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Temperature-dependent refractive index issues using a UV–visible detector in high-performance liquid chromatography

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Abstract

While studying the properties of some potential, UV-detection compatible, mobile phase modifiers for reversed-phase liquid chromatography (RPLC) we encountered serious detector baseline problems. The most severe problem was a periodic saw-tooth-shaped drift. This drift was most noticeable when using water–THF and water–dioxolane mobile phases. We demonstrate that the baseline problems are related to temperature fluctuation in a water bath used to maintain a constant column temperature. The temperature fluctuations change the refractive index (RI) of the mobile phase, which in turn affects the amount of light reaching the detector. Our results show that the magnitude of the baseline drift is related to the RI value of the mobile phases. The baseline problems can be alleviated using a different temperature controller whose feedback mechanism assures smoother operation. Since there is no single and complete source of information in the literature concerning the RI values of typical mobile phases in RPLC, we decided to measure them as a function of the % modifier (between 0 and 100%) and the temperature (between 25°C and 50°C). Knowing the RI values allows us to calculate the excess volume of a binary liquid system. © 2002 Elsevier Science B.V. All rights reserved.

Keywords: Detection, LC; Refractive index detection; Temperature effects; Modifiers; Mobile phase composition

1. Introduction

The number of diverse mobile phases available for use in normal-phase liquid chromatography is relatively large. Reversed-phase chromatography has a much poorer selection of mobile phases. In addition to pH manipulations, typical aqueous mobile phases contain methanol (MeOH), acetonitrile (ACN) or tetrahydrofuran (THF) as the modifier. Mobile phases containing a combination of the above modifiers are used routinely. Of the above three modifiers,

MeOH and ACN are most commonly used. THF is used less frequently, partly because of difficulties with UV detectors baselines.

In our studies we have examined the 1,3-dioxolane as a possible aqueous modifier, since it has the appropriate properties required from a modifier: low viscosity, miscibility with water, UV transparency down to about 220 nm, minimum toxicity, readily available and relatively inexpensive. The purpose of the study was to compare 1,3-dioxolane to the three modifiers mentioned above. The results of this comparison will be reported in a separate publication. In the course of the study we encountered baseline problems, mainly when using water–THF and water–dioxolane mobile phases. We found that

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the detector (UV) baseline wanders, and superimposed on the wandering, was a periodic signal in the shape of saw-teeth (Fig. 1B). The source of the unwanted periodic signal in our case, as will be shown in the Results and discussion section below, is related to temporal fluctuation in the refractive index (RI) of the mobile phase.

While there are papers in the scientific literature discussing the general phenomenon of baseline noise

in chromatography and how to extract useful information from noisy signals (viz. [1–7]), the discussion dealing with the sources of the noise is often limited to books [8,9] and specialized articles [10,11] dealing with various aspects of troubleshooting. Not much appears in the literature concerning refractive index (RI) issues in conjunction with UV–Vis detectors. Peck and Morris [12] describe some of the common noise sources with the UV–Vis detector.

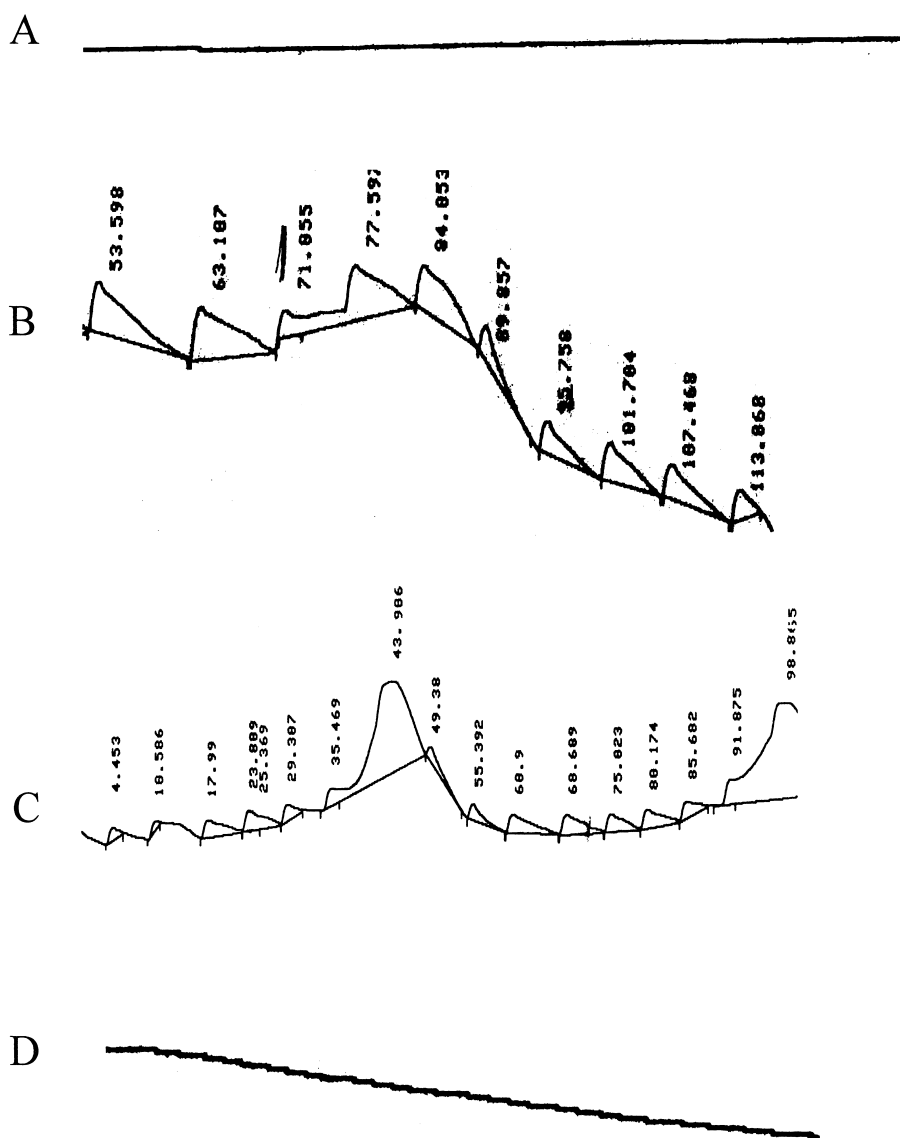


Fig. 1. The shape of the detector (UV–Vis) baseline in the case of a mixture of water and: (A) MeOH or ACN; (B) THF or 1,3-dioxolane (using TEP-1); (C) THF or 1,3-dioxolane (using TEP-3) and (D) THF (using column oven).

They cite RI changes in the flow-cell as a result of mobile phase impurities, temperature and composition gradients in the mobile phase, mobile phase flow-rate and pressure fluctuations, and solvent nonhomogeneity due to incompletely mixed solvents. Also, they mention flow-cell temperature, which may vary during operation due to heat dissipation from the electronic circuits and to the illumination of the light source, as a source of detection error. Evans and co-workers [13] discussed in details the contribution of detector cell geometry to the RI of the mobile phase in that cell and the effect of the RI on the UV–Vis detector signal. Renn and Synovec [14] suggested the use of dual-wavelength absorbance detection to compensate for RI-related baseline drifts in thermal gradient μ LC systems. Carr and his co-workers [15], while not discussing directly RI difficulties, describe the deleterious effect of temperature mismatch in the case of high-temperature LC operation. Furthermore, Poppe and Kraak [16,17] showed that significant differences ($>5^{\circ}\text{C}$) in temperature between the incoming eluent and the column can greatly degrade column efficiency and can cause severe band broadening and peak splitting. Again, while Poppe and Kraak did not discuss RI effects, some of their results could be explained in terms of RI fluctuations.

The contribution of variation in mobile phase RI, due to temperature fluctuation in the column temperature control system, to the noise of a UV–Vis detector was not discussed previously. Also, the importance of the mobile phase composition to this source of noise was not studied. The fact that the RI of the mobile phase can control the signal quality of a UV–Vis detector makes it imperative that we understand the dependence of the RI on the mobile phase temperature and composition.

Much to our surprise, not much can be found in the chromatographic and general scientific literature [18–20] about the RIs of binary aqueous mixtures that are being used as mobile phases in reversed-phase liquid chromatography. We report here the RIs of four binary mixtures; water–methanol, water–acetonitrile, water–THF and water–1,3-dioxolane, over the entire range of composition and over the temperature range of 25–50°C.

In addition, the RIs allow us to calculate the excess volumes of the examined binary mixtures.

Excess volumes are indicative of the nature of the interactions between the components of the binary mixture, and as such they can be of importance to the chromatographer in his assessment of the retention processes that occur in the column during the separation. We report here the excess volumes for the four mobile phases calculated at 298.15°K.

2. Experimental

2.1. Apparatus

The chromatographic system consisted of a LC-10AT pump (Shimadzu, Japan) and a UV-detector model SPD-10A (Shimadzu, Japan) operated at 254 nm wavelength. The data were collected with a Shimadzu C-R5A integrator (Shimadzu, Japan).

We used a reversed-phase column, RP-18 (Merck, Germany), Lichrospher 100, 5 μm .

During the study, the column temperature was maintained constant either by immersing the column in a water bath or by using a forced-air column oven (model CTO-10Avp, Shimadzu, Japan). With the water bath, the temperature was controlled with two different immersion thermostats (TEP-1 and TEP-3) manufactured by Fried (Israel). The specifications of these temperature controllers are listed in Table 1.

The RIs were measured with a Carl Zeiss refractometer model 56477 (Carl Zeiss, Germany). The temperature of the refractometer was controlled by the TEP-1 thermostat unit.

2.2. Materials and methods

Acetonitrile and methanol were supplied from J. T. Baker (USA). Tetrahydrofuran was bought from Bio-Lab Ltd (Israel). 1,3-Dioxolane was supplied from Fluka (Germany). All of the solvents were HPLC grade, except for the 1,3-dioxolane that was 99%. None of the 1,3-dioxolane interfered with our work.

All of the mobile phases used in the chromatographic study consisted of a binary mixture of modifier and water in the mole ratio of 1 modifier to 5.25 mole water. Table 2 shows the ratio of the mobile phases in percent volume.

The binary mixtures used in the RI measurements

Table 1
Heating units specifications

Thermostat, manufacturer	Controller type	Temperature fluctuation around set point (°C)
Immersion thermostat TEP-1, Fried Electric (Israel)	Mechanical	±(0.5–1)
Immersion thermostat TEP-3, Fried Electric (Israel)	Mechanical	±(0.1–0.3)
Column oven (CTO-10Avp), Shimadzu (Japan)	Digital	±(0.1–0.2)

were mixed as percent volume from 0% modifier to 100% modifier in increments of 10%. The measurements were done over the temperature range of 25°C to 50°C in increments of 5°C.

3. Results and discussion

3.1. Baseline fluctuation

In the course of our studies to find new mobile phases for reversed-phase liquid chromatography we found that while mobile phases containing water–methanol and water–acetonitrile gave smooth detector baselines (Fig. 1A), water–THF and water–1,3-dioxolane did not (Fig. 1B). A periodic noise such as shown in Fig. 1B is due, most likely, to the coupling of the detector output to some repeating mechanical or electrical action. Our initial attempts to isolate the source of the periodic noise involved the pumping system. However, changing the pump, or even shutting down the pump altogether, did not eliminate or even reduce the periodic noise of the system. Replacing the detector with a different UV–Vis detector Model L-4000A (Merck-Hitachi, Japan) also

didn't eliminate the periodic noise in the detector's baseline.

When we changed the temperature of the water bath we observed that the frequency of the baseline noise changed as well, increasing with an increase in the bath's temperature. However, shutting down the TEP-1 temperature controller did eliminate the periodic noise. Changing to a more sensitive controller (TEP-3) reduced to some extent the periodic noise but did not eliminate it (Fig. 1C). Replacing the water bath with a forced-air column oven eliminated the periodic noise altogether while controlling the temperature of the column (Fig. 1D).

The fact that we did not see any major baseline fluctuation with the water–methanol and water–acetonitrile mobile phases ruled out electronic noises from the temperature controllers as the cause of the observed noise. This last observation together with the results of all other tests mentioned here led us to suspect that the baseline problems is due to fluctuation in the RI of the mobile phase. The RI is very sensitive to temperature fluctuations. Thus, as the bath temperature oscillated between the controller set points, the RI of the mobile phase changed as well, tracking the temperature changes in the water bath. Changing the RI of the mobile phase can affect the amount of light reaching the detector.

The above explanation concerning the source of the baseline periodic noise in the case of the water–THF and water–1,3-dioxolane mobile phases indicates that (a) these two mobile phases must have substantially higher RIs than water–methanol and water–acetonitrile and (b) that the temperature dependence of the water–THF and water–1,3-dioxolane RIs is more pronounced than with the other two phases. Since a complete set of the RIs of these

Table 2
The % volume ratio equivalent of the 1:5.25 mole ratio modifier–water used in this study

Mobile phase	% Modifier volume/ % water volume
Methanol–water	30/70
Acetonitrile–water	35.7/64.3
THF–water	46.1/53.9
1,3-Dioxolane–water	42.4/57.6

mobile phases, covering the full range of composition (0 to 100% modifier) and the temperature range typically used in HPLC (25–50°C), is not available, we decided to measure these RIs.

3.2. Refractive index measurements

We have measured the RIs of the four mobile phases over the concentration range of 0–100% V/V modifier in increments of 10%. The measurements covered the temperature range of 25–50°C in increments of 5°C. Table 3 summarizes the RIs of the four binary liquid systems. Figs. 2–5 plot the RIs of each modifier–water mobile phase as a function of the modifier concentration at each of the temperatures studied. Our results agree well with the results of Chen et al. [21] who measured the RIs of water–methanol mixtures at 40°C, with the results of Aminabhavi et al. [18] who measured the RIs of water–acetonitrile at 25°C and the results of Signer et al. [19] and Hayduk et al. [20] who measured the RIs of water–tetrahydrofuran at 25°C.

From the figures we observe that the RIs of water–methanol and of water–acetonitrile mobile phases behave in a similar fashion as a function of the percent modifier and of the temperature. The RIs of water–THF and water–1,3-dioxolane are also similar, but substantially higher than the previous two phases. It is to be noted that the scale in Figs. 2 and 3 is more expanded than in Figs. 4 and 5. The RI values for mobile phases containing methanol and acetonitrile have a maximum value at about 50–60% modifier, while with THF and 1,3-dioxolane-containing mobile phases the RIs increase monotonically up to 100% modifier.

As expected, the RIs of all mixtures decrease as the temperature is increased; see Figs. 2–5. This is due to the decrease in the density of the solvent as the temperature goes up.

Fig. 6 combines the RIs of all four mobile phases at 30°C. The figure allows the comparison in the behavior of water–methanol and water–acetonitrile versus the behavior of water–THF and water–1,3-dioxolane. The higher RIs of the water–THF and water–1,3-dioxolane mobile phases confirm our contention that the periodic noise in the UV-detector baseline is due to temperature-induced RI fluctuations. The temperature fluctuations are related direct-

ly to the operation of the thermostat. The frequency of the fluctuation depends on the on/off cycling of the thermostat. At higher temperatures the frequency of the on/off cycle of the temperature controller is higher resulting in a higher frequency of the periodic saw-tooth noise.

3.3. Excess molar volume calculations

The availability of RIs of the four binary mobile phases enables us to calculate the excess molar volumes for these phases. While excess molar volumes do not enter directly into chromatographic calculations, these values are important in the characterization of solutions. Excess molar volumes are indicative of the interactions between the aqueous part of the mobile phase and the organic modifier. Therefore, we decided to report here the excess molar volume values for the four mobile phases. We carried out the calculations for data collected at 25°C. A similar procedure can be used to calculate the excess molar volume values at all the temperatures used in this study.

The excess molar volume, V_E , of a binary solution is related to the molar volume, V , of that solution by the equation:

$$V = X_1V_1 + X_2V_2 + V_E \quad (1)$$

where X_1 and X_2 are the mole fractions of the two components in the binary mixture and V_1 and V_2 are the molar volumes of these components.

Using the Lorenz–Lorentz equation [22] we can relate the molar volume of the mixture to the molar refractive index, R , as well as to the actual refractive index, RI , of the mixture:

$$V = R \frac{RI^2 + 2}{RI^2 - 1} \quad (2)$$

The molar refractive index, R , can be obtained from the molar refractive indices of the two components, R_1 , R_2 , and their respective mole fraction:

$$R = X_1R_1 + X_2R_2 \quad (3)$$

Finally, the molar RI of each of the components can be obtained from the Lorenz–Lorentz equation [22]:

Table 3
Refractive indices of modifier–water as a function of modifier percentage in the temperature range of 25–50°C

	% Modifier	Temperature (°C)					
		25	30	35	40	45	50
Water–MeOH	0	1.3325	1.3321	1.3316	1.3309	1.3300	1.3292
	10	1.3341	1.3336	1.3330	1.3321	1.3310	1.3303
	20	1.3360	1.3357	1.3352	1.3339	1.3330	1.3318
	30	1.3382	1.3378	1.3366	1.3352	1.3343	1.3337
	40	1.3402	1.3393	1.3376	1.3369	1.3358	1.3342
	50	1.3412	1.3400	1.3386	1.3374	1.3360	1.3348
	60	1.3412	1.3400	1.3388	1.3372	1.3355	1.3342
	70	1.3401	1.3389	1.3371	1.3359	1.3340	1.3318
	80	1.3375	1.3356	1.3344	1.3330	1.3313	1.3298
	90	1.3333	1.3316	1.3305	1.3290	1.3259	1.3244
100	1.3269	1.3248	1.3235	1.3212	1.3192	1.3168	
Water–ACN	0	1.3329	1.3322	1.3318	1.3310	1.3300	1.3292
	10	1.3372	1.3362	1.3352	1.3341	1.3332	1.3312
	20	1.3408	1.3397	1.3385	1.3371	1.3360	1.3335
	30	1.3432	1.3416	1.3403	1.3391	1.3375	1.3356
	40	1.3448	1.3443	1.3418	1.3402	1.3382	1.3365
	50	1.3454	1.3440	1.3422	1.3409	1.3388	1.3372
	60	1.3460	1.3441	1.3425	1.3411	1.3391	1.3364
	70	1.3458	1.3438	1.3420	1.3404	1.3382	1.3358
	80	1.3452	1.3430	1.3412	1.3390	1.3373	1.3342
	90	1.3433	1.3413	1.3393	1.3373	1.3346	1.3315
100	1.3422	1.3396	1.3375	1.3350	1.3320	1.3298	
Water–THF	0	1.3326	1.3322	1.3318	1.3310	1.3302	1.3292
	10	1.3398	1.3395	1.3382	1.3379	1.3362	1.3347
	20	1.3487	1.3472	1.3468	1.3452	1.3445	1.3424
	30	1.3583	1.3538	1.3528	1.3512	1.3509	1.3510
	40	1.3650	1.3647	1.3613	1.3593	1.3580	1.3543
	50	1.3741	1.3727	1.3712	1.3672	1.3670	1.3649
	60	1.3818	1.3805	1.3782	1.3745	1.3730	1.3715
	70	1.3889	1.3864	1.3854	1.3820	1.3795	1.3778
	80	1.3943	1.3922	1.3910	1.3882	1.3863	1.3842
	90	1.4010	1.3988	1.3962	1.3938	1.3915	1.3891
100	1.4051	1.4030	1.4000	1.3975	1.3948	1.3925	
Water–1,3-dioxolane	0	1.3326	1.3325	1.3318	1.3310	1.3302	1.3292
	10	1.3393	1.3392	1.3383	1.3375	1.3363	1.3353
	20	1.3478	1.3468	1.3458	1.3434	1.3432	1.3415
	30	1.3550	1.3532	1.3522	1.3495	1.3493	1.3478
	40	1.3610	1.3608	1.3590	1.3568	1.3549	1.3538
	50	1.3697	1.3672	1.3667	1.3653	1.3628	1.3602
	60	1.3768	1.3740	1.3728	1.3702	1.3694	1.3670
	70	1.3830	1.3801	1.3790	1.3763	1.3750	1.3725
	80	1.3890	1.3862	1.3838	1.3820	1.3796	1.3780
	90	1.3940	1.3917	1.3892	1.3872	1.3849	1.3825
100	1.3982	1.3957	1.3938	1.3911	1.3889	1.3862	

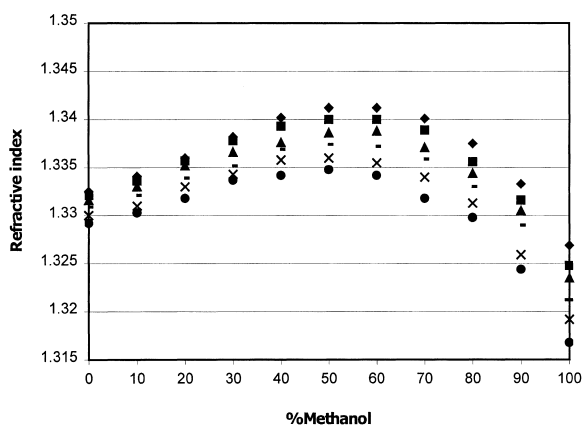


Fig. 2. Refractive index of methanol–water vs. % methanol. ♦, 25°C; ■, 30°C; ▲, 35°C; —, 40°C; ×, 45°C; ●, 50°C.

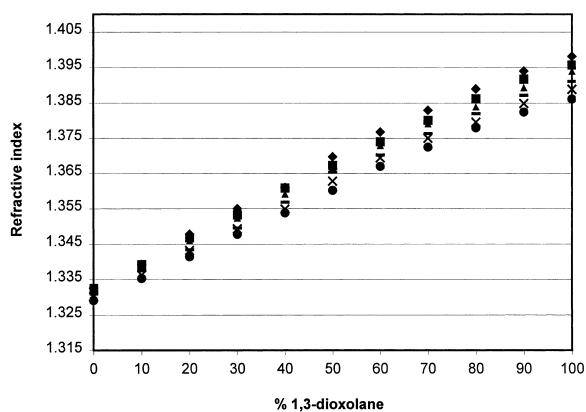


Fig. 5. Refractive index of 1,3-dioxolane–water vs. % 1,3-dioxolane. ♦, 25°C; ■, 30°C; ▲, 35°C; —, 40°C; ×, 45°C; ●, 50°C.

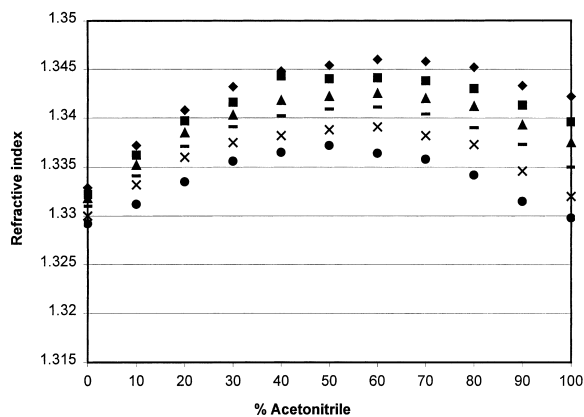


Fig. 3. Refractive index of acetonitrile–water vs. % acetonitrile. ♦, 25°C; ■, 30°C; ▲, 35°C; —, 40°C; ×, 45°C; ●, 50°C.

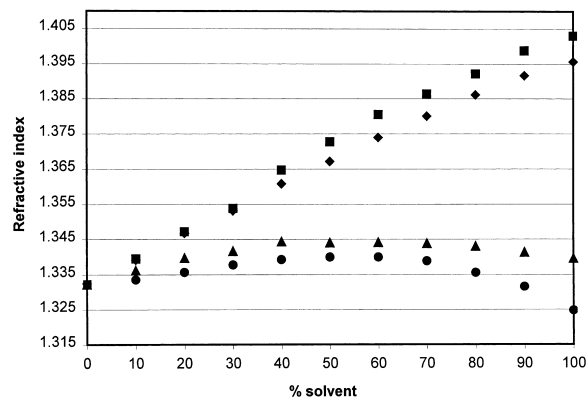


Fig. 6. Refractive indices of the four mobile phases as a function of modifier percentage at 30°C. ♦, 1,3-dioxolane; ■, THF; ▲, ACN; ●, MeOH.

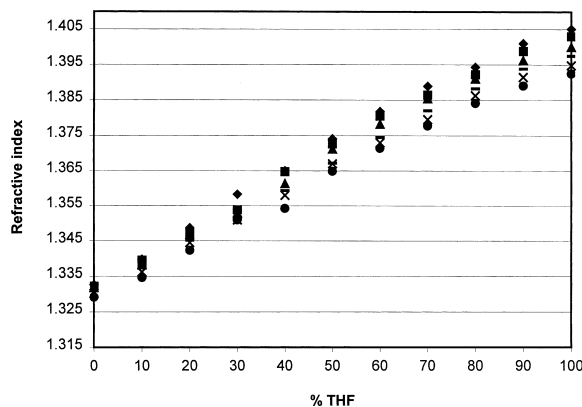


Fig. 4. Refractive index of THF–water vs. % THF. ♦, 25°C; ■, 30°C; ▲, 35°C; —, 40°C; ×, 45°C; ●, 50°C.

$$R_i = V_i \frac{RI_i^2 - 1}{RI_i^2 + 2} \quad (4)$$

where V_i is the molar volume of the component and RI_i is its RI.

The molar volume of each pure liquid was calculated by dividing the molecular mass by the density at 298.15°K. The values obtained, in ml/mol, are 40.7012 for MeOH, 52.8995 for ACN, 81.5699 for THF, 69.5979 for 1,3-dioxolane and 18.0630 for water. With the aid of Eq. (4), and the molar volume values mentioned here, the molar refractive indices of the components were calculated. The values are 8.2330 for MeOH, 11.1540 for ACN, 20.0033 for

Table 4
Molar refractive indices of the binary liquids at 298.15°K

Fraction of modifier	Methanol	ACN	THF	1,3-Dioxolane
0.0	3.7112	3.7112	3.7112	3.7112
0.1	3.9241	3.9876	4.1044	4.0762
0.2	4.1632	4.3049	4.5692	4.5040
0.3	4.4352	4.6752	5.1303	5.0155
0.4	4.7444	5.1087	5.8145	5.6316
0.5	5.1039	5.6301	6.6792	6.3988
0.6	5.5195	6.2578	7.7860	7.3627
0.7	6.0161	7.0454	9.2875	8.6385
0.8	6.6047	8.0360	11.3804	10.3587
0.9	7.3349	9.3602	14.6074	12.8862
1.0	8.2330	11.1540	20.0033	16.8047

THF, 16.8047 for 1,3-dioxolane and 3.7112 for water. Next, the molar refractive indices of the binary mixtures were calculated using Eq. (3) and the molar refractive indices data. The calculated values are given in Table 4. Table 5 gives the molar

Table 5
Molar volumes of the binary mixtures at 298.15°K

Modifier fraction	Methanol	ACN	THF	1,3-Dioxolane
0.0	18.0668	18.0471	18.0619	18.0619
0.1	19.0203	19.1667	19.5914	19.4828
0.2	20.0757	20.4941	21.3062	21.0515
0.3	21.2613	22.1161	23.3450	23.0132
0.4	22.6224	24.0652	26.0218	25.4538
0.5	24.2717	26.4797	29.2402	28.3108
0.6	26.2482	29.3860	33.4719	32.0261
0.7	28.6938	33.1017	39.2781	37.0333
0.8	31.7208	37.8149	47.5443	43.7984
0.9	35.6298	44.2665	60.1228	53.8712
1.0	40.7033	52.9032	81.5958	69.5964

Table 6
Excess molar volumes of the binary mixtures at 298.15°K

Co-solvent fraction	Methanol	ACN	THF	1,3-Dioxolane
0.0	0.0050	-0.0147	0.0001	0.0001
0.1	-0.1076	-0.1889	-0.0037	-0.0156
0.2	-0.2494	-0.3471	-0.1014	-0.1308
0.3	-0.4259	-0.4585	-0.2510	-0.1822
0.4	-0.6130	-0.5386	-0.2422	-0.1666
0.5	-0.7634	-0.5648	-0.3958	-0.3293
0.6	-0.8680	-0.5971	-0.4803	-0.4075
0.7	-0.9093	-0.5682	-0.5296	-0.4219
0.8	-0.8295	-0.4920	-0.4251	-0.4274
0.9	-0.5765	-0.2393	-0.4308	-0.3023
1.0	0.0000	0.0002	0.0000	0.0000

volumes of the binary mixtures as calculated using the molar refractive indices of the binary mixtures (Table 4), the RIs in Table 3 and Eq. (2). Finally, Eq. (1) is used to calculate the excess volume for each of the four binary mixtures studied here; see Table 6.

In the literature we found excess molar volume values for THF [19,23,24], ACN [25] and MeOH [26] at 298.15°K. Our results compare favorably to those in the literature for MeOH and ACN. For THF, in the composition range of about 20 to 80% THF our results are larger (less negative) than the literature values although the trend in the data is the same as observed in the literature.

4. Conclusions

The present work shows a direct correlation between the RI of the mobile phase and the quality

of the signal of a UV–Vis detector. Fluctuation in the temperature of the mobile phase results in RI variation that is manifested by noisy baseline. To ensure a good quality detector signal, a good column thermostat is required to prevent large temperature variations during the analysis. This requirement is especially important in the case of mobile phases having a high RI, e.g., water–THF or water–1,3-dioxolane.

The RIs and excess molar volumes given in this work should aid chromatographers in designing liquid chromatographic systems with minimal baseline disturbances caused by RI variations.

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