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# THIN LAYER CHROMATOGRAPHY OF LYSERGIC ACID DIETHYLAMIDE (LSD), N,N DIMETHYL TRYPTAMINE (DMT), METHYLENE DIOXYPHENYL ISOPROPYLAMINE (STP), AND IBOGAINE

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Quite frequently, the Police Laboratory is faced with the task of identifying a compound, or mixture of compounds, that the narcotic undercover agents have obtained on the illicit market. The days of confining one's examination to heroin, cocaine, or morphine are gone. More and more hallucinogens of a rather exotic nature are appearing, and in many cases, the Police Laboratory is faced with identifying a drug before the methods appear in the literature. Such was the case with LSD and Ibogain (1; 2) until we published procedures suitable for forensic use.

This paper presents the results of work done at the Suffolk County Police Laboratory and gives procedures that have been found to be rapid, convenient, reproducible, and valid for the separation and detection of several of the hallucinogens with which the forensic chemist is faced, and for which there is very little or no data available in the analytical literature.

The technique of thin-layer-chromatography (TLC) has been immeasurably promoted with the advent of commercially prepared sheets and plates instead of requiring the investigator to make his own. We have found commercially prepared plastic sheets and glass plates coated with 250 microns of silica gel quite effective and uniform from batch to batch. We have also prepared small glass plates (3″ x 1″) using microscope slides and coating them with either alumina, silica gel, calcium carbonate, or other adsorbent.

Samples of hallucinogens were obtained from various sources[1] and were identified by physical data such as melting point, UV absorption spectra, and microcrystalline tests, as well as by chemical reactions where such are feasible.

Where solvents were used, the "spectrograde" or reagent grade purity was utilized without further treatment or purification. Instead of using lambda pipettes, glass capillary tubes approximately 0.5 mm in diameter were used to spot the compounds. These have the advantage of being inexpensive and disposable. Adsorbents were purchased, specifying TLC grade where such was available; otherwise reagent grade was used. Slurries were prepared by simply shaking the adsorbent with distilled water at an approximate concentration of 10 per cent.

Many attempts were made to find a relatively simple solvent system. Combinations were tried with varying results. Those which gave excellent results with two hallucinogens would not separate a third or fourth, and since the choice of solvent system is empirical, a great many trials resulted. The system finally adopted consisted of 25 ml of

[1] Sources of material:

| | |
|---|---|
| LSD | Courtesy of Sandoz Pharmaceuticals, Hanover, N. J. |
| Mescaline | Courtesy of N.Y.C. Police Dept. |
| Ibogaine | Courtesy of CIBA Pharmaceutical Co., Summit, N. J. |
| Phencyclidine | Courtesy of Parke Davis & Co., Detroit, Mich. |
| DMT | Purchased from S. T. Baker Chem Co. |
| MDA | Purchased from Aldrich Chemical Co., Inc. |
| STP | Seized contraband, purified by Suffolk County Police Lab |

TABLE 1

| | Rf | U.V. longwave | UV short | $I_2$ fuming | Acid Spray* |
|---|---|---|---|---|---|
| Ibogaine | 0.89 | — | Quenched | Brown | Brown |
| LSD | 0.82 | Fluoresce | Fluoresce | Yellow-Brown | Blue |
| Phencyclidine | 0.77 | — | — | Brown | Yellow-Orange |
| DMT | 0.34 | Quenched | Quenched | Yellow-Brown | Brown |
| STP | 0.26 | — | — | Yellow | Disappears |
| Mescaline | 0.20 | — | — | Yellow | No change |
| MDA | 0.33 | | | | |

* The acid spray consisted of 0.5 N $H_2SO_4$ and then heating to 110° C.

trichlorethylene, 25 ml of ethyl acetate, 35 ml of 1-Butanol and 10 ml of Methanol. This mixture was placed in a covered chromatograph jar and allowed one hour to reach equilibrium. We use this waiting period to spot our TLC plates.

Our known samples were dissolved in methanol and were spotted on Eastman Chromatographic plastic film sheet type K301 with fluorescent indicator or on glass plates similarly coated. Our samples were placed on the paper by means of the previously described capillary tubes. Consistent and reproducible Rf values with a maximum variation from the mean of 0.04 were found after many runs. Table 1 lists the mean Rf values for those drugs studied as well as other means of qualitative identification.

In addition to the separation and identification, several additional factors are of interest. The Rf values of Ibogaine and LSD are close, and yet this is no problem because of the fluorescent characteristic of the LSD. DMT and MDA with close Rf values are easily differentiated on the plate itself due not only to the quenching under UV but the DMT spot is large and very intense after $I_2$ fuming. We tried additional drugs such as quinine, cocaine, heroin, morphine, codeine, and procaine in this system, and we found the system works well with these drugs also. Consistent, repeatable results were obtained during each run.

From a consideration of the behavior of the known hallucinogens on TLC, we accordingly devised an experimental procedure to detect and identify unknowns when they are submitted to the Police Laboratory.

The forms in which the hallucinogens are submitted are many and ingenious. LSD, for example, occurs on blotting paper, chocolate candy "kisses", on the surface of aspirin tablets, on the surface of gelatin capsules which are later filled with some innocuous material, or in the form of pressed colored scored tablets. DMT often is sprayed on parsley leaves, on the surface of gelatin capsules, or in a solution. MDA occurs as a powder in a capsule, Ibogaine mixed with heroin in a capsule. Mixtures of the above with amphetamine or methamphetamine have been submitted by the narcotics investigator. In view of the great diversity of forms of evidence received by the forensic chemist, a procedure suitable for almost any type of hallucinogen-containing evidence would be advantageous. We believe that we now have such a procedure.

The technique is as follows: The evidence, in whatever form, is first examined grossly under ordinary illumination. Extraneous spots or discolored areas on the surface of a tablet or paper, for example, should be noted and delineated. Following this, the same material should be examined under relatively long-wave UV.[2] Fluorescent areas or spots should be noted and marked with a graphite pencil. A similar examination under relatively short-wave UV should be made[3] and again fluorescent areas observed. The fluorescent or discolored areas should be removed and dissolved in methanol. If a gelatin capsule is involved, shaking with chloroform will dissolve enough of the material to enable an identification to be made. Otherwise, whatever the evidence, it can be shaken with, or dissolved in chloroform. The clear solution (centrifuged, if necessary) is then evaporated to dryness at room temperature in a 30 ml beaker by blowing a current of air across the beaker and the residue taken up in a few drops of methanol.

The now concentrated methanol solution is taken up in a capillary tube as heretofore described, and

[2] We use one made by BLAK-RAY Ultra Violet Products Co.

[3] We use Mineralight made by the same company.

spotted on a plate or film along with suitable standards. The spotted surface is placed in the solvent system and allowed to remain until the solvent front has migrated about two-thirds up the sheet. The chromatogram is then removed and dried. It is now examined under relatively long and short wave UV light as described above, and the fluorescent areas outlined with pencil. If this designates the spots satisfactorily, no further treatment of the chromatogram is necessary. If not, iodine fuming should be done. This will indicate other spots (see Table 1). If necessary, acid spraying and heating will demonstrate the remaining spots. Rf values in each case should be determined and compared with the known spots.

As further means of identification, the spots can be scraped off into a small test tube, eluted with 0.1N $H_2SO_4$ and spectra recorded, either in UV or IR, and compared with known spectra.

Using this procedure, we have been able to separate mixtures of several micrograms of each of the hallucinogens studied, and to identify them to our complete satisfaction. As a crude screening test, a small chromatogram with either a microscope slide coated with an appropriate adsorbent or a small strip cut from a TLC film may be spotted with the suspected material and a single or two knowns. These may be chromatographed using a screw-cap 4 oz. jar, with about 10 ml of the solvent system.

We are at present extending this system to separation of other narcotics from hallucinogens or other materials that are submitted from time to time, and will report on these at a later date.

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