# Application of Thin Layer Chromatography to Profiling of 3,4-Methylenedioxymethamphetamine (MDMA)

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## INTRODUCTION

**Drug profiling** (especially profiling of organic impurities in drug samples) helps finding connections between drug sample sized by the police, illicit laboratory and distribution routes. The composition of impurities in drug sample depends on the method of drug synthesis, substrates used, intermediate products, purification process and diluents used in preparation of final products for illegal market. This composition of impurities is usually expressed by chromatogram named a **profile of impurities**.

In the present work thin layer chromatography (TLC) was applied to obtain profiles of 'ecstasy' tablets that contain MDMA (3,4-methylenedioxymethamphetamine) as main psychoactive component. The quality of MDMA profile (TLC chromatogram) was characterized by the symmetrical matrix of dimension equal to the number (n) of spots revealed (fluorescence methods).

## MATRIX CHARACTERIZATION OF TLC PROFILE

$$\begin{bmatrix} a_{11} & a_{12} & a_{13} & a_{14} & \cdots & a_{1n} \\ & a_{22} & a_{23} & a_{24} & \cdots & a_{2n} \\ & & a_{33} & a_{34} & \cdots & a_{3n} \\ & & & a_{44} & \cdots & a_{4n} \\ & & & & \cdots & \\ & & & & & a_{nn} \end{bmatrix}$$

$a_{ij} = 1$ if spots *i* and *j* completely separated
$a_{ij} = 0.5$ if spots *i* and *j* are partly separated
$a_{ij} = 0$ if spots overlap

$\lambda_{exc}$=366nm:
$a_{ii} = 1$ if spot *i* fluoresces intensively
$a_{ii} = 0.5$ if spot *i* exhibits clear fluorescence
$a_{ii} = 0.1$ in case of faint but visible fluorescence
$a_{ii} = 0$ no fluorescence

$\lambda_{exc}$=254nm:
$a_{ii} = 0$ fluorescent indicator of plates is extinguished by separated substances

## PROFILE QUALITY CRITERIA

$Y_1 = \Sigma a_{ij}$ (for $\lambda_{exc}$ = 254 nm)
$Y_2 = \Sigma a_{ij}$ (for $\lambda_{exc}$ = 366 nm)
$Y_3 = \Sigma a_{ii}$ (for $\lambda_{exc}$ = 366 nm)

## PROFILING PROCEDURE

*Preparation of drug samples*
- 75 mg of MDMA was dissolved in 550 µl of buffer solution (pH = 7)
- MDMA solution was shaken for 20 minutes
- centrifugation of sample was performed for 5 minutes at 13000 rpm

*Solid-phase extraction*
- C18 (Baker Bond, 100 mg) extraction columns were used
- 500 µl of MDMA solution after centrifugation was injected onto SPE column
- MDMA was eluted from the column using two portions (each 1 ml) of distilled water
- impurities was eluted using five portion (each 100 µl) of methanol
- the extract was evaporated under a stream of nitrogen
- the residue was dissolved in 40 µl of methanol

*TLC separation*
- horizontal developing chamber (Chromdes, Poland)
- silica gel plates (10x10 cm) with fluorescent indicator $60F_{254}$ (Merck, Germany)
- the mobile phase:
  - mixture of acetone: chloroform: methanol (2:7:1 v/v/v) - *system I*
  - mixture of 1,4-dioxane : chloroform: methanol: 25% aqueous ammonia (6: 2: 2: 1 v/v/v/v) - *system II*
- volume of sample solution placed on TLC plate: 3 µl
- the developing distance: 8 cm
- after development the plates were dried at 100 °C
- detection of spots: $\lambda_{exc}$ = 254 and 366 nm

## RESULTS AND DISCUSSION

From Table 1 it is seen that methanol proved to be the most effective in elution of impurities from C18 SPE column. From among four tested eluents in TLC separation presented in Table 2 the last proved to be the most effective (high values of Y parameters). The examples of chromatograms are presented in Fig. 1. Data presented in Table 3 show that the profiles of MDMA samples obtained by different synthesis method significantly differ.

Table 1 Profile quality criteria obtained by using different washing solvents (MDMA prepared from isosafrol, mobile phase: *system I*)

| washing solvent | quality criteria | | |
|---|---|---|---|
| | $Y_1$ | $Y_2$ | $Y_3$ |
| chloroform | 15 | 19.5 | 1.5 |
| n-hexane | 0 | 0 | 0 |
| methanol | 6.0 | 25 | 3.5 |

Table 2 Profile quality criteria obtained by using different mobile phases (MDMA prepared from isosafrol, mobile phase: *system I*)

| mobile phase | quality criteria | | |
|---|---|---|---|
| | $Y_1$ | $Y_2$ | $Y_3$ |
| chloroform: methanol (9: 1) | 2.5 | 19.5 | 2.8 |
| chloroform: methanol: acetonitrile (5:2:3) | 9.0 | 20.5 | 2.3 |
| acetone: chloroform: methanol (2:7:1) | 6.0 | 25 | 3.5 |
| 1,4-dioxane: chloroform: methanol:ammonia (6:2:2:1) | 8.5 | 27.5 | 2.9 |

Table 3 Profile quality criteria for two samples of MDMA (mobile phase: *system I*)

| MDMA precursor | quality criteria | | |
|---|---|---|---|
| | $Y_1$ | $Y_2$ | $Y_3$ |
| isosafrol | 6 | 25 | 3.5 |
| piperonal | 3 | 11 | 1.6 |

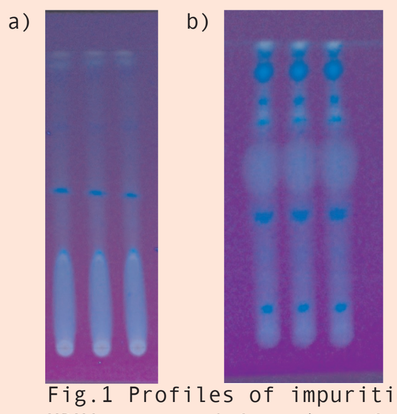


Fig.1 Profiles of impurities MDMA prepared from isosafrol; observed at 366nm;
a) mobile phase - *system I*
b) mobile phase - *system II*

## CONCLUSION

The proposed SPE/TLC profiling methods proved to be an effective tool to differentiate between MDMA samples synthesized according to different synthesis methods

References
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