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A CONVENIENT, RAPID ESTIMATION OF CAFFEINE IN TEA

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Gas liquid chromatography of chloroform extracts of alkalined tea infusions detected caffeine quantitatively. Unbound caffeine availability in different grades of tea was estimated.

INTRODUCTION

Caffeine in beverages has been the focal point of an international controversy in the recent past (Kuhnert 1981) due to the biological activity of this alkaloid (Bury 1975).

Classically quantitative estimation of caffeine has been carried out using the Bailey-Andrew method (AOAC Hand Book 1965, 14.047) which employs a non specific Kjeldahl nitrogen determination. Thus a necessity arose to have a convenient non elaborate method of determining this alkaloid in tea infusions.

The Bailey-Andrew method was complemented by spectrophotometric analysis. Presently spectrophotometric methods have been superseded by chromatographic methods in the determination of caffeine (NEWTON 1969 a & b).

MATERIALS AND METHODS

Apparatus

General laboratory glassware and chemicals.

Tracor 560 gaschromatograph fitted with a Tracor TS 10 recorder.

Glass column 1.5 m × 2 mm prepacked with 2.5% OV 101 on chromasorb WHP 80/100.

Operating parameters of the gas-chromatograph

Carrier gas (Argon)	flow rate 30 ml/min.
Hydrogen	flow rate 30 ml/min.
Air	flow rate 30 ml/min.

Detector Flame ionisation (FID) 250°C, Chart speed 0.25 cm/min.

Column temperature 150°C isothermal. Attenuation 16, Injection 2 μl, 200°C, ethyl acetate.

Standard and Sample preparation

Analar caffeine was accurately weighed and dissolved in distilled ethyl acetate (100 ml) see Table 1. The six grades of tea available at St Coombs were processed for analysis as below.

Black tea (5 g) in boiling distilled water (200 ml) was simmered for 15 minutes, filtered hot and made to 500 ml. An aliquot of the filtrate (50 ml) was made alkaline with 10% ammonium hydroxide (3 ml) and extracted with chloroform (25 ml × 3). The combined organic extracts were washed with, 1% sodium hydroxide (10 ml × 3) followed by distilled water (10 ml × 3) and dried over an hydrous sodium sulphate. The chloroform solution was filtered and the filtrate evaporated to yield a pale yellow solid. The solid was dissolved in distilled ethyl acetate (10 ml) and used directly for gas chromatography.

RESULTS AND DISCUSSION

A standard curve for caffeine was obtained by plotting the peak height Vs the concentration (Fig. 1, 2 and Table 1).

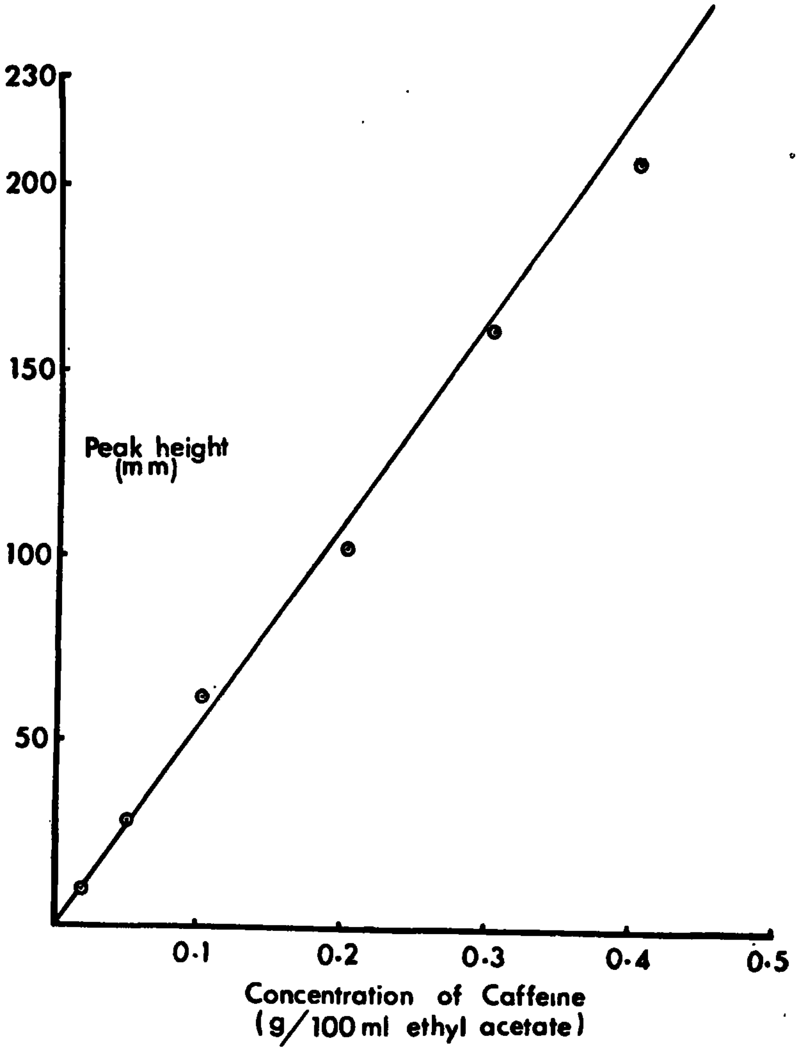

Fig. 1.—A Standard curve for caffeine.

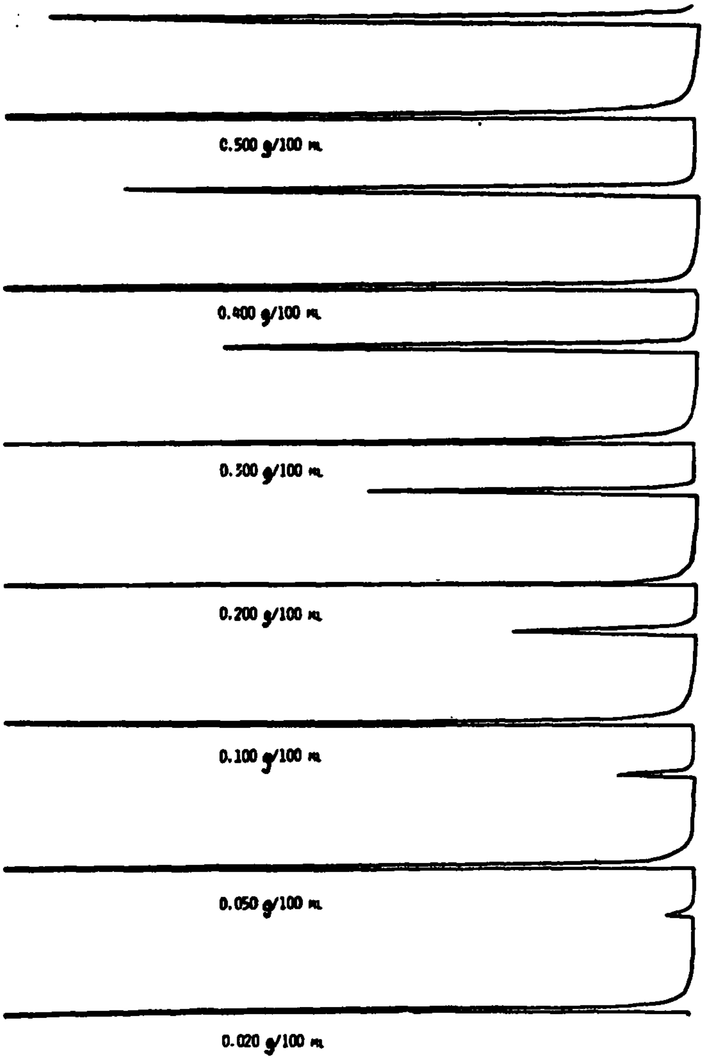

Fig. 2.—Gas chromatogram of caffeine standards.

The Tea samples were then chromatographed and the concentration corresponding to the peak height was read. Based on this reading the caffeine content in brewed tea and black tea was calculated (Fig. 3 and Table 2).

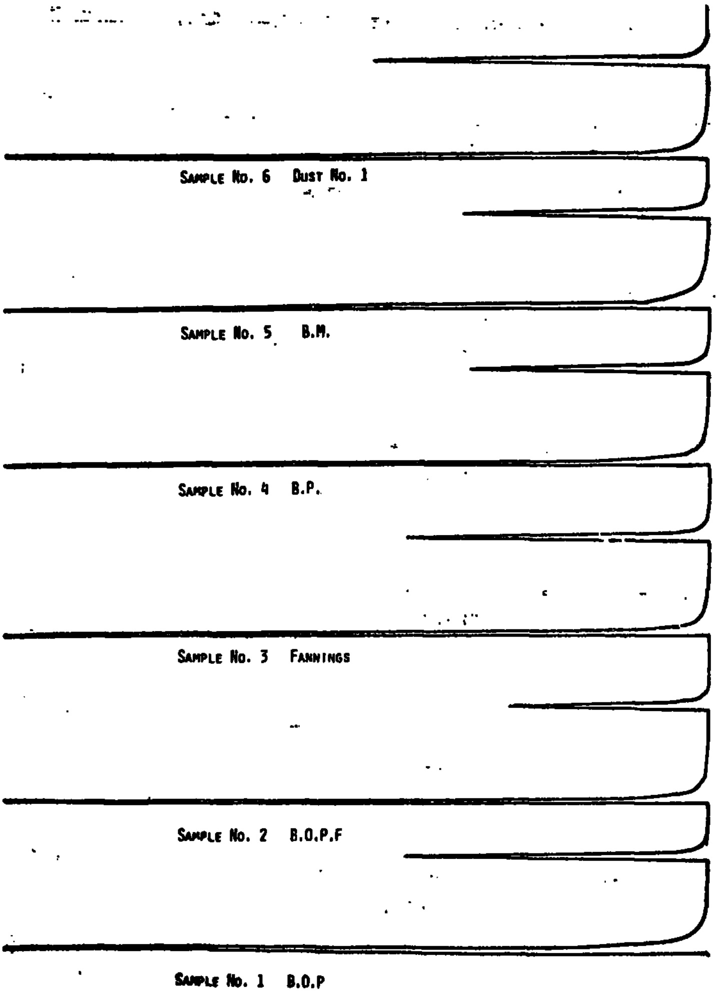

Fig. 3.—Gas chromatogram of extracted caffeine from tea samples.

Table 1

Concentration g/100 ml	*Peak height (mm)*			
	(1)	*(2)*	*(3)*	*Average*
0.020	10	10.5	10	10.2
0.050	28	28	28	28.3
0.100	63	66	63	64
0.200	113	115	113	113.6
0.300	163	168	164	165
0.400	197	200	196	197.6

Table 2

Sample in (10 ml)	*Peak height (mm)*	*Standard Curve reading*	*Caffeine Concentration*	
			Brew g/100ml	*black tea % dry wt.*
BOP	103	0.1878	0.03756	3.75
BOPF	68	0.1250	0.025	2.50
Fannings	103	0.1878	0.03756	3.75
BP	82	0.1500	0.0300	3.00
BM	84	0.155	0.0310	3.10
Dust 1	114	0.210	0.042	4.20
Commercial Black tea	115	0.212	0.0424	4.24
Tea Flush	55	0.101	0.0202	2.02

Hitherto preparation of samples for chromatographic or spectroscopic analysis has been subjected to numerous cleanup procedures of the caffeine extracts (Herath 1981, Yeransian 1963). In the present method we have found that crude chloroform extracts of tea infusions may be used directly for gaschromatographic analysis.

The flame ionisation detector (FID) used by us showed adequate sensitivity, thus a KC1 thermionic detector was found to be unnecessary. We also found that the readily available and common OV 101 column gave reproducible results, thus it was used in preference to a DC 200 column (Newton, 1969 b).

In conclusion we would like to present a table comparing the caffeine estimation in tea by the prevailing popular methods (Table 3).

Caffeine concentration mgs/100 ml

Table 3

Sample	*Method - Bailey-Andrew*	*Spectrophotometric*	*HPLC*	*GC*
1	28	30	30	31
2	29	31	32	32
3	29	30	33	31

H.P.L.C. — High Pressure liquid chromatography (Herath, 1981)
G.C. — Gas Chromatography

Thus the above method of estimating caffeine in tea is much easier and accurate than the spectroscopic method presently used. It is definitely a complementary method to the high pressure liquid chromatography technique.

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