

NMR- und massenspektroskopische Messung des peracetylierten **1** bestimmt. Die β -glykosidische Verknüpfung ergab sich aus der Spaltbarkeit mit β -Glucosidase.²

Nach dem für Saponine üblichen Aufarbeitungswege gelang es nicht, aus *Folia Betula* Saponine zu isolieren, deren Vorkommen in der Literatur angegeben worden ist.^{3,4}

EXPERIMENTELLES

900 g Rohextrakt, bezogen von der Fa. H. Finzelberg's Nachfolger, Andernach, suspendierte man in H_2O , schüttelte zur Entfernung unpolarerer Substanzen mit $CHCl_3$ und anschließend mehrmals mit *n*-BuOH aus. Die BuOH-phasen wurden zur Trockne gebracht und der erhaltene Rückstand (165 g) zur Abtrennung der Farbstoffe über Al_2O_3 mit $CHCl_3$ -MeOH- H_2O (13:7:2)⁵ filtriert. Es fielen 77 g Substanzgemisch an, das an Kieselgel mit einem $CHCl_3$ -MeOH- H_2O -Gemisch steigender Polarität (13:4:2, 13:5:2, 13:7:2) chromatographiert wurde; Ausbeute: 2 g **1**.

3,4'-Dihydroxypropioiphenon. Schmp. 142° (Aceton); ($C_9H_{10}O_3$ (166,2), ber.: C, 65,06; H, 6,02, gef.: C, 65,19; H, 6,04%). UV (MeOH) λ_{max} : 276 nm, $\log \epsilon = 4,18$, 219 nm, $\log \epsilon = 4,04$. IR (KBr): 3430 und 3110 cm^{-1} (Hydroxyl), 1640 cm^{-1} (Carbonyl). Molmassenpeak bei $m/e = 166$.

3,4'-Dihydroxypropioiphenon diacetat. Schmp. 81° (Benzol/Petrol-äther); IR ($CHCl_3$): 1750, 1730 und 1670 cm^{-1} (CO). Molmassenpeak bei $m/e = 250$. NMR: (in $CDCl_3$, alle Angaben in ppm der δ -Skala): (a) 2,05 (s, 3H), (b) 2,35 (s, 3H), (c) 3,3 (t, J 6, 2H), (d) 4,5 (t, J 6, 2H), (e) 7,1–8,0 (2d, J 9, 4H); Zuordnung der Signale: (a) und (b): Acetylprotonen an C-3 und C-4', (c) und (d): Methylenprotonen an C-2 und C-3, (e): aromatische Protonen an den C-Atomen 2', 3', 5', 6'.

3,4'-Dihydroxypropioiphenon-3- β -D-glucopyranosid. Schmp. 158 – 164° (MeOH); $[\alpha]_D^{21} -20,1^\circ$ (c 1,0 MeOH); ($C_{15}H_{20}O_8$ (328,3), ber.: C, 54,88 H, 6,10, gef.: C, 55,17; H, 6,18%). UV (MeOH), λ_{max} : 278 nm, $\log \epsilon = 4,16$, 219 nm, $\log \epsilon = 4,0$, nach Zugabe von NaOMe: 326 nm, $\log \epsilon = 4,29$, 235 nm, $\log \epsilon = 3,73$; IR (KBr): 3320 cm^{-1} (OH), 1640 cm^{-1} (CO).

3,4'-Dihydroxypropioiphenon-3- β -D-glucopyranosid pentaacetat. IR ($CHCl_3$): 1750 und 1670 cm^{-1} (Carbonyl); Molmassenpeak bei $m/e = 538$; NMR ($CDCl_3$): (a) 1,9–2,1 (4s, 12H), (b) 2,3 (s, 3H), (c) 3,1–5,25 (m, 11H), (d) 7,2–8,1 (2d, 4H); Zuordnung der Signale: (a): Acetylprotonen der peracetylierten D-Glucose (b): Acetylprotonen an C-4', (d): aromatische Protonen an den C-Atomen 2', 3', 5', 6'. **1** und **2** gaben in wäßriger Lösung mit $FeCl_3$ eine violette Färbung.

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CUSCOHYGRINE: A CONSTITUENT OF THE ROOTS OF SOME BRITISH CONVULVULACEAE

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Key Word Index—*Calystegia sepium*; *Calystegia soldanella*; *Calystegia sylvestris*; *Convolvulus arvensis*; Convolvulaceae; bindweeds; cuscohygrine.

Plants. *Calystegia sepium* (L.) Roem. and Schult., "Larger bindweed". Roots collected as weed of cultivated land, early summer, white flowered variety; voucher specimen UNPH 101. *Calystegia sylvestris* (Willd.) Roem. and Schult., "Larger bindweed". Collected

as weed, early summer, pink-flushed flowers; voucher specimen UNPH 102. *Calystegia soldanella* (L.) R.Br., "Sea bindweed". Collected Godrevy Towans, Cornwall, late summer; voucher specimen UNPH 103. *Convolvulus arvensis* L., "Bindweed". Pink-flowered variety collected Nottingham University campus, summer; voucher specimen UNPH 104. Voucher specimens deposited in the Herbarium of the Museum of the Pharmaceutical Society of Great Britain, University of Bradford. *Previous work and uses.* *Calystegia soldanella*, purgative,¹ positive alkaloid test.² *Convolvulus arvensis*, phytochemical investigation of aerial parts.³ purgative.⁴ Cuscohygrine in *Convolvulus hamadae* and *C. lineatus*.⁵ Tropane ester alkaloids in *Convolvulus lineatus*, *C. pseudocantabricus*, *C. subhirsutus*.^{5,6}

Isolation of cuscohygrine. Dried, powdered rootstock and roots exhausted with EtOH in the presence of NH_4OH or $\text{Ca}(\text{OH})_2$ and H_2O . Purification of extracted basic material effected by the Stas-Otto procedure, by partition chromatography (kieselguhr supporting phosphate buffer solution, pH 6.0) or by preparative TLC (Si-gel GF₂₅₄, CHCl_3 - Et_2NH) of the evaporated alcoholic extract.

Calystegia sepium. Cuscohygrine. Dipicrate, m.p. and m.m.p. 223–224° (decomp.), satisfactory elemental analysis. IR and ms identical with those of authentic picrate.

Calystegia sylvestris. Cuscohygrine. Dipicrate, crude, m.p. 203–204° (decomp.) and m.m.p. 206–207° (decomp.), IR spectrum identical with that of authentic compound.

Calystegia soldanella. Cuscohygrine. Dipicrate, crude, m.p. 202–204° (decomp.), m.m.p. 204–205° (decomp.), IR and ms identical with those of authentic compound.

Convolvulus arvensis. Cuscohygrine. Dipicrate, crude, m.p. 203–204° (decomp.), m.m.p. 207–208° (decomp.), IR spectrum identical with that of authentic material.

Cuscohygrine commonly, although not exclusively, occurs in association with tropane alkaloids in many species of the Solanaceae and in the genus *Erythroxylum*. A few species (*loc. cit.*) of *Convolvulus* also contain tropane ester alkaloids and in at least one case cuscohygrine has been shown to co-occur. We have been unable to detect tropane alkaloids in the underground organs of the British Convolvulaceae studied but in all cases cuscohygrine appears to be a characteristic component. The possible more widespread occurrence of this base as a single basic component is at present under investigation.

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