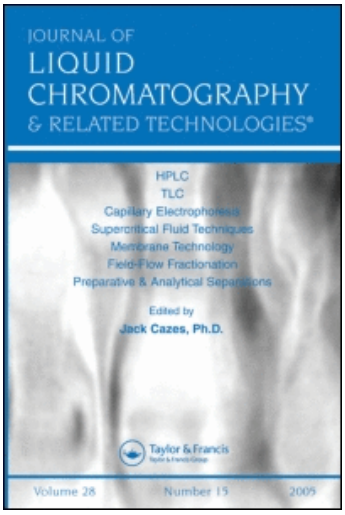

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TLC Screening Method for Identification of Active Components of "Ecstasy" Tablets. Influence of Diluents and Adulterants

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Abstract: The possibility of TLC screening identification of active components of "ecstasy": MDMA, PMA, PMMA, and ephedrine has been studied. For sample dissolution, methanol and phosphate buffer have been used and the results were compared. The usefulness of several multicomponent eluents for TLC has been tested. The simplex method has been employed to find the optimum composition of eluent : chloroform : dioxane : methanol : ammonia : acetonitrile (3.5 : 15 : 2 : 1.5 : 15 v/v/v/v/v).

The influence of adulterants and diluents: magnesium stearate, acetylsalicylic acid (aspirin), p-hydroxyacetanilide (paracetamol), procaine, 1-phenylethylamine, caffeine, glucose, powdered sugar, citric acid, starch, plaster, and chalk on TLC chromatograms of the above drugs was tested.

Keywords: Ecstasy, MDMA, TLC analysis, Simplex optimization, 2^2 Factorial design

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INTRODUCTION

Europe appears to be the area where amphetamines and their analogues are intensively used.[1] Although there has been some fluctuation recently, the number of amphetamine seizures, as well as quantities of drugs produced have grown considerably in the last decade.

"Ecstasy" is a common street name for illicit tablets containing MDMA (3,4-methylenedioxymethamphetamine). The composition of ecstasy tablets exhibits substantial variability. Beside the main active component, MDMA, also amphetamine, MA (methamphetamine), MDA (3,4-methylenedioxyamphetamine), MDEA (3,4-methylenedioxyethamphetamine), and ephedrine were discovered in the seized tablets.[2–7] Recently, tablets sold as ecstasy were found to contain PMA (p-methoxyamphetamine) and/or PMMA (p-methoxymethamphetamine).[8–10] Several cases of fatal overdose outcomes of tablets with PMMA have been reported in Poland.[8] Apart from the psychoactive components, drug tablets contain diluents and adulterants, which are added to the drug before it is brought onto the illegal market. Glucose and other sugars, p-hydroxyacetanilide, acetylsalicylic acid, citric acid, caffeine, magnesium stearate, and starch were reported among others.

Analysis of illicit drug tablets is usually carried out by chromatographic methods: HPLC,[11,12] GC,[13] or capillary electrophoresis.[11,13] Praisler et al. described the application of principal component analysis for the automated identification of amphetamines from vapour-phase FTIR spectra.[14] Spectrophotometric and spectrofluorimetric procedure based on absorption and emission data at three wavelengths has been proposed for identification of amphetamine and/or methamphetamine in street drugs.[15] Reflectance and transmittance near infrared spectroscopy has been employed for analysis of ecstasy tablets.[16] Sägmüller et al. reported the method of identification of illicit drugs by combination of HPLC and surface-enhanced Raman scattering spectroscopy.[17]

In the present paper, a TLC method for screening identification of active components, MDMA, PMA, PMMA, and ephedrine, in 'ecstasy' tablets is described. Before TLC separation is carried out, the solid drug samples are dissolved. In our studies we used methanol, as well as phosphate buffer (pH 7.0) as drug solvents. The influence of the sample solvent used on the quality of TLC separation was examined. The simplex method[18] was used to find the optimum composition of eluent for TLC. The influence of individual additives (adulterants and diluents): magnesium stearate, acetylsalicylic acid (aspirin), p-hydroxyacetanilide (paracetamol), procaine, 1-phenylethylamine, caffeine, glucose, powdered sugar, citric acid (chemical reagent and eatable), starch, plaster, and chalk on TLC chromatograms was tested. Also, the influence of two-component matrices was examined. Their composition was varied in accordance with 2^2 factorial design. Special attention was paid to magnesium stearate, because it is a component of all tablets.

EXPERIMENTAL

Chemicals and Reagents

The following reagents were used: MDMA (3,4-methylenedioxymethamphetamine, synthesized in our laboratory), PMMA (p-methoxymethamphetamine, synthesized in our laboratory), PMA (p-methoxyamphetamine, synthesized in our laboratory), ephedrine (Fluka, Switzerland); methanol, chloroform, dioxane, acetonitrile (all Merck, Germany, HPLC grade), aqueous ammonia (25%, POCh, Poland, analytical grade), xylene (ZK Hajduki, Poland, analytical grade), toluene (Eurochem BGD, Poland, analytical grade), ethyl acetate 99.8% (Aldrich, HPLC grade), phosphate buffer solution pH = 7 (Merck, Germany); glucose (P.P.E."Gemi", Poland), castor sugar (Pfaifer & Langen Polska S.A., Poland), citric acid–chemical reagent (POCh, Poland, analytical grade), citric acid–eatable (Wodzislaw sp. z o.o., Poland), starch (POCh, Poland), plaster (ZPG Dolina Nidy S.A., Poland), chalk (OMYA color, Poland), paracetamol (p-hydroxyacetanilide, synthesised); 1-phenylethylamine hydrochloride, aspirin (acetylsalicylic acid), hydrochloride procaine, caffeine (all Sigma, Aldrich), magnesium stearate (POL-NIL, Poland, analytical grade).

Sample Preparation

The mixtures of tested components (hydrochlorides): 5 mg/mL MDMA, 4 mg/mL PMMA, 4 mg/mL PMA and 10 mg/mL ephedrine in methanol and in phosphate buffer pH 7 were prepared.

To the portions of solutions of drug mixtures the following individual additives were added: glucose, castor sugar, starch; agglutinants: magnesium stearate, chalk, plaster; and adulterants: acetylsalicylic acid, caffeine, citric acid, paracetamol, hydrochloride procaine, 1-phenyloethylamine hydrochloride. It was assumed that MDMA makes 50% of "ecstasy" tablets. The examined samples contained 0, 1, 2, 3, 4, and 5% of magnesium stearate and 0, 5, 10, 20, 30, 40, and 50% of other additives. In further studies, appropriate amounts of magnesium stearate (an agglutinant) and the above mentioned additives were added to drug solutions to make several compositions of two-component drug matrices, according to a 2^2 factorial. The higher and lower concentration levels were, respectively, 0% and 5% for magnesium stearate (present in each matrix tested), and 0% and 50% for other additives.

All prepared sample solutions were shaken for 20 min (Vortex) and, if necessary (precipitate present), centrifugation was performed for 5 min at 13000 rpm.

The simplex optimization of composition of the mobile phase for TLC was carried out on methanol drug solutions (without matrix).

TLC Separation Procedure

Silica gel (0.2 mm) plates with a fluorescent indicator $60F_{254}$ (Merck, Germany) and horizontal developing chamber (Camag, Switzerland) were used. The sample solutions were applied on TLC plates by 4 μL microcapillary (Sigma-Aldrich). In the case of buffer solutions the plates were dried for 5 min after applying the sample. The following mixtures of solvents, the TLC mobile phases, were examined:[19,20] chloroform : acetone : ammonia 25% : methanol (10 : 8 : 1 : 1 v/v/v/v), acetone : xylene : methanol : ammonia 25% (8 : 6 : 1 : 1 v/v/v/v), toluene : acetone : ethanol : ammonia 25% (45 : 45 : 7 : 3 v/v/v/v), chloroform : ethyl acetate : methanol : ammonia 25% (4 : 10 : 1 : 1 v/v/v/v), acetone : xylene : methanol (8 : 6 : 1 v/v/v), chloroform : acetone : methanol (4 : 10 : 1 v/v/v), chloroform : dioxane : methanol : ammonia 25% (10 : 8 : 1 : 1v/v/v/v), chloroform : dioxane : methanol : ammonia 25% (4 : 10 : 1 : 1 v/v/v/v), acetonitrile : ammonia 25% (10 : 1 v/v), dioxane : chloroform : methanol : ammonia 25% : acetonitrile (10 : 4 : 1 : 1 : 1 v/v/v/v/v). The distance of mobile phase development was 8 cm. After development, the plates were dried for 30 min in a heater at 100°C. Then, the spots were observed under UV light ($\lambda_{exe} = 254$ nm).

RESULTS AND DISCUSSION

Mobile Phase Optimization According to the Simplex Method

The variables (factors) taken into account in the optimization process were the portions of solvents in mobile phase. From among mixtures used in preliminary experiments (see point 3.3 above), the best separations were obtained for mixture of dioxane : chloroform : methanol : ammonia 25% : acetonitrile (10 : 4 : 1 : 1 : 1 v/v/v/v/v). This composition, i.e., the portions of the eluent components that corresponded to all six vertices, 1–6, of the starting simplex are presented in Table 1. The optimization progressed by reflecting the vertex at which the worst separation of the drugs was observed in the center of gravity of the remaining vertices. Thus, vertex 7R (see Table 1) was obtained by reflecting point 3 in the center of gravity of points 1,2,4,5, and 6, 8R–after reflection of vertex 2 in the center of gravity of points 1, 4, 5, 6, and 7R. Further reflections did not result in improvement of the chromatogram quality. This is why the simplex 1, 4, 5, 6, 7R, 8R was contracted towards the best vertex 8R (contraction factor = 0.5). The reflection of the worst point, 10C, in the contracted simplex, resulted in vertex 14R, the 'best' one, which, together with points 8R, 9C, 11C, 12C, and 13C made a new simplex. The reflection of the vertices of the latter simplex (13C → 15R, 9C → 16R, 12C → 17R) did not result in an improvement of drugs separation and the best eluent composition corresponded to vertex

Table 1. Simplex optimization of the mobile phase in TLC for the mixture of drugs: MDMA, PMMA, PMA, and ephedrine. Steps 10, 4, 2, 2, 2 were taken for volume of dioxane, chloroform, methanol, acetonitrile, and ammonia 25% respectively

Experiment No.[a]	Dioxane (%)	Chloroform (%)	Methanol (%)	Acetonitrile (%)	Ammonia (%)
1	10	4	1	1	1
2	10	8	1	1	1
3	19	6	1	1	1
4	13	6	2.5	1	1
5	13	6	1.5	1	2.5
6	13	6	1.5	1.5	1.5
7R	4.5	6	2	2	1.5
8R	11	3	2.5	2	2
9C	10.5	3.5	2	1.5	1.5
10C	8	4.5	2.5	2	2
11C	12	4.5	2.5	1.5	1.5
12C	12	4.5	2	2	1.5
13C	12	4.5	2	1.5	2
14R	**15**	**3.5**	**2**	**1.5**	**1.5**
15R	12	3	2.5	1.5	1
16R	14	4	2	2	1.5
17R	14	3	2.5	1.5	1.5

[a]R = reflection; C = contraction.

14R. The linear independence between factors (coordinates of simplex vertices) was controlled in the course of the optimization process.

The quality of separation (optimization parameter) was estimated by inspection of the chromatograms obtained with the use of the mobile phases whose compositions resulted from the simplex optimization strategy. In order to show the effectiveness of optimization, let us compare the differences between R_f's of ephedrine, PMMA, MDMA, and PMA at the vertex 1 of the starting simplex and at vertex 14R (optimum mobile phase composition). At vertex 1, the above mentioned differences amounted to 0.06; 0.02; 0.05, and at vertex 14R: 0.05; 0.04, and 0.08. Moreover, optimum mobile phase ensure better separation between PMMA and MDMA which spots before optimization were only partly separated. In further experiments, the optimum mobile phase was used.

Comparison of TLC Separation of Drug Mixtures Dissolved in Methanol and Phosphate Buffer Solutions

The R_f values of drugs tested proved to be higher if drugs were dissolved in methanol than in buffer medium. A possible explanation of this fact is that

in methanol the drugs are present in the form of hydrochloride, while in buffer solution they probably exist as phosphates. The lower R_f for phosphates than for chlorides may be a result of differences in the size of corresponding salts and their solubility, the phosphates being larger than chlorides then less mobile.

Influence of Individual Additives on TLC Separation

All studied additives influenced the separation of the drugs: they changed the R_f values of the studied drugs. The influence was stronger if drugs were dissolved in methanol. In this case, an addition of more than 5% of glucose or 10% of other additives caused an overlap of the MDMA and PMMA spots. An addition of citric acid and magnesium stearate influenced most significantly the R_f values (see Figure 1). In the case of drugs dissolved in buffer solutions, the strongest influence was noticed for glucose and acetylsalicylic acid (see Figure 2). On TLC chromatograms, four spots corresponded to MDMA, PMMA, PMA, and ephedrine can clearly be distinguished, the presence of 50% acetylsalicylic acid being an exception.

Some additives: caffeine, procaine, glucose, paracetamol, acetylsalicylic acid extinguished fluorescence of the TLC plate at 254 nm, but their spots do not interfere with spots of drugs tested.

Influence of Two-Component Matrices on TLC Separation

On the basis of experiments carried out on two-component matrices, according to a 2^2 factorial design, the regression coefficients, b, in the following polynomial model were calculated:

$$R_f = b_0 + b_1X_1 + b_2X_2 + b_{12}X_1X_2$$

The above equation approximates the dependence of R_f value on concentrations, $X_1$and X_2, of additives in drug matrix.[21,22] The main, b_1 and b_2, and interaction, b_{12}, effects of concentrations of additives in drug matrix are presented in Table 2 for the drugs dissolved in methanol and buffer solutions, respectively.

From Table 2, it is seen that magnesium stearate significantly influences R_f values of the studied drugs in methanol solution. Except for few cases, its effect (b_1) proved to be significant at $\alpha = 0.05$ and 0.01. In most cases the effects of the second additive were significant as well. Matrices with glucose, eatable citric acid, and 1-phenylethylamine show the biggest influence on R_f's.

The separation of drugs appears more resistant to the influence of matrix components if they are dissolved in buffer solution. For example, it is seen that additions of magnesium stearate and procaine hydrochloride did not influence

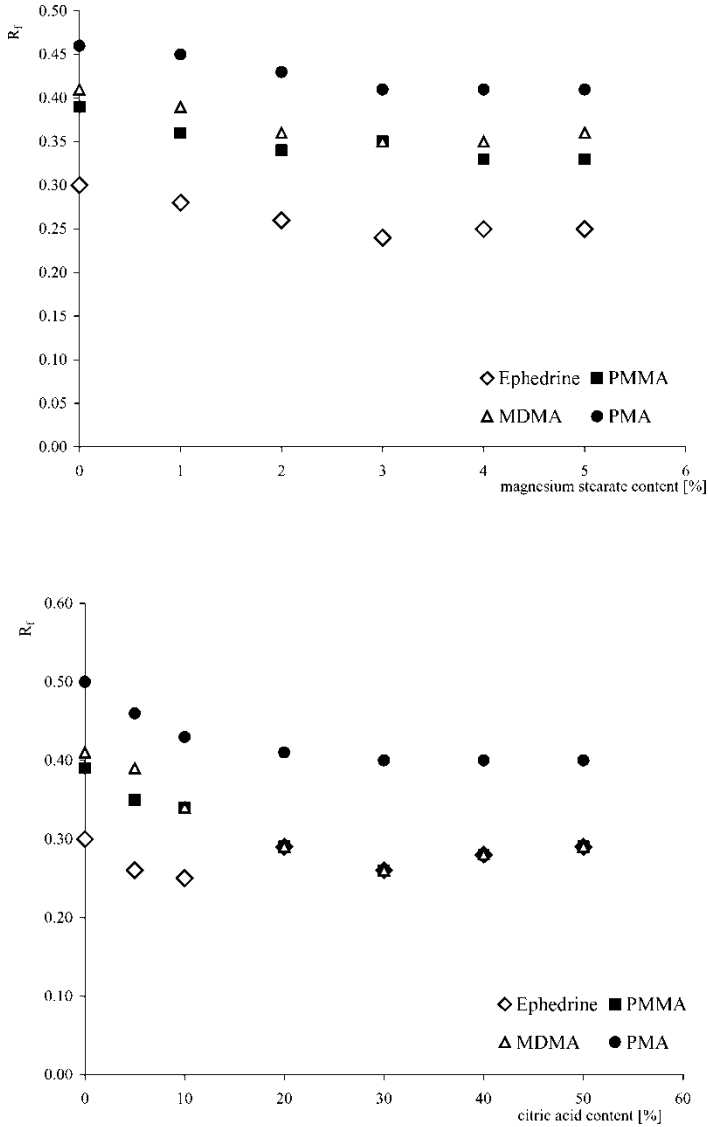

Figure 1. The influence of magnesium stearate and citric acid on R_f of components of drugs mixture in methanol.

the TLC separation of drugs tested. Only one effect (b_{12} for PMMA) proved to be significant ($\alpha = 0.05$) in the case of matrix composed of magnesium stearate and 1-phenylethylamine. A small influence of matrix components was observed when magnesium stearate was accompanied by plaster, parace-

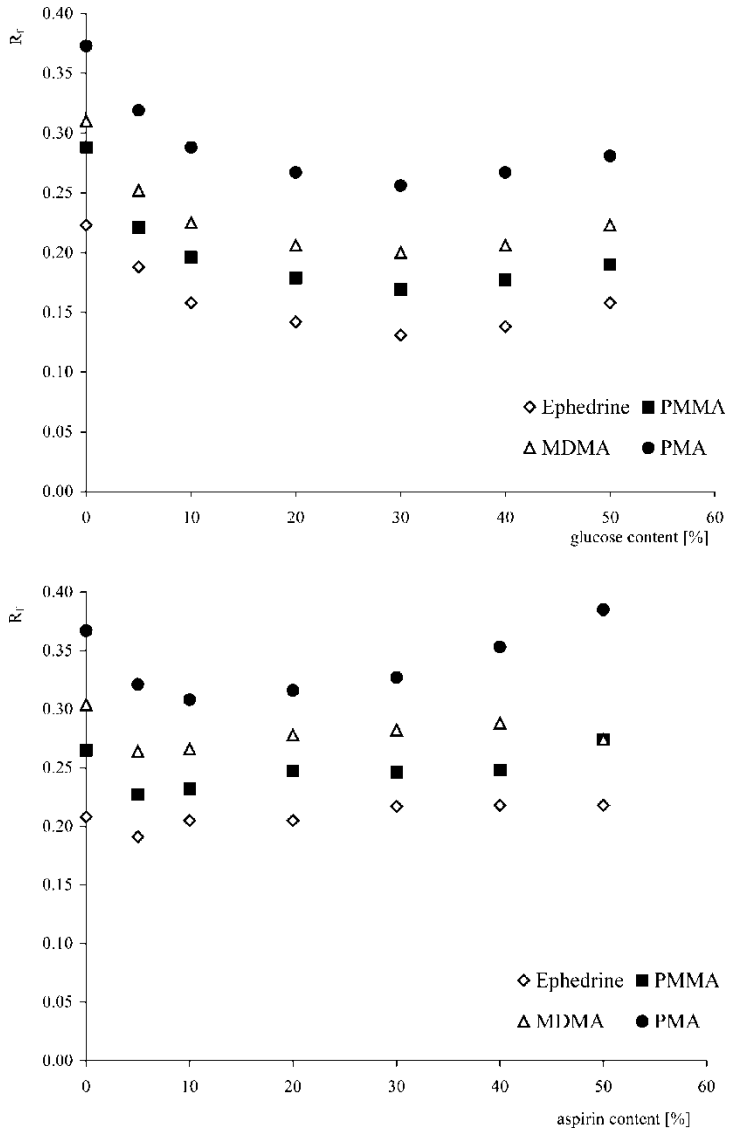

Figure 2. The influence of glucose and aspirin (acetylsalicylic acid) on R_f of components of drugs mixture in buffer solution.

tamol, or 'chemical' citric acid. A larger influence of matrix components was observed for starch, caffeine, and eatable citric acid.

As compared to other drugs tested, the influence of additions on the R_f value of MDMA appeared to be the weakest (see Table 2). Except for

Table 2. Main, (b_1, b_2) and interaction (b_{12}) of concentration of additives on R_f values of drugs and components; bold values indicate the effects significant at $\alpha = 0.05$ while underlined values at $\alpha = 0.01$

Composition of drug matrix	MDMA			PMA			PMMA			Ephedrine		
	b_1	b_2	b_{12}	b_1	b_2	b_{12}	b_1	b_2	b_{12}	b_1	b_2	b_{12}
Magnesium sterate, glucose												
Buffer	−0.002	−0.009	**0.022**	−0.012	−0.009	**0.022**	0.001	−0.01	**0.02**	−0.001	−0.012	0.012
Methanol	**0.02**	**−0.005**	**−0.01**	**−0.008**	0.003	**−0.008**	**0.023**	**0.013**	**−0.013**	**0.01**	**0.005**	**−0.01**
Magnesium sterate, castor sugar												
Buffer	**−0.028**	−0.001	−0.005	**−0.036**	0.002	0.002	**−0.028**	0	−0.007	**−0.02**	−0.005	−0.007
Methanol	**−0.005**	**0.005**	**−0.035**	**0.015**	0	0	**0.018**	0.003	**−0.018**	**0.028**	**0.018**	**0.008**
Magnesium sterate, starch												
Buffer	0.003	−0.013	**0.026**	−0.006	−0.016	**0.028**	0.007	−0.015	**0.028**	0.004	**−0.017**	**0.017**
Methanol	0	**−0.035**	**−0.03**	**−0.005**	**−0.015**	**−0.02**	0.003	**−0.018**	**−0.033**	**−0.005**	**0.015**	**−0.025**
Magnesium sterate, chalk												
Buffer	**−0.03**	0.001	−0.007	**−0.034**	0.002	−0.002	**−0.028**	0.001	−0.007	**−0.024**	−0.001	−0.011
Methanol	**0.028**	**−0.013**	−0.003	**0.02**	**−0.005**	**0.005**	**0.03**	**0.005**	**−0.005**	**0.018**	−0.003	−0.003
Magnesium stearate, plaster												
Buffer	−0.019	0.009	0.004	**−0.028**	0.006	0.006	−0.017	0.008	0.004	**−0.017**	0.004	−0.004
Methanol	**0.028**	−0.003	−0.003	**0.015**	0	0	**0.03**	**0.015**	**−0.005**	**0.015**	**0.005**	**−0.005**
Magnesium stearate, acetylsalicylic acid												
Buffer	−0.013	0.003	0.01	**−0.039**	0.018	−0.006	**−0.021**	0.007	−0.001	−0.005	**0.017**	0.008
Methanol	**0.025**	**−0.005**	**−0.005**	**0.015**	0	0	**0.028**	**0.013**	**−0.008**	**0.018**	**0.018**	−0.003

(*continued*)

Table 2. Continued

Composition of drug matrix	MDMA			PMA			PMMA			Ephedrine		
	b_1	b_2	b_{12}	b_1	b_2	b_{12}	b_1	b_2	b_{12}	b_1	b_2	b_{12}
Magnesium stearate, paracetamol												
Buffer	−0.009	−0.02	0.014	−0.015	−0.018	0.018	−0.004	**−0.023**	0.017	−0.001	**−0.025**	0.012
Methanol	**0.018**	0.003	**−0.013**	**0.008**	0.003	**−0.008**	**0.02**	**0.02**	**−0.015**	**0.008**	0.003	**−0.013**
Magnesium stearate, caffeine												
Buffer	0.004	−0.014	**0.027**	−0.009	−0.012	**0.024**	0.001	−0.016	**0.022**	0.002	**−0.015**	**0.015**
Methanol	**0.013**	**−0.018**	**−0.018**	0	**0.01**	**−0.015**	**0.015**	0	**−0.02**	**0.008**	**0.032**	**−0.013**
Magnesium stearate, citric acid (chemical)												
Buffer	−0.018	−0.012	0.005	**−0.025**	−0.009	−0.09	−0.011	−0.016	0.01	−0.008	**−0.017**	0.005
Methanol	0.003	**−0.033**	**−0.028**	−0.003	**−0.008**	**0.005**	**0.005**	**−0.015**	**−0.03**	−0.003	**0.018**	**−0.023**
Magnesium stearate, citric acid (eatable)												
Buffer	0.006	−0.016	**0.029**	−0.006	−0.015	**0.027**	0.007	−0.015	**0.028**	0.002	**−0.015**	**0.015**
Methanol	**0.025**	**−0.035**	**−0.005**	**0.048**	**−0.038**	**0.033**	**0.028**	**−0.018**	**−0.008**	**0.02**	**0.015**	0
Magnesium stearate, procaine hydrochloride												
Buffer	−0.006	−0.005	0.017	−0.015	0	0.019	−0.008	−0.007	0.013	−0.001	0	0.012
Methanol	**−0.005**	**−0.02**	**−0.035**	**−0.015**	**0.005**	**−0.03**	0.003	−0.003	**−0.038**	**−0.01**	**0.03**	**−0.03**
Magnesium stearate, 1-phenylethylamine												
Buffer	−0.004	−0.006	0.019	−0.015	−0.006	0.019	−0.003	−0.012	**0.025**	−0.002	−0.011	0.011
Methanol	**0.013**	**0.008**	**−0.018**	0	**−0.015**	**−0.015**	**0.015**	**0.025**	**−0.02**	**0.01**	**0.015**	**−0.01**

ephedrine, magnesium stearate changed the R_f values of drugs, its main (b_1) and interaction (b_{12}) effects were significant in most cases.

CONCLUSION

The proposed method can be used for screening identification of drug mixtures composed of MDMA, PMA, PMMA, and ephedrine. But special attention should be paid on possible presence of additives. The TLC identification procedure carried out after a drug sample has been dissolved in buffer solution appears more resistant to the influence of matrix components. From among the additives examined, magnesium stearate exerts the most important influence on TLC separation of the studied mixture of drugs dissolved in buffer solution (pH 7).

The proposed TLC procedure is easy to perform, it does not require sophisticated and expensive equipment. It enables determination of main psychoactive components of 'ecstasy' tablets simultaneously for several drug samples. This makes the method attractive for forensic laboratories as screening procedures.

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