Analytica Chimica Acta, 284 (1993) 291–299
Elsevier Science Publishers B.V., Amsterdam

Application of ion chromatography with indirect spectrophotometric detection to the sensitive determination of alkylphosphonic acids and fosfomycin

G.A. Pianetti and L.M. Moreira de Campos

Laboratoires de Chimie Analytique, Centre d'Études Pharmaceutiques, Université Paris-Sud, 1 Avenue J.B. Clément, F-92290 Châtenay-Malabry (France) and Laboratório de Controle de Qualidade de Produtos Farmacêuticos e Cosméticos, Faculdade de Farmácia da Universidade Federal de Minas Gerais, Belo Horizonte, MG (Brazil)

P. Chaminade, A. Baillet, D. Baylocq-Ferrier and G. Mahuzier

Laboratoires de Chimie Analytique, Centre d'Études Pharmaceutiques, Université Paris-Sud, 1 Avenue J.B. Clément, F-92290 Châtenay-Malabry (France)

(Received 27th April 1993; revised manuscript received 11th August 1993)

Abstract

Indirect spectrophotometric detection was investigated for application in the anion-exchange chromatography of alkylphosphonic acids and fosfomycin. Four chromophoric eluent anions were studied: phthalate, benzoate, phenylphosphonate and *p*-toluenesulphonate. Phthalate showed the best displacing power and the larger dynamic reserve whereas benzoate exhibited the highest absorptivity and transfer ratio. The optimal experimental conditions were found to be 0.4 mM phthalate (pH 8.5) as mobile phase, a flow-rate of 1 ml min^{-1} and a detection wavelength of 272 nm. Selectivity (between 1.02 and 1.85, depending on the pair of vicinal compounds), linearity over a 5–100 μg ml^{-1} concentration range ($r = 0.999$), precision (R.S.D. 0.6–5.3%) and sensitivity (0.4–1.0 μg ml^{-1} according to the phosphonic acid) were determined. The minimum detectable concentrations were improved by an average factor of 50 in comparison with direct non-suppressed conductimetric detection. The application of the technique to the determination of fosfomycin in plasma samples is described.

Keywords: Ion chromatography; Liquid chromatography; Fosfomycin; Phosphonic acids; Plasma

Phosphonic acids are mainly determined by gas or liquid chromatography after derivatization or by ion chromatography. Because these compounds do not absorb or fluoresce, some workers converted them into ester derivatives to allow their separation by gas chromatography [1–4] or to produce fluorescing [5] or UV-absorbing species [6] able to be separated by liquid chromatography. All these methods are time consuming. On the other hand, ion-exchange chromatography is known to provide excellent ion separations, with suppressed conductimetric detection [7] and with non-suppressed conductimetric or spectrophotometric detection [8,9]. Schiff et al. [10] described the determination of phosphonic acids in aqueous and biological samples using direct conductimetric detection with a suppression column.

Recently a rapid method involving anion-ex-

Correspondence to: G.A. Pianetti, Laboratoires de Chimie Analytique, Centre d'Études Pharmaceutiques, Université Paris-Sud, 1 Avenue J.B. Clément, F-92290 Châtenay-Malabry (France)

SSDI 0003-2670(93)E0476-N

change chromatography with direct conductimetric detection [11] was proposed. The separation of a series of homologous alkylphosphonic acids using capillary zone electrophoresis with indirect UV detection has also been reported [12]. Likewise, indirect spectrophotometric detection could be an alternative mode in non-suppressed ion chromatography [13,14]. It provides universal detection of inorganic anions and with the use of low eluent concentrations low limits of detection can be attained. However, the concentration of the mobile phase components cannot be lowered too much without degrading the separation.

The aim of this study was to investigate the applicability of this alternative method of detection coupled with anion-exchange separation. A simple technique was developed for the determination of monophosphonic acids using 0.4 mM potassium phthalate at pH 8.5 as the mobile phase with indirect spectrophotometric detection at 272 nm. The determination of methyl-, ethyl-, propyl- and butylphosphonic acids and fosfomycin in aqueous samples was carried out using phenylphosphonic acid as an internal standard. Detection limits were determined. The technique was then adapted to the determination of fosfomycin in plasma samples. The difficulties encountered and the analytical validation are detailed.

EXPERIMENTAL

Chemicals

Distilled, deionized water was used for liquid chromatography (LC). Methylphosphonic acid (MPA), ethylphosphonic acid (EPA), propylphosphonic acid (PPA), butylphosphonic acid (BPA) and phenylphosphonic acid (FPA) were obtained from Aldrich-Chimie (Strasbourg). The sodium salt of fosfomycin (FOF) was obtained from Clin-Midy (Paris) and sodium phenylphosphonate (SPHP) and potassium benzoate (KBEZ) from Fluka (Mulhouse). Dipotassium phthalate (KPHT) and sodium *p*-toluenesulphonate monohydrate (SPTS) were obtained from Merck (Nogent-sur-Marne). All other chemicals were of analytical-reagent grade.

Instrumentation

LC analyses were carried out using a liquid chromatograph from Touzart et Matignon (Vitry-sur-Seine) consisting of a Shimadzu LC-6A pump, a Shimadzu SPD-6A UV spectrophotometric detector and a Shimadzu CTO-6A oven. This system was connected to a Shimadzu Chromatopac CR-5A integrator which measured the detector response as peak areas. Separations were carried out using a Waters-Millipore (St. Quentin-en-Yvelines) IC-Pak Anion LC column (50 × 4.6 mm i.d.) with a low anion-exchange capacity (30 ± 3 μeq ml^{-1}).

Chromatographic procedure

For aqueous samples, the optimum conditions were determined for the analytical separation as 0.4 mM potassium phthalate (pH 8.5) as mobile phase, a flow-rate of 1.0 ml min^{-1}, a column temperature of 20°C, indirect UV detection at 272 nm (0.08 absorbance full-scale) and a sample size of 20 μl.

For spiked plasma samples, the mobile phase was 0.4 mM potassium phthalate (pH 9.0), the column temperature and indirect UV detection were as above with 0.04 absorbance full-scale and the sample size was 100 μl.

Optimization step

Standard aqueous solutions (100 μg ml^{-1}) of each alkylphosphonic acid and fosfomycin were injected on to the chromatographic column and their retention times were determined.

Calibration

Calibration graphs were obtained from triplicate injections of known concentrations of the phosphonic acids (5–100 μg ml^{-1}) in aqueous solutions, using the optimum mobile phase.

Spiked plasma sample treatment

To 200-μl plasma samples from healthy volunteers, 200, 100 and 40 μl of a 100 μg ml^{-1} solution of FOF dissolved in methanol were added to obtain the required plasma concentrations of 100, 50 and 20 μg ml^{-1}. The volume was completed to 1.0 ml with methanol to precipitate proteins and the solution was vortex mixed for 2

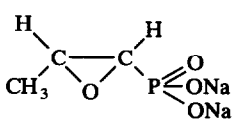
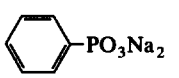
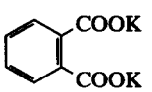
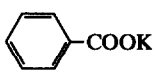
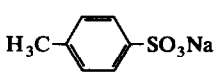
min. The sample was then centrifuged for 20 min at 6000 *g* at 0°C. The supernatant was collected and dried under nitrogen. The residue was dissolved in 1000 μl of the mobile phase, filtered through a 0.11-μm filter (Millipore) and then injected into the LC system.

RESULTS AND DISCUSSION

Several chromophoric eluent anions for the non-suppressed anion-exchange separation of phosphonates using indirect UV detection were studied: potassium phthalate, potassium benzoate, sodium phenylphosphonate and sodium *p*-toluenesulphonate (Table 1). These four UV-absorbing species were selected taking into account the type and number of acidic functional groups in an attempt to compare the displacing powers of the eluent ions. The structure of these acidic groups usually modifies the sample–eluent ion-exchange constants and gives rise to different selectivities. The charge on the eluent molecules plays an important role in the detection performance when modifying the ion-exchange stoichiometry and analysis time. Only singly and doubly charged eluents were investigated. Multiply charged eluents such as trimesate or pyromellilate were eliminated because of the poor retention obtained. All these eluents possess relatively high molar adsorptivities in the UV region. The effects of the type and concentration of

TABLE 1

Formulae of the alkylphosphonic acids and chromophoric eluent anions tested

Compound	Formula	Mol. wt.	p*K* values	λ_{max} (nm)	ϵ_{max} (l mol^{-1} cm^{-1})	Ref.
Methylphosphonic acid (MPA)	$CH_3-P(=O)(OH)OH$	96.02	2.35 7.10	–	–	15
Ethylphosphonic acid (EPA)	$CH_3CH_2-P(=O)(OH)OH$	110.05	2.43 8.05	–	–	16
Propylphosphonic acid (PPA)	$CH_3CH_2CH_2-P(=O)(OH)OH$	124.08	2.49 8.18	–	–	16
Butylphosphonic acid (BPA)	$CH_3CH_2CH_2CH_2-P(=O)(OH)OH$	138.10	2.59 8.19	–	–	16
Sodium fosfomycin (FOF)		182.02	1.50 6.40	–	–	17
Sodium phenylphosphonate (SPHP)		202.06	1.29 6.74	258	231	18
Potassium phthalate (KPHT)		242.32	2.98 5.28	272	821	19
Potassium benzoate (KBEZ)		160.22	4.01	268	1634	20
Sodium *p*-toluene sulphonate (SPTS)		212.20	6.62	261	360	21

chromophoric anion and the wavelength of detection were studied, i.e., the parameters that can affect both the selectivity of separation and the sensitivity of detection. The chromatographic performance of the eluent also requires adjustement of the pH of the mobile phase. As phosphonates are poorly retained in their singly ionized form, the pH was set at 8.5 because at that pH the samples became 74% to 100% doubly ionized.

Anion separation

In a previous study [11], it was observed that the retention of organic anions on low-capacity anion-exchange columns can be attributed to a combination of several phenomena, as the order of elution cannot be explained only as a function of pK_a values or hydrophobicity. The retention times of analyte anions depend on both the type and concentration of the eluent. For a 1 mM concentration of the eluent, the eluting power of phthalate is higher than that of benzoate. This result follows the expected trend of polyvalent ions being stronger displacing species than monovalent ions. Moreover, the displacing power of acidic groups decreased in the order carboxylate > phosphonate > sulphonate (Fig. 1).

The retention times were also measured as a function of the chromophoric eluent anion concentration. When the concentration of the eluent anion decreased, the retention of the analytes increased as expected. Small et al. [7], demonstrated previously that the elution can be described by the expression

$$\log[V_R - V_M] = -(y/x)\log[E^{x-}] + B \qquad (1)$$

where x and y are the charges of the eluent (E) and sample ions, respectively, and B is a constant dependent on the ion-exchange equilibrium constant and the ion-exchange capacity of the stationary phase.

In Table 2, the experimentally observed slopes are compared with the theoretically calculated y/x values. Concurring values were obtained for MPA, FOF and FPA, i.e., the species which have a pK_a far from the pH of the mobile phase. In contrast, larger differences between the experimental and predicted slope values were observed for EPA, PPA and BPA ($pK_a \approx 8$) because of the large variations in the effective y/x values induced by small local pH variations in the mobile phase. pH was then a critical parameter to provide a good prediction of elution. Because of the similar y/x values obtained whatever the alkylphosphonic acid, no changes in elution order could be observed with decreasing phthalate concentration in the mobile phase. The slope values suggest that the optimum selectivity can be ob-

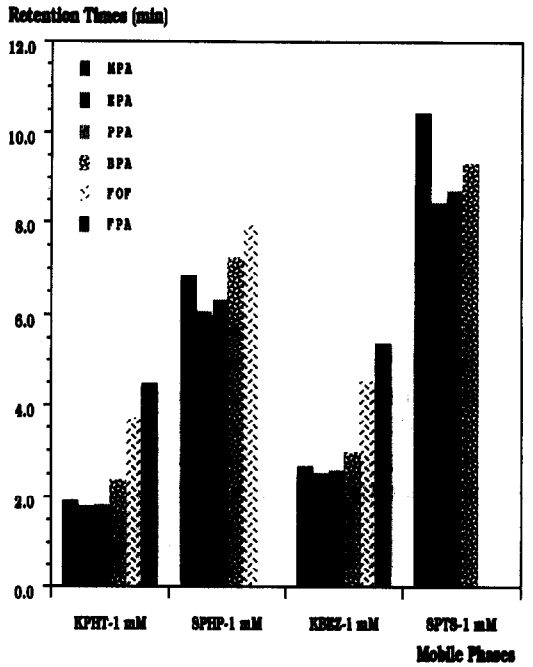


Fig. 1. Retention times of phosphonic acids in various mobile phases at pH 8.5. Elution times longer than 30 min were not determined.

TABLE 2

Comparison between theorical and observed y/x values [a]

Analyte	y/x		Difference (%)
	Calculated	Observed	
MPA	0.98	1.04 ($n = 5$)	5.8
EPA	0.87	1.03 ($n = 5$)	18.4
PPA	0.84	1.04 ($n = 5$)	23.8
BPA	0.83	1.00 ($n = 5$)	20.5
FOF	1.00	0.93 ($n = 9$)	7.0
FPA	1.00	0.94 ($n = 9$)	6.0

[a] Eluent, potassium phthalate (pH 8.5); concentration range, 0.2–2.5 mM; detection wavelength, 272 nm.

TABLE 3

Selectivities per unit time (α/Tr_2) obtained with different concentrations of various chromophoric eluent ions

α/Tr_2 [a]							
KPHT		SPHP		KBEZ		SPTS	
1.0 mM	0.7 mM	2.0 mM	1.0 mM	1.0 mM	0.4 mM	1.4 mM	1.0 mM
PPA/EPA 0.559	PPA/EPA 0.284	PPA/EPA 0.408	PPA/EPA 0.165	PPA/EPA 0.397	PPA/EPA 0.184	PPA/EPA 0.184	PPA/EPA 0.119
MPA/PPA 0.549	MPA/PPA 0.269	MPA/PPA 0.385	MPA/PPA 0.158	MPA/PPA 0.386	MPA/PPA 0.175	MPA/PPA 0.183	BPA/PPA 0.115
BPA/MPA 0.521	BPA/MPA 0.256	BPA/MPA 0.347	BPA/MPA 0.146	BPA/MPA 0.374	BPA/MPA 0.160	BPA/MPA 0.183	MPA/BPA 0.107
FOF/BPA 0.422	FOF/BPA 0.234	FOF/BPA 0.339	FOF/BPA 0.138	FOF/BPA 0.337	– [b]	FOF/MPA 0.182	– [b]
FPA/FOF 0.269	FPA/FOF 0.162	– [b]	– [b]	FPA/FOF 0.221	– [b]	–	– [b]

$\alpha = k'_2/k'_1$, selectivity between two vicinal peaks; Tr_2 = retention time of the more retained compound of the considered pair of eluents. [b] No determined retention times.

tained by increasing the eluent concentration. Indeed, the selectivity or, more accurately, the selectivity per unit time, was improved for all the pairs of compounds with increasing eluent concentration whatever the eluent used (Table 3).

Potassium phthalate was found to offer the best chromatographic performance, i.e., a good separation in a reasonable time. Fig. 2 illustrates the separation of alkylphosphonic acids and fosfomycin with phenylphosphonic acid used as the internal standard.

Sensitivity

In an attempt to study the performance of indirect UV spectrophotometric detection, the

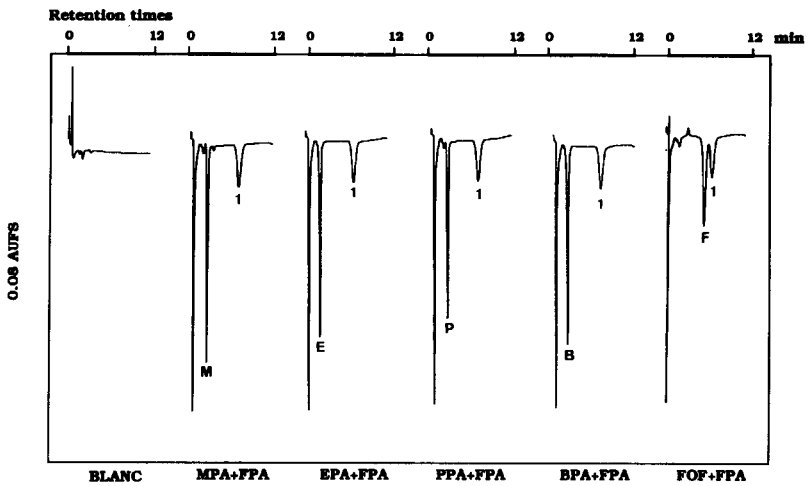


Fig. 2. Typical chromatograms of mixtures of the each alkylphosphonic acid and fosfomycin with phenylphosphonic acid as internal standard with detection at 272 nm (0.08 absorbance full-scale; attenuation = 3). Mobile phase, 0.7 mM potassium phthalate (pH 8.5); solute concentration, 50 μg ml^{-1}; injection volume, 20 μl. Peak identification: $MPA_{(M)} + FPA_{(1)}$, $\alpha = 2.83$; $EPA_{(E)} + FPA_{(1)}$, $\alpha = 3.28$; $PPA_{(P)} + FPA_{(1)}$, $\alpha = 3.21$; $BPA_{(B)} + FPA_{(1)}$, $\alpha = 2.88$; and $FOF_{(F)} + FPA_{(1)}$, $\alpha = 1.19$.

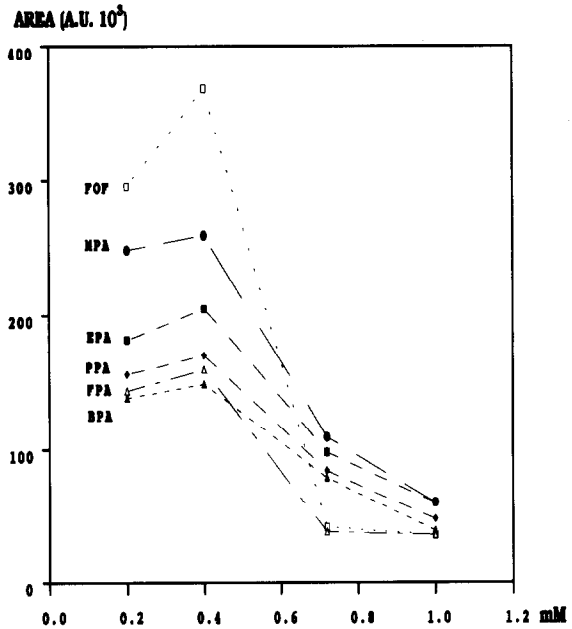


Fig. 3. Plots of peak area as a function of potassium phthalate concentration ($\lambda = 272$ nm). Solute concentration, 100 μg ml^{-1}.

sensitivities obtained when using various chromophoric eluent ions were examined.

Yeung [22] defined the concentration limit of detection, C_{lim}, as a function of the eluent ion concentration, C_m, the dynamic reserve, *DR*, viz., the ability to measure a small change on top of a large background signal, and the transfer ratio, *TR*, i.e., the number of mobile phase molecules exchanged by one analyte molecule:

$$C_{lim} = C_m/(DR \times TR) \quad (2)$$

Accordingly, higher signals were achieved using relatively dilute mobile phase concentrations, C_m. Moreover, the lower is C_m, the larger is the fractional change (larger *DR*) that results for a given analyte concentration. As an example, Fig. 3 shows the increase in area when the potassium phthalate concentration was decreased from 1 to 0.4 mM. However, concentrations less than 0.2 mM were too low to displace sample ions accurately.

The molar absorptivity, ϵ_M, of each chromophoric eluent also plays an important role in determining sensitivities. As expected, a higher *DR* was obtained when the detection wavelength was set at the maximum absorption of the chromophoric eluent.

Finally, the higher was *TR*, the larger became the change per analyte concentration.

Compared with phenylphosphonate [$\epsilon_{M\,(258\text{ nm})} = 231$; $0.84 < TR < 1$, depending on the analyte], *p*-toluene sulphonate [$\epsilon_{M\,(261\text{ nm})} = 360$; $1.67 < TR < 2$] and phthalate [$\epsilon_{M\,(272\text{ nm})} = 821$; $0.83 < TR < 1$], benzoate presented both the highest ϵ_M and *TR* [$\epsilon_{M\,(268\text{ nm})} = 1634$; $1.67 < TR < 2$] and therefore should have led to a better sensitivity. However, its use is limited by its displacing power, which is less favourable than that of phthalate. In fact, the elution of fosfomycin required a higher concentration when benzoate was used as the eluent anion compared with phthalate. This actually compromised the previous advantage.

Validation of the technique

Taking all these criteria into account, we found potassium phthalate to be the most suitable eluent. Thus, the experimental conditions were set as follows: 0.4 mM potassium phthalate as mobile phase, flow-rate 1 ml min^{-1} and detection wavelength 272 nm. The corresponding retention times were 8.6, 8.0, 8.4, 9.1, 9.6 and 13.2 min for MPA, EPA, PPA, BPA, FOF and FPA, respectively. The technique was validated for the determination of phosphonic compounds in aqueous solutions. The specificity was verified with respect to common anions, particularly chloride ($t_R = 3.4$ min), phosphate ($t_R = 15.5$ min) and sulphate ($t_R = 21.8$ min). The calibration graphs (Fig. 4) indicate a satisfactory linear dependence of area (or height) on the amount of each analyte in the range 5–100 μg ml^{-1}. The correlation coefficients were 0.999 for all the phosphonates injected. The relative standard deviation (R.S.D.) of the response factor range from 0.58 to 5.29% for six runs carried out over three days. Detection limits were calculated for a signal-to-noise ratio of 3 (attenuation = 1), corresponding to concentrations of 0.40, 0.20, 0.45, 1.0 and 0.5 μg ml^{-1} for MPA, EPA, PPA, BPA and FOF, respectively. These limits are improved by an average factor of 50 when compared with conductimetric detection [11].

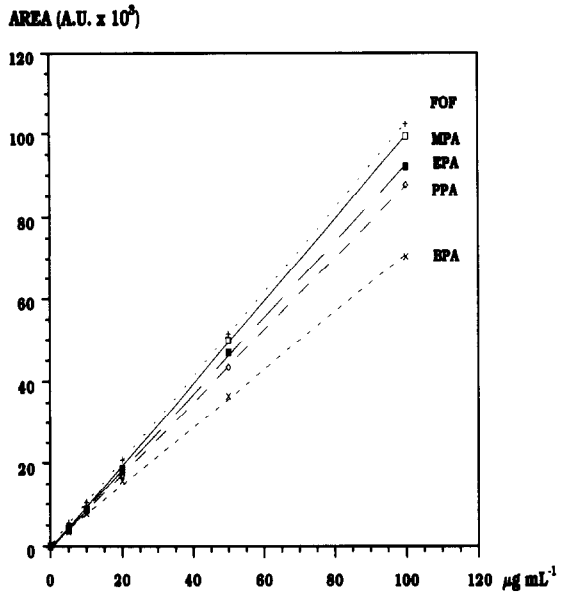


Fig. 4. Calibration graphs obtained for the different phosphonic acids. Mobile phase, 0.4 mM potassium phthalate (pH 8.5); flow-rate, 1 ml min^{-1}; detection wavelength 272 nm.

Application to plasma fosfomycin determination

The method was then applied to a plasma sample spiked at the level of 100 μg ml^{-1}. As explained in a previous paper [11], the yield of fosfomycin extraction with an organic solvent was limited by the high solubility of phosphonates in water. Consequently, the injected samples still contained anionic endogenous compounds, thus setting two problems: first, a large injection peak appeared which prevented the use of the alkylphosphonates as internal standards for the fosfomycin determination; and second, the separation of fosfomycin and phenylphosphonic acid was disturbed by the presence of two unidentified endogenous compounds.

A fivefold dilution of the sample with the mobile phase and the use of an eluent pH of 9.0 (instead of 8.5) solved the first problem, but an overlap was still observed between phenylphosphonic acid and one endogenous compound, thus preventing the use of this analyte as an internal standard.

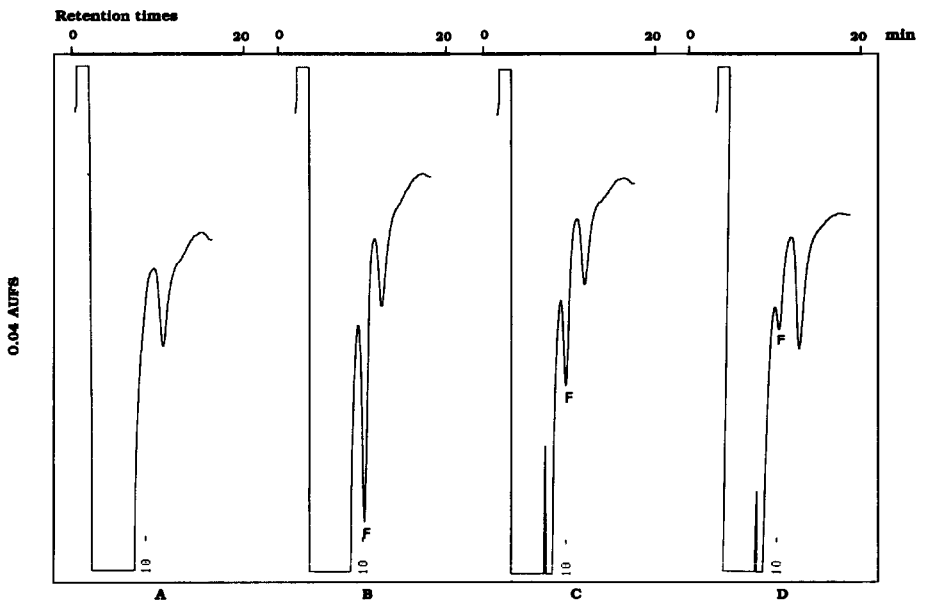


Fig. 5. Typical chromatograms for plasma samples: (A) control human plasma and human plasma spiked with (B) 100, (C) 50 and (D) 20 μg ml^{-1} of a methanolic solution of fosfomycin, both diluted fivefold in the 0.4 mM potassium phthalate mobile phase (pH 9.0). Injection volume, 100 μl; detection wavelength, 272 nm (attenuation = 2).

TABLE 4

Inter- and intra-assay precision for fosfomycin determination in human plasma

Concentration range	Concentration in spiked plasma ($\mu g\ ml^{-1}$)	Concentration found inter-assay ($n = 3$)		Concentration found intra-assay ($n = 4$)	
		Mean ± S.D. ($\mu g\ ml^{-1}$)	R.S.D.	Mean ± S.D. ($\mu g\ ml^{-1}$)	R.S.D.
Low	20	19.13 ± 0.51	2.68	18.60 ± 0.26	1.39
Medium	50	49.27 ± 0.44	0.89	49.57 ± 0.35	0.71
High	100	97.99 ± 0.99	1.01	97.85 ± 0.55	0.56

TABLE 5

Recovery of fosfomycin from human plasma

Concentration range	Concentration in spiked plasma ($\mu g\ ml^{-1}$)	Fosfomycin area		Recovery (%)
		Calibration graph	Sample determination	
Low	20	19600	18600	94.9
Medium	50	49800	49400	99.2
High	100	104700	103000	98.4

A quantitative study with external calibration was then undertaken. Typical chromatograms for spiked human plasma samples are shown in Fig. 5, for FOF plasma concentrations of 0, 100, 50 and 20 $\mu g\ ml^{-1}$. Fosfomycin eluted at ca. 9.62 ± 0.14 min (R.S.D. = 1.47%, $n = 36$) and the endogeneous peak at 12.68 ± 0.20 min (R.S.D. = 1.56%, $n = 36$). The selectivity (α) between these two peaks was 1.37. A calibration graph was obtained by analysis of plasma spiked with fosfomycin in the range 20–100 $\mu g\ ml^{-1}$. The calibration graph was linear up to a concentration (x) of 20 $\mu g\ ml^{-1}$ ($y = -1.0551 + 1.0489x$; $r = 0.993$).

To evaluate the reproducibility of the system and method, inter- and intra-assay precision studies were performed. To evaluate the intra-assay precision of the method, three different samples with high, medium and low concentrations of fosfomycin were chosen and each was injected on to the column four times. The R.S.D. for the four replicate injections for each sample ranged from 0.56 to 1.39%. For inter-assay precision, on three separate occasions different samples with three concentrations were prepared and injected on to the column. The R.S.D. ranged from 0.89 to 2.68% (Table 4). The recovery of fosfomycin in plasma, obtained from the calibration graph for spiked plasma, is shown in Table 5 and ranged from 94.9 to 99.2%.

This investigation was supported by CAPES (Coordenação de Aperfeiçoamento de Pessoal de Nível Superior do Ministério da Educação do Brasil), Brazil, and COFECUB (Comité Français d'Evaluation de la Coopération Universitaire avec le Brésil), France.

REFERENCES

1 M.L. Rueppel, L.A. Suba and J.T. Marvel, Biomed. Mass Spectrom., 3 (1976) 28.
2 C.G. Daughton, A.M. Cook and M. Alexander, Anal. Chem., 51 (1979) 1949.
3 J.Aa. Tornes and B.A. Johnsen, J. Chromatogr., 467 (1989) 129.
4 M.L. Shih, J.R. Smith, J.D. McMonagle, T.W. Dolzine and C. Greshany, Biol. Mass Spectrom., 20 (1991) 717.
5 M.C. Roach, L.W. Ungar, R.N. Zare, L.M. Reimer, D.L. Pompliano and J.W. Frost, Anal. Chem., 59 (1987) 1056.
6 P.C. Bossle, J.J. Martin, E.W. Sarver and H.Z. Sommer, J. Chromatogr., 267 (1983) 209.

7 H. Small, T.S. Stevens and W.C. Bauman, Anal. Chem., 47 (1975) 1801.
8 H. Sato, Anal. Chim. Acta, 206 (1988) 281.
9 S.A. Maki and N.D. Danielson, Anal. Chem., 63 (1991) 699.
10 L.J. Schiff, S.G. Pleva, E.W. Sarver, in J.D. Mulik and E. Sawicki (Eds.), Ion Chromatographic Analysis of Environmental Polluants, Vol. 2, Ann Arbor Sci. Publ., Ann Arbor, 1979, p. 329.
11 G.A. Pianetti, A. Baillet, F. Traoré and G. Mahuzier, Chromatographia, 36 (1993) 263.
12 G.A. Pianetti, M. Taverna, A. Baillet, G. Mahuzier and D. Baylocq-Ferrier, J. Chromatogr., 630 (1993) 371.
13 H. Small and T.E. Miller, Anal. Chem., 54 (1982) 462.
14 H. Small, Anal. Chem., 55 (1983) 235A.
15 A. Rumpf and C. Chavane, Chem. Res., 224 (1947) 919.
16 B. Crofts and K. Kosolapoff, J. Am. Chem. Soc., 75 (1953) 3382.
17 D. Baron and H. Drugeon, Sem. Hôp. Paris, 61 (1985) 2341.
18 O. Mäkitie and V. Konttinen, Acta Chem. Scand., 23 (1969) 1459.
19 W.R. Maxwell and J.R. Partington, Trans Faraday Soc., 31 (1935) 922.
20 M. Yasuda, K. Yamasaki and H. Ohtaki, Bull. Chem. Soc. Jpn., 33 (1960) 1067.
21 Dictionary of Organic Compounds, Vol. 4, Suppl., Chapman and Hall, New York, 5th edn., 1986, p. 3749.
22 E.S. Yeung, Acc. Chem. Res., 22 (1989) 125.