Fig. 2. A stereoscopic view of the packing for compound (I).

The torsion angles indicate that the pyrazole ring in (I) is planar. The conformation of the dihydropyrimidine ring can be described as a distorted sofa with the C(5) atom displaced most from the plane of the ring [puckering coordinates (Zefirov, Palyulin & Dashevskaya, 1990) are: $S = 0.56$, $\varphi = 27.4°$, $\theta = 45.4°$]. The trans substituents at C(5) and C(6) have an axial orientation. The N(3)—C(4)—C(7)—C(12) and N(2)—C(3)—N(3)—C(4) torsion angles are

0.3 (3) and 9.7 (5)°, respectively, indicating conjugation of the π systems of the C(7)—C(8)—C(9)—C(10)—C(11)—C(12) aromatic ring, azomethyne group and pyrazole ring.

A stereoscopic view of the unit cell is shown in Fig. 2. There are no intermolecular distances shorter than the sums of the van der Waals radii.

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Structure of N-[(4-Chlorophenyl)di(methoxy)methyl]acetamide

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Abstract. N-(4-Chloro-α,α-dimethoxybenzyl)acetamide, $C_{11}H_{14}ClNO_3$, $M_r = 243.69$, orthorhombic, $P2_12_12_1$, $a = 12.032$ (4), $b = 10.724$ (4), $c = 9.502$ (3) Å, $V = 1226$ (1) Å^3, $Z = 4$, $D_x = 1.320$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0.71069$ Å, $\mu = 0.301$ mm^{-1}, $F(000) = 512$, $T = 300$ K, final $R = 0.043$, $wR = 0.034$ for 939 unique reflections with $F > 2\sigma(F)$. The conformation is characterized by an

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Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* ($\mathring{A}^2$)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
Cl(1)	-0.3484 (1)	-0.1143 (1)	-0.1209 (1)	0.068 (1)
C(1)	-0.3705 (5)	0.0421 (4)	-0.0720 (6)	0.051 (3)
C(2)	-0.3093 (5)	0.0904 (4)	0.0370 (6)	0.058 (3)
C(3)	0.3294 (5)	0.2116 (4)	0.0762 (5)	0.052 (3)
C(4)	-0.4107 (4)	0.2822 (4)	0.0086 (5)	0.042 (3)
C(5)	-0.4690 (5)	0.2312 (4)	-0.0987 (6)	0.055 (3)
C(6)	-0.4502 (4)	0.1090 (4)	-0.1422 (6)	0.055 (3)
C(7)	-0.4341 (4)	0.4133 (4)	0.0672 (5)	0.044 (2)
O(1)	-0.4736 (3)	0.4032 (3)	0.2077 (4)	0.051 (2)
O(2)	-0.5105 (3)	0.4697 (2)	-0.0202 (4)	0.048 (2)
N(1)	-0.3342 (4)	0.4859 (4)	0.0861 (4)	0.047 (2)
C(8)	-0.5402 (6)	0.5950 (4)	0.0187 (6)	0.066 (3)
C(9)	-0.5767 (4)	0.3357 (5)	0.2227 (6)	0.064 (3)
O(3)	-0.2880 (4)	0.4959 (3)	-0.1435 (4)	0.076 (2)
C(10)	-0.2675 (5)	0.5213 (5)	-0.0198 (6)	0.053 (3)
C(11)	-0.1673 (5)	0.5957 (5)	0.0212 (6)	0.063 (3)

Table 2. *Bond distances* ($\mathring{A}$) *and bond angles* (°)

Cl(1)—C(1)	1.761 (5)	O(1)—C(7)	1.422 (6)
C(1)—C(2)	1.373 (7)	O(2)—C(7)	1.379 (6)
C(1)—C(6)	1.371 (7)	N(1)—C(7)	1.443 (7)
C(2)—C(3)	1.373 (6)	O(1)—C(9)	1.443 (6)
C(3)—C(4)	1.394 (7)	O(2)—C(8)	1.438 (5)
C(4)—C(5)	1.353 (7)	N(1)—C(10)	1.342 (7)
C(5)—C(6)	1.393 (6)	O(3)—C(10)	1.231 (7)
C(4)—C(7)	1.538 (6)	C(10)—C(11)	1.498 (7)
Cl(1)—C(1)—C(2)	118.5 (5)	O(2)—C(7)—C(4)	107.7 (4)
Cl(1)—C(1)—C(6)	118.4 (4)	O(2)—C(7)—O(1)	112.1 (4)
C(2)—C(1)—C(6)	123.0 (5)	N(1)—C(7)—C(4)	112.7 (4)
C(1)—C(2)—C(3)	117.8 (5)	N(1)—C(7)—O(1)	101.7 (4)
C(2)—C(3)—C(4)	120.9 (5)	N(1)—C(7)—O(2)	113.2 (4)
C(3)—C(4)—C(5)	119.5 (4)	C(7)—O(1)—C(9)	114.7 (4)
C(4)—C(5)—C(6)	121.3 (5)	C(7)—O(2)—C(8)	114.8 (4)
C(3)—C(4)—C(7)	117.3 (5)	C(7)—N(1)—C(10)	123.8 (5)
C(5)—C(4)—C(7)	123.2 (5)	N(1)—C(10)—O(3)	122.2 (5)
C(1)—C(6)—C(5)	117.5 (5)	N(1)—C(10)—C(11)	115.9 (5)
O(1)—C(7)—C(4)	109.3 (4)	O(3)—C(10)—C(11)	121.9 (6)

sp^3-hybridized C atom which is connected to the N atom of the acetamide group. The molecules are linked by N—H⋯O hydrogen bonds.

Introduction. The N-acyl imidates have been extensively studied (Bader, 1965; Kaddachi, Hajjem & Baccar, 1989; Matsuda, Tomiyoshi, Yanagiya & Matsumoto, 1988). Hajjem & Baccar (1993) have shown that addition of an alcohol to the C=N double bond produces N-[(dialkoxy)(aryl)methyl]-amides. The title compound was synthesized by the action of methanol on N-acetyl-p-chlorobenzimidate. Slow evaporation of the solution yielded white crystals.

Experimental. A prismatic white crystal with dimensions $0.37 \times 0.55 \times 0.04$ mm was used for X-ray measurements. The final cell parameters were determined from 61 reflections in the range $35.2 < 2\theta < 37.2°$. A total of 2638 reflections up to 45° in 2θ were measured from $\omega/2\theta$ scans on a Stoe Stadi-4

four-circle diffractometer using graphite-monochromated Mo $K\alpha$ radiation (h $0\rightarrow13$, k $-12\rightarrow12$, l $-10\rightarrow10$). 960 reflections were unique ($R_{int} = 0.034$), of which 939 with $F > 2\sigma(F)$ were used for structure refinement. Three standard reflections (212, 211 and 221) were measured every 150 min and the scaling of the intensities gave rise to a scale factor in the range 1.014–1.069. The intensities were corrected for Lp effects but not for absorption. At the end of the refinement an isotropic extinction correction with a coefficient $g = 2.2$ (3) $\times 10^{-3}$ was applied. The structure was solved with direct methods (*SHELXS*86; Sheldrick, 1986) and refined on F with *SHELX*76 (Sheldrick, 1976). Final $R = 0.043$, $wR = 0.034$ and $S = 1.07$; 159 parameters were refined; $w = 3.03/\sigma^2(F)$; $(\Delta/\sigma)_{max} < 0.54$ in the last cycle of refinement. All H atoms were located in the difference Fourier map. Apart from H(1) the final positions of H atoms were determined geometrically assuming C—H = 1.08 $\mathring{A}$. The isotropic thermal parameters of the H atoms were fixed at 0.05 $\mathring{A}^2$ for those on the phenyl ring, and for H(1), and at 0.075 $\mathring{A}^2$ for those on the methyl groups. In the final difference Fourier maps function values were between -0.19 and 0.20 e $\mathring{A}^{-3}$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed on VAX 8530 and IBM 3090 computers.

Discussion. The atomic coordinates are listed in Table 1* and bond lengths and angles in Table 2. The structure (Fig. 1) contains an almost linear

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55776 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: MU1001]
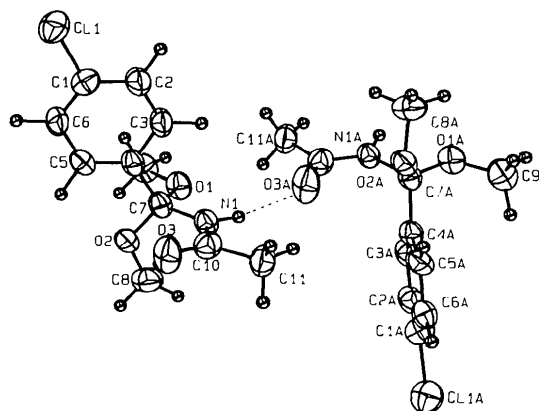

Fig. 1. General view of the molecule with intermolecular hydrogen bonds. Anisotropic ellipsoids are drawn on a 50% probability level; H atoms are represented by spheres of arbitrary radii.

intermolecular hydrogen bond between the amino N atom N(1) and the carbonyl O atom O(3) [N(1)⋯O(3) = 2.97 (1), O(3)⋯H(1) = 2.16, N(1)—H(1) = 0.85 Å, N(1)—H(1)⋯O(3) = 164°]. This type of hydrogen bond is observed in 4,4'-(3-acetamido-2-phenyl-2-propylidene)bisphenyl diacetate (Précigoux, Hospital, Miquel & Gilbert, 1985). The bond lengths in the acetamide group [N(1)—C(7) = 1.443 (7), N(1)—C(10) = 1.342 (7), C(10)—O(3) = 1.231 (7), C(10)—C(11) = 1.498 (7) Å] are very similar to those found by Précigoux *et al.* (1985). The bond angles around C(7) are in the range 102–113° and are compatible with sp^3 hybridization.

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Structure of 15-Phenyl-14-dehydrosparteine

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Abstract. $C_{21}H_{28}N_2$, $M_r = 308.47$, orthorhombic, $P2_12_12_1$, $a = 8.959$ (1), $b = 14.137$ (1), $c = 13.934$ (1) Å, $V = 1764.8$ (3) Å^3, $Z = 4$, $D_x = 1.161$ g cm^{-3}, $\lambda(\text{Cu }K\alpha) = 1.54178$ Å, $\mu = 4.80$ cm^{-1}, $F(000) = 672$, $T = 292$ K, final $R = 0.040$ for 1273 observed reflections and 208 refined parameters. The quinolizidine A/B and dehydroquinolizidine C/D moieties are in *trans* and *cis* configurations, respectively; rings A, B, C are all in chair conformations, ring D is intermediate between sofa and half-chair. This conformation of the molecule is stabilized by steric hindrances preventing an inversion of atom N(16). The phenyl substituent is at -32.5 (4)° to the slightly conjugated bond system C(14)=C(15)—N(16).

Introduction. 15-Phenyl-14-dehydrosparteine (I) is an isomer of 2-phenyl-2-dehydrosparteine (II) investigated most recently by spectroscopic and X-ray methods (Boczoń, 1989; Katrusiak, 1992). Other similar derivatives of sparteine investigated by X-rays include 2-(p-tolyl)-2-dehydrosparteine (Małuszyńska, Boczoń & Kałuski, 1986) and 2-phenylsparteine (Katrusiak, Figas, Kałuski & Lesiewicz, 1987). In the molecule of (II) the phenyl is substituted at C(2) in the outer ring A of the rigid

dehydroquinolizidine fragment A/B, with ring A in a half-chair and ring B in a chair conformation, and the junction between these rings in a *trans* configuration. In (I) the phenyl substituent is at C(15) in the outer ring D of the flexible dehydroquinolizidine moiety C/D. Owing to the possible inversion of N(16), ring C can assume either a chair or a boat conformation, and the junction between rings C and D can change its configuration subsequently to *cis* or *trans*, respectively. The X-ray structural determination of (II) confirmed the inversion of N(16) leading to the boat conformation of ring C, arising from electrostatic repulsion of the electron pairs of the N atoms. It was suggested (Boczoń, 1989) that the substitution of the phenyl ring at C(15) would constitute steric hindrances in the molecule of (I) limiting the possibilities of inversion of N(16). Because of these hindrances, and despite the electrostatic repulsion between N(1) and N(16), it was expected that an inversion of N(16) would not take place and that the molecule would preserve the all-chair conformation. This X-ray study was undertaken to confirm these assumptions.

Experimental. Crystals of (I) suitable for X-ray analysis were obtained from ethanol solution as