

乳香的化学成分研究

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摘要: 目的 研究橄榄科植物卡氏乳香树 *Boswellia carterii* 的树脂氯仿提取物的化学成分。方法 利用色谱方法对乳香化学成分进行分离纯化, 并根据理化性质和波谱数据鉴定化合物的结构。结果 从乳香氯仿提取物中分离得到 14 个化合物, 分别鉴定为 α -香树素 (1)、 β -乳香酸 (2)、乙酰 11 α -甲氧基- β -乳香酸 (3)、11-羰基- β -乳香酸 (4)、乙酰 11-羰基- β -乳香酸 (5)、9, 11-去氢- β -乳香酸 (6)、乙酰 α -乳香酸和乙酰 β -乳香酸 (7)、 α -乳香酸 (8)、9, 11-去氢- α -乳香酸 (9)、 α -乙酰榄香醇酸 (10)、3 α -羟基甘遂-8, 24-二烯-21-酸 (11)、榄香酮酸 (12)、3 α -羟基甘遂-7, 24-二烯-21-酸 (13)、 α -棕榈酸甘油酯 (14)。结论 化合物 1 和 9 为首次从该属植物中分离得到。

关键词: 卡氏乳香树; 乳香; 三萜; α -香树素; 9, 11-去氢- α -乳香酸

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Chemical constituents from frankincense

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Key words: *Boswellia carterii* Birdw.; frankincense; triterpenoids; α -amyrin; 9, 11-dehydro- α -boswellic acid

乳香为橄榄科植物卡氏乳香树 *Boswellia carterii* Birdw. 的树脂。主产于红海沿岸的索马里和埃塞俄比亚。此外尚有同属植物鲍达乳香树 *B. bhaw-dajiana* Birdw. 及野乳香树 *B. neglecta* M. Moore 等数种。现代药理研究表明, 乳香中的乳香酸类化合物具有抗炎作用和对肿瘤细胞有抗增殖、分化诱导和凋亡诱导作用^[1], 郭辉等^[2]报道了乳香中抗哮喘有效部位的化学成分。本课题组采用多种色谱技术, 从卡氏乳香树脂的氯仿提取物中, 分离得到 14 个化合物, 分别鉴定为 α -香树素 (α -amyrin, 1)、 β -乳香酸 (β -boswellic acid, 2)、乙酰 11 α -甲氧基- β -乳香酸 (acetyl 11 α -methoxy- β -boswellic acid, 3)、11-羰基- β -乳香酸 (11-keto- β -boswellic acid, 4)、乙酰 11-羰基- β -乳香酸 (acetyl 11-keto- β -boswellic acid, 5)、9, 11-去氢- β -乳香酸 (9, 11-dehydro- β -boswellic acid, 6)、乙酰 α -乳香酸和乙酰 β -乳香酸 (acetyl α -boswellic acid 和 acetyl β -boswellic acid, 7)、 α -乳香酸 (α -boswellic acid,

8)、9, 11-去氢- α -乳香酸 (9, 11-dehydro- α -boswellic acid, 9)、 α -乙酰榄香醇酸 (α -acetyl elemolic acid, 10)、3 α -羟基甘遂-8, 24-二烯-21-酸 (3 α -hydroxy-tirucalla-8, 24-dien-21-oic acid, 11)、榄香酮酸 (elemonic acid, 12)、3 α -羟基甘遂烷-7, 24-二烯-21-酸 (3 α -Hydroxytirucalla-7, 24-dien-21-oic acid, 13)、 α -棕榈酸甘油酯 (α -glyceryl palmitate, 14)。

1 仪器和材料

核磁共振波谱仪为 Bruker ARX 300 NMR spectrometer 和 Bruker ARX 600 NMR Spectrometer, TMS 做内标; 质谱仪为 Shimadzu GCMS—QP5050A, Agilent 1100 SL, Autospec-Ultima ETOF。色谱柱硅胶 (200~300 目) 和薄层色谱用硅胶 H、GF₂₅₄ 购于青岛海洋化工厂; ODS、Sephadex LH-20 购于 GE Healthcare。

乳香购于辽宁省药材公司, 经沈阳药科大学中药学院药用植物教研室孙启时教授鉴定为卡氏乳香树 *Boswellia carterii* Birdw. 的树脂, 标本(20070918)

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2 提取与分离

乳香树脂 1 kg, 经粉碎后, 以氯仿回流提取 3 次, 合并 3 次提取液, 浓缩干燥, 得浸膏 678 g。进行硅胶柱色谱 (200~300 目) 分离, 以石油醚-醋酸乙酯梯度洗脱, 将各个梯度所得流份合并, 再经反复硅胶、Sephadex LH-20 及反相 ODS 开放柱色谱分离, 并重结晶得化合物 **1** (18 mg)、**2** (6 mg)、**3** (7 mg)、**4** (5 mg)、**5** (1 700 mg)、**6** (8 mg)、**7** (3 432 mg)、**8** (9 mg)、**9** (8 mg)、**10** (3 173 mg)、**11** (7 mg)、**12** (1 500 mg)、**13** (9 mg)、**14** (30 mg)。

3 结构鉴定

化合物 **1**: 白色无定形粉末 (丙酮), 10% 硫酸乙醇溶液显色为红色。¹H-NMR (300 MHz, CDCl₃) δ : 0.79 (6H, s), 0.80 (3H, s), 0.96 (3H, s), 1.00 (6H, s), 1.01 (3H, s), 1.07 (3H, s), 3.25 (1H, dd, J = 10.1, 4.5 Hz, H-3), 5.31 (1H, br s, H-12); ¹³C-NMR (75 MHz, CDCl₃) δ : 38.8 (C-1), 27.3 (C-2), 79.1 (C-3), 38.8 (C-4), 55.2 (C-5), 18.3 (C-6), 32.9 (C-7), 40.0 (C-8), 47.7 (C-9), 36.9 (C-10), 23.3 (C-11), 124.4 (C-12), 139.6 (C-13), 42.0 (C-14), 28.7 (C-15), 26.6 (C-16), 33.7 (C-17), 59.1 (C-18), 39.6 (C-19), 39.6 (C-20), 31.2 (C-21), 41.5 (C-22), 28.1 (C-23), 15.6 (C-24), 15.6 (C-25), 16.9 (C-26), 23.3 (C-27), 28.1 (C-28), 17.5 (C-29), 21.4 (C-30)。以上数据与文献报道一致^[3], 故鉴定化合物 **1** 为 α -香树脂素。

化合物 **2**: 无色针晶 (丙酮), 10% 硫酸乙醇溶液显色为红色。¹H-NMR (300 MHz, CDCl₃) δ : 0.80 (6H, s), 0.90 (6H, s), 1.04 (3H, s), 1.09 (3H, s), 1.35 (3H, s), 4.08 (1H, br s, H-3), 5.14 (1H, br s, H-12); ¹³C-NMR (75 MHz, CDCl₃) δ : 33.9 (C-1), 26.2 (C-2), 70.7 (C-3), 47.3 (C-4), 49.1 (C-5), 19.7 (C-6), 33.1 (C-7), 40.0 (C-8), 46.8 (C-9), 37.5 (C-10), 23.4 (C-11), 124.5 (C-12), 139.6 (C-13), 42.3 (C-14), 26.5 (C-15), 28.1 (C-16), 33.8 (C-17), 59.2 (C-18), 39.7 (C-19), 39.6 (C-20), 31.3 (C-21), 41.5 (C-22), 24.2 (C-23), 182.7 (C-24), 13.3 (C-25), 16.9 (C-26), 23.2 (C-27), 28.8 (C-28), 17.4 (C-29), 21.4 (C-30)。以上数据与文献报道一致^[4], 故鉴定化合物 **2** 为 β -乳香酸。

化合物 **3**: 无色针晶 (丙酮), 10% 硫酸乙醇溶液显色为红色。EI-MS m/z : 528 [M]⁺, 496, 323, 248 (100), 119。¹H-NMR (300 MHz, CDCl₃) δ : 0.81 (3H, s, Me-28), 0.90 (3H, d, J = 5.8 Hz, Me-29), 0.93 (3H,

s, Me-30), 1.03 (3H, s, Me-25), 1.09 (3H, s, Me-26), 1.20 (3H, s, Me-27), 1.24 (3H, s, Me-23), 2.06 (CH₃CO), 3.28 (3H, s, CH₃O), 3.82 (1H, dd, J = 9.0, 3.0 Hz, H-11), 5.28 (1H, br s, H-3), 5.36 (1H, d, J = 3.0 Hz, H-12); ¹³C-NMR (75 MHz, CDCl₃) δ : 35.6 (C-1), 23.7 (C-2), 73.2 (C-3), 46.7 (C-4), 50.4 (C-5), 19.4 (C-6), 33.1 (C-7), 42.2 (C-8), 51.4 (C-9), 38.5 (C-10), 77.0 (C-11), 124.0 (C-12), 143.5 (C-13), 42.8 (C-14), 26.6 (C-15), 27.9 (C-16), 33.6 (C-17), 58.6 (C-18), 39.5 (C-19), 39.3 (C-20), 31.1 (C-21), 41.3 (C-22), 24.0 (C-23), 182.2 (C-24), 14.4 (C-25), 18.1 (C-26), 22.4 (C-27), 28.7 (C-28), 17.3 (C-29), 21.3 (C-30), 54.5 (CH₃O), 170.4 (CH₃CO), 21.3 (CH₃CO)。以上数据与文献报道一致^[5], 故鉴定化合物 **3** 为乙酰 11 α -甲氧基- β -乳香酸。

化合物 **4**: 无色针晶 (丙酮), 10% 硫酸乙醇溶液不显色。¹H-NMR (300 MHz, CDCl₃) δ : 0.79 (3H, d, J = 6.3 Hz), 0.82 (3H, s), 0.94 (3H, s), 1.13 (3H, s), 1.18 (3H, s), 1.31 (3H, s), 1.35 (3H, s), 4.08 (1H, br s, H-3), 5.55 (1H, br s, H-12); ¹³C-NMR (75 MHz, CDCl₃) δ : 33.9 (C-1), 23.5 (C-2), 73.4 (C-3), 45.1 (C-4), 48.8 (C-5), 18.3 (C-6), 32.6 (C-7), 43.7 (C-8), 60.4 (C-9), 37.4 (C-10), 200.6 (C-11), 130.4 (C-12), 165.2 (C-13), 45.0 (C-14), 27.5 (C-15), 27.3 (C-16), 33.9 (C-17), 58.9 (C-18), 39.2 (C-19), 39.2 (C-20), 30.9 (C-21), 40.9 (C-22), 24.4 (C-23), 182.2 (C-24), 13.1 (C-25), 18.3 (C-26), 21.1 (C-27), 28.8 (C-28), 17.4 (C-29), 20.5 (C-30)。以上数据与文献报道一致^[5], 故鉴定化合物 **4** 为 11-羰基- β -乳香酸。

化合物 **5**: 无色针晶 (丙酮), 10% 硫酸乙醇溶液不显色。EI-MS m/z : 512 [M]⁺, 497, 453, 273 (100), 232, 135。¹H-NMR (300 MHz, CDCl₃) δ : 0.78 (3H, d, J = 6.4 Hz, CH₃-29), 0.80 (3H, s, CH₃-28), 0.93 (3H, br s, CH₃-30), 1.12 (3H, s, CH₃-25), 1.18 (3H, s, CH₃-26), 1.22 (3H, s, CH₃-23), 1.33 (3H, s, CH₃-27), 2.07 (3H, s, CH₃CO), 5.28 (1H, br s, H-3), 5.54 (1H, s, H-12); ¹³C-NMR (75 MHz, CDCl₃) δ : 34.6 (C-1), 23.5 (C-2), 73.1 (C-3), 46.5 (C-4), 50.4 (C-5), 18.7 (C-6), 32.8 (C-7), 45.1 (C-8), 60.3 (C-9), 37.4 (C-10), 199.4 (C-11), 130.5 (C-12), 165.1 (C-13), 43.8 (C-14), 27.5 (C-15), 27.2 (C-16), 34.0 (C-17), 59.0 (C-18), 39.3 (C-19), 39.3 (C-20), 30.9 (C-21), 40.9 (C-22), 23.8 (C-23), 182.1 (C-24), 13.2 (C-25), 18.4 (C-26), 20.5

(C-27), 28.9 (C-28), 17.4 (C-29), 21.1 (C-30), 170.3 (CH₃CO), 21.3 (CH₃CO)。以上数据与文献报道一致^[5], 故鉴定化合物 **5** 为乙酰 11-羧基-β-乳香酸。

化合物 **6**: 白色无定形粉末 (甲醇), 10%硫酸乙醇溶液显色为红色。¹H-NMR (300 MHz, CDCl₃) δ: 0.80 (3H, d, *J* = 6.3 Hz), 0.86 (3H, s), 0.93 (6H, s), 1.11 (3H, s), 1.19 (3H, s), 1.37 (3H, s), 4.09 (1H, br s, H-3), 5.46 (1H, d, *J* = 6.0 Hz), 5.65 (1H, d, *J* = 6.0 Hz); ¹³C-NMR (75 MHz, CDCl₃) δ: 33.6 (C-1), 22.4 (C-2), 72.9 (C-3), 46.9 (C-4), 47.4 (C-5), 19.5 (C-6), 31.8 (C-7), 43.4 (C-8), 152.7 (C-9), 39.0 (C-10), 122.9 (C-11), 116.4 (C-12), 141.6 (C-13), 40.6 (C-14), 26.2 (C-15), 28.2 (C-16), 33.7 (C-17), 57.3 (C-18), 39.4 (C-19), 39.0 (C-20), 31.2 (C-21), 41.3 (C-22), 23.7 (C-23), 183.2 (C-24), 23.4 (C-25), 21.6 (C-26), 17.4 (C-27), 28.7 (C-28), 17.4 (C-29), 21.8 (C-30)。以上数据与文献报道一致^[6], 故鉴定化合物 **6** 为 9, 11-去氢-β-乳香酸。

化合物 **7**: 无色结晶 (石油醚-醋酸乙酯), 10%硫酸乙醇溶液显色为紫色。EI-MS *m/z*: 498 [M]⁺, 483, 423, 394, 218 (100)。¹H-NMR (300 MHz, CDCl₃) δ: 0.80 ~ 1.30 (CH₃ × 14), 2.11 (6H, s, CH₃COO), 5.30 (2H, br s, H-3), 5.20 (1H, br s, H-12), 5.15 (1H, br s, H-12)。以上数据与文献报道的 α-乙酰乳香酸和 β-乙酰乳香酸混合物一致^[5], 故鉴定化合物 **7** 为 α-乙酰乳香酸和 β-乙酰乳香酸混合物。

化合物 **8**: 无色针晶 (丙酮), 10%硫酸乙醇溶液显色为红色。EI-MS *m/z*: 456 [M]⁺, 441, 423, 395, 218 (100), 203。UV λ_{max}^{MeOH} (nm): 205。IR ν_{max}^{KBr} (cm⁻¹): 3 445, 2 948, 1 696, 1 457, 1 381, 1 234, 1 194, 1 055, 1 026, 994。¹H-NMR (300 MHz, CDCl₃) δ: 0.83 (3H, s, CH₃-28), 0.86 (3H, s, CH₃-29), 0.87 (3H, s, CH₃-30), 0.88 (3H, s, CH₃-25), 1.00 (3H, s, CH₃-26), 1.15 (3H, s, CH₃-27), 1.34 (3H, s, CH₃-23), 4.08 (1H, br s, H-3), 5.19 (1H, br s, H-12); ¹³C-NMR (75 MHz, CDCl₃) δ: 33.6 (C-1), 26.1 (C-2), 70.7 (C-3), 47.4 (C-4), 49.1 (C-5), 19.7 (C-6), 32.7 (C-7), 39.8 (C-8), 46.7 (C-9), 37.6 (C-10), 23.5 (C-11), 121.7 (C-12), 145.1 (C-13), 41.9 (C-14), 26.0 (C-15), 26.9 (C-16), 32.5 (C-17), 47.3 (C-18), 46.7 (C-19), 31.1 (C-20), 34.7 (C-21), 37.1 (C-22), 24.2 (C-23), 183.2 (C-24), 13.0 (C-25), 16.7 (C-26), 25.9 (C-27), 28.4 (C-28), 33.3 (C-29), 23.7 (C-30)。以上数据与文献报道一致^[4], 故鉴定化合物 **8** 为 α-乳香酸。

化合物 **9**: 白色无定形粉末 (甲醇), 易溶于氯

仿, 10%硫酸乙醇溶液显色为红色。¹H-NMR (300 MHz, CDCl₃) δ: 0.88 (3H, s), 0.89 (3H, s), 0.90 (3H, s), 1.03 (3H, s), 1.11 (3H, s), 1.16 (3H, s), 1.38 (3H, s), 4.10 (1H, br s, H-3), 5.51 (1H, d, *J* = 6.0 Hz), 5.65 (1H, d, *J* = 6.0 Hz); ¹³C-NMR (75 MHz, CDCl₃) δ: 33.2 (C-1), 23.2 (C-2), 70.3 (C-3), 46.9 (C-8), 47.6 (C-5), 19.6 (C-6), 31.9 (C-7), 43.0 (C-8), 152.3 (C-9), 39.2 (C-10), 120.6 (C-11), 116.9 (C-12), 147.5 (C-13), 40.6 (C-14), 26.9 (C-15), 28.7 (C-16), 33.7 (C-17), 46.9 (C-18), 45.6 (C-19), 31.1 (C-20), 34.6 (C-21), 37.0 (C-22), 23.7 (C-23), 181.2 (C-24), 23.2 (C-25), 20.5 (C-26), 20.0 (C-27), 28.7 (C-28), 33.2 (C-29), 24.1 (C-30)。以上数据与文献报道一致^[7], 故鉴定化合物 **9** 为 9, 11-去氢-α-乳香酸。

化合物 **10**: 无色针晶 (丙酮), 10%硫酸乙醇溶液显色为红色。¹H-NMR (300 MHz, CDCl₃) δ: 0.75 ~ 0.97 (CH₃ × 5), 1.59 (3H, s), 1.68 (3H, s), 2.06 (3H, s, CH₃CO), 4.65 (1H, br s, H-3), 5.10 (1H, t, *J* = 6.6 Hz, H-24); ¹³C-NMR (75 MHz, CDCl₃) δ: 31.2 (C-1), 27.3 (C-2), 76.5 (C-3), 37.3 (C-4), 44.3 (C-5), 20.1 (C-6), 28.9 (C-7), 133.4 (C-8), 133.2 (C-9), 37.0 (C-10), 25.1 (C-11), 31.3 (C-12), 43.7 (C-13), 49.6 (C-14), 28.4 (C-15), 28.5 (C-16), 46.9 (C-17), 19.7 (C-18), 16.6 (C-19), 47.3 (C-20), 182.5 (C-21), 22.4 (C-22), 26.5 (C-23), 123.6 (C-24), 132.7 (C-25), 17.6 (C-26), 23.7 (C-27), 24.2 (C-28), 19.2 (C-29), 26.8 (C-30), 170.3 (CH₃CO), 21.7 (CH₃CO)。以上数据与文献报道一致^[8-9], 故鉴定化合物 **10** 为 α-乙酰榄香醇酸。

化合物 **11**: 无色针晶 (丙酮), 10%硫酸乙醇溶液显色为红色。EI-MS *m/z*: 456 [M]⁺, 441 [M-CH₃], 423 [M-CH₃-H₂O]。¹H-NMR (300 MHz, CDCl₃) δ: 0.79 (3H, s), 0.82 (3H, s), 0.88 (3H, s), 0.93 (3H, s), 0.97 (3H, s), 1.00 (3H, s), 1.55 (3H, s), 1.68 (3H, s), 3.25 (1H, br s, H-3), 5.09 (1H, t, *J* = 6.6 Hz, H-24); ¹³C-NMR (75 MHz, CDCl₃) δ: 35.5 (C-1), 27.1 (C-2), 79.1 (C-3), 39.0 (C-4), 51.1 (C-5), 19.1 (C-6), 28.8 (C-7), 134.1 (C-8), 132.3 (C-9), 37.2 (C-10), 26.1 (C-11), 32.5 (C-12), 44.0 (C-13), 50.0 (C-14), 29.4 (C-15), 28.0 (C-16), 47.0 (C-17), 20.1 (C-18), 15.5 (C-19), 48.0 (C-20), 183.1 (C-21), 21.7 (C-22), 27.0 (C-23), 124.0 (C-24), 133.4 (C-25), 24.6 (C-26), 17.8 (C-27), 28.1 (C-28), 16.0 (C-29), 25.8 (C-30)。以上数

据与文献报道一致^[10], 故鉴定化合物 **11** 为 3 α -羟基甘遂-8, 24-二烯-21-酸。

化合物 **12**: 无色针晶 (丙酮), 10%硫酸乙醇溶液显色为红色。EI-MS m/z : 454 $[M]^+$, 439 $[M-CH_3]$, 421 $[M-CH_3-H_2O]$, 393, 297, 159, 133, 119 (100)。¹H-NMR (300 MHz, CDCl₃) δ : 0.82 (3H, s), 0.90 (3H, s), 1.03 (3H, s), 1.05 (3H, s), 1.10 (3H, s), 1.59 (3H, s), 1.68 (3H, s), 5.09 (1H, t, $J=6.6$ Hz, H-24); ¹³C-NMR (75 MHz, CDCl₃) δ : 35.6 (C-1), 34.5 (C-2), 217.7 (C-3), 47.3 (C-4), 51.5 (C-5), 20.1 (C-6), 25.9 (C-7), 133.2 (C-8), 134.4 (C-9), 37.1 (C-10), 21.4 (C-11), 29.3 (C-12), 43.9 (C-13), 49.7 (C-14), 27.4 (C-15), 28.7 (C-16), 46.9 (C-17), 15.8 (C-18), 19.6 (C-19), 47.6 (C-20), 182.8 (C-21), 32.4 (C-22), 26.8 (C-23), 123.6 (C-24), 132.7 (C-25), 17.6 (C-26), 25.7 (C-27), 24.2 (C-28), 21.2 (C-29), 26.8 (C-30)。以上数据与文献报道一致^[11], 故鉴定化合物 **12** 为榄香酮酸。

化合物 **13**: 白色无定形粉末 (丙酮), 易溶于氯仿, 10%硫酸乙醇溶液显色为红色。¹H-NMR (300 MHz, CDCl₃) δ : 0.75 (3H, s), 0.90 (3H, s), 0.91 (3H, s), 0.93 (3H, s), 0.97 (3H, s), 1.58 (3H, s), 1.71 (3H, s), 3.49 (1H, br s, H-3), 5.09 (1H, br s, H-7), 5.25 (1H, br s, H-24); ¹³C-NMR (75 MHz, CDCl₃) δ : 31.2 (C-1), 25.3 (C-2), 76.5 (C-3), 37.3 (C-4), 44.5 (C-5), 23.9 (C-6), 118.2 (C-7), 145.7 (C-8), 48.3 (C-9), 34.8 (C-10), 17.5 (C-11), 33.4 (C-12), 43.3 (C-13), 51.0 (C-14), 33.9 (C-15), 29.7 (C-16), 47.1 (C-17), 12.9 (C-18), 21.8 (C-19), 49.7 (C-20), 181.4 (C-21), 32.4 (C-22), 27.0 (C-23), 123.6 (C-24), 132.2 (C-25), 17.7 (C-26), 25.7 (C-27), 27.8 (C-28), 21.8 (C-29), 27.3 (C-30)。以上数据与文献报道一致^[12], 故鉴定化合物 **13** 为 3 α -羟基甘遂-7, 24-二烯-21-酸。

化合物 **14**: 白色无定形粉末 (甲醇), 10%硫酸乙醇溶液显色为红色。EI-MS m/z : 310, 299, 256, 239, 134, 98, 57, 43。¹H-NMR (CDCl₃) δ : 0.88 (3H, $J=7.2$ Hz, CH₃), 1.25 (br s, CH₂×12), 1.63 (2H, t, $J=6.9$ Hz, H-3'), 2.35 (2H, t, $J=7.5$ Hz, H-2'), 3.59 (1H, dd, $J=12.0, 5.7$ Hz, H-3), 3.70 (1H, dd, $J=12.0, 3.9$ Hz, H-3), 3.93 (1H, m, H-2), 4.14 (1H, dd, $J=12.0, 6.2$ Hz, H-1), 4.20 (1H, dd, $J=12.0, 5.0$ Hz, H-1)。以上数据与文献报道一致^[13], 故鉴定该化合物 **14** 为 α -棕榈酸甘油酯。

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