A COLOR REACTION OF STEROID AGLYCONES OF THE SPIROSTAN SERIES WITH SULFURIC ACID SOLUTIONS

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UDC 543.857.6+547.918

We have used color reaction of pennogenin (17α-diosgenin) with 72% aqueous sulfuric acid catalyzed by Fe(III) ions [1] to determine diosgenin. It was found that the absorption spectra (300-700 nm) of diosgenin in aqueous solutions of acid, like those of pennogenin, have two maxima at 413 and 486 nm the intensities of which depend on the concentration of the acid and the reaction conditions.

Thus, the most intense maximum with $\varepsilon_{413} = 12 \cdot 10^3$ is observed in the reaction of diosgenin with 96% acid at 60°C for 30 min, while ε_{486} reaches its maximum value, of $23 \cdot 10^3$, under the conditions of the reaction of diosgenin with a 72% aqueous solution of the acid in the presence of catalytic amounts (0.0001%) of Fe(III). The coloration develops at 50°C in 60 min and remains unchanged for not less than 5 h. A calibration graph is linear in the range of 0.01-0.1 mmole of diosgenin in 4 ml of acid.

With Fe(III) ions the coloration develops very slowly; the addition of large amounts (0.01%) of Fe(III) is accompanied by the appearance of a bright crimson coloration; the maximum at 486 nm in the absorptions spectrum disappears and a new maximum appears at 540 nm but under these conditions ε_{540} reaches only $14 \cdot 10^3$.

The use of ethanol as a diluent of the acid in the reaction of pennigenin and diosgenin (optimum conditions: 96% acid–ethanol (1.5:1, by volume), 60°C, 30 min, 0.01-0.1 μmole of aglycone in 4 ml of acid) cannot be recommended since the replaced of water by ethanol gives no advantages in sensitivity ($\varepsilon_{486} = 18 \cdot 10^3$) and Beer's law is not observed in the given range of concentrations.

On turning to literature information, it can be seen that the reaction with 72% acid described here is more sensitive than the known color reactions for diosgenin [2-5], the maximum value of ε_{max} of which does not exceed $13 \cdot 10^3$. The comparatively mild conditions and high sensitivity permit diosgenin to be determined in a combined glycoside fraction without passing through a hydrolysis stage, or the individual glycoside to be determined after chromatographic separation.

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 0009-3130/83/1902-0238$07.50