Bond lengths and angles are comparable to those in other silicon–nitrogen compounds and in Si-substituted acetylenes (Zeil, Haase & Dakkouri, 1969). The Bu groups are well-spaced around the molecule, with a staggered arrangement about the Si···Si line (as required by the crystallographic symmetry). Their steric bulk produces deviations of several degrees in the bond angles for Si from the ideal tetrahedral value of 109·5°.

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Structure of 2′-Acetyl-2′-(*p*-chlorobenzoyl)-2-(*p*-methoxyphenyl)glyoxylohydrazide (ACMGH)

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Abstract. $C_{18}H_{15}ClN_2O_5$, M_r = 374·785, triclinic, $P\bar{1}$, a = 10·169 (1), b = 9·458 (1), c = 9·974 (1) Å, α = 102·23 (1), β = 110·89 (1), γ = 87·68 (1)°, Z = 2, V = 916·15 Å^3, λ(Mo $K\alpha$) = 0·71069 Å, F(000) = 388, D_c = 1·36, D_m = 1·37 Mg m^{-3} (by flotation, KBr solution), m.p. 390–392 K, μ = 0·247 mm^{-1}; 1477 independent non-zero reflexions. The structure was solved by direct phase determination with *MULTAN*. The positional and vibrational parameters, with anisotropic temperature factors for the non-hydrogen atoms, were refined by full-matrix least-squares calculations to a final R = 0·051. The compound is an open-chain hydrazine derivative, with two characteristic planar conjugated systems, forming a small angle (13·5°) with each other.

Introduction. In the course of a study on the oxidation of bis(aroyl)hydrazones of α-dicarbonyl compounds a number of oxidation products of 1-arylglyoxal-2-aroylhydrazones with lead tetraacetate have been prepared at the Laboratory of Organic Chemistry of the Aristotle University of Thessaloniki (Rodios, 1976; Rodios & Alexandrou, 1980).

Since chemical and spectroscopic data were inconclusive as to whether these compounds contained an open chain or a heterocyclic ring, an X-ray structure analysis was considered necessary and to this end the crystal structure determination of ACMGH was carried out.

Pure, colourless crystals of ACMGH were kindly provided by Professor N. E. Alexandrou and Dr N. Rodios. A transparent prismatic single crystal, with dimensions 0·15 × 0·20 × 0·30 mm, was selected and centred on our computer-controlled Philips PW 1100 four-circle single-crystal diffractometer. With Mo $K\alpha$ radiation (λ = 0·71069 Å) the cell constants were determined by measuring the θ angles of 180 strong reflexions with large θ values directly on the diffractometer and subsequently processing them with the least-squares program *PARAM* [part of the XRAY system of crystallographic programs (Stewart, Kruger, Ammon, Dickinson & Hall, 1976)]. Intensity statistics indicated the space group $P\bar{1}$, which was confirmed by the final structure.

Three-dimensional intensity data were collected with a scintillation counter in the ω-scan mode, using Mo $K\alpha$ radiation and a graphite monochromator. The intensities of 2221 independent reflexions up to 2θ = 42° (max. hkl = 11, ±10, ±10 respectively) were examined and measured. Of these, 1477 with intensities

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greater than 2σ were considered as observed and included in all subsequent computations.

Integrated intensities were converted to $|F_o|$ values in the usual way, using the special measurement-treatment program *DATRED* (Main, 1970). Since μ is very small, no absorption correction was applied. For further calculations the programs of the *MULTAN* (Main, Woolfson, Lessinger, Germain & Declercq, 1974) and XRAY systems were used.

The structure was essentially solved by direct phase determination with the *MULTAN* system. The phases of 185 strong reflexions were determined and on the resulting E map it was possible to locate all the 26 non-hydrogen atoms of the asymmetric unit. A structure factor calculation with the 185 reflexions and an overall $B = 3{\cdot}44$ Å^2, gave $R = 0{\cdot}157$.

Refinement of the structure was carried out by full-matrix least-squares calculations with the XRAY system, using unit weights. The atomic scattering factors for Cl, O, N and C were taken from Cromer & Waber (1965), and for H from Stewart, Davidson & Simpson (1965). A single scale factor was used for the whole set of reflexion data.

After several cycles of isotropic and anisotropic refinement R reduced to 0·076. The H atoms were then located by a difference Fourier synthesis and included in three further refinement cycles with individual isotropic temperature factors equal to those of the C atoms to which they are bonded. The R factor reduced to 0·051. The average shift/error ratio of the last cycle is less than 0·18.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors for the non-hydrogen atoms in ACMGH*

Here and throughout this paper the e.s.d.'s are given in parentheses and refer to the last digit.

	x	y	z	B_{eq} (Å^2)*
Cl	3018 (2)	4327 (2)	4230 (2)	4·9
O(1)	1319 (4)	1278 (4)	5143 (4)	4·5
O(2)	9986 (4)	2326 (4)	1117 (4)	4·1
O(3)	3511 (4)	1011 (4)	2794 (4)	4·0
O(4)	2304 (4)	9874 (4)	9241 (4)	4·3
O(5)	7388 (4)	5638 (5)	940 (4)	5·0
N(1)	801 (4)	787 (4)	2649 (4)	2·9
N(2)	1322 (4)	9992 (4)	1623 (4)	2·9
C(1)	1272 (5)	425 (6)	4049 (6)	3·2
C(2)	1699 (5)	8895 (6)	4050 (5)	3·0
C(3)	2994 (6)	8625 (6)	5037 (6)	3·3
C(4)	3433 (6)	7232 (6)	5102 (6)	3·6
C(5)	2524 (6)	6105 (6)	4179 (6)	3·5
C(6)	1197 (6)	6347 (6)	3199 (6)	3·6
C(7)	802 (5)	7742 (6)	3134 (5)	3·1
C(8)	86 (5)	2040 (5)	2286 (6)	3·1
C(9)	9411 (6)	2877 (7)	3282 (7)	4·5
C(10)	2649 (6)	233 (6)	1747 (6)	3·4
C(11)	2997 (5)	9507 (6)	382 (6)	3·2
C(12)	4131 (5)	8495 (5)	524 (6)	3·0
C(13)	4544 (6)	7953 (7)	9334 (6)	3·7
C(14)	5613 (6)	7000 (7)	9411 (7)	4·0
C(15)	6310 (6)	6558 (6)	730 (6)	3·6
C(16)	5894 (7)	7074 (7)	1922 (7)	4·5
C(17)	4825 (7)	8023 (7)	1837 (7)	3·9
C(18)	7859 (8)	5089 (8)	9754 (8)	5·1

* $B_{eq} = \frac{8}{3}\pi^2$ trace $\tilde{\mathbf{U}}$.

Table 2. *Atomic coordinates* ($\times 10^3$) *and isotropic temperature factors for the hydrogen atoms in ACMGH*

	x	y	z	B (Å^2)
H(N2)	77 (5)	947 (5)	88 (5)	3·4 (1·2)
H(C4)	431 (6)	702 (6)	571 (6)	4·9 (1·4)
H(C3)	366 (5)	933 (5)	570 (5)	3·2 (1·1)
H(C6)	55 (5)	551 (5)	253 (5)	3·9 (1·2)
H(C7)	994 (5)	798 (5)	250 (5)	2·5 (1·0)
H1(C9)	871 (7)	339 (7)	280 (7)	6·7 (1·9)
H2(C9)	891 (6)	222 (6)	366 (6)	5·7 (1·4)
H3(C9)	9 (7)	347 (7)	406 (7)	5·8 (1·5)
H(C13)	405 (6)	830 (6)	840 (6)	4·3 (1·4)
H(C14)	588 (7)	664 (8)	852 (7)	7·1 (1·9)
H(C16)	632 (6)	677 (7)	273 (6)	5·1 (1·5)
H(C17)	461 (6)	840 (6)	268 (6)	4·3 (1·5)
H1(C18)	861 (9)	433 (9)	6 (9)	10·1 (2·5)
H2(C18)	707 (8)	456 (8)	887 (8)	7·2 (2·0)
H3(C18)	819 (6)	593 (7)	948 (6)	5·4 (1·5)

Table 3. *Interatomic distances* (Å) *in ACMGH*

N(1)–C(1)	1·417 (7)	C(3)–C(4)	1·381 (8)
N(1)–C(8)	1·411 (7)	C(4)–C(5)	1·377 (7)
N(1)–N(2)	1·386 (6)	C(5)–C(6)	1·399 (7)
N(2)–C(10)	1·335 (8)	C(5)–Cl	1·741 (6)
C(1)–C(2)	1·494 (8)	C(6)–C(7)	1·371 (8)
C(1)–O(1)	1·199 (7)	C(12)–C(13)	1·388 (9)
C(8)–O(2)	1·221 (8)	C(12)–C(17)	1·406 (8)
C(8)–C(9)	1·481 (9)	C(13)–C(14)	1·375 (9)
C(10)–O(3)	1·214 (6)	C(14)–C(15)	1·397 (9)
C(10)–C(11)	1·542 (8)	C(15)–C(16)	1·387 (10)
C(11)–O(4)	1·220 (6)	C(16)–C(17)	1·371 (10)
C(11)–C(12)	1·455 (8)	C(15)–O(5)	1·356 (7)
C(2)–C(3)	1·386 (7)	C(18)–O(5)	1·423 (10)
C(2)–C(7)	1·386 (7)		

Table 4. *Bond angles* (°) *in ACMGH*

C(1)–N(1)–N(2)	116·7 (0·4)	C(3)–C(2)–C(7)	119·4 (0·5)
C(1)–N(1)–C(8)	126·2 (0·4)	C(2)–C(3)–C(4)	121·6 (0·4)
C(8)–N(1)–N(2)	115·9 (0·4)	C(3)–C(4)–C(5)	117·9 (0·5)
C(2)–C(1)–O(1)	122·8 (0·5)	C(4)–C(5)–C(6)	121·6 (0·5)
C(2)–C(1)–N(1)	114·6 (0·4)	C(5)–C(6)–C(7)	119·2 (0·5)
N(1)–C(1)–O(1)	112·6 (0·5)	C(6)–C(7)–C(2)	120·2 (0·4)
C(1)–C(2)–C(3)	119·2 (0·4)	C(4)–C(5)–Cl	119·7 (0·4)
C(1)–C(2)–C(7)	121·4 (0·4)	C(6)–C(5)–Cl	118·6 (0·4)
C(9)–C(8)–N(1)	118·6 (0·5)		
C(9)–C(8)–O(2)	123·5 (0·5)	C(13)–C(12)–C(17)	118·1 (0·5)
N(1)–C(8)–O(2)	117·7 (0·5)	C(12)–C(13)–C(14)	122·3 (0·5)
N(1)–N(2)–C(10)	119·8 (0·4)	C(13)–C(14)–C(15)	119·6 (0·6)
C(11)–C(10)–N(2)	114·1 (0·4)	C(14)–C(15)–C(16)	119·3 (0·6)
N(2)–C(10)–O(3)	124·4 (0·6)	C(15)–C(16)–C(17)	121·3 (0·6)
O(3)–C(10)–C(11)	121·4 (0·6)	C(12)–C(17)–C(16)	119·9 (0·6)
C(10)–C(11)–C(12)	119·7 (0·5)	C(14)–C(15)–O(5)	124·2 (0·6)
C(10)–C(11)–O(4)	115·9 (0·5)	C(16)–C(15)–O(5)	116·4 (0·5)
C(12)–C(11)–O(4)	124·3 (0·5)	C(15)–O(5)–C(18)	118·5 (0·5)
C(11)–C(12)–C(13)	119·9 (0·5)		
C(11)–C(12)–C(17)	122·0 (0·6)		

The final positional parameters and equivalent isotropic temperature coefficients for the non-hydrogen atoms (Willis & Pryor, 1975) are given in Table 1, for H atoms in Table 2. Interatomic distances and bond angles are given in Tables 3 and 4.[*]

Discussion. The structural and conformational features of the ACMGH molecule are shown in the projection of Fig. 1. Clearly, the compound is not heterocyclic but an open-chain hydrazine derivative.

The characteristic feature of the molecule is the almost completely coplanar conjugated system formed by the methoxyphenyl group with the attached carbonyl of the glyoxyloyl group (details of least-squares planes have been deposited). The two CO groups of the latter adopt an *anti* orientation.

A second coplanar conjugated system is formed by the two N atoms of the glyoxyloyl group with the C atoms of the acetyl and benzoyl groups attached to N(1). The two conjugated systems form with each other a small angle of 13·5°.

The CO of the *p*-chlorobenzoyl group is at an *anti* position with the CO of the acetyl group and lies completely out of the benzene plane *B*1 (Fig. 1), forming with it an angle of 67·9°. This unfavoured conformation may be attributed to van der Waals or steric interactions between the two neighbouring carbonyl groups containing the O(1) and O(3) atoms.

The geometrical features of the benzene rings *B*1 and *B*2 are in good agreement with the usually accepted values. Mean bond lengths of the rings are 1·383 and 1·387 Å respectively. The two rings form with each other an angle of 70°.

The intermolecular contact distances O(2)···H(N2) = 2·77 (5), O(4)···H(N2) = 2·72 (6), O(1)···H1(C9) = 2·60 (5), O(1)···H2(C9) = 2·61 (6), and O(2)···H3(C9) = 2·50 (8) Å are comparable with the sum (2·48–2·72 Å) of the van der Waals radii of the corresponding atoms (Bondi, 1964). All other intramolecular contact distances are within expected values.

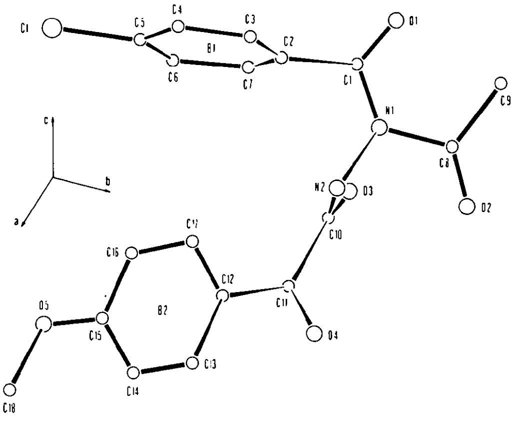

Fig. 1. Clinographic projection of the ACMGH molecule.

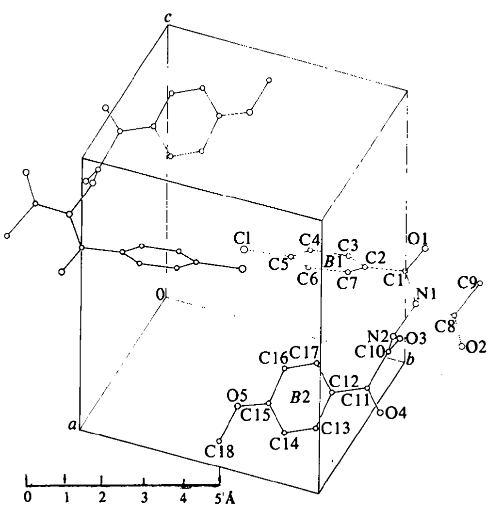

Fig. 2. Clinographic projection of ACMGH showing the molecular packing in the unit cell.

Fig. 2 is a clinographic projection of the structure showing the molecular packing of ACMGH in the unit cell. The various intermolecular distances are normal.

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[*] Lists of structure factors, anisotropic thermal parameters of the non-hydrogen atoms, bond distances and angles involving H atoms and various least-squares planes of the ACMGH molecule have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35771 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.