LETTERS TO THE EDITORS

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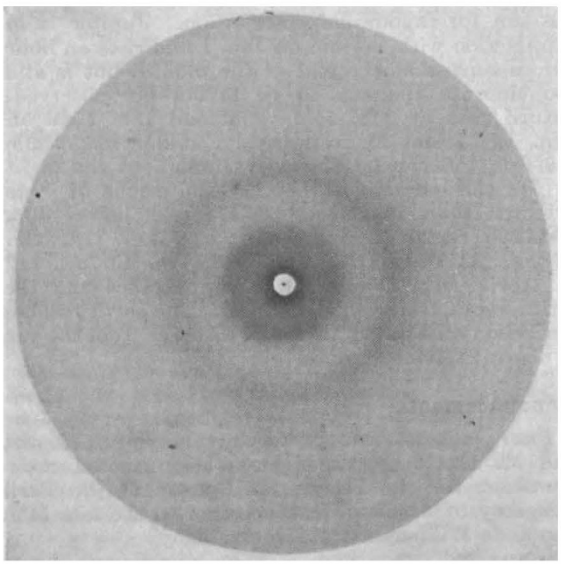
Regenerated Keratin Fibres

In the past few years considerable progress has been made in the regeneration of certain protein fibres from solution; in particular, casein[1] and peanut globulin[2] have been spun with considerable success. Hewitt and Coleman[3] have dissolved and respun silk, Lundgren[4] and his co-workers have made fibres from dissolved chicken feathers, and one of us (R. L. W.) has described the regeneration of a protein from a mixture of casein and soluble keratin[5]. In the following, a short description is given of the regeneration of wool keratin fibres from solution. This material was made by the method previously described by Wormell[5], and its X-ray photograph is reproduced herewith. It can be seen from this diagram that the keratin molecule is in the β or fully extended form, and there is no evidence of the presence of the typical α-fold of the original wool molecule.



X-ray photograph of soluble protein molecules from wool, showing a typical denatured β-keratin structure. Copper *Ka* radiation. $D = 3$ cm.

The soluble keratin curd was dissolved in dilute cuprammonium solution, centrifuged and precipitated with sulphuric acid. The curd was then washed in water, redissolved in cuprammonium solution, and after further centrifuging, the solution was spun, as previously described for the casein/keratin mixtures[5], into a saturated solution of sodium sulphate (*p*H=7) containing 0·1 per cent 'Fixanol'. Finally, the resulting fibres were hardened in a bath of sodium sulphate and formaldehyde, and insolubilized in a solution of sulphuric acid, sodium sulphate and formaldehyde at approximately 50° C.

An X-ray study of the resulting fibres showed that they had retained the β-keratin structure. Traces of orientation could be detected, but there was a definite decrease in the crystallinity of the protein at this stage. In particular, the definition of the 4·65 A. backbone spacing was not so good in the fibre as in the original protein curd. It was found, however, that the fibres could be readily stretched in hot water or steam to extensions greater than 100 per cent of their original length. The X-ray diagram of a fibre so stretched showed a redevelopment of a crystalline β-keratin structure, with a reasonably good orientation of the molecules along the fibre axis.

From the above experiments, it is clear that the dissolution and extrusion of wool can readily be carried out after the cystine cross-links have been broken down by swelling and dispersion of the protein in aqueous sodium sulphide. The long-chain keratin molecules remain in solution without extensive degradation; the specific α-configuration of the molecule is apparently destroyed, and the resulting protein is capable of reformation from a dispersion in cuprammonium hydroxide into fibres showing the crystalline β-form. Hardening and insolubilizing processes can then be applied to the material to produce a regenerated protein fibre in a manner somewhat similar to that used in casein fibre manufacture.

Note added in proof.—Since this note was submitted for publication, regenerated keratin fibres have been made with an average tenacity of 0·8 gm./denier and 30 per cent extension at break, measured at 65 per cent relative humidity and 70° F. From this point of view at least, the fibres can be considered as being in the range of usable textile materials.

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[1] Happey and Wormell, *J. Soc. Dyers and Colourists*, Symposium on Fibrous Proteins, 160 (May 1946).
[2] Traill, *Chem. and Ind.*, **8**, 58 (1945). Chibnall, Bailey and Astbury, U.K. Pat. 467,704.
[3] Hewitt and Coleman, *Proc. Roy. Soc.*, A, **190**, 145 (1947).
[4] Ward, High and Lundgren, *J. Polymer Res.*, 1, 22 (1948).
[5] Wormell, *J. Tex. Inst.*, **39**, P.T. 219 (1948).

The elastic properties of the keratin fibres, wool and hair, have been shown to result from a folded molecular structure capable of reversible extensibility[1]. This folded structure gives rise to a large-angle X-ray pattern, referred to as the α-pattern, which characterizes an extensive class of proteins, the α-proteins[2]. In view of the dependence of the elastic properties of the keratins on the α-structure, it is a desirable goal in the preparation of artificial fibres to obtain a regenerated protein from suitable raw materials possessing this structure.

Most regenerated proteins when sufficiently oriented in the fibrous form yield the β-type X-ray pattern, indicating the presence of straight rather than folded chains[3,4]. It has been found possible, however, by using stronger solutions of the type employed by Lundgren and colleagues[3] (saturated urea containing reducing agents) to dissolve a substantial fraction of a 'hard' keratin such as wool. The dissolved protein may be readily salted out of solution. Two kinds of precipitated protein have been prepared: (*a*) a non-orientable material, obtained at *p*H 6–7 and temperature 40° C., with properties resembling those of chewing-gum; and (*b*) an orientable fibrous form at *p*H 8–9 and 50° C. The first material may be drawn into threads, but these are non-birefringent and yield an X-ray pattern consisting only of two diffuse rings. The second may be drawn into fine fibres with anisotropic physical properties, marked birefringence and yielding the α-type X-ray pattern. It strongly resembles Rudall's 'epidermin'[5] prepared by extract-