Isolation of Adenosine from the Rhizomes of Polygonatum sibiricum

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Abstract ☐ The purine nucleoside, adenosine, mp. 233-4°, was isolated from the rhizomes of *Polygonatum sibiricum*. Its structure was characterized on the basis of spectral data.

Keywords Polygonatum sibiricum, Liliaceae, purine nucleoside, adenosine.

The rhizomes of *Polygonatum sibiricum* Redoute (Liliaceae) are one of the important traditional medicine in Korea. Recently we reported the isolation of two new steroidal saponins, sibiricosides A and B together with two known ones¹. Further investigation on this plant led to the isolation of an additional compound, adenosine (1) as a minor component. The present paper describes the isolation and characterization of this compound.

EXPERIMENTAL

General experimental procedure

Instrumentation used and isolation procedure of 1 were as described previously¹⁾.

Compound 1

Crystallized from aqueous MeOH as colorless needles: mp. 233-4°C, $[\alpha]_D^{\infty} = -59.5^{\circ}$ (c = 0.5. H₂O) [lit¹⁰, mp. 233-5°C; $[\alpha]_D = -59.7^{\circ}$]; IR v_{max}^{KBr} cm $^{-1}$ 3430, 3350 (NH₂), 3150 (OH), 1685, 1670, 1610 (NH), 1105 (C-N), 1032 (C-O), 825 (NH₂); UV $\lambda_{max}^{\text{MeOH}}$ nm (log ϵ), 208 (4.03), 260 (3.83); $^{-1}$ H-NMR (300 MHz, DMSO-d₆) and $^{-13}$ C-NMR (75.5 MHz, DMSO-d₆); see Table I; MS (30 eV), m/z (rel. int.) 267 (M $^{+}$, 1.2), 250 (1.0), 238 (3.4), 237 (9.7), 220 (1.4), 194 (1.7), 179 (10.1), 178 (35.1), 165 (21.0), 164 (96.5), 137 (19.9), 136 (95.5), 135 (100), 134 (1.7), 121 (1.3), 119 (8.5), 109 (7.5), 108 (29.3), 81 (4.0), 73 (7.2).

RESULTS AND DISCUSSION

The compound (1), mp. 233-4°C, was positive to anisaldehyde-H₂SO₄ reagent and showed characteristic bands at 3150-3430 (OH and NH₂), 1685, 1670, 1610 (NH), 1105 (C-N), and 825 (NH₂) cm⁻¹ in its IR spectrum. In the UV spectrum, it exhibited strong absorption peaks at 208 and 260 nm which was suggested that compound 1 was a purine nucleoside²⁾. This result was further supported by the EI mass spectrum of 1. The mass spectrum of 1 showed a molecular ion at m/z 267 along with a base peak at m/z 135. Other important peaks at m/z 237, 178, 164 and 136 with great relative abundance are characteristic of adenosine and its analogs³⁻⁵⁾. The ¹H-NMR spectrum of 1 showed two aromatic singlet signals at δ 8.133 and 8.335. The ¹H-¹H COSY spectra of 1 enabled the complete assignment of all proton signals as indicated in Table I and were consistent with the structure assigned. Finally the ¹³C-NMR spectral data of 1 confirmed the structure of compound 1. Further confirmation of the assignment of the structure of 1 as adenosine was obtained by the direct comparison with an authentic sample (mmp and ¹H-NMR).

Adenosine is widely distributed in plants⁽⁻⁰⁾ and has been isolated as a platelet aggregation inhibitor from *Angelica acutiloba*⁽⁰⁾, *Ganoderma lucidum*⁽¹⁾ and two *Allium* plants such as *A. bakeri* and *A. sativum*⁽²⁾.

Proton	δ H[multiplicity, J (Hz)]	¹H-¹H COSY	Carbon	δС
H-2	8.133 s		C-2	152.34
H-8	8.335 s	H-1'	C-4	149.03
H-1'	5.879 d (6.2)	H-2', H-8	C-5	119.36
H-2'	4.604 m	H-3', H-1'	C-6	156.09
H-3'	4.147 m	H-4', H-2'	C-8	139.90
H-4'	3.966 dd (6.6, 3.4)	H-5'ab, H-3'	C-1'	87.91
H-5'a	3.677 dt (3.6, 12.2)	H-5'b, H-4'	C-2'	73.42
H-5'b	3.588 ddd (3.4, 6.6, 12.2)	H-5'a, H-4'	C-3'	70.64
			C-4'	85.88
			C-5'	61.66

Table I. 1H- and 13C-NMR data for compound 1 in DMSO-d₆

However, this is the first isolation from this plant.

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