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THIN-LAYER CHROMATOGRAPHY OF ALKALOIDS ON POLYAMIDE

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To detect the separated substances in chromatography on a thin film of polyamide, many of the usual methods of detection are used. However, polyamide films withstand only mild, nondestructive methods of detection, excluding such aggressive reagents as concentrated acids and alkalis. For this purpose it is possible to use color reactions of various mechanisms (azo coupling [1], oxidation-reduction [2], electrophilic addition [3]), and, in addition, such properties as the intrinsic fluorescence of the substances or their derivatives [4], luminescence [5], and also the quenching of fluorescence [6] are widely employed.

We give information on the separation of alkaloids in a thin film of polyamide with their subsequent localization from the quenching of fluorescence. The polyamide films were treated with a 0.005% alkaline solution of fluorescein in butanol for a day and wer dried in the air. The separated substances were detected in the form of dark zones on the yellow-green-fluorescing background on examination in UV light (254 nm). The mobile phases used were ethyl acetate–cyclohexane–methanol–25% ammonia in the following proportions: (70 : 15 : 10 : 5) (1), (100 : 15 : 10 : 5) (2), (70 : 20 : 10 : 5) (3), and (70 : 15 : 2.5 : 5) (4).

The R_f values of the alkaloids in these systems are given below:

Alkaloid	1	2	3	4
Atropine	0.84	0.91	0.91	0.80
Delsemine	0.56	0.68	0.84	0.50
Delsoline	0.93	0.84	0.89	0.80
Codeine	0.70	0.73	0.86	0.43
Condelphine	0.89	0.91	0.93	0.78
Caffeine	0.84	0.84	0.95	0.95
Methyllycaconitine	0.77	0.86	0.91	0.86
Morphine	0.57	0.55	0.68	0.39
Narcotine	0.93	0.95	0.91	0.95
Papaverine	0.91	0.91	0.89	0.84
Pilocarpine	0.80	0.77	0.84	0.66
Salsoline	0.68	0.68	0.80	0.55
Scopolamine	0.91	0.94	0.91	0.84
Spasmolytin (adephenine)	0.98	0.98	0.99	0.98
Theobromine	0	0	0	0.84
Theophylline	0.75	0	0.32	0
Elatine	0.86	0.91	0.93	0.93
Eldeline (deltaline)	0.81	0.89	0.91	0.86
Rphedrine	0.77	0.86	0.89	0.73

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Sedum ALKALOIDS

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The alkaloids of Sedum acre (goldmoss stonecrop) have been considered in various publications [1-5]. We have investigated the alkaloid compositions of the epigeal parts of four Siberian species of Sedum: S. aizoon L. (aizoon stonecrop), S. purpureum (L.) Shult. (purple stonecrop), S. hydridum L. (evergreen stonecrop), and S. ewersii Ledeb. (Ewers stonecrop), collected in the Tomsk oblast (village of Urtam) and in the Gorno-Altai Autonomous Region (villages of Maima and Kebezen') in the flowering phase.

The combined bases were isolated by Franck's method [4]. TLC on plates coated with Al_2O_3 in the benzene–methanol (85 : 15) (system 1) and ethyl acetate–chloroform–methanol (2.5 : 2.0 : 0.5) (system 2) systems showed that the first three species have the same qualitative composition (each containing three bases), while the chloroform extract from S. ewersii revealed the presence of only one alkaloid (III). The alkaloids were separated on comumn of alunina (activity grade II, neutral) being eluted successively with benzene and mixtures of benzene with 5-25% of methanol.

Base (I) was isolated in the form of a colorless oily residue with R_f 0.89 (system 1) and 0.86 (system 2) and gave a picrate with mp 156-158°C. IR spectrum, cm^{-1}: 2950 (CH_3), 1690 ($C=O$). On the basis of these results, the substance was identified as (±)-methlyisopelletierine [6].

Base (II), mp 119-121°C, R_f 0.28 (system 1) and 0.75 (system 2) decolorized a solution of potassium permanganate and bromine water. Its IR spectrum showed absorption bands at (cm^{-1}) 3385 (OH), 2935 (CH_3), 1630 (π-bond), 1590 (aromatic ring), and 760, 745 (monosubstituted benzene nucleus). It formed a hydrochloride with mp 169-170°C, $[\alpha]_D^{20} - 140°C$ (c 0.04; CH_3OH). The base was identified as (−)-sedinine [7].

Base (III), mp 89-90°C, R_f 0.22 (system 1) and 0.61 (system 2). IR spectrum, cm^{-1}: 3280 (OH), 2955 (CH_3), 1570, 1485 (benzene ring). The substance formed a hydrochloride with mp 186-188°C. The results obtained permitted the conclusion that base (III) was (±)-sedamine [4, 8]. A direct comparison with (±)-sedamine confirmed their identity.

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