

CHROM. 15,157

LINEAR RETENTION INDEX SYSTEM FOR POLYCYCLIC AROMATIC COMPOUNDS

CRITICAL EVALUATION AND ADDITIONAL INDICES

DANIEL L. VASSILAROS

Midwest Research Institute, 425 Volker Blvd, Kansas City, MO 94110 (U.S.A.)

ROBERT C. KONG

Department of Chemistry, Brigham Young University, Provo, UT 84602 (U.S.A.)

DOUGLAS W. LATER

Battelle PNL, Richland, WA 99352 (U.S.A.)

and

MILTON L. LEE*

Department of Chemistry, Brigham Young University, Provo, UT 84602 (U.S.A.)

(Received June 18th, 1982)

SUMMARY

A linear retention index system for temperature-programmed capillary gas chromatography of polycyclic aromatic compounds using naphthalene, phenanthrene, chrysene, and picene as internal standards has been used in this laboratory for routine analysis since 1979. Conclusions from a data set of 29 routine capillary gas chromatographic analyses of a complex mixture of polycyclic aromatic compounds are used to evaluate, describe, and justify the polycyclic aromatic compound retention index system. The number, width, and linearity of the interpolation intervals are described. The effects on the retention indices of variations in programming rate, column internal diameter, and initial temperature are discussed. The polycyclic aromatic compound retention index system is shown to be a consistent, useful tool for preliminary identification of polycyclic aromatic compounds in complex mixtures. Tables listing standard retention indices for polycyclic aromatic hydrocarbons, polycyclic aromatic sulfur heterocycles, and nitrogen-containing polycyclic aromatic compounds are included.

INTRODUCTION

Because absolute gas chromatographic (GC) retention is a complicated function of many parameters, and thus useless as a means of reporting and comparing data, various schemes have been devised to normalize retention data. Haken¹ and Ettre² have reviewed the development of relative retention concepts and evaluated their application and utility. The most widely applied development is the Kovats retention index (*I*) system³, which is theoretically founded upon the linear relation-

ship between the logarithm of the retention time ($\log t_R$) and the carbon number of a homologous series of *n*-alkanes under constant-temperature GC operating conditions. Since its introduction, many scientists have examined, applied, evaluated, and modified the Kovats system¹⁻⁹. Two observations from the literature serve to characterize the Kovats system with respect to its applicability to routine analysis of non-aliphatic hydrocarbon mixtures:

(a) Ettre² and Mathiasson *et al.*⁷ have shown that the *I* values can be disproportionately affected by selective retardation of the standards or the solutes as the nature and film thickness of the stationary phase change with usage.

(b) The theoretical basis of the Kovats system obviously limits its application to mixtures containing a narrow boiling point range of components⁶. Complex mixtures are laboriously analyzed by isothermal GC at several different temperatures⁸.

The linear relationship between $\log t_R$ and carbon number does not hold under programmed temperature conditions. Van den Dool and Kratz¹⁰ generalized Kovats' logarithmic formulation to allow calculation of *I* values from chromatographic data generated by temperature programming, assuming a linear relationship between t_R and carbon number of the standards. Temperature programming is preferred over isothermal operation because narrow peaks are normally obtained throughout the chromatogram and a wide boiling point range of compounds can be chromatographed. This is especially useful when dealing with the analysis of complex environmental mixtures containing several hundred compounds boiling from 80°C to 500°C.

The purposes of this paper are to review the linear *I* system specifically devised for the qualitative analysis of the polycyclic aromatic compounds (PACs)¹¹, to support empirically its fundamental assumptions, to make observations based on three years application of the *I* system to the analysis of PAC mixtures from a variety of samples under a wide range of GC conditions, and to present *I* values for a large number of PACs, including polycyclic aromatic sulfur heterocycles (PASHs), and nitrogen-containing polycyclic aromatic compounds (N-PAC).

EXPERIMENTAL

All chromatography was carried out on SE-52 coated fused-silica columns. The columns varied in length from 15 to 20 m, and were either 0.3 mm I.D. (0.25 μm film thickness) or 0.2 mm I.D. (0.17 μm film thickness). Hydrogen was used as the carrier gas with a linear velocity of 100 cm/sec.

GC was performed using Hewlett-Packard 5880A gas chromatographs equipped with capillary injection systems (operated in the splitless mode) and BASIC programming capability. Unless specifically mentioned, the oven temperature profile was 40°C to 265°C at 4°C/min with a 2-min initial isothermal period. A standard set of all GC operating parameters (attenuation, threshold, baseline, temperature of heated zones, etc.) was stored on tape in an "Analysis" file. Upon retrieval of the "Analysis" file all operating parameters were automatically set to the standard values.

The *I* values were generated from the raw retention data by use of a BASIC program written for the HP 5880 GC and based on the equation of Van den Dool and Kratz¹⁰. The program calculates *I* values, compares the experimental values with

“standard” indices¹¹ stored in DATA statements, outputs all calculated I values, and names those peaks whose indices fall within a given tolerance window. Fifty PACs are normally stored in the DATA set for routine analyses, although additional compound names and respective I values could be stored.

The internal standards employed in this particular I system are naphthalene (200.000), phenanthrene (300.000), chrysene (400.000), and picene (500.000). Three of these standards are present in most complex PAC mixtures; picene oftentimes is not. Rather than add picene to the PAC sample being analyzed, a converging algorithm which uses three data points from the chromatogram for the post-run calculation of the t_R of picene was written and added to the BASIC program. All I values in the region between chrysene and picene ($I = 400-500$) were generated using a calculated t_R for picene.

A coal tar sample is used in this laboratory as a standard complex mixture for GC quality control. Compounds in this sample have been identified by GC-mass spectrometry (MS) and by comparison of retention data with standard compounds. I values from the coal tar sample have been included in this report as an example of the application of the PAC I system to an authentic complex PAC mixture. In addition, I values have been measured for 232 standard PACs which contain either a sulfur or nitrogen heteroatom by co-chromatographing standard mixtures with all four PAC internal standards.

RESULTS AND DISCUSSION

The I system for PACs was described by Lee *et al.*¹¹, and is based on the internal standards naphthalene, phenanthrene, chrysene, and picene. Although the system has no rigorous foundation in chromatographic thermodynamics and does not assume or require a linear relationship between the retention and ring number of the four internal standards, a linear regression analysis of the retention times of these standards from nine different coal tar chromatograms gave an $r^2 = 0.9956$. These four compounds were used as the internal standards instead of the n -alkanes¹² to ensure constant elution behavior among solutes and standards under all operating conditions. The following equation, suggested by Van den Dool and Kratz¹⁰, was used to calculate retention indices:

$$I = 100z + 100 \left[\frac{t_{R_x} - t_{R_z}}{t_{R_{z+1}} - t_{R_z}} \right] \quad (1)$$

where z and $z + 1$ are the numbers of aromatic rings in the bracketing standards.

The ratio inside the brackets in eqn. 1 is a measure of the elution position of compound “ x ” relative to the interpolation interval between the two standards. Assuming that stationary phase, surface activity, and instrumental conditions affect the elution of all polycyclic aromatic hydrocarbons (PAHs) proportionally, this ratio should be a constant on a given stationary phase, within normal GC operating parameter limits. There are indications that a good correlation of the ratio on a wide polarity range of liquid phases will be obtained¹³.

Fig. 1 is a capillary chromatogram of the coal tar sample. Peak numbers refer to the compound identifications listed in Table I. The I values of many major and

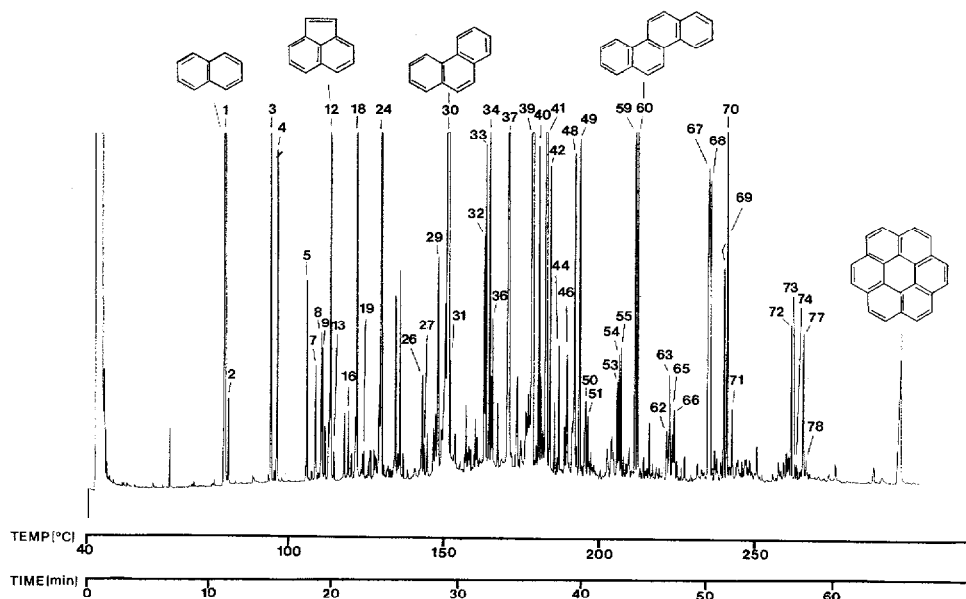


Fig. 1. Capillary gas chromatogram of coal tar sample. Column, 20 m \times 0.20 mm I.D. SE-52 fused silica; see Experimental section for further details, and Table I for peak identifications.

minor components of this sample from 29 GC analyses over the past year were compiled and statistically evaluated. The average I values, the standard deviations, and the 95% confidence limits of 78 peaks are tabulated. The precision obtained for the compounds listed in Table I is excellent considering the time span over which the data were collected, and the normal variables of different instrument operators and columns of different lengths and inner diameters.

The average, standard deviation, and 95% confidence limits of the width in minutes of the interval between each pair of standards from the 29 coal tar analyses described above were compared with parallel statistical data from ten recent chromatograms of the same coal tar sample obtained within a 2-day period. The differences in the averages from one data set to the other are 0.2 sec, 0.3 sec, and 5 sec, for Regions II (naphthalene-phenanthrene), III (phenanthrene-chrysene), and IV (chrysene-picene), respectively. The interval widths over 39 runs are remarkably constant.

The widths of the interpolation intervals are different, each successive interval being *ca.* 2 min shorter than the previous one: an index unit in Region II is *ca.* 10 sec, an index unit in Region III is *ca.* 9 sec, and an index unit in Region IV is *ca.* 8 sec. This slight deviation from linearity would have to be taken into account for structure-retention correlation calculations, but has no effect on the analysis of complex mixtures.

The absolute width of the interpolation intervals is a function of the number of internal standards employed. The use of shorter retention windows was evaluated by selecting additional parent PAHs which elute roughly in the middle of each region. The I values of at least two peaks in each of the smaller regions were calculated and then compared with the I values obtained for the same peaks from the same chromatograms using only the original four retention index standards. Results of this com-

TABLE I
RETENTION INDICES FOR COAL TAR PACs

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of determinations
1	Naphthalene	200.00			
2	Benzo[<i>b</i>]thiophene	201.43	0.28	±0.03	30
3	2-Methylnaphthalene	220.22	0.23	±0.08	30
4	1-Methylnaphthalene	223.01	0.28	±0.10	30
5	Biphenyl	236.44	0.19	±0.07	30
6	2-Ethynaphthalene	238.55	0.24	±0.11	22
7	2,6- or 2,7-Dimethylnaphthalene	240.28	0.22	±0.08	30
8	1,7-Dimethylnaphthalene	242.77	0.23	±0.09	29
9	1,3- or 1,6-Dimethylnaphthalene	243.30	0.20	±0.10	18
10	2,3- or 1,4-Dimethylnaphthalene	246.03	0.22	±0.09	23
11	1,5-Dimethylnaphthalene	246.92	0.31	±0.12	30
12	Acenaphthylene	246.92	0.31	±0.12	30
13	1,2-Dimethylnaphthalene	248.50	0.26	±0.10	27
14	1,8-Dimethylnaphthalene	252*			1
15	Acenaphthene	253.14	0.28	±0.10	30
16	Methylbiphenyl	255.41	0.19	±0.07	30
17	4- or 3-Methylbiphenyl	256.69	0.20	±0.08	27
18	Dibenzofuran	258.77	0.24	±0.09	30
19	C-3 Naphthalene	261.50	0.20	±0.08	27
20	C-3 Naphthalene	262.13	0.20	±0.08	28
21	2,3,6-Trimethylnaphthalene	265.09	0.18	±0.07	27
22	1-Methylacenaphthylene	266.57	0.23	±0.09	29
23	2,3,5-Trimethylnaphthalene	267.54	0.17	±0.07	28
24	Fluorene	269.73	0.30	±0.11	30
25	9-Methylfluorene	273.79	0.18	±0.11	12
26	2-Methylfluorene	288.42	0.15	±0.06	30
27	1-Methylfluorene	289.20	0.21	±0.08	27
28	Methylfluorene	290.83	0.22	±0.08	30

(Continued on p. 6)

TABLE I (continued)

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of determinations
29	Dibenzothiophene	295.39	0.17	±0.06	30
30	Phenanthrene	300.00			
31	Anthracene	301.08	0.11	±0.04	30
32	3-Methylphenanthrene	319.19	0.24	±0.09	30
33	2-Methylphenanthrene	319.93	0.20	±0.08	30
34	4H-Cyclopenta[<i>def</i>]phenanthrene	321.77	0.12	±0.04	30
35	9- or 4-Methylphenanthrene	322.81	0.21	±0.08	30
36	1-Methylphenanthrene	323.64	0.42	±0.16	30
37	Anthrone	331.39	0.12	±0.04	30
38	2-Phenylanthralene	332.64	0.26	±0.10	29
39	Fluoranthene	344.51	0.06	±0.02	30
40	Acphenanthrylene	347.67	0.08	±0.03	30
41	Pyrene	351.51	0.15	±0.06	30
42	MW 218**	353.88	0.14	±0.05	30
43	MW 218	356.18	0.16	±0.06	30
44	MW 218	358.48	0.16	±0.06	30
45	MW 218	361.43	0.14	±0.05	30
46	Methylfluoranthene or methylacephenanthrylene	362.76	0.14	±0.05	30
47	Methylfluoranthene or methylacephenanthrylene	365.42	0.14	±0.06	21
48	Benzo[<i>a</i>]fluorene	366.72	0.10	±0.04	30
49	Benzo[<i>b</i>]fluorene or 4-methylpyrene or 2-methylpyrene	369.40	0.10	±0.04	30
50	Methylfluoranthene or methylacephenanthrylene	372.49	0.10	±0.04	30
51	1-Methylpyrene	373.45	0.13	±0.05	29
52	Methylfluoranthene	374.68	0.18	±0.09	18
53	Benzo[<i>b</i>]naphtho(2,1- <i>d</i>)thiophene	389.09	0.09	±0.03	30

54	Benzol[<i>ghi</i>]fluoranthene	389.92	0.11	± 0.04	30
55	Benzol[<i>c</i>]phenanthrene	391.24	0.10	± 0.04	30
56	Benzol[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	392.59	0.10	± 0.04	26
57	Benzol[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	395.61	0.10	± 0.04	28
58	Cyclopenta[<i>c,d</i>]pyrene	396.55	0.17	± 0.07	23
59	Benz[<i>a</i>]anthracene	398.76	0.04	± 0.02	30
60	Chrysene	400.00			
61	Triphenylene	400.00			
62	3-Methylchrysene	418.02	0.07	± 0.07	28
63	2-Methylchrysene or 12-methylbenz[<i>a</i>]anthracene or 4-methylbenz[<i>a</i>]anthracene and MW 240***	419.66			
64	5-Methylchrysene	420.20*	0.12	+ 0.05	29
65	6-Methylchrysene or 4-methylchrysene and MW 240	421.25	0.09	± 0.03	28
66	1-Methylchrysene or 7-methylbenz[<i>a</i>]anthracene and MW 240	422.99	0.08	± 0.08	7
67	Benzol[<i>h</i>]fluoranthene or benzol[<i>b</i>]fluoranthene	443.13	0.11	± 0.04	29
68	Benzol[<i>k</i>]fluoranthene	444.02	0.07	± 0.03	27
69	Benzol[<i>e</i>]pyrene §	452.29			
70	Benzol[<i>a</i>]pyrene	454.02	0.07	± 0.07	30
71	Perylene	457.17	0.06	± 0.02	30
72	Indeno[1,2,3- <i>c,d</i>]pyrene	493.24	0.09	± 0.03	30
73	Dibenz[<i>a,h</i>]anthracene	496.20	0.30	± 0.13	24
74	Dibenz[<i>a,c</i>]anthracene	497.09	0.36	± 0.17	18
75	Benzol[<i>b</i>]chrysene	498.90	0.23	± 0.15	13
76	Picene	500.00			
77	Benzol[<i>ghi</i>]perylene	500.29	0.05	± 0.02	29
78	Dibenzol[<i>de</i> / <i>ino</i>]chrysene	504.10	0.11	± 0.05	21

* Standard compound; not present in coal tar PAH fraction.

** Could be methylphenylanthralene.

*** Could be cyclopentabenz[*a*]anthracene or cyclopentachrysene.

§ Standard *I* of "known" compounds used to calculate *I_R* for picene. See text. *I* determined by co-injection of compound with internal standards.

parison showed no significant improvement in precision or accuracy using the additional internal standards (*i.e.*, shorter intervals).

The GC operating parameters described in the Experimental section were designed for routine analysis of complex PAC mixtures. Subambient operating temperatures are avoided for practical reasons. Under these routine analytical conditions the major variations seen in the PAC *I* values are caused by differences in initial oven temperature, the temperature programming rate, and the initial isothermal period. Increasing the initial column temperature beyond the point at which naphthalene begins to migrate through the column will lead to decreased *I* values of the more volatile PACs (naphthalene to 2-methylfluorene). The converse also holds: lower initial temperatures result in increased *I* values. Subambient temperatures may be necessary to eliminate the dependency of the *I* values in Region II on initial column temperature. The *I* values of the three- and four-ring compounds are independent of the initial oven temperature at temperatures lower than 60°C. With the same carrier gas linear velocity and oven programming parameters (see Experimental section), the *I* values in the early part of Region II are generally higher with 0.2 mm I.D. columns than with 0.3 mm I.D. columns, a result of decreased solute retention in the larger-diameter columns. Operating the 0.3 mm I.D. column at a lower initial temperature would produce *I* values similar to those obtained on the 0.2 mm I.D. column. The effect of temperature programming rate on the PAC *I* values was evaluated on the same columns for rates of 3°C/min to 10°C/min. The more volatile PACs in Region II showed *I* increases at the faster program rates.

An initial isothermal period is useful for resolving compounds which are only slightly less volatile than the solvent. A 2-min initial time at the injection temperature

TABLE II
EFFECT OF VARIATION OF DURATION OF INITIAL ISOTHERMAL PERIOD ON *I*

<i>Compound</i>	0*	1**	2***
2-Methylnaphthalene	219.507	219.945	220.454
Biphenyl	235.601	236.146	236.756
Acenaphthylene	246.094	246.572	247.155
Dibenzofuran	258.150	258.586	259.068
Fluorene	269.263	269.588	269.955
Dibenzothiophene	295.514	295.552	295.635
Anthracene	301.359	301.351	301.364
3-Methylphenanthrene	319.442	319.417	319.468
1-Methylphenanthrene	323.818	323.823	323.872
Fluoranthene	344.432	344.439	344.471
Acephenanthrylene	347.762	347.793	347.811
Pyrene	351.412	351.428	351.484
Benzo[<i>a</i>]fluorene	366.838	366.831	366.886
Benzo[<i>b</i>]fluorene	369.522	369.527	369.554
Benzo[<i>b</i>]naphtho[2,1- <i>d</i>]- thiophene	389.323	389.263	389.295
Benzo[<i>ghi</i>]fluoranthene	390.129	390.075	390.106

* 0 = no initial isothermal period; temperature program started with injection.

** 1 = 1-min isothermal period of initial temperature.

*** 2 = 2-min isothermal period of initial temperature.

has been a routine part of the temperature program profile in this laboratory, and most retention data reported in this paper include that isothermal contribution. Obviously, the isothermal period prolongs the retention of compounds eluting from naphthalene up to approximately the methylfluorenes ($I = 288$); this is the reason for the less than exact agreement of the Region II data in this paper and the same data in the original paper¹¹. The original work did not include an isothermal period at the start of the GC run. Table II lists the I values for a number of PAHs which elute between naphthalene and phenanthrene, and representative compounds after phenanthrene from three coal tar analyses which had initial isothermal time periods of 0, 1, and 2 min. The I values in the first column are similar to the published values, while the next two columns show an increment in I with increasing initial isothermal time. Although Region II shows significant changes, the other regions do not.

TABLE III
COMPARISON OF ΔI UNDER WIDELY VARYING OPERATING CONDITIONS

<i>Compound</i>	<i>Coal tar*</i>	<i>Standard compounds**</i>	ΔI_1^{***}	ΔI_2^{\S}
2-Methylnaphthalene	220.524	218.14	2.78	2.90
1-Methylnaphthalene	223.308	221.04	13.54	12.92
Biphenyl	236.851	233.96	3.88	3.62
2,6-Dimethylnaphthalene	240.726	237.58	2.36	2.87
1,3- and 1,6-Dimethylnaphthalene	243.085	240.45	4.08	4.18
Acenaphthylene	247.167	244.63	1.77	1.86
1,2-Dimethylnaphthalene	248.935	246.49	4.59	4.80
Acenaphthene	253.528	251.29	5.57	5.88
Dibenzofuran	259.096	257.17	10.92	11.00
Fluorene	270.016	268.17	18.87	20.04
2-Methylfluorene	288.886	288.21	6.86	7.60
Dibenzothiophene	295.746	295.81	4.25	4.19
Phenanthrene	300.000	300.00	1.50	1.69
Anthracene	301.495	301.69	18.04	17.77
3-Methylphenanthrene	319.539	319.46	0.75	0.71
2-Methylphenanthrene	320.290	320.17	1.73	1.91

(Continued on p. 10)

TABLE III (continued)

Compound	Coal tar*	Standard compounds**	ΔI_1 ***	ΔI_2 §
4H-Cyclopenta[def]phenanthrene	322.018	322.08	1.18	1.02
9- and 4-Methylphenanthrene	323.194	323.10	0.78	0.80
1-Methylphenanthrene	323.972	323.90	8.92	8.69
2-Phenylnaphthalene	332.890	332.59	11.56	11.42
Fluoranthene	344.447	344.01	6.96	7.21
Pyrene	351.406	351.22	15.51	15.52
Benzo[a]fluorene	366.917	366.74	2.67	2.65
Benzo[b]fluorene	369.582	369.39	19.77	19.87
Benzo[b]naphtho[2,1-d]thiophene	389.353	389.26	8.67	8.90
Benz[a]anthracene	398.804	398.50	1.20	1.50
Chrysene	400.000	400.000		

* 0.30 mm I.D. column; 4°C/min program.

** Ref. 11.

*** ΔI from coal tar.

§ ΔI from standard compounds.

Table III lists I values obtained recently using the initial 2-min isothermal period and those obtained in the original work¹¹. In addition, the difference between each successive pair of indices from each data set is tabulated. It can be seen from these comparisons that the utility of the PAC I values lies not necessarily in the published I values themselves, but possibly in their description of relative elution order and position.

TABLE IV

COMPARISON OF REGION IV WIDTHS USING ACTUAL AND CALCULATED VALUES FOR PICENE t_R *

Retention parameter	Average	Standard deviation	95% confidence limits
t_R Picene (measured)	53.521	0.020	± 0.021
t_R Picene (calc.)	53.498	0.031	± 0.032
t_R Chrysene (measured)	40.584	0.012	± 0.013
Δt_R (Measured)	12.937	0.011	± 0.011
Δt_R (Calc.)	12.914	0.030	± 0.031

* Data in minutes.

TABLE V

RETENTION INDICES OF SULFUR POLYCYCLIC AROMATIC COMPOUNDS

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of determinations
1	Benzo[<i>b</i>]thiophene	201.57	0.02	± 0.03	4
2	7-Methylbenzo[<i>b</i>]thiophene	219.16	0.02	± 0.02	4
3	2-Methylbenzo[<i>b</i>]thiophene	220.76	0.04	± 0.03	7
4	5-Methylbenzo[<i>b</i>]thiophene	222.09	0.08	± 0.09	4
5	6-Methylbenzo[<i>b</i>]thiophene	222.11	0.05	± 0.09	3
6	3-Methylbenzo[<i>b</i>]thiophene	223.08	0.08	± 0.05	10
7	4-Methylbenzo[<i>b</i>]thiophene	223.15	0.03	± 0.05	3
8	5-Ethylbenzo[<i>b</i>]thiophene	236.14	0.04	± 0.04	5
9	3,5-Dimethylbenzo[<i>b</i>]thiophene	243.56	0.04	± 0.07	3
10	1,2,3,4,4a,4b-Hexahydrodibenzothiophene	271.69	0.04	± 0.05	4
11	Naphtho[1,2- <i>b</i>]thiophene	295.80	0.03	± 0.03	5
12	Dibenzothiophene	296.01	0.04	± 0.03	6
13	Naphtho[2,1- <i>b</i>]thiophene	300.00			3
14	Naphtho[2,3- <i>b</i>]thiophene	304.47	0.08	± 0.09	4
15	5-Methylnaphtho[2,1- <i>b</i>]thiophene	306.53	0.04	± 0.05	4
16	2-Methylnaphtho[2,1- <i>b</i>]thiophene	311.77	0.08	± 0.06	7
17	4-Methyldibenzothiophene	312.72	0.05	± 0.08	3
18	8-Methylnaphtho[1,2- <i>b</i>]thiophene	315.61	0.11	± 0.09	6
19	2-Methyldibenzothiophene	316.19	0.03	± 0.05	3
20	3-Methyldibenzothiophene	316.32	0.04	± 0.08	3
21	4-Methylnaphtho[1,2- <i>b</i>]thiophene	317.19	0.16	± 0.11	8
22	4-Methylnaphtho[2,1- <i>b</i>]thiophene	318.12	0.12	± 0.08	8
23	6-Methylnaphtho[1,2- <i>b</i>]thiophene	319.55	0.08	± 0.07	6
24	1-Methyldibenzothiophene	319.69	0.08	± 0.13	3
25	8-Methylnaphtho[2,1- <i>b</i>]thiophene	319.86	0.05	± 0.03	8
26	7-Methylnaphtho[2,1- <i>b</i>]thiophene	320.26	0.13	± 0.11	6
27	6-Methylnaphtho[2,1- <i>b</i>]thiophene	323.57	0.05	± 0.12	3
28	1-Methylnaphtho[2,1- <i>b</i>]thiophene	323.58	0.10	± 0.06	8

(Continued on p. 12)

TABLE V (continued)

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of determinations
29	9-Methylnaphtho[2,1- <i>b</i>]thiophene	325.25	0.02	± 0.02	7
30	3-Ethyl dibenzothiophene	328.34	0.02	± 0.06	3
31	4,6-Dimethyldibenzothiophene	329.17	0.10	± 0.25	3
32	2,6-Dimethyldibenzothiophene	332.42	0.15	± 0.13	3
33	2-Ethyl dibenzothiophene	332.65	0.07	± 0.78	3
34	3,6-Dimethyldibenzothiophene	332.88	0.03	± 0.05	3
35	2,8-Dimethyldibenzothiophene	335.90	0.04	± 0.07	4
36	3,7-Dimethyldibenzothiophene	336.02	0.07	± 0.11	4
37	3,8-Dimethyldibenzothiophene	336.09	0.14	± 0.25	3
38	1,7-Dimethyldibenzothiophene	339.36	0.06	± 0.14	3
39	Phenanthro[4,5- <i>bcd</i>]thiophene	348.75	0.12	± 0.05	30*
40	Phenalenol[6,7- <i>bc</i>]thiophene	353.45	0.01	± 0.01	4
41	Benzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	389.37	0.05	± 0.06	4
42	Benzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	392.92	0.07	± 0.08	4
43	Phenanthro[9,10- <i>b</i>]thiophene	394.96	0.02	± 0.03	4
44	Phenanthro[4,3- <i>b</i>]thiophene	395.03	0.05	± 0.05	5
45	Anthra[1,2- <i>b</i>]thiophene	395.39	0.10	± 0.17	3
46	Benzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	395.97	0.24	± 0.40	3
47	Phenanthro[1,2- <i>b</i>]thiophene	396.01	0.04	± 0.05	4
48	Phenanthro[3,4- <i>b</i>]thiophene	396.43	0.07	± 0.10	3
49	Anthra[2,1- <i>b</i>]thiophene	399.31	0.01	± 0.02	3
50	Phenanthro[2,1- <i>b</i>]thiophene	400.59	0.07	± 0.13	3
51	Phenanthro[3,2- <i>b</i>]thiophene	401.89	0.07	± 0.06	5
52	Phenanthro[2,3- <i>b</i>]thiophene	402.19	0.05	± 0.05	5
53	1-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	402.59	0.06	± 0.04	8
54	11-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	404.15	0.04	± 0.03	7
55	10-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	404.28	0.06	± 0.10	3
56	3-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	407.55	0.10	± 0.17	3
57	Anthra[2,3- <i>b</i>]thiophene	407.57	0.10	± 0.16	3
58	2-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	407.63	0.08	± 0.08	5
59	8-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	407.69	0.09	± 0.10	4
60	9-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	407.93	0.08	± 0.08	5
61	2-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	408.00	0.03	± 0.02	6

62	8-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	409.04	0.03	± 0.02	8
63	10-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	409.04	0.04	± 0.03	8
64	6-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	409.48	0.06	± 0.04	7
65	5-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	410.58	0.05	± 0.79	3
66	3-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	411.48	0.04	± 0.04	5
67	4-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	411.60	0.09	± 0.08	6
68	4-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	411.65	0.11	± 0.13	4
69	6-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	411.71	0.06	± 0.05	6
70	9-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	411.81	0.17	± 0.14	6
71	7-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	412.08	0.06	± 0.05	6
72	10-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	414.26	0.06	± 0.07	4
73	9-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	414.62	0.05	± 0.08	3
74	1-Methylbenzo[<i>b</i>]naphtho[2,1- <i>d</i>]thiophene	414.62	0.10	± 0.12	4
75	8-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	414.68	0.10	± 0.12	4
76	2-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	414.69	0.09	± 0.08	5
77	6-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	415.02	0.04	± 0.07	3
78	3-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	415.11	0.11	± 0.12	4
79	4-Methylbenzo[<i>b</i>]naphtho[1,2- <i>d</i>]thiophene	415.41	0.09	± 0.08	5
80	1-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	415.54	0.09	± 0.15	3
81	7-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	417.07	0.09	± 0.10	4
82	3-Methylphenanthro[9,10- <i>b</i>]thiophene	417.70	0.05	± 0.09	3
83	1-Methylanthra[2,1- <i>b</i>]thiophene	418.22	0.08	± 0.07	6
84	10-Methylphenanthro[2,1- <i>b</i>]thiophene	422.14	0.07	± 0.06	6
85	11-Methylbenzo[<i>b</i>]naphtho[2,3- <i>d</i>]thiophene	422.85	0.03	± 0.05	3
86	3-Methylphenanthro[2,1- <i>b</i>]thiophene	423.48	0.08	± 0.08	5
87	2-(2-Naphthyl)benzo[<i>b</i>]thiophene	430.65	0.09	± 0.09	5
88	Benzo[2,3]phenanthro[4,5- <i>bcd</i>]thiophene	443.29	0.06	± 0.05	5
89	Pyreno[4,5- <i>b</i>]thiophene	446.51	0.05	± 0.06	4
90	Benzo[1,2]phenaleno[3,4- <i>bc</i>]thiophene	447.66	0.11	± 0.11	5
91	Triphenyleno[4,5- <i>bcd</i>]thiophene	448.45	0.06	± 0.10	3
92	Pyreno[1,2- <i>b</i>]thiophene	449.30	0.08	± 0.14	3
93	Chryseno[4,5- <i>bcd</i>]thiophene	450.62	0.08	± 0.07	5
94	Pyreno[2,1- <i>b</i>]thiophene	455.01	0.18	± 0.17	5
95	Benzo[4,5]phenaleno[1,9- <i>bc</i>]thiophene	455.99	0.20	± 0.24	4
96	Benzo[4,5]phenaleno[9,1- <i>bc</i>]thiophene	457.30	0.16	± 0.15	5
97	Benzo[<i>b</i>]phenanthro[4,3- <i>d</i>]thiophene	470.47	0.06	± 0.07	4
98	Dinaphtho[2,1- <i>b</i> :1',2'- <i>d</i>]thiophene	472.62	0.08	± 0.14	3

(Continued on p. 14)

TABLE V (continued)

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of deviations
99	Dinaphtho[1,2- <i>b</i> :2',1'- <i>d</i>]thiophene	482.60	0.20	±0.19	5
100	Benzo[1,2]phenaleno[4,3- <i>bc</i>]thiophene	482.99	0.05	±0.05	5
101	Dinaphtho[1,2- <i>b</i> :1',2'- <i>d</i>]thiophene	486.58	0.14	±0.16	4
102	Benzo[<i>b</i>]phenanthro[9,10- <i>d</i>]thiophene	487.32	0.03	±0.31	5
103	Benzo[<i>b</i>]phenanthro[3,4- <i>d</i>]thiophene	487.76	0.05	±0.05	4
104	Anthra[1,2- <i>b</i>]benzo[<i>d</i>]thiophene	488.45	0.08	±0.14	3
105	Benzo[<i>b</i>]phenanthro[2,1- <i>d</i>]thiophene	488.89	0.13	±0.23	3
106	Dinaphtho[1,2- <i>b</i> :2',3'- <i>d</i>]thiophene	489.14	0.04	±0.05	4
107	9,13- <i>H</i> -Triphenyleno[2,3- <i>b</i>]thiophene	489.81	0.06	±0.04	6
108	Benzo[<i>b</i>]phenanthro[3,2- <i>d</i>]thiophene	491.02	0.03	±0.05	3
109	Benzo[<i>b</i>]phenanthro[1,2- <i>d</i>]thiophene	492.31	0.04	±0.07	3
110	Benzo[<i>b</i>]phenanthro[2,3- <i>d</i>]thiophene	493.31	0.06	±0.07	4
111	Triphenyleno[2,1- <i>b</i>]thiophene	493.90	0.05	±0.04	6
112	Triphenyleno[1,2- <i>b</i>]thiophene	494.41	0.10	±0.10	5
113	Dinaphtho[2,3- <i>b</i> :2',3'- <i>d</i>]thiophene	495.17	0.06	±0.06	5
114	Triphenyleno[2,3- <i>b</i>]thiophene	500.00			3
115	13-Methylbenzo[<i>b</i>]phenanthro[3,2- <i>d</i>]thiophene	511.19	0.04	±0.06	3

* Data from coal tar PAH analyses.

TABLE VI
RETENTION INDICES FOR NITROGEN POLYCYCLIC AROMATIC COMPOUNDS

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of determinations
1	1-Aminoindan	207.63	0.03	± 0.03	6
2	Quinoline	210.26	0.26	± 0.24	7
3	Isoquinoline	214.14	0.28	± 0.26	7
4	1-Methylindole	216.90	0.02	± 0.03	4
5	Indole	222.66	0.15	± 0.24	4
6	7-Azatindole	223.70	0.05	± 0.12	3
7	2-Methylquinoline	224.13	0.10	± 0.25	3
8	8-Methylquinoline	225.18	0.01	± 0.02	3
9	1-Methylisoquinoline	229.21	0.03	± 0.05	4
10	7-Methylquinoline	231.37	0.02	± 0.05	3
11	5-Aminoindan	232.12	0.14	± 0.22	4
12	3-Methylquinoline	232.47	0.32	± 0.40	5
13	7-Methylindole	235.49	0.05	± 0.06	5
14	4-Methylquinoline	235.77	0.04	± 0.06	4
15	3-Methylindole	239.20	0.15	± 0.24	4
16	2-Methylindole	240.10	0.30	± 0.37	5
17	2,7-Dimethylquinoline	244.04	0.03	± 0.05	4
18	2,6-Dimethylquinoline	244.19	0.27	± 0.25	7
19	1,2-Dimethylindole	244.42	0.12	± 0.15	5
20	2,2'-Bipyridyl	247.15	0.15	± 0.24	4
21	2,4-Dimethylquinoline	247.96	0.06	± 0.15	3
22	4-Azabiphenyl	252.33	0.03	± 0.05	4
23	2,5-Dimethylindole	256.65	0.09	± 0.14	4
24	1-Cyanonaphthalene	256.75	0.37	± 0.59	4
25	2,3-Dimethylindole	257.32	0.17	± 0.27	4
26	2-Cyanonaphthalene	260.88	0.08	± 0.20	3
27	5-Nitroindan	261.55	0.06	± 0.06	6
28	1-Aminonaphthalene	262.98	0.10	± 0.16	4

(Continued on p. 16)

TABLE VI (continued)

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of determinations
29	2-Aminonaphthalene	265.53	0.11	±0.18	4
30	2,3,5-Trimethylindole	273.61	0.08	±0.13	4
31	2-Aminobiphenyl	273.63	0.16	±0.25	4
32	1-Nitronaphthalene	274.95	0.09	±0.09	6
33	4-Azafluorene	279.85	0.11	±0.10	7
34	2-Nitronaphthalene	280.63	0.05	±0.06	5
35	3-Methyl-2-aminonaphthalene	283.73	0.05	±0.05	6
36	2-Nitrobiphenyl	290.25	0.12	±0.19	4
37	Phenazine	294.37	0.26	±0.32	5
38	4-Aminobiphenyl	298.05	0.08	±0.13	4
39	Benzofluquinoline	301.94	0.07	±0.06	7
40	Acridine	304.04	0.07	±0.06	7
41	Acridan (9,10-dihydroacridine)	304.11	0.08	±0.13	4
42	Benzofluquinoline	307.94	0.12	±0.11	7
43	Phenanthridine	307.94	0.12	±0.11	7
44	3-Nitrobiphenyl	310.09	0.11	±0.12	6
45	Carbazole	311.71	0.19	±0.24	5
46	4-Nitrobiphenyl	314.59	0.13	+0.21	4
47	3-Methylbenzofluquinoline	320.26	0.08	±0.07	7
48	2-Methylbenzofluquinoline	320.50	0.09	±0.14	4
49	2-Methylacridine	324.34	0.06	±0.06	7
50	1-Methylcarbazole	324.45	0.17	±0.27	4
51	4-Amino fluorene	325.11	0.09	±0.09	6
52	1-Amino fluorene	327.21	0.29	±0.36	5
53	3-Methylcarbazole	328.81	0.03	±0.04	5
54	3-Amino fluorene	329.08	0.22	±0.27	5
55	2-Methylcarbazole	329.61	0.09	±0.14	4
56	9-Methylacridine	331.15	0.05	±0.08	4
57	4-Methylcarbazole	331.88	0.03	±0.07	3
58	2-Amino fluorene	331.91	0.04	±0.06	4
59	6-Phenylquinoline	340.84	0.06	±0.07	5
60	1,4-Dimethylcarbazole	343.16	0.03	±0.07	3
61	2-Phenylindole	346.18	0.08	±0.10	5
62	1,2-Dimethylcarbazole	347.31	0.07	±0.11	4
63	2-Azafluoranthene	347.39	0.14	±0.13	7
64	1-Azafluoranthene	348.17	0.17	±0.21	5

65	1,3-Dimethylcarbazole	348.45	0.02	± 0.02	5
66	9-Cyanoanthracene	350.46	0.04	± 0.10	3
67	7-Azafluoranthene	350.50	0.06	± 0.15	3
68	9-Cyanophenanthrene	351.84	0.14	± 0.22	4
69	2-Nitrofluorene	353.06	0.19	± 0.20	6
70	4-Aminophenanthrene	353.97	0.17	± 0.18	6
71	9-Nitroanthracene	357.42	0.11	± 0.12	6
72	1-Azapyrene	357.73	0.28	± 0.26	7
73	4-Azapyrene	357.94	0.05	± 0.08	4
74	2-Azapyrene	362.43	0.10	± 0.16	4
75	1-Aminophenanthrene	362.62	0.08	± 0.10	5
76	1-Aminoanthracene	362.83	0.01	± 0.02	4
77	9-Aminophenanthrene	362.83	0.01	± 0.02	4
78	9-Aminoanthracene	363.91	0.25	± 0.31	5
79	Benzo[<i>le</i>]carbazole	363.92	0.10	± 0.16	4
80	3-Aminophenanthrene	365.60	0.44	± 0.70	4
81	2-Aminophenanthrene	365.80	0.26	± 0.65	3
82	2-Aminoanthracene	367.45	0.01	± 0.02	4
83	3,5-Diphenylpyridine	372.84	0.51	± 0.63	5
84	9-Phenylcarbazole	381.51	0.09	± 0.14	4
85	Benzo[<i>c</i>]acridine	392.60	0.15	± 0.14	7
86	Benzo[<i>d</i>]acridine	398.65	0.24	± 0.22	7
87	1-Azabenz[<i>g</i>]anthracene	400.00			4
88	4-Azachrysene	401.16	0.04	± 0.06	4
89	Benzo[<i>d</i>]carbazole	402.22	0.27	± 0.43	4
90	1-Azachrysene	407.18	0.03	± 0.04	5
91	Benzo[<i>b</i>]carbazole	409.63	0.43	± 0.68	4
92	3-Aminoanthracene	409.97	0.04	± 0.06	4
93	2-Azachrysene	411.49	0.11	± 0.12	6
94	Benzo[<i>c</i>]carbazole	411.89	0.13	± 0.32	3
95	4-Aminopyrene	412.31	0.22	± 0.27	5
96	2-Aminopyrene	413.83	0.26	± 0.41	4
97	1-Aminopyrene	415.39	0.17	± 0.19	4
98	1-Nitropyrene	421.48	0.24	± 0.30	5
99	2,2'-Biquinoline	422.56	0.03	± 0.04	5
100	7,9-Dimethylbenzo[<i>c</i>]acridine	438.32	0.01	± 0.02	4
101	5,7-Dimethylbenzo[<i>c</i>]acridine	438.38	0.03	± 0.05	4

(Continued on p. 18)

TABLE VI (continued)

Compound number	Compound name	Average I	Standard deviation	95% confidence limits	Number of determinations
102	7,10-Dimethylbenz[<i>a</i>]acridine	439.46	0.12	± 0.15	5
103	2-Aminobenzo[<i>c</i>]phenanthrene	450.10	0.07	± 0.09	5
104	4-Aminobenzo[<i>c</i>]phenanthrene	451.51	0.09	± 0.22	3
105	10-Azabenzo[<i>a</i>]pyrene	455.40	0.39	± 0.48	5
106	6-Aminochrysene	463.19	0.08	± 0.13	4
107	9,10,12-Trimethylbenz[<i>a</i>]acridine	466.79	0.20	± 0.32	4
108	Dibenz[<i>a,c</i>]phenazine	474.08	0.02	± 0.03	4
109	5-Aminochrysene	487.88	0.26	± 0.32	5
110	Dibenz[<i>a,h</i>]acridine	488.55	0.29	± 0.30	6
111	Dibenzo[<i>a,i</i>]carbazole	490.57	0.22	± 0.35	4
112	Dibenz[<i>a,j</i>]acridine	490.66	0.05	± 0.05	6
113	6-Nitrobenzo[<i>a</i>]pyrene	501.71	0.10	± 0.25	3
114	Dibenzo[<i>a,g</i>]carbazole	502.30	0.49	± 1.22	3
115	Dibenzo[<i>c,g</i>]carbazole	502.92	0.24	± 0.38	4
116	7-Aminobenzo[<i>a</i>]pyrene	511.98	0.15	± 0.24	4
117	6-Aminobenzo[<i>a</i>]pyrene	515.66	0.13	± 0.21	4

The concept of "routine analysis" implies rapid, simple, and reproducible analytical methodologies. An *I* system can facilitate routine GC analysis of complex mixtures by providing tentative identifications; however, spiking a complex sample with the internal standards can lead to co-elution problems and will definitely make quantitative determinations of masked or partly resolved peaks impossible. Ideally the internal standards should be indigenous to the sample being analyzed; the advantages of this situation are obvious. PAC samples generally contain relatively large amounts of three of the four PAC *I* standards. Rather than spiking the fourth standard (picene) into the mixture where it would not be resolved from benzo[ghi]perylene, would mask benzo[b]chrysene, and would interfere with the quantification of both peaks, a converging algorithm was written which uses the t_R of chrysene and the t_R and "standard" *I* of a known compound eluting in Region IV (benzo[e]pyrene is a good candidate) to optimize a calculated t_R for picene. This calculation assumes proportional elution behavior among all three compounds involved. Evidence supporting this assumption can be seen in Table IV where the average measured t_R values of chrysene and picene, the average width of the interval between them, the average calculated t_R of picene and respective interval width, and the standard deviations and 95% confidence limits of both intervals are given. The source of the data is six standard mixture analyses under the standard *I* conditions of this work in which the four PAC *I* standards and benzo[e]pyrene were present. The interval width between the t_R of chrysene and the calculated t_R of picene has three times the standard deviation of the actual width, but the averages vary only by 1.4 sec. Application of the Student's *t*-test suggests that the difference between the two means is statistically insignificant.

The relative nature of the PAC *I* system makes the statistical considerations in the preceding paragraph trivial. As long as retention data are generated in a consistent manner, it does not matter how the necessary cardinal points are defined; it is simply convenient to design a calculation to give a picene t_R similar to its actual retention time. All of the Region IV *I* values from the coal tar PAC data base reported in Table I are based upon a calculated t_R of picene.

The PAC *I* system has been applied to the analysis of diesel particulate material^{14,15}, coal extracts¹³, synthetic fuels^{16,17}, tissues and sediments¹⁸⁻²¹, air particulate matter²¹, and combustion effluents²². To broaden the applicability of the PAC *I* system, the *I* values of 232 PASHs and N-PACs were generated from standard mixtures, and are given in Tables V and VI, respectively.

It is not reasonable to expect ppm reproducibility from any *I* system within one's own laboratory, let alone from one laboratory to another. The degree of fine control over relevant chromatographic parameters required for that level of precision is simply not presently possible; and each researcher must invest developmental time in the analysis of a "standard" complex mixture in order to become familiar with the variations inherent in his own system. Published *I* values should be presented as good working estimates under given conditions, and should be viewed with the same attitude. Carefully generated linear *I* data provide essential information on elution order and intervals, but the actual numbers should not be expected to be absolute.

ACKNOWLEDGEMENT

This work was supported by the Department of Energy, Office of Health and Environmental Research, contract number DE-AC02-79EV10237.

REFERENCES

- 1 J. K. Haken, *Advan. Chromatogr.*, 154 (1976) 367.
- 2 L. S. Ettre, *Chromatographia*, 6 (1973) 489.
- 3 E. Kovats, *Helv. Chim. Acta*, 41 (1958) 1915.
- 4 F. J. Heeg, R. Zinburg, H. J. Neu and K. Ballschmiter, *Chromatographia*, 12 (1979) 451.
- 5 G. Schomburg and G. Dielmann, *J. Chromatogr. Sci.*, 11 (1973) 151.
- 6 J. Albaiges and X. Guerdino, *Chromatographia*, 13 (1980) 755.
- 7 L. Mathiasson, J. A. Jönsson, Å. M. Olsson and L. Haraldson, *J. Chromatogr.*, 152 (1978) 11.
- 8 H. Borowitzky, *PhD Dissertation*, Ruhr-Universität Bochum, Bochum, G.F.R., 1981.
- 9 L. Soják and J. A. Rijks, *J. Chromatogr.*, 119 (1976) 505.
- 10 H. Van den Dool and P. Dec. Kratz, *J. Chromatogr.*, 11 (1963) 463.
- 11 M. L. Lee, D. L. Vassilaros, C. M. White and M. Novotny, *Anal. Chem.*, 51 (1979) 768.
- 12 F. Sellier, G. Tersac and G. Guiochon, *J. Chromatogr.*, 219 (1981) 213.
- 13 G. Alexander and I. Hazai, *J. Chromatogr.*, 217 (1981) 19.
- 14 M.-L. Yu and R. A. Hites, *Anal. Chem.*, 53 (1981) 951.
- 15 J. A. Yergey, T. H. Risby and S. S. Lestz, *Anal. Chem.*, 54 (1982) 354.
- 16 C. Willey, M. Iwao, R. N. Castle and M. L. Lee, *Anal. Chem.*, 53 (1981) 400.
- 17 D. W. Later, M. L. Lee, K. D. Bartle, R. C. Kong and D. L. Vassilaros, *Anal. Chem.*, 53 (1981) 1612.
- 18 D. L. Vassilaros, P. W. Stoker, G. M. Booth and M. L. Lee, *Anal. Chem.*, 54 (1982) 106.
- 19 D. L. Vassilaros, D. A. Eastmond, W. R. West, M. L. Lee and G. M. Booth, *Proc. 6th Int. Symp. on Polynuclear Aromatic Hydrocarbons, Columbus, Ohio, October 27-29, 1981*.
- 20 G. V. Graas, J. W. deLeeuw, P. A. Schenk and J. Haverkamp, *Geochim. Cosmochim. Acta*, 45 (1981) 2465.
- 21 M. L. Lee, D. L. Vassilaros and D. W. Later, *Int. J. Environ. Anal. Chem.*, 11 (1982) 251.
- 22 S. Krishnan and R. A. Hites, *Anal. Chem.*, 53 (1981) 342.