 DOI: 10.33892/aph.2021.91.222-223

P-30

Determination of Psychotropic Drugs by Thin-Layer Chromatography

GEGECHKORI VLADIMIR I.[1]; SALTYKOVA OLESIA V[1]; RODIONOVA GALINA M.[1]

[1]I.M. Sechenov First Moscow State Medical University of the Ministry of Health of the Russian Federation (Sechenov University), 8, Trubetskaya str., Moscow, 119991, Russia

Correspondence: vgegechkori@gmail.com

Keywords: *Paroxetine, fluvoxamine, sertraline, fluoxetine, pregabalin, thin-layer chromatography*

1. Introduction

Antidepressants are the most prescribed drugs in developed countries; however, a third of all prescribed drugs have non-psychiatric indications. Selective serotonin reuptake inhibitors (SSRIs) stand out among antidepressantsas the safest and most well-tolerated ones compared to earlier drugs such as tricyclic antidepressants, while showing similar efficacy. The main advantage of SSRIs over other groups of antidepressants is the selective inhibition of only one type of biogenic amines, which helps to prevent undesirable side effects. This has a positive effect on the tolerability of this group of drugs by the body. As a result, their popularity among patients and specialists is growing every year [1]. Combined poisoning often occurs due to a combination of psychotropic drugs and potentiation of effects, in particular with pregabalin.

Thin-layer chromatography is one of the most common methods of preliminary analysis used in forensic chemical studies. This method occupies one of the leading places in the qualitative analysis of complex natural, pharmaceutical and chemical objects [2].

2. Materials and methods

During the TLC authenticity tests of SSRI antidepressants (paroxetine, fluvoxamine, sertraline, fluoxetine) and one of the antiepileptic drugs (pregabalin), Russian ready-made chromatographic plates with a fixed layer of silica gel were used.

50 μl of 0.1% alcohol solutions of paroxetine, fluvoxamine, sertraline, fluoxetine and pregabalin (1 mcg) were applied to the start line of the chromatographic plate at a distance of 1 cm from each edge using an Agilent Gold Standard micro syringe with a straight needle volume of 1 to 100 μl.

To determine the optimal applied volume for the analysis, the linear capacity of the sorbent was determined by applying a series of samples of a standard solution of 0.1% pregabalin (in ethyl alcohol). As a result of the experiment, it was found that when applying from 50 to 600 μg of a standard solution of pregabalin, the minimum sorbent capacity and the R_f value of the test compound fall within 10% of their values for the linear part of the adsorption isotherm.Thus, when 50 to 100 μl of a standard solution of pregabalin was applied to a chromatographic plate, R_f values remained unchanged. As the load increased, $R_f$value began to plummet and reached the point where it markedly decreased.

At the stage of application of the samples, the plates were heated using the USP-1 Heating Device with a given temperature of 55 °C to obtain the most compact spots, which in turn increases the efficiency and clarity of separation.

To detect colorless substances, first of all, one should use physical methods based on light absorption and fluorescence. When the plate is irradiated with UV radiation, absorbent substances in this part of the spectrum are detected in the form of dark zones (spots).

Chemical methods include the use of "universal reagents" and reagents that selectively react with certain functional groups of the analyzed compounds.

The chromatogram was air-dried and the analyzed drugs were detected in UV light (λ = 254 nm), iodine vapor, and then using the Dragendorff reagent in the Mounier modification.

3. Results

An out-of-laboratory assessment of solvent systems was carried out. On this basis, the most effective systems were selected for the separation of

the analyzed medicinal compounds of weakly basic and basic nature. A large number of factors influence the quality of separation of the components of a mixture in thin-layer chromatography: the type of separation chamber; preliminary saturation of the chamber and the sorbent layer with vapors of the mobile phase; starting spot size; distance from start to bottom edge of plate; relative humidity and temperature of the air and the laboratory premises; the presence of microdamage to the layer; elution rate; volume of solvent in the chamber; the presence of impurities in the eluent [3].

To obtain optimal separation of the analytes, mixtures of solvents with different polarity were prepared in certain proportions characterized by maximum separation ability. Solvent systems: S_1: toluene-acetone-ethanol-25% ammonia (45:45:7:3); S_2: isoropanol-acetone-25% ammonia-water (22:25:4:7); S_3: acetone-25% ammonia (9:1); S_4: toluene-acetone-ammonia 25% (50:50:4); S_5: methanol-25% ammonia (100:1.5).

Table 1 *Chromatographic mobility of the analyzed medicinal substances in the solvent system*

Analyzed substance	Solvent system / R_f value				
	(S_1)	(S_2)	(S_3)	(S_4)	(S_5)
Paroxetine	0,77	0,87	0	0,67	0,41
Fluvoxamine	0,91	0,93	0,86	0,91	0,69
Sertraline	0,94	0,92	0	0,93	0,70
Fluoxetine	0,84	0,86	0,82	0,78	0,56
Pregabalin	0,80	0,51	0,10	0,27	0

According to the obtained data, only one of the mobile phases we studied, the acetone-25% ammonia system (9:1) (S_3) makes it impossible to separate the analyzed substances from one another. In the four remaining systems, separation is sufficient to identify these drugs. However, fluvoxamine and sertraline have similar R_f values in these systems; therefore, the acetone-25% ammonia (9:1) system can be used to separate the analyzed medicinal substances from one another.

4. Conclusions

From the obtained data we can conclude that the following mobile phases are recommended in TLC screening for detection and separation of fluoxetine, paroxetine, fluvoxamine, sertraline and pregabalin: S_1: toluene-acetone-ethanol-25% ammonia (45:45:7:3); S_2: isopropanol-acetone-25% ammonia-water (22:25:4:7); S_4: toluene-acetone-25% ammonia (50:50:4); S_5: methanol-25% ammonia (100:1.5).

References

1. *Drobizhev, M. Yu. Selektivnye ingibitory obratnogozahvata serotonina i noradrenalina: vozmozhno li sochetanie effektivnosti i bezopasnosti? [Selective serotonin and norepinephrine reuptake inhibitors: is a combination of efficacy and safety possible?] // "Psychiatry and Psychopharmacotherapy" – 2004. – № 6(5), p. 248–250.*
2. *Berezkin, V.G. Konusnaya tonkoslojnaya-hromatografiya [Cone thin layer chromatography] // Academic paper. – 2008. – Ed.8. – p. 556-559.*
3. *Shapovalova, E.N., Pirogov, A.V. Hromatograficheskie metody analiza: metodicheskoe posobie [Chromatographic analysis methods: a guidance manual] – Moscow – MSU publishing house –2007*