Table 1. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic thermal factors* ($\times 10^3$ Å$^2$) *for α-cyclopiazonic acid*

$$U_{eq} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

| | *x* | *y* | *z* | $U_{eq}$ |
|---|---|---|---|---|
| N1*A* | 3016 (6) | 86 (5) | 9126 (1) | 69 (2) |
| C1*A* | 3841 (7) | 1089 (6) | 9073 (1) | 62 (2) |
| C2*A* | 3123 (7) | 2132 (6) | 9017 (1) | 56 (2) |
| C3*A* | 3468 (6) | 3485 (6) | 8948 (1) | 55 (2) |
| C4*A* | 4437 (6) | 4147 (6) | 9107 (1) | 51 (2) |
| C5*A* | 5730 (6) | 4557 (7) | 9018 (1) | 59 (2) |
| C6*A* | 5817 (7) | 5919 (7) | 9040 (1) | 57 (2) |
| C7*A* | 4593 (6) | 6405 (7) | 9125 (1) | 54 (2) |
| N2*A* | 3822 (5) | 5382 (5) | 9165 (1) | 52 (1) |
| C8*A* | 2403 (6) | 5239 (6) | 9154 (1) | 54 (2) |
| C9*A* | 2286 (6) | 4444 (6) | 8946 (1) | 55 (2) |
| C10*A* | 953 (6) | 3823 (6) | 8903 (1) | 62 (2) |
| C11*A* | 725 (7) | 2505 (6) | 8996 (1) | 57 (2) |
| C12*A* | −449 (7) | 1960 (8) | 9037 (1) | 75 (2) |
| C13*A* | −515 (8) | 641 (8) | 9104 (1) | 81 (2) |
| C14*A* | 573 (8) | −103 (7) | 9138 (1) | 73 (2) |
| C15*A* | 1740 (8) | 511 (7) | 9105 (1) | 61 (2) |
| C16*A* | 1825 (7) | 1796 (6) | 9037 (1) | 51 (2) |
| C17*A* | 6833 (8) | 6654 (8) | 8986 (1) | 75 (2) |
| C18*A* | 7977 (6) | 6151 (9) | 8892 (1) | 98 (3) |
| C19*A* | 1942 (6) | 4537 (6) | 9360 (1) | 66 (2) |
| C20*A* | 1721 (7) | 6533 (6) | 9128 (1) | 68 (2) |
| O1*A* | 6531 (5) | 3771 (5) | 8947 (1) | 85 (1) |
| O2*A* | 6763 (6) | 7997 (6) | 9016 (1) | 119 (2) |
| O3*A* | 4334 (4) | 7552 (4) | 9151 (1) | 65 (1) |
| N1*B* | 4884 (5) | −5635 (5) | 9690 (1) | 58 (1) |
| C1*B* | 4211 (6) | −4590 (6) | 9778 (1) | 59 (2) |
| C2*B* | 4928 (6) | −3503 (6) | 9755 (1) | 49 (2) |
| C3*B* | 4749 (6) | −2143 (5) | 9822 (1) | 53 (2) |
| C4*B* | 4001 (6) | −1302 (6) | 9662 (1) | 53 (2) |
| C5*B* | 2550 (7) | −1198 (7) | 9674 (1) | 57 (2) |
| C6*B* | 2267 (7) | 155 (7) | 9695 (1) | 59 (2) |
| C7*B* | 3443 (7) | 857 (7) | 9723 (1) | 65 (2) |
| N2*B* | 4451 (5) | 10 (5) | 9705 (1) | 59 (1) |
| C8*B* | 5600 (7) | 37 (6) | 9839 (1) | 66 (2) |
| C9*B* | 6049 (6) | −1402 (6) | 9833 (1) | 60 (2) |
| C10*B* | 6909 (7) | −1671 (6) | 9631 (1) | 63 (2) |
| C11*B* | 7103 (6) | −3086 (6) | 9591 (1) | 57 (2) |
| C12*B* | 8152 (7) | −3676 (7) | 9493 (1) | 71 (2) |
| C13*B* | 8115 (7) | −5015 (7) | 9455 (1) | 70 (2) |
| C14*B* | 7102 (7) | −5798 (7) | 9508 (1) | 66 (2) |
| C15*B* | 6059 (7) | −5192 (6) | 9613 (1) | 55 (2) |
| C16*B* | 6078 (6) | −3874 (6) | 9650 (1) | 52 (2) |
| C17*B* | 1043 (8) | 650 (8) | 9700 (1) | 80 (2) |
| C18*B* | 776 (11) | 1963 (7) | 9713 (2) | 143 (4) |
| C19*B* | 5260 (8) | 374 (7) | 10082 (1) | 81 (2) |
| C20*B* | 6599 (7) | 1004 (7) | 9759 (1) | 92 (2) |
| O1*B* | 1783 (4) | −2105 (4) | 9665 (1) | 79 (1) |
| O2*B* | −23 (5) | −215 (7) | 9694 (1) | 128 (2) |
| O3*B* | 3552 (5) | 2018 (5) | 9759 (1) | 90 (2) |

Table 2. *Selected bond distances* (Å) *and bond angles* (°) *for α-cyclopiazonic acid*

| | *A* | *B* |
|---|---|---|
| C5—C6 | 1.427 (9) | 1.444 (8) |
| C6—C7 | 1.467 (8) | 1.435 (8) |
| C6—C17 | 1.347 (9) | 1.374 (9) |
| C17—O2 | 1.411 (8) | 1.429 (9) |
| C17—C18 | 1.422 (9) | 1.397 (9) |
| | | |
| C5—C6—C7 | 108.7 (6) | 109.4 (6) |
| C5—C6—C17 | 126.2 (7) | 123.8 (7) |
| C7—C6—C17 | 125.1 (7) | 126.7 (7) |
| C6—C17—O2 | 119.3 (8) | 118.9 (7) |
| C6—C17—C18 | 123.2 (8) | 123.5 (9) |
| O2—C17—C18 | 117.5 (8) | 117.6 (8) |

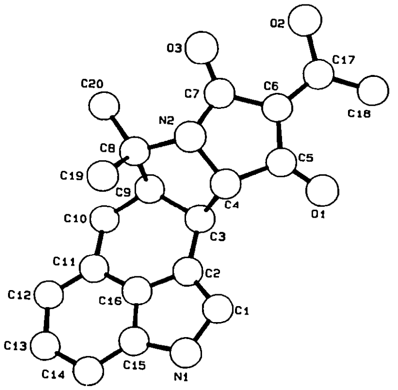

Fig. 1. Perspective view with atomic numbering scheme.

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# Structure of Citreohybridone A

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**Abstract.** $C_{30}H_{38}O_9$, $M_r = 542.6$, orthorhombic, $P2_12_12_1$, $a = 13.119$ (1), $b = 22.204$ (3), $c = 9.868$ (1) Å, $V = 2874.5$ (5) Å$^3$, $Z = 4$, $D_x = 1.25$ Mg m$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71073 Å, $\mu = 0.086$ mm$^{-1}$, $F(000) = 1160$, $T = 297$ K, $R = 0.067$ for 1914 observed unique reflections. The relative structure of a new cytotoxic substance against HeLa cells has been determined by single-crystal X-ray

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diffraction. The C—C bond at the junction of the five- and six-membered rings is as long as 1.619 (9) Å, which may be due to strain in the fused ring structure.

Table 1. *Positional parameters* ( × $10^4$) *and equivalent isotropic temperature factors* (*Hamilton*, 1959)

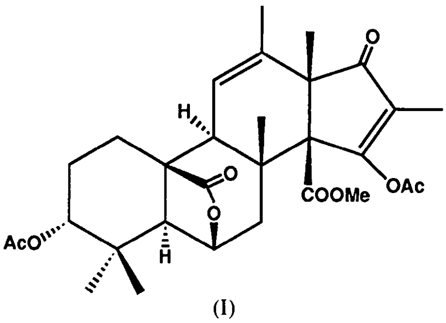
| | $x$ | $y$ | $z$ | $B/B_{eq}$(Å$^2$) × 10 |
|---|---|---|---|---|
| C1 | 630 (5) | 3030 (3) | 6841 (7) | 30 |
| C2 | 622 (5) | 3677 (3) | 6340 (8) | 38 |
| C3 | 1695 (6) | 3946 (3) | 6385 (8) | 40 |
| C4 | 2441 (5) | 3618 (3) | 5486 (8) | 39 |
| C5 | 2476 (5) | 2940 (3) | 5962 (7) | 29 |
| C6 | 2905 (5) | 2494 (3) | 4936 (8) | 34 |
| C7 | 3256 (5) | 1924 (3) | 5621 (7) | 30 |
| C8 | 2400 (4) | 1592 (3) | 6400 (6) | 22 |
| C9 | 1670 (4) | 2059 (3) | 7052 (6) | 23 |
| C10 | 1429 (4) | 2634 (3) | 6197 (7) | 23 |
| C11 | 749 (5) | 1726 (3) | 7584 (7) | 30 |
| C12 | 815 (5) | 1179 (3) | 8133 (7) | 29 |
| C13 | 1826 (5) | 848 (3) | 8338 (7) | 34 |
| C14 | 2774 (5) | 1200 (3) | 7647 (6) | 26 |
| C15 | 3160 (5) | 1551 (3) | 8853 (7) | 29 |
| C16 | 2794 (5) | 1429 (3) | 10016 (7) | 33 |
| C17 | 2042 (6) | 949 (3) | 9842 (7) | 40 |
| C18 | 3106 (7) | 1627 (4) | 11406 (8) | 57 |
| C19 | 3645 (5) | 770 (3) | 7240 (7) | 31 |
| C20 | 1739 (6) | 176 (3) | 7995 (9) | 49 |
| C21 | − 127 (6) | 865 (4) | 8652 (9) | 55 |
| C22 | 1862 (5) | 1159 (3) | 5373 (7) | 32 |
| C23 | 1182 (5) | 2446 (3) | 4769 (7) | 29 |
| C24 | 3510 (6) | 3883 (4) | 5638 (11) | 61 |
| C25 | 2139 (7) | 3701 (4) | 3991 (9) | 55 |
| C26 | 1929 (8) | 4344 (4) | 8609 (11) | 76 |
| C27 | 2310 (10) | 4179 (6) | 9979 (13) | 123 |
| C28 | 4790 (7) | 38 (4) | 8084 (10) | 64 |
| C29 | 4154 (9) | 2467 (5) | 9094 (10) | 85 |
| C30 | 5197 (9) | 2675 (5) | 8973 (11) | 100 |
| O1 | 2069 (4) | 3903 (2) | 7775 (5) | 48 |
| O2 | 1478 (9) | 4789 (3) | 8325 (9) | 158 |
| O3 | 2048 (3) | 2366 (2) | 4040 (4) | 34 |
| O4 | 345 (3) | 2344 (2) | 4281 (5) | 41 |
| O5 | 3960 (4) | 474 (2) | 8319 (5) | 50 |
| O6 | 4009 (4) | 717 (2) | 6157 (5) | 49 |
| O7 | 3997 (4) | 1911 (2) | 8624 (5) | 48 |
| O8 | 3418 (6) | 2722 (3) | 9613 (8) | 96 |
| O9 | 1634 (4) | 659 (3) | 10745 (5) | 63 |

Table 2. *Selected bond lengths* (Å) *and bond angles* (°)

| | | | |
|---|---|---|---|
| C1—C2 | 1.519 (10) | C9—C10 | 1.563 (9) |
| C1—C10 | 1.509 (9) | C9—C11 | 1.511 (9) |
| C2—C3 | 1.530 (10) | C10—C23 | 1.505 (10) |
| C3—C4 | 1.508 (10) | C11—C12 | 1.333 (9) |
| C4—C5 | 1.578 (10) | C12—C13 | 1.530 (9) |
| C5—C6 | 1.524 (10) | C13—C14 | 1.619 (9) |
| C5—C10 | 1.550 (9) | C13—C17 | 1.528 (10) |
| C6—C7 | 1.507 (10) | C14—C15 | 1.510 (9) |
| C6—O3 | 1.458 (8) | C15—C16 | 1.273 (10) |
| C7—C8 | 1.548 (9) | C16—C17 | 1.462 (10) |
| C8—C9 | 1.551 (9) | C23—O3 | 1.356 (8) |
| C8—C14 | 1.585 (9) | | |

| | | | |
|---|---|---|---|
| C2—C1—C10 | 114.8 (6) | C1—C10—C23 | 114.0 (5) |
| C1—C2—C3 | 110.7 (6) | C5—C10—C9 | 105.0 (5) |
| C2—C3—C4 | 113.0 (6) | C5—C10—C23 | 99.9 (5) |
| C3—C4—C5 | 107.7 (6) | C9—C10—C23 | 108.8 (5) |
| C4—C5—C6 | 115.7 (6) | C9—C11—C12 | 122.4 (6) |
| C4—C5—C10 | 115.9 (5) | C11—C12—C13 | 123.2 (6) |
| C6—C5—C10 | 98.2 (5) | C12—C13—C14 | 112.2 (5) |
| C5—C6—C7 | 111.1 (6) | C12—C13—C17 | 102.6 (5) |
| C5—C6—O3 | 104.2 (5) | C14—C13—C17 | 101.3 (5) |
| C7—C6—O3 | 110.1 (5) | C8—C14—C13 | 110.7 (5) |
| C6—C7—C8 | 113.6 (5) | C8—C14—C15 | 115.6 (5) |
| C7—C8—C9 | 109.6 (5) | C13—C14—C15 | 110.1 (5) |
| C7—C8—C14 | 115.0 (5) | C14—C15—C16 | 118.3 (6) |
| C9—C8—C14 | 103.7 (5) | C15—C16—C17 | 107.7 (6) |
| C8—C9—C10 | 116.6 (5) | C13—C17—C16 | 110.3 (6) |
| C8—C9—C11 | 108.1 (5) | C10—C23—O3 | 110.6 (5) |
| C10—C9—C11 | 115.2 (5) | C5—C6—O3 | 104.2 (5) |
| C1—C10—C5 | 115.0 (5) | C6—O3—C23 | 107.4 (5) |
| C1—C10—C9 | 112.9 (5) | | |

**Experimental.** Crystals (I) were grown from an *n*-hexane and benzene solution. X-ray intensities were measured on a Rigaku AFC-5 four-circle diffractometer with graphite-monochromatized Mo $K\alpha$ radiation, $\theta$–$2\theta$, scan speed 6° $\text{min}^{-1}$ in $\theta$, crystal size $0.32 \times 0.47 \times 0.35$ mm, $0 \le h \le 17$, $0 \le k \le 28$, $0 \le l \le 22$ ($4 < 2\theta \le 55°$), 3703 reflections measured, 1914 reflections observed with $|F_o| > 3\sigma(|F_o|)$; lattice constants based on 22 $2\theta$ values ($20 < 2\theta < 30°$). Mean ratio of $|F_o|$ of five standard reflections, $0.99 \le \sum(|F_o|/|F_o|_{\text{initial}})/5 \le 1.00$. Absorption correction by Gauss numerical integration method (Busing & Levy, 1957; relative transmission factors 0.94–0.98). Systematic absences ($h00$ $h$ odd; $0k0$ $k$ odd; $00l$, $l$ odd) indicated the space group to be $P2_12_12_1$.



(I)

The structure was solved by direct methods with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978); coordinates of all the non-H atoms refined by block-diagonal least squares with anisotropic thermal parameters using the *UNICS*III program system (Sakurai & Kobayashi, 1979); 23 of the 38 H atoms were located by difference syntheses and the others were calculated and refined isotropically. Function $\sum w(|F_o| - |F_c|)^2$ was minimized with weight $w^{-1} = \sigma^2(|F_o|) + (0.015|F_o|)^2$. Final $R = 0.067$, $wR = 0.072$, $S = 2.76$, $\Delta/\sigma < 0.18$, number of reflections/parameters = 3.8, $-0.32 < \Delta\rho < 0.64$ e Å$^{-3}$. Complex neutral-atom scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Calculations were performed with a FACOM M-780/10 computer at Keio University. Atomic coordinates are given in Table 1, and selected bond lengths and bond angles in Table 2.* The molecular structure is shown in Fig. 1. Absolute configuration was not determined, because the anomalous dispersion was negligibly small.

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* Lists of structure factors, anisotropic thermal parameters and positional and thermal parameters for H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54499 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0528]

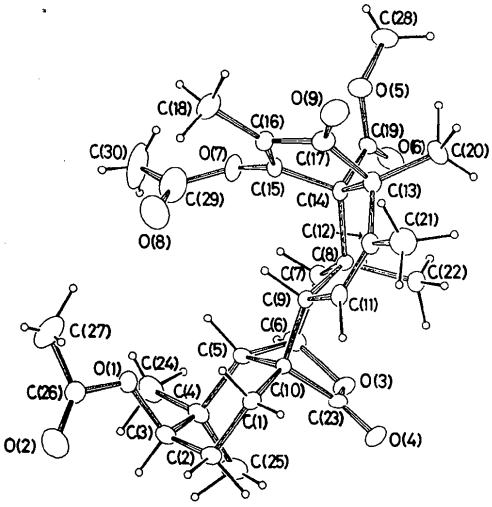

Fig. 1. An *ORTEP* drawing (Johnson, 1965) of the molecule with 20% probability ellipsoids. H atoms are represented by circles of radius 0.08 Å.

**Related literature.** Citreohybridone A and B are new sesterterpenoid-type metabolites of a hybrid strain KO 0031 derived from *Penicillium citreo-viride* B. (IFO 6200 and 4692) (Kosemura, Matsunaga, Yamamura, Kubota & Ohba, 1991).

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# Structure of 2-(4-Aminophenyl)-1,3-propanedinitrile

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(*Received* 10 *June* 1991; *accepted* 12 *September* 1991)

**Abstract.** $C_9H_7N_3$, $M_r = 157.17$, orthorhombic, $Pna2_1$, $a = 8.758\,(5)$, $b = 16.795\,(6)$, $c = 5.646\,(4)$ Å, $V = 831\,(1)$ Å$^3$, $Z = 4$, $D_x = 1.257$ g cm$^{-3}$, $\lambda$(Mo $K\alpha$) = 0.71069 Å, $\mu = 0.75$ cm$^{-1}$, $F(000) = 328$, $T = 296$ K, $R = 0.044$, 475 unique observed reflections. The solid-state structure for 2-(4-aminophenyl)-1,3-propanedinitrile indicates that an H atom is bound at C(2).

**Experimental.** The title compound was prepared following the procedure of Hartzler (1964). A solution of acetone (20 cm$^3$), Raney nickel active catalyst (1.8 g) and 4-nitrophenylmalononitrile (2.5 g, 0.013 mol) was subjected to $3.45 \times 10^5$ Pa of hydrogen for $2\frac{1}{2}$ h in a Parr apparatus, at room temperature with agitation. The solution was then concentrated to give deep orange needles which were washed with water and recrystallized from a boiling ethanol:water (1:1) mixture. The isolated needles (1.14 g, 56% yield) melted at 408–409 K (lit. 408–409 K; Hartzler, 1964). NMR ($CDCl_3$): $\delta$ 7.237, 7.204, 6.721, 6.693 (*AA′BB′*, 4H), $\delta$ 4.905 (*s*, 1H), $\delta$ 3.89 (broad *s*).

The crystal used for data collection was an orange fragment with dimensions $0.27 \times 0.22 \times 0.13$ mm. Data were collected with a Rigaku AFC5S diffractometer using graphite-monochromated Mo $K\alpha$ radiation, $\omega$–$2\theta$ scans, and a scan speed of 4° min$^{-1}$ (in $\omega$). Weak reflections [$I < 10.0\sigma(I)$] were rescanned (maximum of two rescans) and the counts accumulated to assure improved counting statistics. The lattice parameters were obtained from a least-squares fit of 25 strong reflections in the $2\theta$ range 20–29°. A total of 814 reflections were measured ($h$ 0 to 10, $k$ 0 to 19, $l$ 0 to 6) [$(\sin\theta/\lambda)_{\max} = 0.60$ Å$^{-1}$] and 339 reflections were considered unobserved with [$I <$

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