Alkaloid extraction methods pdf

Common methods of extracting and isolating alkaloids from plant sources need to take into account the following steps consistently, namely: (i) Separating alkaloids (s) from the bulk of non-akalcaloid substances, (ii) Most alkaloid-containing plants, several alkaloids with closely related chemical structures are usually present, such as: cinchona alkaloids consist of more than twenty-native alkaloids. There is hardly any known plant source that contains only one alkaloid exclusively, (iii) separating each individual alkaloid from a mixture of alkaloids derived from a specific plant source (e.g. cinchona bark) using the latest separation techniques, such as the drug-producing high-performance liquid chromatography, (HPLC) column chromatography, using chromatotron, and high performance for example: (a) the alkaline nature of most alkaloids, b) the ability and simplicity of the formation of alkaloid salts with acids, and (c) the relative solubility of alkaloid salts obtained or in polar organic solvents such as ethanol, chloroform, etc. It is also based on the quantum and main parts of the raw materials that will be used in the operation. Of course, for research purposes, the chromatography column using ion-exchange resins is successfully and effectively used for strips of plant materials of their alkaloid content. However, on a commercial scale, large volumes of plant extracts of plant materials are usually pumped through huge metal columns, packed cation resins, which in turn pick up all the main components (katia). Subsequently, alkaloids (i.e. the main components are conveniently washed away by rinsing the column with moderately strong acid. , the following five steps are the most important and vital, namely: (i) Preparation of a sample (ii) Release of a free alkaloid base (iii) Extracting an alkaloid base with an organic solvent (i) extract (i) a fraction of raw alkaloids All these five steps should be discussed individually, as in accordance with: 1.7.1 Preparation of sample First and a step is to prepare a sample. The plant material is reduced to moderately coarse powder with grinding plants and a sieve to facilitate the most effective contact of the solvent with torn alkaloid bearing tissues and cells. In the case of plant substances rich in oils and fats, such as: seeds, nuclei, these non-alcaloid chemical components should be completely eliminated by extraction with a suitable non-polar solvent, such as neksan, light oil ester, in the apparatus soxhlet, which will not extract the alkaloids in question. However, it is always advisable to shake the light-oil ether or n-hexane fraction with diluted mineral acid and then check the acidic solution for the presence of alkaloids. 1.7.2 Release of the free alkaloid base It has been observed that alkaloids are invariably found in plant sources as salt acids, such as oxalates, tanates, etc. Choosing alkaline indeed, the choice of a suitable mineral base (alkaline) for the simplicity of releasing alkaloids from salts is not only very important, but also just as significant and largely depends on the following factors, namely: a) The natural state of alkaloids: It has been observed that salt is strongly the main alkaloid with mineral acid, usually tends to split under the influence of a stronger base. Similarly, the corresponding salt of the weak underlying alkaloid and relatively weak organic acid requires a rather weak basis for its cleavage. (b) The chemical characteristics of the alkaloid base: Strong alkaline, such as NaOH or KOH, should be avoided due to the fact that some alkaloids are hydrolysed in prolonged contact with a strong base. Example (i) hydrolysis of ester alkaloids, such as cocaine, hyosiamine; (ii) phenolic alkaloids such as cepheline, morphine. These alkaloids are usually dissolved when exposed to strong alkaline and therefore milder alkaline reagents, such as diluted ammonia solution necessary for their release. (c) The presence of fatty substances: the use of strong alkaline acid is strictly prohibited in the case of fat-containing plant materials due to the formation of saponified products that cause troublesome emulsions. In such cases, it is always preferable to defate the plant matter before releasing free alkaloids. The ammonium hydroxide solution dilutes the akim hydroxide solution is one of the most commonly used to release alkaloids from plant sources. It has a good advantage. First, being its adequate alkalinity to release most common alkaloids, and secondly, his character, so that it can be removed Solvent. Since it tends to be extracted by the ester solvent from the aquier solution, therefore it is almost necessary to get rid of it by evaporating and then washing again. In normal practice, usually even the last traces of ammonia are removed when the combined essential extract is reduced to half of its original volume in a vacuum. NaOH or KOH Solution are alkaloids that occur naturally as their salt tanata specifically require either a NaOH or KOH solution for their subsequent release. In some typical cases, even the use of KOH or NaOH is unable to break down tanata salts due to their closely strong bondage with alkaloid and extremely insoluble nature. Example (i) Cora Sincona: It must be processed first by heating with diluted HCl in order to decompose salts and release alkaloids in the form of water-soluble hydrochlorides, and ii) pomegranate bark: It has no tannin so tenaciously associated with alkaloids as in the case of the cionanch bark. Thus, the NaOH solution is strong enough to cause an effective split of alkaloid salts. It also acts to control the solubleness of water-soluble pomegranate alkaloids, preventing their dissociation. 1.7.3 The extraction of the alkaloid base of alkaloid base extraction can be carried out by three different types of solvents, which are discussed below, namely: A Mining with water-producing solvents Many alkaloids and their corresponding salts are soluble in alcohols, such as: methanol, ethanol, isopropaneol; thus, these very solvents can also be used to extract plant substances. The usual pre-treatment of raw lye can be avoided completely, as alcohol seems to affect the dissolution not only of alkaloid salts, but also of the loose bases contained in plant substances. It is believed, however, that alcohol mainly has a hydrolysis effect on alkaloid tanates and other salts. In practice, there is no need for pre-treatment of raw drug alkaline, or acidification of alcohol with a small amount of mineral acid or organic acid. Note 1. The penetration and therefore subsequent extraction of the raw drug is almost completed with the help of four consecutive extractions with alcohol. In addition, the loss of solvent is relatively less than chlorinated solvents, such as chloroform. 2. The extraction of full alkaloids with alcohol is highly recommended because of its maximum effectiveness and economic viability. Mining with water solvents In fact, the most widely used water-immy solvents for the extraction of alkaloids are: chloroform, ditil ether (ether solvent) and isopropyl ether. However, several other specific organic solvents, namely: ethylene chloride, carbon tetrachloride and benzene can used with obvious obvious for some specific alkaloids. Interestingly, chloroform is regarded as the best water-measuring solvent for a wide range of alkaloids present in the plant kingdom, and extracts them with varying degrees of lightness. Note: Chloroform is not suitable for the extraction of quarter alkaloids, such as tulorarine. The raw drug is extracted with water pre-acidized with diluted solution HCl, H2SO4 or CH3COOH, which subsequently turns out to be alkaline, preferably with diluted NH4OH solution and is finally extracted with water solvent as indicated in the above. Undoubtedly, water is an excellent and absolutely inexpensive polar solvent for the extraction of alkaloids, but if it offers a huge amount of disadvantages, because it carries with it a large number of other components of plants, for example: sugar, pigments (e.g. chlorophyll), starches, tannins, proteins, etc., which ultimately puts through a collosity of time, energy and chemicals. Thus, its use has resulted in a bear minimum level. Typically, alkaloids can be extracted by any of the following three well-defined and widely accepted processes, namely(a) the Soxhlet (b) Production Process of Stas-Otto and c) the Kippenberger process. All three processes will now be briefly discussed in the sections that follow: a) Soxhlet extraction process: Soxhlet assembly is a continuous extractor that is usually suitable for extracting alkaloids from powdered plant materials using organic solvents. In this case, the powdered drug is usually moisturized with a solution of diluted ammonia, and then freely packed into the thimble of the Soxhlet apparatus; and organic solvent provides deep penetration of the moist drug, allowing the maximum possible extraction of alkaloids from the open surfaces of cells and tissues of crude drugs. Once the extraction is found to be complete, the solvent is filtered and evaporated into the rotary thin-film vaporizer and the residues are processed further to isolate the individual alkaloids. (b) Stas-Otto process: The Stas-Otto process essentially consists of the treatment of a powdered and sifted drug 90-95% (v/v) of ethanol, previously acidified with tartare acid. The proportion of raw food in the solvent should be maintained at a rate of 1 kg to 1 litre. If any alkaloid is removed by the oil ester, it should be restored by treating it with diluted mineral acid. Thus, the resulting aka extract is mixed with the bulk of the aquitay extract. Combined aqueous extract filtered evaporates to dry preferably in a Rotary thin film vaporizer under a vacuum. Vacuum. residues are extracted by absolute ethanol, thus dissolving the common alkaloid. (c) Kippenberger process: In the Kippenberger process, the powdered and sifted vegetable matter is primarily digested with a solution of tannin (100 g) in glycerol (500 g) at a constant temperature of 40 degrees Celsius for 48 hours. The resulting mixture is additionally heated to 50 degrees Celsius to help in the full clotting of the proteins, cooled to the ambient temperature and finally filtered. The resulting filter is carefully shaken by the oil ester to get rid of faulty materials (oils, fats and waxes), and the last traces of the oil ester are removed from the extract by heating either in a water bath (electric) or when exposed to an infrared lamp. The skimmed extract of the raw plant is subsequently acidified and shaken by chloroform, consistently removing the bulk of the alkaloids, namely atropine, codeine, colchicine, narcotin, nicotine, papaverin, spantin and thebaine. The resulting residual extract may still contain narsein, kurarin and morphine. However, narsein and morphine can be isolated by transferring freshly generated CO2 directly into the extract in order to convert alkaline hydroxide into the corresponding carbonate, which is then eventually extracted by solvent using a mixture of alcohol and chloroform. Finally, the third alkaloid, kurarin, can be extracted by arousing with a mixture of equal volumes of ether and chloroform. However, the combination of the Kippenberger process and the Stas-Otto process by its application to the final alcoholic extract obtained by the latter process is found to give a better separation of the alkaloids. 1.7.4 Cleaning of alkaloid extract The bulk of raw alkaloid extract is invariably further cleaned with any or combination of the following methods: (a) Extraction with acid solution Extracting alkaloids from the bulk of raw alkaloid soluble in non-immimic organic solvents is invariably carried out by shaking acid solution. In normal practice, the use of HCl is limited when chloroform remains solvent due to the fact that quite a few alkaloids are distinctly soluble in the latter. However, diluted H2SO4 is always preferable to HCl for general use in alkaloid extraction. Subsequently, the acid solution is alkaline with a diluted NH4OH solution to release alkaloids, which are then extracted with an organic solvent. The solvent is removed under low pressure and traces of moisture are removed using sodium sulfate anhydrose. Note: The following two precautions may be observed, namely (i) To avoid the formation of stubborn and troublesome emulsion solution gumtragacanta is often added to the aquelus phase. In the if it still persists the two stages can be separated and (ii) To avoid the presence of foreign interfent extractive ingredients present in plant substances such as: pigments, resins, waxes, oils and fats, the use of 2.5-5% (w/v) solution of lead acetate is produced in an alkaloid extract that precipitates them effectively. Excess lead present in the filter is removed either by transferring H2S gas through the Kippa machine or by adding sodium phosphate. (b) Precipitation of the alkaloide from the besieging reagents Conventional precipitation of the alkaloid as a complex compound is achieved by adding a suitable precipitate reagent. The resulting alkaloid complex is additionally cleaned by filtration, recrystalization and eventually decomposes to obtain the desired free alkaloid (s). Example (i) Tannico-acid Complex: Usually decomposed by processing freshly prepared Pb (OH)2 or Pb(CO3)2. (ii) Precipitation obtained with HgCl2, AuCl3, PtCl4, Myer reagent: These precipitation decomposes as H2S gas flows through its suspension. (iii) Double salt precipitation: Double salt obtained from the Dragendorf regent is quickly cooked with BaCO3 5%. (iv) Nitrogen acid precipitation: nitrogen acid precipitation, such as picric acid and picrononic acid, is usually decomposed by either NH4OH or NaOH. (v) Reinecata Complex: Complex derived from alkaloide with Reineke salt, NH4 (NH3)2 (SCN)4, usually decomposes by treating its solution in a mixture of acetone and water (1:1) with a solution of silver sulfate. It is appropriate to mention here that the free released alkaloid from the complexes mentioned above (i) via (v) can be further extracted for its final recovery with an appropriate organic solvent, such as: chloroform. (c) The cleaning of alkaloids can also be done by the formation of crystallized alkaloid salt by adding appropriate mineral or organic acid, such as salt, hydrobromic, perchloric, sulphuric, sorrel and tartare acid. (d) Various known methods of separation, namely, partition, ion and column chromatography are invariably used to clean the host alkaloid. In addition, various physical parameters, such as: specific rotation, melting point, solubility are often used as a certain criterion for establishing the purity of alkaloids. 1.7.5 A fraction of raw alkaloids have been observed mainly that most alkaloid plant materials usually contain a mixture of closely related alkaloids. Therefore, it became almost necessary to carry out an effective fraction of raw alkaloids from the extract or solution of all raw alkaloid. However, traditional and orthodox methods of separation are not only difficult, but also tedious and cumbersome. Widely used separation methods found reliable and reliable can be a short list as follows: (i) factional crystallization, (ii) fractional distillation, and (iii) excretion with low salt products. The newest methods of alkaloid separation are the drug-high-performance liquid chromatography (HPLC), high performance of thin-layer chromatography (HPTLC), chromatotron, counter-flowing distribution and other chromatographic methods, including chromatography column, ion-exchange chromatography. Below are some of the typical situations in which the alkaloid mixture can be separated effectively, such as: (a) Most alkaloids are easily soluble in chloroform and relatively less soluble in other organic solvents. In general, the order of solubility, as indicated below chloroform and acetone, ethanol and methanol , ethyl acetate, ether n-hexane. Taking into account the above profile of alkaloid soluble in organic solvents, if one of the alkaloids is much less soluble in ethanol than chloroform, fractional crystallization of this alkaloid is possible. In this particular case, the chloroform fraction is concentrated to the appropriate level, and hot ethanol is added in small proportions at intervals. Thus, when cooling the alkaloid, which is less soluble in ethanol, separates comfortably. (b) If the fractional crystallization of a mixture of closely related alkaloids becomes tedious and inefficient, you can try to form their respective salts and then separate them as above. (c) Various acids, namely: HCl, HBr, HI, HClO4, HNO3, C2H2O4 and C6H3N3O7, can be used in aquier or methanol solution. Thus, from the resulting methanol solution, the salt of the respective alkaloids can be besieged by the addition of ether. Beleaguered raw alkaloid salts can be further redrawn from hot acetone containing a small proportion of methanol. (d) In some other specific cases, salts of the respective oxalates, picrats and perchlorates may be caused by their solutions in acetone by adding ethyl acetate. --- benzene: It is a carcinogenic chemical and therefore its use can be avoided or done in a highly effective smoke cabinet. Alkaloid salts: which are often used hydrochloride, hydrobromide, hydroiodide perchlorate, nitrate, oxalate and picrat. Source: Pharmacognosia and Pharmacobiotechnology Ashutosh Kar Kar alkaloid extraction methods pdf

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