二羟二丁基醚及其手性杂质的毛细管气相色谱法

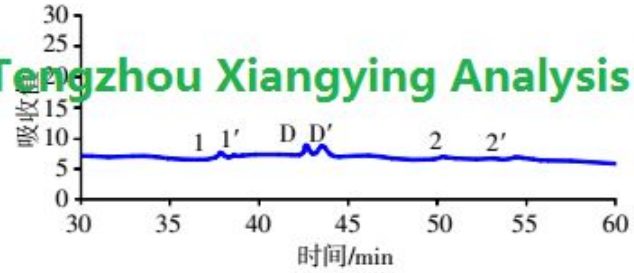
Two - butyl ether and chiral impurities by capillary gas chromatography

Dihydroxy butyl ether (DHDB) belongs to duplex promoters of bile secretion, cholagogue, anti-inflammatory, antispasmodic, hepatoprotective, lipid-lowering, a row of stone, increase the secretion of bile effects, and can be used in many kinds of chronic cholelithiasis and cholecystitis, cholangitis, biliary tract infection treatment is a kind of efficient cholagogue, gallstone dissolution agent and liver protecting drugs, it has attracted more and more attention. However, the DHDB structure of the two chiral carbon and potential.

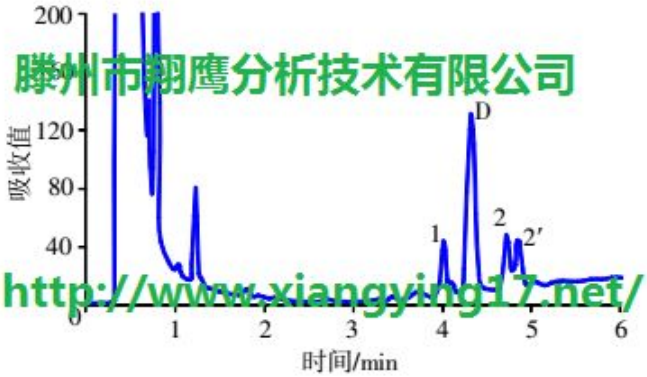
Tengzhou City Xiangying Analysis Technology Co., Ltd. the (CGC) XiangyingGC7990plus capillary column and gas chromatography respectively of dihydroxy butyl ether (DHDB) and chiral impurity separation conditions were investigated and to establish a method for determination of the content of dihydroxy butyl ether (DHDB). (30 m x 0.53 mm x 1.0 m) capillary column chromatography FID detector,, internal standard method, DHDB chiral impurity program heating conditions for initial temperature of 120 DEG C, to 0.5 DEG C / min is heated to 180 DEG C, keeping 40 min; DHDB content was measured by temperature programmed conditions to 140 DEG C for 1 min, 20 C / min up to 200 DEG C. DHDB and its isomer can be completely separated; DHDB in 66.70~800.1 mu g / L has a good linear relationship (y=0.007 0x+0.055 4; R2 = 0.999 6). The average recovery rate was 99.54% and repeatability RSD was 0.68%, intermediate precision (RSD) was 0.95%, the average content of the samples is 77.92% (RSD=0.25%). The method is simple and rapid determination of chiral impurity analysis and content for DHDB.

二羟二丁基醚(DHDB)属于胆汁分泌双相促进剂,具有利胆、消炎、解痉、护肝、降脂、排石、增加胆汁分泌的作用,可用于胆道结石及胆囊炎、胆管炎等多种慢性胆道感染的治疗,是一种高效的利胆剂、胆石溶解剂和保肝药,因此倍受关注。然而,DHDB 结构中的两个手性碳和潜在的。

滕州市翔鹰分析技术有限公司采用 XiangyingGC7990plus 毛细管气相色谱法(CGC)分别对二羟二丁基醚(DHDB)及手性杂质的分离条件进行考察,建立二羟二丁基醚(DHDB)含量的测定方法。(30 m×0.53 mm×1.0μm)毛细管柱为色谱柱,FID 为检测器,内标法,DHDB 手性杂质程序升温条件为初温 120℃,以 0.5℃/min升温至180℃,保持40 min;DHDB 含量测定程序升温条件为140℃保持1 min,以 20℃/min 升至 200℃。DHDB 及其手性异构体得到完全分离;DHDB 在 66.70~800.1μg/mL 的范围具有良好的线性关系(y=0.007 0x+0.055 4;r2=0.999 6);平均回收率为 99.54%,重复性 RSD 为 0.68%,中间精密度 RSD 为 0.95%,样品的平均含量为 77.92%(RSD=0.25%)。该方法快速、简便,适于 DHDB 手性杂质分析和含量测定。



(a)二羟二丁基醚和手性杂质色谱图



(b)二羟二丁基醚定量色谱图

D,D′:二羟二丁基醚及手性异构体;1,1′:杂质及手性异构体;
2,2′:杂质及手性异构体。

图 1　二羟二丁基醚定量色谱图和手性杂质色谱图

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
INJ
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*10 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：≤2.5*10 -12 g/s(n-C16)
线性范围：107（±10%）