* (Nonius, 1997); cell refinement: *DENZO/SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *DENZO/SCALEPACK*; program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEPII* (Johnson, 1976) in *TEXSAN* (Molecular Structure Corporation, 1997); software used to prepare material for publication: *CRYSTALS*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HG2268).

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# supplementary materials

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# Methyl 3,4-dibromo-2-(triisopropylsilyl)-1*H*-pyrrole-1-carboxylate

**M. G. Banwell, K. E. Holden and A. C. Willis**

## Comment

During attempts to prepare compound (I) by sequential treatment of the known tribromide (II) (Bray *et al.*, 1990) with phenyllithium then methyl chloroformate an isomeric product was sometimes formed (and occasionally to the exclusion of the desired material). The isomer was subjected to a single-crystal X-ray analysis and thus establishing it was the title compound (III). This previously unreported tetra-substituted pyrrole most likely arises from an initial and selective lithium-for-bromine exchange reaction between phenyllithium and tribromide (II) and so forming the corresponding C2-lithiated compound. This last species presumably undergoes silyl group migration to give the more stable N1-lithiated isomer that then reacts with added methyl chloroformate and so affording the observed product. The silyl group migration proved to be a temperature sensitive process that could be suppressed by strict maintenance of the reaction temperature at 195 K and with the result that the desired compound, (I), was formed in preparatively useful yields.

Compound (III) represents the first example of a C2-triisopropylsilyl-substituted pyrrole for which a single-crystal X-ray analysis has been reported although a few such analyses of other types of C2-silylated pyrroles have been described (König *et al.*, 1995; Frenzel *et al.*, 1996; Kang *et al.*, 1999; Liu *et al.*, 2000; Kang *et al.*, 2000; Couzijn *et al.*, 2004; Marsh, 2004). All of the non-hydrogen-containing bond lengths and angles (Table 1) associated with compound (III) fall within the expected ranges. The most conspicuous feature associated with the structure is the *s-transoid* arrangement adopted by the carbomethoxy group about the C6–N1 bond and this undoubtedly results from the steric effects exerted by the adjacent and bulky triisopropylsilyl group. The C2–C3 bond is notably longer than its C4–C5 counterpart (1.382 (3) *vs* 1.342 (3) Å) and may reflect the repulsive effectsoperating between the C2-triisopropylsilyl and C3-bromine groups attached to the carbons of the former bond. The N1–C2 bond is also longer than the equivalent N1–C5 bond (1.423 *vs* 1.385 Å) and this situation is probably the result of similar effects. In contrast the C3- and C4-bromine bonds are of similar length (1.879 (2) *vs* 1.871 (2) Å).

## Experimental

Phenyllithium (0.41 ml of a 1.8 *M* solution in cyclohexane/diethyl ether, 0.74 mmol, *ex* Aldrich Chemical Co.) was added, dropwise, to a magnetically stirred solution of tribromopyrrole (II) (341 mg, 0.74 mmol) in dry THF (15 ml) maintained at 195 K under a nitrogen atmosphere. After 0.25 h unchilled methyl chloroformate (60 µl, 0.78 mmol) was added to the reaction mixture and then the cooling bath removed. Once the reaction mixture had warmed to 291 K it was diluted with water (10 ml) and extracted with ethyl acetate (3 × 15 ml). The combined organic extracts were then dried ($MgSO_4$), filtered and concentrated under reduced pressure. Subjection of the ensuing light-yellow oil to flash chromatography (silica, hexane elution) and concentration of the relevant fractions ($R_f$ = 1/5) under reduced pressure afforded a white solid. Recrystallization of this material (methanol or THF) then gave the title compound (III) (42 mg, 13%) as colourless needles, m.p. 366–367 K [Found: $(M - C_3H_7^{\cdot})^+$, 393.9485. $C_{15}H_{25}{}^{79}Br_2NO_2Si$ requires $(M - C_3H_7^{\cdot})^+$, 393.9474]. $^1$H NMR (300 MHz, $CDCl_3$): $\delta$ 7.53 (*s*, 1H), 3.94 (*s*, 3H), 1.84 (*septet*, *J* = 7.5 Hz, 3H), 1.10 (*d*, *J* = 7.5 Hz, 18H); $^{13}$C NMR (75 MHz, $CDCl_3$): $\delta$ 150.4 (C),

130.7 (C), 125.1 (CH), 117.7 (C), 106.3 (C), 54.7 ($CH_3$), 19.2 (6 × $CH_3$), 12.8 (3 × CH); IR (KBr, $\nu_{max}$, $cm^{-1}$): 2945, 2866, 1757, 1440, 1353, 1302, 1280, 1215, 1127, 1036; MS (EI, 70 eV): 398, 396 and 394 [$(M - C_3H_7^{\cdot})^+$, 70, 100 and 69%].

### Refinement

All H atoms were observed in a difference electron density map prior to their inclusion. They were added at calculated positions, and then refined positionally. The largest peaks in the final difference electron density map are located near the bromine atoms.

### Figures

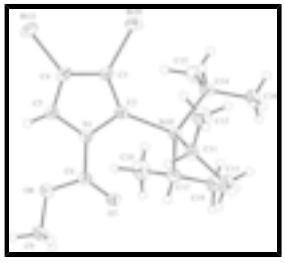
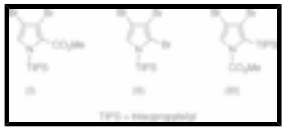


Figure 1. Molecular structure of (III) with labelling of selected atoms. Anisotropic displacement ellipsoids show 30% probability levels. Hydrogen atoms are drawn as circles with small radii.

Figure 2. Unit cell packing diagram of (III) projected down the *b* axis. Hydrogen atoms are drawn as circles with small radii.



### Methyl 3,4-dibromo-2-(triisopropylsilyl)-1*H*-pyrrole-1-carboxylate

*Crystal data*

| | |
|---|---|
| $C_{15}H_{25}Br_2NO_2Si$ | $F_{000}$ = 888 |
| $M_r$ = 439.26 | $D_x$ = 1.547 Mg $m^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation<br>λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 92459 reflections |
| $a$ = 14.6807 (4) Å | θ = 3–27º |
| $b$ = 7.3800 (2) Å | μ = 4.37 $mm^{-1}$ |
| $c$ = 17.4291 (5) Å | $T$ = 200 K |
| β = 92.8487 (15)º | Needle, colourless |
| $V$ = 1886.00 (9) $Å^3$ | 0.36 × 0.10 × 0.07 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 2605 reflections with $I > 3\sigma(I)$ |
| Monochromator: graphite | $R_{int} = 0.059$ |
| $T = 200$ K | $\theta_{max} = 27.5^{o}$ |
| φ and ω scans with CCD | $\theta_{min} = 3.1^{o}$ |
| Absorption correction: integration [via Gaussian method (Coppens, 1970) implemented in maXus (Mackay *et al.*, 1999)] | $h = -19\rightarrow19$ |
| $T_{min} = 0.447$, $T_{max} = 0.762$ | $k = -8\rightarrow9$ |
| 41120 measured reflections | $l = -22\rightarrow22$ |
| 4336 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F$ | Hydrogen site location: inferred from neighbouring sites |
| Least-squares matrix: full | Only H-atom coordinates refined |
| $R[F^2 > 2\sigma(F^2)] = 0.024$ | Method, part 1, Chebychev polynomial [Carruthers & Watkin (1979). Acta Cryst. A35, 698–699; *P*rince (1982). Mathematical Techniques in Crystallography and Materials Science. Ne*w* York: Springer–Verlag] [*w*eight] = $1/[A_0*T_0(x) + A_1*T_1(x) \cdots + A_{n-1}]*T_{n-1}(x)]$ where $A_i$ are the Chebychev coefficients listed below and x = $F/F$max Method = Robust Weighting (*P*rince, 1982) W = [*w*eight][1-($\delta F/6\sigma F$)$^2$]$^2$, $A_i$ are: 2.01 -0.979 2.14 -0.341 0.473 |
| $wR(F^2) = 0.027$ | $(\Delta/\sigma)_{max} = 0.002$ |
| $S = 1.15$ | $\Delta\rho_{max} = 0.33$ e Å$^{-3}$ |
| 2605 reflections | $\Delta\rho_{min} = -0.49$ e Å$^{-3}$ |
| 265 parameters | Extinction correction: None |
| Primary atom site location: structure-invariant direct methods | |

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| N1 | 0.36958 (13) | 0.5984 (3) | 0.44081 (10) | 0.0297 |
| C2 | 0.28285 (16) | 0.5206 (3) | 0.45316 (12) | 0.0311 |
| C3 | 0.26276 (17) | 0.5898 (4) | 0.52402 (13) | 0.0359 |
| C4 | 0.33465 (18) | 0.7017 (3) | 0.55408 (13) | 0.0362 |
| C5 | 0.39939 (18) | 0.7052 (4) | 0.50238 (13) | 0.0354 |
| C6 | 0.41628 (16) | 0.5914 (3) | 0.37249 (13) | 0.0313 |
| O7 | 0.38537 (12) | 0.5309 (2) | 0.31321 (9) | 0.0383 |
| O8 | 0.49883 (12) | 0.6636 (3) | 0.38432 (10) | 0.0445 |
| C9 | 0.5537 (2) | 0.6726 (5) | 0.3175 (2) | 0.0534 |
| Si10 | 0.22018 (4) | 0.35292 (9) | 0.38468 (3) | 0.0298 |

| | | | | |
|---|---|---|---|---|
| C11 | 0.16792 (19) | 0.4966 (4) | 0.30420 (14) | 0.0388 |
| C12 | 0.0864 (2) | 0.6051 (5) | 0.3294 (2) | 0.0580 |
| C13 | 0.1456 (3) | 0.3964 (5) | 0.22850 (17) | 0.0550 |
| C14 | 0.12861 (19) | 0.2398 (4) | 0.44087 (15) | 0.0421 |
| C15 | 0.1679 (3) | 0.1198 (5) | 0.5064 (2) | 0.0597 |
| C16 | 0.0573 (2) | 0.1367 (5) | 0.3905 (2) | 0.0555 |
| C17 | 0.30387 (18) | 0.1785 (4) | 0.34943 (15) | 0.0389 |
| C18 | 0.3822 (2) | 0.1358 (5) | 0.4070 (2) | 0.0571 |
| C19 | 0.2602 (3) | 0.0001 (4) | 0.3208 (2) | 0.0539 |
| Br20 | 0.15689 (2) | 0.56204 (5) | 0.579168 (18) | 0.0638 |
| Br21 | 0.34204 (2) | 0.82451 (4) | 0.648093 (14) | 0.0502 |
| H51 | 0.452 (2) | 0.765 (4) | 0.5030 (16) | 0.0425* |
| H91 | 0.609 (3) | 0.718 (5) | 0.3363 (19) | 0.0648* |
| H92 | 0.565 (2) | 0.564 (5) | 0.301 (2) | 0.0648* |
| H93 | 0.521 (2) | 0.745 (5) | 0.280 (2) | 0.0648* |
| H111 | 0.215 (2) | 0.581 (4) | 0.2954 (16) | 0.0462* |
| H121 | 0.068 (2) | 0.691 (5) | 0.291 (2) | 0.0692* |
| H122 | 0.094 (3) | 0.661 (5) | 0.376 (2) | 0.0692* |
| H123 | 0.035 (3) | 0.524 (5) | 0.332 (2) | 0.0692* |
| H131 | 0.121 (2) | 0.477 (5) | 0.191 (2) | 0.0654* |
| H132 | 0.198 (3) | 0.336 (5) | 0.211 (2) | 0.0654* |
| H133 | 0.100 (2) | 0.308 (5) | 0.237 (2) | 0.0654* |
| H141 | 0.097 (2) | 0.340 (4) | 0.4636 (18) | 0.0510* |
| H151 | 0.218 (3) | 0.180 (5) | 0.540 (2) | 0.0720* |
| H152 | 0.189 (3) | 0.011 (6) | 0.487 (2) | 0.0720* |
| H153 | 0.119 (3) | 0.076 (5) | 0.536 (2) | 0.0720* |
| H161 | 0.025 (2) | 0.218 (5) | 0.349 (2) | 0.0665* |
| H162 | 0.087 (3) | 0.033 (5) | 0.369 (2) | 0.0665* |
| H163 | 0.014 (2) | 0.089 (5) | 0.422 (2) | 0.0665* |
| H171 | 0.330 (2) | 0.235 (4) | 0.3058 (17) | 0.0468* |
| H181 | 0.417 (2) | 0.244 (6) | 0.423 (2) | 0.0683* |
| H182 | 0.424 (2) | 0.046 (5) | 0.382 (2) | 0.0683* |
| H183 | 0.363 (2) | 0.080 (5) | 0.454 (2) | 0.0683* |
| H191 | 0.211 (3) | 0.021 (5) | 0.282 (2) | 0.0649* |
| H192 | 0.307 (2) | −0.077 (5) | 0.296 (2) | 0.0649* |
| H193 | 0.241 (2) | −0.066 (5) | 0.361 (2) | 0.0649* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| N1 | 0.0299 (10) | 0.0344 (10) | 0.0250 (8) | −0.0019 (8) | 0.0032 (7) | −0.0007 (8) |
| C2 | 0.0346 (12) | 0.0344 (12) | 0.0246 (10) | −0.0012 (10) | 0.0044 (9) | 0.0009 (9) |
| C3 | 0.0422 (14) | 0.0371 (13) | 0.0288 (11) | −0.0024 (11) | 0.0062 (10) | −0.0026 (10) |
| C4 | 0.0484 (15) | 0.0337 (13) | 0.0262 (10) | 0.0012 (11) | −0.0007 (10) | −0.0015 (9) |
| C5 | 0.0402 (14) | 0.0359 (14) | 0.0298 (11) | −0.0039 (11) | −0.0015 (10) | −0.0008 (9) |
| C6 | 0.0355 (12) | 0.0279 (12) | 0.0308 (11) | 0.0016 (10) | 0.0056 (9) | 0.0047 (9) |
| O7 | 0.0454 (10) | 0.0455 (10) | 0.0243 (8) | −0.0084 (8) | 0.0061 (7) | −0.0006 (7) |
| O8 | 0.0348 (9) | 0.0565 (12) | 0.0430 (9) | −0.0121 (9) | 0.0105 (7) | −0.0059 (9) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C9 | 0.0472 (17) | 0.0580 (19) | 0.0571 (18) | −0.0137 (16) | 0.0232 (14) | −0.0044 (16) |
| Si10 | 0.0305 (3) | 0.0318 (3) | 0.0271 (3) | −0.0021 (3) | 0.0026 (2) | −0.0004 (2) |
| C11 | 0.0380 (14) | 0.0409 (14) | 0.0368 (12) | −0.0017 (12) | −0.0047 (10) | 0.0031 (11) |
| C12 | 0.0503 (18) | 0.055 (2) | 0.068 (2) | 0.0140 (16) | −0.0045 (16) | 0.0031 (17) |
| C13 | 0.060 (2) | 0.067 (2) | 0.0368 (14) | −0.0038 (18) | −0.0100 (14) | 0.0001 (14) |
| C14 | 0.0426 (15) | 0.0432 (15) | 0.0412 (13) | −0.0079 (13) | 0.0094 (11) | −0.0042 (12) |
| C15 | 0.071 (2) | 0.060 (2) | 0.0494 (17) | −0.0170 (17) | 0.0144 (16) | 0.0175 (15) |
| C16 | 0.0421 (16) | 0.058 (2) | 0.067 (2) | −0.0181 (15) | 0.0087 (15) | −0.0077 (16) |
| C17 | 0.0395 (13) | 0.0332 (13) | 0.0445 (13) | −0.0016 (11) | 0.0075 (11) | −0.0029 (11) |
| C18 | 0.0496 (18) | 0.0403 (17) | 0.081 (2) | 0.0089 (14) | −0.0040 (17) | 0.0041 (16) |
| C19 | 0.060 (2) | 0.0400 (16) | 0.0632 (19) | −0.0017 (15) | 0.0128 (16) | −0.0114 (14) |
| Br20 | 0.06063 (19) | 0.0831 (3) | 0.05041 (17) | −0.01778 (18) | 0.03013 (14) | −0.02307 (16) |
| Br21 | 0.0718 (2) | 0.04899 (16) | 0.03003 (12) | −0.00375 (15) | 0.00367 (11) | −0.01156 (11) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| N1—C2 | 1.423 (3) | C12—H122 | 0.91 (4) |
| N1—C5 | 1.385 (3) | C12—H123 | 0.96 (4) |
| N1—C6 | 1.404 (3) | C13—H131 | 0.94 (4) |
| C2—C3 | 1.382 (3) | C13—H132 | 0.95 (4) |
| C2—Si10 | 1.922 (2) | C13—H133 | 0.95 (4) |
| C3—C4 | 1.419 (4) | C14—C15 | 1.535 (4) |
| C3—Br20 | 1.879 (2) | C14—C16 | 1.535 (4) |
| C4—C5 | 1.342 (3) | C14—H141 | 0.97 (3) |
| C4—Br21 | 1.871 (2) | C15—H151 | 1.02 (4) |
| C5—H51 | 0.89 (3) | C15—H152 | 0.93 (4) |
| C6—O7 | 1.194 (3) | C15—H153 | 0.96 (4) |
| C6—O8 | 1.331 (3) | C16—H161 | 1.04 (4) |
| O8—C9 | 1.450 (3) | C16—H162 | 0.97 (4) |
| C9—H91 | 0.93 (4) | C16—H163 | 0.93 (4) |
| C9—H92 | 0.88 (4) | C17—C18 | 1.521 (4) |
| C9—H93 | 0.95 (4) | C17—C19 | 1.537 (4) |
| Si10—C11 | 1.890 (3) | C17—H171 | 0.96 (3) |
| Si10—C14 | 1.896 (3) | C18—H181 | 0.98 (4) |
| Si10—C17 | 1.902 (3) | C18—H182 | 1.02 (4) |
| C11—C12 | 1.523 (4) | C18—H183 | 0.98 (4) |
| C11—C13 | 1.534 (4) | C19—H191 | 0.98 (4) |
| C11—H111 | 0.95 (3) | C19—H192 | 1.00 (4) |
| C12—H121 | 0.95 (4) | C19—H193 | 0.91 (4) |
| Br21···O7$^{i}$ | 3.105 (2) | C5···C18$^{ii}$ | 3.589 (5) |
| C2—N1—C5 | 111.29 (18) | C11—C13—H131 | 110 (2) |
| C2—N1—C6 | 126.27 (19) | C11—C13—H132 | 111 (2) |
| C5—N1—C6 | 122.0 (2) | H131—C13—H132 | 111 (3) |
| N1—C2—C3 | 102.6 (2) | C11—C13—H133 | 109 (2) |
| N1—C2—Si10 | 124.77 (15) | H131—C13—H133 | 107 (3) |
| C3—C2—Si10 | 132.56 (18) | H132—C13—H133 | 109 (3) |
| C2—C3—C4 | 110.9 (2) | Si10—C14—C15 | 112.8 (2) |
| C2—C3—Br20 | 129.47 (19) | Si10—C14—C16 | 113.8 (2) |

| | | | |
|---|---|---|---|
| C4—C3—Br20 | 119.57 (17) | C15—C14—C16 | 111.1 (3) |
| C3—C4—C5 | 107.6 (2) | Si10—C14—H141 | 104.0 (18) |
| C3—C4—Br21 | 127.93 (18) | C15—C14—H141 | 107.8 (18) |
| C5—C4—Br21 | 124.5 (2) | C16—C14—H141 | 106.7 (18) |
| N1—C5—C4 | 107.6 (2) | C14—C15—H151 | 114 (2) |
| N1—C5—H51 | 122.1 (18) | C14—C15—H152 | 110 (2) |
| C4—C5—H51 | 130.3 (19) | H151—C15—H152 | 110 (3) |
| N1—C6—O7 | 124.5 (2) | C14—C15—H153 | 109 (2) |
| N1—C6—O8 | 109.5 (2) | H151—C15—H153 | 112 (3) |
| O7—C6—O8 | 126.0 (2) | H152—C15—H153 | 100 (3) |
| C6—O8—C9 | 115.6 (2) | C14—C16—H161 | 113.1 (20) |
| O8—C9—H91 | 104 (2) | C14—C16—H162 | 108 (2) |
| O8—C9—H92 | 111 (2) | H161—C16—H162 | 113 (3) |
| H91—C9—H92 | 106 (3) | C14—C16—H163 | 109 (2) |
| O8—C9—H93 | 107 (2) | H161—C16—H163 | 108 (3) |
| H91—C9—H93 | 116 (3) | H162—C16—H163 | 105 (3) |
| H92—C9—H93 | 113 (3) | Si10—C17—C18 | 114.0 (2) |
| C2—Si10—C11 | 105.30 (11) | Si10—C17—C19 | 114.7 (2) |
| C2—Si10—C14 | 107.01 (11) | C18—C17—C19 | 109.0 (3) |
| C11—Si10—C14 | 110.99 (13) | Si10—C17—H171 | 104.3 (18) |
| C2—Si10—C17 | 109.84 (11) | C18—C17—H171 | 107.2 (17) |
| C11—Si10—C17 | 112.63 (12) | C19—C17—H171 | 107.0 (18) |
| C14—Si10—C17 | 110.78 (13) | C17—C18—H181 | 113 (2) |
| Si10—C11—C12 | 112.1 (2) | C17—C18—H182 | 107 (2) |
| Si10—C11—C13 | 115.4 (2) | H181—C18—H182 | 109 (3) |
| C12—C11—C13 | 111.4 (3) | C17—C18—H183 | 114 (2) |
| Si10—C11—H111 | 102.3 (18) | H181—C18—H183 | 106 (3) |
| C12—C11—H111 | 107.2 (18) | H182—C18—H183 | 107 (3) |
| C13—C11—H111 | 107.6 (18) | C17—C19—H191 | 112 (2) |
| C11—C12—H121 | 110 (2) | C17—C19—H192 | 110 (2) |
| C11—C12—H122 | 116 (2) | H191—C19—H192 | 107 (3) |
| H121—C12—H122 | 110 (3) | C17—C19—H193 | 110 (2) |
| C11—C12—H123 | 108 (2) | H191—C19—H193 | 112 (3) |
| H121—C12—H123 | 105 (3) | H192—C19—H193 | 106 (3) |
| H122—C12—H123 | 108 (3) | | |
| Br20—C3—C2—Si10 | −6.5 (4) | C2—Si10—C14—C16 | 165.9 (2) |
| Br20—C3—C2—N1 | 176.4 (2) | C2—Si10—C17—C18 | 31.0 (2) |
| Br20—C3—C4—Br21 | 3.2 (3) | C2—Si10—C17—C19 | 157.7 (2) |
| Br20—C3—C4—C5 | −177.1 (2) | C2—N1—C5—C4 | −0.8 (3) |
| Br21—C4—C3—C2 | −179.1 (2) | C2—C3—C4—C5 | 0.7 (3) |
| Br21—C4—C5—N1 | 179.8 (2) | C3—C2—Si10—C11 | 104.7 (3) |
| Si10—C2—N1—C5 | −176.3 (2) | C3—C2—Si10—C14 | −13.4 (3) |
| Si10—C2—N1—C6 | 11.5 (3) | C3—C2—Si10—C17 | −133.8 (2) |
| Si10—C2—C3—C4 | 176.1 (2) | C3—C2—N1—C5 | 1.1 (3) |
| O7—C6—O8—C9 | 1.9 (4) | C3—C2—N1—C6 | −171.1 (2) |
| O7—C6—N1—C2 | 6.5 (4) | C4—C5—N1—C6 | 171.9 (2) |
| O7—C6—N1—C5 | −165.0 (2) | C11—Si10—C14—C15 | 179.4 (2) |
| O8—C6—N1—C2 | −174.1 (2) | C11—Si10—C14—C16 | 51.5 (2) |
| O8—C6—N1—C5 | 14.4 (3) | C11—Si10—C17—C18 | 148.0 (2) |

| | | | |
|---|---|---|---|
| N1—C2—Si10—C11 | −78.7 (2) | C11—Si10—C17—C19 | −85.3 (2) |
| N1—C2—Si10—C14 | 163.2 (2) | C12—C11—Si10—C14 | 42.3 (2) |
| N1—C2—Si10—C17 | 42.8 (2) | C12—C11—Si10—C17 | 167.1 (2) |
| N1—C2—C3—C4 | −1.1 (3) | C13—C11—Si10—C14 | −86.7 (3) |
| N1—C5—C4—C3 | 0.1 (3) | C13—C11—Si10—C17 | 38.2 (3) |
| N1—C6—O8—C9 | −177.5 (2) | C14—Si10—C17—C18 | −87.1 (2) |
| C2—Si10—C11—C12 | −73.2 (2) | C14—Si10—C17—C19 | 39.7 (2) |
| C2—Si10—C11—C13 | 157.8 (2) | C15—C14—Si10—C17 | 53.5 (2) |
| C2—Si10—C14—C15 | −66.2 (2) | C16—C14—Si10—C17 | −74.4 (2) |

Symmetry codes: (i) *x*, −*y*+3/2, *z*+1/2; (ii) *x*, *y*+1, *z*.

**Fig. 1**

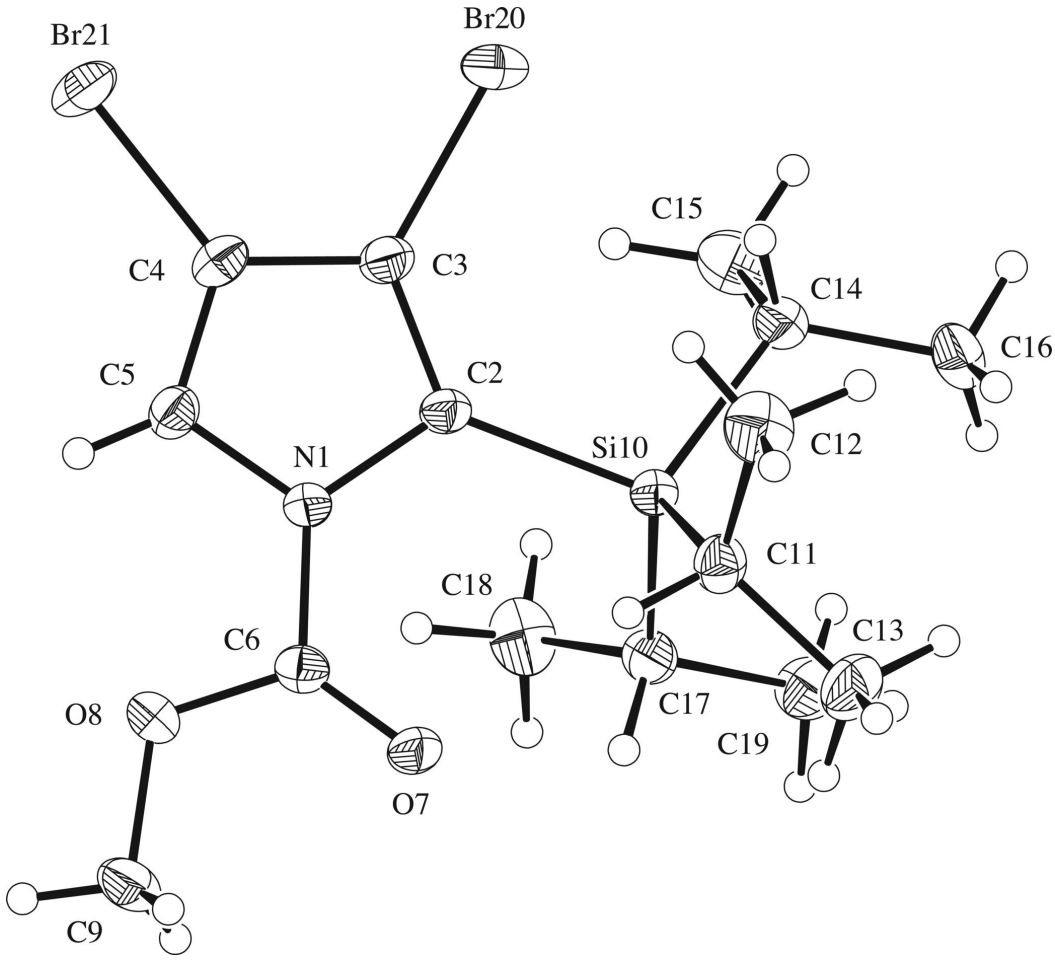

Br21
Br20
C15
C4
C3
C14
C16
C5
C2
C12
N1
Si10
C11
C18
C6
C13
O8
C17
C19
O7
C9

**Fig. 2**

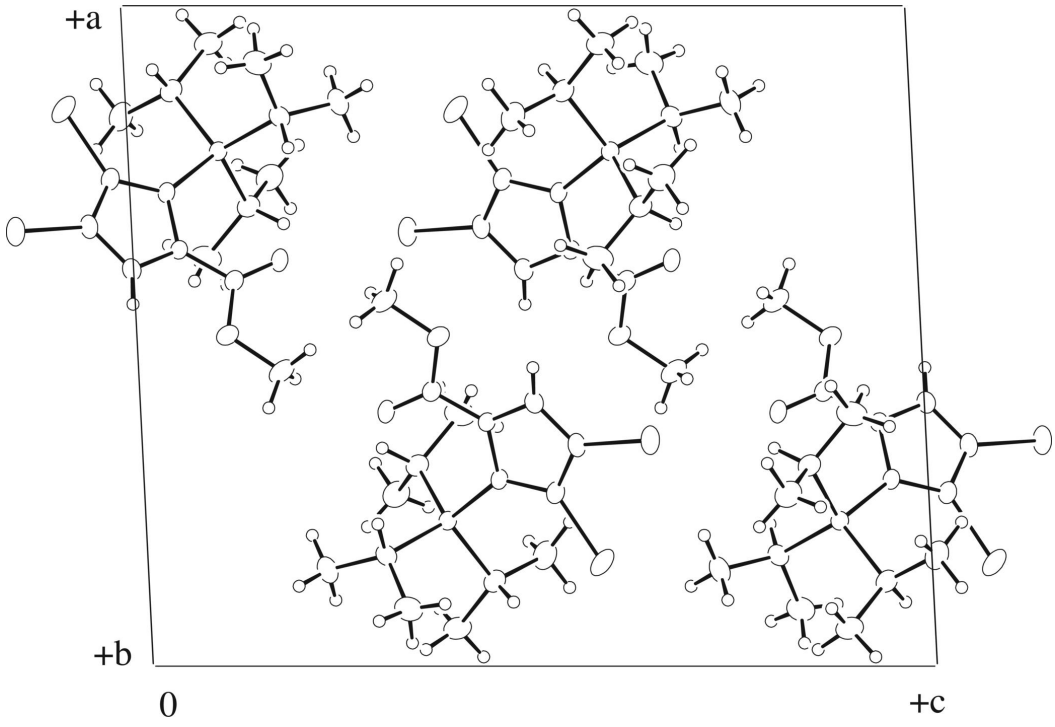
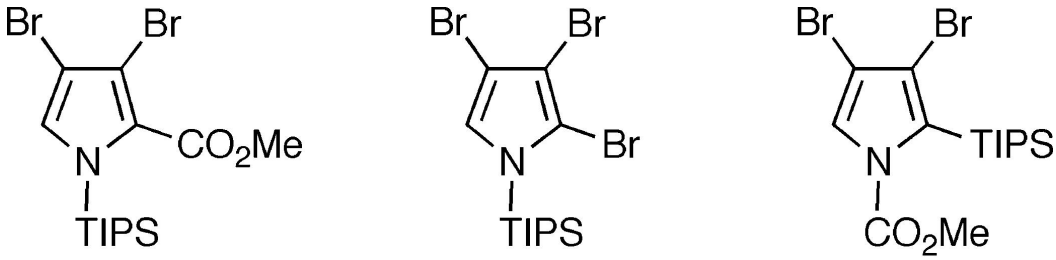


**Fig. 3**




(I) (II) (III)

TIPS = triisopropylsilyl