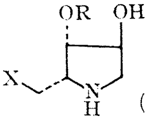
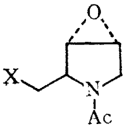
Synthesis of *N*-Acetyl Deacetyl Anisomycin Isomers

By M. SALMÓN and F. WALLS*

(*Instituto de Química de la Universidad Nacional Autónoma de México, México* 20, *D.F.*)



(I)



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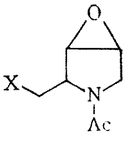

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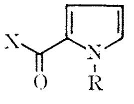
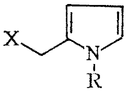
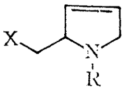


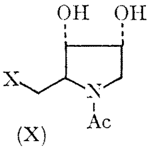
(IV) R=H
(V) R=CH₂Ph



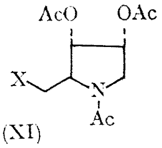
(VI) R=H
(VII) R=CH₂Ph



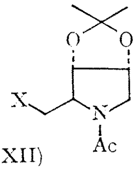
(VIII) R=Ac
(IX) R=CH₂Ph



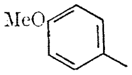
(X)



(XI)



(XII)

X=


WE report work on compounds closely related to anisomycin (I; R = Ac),[1,2] as possible antibiotics. The (±)-isomer (*trans-cis*) (XII) of deacetylanisomycin (1; R = H) has been synthesised as follows. 2-*p*-Anisoylpyrrole[1] (IV), m.p. 111—112°, by Wolf–Kishner reduction, gave 2-anisylpyrrole[1] (VI) (83%, b.p. 118—120°/0·05 mm.) which, with Zn–Hg and conc. HCl–MeOH, and after removal of solvent under reduced pressure and treatment with an excess of pyridine and acetic anhydride, gave the *N*-acetyl compound (IX) [b.p. 178°/0·01 mm.; M^+ 231; n.m.r. (CCl_4)†: 337 (s. 2H 3-pyrroline system) and 114·5 and 120·5, relative intensities 100 : 23, total absorption integrating for 3H, acetyl group].

The *N*-acetyl-3-pyrroline (VIII), with 30% H_2O_2 (Me_2CO–Et_2O with OsO_4 as catalyst[4] gave the *cis*-glycol (X), which, after chromatography in silica gel, melted at 135·5—136. Compound (X) could be acetylated with pyridine and acetic anhydride, to yield (XI). The acetonide (XII), M^+ 305, could be formed with acetone and toluene-*p*-sulphonic acid. When (X) was tested against six bacteria, seven fungi, and one yeast (*Saccharomyces cerevisiae*) it was totally inactive up to a level of 100 mg. per ml.

A new series of *N*-benzyl compounds was made. Thus (IV), with sodamide (hot toluene) and benzyl chloride gave

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† N.m.r. values in c./sec. determined in a varian A-60-A machine with Me_4Si as internal reference.

(V), which on Wolff-Kishner reduction (or Zn–Hg and HCl–MeOH) produced the corresponding *N*-benzyl-3-pyrroline. This compound was readily oxidized, reverting to the parent pyrrole (VII).

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[2] J. P. Schaefer and P. J. Wheatley, *Chem. Comm.*, 1967, 578; *J. Org. Chem.*, 1968, **33**, 166; K. Butler, *ibid.*, p. 2136.

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