Thus, Ni could have a distorted tetrahedral-type configuration to relieve steric crowding with a Ge—Ni—P angle of 100·7 (7)° and with C(3), C(4), C(5) being considered as a π-allyl system and the rather short C(1)—C(2) bond as a double bond. Although the C_5H_5 ring in $(C_5H_5)_2Ni$ is irregular, the rings still maintain planarity (Seiler & Dunitz, 1980).

The Ge—Cl bonds have an average length of 2·189 (3) Å and, whereas Ge—Cl(2) and Ge—Cl(3) are the same within experimental error, Ge—Cl(1) is shorter. The angles at Ge are distorted, the Cl—Ge—Cl angles varying from 98·6 (1) to 100·9 (1)° and the Ni—Ge—Cl angles from 112·7 (1) to 125·4 (1)°. In all cases the length and angles involving Cl(1) deviate the most and this is the atom which is almost coplanar with P, Ge and Ni and is located opposite to the C_5H_5 ring.

The Ni—Ge bond length [2·248 (1) Å] is shorter than those in the only other two compounds for which this is known [2·308 (3) and 2·329 (4) Å] (Titova *et al.*, 1980; Zakharov *et al.*, 1980) and the sum of the covalent radii (2·61 Å) (Pauling, 1960). This short bond length, the small Cl—Ge—Cl angles and the closeness of the Ge—Cl distance to the sum of the covalent radii (2·21 Å) (Pauling, 1960) suggest appreciable d_π–d_π bonding between Ni and Ge rather than d_π–p_π bonding with the Cl atoms.

The benzene molecule lies on the twofold axis located at 0, y, $\frac{1}{4}$. The surrounding groups are phenyl(III) and the C_5H_5 ring.* There is no evidence for disorder, but the thermal parameters are large and the refinement of the atomic parameters was slow, which could explain the rather short C(24)—C(25) bond length of 1·30 (2) Å.

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* A packing diagram has been deposited. See deposit footnote.

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Carbonyldihydrido(trimethylgermyl)bis(triphenylphosphine)iridium(III) Hemibenzene Solvate, $C_{40}H_{41}GeIrOP_2.\frac{1}{2}C_6H_6$

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Abstract. $M_r = 903·5$, monoclinic, $P2_1/c$, $a = 13·63\,(1)$, $b = 11·044\,(5)$, $c = 26·07\,(1)$ Å, $\beta = 92·52\,(3)°$, $U = 3920·5$ Å^3, $Z = 4$, $D_m = 1·54\,(2)$, $D_x = 1·53$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·7107$ Å, $\mu = 4·10$ mm^{-1}, $F(000) = 1796$, $T = 295$ K, $R = 0·052$ for 3560 observed reflections. The structure of $H_2(Me_3Ge)CO(Ph_3P)_2Ir$, isolated as a benzene solvate, has distorted octahedral coordination round iridium. The two phosphine groups are *cis* to each other, one being *trans* to a hydride hydrogen atom and the other *trans* to the germyl group.

Introduction. Addition of R_3GeH (R = Me, Et) to *trans*-$(Ph_3P)_2CO(Cl)Ir$ unexpectedly results in the slow formation of the six-coordinate dihydrido complexes $H_2(R_3Ge)CO(Ph_3P)_2Ir$ (Glockling & Wilbey, 1970). This contrasts markedly with analogous hydrosilation (Chalk & Harrod, 1965; Hazeldine, Parish & Parry, 1967) and hydrostannation (Lappert & Travers, 1968) where addition of R_3MH results in the formation of monohydrido complexes $H(R_3M)CO(Cl)(Ph_3P)_2Ir$ (M = Si or Sn; R = alkyl, alkoxy or halide).

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The ^{1}H NMR spectrum unambiguously defines the spatial relationship of the two H and two P atoms bonded to Ir, and its IR spectrum allows tentative conclusions to be drawn concerning the positions of the R_3Ge and CO ligands. We have determined the structure by X-ray analysis of one of these compounds, $H_2(Me_3Ge)CO(Ph_3P)_2Ir$, isolated as the benzene solvate. A preliminary report on this structure, which is the first structural determination of a compound containing an Ir—Ge bond, has been published (Schneider & Shearer, 1969).

Experimental. Prepared by reaction of trimethylgermane with *trans*-(CO)Cl$(Ph_3P)_2$Ir for two weeks at 293 K; buff-coloured solid, isolated from the reaction, formed colourless crystals when recrystallized from benzene (Glockling & Wilbey, 1970); crystal 0·40 × 0·50 × 0·09 mm with well developed (001) faces, D_m by flotation in iodobenzene/bromobenzene, unit-cell dimensions from precession photographs with the crystal mounted about **b*** from the 0*kl* and *hk*0 nets, Hilger & Watts four-circle diffractometer, θ–2θ scan of 60 steps (0·01°/step), 1·5 s per step; 3 standard reflections every 50 reflections; two shells of data collected, 0–15° measuring one half of the sphere of reflection and 15–20° measuring one quarter of the sphere; 5763 independent reflections measured, 3560 $[I \geq 3\sigma(I)]$ corrected for Lorentz and polarization effects, *h* 0–13, *k* 0–12, *l* –27–28; structure solved by heavy-atom method, three peaks with heights > 3 e Å^{-3}, located round the centre of symmetry at $(\frac{1}{2},\frac{1}{2},0)$, identified as half of a benzene molecule; H atoms not considered, parameters refined by least squares using block-diagonal approximation; scattering factors and anomalous-scattering corrections for the heavy atoms from *International Tables for X-ray Crystallography* (1962); in the final cycle of refinement, with anisotropic thermal parameters, parameter shifts all $<0{\cdot}33\sigma$, $R = 0{\cdot}052$; $w = 1{\cdot}0/(300{\cdot}0 + |F_o| + 0{\cdot}003|F_o|^2 + 0{\cdot}0003|F_o|^3)$, $wR = 0{\cdot}055$, function minimized $\sum(w\Delta^2)$ where $\Delta = |F_o| - |F_c|$, residual electron density within $-1{\cdot}3$ and $+0{\cdot}7$ e Å^{-3}; computer programs supplied by Dr F. R. Ahmed and his associates.

Table 1. *Fractional positional parameters* (Ir, Ge $\times 10^5$; O, P, C $\times 10^4$) *and equivalent isotropic thermal parameters with e.s.d.'s in parentheses*

	x	y	z	B_{eq}(Å^2)*
Ir	20424 (4)	12455 (5)	11673 (2)	3·05 (2)
Ge	31056 (13)	–3637 (17)	8249 (8)	5·30 (9)
O	2325 (9)	2854 (13)	252 (4)	6·4 (7)
P(1)	486 (3)	1941 (4)	1390 (2)	3·6 (2)
P(2)	3223 (3)	2115 (3)	1750 (2)	3·2 (2)
C(1)	2225 (11)	2272 (16)	617 (6)	4·5 (8)
C(2)	4235 (13)	–983 (14)	1259 (9)	7·1 (11)
C(3)	2299 (15)	–1853 (17)	679 (9)	7·3 (11)
C(4)	3649 (14)	69 (21)	156 (8)	7·5 (12)
C(5)	221 (10)	2206 (13)	2061 (6)	3·6 (7)
C(6)	–475 (12)	3085 (15)	2202 (7)	5·2 (9)
C(7)	–653 (14)	3242 (16)	2732 (8)	5·8 (10)
C(8)	–135 (12)	2549 (15)	3105 (7)	4·9 (8)
C(9)	561 (12)	1694 (15)	2952 (6)	4·5 (8)
C(10)	748 (11)	1544 (15)	2434 (6)	4·4 (8)
C(11)	–458 (105)	801 (15)	1200 (6)	4·4 (8)
C(12)	–552 (13)	487 (23)	684 (7)	7·3 (11)
C(13)	–1230 (14)	–448 (25)	533 (9)	8·6 (14)
C(14)	–1788 (14)	–1002 (20)	902 (9)	7·0 (11)
C(15)	–1703 (12)	–655 (16)	1405 (7)	5·3 (9)
C(16)	–1051 (12)	277 (15)	1571 (7)	4·8 (8)
C(17)	17 (12)	3301 (16)	1068 (6)	4·8 (8)
C(18)	664 (13)	4280 (15)	991 (6)	4·7 (8)
C(19)	352 (17)	5346 (19)	753 (7)	7·2 (11)
C(20)	–621 (18)	5480 (21)	579 (9)	8·5 (14)
C(21)	–1264 (15)	4576 (24)	650 (9)	9·0 (14)
C(22)	–974 (14)	3419 (22)	881 (9)	7·8 (12)
C(23)	3116 (10)	3760 (14)	1852 (6)	3·4 (7)
C(24)	2249 (11)	4195 (13)	2058 (6)	4·1 (7)
C(25)	2098 (12)	5441 (16)	2114 (7)	5·0 (8)
C(26)	2810 (13)	6271 (16)	1977 (7)	5·4 (9)
C(27)	3696 (14)	5824 (16)	1777 (8)	6·0 (10)
C(28)	3848 (12)	4568 (15)	1726 (7)	4·9 (8)
C(29)	4501 (10)	2000 (12)	1556 (6)	3·4 (7)
C(30)	5252 (11)	1560 (15)	1898 (7)	4·8 (8)
C(31)	6205 (12)	1443 (15)	1712 (7)	4·8 (8)
C(32)	6406 (11)	1763 (15)	1215 (6)	4·4 (8)
C(33)	5675 (11)	2240 (16)	874 (6)	4·6 (8)
C(34)	4701 (11)	2354 (14)	1060 (6)	4·0 (7)
C(35)	3293 (10)	1509 (15)	2398 (6)	4·0 (7)
C(36)	3140 (12)	252 (15)	2478 (6)	4·7 (8)
C(37)	3217 (13)	–221 (19)	2974 (7)	6·2 (10)
C(38)	3431 (12)	513 (19)	3396 (7)	6·0 (10)
C(39)	3596 (12)	1752 (19)	3317 (6)	5·5 (9)
C(40)	3533 (11)	2237 (17)	2819 (6)	4·4 (8)
C(41)	4251 (17)	4207 (23)	–169 (9)	8·2 (13)
C(42)	5242 (20)	4175 (22)	–374 (9)	8·7 (14)
C(43)	5965 (17)	4986 (21)	–181 (8)	7·7 (12)

* B_{eq} is defined according to Willis & Pryor (1975).

Discussion. The atomic parameters are given in Table 1† and bond lengths and angles in Table 2. The crystallographic study confirmed the structure deduced from the NMR spectra (Glockling & Wilbey, 1970) with Ir in a distorted octahedral environment. One phosphine ligand is *trans* to the germyl group and the other *cis* to the CO group so that the two hydride H atoms must be *cis* to each other although their positions were not established (Fig. 1). The resulting coordination polyhedron about Ir is highly distorted, the extent and nature of which appear to reflect the differing steric demands of the bulky triphenylphosphine and trimethylgermyl groups and that of the hydride ligands. Most noteworthy of the angular distortions is that found for the P(1)—Ir—Ge grouping [150·3 (1)°] and may be attributed to the relief of steric strain between the *cis* triphenylphosphine ligands. The two Ir—P bond distances are not significantly different at the 1% probability level and thus the *trans* influence of H and $GeMe_3$ is similar.

Ir, C(1) and O are almost collinear and the C—O bond length 1·16 (2) Å is one of the longest found in Ir carbonyl complexes which range from 1·05 Å (McGinnety, Doedens & Ibers, 1967) to 1·165 (24) Å (Manojlović-Muir, Muir & Ibers, 1969). The Ir—C(1) bond length, 1·85 (2) Å, is one of the shortest found, *cf.*

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† Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39102 (23 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

1·94 (1) Å in $[IrH(CO)_2(PPh_3)_2]_2SO_2$ (Angoletta, Bellon, Manassero & Sansoni, 1974) and this together with the long C—O bond indicates that there may be strong back donation from the Ir to the CO group.

The Ge—Ir bond length, 2·484 (2) Å, appears to be the first reported and can only be compared with the sum of the covalent radii, 2·56 Å (Pauling, 1960); it appears to be consistent with some partial double bonding arising from d_π–d_π interactions between Ge and Ir. The Ir—P distances 2·353 (4), 2·367 (4) Å are longer than those found in other hydridoiridium complexes with phosphine ligands (Angoletta *et al.*, 1974; Bird, Harrod & Than, 1974; Immirzi, Musco, Pregosin & Venanzi, 1980).

The Ge—C bond lengths, average 1·99 (2) Å, are in good agreement with other values reported, such as the value of 1·945 (3) Å in $GeMe_4$ (Hencher & Mustoe, 1975). The C—Ge—C angles are similar and have an average value of 104·6 (6)°, significantly less than the tetrahedral value, due to multiple bonding between Ge and Ir, similar to the effect found for Cl—Ge—Cl angles in $(\eta^5\text{-}C_5H_5)(Ph_3P)(GeCl_3)Ni.\frac{1}{2}C_6H_6$ (Bell, Glockling, McGregor, Schneider & Shearer, 1984).

The unit cell contains, in addition to the complex, two molecules of benzene, the solvent from which the crystals were grown. Thus, in the crystalline state the overall formula is $H_2(Me_3Ge)CO(Ph_3P)_2Ir.\frac{1}{2}C_6H_6$. The two benzene solvate molecules are located on centres of symmetry (Fig. 2) and, although the large temperature factors indicate considerable thermal motion, they do not appear to be disordered.

Table 2. *Bond distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Symmetry code: (i) $1-x$, $1-y$, $-z$

Ir—Ge	2·484 (2)	C(17)—C(18)	1·42 (2)
Ir—P(1)	2·353 (4)	C(18)—C(19)	1·39 (3)
Ir—P(2)	2·367 (4)	C(19)—C(20)	1·39 (3)
Ir—C(1)	1·85 (2)	C(20)—C(21)	1·35 (3)
Ge—C(2)	1·99 (2)	C(21)—C(22)	1·46 (3)
Ge—C(3)	2·01 (2)	C(22)—C(17)	1·42 (3)
Ge—C(4)	1·98 (2)	C(23)—C(24)	1·41 (2)
C(1)—O	1·16 (2)	C(24)—C(25)	1·40 (3)
P(1)—C(5)	1·82 (2)	C(25)—C(26)	1·39 (2)
P(1)—C(11)	1·85 (2)	C(26)—C(27)	1·42 (3)
P(1)—C(17)	1·82 (2)	C(27)—C(28)	1·41 (2)
P(2)—C(23)	1·84 (2)	C(28)—C(23)	1·39 (2)
P(2)—C(29)	1·84 (1)	C(29)—C(30)	1·41 (2)
P(2)—C(35)	1·82 (2)	C(30)—C(31)	1·41 (2)
C(5)—C(6)	1·42 (2)	C(31)—C(32)	1·38 (2)
C(6)—C(7)	1·42 (3)	C(32)—C(33)	1·41 (2)
C(7)—C(8)	1·40 (3)	C(33)—C(34)	1·44 (2)
C(8)—C(9)	1·41 (2)	C(34)—C(29)	1·39 (2)
C(9)—C(10)	1·40 (2)	C(35)—C(36)	1·42 (2)
C(10)—C(5)	1·39 (2)	C(36)—C(37)	1·40 (3)
C(11)—C(12)	1·39 (3)	C(37)—C(38)	1·39 (3)
C(12)—C(13)	1·43 (3)	C(38)—C(39)	1·40 (3)
C(13)—C(14)	1·39 (3)	C(39)—C(40)	1·40 (2)
C(14)—C(15)	1·37 (3)	C(40)—C(35)	1·39 (2)
C(15)—C(16)	1·42 (2)	C(41)—C(42)	1·48 (3)
C(16)—C(11)	1·41 (2)	C(42)—C(43)	1·41 (3)
		C(43)—C(41$^{\text{i}}$)	1·32 (3)

Ge—Ir—P(1)	150·3 (1)	P(1)—C(17)—C(18)	119 (1)
Ge—Ir—P(2)	97·5 (1)	P(1)—C(17)—C(22)	123 (1)
Ge—Ir—C(1)	93·4 (5)	C(22)—C(17)—C(18)	118 (2)
P(1)—Ir—P(2)	107·7 (1)	C(17)—C(18)—C(19)	122 (2)
P(1)—Ir—C(1)	98·3 (5)	C(18)—C(19)—C(20)	120 (2)
P(2)—Ir—C(1)	97·9 (5)	C(19)—C(20)—C(21)	120 (2)
C(2)—Ge—C(3)	103·3 (8)	C(20)—C(21)—C(22)	123 (2)
C(2)—Ge—C(4)	105·8 (9)	C(21)—C(22)—C(17)	117 (2)
C(3)—Ge—C(4)	104·7 (9)	P(2)—C(23)—C(24)	118 (1)
Ir—Ge—C(2)	119·4 (6)	P(2)—C(23)—C(28)	122 (1)
Ir—Ge—C(3)	109·4 (6)	C(28)—C(23)—C(24)	120 (1)
Ir—Ge—C(4)	112·9 (6)	C(23)—C(24)—C(25)	120 (1)
Ir—C(1)—O	176 (1)	C(24)—C(25)—C(26)	121 (2)
C(5)—P(1)—C(11)	101·7 (7)	C(25)—C(26)—C(27)	119 (2)
C(5)—P(1)—C(17)	103·3 (7)	C(26)—C(27)—C(28)	120 (2)
C(11)—P(1)—C(17)	102·2 (7)	C(27)—C(28)—C(23)	120 (2)
C(23)—P(2)—C(29)	100·9 (7)	P(2)—C(29)—C(30)	121 (1)
C(23)—P(2)—C(35)	103·3 (7)	P(2)—C(29)—C(34)	118 (1)
C(29)—P(2)—C(35)	102·6 (7)	C(34)—C(29)—C(30)	121 (1)
Ir—P(1)—C(5)	120·5 (5)	C(29)—C(30)—C(31)	118 (1)
Ir—P(1)—C(11)	109·6 (5)	C(30)—C(31)—C(32)	121 (1)
Ir—P(1)—C(17)	117·2 (6)	C(31)—C(32)—C(33)	122 (1)
Ir—P(2)—C(23)	115·9 (5)	C(32)—C(33)—C(34)	117 (1)
Ir—P(2)—C(29)	114·9 (5)	C(33)—C(34)—C(29)	121 (1)
Ir—P(2)—C(35)	117·0 (5)	P(2)—C(35)—C(36)	120 (1)
P(1)—C(5)—C(6)	121 (1)	P(2)—C(35)—C(40)	122 (1)
P(1)—C(5)—C(10)	118 (1)	C(40)—C(35)—C(36)	119 (1)
C(10)—C(5)—C(6)	121 (1)	C(35)—C(36)—C(37)	120 (1)
C(5)—C(6)—C(7)	119 (1)	C(36)—C(37)—C(38)	121 (2)
C(6)—C(7)—C(8)	120 (2)	C(37)—C(38)—C(39)	119 (2)
C(7)—C(8)—C(9)	120 (2)	C(38)—C(39)—C(40)	120 (2)
C(8)—C(9)—C(10)	121 (2)	C(39)—C(40)—C(35)	121 (2)
C(9)—C(10)—C(5)	120 (1)	C(43$^{\text{i}}$)—C(41)—C(42)	120 (2)
P(1)—C(11)—C(12)	118 (1)	C(41)—C(42)—C(43)	119 (2)
P(1)—C(11)—C(16)	120 (1)	C(42)—C(43)—C(41$^{\text{i}}$)	120 (2)
C(16)—C(11)—C(12)	122 (2)		
C(11)—C(12)—C(13)	119 (2)		
C(12)—C(13)—C(14)	120 (2)		
C(13)—C(14)—C(15)	121 (2)		
C(14)—C(15)—C(16)	122 (2)		
C(15)—C(16)—C(11)	117 (2)		

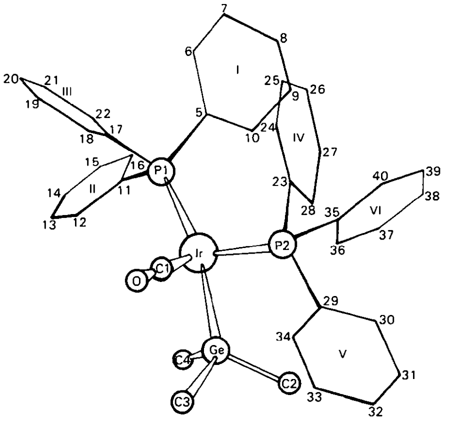

Fig. 1. Structure of $H_2(Me_3Ge)CO(Ph_3P)_2Ir$.

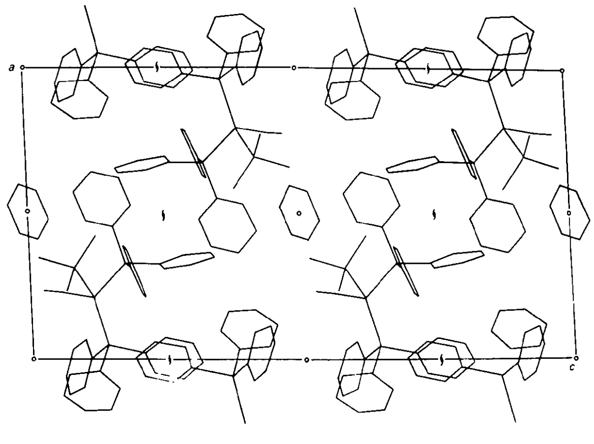

Fig. 2. Projection on the (010) plane.

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Bis(morpholine)aluminium Trichloride, $[Al(C_4H_9NO)_2]Cl_3$

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Abstract. $M_r = 307\cdot585$, triclinic, $P\bar{1}$, $a = 7\cdot3110\,(5)$, $b = 8\cdot0598\,(5)$, $c = 12\cdot8353\,(4)$ Å, $\alpha = 80\cdot583\,(3)$, $\beta = 89\cdot959\,(3)$, $\gamma = 71\cdot148\,(4)°$, $V = 705\cdot005$ Å^3, $Z = 2$, $D_x = 1\cdot449$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1\cdot54178$ Å (Ni filter), $\mu = 65\cdot64$ cm^{-1}, $F(000) = 320$, $T = 294$ K. Final $R = 0\cdot059$ for 2632 unique observed reflections. The structure consists of discrete molecules, all intermolecular distances being longer than 2·8 Å. The coordination geometry around aluminium is slightly distorted trigonal bipyramidal with chlorine in equatorial and nitrogen in axial positions. The Al—N bond lengths are 2·093 (3) and 2·064 (3) Å, Al—Cl 2·173 (1), 2·197 (1) and 2·181 (1) Å.

Introduction. Alkylaluminium halides, in conjunction with Group V*b* Lewis bases, notably phosphines, play an important role as cocatalysts in the oligomerization of olefins catalysed by nickel compounds (Wilke, 1979; Bogdanovic, Spliethoff & Wilke, 1980; Brandes, Goddard, Jolly, Krüger, Mynott & Wilke, 1983). Recent results indicate that morpholine, which is a cofactor in nickel-based catalytic systems even in the absence of aluminium alkyls (Heimbach & Schenkluhn, 1980), may be important in controlling the stereochemical course and product distribution of olefin oligomerizations with homogeneous nickel/aluminium catalysts (Heimbach & Sturm, 1983, unpublished observations). In order to gain more insight into the nature of the active species P. Heimbach and H. Sturm reacted $Al(C_2H_5)_3$ with an excess of morpholine and obtained a material which was tentatively formulated as the 1 : 1 morpholine adduct of aluminium tris(morpholide). Its very low solubility in common solvents indicates a polymeric structure with morpholine acting as a bridging ligand with both of its heteroatoms as donor functions. Unfortunately, no single crystals suitable for X-ray diffraction could be obtained due to the insolubility of the compound. However, the observation that the compound reacted slowly with $CHCl_3$ led to the isolation of a 2 : 1 adduct of morpholine with aluminium trichloride whose structure was determined subsequently to establish the mode of bonding exhibited by morpholine in such complexes.

Experimental. Plate-like crystals (0·5 × 0·4 × 0·2 mm) grown from chloroform; Enraf–Nonius CAD-4 diffractometer; cell dimensions obtained by least squares from setting angles of 75 reflections ($18\cdot86 \leq \theta \leq 64\cdot22°$); 2988 measured reflections (θ–2θ scans, $5\cdot89 \leq \theta \leq 76\cdot48°$, $-10 \leq h \leq 10$, $-12 \leq k \leq 12$, $-17 \leq l \leq 17$) merged to give 2970 unique reflections ($R_{\text{int}} = 0\cdot019$) of which 338 unobserved [$I < 2\cdot0\,\sigma(I)$]; orientation (every 250 reflections) and intensity control (every 4000 s of X-ray exposure) by means of three standard reflections ($2\bar{3}\bar{2}$, $0\bar{1}\bar{5}$, $\bar{2}\bar{3}\bar{2}$), no detectable decomposition;

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