



Comparison and evaluation of the quick purification methods of methamphetamine hydrochloride from dimethyl sulfone for spectroscopic identification



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ABSTRACT

Methods to quickly purify methamphetamine hydrochloride from the cutting agent dimethyl sulfone for subsequent identification of confiscated crystalline samples using infrared absorption spectroscopy were compared and evaluated. Although sequential solvation and reprecipitation methods were simple, spectral contamination from dimethyl sulfone was inevitable and might affect the interpretation of the spectra. In addition, methamphetamine hydrochloride and dimethyl sulfone could form a solid solution because of solvation of both crystals into a single solution layer. By contrast, sublimation was an effective method for separation of methamphetamine hydrochloride and dimethyl sulfone. Sublimation combined with infrared absorption spectroscopy enabled rapid identification of crystalline methamphetamine hydrochloride.

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1. Introduction

Methamphetamine (MA) is one of the most widely abused drugs in the world. According to the United Nations Office on Drugs and Crime, seizures of amphetamine-type stimulants, including MA, are rapidly increasing [1]. For a forensic laboratory, fast and robust analytical methods to identify MA are critically important. In general, MA is distributed in many forms, including a crystalline form known as ice, liquid form, and tablet form called Ya-ba [1]. Appropriate pretreatment methods are required before applying the MA seizure to the instrumental analysis, and the selection of pretreatment methods should depend on the form of the specimen.

Infrared absorption (IR) spectroscopy is a feasible analytical technique in the field of forensic drug analysis. IR spectra provide information on the molecular vibration of functional groups in a compound. Since the molecular vibration is unique and highly specific to the chemical structure, this method can be used to identify drugs. For example, different from chromatography, changes of compounds' salt forms could be probed by IR spectroscopy [2]. There are many reports on the application of

IR spectroscopy to drug analysis such as for amphetamine type stimulants [3–5], cocaine [6–9], heroin [10,11], and many types of new psychoactive substances [12]. In particular, attenuated total reflectance (ATR) IR spectroscopy is a powerful technique that uses only a small quantity of sample to perform robust measurement. In addition, this method is very simple and any sample pretreatment processes are unnecessary if the sample form is suitable for measurement (e.g. crystalline or powder).

However, IR spectroscopy has a major disadvantage. Analysis of mixtures is difficult because the spectra of mixtures are complicated and hard to interpret. There have been many studies attempting to overcome this problem by using spectral analysis. A chemometric approach using principal component analysis, partial least squares modeling, or non-negative matrix factorization, has previously been explored for the analysis of complex spectra [13–18]. There are many software packages available to perform multivariate analysis, and they enable us to use multivariate analysis without the deep knowledge on the mathematical principles of chemometrics. Although these methods can provide good results for decomposing the spectra of the target compound and any contaminants, maturity of the multivariate analysis is indispensable for correct interpretation of the results. Without the knowledge on multivariate analysis, at worst misinterpretations can occur [19]. Therefore, physical separation of a mixture provides an important pathway for resolving the IR spectra of mixtures.

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In this study, we focused on rapid separation method of MA hydrochloride (HCl) and dimethyl sulfone (DMSO₂) in crystalline samples. DMSO₂, or methylsulfonylmethane, is used industrially as a high-temperature solvent due to its high polarity and stability. This compound is also used as a dietary supplement and in cosmetics, although its pharmacological effects are under discussion. Solid DMSO₂ is in the form of transparent crystals, and apparently resembles solid MA HCl. Due to this similarity, DMSO₂ is frequently used as a cutting agent for crystalline MA HCl [20–23]. The IR spectra of MA HCl and DMSO₂ are clearly different and easy to distinguish. However, a mixture of these compounds gives a complicated IR spectrum. To rapidly and conclusively identify MA in a seizure, quick and complete separation of MA HCl and DMSO₂ is very important. For this purpose, we compared sublimation [24], reprecipitation [25], and sequential solvation [26] methods for separation of these chemicals. The IR spectra obtained were evaluated from the view point of spectral purity. During the evaluation, peak shifts were observed for the DMSO₂ bands, and powder X-ray diffraction (XRD) measurements was used to investigate why this occurred.

2. Experimental

2.1. Chemicals

Methanol, diethyl ether, chloroform, and acetone were purchased from Wako Pure Chemical Industries (Osaka, Japan). All solvents were of analytical grade or better. MA HCl authentic standard (tradename Philopon) was obtained from Dainippon

Pharmaceutical (Osaka, Japan). MA HCl for preparation of samples was issued from the Narcotic Agent, Ministry of Health, Labor and Welfare (Tokyo, Japan). DMSO₂ was purchased from Tokyo Chemical Industry (Tokyo, Japan).

2.2. Sample preparation

MA HCl and DMSO₂ were weighed and mixed in 1:1 by weight. Samples of MA HCl, DMSO₂, and their mixture were crushed and homogenized in agate mortars. These homogenized powder samples were used for following experiments.

2.3. Purification by sublimation

This method used sublimation of DMSO₂ [24]. The powdered 1:1 mixture of MA HCl and DMSO₂ (10 mg) was placed in a glass petri dish. The dish was covered and placed on a hotplate heated to 100 °C. Subsequently sublimation of DMSO₂ occurred, as evidenced by the deposition of white powder on the petri dish cover. The dish cover was changed to a clean one after a certain period, and we confirmed that no further deposition of the white powder occurred. The remnant in the base of the petri dish was retained as a sample for ATR-IR spectroscopy (Fig. 1a).

2.4. Purification by reprecipitation

This method used the different solubilities of MA HCl and DMSO₂ in diethyl ether [25]. The powdered 1:1 mixture of MA HCl and DMSO₂ (10 mg) was placed in a test tube. Then, 100 μL of

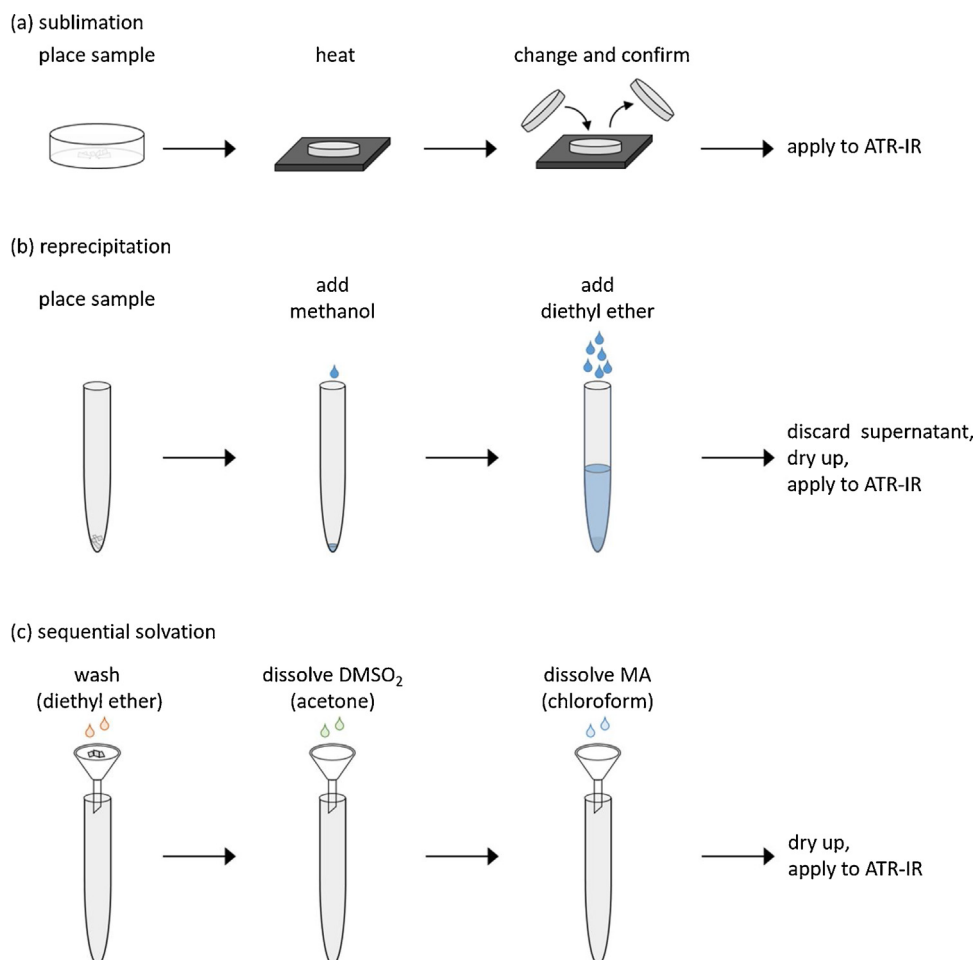


Fig. 1. Schematic of methods for separating MA HCl and DMSO₂ by (a) sublimation, (b) reprecipitation, and (c) sequential solvation followed by ATR-IR spectroscopy.

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