

METHOD FOR OBTAINING A TROPA-ALKALOIDS RAW EXTRACT FROM UNDRIED PLANT PARTS

Abstract

New process for obtaining tropa-alkaloid raw extracts, in particular scopolamine and/or hyoscyamine from undried plant elements.

Classifications

[C07D451/10](#) Oxygen atoms acylated by aliphatic or araliphatic carboxylic acids, e.g. atropine, scopolamine

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DE3603678A1

Germany

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Worldwide applications

1986 [DE](#) 1987 [AU](#) [WO](#)

Application DE19863603678 events

1986-02-06 Application filed by [Boehringer Ingelheim GmbH](#)

1986-02-06 Priority to [DE19863603678](#)

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1987-01-31 Priority to [PCT/EP1987/000048](#)

1987-08-13 Publication of [DE3603678A1](#)

Status [Withdrawn](#)

Info: [Patent citations \(1\)](#), [Non-patent citations \(6\)](#), [Cited by \(2\)](#), [Legal events](#), [Similar documents](#), [Priority and Related Applications](#)

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Claims (4)

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translated from German

1) Process for the production of a crude alkaloid extract from undried plant parts of an alkaloid-containing plant, characterized in that

- a) the plant parts are extracted directly or repeatedly with water and / or
- b) the plant parts are pressed out using a press and, if desired, the pomace
 - b₁) treated one or more times with water and squeezed again and that if desired
- c) the aqueous crude extracts (raw miscella) obtained according to a) or b) are concentrated after separation of the solids contained therein and if desired
- d) the concentrate obtainable according to c) is extracted with a suitable organic solvent.

2) Method according to claim 1, characterized in that the plant parts in the first stage with a screw press presses the thus obtained Squeezed juice, and the pomace in or extracted several times with water. 3) Method according to claim 1 or 2, characterized characterized in that the extraction of the Plant material with water at temperatures between 10 and 70 ° C, preferably between 15 and 40 ° C is carried out. 4) Method according to claim 1 to 3 for the production of Scopolamine and / or atropine and / or hyoscyamine.

Description

translated from German

The invention relates to a new method for extraction of Tropa alkaloid crude extracts, especially from Scopolamine and / or hyoscyamine crude extracts undried parts of plants.

It is known to use scopolamine besides hyoscyamine or also both components by extraction from plants alone or parts of plants to win (F. E. Hamerslag; The Technology and Chemistry of Alkaloids; D. van Nostrand Comp. Inc. Toronto, New York, London, 1950).

Dried are used to produce the crude alkaloid extract Plant parts either with aqueous acids or with treated aqueous bases and then the extract with a suitable solvent extracted again.

From the multitude of publications published for this are to be mentioned:

Obtaining scopolamine from *Datura stramonium* or *Atropa belladonna* by extraction with aqueous ammoniacal Solution and benzene, 1,2-dichloroethane or toluene according to IN 1 30 397 (CA 91 1 81 445 w).

Extraction of *Duboisia Leichhardtii*, *Datura alba* or *Scopolia carniolica* with aqueous ammoniacal solution and a hydrocarbon fraction (naphthanol) JP-A-75-49 412 (CA 83 65 453 h).

Extraction of plants of the genus *Datura* with a ammoniacal water-ethanol mixture FR-A-24 05 947 (CA 91 1 98 926 b).

Extraction of *Datura sanguinea* or *Datura metel* with an aqueous acid - preferably sulfuric acid or Hydrochloric acid - and di-, tri- or tetrachloromethane according to EC 151.

A recent study compared the extraction of scopolamine from *Datura innoxia* in alkaline (NH_3) and acidic (H_2SO_4 , HCl) aqueous medium, with the best results being obtained with 1% hydrochloric acid (Farmacia, (1973) 21, 403; CA 80 1 00 142 t).

The known from the prior art Extraction processes have the disadvantage that the quality by aqueous-alkaline or aqueous-acidic extraction obtained crude extract very strongly from the pH of the depends on the respective extractant, whereby only at defined pH values and extraction times Side reactions such as hydrolytic decomposition of the Alkaloids or the alkaloids ester can be avoided. Due to the acidic or basic character of the Extractants also become more unwanted plant ingredients dissolved and complicate the further processing of the raw extract.

Surprisingly, it has now been found that Tropa alkaloid extracts in high yield and good Purity from undried parts of plants alkaloid plant can be obtained.

The invention thus relates to a method of manufacture of a Tropa alkaloid crude extract from undried Parts of a plant containing an alkaloid, characterized in that one

- a) the plant parts extracted directly or repeatedly with water
or
- b) the plant parts are pressed out using a press and, if desired, the pomace
b₁) treated one or more times with water and squeezed again and that if desired
- c) the aqueous crude extracts obtained according to a) or b) (Raw miscella) after separation of the contained therein Concentrate solids and if desired
- d) the concentrate obtainable according to c) with a extracted suitable organic solvent.

The obtained by the method according to the invention Raw miscella, the raw miscella concentrate or raw concentrate is largely free of unwanted accompanying substances, which the quality of the final product (scopolamine and / or Hyoscyamine) adversely affect.

Plants containing Tropa alkaloid include:

Datura species, such as *Datura stramonium*, *Datura alba*, *Datura metel*, *Datura sanguinea* or *Datura innoxia*

Atropa species, such as *Atropa belladonna*,

Duboisia species, such as *Duboisia leuichhardtii*

Scopolia species, such as *Scopolia carniolica*

Tropa alkaloids are an alkaloid group known per se; scopolamine, hyoscyamine and atropine are mentioned.

Among the undried plant parts are primarily the aerial parts of plants such as stems, leaves, Understand flowers and fruit clusters immediately after the Harvest can be processed without drying.

Plant parts are also plant parts made of cell or Suspension cultures.

The extraction of the plant parts with water can any temperature, d. H. at temperatures between 0 and 100 ° C, the boiling point of the water. However, temperatures of 10 to 70 ° C. are preferred the temperature range from 15 ° C. to is particularly preferred 40 ° C.

The method according to the invention has a number essential advantages. In addition to improving Product quality of raw miscelles or Raw miscella concentrate or the raw alkaloids extract to quote:

- Simplification of processing:

The drying of the harvested plant parts necessary according to the prior art is not necessary.

- The production of raw miscella or extracts can be on or in the immediate vicinity of the installation site take place so that as a process backlog resulting pomace as a valuable fertilizer or Mulch material returned to the cultivated area can be. It is particularly advantageous that the plant extraction residues are not with organic solvents or others non-plant chemicals were in contact. they can thus be safely deposited or used as fertilizer or mulch material can be used.

- Furthermore, the inventive Process also stems and other parts of plants are used, whereas in the case of the state of the Extraction process known in the art use only dried leaves.

- According to the method of the invention is also omitted otherwise with alkaline or acid extraction necessary neutralization step, what both in terms of procedural security, as well is advantageous from an ecological point of view.

In addition, a neutralization Racemization of the alkaloids observed what Product quality clearly deteriorates.

The raw miscella is the aqueous extract or the pressed juice, d. H. an aqueous solution or emulsion or suspension of Crude alkaloids.

The crude extract is that obtained by extracting the raw miscella with an immiscible or only miscible with water Extract extract available.

Such extraction methods are state of the art. As Suitable extraction agents are:

Chlorinated hydrocarbons, such as dichloromethane, Trichloromethane, carbon tetrachloride, dichloroethane, Trichloroethane, tetrachloroethane;

Aromatic hydrocarbons, such as benzene, toluene, xylene;

Ethers such as diethyl ether; Cyclic ether like Tetrahydrofuran u. a.

Hydrocarbons such as naphtha fractions, petroleum ether

In a preferred embodiment, the inventive method as a combination of Squeezing and extraction in a multi-stage process carried out. In the first stage, the undried (fresh) alkaloid plant parts using pressed out in a suitable press and the resultant Pomace in a two-step process with water in the Extracted countercurrent and then the resulting aqueous Extract (raw miscella) with the pressed juice (raw miscella) united and worked up further.

Screw presses are particularly preferred as presses, because by the shear forces that occur Plant material is pressed particularly efficiently.

The from the aqueous extract and / or pressed juice Raw miscella obtained can, if desired, be filtered and to the raw miscella concentrate according to known Process by distillation or by diffusion process be concentrated.

The gentle distillative is particularly preferred Concentrate on a plate evaporator.

The following example illustrates the invention without restricting it to them.

example 1

1a Preparation of a plant-miscella concentrate

In the first stage, 1000 kg of freshly harvested ones *Datura-sanguinea* wet drug (equivalent to 120 kg Dry drug) with a chopper and with a Grind the mill with an 8 x 8 mm sieve.

The wet drug obtained in this way is pressed out using a screw press with a 1 mm round perforated sieve. 798 kg of pressed juice I and 202 kg of pomace I are obtained.

In the second stage, the pomace is then combined with the pressed juice III in a stirred kettle and 45 min. Stirred at room temperature and then pressed again with a screw press with 1 mm round sieve. 1052 kg of pressed juice II and 168 kg of pomace II are obtained. The pomace II thus obtained is stirred in the third stage in a stirred

kettle with 1000 kg of water for 45 minutes at room temperature.

The entire kettle contents are then pressed out again using a screw press with a 1 mm round perforated screen. 1018 kg of pressed juice III and 150 kg of pomace III are obtained. The pressed juice III produced at this stage is added to the pomace I in the subsequent batch at stage 2. The pomace III obtained in the third stage is deposited.

The press juices I and II obtained from the preceding stages are mixed in a suitable kettle, the press juice being loaded with a solids content of about 3.5% by volume.

The solid is separated off in a separator. 167 kg of solid suspension are obtained in addition to 1899 kg Separately (raw miscella) with a solids content of approx 0.2 vol%.

The separation is carried out in a vacuum using a plate evaporator concentrated to 185 kg of concentrate.

1b Preparation of the crude extract

185 kg of the concentrate obtained according to Example 1a (Solids content: approx. 3 vol.%) Are added via a Separator separated (operating mode: total purification with Displacement). 10 kg of solid suspension and 175 kg are obtained separately.

The separator is then placed over a plate filter clear filtered. After rinsing the filter with 50 kg of water you get 195 kg of filtrate.

The filtrate is passed through a pulsed sieve tray column Chloroform extracted.

289 kg (195 l) of chloroform extract, which is obtained with 106 g of 48% hydrobromic acid are added and up to 1s evaporated to dryness. 0.746 kg of crude scopolamine HBr extract are isolated with a Scopolamine hydrobromide content of about 70%.

Patent Citations (1)

Publication number	Priority date	Publication date	Assignee	Title
US2509051A *	1946-04-17	1950-05-23	Appelzweig Norman	Extraction of alkaloids from fresh plants
Family To Family Citations				

* Cited by examiner, † Cited by third party

Non-Patent Citations (6)

Title
Chem. Abstr. 79, 1973, 96 882 u *
Chem. Abstr. 85, 1976, 11 2692 p *
Chem. Abstr. 86, 1977, 16 1179 f *
Chem. Abstr. 92, 1980, 82 421 z *
Chem. Abstr. 99, 1983, 36 003 m *
Khim. Farm. Zh. 7, 1973, 54-57 *

* Cited by examiner, † Cited by third party

Cited By (2)

Publication number	Priority date	Publication date	Assignee	Title
US8658221B2	2007-12-17	2014-02-25	Bayer Cropscience Ag	Extraction process for plant ingredients
Family To Family Citations				
GB9903034D0 *	1999-02-10	1999-03-31	Phytopharm Ltd	Extraction of volatile oils

* Cited by examiner, † Cited by third party, ‡ Family to family citation

Similar Documents

Publication	Publication Date	Title
DE69915241T2	2004-12-30	Isolation and purification of paclitaxel and other related taxanes by commercial preparative low pressure chromatography on a polymeric resinous column
DE2753478A1	1978-06-08	PROCESS FOR THE RECOVERY OF FLAVANOL OLIGOMERS
EP0971931B1	2003-02-19	Method for debenzoylation of dibenzyl biotin
DE602004011034T2	2009-02-12	IMPROVED METHOD FOR ISOLATING AND PURIFICATION OF PACLITAXEL FROM NATURAL PRODUCTS
DE3603678A1	1987-08-13	METHOD FOR OBTAINING A TROPA-ALKALOIDS RAW EXTRACT FROM UNDRIED PLANT PARTS
EP0111699B1	1987-03-25	Process for recovering furfural and monocarboxylic acids from aqueous solutions
DE3729471C2	1992-04-16	
EP1937707B1	2010-12-29	Improved method for the production of betulinic acid

Publication	Publication Date	Title
DE1793642C3	1975-05-07	Process for the production of isoionocosterone
DE2439883A1	1975-03-13	PROCESS FOR THE CONCENTRATION AND EXTRACTION OF WATER-SOLUBLE GLYCOSIDES
DE60213447T2	2007-08-23	Process for the preparation of hydroxymatairesinol or a hydroxymatairesinol complex of wood
DE1040543B	1958-10-09	Process for purifying a raw sapogenin-containing material obtained by hydrolysis of natural saponin-containing material
US2995574A	1961-08-08	Preparation of visnadin from ammi visnaga
DE3519039C2	1989-02-02	
EP0881211A1	1998-12-02	Process for the preparation of anhydrous 2-amino-1-methoxypropane
DE2837694A1	1979-03-15	PROCESS AND EXTRACTION AGENT FOR THE MANUFACTURE OF PURE PHOSPHORIC ACID
DE1445934C3	1974-02-28	Process for purifying bipyridyls
DE2358472C3	1976-08-05	Process for the production of XyloseHteungen
DE1193061B	1965-05-20	Process for the production of galanthamine hydrobromide from plants of the Amaryllidaceae family
CH395050A	1965-07-15	Process for the production of catalysts and acetic acid from an acetic acid synthesis mixture
DE69804753T2	2002-11-28	METHOD FOR SEPARATING ISOMERS OF NITROTOLUOLIC ACID
DE684014C	1939-11-20	Process for the production of fermentation glycerine from betaine-containing vinasse
DE535149C	1931-10-07	Process for obtaining alkaloids from cocoa residues by extraction with simultaneous deoiling
EP4450465A1	2024-10-23	Reactive liquid-liquid extraction of boric acid from an aqueous phase using 2-ethyl-1,3-hexanediol
AT211957B	1960-11-25	Process for the production of an isoquinuclidine alkaloid from Conopharyngia species

Priority And Related Applications

Priority Applications (4)

Application	Priority date	Filing date	Title
DE19863603678	1986-02-06	1986-02-06	METHOD FOR OBTAINING A TROPA-ALKALOIDS RAW EXTRACT FROM UNDRIED PLANT PARTS
BR8705408A	1986-02-06	1987-01-31	PROCESS FOR PREPARING A CRUDE ALKALOID EXTRACT TROOP OF UNDRIED PLANT PARTS
AU69495/87A	1986-02-06	1987-01-31	Process for obtaining a tropa-alkaloid raw extract from undried plant elements
PCT/EP1987/000048	1986-02-06	1987-01-31	Process for obtaining a tropa-alkaloid raw extract from undried plant elements

Applications Claiming Priority (1)

Application	Filing date	Title
DE19863603678	1986-02-06	METHOD FOR OBTAINING A TROPA-ALKALOIDS RAW EXTRACT FROM UNDRIED PLANT PARTS

Legal Events

Date	Code	Title	Description
1987-08-13	OP8	Request for examination as to paragraph 44 patent law	
1990-07-12	8130	Withdrawal	

Concepts

machine-extracted

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Name	Image	Sections	Count	Query match
method		title,claims,abstract,description	16	0.000
extract		title,claims,abstract,description	12	0.000
Hyosciamin-hydrochlorid		claims,abstract,description	7	0.000
extraction		claims,description	18	0.000
alkaloid		claims,description	17	0.000

Name		Image	Sections	Count	Query match
■ water	claims,description			12	0.000
■ alkaloid derivatives	claims,description			11	0.000
■ concentrate	claims,description			11	0.000
■ fruit/vegetable juice	claims,description			11	0.000
■ crude extract	claims,description			8	0.000
■ solid	claims,description			8	0.000
■ manufacturing process	claims,description			4	0.000
■ material	claims,description			4	0.000
■ organic solvent	claims,description			3	0.000
■ separation method	claims,description			3	0.000
■ Atropine	claims,description			2	0.000
■ atropine	claims,description			2	0.000
■ atropine	claims,description			2	0.000
■ A04AD01 - Scopolamine	abstract,description			7	0.000
■ Hyoscine	abstract,description			7	0.000
■ N-Methyl-scopolamin	abstract,description			7	0.000
■ scopolamine	abstract,description			7	0.000
■ scopolamine	abstract,description			7	0.000
■ (S)-atropine	abstract,description			5	0.000
■ hyoscyamine	abstract,description			5	0.000
■ hyoscyamine	abstract,description			5	0.000

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