药品红外光谱集

ATLAS OF INFRARED SPECTRA OF DRUGS

第四卷（2010）

国家药典委员会 编

中国医药科技出版社

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前　　言

红外光谱法是有机化合物分析中广泛应用的方法。由于红外光谱的高度专属性，在药品检验中，红外光谱法常与其他理化方法联合使用，作为有机药品重要的鉴别方法。鉴于有机药品品种不断增加，特别是许多药品化学结构比较复杂或相互之间化学结构差异较小，当用颜色反应、沉淀、结晶形成或紫外-可见分光光度法等常用方法不足以相互区分时，红外光谱法更是行之有效的鉴别手段。

《中华人民共和国药典》（二部）自1977年版开始采用红外光谱法用于一些药品的鉴别，在该版药典附录中收载了对照图谱。为了适应我国对药品监督检验的需要，1985年，我委委托部分省、市药品检验所收集绘制了国产药品红外光谱图423幅，编制出版了《药品红外光谱集》1985年版，作为药品鉴别用红外对照图谱。鉴于《中华人民共和国药典》及国家药品标准均收载红外光谱法，应用红外光谱鉴别的品种不断增加，有必要在原有基础上扩大收载范围，为此，国家药典委员会正式组织编制出版《药品红外光谱集》作为国家标准系列配套丛书，广泛用于药品的鉴别检验。1986年我会组织中国药品生物制品检定所及湖北、北京、湖南、上海、天津、辽宁、黑龙江、江苏等省市药品检验所的部分专家组成编审组，负责绘制和审定图谱。1990年，出版了《药品红外光谱集》1990年版，该版光谱集共收载582幅图谱。为了适应光谱集编制工作的延续性，经编审组研究决定，分卷出版《药品红外光谱集》，1995年出版了第一卷，收载了光栅型红外分光光度计绘制的药品红外光谱图共685幅。2000年出版了第二卷，收载药品红外光谱图208幅，并全部改由傅立叶红外光谱仪绘制。2005年版出版第三卷，共收载药品红外光谱图210幅（其中172个为新增品种，38个老品种重新绘制了图谱）。2010年出版第四卷，共收载药品红外光谱图124幅。凡在《中华人民共和国药典》和国家药品标准中收载红外鉴别或检查的品种，除特殊情况外，本光谱集中均有相应收载，以供比对。《中华人民共和国药典》和国家药品标准中不另收载红外光谱图。其他光谱图可供药品检验中作对照用。

本光谱集所需的样品和资料得到了全国各省、市、自治区药品检验所及有关单位的大力支持，在此一并致谢。对于本光谱集的不妥之处，希望各有关单位在实践过程中及时提出宝贵意见，以便予以修订。

国家药典委员会

2009年12月

PREFACE

Infrared spectrophotometry is a means widely used to analyze organic compounds. As the high degree of specificity of the infrared spectrum for a given compound, infrared spectrophotometry is commonly adopted in pharmaceutical analysis combined with the other physical and chemical methods in the identification test of organic drug substances. With the number of organic drug substances increases continuously, especially their chemical structures are complicated or rare different in case of their analogs, the tests based on infrared spectrophotometry are always found to be an effective one when the usual methods for identification, such as color reactions, precipitation, crystal tests or Ultra-vis spectrophotometry, etc., are inadequate to differentiate the drug substances with closely related structures.

Identification tests of some drug substances based on infrared spectrophotometry were introduced for the first time into the Chinese Pharmacopoeia volume Ⅱ, 1977 edition, and the infrared reference spectra were compiled in the appendix. In 1985, to meet the requirement of drug control in China, the Pharmacopoeia Commission entrusted some institutes for drug control to compile and publish the Atlas of Infrared Spectra of Drugs (1985) in which the infrared spectra for 423 drug substances supplied by domestic manufactures were recorded as the infrared reference spectra being adopted in specifications of pharmaceutical substances. Seeing that the infrared spectrophotometry had been adopted in the Chinese Pharmacopoeia as well as in other National Pharmaceutical Specifications and the number of specifications with tests based on infrared spectrophotometry was increasing steadily, there was a demand for further extension of this work. So the Atlas was officially published as a companion volume of National Pharmaceutical Specifications. Therefore, the Commission organized a collaborative study & reviewing experts group, composed of scientists from National Institute for the Control of Pharmaceutical and Biological Products, Hubei, Beijing, Hunan, Shanghai, Tianjin, Liaoning, Jiangsu and Heilongjiang Institutes for Drug Control, to carry out the work of recording, examining, verifying and compiling the spectra. In 1990, the Atlas of Infrared Spectra of Drugs (1990 edition) including 582 infrared spectra of drug substances was published. For the sake of continuity of this work, the Atlas of Infrared Spectra of Drugs would be published volume by volume.

There were 685 infrared spectra of the drug substances recorded by Grating Infrared Spectrophotometer in the volume Ⅰ published in 1995. The volume Ⅱ published in 2000 included 208 infrared spectra of the drug substances, which were all recorded by Fourier Transform Infrared Spectraphotometer instead. The volume Ⅲ published in 2005 included 210 infrared spectra of the drug substances (172 new spectra and 38 re-recorded ones). The volume Ⅳ published in 2010 consists of

124 infrared spectra of the drug substances. With the exception of particular situations, the corresponding spectra in this Atlas are used as reference spectra of drug substances for identification or purity test required in monographs concerned in the Chinese Pharmacopoeia as well as in other National Pharmaceutical Specifications, which will not include infrared spectrum any more. The other spectra in this Atlas may be used also as reference spectra in pharmaceutical analysis.

In the course of compilation, the group is indebted to the full support in providing the specimens of drug substances and reference materials from various Institutes for Drug Control as well as manufacturers throughout the country.

All comments and suggestions concerning the contents of the Atlas will be welcome and subjected to careful consideration and necessary amendments be made for inclusion in subsequent supplement or next volume.

Pharmacopoeia Commission of P.R. China

说　　明

一、《药品红外光谱集》每卷有三个部分，即说明、光谱图和索引。光谱图系由《中华人民共和国药典》、国家药品标准中所收载的药品，用红外光谱仪录制而得。每幅光谱图并记载该药品的中文名、英文名、结构式、分子式、光谱号及试样的制备方法等。

索引有中文名索引、英文名索引、分子式索引，索引中列出的数字系指光谱号。

二、红外光谱仪

本光谱集第四卷所收载的光谱图系由不同型号的傅立叶红外光谱仪录制，再用伯乐 WIN-IR 软件统一格式化。

三、光谱图的录制

除少数为鉴别药品必需的有关化合物外，本光谱集所收载的药品，均符合其药品质量标准的规定。

试样的制备

1．压片法　取供试品约 1mg，置玛瑙研钵中，加入干燥的溴化钾或氯化钾细粉约 200mg，充分研磨混匀，移置于直径为 13mm 的压模中，使铺布均匀，抽真空约 2min 后，加压至 0.8 ～ 1GPa，保持 2 ～ 5min，除去真空，取出制成的供试片，目视检查应均匀透明，无明显颗粒（也可采用其它直径的压模制片，样品与分散剂的用量可相应调整以制得浓度合适的片子）。将供试片置于仪器的样品光路中，并扣除用同法制成的空白溴化钾或氯化钾片的背景，录制光谱图。

对溴化钾或氯化钾的质量要求　用溴化钾或氯化钾制成空白片，录制光谱图，基线应大于 75%透光率；除在 3440cm^{-1} 及 1630cm^{-1} 附近因残留或附着水而呈现一定的吸收峰外，其他区域不应出现大于基线 3%透光率的吸收谱带。

2．糊法　取供试品约 5mg，置玛瑙研钵中，滴加少量液状石蜡或其他适宜的液体，制成均匀的糊状物，取适量夹于两个溴化钾片（每片重约 150mg）之间，作为供试片；以溴化钾约 300mg 制成空白片作为背景补偿，录制光谱图。亦可用其他适宜的盐片夹持糊状物。

3．膜法　参照上述糊法所述的方法，将液体供试品铺展于溴化钾片或其他适宜的盐片中录制；或将供试品置于适宜的液体池内录制光谱图。若供试品为高分子聚合物，可先制成适宜厚度的薄膜，然后置样品光路中测定。

4．溶液法　将供试品溶于适宜的溶剂内，制成 1%～ 10%浓度的溶液，置于 0.1 ～ 0.5mm 厚度的液体池中录制光谱图，并以相同厚度装有同一溶剂的液体池作为背景补偿。

5．衰减全反射法　将供试品均匀地铺展在衰减全反射棱镜的底面上，使紧密接触，依法录制反射光谱图。

6．气体法　采用光路长度约为 10cm 的气体池，首先将气体池抽真空，然后充以适当压力（例如 30 ～ 50mmHg）的供试气体，录制光谱图。

制图

本卷中光谱图的横坐标为波数（cm^{-1}），纵坐标为透光率（*T*%）。

本卷收载的光谱图，系用分辨率为 $2cm^{-1}$ 条件绘制，基线一般控制在 90%透光率以上，供试品取量一般控制在使其最强吸收峰在 10%透光率以下。

四、光谱图的使用

1．凡《中华人民共和国药典》、国家药品标准已收载用红外光谱法作为鉴别的原料药，本卷中收载的相应光谱图供比对用。

2．本卷光谱图的波数范围为 4000 ～ $400cm^{-1}$，但有的红外光谱仪的光谱录制范围不同，用此类仪器录制的光谱图，除另有规定外，亦可使用本卷所收载的光谱图中相应的波数区间比对。所用仪器的性能应符合《中华人民共和国药典》附录Ⅳ C 红外分光光度法项下的要求。

3．固体药品在测定时，可能由于晶型的影响，致使录制的光谱图与本卷所收载的光谱图不一致，遇此情况，应按本卷中各相应光谱图中备注的方法或该品种正文中规定的方法进行预处理后，再行录制。

4．采用压片法时，影响图谱形状的因素较多，使用本卷对照时，应注意供试片的制备条件对图谱形状及各谱带的相对吸收强度可能产生的影响。

压片时，若样品（盐酸盐）与溴化钾之间不发生离子交换反应，则采用溴化钾作为制片基质。否则，盐酸盐样品制片时必须使用氯化钾基质。

5．常用的傅立叶变换红外光谱仪系单光束型仪器。因此，应注意二氧化碳和水汽等的大气干扰，必要时，应采取适当措施（如采用干燥氮气吹扫）予以改善。

6．为便于光谱的比对，本卷收载了聚苯乙烯薄膜的光谱图。在比对所测药品的光谱图与本卷所收载的药品的光谱图时，宜首先在测定药品所用的仪器上录制聚苯乙烯薄膜的光谱图，与本卷收载的聚苯乙烯薄膜的光谱图加以比较，由于仪器间的分辨率存在差异及不同操作条件的影响，聚苯乙烯薄膜光谱图的比较，将有助于药品光谱图比对时的判断。

7．由于图谱的质量或供试品的多晶型等原因，有些化合物的光谱图作了重新绘制，并收入后续卷中。若同一化合物的光谱图在不同卷中均有收载，用于鉴别时以后卷光谱图作为比对依据，前卷光谱图仅作为参考。

NOTICES

Ⅰ. Each volume, the Atlas of Infrared Spectra of Drugs, consists of three parts: notices, spectra and indexes. The spectra were recorded using an infrared spectrophotometer from the drug substances described in the Chinese Pharmacopoeia and National Pharmaceutical Specifications promulgated by the State Food and Drug Administration. Under each spectrum are described both Chinese and English generic names, structural and molecular formulas, spectrum number and preparation method for sample of the drug substance concerned.

Indexes are arranged in Chinese titles, English titles as well as molecular formulas of the drug substances, respectively. The numeral listed in the index indicates the spectrum number.

Ⅱ. Infrared spectrophotometer

The spectra in Volume Ⅳ were recorded using various models of Fourier Transform Infrared Spectrophotometers and all these digital raw IR data from various FTIRs were processed with Bio-Rad WIN-IR software to achieve the format unification of spectra.

Ⅲ. Recording of spectra

In Volume Ⅳ, all drug substances used for recording the spectra comply with their requirements described in the monographs concerned with the exception of a few related compounds which are necessary in identification test of certain drugs.

Procedures for preparation of samples

1. Disc Method

Triturate about 1mg of the substance being examined with approximate 200 mg of dried, finely powdered potassium bromide or potassium chloride in an agate mortar. Grind the mixture thoroughly and spread it uniformly in a die of 13 mm in diameter. Compress the mixture under vacuum with a pressure applied to the die of 0.8~1 GPa for 2 to 5 minutes, after the die assembly has been evacuated about 2 minutes. Remove the vacuum and take off the disc. The resultant disc should be uniform transparent and free from any obvious particles by visual inspection. When and if the die of other diameters is used, the dosages of sample and dispersive reagent should be adjusted accordingly to prepare the disc with suitable concentration. Mount the disc in a suitable holder and place it into the sample beam of the spectrophotometer.

Place a similarly prepared blank disc of potassium bromide or potassium chloride into the sample beam for background compensation. Record the spectrum with background deducted.

Quality Requirement for potassium bromide or potassium chloride Record the spectrum of a blank disc of potassium bromide or potassium chloride prepared as described above. The spectrum has a substantially flat baseline exhibiting no maxima with an absorbance greater than 3% of transmittance above the baseline, with the exception of maxima due to residual or absorbed water at about 3440 cm^{-1} and 1630 cm^{-1}. The baseline should be more than 75% of transmittance.

2. Mull method

Triturate about 5 mg of the substance being examined with a little amount of liquid paraffin or other suitable liquid to give a homogeneous creamy paste in an agate mortar. Compress and hold a portion of the mull between two flat potassium bromide plates (about 150 mg each). Record the spectrum by using a blank disc of potassium bromide with about 300 mg in weight for background compensation. Other suitable salt plates may be used instead of potassium bromide plates.

3. Film method

Use a capillary film of the liquid substance being examined held between two potassium bromide plates or other suitable salt plates with the method as described in the mull method. A filled cell of suitable thickness may be also used. For high polymer, prepare a film with suitable thickness. Mount the film in a suitable holder and place it into the sample beam. Record the spectrum.

4. Solution method

Prepare a solution of the substance being examined in a suitable solvent to the concentrations of 1%~10%. Place the solution in a filled cell with a thickness of 0.1 to 0.5 mm. Record the spectrum when a matched cell filled with the same solvent as background.

5. ATR method

Place the substance being examined in a manner of homogeneous and close contact with an ATR (Attenuated Total Reflectance) prism, and record its reflectance spectrum.

6. Gas method

Examine gases in a cell with optical path length of about 10cm. Evacuate the cell and fill the gas being examined to a suitable pressure (for example, 30~50mmHg). Record the spectrum.

Spectrum recording

The linear abscissa of the spectrum shows wave number (cm^{-1}) and the ordinate of the spectrum indicates transmittance (T %).

The spectra were recorded at 2 cm^{-1} resolution. In general, the baseline in spectrum was controlled to be more than 90% transmittance and the transmittance of the strongest absorbance peak was controlled to be less than 10% by appropriately adjusting the quantity of substance being examined.

Ⅳ. Uses of the spectra

1. The corresponding spectra in this Atlas are used as reference spectra for drug substance when the identification by the use of infrared spectrophotometry is required in monographs of the Chinese Pharmacopoeia and National Pharmaceutical Specifications promulgated by the State Food and Drug Administration.

2. In volume Ⅳ, the spectrum was scanned in the range from 4000 cm^{-1} to 400 cm^{-1}. However, the spectrum recorded on different models of infrared spectrophotometer, which may have different scanning range, can be compared with the relevant spectrum included in this volume within the corresponding spectrum region. Of course, the performance of the instrument used should meet the requirements of *Infrared Spectrophotometry* as described in the appendix Ⅳ C of the Chinese Pharmacopoeia.

3. Due to polymorphism, the difference between the spectrum recorded from the substance being examined and the relevant spectrum included in this volume may occur. In this case, the preparation method of the substance being examined as described in the note of the spectrum or that described in the monograph should be followed.

4. Various factors may affect the character of spectrum recorded by disc method. Therefore, the possible influence of preparation conditions of disc to the positions and the relative intensities of the absorbance bands should be considered when the spectrum in this volume is used for comparison.

If no ion-exchange reaction happens between the substance (chloride salts) being examined and the matrix when preparing disc, potassium bromide should be used as matrix for all solid specimens. Otherwise, potassium chloride must be used as matrix for chloride salts.

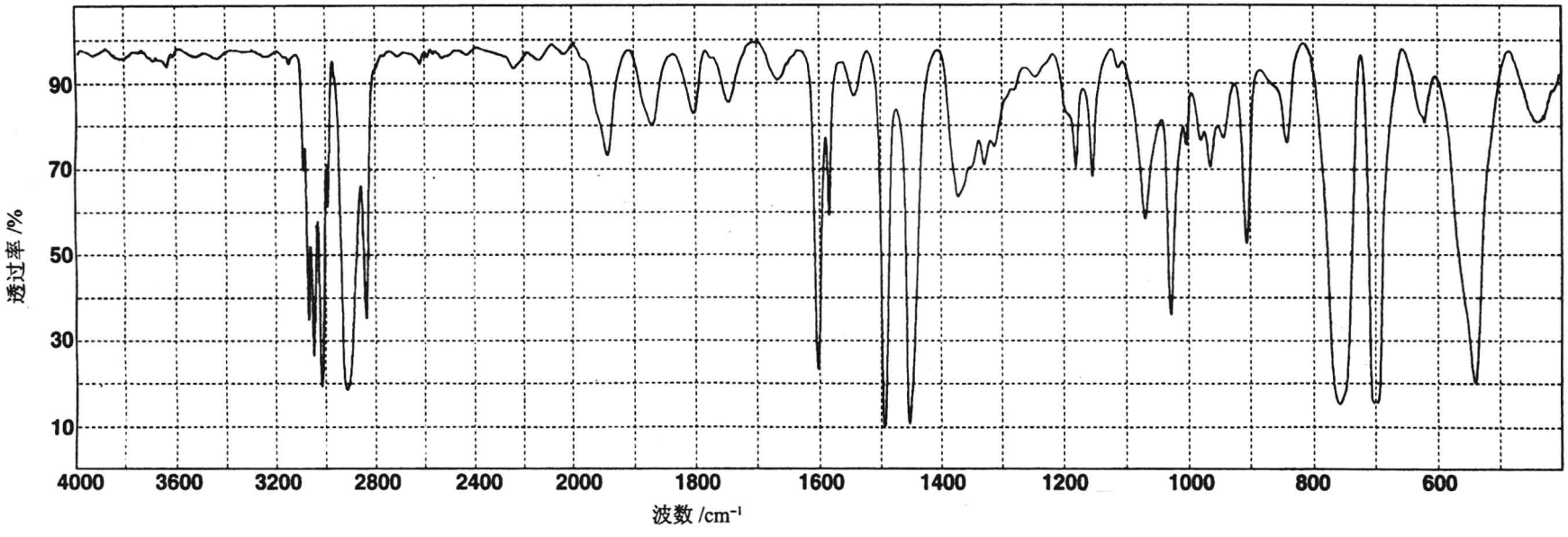
5. Care should be taken to the interference of atmosphere including carbon dioxide and water, because FT-IR spectrophotometer is usually a single beam type instrument. Some suitable measures, such as blow with dried nitrogen, should be adopted if necessary.

6. A spectrum of a polystyrene film is included in this volume for the convenience of comparison. It is suggested that a polystyrene spectrum is recorded on the instrument being used for examination of the substance being examined. Both spectra should be compared at first to observe any possible differences due to the potential variations of resolving power and operating conditions of the instruments being used. With reference to these factors, it would be useful for assessing the concordance of the spectrum of the substance being examined with that of the reference spectrum in this volume.

7. Due to some reasons, such as the quality of spectrum or polymorphism of the substance being examined, the spectra of some compounds were rerecorded and inscrolled in the subsequent volume. If the spectra of some compounds were inscrolled in different volumes, the spectrum inscrolled in latter volume should be used as criteria for identification, and the spectrum in former volume only as reference.

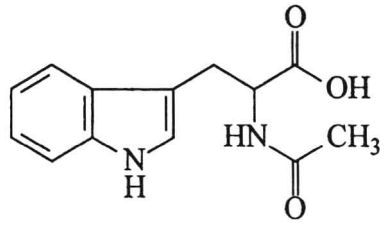
目　　录

聚苯乙烯薄膜标准红外光谱图



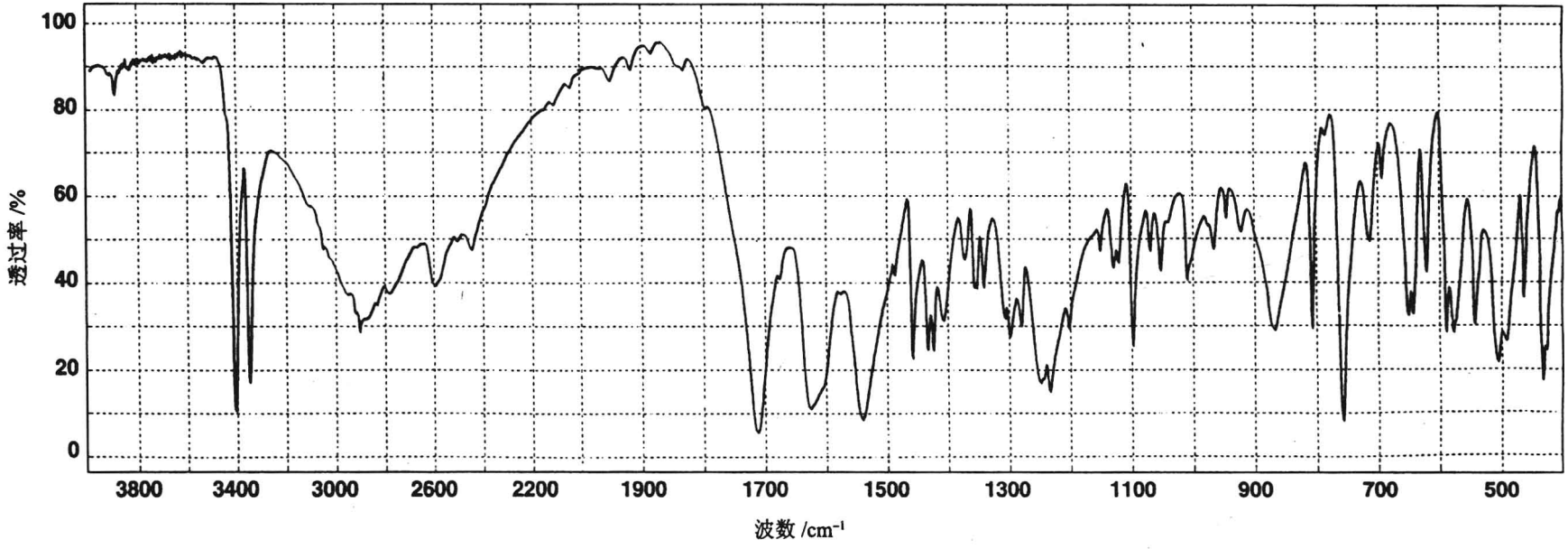
中文名：*N*- 乙酰 -L- 色氨酸

英文名：*N*-Acetyl-L-tryptophan



分子式：$C_{13}H_{14}N_2O_3$

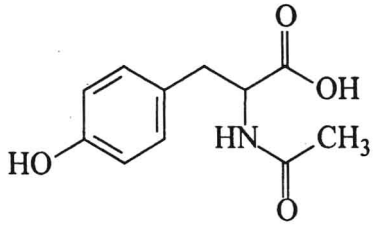
试样制备：KBr 压片法



光谱号 1104

中文名：*N*- 乙酰 -L- 酪氨酸

英文名：*N*-Acetyl-L-tyrosine



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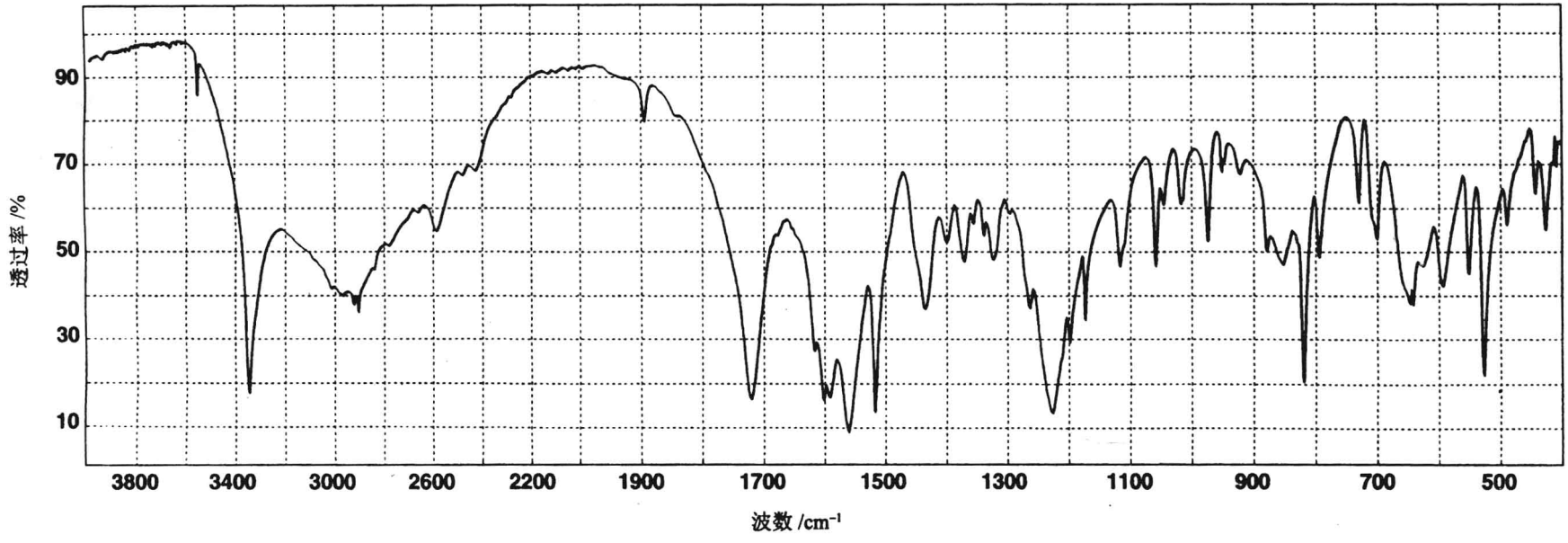
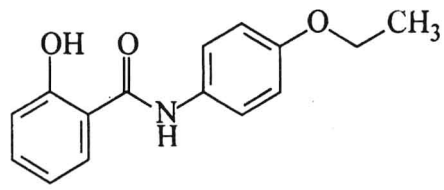
试样制备：KBr 压片法



光谱号 1105

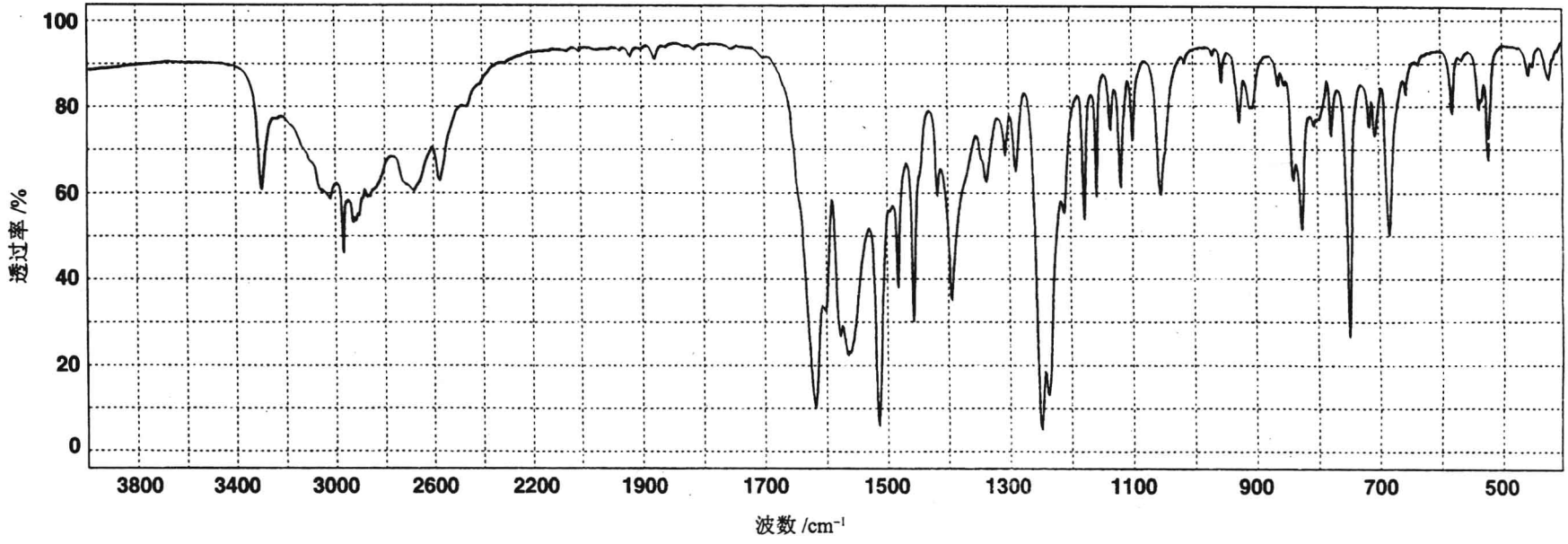
中文名：乙氧苯柳胺

英文名：Etofesalamide



分子式：$C_{15}H_{15}NO_3$

试样制备：KBr 压片法



光谱号 1106