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A study of interlaboratory influence on column evaluation

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Abstract

A liquid chromatography method for the characterization of base deactivated columns was investigated in a collaborative study involving six laboratories. This work was carried out on two chromatographic supports (Xterra RP 18 and Symmetry Shield). Different cooling systems, namely water bath and air oven, were tested and it was shown that column thermoregulation did not significantly influence chromatographic data. In order to control the mobile phase composition, the latter was prepared by weight rather than volume. Thanks to the injection of a set of selected neutral compounds, extra-column effects were evaluated in each of the participating laboratories. The results showed that chromatographic supports tested in different laboratories and following the same test protocol could be effectively compared.

Keywords: Interlaboratory study; High-performance liquid chromatography; Basic compounds; Neutral compounds

1. Introduction

Interlaboratory studies are considered as an important aspect in method validation during an analytical transfer. Besides reproducibility, it is essential to determine whether factors such as "laboratory equipment" or "preparation of mobile phases" introduce significant result dispersion [1,2]. For that purpose, it is necessary to perform a collaborative study involving at least six laboratories using the same procedure when analysing the same products [1]. Effectively, data stemming from a single laboratory are in no way sufficient to estimate method reproducibility [3].

In this work, a collaborative study was undertaken by six laboratories following the same experimental protocol related to a chromatographic test for the evaluation of base deactivated chromatographic columns [4,5]. All experiments were carried out with the same chromatographic columns to avoid possible variations due to the stationary phase. Each laboratory used its regular HPLC equipment. Buffer salts and mobile phase solvents were provided from local sources. The generating laboratory (laboratory 1) was in charge of providing test solutions in sealed ampoules to each participant.

A set of 10 (five neutral and five basic) test compounds was selected. Basic compounds were chosen to assess interlaboratory variability of retention, and asymmetry factors were used for the evaluation of base deactivated supports. Neutral compounds were included for the estimation of extra-column effects. In addition, the possible correlation between the nature of the analyte and interlaboratory variability was investigated for all measured chromatographic parameters (k, As and N).

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As already reported in the literature, column thermoregulation system is one of the main variables affecting interlaboratory variability [6]. Therefore, a comparison between air oven and water bath thermoregulation system was also performed by a limited number of laboratories.

Laboratory 1 was in charge of testing chromatographic supports at the beginning and at the end of the study to evaluate possible column performance deterioration.

In addition, for one of the two tested chromatographic supports (Xterra RP 18), inter-batch variability ($n = 4$) data, measured in the same laboratory, were determined and compared to interlaboratory data ($n = 6$).

2. Experimental

2.1. Chemicals and materials

The test solutes for this interlaboratory study were of analytical reagent grade. Chloroprocaine hydrochloride (CL) was provided by Orgamol (Evionnaz, Switzerland). Diphenhydramine hydrochloride (DP) and codeine (CO) were supplied by Siegfried (Zofingen, Switzerland). Fentanyl citrate (FN) was from Mcfarlan Smith Limited (Edinburgh, Scotland) and quinine hydrochloride (QN) from Hänseler AG (Herisau, Switzerland). All neutral compounds were obtained from Merck (Darmstadt, Germany), Fluka (Buchs, Switzerland), Janssen (Beerse, Belgium), Aldrich (Steinheim, Germany) at the highest available purity.

Acetonitrile was from SDS (Peypin, France). Water was obtained with the Milli-Q Water Purification System from Millipore (Milford, MA, USA). Aqueous buffer was prepared with di-potassium hydrogen phosphate anhydrous and potassium di-hydrogen phosphate (Fluka-Buchs, Switzerland) by measuring the pH with a Metrohm pH meter (Herisau, Switzerland).

Principal component analysis was performed with the Simca P software package (Umetrics, Sweden).

2.2. Test solutions and columns

Each laboratory was provided with sealed ampoules containing the test solutions, which were simply diluted to 10 ppm in the mobile phase immediately before injection in the HPLC system.

The participants received two columns: a Symmetry Shield (150 mm × 4.6 mm i.d., 5 μm) and an Xterra RP18 (150 mm × 4.6 mm i.d., 5 μm), both manufactured by Waters® (Milford, USA). Both columns were sent from laboratory to laboratory to make sure that all possible interlaboratory differences were not due to column (or batch) variability.

2.3. Conditions and procedure

In order to assess the most realistic estimation of interlaboratory variability, each participating laboratory was asked to use its routine HPLC equipment. A list of general criteria was specified concerning operating conditions. As an example, data acquisition rate and detector response time were, respectively, fixed at 20 Hz and 0.1 s. Both equilibrating and cleaning procedures were specified in the analytical protocol. The mobile phase was prepared by weight rather than volume and it was composed of acetonitrile – pH 7.0, 0.0375 M phosphate buffer (31.1:60.0 w/w), corresponding to (40:60; v/v) of reference [4]. The injection sequence was randomised for each laboratory. The detection wavelength was set at 215 nm, flow rate at 1.0 ml/min and analyses were carried out at 30 °C.

Three chromatographic parameters (k, As and N) were measured according to the following equations:

Retention factor:

$$k = \left(\frac{t_r - t_0}{t_0}\right) \quad (1)$$

where t_r was the compound retention time and t_0 the column void volume retention time (measured with $NaNO_3$);

Asymmetry:

$$As = \frac{1}{2} \times \left(1 + \frac{B}{A}\right) \quad (2)$$

where A and B were evaluated at 5% of the peak height;

Efficiency:

$$N = 5.54 \times \left(\frac{t_r}{w_{1/2}}\right)^2 \quad (3)$$

where $w_{1/2}$ was the peak width at 50% of the peak height.

3. Results and discussion

According to the previously developed chromatographic test [5], column performance was assessed by measuring retention (k) and asymmetry factors (As) of a reduced number of basic test compounds. Moreover, neutral compounds (*N*,*N*-diethylacetamide, phenol, nitrobenzene, anisole and naphthalene) were included to determine extra-column effects in this study. Interlaboratory variability ($n = 6$) of k and As was calculated in terms of relative standard deviation (R.S.D., in %) for each test compound on both stationary phases.

3.1. Estimation of extra-column effects

It is well known that extra-column volumes, such as tubing, injector, detector cell, etc. can decrease chromatographic performance. Therefore, the observed efficiency, N_{obs}, determined from Eq. (3) may be lower than the actual column efficiency, N_{col}, particularly for the less retained solutes. For this reason, the set of neutral compounds selected for this collaborative study was injected by each participating laboratory to estimate extracolumn effects. For a given compound, the

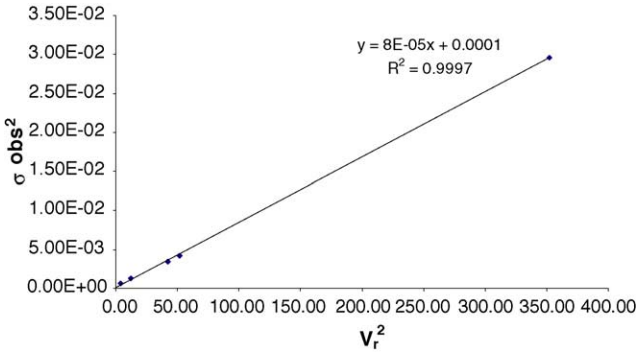

Fig. 1. Estimation of extra-column effects in lab 1.

observed peak variance, σ^2_{obs} is related to N_{obs} by

$$\sigma^2_{obs} = \left(\frac{V_r^2}{N_{obs}}\right) \quad (4)$$

where V_r is the retention volume of the compound, σ^2_{obs} is the sum of variances due to column dispersion, σ^2_{col} and to extracolumn dispersion, σ^2_{ec} according to

$$\sigma^2_{obs} = \sigma^2_{col} + \sigma^2_{ec} \quad (5)$$

σ^2_{col} being given by

$$\sigma^2_{col} = \left(\frac{V_r^2}{N_{col}}\right) \quad (6)$$

For this reason, the various HPLC systems afford a chromatographic peak efficiency (N_{obs}), which is always lower than the real column efficiency (N_{col}).

The σ^2_{ec} value is specific to a particular HPLC system and therefore may be very different in the laboratories. N_{col} and σ^2_{ec} were estimated in all participating laboratories from the following linear equation:

$$\sigma^2_{obs} = \frac{V_r^2}{N_{col}} + \sigma^2_{ec} \quad (7)$$

Thus, by plotting σ^2_{obs} as a function of V_r^2 for neutral compounds, it is possible to determine N_{col} and σ^2_{ec} (see Fig. 1 for lab 1).

The maximum peak variance increase tolerated from extra-column dispersion is 10% ($\sigma^2_{ec}/\sigma^2_{obs} < 0.1$). This value was suggested by Klinkenberg in 1960 and has been accepted as the criteria for extra-column dispersion [7]. In the case of two laboratories, significative extra-column effects ($\sigma^2_{ext}/\sigma^2_{obs} > 0.1$) were observed for early eluting peaks. In all other laboratories, extra-column effects were negligible. Thus, a significant "laboratory effect" was reported on measured efficiency (N_{obs}), a chromatographic parameter which is particularly affected by extra-column effects. The estimated column efficiency values (N_{col} in Eq. (7)) were 11,950, 11,900, 11,660, 11,970, 11,630, 11,920 for labs 1–6, respectively. It is interesting to note that column efficiency did not change from lab to lab and that it corresponds to a reduced plate height $h = 2.5$. It is somewhat higher than the one expected (9000) by the empirical rule $N \approx 3000 \times L/d_p$ [8], L being the column length in cm and d_p the packing particle diameter in μm.

In fact, this empirical equation has been calculated for $h = 3.3$. It will be verified (at least) only if $h \leq 3.3$, i.e. if:

$$\sigma_{tot} \leq \frac{V_R}{\sqrt{3000}} \times \sqrt{\frac{d_p}{L}} \quad (8)$$

corresponding to

$$w_{1/2} \leq 0.043 \times t_r \times \sqrt{\frac{d_p}{L}} \quad (9)$$

This case is not verified for early eluting peaks for which extra-column effects are important. Efficiency values of 5931, 9354, 12,121 and 12,281 were, respectively, observed for *N*,*N*-diethylacetamide, phenol, nitrobenzene and anisole (in one of the participating laboratories), confirming the fact that only early eluted peaks were affected by extra-column effects (only *N*,*N*-diethylacetamide does not satisfy the condition given by Eq. (9)).

3.2. Comparison of two column thermoregulation systems

As reported in the literature, a major variable during method transfer was identified as the temperature control of the columns [9,10]. The worst reproducibility came from metal block ovens, without a fan driven circulating air supply. This would mean that not all laboratories could take part in interlaboratory studies [6] if tight specifications have to be respected concerning temperature control.

For this reason and in order to assess interlaboratory variability of column evaluation as close as possible to reality, a comparison between two temperature control systems was performed.

Three out of the six laboratories accepted to perform the chromatographic test with both an air oven and a water jacket thermoregulation system. This study was assessed

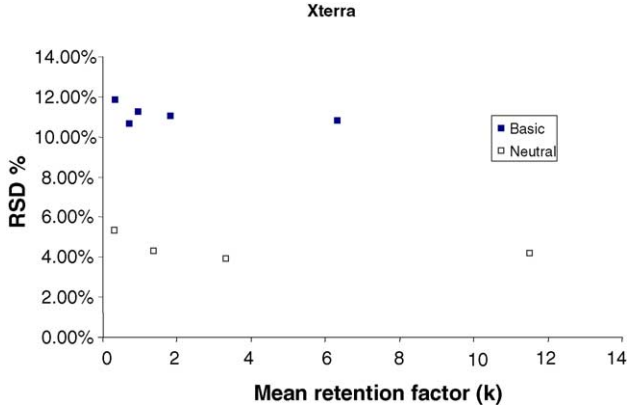

Fig. 2. R.S.D. (%) of retention factors on Xterra RP_{18}.

Table 1
Retention factors obtained in the three laboratories with both thermoregulation systems and Fisher test

	Water jacket						Air oven						s^2_{water}	s^2_{air}	F_{obs}	F_{theo}
	Lab 1		Lab 2		Lab 3		Lab 1		Lab 2		Lab 3					
	Inj 1	Inj 2	Inj 1	Inj 2	Inj 1	Inj 2	Inj 1	Inj 2	Inj 1	Inj 2	Inj 1	Inj 2				
Xterra RP18																
Codeine	0.40	0.40	0.37	0.36	0.31	0.31	0.39	0.39	0.36	0.35	0.30	0.31	0.002	0.002	1.133	5.050
Chloroprocaine	1.09	1.10	0.98	0.98	0.85	0.85	1.07	1.07	0.96	0.94	0.83	0.82	0.012	0.012	1.000	5.050
Quinine	0.83	0.84	0.74	0.74	0.66	0.66	0.82	0.81	0.73	0.72	0.63	0.63	0.006	0.007	1.087	5.050
Diphenhydramine	2.09	2.09	1.85	1.84	1.65	1.65	2.08	2.08	1.78	1.78	1.59	1.59	0.039	0.048	1.234	5.050
Fentanyl	7.03	7.04	6.30	6.32	5.70	5.70	6.95	6.96	6.20	6.21	5.52	5.53	0.358	0.409	1.142	5.050
N,*N*-Diethylacetamide	0.34	0.34	0.34	0.34	0.32	0.32	0.34	0.35	0.35	0.34	0.32	0.32	0.000	0.000	2.996	5.050
Phenol	1.36	1.36	1.35	1.35	1.30	1.30	1.42	1.42	1.38	1.38	1.32	1.32	0.001	0.002	2.499	5.050
Nitrobenzene	3.32	3.33	3.28	3.28	3.15	3.16	3.45	3.45	3.36	3.35	3.21	3.21	0.006	0.011	1.790	5.050
Anisole	3.79	3.79	3.64	3.63	6.27	6.26	3.92	3.93	3.83	3.84	6.38	6.38	1.748	1.670	1.046	5.050
Naphthalene	11.41	11.43	10.79	10.84	10.61	10.61	11.85	11.87	11.41	11.44	10.83	10.83	0.142	0.216	1.520	5.050
Symmetry Shield																
Codeine	0.64	0.64	0.65	0.65	0.55	0.55	0.66	0.66	0.61	0.62	0.57	0.57	0.002	0.001	3.67	5.050
Chloroprocaine	2.02	2.02	1.91	1.90	1.65	1.65	2.05	2.05	1.87	1.86	1.70	1.71	0.028	0.009	3.29	5.050
Quinine	1.48	1.48	1.46	1.46	1.28	1.27	1.51	1.51	1.44	1.44	1.31	1.31	0.010	0.006	1.79	5.050
Diphenhydramine	4.38	4.38	4.28	4.27	3.77	3.77	4.46	4.47	4.22	4.22	3.85	3.85	0.085	0.046	1.85	5.050
Fentanyl	11.23	11.25	11.19	11.18	9.97	9.95	11.48	11.48	10.90	10.88	10.08	10.10	0.421	0.214	1.97	5.050
N,*N*-Diethylacetamide	0.53	0.53	0.56	0.55	0.51	0.51	0.55	0.55	0.55	0.55	0.52	0.51	0.000	0.000	1.20	5.050
Phenol	2.02	2.02	2.15	2.15	2.01	2.01	2.11	2.11	2.25	2.25	2.00	2.00	0.005	0.021	4.23	5.050
Nitrobenzene	5.02	5.02	5.34	5.35	5.05	5.06	5.23	5.23	5.60	5.60	5.04	5.04	0.025	0.105	4.12	5.050
Anisole	5.92	5.93	6.26	6.27	11.13	11.13	6.14	6.17	6.56	6.56	11.14	11.12	6.786	6.956	1.03	5.050
Naphthalene	18.76	18.78	20.10	20.14	19.16	19.12	19.54	19.55	21.36	21.40	19.18	19.18	0.390	1.619	4.15	5.050

on chromatographic parameters of basic and neutral test compounds measured on the selected stationary phases.

Retention factors measured in the three labs with the two temperature control systems are reported in Table 1 and a Fisher test was performed on this set of chromatographic parameters in order to compare interlaboratory variability with both water jacket and air oven system. For all the tested compounds, and on both chromatographic supports, the observed F values were lower than the theoretical F value. This indicates that the temperature control system does not influence the interlaboratory variability of retention factors, for both basic and neutral compounds. All participating laboratories selected the air oven as temperature controller.

3.3. Interlaboratory variability of retention and asymmetry factor

According to the previously developed chromatographic test [5], column performance was assessed by measuring retention (k) and asymmetry (As) factors of a reduced number of basic test compounds. Moreover, neutral compounds (*N*,*N*-diethylacetamide, phenol, nitrobenzene, anisole and naphthalene) included in this study were used for determining a possible correlation between lab-to-lab variability and the nature of the tested compound. Interlaboratory variability ($n = 6$) of k and As was calculated in terms of relative standard deviation (R.S.D., in %) for each test compound, on both stationary phases. Retention factors measured on Xterra RP 18 support were reported in Fig. 2 and a marked split between the

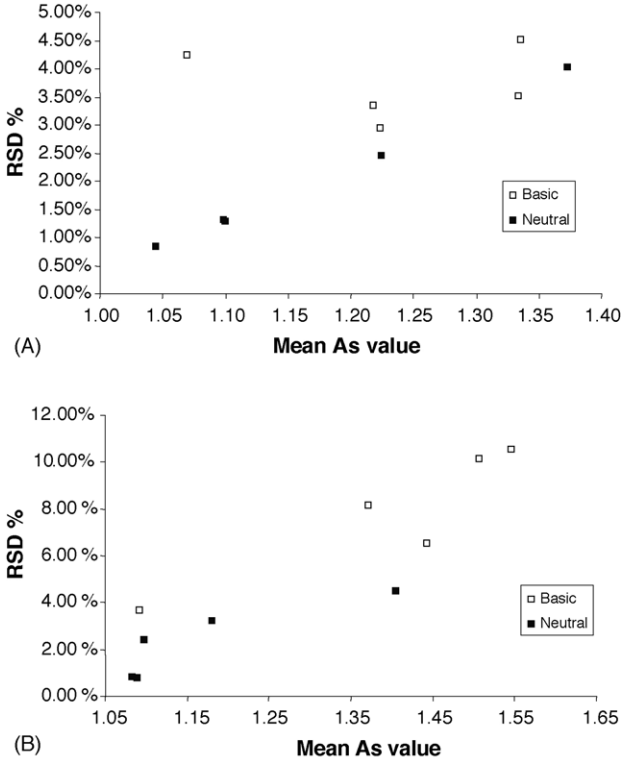

Fig. 3. R.S.D. (%) of asymmetry factors on (A) Xterra RP_{18} and (B) Symmetry Shield.

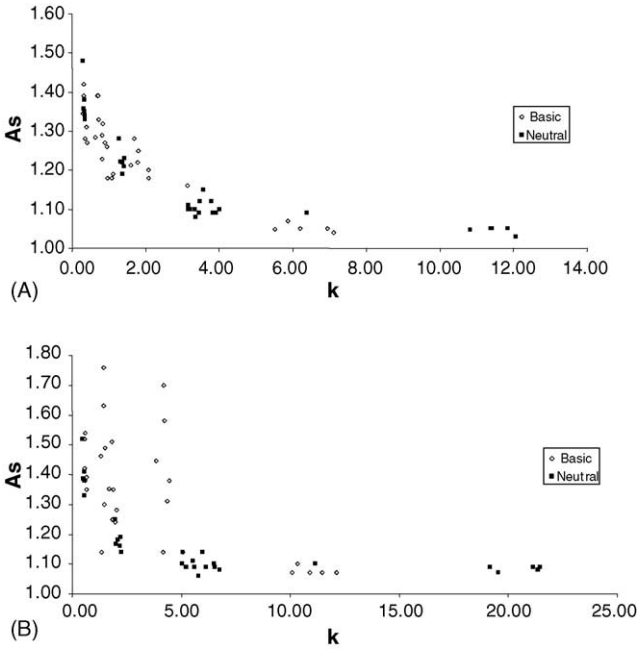

Fig. 4. Asymmetry values vs. retention factors of all participating laboratories (A: Xterra RP 18 support and B: Symmetry Shield support).

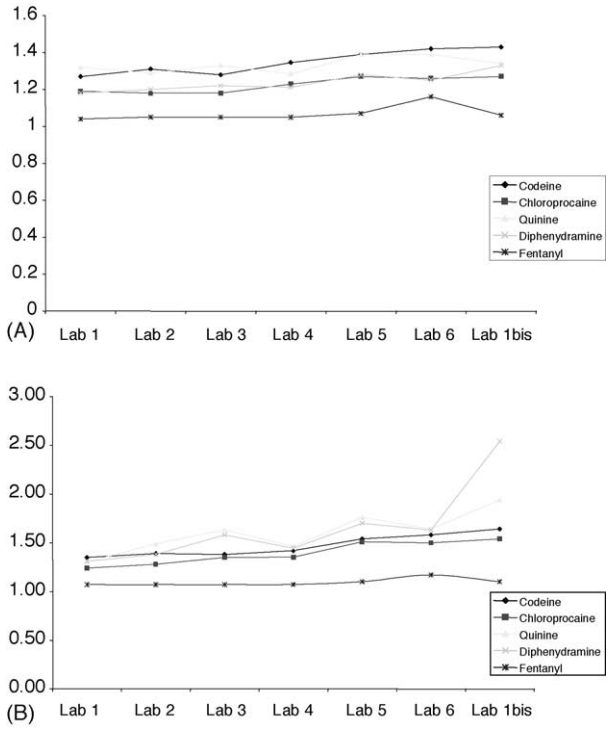

Fig. 5. Asymmetry values measured on both chromatographic supports (A: Xterra and B: Symmetry Shield) at the beginning and at the end of the collaborative study.

interlaboratory variability of basic and neutral compounds was observed. R.S.D. value was lower than 6.00% for neutral compounds and higher than 10.00% for basic compounds. With the Symmetry Shield column, the interlaboratory variability was of the same magnitude (≈6.00%) both for basic and neutral compounds (data not shown). In all cases, interlaboratory variability seems to be independent of solute retention.

Concerning asymmetry factors, an interlaboratory variability (R.S.D., in %) increase with peak tailing (Fig. 3) was observed on both studied supports. In addition, when plotting As in function of *k*, there was a correlation (Fig. 4), indicating that the high interlaboratory variability for the less-retained solutes is partly due to extra-column effects. On the Symmetry Shield column, a higher variability was found for the asymmetry values of basic compound. These results indicate that chromatographic reproducibility is certainly affected by secondary unwanted interactions with silanol groups and thus, particular care should be taken when evaluating the performance of base deactivated supports with a set of basic test compounds.

3.4. Assessment of chromatographic support performance

In order to make sure that column degradation did not occur during the study, the originating laboratory was in charge of testing the chromatographic supports at the beginning and at the end of the process.

Fig. 5 gives the asymmetry values of basic compounds on both chromatographic supports, from the beginning to the end of the interlaboratory study.

Chromatographic performances of the two supports kept constant along the study (from laboratories 1 to 6) and a significant peak tailing increase was observed only for two basic substances (quinine and diphenhydramine) on Xterra support at the end of the study.

3.5. Comparison of interlaboratory and batch variability

Xterra support was previously evaluated with the chromatographic test [11]. The batch-to-batch variability for this support was one of the lowest among all the tested base deactivated supports. For this reason, this support was retained to compare the interlaboratory and batch variability.

In the case of Xterra RP_{18} support, both interlaboratory ($n = 6$) and batch ($n = 4$, four different batches tested in one laboratory) variability data on k, As and *N* chromatographic parameters were investigated. In order to compare the lab-to-lab and batch-to-batch variability, R.S.D. values were calculated for each tested compound and chromatographic parameters as well as squared mean R.S.D. were measured on all the neutral and basic compound chromatographic parameters. Results for retention and asymmetry factor of Xterra support are, respectively, reported in Tables 2 and 3.

With squared mean R.S.D. values, variability was generally higher on the chromatographic parameters of basic compounds, in comparison to neutral compounds, which confirmed the previous results on only “pure” interlaboratory

Table 2
Interlaboratory and batch variability of retention factor (Xterra RP 18)

Substance	R.S.D. (%)			
	Lab-to-lab ($n = 6$)	Squared mean	Batch-to-batch ($n = 4$)	Squared mean
Codeine	11.83		8.45	
Chloroprocaine	11.23		2.66	
Quinine	10.64	11.11	28.89	15.08
Diphenhydramine	11.01		14.88	
Fentanyl	10.81		1.71	
N,*N*-Diethylacetamide	5.29		6.37	
Phenol	4.31	4.45	2.15	4.85
Nitrobenzene	3.90		2.48	
Naphthalene	4.16		6.53	

Table 3
Interlaboratory and batch variability of asymmetry factor (Xterra RP 18)

Substance	R.S.D. (%)			
	Lab-to-lab ($n = 6$)	Squared mean	Batch-to-batch ($n = 4$)	Squared mean
Codeine	4.52		1.16	
Chloroprocaine	3.34		3.92	
Quinine	3.50	3.27	25.34	13.77
Diphenhydramine	2.94		15.24	
Fentanyl	1.05		7.53	
N,*N*-Diethylacetamide	4.02		2.30	
Phenol	2.46	2.48	7.24	
Nitrobenzene	1.29		2.92	4.08
Naphthalene	0.83		0.54	

variability. It is interesting to note that batch-to-batch variability was even higher than interlaboratory variability for the same substances. It was mainly due to the high variability between the four batches, for two basic substances (quinine and diphenhydramine), in terms of retention factor as well as asymmetry value.

Finally, chromatographic parameters (of basic compounds) were treated by principal component analysis (PCA), according to a previously developed methodology [5]. The score plot is presented in Fig. 6. The first axis (PC 1) represents 64% of total variance. Asymmetry factors contribute more than retention factors to this axis, whereas for the second axis (PC 2), which represents 24% of total variance, retention factors contribute more than asymmetry factors. Interlaboratory and batch variability were of the same order of magnitude in the score plot, but in orthogonal direction. According to the loading plot (plot not shown), interlaboratory differences were mainly correlated to operating conditions, such as mobile phase preparation, column temperature, etc., which affect the retention factor rather than asymmetry. On the other hand, differences between batches were mostly related to the stationary phase properties, such as column packing, silica support properties, etc., which can influence the asymmetry factor.

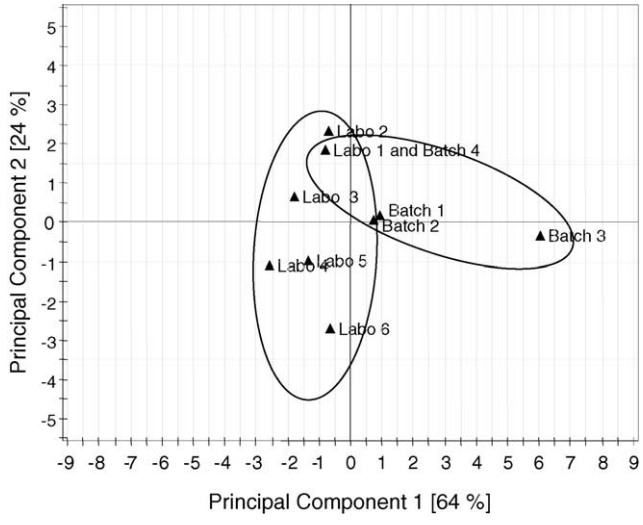

Fig. 6. PCA score plot of interlaboratory and batch variability.

4. Concluding remarks

This work sought to demonstrate that the chromatographic supports tested in different laboratories could be compared. The following conclusions were drawn:

- basic compounds show a higher interlaboratory (and batch) variability in comparison to neutral compounds in the same chromatographic conditions. Thus, particular care should

be taken when evaluating base deactivated supports for the analysis of these substances;

- the use of neutral compounds is a quick and efficient method for the estimation of extra-column effects—a necessary and important point to assess in a collaborative study;
- comparison of the influence on interlaboratory variability of two different thermoregulation systems allows to conclude that this parameter does not have a significant influence on the response. For this reason, there are no tight restrictions concerning the use of a water jacket system rather than an air oven system;
- by comparing the lab-to-lab and batch-to-batch variability of same supports, it was demonstrated that columns, tested in different laboratories but following strictly the same protocol, can be compared. Interlaboratory variability ($n = 6$) with a particular stationary phase was in fact comparable, and even lower than batch variability ($n = 4$).

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