

others (figures 1-4). The onion root tips treated with pure sodium 10% bicarbonate solution showed very few aberrations. However the total percentage of aberrations have varied in relation to the concentration of lecanoric acid (Control—nil, NaHCO₃—0.93, 0.1%, lecanoric acid 2.87, 0.2%—4.73 and 0.3%—0.47).

The C-metaphase activity of the lecanoric acid has also been noticed in the present study and its high incidence has been observed particularly at 0.2% concentration. The changes noted above are due to the effect of lecanoric acid dissolved in 10% sodium bicarbonate solution.

The authors are thankful to the Head, Department of Chemistry and Head, Department of Botany, Osmania University for facilities and encouragement. One of us (BS) is thankful to UGC for the award of FIP fellowship.

19 August 1982

1. Venugopal Rao, K. and Manoharachary, C., *Geobios.*, 1980, 7, 176.
2. Malleshwar, D., Sundara Murthy, V. and Subba Rao, N. V., *Curr. Sci.*, 1974, 43, 74.
3. Asahina, Y. and Shibata, S., *Chemistry of Lichen Substances*, (Tokyo, Japan Society for Promotion of Sciences, Publ.), 1954.
4. Henningson, B. and Lundstrom, H., *Material and Organism*, 1970, 5, 19.
5. Sturelid, S. and Lundstrom, H., *Experientia*, 1972, 28, 1238.
6. Swami, U. B. S. and Vijayakumar, C., *Curr. Sci.*, 1976, 45, 636.
7. Venkat Reddy, P., Rao, P. S. and Subramanyam, S., *Indian J. Exp. Biol.*, 1978, 16, 1019.
8. Deysson, G., *Int. Rev. Cytol.*, 1968, 24, 99.

GAS CHROMATOGRAPHIC DETERMINATION OF TRACES OF 2-NAPHTHYLAMINE IN 1-NAPHTHYLAMINE

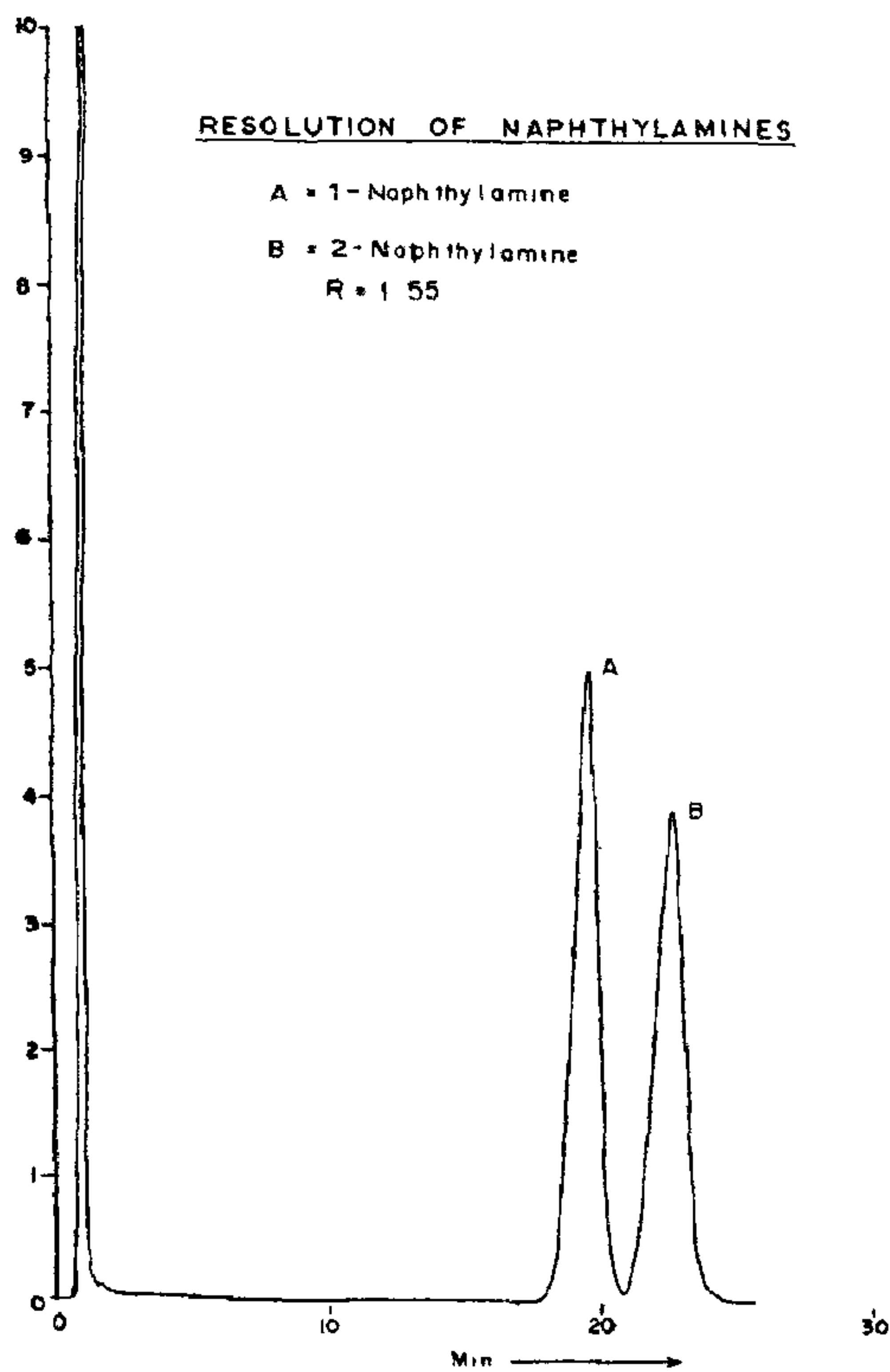
C. M. FARIAS AND A. K. DURVE

Netel Chromatographs, (Applications Laboratory), S.V. Road, Manpada, Thane 400 607, India.

NAPHTHYLAMINES are used in the manufacture of dyes which are in turn used as food colours; 2-naphthylamine has been reported to be toxic¹ and hence its determination in trace levels in various species such as food colours² and food additives³ has gained importance.

Methods reported for the determination of 2-naphthylamine in 1-naphthylamine include both chromatographic as well as non chromatographic techniques. Various titrimetric⁴, spectrophotometric³ and conductometric⁵ methods have also been investigated. Chromatographic methods reported include paper⁶ and thin layer⁷, high performance liquid⁸ and gas chromatography techniques. The gas chromatographic methods reported either involve conversion of the amines to a suitable derivative^{9,10} or direct injection onto a column packed with specialised liquid crystals¹¹.

The present paper describes a sensitive, rapid and accurate gas chromatographic method making use of a glass column pretreated with alkali and packed with a mixture of FFAP and KOH on a silanized solid support. The main advantage of the method is direct injection of the sample without derivatization resulting in a resolution factor of 1.55; 2-naphthylamine in levels less than 0.1% in 1-naphthylamine or in levels lower than 5 ng has been determined.



Gas chromatographic measurements were made on a Netel model 'Omega' instrument equipped with a dual flame ionization detector (FID) and a single pen recorder with an input of 1 mv. A glass column pre-treated with KOH, 2 meters in length with outer diameter of 1/4 inch and packed with 10% FFAP + 2% KOH on chromosorb W-HP, 80/100 mesh was used. The oven temperature was maintained at 210°C. The temperature of the detector and injection port were adjusted to 230°C and 300°C respectively. Nitrogen was used as a carrier gas at a flow rate of 45 ml/min and the FID was used with flow rates of hydrogen and air maintained at 30 ml/min and 300 ml/min respectively. The chart speed was 0.5 cm/min. The retention time of 1-naphthylamine under these conditions was 20 minutes and that of 2-naphthylamine 23 minutes (figure 1).

Preparation of solutions: Stock solutions of 1-naphthylamine (250 mg) and 2-naphthylamine (100 mg) were prepared in benzene (5 ml). Standard mixtures of 2-naphthylamine in 1-naphthylamine were prepared to give a series of standard solutions in the range of 0.1% to 0.5%. The sample (250 mg) was dissolved in benzene (5 ml) and this solution was used for injection.

Procedure: Standard solutions (0.6 µl) were injected sequentially and a calibration curve was prepared. A plot of peak heights against concentrations gave a straight line in the range 0.1-0.5% of 2-naphthylamine. The sample was treated in the same manner as the standards and the peak height was extrapolated to give the 2-naphthylamine content. Results obtained on various samples are as shown in table I.

TABLE I

2-Naphthylamine content in 1-naphthylamine

Sample	I	II	III
Amount of 2-naphthylamine estimated in sample	0.58%	1.10%	2.03%
Number of observations	10	10	10
Standard deviation	0.06	0.04	0.07
Per cent recovery after standard addition	101.90	100.58	101.87

30 July 1982

- Malkus, Z., *Cesk. Hyg.*, 1977, **22**, 259.
- Dixon, E. J. and Groffman, D. M., *Analyst (London)*, 1975, **100**, 476.
- Albert, F. M., Butuceanu, E. and Cupfer, M., *Rev. Roumaine Chim.*, 1964, **9**, 835; *Chem. Abstr.*, 63, 14046d.
- Albert, F., *Butuceanu Rev. Chim. (Bucharest)*, 1974, **25**, 751.
- Chetti, G., Bartalini, E., Armeli, G. and Pozzoli, L., *Lav. Um.*, 1968, **20**, 389; *Chem. Abstr.*, 1969, **71**, 24552y.
- Matsushita, H., *Ind. Health*, 1967, **5**, 260; *Chem. Abstr.*, 1968, **69**, 92774f.
- Okamoto, M., Yamada, F. and Nakamura, A., *Eisei Kagaku*, 1979, **25**, 48, (Japan); *Chem. Abstr.*, 91, 75677p.
- Marmion, D. M., White, R. G., Bille, L. H. and Ferbev, K. H., *J. Gas Chromatog.*, 1966, **4**, 190.
- Chiavari, G. and Glumanini, A. G., *J. Chromatogr.*, 1981, **206**, 555.
- Ciosek, M., Witkiewicz, Z. and Dabrowski, R., *Chem. Anal. (Warsaw)*, 1980, **25**, 567.

STUDIES IN THE REARRANGEMENT OF SPIRANES: SYNTHESIS OF 1',2',3',4'-TETRAHYDRO SPIRO [2,3,4-TRIMETHYL CYCLOPENTANE-1,2'-NAPHTHALENE] AND ITS REARRANGEMENT ON CATALYTIC DEHYDROGENATION

D. N. CHATTERJEE AND BHARATI CHAKRABORTY
Department of Chemistry, Presidency College,
Calcutta 700 073, India.

SPIRANES of the type 1',2',3',4'-Tetrahydrospiro cyclopentane-1,2'-naphthalene are known to undergo rearrangement on dehydrogenation with 10% Pd-C catalyst¹. Recently we synthesised a substituted spirane of the above type² and observed that it underwent smooth ring transformation on catalytic dehydrogenation without any loss of carbon atom giving substituted phenanthrene. We now report the synthesis of highly substituted spirane, 1',2',3',4'-tetrahydrospiro-[2,3,4-trimethyl cyclopentane-1,2'-naphthalene] (I) with a view to studying its rearrangement on dehydrogenation.

The hydrocarbon (I) was synthesised starting from 2,3,4-trimethyl cyclopentane prepared from crotonic acid by addition of potassium iodide in phosphoric acid solution followed by coupling of the

1. Bowman, M. C., *J. Assoc. Off. Anal. Chem.*, 1978, **61**, 1253.