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HPLC Analysis

HPLC fractions analysis(Bruker DRX600, 2.5-mm SEI probe head,2H2O, 30°C) of the compound yielded data consistent with 3,4-dihydroxycyclohexane-1-carboxylic acid. Subsequently, this compound was synthesized by a methodpreviously published (13). 3-Cyclohexene-1-carboxylic acid (99.5 mg, 0.79 mmol)was dissolved in 100 ml of 20 mM KOH and cooled to 0°C.

To this solution, 100mg of KMnO4(0.63 mmol) in 20 ml of H2O was added drop wise at 0°C, and the solution was stirred for1hato°C. Subsequently, 4 ml of 1 M HCl was added, the precipitated MnO2was removed by filtration, and the resulting solution was freeze-dried.

The resulting material was dissolved in 6 ml of aqueous 7.5%MeCN (0.1% trifluoroacetic acid) and fractionated (120.5 ml injected) on agraphitized carbon HPLC column (21.2 by 100 mm, 5m; ThermoQuest, Run-corn, Cheshire, United Kingdom) eluted with aqueous 7.5% MeCN (0.1% tri-fluoroacetic acid) at 10 ml min1, with UV detection at 210 nm.

Fractions were pooled and subjected to NMR analysis, resulting in 27 mg of (trans,trans)-3,4-dihydroxycyclohexane-1-carboxylic acid, which was the main product.
HPLC-electrospray ionization (ESI)-tandem MS (MS/MS) analysis of silageextracts.

The MeCN fractions obtained by SPE were analyzed by HPLC-ESI-MSand HPLC-ESI-MS/MS (HP1100 [Hewlett-Packard, Palo Alto, CA], BrukerEsquire, with a Hypercarb graphitized carbon HPLC column (2.1 by 100 mm) eluted with a gradient of MeOH inH2O (35 to 100% in 8 min, followed by 12 min at 100%) containing 0.1% formicacid at 0.2 ml min1. Diketopiperazines were identified by comparison of retention times and fragmentation patterns with those of authentic standards.

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