followed by condensation of the resulting 6-amino-1,3-dialkyl-5-nitrosouracil with unchanged II (or V). On the other hand, treatment of I alone with N-nitrosodimethylamine under the same conditions gave III (65%) as the sole product.

The reaction of I and 6-benzylamino-1,3-dimethyluracil in N-nitrosodimethylamine gave a mixture of III (76%) and 8-phenyltheophylline (VII)[8] (84%).

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Synthesis of Viscosic Acid[1]

Recently we[2] have proposed the structures of viscosin (I), an antibiotic active to tubercle bacillus and virus, and of viscosic acid (II), the saponification product of I [mp 179—181°, $[\alpha]_D$ −13°; $[\alpha]_D$ −10° (c=1, EtOH)], by the degradative and physical methods. We have now synthesized D-3-hydroxydecanoylnonapeptide (II) and identified it with viscosic acid derived from the natural source.

D-Dec(OH)–Leu–D-Glu–D-*a*Thr–D-Val–Leu–D-Ser—
O–Ile←D-Ser←Leu← (I)

D-Dec(OH)–Leu–D-Glu–D-*a*Thr–D-Val–Leu–D-Ser–Leu–D-Ser–Ile–OH (II)

pMZ-Leu-D-Ser-Leu-D-Ser-Ile-OBzl (III),[3] mp 160—162°, $[\alpha]_D$ −22°, Rf_1 0.40, was prepared in 41% overall yield by repeated azide couplings with pMZ-Leu-D-Ser-$NHNH_2$ [mp 200.5—201° (decomp.), $[\alpha]_D$ −5°] starting from H-Ile-OBzl. pMZ-D-*a*Thr-D-Val-Leu-D-Ser-Leu-D-Ser-Ile-OBzl (IV), mp 188—189°, $[\alpha]_D$ −28°, Rf_1 0.43 [prepared (62%) by azide coupling of pMZ-D-*a*Thr-D-Val-$NHNH_2$, mp 245—245.5° (decomp.), $[\alpha]_D$ +20° (c=0.5, AcOH), with H-Leu-D-Ser-Leu-D-Ser-Ile-OBzl obtained by the acidolysis of III] was treated with cold trifluoroacetic acid in the presence of anisole to remove the pMZ-group and then coupled

1) Unless otherwise noted, amino acid symbols denote the L-configuration and $[\alpha]_D$ refers to 1% solution in DMF at 18—20°. The following abbreviations are used: Dec(OH)=3-hydroxydecanoyl-; pMZ=*p*-methoxybenzyloxycarbonyl; OBzl=benzyl ester; ONp=*p*-nitrophenyl ester; N,N′-dicyclohexylcarbodiimide. Thin-layer chromatography (TLC) was carried out on Kieselgel G (Merck) with the solvent systems of $CHCl_3$–MeOH–AcOH (95:5:3 v/v, Rf_1), *n*-BuOH–AcOH–H_2O (4:1:5 v/v, Rf_2) and AcOEt–pyridine–AcOH–H_2O (60:20:6:11 v/v, Rf_3).
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3) Satisfactory elementary analyses were obtained for all crystalline compounds.

with pMZ-D-Glu(γ-OBzl)-ONp (mp 125—126°, $[\alpha]_D$ +24°, Rf_1 0.92) to yield pMZ-D-Glu(γ-OBzl)-D-*a*Thr-D-Val-Leu-D-Ser-Leu-D-Ser-Ile-OBzl (V) (81%), mp 194—195°, $[\alpha]_D$ −16.5, Rf_1 0.32. After elimination of the pMZ-group by the method as described in IV, the resulting octapeptide was condensed with D-3-hydroxydecanoyl-Leu-$NHNH_2$[4] *via* the corresponding azide. The resulting protected N-acylnonapeptide D-Dec(OH)-Leu-D-Glu (γ-OBzl)-D-*a*Thr-D-Val-Leu-D-Ser-Leu-D-Ser-Ile-OBzl (VI) was isolated by column chromatography on silica gel using $CHCl_3$–MeOH–AcOH (95:5:3 v/v) as a solvent; 74%, mp 184—186°, $[\alpha]_D$ −18°, Rf_1 0.30. Finally the removal of the protecting group from VI by catalytic hydrogenation furnished D-Dec (OH) -Leu-D-Glu-D-*a*Thr-D-Val-Leu-D-Ser-Leu-D-Ser-Ile-OH (II) (73 %), mp 181—182°, $[\alpha]_D$ −10°; $[\alpha]_D$ −9.5° (c=1, EtOH) [*Anal.* Calcd. for $C_{54}H_{97}O_{17}N_9 \cdot 2H_2O$: C, 54.95; H, 8.64; N, 10.68. Found: C, 54.94; H, 8.57; N, 10.46. IR ν_{max}^{KBr} cm^{-1}: 1715 (C=O), 1630 (amide I), 1530 (amide II). Mass spectrum of the dimethyl ester *m/e*: 1171 (M^+). Amino acid ratios in acid hydrolysate (6N HCl, 110°, 24 hr): Glu 1.01, *a*Thr 0.89, Ser 1.78, Val 1.03, Ile 1.00, Leu 2.98], which gave a single spot (iodine-toluidine reaction) on TLC in different solvent systems (Rf_1 0.04, Rf_2 0.85, Rf_3 0.70) and was shown to be identical with viscosic acid obtained from the natural specimen in mp, mixed mp, $[\alpha]_D$, TLC, infrared (KBr) and mass spectra.

The dimethyl ester of the synthetic viscosic acid was prepared by CH_2N_2 treatment, which showed 203—205° and $[\alpha]_D$ −11°; the physical constants were identical with those of an authentic sample of viscosic acid dimethyl ester.

Confirmation of the structure of viscosic acid thus presents strong support to the assignd structure (I) of viscosin.

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On the Tentative Structure of Wilforine

In 1966, Mitsuhashi, *et al.* reported the components of *Cynanchum Wilfordi* Hemsley (Japanese name Koikema), Asclepiadaceae family, containing polyoxypregnane ester glycosides.[1] They isolated three polyoxypregnane derivatives, sarcostin (I),[2] deacylmetaplexigenin (II),[3] and lineolon (III)[2] from the plants, after the alkali hydrolysis of the ester type aglycone mixture. In this communication, we wish to describe the separation and the structure of a new aglycone, wilforine (IV) from the ester type aglycone mixture.

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