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Key Words

Cantharidin
N-4-methylbenzylcantharidinimide (4b)
mp 124-125 °C; ¹H NMR (CDCl₃, 300 MHz): δ 7.06 (m, d, J = 7.2 Hz, phenyl H-3, H-5), 7.16 (m, d, J = 7.2 Hz, phenyl H-2, H-6), IR (KBr): 1734 cm⁻¹ (amide); MS m/z (rel. int.): 299 [M]⁺ (30), 105 (100), 138 (60).
N-4-methylbenzylcantharidinimide (4a)
mp 108-109 °C; ¹H NMR (CDCl₃, 300 MHz): δ 7.17 (m, t, CH, × 2), 1.68-1.72 (m, CH₂ × 2), 4.26 (m, s, OCH₂), 4.60 (m, s, NCH₂), 7.42 (m, d, J = 6.8 Hz, phenyl H-2, H-6), 7.62 (m, d, J = 6.8 Hz, phenyl H-3, H-5), IR (KBr): 1700 cm⁻¹ (amide); MS m/z (rel. int.): 319 [M]⁺ (20), 138 (10), 122 (20), 105 (100).
N-4-methylbenzylcantharidinimide (4c)
mp 108-109 °C; ¹H NMR (CDCl₃, 300 MHz): δ 7.17 (m, t, CH, × 2), 1.68-1.72 (m, CH₂ × 2), 4.26 (m, s, OCH₂), 4.52 (m, s, NCH₂), 7.42 (m, d, J = 6.8 Hz, phenyl H-2, H-6), 7.62 (m, d, J = 6.8 Hz, phenyl H-3, H-5), IR (KBr): 1700 cm⁻¹ (amide); MS m/z (rel. int.): 319 [M]⁺ (20), 138 (10), 122 (20), 105 (100).

ACKNOWLEDGEMENTS

The authors thank the National Science Council, R.O.C. for financial support (NSC 87-2311-B-001). We also thank the National Science Council, R.O.C. for financial support (NSC 87-2311-B-001).

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EXPERIMENTAL SECTION

General Procedures
The reaction of compound 1 with alcohol (3a-c) was carried out in a 100 mL round-bottomed flask equipped with a magnetic stirring bar and a nitrogen inlet. Compound 1 (0.4 mmol) and the corresponding alcohol (0.4 mmol) were dissolved in 10 mL of dry THF. The reaction mixture was stirred for 12 h under N₂. After the reaction was removed under reduced pressure and the solvent was removed under reduced pressure and the resulting residue was extracted with 1-hexanol. To remove the PMPO by-product, the crude product was purified by SiO₂ chromatography. The residue was crystallized from MeOH.

N-4-methylbenzylcantharidinimide (4a)
mp 120-122 °C; ¹H NMR (CDCl₃, 300 MHz): δ 7.10 (m, d, J = 7.2 Hz, phenyl H-3, H-5), 7.16 (m, d, J = 7.2 Hz, phenyl H-2, H-6), IR (KBr): 1734 cm⁻¹ (amide); MS m/z (rel. int.): 299 [M]⁺ (30), 105 (100), 138 (60).

N-4-methylbenzylcantharidinimide (4b)
mp 124-125 °C; ¹H NMR (CDCl₃, 300 MHz): δ 7.06 (m, d, J = 7.2 Hz, phenyl H-3, H-5), 7.16 (m, d, J = 7.2 Hz, phenyl H-2, H-6), IR (KBr): 1734 cm⁻¹ (amide); MS m/z (rel. int.): 299 [M]⁺ (30), 105 (100), 138 (60).

N-4-methylbenzylcantharidinimide (4c)
mp 108-109 °C; ¹H NMR (CDCl₃, 300 MHz): δ 7.17 (m, t, CH, × 2), 1.68-1.72 (m, CH₂ × 2), 4.26 (m, s, OCH₂), 4.52 (m, s, NCH₂), 7.42 (m, d, J = 6.8 Hz, phenyl H-2, H-6), 7.62 (m, d, J = 6.8 Hz, phenyl H-3, H-5), IR (KBr): 1700 cm⁻¹ (amide); MS m/z (rel. int.): 319 [M]⁺ (20), 138 (10), 122 (20), 105 (100).