Table 1. *Atomic coordinates* (× 10^4) *and equivalent isotropic displacement parameters* (Å^2 × 10^3)

	x	y	z	U_{eq}*
O(1)	2627 (2)	4506 (1)	5822 (1)	40 (1)
O(2)	−507 (2)	4930 (1)	7112 (1)	39 (1)
C(1)	1514 (2)	4615 (1)	7177 (2)	30 (1)
C(2)	2694 (2)	4348 (1)	8859 (2)	30 (1)
C(3)	4795 (2)	3981 (1)	9006 (2)	29 (1)
C(4)	6095 (2)	3684 (1)	10618 (2)	28 (1)
C(5)	8204 (2)	3263 (1)	10531 (2)	33 (1)
C(6)	9469 (2)	2970 (1)	12032 (2)	38 (1)
C(7)	8669 (3)	3097 (1)	13633 (2)	41 (1)
C(8)	6587 (3)	3513 (1)	13737 (2)	41 (1)
C(9)	5306 (2)	3804 (1)	12244 (2)	33 (1)
H(10)	1638 (51)	4724 (15)	4669 (43)	128 (10) (U_{iso})

* Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

Table 2. *Bond lengths* (Å) *and angles* (°)

O(1)—C(1)	1·292 (2)	O(1)—C(1)—C(2)	117·5 (1)
O(2)—C(1)	1·254 (2)	O(1)—C(1)—O(2)	123·1 (1)
C(1)—C(2)	1·469 (2)	C(1)—C(2)—C(3)	122·3 (1)
C(2)—C(3)	1·334 (2)	O(2)—C(1)—C(2)	119·4 (1)
C(3)—C(4)	1·467 (2)	C(2)—C(3)—C(4)	126·3 (1)
C(4)—C(5)	1·401 (2)	C(3)—C(4)—C(5)	119·2 (1)
C(5)—C(6)	1·389 (2)	C(3)—C(4)—C(9)	122·4 (1)
C(6)—C(7)	1·381 (2)	C(4)—C(9)—C(8)	120·6 (1)
C(7)—C(8)	1·386 (2)	C(4)—C(5)—C(6)	120·6 (1)
C(8)—C(9)	1·387 (2)	C(5)—C(4)—C(9)	118·4 (1)
C(4)—C(9)	1·395 (2)	C(5)—C(6)—C(7)	120·2 (1)
O(1)—O(2)	2·634 (2)	C(6)—C(7)—C(8)	119·8 (1)
		C(7)—C(8)—C(9)	120·4 (1)

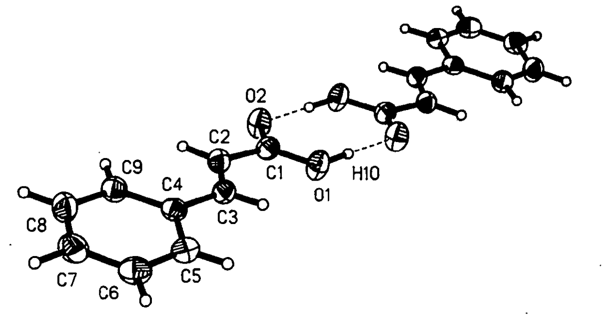

Fig. 1. The atom numbering.

solved by a routine application of *SHELXTL-Plus* (Sheldrick, 1987). All H atoms found from a difference synthesis. Full-matrix least-squares [using $w(F_o - F_c)^2$] anisotropic refinement of the positions of non-H atoms and isotropic refinement of H atoms gave a final $R = 0{\cdot}0412$ [$wR = 0{\cdot}0540$; $w^{-1} = \sigma^2(F) + 0{\cdot}0002F^2$]. The model was refined to convergence: $\Delta/\sigma = 0{\cdot}004$. Data to parameter ratio 11·4:1.

The final difference map was essentially featureless: 0·24 e Å^{-3} (max), −0·19 e Å^{-3} (min). Scattering factors from *International Tables for X-ray Crystallography* (1974).

Final atomic coordinates are given in Table 1, and bond lengths and angles in Table 2. The atom-numbering scheme is shown in Fig. 1.*

Related literature. Lattice constants at room temperature and 90 K (Ladell, McDonald & Schmidt, 1956). Magnetic anisotropies and susceptibilities (Lonsdale, 1939; Lasheen, 1964).

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* Lists of structure factors, anisotropic thermal parameters and atomic paramaters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51390 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1989). C**45**, 339–341

α-Cyano-*N*,*N*-diisopropyl-α-(3-methoxy-6-methylphenyl)acetamide

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Abstract. $C_{17}H_{24}N_2O_2$, $M_r = 288{\cdot}4$, orthorhombic, *Pbca*, $a = 8{\cdot}032$ (3), $b = 17{\cdot}076$ (7), $c = 24{\cdot}83$ (1) Å, $V = 3405$ (1) Å^3, $Z = 8$, $D_x = 1{\cdot}12$ g cm^{-3}, λ(Mo $K\alpha$) $= 0{\cdot}71073$ Å, $\mu = 0{\cdot}69$ cm^{-1}, $F(000) = 1248$, $T = 295$ K, final $R = 0{\cdot}056$ for 1308 observed reflections. The phenyl, amide and nitrile groups and the H atoms are arranged in a distorted tetrahedron with respect to the α-C atom. The X-ray structure reveals the de-

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Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement coefficients* (Å$^2 \times 10^3$)

Equivalent isotropic U defined as one third of the trace of the orthogonalized U_{ij} tensor.

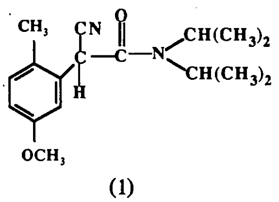
	x	y	z	U_{eq}
C(1)	−1515 (5)	1166 (2)	4152 (2)	36 (2)
C(2)	−1913 (5)	640 (3)	4557 (2)	46 (2)
C(3)	−2594 (6)	−69 (3)	4403 (2)	59 (2)
C(4)	−2912 (6)	−255 (3)	3874 (2)	57 (2)
C(5)	−2570 (6)	281 (3)	3481 (2)	52 (2)
C(6)	−1857 (5)	992 (2)	3620 (2)	46 (2)
C(7)	−717 (5)	1955 (2)	4286 (2)	38 (1)
C(8)	292 (6)	2323 (2)	3826 (2)	41 (2)
N(9)	1719 (4)	1975 (2)	3683 (1)	43 (1)
C(10)	−1680 (7)	829 (3)	5140 (2)	74 (2)
O(11)	−2876 (5)	184 (2)	2943 (1)	86 (2)
C(12)	−3618 (9)	−518 (3)	2783 (2)	110 (3)
C(13)	−2023 (6)	2506 (3)	4454 (2)	47 (2)
N(14)	−3052 (6)	2904 (2)	4608 (2)	69 (2)
O(15)	−244 (4)	2917 (2)	3608 (1)	60 (1)
C(16)	2384 (6)	1259 (3)	3938 (2)	52 (2)
C(17)	2517 (7)	595 (3)	3543 (2)	77 (2)
C(18)	4018 (6)	1415 (3)	4218 (2)	74 (2)
C(19)	2724 (6)	2325 (3)	3247 (2)	55 (2)
C(20)	1826 (7)	2335 (3)	2712 (2)	79 (2)
C(21)	3410 (6)	3122 (3)	3401 (2)	78 (2)

Table 2. *Bond lengths* (Å), *bond angles* (°) *and selected torsion angles* (°)

C(1)—C(2)	1·386 (6)	C(1)—C(6)	1·381 (6)
C(1)—C(7)	1·528 (5)	C(2)—C(3)	1·383 (6)
C(2)—C(10)	1·495 (6)	C(3)—C(4)	1·376 (7)
C(4)—C(5)	1·366 (7)	C(5)—C(6)	1·385 (6)
C(5)—O(11)	1·367 (6)	C(7)—C(8)	1·534 (6)
C(7)—C(13)	1·470 (6)	C(8)—N(9)	1·339 (6)
C(8)—O(15)	1·229 (5)	N(9)—C(16)	1·477 (5)
N(9)—C(19)	1·478 (6)	O(11)—C(12)	1·397 (7)
C(13)—N(14)	1·136 (6)	C(16)—C(17)	1·504 (6)
C(16)—C(18)	1·509 (6)	C(19)—C(20)	1·511 (7)
C(19)—C(21)	1·518 (7)		
C(2)—C(1)—C(6)	120·5 (4)	C(2)—C(1)—C(7)	120·7 (4)
C(6)—C(1)—C(7)	118·8 (4)	C(1)—C(2)—C(3)	117·2 (4)
C(1)—C(2)—C(10)	122·3 (4)	C(3)—C(2)—C(10)	120·5 (4)
C(2)—C(3)—C(4)	122·7 (4)	C(3)—C(4)—C(5)	119·4 (4)
C(4)—C(5)—C(6)	119·5 (4)	C(4)—C(5)—O(11)	125·4 (4)
C(6)—C(5)—O(11)	115·1 (4)	C(1)—C(6)—C(5)	120·6 (4)
C(1)—C(7)—C(8)	114·9 (3)	C(1)—C(7)—C(13)	109·1 (3)
C(8)—C(7)—C(13)	109·0 (3)	C(7)—C(8)—N(9)	117·9 (3)
C(7)—C(8)—O(15)	118·8 (4)	N(9)—C(8)—O(15)	123·3 (4)
C(8)—N(9)—C(16)	124·3 (3)	C(8)—N(9)—C(19)	118·9 (3)
C(16)—N(9)—C(19)	116·9 (3)	C(5)—O(11)—C(12)	117·3 (4)
C(7)—C(13)—N(14)	176·0 (5)	N(9)—C(16)—C(17)	111·7 (4)
N(9)—C(16)—C(18)	111·5 (4)	C(17)—C(16)—C(18)	111·9 (4)
N(9)—C(19)—C(20)	112·8 (4)	N(9)—C(19)—C(21)	112·1 (4)
C(20)—C(19)—C(21)	112·7 (4)		
C(7)—C(1)—C(2)—C(10)	3·5 (6)	C(1)—C(7)—C(8)—N(9)	−69·0 (5)
C(1)—C(7)—C(8)—O(15)	110·8 (4)	C(13)—C(7)—C(8)—O(15)	−12·0 (5)
O(15)—C(8)—N(9)—C(16)	−179·7 (4)	O(15)—C(8)—N(9)—C(19)	1·1 (6)
C(4)—C(5)—O(11)—C(12)	0·8 (7)	C(8)—C(7)—C(13)—N(14)	−173 (8)

localization of electrons over O and N in the amide group.

Experimental. The title compound (1) was prepared by adding ethyl cyanoacetate (1 mmol) to a cold (195 K) solution of lithium diisopropylamide (LDA) in tetrahydrofuran (THF), which was prepared by reacting diisopropylamine (3 mmol) and *n*-butyllithium (3 mmol) in 25 ml of dry THF at 195 K. After the addition of ethyl cyanoacetate, the reaction solution was warmed to 233 K, 2-bromo-4-methylanisole was added, and the resulting solution was stirred for 10 min. The solution was then allowed to warm to ambient temperature, quenched with aqueous sodium bicarbonate and concentrated (rotary evaporator) to yield an oil from which (1) was isolated by flash chromatography (silica gel/5% ethyl acetate: 95% hexane). Crystals of (1) were grown in ethanol to yield transparent needles. Unit-cell parameters by least-squares fit of 25 reflections in the range $10 < 2\theta < 25°$, crystal dimensions $0{\cdot}3 \times 0{\cdot}6 \times 0{\cdot}2$ mm, space group *Pbca* from systematic absences (space group No. 61); automatic Nicolet *R*3*m*/V diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode (scan rate 3·0–15·0° min^{-1}, depending on intensity), 4473 measured reflections, 3916 independent reflections in the ranges $3 < 2\theta < 50°$, $R_{\rm int} = 0{\cdot}060$, hkl range $h\,0\rightarrow10$, $k\,0\rightarrow22$, $l\,0\rightarrow32$, 1308 observed reflections with $F > 6\sigma(F)$, $\sigma(F)$ from counting statistics; three standard reflections ($\bar{3}5\bar{5}$, $\bar{5}0\bar{1}$, $04\bar{4}$) remeasured after every 100 reflections did not show any significant change in intensity during data collection; Lorentz–polarization correction, no absorption or extinction corrections. Structure solved by direct methods (*SHELXTL-Plus*; Sheldrick, 1986) and subsequent difference Fourier methods. H-atom positions were calculated and included in the refinement with fixed isotropic temperature factors and geometrically constrained residing carbon as primary, tertiary and phenyl H atoms. Final full-matrix least-squares refinement (*SHELXTL-Plus*) converged to $R = 0{\cdot}056$, $wR = 0{\cdot}060$, $S = 1{\cdot}78$ for 190 parameters; $w = 1/[\sigma^2(F) + 0{\cdot}0003F^2]$, $\sum w(|F_o| - |F_c|)^2$ minimized; $(\Delta/\sigma)_{\rm max} = 0{\cdot}007$, $\Delta\rho_{\rm max,\,min} = 0{\cdot}26$, $-0{\cdot}27$ e Å^{-3} in final difference Fourier map. Atomic scattering factors for C, H, N and O were those stored in *SHELXTL-Plus* which were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters are given in Table 1,[*] bond lengths, angles and selected torsion angles in Table 2. The identification of the atoms and the configuration are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1.



(1)

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[*] Lists of structure factors, anisotropic temperature factors, selected torsion angles, least-square planes and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51300 (29 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

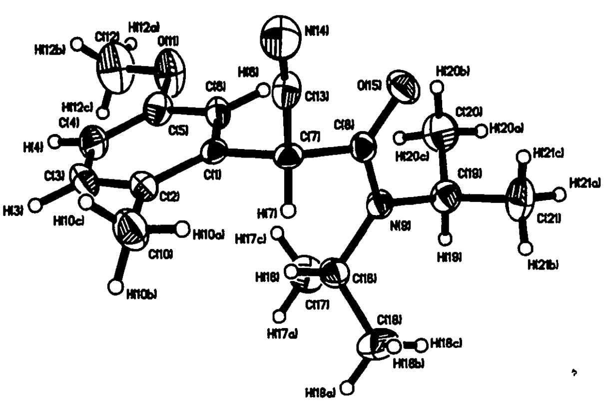

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule. Thermal ellipsoids scaled to enclose 35% probability. H atoms are represented as spheres of arbitrary radii.

Related literature. The mechanism by which compound (1) is formed involves the addition of the anion of α-cyano-*N*,*N*-diisopropylacetamide (formed *in situ* by the reaction of LDA and ethyl cyanoacetate between 195 and 233 K) to 3-methoxy-6-methylbenzyne. To our knowledge, both the addition of an amide to aryne and the low-temperature amination of an ester is unprecedented in the literature. We currently are investigating the mechanism and scope of these novel reactions.

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Structure of Medicagenic Acid: a Triterpene

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Abstract. 2*β*,3*β*-Dihydroxyolean-12-ene-23,28-dioic acid, $C_{30}H_{46}O_6$, $M_r = 502$, orthorhombic, $P2_12_12_1$, $a = 11{\cdot}847\,(1)$, $b = 14{\cdot}654\,(1)$, $c = 15{\cdot}841\,(1)$ Å, $V = 2750{\cdot}1$ Å^3, $Z = 4$, $D_x = 1{\cdot}212$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}47$ cm^{-1}, $F(000) = 1096$, $T = 293\,(2)$ K, $R = 0{\cdot}061$ for 1455 independent reflections with $F_o > 2\sigma(F_o)$. The molecule consists of five six-membered fused rings, rings *D* and *E* are *cis*-fused. Ring *C* has a slightly distorted sofa conformation while rings *A*, *B*, *D* and *E* have chair conformations. The H atom at the C(18) asymmetric center is equatorial relative to the *D* ring. The molecules related by the twofold screw axes in the **a** direction are linked 'head-to-tail' *via* intermolecular hydrogen bonds involving the carboxylic groups.

Experimental. The methanol extract of the roots of *Dolichos kilimandscharicus* Leguminosae yielded the saponin 3-*O*-glucosylmedicagenic acid, which on acid hydrolysis gave medicagenic acid. Transparent rectangular crystals were grown from MeOH. Stoe–Siemens AED-2 four-circle diffractometer, $\theta_{\max} = 22{\cdot}5°$. Four check reflections, no intensity variation. Crystal size 0·38 × 0·30 × 0·27 mm. 2555 unique reflections, 1460 with $F_o > 2\sigma(F_o)$, 1455 used for all calculations, five (probably suffering from extinction) removed. Index range *h* 0–12, *k* 0–15, *l* 0–17. Cell constants from ±*ω* values of 56 reflections in the 2*θ* range 15–27°. No correction for absorption. Structure solved by direct methods using the program system *SHELX*76 (Sheldrick, 1976) which was also used for all further calculations. The –CH and –CH_2 H atoms

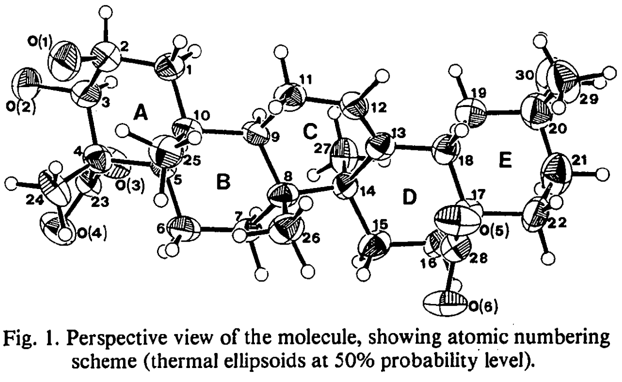

Fig. 1. Perspective view of the molecule, showing atomic numbering scheme (thermal ellipsoids at 50% probability level).

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