

植物油中 6 号溶剂残留测定的气相色谱新方法



Statement:

The method of Tengzhou City Xiangying Analysis Technology Co., Ltd. (Chromatographic engineering research and analysis and test center) exclusive research and development with the nature of the patent, protected by the patent law, where the identical, belong to the fake, Xiangying technology will be prosecuted according to law the legal liability of the infringer.

preface

Years of grain and oil testing work enable us to clearly understood: strict implementation of national standards, in accordance with the provisions of the standard procedures and steps were detected, is the bounden duty of every inspection of grain and oil workers. However, in accordance with the people's Republic of China national standard GB5009.37 the edible method for analysis of hygienic standard of plant oil, residual solvent determination steps are performed to detect, often appear some non-human but inevitable problems. Therefore, Tengzhou City Xiangying Analysis Technology Co., Ltd. according to all the year round in the actual testing process encountered some problems, of GB were some amendment and supplement, developed a new approach to the analysis, and achieved good results, special to share with the new and old customers. Deficiencies in the national standard:

1. GB successful sample preparation using N, N - dimethyl acetamide (DMA) is used as the base fluid, (1) and vegetable oil samples at the bottom of the liquid is different, i. e. the operation is not the same conditions,

the existence of more or less error, (2) furthermore, MDEA to use chromatography pure, the price is expensive; otherwise, use the analysis of pure and easy to introduce impurities, increase the complexity of the analysis, (3) boiling point of MDEA, although high, but always more or less volatile, into the chromatographic column, due to the lower column temperature, MDEA residual in the chromatographic column, column efficiency and separation degree, causing unnecessary interference.

One

2 GB in a packed column with DEGS/102. The practice proves that this post is not too high column efficiency, peak shape easy trailing, short service life; therefore Xiangying technology R & D out special packed column xy-6 solvent, solvent peak shape and symmetry, the column efficiency high.

A new method for the improvement of national standard:

(1) without using MDEA, (I) buy city selling pressure oil (solvent-free) as bottom liquid (II) by their own oil as base solution (heated volatile production, no solvent peaks can be, this method, worthy of promotion)
(2) the Tengzhou City Xiangying Analysis Technology Co., Ltd. R & D special packed column xy-6 solvent, solvent peak shape and symmetry, the column efficiency high.

Appendix:

Solvent XY-6 packed column test plant oil example reference spectrum

声明:

本方法系滕州市翔鹰分析技术有限公司(色谱工程研究所和分析与测试中心)独家研发具有专利性质,受专利法保护,凡与此雷同者,均属假冒,翔鹰技术将依法追究侵权者的法律责任。

前言:

多年的粮油检测工作使我们清楚懂得:严格执行国家标准,按标准规定的程序和步骤进行检测,是每一位粮油检测工作者的天职。然而,在按中华人民共和国国家标准(GB5009.37)中《食用植物油卫生标准的分析方法》“残留溶剂”的测定步骤进行检测时,经常出现一些非人为但又不可避免的问题。为此,滕州市翔鹰分析技术有限公司根据常年在实际检测过程中遇到的一些问题,对国标进行了一些修改与补充,研发出新的分析方法,取得了不错的效果,特与广大新老客户分享。

国标中不足之处:

1. 国标中标样配制采用的 N, N-二甲基乙酰胺(简称 DMA)作底液, (1) 这和植物油样品的底液是不同的,即不是同一条件下的操作,或多或少存在着误差, (2) 再者 MDEA 要使用色谱纯,价格昂贵;否则使用分析纯,易引入杂质,增加分析的复杂性, (3) MDEA 沸点虽然高,但总会或多或少的挥发,就入色谱柱,由于柱温较低, MDEA 将残留在色谱柱中,影响柱效及分离度,造成不必要的干

扰。

2. 国标中填充柱采用 DEGS/102, 实践证明这种柱子存在的问题是: 柱效不是太高, 峰形易拖尾, 使用寿命短; 为此翔鹰技术研发出专用填充柱 XY-6 solvent, 溶剂峰形对称, 柱效高。

对国标改进后的新方法:

(1) 无需采用 MDEA, (I) 购买市售压榨油 (无溶剂) 作为底液 (II) 利用企业自身生产油作为底液 (需加热挥发, 无溶剂色谱峰即可, 此种方法, 值得推广)

(2) 采用滕州市翔鹰分析技术有限公司研发的专用填充柱 XY-6 solvent, 溶剂峰形对称, 柱效高。

附录:

XY-6 solvent 填充柱测试植物油实例参考谱图



植物油 6 号溶剂检测专用填充柱

Vegetable oil solvent testing for packed column 6

规格: 1m*3mm (ID)*4mm (OD)

Specifications: 1m*3mm (ID)*4mm (OD)

材质: 不锈钢

The material: Stainless steel

填料: 6 号溶剂专用

Packing: No. 6 solvent is special

目数: 60-80

Mesh: 60-80

柱温: 60° C

Column temperature: 60° C

气化室: 140° C

Gasification chamber: 140° C

检测器: 140° C

The detector: 140° C

载气: N2

The carrier gas: N2

流量: 30ml/min 0.01MPa

Traffic: 30ml/min

进样: 1ml

Sample: 1ml

灵敏度: 1 连接: 柱接头

The sensitivity: 1

使用说明:

出厂前已老化, 使用一段使用一段时间后, 如基线不稳, 请重新老化。

Before delivery has been aging, use for a period of use after a period of time, such as baseline instability, please aging again.

色谱柱入口端接进样器, 另一端不接检测器。

Chromatographic column entry termination sampler, the other end without a detector.

通高纯氮气, 流速 40ml/min, 50° C 初温, 以 5° C/min 程序升温到 150° C, 恒温 12 小时。

High purity nitrogen, 40 ml/min, flow velocity at the beginning of 50 ° C temperature, with 5 ° C/min to 150 ° C, temperature programmed temperature 12 hours.

降温后, 将另一端接到检测器上即可使用。

After cooling, the other end received the detector can be used on.

老化好以后, 应达到基线稳定, 分离良好。

After aging, should reach the baseline is stable, good separation.

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Tengzhou Xiangying Analysis Technology Co. Ltd.

电话：0632-5817578, 13963221227

The phone：0632-5817578, 13963221227

<http://www.xiangying17.net/>