proportion of moderately acid albumins than the other species. The proportion of basic, non-absorbing proteins is smaller in oat (20 %) than in the other species (winter wheat 38 %, 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 organophosphorous anticholinesterase poisoning. Very promising results have also been obtained with oximes such as pyridine-aldoxime methiodide or mono-isonitrosocetone. 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 method 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 intra-molecular 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 cm⁻¹ 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 \( \equiv \text{NH} \).

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.

\[
\begin{align*}
\text{H}_2\text{C} & \quad \text{CH}_2 \\
\text{H} & \quad \text{C} \quad \text{N} \quad \text{CH} \\
\text{H}_2\text{C} & \quad \text{CH} \quad \text{CH}_2 \\
\text{H} & \quad \text{C}=\text{O} \\
\text{H} & \quad \text{C}=\text{O} \\
\text{H} & \quad \text{C}=\text{N} \quad \text{OH} \\
\text{H} & \quad \text{C}=\text{N} \quad \text{OH} \\
\end{align*}
\]

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

The $1600 - 1400$ cm$^{-1}$ region: At $1575$ cm$^{-1}$ and $1500$ cm$^{-1}$ weak absorption bands characteristic of the benzene ring in a conjugated system are found.

The $1400 - 1100$ cm$^{-1}$ region: At $1290$ cm$^{-1}$ an absorption band occurs which might be ascribed to OH and at $1205$ a strong band indicates ester C=O.

The IR-spectra and earlier findings strongly support the structure given in the preceding formulae. The absorption bands between 2800 and 1800 cm$^{-1}$ show that formula III is dominating. Preliminary pharmacological tests indicate that phenylglyoxylic 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 Grützner. Found m.p. 112°C.

Phenyl-acetic acid tropylester was prepared as described by Barrowcliffe and Tutin. Found m.p. of picrate 171°C.

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 ice-cooled 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. Calcd. for $\text{C}_{14}\text{H}_{16}\text{N}_{2}\text{O}_{3}$ (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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