yellow oil with a sharp smell, was distilled under reduced pressure in a current of nitrogen. This gave a clear yellow oil (7.5 g), which rapidly became red in the air and even on storage under nitrogen.


## SUMMARY

It has been shown that the anhydrolupinine obtained from lupinine by the action of water-abstracting agents is a mixture of two isomers differing by the position of a double bond in the ring system of the molecule.


## LITERATURE CITED


1. G. Clemo and P. Raper, J. Chem. Soc., 1927 (1929).
2. P. Karrer and A. Vogt, Helv. Chim. Acta, B, 1073 (1930).
3. E. Whitte, New Zealand, J. Sci. Technol., B, 33, 47 (1951).
4. Heterocyclic Compounds [in Russian], Vol. 3, Moscow (1954), p. 314.
5. G. Clemo and G. Ramage, J. Chem. Soc., 437 (1931).
6. R. Willstätter and E. Fourneau, Ber., 35, 1910 (1902).


# ELECTROCHEMICAL EXTRACTION OF ALKALOIDS OF THE TROPANE GROUP FROM PLANT RAW MATERIAL


E. A. Vdoviko, S. A. Pokhmelkina, and O. R. Pryakhin



UDC 547.944/945+541.138


Electrodialysis is an extremely promising method of isolating alkaloids from plant raw material, since it leads to the isolation of a purified product, avoids the use of organic solvents, and shortens the technological process [1]. The aim of the present work was to investigate further the conditions for extracting alkaloids of the tropane group from acid aqueous extracts of the leaves of *Atropa belladonna* (Z.).

To study the conditions of isolating the alkaloids we used model systems with concentrations of aqueous solutions of atropine sulfate approximately equal to the amount of the alkaloids in the plant raw material. The use of model systems permitted the influence of the impurities present in acid extracts of belladonna on the process of electrodialysis to be excluded. In addition to traditional methods [2], the possibility has been considered of using conductometric and photonephelometric methods for determining the concentration of atropine sulfate in model systems. The amounts of atropine salt in the initial solutions and the catholyte were determined from the characteristic inflection on the conductometric titration curve (Fig. 1). In the photonephelometric method, the optical densities of the solutions were measured at a wavelength of 430 nm, gelatin being used as a stabilizer for the system.

The experimental results show that the size of the current has a fundamental influence on the process of extracting atropine into the catholyte when using model systems. Thus, for example, at an initial concentration of atropine salt of $0.6 \cdot 10^{-3}$ kmole/m$^3$ the amount of alkaloid that passed into the catholyte in the first 10 min of the experiment was $0.08 \cdot 10^{-3}$ kmole/m$^3$ at 0.1 A and $0.24 \cdot 10^{-3}$ kmole/m$^3$ at 0.5 A. With a further increase in the current, the amount of alkaloid fell because of a sharp rise in the temperature of the solution (to 60°C) and its boiling, while small currents caused a considerable lengthening of the electrodialysis process. In view of this, we checked the dependence of the degree of isolation of the alkaloid on the time of electrodialysis at an optimum current of 0.5 A.


Zaporozh'e State Medical Institute. Translated from Khimiya Prirodnykh Soedinenii, No. 4, pp. 497-500, July-August, 1976. Original article submitted December 23, 1975.

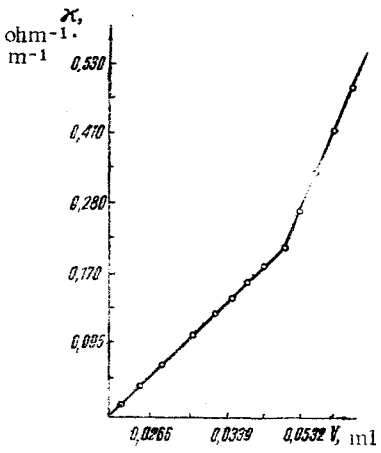


Fig. 1

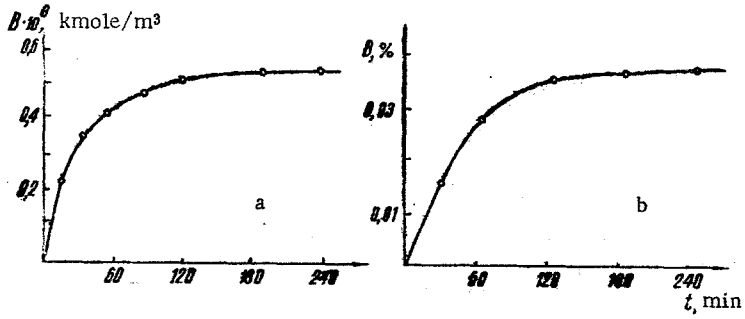


Fig. 2

Fig. 1. Conductometric titration curve (titrant 0.01 N KOH).

Fig. 2. Dependence of the yield of alkaloid on the time of electrodialysis: a) for atropine sulfate; b) for a tincture of belladonna (B — degree of extraction; t — time of electrodialysis).

After the lapse of a predetermined time of electrodialysis (120 min), the yield of alkaloid into the catholyte ceased to change appreciably with time (Fig. 2a). The inflection in the curve enables the optimum time for the performance of the process to be determined.

As observed previously [2], during electrodialysis the catholyte becomes alkaline as a result of the electrode reaction, and therefore the process was performed at a controlled pH not exceeding 5.5 to prevent the precipitation of the alkaloid.

The completeness of the extraction of the alkaloid at 0.5 A was 92% (see Fig. 2a), and, as quantitative analysis showed, the residual amount of alkaloid was present in the central part of the cell, no adsorption of the alkaloid on the membrane being observed.

The laws of the process of extracting atropine in model systems that were found enabled us to carry out an investigation of the isolation of alkaloids of the tropane group from plant raw material. The initial solutions were acid aqueous extracts of belladonna. The quantitative analysis of the amounts of alkaloids in the extracts and the catholyte was performed by the volume-extraction method [2] using potentiometric titration in the concluding stage of the analysis. The amount of alkaloids in the acid extracts, calculated as atropine, was 0.05 ± 0.003%.

The dependence of the degree of extraction of the alkaloid from the plant raw material on the time of electrodialysis was similar to the same dependence for the model systems (see Fig. 2b), which may show the achievement of a limiting yield of alkaloids with time under the given conditions of performing the process, amounting to about 70%, the optimum current being 0.7 A and the time of electrodialysis 120 min.

## EXPERIMENTAL

Electrodialysis was performed in a three-chamber cell with a capacity of 300 $cm^3$ in which the cathode and anode regions were separated. The solutions under investigation were placed in the central part of the cell separated by parchment semipermeable membranes from the cathode and anode spaces, which were filled with a 2% solution of sulfuric acid and with distilled water, respectively. The electrodes were graphite plates (with a surface area of 10 $cm^2$). The source of constant current was a type VSA-5A selenium rectifier. The measurements were performed at currents of from 0.1 to 1 A. For conductometric analysis we used a P-568 alternating-current bridge with a conductometric cell the constant of which was 61 $m^{-1}$ and with platinized platinum electrodes. The measurements were thermostatted at 25 ± 0.02°C, and the titrant was a 0.01 N solution of potassium hydroxide.

The photonephelometric analysis was performed on an FÉK-60 photocolorimeter. For potentiometric titration and for monitoring the pH of the catholyte we used a type LPM-60M pH-meter with 0.01 N potassium hydroxide solution as the titrant.

## SUMMARY

1. The possibility has been shown of studying the isolation of alkaloids from model systems with a quantitative yield of salt of up to 92%.

2. The alkaloids of the tropane group have been isolated from acid extracts of belladonna by the electrochemical method. The conditions ensuring the maximum yield of alkaloids into the catholyte as a function of the time of electrodialysis have been determined.

## LITERATURE CITED

1. S. Kh. Babich, Zh. Prikl. Khim., 652 (1947); T. P. Dorosh and V. S. Alekseev, Farmats. Zh., No. 6, 44 (1960); G. K. Nikonov, Aptechnoe Delo, No. 1, 13 (1955).
2. E. A. Vdoviko, S. A. Pokhmelkina, and V. V. Petrenko, Khim. Prirodn. Soedin., 334 (1972).