NOTE

PARTIAL SYNTHESIS OF [6α-^{3}H]GIBBERELLIN-A_3 OF HIGH SPECIFIC ACTIVITY [1]

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SUMMARY

The phytohormone gibberellin-A_3 was tritium-labelled by a chemical route. The specific activity of the [6α-^{3}H]-gibberellin-A_3 obtained was 22.5 Ci/mmol.

Keywords: Gibberellin-A_3, Tritium, High Specific activity labelling

INTRODUCTION

The availability of high specific activity tritiated gibberellin-A_3 (GA_3) is very important, for example, for the investigation of hormone-receptor-relationships in plants and for use in radioimmunoassay.

Recently, we reported new procedures for the preparation of [15-^{3}H]GA_3 [2] and [6α-^{3}H]GA_3 (4)[3]. In addition to the latter synthesis, we wish to describe a preparation of [6α-^{3}H]GA_3 (4) at high molar specific activity.

EXPERIMENTAL AND PURIFICATION

GA_3-7-aldehyde (1) (115.2 mg)[4] was silylated with Me_3Si-NH-$SiMe_3$ and Me_3SiCl in pyridine and enolized with KH[3]. The resulting eno-

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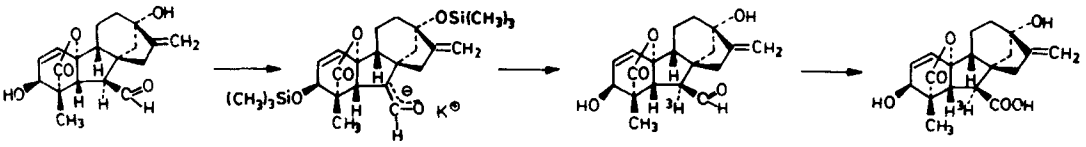
late 2 was tritium-labelled by addition of ^3H_2O [prepared by platinum oxide-oxidation of carrier-free tritium gas (100 Ci)[5] and destilled into the enolate solution 2 using a high vacuum line]. Analogous working up and SiO_2-chromatography as described[3] gave in 26 % yield [6α-^{3}H]GA_3-7-aldehyde (3) [30.2 mg not optimized; specific radioactivity approximately 22 Ci/mmol; radiochemical purity > 97 % determined by TLC using $CHCl_3$/EtOAc/AcOH (90:10:5)]. After storage for one month in ethanol at -30 °C, the radiochemical purity of 3 decreased to approximately 60 %. The by-product



1 2 3 4

[6-^{3}H]6-epi-GA_3-7-aldehyde was not isolated. Acetylation, oxidation and deacetylation of 3 (3 mg) as described[3] yielded [6α-^{3}H]-GA_3 (4), which was purified four times on a preparative plate (silica 60 from Merck, 0.3 mm) in acetone/ethylacetate/toluene/water (70:30:15:10) as well as chloroform/methanol/acetic acid (80:15:5). We obtained 8.3 mCi (not optimized) [6α-^{3}H]GA_3: radiochemical purity > 97 %; specific radioactivity 22.5 Ci/mmol (determined by liquid scintillation counting and by fluorimetry. [6α-^{3}H]GA_3 (1 mCi) was stored in ethanol (3 ml) at -30° for three monthsduring which time the radiochemical purity dropped to 85 %.

REFERENCES

1. Gibberelline part 105; for part 104 see Voigt, B. and Adam, G. - Z. Chem. 23: 177 (1983)
2. Lischewski, M. and Adam, G. - J. Lab. Comp. Radiopharm. 19: 1231 (1982)
3. Lischewski, M., Adam, G., Liebisch, H.-W. and Pleiß, U. - J. Lab. Comp. Radiopharm. 19: 725 (1982)
4. Lischewski, M. and Adam, G. - Tetrahedron 36: 1237 (1980)
5. Pleiß, U. and Römer, J. - GDR patent 248 680/8 (8.2.1983)