THE IDENTIFICATION AND QUANTITATIVE DETERMINATION OF PHENYLEPHRINE IN ADRENAL GLAND

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Phenylephrine, the N-methyl derivative of m-octopamine (m-hydroxyphenylethanolamine) is a sympathomimetic amine with strong presser and mydriatic activity (Innes & Nickerson 1970) and it has not hitherto been identified in animal tissue. m-Octopamine has recently been detected in mammalian tissue (Robertson et al 1977), particularly in rat and bovine adrenal gland (Williams & Couch 1978). Since the adrenal medulla contains large amounts of phenylethanolamine N-methyltransferase and m-octopamine is an active substrate for this enzyme (Axelrod 1962), bovine adrenal gland was investigated for the presence of phenylephrine.

Authentic phenylephrine was reacted with N-methyl bis(trifluoroacetamide)/pyridine to give the tris-trifluoracetyl (TFA) derivative, which was resolved on gc from its congeners which had been similarly derivatized. The TFA derivative afforded characteristic ions in the mass spectrum at m/e 140 (base peak) and m/e 455 (M^+): the ratio of the intensities of these two ions was 91 (± 6) at a retention time of 5.52 min on 5% OV-101 at 196°. Fresh adrenal glands were homogenized in formic acid-acetone and, after centrifugation, the supernatant was fractionated by ion-exchange chromatography. The dried amine fraction was derivatized as before and gas chromatography-mass spectrometry-selected ion monitoring (gc-ms-sim) revealed the presence of a compound with a retention time of 5.53 min, fragment ions of m/e 140 (base peak) and m/e 455 (M^+) which had a ratio of intensities of 90 (± 17). The N-trideuteromethyl derivative of phenylephrine was synthesized from m-hydroxybenzaldehyde by a seven stage procedure and used as an internal standard for the quantitative determination of phenylephrine by gc-ms-sim of the TFA derivative. The concentration of phenylephrine in the adrenal glands from several animals varied from 18-66 ng g^{-1}.

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