S(31), 0·0714 Å] and hetero N [N(1), –0·0336 Å; N(4), –0·0510 Å]. These displacements are also a consequence of S,N coordination by the ligand.

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Absolute Configuration and Structure of Carminic Acid* Existing as the Potassium Salt in *Dactylopius cacti* L.

By Toshimasa Ishida and Masatoshi Inoue

Laboratory of Physical Chemistry, Osaka University of Pharmaceutical Sciences, 2-10-65 *Kawai, Matsubara, Osaka* 580, *Japan*

Kimiye Baba and Mitsugu Kozawa

Laboratory of Pharmacognosy, Osaka University of Pharmaceutical Sciences, 2-10-65 *Kawai, Matsubara, Osaka* 580, *Japan*

and Kenichiro Inoue and Hiroyuki Inouye

Laboratory of Phytochemistry, Faculty of Pharmaceutical Sciences, Kyoto University, Sakyo-ku, Kyoto 606, *Japan*

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Abstract. $C_{22}H_{19}O_{13}^{-}.K^{+}.3H_2O$, $M_r = 584·53$, monoclinic, $P2_1$, $a = 8·867\,(2)$, $b = 7·126\,(1)$, $c = 19·207\,(3)$ Å, $\beta = 103·67\,(2)°$, $V = 1179·2\,(4)$ Å^3, $Z = 2$, $D_m = 1·621\,(1)$, $D_x = 1·646$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1·5418$ Å, $\mu = 26·99$ cm^{-1}, $F(000) = 608$, $T = 297$ K, $R = 0·042$ for 2196 independent reflections. Careful isolation of carminic acid has shown equimolar coexistence with potassium ions in *Dactylopius cacti* L., and the stereogeometry and the structural characteristics have been elucidated by X-ray analysis.

Introduction. Carminic acid is a natural anthraquinone dyestuff obtained from the insect *Dactylopius cacti* L. and is widely used as one of the most important red

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* Carminic acid is 7-β-D-glucopyranosyl-9,10-dihydro-3,5,6,8-tetrahydroxy-1-methyl-9,10-dioxo-2-anthracenecarboxylic acid (*Chemical Abstracts* name).

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Table 1. ^{13}C *NMR data for potassium carminate*

C1	C2	C3	C4	C4a	C5
160·6	120·2	155·7	149·1	111·2	113·1
C6	C7	C8	C8a	C9	C9a
166·3	127·0	146·6	121·3	184·7	104·5
C10	C10a	COOH	CH	C1′	C2′
185·9	135·3	170·0	20·5	73·6	70·0
C3′	C4′	C5′	C6′		
78·6	81·4	70·2	61·1		

110 mg/0·5 ml Me_2SO-d_6, δ values in p.p.m. relative to TMS.

colouring agents. Because of its unique structure, carminic acid is not only a useful compound for the quantitative determination of trace metals (Aznarez, Ferrer, Rabadan & Macro, 1985; Norwitz & Gordon, 1977; Ogner, 1980), but it has been thought of as an important antitumour agent (Fumero, Merigg, Mondino & Silvestri, 1978; Gutteridge & Quinlan, 1985; Lown, Chen, Sim & Plambeck, 1979; Mihail & Cracium, 1970). The structure of carminic acid was established by chemical and spectral studies (Bhatia & Venkataraman, 1965; Fiecchi, Anastasia, Galli & Gariboldi, 1981; Meloan, Valentine & Puchtler, 1971), and by an NMR investigation including ^{13}C—^{1}H shift correlation (Schmitt, Gunther, Hagele & Stilke, 1984). The necessity of knowing the detailed stereochemistry to elucidate the mode of action at atomic level compelled us to determine the structure and absolute configuration of carminic acid.

Experimental. The dried and crushed *Dactylopius cacti* L. (200 g) was extracted ten times by refluxing with MeOH for 8 h. The solution was concentrated under reduced pressure and treated with hexane and $CHCl_3$. After removal of the components soluble in hexane and $CHCl_3$, the residue was applied to column chromatography (Sephadex LH20, 30% MeOH), and crystallized from 70% MeOH (10·3 g) to afford a red compound, $C_{22}H_{19}O_{13}^{-}.K^{+}$, SIMS: m/z 530 (M^{+}), m.p. 511 K (dec.); UV$\lambda_{\max}^{\text{MeOH}}$ (nm) (log ε): 282·0 (4·75), 320·0 (4·33), 510·0 (4·14); IR$\nu_{\max}^{\text{Nujol}}$ (cm^{-1}): 3650–2000 (—OH), 1660 (CO), 1610, 1585 (arom.); ORD (c=0·38, H_2O) $[\alpha]^{24\,°\text{C}}$ (nm): −36·7° (600), −52·5° (589), −78·7° (580). The ^{13}C NMR spectrum showed some significant differences from that of carminic acid (Table 1).

Dark red crystal, 0·2 × 0·4 × 0·3 mm, from 70% MeOH. D_m by flotation (benzene–ethylene dibromide mixture). Rigaku four-circle diffractometer with graphite-monochromated Cu $K\alpha$ radiation (λ = 1·5418 Å). Cell parameters from 46 reflections with $20 < \theta < 25°$. Intensity data measured by ω–2θ scan mode, scan speed 5° min^{-1} (θ), scan range $(1{\cdot}0 + 0{\cdot}15\tan\theta)°$, 5 s stationary background counts. $2\theta_{\max}$ = 130° (h: 0→10, k: 0→8, l: −22→22). Three reference reflections monitored every 100 reflections showed no significant variation in intensity during data collection (<5%). 2196 independent reflections measured. Absorption correction (Takano, Tanaka & Kusunoki, 1979): transmission factors 0·825–1·213. After the failure of attempts to solve the structure using direct methods, because of the extensive planar arrangement of the anthraquinone rings, the structure was finally solved by a vector search of the aromatic ring in a Patterson map (Tanaka & Yasuoka, 1979). All H atoms were found from a difference Fourier map

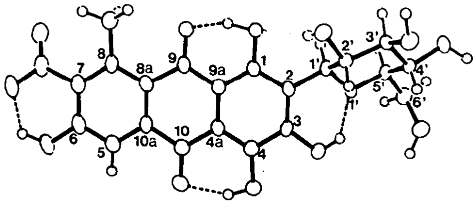

Fig. 1. Conformation of carminic acid, along with atomic numbering. The dotted lines represent the intramolecular hydrogen bonds.

Table 2. *Atomic coordinates and equivalent isotropic thermal parameters of non-*H *atoms with e.s.d.'s in parentheses*

$$B_{\text{eq}} = \tfrac{4}{3}(a^2B_{11} + b^2B_{22} + c^2B_{33} + 2acB_{12}\cos\beta).$$

	x	y	z	B_{eq}(Å^2)
K$^+$	1·1257 (1)	0·8803	1·28100 (6)	3·46 (4)
C1	0·7062 (5)	0·7406 (8)	1·1801 (2)	2·4 (2)
O1	0·8623 (4)	0·7242 (7)	1·1950 (2)	3·5 (2)
C2	0·6343 (5)	0·7383 (8)	1·2386 (2)	2·3 (2)
C3	0·4751 (5)	0·7479 (8)	1·2239 (2)	2·4 (2)
O3	0·3962 (4)	0·7434 (7)	1·2761 (2)	3·3 (2)
C4	0·3845 (5)	0·7626 (8)	1·1533 (2)	2·5 (2)
O4a	0·2283 (4)	0·7667 (7)	1·1454 (2)	3·5 (2)
C4a	0·4546 (5)	0·7655 (8)	1·0964 (2)	2·3 (2)
C5	0·3520 (6)	0·7720 (9)	0·8952 (3)	2·9 (2)
C6	0·4178 (6)	0·7722 (8)	0·8366 (2)	2·7 (2)
O6	0·3261 (4)	0·7852 (6)	0·7709 (2)	3·3 (1)
C7	0·5819 (6)	0·7653 (8)	0·8473 (2)	2·8 (2)
C7′	0·6447 (7)	0·7459 (9)	0·7814 (2)	3·2 (2)
O7′	0·7780 (5)	0·6904 (7)	0·7848 (2)	4·0 (2)
O7″	0·5492 (5)	0·7810 (7)	0·7209 (2)	4·1 (2)
C8	0·6767 (6)	0·7724 (8)	0·9175 (2)	2·7 (2)
C8*M*	0·8508 (6)	0·800 (1)	0·9287 (3)	4·1 (3)
C8a	0·6071 (5)	0·7665 (8)	0·9758 (2)	2·5 (2)
C9	0·6985 (6)	0·7522 (9)	1·0511 (2)	2·7 (2)
O9	0·8423 (4)	0·7345 (8)	1·0655 (2)	4·5 (2)
C9a	0·6195 (5)	0·7544 (8)	1·1096 (2)	2·5 (2)
C10	0·3629 (6)	0·7708 (9)	1·0235 (2)	2·8 (2)
O10a	0·2194 (4)	0·7746 (8)	1·0104 (2)	4·2 (2)
C10a	0·4416 (5)	0·7674 (8)	0·9634 (2)	2·5 (2)
C1′	0·7324 (5)	0·7240 (7)	1·3143 (2)	2·1 (2)
O1′	0·6489 (4)	0·6127 (5)	1·3545 (2)	2·4 (1)
C2′	0·7670 (5)	0·9181 (8)	1·3504 (2)	2·1 (2)
O2′	0·8651 (4)	1·0222 (6)	1·3167 (2)	3·0 (1)
C3′	0·8507 (5)	0·8951 (8)	1·4285 (2)	2·2 (2)
O3′	0·8590 (4)	1·0716 (6)	1·4642 (2)	3·0 (1)
C4′	0·7680 (5)	0·7593 (8)	1·4670 (2)	2·0 (2)
O4′	0·8647 (4)	0·7278 (6)	1·5367 (2)	2·8 (1)
C5′	0·7350 (5)	0·5737 (7)	1·4262 (2)	2·2 (2)
C6′	0·6405 (6)	0·4389 (8)	1·4583 (3)	2·9 (2)
O6′	0·5116 (4)	0·5236 (7)	1·4782 (2)	3·6 (2)
O1*W*	1·3312 (5)	1·1096 (7)	1·3830 (2)	4·0 (2)
O2*W*	1·2083 (5)	0·6148 (7)	1·3957 (2)	4·4 (2)
O3*W*	1·9580 (6)	0·909 (1)	1·7164 (3)	6·6 (3)

(Ashida, 1979). Block-diagonal least-squares refinements (Ashida, 1979) on *F*. *R* and *wR* values for all reflections were 0·042 and 0·041, respectively, where $w = 1{\cdot}0$. $S = 0{\cdot}86$, $(\Delta/\sigma)_{\max} = 0{\cdot}10$ for non-H atoms; $(\Delta\rho)_{\max} = 0{\cdot}23$ and $(\Delta\rho)_{\min} = -0{\cdot}21$ e Å^{-3}. Scattering factors from *International Tables for X-ray Crystallography* (1974). All numerical calculations concerning structure analysis and bonding parameters (Fujii, 1979) were performed on an ACOS 900 computer at the Crystallographic Research Center, Institute for Protein Research Osaka University.

The absolute structure was determined based on the anomalous-dispersion effects of K, C and O atoms; among the 55 measured Bijvoet pairs showing a large difference between $F(hkl)$ and $F(\bar{h}\bar{k}\bar{l})$, 53 pairs clearly suggested the *β*-D-glucopyranosyl configuration of carminic acid.

Discussion. A perspective drawing of the molecule is presented in Fig. 1. The atomic coordinates of the non-H atoms are given in Table 2.* The bond lengths and angles are listed in Table 3. Hydrogen-bond, short contact and ionic interaction parameters are listed in Table 4.

Table 3. *Bond lengths* (Å) *and angles* (°) *of carminic acid*

C1–O1	1·350 (8)	C8–C8*M*	1·52 (1)
C1–C2	1·416 (8)	C8–C8a	1·402 (8)
C1–C9a	1·394 (8)	C8a–C9	1·485 (9)
C2–C3	1·375 (8)	C8a–C10a	1·430 (8)
C2–C1′	1·511 (8)	C9–O9	1·245 (9)
C3–O3	1·351 (8)	C9–C9a	1·458 (9)
C3–C4	1·408 (8)	C10–O10a	1·238 (8)
C4–O4	1·358 (8)	C10–C10a	1·484 (9)
C4–C4a	1·379 (8)	C1′–O1′	1·429 (7)
C4a–C9a	1·426 (8)	C1′–C2′	1·545 (8)
C4a–C10	1·444 (8)	O1′–C5′	1·437 (6)
C5–C6	1·384 (9)	C2′–O2′	1·412 (7)
C5–C10a	1·362 (9)	C2′–C3′	1·518 (8)
C6–O6	1·333 (7)	C3′–O3′	1·426 (7)
C6–C7	1·421 (8)	C3′–C4′	1·509 (8)
C7–C7′	1·504 (9)	C4′–O4′	1·426 (7)
C7–C8	1·411 (8)	C4′–C5′	1·530 (7)
C7′–O7′	1·234 (8)	C5′–C6′	1·500 (8)
C7′–O7″	1·291 (8)	C6′–O6′	1·422 (8)

O1–C1–C2	117·5 (5)	C9–C8a–C10a	117·7 (5)
O1–C1–C9a	120·9 (5)	C8a–C9–O9	121·1 (6)
C2–C1–C9a	121·5 (5)	C8a–C9–C9a	120·0 (5)
C1–C2–C3	118·0 (5)	O9–C9–C9a	118·9 (6)
C1–C2–C1′	120·0 (5)	C1–C9a–C4a	118·9 (5)
C3–C2–C1′	122·0 (5)	C1–C9a–C9	119·6 (5)
C2–C3–O3	122·2 (5)	C4a–C9a–C9	121·5 (5)
C2–C3–C4	121·7 (5)	C4a–C10–O10	120·9 (6)
O3–C3–C4	116·0 (5)	C4a–C10–C10a	119·6 (5)
C3–C4–O4	116·3 (5)	O10–C10–C10a	119·5 (6)
C3–C4–C4a	120·2 (5)	C5–C10a–C8a	120·2 (6)
O4–C4–C4a	123·4 (5)	C5–C10a–C10	118·3 (5)
C4–C4a–C9a	119·6 (5)	C8a–C10a–C10	121·5 (5)
C4–C4a–C10	120·8 (5)	C2–C1′–O1′	107·7 (4)
C9a–C4a–C10	119·5 (5)	C2–C1′–C2′	112·3 (4)
C6–C5–C10a	121·3 (6)	O1′–C1′–C2′	109·1 (4)
C5–C6–O6	119·3 (5)	C1′–O1′–C5′	113·6 (4)
C5–C6–C7	119·7 (5)	C1′–C2′–O2′	110·0 (4)
O6–C6–C7	120·9 (5)	C1′–C2′–C3′	110·3 (4)
C6–C7–C7′	116·8 (5)	O2′–C2′–C3′	107·5 (4)
C6–C7–C8	119·7 (5)	C2′–C3′–O3′	109·7 (5)
C7′–C7–C8	123·5 (5)	C2′–C3′–C4′	111·9 (5)
C7–C7′–O7′	121·7 (6)	O3′–C3′–C4′	107·8 (5)
C7–C7′–O7″	116·5 (5)	C3′–C4′–O4′	107·8 (4)
O7′–C7′–O7″	121·8 (6)	C3′–C4′–C5′	111·1 (4)
C7–C8–C8*M*	119·7 (6)	O4′–C4′–C5′	110·2 (4)
C7–C8–C8a	119·1 (5)	O1′–C5′–C4′	108·5 (4)
C8*M*–C8–C8a	121·0 (6)	O1′–C5′–C6′	107·2 (4)
C8–C8a–C9	122·6 (5)	C4′–C5′–C6′	113·6 (4)
C8–C8a–C10a	119·6 (5)	C5′–C6′–O6′	113·6 (5)

Table 4. *Hydrogen-bond, short contact and ionic interaction parameters*

Donor (at *x*, *y*, *z*)	Acceptor	Symmetry operation	*D*···*A* (Å)	H···*A* (Å)	∠*D*–H···*A* (°)
O1	O9	*x*, *y*, *z*	2·453 (8)	1·53 (8)	145 (7)
O3	O1′	*x*, *y*, *z*	2·561 (6)	1·82 (7)	140 (7)
O4	O10	*x*, *y*, *z*	2·577 (8)	1·72 (8)	151 (7)
O6	O7″	*x*, *y*, *z*	2·395 (7)	1·52 (9)	147 (8)
O2′	O6	1 − *x*, 0·5 + *y*, 2 − *z*	2·805 (6)	1·91 (9)	175 (8)
O3′	O4′	2 − *x*, *y* + 0·5, 3 − *z*	2·695 (6)	1·88 (9)	171 (9)
O4′	O1*W*	2 − *x*, *y* − 0·5, 3 − *z*	2·715 (7)	1·87 (7)	156 (7)
O6′	O2*W*	*x* − 1, *y*, *z*	2·855 (7)	2·11 (9)	163 (9)
O1*W*	O7″	2 − *x*, *y* + 0·5, 2 − *z*	2·756 (7)	1·78 (10)	179 (9)
O1*W*	O6′	2 − *x*, *y* + 0·5, 3 − *z*	2·769 (7)	1·83 (10)	176 (9)
O2*W*	O3′	2 − *x*, *y* − 0·5, 3 − *z*	2·906 (6)	1·98 (10)	178 (9)
O3*W*	O7′	*x* + 1, *y*, *z* + 1	2·774 (9)	1·85 (10)	179 (9)
O3*W*	O1	3 − *x*, *y* + 0·5, 3 − *z*	3·032 (9)	2·05 (11)	176 (9)

Atom (at *x*, *y*, *z*)	Atom	Symmetry operation	Distance (Å)
O1	O2′	*x*, *y*, *z*	3·153 (7)
O3	O4	*x*, *y*, *z*	2·603 (7)
O7′	O1*W*	2 − *x*, *y* − 0·5, 2 − *z*	3·192 (7)
O7″	O1′	1 − *x*, *y* + 0·5, 2 − *z*	3·093 (6)
O1′	O6′	*x*, *y*, *z*	2·984 (6)
O2′	O3′	*x*, *y*, *z*	2·869 (6)
O3′	O4′	*x*, *y*, *z*	2·812 (6)
O4′	O2*W*	2 − *x*, *y* + 0·5, 3 − *z*	3·178 (6)

K^+ (at *x*, *y*, *z*)	Atom	Symmetry operation	Distance (Å)
	O1	*x*, *y*, *z*	2·756 (5)
	O3	1 + *x*, *y*, *z*	2·612 (5)
	O4	1 + *x*, *y*, *z*	3·063 (5)
	O7′	2 − *x*, *y* + 0·5, 2 − *z*	2·779 (5)
	O2′	*x*, *y*, *z*	2·753 (5)
	O1*W*	*x*, *y*, *z*	2·853 (5)
	O2*W*	*x*, *y*, *z*	2·865 (5)

This work has confirmed that carminic acid is 7-*β*-D-glucopyranosyl-1-methyl-3,5,6,8-tetrahydroxy-2-anthraquinone carboxylic acid; the configuration at the C-glycosyl bond has also been determined by other chemical methods (Fiecchi, Anastasia, Galli & Gariboldi, 1981). There are four intramolecular hydrogen bonds which form a stable six-membered ring. Because of an intramolecular hydrogen bond (O6···O1′) and a short contact between O1 and O2′, the rotation around the glycosyl bond is strictly constrained. The anthraquinone ring is almost planar, and the dihedral angle between the two benzene moieties of the ring is 3·8 (3)°. The molecular dimensions of the carboxyl group, which is in anionic form and makes a dihedral angle of 15·9 (3)° with the ring, and of the glucose ring (chair form) are in the normal range.

Careful isolation from *Dactylopius cacti* L. showed that the potassium salt of carminic acid is the natural

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* Lists of structure factors, anisotropic temperature factors, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43774 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

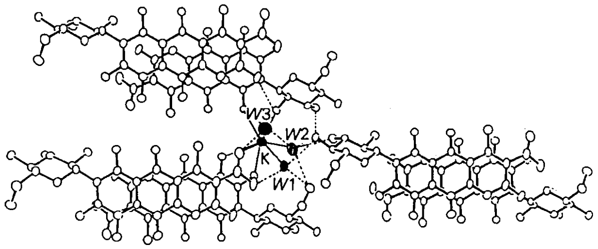

Fig. 2. Molecular packing environment of carminic acids around a potassium ion, viewed along the *b* axis. Ionic interactions with the potassium ion (full circle) are shown by thin lines, and hydrogen bonds by the dotted lines. Shaded circles represent waters of crystallization.

form. This is the first insight. Although its physiological role is at present unclear, the potassium ion appears to stabilize the molecular structure of anionic carminic acid by the neutralization. As is obvious from Fig. 2 and Table 4, extensive ionic interactions are observed between K^+ and the polarizable atoms of the carminic acid and water molecules. In addition to the oxygen atom of the carboxyl anion, those of the 5-, 6- and 8-hydroxyl groups also participate in the interactions.

A structural feature of the crystal is the extensive stacking of anthraquinone rings. The dihedral angle between the aromatic rings is 4·9 (2)° and their average interplanar spacing is 3·5 Å: the carboxyl group and glucose ring jut out from the stacking layers. The formation of these infinite stacking layers would result in the profound red colouration characteristic of carminic acid.

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Structure of Bis(18-crown-6)potassium* Dioxotetrathiocyanatouranate–Water

BY WANG MING AND ZHENG PEI-JU

Center of Analysis and Measurement, Fudan University, Shanghai, People's Republic of China

AND ZHANG JING-ZHI, CHEN ZHONG, SHEN JIN-MING AND YANG YONG-HUI

Department of Chemistry, Shandong University, Jinan, Shandong, People's Republic of China

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Abstract. $[K(C_{12}H_{24}O_6)]_2[UO_2(NCS)_4(H_2O)]$, M_r = 1127·22, monoclinic, $C2/c$, a = 26·780 (4), b = 8·661 (2), c = 24·814 (2) Å, β = 129·08 (1)°, V = 4467·6 Å^3, D_x = 1·676 g cm^{-3}, Z = 4, λ(Mo $K\alpha$) = 0·71073 Å, μ = 38·709 cm^{-1}, $F(000)$ = 2240, T = 293 K, R = 0·035 for 4097 observed reflections with

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* 18-Crown-6 = 1,4,7,10,13,16-hexaoxacyclooctadecane.

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