Journal of Chromatography, 179 (1979) 392–393

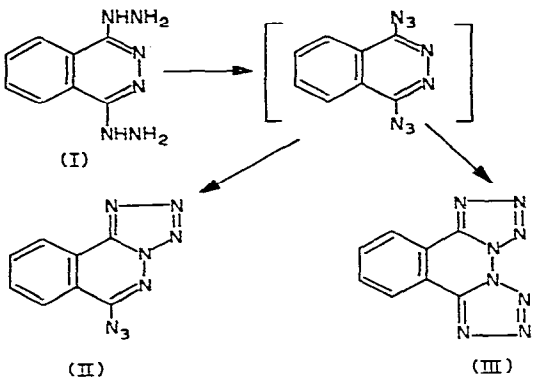
CHROM. 12,259

Letter to the Editor

Structure of the product of reaction between dihydralazine and nitrous acid

Sir,

We thank Dr. Jack[1] for drawing the work of Reynolds *et al.*[2] to our attention. Their studies[2] indicated that reaction of dihydralazine (I) with nitrous acid resulted in 5-azido-1,2,3,3*a*,4-pentazacyclopent[*e*]indene (II) implying that cyclisation of only one of the two intermediate azide substituents had occurred.



Our mass spectral data[3] for the product of this reaction was consistent with either the monotetrazolo (II) or ditetrazolo (III) structures. Accordingly we have examined our reaction product by infrared spectroscopy since the presence of an azide substituent would be revealed by a characteristic band at about 2150 cm^{-1} (ref. 4). The resulting spectrum (Fig. 1) contained this band (at 2140 cm^{-1}) which was absent in the infrared spectra of (I) and of tetrazolo(1,5-*a*)phthalazine formed by the reaction of

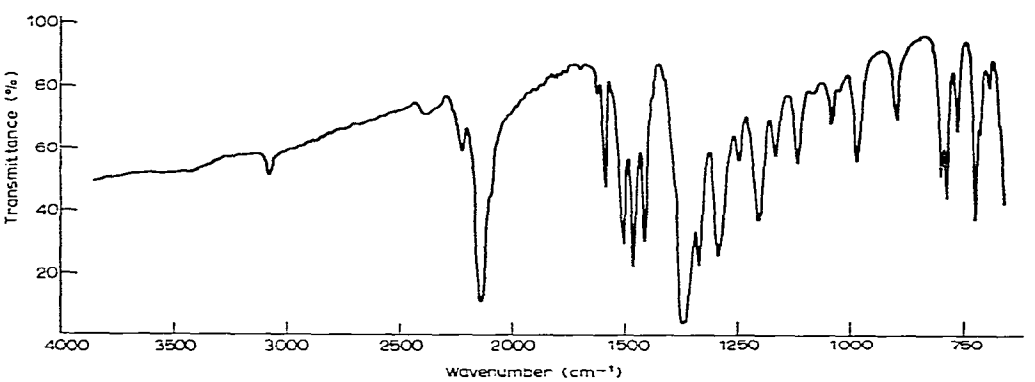


Fig. 1.

nitrous acid with hydralazine. The structure of the reaction product of dihydralazine (I) with nitrous acid should therefore be presented as (II) and not as (III) as indicated previously[3]. This revision of structure does not alter the utility of the analytical method for the measurement of dihydralazine in plasma by high-performance liquid chromatography[3]. The reaction product (II) is potentially rather explosive and should therefore be handled with great care[5].

Department of Metabolism and Pharmacokinetics,
Huntingdon Research Centre,
Huntingdon (Great Britain)

A. R. WALLER,
L. F. CHASSEAUD and
T. TAYLOR

1 D. B. Jack, *J. Chromatogr.*, 179 (1979) 390.
2 G. A. Reynolds, J. A. Van Allan and J. F. Tinker, *J. Org. Chem.*, 24 (1959) 1205.
3 A. R. Waller, L. F. Chasseaud and T. Taylor, *J. Chromatogr.*, 173 (1979) 202.
4 F. L. J. Sixma and H. Wynberg, *A Manual of Physical Methods in Organic Chemistry*, Wiley, London, 1964, p. 203.
5 W. Riess, personal communication.

(Received July 30th, 1979)