H 6.03; N 9.86.) Electrophoresis was performed with a buffer pH 6.5 (100 ml pyridine and 10 ml HOAc diluted to 1000 ml). The migration was 45 mm (15 min at 900 V and 35 mA).

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Chemical Studies on Bryophytes*

11. (—)-16 α-Hydroxykaurane from *Saelania glaucescens* (Hedw.) Broth.

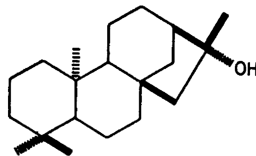
ERLING NILSSON and OLLE MÅRTENSSON

Institute of Chemistry, Organic Chemistry Department, University of Uppsala, Box 531, S-751 21 Uppsala 1, Sweden

A few mosses and liverworts are recognized by b bluish green or bluish grey colour. As the specific epither indicates this is an important taxonomic character of the moss *Saelania glaucescens* (Hedw.) Broth. The bluish grey tinge is especially pronounced when the moss grows in relatively pure tufts, and it can generally be observed also on isolated plants. This special colour originates from a granular or filamentous covering on the back of the leaves (particularly the lower parts) and adjacent parts of the stem. Although the waxy character of this coating was pointed out early [1] some authors of modern handbooks and floras consider it to be composed of fungal or algal threads.

Recently Huneck and Vevle [2] identified the main component of the waxy coating on two liverworts. *Anthelia juratzkana* (Limpr.) Trev. and *Anthelia julacea* (L.) Dum. as the tetracyclic diterpene (–)-16α-hydroxykaurane (I).

The present investigation shows that compound I is the main component of the white covering on *Saelania glaucescens*. Brief treatment with benzene removes the white material outside the plant completely and only minor amounts of carotenoids and chlorophylls are extracted. The infrared spectrum indicates contamination by waxes, but the main compound is sufficiently abundant to allow purification simply by sublimation and one recrystallization. A sufficient quantity of material (4 mg) was obtained from only a few grams of herbarium material available. By comparison with an authentic sample the moss compound was proved to be (–)-16α-hydroxykaurane (I) (IR, MS, NMR, mixed m.p., and optical activity).



I

The occurrence of triterpenes in some mosses has been reported by other authors [3] but 16-kauranol seems to be the first diterpene identified in a member of the Musci.

Experimental. Moss material (collections made in the sixties in the western parts of the province of Lycksele lappmark) was rinsed rapidly with benzene. The extract was centrifuged and evaporated to dryness. Sublimation at 110°C and 0.2 mmHg, and subsequent crystallization from a dichloromethane-meth-

* Previous publications in this series have appeared under the heading "Moss Pigments". Part 10: *Acta Chem. Scand.* **23** (1969) 2910. Future papers will be published under the new heading independent of the type of compounds investigated.

anol mixture gave minute white needles (4 mg) with m.p. 218–219°C (uncorr.). Mixed m.p. with authentic (–)-16α-hydroxykaurane (I) 217–218°, $[\alpha]_D^{25} = -46°$ (*c* 0.40, $CHCl_3$), I: $[\alpha]_D^{25} = -45°$ (*c* 1.0, $CHCl_3$). The molecular weight found (mass spectrometry) was 290 ($C_{20}H_{34}O$: m.w. 290.5). The mass spectrum differed from that of I (*cf.* Ref. 4) only in the relative intensities of some peaks. Infrared spectra (KBr phase) of the two samples were indistinguishable.

NMR spectra were recorded on a Varian A60 instrument equipped with a C 1024 time averaging computer. Signals from the four methyl groups occurred at the following δ-values (TMS as internal reference, solvent $CDCl_3$).

I	0.80	0.84	1.02	1.35
Moss substance	0.80	0.83	1.04	1.35

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Studies on the Coupling Step in Solid Phase Peptide Synthesis. Some Preliminary Results from Competition Experiments

ULF RAGNARSSON, SUNE KARLSSON and BENGT SANDBERG

Biokemiska Institutionen, Box 531, S-751 21 Uppsala 1, Sweden

This preliminary study deals with the reactivity of Boc *-protected amino acids under the standard coupling conditions used in solid phase peptide synthesis as described by Merrifield.[1] The aim has been to estimate roughly the relative coupling rates of properly protected derivatives in order to have a rational basis for considerations by which differences in reactivity can be compensated for in some way or another. Such differences in reactivity may be assumed to exist for sterical and other reasons but have not received due attention so far, though there are some cases already known when certain amino acids have caused special difficulties in the coupling step.[2,3]

We recently started an investigation, the first results of which are reported below. Thus polymer-bound amino acid ** (1 equiv.) was reacted with altogether 4 equiv. of 4 generally *different* Boc-amino acids and the same quantity of dicyclohexylcarbodiimide for 2 h at room temperature. Boc-glycine was present in all experiments to make a rough comparison possible. From a practical point of view, the general procedure was essentially that of Merrifield. The Boc-group was removed from the dipeptide resin with 50 % (v/v) trifluoroacetic acid in methylene chloride before the dipeptide mixture was split off from the resin with liquid hydrogen fluoride,[4] and a peptide aliquot after acid hydrolysis analyzed for free amino acids.[5] One series of experiments was performed with polymer-bound alanine (Table 1), another with polymer-bound valine (Table

* Boc stands for α-*t*-butyloxycarbonyl.

** Prepared from Bio-Beads S·X-1, 200–400 mesh, chloromethylated, capacity 0.75 mequiv./g, obtained from Bio-Rad Laboratories, Richmond, Calif., USA, following the standard procedure.