Table 2 Taste of test samples based on Ryokeijutsukanto by Human Gustatory Sensation Test

No.	Samples	Taste
1.	Ryokeijutsukanto (A)*	Bitter and fairly sweet
2.	Ryokeijutsukanto (AL)*	Sweet and fairly bitter
3.	Poria Sclerotium removing Ryokeijutsukanto (A)	Bitter and astringrnt and fairly sweet
4.	Poria Sclerotium removing Ryokeijutsukanto (AL)	Fairly bitter and sweet
5.	Glycyrrhiza removing Ryokeijutsukanto (A)	Strongly bitter
6.	Glycyrrhiza removing Ryokeijutsukanto (AL)	Rather astringent, slightly sweet and slightly sour
7.	Cinnamon Bark removing Ryokeijutsukanto (A)	Bitter and sweet
8.	Cinnamon Bark removing Ryokeijutsukanto (AL)	Sweet and fairly bitter
9.	Atractylodes Rhizome removing or Atractylodes Lancea Rhizome removing Ryokeijutsukanto	Fairly bitter and sweet
10.	Poria Sclerotium	Slightly sweet and slightly bitter
11.	Glycyrrhiza	Sugary sweet and bitter
12.	Cinnamon Bark	Slightly sweet, bitter and astringent
13.	Atractylodes Rhizome	Extremely bitter
14.	Atractylodes Lancea Rhizome	Fairly sweet, bitter and rather sour
15.	Separately decocted mixture 1	Fairly sweet, bitter and astringent
16.	Separately decocted mixture 2	Fairly sweet and rather bitter
17.	Taste comparison of 1 with 2	1 is bitterer than 2
18.	Taste comparison of 15 with16	15 is bitterer than 16
19.	Taste comparison of 1 with 15	1 is slightly bitterer than 15
20.	Taste comparison of 2 with 16	They show similar taste, but 16 is bitterer and more astringent than 2
21.	Taste comparison of 1 with 3	3 is bitterer than 1
22.	Taste comparison of 2 with 4	4 is sweeter than 2, but 2 is more quaffable than 4

* (A) and (AL) indicate that the corresponding is prepared samples with Atractylodes Rhizome and Atractylodes Lancea Rhizome, respectively. No. 2, 4, 6, 8, 14 and 16 have characteristic odor of Atractylodes Lancea Rhizome.

1-2. 酸性苦味

1-2-1. ビャクジュツを使用した場合

味認識装置による測定において，苓桂朮甘湯と同時煎一味抜き苓桂朮甘湯を比較したとき，どの構成生薬を除いても，酸性苦味の数値に変化は見られなかった．例として，苓桂朮甘湯とケイヒを除いた煎出液及びケイヒの単味煎出液の比較チャートを示す (Fig. 3a)．また，各構成生薬の単味煎出液と苓桂朮甘湯の酸性苦味の数値を比較すると，ブクリョウの単味煎出液は非常に小さな数値を示し，カンゾウ及びケイヒの単味煎出液は苓桂朮甘湯とほぼ同等の数値を示したが (Fig. 2b, 2c)，ビャクジュツの単味煎出液においては苓桂朮甘湯より大きな数値を示した (Fig. 2d)．従って，味認識装置による測定においてビャクジュツを使用した苓桂朮甘湯が示す酸性苦味は，ビャクジュツに由来していると考えられる．また，ヒトによる味覚試験において，単味煎出液の味の表現は，ビャクジュツ以外の生薬は程度の差はあっても『甘い』という表現が含まれていたのに対し，ビャクジュツの単味煎出液の味は『非常に苦い』と表現された (Table 2, No. 10 ~ 14)．また，苓桂朮甘湯及びその他関連試料液についても，ビャクジュツを使用した試料液については，味を表現する際，苦味に関連する表現が多い傾向が認められ，味認識装置による結果が支持された．

1-2-2. ソウジュツを使用した場合

味認識装置による測定において，苓桂朮甘湯と同時煎一味抜き苓桂朮甘湯を比較したとき，ケイヒを除いた煎出液の酸性苦味の数値が小さかった (Fig. 3b)．また，各構成生薬の単味煎出液と苓桂朮甘湯の酸性苦味の数値を比較すると，ブクリョウの単味煎出液は非常に小さい数値を示し，カンゾウの単味煎出液は苓桂朮甘湯とほぼ同等であり (Fig. 2B)，ソウジュツの単味煎出液の数値は苓桂朮甘湯より小さかった (Fig. 2D)．一方で，ケイヒの単味煎出液は苓桂朮甘湯より大きい数値を示した (Fig. 2C)．従って，味認識装置による測定においてソウジュツを使用した苓桂朮甘湯が示す酸性苦味は，ケイヒによる寄与の割合が大きいものと考えられる．尚，ヒトによる味覚試験において，単味煎出液の味は，程度の差はあるが，どの生薬にも『甘い』と『苦い』が共に表現されていた (Table 2, No. 10 ~ 12, 14)．

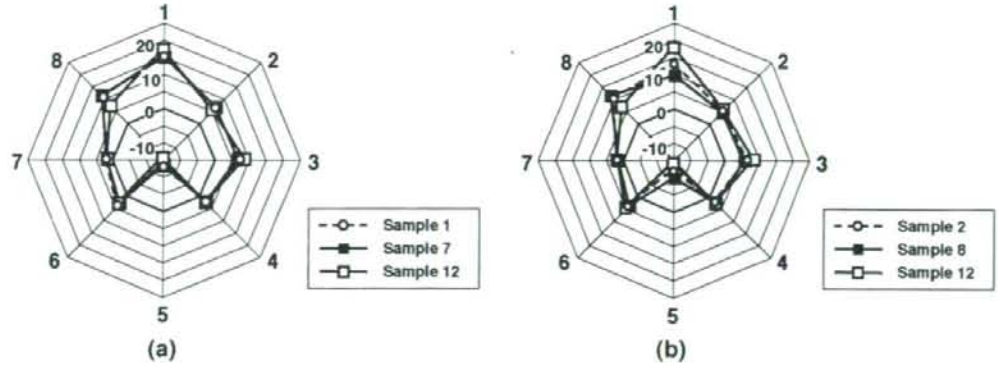

Fig. 3 Change of taste patterns by removing Cinnamon Bark from the Ryokeijutsukanto formula
The data were obtained by the taste-sensing system. The numbers on circumference of graphs are as described in Fig. 1. Sample numbers on each legends as described in Table 1.

2. ビャクジュツを使用した苓桂朮甘湯とソウジュツを使用した苓桂朮甘湯の比較

苓桂朮甘湯，別煎混合苓桂朮甘湯それぞれについて，ビャクジュツを使用した場合とソウジュツを使用した場合を比較すると，味認識装置による測定において，どちらもビャクジュツを使用した方が酸性苦味において大きな数値を示す傾向があり，特に，別煎混合苓桂朮甘湯においては，ソウジュツを使用した場合より 2 以上大きかった (Fig. 4). 一方，ヒトによる味覚試験では，苓桂朮甘湯，別煎混合苓桂朮甘湯のどちらも，『ビャクジュツを使用した方が苦味が強い』と表現された (Table 2, No. 17, 18). また，ソウジュツを使用した苓桂朮甘湯では，ソウジュツ独特の匂いが感じられ，この匂いによっても味の感じ方に違いが出るものと考えられる．尚，ソウジュツの匂いは非常に特徴的であり，匂いの識別に特別な訓練は要しなかった．

3. 苓桂朮甘湯と別煎混合苓桂朮甘湯の比較

味認識装置による測定において，苓桂朮甘湯と別煎混合苓桂朮甘湯を比較すると，ビャクジュツを使用した場合も，ソウジュツを使用した場合も，両者の間に，味分布の違いは見られなかった (Fig. 5). 一方，ヒトによる味覚試験においては，苓桂朮甘湯と別煎混合苓桂朮甘湯を比較すると，ビャクジュツを使用した場合は，『苓桂朮甘湯の方が多少苦味が強い』と表現された (Table 2, No. 19). また，ソウジュツを使用した場合は，『どちらもほぼ同じ様な味だが，別煎混合苓桂朮甘湯の方が少し苦く渋い』と表現された (Table 2, No. 20). 以上の結果を考慮すると，

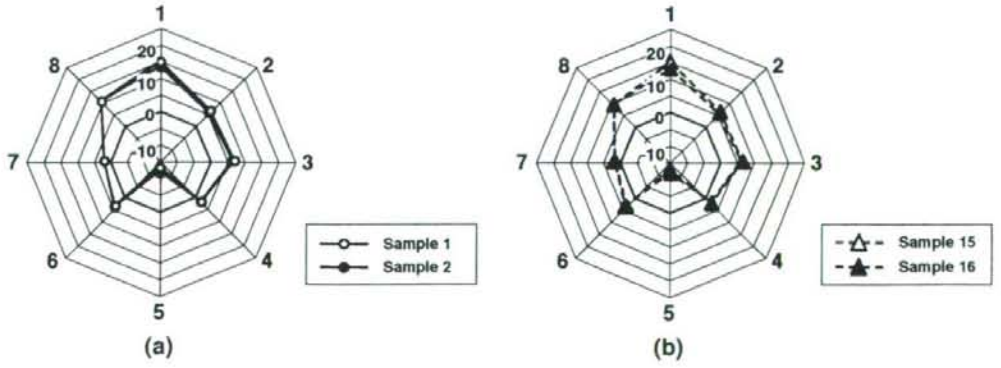

Fig. 4 Taste patterns of Atractylodes Rhizome used Ryokeijutsukanto and Atractylodes Lancea Rhizome used Ryokeijutsukanto
The data were obtained by the taste-sensing system. Ryokeijutsukanto (a). First-decocted and last-mixed Ryokeijutsukanto (b). The numbers on circumference of graphs are as described in Fig. 1. Sample numbers on each legends are as described in Table 1.

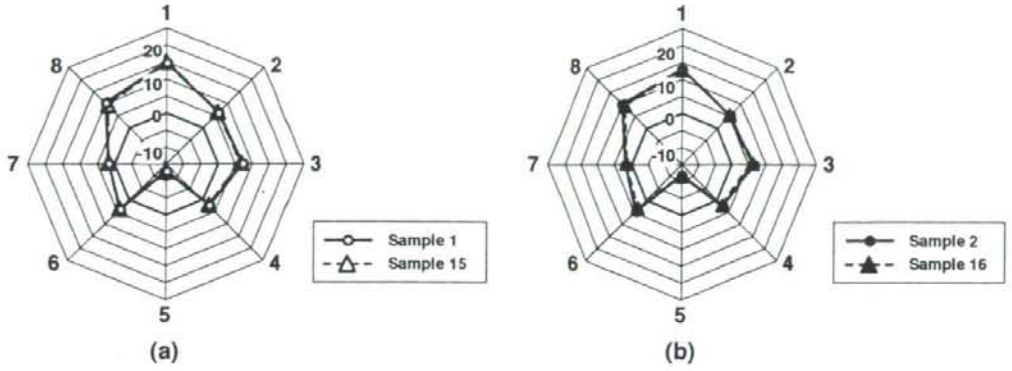

Fig. 5 Taste patterns of Ryokeijutsukanto and separately decocted mixture of Ryokeijutsukanto
The data were obtained by the taste-sensing system. Atractylodes Rhizome was used as a component for Ryokeijutsukanto (a). Atractylodes Lancea Rhizome was used as a component for Ryokeijutsukanto (b). The numbers on circumference of graphs are as described in Fig. 1. Sample numbers on each legends as described in Table 1.

苓桂朮甘湯の場合，味認識装置では，同時煎と別煎混合の両者を区別できないのに対し，ヒトでは味のわずかな違いを感じ取っている可能性が高いことが明らかとなった．

4. その他

味認識装置による測定において，ブクリョウの単味煎出液にはほとんど味が検出されない一方，ブクリョウを除いた煎出液は全体的に各味要素の数値が大きくなる傾向が見られた (Fig. 1a)．また，ヒトによる味覚試験において苓桂朮甘湯と比較したとき，ビャクジュツを使用した場合は，『ブクリョウを除いた煎出液の方が苦い』と表現され (Table 2, No. 21)，ソウジュツを使用した場合は，『ブクリョウを除いた煎出液の方が甘いが，苓桂朮甘湯の方が飲みやすい』と表現された (Table 2, No. 22)．従って，ブクリョウは処方の味をまろやかで飲みやすくする作用を有するものと考えられる．ブクリョウは，主に利水滲湿（水分代謝機能の調節）作用や精神安定及び鎮静作用を期待して使用されることが知られている [13-15]．また，各種漢方処方に配合される頻度が非常に高い．今回得られた知見をもとに考えると，処方の味を調え飲みやすくできるというブクリョウの特性は，ブクリョウが多くの漢方処方に配合される理由のひとつであるかもしれない．

今回検討に用いた苓桂朮甘湯は，前報 [2] で示した葛根湯と比べて，味がマイルドで飲みやすく，また，通常ヒトが服用する湯液と同じ濃度の試料液を味認識装置で測定することが可能であったため，ヒトと本装置の味認識について直接的に比較検討することができた．その結果，苓桂朮甘湯の苦味について，本装置による測定で得られる酸性苦味と，ヒトによる味覚試験で得られる苦味の評価に関して同等の結果が得られることが判明した．

今回，味認識装置による測定において，苓桂朮甘湯の構成生薬中のカンゾウに由来する塩味と，ビャクジュツを使用した苓桂朮甘湯についてはビャクジュツに由来する酸性苦味，また，ソウジュツを使用した苓桂朮甘湯についてはケイヒに由来する酸性苦味が，苓桂朮甘湯を特徴付ける味要素であることが示された．しかし，ヒトによる味覚試験において，前報 [2] における葛根湯の構成生薬中のマオウのように，処方独自の味を単独で強く示す構成生薬は無かった．これは，味認識装置による測定によって得られた味分布のパターンから合理的に説明可能である．即ち，葛根湯では，処方を構成する 7 種類の生薬のうち，葛根湯独自の味を強く示したマオウ 1 種類のみが葛根湯に近い数値の味分布のパターンを示した．これに対し，苓桂朮甘湯では構成生薬が 4 種類と葛根湯に比べて少ない上，そのうちの 2 種類（ケイヒとビャクジュツもしくはソウジュツ）が苓桂朮甘湯と比較的似た数値の味分布のパターンを示したものの，カンゾウは塩味及び旨味後味で数値が突出し，ブクリョウは全般的に数値が非常に小さかった (Fig. 2)．従って，苓桂朮甘湯では，葛根湯におけるマオウの様に処方の味を強く決定付ける構成生薬は無く，その味は 4 種の構成生薬の味が組み合わさることで構成されたものであることが示された．

結論

本研究では，漢方処方の品質評価の一環として，処方の味に関する客観性の高い評価法を確立することを目的とし，繁用処方のひとつである苓桂朮甘湯について味認識装置の有用性の検討を行った．その結果，以下のことが明らかになった．

1) 前報の葛根湯と同様に，味認識装置による測定により，苓桂朮甘湯においても処方の味の特徴を示す味の要素を明確にすることが可能である．2) 味認識装置による測定において，苓桂朮甘湯が示す塩味にはカンゾウが大きく寄与しており，カンゾウが示す強い塩味を主にブクリョウが低下させている．3) 味認識装置による測定において，ビャクジュツを使用した苓桂朮甘湯が示す酸性苦味はビャクジュツが大きく寄与しており，ソウジュツを使用した苓桂朮甘湯が示す酸性苦味はケイヒによる寄与が大きい．4) 味認識装置は，苓桂朮甘湯と，その構成生薬を別々に煎出した後混合した液の味の差を認識しない．5) 苓桂朮甘湯には，葛根湯におけるマオウの様に単独で処方独自の味を決定付ける構成生薬は存在しない．

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生薬学雑誌 61 (1), 44-57 (2007)

西太平洋地区４カ国（日本、中国、韓国、ベトナム）の薬局方収載生薬の各種試験法並びに規格値の比較に関する研究（第３報）生薬関連一般試験法の比較

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Comparative Study on Testing Methods and Specification Values for Crude Drugs in Pharmacopoeias among Four Western Pacific Regional Countries (Japan, China, Korea and Vietnam) (III) Comparative Study on General Testing Methods for Crude Drugs

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The Western Pacific Regional Forum for the Harmonization of Herbal Medicines (FHH) has three Sub-Committees (Sub-C). Of them, Sub-C 1 deals with the nomenclature and standardization and consists of five Expert Working Groups (EWGs 1-5). The task of EWG 5 is to list the information on general testing methods for crude drugs described in the general test section of each Pharmacopoeia among four countries (Japan, China, Korea and Vietnam). In this paper, we show the results of the task work.

In the sections of sampling, foreign matter, loss on drying, total ash, acid-insoluble ash, extract content, essential oil content, arsenic limit test and heavy metals limit test, there are many similarities among the four Pharmacopoeias. However, the method of microscopic examination in the Pharmacopoeias of China and Vietnam were completely different from those of Japan and Korea. Namely, the former describe detailed techniques and observation points.

Keywords: FHH, Crude drug, Comparative table, General testing methods

はじめに

「生薬・薬用植物に関する国際調和のための西太平洋地区討論会」(FHH : Western Pacific Regional Forum for the Harmonization of Herbal Medicines) は，西太平洋地区の６カ国７地域（日本，中国，韓国，ベトナム，シンガポール，オーストラリア，香港）における生薬・薬用植物の安全性，有効性及び品質に関する技術的な記録とコンセンサスを提供することを目的としている．日本はその下部組織である Sub-Committee 1 会議 (Nomenclature and Standardization) を主催し，5つの専門部会 (Expert working group, EWG 1-5) を設立した．これらの EWG では，それぞれの分野における各国薬局方の比較表を作成することが課題事項として議決された．著者の一人である川原は EWG 2 (Testing Methods used in Monographs) の責任者であり，著者らは前報及び前々報において日本，中国，韓国，ベトナム４カ国の薬局方に収載された生薬の試験法及び規格値に関する比較表を作成するとともに，確認試験法における TLC 条件並びに定量法（成分含量測定法）における分析条件の詳細な比較表を作成し，それらの共通点，相違点の詳細について報告した[1,2]．

本報ではさらに引き続き，EWG 5 (Information on General Tests) の課題事項である日本，中国，韓国，ベトナム 4 カ国の薬局方に収載された生薬関連一般試験法の詳細について比較表を作成し，比較検討を行った．

なお，本報では各国の局方に収載されている一般試験法の中で，特に生薬を規定するために必要な試験法を「生薬関連一般試験法」として比較検討の対象とした．

方法

本研究では FHH 参加国及び地域のうち，独自の薬局方を保有している日本，中国，韓国，ベトナムの 4 カ国の生薬に関する一般試験法を精査し，各国の試験法（試料の採取，異物，分析用試料の作成，乾燥減量，灰分，酸不溶性灰分，エキス含量，精油含量，鏡検，重金属，ヒ素等）の各項目について試験法の設定の有無，試験方法について比較表を作成した．本比較表の作成に使用した各国薬局方を Table 1 に示す．

Table 1 Pharmacopoeias Used in Preparation of Comparative Table

日本薬局方 (JP)	第 14 改正日本語版，英語版 第 14 改正第一追補日本語版，英語版 第 14 改正第二追補日本語版，英語版 第 15 改正日本語版 日本薬局方外生薬規格 1989 年日本語版
中華人民共和国薬典 (CP)	2000 年版中国語版，英語版 2005 年版中国語版
大韓民国薬局方 (KP)	1997 年第 7 版英語版 2002 年第 8 版韓国語版，英語版
ベトナム薬局方 (VP)	2002 年第 3 版ベトナム語版 2005 年第 3 版英語版

結果

作成した比較表を Table 2. に示す．この結果，日本薬局方（JP）と大韓民国薬局方（KP）の一般試験法における試験項目，記載内容は，重金属試験法において JP では第 1 法ー第 4 法が記載されているのに対し，KP では第 5 法まで記載されている以外はほぼ同一であった．他方，中華人民共和国薬典 (CP) とベトナム薬局方 (VP) の一般試験法における試験項目，記載内容はほぼ同一であった．また，CP 及び VP において，分析用試料の作成の項目は認められないが，生薬の品質評価法，生薬の調製・加工，タンニン量及びシネオール量についての項目が収載されていた．エキス含量の項においては，JP 及び KP では希エタノールエキス，水製エキス及びエーテルエキス定量法が収載されているのに対し，CP 及び VP ではエーテルエキス定量法は収載されていなかった．さらに VP では硫酸処理灰分及び水不溶性灰分の項目設定がなされていた．

一方，鏡検に関して JP 及び KP では装置，鏡検用プレパラートの作成及び性状の項の各要素の観察の各小項目で比較的簡単に記載されているのに対し，CP 及び VP では崩壊した組織のスライド作成法，花粉や胞子のスライド作成法，細胞や細胞内容物の測定法，細胞壁及び細胞内容物の観察方法等，詳細な記載が認められた．

考察及び結論

今回の比較表作成より，東アジア地区 4 カ国の薬局方における生薬関連一般試験法の共通点，相違点が明らかとなった．特に JP と KP，CP と VP については一般試験法における試験項目，記載内容がほぼ同一であり，これは局方英語版作成に当り，KP は JP を，CP は VP をそれぞれ参考にして作成されているためこのような結果が得られたものと推測された．また鏡検に関して CP 及び VP では小項目ごとに具体的かつ詳細な記載がなされており，鏡検による生薬の鑑別が現在においても重要視されていることが示唆された．さらに生薬の品質評価法，生薬の調製・加工等，CP 及び VP にのみに収載されている項目も多く認められており，興味深い．

前報に引き続き，各国の薬局方について共通点と相違点を認識することを目的として，特に日本，中国，韓国，ベトナム 4 カ国の薬局方に収載された生薬関連一般試験法について比較表の作成を試みた．この結果，試料の採取，異物，乾燥減量，灰分，酸不溶性灰分，エキス含量，精油含量，重金属，ヒ素等の設定及びその内容に関して共通点が多く認められたが，CP 及び VP では鏡検の方法，特にスライド作成法や観察方法の詳細な記載及びその内容に関して，相違点が認められた．

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Table 2. Comparative Table on General Testing Methods for Crude Drugs in JP, KP, CP and VP

JP	KP	CP	VP
Sampling	**Sampling**	**Sampling of Crude Drugs**	**SAMPLING OF CRUDE DRUGS**
Unless Otherwise specified, sample should be taken by the following methods. If necessary, preserve the samples in tight containers. (1) When crude drugs to be sampled are small-sized, cut or powdered, 50 to 250 g of sample should be taken after mixing thoroughly. (2) When crude drugs to be sampled are large-sized, 250 to 500 g of sample should be taken after mixing thoroughly. (3) When the mass of each single piece of the crude drugs is not less than 100 g, not less than 5 pieces should be taken for a sample, or not less than 500 g of the sample should be taken after cutting to a suitable size and mixing thoroughly.	Unless Otherwise specified, sample should be taken by the following methods. If necessary, preserve the samples in tight containers. (1) When crude drugs to be sampled are small-sized, cut or powdered, 50 to 250 g of sample should be taken after mixing thoroughly. (2) When crude drugs to be sampled are large-sized, 250 to 500 g of sample should be taken after mixing thoroughly. (3) When the mass of each single piece of the crude drugs is not less than 100 g, not less than 5 pieces should be taken for a sample, or not less than 500 g of the sample should be taken after cutting to a suitable size and mixing thoroughly.	Sampling of Crude Drugs refers to the method used to sort the crude drugs for examination. The validity of sampling affects directly the precision and accuracy of the examination. The procedure for sampling should be followed in details. 1. Examine the confirmation of the name, source of material, specification and package form of the cargo before sampling. Examine the intactness cleanliness of package and contamination of moulds and foreign matter, make notes in detail. The abnormal packages should be examined separately. 2. The general requirements for sampling of crude drugs in a consignment are as follows: Total number of package less than 100, 5 packages are sampled; 100-1000 packages, 5% are sampled; when the total number of package is more than 1000, 1% of the part in excess of 1000 packages are sampled; when the total number of package is more than 5, the packages are sampled one by one. Precious crude drugs are sampled one by one, regardless of the number of packages. 3. If the material is in crushed or powdered form or in pieces of less than 1 cm in size, at least 2-3 portions of sample are taken by suitable means from different parts in each package. If the number of package is small, the quantity of samples taken should be not less than 3 times of the quantity required for the testing. If the number of package is large, the quantity of samples taken is defined as follows: Common drugs: 100-500 g Powdered drugs: 25 g Precious drugs: 5-10 g As for the drugs of large size, representative samples can be taken from different parts of a package (10 cm in depth below the surface for large packages). 4. Mix the samples thoroughly, if the size of the drug is small, take an average sample by quartering, until sufficient quantity of sample is obtained for testing and retention. In the case of large size drugs, the average sample can be obtained with any proper method. The quantity or average sample taken should be not less than 3 times of that required for the testing, using one third for analysis, another one third for verification and the remaining as a retention which should be kept at least for one year.	Sampling of clude drugs refers to the method used to sort the crude drugs for examination. The representativeness of samples affects directly the prescision and accuracy of the examination. Attention should be paied to the following points while sampling: a) Valify the name, source of the material, specifications and forms of packages before sampling. Examine the intactness, cleanliness of the packagem the contamination of modules and foreign matter, make notes in details. Abnormal packages should be eamined more carefully. b) The general requirements for sampling of crude drugs are as follows: For a number of packages: less tha 5, every package is sampled; less than 100, 5 packages are sampled; from 100 to 1000, 5% of packages are sampled; over 1000, 50 packages and 1% of the number in excess of 1000 packages are sampled. For precious crude drugs every package is sampled, regardless of the number of packages. c) If the material is in scraps or powder form or in pieces of less than 1 cm in size, at least 2-3 portions of sample are taken by suitable means from different places in each package. If the number of packages is small, the amount of sample taken should be not less than 3 times the quantity required for testing. If the number of packages is large, the amount of sample taken is as follows: Common drugs: 100-500 g Powdered drugs: 25 g Precious drugs: 5-10 g (unless otherwis specified) For the drugs in large size, a representative sample can be taken from different places of a package (at 10 cm in depth below the surface for package). d) Mix the samples taken as required for the test sample. If the sample size of drug is small, take an aberage sample by quartering method as follows: Spread the samples (after mixing throughly) in a square, then divide the sample into 4 equal parts by diagonals; take two opposite parts and mix again. With the mixture obtained, repeat the quartering in the wame way until a sufficient amount of sample is obtained for testing and retention. In the case of large size drugs, the avarage samples can be obtained with any appropriate methods. The amount of an average sample should not less than 3 times of that required for testing, using one third for analysis, another for verification and the remaining as retained sample which should be kept at least for one year.
Foreign matter	**Foreign matter**	**Determination of Foreign Matter**	**DETERMINATION OF FOREIGN MATTER IN CRUDE DRUGS**
Unless otherwise specified, weigh 25 to 500 g of the sample, spread out in a thin layer, and separate the foreign matter by inspecting with the naked eye or with the use of a magnifying glass of 10 magnifications. Weigh, and determine the percentage of foreign matter.	Unless otherwise specified, weigh 25 to 500 g of the sample, spread out in a thin layer, and separate the foreign matter by inspecting with the naked eye or with the use of a magnifying glass of 10 magnifications. Weigh, and determine the percentage of foreign matter.	Foreign mater consists of any or all of the following: 1. The biological origin of which is the same as that specified in the monograph concerned but the appearance or botanical parts is different. 2. The biological origin of which differs from that specified in the monograph concerned. 3. Foreign mineral matters such as stones, sand, lumps of soil. **Method** (1) Weight a quantity of the drug as specified in the monograph and spread out in a thin layer. Detect the foreign matter by inspection with naked eye or with a lens (5-10 X), or by the use of a suitable sieve, If necessary, to separate the foreign matter. (2) Weight separately each kind of foreign matter and calculate the percentage content.	Foreign matter in herbal drugs consists of any or all of the following: Foreign mineral mannter such as stons, sand, lumps of soil. Other herbs and other parts of the plant that are not specified as crude drugs. Remains of insects. **Method:** Weigh a quantity of the crude drug as specified in the monograph and spread out in a thin layer. Detect the foreign matter by inspection with naked eye or with a lens or by use of a suitable sieve, if necessary, to separate the foreign matter. Weigh the foreign matter and calculate the percentage, using the expression: $X\% = a/p \times 100$ where: a: Mass of foreign matter (g). p: Mass of test sample being examined (g)
Preparation of the test sample for analysis	**Preparation of the test sample for analysis**		
Preparations are to be made by mixing the sample well. Powdered drugs should be used as they are, and in the case of unpowdered drugs, unless otherwise specified, grind the sample into powder. If the sample cannot be ground into powder, reduce it as finely as possible, spread it out in a thin layer, and withdraw a typical portion for analysis. If necessary, preserve the test sample in a tight container.	Preparations are to be made by mixing the sample well. Powdered drugs should be used as they are, and in the case of unpowdered drugs, unless otherwise specified, grind the sample into powder. If the sample cannot be ground into powder, reduce it as finely as possible, spread it out in a thin layer, and withdraw a typical portion for analysis. If necessary, preserve the test sample in a tight container.		

JP	KP	CP	VP
Loss on drying	**Loss on drying**	**Determination of Loss on Drying**	DETERMINAITON OF LOSS ON DRYING
Unless otherwise specified, transfer 2 to 6 g of the test sample for analysis to a tared weighing bottle, and weigh accurately. Dry at 105℃ for 5 hours, allow to cool in a desiccator (silica gel), and weigh accurately. Continue the drying at 105℃, and weigh accurately at 1-hour intervals. When the mass of the sample becomes constant, the loss of mass represents the percentage of loss on drying (%). When the period of time for drying is specified, weigh accurately after drying for the period of time specified, and determine the loss on drying (%).	Unless otherwise specified, transfer 2 to 6 g of the test sample for analysis to a tared weighing bottle, and weigh accurately. Dry at 105℃ for 5 hours, allow to cool in a desiccator (silica gel), and weigh accurately. Continue the drying at 105℃, and weigh accurately at 1-hour intervals. When the mass of the sample becomes constant, the loss of mass represents the percentage of loss on drying (%). When the period of time for drying is specified, weigh accurately after drying for the period of time specified, and determine the loss on drying (%).	Mix the substance being examined thoroughly, if it is in the form of large crystals, reduce them to a size of about 2 mm by crushing. Place 1 g or the amount specified under individual monographs of the substance being examined in a tarred, shallow weighing bottle, previously dried to constant weight under the conditions specified in individual monographs, unless otherwise directed. The substance being examined should be evenly distributed to form a layer of not more than 5 mm in thickness, or not more than 10 mm in the case of bulky material. When the loaded bottle is placed in the chamber of desiccator, remove the stopper and put in beside the bottle, or leave it on the bottle in half open position. Upon the opening of the drying chamber or desiccator, the bottle should be closed promptly. If the substance is dried by heating, allow it to cool to room temperature in a desiccator before weighing. If the substance melts temperature than the specified drying temperature, maintain the bottle with its content below the melting temperature until most of water is removed, then dry it under the specified conditions. If a vacuum desiccator or constant temperature vacuum desiccator is to be used, a pressure of 2.67 kPa (20 mm Hg) or less should be maintained unless otherwise directed. The desiccants used in a desiccator are usually anhydrous calcium chloride, silica gel or phosphorus pentoxide. Phosphorus pentoxide is often used in a constant temperature vacuum desiccataor. The desiccants should be kept fully effective.	Loss on drying is the loss of mass, expressed as percentage (m/m), of the test sample being dried under conditions specified in the individual monograph. The loss of mass after during represents the loss of the absorbed water, one part or the whole water of crystallisation and other volatile substances present in the sample being examined. The determination of loss of drying should not affect basic physico-chemical properties of the substance being examined; so in each individual monograph the drying method is specified and selected among the following methods: Method 1: drying in an oven under atmospheric pressure Method 2: drying under reduced pressure Method 3: drying in a desiccator over a strong desiccant such as concentrated sulfuric aciem phosphorus pent oxide, anhydrous calcium chloride, silica gel, etc.. For each method, detailed specific conditions are prescribed in the individual monograph for the substance being examined. When prescribed in the monograph: "Not exceed 1% (1 g, 105℃, 4 hours)", it means method 1 used: one gram of the sample being examined is dried in an oven at 105℃ for 4 hours and the loss mass should not exceed 10 mg. "Not exceed 0.5% (1 g, phosphorous pent oxide, 24 hours)" means method 2 is used: one gram of the substance being examined is dried in a drying device for 24 hours under reduced pressure (2 kPa) with the presence of phosphorus pent oxide as desiccant and the loss of mass should not exceed 5mg. Not exceed 0.2% (1 g, silica gel, 24 hours) means method 3 is used: one gram of the substance being examined is dried in a drying device for 24 hours under reduced pressure (2kPa) with the presence of desiccant silica gel and the loss of mass should not exceed 2 mg. When the drying time is not specified in the monograph, the sample should be dried to constant weight (this means two consecutive weighings should not differ by more than 0.5 milligram, the second weighing being made after an additional period of drying (1 hour in an oven or 6 hours in a desiccator). **Method** The container used in weightings ca be a Petri dish or a weighing bottle which is dried for 30 minutes following the method and conditions specified in the monograph; and then the container is weighed to determine its mass. Place immediately a quantity of the substance being examined (the quantity prescribed in the monograph, with a deviation of ± 10%) in the container and weigh it accurately. Unless otherwise stated in the monograph, the sample being examined is evenly spread to form a layer of a thickness not more than 5 mm. If the sample being examined contains large pieces, it should be quickly ground to obtain particles of size under 2 mm before weighing. Dry the sample under the conditions prescribed in the monograph using the same drying device as that has been used for drying the container. When drying in an oven, the temperature in the oven used should not differ by more than ±2℃ from the specified temperature. After drying, the sample is allowed to cool in a desiccator, over silica gel as desiccant, down to room temperature, then weighed immediately. If the substance being examined melts at a temperature lower than the specified temperature, it should be kept for 1 to 2 hours at a temperature 5℃ to 10℃ lower than its melting point before heating up to the described temperature. For sample in the form of capsules or draggers, the shells should be discarded and the sample being examined is quickly ground to form a powder of 2 mm particles, and amount of powder equivalent to at least 4 draggers or capsules is taken for testing. For materia medica, unless otherwise prescribed, method 1 is applied. The sample is ground into pieces not larger than 3 mm in diameter, then an amount of 2 g to 5 g is taken and evenly spread to form a layer of a thickness not more than 5 mm (or not more than 10 mm when the sample is porous material). The sample is dried as described in the monograph at the specified temperature for the prescribed period of time.

JP	KP	CP	VP
Total ash	**Total ash**	**Determination of Ash (Total ash)**	**DETERMINATION OF ASH**
Ignite previously a crucible of platinum, quartz or porcelain between 500℃ and 550℃ for 1 hour. Cool, and weigh accurately the crucible. Unless otherwise specified, weigh accurately 2 to 4 g of the test sample for analysis in this crucible, take off the lid or keep it open a little if necessary, heat the crucible at a low temperature at first, then gradually heat to a temperature between 500℃ and 550℃, ignite to incinerate the residue for more than 4 hours until no carbonized substance remains in the ash, cool, and weigh accurately the ash. Incinerate repeatedly to constant mass, cool, weigh accurately, and determine the amount (%) of total ash. If a carbonized substance remains andconstant mass cannot be obtained in the above-mentioned method, extract the charred mass with hot water, collect the insoluble residue on filter paper for assay, and incinerate the residue and filter paper until no carbonized substance remain in the ash. Then add the filtrate, evaporate it to dryness, and incinerate. Cool, weigh accurately, and determine the mass (%) of the total ash. If a carbon-free ash cannot be obtained even in this way, moisten the ash with a small amount of ethanol (95), break up the ash with a glass rod, wash the rod with a small amount of ethanol (95), evaporate carefully, and determine the mass of the total ash as described above. A desiccator (silica gel) is used for cooling.	Ignite previously a crucible of platinum, quartz or porcelain between 500℃ and 550℃ for 1 hour. Cool, and weigh accurately the crucible. Unless otherwise specified, weigh accurately 2 to 4 g of the test sample for analysis in this crucible, take off the lid or keep it open a little if necessary, heat the crucible at a low temperature at first, then gradually heat to a temperature between 500℃ and 550℃, ignite to incinerate the residue for more than 4 hours until no carbonized substance remains in the ash, cool, and weigh accurately the ash. Incinerate repeatedly to constant mass, cool, weigh accurately, and determine the amount (%) of total ash. If a carbonized substance remains andconstant mass cannot be obtained in the above-mentioned method, extract the charred mass with hot water, collect the insoluble residue on filter paper for assay, and incinerate the residue and filter paper until no carbonized substance remain in the ash. Then add the filtrate, evaporate it to dryness, and incinerate. Cool, weigh accurately, and determine the mass (%) of the total ash. If a carbon-free ash cannot be obtained even in this way, moisten the ash with a small amount of ethanol (95), break up the ash with a glass rod, wash the rod with a small amount of ethanol (95), evaporate carefully, and determine the mass of the total ash as described above. A desiccator (silica gel) is used for cooling.	Pulverize the material being examine, pass through No.2 sieve, mix well. Place 2—3 g (3—5 g for the determination of acid-insoluble ash) of powdered drug in a tarred crucible, weigh accurately (to nearest 0.01 g), ignite slowly till the sample is completely carbonized, keep it from burning with care, raise the temperature gradually to 500—600℃, incinerate to constant weight and the ash is carbon-free. Calculate the percentage of ash with reference to the air-dried drug. If carbon-free ash cannot be obtained in this way, cool the crucible and moisten the residue with hot water or 2 ml of 10% ammonium nitrate solution. Evaporate to dryness on a water bath, ignite the residue as above until carbonfree ash is obtained.	Use method 1 unless otherwise directed in the monograph. **Method 1:** For vegetable drugs: Incinerate 2 to 3 of the ground drug in a tarred platinum or silica crucible at a temperature not exceed 450℃ until free from carbon, cool and weigh. If a carbon-free ash cannot be obtained in this way, exhaust the charred mass with hot water, stair with glass rod, filter through an ashless filter paper. Wash the glass rod and filter paper, combine the washings and the filtrate. Place the filter paper and the residue in a crucible and ignite until a white or almost white ash obtained. Add the filtrate to residue in the crucible, evaporate to dryness, and ignite at a temperature not exceeding 450℃ to constant mass. Calculate the percentage of ash with reference to air dried drug. For other substances: Carry out the above method using 1g, unless otherwise directed in the monograph. Calculate the percentage of ash. **Method 2:** Heat a porcelain or platinum crucible to red heat for 30 minutes, allow to cool in a desiccator and weigh. Unless otherwise specified in the monograph, evenly distribute 1 g of the substance being examined in the crucible, dry at 100℃ to 150℃ for 1 hour and ignite to constant weight in a muffle furnace at 575℃ to 625℃. Allow the crucible to cool in a desiccator and weigh after each ignition. Flames should not be produced at any time during the procedure. If after prolonged ignition a carbon-free ash cannot be obtained, take up with hot water, filter through an ashless filter paper and ignite again the residue and the filter paper. Combine the filtrate with the ash, carefully evaporate to dryness and ignite to constant weight. Calculate the percentage of ash with reference to the air-dried drug.
Acid-insoluble ash	**Acid-insoluble ash**	**Determination of Ash (Acid-insoluble ash)**	**DETERMINATION OF ACID INSOLUBLE ASH**
Add carefully 25 mL of dilute hydrochloric acid to the total ash, boil gently for 5 minutes, collect the insoluble matter on filter paper for assay, and wash thoroughly with hot water. Dry the residue together with the filter paper, and ignite to incinerate in a tared crucible of platinum, quartz or porcelain for 3 hours. Cool in a desiccator (silica gel), weigh, and determine the amount (%) of acid-insoluble ash. When the amount determined exceeds the limit specified, incinerate repeatedly to constant mass.	Add carefully 25 mL of dilute hydrochloric acid to the total ash, boil gently for 5 minutes, collect the insoluble matter on filter paper for assay, and wash thoroughly with hot water. Dry the residue together with the filter paper, and ignite to incinerate in a tared crucible of platinum, quartz or porcelain for 3 hours. Cool in a desiccator (silica gel), weigh, and determine the amount (%) of acid-insoluble ash. When the amount determined exceeds the limit specified, incinerate repeatedly to constant mass.	Place the obtained in the determination of total ash in crucible, add 10 ml of dilute hydrochloric acid with great care, cover with a watch glass, heat on a water bath for 10 minutes. Rinse the watch glass, with 5 ml of hot water and add the rinsings to the crucible, filter with an ashless filter paper, transfer the residue to the filter paper with water, wash till the filtrate yields no reactions of chlorides. Transfer the filter paper together with the residue to the original crucible, dry and ignite to constant weight. Calculate the percentage of acid-insoluble ash with reference to the air-dried drug.	Use method 1 unless otherwise directed in the monograph. **Method 1:** Boil the ash for 5 minutes with 25 ml of 2 M hydrochloric acid *R* , filter, collect the insoluble matter in a previously weighed sintered-glass crucible or on an ashless filter paper, wash with hot water and ignite. Calculate the percentage of acid-insoluble ash with reference to the air-dried drug. **Method 2:** Place the ash or the sulphated ash, as specified in the monograph, in a crucible, add 15 ml of water and 10 ml of hydrochloric acid *R* , cover with a watch glass, boil gently for 10 minutes and allow to cool. Wash the watch glass with 5ml of hot water, collect the washings in the crucible. Collect the insoluble matter in a previously weighed sintered-glass funnel or on ashless filter paper, wash with hot water until the filtrate is neutral. Dry, ignite to dull redness, allow to cool in a desiccator and weigh. Repeat until the difference between tow successive weightings is not more than 1 mg. Calculate the percentage of acid-insoluble ash with reference to air-dried drug.
			DETERMINATION OF SULPHATED ASH
			Use method 1 unless otherwise directed in the monograph. **Method 1:** Heat a porcelain or platinum crucible to redness for 10 minutes, allow to cool in a desiccator and weigh. Unless otherwise specified in the monograph, place 1 g of the substance being examined in the crucible, moisten with sulphuric acid *R* , ignite gently, again moisten with sulphuric acid and ignite at about 800℃. Cool, weigh again, ignite for 15 minutes and cool, weigh again. Repeat this procedure until tow successive weightings do not differ by more than 0.5 mg. If the residue is reserved for the test of heavy metals, ignition should be carried out at 500℃ to 600℃. **Method 2:** Heat a porcelain or platinum crucible to redness for 10 minutes, allow to cool in a desiccator and weigh. Place a suitable quantity of the substance being examined in the crucible, add 2 ml of 1 M sulphuric acid *R* and heat, first on a water bath , then cautiously over a flame and then progressively to about 600℃. Continue incineration until all black particles have disappeared and then allow to cool. Add a few drops of 1 M sulphuric acid *R* , incinerate as before and allow to cool. Add a few

JP	KP	CP	VP
			DETERMINATION OF SULPHATED ASH
			drops of a 15.8 % m/v solution of ammonium carbonate R , evaporate to dryness. Incinerate carefully, allow to cool, weigh. Incinerate for 15 minutes and repeat this procedure to constant mass.
			DETERMINATION OF WATER-SOLUBLE ASH
			Boil the ash (Appendix 7.6) for 5 minutes with 25 ml of water. Collect the insoluble matter in a previously weighed sintered-glass funnel or filter crucible or on an ashless filter paper, wash with hot water and ignite for 15 minutes at a temperature not exceeding 450°C. Allow to cool in a desiccator and weigh to determine the quantity of water insoluble residue. The difference between the weight of ash add the weight of water-insoluble residue is the mass of water-soluble ash. Calculate the percentage of water-soluble ash with reference to the air-dried drug.
Extract content	**Extract content**	**Determination of Extractives**	DETERMINATION OF EXTRACTIVES IN HERBAL DRUGS
The test for the extract content in crude is performed as directed in the following methods: (1) Dilute ethanol-soluble extract-Unless otherwise specified, weigh accurately about 2.3 g of the sample for analysis, extract with 70 mL of dilute ethanol in a suitable flask with intermittent shaking for 5 hours, and allow to stand for 16 to 20 hours. Filter, and wash flask and residue with small portions of dilute ethanol until the filtrate measures 100 mL. Evaporate a 50 mL aliquot of filtrate to dryness, dry at 105℃ for 4 hours, and cool in a desiccator (silica gel). Weigh accurately the amount, multiply it by 2, and determine the amount of dilute ethanol-soluble extract. Calculate the extract content (%) with respect to the amount of the sample on the dried basis, obtained under the loss on drying. (2) Water-soluble extract-Proceed as directed in (1), using water instead of dilute ethanol, weigh accurately the amount, multiply by 2, and determine the amount of water-soluble extract. Calculate the extract content (%) with respect to amount of the sample on the dried basis, obtained under the loss on drying. (3) Diethyl ether-soluble extract-Unless otherwise specified, dry the sample for analysis in a desiccator (silica gel) for 48 hours, weigh accurately about 2 g of it, and place in a suitable flask. Add 70 mL of diethyl ether, attach a reflux condenser to the flask, and boil gently on a water bath for 4 hours. Cool, filter, and wash the flask and the residue with small portions of diethyl ether until the filtrate measures 100 mL. Evaporate a 50 mL aliquot of the filtrate to dryness on a water bath, dry in a desiccator (silica gel) for 24 hours, weigh accurately the amount, multiply it by 2, determine the amount of diethyl ether-soluble extract, and calculate the extract content (%).	The test for the extract content in crude is performed as directed in the following methods: (1) Dilute ethanol-soluble extract-Unless otherwise specified, weigh accurately about 2.3 g of the sample for analysis, extract with 70 mL of dilute ethanol in a suitable flask with intermittent shaking for 5 hours, and allow to stand for 16 to 20 hours. Filter, and wash flask and residue with small portions of dilute ethanol until the filtrate measures 100 mL. Evaporate a 50 mL aliquot of filtrate to dryness, dry at 105℃ for 4 hours, and cool in a desiccator (silica gel). Weigh accurately the amount, multiply it by 2, and determine the amount of dilute ethanol-soluble extract. Calculate the extract content (%) with respect to the amount of the sample on the dried basis, obtained under the loss on drying. (2) Water-soluble extract-Proceed as directed in (1), using water instead of dilute ethanol, weigh accurately the amount, multiply by 2, and determine the amount of water-soluble extract. Calculate the extract content (%) with respect to amount of the sample on the dried basis, obtained under the loss on drying. (3) Diethyl ether-soluble extract-Unless otherwise specified, dry the sample for analysis in a desiccator (silica gel) for 48 hours, weigh accurately about 2 g of it, and place in a suitable flask. Add 70 mL of diethyl ether, attach a reflux condenser to the flask, and boil gently on a water bath for 4 hours. Cool, filter, and wash the flask and the residue with small portions of diethyl ether until the filtrate measures 100 mL. Evaporate a 50 mL aliquot of the filtrate to dryness on a water bath, dry in a desiccator (silica gel) for 24 hours, weigh accurately the amount, multiply it by 2, determine the amount of diethyl ether-soluble extract, and calculate the extract content (%).	**Determination of Water-soluble Extractives** Pulverize the material being examined, pass through No.2 sieve, mix well. **Cold maceration method** Place 4 g of the powdered material, accurately weight (to the nearest 0.01 g), in a 250－300 ml stoppered conical flash, add accurately 100 ml of water, stopper well. Macerate the drug with shaking for 6 hours and allow to stand for 18 hours. Filter rapidly through a dry filter, transfer accurately 20 ml of filtrate to an evaporating dish, previously dried to constant weight, and evaporate to dryness on a water bath. Dry at 150℃ for 3 hours and allow to cool for 30 minutes in a desiccator. Weigh rapidly and accurately, unless specified otherwise in the monograph, calculate the percentage of water-soluble extractives on the dried basis. **Hot extraction method** Place 2－4 g of the powdered material, accurately weighted in a 100－250 ml stoppered conical flask, add a accurately 50－100 ml of water, stopper well and weigh, allow to stand for 1 hour. Boil gently under reflux for 1 hour. Allow to cool, take off the flask, stopper well and weigh, add water to restore its original weight, shake well and filter through a dry filter. Place 25 ml of the filter, accurately, in an evaporating dish, previously dried to constant weight, and evaporate to dryness on water bath. Dry at 105℃ for 3 hours and allow to cool for 30 minutes in a desiccator. Weigh rapidly accurately, unless specified otherwise in the monograph, calculate the percentage of water-soluble extractives on the dried basis. **Determination of Ethanol-soluble Extractives** Proceed as directed under determination of water-soluble extractive (hot extraction method should be heating on a water bath), using ethanol or methanol of a strength specified in individual monograph as the solvent instead of water.	**Determination of water-soluble extractives** **Cold maceration method:** Unless otherwise specified in the monograph, place about 4.000 g of the moderately coarse powdered material, accurately weighed, in a 250-300 ml stoppered conical flask. Add accurately 100.0 ml of water, close well, allow to macerate cold occasionally shaking for 6 hours, then allow to stand fro 18 hours. Filter through a dry filter into a suitable dry flask. Pipette 20 ml of the filtrate to a glass beaker, previously dried to constant mass, and evaporate to dryness in a water bath. Dry the residue at 105℃ for 3 hours and allow to cool for 30 minutes in a desiccator, weigh rapidly to determine the mass of the residue, calculate the percentage of water-soluble extractives with reference to the air-dried drug. **Hot extraction method:** Unless otherwise specified in the monograph, place about 2.000 g to 4.000 g of the moderately coarse powdered material, accurately weighed, in a 100 ml or 250 ml close conical flask. Add accurately 50.0 or 100.0 ml of water, close well and weigh, allow to stand for 1 hour, then heat under a reflux condenser in a water bath for 1hour, allow to cool, take off the flask, closes well and weigh, add water to restore its original mass, filter though a dry filter into a suitable dry flask. Pipette 25 ml of the filtrate to a glass beaker, previously dried to constant mass, and evaporate to dryness in a water bath. Dry the residue a 105℃ for 3 hours and allow to cool for 30 minutes in a desiccator, weigh rapidly to determine the mass of the residue, calculate the percentage of water-solbule extractives with reference to the air-dried drug. **Determination of alcohol-solble extractives** Process as directed under determination of water-soluble extractives using ethanol or methanol of strength specified in individual monograph as extraction solvent instead of water.
Essential oil content	**Essential oil content**	**Determination of Volatile Oil**	DETERMINATION OF VOLATILE OIL IN DRUGS
The test of essential oil content in crude drugs is performed as directed in the following method: Essential oil determination: Weigh the quantity of the test sample for analysis directed in the monograph in a 1-L hard glass-stoppered flask, and add from 5 to 10 times as much water as the drug. Set up apparatus for essential oil determination in the upper mouth of it, and heat the content of the flask in an oil bath between	The test of essential oil content in crude drugs is performed as directed in the following method: Essential oil determination: Weigh the quantity of the test sample for analysis directed in the monograph in a 1-L hard glass-stoppered flask, and add from 5 to 10 times as much water as the drug. Set up apparatus for essential oil determination in the upper mouth of it, and heat the content of the flask in an oil bath between	The drug being examined should be pulverized to pass through No.2 or No.3 sieves and then mixed well, unless otherwise specified. **Method 1** This method is used for determining volatile oil of which the relative density is less than 1.0. Weigh accurately to the nearest 0.01 g, a quantity of the substance being examined equivalent to 0.5－1.0 ml of volatile oil, into flask A. Add 300－500 ml of water and a few glass beads, shake and mix well. Connect flask A to volatile oil determination tube B and then connect B to reflux condenser C. Add water through the top of reflux condenser C until Heat the flask gently in an electric	The determination of volatile oil in drugs is carried out by steam distillation in the apparatus described in the Fig 9.2. The distillate is collected in a tube graduated into divisions of 0.05 ml and the aqueous phase is automatically recalculated into the distillation flask. The volume of volatile oil may be measured directly on the graduated tube or xylene may be used to take up the volatile oil to the graduated part of the tube (for the volatile oils the relative density of which is more than 1.0), and then total volume of the mixture of xylene and volatile oil is measured. The content of volatile oil is expressed as a percentage v/m.

JP	KP	CP	VP
Essential oil content	**Essential oil content**	**Determination of Volatile Oil**	**DETERMINATION OF VOLATILE OIL IN DRUGS**
130℃ and 150℃ to boiling. The graduated tube of the apparatus is to be previously filled with water to the standard line, and 2.0 mL of xylene is added to the graduated tube. Unless otherwise specified, continue boiling for 5 hours, allow to stand for some time, and open the stopper of the apparatus. Draw off the water slowly until the surface of the oil layer corresponds to the preparation line, and allow it to stand for than 1 hour at ordinary temperature. Then lower the surface of the oil layer to the zero line, and read the volume (mL) of the oil at ordinary temperature. Subtract the volume (mL) of xylene from the volume of the total oil.	130℃ and 150℃ to boiling. The graduated tube of the apparatus is to be previously filled with water to the standard line, and 2.0 mL of xylene is added to the graduated tube. Unless otherwise specified, continue boiling for 5 hours, allow to stand for some time, and open the stopper of the apparatus. Draw off the water slowly until the surface of the oil layer corresponds to the preparation line, and allow it to stand for than 1 hour at ordinary temperature. Then lower the surface of the oil layer to the zero line, and read the volume (mL) of the oil at ordinary temperature. Subtract the volume (mL) of xylene from the volume of the total oil.	heating jacket or by other suitable means until boiling begins-continue heating for about 5 hours, until the volume of oil does not increase. Stop heating, allow to stand for a few minutes, and open the stopcock at the lower part of B, run off the water layer slowly until the oil layer is 5 mm above the zero mark. Allow to stand for at least 1 hours, open the stopcock again, run off the remaining water layer carefully until the oily layer is just on the zero mark. Read the volume of oil in the graduated portion of the tube and calculate the content of volatile oil, expressed as percentage ml/g. **Method 2** This method is used for determination volatile oils of which the relative density is more than 1.0. Transfer 300 ml of water and a few glass beads to flask A. Connect flask A to volatile oil determination assembly B. Add water through the top of B until the graduated measuring tube of B is filled and water overflows to flask A. Add 1 ml of xylene with pipette and then connect the reflux condenser C to B. Heat the flask until boiling begins and continue the distillation at a rate that will keep the middle part of the condenser cold. Stop heating after 30 minutes, allow to stand for at least 15 minutes. Read the volume of xylene in the graduate portion of the tube. Carry out the procedure described under Method I. Beginning at the words "Weigh accurately to the nearest 0.01 g...". Subtract the volume of xylene previously from the volume of the oil layer, Subtract the volume of xylene previously from the volume of the oil layer, the remainder is taken to the content of volatile oil in the drug being examined, expressed as percentage ml/g.	***Determination of the volatile oils the relative density of which is less than 1.0.*** Weigh accurately the nearest 0.01 g, a quantity of the substance being examined passed through sieve No.2000 equivalent to 0.5-1.0 ml of volatile oil in to the distillation flask. Add 300-500 ml of water and a few pieces of porous earthenware. Connect the distillation flask to the still head A of the apparatus. Add water through the funnel N until it is at the level B. Heat the flask until ebullition begins and adjust the distillation rate 2 to 3 ml per minute unless otherwise prescribed. Determine the rate of distillation by lowering the level of distillation liquid by means of the three-way tap M until the meniscus is level with the lower mark I, J, closing the tap M and simultaneously starting a stop watch. When the level reaches the mark H, stop the watch and note the time. Open the tap M and continue the distillation for 5 hours, unless otherwise prescribed, until the volume of volatile oil stops to increase. Stop heating and after at least 10 minutes read the volume of the oil collector in the graduated tube. ***Determination of the volatile oils the relative density of which is more than 1.0.*** Connect the distillation flask containing about 300-500 ml of water and a far small pieces of porous earthenware, to the still head A of the apparatus. Add water though the funnel N untie it is at the level B. Introduce 1 ml of xylene *R* at K by means of a pipette (the tip of which is inserted the lower part of orifice K). Heat the flask until ebullition begins and adjust the distillation rate as the way described under the method for determination of the volatile oils relative density of which is less than 1.0. After 30 minutes discontinue heating and after at least a further 10 minutes read the volume of xylene *R* collected in the graduated tube. Introduce the specified quantity of drug passed the through No.2000 sieve equivalent to 0.5-1.0 ml of volatile oil into the distillation flask. Carry out the distillation at the distillation rate from 2 to 3 ml per minute for 5 hours, unless otherwise prescribed, until the volume of the volatile oil stops to increase. Stop heating and after at least 10 minutes read the volume of the mixture of xylene *R* and volatile oil. Subtract the volume of xylene R previously observed from the volume of the oily layer. The difference in volume and the quantity of drug are taken to be the content of volatile oil in the drug being examined.
Microscopic examination	**Microscopic examination**	**Microscopical Identification for Crude Drugs and Patent Medicines**	**MICROSCOPICAL IDENTIFICATION FOR CRUDE DRUGS AND PATENT MEDICINES**
(1) Apparatus Use an optical microscope with objective of 10 and 40 magnifications, and an ocular of 10 magnifications. (2) Preparation for microscopic examination (i) Section: To a section an a slide glass add 1 to 2 drops of a mounting agent, and put a cover glass in it, taking precaution against inclusion of bubbles. Usually use a section 10 to 20 mm in thickness. (ii) Powder: Place about 0.1 g of powdered sample in a watch glass containing 2 to 3 drops of a swelling agent, stir well with a small rod preventing inclusion of bubbles, and allow to stand for more than 10 minutes to swell the sample. Smear, using a small glass rod, the slide glass with a small amount of the swollen sample, add 1 drop of the mounting agent, and put a cover glass on it so that the tissue sections soread evenly without overlapping each other, taking precaution against inclusion of bubbles. Unless otherwise specified, use a mixture of glycerin and water (1:1) as mounting agent and swelling agent. (3) Observation of components in the description In each monograph, description is usually given of the outer portion and the inner portion of section in this order, followed by a specification of cell contents. Observation should be made in	(1) Apparatus Use an optical microscope with objective of 10 and 40 magnifications, and an ocular of 10 magnifications. (2) Preparation for microscopic examination (i) Section: To a section an a slide glass add 1 to 2 drops of a mounting agent, and put a cover glass in it, taking precaution against inclusion of bubbles. Usually use a section 10 to 20 mm in thickness. (ii) Powder: Place about 0.1 g of powdered sample in a watch glass containing 2 to 3 drops of a swelling agent, stir well with a small rod preventing inclusion of bubbles, and allow to stand for more than 10 minutes to swell the sample. Smear, using a small glass rod, the slide glass with a small amount of the swollen sample, add 1 drop of the mounting agent, and put a cover glass on it so that the tissue sections soread evenly without overlapping each other, taking precaution against inclusion of bubbles. Unless otherwise specified, use a mixture of glycerin and water (1:1) as mounting agent and swelling agent. (3) Observation of components in the description In each monograph, description is usually given of the outer portion and the inner portion of section in this order, followed by a specification of cell contents. Observation should be made in	Microscopical identification is method with the application of the microscope to identify the characters of tissues, cells or cell contents in sections, powders disintegrated tissues or surface slides of crude drugs and patent medicines. Representative to meet the requirements of identifications for each drugs. The slides of patent medicines are made after appropriate treatment with reference to their different dosage forms. **1. Transverse or Longitudinal Sections** Select a suitable part of the drug, cut into sections of 10—20mm in thickness with a razor blade or using sliding microtome after softened. Examine after treated with glycerolacetic acid TS, choral hydrate TS or other test solutions. Material may be embedded in hard paraffin before cutting if necessary. **2. Slides of Powder** Spread a small quantify of the powder on a slide, and examine after treated with glycerol-acetic acid TS, chloral hydrate TS, or other suitable test solutions. **3. Slides of Surface** After moistening and softening the materials, cut a part or tear its epidermis. add suitable test solutions and examine. **4. Slides of Disintegrated Tissue** Potassium hydroxide method can be used parenchyma makes most part of the material or the material with few or scattered woody tissues; chromic-nitric acids method or potassium chlorate method can be used if the material is hard, with the presence of more woody tissues or the woody grouped to lager bundles. The material should be cut into small strips or pieces of about 2 mm wide or thick before being disintegrated. (1) **Potassium Hydroxide Method** (2) **Chromic-Nitric Acids Method**	Microscopical identification is a method using a microscope to identify the characters of tissues, cells or cell contents in sections, powders, disintegrated tissues or surface slides of crude drugs and patent medicines. Representative samples are chosen to be identified and slides are prepared to meet the requirements of identification for each drug. The slide of patent medicines are after appropriate treatment with reference ton their different dosage forms. **Transverse of longitudinal sections** Select a suitable part of the drug having enough required botanical characteristics as specified below: **Stems and small roots:** Take a piece with a full sartorial trance verse section. **Stems, big roots:** Take a piece with a spectral transverse section (showing from the epidermis to the centre). **Stem bark:** Take a piece with a rectangular transverse section (showing from cork to xylem). **Leaves:** Take a piece with central vein and part of the lobes on both of its side. **Flowers:** Take the epiderma or cut transversely every part of the flower. **Small fruits and seeds:** Take the whole fruit or seed. **Big fruits and seeds:** Take a part of fruit or seed so that a section of which shows all botanical characteristics. Cut into thin sections with razor bale or using sliding microtome after being softened. Material may be embedded in herd paraffin before cutting if necessary. The section is examined immediately under a microscope unless otherwise specified or after being treated by the following ways: Macerate the section in 5% solution of chloramines *TR* until it is white, thoroughly wash with water.

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Microscopic examination	**Microscopic examination**	**Microscopical Identification for Crude Drugs and Patent Medicines**	**MICROSCOPICAL IDENTIFICATION FOR CRUDE DRUGS AND PATENT MEDICINES**
the same order. In the case of a powdered sample, description is given of a characteristic component or a matter present in large amount, existing matter, and cell contents in this order. Observation should be made in the same order.	the same order. In the case of a powdered sample, description is given of a characteristic component or a matter present in large amount, existing matter, and cell contents in this order. Observation should be made in the same order.	(3) **Potassium Chlorate Method** **5. Slides of Pollen and Spore** Grind Pollens, anthers (or small flowers) or sori (soften the dry material in glacial acetic acid) with a glass rod and filter into a centrifugal tube, centrifuge. To the precipitate add 1－3 ml of a freshly prepared mixture of acetic anhydride-sulfuric acid (9:1), heat on a water bath for 2－3 minutes, centrifuge. Wash the precipitate with 2 quantities of water, add 3－4 drops of 50% glycerin and 1% phenol, mount in fuchsin-glycerin gelatin and examine. Chloral hydrate TS may also be used as mountant for the examination. **6. Measurements of Cells and Cell Contents** To measure the sizes of cells and cell contents, etc, under the microscope, ocular micrometer can be used. Place the ocular micrometer in an first. Then calibrate with a stage micrometer. For the calibration, turn the eyepiece and move the stage micrometer to make the divisions on the two scales parallel and their left "O" lines coincide, then look for another coincide lines to the light. The value (mm) of 1 ocular micrometer division can be calculated on the basis of divisions of the two micrometer scales between the coincide lines. To measure the object , multiply the number of object-measuring divisions of ocular micrometer by the value (mm) of each division. Generally, it is carried out under an high power objective, but a low power objective would be more convenient to measurer the length of longer fibres and non-glandular hairs, etc. Record the maximal and minimal values (mm), permitting a few numerical values slightly high or lower than the values specified in pharmacopoeial requirement. **7. Detection of cell wall** (1) **Lignified cell wall** (2) **Suberized or Cuticularized Cell Wall** (3) **Cellulose Cell Wall** (4) **Siliceous Cell Wall** **8. Detection of Cell Content** (1) **Starch** (2) **Aleurone** (3) **Fatty oil, Volatile Oil or Resin** (4) **Inulin** (5) **Mucilage** (6) **Calcium Oxalte Crystals** (7) **Calcium Carbonate (stalactile)** (8) **Silicum**	Macerate the section in a 1 % solution of acetic acid *R* for 2 minutes, thoroughly wash with water. Macerate the section in green lod solution *R* or methylene blue for 1-5 s, quickly wash with ethanol (60%) *R* then with water. Macerate the section in carmine 40 solution *R* until it is coloured, wash with water. **Slides of powder** Spread a small quantity of the powder on a slide, and examine under a microscope after being treated with either water, glycerol, chloral hydrate *R* , or other suitable test solutions. **Slide of surface** After moistening and softening the materials (when necessary) cut a part or tear its epidermis, add suitable test solutions and examine. **Slide of disintegrated tissue** Potassium hydroxide method can be used if parenchyma makes most part of the material or the material with a few or scattered woody tissues; chromic-nitric acids method or potassium chlorate method can be used if the material is hard, with the presence of more woody tissues or the woody tissues propped into larger bundles. The material should be cut into small strips or pieces of about 2 mm wide or thick before being disintegrated. **a. Potassium hydroxide method** **b. Chromic-nitric acids method** **c. Potassium chlorate method** **Pollen and spore slides** Grind pollens, anthers, small flowers or sore (soften the dry material in glacial acetic acid *R*) with a glass rod and filter into a centrifugal tube, centrifuge. To the precipitate add 1－3 ml of a freshly prepared mixture of acetic anhydride-sulfuric acid (9:1), heat on a water bath for 2-3 minutes, centrifuge. Wash the precipitate with water twice, add 3-4 drops of glycerine gelatine and examine. Chloral hydrate *R* may also be used as mount ant for the examination. **Measurements of cells and cell contents** To measure the sizes of cells and cell contents, etc, under the microscope, ocular micrometer can be used. Place the ocular micrometer in an eyepiece first, then calibrate with a stage micrometer. For the calibration, turn the eyepiece and move the stage micrometer to make the divisions on the two scales parallel and their left "O" lines coincide, then look for another coincident lines to the right. **Measurements of cells and cell contents** The value (μm) of 1 ocular micrometer division can be calculated on the basis of divisions of the two micrometer scales between the coincident lines. To measure the object, multiply the number of object-measuring divisions of ocular micrometer by the value (μm) of each division. Generally, it is carried out under a high power objective, but a low power objective would be more convenient to measure the length of longer fibres and non-glandular hairs, etc. Record the maximal and minimal values (μm), permitting a few numerical values slightly higher or lower than the values specified in pharmacopoeial requirement. **Detection of cell wall** *Lignified cell wall* *Suberized or Cuticularized cell wall* *Cellulose cell wall* *Siliceous cell Wall* **Detection of cell contents** *Starch* *Aleuronic* *Fatty oil, volatile oil or resin* *Inulin* *Calcium oxalate crystals* *Calcium carbonate* **Silicum**

JP	KP	CP	VP
Microscopic examination	**Microscopic examination**	**Microscopical Identification for Crude Drugs and Patent Medicines**	**MICROSCOPICAL IDENTIFICATION FOR CRUDE DRUGS AND PATENT MEDICINES**
			Insoluble in sulphuric acid Identify the patent medicines made from pulverized drugs, slides for powders are prepared according to the method for powder slides mentioned above, for pills and tablets, ets..., grind 2 -3 pills (tablets) into fine powder, to a small quantity of the sample add drop wise the required test solutions, stair thoroughly to separate the stuck cells and tissues, then carry out the identification method for powder characters, slides of honeyed pills can be prepared directly by picking a little sample, or de-honeyed with hot water for the examination.
Arsenic Limit Test	**Arsenic Limit Test**	**Limit Test for Arsenic**	**LIMIT TESTS FOR IMPURITIES (ARSENIC)**
The Arsenic Limit Test is a limit test of arsenic contained in drugs. The limit is expressed in terms of arsenic (III) trioxide (As_2O_3). In each monograph, the permissible limit for arsenic (as As_2O_3) is described in terms of ppm in parentheses. **Preparation of the test solution** Unless otherwise specified, proceed in the following. **(1)** Method 1 Weigh the amount of the sample direct in the monograph, add 5 mL of water, dissolve by heating if necessary, and designate the solution as the test solution. **(2)** Method 2 Weigh the amount of the sample directed in the monograph, add 5 mL of sulfuric acid except in the cases that the samples are inorganic acids. Add 10 mL of sulfurous acid solution, transfer to a small beaker, and evaporate the mixture on a water bath until it is free from sulfurous acid is reduced to about 2 mL in volume. Dilute with water to make 5 mL, and designate it as the test solution. **(3)** Method 3 Weigh the amount of the sample directed in the monograph, and place it in a crucible of platinum, quartz or porcelain. Add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol(95)(1 in 50), ignite the ethanol, and heat gradually to incinerate. If carbonized material still remains by this procedure, moisten with a small quantity of nitric acid, and ignite again to incinerate. After cooling, add 3 mL of hydrochloric acid, heat on a water bath to dissolve the residue, and designate it as the test solution. **(4)** Method 4 Weigh the amount of the sample directed in the monograph, and place it in a crucibleof platinum, quartz or porcelain. Add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95)(1 in 10), burn the ethanol, heat gradually, and ignite to incinerate. If carbonized material still remains by this procedure, moisten with a small quantity of nitric acid, and ignite again to incinerate in the same manner. After cooling, add 3 mL of hydrochloric acid, heat on a water bath to dissolve the residue, and designate it as the test solution. **(5)** Method 5 Weigh the amount of the sample directed in the monograph, add 10 mL of *N ,N* -dimethylformamide, dissolve by heating if necessary, and designate the solution as the test solution.	The Arsenic Limit Test is a limit test of arsenic contained in drugs. The limit is expressed in terms of arsenic (III) trioxide (As_2O_3). In each monograph, the permissible limit for arsenic (as As_2O_3) is described in terms of ppm in parentheses. **Preparation of the test solution** Unless otherwise specified, proceed in the following. ***(1) Method 1*** Weigh the amount of the sample direct in the monograph, add 5 mL of water, dissolve by heating if necessary, and designate the solution as the test solution. ***(2) Method 2*** Weigh the amount of the sample directed in the monograph, add 5 mL of sulfuric acid except in the cases that the samples are inorganic acids. Add 10 mL of sulfurous acid solution, transfer to a small beaker, and evaporate the mixture on a water bath until it is free from sulfurous acid is reduced to about 2 mL in volume. Dilute with water to make 5 mL, and designate it as the test solution. ***(3) Method 3*** Weigh the amount of the sample directed in the monograph, and place it in a crucible of platinum, quartz or porcelain. Add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (1 → 50), ignite the ethanol, and heat gradually to incinerate. If carbonized material still remains by this procedure, moisten with a small quantity of nitric acid, and ignite again to incinerate. After cooling, add 3 mL of hydrochloric acid, heat on a water bath to dissolve the residue, and designate it as the test solution. ***(4) Method 4*** Weigh the amount of the sample directed in the monograph, and place it in a crucible of platinum, quartz or porcelain. Add 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (1 → 10), burn the ethanol, heat gradually, and ignite to incinerate. If carbonized material still remains by this procedure, moisten with a small quantity of nitric acid, and ignite again to incinerate in the same manner. After cooling, add 3 mL of hydrochloric acid, heat on a water bath to dissolve the residue, and designate it as the test solution. ***(5) Method 5*** Weigh the amount of the sample directed in the monograph, add 10 mL of *N ,N* -dimethylformamide, dissolve by heating if necessary, and designate the solution as the test solution.	**Method 1** (Gutzeit's method) **Apparatus** A is a 100 ml conical flask with standard ground joint : B is a standard hollow ground glass stopper connected to glass conduit C (external diameter 8.0 mm, internal diameter 6.0 mm), the total length of B and C is about 180 mm. D is a plastic screw, the upper part of which has an aperture 6.0 mm in diameter and the lower part of which has an aperture 8.0 mm in diameter; E is a plastic screw cap which has an aperture 6.0 mm in diameter. A wad of lead acetate cotton wool weighting about 60 mg is packed into tube C to a depth of about 60—80 mm. A disc of mercuric bromide test paper is placed between the contacting surfaces of D and E. **Arsenic standard stain** Place 2 ml of standard arsenic solution, accurately measured, in flask A, add 5 ml of hydrochloric acid and 21 ml water. Then add 5 ml of potassium iodide TS and 5 drops pf acid stannous chloride TS, allow to stand at room temperature for 10 minutes and add 2 g of zinc granules. Insert the stopper B and conduit C into the mouth of flask A and immerse the flask in a water bath at 25—40℃ for 45 minutes. Remove the mercuric bromide test paper. **Procedure** Transfer the preparation prepared as described under individual monographs to flask A and proceed as described under Arsenic standard stain, beginning with the wards "Then add 5 ml of potassium iodide TS ...". Any stain produced is not more intense than the standard stain. **Method 2** (Silver diethyldithiocarbamate method) **Apparatus** A is a 100 ml conical flask with standard ground joint; B is a standard hollow ground glass stopper connected to glass conduit C (at one end, the external diameter is 8.0 mm and the internal diameter is 6.0 mm; the other end is in length of 180 mm, in external diameter of 4 mm and in internal diameter of 1.6 mm, the internal diameter of sharp end is 1 mm). D is a glass tube with flat bottom (length 180 mm, internal diameter 10 mm, and with a graduation at 5.0 ml). A wad of cotton wool previously moistened with lead acetate TS and dried weighing about 0.1 g is packed into conduit C to a depth of about 80 mm, and measure 3 ml of silver diethyldithiocarbamate TS in tube D. Standard arsenic reference solution Transfer 2 ml of arsenic standard solution as described under Method I to flask A, accurately measured, add 5 ml of hydrochloric acid and 21 ml of water. Then add 5 ml of potassium iodide TS and 5 drops of acid stannous chloride TS, allow to stand at room temperature for 10 minutes and add 2 g of zinc granules. Connect conduit C into flask A immediately, and allow the evolved arsine to enter tube D. Immerse the flask A in a water bath at 25—40℃ for 45 minutes. Remove tube D, add chloroform to the graduation, mix well. **Procedure** Transfer the test preparation prepared as described under individual monographs to flask A and proceed as described under standard arsenic reference solution beginning with the words "Then add 5 ml of potassium iodide TS ...". Compare the above two solution against a white background. Any colour produced by the preparation is not more intense than produced by the standard arsenic reference solution. If necessary, determine the absorbance at the wavelength of 510 nm, with a suitable spectrophotometer or colorimeter, using silver diethyldithiocarbamate TS as the blank.	LIMIT TESTS FOR IMPURITIES (ARSENIC) Use Method A unless otherwise directed in the monograph **Method A** The Apparatus consists of a 100 ml conical flask closed with ground-glass stopper through which passes a glass tube about 200 mm long and 5 mm in internal diameter. The lower part of the tube is drawn to an in infernal diameter of 1 mm. 15 mm from its tip there is a lateral orifice 2 to 3 mm in diameter. When the tube is in position in the stopper the lateral orifice should be at least 3 mm below the lower surface of the stopper. The upper end of the tube has a perfectly flat, ground surface at right angles to the axis of the tube. A second glass tube of the same internal diameter and 30 mm long, with a similar flat ground surface, is placed in contact with the first and held in position by two spiral springs. *Procedure*: Into the longer tube insert 50 to 60 mg of lead acetate cotton *R* . Between the flat surfaces of the 2 tubes place a disc or a small square of mercury (II) bromide paper *R* large enough to cover the orifice of the tube, hold the 2 tubes in position by two spiral springs. In the conical flask dissolve or dilute the prescribed quantity of the substance being examined in sufficient water to produce 25 ml. Add 15 ml of hydrochloric acid *R* , 0.1 ml of tin (II) chloride solution As *TR* and 5 ml of a 20% solution of potassium iodide *R* . Allow to stand for 15 minutes and add 5 g of arsenic-free zinc *R* . Immediately assemble the two parts of the apparatus and immerse the flask in a water bath at a temperature such that a uniform evolution of gas is maintained. Prepare a standard at the same time and in the same manner using 1 ml of arsenic standard solution (1 ppm As) in place of the substance being examined and diluted to 25 ml with water. After not less than 2 hours compare the stains produced on the mercury (II) bromide papers. Any stain produced on the paper of the test flask is not more intense than that of the standard. **Method B** Add the prescribed quantity of the substance being examined to a test tube containing 4 ml of hydrochloric acid R and about 5 mg of potassium iodide R and add 3 ml of hydrophosphite solution R. Heat the mixture on a water bath for 15 minutes, shaking occasionally. Prepare a standard at the same time and in the same manner using 0.5 ml of arsenic standard solution (1 ppm As) in place of the substance being examined. Compare the colour produced in the test solution with that in the standard solution. Any colour produced in the test solution is not more intese than that obtained in the standard solution.

JP	KP	CP	VP
Heavy Metals Limit Test	**Heavy Metals Limit Test**	**Limit Test for Heavy Metals**	**LIMIT TESTS FOR IMPURITIES (HEAVY METALS)**
The Heavy Metals Limit Test is a limit test of the quantity of heavy metals contained as impurities in drugs. The heavy metals are the metallic inclusions that are darkened with sodium sulfide TS in acidic solution, as their quantity is expressed in terms of the quantity of lead (Pb). In each monograph, the permissible limit for heavy metals (as Pb) is described in terms of ppm in parentheses.	The Heavy Metals Limit Test is a limit test of the quantity of heavy metals contained as impurities in drugs. The heavy metals are the metallic inclusions that are darkened with sodium sulfide TS in acidic solution, as their quantity is expressed in terms of the quantity of lead (Pb). In each monograph, the permissible limit for heavy metals (as Pb) is described in terms of ppm in parentheses.	The Term "heavy metals" refers to those metals that react with thioacetamide or sodium under the specified conditions to produce a coloured compound.	Use one of the following methods as prescribed in the monograph.
Preparation of test solutions and control solutions	**Preparation of test solutions and control solutions**	**Method 1**	**Method 1**
Unless otherwise specified, test solution and control solution are prepared as directed in the following:	Unless otherwise specified, test solution and control solution are prepared as directed in the following:	Unless otherwise specified, use two 25 ml Nessler cylinders. To cylinder A add the specified volume of lead standard solution and 2 ml of acetate BS (pH 3.5). dilute with water or other solvent as specified under individual monographs to 25 ml. To cylinder B add 25 ml of the test preparation containing a quantity of the substance being examined as specified under individual monographs.	To 12 ml of the prescribed solution in a tube, add 2 ml of aceiate buffer pH 3.5 and mix. Add 1.2 ml of thioacetamide solution *R* , mix immediately and allow to stand for 2 minutes. Prepare a standard solution in the same manner using a mixture of 10 ml of either lead standard solution (1 ppm Pb) or lead standard solution (2 ppm Pb), as prescribed, and 2 ml of the solution being examined. Compare the colour produced in the test solution with that in the standard solution. Any brown colour produced in the test solution is not more intense than that obtained in the standard solution. The standard solution exhibits astlightly brown colour when compared to a blank solution prepared by tracting in the same manner a mixture of 10 ml water and 2 ml of the solution being examined.
(1) Method 1 Place an amount of the sample, directed in the monograph, in Nessler tube. Dissolve in water to make 40 mL. Add 2 mL of dilute acetic acid and water to make 50 mL, and designate it as the test solution. The control solution is prepared by placing the volume of Standard Lead Solution directed in the monograph in a Nessler tube, and adding 2 mL of dilute acetic acid and water to make 50 mL.	(1) *Method 1* Place an amount of the sample, directed in the monograph, in Nessler tube. Dissolve in water to make 40 mL. Add 2 mL of dilute acetic acid and water to make 50 mL, and designate it as the test solution. The control solution is prepared by placing the volume of Standard Lead Solution directed in the monograph in a Nessler tube, and adding 2 mL of dilute acetic acid and water to make 50 mL.	If the original test preparation is coloured, its colour can be matched by the addition of a few drops of dilute caramel solution or other suitable solution to cylinder A. To each cylinder add 2 ml of thioacetamide TS and mix well, allow to stand for 2 minutes, compare the colour produced by viewing down the vertical axis of the cylinder against a white background. The colour produced in cylinder B is not more intense than that produced in cylinder A. If the colour cannot be matched by the addition of caramel solution, duplicate the quantity of the substance being examined and the regent, add water or other solvent as specified under individual monographs to produce 30 ml of test preparation. Divide the test preparation into two equal portions and transfer to Nessler cylinder A and B. To cylinder B add sufficient water or other solvent as specified under individual monograph to produce 25 ml. To cylinder A add 2 ml of thioacetamide TS, mix well and allow to stand for 2 minutes, filter through filter membrane of 3 μ m in porosity. To cylinder A add the prescribed volume of lead standard solution and dilute with water of other solvent as specified under individual monographs to produce 25 ml. Then add 2 ml of thioacetamide TS to cylinder B and 2 ml of water to cylinder A and compare the colour as described above.	**Method 2** Dissolve the specified quantity of the substance being examined in an organic solvent containing a minimum percentage of water, such as 1, 4-dioxan *R* or acetone *R* containing 15% of water. Carry out Method 1 but prepare the lead standard solution by diluting lead standard solution (100 ppm Pb) with the solvent used to prepare the test solution to contain 1 or 2 ppm of Pb, as specified.
(2) Method 2 Place an amount of the sample, directed in the monograph, in a quartz or porcelain crucible, cover loosely with a lid, and carbonize by gentle ignition. After cooling, add 2 mL of nitric acid and 5 drops of sulfuric acid, heat cautiously until white fumes are no longer evolved, and incinerate by ignition between 500℃ and 600℃. Cool, add 2 mL of hydrochloric acid, evaporate to dryness on a water bath, moisten the residue with 3 drops of hydrochloric acid, add 10 mL of hot water, and warm for 2 minutes. Then add 1 drop if phenolphthalein TS, add ammonia TS dropwise until the solution develops a pale red color, add 2 mL of dilute acetic acid, filter if necessary, and wash with 10 mL of water. Transfer the filtrate and washing to a Nessler tube, and add water to make 50 mL. Designate it as the test solution. The control solution is prepared as follows: Evaporate a mixture of 2 mL of nitric acid, 5 drops of sulfuric acid and 2 mL of hydrochloric acid on a water bath, further evaporate to dryness on a sand bath, and moisten the residue with 3 drops of hydrochloric acid. Hereinafter, proceed as directed in the test solution, then add the volume of Standard Lead Solution directed in the monograph and water to make 50mL.	(2) *Method 2* Place an amount of the sample, directed in the monograph, in a quartz or porcelain crucible, cover loosely with a lid, and carbonize by gentle ignition. After cooling, add 2 mL of nitric acid and 5 drops of sulfuric acid, heat cautiously until white fumes are no longer evolved, and incinerate by ignition between 500℃ and 600℃. Cool, add 2 mL of hydrochloric acid, evaporate to dryness on a water bath, moisten the residue with 3 drops of hydrochloric acid, add 10 mL of hot water, and warm for 2 minutes. Then add 1 drop if phenolphthalein TS, add ammonia TS dropwise until the solution develops a pale red color, add 2 mL of dilute acetic acid, filter if necessary, and wash with 10 mL of water. Transfer the filtrate and washing to a Nessler tube, and add water to make 50 mL. Designate it as the test solution. The control solution is prepared as follows: Evaporate a mixture of 2 mL of nitric acid, 5 drops of sulfuric acid and 2 mL of hydrochloric acid on a water bath, further evaporate to dryness on a sand bath, and moisten the residue with 3 drops of hydrochloric acid. Hereinafter, proceed as directed in the test solution, then add the volume of Standard Lead Solution directed in the monograph and water to make 50mL.	If the substance being examined contains a ferric salt which interferes the test, 0.5— 1.0 g of ascorbic acid should be added to each cylinder. Unless otherwise specified, evaporate the same quantity of the same reagents to dryness in a porcelain dish. Dissolve the residue in 2 ml of acetate buffer (pH 2.5) and 15 ml of water. Transfer the solution to a Nessler cylinder, add the specified quantity of lead standard solution and water to 25 ml. The solution is used as reference solution for the test solution which is prepared by using more than 1.0 ml of hydrochloric acid or equivalent amount of dilute hydrochloric acid, 2 ml of ammonia TS or by treating with other regents.	**Method 3** Place the prescribed quantity (usually not more than 2 g) of the substance being examined in a silica crucible. Add 4 ml of a 25% solution of magnesium sulphate in 2 N sulphuric acid *R* . Mix using a fine glass rod and heat cautiously. If the mixture is liquid, evaporate gently to dryness on a water bath. Progressively heat to ignition, not allowing the temperature to exceed 800℃, and continue heating until a white or at most greyish residue is produced. Allow to cool, moisten the residue with 0.2 ml of 2 N sulphuric acid *R* , evaporate, ignition again and allow to cool. The total period of ignition must not exceed 2 hours. Dissolve the residue using two 5 ml quantities of 2 N hydrochloric acid *R* . Add 0.1 ml of phenolphthalein solution *I* and concentrated ammonia solution *R* dropwise until a pink colour is produced. Cool, add glacial acetic acid *R* until the solution is decolorized and add a further 0.5 ml. Filter if necessary and dilute the solution to 20 ml with water. To 12 ml of the resulting solution in a tube, add 2 ml of acetate buffer pH 3.5 and mix. Add to 1.2 ml of thioacetamide solution *R* , mix immediately and allow to stand for 2 minutes. Compare the colour produced in the test solution with that in a standard solution prepared simultaneously in the same manner. Any colour producedin the test solution is not more intense than that obtained in the standard solution.
(3) Method 3 Place an amount of the sample, directed in the monograph, in quartz or porcelain crucible, heat cautiously, gently at first, and then increase the heat until incineration is completed. After cooling, add 1 mL of aqua regia, evaporate to dryness on a water bath, moisten the residue with 3 drops of hydrochloric acid, add 10 mL of hot water, and warm for 2 minutes. Add 1 drop of phenolphthalein TS, add ammonia TS dropwise until the solution develops a pale red color, add 2 mL of dilute acetic acid, filter if necessary, wash with 10 mL of water, transfer the filtrate and	(3) *Method 3* Place an amount of the sample, directed in the monograph, in quartz or porcelain crucible, heat cautiously, gently at first, and then increase the heat until incineration is completed. After cooling, add 1 mL of aqua regia, evaporate to dryness on a water bath, moisten the residue with 3 drops of hydrochloric acid, add 10 mL of hot water, and warm for 2 minutes. Add 1 drop of phenolphthalein TS, add ammonia TS dropwise until the solution develops a pale red color, add 2 mL of dilute acetic acid, filter if necessary, wash with 10 mL of water, transfer the filtrate and	**Method 2** Unless otherwise specified, use the residue obtained from the Determination of residue on Ignition, add 0.5 ml of nitric acid, evaporate to dryness, heat until nitrous oxide fumes are no longer evolved (or alternatively, ignite a quantity of the substance being examined in crucible until thoroughly charred, cool, moisten the residue with 0.5— 1.0 ml of sulfuric acid, ignite at a low temperature until sulfurous acid fumes are no longer evolved, add 0.5 ml of nitric acid, evaporate to dryness, heat until nitrous oxide fumes are no longer evolved and ignite at 500— 600℃ until the incineration is complete). Cool, add 2 ml of hydrochloric acid evaporate to dryness on a water bath, add 15 ml of water, followed by ammonia TS dropwise until the solution is neutral to phenolphthalein IS, then add 2 ml of acetate BS (pH 3.5) and warm to effect dissolution. Transfer the resulting solution to Nessler cylinder B, dilute with water to 25 ml and produced as described under method 1. The reference preparation should be prepared as follows. Place the same quantify of the same regents used for the preparation of test solution in a porcelain dish and evaporate to dryness, heat gently and dissolve in 2 ml of acetate BS (pH 3.5) and 15 ml of water, transfer to the Nessler cylinder A and add the specified volume of standard lead solution, dilute with water to 25 ml.	**Method 4** Mix the prescribed quantity of the substance being examined with 0.5 g of magnesium oxide *R* in a silica crucible. Ignite to dull red heat until a homogeneous white or greyish white mass is produced. If after 30 minutes of ignition the mixture remains coloured, allow to cool, mix with a fine glass rod and repeat the ignition. If necessary, repeat the operation. Finally heat at 800℃ for about 1 hour. Dissolve the residue using two 5 ml quantities of 5 N hydrochloric acid solution *R* and carry out the procedure described under Method 3 beginning at the word "Add 0.1 ml of phenolphthalein solution I...". To prepare the standard solution place the prescribed volume of lead standard solution (10 ppm Pb) in a silica crucible, add 0.5 g of magnesium oxide *R* and mix. Dry the mixture in an oven at 100℃ to 105℃, ignite as described above. Dissolve the residue using two 5 ml quantities of 5 N hydrochloric acid solution *R* and carry out the procedure described under Method 3 from the substance "Add 0.1 ml of phenolphthalein solution I..." and use a mixture of 10 ml of the above treated lead standard solution and 2 ml of the test solution.
		Method 3 Unless otherwise specified, dissolve a quantity of the substance being examined in 5 ml if sodium hydroxide TS and 20 ml of water. Transfer	**Method 5** Use a membrane filter holder, the dimensions of which are shown in Figure, fitted with a 50 ml syringe. The membrane filter disk (C) is made of a suitable material with a nominal pore diameter of 3 μ m and protected by a prefilter (B) that is made of borosilicate glass wire. Dissolve

JP	KP	CP	VP
Heavy Metals Limit Test	**Heavy Metals Limit Test**	**Limit Test for Heavy Metals**	**LIMIT TESTS FOR IMPURITIES (HEAVY METALS)**
washings to a Nessler tube, and add water to make 50 mL. Designate it as the test solution. The control solution is prepared as follows: Evaporate 1 mL of aqua regia to dryness on a water bath. Hereinafter, proceed as directed for the test solution, and add the volume of Standard Lead Solution directed in the monograph and water to make 50 mL. (4) Method 4 Place an amount of the sample, directed in the monograph, in a platinum or porcelain crucible, mix with 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 10), fire the ethanol to burn, and carbonize by gradual heating. Cool, add 1mL of sulfuric acid, heat carefully, and incinerate by ignition between 500℃ and 600℃. If a carbonized substance remains, moisten with a small amount of sulfuric acid, and incinerate by ignition. Cool, dissolve the residue in 3 mL of hydrochloric acid, evaporate on a water bath to dryness, wet the residue with 3 drops of hydrochloric acid, add 10 mL of water, and dissolve by warming. Add 1 drop of phenolphthalein TS, add ammonia TS dropwise until a pale red color develops, then add 2 mL of dilute acetic acid, filter if necessary, wash with 10 mL of water, transfer the filtrate and the washing to Nessler tube, add water to make 50 mL, and use this solution as the test The control solution is prepared as follows: Take 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 10), and fire the ethanol to burn. Cool, add 1 mL of sulfuric acid, heat carefully, and ignite between 500℃ and 600℃. Cool, and add 3 mL of hydrochloric acid. Hereinafter, proceed as directed in the test solution, then add the volume of Standard Lead Solution directed in the monograph and water to make 50 mL.	washings to a Nessler tube, and add water to make 50 mL. Designate it as the test solution. The control solution is prepared as follows: Evaporate 1 mL of aqua regia to dryness on a water bath. Hereinafter, proceed as directed for the test solution, and add the volume of Standard Lead Solution directed in the monograph and water to make 50 mL. ***(4) Method 4*** Place an amount of the sample, directed in the monograph, in a platinum or porcelain crucible, mix with 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 10), fire the ethanol to burn, and carbonize by gradual heating. Cool, add 1mL of sulfuric acid, heat carefully, and incinerate by ignition between 500℃ and 600℃. If a carbonized substance remains, moisten with a small amount of sulfuric acid, and incinerate by ignition. Cool, dissolve the residue in 3 mL of hydrochloric acid, evaporate on a water bath to dryness, wet the residue with 3 drops of hydrochloric acid, add 10 mL of water, and dissolve by warming. Add 1 drop of phenolphthalein TS, add ammonia TS dropwise until a pale red color develops, then add 2 mL of dilute acetic acid, filter if necessary, wash with 10 mL of water, transfer the filtrate and the washing to Nessler tube, add water to make 50 mL, and use this solution as the test The control solution is prepared as follows: Take 10 mL of a solution of magnesium nitrate hexahydrate in ethanol (95) (1 in 10), and fire the ethanol to burn. Cool, add 1 mL of sulfuric acid, heat carefully, and ignite between 500℃ and 600℃. Cool, and add 3 mL of hydrochloric acid. Hereinafter, proceed as directed in the test solution, then add the volume of Standard Lead Solution directed in the monograph and water to make 50 mL. ***(5) Method 5*** Unless otherwise specified, in the monograph, place 0.5 g of extract or 1.0 g of fluidextract in a platinum or porcelain crucible, evaporate to dryness on a water bath, incinerate by ignition between 500℃ and 600℃. Cool, dissolve the residue in 3 mL of hydrochloric acid by warming, filter and wash the residue 5 mL of water two times. Transfer the filtrate and washings to a Nessler tube, add 1 drop of phenolphthalein TS, add ammonia TS dropwise until a pale red color develops, then add 2 mL of dilute acetic acid, and add water to make 50 mL. Designate it as the test solution. The control solution is prepared as follows: add 3 mL of hydrochloric acid. Hereinafter, proceed as directed in the test solution, then add 3.0 mL of Standard Lead Solution and water to make 50 mL.	the solution to a Nessler cylinder, add 5 drops of sodium sulphide TS and mix well the colour produced is not more intense than of a reference preparation containing the specified volume of lead standard solution and treated in the same manner. **Method 4** **Apparatus** The filter holder is compared of tightly sealed upper and lower parts with screw thread, washer, filter A is the upper cap part pf the filter holder the entrance may be fitted with a 50 ml syringe; B is ioint : C is washer (external diameter is 10 mm, internal diameter is 6 mm) : D is filter membrane with 10 mm in diameter and 3.0 μ m of porosity, soaked in water for more than 24 hours before use; E is auxiliary filter plate made of No.3 sintered glass filter plate with 10 mm in diameter and 1 mm in thickness; F is the lower part of the filter holder, the exit is fitted with a suitable rubber tube. **Lead standard stain** Measure accurately a quantity of lead standard solution to a small beaker, dilute to 10 ml with water or other solvent as and 1.0 ml of thioacetamide TS, mix well, allow to stand for 10 minutes. Transfer to a filter holder with a 50 ml syringe and filter it on applying an even pressure (filter rate is about 1 ml per minute), then place the filter membrane on a piece of filter paper and dry it.	the prescribed quantity of the substance being examined in 30 ml of water unless otherwise specified in the monograph. Filter the solution applying an even pressure. Dismantle the holder and check that the membrane filter remains uncontaminated; if necessary replace the membrane filter and repeat the filtration. To the whole filtrate, or the prescribed volume of the filtrate, add 2 ml of acetate buffer pH 3.5 and add to 1.2 ml of thioacetamide solution *R* , mix and allow to stand for 10 minutes. Invert the order of the filters, and filter the solution anolving slow and even pressure. Remove the membrane filter is not move intense than that obtained by standard which is treated using the prescribed volume of lead standard solution (1 ppm Pb) in the same manner from the sentence "Add 2 ml of acetate buffer pH 3.5...".
		General Quality Control Method for Crude Drugs	
		General quality control method for crude drugs includes the "Description", "Identification", "Tests", "Determination of Extractives" and "Assay" of crude drugs. A scheme for the examination of crude drugs is outlined below.	

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		General Quality Control Method for Crude Drugs	
		1. Carry out the method for sampling of crude drugs to take the drugs being examined. 2. Use a reference drug concerned which complies with the requirements specified under individual monograph to verify the result of tests or assays of a crude drug. 3. If the crude drugs being examined are broken, they should comply with the general requirement, except that described under "Description" in the monograph concerned. 4. "Description" consists of the form, size, colour, surface characters, texture, cut surface or fracture characters, odour and taste. 5. Identification indicates the methods for the examination of the identify of crude drugs, consisting of the traditional experientional, microscopic, physical and chemical methods. 6. Tests refers to test for the purity of crude drugs, such as the content of water, ash or foreign matter. 7. Determination of extractive refers to determine the content of soluble substances in crude drugs extracted with water or other solvents. 8. Assay refers to examine the crude drugs quantitively with chemical, physical or biological methods, including the determination of volatile oils, the content of active principles and potency by biological assay.	
		The Processing of Crude Drugs	THE PROCESSING OF CRUDE DRUGS
		Processing of crude drugs is to make the crude drugs into small processed pieces through processing procedures such as cleaning, cutting and stir-baking, so that to obtain the processed drugs fulfilling the requirements of therapy, dispensing and making preparations thus assuring the safety and efficacy of the drugs. The water used for processing should be unpolluted drinking water. Unless specified otherwise, the processing should meet the following requirements. **1. Cleaning** The crude drugs after cleaning are called "clean crude drugs". Clean crude drugs should be used in cutting, processing, dispensing or compounding. The crude drugs can be cleaned with the method of sorting, winnowing, washing, sifting, cutting, scraping, paring, rejecting, brushing, rubbing and grinding, soaking, rinsing etc. to reach the quality standard on the basis of specific conditions. **2. Cutting** Unless cutted in fresh or dry form, the crude drugs should be moistened to soft for cutting, it is better to keep moisten than to soak in water to prevent the elimination of active principles, the crude drugs should be treated separately and appropriately according to their size, diameter and hardness, nothing the temperature, quantity of water and duration of treatment. The drugs should be dried in time after cutting. The crude drugs may be cut into slices, sections, pieces and slivers, etc. Their size and thickness are generally as follows. *Slices* Less than 0.5 mm in thickness for very thin slices, 1-2 mm in thickness for thin slices: more than 2-4 mm in thickness for thick slices. *Sections or segments* 10-15 mm in length. *Pieces* Cubes of 8-12 mm. *Slivers* 2-3 mm in width for barks; 5-10 mm in width for leaves. The crude drugs other than those treated by cutting are usually treated by pounding. **3. Roasting and Broiling** Unless specified otherwise, the general methods and requirments are as follows. (1) **Stir-baking** (2) **Scalding** (3) **Calcing** (4) **Carbonizing** (5) **Steaming** (6) **Boiling** (7) **Stewing** (8) **Blanching in boiling water** (9) **Processing with wine** (10) **Processing with vinegar**	In traditional Vietnamese medicine, the medicaments used by oral administration are always to undergo stages of processing. *Preprocessing* (preliminary processing): The preprocessing aims at removing parts that are not indented for medicinal use (roodets, cores, roots, stones...) or stabilising the crude drugs right away at the beginning (exposure to sunlight, drying, sulphuration...). Thus, after preprocessing the initial materials are obtained and called "raw drugs" that however have to comply with certain requirements of quality standard. *Complex-processing* (processing): This is more complicated process with a view to reducing toxicity, adverse and side effects or changing therapeutic categories, increasing channel tropism and still affecting very often the active ingredient structure and effects of the crude drugs to be processed. Thus, after complex-processing the materials with official meaning are obtained and called "processed drugs", complying with the requirement of therapy. **Aqueous methods** (water-processing) *Washing* *Soaking* *Wrapping up* *Levitating* **Thermal methods** (fire-processing) *Stir-baking* *Simple stir-baking* *Stir-baking with gentle heat* *Stir-baking to yellowing* *Stir-baking to yellowing and laying down on the ground* *Stir-baking to yellowing with darkened fractures* *Stir-baking with nature preservation (Stir-baking to darkening)* *Stir-baking to carbonizing* *Stir-baking with liquid excipients* *Stir-baking with wine* *Stir-baking with vinegar (processing with vinegar)* *Stir-baking with honey* *Stir-baking with ginger juice* *Stir-baking with ginger loses* *Stir-baking with milk* *Stir-baking with rice-washing water* *Stir-baking with urine* *Stir-baking with black-bean water* ***Stir-baking through an intermediary***

JP	KP	CP	VP
		The Processing of Crude Drugs	**THE PROCESSING OF CRUDE DRUGS**
		(11) **Processing with solt-water** (12) **Stir-baking with ginger juice** (13) **Stir -Baking with honey** (14) **Frost-like powder** (15) **Levigating**	*Stir-baking in a sand-bath* *Stir-baking in a bath of powdered talc or clam-shell* *Broiling* *Burning with ethanol* *Calcinating* **Drying** *Drying in a stove at normal pressure* *Drying over a cooking fire or charcoal oven* **Aqueous-thermal methods** **Stewing** *Steaming* *Boiling* *Quenching*
		Determination of Tanninoids	**DETERMINATION OF TANNINOIDS IN HERBAL DRUGS**
		Weigh accurately a quantity of powdered crude drug (passed through No.3 sieve and containing 1 g of tanninoids) to a conical flask, add 150 ml of water and heat on a water bath for 30 minutes. Allow to cool, transfer the mixture to a 250 ml volumetric flask, dilute to volume with water, filter, use the filtrate as the test solution. **Determination of total water-extractives** Evaporate 25 ml of the test solution, accurately measured, to dryness, dry the residue at 105℃ for 3 hours and weigh (T1). **Determination of water-extractives not bound with hide powder** To 100 ml of the test solution, measured accurately, add 6 g of dry hide power RS, shake well for 15 minutes and filter. Evaporate 25 ml of the filtrate, accurately measured, to dryness, dry the residue at 105℃ for 3 hours and weigh (T2). **Determination of water-extractives of hide powder** To 100 ml water, accurately measured, add 6 g dry hide powder RS, shake well for 15 minutes and filter. Evaporate 25 ml of the filtrate, accurately measured, to dryness, dry the residue at 105℃ for 3 hours and weigh (T0).	Weigh accurately a quantity of powdered crude drug (passed through a NO 355 sieve) containing about 1g of tannoids. Place in a conical flask, add 150 ml of water and heat on a bath for 30 minutes. Allow to cool, transfer the mixture to a 250 ml volumetric flask. Dilute to volume with water, filter and use the filtrate as the test solution. **Determination of total water-soluble extractives** Take accurately 25 ml of the test solution, evaporate to dryness, dry the residue at 105℃ for 3 hours. Weigh (T1 g). **Determination of water-soluble extractives not bound with hide powder** To 100 ml of the test solution, measured accurately, add 6 g of dry hide powder *R*. Shake well fore 15 minutes and filter, Take accurately 25 ml of the filtrate, evaporate to dryness, dry the residue at 105℃ for 3 hours. Weigh (T2 g). **Determination of water-soluble extractives of hide powder** To 100 ml of water, measured accurately, add 6 g of dry hide powder (R). Shake well fore 15 minutes and filter, Take accurately 25 ml of the filtrate, evaporate to dryness, dry the residue at 105℃ for 3 hours. Weigh (T0 g). Calculate the percentage of tanninoids in herbal drugs from the expression: (T1-T2+T0) x 10/a x100 where: a is the mass taken (in g) of the drug being examined, calculated on the dried basis.
		Determination of Cineol	**DETERMINATION OF CINEOLE IN THE VOLATILE OIL**
		Carry out the method for gas chromatography. Test of the suitability of the system Pack a column with 7:3 (g/g) of 10.0% polyethylene glycol (PEG)-20M and 2.0% silicon (OV-17), with PEG at the end of injection; maintain the column temperature 110±5℃; the number of theoretical plate of the column is not less than 2500, calculated with reference to cineol; the resolution factor of the peaks of cineol and its neighbouring impurities should meet the requirement.	Weigh 3.00g of the sample, recently dried with anhydrous sodium sulphate *R* , into a dry test tube and add 2.10g of melted *o* -cresol. Place the tube in the apparatus for the determination of freezing point and allow to cool, stirring continuously. When crystallisation takes place there is a small rise in temperature; note the highest temperature reached (t1). Remelt the mixture on a water bath ensuring that the temperature douse not exceed t1 by more than 5℃ and place the tube in the apparatus maintained at a temperature 5℃ below t1. When recrystallisation takes place, or when the temperature of the mixture has fallen 3℃ below t1, stair continuously, note the highest temperature at which the mixture freezes (t2). Repeat the operation until the tow highest values obtained for t2 not differ by more than 0.2℃. If super cooling occurs, induce crystallisation by the addition of small crystal of a complex consisting of 3.00g of cineol and 2.10g of melted o-cresol. If t2 is below 27.4℃, repeat the determination after the addition of 5-10g of the complex. Determine the percentage (m/m) of cineole correspondent to the freezing point (2) from the Table, obtaining intermediate values by interpolation. If 5.10g of the cineol *o* -cresol complex was added, calculate the percentage m/m of cineole from the expression 2 (A-50), where A is the value corresponding to a freezing point of t2 taken from the Table.

ための有用性評価（EBM 確保）手法及び安全性確保等に関する研究」，並びに平成 16 及び 17 年度厚生労働科学研究費補助金（医薬品・医療機器等レギュラトリーサイエンス総合研究事業）「一般用漢方処方の見直しに資するための有用性評価（EBM 確保）手法及び安全性確保等に関する研究」によった．

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Medical and Pharmaceutical Society for WAKAN-YAKU

一般用漢方処方の使用実態調査AUR (Actual Use Research) およびAURを用いた加味逍遙散の特徴解析

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Actual Use Research, a new method for evaluating the effectiveness of OTC Kampo drugs and its application to Kamishoyosan formulation

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Actual Use Research (AUR) is a new pharmacist-centered research system to evaluate the usefulness of OTC Kampo medicine. The system uses a commercially available OTC drug. First, after an explanatory meeting of the AUR system, a pharmacist (or pharmacy) is contracted by the AUR implementation committee. The pharmacist invites the customers of the pharmacy to participate in AUR. After consent and answering several questions from the pharmacist, the AUR participant purchased the test OTC drug and begins to keep a daily use record of dosage and time of intake, the condition of disease and use of other drugs. After a predetermined number of days, or when the symptoms of the disease disappear, the participant returns to the pharmacy, and submits the daily record to the pharmacist, and answers a questionnaire evaluating the usefulness of the drug as well as some questions from the pharmacist. The participant then receives a gratuity. Independent from the participant evaluation, the pharmacist evaluates the usefulness of the drug on the basis of information obtained by the interview with the participant at the second meeting. Then, the pharmacist submits the daily record and the questionnaire from the participant, and also his/her evaluation to the AUR implementation committee. In this report, we describe the first trial of AUR using the commercially available OTC "Kamishoyosan" (a Kampo formula used to treat women with painful tension in the shoulders, who tire easily, suffer from fear, neuro-psychic disturbance, have a tendency toward constipation, sensitivity to cold, and/or dysmenorrheal and/or oligomenorrhea).

Key words OTC Kampo medicines, Kamishoyosan, pharmacist, Actual Use Research.

緒　言

近年，急速な高齢化の進展や生活習慣病の増加などの疾病構造の変化，生活の質（QOL）の追求等に伴い，自分の健康に強い関心を持つ国民が増えている。それとともに，薬局や薬店の薬剤師等による適切なアドバイスの下で，身近にある一般用医薬品を利用するセルフメディケーションの考え方が広がりつつある。一般用医薬品のあり方等に関しては，これまでも様々な場で検討されてきた。しかしながら，以前に比べて高齢者の全人口に占める割合がさらに増加し，国民の健康ニーズも多様化している。その現状において，今後，保健・医療資源としての一般用医薬品の有効活用を進めていくためには，国民の新たなニーズに対応し得る一般用医薬品を育成していく必要がある。この様な背景から，厚生労働省では国民の新たなニーズに対応し得る一般用医薬品の育成を考

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え，平成14年の6月から一般用医薬品承認審査合理化等検討会を開催し，11月にその中間報告として，「セルフメディケーションにおける一般用医薬品のあり方について　提言-具体的な方策-」を発表している[1]。一般用漢方処方「210処方」は，昭和40年代末に当時の厚生省より承認審査の内規として発表され，そのまま現在に至っている。中間報告はこの点を指摘し，「現在の生活環境の変化や急激な人口の高齢化に伴う疾病構造の変化等に伴い，国民のニーズに合致しなくなってきている」として見直しの必要性を提言している。一般用漢方処方を見直す為には，先ず一般用漢方処方の有用性評価と，その安全性確保が重要となる。

漢方処方（方剤）は，西洋薬と比較して，効能効果が顕著に現れにくく，長期間の使用による体質改善を目的とするなど，その有用性の評価が困難であることがいわれ，長年の使用経験に基づいて承認されている。医療用製剤の場合，平成3年の漢方8処方について再評価指定以来，それらの処方について臨床評価試験が行われている。他方，一般用漢方製剤の場合，これまでその有用性を評価する手法は示されていない。本研究は，上記提言に即し，薬剤師研修センター，日本漢方生薬製剤協会一般用漢方製剤委員会，日本大衆薬工業協会薬制委員会，大学関係者，薬局薬剤師の協力を得て，一般用漢方処方の有用性評価の手法AUR（Actual Use Research）の確立を目的として行った。

AURは，2001年に清水らがインドメタシンハップ剤を用い薬剤師会，薬局，薬剤師ベースで行った使用実態試験AUT（Actual Use Trial）[2]を参考にし，著者らの研究グループが，一般用であること，証の概念のある漢方処方であることを念頭に確立しようとする薬局，薬剤師ベースの有用性評価手法である。著者らは，一般用医薬品の場合，①薬剤師と消費者が効いたと判断することが重要，②一般用漢方処方の再評価モデルと考えた場合，プラセボを用いず市販薬をそのまま使用するスタイルが適当，③選択基準，除外基準に漢方処方の「証」の考えをとりいれることで漢方処方そのものの有効性を判断することも可能と考え，AURパイロット研究調査実行委員会を設立した。調査実行委員会では，AURの実行方法を論議するとともに売り上げ上位の代表的な漢方処方の中から，①長期疾病に用いる処方として加味逍遙散，②短期疾病（数日）に用いる処方として葛根湯，③中間的な疾病期間（10日間）をもつ猪苓湯を選択し，2004年から2005年において3度AURを実施した。本論文では加味逍遙散を用いて行ったAURパイロット調査研究の結果をもとに，一般用漢方処方の評価手法としてAURの特徴と得られた問題点等について述べる。さらに，一般用医薬品として加味逍遙散の有用性とその特徴について解析する。なお，加味逍遙散は臨床的には更年期障害に対しホルモン補充療法と同程度に有用であり[3]，かつエストロゲン活性がほとんどないため，ホルモン補充療法より安全性が高いことが示されている[4]。また，基礎研究においては瘀血モデルマウスに対して末梢血管における血流量を増加させることが報告されている[5]。加味逍遙散についてはこの他にも多数の臨床研究および基礎研究が報告されている[6-17]。

対象と方法

調査方法は，薬局にて薬剤師が行う医師の介在がない調査としてデザインした。これは，現在医療用漢方処方の再評価のために実施されている，医師の介在を必要とする治験のスタイルとは異なる。また，治験と異なってプラセボを用いないone-armのコホート研究とした。

実施体制の概略

調査の実施体制の概略をFig.1に示した。調査依頼者はAURパイロット研究調査実行委員会とした。調査は，調査実施薬局と定めた薬局の調査責任薬剤師と契約を結び，実施した。調査実施薬局では，調査責任薬剤師（あるいは調査責任薬剤師から説明を受けた調査薬剤師）が，購入目的で来店した消費者に対して『一般用漢方処方加味逍遙散のパイロット使用実態調査参加についての説明書および同意書』[18]を用いて本調査研究に関する説明を行い，同意が得られた消費者を調査協力者とした。調査使用薬としては『カミセーヌ「コタロー」（小太郎漢方製薬株式会社製加味逍遙散エキス製剤）』を使用し，これを通常の販売形態にて販売した上で，漢方処方服用に対する満足度をアンケート方式にて調査した。調査協力者の募集目標は100人とし，用法・用量を遵守して調査使用薬の1パッケージ（180錠）を服用し終わった時，あるいは症状が消失した時をエンドポイントとした。

既に一般用医薬品として承認されている製品であることから，重篤な有害作用の出現の可能性は低いと考えられるが，有害作用が出現したときに備えて調査協力医師を定め，有害作用が出現した際に，調査薬剤師が調査協力医師より適切な助言が得られるように取り計らった。

実施方法

本研究方法は国立医薬品食品衛生研究所の倫理委員会において承認を受けたものである。

まず，調査実行委員会が調査使用薬と調査実施地区（今回は東京近郊地区）を指定し，日本漢方生薬製剤協会より，調査使用薬を日常的に販売している薬局を推薦いただいた。次いで，推薦された薬局に調査実施説明会（平成16年5月30日）の案内状を送付した。調査実施説明会には，調査責任薬剤師の参加を必須とした。調査実施説明会では，実施期間・実施方法・実施上の注意点等について説明を行った。特に調査記録票や調査協力者日誌の記入方法や個々の判断基準は，本説明会で詳細に説明した。説明会終了後，調査実施薬局として調査への参加に同意できる薬局の調査責任薬剤師と，調査実施薬局としての契約を結んだ。調査の実施は全て調査実施薬局において行った。

調査は平成16年年6月1日～9月30日の4ヶ月間にわたり，