ATLAS OF INFRARED SPECTRA OF DRUGS

药品红外光谱集

中华人民共和国卫生部药典委员会 编

一九九〇年

化学工业出版社

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内 容 提 要

本书收载药品的红外光谱图共582幅，包括《中华人民共和国药典》(1990年版)、卫生部部颁药品标准以及全国各省、市、自治区地方药品标准中收载的各类药品的红外光谱图。今后，《中华人民共和国药典》及《中华人民共和国卫生部药品标准》不再收载红外光谱图，凡以红外光谱法作为鉴别和检查的药品，一律以本书收载的红外光谱图作为对照图谱。本书是上述各药品标准不可缺少的配套用书，对药品分析和检验工作有很大的实用价值。本书也可供有机化学、有机分析专业人员参考。

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一九九〇年

中华人民共和国卫生部

药典委员会 编

责任编辑：潘正安

封面设计：任 辉

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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，作为有机药品的鉴别项目。特别是鉴于有机药品品种不断增加，一般药品化学结构比较复杂或药品化学结构相互之间差异较小，用颜色反应、沉淀、结晶形成或紫外分光光度法等常用方法不足以相互区分，而采用红外光谱法常可有效地解决。

《中国药典》自1977年版开始采用红外光谱法作为一些药品的鉴别，在附录中收载对照图谱。为了适应我国药品质量监督体系的需要，1985年，我会委托一些药品检验所收集录制了国内生产的药品红外光谱图423幅，编制出版了《药品红外光谱集》(1985年)，为药品标准建立红外对照图谱。鉴于《中国药典》1990年版及卫生部药品标准均收载红外光谱法，应用红外光谱方法的品种不断增加，有必要继续工作，扩大收载范围，出版一本供上述各药品标准中需要的对照红外光谱图集。因此，由我会组织湖北省药品检验所(牵头单位)、中国药品生物制品检定所和湖南、上海、天津、辽宁、黑龙江等省市药品检验所组成协作组，负责绘制和审定图谱，以《药品红外光谱集》(1985年)为基础进行补充和修订，编制了本光谱集。完成本光谱集所需的样品和资料得到了全国各省、市、自治区药品检验所的大力支持。

本光谱集收载药品的红外光谱图共582幅。凡在《中国药典》及卫生部药品标准已收载作为鉴别和检查的药品，本光谱集中相应的光谱图用作对照图谱，《中国药典》及卫生部药品标准中不再收载红外光谱图。其他光谱图可供在药品检验中对照参考。

今后药典、卫生部药品标准改版，但《药品红外光谱集》不一定改版，根据药品品种的增加以及在使用中发现本光谱集的不妥之处，将不定期出版增补本。希望各有关单位在实践过程中及时提出宝贵意见，以便在增补本中予以修订补充。

卫生部药典委员会

1989年8月

PREFACE

Infrared spectroscopy has been widely used in analysis of organic compounds. Due to the high specificity of the infrared spectrum of a compound, infrared spectroscopy is commonly adopted in pharmaceutical analysis in conjunction with the other physical and chemical methods in the test for identification of organic drug substances. As the number of organic drug substances, in particular, increases continuously and their chemical structures are generally complex or slightly different in case of their analogs. The usual methods for identification such as color reactions, precipitation, crystal tests or ultraviolet spectrophotometry, etc. are inadquate to differenciate the drug substances with closely related structures. On the contrary, tests based on infrared spectroscopy are always found to be effective for solving the problem

Tests for identification of some drug substances based on infrared spectroscopy were introduced for the first time into the Chinese Pharmacopoeia, 1977 edition, and the infrared reference spectra were included in the appendix. In 1985, the Pharmacopoeia Commission entrusted some institutes for drug control to compile and publish the Atlas of Infrared Spectra of Drugs (1985) in which infrared spectra for 423 drug substances supplied by domestic manufacturers were recorded with a purpose of establishment of infrared reference spectra being adopted in specifications of pharmaceutical substances in the drug quality testing scheme in China. Since the infrared spectrophotometry will be adopted in the Chinese Pharmacopoeia 1990 edition as well as in the pharmaceutical specfications promulgated by the Ministry of Public Health and the number of specifications with tests based on infrared spectroscopy is increasing steadily, there is a demand for further extension of this work. The result is to be officially published as a companion volume of infrared reference spectra to meet the requirements of the above mentioned monographs. Therefore, the Commission organized a collaborative study group, composed of scientists from Hubei Institute for Drug Control (the leading member), National Institute for the Control of Pharmaceutical and Biological Products, Hunan, Shanghai, Tianjin, Liaoning and Heilongjiang Institutes for Drug Control, to carry out the work for recording, examining, verifying and compiling the spectra. The work was performed, revised and amended on the basis of the 1985 edition of the Atlas. In the course of compilation, the group enjoyed the full support in providing the specimens of drug substances and reference materials from various Institutes for Drug Control throughout the country.

There are 582 infrared spectra of the drug substances included in this volume. The corresponding spectra in this volume are used

as reference spectra for drug substances in testing for identification or purity required in monographs concerned in the Chinese Pharmacopoeia as well as the pharmaceutical specfications promulgated by the Ministry of Public Health, and no infrared spectrum will further be included in the Pharmacopoeia and the pharmaceutical specifications promulgated by the Ministry of Public Health thereafter. The other spectra in this volume may be used also as reference spectra in pharmaceutical analysis

The Chinese Pharmacopoeia and the pharmaceutical specifications promulgated by the Ministry of Public Health will be revised routinely and periodically, but this volume, Atlas of Infrared Spectra of Drugs (1990), may not be revised concomitantly. Supplement to this volume will be published as required to cover the increasing number of drug substances and for correcting any discrepancies found in use. All comments and suggestions concerning the contents of this volume will be welcome and subjected to careful consideration and necessary amendments be made for inclusion in subsequent supplements

Pharmacopoeia Commission of
the Ministry of Public Health,
the People's Republic of China

说　　明

一、《药品红外光谱集》有三个部分，即说明、光谱图和索引。光谱图系由《中国药典》、卫生部药品标准和各省、自治区、直辖市药品标准中所收载的药品，用红外分光光度计录制而得。每幅光谱图并记载该药品的中文名、拉丁名、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、拉丁名索引、分子式索引，索引中列出的数字系指光谱号。

二、红外分光光度计

本光谱集所收载的光谱图系在光栅型Perkin-Elmer 77及99系列和上海分析仪器厂4010型仪器上录制而得。

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，压模与真空泵相连，抽气约2分钟后，加压至0.8～1×10^{6}kPa(约8～10t·cm^{-2})，保持2～5分钟，除去真空，取出制成的供试片，用目视检查应均匀，无明显颗粒。将供试片置于仪器的样品光路中，另在参比光路中置一按同法制成的空白溴化钾或氯化钾片作为补偿，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，以空气作参比，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，取适量糊状物夹于两个溴化钾片（每片重约150mg)之间，作为供试片；以溴化钾约300mg制成空白片作为补偿，录制光谱图。亦可用其他适宜的盐片压夹糊状物。

3．膜　法

参照上述糊法所述的方法，将液体供试品夹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。

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。录制时应在参比光路中置一和试

样池配对的装有溶剂的液体池作为补偿。

制　　图

本光谱集中光谱图的线性横座标为波数(cm^{-1})，同时标出相应的波长(μm)，光谱图的纵座标为透光率(T%)。

本光谱集收载的光谱图，录制的扫描速度为12～15分钟，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《中国药典》、卫生部药品标准已收载用红外光谱法作为鉴别的药品，本光谱集中相应的光谱图供作对照用。

其他光谱图亦可作《中国药典》、卫生部药品标准和各省、自治区、直辖市地方药品标准等有关药品鉴别的对照。

2．本光谱集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，亦可使用本光谱集所收载的光谱图中相应的波数区间对照。所用仪器的性能应符合《中国药典》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光谱集所收载的光谱图不一致，遇此情况时，应按本光谱集中各相应光谱图中备注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，因此，使用本光谱集对照时，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。

为避免压片时可能发生的离子交换现象，本光谱集所收载的各盐酸盐品种均采用氯化钾压片法。

5．为了方便对照，本光谱集收载了在本说明第二部分所述的仪器上录制的聚苯乙烯薄膜的光谱图。在对照所测药品的光谱图与本光谱集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，与本光谱集所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（例如狭缝程序、扫描速度等）的影响，聚苯乙烯薄膜光谱图的比较，将有助于药品光谱图对照的判断。

Notices

Ⅰ. This volume, Atlas of Infrared spectra of Drugs, consists of three parts: notices, spectra and indexes. The spectra were recorded on an infrared spectrophotometer from the drug substances described in the Pharmacopoeia of the People's Republic of China, pharmaceutical specifications promulgated by the Ministry of Public Health and pharmaceutical specifications promulgated by authorities of provinces, autonomous regions or Municipalities. Under each spectrum it has been described also both Chinese and Latin titles, structural and molecular formulas, spectrum number and method of preparation of sample of the drug substance concerned.

Indexes are arranged in Chinese titles, Latin 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 this volume were recorded on the instruments of the grating type spectrophotometers of the Perkin-Elmer 77 and 99 series as well as the 4010 model of Shanghai Analytical Instrument Factory

Ⅲ. Recording of spectra

In this Volume, 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 test for identity of certain drugs

Procedures for preparation of samples

1. Disc Method

Triturate about 1 mg of the substance being examined with approximately 200 mg of dried, finely powdered potassium bromide or potassium chloride in an agate mortar. Grind the mixture thoroughly. Spread it uniformly in a die of 13 mm in diameter. Connect the die with a vacuum pump. Compress the mixture under vacuum at a pressure of $0.8-1\times10^{6}$ kPa(about 8–10 $t\cdot cm^{-2}$) for 2–5 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 and free from any obvious particles by visual inspection. Mount the disc in a suitable holder and place it in the sample beam of the spectrophotometer. Place a similarly prepared blank potassium bromide or potassium chloride disc in the reference beam for compensation. Record the spectrum.

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 against air as reference. 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 3440 and 1630 cm^{-1}. The baseline should be not less than 75% of transmittance.

2. Mull method

Triturate about 5 mg of the substance being examined with the minimum amount of liquid paraffin or other suitable liquid to give a smooth creamy paste in an agate mortar. Compress a portion of the mull between two flat potassium bromide plates (about 150 mg each). Record the spectrum by using a blank potassium bromide disc of 300 mg in weight for 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. Record the spectrum. A filled cell of suitable thickness may be also used.

4. Solution method

Prepare a solution of the substance being examined in a suitable solvent to concentrations of 1–10%. Place the solution in a filled cell with a path-length of 0.1–0.5mm. Record the spectrum when a matched cell filled with the same solvent is placed in the reference beam for compensation.

Spectrum recording

The linear abscissa of the spectrum shows wavenumber (cm^{-1}) and the corresponding wavelenth (μm) is also indicated. The ordinate of the spectrum shows transmittance (T%).

The spectra were recorded at a scanning speed of 12–15 minutes. 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 volume are used as reference spectra for drug substance as an infrared spectiophotometric identification is required in monographs of the Chinese Pharmacopoeia and pharmaceutical specifications promulgated by the Ministry of Public Health.

The other spectra may be used as reference spectra for testing or verification of identification of drug substances included in the

Chinese Pharmacopoeia, pharm aceutical specifications promulgated by the Ministry of Public Health and the pharmaceutical specifications promulgated by authorities of the provinces, autonomous regions or municipalities.

2. In this volume, the spectrum was scanned in the range from 4000–400cm^{-1}. However, the spectrum recorded on various models of infrared spectrometer, 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 resolution performance of the instrument used should meet the requirements as described in *Spectrophotometry* of the Chinese Pharmacopoeia.

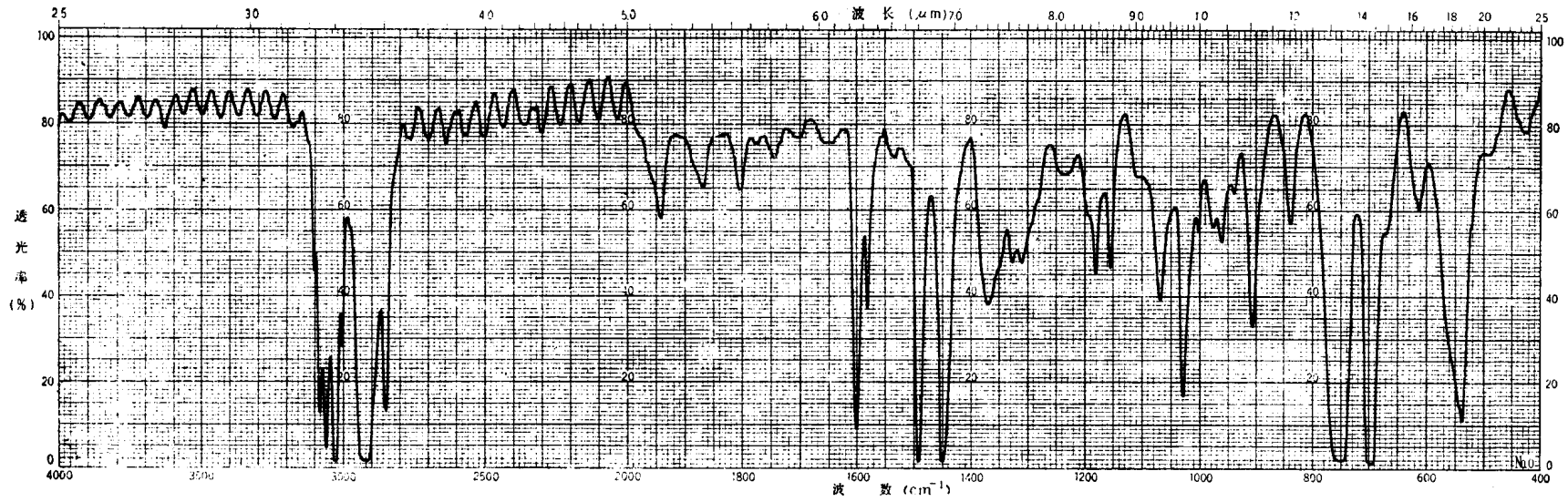
3. Due to polymorphism, difference between the spectrum recorded from the substance being examined and relevant spectrum included in this volume may occur. In this case, the method of preparation of the substance being examined as described in note of the spectrum should be followed.

4. Various factors may affect the character of spectrum recorded by disc method. Therefore, the possible influence of conditions for preparation 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.

In this volume, potassium chloride was used to avoid the possible ion-exchange when spectra of chloride salts were recorded by disc method.

5. A spectrum of a polystyrene film recorded on the spectrophotometer described in paragraph Ⅱ 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 (i. e. slit programme, scanning speed, etc.) 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.

聚苯乙烯薄膜标准红外光谱图

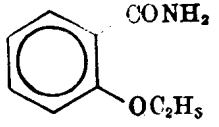


目　　录

中文名：乙水杨胺

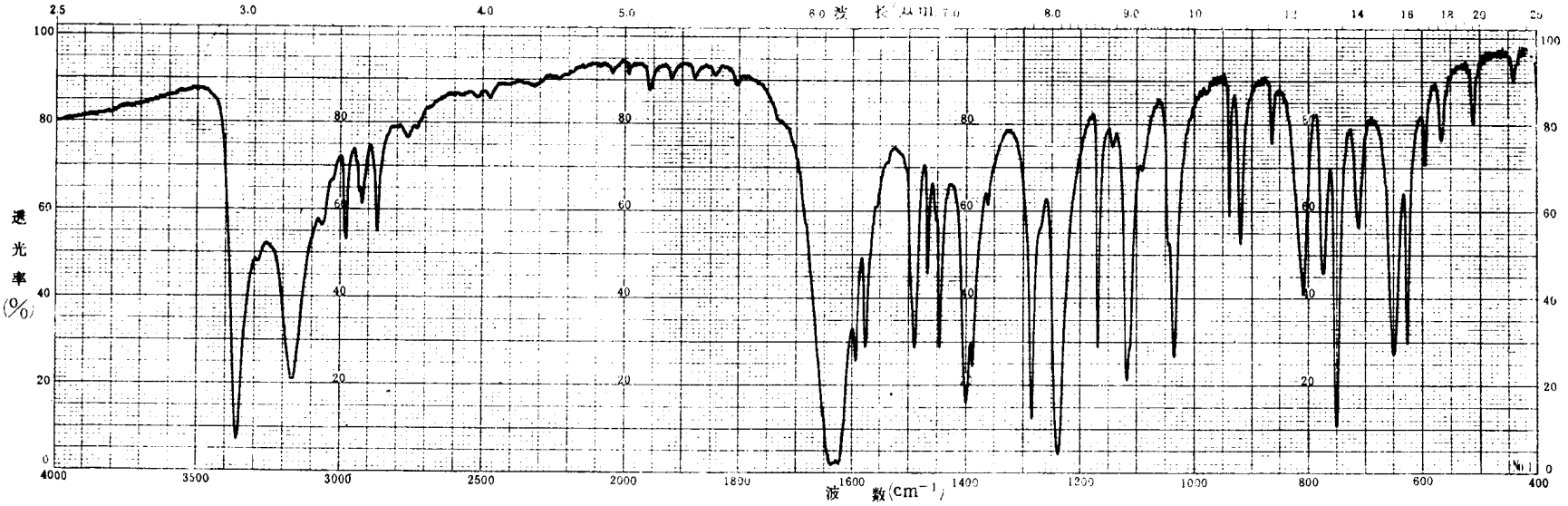
（乙氧苯酰胺，止痛灵）

拉丁名：Ethenzamidum



分子式：$C_9H_{11}NO_2$

试样制备：KBr压片法

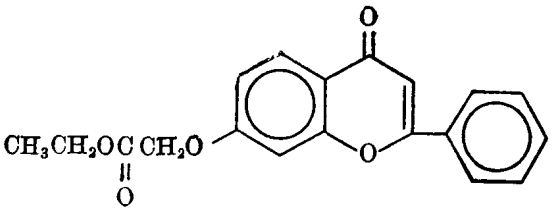


光谱号 1

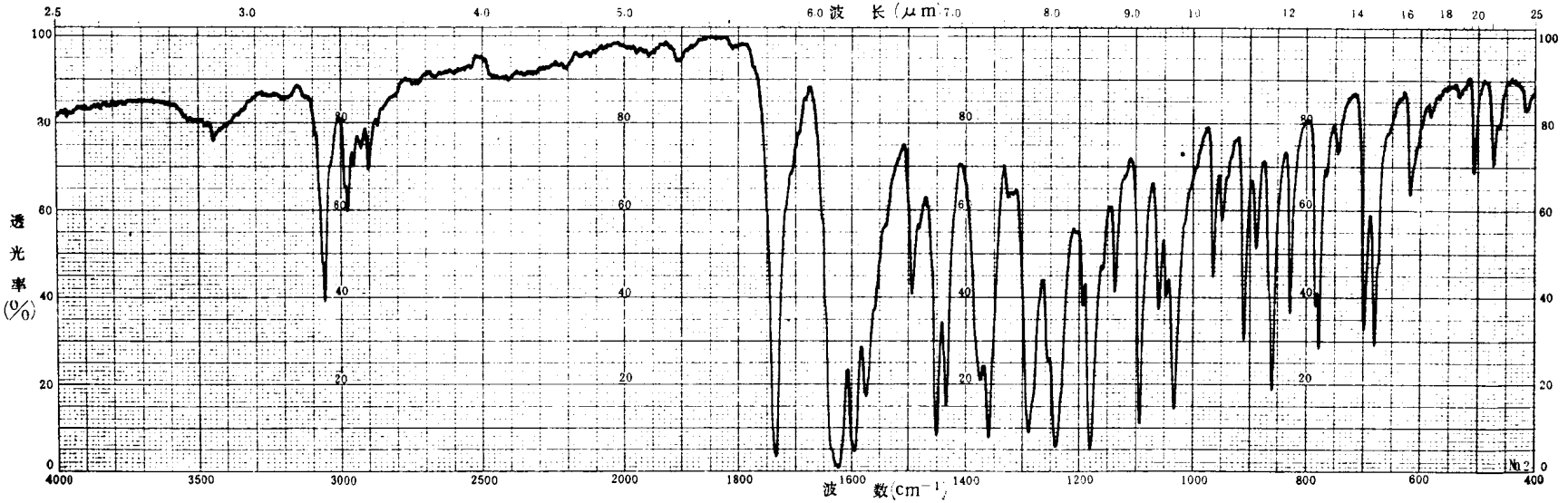
中文名：乙氧黄酮
（舒心酮）

拉丁名：Efloxatum

分子式：$C_{19}H_{16}O_5$



试样制备：KBr压片法

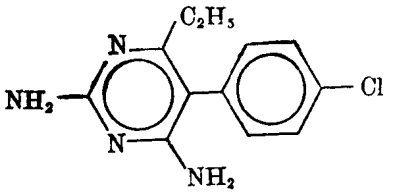


光谱号 2

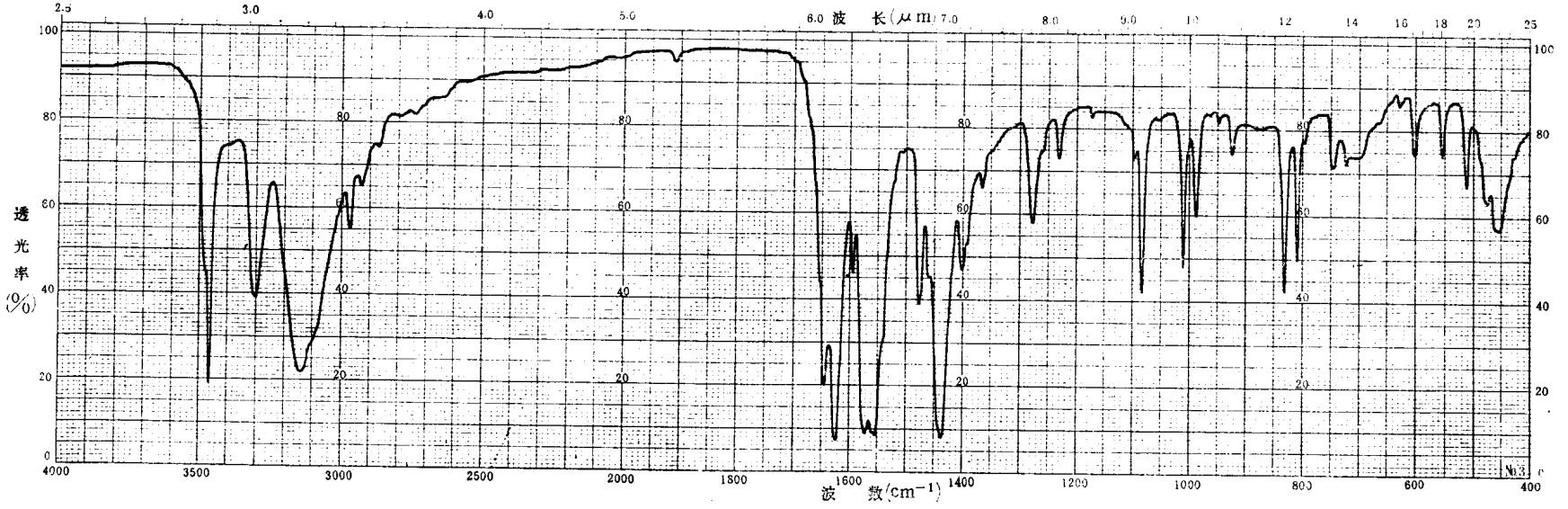
中文名：乙胺嘧啶

拉丁名：Pyrimethaminum

分子式：$C_{12}H_{13}ClN_4$

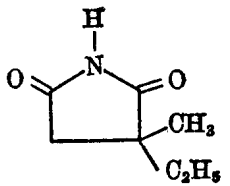


试样制备：KBr压片法



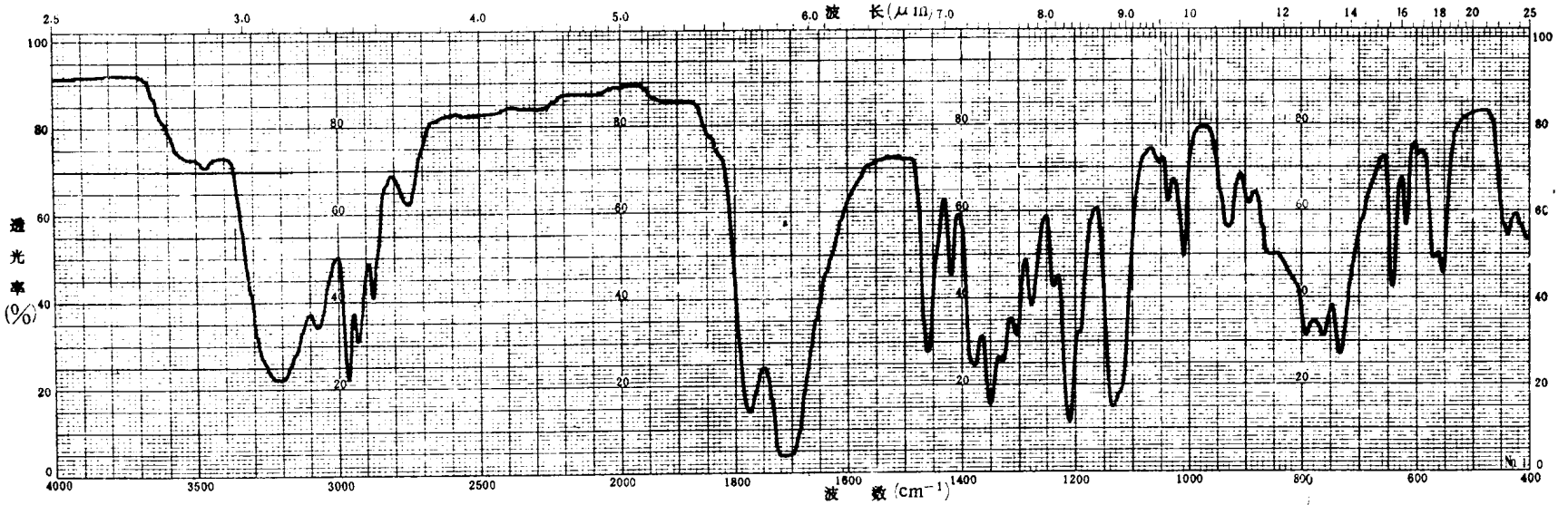
中文名：乙琥胺

拉丁名：Ethosuximidum



分子式：$C_7H_{11}NO_2$

试样制备：KBr压片法



光谱号 4