

顶空气相色谱法测定乙氧基含量

Determination of the content of B oxygen by Headspace Gas Chromatography

Ethyl cellulose (ethylcellulose) as a pharmaceutical excipient, can be used as drug matrix, coating material, carrier, adhesive and capsule wall material, and so on, the quality standard were received contained in the 2005 edition of the Pharmacopoeia of the people's Republic of China, the United States Pharmacopoeia version 30, 2007 edition of the British Pharmacopoeia and European Pharmacopoeia version 5 [2~5]. The 2005 edition of "Chinese Pharmacopoeia" of the people's Republic of China Annex VII G ethyl cellulose.

Ethyl cellulose (EC) is a thermoplastic nonionic cellulose ether, soluble in various organic solvents, at low temperatures can still retaining the with adhesive, filling and film good flexibility. EC can be used for synthetic plastics, paint, ink, insulating materials such as. Due to lack of toxicity in EC, is applied more and more widely in the medicinal materials and food packaging. Ethoxy in ethylcellulose product content of product.

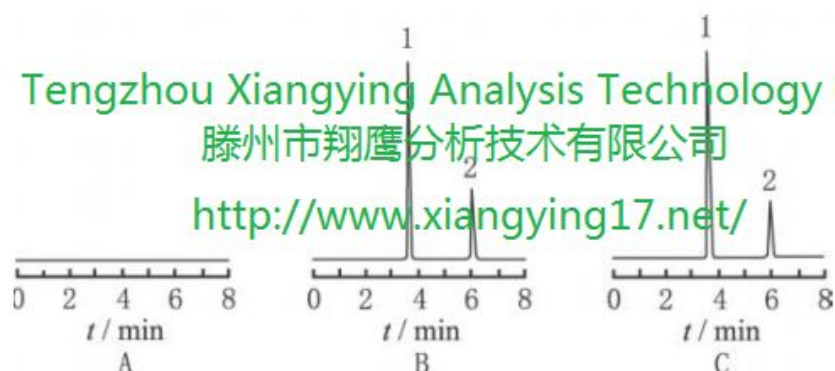
Tengzhou Xiangying Analysis Technology Co. Ltd. according to the shortages of existing methods for determination of ethoxy ethyl cellulose, a gas chromatography with XiangyingGC7990Plus headspace (HS-GC) determination of ethyl cellulose ethoxyl content method in the vial through hydroiodic acid and ethyl cellulose reaction, the ethoxy ethyl iodide into the reaction. After adding lye and excess hydrogen iodate to prevent corrosion of the injector and the column, followed by headspace gas chromatographic analysis using toluene as internal standard, determination of iodine ethane content by internal standard method, and calculated the ethoxyl content of ethyl cellulose was studied. The hydrolysis temperature and time, headspace sampler in the balance of temperature and time, matrix effect and other factors impact on the determination results showed that the hydrolysis temperature at 140 to 150 DEG C, the temperature changed little, 1.0h can complete hydrolysis reaction ; headspace vial sample in 80 DEG C after 30 min can achieve gas-liquid equilibrium; adding cellulose can eliminate matrix effects and iodine ethane concentration and peak area showed a good linear relationship. In this paper also studied the the method reproducibility and recovery rate, found that the reproducibility of the method better, ethoxy content determination results of the relative standard deviation was 2.83%, recovery rate of 99.6%. This method and the chemical titration method, be consistent with the determination results of samples.

乙基纤维素(ethylcellulose)作为药物辅料,可用作药物骨架、包衣材料、载体、黏合剂和囊材等,质量标准分别收载于《中华人民共和国药典》2005年版、《美国药典》30版、《英国药典》2007年版和《欧洲药典》5版[2~5]。2005年版《中华人民共和国药典》附录VII G 将乙基纤维素。

乙基纤维素(EC)是一种热塑性的非离子型纤维素醚,可溶于多种有机溶剂,在低温下仍能保持良好的挠曲性。EC具有粘合、填充和成膜作用,可用于合成塑料、涂料、油墨、绝缘材料等。由于EC无毒,在药用辅料和食品包装中的应用越来越广泛。乙基纤维素产品中乙氧

基的含量对产品的性。

滕州市翔鹰分析技术有限公司针对现有乙基纤维素乙氧基测定方法的不足,提出一种用XiangyingGC7990Plus 顶空气相色谱(HS-GC)测定乙基纤维素乙氧基含量的方法——在顶空瓶中通过氢碘酸与乙基纤维素反应,使乙氧基转化为碘乙烷,反应结束后加碱液中和过量氢碘酸防止腐蚀进样器和色谱柱,随后顶空进样进行气相色谱分析,以甲苯为内标物,采用内标法测定碘乙烷含量,从而计算得出乙基纤维素乙氧基含量.考察了水解温度和时间、顶空进样器内平衡温度和时间、基质效应等因素对测定结果的影响,结果表明:水解温度在 140~150℃ 范围内时,结果随温度变化不大,1.0h 可完成水解反应;顶空瓶内样品在 80℃ 下经 30min 可达到气液平衡;添加纤维素可消除基质效应,碘乙烷浓度与峰面积呈良好的线性关系.文中还研究了该方法的重现性与加样回收率,发现该方法重现性较好,乙氧基含量测定结果的相对标准偏差为 2.83%,加样回收率达 99.6%.将该方法与化学滴定法比较,可得到一致的样品测定结果.



空白 (A)、对照品 (B) 与样品 (C) GC图谱

1. 内标 (3.5 min); 2. 碘乙烷 (5.9 min)

XiangyingGC7990plus features:

1 excellent performance, competitive price, to meet the customers' choice

2 multi industry adaptability

3 outstanding performance in all kinds of harsh environment

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A variety of sampling ports can be equipped with: packed column inlet, shunt / non shunt capillary inlet,

Hydrogen flame ionization detector (FID)

Maximum use temperature: 400

The minimum detection limit: $-12 = 2.5 \times 10^{-12}$ g/s (n-C16)

Linear range: 107 (+ 10%)

XiangyingGC7990plus 特点:

1. 卓越的性能, 具有竞争力的价格, 满足广大客户的选择

2. 多行业适应性

3. 在各种苛刻环境下均表现出色

进样口

多种进样口可配: 填充柱进样口、分流/不分流毛细管进样口、

氢火焰离子化检测器 (FID)

最高使用温度：400℃

最小检出限： $\leq 2.5 \times 10^{-12}$ g/s (n-C16)

线性范围：107 (±10%)