

CATALYTIC EXCHANGE LABELLING OF GOSSYPOL WITH TRITIUM

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ABSTRACT

Gossypol (8, 8'-dicarboxyaldehyde-1,1', 6,6', 7,7'-hexahydroxy-5,5'-diisopropyl-3,3'-dimethyl-2,2'-binaphthalene) is a constituent of cotton seed. As part of a study of the properties of gossypol, a sample of radioactive material is needed for in vitro experiments. Tritiated gossypol is obtained by catalyzed isotopic exchange in ethyl acetate solution with tritium gas in the presence of PdO/BaSO₄. The specific activity of the tritiated material purified by silicon gel-loaded paper chromatography is 2.2 Ci/mM with radiochemical purity over 95%.

Key words Gossypol, Tritium, Isotopic exchange, Labelled compound.

枸橼酸镓-67注射液放化纯的测定

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关键词 枸橼酸镓-67, 上行纸色层法, 放化纯, R_f 值, 镓放射性同位素。

关于枸橼酸镓-67注射液放化纯度的鉴定, 文献已有报道^[1-11]。Hnatowich^[1,2]比较了几种展开剂对不同形式镓的 R_f 值的影响; Waxmax^[3]用甲醇-水的硅胶薄板色层法对几种枸橼酸镓-67商业产品作了放化纯分析; 日本是采用柠檬酸钠-乙醇展开剂的纸色层分析法^[4]。

本文比较了五种展开剂在中性产品条件下及三种展开剂在不同 pH 值条件下对不同形式镓的纸色层图谱的影响, 试图选择1—2种展开剂作为生产枸橼酸镓-67注射液放化纯的常规鉴定方法。

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实 验 方 法

(1) **展开剂** 试剂为分析纯, 按体积比配制。展开剂的 pH 值用 HCl、NaOH 调节, 用 25 型酸度计测定 pH 值。

(2) **镓制剂** 用加速器生产的 ^{67}Ga , 以 6—7 N HCl 溶靶, 经分离后制成母液, 分别配制成 $^{67}\text{GaCit}$, $^{67}\text{GaCl}_4^-$, $^{67}\text{GaCl}_3$ 溶液。

(3) **色层纸** 新华 No. 1, $2.5 \times 25\text{cm}$ 和 $6.5 \times 25\text{cm}$ 两种规格。

(4) **实验方法** 用微量注射器或毛细管吸取配制的镓制剂 1—5 μl , 滴于色层纸原始线中心, 风干。悬挂于预先用展开剂饱和过的色层缸中, 室温上行展开 12 cm, 取出风干后切成 1 cm 小块, 在 FJ-336 NaI (TI) 探测器上测 γ 放射性强度。在相同条件下做放射性自显影照相进行对比。

实 验 结 果

(1) **不同展开剂对不同形式镓展开的 R_f 值及图谱的影响** 结果示于图 1。

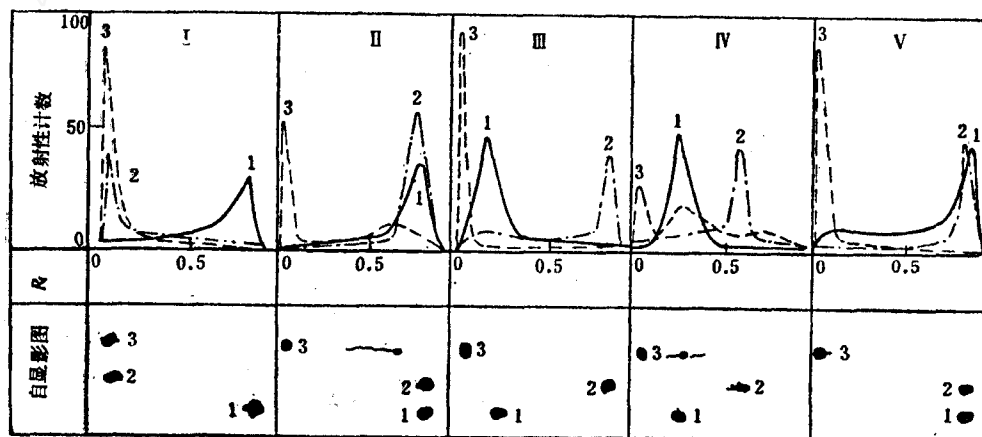


图 1 不同展开剂对不同形式镓-67的展开图谱及自显影照相的影响

展开剂: I —— 吡啶:乙醇:水=1:2:4; II —— 柠檬酸钠 (0.1M):乙醇=5:3; III —— 甲醇:水=85:15;
IV —— 吡啶:正丁醇:冰醋酸:水=8:8:9:4; V —— 乙醇:水:氢氧化氨=200:700:0.35。
1——GaCit; 2——GaCl₄⁻; 3——GaCl₃。

由图看出: 五种展开剂对GaCit展开的 R_f 值分别为0.92、0.84、0.20、0.31、0.88, 除 II 号的 R_f 值比文献值 (0.65)^[4] 稍高外, 其余结果与文献相符。I、III 号展开剂使GaCit的色谱峰与GaCl₄⁻、GaCl₃的明显分开, 故宜用于鉴定枸橼酸镓-67 注射液放化纯。II、IV、V 号展开剂使GaCit的色谱峰与GaCl₄⁻、GaCl₃的色谱峰重叠或部分重叠, 但 II、V 号展开剂有它的优点, 在中性产品条件下, 加过量NaCit时仍可考虑采用。

(2) **展开剂pH值对不同形式镓的展开图谱的影响** 结果示于图 2。

由图看出: 展开剂的 pH 值对不同形式镓的展开图谱有一定影响。

应用 II、V 号展开剂时, GaCit 与 GaCl₄⁻ 的展开峰接近或重合, pH 值的改变对峰的分开影响不大。II 号展开剂的 GaCl₃ 峰, 随 pH 值增大, 与 GaCit、GaCl₄⁻ 的峰分离越明显。pH 值改

变时,对Ⅱ号展开剂的GaCit、GaCl₄⁻、GaCl₃的展开峰影响不突出。

枸橼酸镓-67注射液一般为中性,这时GaCl₃主要以Ga(OH)₃存在, R_f 值在0.83、0.17、0.96,峰可分开。如前所述,过量NaCit存在下的中性产品,可用Ⅱ、Ⅴ号展开剂对GaCit进行放化纯鉴定。

随着pH值增大,GaCit、GaCl₃有不同程度水解,其水解产物[C₃H₄O₂Ga(COOH)₃]²⁺、[C₃H₄O₂Ga(OH)₂(COOH)₃]⁻、[C₃H₄O₂Ga(OH)₂(COO)(COOH)₂]⁻、[C₃H₄O₂Ga(OH)₂(COO)₃]³⁻、Ga(OH)₂⁺、Ga(OH)₃、GaO(OH)、Ga(OH)₄⁻的 R_f 值在0-0.9₆之间,这是展开图谱存在多峰、拖尾的原因。

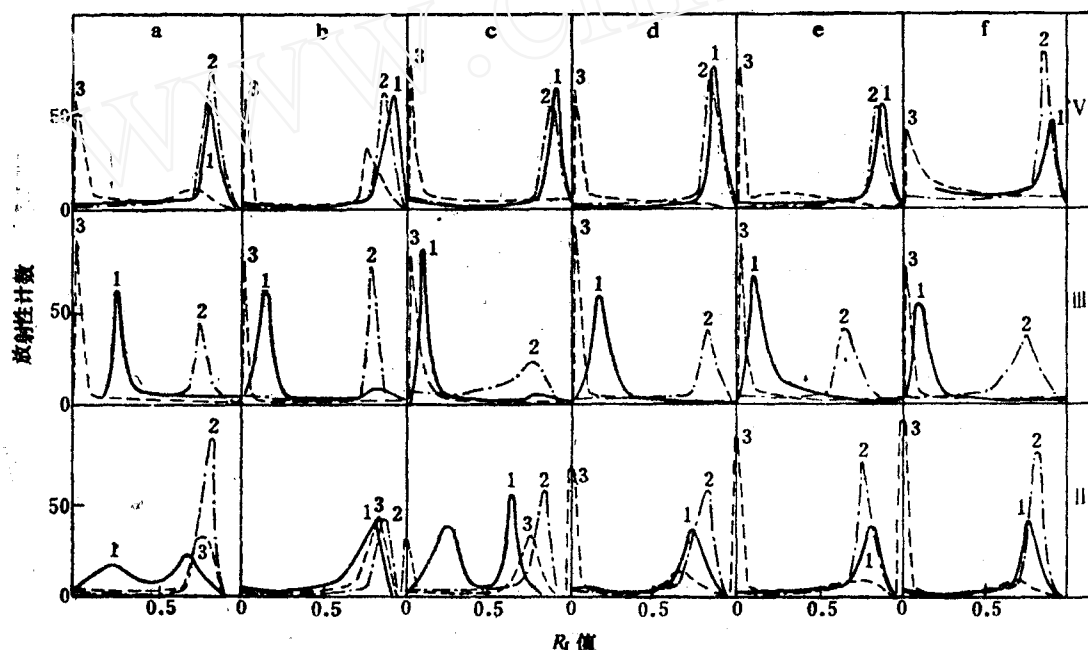


图2 展开剂pH值对不同形式铈的展开图谱的影响

pH值: a—2.5; b—4.8; c—6.2; d—7.2; e—8.4; f—10。
1—GaCit; 2—GaCl₄⁻; 3—GaCl₃。Ⅱ, Ⅲ, Ⅴ——展开剂号。

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ABSTRACT

A technique for the analysis of trace elements in serum is developed using proton-induced X-ray emission. The concentrations of K, Ca, Cr, Mn, Fe, Ni, Cu, Zn, As, Se, Br and Rb in serum of mice, normal and fed gossypol acetic acid, are determined. An obvious increase in the concentration of Cu and the decrease in the concentration of Zn of mice fed with gossypol acetic acid, as compared with those of normal mice, are observed, so that the Cu/Zn ratio of the former is significantly higher than the latter ($P=0.05$). No significant changes in the concentrations of K and Ca are observed, although the concentration of Ca of mice fed with gossypol acetic acid is lower.

Key words Proton-induced X-ray emission, Trace element, Spectrometer scale, Male contraceptive, Gossypol acetic acid.

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METHOD OF DETERMINATION OF RADIOCHEMICAL PURITY OF GALLIUM-67 CITRATE INJECTION

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ABSTRACT

A simple method is used to compare the effect of five developing agents on the radiochemical purity of neutral products of $^{67}\text{GaCit}$ and on R_f values. Two preferable developing agents are recommended as suitable for the identification of $^{67}\text{GaCit}$ injection in its production. The effect of six pH values of different developing agents on radiochemical purity, R_f and chromatogram are compared for the neutral products. The results of the experiments show that the ascending paper chromatography with 1:2:4 pyridine/ethanol/water and 85:15 methanol/water is preferable for the determination of the radiochemical purity of $^{67}\text{GaCit}$. The other developing agents also can be used if there are not any impurities except gallium radioisotopes.

Key words Gallium-67 citrate injection, Ascending paper chromatography, Radiochemical purity, R_f value, Gallium radioisotopes.