

proportion of moderately acid albumins than the other species. The proportion of basic, non-adsorbing proteins is smaller in oat (20 %) than in the other species (winter wheat 36 %, summer wheat 33 %, rye 32 % and barley 32 %).

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Synthesis of an Oxime Analogue to Atropin

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Atropin is the drug of choice in treatment of organophosphorus anticholinesterase poisoning. Very promising results have also been obtained with oximes such as pyridine-aldoxime methiodide or mono-*iso*-nitrosoacetone¹. It seemed thus, without any pretention of strict pharmacological thinking, tempting to prepare an oxime closely analogous to atropin such as phenylglyoxylic acid tropylester oxime, see formulae I–III.

Iso-nitrosation by butyl nitrite was used as a final step in the synthesis. This meth-

od may result in *iso*-nitrosation of other groups than the methylene group of the phenyl-acetic acid. Thus the structure of the final product was studied by infrared spectroscopy.

Results. Phenyl-glyoxylic acid tropylester oxime has been prepared and the structure of the compound has been confirmed by the following results from IR-spectra.

Phenyl-glyoxylic acid ethylester oxime. The 3 500–2 500 cm⁻¹ region: An absorption band at 3 220 cm⁻¹ can be ascribed to intramolecular bonded OH. Between 3 180 and 2 990 cm⁻¹ the CH absorption bands are found.

The 1 800–1 600 cm⁻¹ region: At 1 725 cm⁻¹ a strong absorption band can be ascribed to C=O and at 1 685 cm⁻¹ a weaker band may indicate presence of C=N.

The 1 600–1 400 cm⁻¹ region: At 1 575 cm⁻¹ and 1 490 cm⁻¹ weak absorption bands characteristic of the benzene ring in a conjugated system are found.

The 1 400–1 100 cm⁻¹ region: At 1 300 cm⁻¹ an absorption band occurs which might be ascribed to OH and at 1 195 a strong band indicates ester C–O.

Phenyl-glyoxylic acid tropylester oxime. The 3 500–1 800 cm⁻¹ region: An absorption band at 2 990 can be ascribed to CH. At 2 800–2 200 cm⁻¹ and 2 100–1 800 two broad bands occur which can be ascribed to ≡NH.

The 1 800–1 600 cm⁻¹ region: At 1 710 a strong absorption band can be ascribed to C=O and at 1 665 a weak band may be ascribed to C=N.

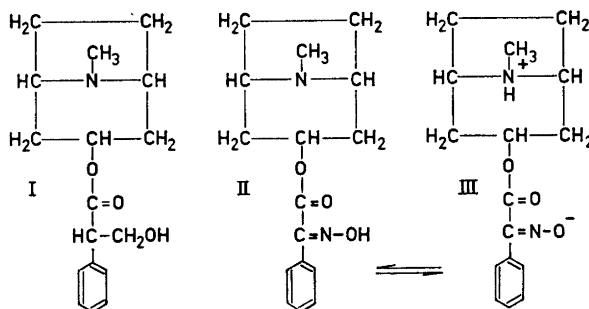


Fig. 1. I is atropin. II and III are protomeric forms of phenyl-glyoxylic acid tropylester oxime.

The 1 600–1 400 cm^{-1} region: At 1 575 cm^{-1} and 1 500 cm^{-1} weak absorption bands characteristic of the benzene ring in a conjugated system are found.

The 1 400–1 100 cm^{-1} region: At 1 290 cm^{-1} an absorption band occurs which might be ascribed to OH and at 1 205 a strong band indicates ester C–O.

The IR-spectra and earlier findings² strongly support the structure given in the preceding formulae. The absorption bands between 2 800 and 1 800 cm^{-1} show that formula III is dominating. Preliminary pharmacological tests indicate that phenyl-glyoxylic acid tropylester oxime has a weak atropin effect.

Experimental. The syntheses were performed as follows.

Phenyl-glyoxylic acid ethylester oxime was prepared as described by Wislicenus and Grützner². Found m.p. 112°C.

Phenyl-acetic acid tropylester was prepared as described by Barrowcliff and Tutin³. Found m.p. of picrate 171°C⁴.

Phenyl-glyoxylic acid tropylester oxime was prepared as follows. 4.4 g of phenyl-acetic acid tropylester and 1.75 g butyl nitrite in 100 ml sodium distilled ether at -20°C were added

to potassium ethoxide in 50 ml of ether at -20°C , prepared from 0.66 g potassium. The mixture was stirred for 45 min and temperature rose to 0°C . After cooling 30 ml of icecooled water were added. After shaking, the aqueous phase was separated and treated with carbon dioxide while cooling. After 3 min a yellow red precipitate was formed. The precipitate was filtered off and washed with water and ether. Yield 0.75 g. The compound was recrystallized twice from ethyl acetate. M.p. 196°C . (Found: C 66.8; H 7.0; N 9.8. Calc. for $\text{C}_{16}\text{H}_{20}\text{N}_2\text{O}_3$ (288.3): C 66.7; H 7.0; N 9.7).

Infrared spectra of phenyl-glyoxylic acid tropylester oxime and phenyl-glyoxylic acid ethylester oxime were recorded using potassium bromide pellets.

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