

FISHERIES AND MARINE SERVICE

Translation Series No. 4168

Gas chromatographic analysis of isovaleric acid esters

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Original title: Gazo-khromatograficheski analiz efirov izovalerianovoi kisloty

From: Dostizh. Razvit. Nov. Metodov Khim. Anal. 3-4 p., 1974

Translated by the Translation Bureau (JW)
Multilingual Services Division
Department of the Secretary of State of Canada

Department of Fisheries and the Environment
Fisheries and Marine Service
Halifax Laboratory
Halifax, N.S.

1977

3 pages typescript

DEPARTMENT OF THE SECRETARY OF STATE
TRANSLATION BUREAU

MULTILINGUAL SERVICES
DIVISION



SECRETARIAT D'ÉTAT
BUREAU DES TRADUCTIONS

DIVISION DES SERVICES
MULTILINGUES

TRANSLATED FROM - TRADUCTION DE

Russian

INTO - EN

English

AUTHOR - AUTEUR

T.P. Gukalova and others

TITLE IN ENGLISH - TITRE ANGLAIS

Gas chromatographic analysis of isovaleric acid esters

TITLE IN FOREIGN LANGUAGE (TRANSLITERATE FOREIGN CHARACTERS)
TITRE EN LANGUE ÉTRANGÈRE (TRANSCRIRE EN CARACTÈRES ROMAINS)

Gazo-khromatograficheskii analiz efirov izovalerianovoi kisloty

REFERENCE IN FOREIGN LANGUAGE (NAME OF BOOK OR PUBLICATION) IN FULL. TRANSLITERATE FOREIGN CHARACTERS.
RÉFÉRENCE EN LANGUE ÉTRANGÈRE (NOM DU LIVRE OU PUBLICATION), AU COMPLET, TRANSCRIRE EN CARACTÈRES ROMAINS.

Dostizh. Razvit, Nov. Metodov Khim. Anal.

REFERENCE IN ENGLISH - RÉFÉRENCE EN ANGLAIS

New Methods of Chemical Analysis

PUBLISHER - ÉDITEUR	DATE OF PUBLICATION DATE DE PUBLICATION			PAGE NUMBERS IN ORIGINAL NUMÉROS DES PAGES DANS L'ORIGINAL
	YEAR ANNÉE	VOLUME	ISSUE NO. NUMÉRO	
Not given				3--4
PLACE OF PUBLICATION LIEU DE PUBLICATION				NUMBER OF TYPED PAGES NOMBRE DE PAGES DACTYLOGRAPHIÉES
USSR	'74	Not given	Not given	3

REQUESTING DEPARTMENT
MINISTÈRE-CLIENT

Environment

TRANSLATION BUREAU NO.
NOTRE DOSSIER N°

1448525

BRANCH OR DIVISION
DIRECTION OU DIVISION

Scientific Information and
Publications Branch

TRANSLATOR (INITIALS)
TRADUCTEUR (INITIALES)

JW

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DEMANDÉ PAR

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YOUR NUMBER
VOTRE DOSSIER N°

5053-1

DATE OF REQUEST
DATE DE LA DEMANDE

12. 12. 77

UNEDITED TRANSLATION
For information only
TRADUCTION NON REVISEE
Information seulement

JAN 23 1978



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DIVISION DES SERVICES
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CLIENT'S NO. N° DU CLIENT	DEPARTMENT MINISTÈRE	DIVISION/BRANCH DIVISION/DIRECTION	CITY VILLE
	Environment	Fisheries	Halifax, N.S.
BUREAU NO. N° DU BUREAU	LANGUAGE LANGUE	TRANSLATOR (INITIALS) TRADUCTEUR (INITIALES)	
1448525	Russian	JW	JAN 23 1978

Source: Dostizh, Razvit. Nov. Metodov Khim. Anal. (New Methods of Chemical Analysis), 1974, pp. 3--4 (USSR).

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At present the method of gas-liquid chromatography is widely used for analyzing medicinal preparations. This method proved convenient for separating and analyzing ethyl ester of α -bromisovaleric acid** (EEBIK) and validol. EEBIK is the therapeutic substance of the preparation korvalol. Validol is a mixture of menthyl ester of isovaleric acid and menthol.

Of the investigated stationary phases for separating the constituents in EEBIK and in validol, polyethylene-glycoladipinate (PEGA) turned out to be the most selective. To determine the optimal conditions for the analysis, we investigated the dependence of VETT [height equivalent to a theoretical plate] on the rate of flow of the carrier gas and the column temperature and the dependence of the separation factor on the temperature. These experiments provided a basis for selecting the optimal conditions for analyzing one column of

*The figures in the right-hand column indicate page numbers of the original (Tr.).

**In this paper names of compounds and chemical expressions are translated literally (Tr.).

EEBIK and validol.

For identification and quantitative analysis of EEBIK the components of the mixture were separated on a preparatory chromatograph and subsequently identified on an MKh 1303 mass spectrometer under the following operating conditions:

- temperature of inlet system and ion source : 200°
- ionization potential : 50 electron volts
- emission current : 1.5 ma
- accelerating voltage : 2 kilovolts.

The study of the mass spectra enabled us to determine the molecular weight and structure of the compounds on the basis of the molecular and fission-produced ions. The structure of the hydrocarbon chains governs the emergence of a high peak (m/e 55) in the mass spectrum of the first component and a peak (with m/e 41) in the spectrum of the second component, which suggests a structure of ethyl ester of α -brommethylethyl acetic acid for the first component and of ethyl ester of α -bromisovaleric acid for the second. The presence of ethyl ester of α -brommethylethyl acetic acid stems from the fact that the initial constituent in the synthesis of EEBIK is industrial isovaleric acid whose main constituent is methylethyl acetic acid which is difficult to separate. /4

Since validol is also synthesized from isovaleric acid, we must expect that it, too, contains menthyl [sic] ester of methyl-ethyl acetic acid, which was demonstrated by the method of adding. This same method was used to determine menthol and menthyl ester of isovaleric acid.

It is conceivable that the esterification of isovaleric acid is accompanied by dehydration of the menthol, resulting in the formation of menthene hydrocarbons. The latter's presence in validol was demonstrated with

the aid of mass spectrometry. The quantitative analysis of validol and EEBIK was conducted by the method of internal normalization. The results of the analysis were treated statistically.