The product was too deliquescent to make it practical to remove it from the reaction flask.

(b) **Trimethyl-(7-methyl-8-carbethoxy)-octylammonium Bromide.**—This compound was prepared in the same manner from 16.9 g. of ethyl ω-bromo-β-methylpelargonate and 39 g. of an approximately 16% solution of trimethylamine in anhydrous benzene. It also was extremely deliquescent, and after washing with 500 ml. of anhydrous ether, was used directly for the next synthesis.

Hydrazides of Trimethyl-ω-carboxyalkylammonium Bromides. (a) Hydrazide of Trimethyl-(5-methyl-6-carboxy)-hexylammonium Bromide.—A mixture of the crude trimethyl-(5-methyl-6-carbethoxy)-hexylammonium bromide and 7 g. of 85% hydrazine hydrate was heated to reflux for 15 minutes, enough alcohol was added to give a clear solution, and then refluxing was continued for an additional 2 hours. The solvent was removed under reduced pressure and the residual oil was washed with dry ether and chilled. There was obtained 2.5 g. (26%) of a white powder which melted at 118–122°.

Anal. Calcd. for $C_{11}H_{26}ON_3Br$: C, 44.59; H, 8.84. Found: C, 44.23; H, 9.16.

(b) **Hydrazide of Trimethyl-(7-methyl-8-carboxy)-octylammonium Bromide.**—The crude trimethyl-(7-methyl-8-carbethoxy)-octylammonium bromide, obtained previously, and 20 g. of 85% hydrazine hydrate were caused to react as described in the preceding experiment. The waxy appearing product was dissolved in hot absolute alcohol and precipitated by the addition of anhydrous ether. The solid was removed by filtration, dissolved in a small amount of hot absolute alcohol, clouded with petroleum ether (b.p. 60–68°) and allowed to solidify. There was obtained 5 g. (23%) of a hygroscopic, white powder, m.p. 136–139°.

Anal. Calcd. for $C_{18}H_{30}ON_3Br$: C, 48.13; H, 9.32. Found: C, 48.41; H, 9.60.

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Constituents of U. S. P. Colchicine. N-Formyltrimethylcolchicinic Acid Methyl Ether[1]

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An earlier report from these laboratories[3] called attention to the presence of *ca.* 4% of 2-desmethylcolchicine[4] in commercial samples of U.S.P. colchicine. When a new sample[5] was subjected to chromatographic purification by the usual procedure[3,6] using chloroform–methanol (99:1) as eluant, an alkaloid (*ca.* 1.5% yield) having the properties of Šantavý's Substance B (N-formyltrimethylcolchicinic acid methyl ether)[7] was isolated; no 2-desmethylcolchicine was encountered. The new compound crystallized readily from ethyl acetate as pale yellow prisms which melted with decompositions at 260–262° (capillary). A comparison of this substance with Šantavý's Substance B is given in Table I.

TABLE I

	N-Formyltrimethylcolchicinic acid methyl ether from U.S.P. Colchicine[a]	Šantavý's Substance B	N-Formyliso-trimethylcolchicinic acid methyl ether
M.p., °C., dec.	260–262 (capillary)	264–267 (Kofler block)	252–253 (capillary)
$[\alpha]_D$ chloroform	$-175 \pm 1°$	$-171.2°$	$-315 \pm 1°$
	c 1.01, $t = 25°$	c 1.08, $t = 22°$	c 0.719, $t = 25°$
λ_{max} (log ϵ)	242.5 (4.48)	247 (4.51)	244 (4.50)
(95% ethanol)	350.0 (4.24)	350 (4.27)	342.5 (4.29)
	(c 5.22 × 10^{-5} M)		(c 5.6 × 10^{-5} M)

[a] Also synthesized from trimethylcolchicinic acid methyl ether.

The product was synthesized by formylation of trimethylcolchicinic acid methyl ether[8] using 98% formic acid in pyridine. Solvents were removed *in vacuo*, the residue was taken up in chloroform, washed with water and dried. Evaporation left a residue which crystallized readily from ethyl acetate to give pale yellow prisms, m.p. 260–262° dec. alone and when mixed with a sample isolated from U.S.P. colchicine.

Further confirmation of the configuration of our product was obtained by comparison with the iso-derivative prepared from iso-trimethylcolchicinic acid methyl ether[8] in the same manner. The product crystallized from ethyl acetate containing a little chloroform or methylene chloride as pale yellow prisms, m.p. 252–253° dec.; mixed m.p. with Substance B, 224–233° dec. For analysis it was dried to constant weight at 80° *in vacuo*.

Anal. Calcd. for $C_{21}H_{23}NO_6$: C, 65.44; H, 6.02. Found: C, 65.20; H, 5.94.

Comparative data are given in Table I; these are in agreement with previous findings[8,9] with respect to the properties of the iso- *vs.* the normal-forms in the colchicine and trimethylcolchicinic acid series.

Minor amounts of other alkaloids are present in the samples of U.S.P. colchicine which we have examined. Investigation of them will be continued. The biological effects of the N-formyltrimethylcolchicinic acid methyl ethers are being studied and will be reported elsewhere.

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The Characterization and Degradation of Isotopic Acetic and Lactic Acids

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During a study of the biosynthesis of hyaluronic acid,[1] it became necessary to characterize and degrade small quantities of isotopic acetic and lactic acids. As the procedures developed may be of general interest, details are presented here.

The chemistry of benzimidazole derivatives of aliphatic acids has been described in a recent comprehensive review.[2] In contrast to the usual technique for characterization of aliphatic acids,[3–5] the present method involves the use of a large excess of the reagent, *o*-phenylenediamine, and removal

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