Table 3. *Geometry of the hydrogen bonds* (Å,°); *e.s.d.'s are given in parentheses*

D—H···*A*	*D*—H	*D*···*A*	H···*A*	*D*—H···*A*
O(1)—H(10)···O(7$^{\rm i}$)	0·90 (3)	2·731 (3)	1·84 (2)	171 (1)
O(2)—H(20)···O(1$^{\rm ii}$)	0·90 (1)	2·747 (3)	1·89 (2)	158 (2)
O(3)—H(30)···O(2$^{\rm iii}$)	0·93 (1)	2·696 (2)	1·77 (1)	174 (1)
O(4)—H(40)···O(3$^{\rm iv}$)	0·90 (1)	2·738 (2)	1·88 (1)	158 (1)
O(5)—H(50)···O(4$^{\rm iii}$)	0·89 (1)	2·688 (2)	1·80 (1)	175 (2)
O(6)—H(60)···O(5$^{\rm v}$)	0·90 (1)	2·747 (3)	1·89 (2)	159 (1)
O(7)—H(70)···O(6$^{\rm iii}$)	0·91 (1)	2·730 (2)	1·82 (1)	175 (1)

Symmetry operations: (i) $-\frac{1}{2}+x, \frac{1}{2}-y, -\frac{1}{2}+z$; (ii) $x, 1-y, \frac{1}{2}+z$; (iii) $x, y-1, z$; (iv) $x, 1-y, -\frac{1}{2}+z$; (v) $x, 1-y, \frac{1}{2}+z$.

and the orientations of molecules within the cell show substantial differences. In the racemate the molecular planes through the C atoms of each molecule are aligned parallel to each other, whereas the molecular planes of the two sets of crystallographically independent molecules in the pure enantiomer include an angle of approximately 90°. Furthermore, in the pure enantiomer the molecules are orientated head to head, while the D and L enantiomers, respectively, in the racemate are found in a head-to-tail relationship. The unit cell displayed in Fig. 2 demonstrates this orientation. In accordance with the Wallach 'rule', the density D_x of the racemate (1·529 g cm^{-3}) is slightly higher than that of the pure enantiomer (1·510 g cm^{-3}).

All seven hydroxyl group H atoms are involved in intermolecular hydrogen bonds (Table 3). In contrast to the pure enantiomer, no bifurcated hydrogen bonds are observed in the title compound. The pattern of these bonds can partially be discerned in Fig. 2. It is totally different from the hydrogen-bond network found in the pure enantiomer.

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Structure of 4,7-Didehydroneophysalin B, Acid-Induced Rearrangement Product of Physalin A

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Abstract. $C_{28}H_{28}O_9$, $M_r = 508·5$, orthorhombic, $P2_12_12_1$, $a = 14·333\,(2)$, $b = 13·120\,(2)$, $c = 12·445\,(2)$ Å, $V = 2340·3\,(1)$ Å^3, $Z = 4$, $D_x = 1·443$ g cm^{-3}, $\lambda(\mathrm{Cu}\ K\alpha) = 1·54178$ Å, $\mu = 8·57$ cm^{-1}, $F(000) = 1072$, $T = 295$ K, $R = 0·042$ for 1910 reflections. The rigid molecule consists of eight fused rings involving three lactone rings. An intramolecular hydrogen bond is formed between the ketone and hydroxyl groups. The spiro-fused γ-lactone rings in an envelope conformation are fused to a cyclohexane ring in a half-chair conformation flattened at the distorted spiro junction. The δ-lactone ring fused to the cyclohexane ring also adopts a half-chair conformation.

Introduction. Physalin A (I), isolated as a bitter principle of *Physalis alkekengi var. francheti*, is the first member of a novel class of steroids, physalins,

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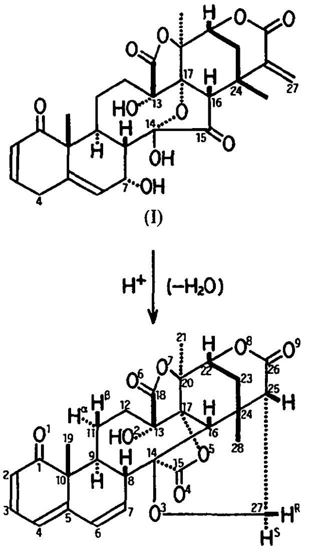
possessing a 13,14-seco-16,24-cycloergostane skeleton (Matsuura, Kawai, Nakashima & Butsugan, 1970). The structure of (I) is based on the X-ray analysis of a bromine-containing derivative of (I) (Kawai, Matsuura, Taga & Osaki, 1970). Reinvestigation of the acid-induced dehydration products of (I) using ^{1}H and ^{13}C NMR spectroscopy, however, revealed that one of the products, (II), does not maintain the original carbon skeleton (Kawai, Ogura, Butsugan, Taga & Hayashi, 1991). The present X-ray study was undertaken to establish the structure of (II) and to investigate its stereochemistry.



Experimental. Yellow prismatic crystals were grown from methanol solution. A crystal of dimensions 0·30 × 0·3 × 0·4 mm was used for data collection on a Rigaku AFC-5RU diffractometer using graphite-monochromated Cu $K\alpha$ radiation. Cell dimensions were determined by a least-squares refinement of the setting angles of 24 reflections having $25 < \theta < 27°$. Intensities of reflections with indices, $0 \leq h \leq 16$, $0 \leq k \leq 14$, $0 \leq l \leq 13$, within a range of $2\theta \leq 120°$ were measured by 2θ–ω scans at speed of 4° min^{-1} with scan width (0·8 + 0·45tanθ)°. Three check reflections showed no significant fluctuation during data collection. Data were corrected for Lorentz and polarization factors, but not for absorption. 1950 reflections were collected, of which 1932 were unique ($R_{\rm int}$ = 0·013). The structure was solved with *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and refined by block-diagonal least squares based on F with $w = [\sigma^2(F) + (0{\cdot}023F)^2]^{-1}$. Non-H atoms were refined anisotropically. H atoms located by difference Fourier synthesis were refined isotropically. The final R value was 0·042 (wR = 0·053, S = 3·0) for 1910 reflections with $F_o > 3\sigma(F_o)$; parameter shifts were less than 0·12σ, and residual densities in the final density map ranged from −0·35 to 0·24 e Å^{-3}. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). All computations were performed on a FACOM M780 in the Data Processing Center of Kyoto University, using the program *KPPXRAY* (Taga, Higashi & Iizuka, 1985).

Discussion. The final atomic parameters are listed in Table 1.* Bond distances and angles are listed in Table 2. A stereoview of the molecule (II) is shown in Fig. 1. The rigid molecule consists of eight fused rings involving three lactone rings. The structure includes a newly formed γ-lactone ring instead of the oxacyclopentanone ring of the physalin skeleton (I). The carbon skeletal rearrangement from (I) to (II) involves C(15)—C(16) bond cleavage and C(14)—C(16) bond formation, accompanied by bond migration of the ether oxygen at C(14) to the C(15) position, and it also involves O(3)—C(27) bond formation, resulting in a new oxacyclohexane ring in a boat form. In the subsequent structure, an intramolecular hydrogen bond with O···O 2·887 (4) Å and O—H···O 167 (3)° is formed between O(1) and O(2). The two γ-lactone rings adopt envelope conformations; C(16) deviates from the least-squares plane through C(14), C(15), C(17), O(4) and O(5) by 0·38 Å, and C(20) deviates from the least-squares plane through C(13), C(17), C(18), O(6) and O(7) by 0·48 Å. Both rings are fused at C(17) in a spiro form. The bonding feature about the junction atom is distorted from the ideal sp^3 hybridization; the C(17)—C(13) bond is elongated to 1·585 (4) Å, and the C(16)—C(17)—C(20) angle is opened to 117·7 (3)°. Such bond distortion about C(17) causes a half-chair conformation of the cyclohexane ring fused to these γ-lactone rings; C(16), C(17), C(20), C(22) and C(24) are planar within 0·08 Å, and C(23) deviates from the plane by 0·68 Å. The δ-lactone ring fused to this six-membered ring also adopts a half-chair conformation flattened at the carbonyl end; C(23) deviates from the plane through the other five atoms by 0·61 Å. The C(5)—C(10) fused six-membered rings are planar within 0·10 Å except for C(10) and the C(sp^2)—C(sp^2) bonds in the rings are conjugated by the through-bond coupling of the π electrons. C(10) and O(1) deviate from the plane by 0·47 and 0·44 Å, respectively. The C(9)—C(11)—C(12) angle [118·1 (3)°] and the C(11)—C(12)—C(13)

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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 54132 (8 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates and equivalent isotropic temperature factors with e.s.d.'s in parentheses*

$B_{eq} = (4/3)\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}(Å^2)
C(1)	0·6243 (3)	0·1285 (3)	0·4019 (3)	3·73 (8)
C(2)	0·6337 (3)	0·0726 (4)	0·3017 (3)	5·0 (1)
C(3)	0·5635 (4)	0·0187 (4)	0·2639 (3)	5·4 (1)
C(4)	0·4720 (3)	0·0210 (3)	0·3158 (3)	5·1 (1)
C(5)	0·4545 (3)	0·0848 (3)	0·3978 (3)	3·91 (9)
C(6)	0·3645 (3)	0·0886 (3)	0·4494 (3)	4·6 (1)
C(7)	0·3485 (2)	0·1416 (3)	0·5387 (3)	4·2 (1)
C(8)	0·4216 (2)	0·2017 (3)	0·5981 (3)	3·19 (9)
C(9)	0·5217 (2)	0·1886 (2)	0·5534 (3)	2·84 (8)
C(10)	0·5254 (2)	0·1638 (3)	0·4319 (3)	3·37 (8)
C(11)	0·5825 (2)	0·2830 (2)	0·5821 (3)	3·39 (9)
C(12)	0·6545 (2)	0·2722 (3)	0·6727 (3)	3·27 (8)
C(13)	0·6571 (2)	0·1768 (3)	0·7428 (3)	3·03 (7)
C(14)	0·4159 (2)	0·1739 (2)	0·7208 (3)	3·02 (8)
C(15)	0·4431 (2)	0·0623 (3)	0·7326 (3)	3·08 (8)
C(16)	0·4845 (2)	0·2283 (2)	0·7977 (3)	2·83 (8)
C(17)	0·5661 (2)	0·1511 (2)	0·8099 (3)	2·66 (7)
C(18)	0·7280 (2)	0·2018 (3)	0·8288 (3)	4·0 (1)
C(19)	0·5025 (3)	0·2579 (3)	0·3590 (3)	4·8 (1)
C(20)	0·6068 (2)	0·1337 (3)	0·9254 (3)	3·47 (8)
C(21)	0·6387 (3)	0·0244 (3)	0·9457 (3)	4·5 (1)
C(22)	0·5479 (3)	0·1739 (3)	1·0180 (3)	3·8 (1)
C(23)	0·5041 (3)	0·2737 (3)	0·9940 (3)	4·0 (1)
C(24)	0·4336 (2)	0·2617 (2)	0·9032 (3)	3·42 (9)
C(25)	0·3617 (2)	0·1824 (3)	0·9394 (3)	3·71 (9)
C(26)	0·3988 (3)	0·0885 (3)	0·9941 (3)	3·77 (9)
C(27)	0·2934 (2)	0·1549 (4)	0·8502 (4)	5·0 (1)
C(28)	0·3845 (3)	0·3636 (3)	0·8845 (3)	4·8 (1)
O(1)	0·6915 (2)	0·1426 (2)	0·4599 (2)	4·60 (7)
O(2)	0·6884 (1)	0·0904 (2)	0·6854 (2)	3·27 (5)
O(3)	0·3213 (1)	0·1923 (2)	0·7473 (2)	4·08 (7)
O(4)	0·4001 (2)	−0·0114 (2)	0·7056 (2)	4·45 (5)
O(5)	0·5272 (1)	0·0536 (2)	0·7788 (2)	2·74 (5)
O(6)	0·8082 (2)	0·2246 (3)	0·8158 (2)	5·96 (8)
O(7)	0·6905 (2)	0·1976 (2)	0·9279 (2)	4·32 (7)
O(8)	0·4819 (2)	0·0955 (2)	1·0473 (2)	4·49 (7)
O(9)	0·3572 (2)	0·0104 (2)	1·0026 (3)	5·76 (9)

Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

C(1)—C(2)	1·453 (6)	C(1)—C(10)	1·537 (5)
C(1)—O(1)	1·218 (5)	C(2)—C(3)	1·317 (7)
C(3)—C(4)	1·462 (7)	C(4)—C(5)	1·343 (5)
C(5)—C(6)	1·442 (6)	C(5)—C(10)	1·512 (5)
C(6)—C(7)	1·331 (5)	C(7)—C(8)	1·505 (5)
C(8)—C(9)	1·548 (4)	C(8)—C(14)	1·572 (5)
C(9)—C(10)	1·548 (5)	C(9)—C(11)	1·556 (4)
C(10)—C(19)	1·567 (5)	C(11)—C(12)	1·535 (5)
C(12)—C(13)	1·526 (5)	C(13)—C(17)	1·585 (4)
C(13)—C(18)	1·512 (5)	C(13)—O(2)	1·413 (5)
C(14)—C(15)	1·522 (5)	C(14)—C(16)	1·547 (5)
C(14)—O(3)	1·416 (3)	C(15)—O(4)	1·195 (5)
C(15)—O(5)	1·340 (3)	C(16)—C(17)	1·555 (4)
C(16)—C(24)	1·565 (5)	C(17)—C(20)	1·568 (5)
C(17)—O(5)	1·448 (4)	C(18)—O(6)	1·199 (4)
C(18)—O(7)	1·346 (4)	C(20)—C(21)	1·526 (6)
C(20)—C(22)	1·523 (5)	C(20)—O(7)	1·464 (4)
C(22)—C(23)	1·482 (6)	C(22)—O(8)	1·444 (5)
C(23)—C(24)	1·524 (5)	C(24)—C(25)	1·532 (4)
C(24)—C(28)	1·529 (5)	C(25)—C(26)	1·505 (5)
C(25)—C(27)	1·523 (5)	C(26)—O(8)	1·366 (5)
C(26)—O(9)	1·190 (5)	C(27)—O(3)	1·428 (5)

C(2)—C(1)—C(10)	116·5 (3)	C(2)—C(1)—O(1)	120·8 (4)
C(10)—C(1)—O(1)	122·7 (3)	C(1)—C(2)—C(3)	120·5 (4)
C(2)—C(3)—C(4)	121·1 (4)	C(3)—C(4)—C(5)	121·1 (4)
C(4)—C(5)—C(6)	121·8 (4)	C(4)—C(5)—C(10)	121·0 (4)
C(6)—C(5)—C(10)	116·9 (3)	C(5)—C(6)—C(7)	123·0 (4)
C(6)—C(7)—C(8)	124·3 (3)	C(7)—C(8)—C(9)	114·2 (3)
C(7)—C(8)—C(14)	108·6 (3)	C(9)—C(8)—C(14)	111·8 (3)
C(8)—C(9)—C(10)	114·0 (3)	C(8)—C(9)—C(11)	110·4 (3)
C(10)—C(9)—C(11)	111·9 (3)	C(1)—C(10)—C(5)	110·2 (3)
C(1)—C(10)—C(9)	109·4 (3)	C(1)—C(10)—C(19)	106·9 (3)
C(5)—C(10)—C(9)	113·3 (3)	C(5)—C(10)—C(19)	103·7 (3)
C(9)—C(10)—C(19)	113·1 (3)	C(9)—C(11)—C(12)	118·1 (3)
C(11)—C(12)—C(13)	120·8 (3)	C(12)—C(13)—C(17)	117·1 (3)
C(12)—C(13)—C(18)	104·1 (3)	C(12)—C(13)—O(2)	112·1 (3)
C(17)—C(13)—C(18)	103·1 (3)	C(17)—C(13)—O(2)	110·9 (3)
C(18)—C(13)—O(2)	108·6 (3)	C(8)—C(14)—C(15)	107·7 (3)
C(8)—C(14)—C(16)	117·4 (3)	C(8)—C(14)—O(3)	103·7 (2)
C(15)—C(14)—C(16)	102·8 (2)	C(15)—C(14)—O(3)	112·7 (2)
C(16)—C(14)—O(3)	112·7 (3)	C(14)—C(15)—O(4)	128·3 (3)
C(14)—C(15)—O(5)	110·7 (3)	O(4)—C(15)—O(5)	121·0 (3)
C(14)—C(16)—C(17)	103·8 (2)	C(14)—C(16)—C(24)	110·6 (2)
C(17)—C(16)—C(24)	116·8 (3)	C(13)—C(17)—C(16)	115·4 (3)
C(13)—C(17)—C(20)	102·0 (2)	C(13)—C(17)—O(5)	111·3 (3)
C(16)—C(17)—C(20)	117·7 (3)	C(16)—C(17)—O(5)	105·1 (2)
C(20)—C(17)—O(5)	105·0 (2)	C(13)—C(18)—O(6)	127·1 (3)
C(13)—C(18)—O(7)	111·8 (3)	O(6)—C(18)—O(7)	121·1 (3)
C(17)—C(20)—C(21)	113·6 (3)	C(17)—C(20)—C(22)	115·9 (3)
C(17)—C(20)—O(7)	103·9 (3)	C(21)—C(20)—C(22)	111·5 (3)
C(21)—C(20)—O(7)	106·8 (3)	C(22)—C(20)—O(7)	103·9 (3)
C(20)—C(22)—C(23)	112·8 (3)	C(20)—C(22)—O(8)	107·9 (3)
C(23)—C(22)—O(8)	113·7 (3)	C(22)—C(23)—C(24)	109·8 (3)
C(16)—C(24)—C(23)	110·0 (3)	C(16)—C(24)—C(25)	111·7 (2)
C(16)—C(24)—C(28)	109·4 (3)	C(23)—C(24)—C(25)	107·3 (3)
C(23)—C(24)—C(28)	109·1 (3)	C(25)—C(24)—C(28)	109·2 (3)
C(24)—C(25)—C(26)	116·8 (3)	C(24)—C(25)—C(27)	112·3 (3)
C(26)—C(25)—C(27)	111·3 (3)	C(25)—C(26)—O(8)	118·2 (3)
C(25)—C(26)—O(9)	124·6 (4)	O(8)—C(26)—O(9)	116·9 (4)
C(25)—C(27)—O(3)	113·1 (3)	C(14)—O(3)—C(27)	114·7 (2)
C(15)—O(5)—C(17)	112·7 (3)	C(18)—O(7)—C(20)	109·3 (3)
C(22)—O(8)—C(26)	119·8 (3)		

angle [120·8 (3)°] of the cyclooctane ring deviate considerably from the normal C(sp^3) angle. Comparison with the structure of (I) (Kawai, Matsuura, Taga & Osaki, 1970) shows a similar bond distortion at the central macrocyclic moiety.

The established stereostructure of (II) is consistent with the ^{13}C NMR spectra exhibiting a single ketone and three lactone carbonyl signals, and it has enabled unequivocal ^{1}H NMR spectral assignments of the nonequivalent methylene protons which exhibit large chemical shift differences (Kawai, Ogura, Butsugan, Taga & Hayashi, 1991). In Me$_2$SO-d_6 solution the methylene protons at C(11) exhibit two multiplets at δ 1·74 and 2·57, to which β and α configurations, respectively, are assigned, since a downfield shift can be expected for the H$^\alpha$ atom located near the plane of the C(1) carbonyl group [H$^\alpha\cdots$O(1) 2·46 (4) Å]. As for the C(27) methylene group a double doublet at δ 4·01 (J = 12 and 3·5 Hz) and a triplet at δ 4·26 (J = 12 Hz) are observed. The higher field signal (δ 4·01) is assigned to the H^S atom (designated in the structural formula) which lies in the anisotropic shielding zone of the C(15) carbonyl group [H$^S\cdots$O(4) 2·66 (5) Å]. The vicinal coupling constants (12 for H^R and 3·5 Hz for H^S) are also consistent with the torsion angles H—C(25)—

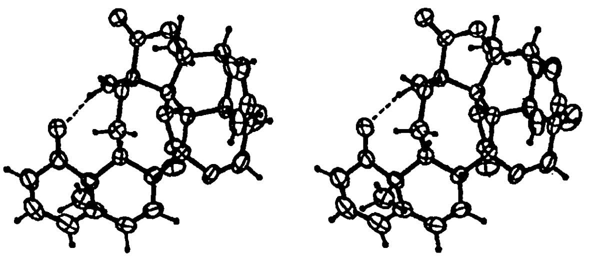

Fig. 1. Stereoscopic view of molecule (II). Thermal ellipsoids are drawn at the 50% probability level. The broken line indicates an intramolecular hydrogen bond.

C(27)—H^R 8 (4) and H—C(25)—C(27)—H^S −112 (4)° in the crystal (Karplus, 1959).

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Structure of Capnoidine, a Phthalide Isoquinoline Alkaloid

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Abstract. $C_{20}H_{17}NO_6$, M_r = 367·36, orthorhombic, $P2_12_12_1$, a = 7·446 (3), b = 13·239 (5), c = 16·634 (5) Å, V = 1640 (1) Å^3, Z = 4, D_x = 1·488 Mg m^{-3}, λ(Mo $K\alpha$) = 0·7107 Å, μ = 0·104 mm^{-1}, F(000) = 768, T = 293 (1) K, R = 0·038 for 1300 unique observed reflections. Ring *B* has almost screw-boat shape. Both C5 and C11 chiral centers possess the *R* configuration. The molecule consists of two approximate planes of atoms with a dihedral angle of 42·3 (2)° and joined by the C5—C11 bond. The epimer of capnoidine is bicuculline.

Introduction. In the course of our studies of natural products we investigated the crystal and molecular structure of capnoidine isolated from *Corydalis cava* collected from Vojvodinas Danube region. Extraction, isolation, separation and identification of capnoidine were performed as described by Slavik & Slavikova (1961), Preininger, Šimanek, Gašić & Šantavy (1973) and Gašić, Popović & Dragutinović (1985). The interest in this work lies in the accurate determination of molecular conformation of the compound which might assist in a better understanding of its biological action, studies of which are in progress.

Experimental. A crystal 0·22 × 0·35 × 0·15 mm was mounted on a CAD-4 diffractometer (University of Berne) using graphite-monochromated Mo $K\alpha$ radiation. Cell constants were refined by a least-squares fit for 22 centered reflections with θ range from 4·2 to 8·5°. Intensity data were collected with ω–2θ scan in the range $2\theta < 50°$; h 0 to 8, k 0 to 15, l 0 to 19. The systematic absences (h00: $h = 2n + 1$, 0k0: $k = 2n + 1$, 00l: $l = 2n + 1$) corresponded to those of orthorhombic space group $P2_12_12_1$. Standard reflection ($\bar{2}01$) was measured every 200 min, but no intensity variations were recorded. 1669 unique reflections, 1300 with $I > 3\cdot5\sigma(I)$ were considered as observed. Data were corrected for Lorentz and polarization effects. The structure was solved using *SHELXS*86 (Sheldrick, 1986) with all non-H atoms obtained from the *E* map (R = 0·173). Full-matrix least-squares (on *F*) isotropic (R = 0·096) and anisotropic refinement with program *SHELX*76 (Sheldrick, 1976) minimized $\sum w(\Delta F)^2$ for 245 parameters with unit weights. Final R = 0·038, wR = 0·038, $(\Delta/\sigma)_{\rm max}$ = 0·007. Positions of H atoms were generated from assumed geometries and their positions were taken into account without refinement in structure-factor calculations with common isotropic temperature factor U = 0·072 (4) Å^2. A final

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