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ALKALOIDS AND OTHER LOW-MOLECULAR-WEIGHT METABOLITES FROM *Crambe kotschyana*

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The chemical composition of the plant *Crambe kotschyana* Boiss. [1, 2] has not been previously studied. We first studied alkaloids and other low-molecular-weight metabolites from the aerial part of *C. kotschyana* growing in Uzbekistan [3]. The presence of alkaloids in this plant was first established by us [4, 5].

In continuation of this work, total alkaloids obtained by extraction with $CHCl_3$ (fraction 1), work up of the alkaline solution after isolating total tertiary bases with EtOAc (fraction 2) and *n*-BuOH (fraction 3), and drying of the remaining alkaline solution to dryness (fraction 4) produced four fractions that were analyzed by GC–MS.

The first fraction (total alkaloids) contained goitrin (**1**), 3-methyldodecane (**2**), and tetradecyliodide (**3**). The second fraction (*n*-BuOH total) included three neutral compounds, methyloleate (**4**), methylpalmitate (**5**), and 1,3-dimethoxybutane (**6**) and probably 5-dimethylhexan-2-ol.

Two compounds, *N*-methylaminoheptane (**7**) and 5-hydroxymethyl-2-furancarboxaldehyde (**8**) were found in the third fraction.

The fourth fraction was obtained from the alkaline solution by work up with EtOAc. GC–MS analysis showed the probable presence of two compounds, coumaran (dihydrobenzofuran) and 2,4-dimethylthietane [CH_3–($CHCH_2CHS$) –CH_3] with retention times of 6.595 and 27.337, respectively [6].

GC–MS of the plant constituents was performed on an Agilent 7890A GC with an Agilent 5975C Inert MSD quadrupole mass-spectrometric detector. We used a quartz capillary column (30 m × 250 μm, 0.25 μm stationary film thickness), He carrier gas, flow rate 1 mL/min (splitless), injector temperature 280, programmed furnace from 180 to 280°C, and sample volume 1 μL. Compounds were identified by comparison of retention times and mass spectra with data in the W8N05ST.L electronic library. Column chromatography of the obtained $CHCl_3$ total alkaloids and fractions was carried out using KSK silica gel adsorbent.

Extraction and isolation of total alkaloids were performed by the usual procedure of extracting ground aerial part with $CHCl_3$ with preliminary wetting of the raw material with ammonia solution (5%), condensing the combined $CHCl_3$ extracts, working up with H_2SO_4 (10%), washing with hydrocarbons, making basic with conc. ammonia solution to pH 10–11, treating once with $CHCl_3$, condensing the extract, and drying the residue.

Preparation of Fractions from Total Substances Extracted from *C. kotschyana*. The $CHCl_3$ total alkaloids isolated from the aerial part of *C. kotschyana* (6.0 kg) was chromatographed over a column of silica gel with elution by hydrocarbons:$CHCl_3$ (1:1) to produce five fractions (1–5) with similar R_f values. The combined effluents of the first fraction were analyzed by GC–MS (data given in Table 1).

After separating total tertiary bases, the alkaline solution was worked up first with EtOAc and then *n*-BuOH. The BuOH fraction contained according to TLC (detection by I_2 vapor) 5–6 minor compounds. This part of the fraction was chromatographed over a column of silica gel with elution by $CHCl_3$ and a $CHCl_3$:MeOH gradient. The first fractions of the $CHCl_3$:MeOH mixture (100:1) contained compounds with similar R_f values and were combined. A second fraction was obtained and analyzed by GC–MS (Table 1).

The alkaline solution remaining after work up with *n*-BuOH was evaporated to dryness to produce a dry residue (third fraction), which was analyzed by GC–MS (Table 1).

Work up of the alkaline solution with EtOAC produced total extracted substances (fourth fraction). Table 1 presents data from the GC–MS analysis.

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TABLE 1. GC–MS Analysis of Fractions from the Aerial Part of *Crambe kotschyana*, Content, %

Compound	Retention time	%
5-Vinyl-2-oxazolidinethione (goitrin)	17.875	63.42
3-Methyldodecane	7.985	19.13
Tetradecyliodide	8.895	9.13
Methyloleate	15.326	36.79
1,3-Dimethoxybutane	8.664	15.37
Methylpalmitate	13.976	9.15
N-Methylaminoheptane	15.773	77.89
5-Hydroxymethyl-2-formylfuran	7.99	14.58

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