A Colorimetric Method for the Estimation of Chromium in Wool

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A colorimetric method for the estimation of chromium in wool based on the use of EDTA as the chelating agent is reported. The method involves dry ashing of the wool sample, oxidation of Cr(III) to Cr(VI) using nitric acid and potassium chlorate, reduction of Cr(VI) to Cr(III) by treatment with ascorbic acid and finally complex formation with EDTA solution. The chromium-EDTA complex gives an absorption maximum at 546 nm. The stability of the complex is much greater than those of the complexes obtained with the currently used chelating agents, viz. diphenylcarbazide and haematoxylin. Therefore, the results obtained with EDTA are more accurate.

The chromium content of dyed or treated wool often gives useful information in the investigation of problems associated with dyeing. For example, it gives an indication whether wool has been dyed with chrome mordant dyes or with premetallized dyes.

The methods for the estimation of chromium in wool mostly involve oxidation of chromium to the hexavalent state followed by determination of Cr(VI) either (i) volumetrically, e.g. by titration with ferrous ammonium sulphate using n-phenyl anthranilic acid as indicator or (ii) colorimetrically, e.g. as the diphenylcarbazide complex or the haematoxylin complex.

The complex produced by reaction between diphenylcarbazide and chromium is not very stable and fading occurs. Therefore, rapid measurement of the colour intensity is necessary. Moreover, the colour intensity is affected by the quality of diphenylcarbazide reagent used. With haematoxylin, precipitation of the complex occurs on standing. These factors are likely to lead to erroneous results. It is possible to avoid the influence of these factors by using a more powerful chelating agent than diphenylcarbazide or haematoxylin. In the present study, ethylenediaminetetraacetic acid (EDTA) has been used as a chelating agent in the colorimetric estimation of chromium in wool.

It has been reported that under proper conditions, EDTA forms a well-defined coloured complex with trivalent chromium. The complex is violet in colour and can be used for the colorimetric estimation of chromium in wool. The stability constant of the chromium-EDTA complex is sufficiently high to preclude the possibility of interference due to other cations.

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It is now well established that Cr(VI) from the chromate or dichromate anions adsorbed by wool during dyeing with chrome mordant dyes is reduced to Cr(III) involving cystine bonds (CH₂-S-S-CH₂) in the fibre. Although Cr(III) readily forms complexes with EDTA, it is not possible to remove it quantitatively from the dyed material directly and it is necessary to apply the standard method of dry ashing the wool sample followed by oxidation to Cr(VI) using nitric acid and potassium chlorate. The Cr(VI) obtained in this way is then reduced to Cr(III) for complex formation with treatment with ascorbic acid.

The quantity of ascorbic acid required for reduction and the conditions for complex formation were established in preliminary experiments. Fig. 1 is a plot of the absorbance curves for chromium-EDTA complexes in which samples of Cr(III) obtained from different sources were used. It is seen that the chromium-EDTA complex gives an absorption maximum at 546 nm.

Estimation of Chromium Content in Wool

Samples of wool fabrics were dyed with chrome mordant dyes (1-4% on weight of fibre) at boil for 1-2 hr using the afterchrome method. One undyed sample of wool fabric was also treated with potassium dichromate (2% on the weight of fibre) at boil for 3 hr. The chromium contents of both the dyed and the chromium treated samples were estimated by the colorimetric method reported here as well as by the volumetric method. In a typical experiment, a dyed sample (2-3 g) was dried to constant weight at 105°C, dry ashed in a silica crucible at about 500°C and chromium brought into solution by a method reported in the literature. The resulting solution was made up to 100 ml with distilled water and to 20 ml of this
solution were added 5% ascorbic acid solution (6 ml) and 2% EDTA solution (3 ml). The pH was adjusted to 3-3.5 and the mixture boiled gently for 10 min.

After cooling, the volume was made up to 50 ml with distilled water, the absorbance measured at 546 nm and the concentration of chromium calculated from the calibration plot. The calibration plot was obtained by two methods. In the first method, an aliquot of a standard Cr(III) solution was mixed with excess EDTA, the pH adjusted to 3-3.5 and the solution boiled gently for 10 min. The solution was then cooled, the volume made up to 50 ml with distilled water and the absorbance measured on a Cecil CE 303 grating spectrophotometer at 546 nm against a reagent blank.

In the second method, a solution of potassium dichromate of known concentration was treated as described above, but with the addition of ascorbic acid to reduce Cr(VI) to Cr(III) during boiling. The plot of absorbance against concentration is shown in Fig. 2. It is seen that both sets of points fall on the same straight line.

The values for a number of dyed and treated samples obtained by the colorimetric method are compared with those obtained by the volumetric method in Table 1. It is evident that the Cr(III)-EDTA complex can be used to estimate the chromium content of wool. The method is simple and reliable and the stability of the complex formed is much greater than that of the complex formed in the other methods. The absorbance of a solution containing the complex was checked at different time intervals and found to remain constant for at least one week.

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References
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