Short Communications

Some Chemical Data Concerning The Epicuticle of Wool

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The wool fibre, as well as hairs from other animals, is enveloped by a thin membrane, the epicuticle ¹, with a thickness varying from 50 to 250 Å.² Similar structures are found on feathers ^{3,4} and also in the surface layers of human skin ⁵.

There are two main methods for isolating the epicuticle from the wool. A common feature is, however, that the epicuticle, which amounts to 0.1-0.2% of the scoured material, is always contaminated by material from the underlaying layers. — The one method is to dissolve the main part of the wool by treatment with dilute Na_2S_2 whereby the epicuticle is left as a residue ^{1,2}. The other is to treat the wool with chlorine or bromine for a short time, destroy the excess of halogen by treatment with dilute $NaHSO_3$ and finally shake off the epicuticle ¹.

A sample of epicuticle from wool, isolated by the bromine method and containing approximately 30 % pure epicuticle, has been treated with 20 % HCl at 100°C for 10 hours. (The estimation of the percentage of pure epicuticle is based upon the fraction isolated from the original material and also on electron microscopical observation.) Part of the substance was dissolved during the hydrolysis. The

hydrolysate was used for paper chromatography on sugars using a mixture of butanol, acetic acid and water as solvent and different types of developers in the different runs. The procedure is described by Werner and Odin 6. In most cases rhamnose was used as a front sugar and other substances used for identification of the different spots were glucosamine, fucose, galactose and glucose. The method suggested by Novellie 7 using a developing agent containing β -naphtylamine and the method of Trevelyan, Procter and Harrison 8 using a solution of AgNO3 in acetone and a spray of alcoholic NaOH gave similar results. In these cases spots indicating the presence of galactose or a sugar with almost the same R_f -value as galactose for this system were obtained. diffuse spots, or rather a band of diffuse spots, occurred in the test runs. This band indicates the presence of a reducing agent with an R_t -value between rhamnose and mannose and also that glucosamine may be present. The same results were obtained when the 20 % HCl in the hydrolysis was replaced by 36 % HCl. The original method of Partridge 9 utilizing ammonia + AgNO₃ gave diagrams with less distinct spots and the paper also had a tendency of blackening after some time. No paper chromatography on the amino acid composition of the sample was carried out as the contaminations most probably are constituted by amino acids more or less modified by the chemical treatment of the material. The carbohydrates are, however, probably constituents of the epicuticle. They probably form compounds with other

Table 1.

	Per cent epicuticle (approx.)	% 0	% 8	% N	% Br
Residue from Na ₂ S-treatment	10	22.3	3.5	14.3	
Sample obtained by brominating the wool	30	27.0	3.0	13.0	8.3
Untreated wool	0.1 - 0.2		2.8	14.7	

constituents such as proteins or possibly fatty acids. — The X-ray diagrams from samples isolated from wool give some interferences found neither in the a- nor in the β -diagram of wool ¹⁰, which are possible indications of structures of the above mentioned type.

As to the modification caused by the chemical treatment Table 1 should give an idea and at the same time give a few figures for the chemical composition of the epicuticle compared with ordinary wool. The elementary analysis has been carried out by the Microchemical Laboratory of the Medico-Chemical Institute of The University of Uppsala.

The nitrogen content of the epicuticle seems to be lower than that of the main part of the wool, the sulphur content is slightly higher. In the case of the Na₂S-treated sample some impurities may originate from polysulphides and sulphur containing products formed by the reagent and the original material. In the brominated sample it is evident that the bromine has reacted with the organic material. Moreover it is indicated that some oxidation has occurred as the oxygen content is higher than in the other samples. This oxidation may have led to the formation of carboxyl groups which may be an explanation to the fact that samples obtained in this way are to great extent soluble in dilute alkali.

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- Lindberg, J., Philip, B., and Gralén, N. Nature 162 (1948) 458.
- Lagermalm, G., and Philip, B. Textile Research J. 20 (1951) 668.
- Philip, B., Lagermalm, G., and Gralén, N. Nature 166 (1951) 1070.
- Philip, B., Lagermalm, G., and Gralén, N. Biochim. et Biophys. Acta 6 (1951) 497.
- Lagermalm, G., Philip, B., and Lindberg, J. Nature. In press.
- Werner, I., and Odin, L. Upsala Läkarefören. Förh. 54 (1949) 69.
- 7. Novellie, L. Nature 166 (1950) 745.
- Trevelyan, W. E., Procter, D. P., and Harrison, J. S. Nature 166 (1951) 444.
- 9. Partridge, S. M. Nature 158 (1946) 270. 10. Lagermalm, G. Proc. Swed. Inst. Textile
 - Research, Gothenburg, Swed. 14 (1951) 65.

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Investigations in the Retene Field. IV. Nitration of Retene in the Presence of Boron Trifluoride

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Several attempts have been made to prepare nitro-derivatives of retene by direct nitration, the most successful being those reported by Fredriksen and Nielsen ¹ (who also give further references on this subject). By nitration of retene under mild conditions these workers obtained a crude product from which they isolated 9-nitroretene in a yield of about 5 %. According to a private communication, they were also able to isolate 3-nitroretene but this