

-三芳基锗()-取代丙酸同 1-乙氧基锡杂^噻唑烷的反应

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摘要 利用 -三芳基锗()-取代丙酸同 1-乙氧基锡杂^噻唑烷的 1-位取代反应,合成了 15 个 1-[(-三芳基锗()-丙酰氧基)-2,8,9-三氧杂-5-氮杂-1-锡杂三环[3,3,3,0^{1,5}]-十一烷,并研究该反应规律,利用 IR,NMR 和 MS 表征了该类化合物的结构和质谱裂解机制.生物活性测定表明,它们只对某些细菌如溶血链球菌和金黄葡萄糖球菌有较好的抑制作用.

关键词 三芳基锗,锡杂^噻唑烷,结构表征,杀菌活性

The Reaction of -Trialkylgermanium- ()-substituted Propionic Acid with 1-Ethoxystannatrane

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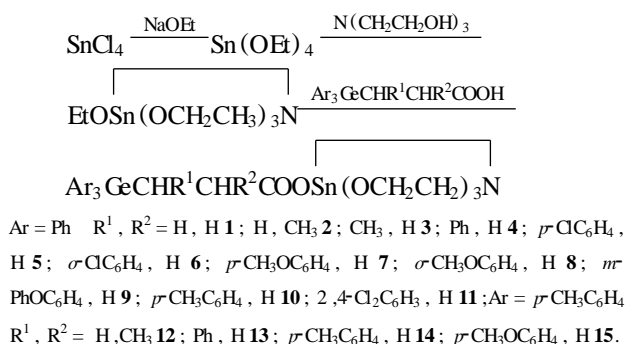
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Abstract The reaction of -trialkylgermanium- ()-substituted propionic acid with 1-ethoxystannatrane was studied and fifteen organometallic compounds containing germanium and tin were synthesized. The procedure of this reaction was described. The structures of prepared compounds were characterized by means of IR, NMR (¹H, ¹¹⁹Sn) and MS. The fragmentation mechanism of MS was proposed. The tests of biological activity showed that they have better inhibition activity only for some bacteria such as *Beta hemolytic strepto coai* and *Staphylococcus aureus*.

Key words trialkylgermanium, stannatrane, structure characterization, antibacterial activity

1-取代-2,8,9-三氧杂-5-氮杂-1-锡杂三环[3,3,3,0^{1,5}]-十一烷(简称 1-取代锡杂^噻唑烷)是一类具有笼状结构的金属有机化合物,由于其稳定性较差,至今报道很少^[1~3],我们曾经报道过 1-芳酰氧基锡杂^噻唑烷的合成及结构研究^[4],确证其是一类既含有 Sn-N 配键,又含有羰基氧同锡原子配位的聚合六配位有机锡化合物.本文以 -三芳基锗()-取代锗丙酸同 1-乙氧基锡杂^噻唑烷进行 1-位反应,研究该反应规律,并且制得 15 个含锗锡的双金属有机化

合物,反应方程式如下:



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1 实验部分

1.1 实验仪器及原料合成

元素分析用 YANACO CHN Corder MT-3 型自动元素分析仪; IR 用 SHIMADZU-IR435 型红外光谱仪, KBr 压片; ^1H , ^{119}Sn NMR 用 JECL FX 90 型或 BRACKER AC-p200 核磁共振仪, CDCl_3 或 $\text{DMSO}-d_6$ 为溶剂, TMS 或 Me_4Sn 作为内标或外标; HP-5988A 型质谱仪, EI 源; 三芳基锗-取代丙酸的合成参照文献[5], 按以下方程式合成:



1.2 目标化合物合成

所用溶剂甲苯、二氯甲烷、石油醚均按标准方法经无水处理, 反应在 schlenk 真空线上 N_2 保护下进行。

取 2.0 mmol $\text{Ar}_3\text{GeCHR}^1\text{CHR}^2\text{COOH}$ 和 2.0 mmol 1-乙氧基锡杂噻唑烷和 15 mL 甲苯加热 10 min, 反应物全部溶解, 继续在 110~115 °C 下搅拌反应 5 h, 冷却, 氮气保护下过滤, 减压蒸去溶剂, 得白色固体, 用二氯甲烷/石油醚 (60~90 °C) 重结晶得白色晶体产物。所得产物的有关数据如下:

1.06 g 白色固体 **1**, 产率 82.4 %, m.p. 100~104 °C; ^1H NMR : 2.92 (br, 6H, 3 $\times\text{NCH}_2$), 3.68 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.50 (t, 2H, CH_2COO), 1.82 (t, 2H, GeCH_2), 7.32 (s, 15H, Ph_3Ge); IR (KBr) : 1621 (s, CO_2), 461 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{27}\text{H}_{31}\text{NO}_5\text{GeSn}$: C 50.61, H 4.88, N 2.19; found C 50.47, H 5.13, N 2.16.

1.04 g 白色固体 **2**, 产率 79.6 %, m.p. 108~110 °C; ^1H NMR : 2.90 (br, 6H, 3 $\times\text{NCH}_2$), 3.67 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 1.68~2.38 (m, 3H, CH_2CHCO_2), 1.13 (d, 3H, CH_3), 7.34 (s, 15H, Ph_3Ge); IR (KBr) : 1625 (s, CO_2), 461 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{28}\text{H}_{33}\text{NO}_5\text{GeSn}$: C 51.36, H 5.08, N 2.14; found C 51.44, H 5.20, N 2.11.

1.28 g 白色固体 **3**, 产率 97.5 %, m.p. 117~119 °C; ^1H NMR : 2.86 (t, 6H, 3 $\times\text{NCH}_2$), 3.68 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 1.88~2.52 (3H, m, CHCH_2CO_2), 1.26 (d, 3H, CH_3), 7.36 (s, 15H, Ph_3Ge); IR (KBr) : 1618 (s, CO_2), 459 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{28}\text{H}_{33}\text{NO}_5\text{GeSn}$: C 51.36, H 5.08, N 2.14; found C 51.28, H 5.23, N 2.11.

1.17 g 白色固体 **4**, 产率 81.6 %, m.p. 112~114 °C; ^1H NMR : 2.90 (br, 6H, 3 $\times\text{NCH}_2$), 3.70 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.88 (m, 2H, CH_2CO_2), 3.64 (m, 1H, GeCH), 7.14 (s, 5H, R^1), 7.36 (s, 15H, Ph_3Ge); IR (KBr) : 1620 (s, CO_2), 460 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{33}\text{H}_{35}\text{NO}_5\text{GeSn}$: C 55.29, H 4.92, N 1.95; found C 55.58, H 4.89, N 1.91.

1.01 g 白色固体 **5**, 产率 67.2 %, m.p. 127~129 °C; ^1H NMR : 2.90 (br, 6H, 3 $\times\text{NCH}_2$), 3.64 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.80 (m, 2H, CH_2CO_2), 3.60 (m, 1H, GeCH), 6.86~7.08 (m, 4H, R^1), 7.40 (s, 15H, Ph_3Ge); IR (KBr) : 1630 (s, CO_2), 470 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{33}\text{H}_{34}\text{NO}_5\text{ClGeSn}$: C 52.75, H 4.56, N 1.86; found C 53.15, H 4.18, N 2.17.

1.07 g 白色固体 **6**, 产率 71.5 %, m.p. 142~144 °C; ^1H NMR : 2.88 (br, 6H, 3 $\times\text{NCH}_2$), 3.60 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.90 (m, 2H, CH_2CO_2), 3.60 (m, 1H, GeCH), 6.84~7.21 (m, 4H, R^1), 7.30 (s, 15H, Ph_3Ge); IR (KBr) : 1637 (s, CO_2), 470 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{33}\text{H}_{34}\text{NO}_5\text{ClGeSn}$: C 52.75, H 4.56, N 1.86; found C 52.38, H 4.88, N 1.97.

1.10 g 白色固体 **7**, 产率 73.8 %, m.p. 165~167 °C; ^1H NMR : 2.80 (br, 6H, 3 $\times\text{NCH}_2$), 3.60 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.84 (m, 2H, CH_2CO_2), 3.60 (m, 4H, $\text{GeCH} + \text{CH}_3\text{OR}^1$), 6.42~6.80 (m, 4H, R^1), 7.22 (s, 15H, Ph_3Ge); IR (KBr) : 1626 (s, CO_2), 470 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{34}\text{H}_{37}\text{NO}_6\text{GeSn}$: C 54.67, H 4.99, N 1.87; found C 54.91, H 4.63, N 1.55.

1.20 g 白色固体 **8**, 产率 80.1 %, m.p. 147~149 °C; ^1H NMR : 2.88 (br, 6H, 3 $\times\text{NCH}_2$), 3.64 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.88 (m, 2H, CH_2CO_2), 3.60~3.82 (m, 4H, $\text{GeCH} + \text{CH}_3\text{OR}^1$), 6.38~7.08 (m, 4H, R^1), 7.28 (s, 15H, Ph_3Ge); IR (KBr) : 1641 (s, CO_2), 474 (m, Sn-N) cm^{-1} ; Anal. calcd for $\text{C}_{34}\text{H}_{37}\text{NO}_6\text{GeSn}$: C 54.67, H 4.99, N 1.87; found C 54.36, H 5.25, N 2.09.

1.25 g 白色固体 **9**, 产率 77.3 %, m.p. 116~118 °C; ^1H NMR : 2.88 (br, 6H, 3 $\times\text{NCH}_2$), 3.68 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.88 (m, 2H, CH_2CO_2), 3.64 (m, 1H, GeCH), 6.64~7.16 (m, 9H, R^1), 7.32 (s, 15H, Ph_3Ge); IR (KBr) : 1604 (s, CO_2), 470 (m,

Sn N) cm^{-1} ; Anal. calcd for $\text{C}_{39}\text{H}_{39}\text{NO}_6\text{GeSn}$: C 57.90, H 4.86, N 1.73; found C 57.78, H 5.18, N 1.96.

1.27 g 白色固体 **10**, 产率 87.0%, m. p. 121 ~ 123; ^1H NMR: 2.88 (br, 6H, 3 $\times\text{NCH}_2$), 3.66 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.90 (m, 2H, CH_2CO_2), 3.65 (m, 1H, GeCH), 6.92 ~ 7.14 (m, 4H, R^1), 2.30 (s, 3H, CH_3), 7.38 (s, 15H, Ph_3Ge); IR (KBr): 1621 (s, CO_2), 456 (m, Sn N) cm^{-1} ; Anal. calcd for $\text{C}_{34}\text{H}_{37}\text{NO}_5\text{GeSn}$: C 55.87, H 5.10, N 1.92; found C 55.96, H 5.17, N 1.75.

1.43 g 白色固体 **11**, 产率 91.2%, m. p. 147 ~ 149; ^1H NMR: 2.92 (br, 6H, 3 $\times\text{NCH}_2$), 3.65 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.94 (m, 2H, CH_2CO_2), 3.62 (m, 1H, GeCH), 6.84 ~ 7.26 (m, 3H, R^1), 7.40 (s, 15H, Ph_3Ge); IR (KBr): 1624 (s, CO_2), 461 (m, Sn N) cm^{-1} ; Anal. calcd for $\text{C}_{33}\text{H}_{33}\text{NO}_5\text{Cl}_2\text{GeSn}$: C 50.44, H 4.23, N 1.78; found C 50.51, H 4.00, N 1.72.

1.23 g 白色固体 **12**, 产率 88.6%, m. p. 150 ~ 152; ^1H NMR: 2.90 (br, 6H, 3 $\times\text{NCH}_2$), 3.64 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 1.16 (d, 3H, CH_3), 1.70 ~ 2.42 (m, 3H, CH_2CHCOO), 6.92 ~ 7.16 (m, 12H, GeC_6H_4), 2.32 (s, 9H, PhCH_3); IR (KBr): 1628 (s, CO_2), 488 (m, Sn N) cm^{-1} ; Anal. calcd for $\text{C}_{31}\text{H}_{39}\text{NO}_5\text{GeSn}$: C 53.43, H 5.64, N 2.01; found C 53.32, H 5.56, N 1.90.

1.14 g 白色固体 **13**, 产率 75.3%, m. p. 143 ~ 145; ^1H NMR: 2.88 (br, 6H, 3 $\times\text{NCH}_2$), 3.64 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.88 (m, 2H, CH_2CO_2), 3.64 (m, 1H, GeCH), 7.16 (s, 5H, R^1), 6.96 ~ 7.16 (m, 12H, GeC_6H_4), 2.32 (s, 9H, PhCH_3); IR (KBr): 1616 (s, CO_2), 478 (m, Sn N) cm^{-1} ; Anal. calcd for $\text{C}_{36}\text{H}_{41}\text{NO}_5\text{GeSn}$: C 57.00, H 4.45, N 1.84; found C 57.31, H 4.85, N 1.63.

1.39 g 白色固体 **14**, 产率 90.1%, m. p. 170 ~ 172; ^1H NMR: 2.88 (br, 6H, 3 $\times\text{NCH}_2$), 3.62 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.92 (m, 2H, CH_2CO_2), 3.60 (m, 1H, GeCH), 6.82 ~ 7.28 (m, 4H, R^1), 2.40 (s, 3H, R^1CH_3), 6.94 ~ 7.16 (m, 12H, GeC_6H_4), 2.30 (s, 9H, CH_3Ph); IR (KBr): 1627 (s, CO_2), 489 (m, Sn N) cm^{-1} ; Anal. calcd for $\text{C}_{37}\text{H}_{41}\text{NO}_5\text{GeSn}$: C 57.49, H 5.61, N 2.01; found C 57.22, H 5.58, N 1.80.

1.34 g 白色固体 **15**, 产率 84.7%, m. p. 186 ~ 189; ^1H NMR: 2.89 (br, 6H, 3 $\times\text{NCH}_2$), 3.62 (br, 6H, 3 $\times\text{CH}_2\text{O}$), 2.94 (m, 2H, CH_2CO_2), 3.62 (m, 1H, GeCH), 6.40 ~ 7.14 (m, 4H, R^1), 3.80 (s, 3H, CH_3OR^1), 6.98 ~ 7.24 (m, 12H, GeC_6H_4), 2.34 (s, 9H, CH_3Ph); IR (KBr): 1604 (s, CO_2), 489 (m, Sn N) cm^{-1} ; Anal. calcd for $\text{C}_{37}\text{H}_{43}\text{NO}_6\text{GeSn}$: C 56.31, H 5.49, N 1.78; found C 56.10, H 5.43, N 1.75.

2 结果与讨论

羧酸同金属杂噻唑烷的 1-位取代反应是合成各类 1-酰氧基金属杂噻唑烷的方法之一, 由于金属杂噻唑烷的稳定性与金属元素密切相关, 相对 Si 和 Ge 来说, 锡杂噻唑烷的稳定性较差, 因此可以不分

离中间体 $\text{EtOSn}(\text{OCH}_2\text{CH}_2)\text{N}$ 直接由 $\text{Sn}(\text{OEt})_4 + \text{N}(\text{CH}_2\text{CH}_2\text{OH})_3 + \text{Ar}_3\text{GeCHR}^1\text{CHR}^2\text{COOH}$ 一锅法来合成, 产率有明显提高, 另外反应应在 N_2 保护下进行.

在反应中及时把生成的乙醇排除出体系也是至关重要, 因为反应生成的乙醇有可能同未反应的取代丙酸发生酯化反应^[6]:



化合物的红外光谱数据表明, 它的 Sn N 伸缩振动吸收峰在 459 ~ 489 cm^{-1} 之间, 比类似的 Silatrane 化合物的波数 573 ~ 589 cm^{-1} 低^[6], 这可能是 Sn 原子接受了酰氧基中羰基氧的配位, 使其电子云密度增大, 减弱了 Sn N 的强度. 红外光谱中 C=O 基的吸收峰在 1604 ~ 1641 cm^{-1} 范围内, 也大大低于相应的 Silatrane 化合物 (1695 ~ 1718 cm^{-1})^[6], 这进一步说明这类化合物中 O—C=O 基的碳氧键长被平均化.

本类化合物的 ^1H NMR 与相应的含锗 Silatrane 不同^[6], 杂噻唑烷中的 $\text{OCH}_2\text{CH}_2\text{N}$ 并未表现出明显的双三重峰, 而是两个较宽的多重峰, 分别在 2.80 ~ 2.92 和 3.60 ~ 3.70 之间, 这可能是因为锡原子易变形的 5d 空轨道使 Sn 原子同杂噻唑烷中的氧原子也有配位作用, 造成杂噻唑环上三个 $\text{OCH}_2\text{CH}_2\text{N}$ 碳不等价所致. 由于 $\text{GeCHR}^1\text{CHR}^2$ 两个 C 原子上的氢质子化学位移有时与噻唑环的 $\text{CH}_2\text{CH}_2\text{N}$ 氢质子的化学位移相重叠, 所以表现为多重峰.

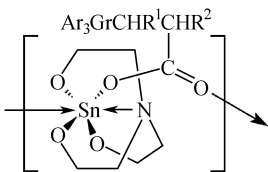


图 1 化合物的结构

Figure 1 The Structure of compounds

¹¹⁹Sn NMR 化学位移值在一定程度上反映出 Sn 原子的配位关系,Sn 原子的配位数增加,¹¹⁹Sn 的化学位移向高场移动,下面列出部分化合物的¹¹⁹Sn NMR 化学位移数据() (溶剂 DMSO-*d*₆):化合物 1 - 544.99;2 - 544.96;10 - 544.98;11 - 544.98;14 - 545.01. 结果表明,它们均在 - 545 处出现一个单峰,说明它们在溶剂中只存在一种锡核环境,可能

是六配位有机锡化合物(见图 1),这与 IR 和¹H NMR 谱的推测相一致,同时也说明具有强配位能力的 DMSO 并未破坏本类化合物的 C=O Sn 配位,使其在溶液和固体时结构保持一致.

化合物的裂解机制见图 2. 从图中可以看到它与相应硅杂^噁唑烷^[6]明显不同:(1)不存在分子离子峰,表明该类化合物在质谱条件下,不如 Silatrane 化合物稳定.(2)本类化合物的裂解首先发生在两个金属键上,形成三个独立的碎片离子:Ph₃Ge⁺ *m/e*: 305 (100%), Ph₃GeCH(C₆H₄OCH₃-*p*)CH₂COO⁺ *m/e*: 483 (2.0%), 和 Sn(OCH₂CH₂)₃N⁺ *m/e*: 266 (3.9%). 再由它们继续断裂,而形成更小的碎片离子峰. 化合物的质谱裂解也同样证明该锡杂^噁唑烷比相应的硅杂^噁唑烷较不稳定.

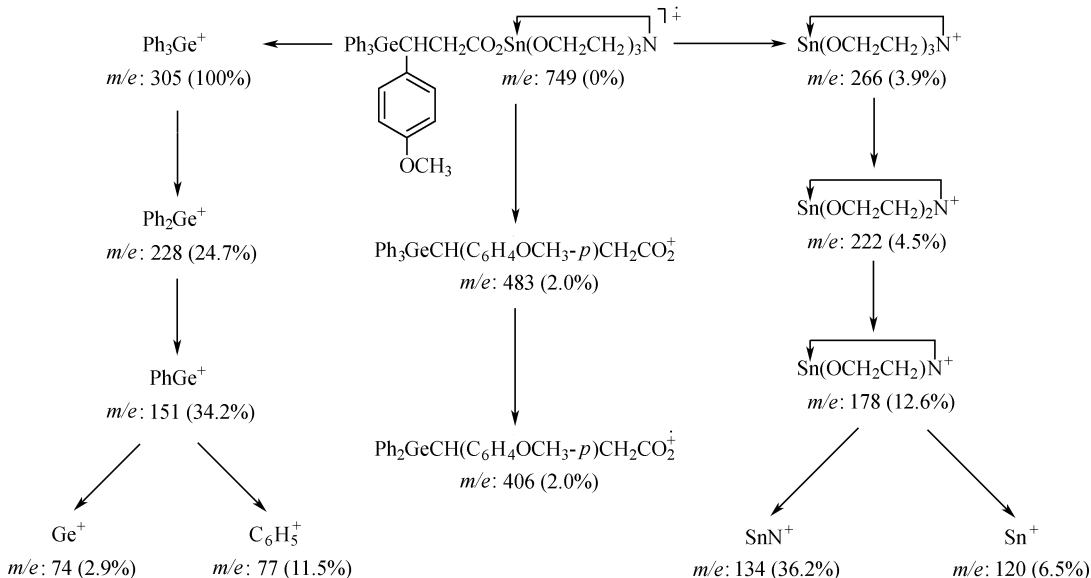


图 2 化合物 7 的质谱裂解机理

Figure 2 Fragmentation mechanism of compound 7

表 1 化合物对细菌的抑制活性

Table 1 Inhibition activities of compounds against some bacteria

	1 (μg/ mL)				2 (μg/ mL)				3 (μg/ mL)			
	200	100	50	25	200	100	50	25	200	100	50	25
A	++	+	+-	-	++	++	+	+-	++	+	+	+-
B	++	+	+-	-	++	++	+	+-	++	+	+	+-
C	+	+-	-	--	+-	-	-	-	+-	-	-	-
D	+-	-	-	--	+-	+-	-	-	-	-	-	-
E	-	-	-	--	+-	+-	-	-	+	+-	-	--
F	-	-	-	-	+-	-	-	--	-	-	-	--
G	-	-	-	--	-	-	-	--	+-	-	--	--
H	-	-	-	--	-	-	-	--	-	-	-	--

A—Beta heamolytic strepto coai; B—Staphylococcus aureus; C—Salmonella para typhi A; D—Salmonella typhi; E—Klebsiella; F—Boteus vulgaris; G—Pseudomones; H—E. coli. ++—Complete no growth and good activity; +—No growth and activity; +-—Some activity; -—Growth and almost no activity; --—Complete growth and activity.

众所周知金属杂^噁唑烷和有机锗丙酸类化合物都是具有某些生物活性的有机金属化合物. 为此, 我们选择了部分化合物进行了杀菌活性筛选, 结果

见表 1. 从表中可以看到, 化合物对溶血链球菌和金黄葡萄球菌有较好的抑制作用.

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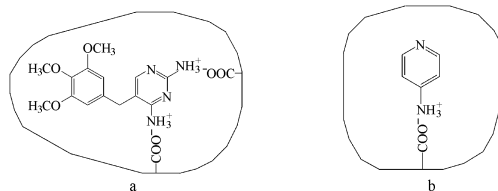
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Study on Recognition Mechanism of Molecularly Imprinted Microsphere Synthesized from Aqueous Solution

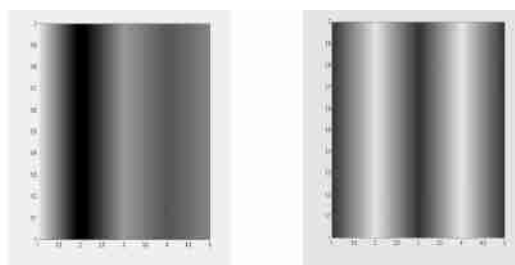


The molecularly imprinted polymer microspheres against 4-aminopyridine (4-AP) and trimethoprim (TMP) were synthesized. Chromatographic analyses showed that the interaction between acidic monomer/polymer and template with amino group mainly depended on the ionic (electrostatic) interaction, but not the simply added interaction.

LAI, Jia-Ping; CAO, Xi-Feng; HE, Xi-Wen; LI, Yuan-Yuan

Acta Chimica Sinica **2002**, 60(2), 322

A Visualization Method of Chromatographic Data for Discovering Fingerprint Features of Natural Herbal Medicines

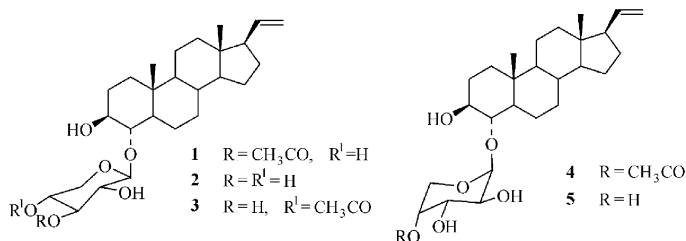


A novel visualization method of chromatographic data for discovering the fingerprint features of natural herbal medicines is proposed. The method can be used to effectively extract the hidden fingerprint features from analytical data set, and visually represent the chemical pattern discrepancy between different classes of herbal medicine.

CHENG, Yi-Yu; YU, Jie; WU, Yong-Jiang

Acta Chimica Sinica **2002**, 60(2), 328

Studies on the Secondary Metabolite of the Soft Coral *Lobophytum* sp.

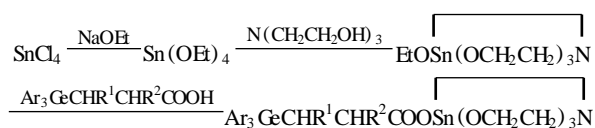


Five steroidal glycosides (1) ~ (5) were isolated from the soft coral *Lobophytum* sp. collected from Sanya Bay, Hainan Island. 1 is a new compound. These steroidal glycosides exhibited cytotoxic activity toward human tumor cell lines SKMG-4Hep-G2 and CNE2.

HE, Xi-Xin; SU, Jing-Yu; ZENG, Long-Mei; YANG, Xiao-Ping; LIANG, Yong-Ju

Acta Chimica Sinica **2002**, 60(2), 334

The Reaction of α -Trialkylgermanium (α)-substituted Propionic Acid with 1-Ethoxystannatrane



The reaction of α -trialkylgermanium (α)-substituted propionic acid with 1-ethoxystannatrane was studied and fifteen organometallic compounds containing germanium and tin were synthesized. The structures of prepared compounds were characterized.

SONG, Xue-Qing; LUO, Ning; SUN, Li-Juan; XIE, Qing-Lan

Acta Chimica Sinica **2002**, 60(2), 338