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Structures of Three Sesquiterpene γ-Lactones from *Rudbeckia mollis*

BY MARTA VASQUEZ, FRANK R. FRONCZEK, LEOVIGILDO QUIJANO AND NIKOLAUS H. FISCHER

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803, *USA*

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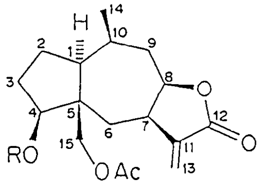
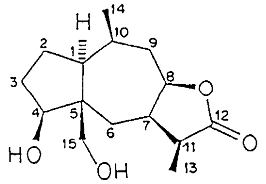
Abstract. (1): 15-Acetoxyrudmollin, $C_{17}H_{24}O_5$, $M_r = 308.4$, orthorhombic, $P2_12_12_1$, $a = 9.0071$ (10), $b = 10.053$ (2), $c = 18.144$ (2) Å, $V = 1642.9$ (7) Å^3, $Z = 4$, $D_x = 1.247$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.85$ cm^{-1}, $F(000) = 664$, $T = 295$ K, $R = 0.049$ for 1540 observations having $I > 3\sigma(I)$ (of 2158 unique data). (2): Rudmollin diacetate, $C_{19}H_{26}O_6$, $M_r = 350.4$, monoclinic, $P2_1$, $a = 8.117$ (2), $b = 7.681$ (4), $c = 14.865$ (3) Å, $\beta = 92.47$ (2)°, $V = 926.0$ (6) Å^3, $Z = 2$, $D_x = 1.257$ g cm^{-3}, λ(Cu $K\alpha$) = 1.54184 Å, $\mu = 7.29$ cm^{-1}, $F(000) = 376$, $T = 295$ K, $R = 0.044$ for 3170 observations having $I > 3\sigma(I)$ (of 3747 unique data). (3): Dihydrorudmollin, $C_{15}H_{24}O_4$, $M_r = 268.4$, orthorhombic, $P2_12_12_1$, $a = 9.441$ (2), $b = 10.731$ (2), $c = 13.349$ (2) Å, $V = 1352.3$ (7) Å^3, $Z = 4$, $D_x = 1.320$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.88$ cm^{-1}, $F(000) = 584$, $T = 125$ K, $R = 0.043$ for 2293 observations having $I > 3\sigma(I)$ (of 3335 unqiue data). Crystals of the three compounds were isolated from *Rudbeckia mollis* Ell (Asteraceae) which was collected in Putman County, Florida. All three compounds are ambrosanolide-type pseudoguaianolides. Dihydrorudmollin (3) has its methyl group β-oriented on the lactone. All compounds have the seven-membered rings in twist-boat conformations with the methyl-bearing C atom on the pseudodiad and the cyclopentane ring in envelope conformations with the quaternary C atom C5 at the flap. Hydroxyl groups of the two OH-bearing compounds [(1) and (3)] are involved in hydrogen bonding.

Introduction. Recently, we have isolated a new sesquiterpene lactone, 11α*H*,13-dihydrorudmollin (3) from *R. mollis* (Vasquez, Quijano, Urbatsch & Fischer, 1991). From the same plant we obtained two related sesquiterpene lactones, 15-acetoxyrudmollin (1) and rudmollin diacetate (2), which had been previously reported by Herz, Kumar & Blount (1981). The chemical structures of compounds (1), (2) and (3) were established on the basis of ^{1}H and ^{13}C NMR spectroscopic studies and chemical transformations. The molecular structures of pseudoguaianolides (1), (2) and (3) were determined in order to learn about the influence of the presence of acetate groups upon the conformation of the rudmollin skeleton in lactones (1) and (2) as well as the effects of saturation of the 11,13-double bond found in (3), when compared to the molecular structure of rudmollin (Herz, Kumar & Blount, 1981).



(1) *R* = H
(2) *R* = Ac



(3)

Experimental. Intensity data for all three compounds were obtained from fragments of colorless needles, on Enraf–Nonius CAD-4 diffractometers equipped with either Mo $K\alpha$ ($\lambda = 0.71073$ Å) or Cu $K\alpha$ ($\lambda = 1.54184$ Å) radiation and graphite monochromators. Data-collection parameters are summarized in Table 1. The cryogenic (125 K) data for (3) were collected using an N_2 gas stream cryostat. Variable scan rates were employed in the ω–2θ scans, and a maximum

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Table 1. *Summary of data collection and structure refinement parameters*

	(1)	(2)	(3)
Crystal size (mm)	0.33 × 0.40 × 0.58	0.33 × 0.38 × 0.53	0.35 × 0.40 × 0.52
Cell constants from θ range (°)	10–13	25–30	11–13
ω-scan width (°)	$0.70 + 0.35\tan\theta$	$0.70 + 0.14\tan\theta$	$0.80 + 0.35\tan\theta$
Scan speeds (° min^{-1})	0.56–4.00	1.03–3.33	0.71–4.00
Max. scan time per reflection (s)	120	60	120
Range for data collection			
θ (°)	1–27.5	2 – 75	1–35
h,k,l	0,11;0,13;0,23	−10,10; −9,9; −18,18	0,15;0,17;0,21
Standard reflections	200, 020, 006	300, 020, 002	300, 060, 002
Intensity decay (%)		14.9	5.9
Empirical absorption correction max., min.		0.9960, 0.8802	
Reflections measured			
Total	2255	7695	3492
Unique	2158	3747	3335
Observed [$I > 3\sigma(I)$]	1540	3170	2293
R_{int}		0.020	
R, wR	0.049, 0.056	0.044, 0.055	0.043, 0.043
Variables	200	306	269
Max. shift/e.s.d. ratio	< 0.01	0.09	< 0.01
Min., max. height in final ΔF (e Å^{-3})	−0.19, 0.19	−0.27, 0.26	−0.24, 0.39
Goodness of fit, S	2.404	1.930	1.439
Extinction, g	$5.0\ (9) \times 10^{-7}$	$6.5\ (6) \times 10^{-6}$	$3.1\ (10) \times 10^{-7}$

Table 2. *Positional parameters and equivalent isotropic thermal parameters with e.s.d.'s*

$$B_{\text{eq}} = (8\pi^2/3)\textstyle\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq} (Å^2)
Compound (1)				
O1	0.0478 (3)	0.3790 (2)	−0.0041 (1)	5.52 (6)
O2	0.1494 (4)	−0.2410 (2)	0.0696 (1)	6.33 (7)
O3	0.0799 (5)	−0.3536 (2)	−0.0302 (2)	9.1 (1)
O4	−0.1666 (2)	0.1378 (2)	0.1398 (1)	3.89 (4)
O5	−0.3337 (3)	0.2859 (3)	0.1782 (2)	6.75 (7)
C1	0.1968 (3)	0.1670 (3)	0.1419 (2)	3.57 (6)
C2	0.2375 (4)	0.3044 (3)	0.1717 (2)	4.60 (8)
C3	0.2087 (4)	0.4006 (3)	0.1072 (2)	4.70 (8)
C4	0.1466 (4)	0.3135 (3)	0.0451 (2)	3.90 (7)
C5	0.0730 (3)	0.1938 (3)	0.0839 (2)	3.18 (6)
C6	0.0443 (4)	0.0800 (3)	0.0290 (2)	3.61 (6)
C7	0.1724 (4)	−0.0199 (3)	0.0217 (2)	3.97 (7)
C8	0.1991 (4)	−0.1073 (3)	0.0906 (2)	4.34 (7)
C9	0.1206 (4)	−0.0721 (3)	0.1611 (2)	4.24 (7)
C10	0.1753 (4)	0.0538 (3)	0.1984 (2)	3.99 (7)
C11	0.1430 (6)	−0.1219 (3)	−0.0381 (2)	5.93 (9)
C12	0.1210 (5)	−0.2501 (4)	−0.0019 (2)	5.78 (9)
C13	0.0973 (6)	−0.1020 (3)	−0.1071 (2)	5.8 (1)
C14	0.0812 (4)	0.0871 (4)	0.2654 (2)	4.93 (8)
C15	−0.0724 (4)	0.2456 (3)	0.1165 (2)	3.62 (6)
C16	−0.2971 (4)	0.1724 (3)	0.1702 (2)	4.25 (7)
C17	−0.3832 (4)	0.0542 (4)	0.1915 (2)	5.59 (9)
Compound (2)				
O1	0.2655 (2)	0	0.5458 (1)	8.05 (5)
O2	0.1013 (3)	−0.2321 (4)	0.5452 (1)	10.19 (6)
O3	0.4870 (2)	0.2108 (2)	0.83888 (7)	4.56 (3)
O4	0.5483 (2)	0.2050 (3)	0.98566 (9)	6.82 (4)
O5	0.0686 (2)	0.4835 (2)	0.87979 (9)	5.27 (3)
O6	−0.1389 (3)	0.6641 (4)	0.8481 (2)	10.98 (6)
C1	0.3447 (3)	0.4884 (3)	0.6922 (1)	4.97 (4)
C2	0.3787 (3)	0.6680 (4)	0.7324 (2)	6.44 (6)
C3	0.2358 (3)	0.6983 (4)	0.7960 (2)	6.25 (5)
C4	0.1406 (3)	0.5270 (3)	0.7943 (1)	4.85 (4)
C5	0.2645 (2)	0.3850 (3)	0.7694 (1)	4.02 (4)
C6	0.1778 (2)	0.2139 (3)	0.7413 (1)	4.00 (3)
C7	0.1235 (2)	0.1981 (3)	0.6410 (1)	4.56 (4)
C8	0.2690 (3)	0.1829 (4)	0.5769 (1)	5.83 (5)
C9	0.4432 (3)	0.2158 (4)	0.6141 (1)	5.59 (5)
C10	0.4816 (3)	0.4061 (4)	0.6378 (1)	5.95 (5)
C11	0.0310 (3)	0.0337 (4)	0.6229 (1)	5.54 (5)
C12	0.1302 (3)	−0.0810 (4)	0.5675 (2)	7.20 (6)
C13	−0.1102 (4)	−0.0213 (5)	0.6552 (2)	7.39 (7)
C14	0.6565 (3)	0.4234 (5)	0.6779 (2)	7.13 (7)
C15	0.3761 (2)	0.3544 (3)	0.8535 (1)	4.29 (4)
C16	0.5658 (2)	0.1474 (3)	0.9118 (1)	4.28 (4)
C17	0.6724 (3)	−0.0031 (4)	0.8905 (2)	6.46 (5)
C18	−0.0713 (3)	0.5629 (4)	0.8982 (2)	6.53 (5)
C19	−0.1281 (3)	0.5117 (5)	0.9885 (2)	7.95 (7)
Compound (3)				
O1	0.7149 (2)	0.1303 (1)	1.0131 (1)	1.42 (2)
O2	0.7896 (2)	0.4811 (1)	0.6248 (1)	1.80 (3)
O3	0.8462 (2)	0.6670 (1)	0.6881 (1)	2.39 (3)
O4	0.4785 (2)	0.2531 (1)	0.9449 (1)	1.55 (2)
C1	0.7116 (2)	0.1082 (2)	0.7389 (1)	0.86 (3)
C2	0.6852 (2)	−0.0280 (2)	0.7703 (1)	1.14 (3)
C3	0.7214 (2)	−0.0318 (2)	0.8826 (2)	1.23 (3)
C4	0.7533 (2)	0.1048 (2)	0.9115 (1)	1.02 (3)
C5	0.6725 (2)	0.1844 (2)	0.8335 (1)	0.82 (2)
C6	0.7251 (2)	0.3213 (2)	0.8338 (1)	0.98 (3)
C7	0.8490 (2)	0.3466 (2)	0.7635 (1)	1.02 (3)
C8	0.8046 (2)	0.3490 (2)	0.6513 (1)	1.20 (3)
C9	0.6650 (2)	0.2889 (2)	0.6218 (2)	1.25 (3)
C10	0.6576 (2)	0.1474 (2)	0.6347 (1)	1.22 (3)
C11	0.9175 (2)	0.4763 (2)	0.7753 (2)	1.29 (3)
C12	0.8491 (2)	0.5550 (2)	0.6952 (2)	1.61 (3)
C13	0.9157 (3)	0.5381 (2)	0.8775 (2)	1.87 (4)
C14	0.5102 (2)	0.1008 (2)	0.6067 (2)	1.79 (4)
C15	0.5131 (2)	0.1830 (2)	0.8566 (2)	1.15 (3)

was set on the time spent on a weak reflection. Cell dimensions were obtained from the setting angles of 25 reflections, including measurements at $\pm 2\theta$. Intensity standards were remeasured every 10 000 s, and linear decay corrections were applied for (2) and (3). One octant of data was collected within the specified θ limits for (1) and (3), while a full sphere of data was collected for (2). Data reduction included corrections for background, Lorentz and polarization effects. Absorption corrections for (2) were based on ψ scans.

Space groups for (1) and (3) were uniquely determined from systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd. The space group of (2) was determined from systematic absences $0k0$ with k odd and the known chirality of the compound. The structures of (1) and (2) were solved by direct methods, using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Compound (3) is isomorphous with rudmollin (Herz, Kumar & Blount, 1981) and coordinates for all non-H atoms except C13 of rudmollin were used as an initial refinement model.

Structures were refined by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$, using the Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients for (2) of Cromer (1974) and data having $I > 3\sigma(I)$. Non-H atoms were refined anisotropically and H atoms were located by ΔF syntheses. For (1), H atoms were included as fixed contributors with a C—H distance of 0.95 Å and $B = 1.3B_{\text{eq}}$ for the bonded atom. For (2), acetate H atoms were treated as above, while other H atoms were refined with isotropic U's. For (3), all H atoms were refined with isotropic U's. Secondary-extinction coefficients, g, were refined for all structures, with the correction factor $(1 + gI_c)^{-1}$ applied to F_c. Final values of the extinction coefficients, residual densities in final ΔF maps, R factors and other details of the refinements are given in Table 1.

Atomic parameters for non-H atoms are given in Table 2, the molecular structures are shown in Figs. 1, 2 and 3, and selected bond distances, angles and torsion angles are given in Table 3.*

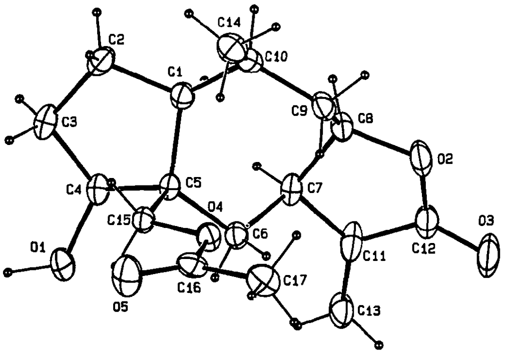

Fig. 1. The molecular structure of (1), with thermal ellipsoids drawn at the 20% probability level and H atoms represented by spheres of arbitrary size.

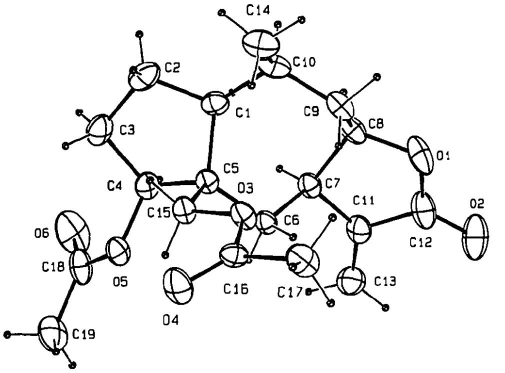

Fig. 2. The molecular structure of (2), with 20% ellipsoids.

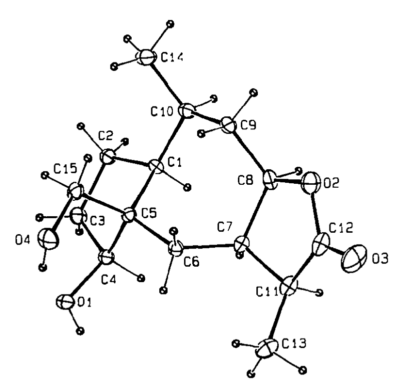

Fig. 3. The molecular structure of (3), with 50% ellipsoids.

Table 3. *Selected bond distances* (Å), *angles* (°) *and torsion angles* (°) *for compounds* (1), (2) *and* (3)

	(1)	(2)	(3)
C1—C5	1.557 (4)	1.561 (3)	1.549 (3)
C5—C15	1.529 (4)	1.530 (2)	1.535 (3)
C7—C8	1.548 (5)	1.554 (3)	1.556 (3)
C10—C14	1.519 (5)	1.522 (3)	1.526 (3)
C11—C13	1.333 (5)	1.330 (4)	1.517 (3)
C2—C1—C5	104.7 (2)	104.3 (2)	103.9 (1)
C1—C5—C4	97.8 (2)	96.6 (2)	98.0 (1)
C1—C5—C15	114.3 (2)	115.3 (2)	113.1 (1)
C6—C7—C8	114.7 (3)	114.0 (1)	112.9 (2)
C7—C8—C9	118.7 (3)	119.0 (1)	118.5 (2)
C7—C11—C12	107.4 (3)	109.0 (2)	104.7 (2)
C5—C1—C2—C3	−30.1 (3)	−33.5 (2)	−32.8 (2)
C1—C2—C3—C4	2.4 (4)	5.3 (3)	5.0 (2)
C2—C3—C4—C5	26.4 (4)	25.3 (2)	24.6 (2)
C3—C4—C5—C1	−43.3 (3)	−44.0 (2)	−43.5 (2)
C2—C1—C5—C4	44.8 (3)	47.1 (2)	46.6 (2)
C10—C1—C5—C6	−63.8 (4)	−60.2 (3)	−61.6 (2)
C1—C5—C6—C7	−20.0 (4)	−22.0 (3)	−21.5 (2)
C5—C6—C7—C8	68.6 (2)	68.7 (3)	73.8 (2)
C6—C7—C8—C9	−10.9 (4)	−10.4 (4)	−18.6 (2)
C7—C8—C9—C10	−70.4 (4)	−70.6 (3)	−66.4 (2)
C8—C9—C10—C1	44.6 (4)	46.8 (2)	48.3 (2)
C9—C10—C1—C5	46.2 (4)	42.2 (3)	41.0 (3)

Discussion. All three compounds are shown to be pseudoguaianolides of the ambrosanolide class, which typically contain a C14 β-methyl group. The methyl group C13 of compound (3) is also shown to be β-oriented. In all compounds, the lactone ring is *cis*-fused to the seven-membered ring and the other five-membered ring is *trans*-fused. In all three compounds, the seven-membered ring is in a twist-boat conformation with the pseudodiad passing through C10 and bisecting the C6—C7 bond. The asymmetry parameter ΔC_2 (Duax & Norton, 1975) has a value of 5.9 for (1), 8.9 for (2) and 5.0° for (3). In all three structures, the cyclopentane ring has the envelope conformation with C5 at the flap. Asymmetry parameters ΔC_s are 2.6 for (1), 5.7 for (2) and 5.7° for (3). The lactone ring is much flatter, and has the envelope conformation with C7 at the flap for compounds (1) and (3), which have asymmetry parameters ΔC_s 1.9 and 2.8°, respectively, while diacetate (2) has its lactone ring in the half-chair conformation, with C12 on the pseudodiad, and ΔC_2 0.8°. The conformations of these three pseudoguaianolides are analogous to that of rudmollin (Herz, Kumar & Blount, 1981). Rudmollin also has both five-membered rings in envelope conformations with C5 and C7 at the flaps. Its seven-membered ring is an intermediate conformation and has been described by Herz, Kumar & Blount (1981) as a boat, with a pseudomirror passing through C1. Our computations show that ΔC_s for that description is 14.3°, while ΔC_2 for the twist boat with C10 on the pseudodiad [the conformation of (1), (2) and (3)] is smaller, at 9.2°. Bond distances for our compounds are normal and show good agreement. Bond distances in dihydrorudmollin (3) agree extremely well with those of rudmollin. The 18 bond lengths not involving

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* Complete tables of distances, angles, torsion angles, H-atom parameters, anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54441 (69 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

C11, in which the hybridization state differs, exhibit an r.m.s. deviation of 0.005 Å, with the largest individual difference, 0.013 Å, for C8—C9. Both our compounds containing OH groups exhibit hydrogen bonding in the solid. The hydroxyl group of O1 of acetate (1) forms an intermolecular hydrogen bond with lactone carbonyl oxygen O3 at $x, y+1, z$ having O—O distance 2.746 (3) Å. The angle about the unrefined H-atom position is 173°. The hydroxyl compound (3) exhibits both intramolecular and intermolecular hydrogen bonding. The intramolecular bond involves O4 as donor and hydroxyl group O1 as acceptor, O—O distance 2.747 (2) Å, angle at H 143 (3)°. The intermolecular hydrogen bond involves O1 as donor and hydroxyl group O4 at $\frac{1}{2}+x, \frac{1}{2}-y, 2-z$, with O—O distance 2.841 (2) Å and angle at H 157 (3)°.

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Structure of a Potential Anticonvulsant: 5′-Bromospiro[1,3-dioxolane-2,3′-indolin]-2′-one

By Amitabha De*

Department of Physics, University College of Science, 92 Acharyya Prafulla Chandra Road,
Calcutta-700 009, India

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Abstract. $C_{10}H_8BrNO_3$, $M_r = 270.08$, monoclinic, $C2/c$, $a = 22.924$ (1), $b = 7.5051$ (4), $c = 15.274$ (10) Å, $\beta = 129.071$ (3)°, $V = 2040.2$ (2) Å^3, $Z = 8$, $D_m = 1.760$ (by flotation), $D_x = 1.758$ Mg m^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1.5418$ Å, $\mu = 5.44$ mm^{-1}, $F(000) = 1072$, $T = 298$ K, $R = 0.071$ for 1424 reflections with $I > 2\sigma(I)$. The bond linking the C(=O) atom to the tetrahedral C atom is a single bond. The amide group exhibits delocalization of the π-electrons over the three atoms (N, C and O). The five- and six-membered rings are planar and inclined at a dihedral angle of 2.6 (5)°. The dioxolane ring is almost perpendicular [89.4 (7)°] to the indolinone group. The crystal structure is stabilized by a three-dimensional network of N(1)—H(N1)···O(1) hydrogen bonds.

Introduction. Anti-epileptic drugs have diverse chemical structures and complex physiological and pharmacological actions. The search for potential anti-epileptic drugs and their mechanism of action has been difficult because of their complexity. There is no single mechanism to explain the action of all anti-epileptic drugs. Some of these drugs have specific modes of action but others have not yet been linked with a specific binding site. A series of spiro[1,3-dioxane-2,3′-indolin]-2′-ones and structural analogues active against electrically and chemically induced seizures have been studied. These compounds contain both an oxoindole and a dioxolane moiety which have independently been seen in other anticonvulsants (Popp, 1977, 1984). The basic model compound, mentioned above, was used to study the effect of various electron-donating, electron-withdrawing and hydrophobic groups on the activity of the molecule. In general a bulky hydrophobic

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* Present address: Structural Studies Division, MRC Laboratory of Molecular Biology, Hills Road, Cambridge CB2 2QH, England.

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