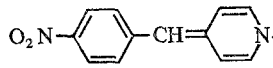
Table 1. $R_f \times 100$ values for N-alkyl-4-(4-nitrobenzylidene)-1,4-dihydropyridines[1]

Compounds	Silica gel G benzene/dimethoxyethane (6:4) plus 5% diethylamine
	
	$-CH_3$ — 13
	$-C_2H_5$ — 29
	$-C_3H_7$ — 36
	$-C_4H_9$ — 46

[1] The standards were applied in their salt form to the TLC-plate and only during the run, the N-alkyl-4-(4-nitrobenzylidine) 1,4-dihydropyridines are formed.

depend on the nature of the masking components present.

The method was employed in studies in the nature of alkylating components present in the gas phase of cigarette smoke. In this regard, most of the alkylating activity was found to be a methylation, caused mainly by methyl chloride, a known constituent of the gas phase of cigarette smoke [4, 7].

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Polarographic Determination of Indium(III) with Tetraethylene Pentamine

Polarographische Bestimmung von Indium(III) mit Tetraäthylenpentamin

Best. von Indium(III); Polarographie; Tetraäthylenpentamin

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A method is proposed for the polarographic determination of indium(III) using tetraethylene pentamine (tetren) as a base electrolyte.

Experimental. Polarograms were recorded with manual Adept polarograph and a Philips pH-meter was used for pH-measurements. D.M.E. and S.C.E. connected to the cell through KCl-agar bridge, were used as electrodes and the values of m and t were 1.85 mg/sec and 3.20 sec, respectively, in distilled water and open circuit.

Indium solution was prepared from B.D.H. (AR) Indium sulphate and 2 M solution of tetren was used for the measurements.

The wave remained well defined over the 0.1 to 1.0 M range of tetren concentration. The value of $E_{1/2}$ with 0.4 M tetren was found to be -1.125 V, however, $E_{1/2}$ shifted to more negative potential values with the increase in tetren concentration.

The effect of pH on the polarograms of indium was also studied in detail. The reduction wave remained well defined in the pH range of 8.0 to 10.5. With the decrease in pH, $E_{1/2}$ shifts to positive potential.

Discussion. The plot of i_d vs. $\sqrt{h}$, which was linear, indicated the diffusion controlled nature of the wave. The slope of the plot of log $(i/i_d - i)$ vs. E and the values of $E_{3/4} - E_{1/4}$ both indicated the irreversibility of the reduction. Due to this irreversibility no deduction could be made about the nature of the complex formed. By comparing the diffusion current with some known reduction, the three electron involvement was indicated in the reduction of indium.

Polarograms with 0.4 M tetren and different concentrations of indium were recorded and the diffusion currents were measured by extrapolation method. When i_d was plotted against metal ion concentration, a straight line was obtained. So these results can be

used for the polarographic determination of indium. A calibration graph was prepared for concentrations of 0.2 to 1.5 M of indium and 0.4 M of tetren, the polarogram of the unknown solution was recorded under identical conditions and the values of i_d were referred to the calibration graph. The results were found to be within an error of $\pm 0.5\%$.

Metals like Cu, Bi, Ni, Pd, Pb, Zn, Ag, Mo, W and Au do not undergo reduction in the potential range of indium and cause no interference. Fe, Th and Mn get precipitated, so also do not interfere, Cd and Sn give rise to interferences. Simultaneous determinations of In-Tl and Cu-In are possible.

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Spectrophotometric Determination of Traces of Cobalt with 1-Naphthamidoxime

Spektralphotometrische Bestimmung von Kobaltspuren mit 1-Naphthamidoxim

Best. von Kobalt mit 1-Naphthamidoxim; Spektralphotometrie; Spuren

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In alkaline medium cobalt(II) reacts with 1-naphthamidoxime to form a soluble blue complex extractable with alcohols (C_4—C_8). The reaction is very sensitive and allows the detection of 1 ppm Co. Almost all other amidoximes react similarly.

Reagent. 1-Naphthamidoxime was prepared from naphthonitrile and hydroxylamine in water-alcohol solution at 70° C (3 days), recrystallised and purified with active coal; m.p. 149—150° C [1, 2]. If kept in the dark the alcoholic solutions are stable for some months.

Procedure. Mix in a separating funnel 10 ml of neutral sample solution (with 5—90 µg of Co) with 1 ml of 0.05 M reagent solution and 1 ml of 0.4 N KOH solution, leave for 10 min to develop the colour, extract with 5.5 ml of isobutyl alcohol for 1 min, dry the organic phase with sodium sulphate and measure the absorption at 581 nm.

Traces of cobalt can be *detected* as follows: Add 0.5 ml of 0.05 M reagent solution to 1 ml of sample solution, alkalise with 0.5 ml of 0.4 N KOH solution and dilute with 1.2 ml of isobutyl alcohol. Dilute to 10 ml with water after 10 min, shake and leave to stay until two layers appear. The organic layer becomes light blue if 1 ppm Co is present in the sample.

The absorptivity at the absorption maximum of 581 nm increases with increasing ratio reagent/Co, but remains constant when reagent/Co is larger than 25. The colour is stable for 2 h and decreases thereafter slowly.

The decadic molar absorptivity is $\bar{\varepsilon} = 4400 \pm 50$ at 581 nm. The stability constant as calculated by the logarithmic method is $\log K = 5.80$ for $n = 2$ and $K = \beta_1\beta_2$. Beer's law is obeyed in the range of 0.5 to 9 ppm Co (optimum 2—7 ppm). The relative error is $\pm 2.5\%$.

Oxidising agents destroy the complex completely. Ni, Cr(III) and Mn(II) interfere. Small amounts of Cu(II) and Fe(III) are tolerable, if masked by tartrate (Co/Cu 1:5) and fluoride (Co/Fe 1:3), respectively. Fluoride, chloride, bromide, nitrate, nitrite, sulphate and tartrate anions may be present in rather large amounts (anion/Co > 500).

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