

1 $\mu$ . The purpose of this communication is to report the result of a re-examination of the reaction of  $\beta$ -carotene and antimony trichloride.

The sample of  $\beta$ -carotene used had the following characteristics: m.p. 179°; in light petroleum (b.p. 70–80°) the maxima were at 478 m $\mu$  and 451 m $\mu$ ,  $E^{1\text{ per cent}}_{1\text{ cm}}$  (451 m $\mu$ ), 2,500 and  $E^{1\text{ per cent}}_{1\text{ cm}}$  (340 m $\mu$ ), 136, and in carbon disulphide the maxima were at 512 m $\mu$  and 482 m $\mu$ . The sample probably contained some *cis*-isomers. 1 ml. of a solution of  $\beta$ -carotene in chloroform was mixed with about 2 ml. of Carr–Price reagent and the absorption measured at one wavelength in a Beckmann spectrophotometer. It was found that the absorption reached a value which remained approximately the same for about 1 min., 45 sec. after mixing. The value of the extinction 60 sec. after mixing was recorded for a number of wave-lengths using separate solutions for each wave-length. In this manner it has been shown that  $\lambda_{\text{max}}$  is about 1,020 m $\mu$  and that  $\epsilon_{\text{max}}$  is about 115,000. Antimony trichloride is transparent in this region of the spectrum.

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<sup>1</sup> Morton, R. A., "Absorption Spectra" (Adam Hilger, Ltd., London, 1942).

<sup>2</sup> Ferguson, L. N., *Chem. Rev.*, **43**, 385 (1948).

<sup>3</sup> Karrer, P., and Schwyzer, R., *Helv. Chim. Acta*, **31**, 1055 (1948).

<sup>4</sup> Shantz, E. M., *Science*, **108**, 417 (1948).

<sup>5</sup> Karrer, P., and Benz, J., *Helv. Chim. Acta*, **32**, 232 (1949).

<sup>6</sup> Karrer, P., Karanth, K. P., and Benz, J., *Helv. Chim. Acta*, **32**, 1036 (1949).

### A Method for the Determination of Procaine Penicillin

THE iodimetric estimation of procaine penicillin presents difficulties because of the interaction of procaine with iodine. Sodium penicillin to which an equivalent weight of procaine hydrochloride has been added gives results which may be from 3 to 5 per cent lower than for the untreated penicillin. Although the ferricyanide method of Hiscox<sup>1</sup> is said to be free from the above objection, we could not obtain consistent results with it, a spread of  $\pm 4$  per cent being obtained in a series of determinations on a sample of sodium penicillin. The removal of the procaine ion by precipitation followed by iodimetric determination of the penicillin seemed the most promising line of attack, and sodium silicomolybdate and sodium silicotungstate were both found suitable for this purpose, although the latter possesses certain advantages over the former and is obtainable commercially. Both substances give with procaine salts heavy precipitates which are granular in the presence of sodium chloride (3 per cent) and can readily be centrifuged to a compact mass permitting decantation of the supernatant liquid. Precipitation is virtually complete, and neither precipitant has any action upon iodine or penicillin.

Results obtained by adding procaine hydrochloride to sodium penicillin and assaying iodimetrically after precipitation were about 0.5 per cent higher than for the untreated sodium penicillin. This difference, which may be real, is nevertheless well within the accepted limits of experimental error of the two different iodimetric methods which were employed for the estimations<sup>2,3</sup>. For the first method<sup>2</sup>, results

for sodium penicillin ranged from 98.3 to 100.8 per cent with a mean value of 99.5 per cent and standard deviation of 0.30, while for procaine penicillin the corresponding figures were 98.7–101.3 per cent with a mean figure of 99.95 per cent and standard deviation of 0.36. For the second iodimetric method<sup>3</sup> the results for sodium penicillin ranged from 98.6 to 99.9 per cent with a mean value of 99.35 per cent and standard deviation of 0.17, and for procaine penicillin from 99.5 to 100.3 per cent with a mean value of 100.0 per cent and standard deviation of 0.18.

Full details of the work carried out in connexion with the above will be published elsewhere at the earliest opportunity.

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<sup>1</sup> Hiscox, D. J., *Anal. Chem.*, **21**, 658 (1949).

<sup>2</sup> Wild, A. M., *J. Soc. Chem. Indust.*, **67**, 90 (1948).

<sup>3</sup> Report of the Analysts Sub-Committee to the Ministry of Health Conference on the Differential Assay of Penicillin, Part II. *Analyst*, **74**, 550 (1949) (with modifications).

### Mercerization of Jute

THE transformation of crystalline native cellulose (cellulose-I) into the hydrate modification (cellulose-II) by the action of caustic soda solutions is known to proceed by the formation of intermediate crystalline soda-cellulose complexes<sup>1</sup>. In a previous publication<sup>2</sup> from this Laboratory, it was reported that the complete native-hydrate transformation is impossible in raw jute, the presence of appreciable quantities of cellulose-I being always apparent from the X-ray photographs. In ramie the conditions for a partial transformation of this kind are much more critical, and appear to be such that only part of the cellulose has reacted with the alkali. In order to determine whether the effect in jute can also be explained in this way, we have studied the 'soda-cellulose' obtained by treating raw jute without tension in caustic soda solutions of various concentrations, and find that in general the native modification is absent for concentrations greater than about 10 per cent (w/w), but that it reappears when the alkali is removed by washing in water. In jute, therefore, incomplete transformation to cellulose-II is not due to the failure of the alkali to penetrate into the cellulose crystallites, but to at least partial reversibility of the native  $\rightarrow$  soda-cellulose reaction.

We have ascribed the difference in behaviour of jute and ramie during mercerization to the effect of lignin in the former, where, perhaps by forming cross-linkages between the cellulose chains near the boundaries of the crystallites, it introduces an element of stability absent in the purer fibres. In this connexion we should also like to direct attention to an abnormal soda-cellulose-I which appears in jute treated with 10 per cent caustic soda solution. The X-ray photograph is then that of soda-cellulose-I with the strong inner equatorial reflexion spread out into a streak, indicating either a distortion of the soda-cellulose lattice or the co-existence of a range of transitional states between native cellulose and soda-cellulose-I in jute. This effect has not, so far as we know, been