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FLAVONOIDS ISOLATED FROM THE AERIAL PARTS OF *CICER SONGARICUM*

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Abstract. The flavonoid composition of the aerial parts of *Cicer songaricum* Stephan ex DC. was investigated. Five flavonoid compounds were isolated from the ethyl acetate and n-butanol fractions using column chromatography. Their chemical structures were elucidated based on UV, IR, ^1H NMR, and ^13C NMR spectral data. The isolated compounds were identified as kaempferol, quercetin, isorhamnetin, isorhamnetin-3-*O*-β-D-glucopyranoside, and isoquercitrin. The obtained results indicate that *Cicer songaricum* is an important natural source of biologically active flavonoids.

Keywords: *Cicer songaricum*, flavonoids, kaempferol, quercetin, isorhamnetin, isoquercitrin, NMR spectroscopy.

Introduction. *Cicer songaricum* Stephan ex DC. is a perennial herbaceous plant belonging to the Fabaceae family and is distributed from foothill areas to middle mountain regions. It is widely distributed in the Tashkent, Namangan, Andijan, Fergana, Samarkand, Kashkadarya, Bukhara, and Surkhandarya regions of Uzbekistan. Previous studies on the aerial parts of *Cicer songaricum* growing in the mountainous regions of the Republic of Tajikistan reported the presence of approximately ten flavonoids, which were identified using chromatographic

methods as well as UV and IR spectral analyses [1]. However, detailed ^{1}H NMR and ^{13}C NMR spectroscopic investigations of these compounds were not performed. In the present study, the flavonoid composition of the aerial parts of *Cicer songaricum* collected from the mountainous regions of Uzbekistan was investigated, and the chemical structures of the isolated individual compounds were elucidated using ^{1}H NMR and ^{13}C NMR spectroscopy.

Materials and methods. The present study investigated the phenolic compounds present in the ethyl acetate fraction (2.0 g) and *n*-butanol fraction (4.0 g) obtained from a 96% ethanol extract of the aerial parts of *Cicer songaricum* (300 g) collected during the flowering stage. The fractions were separated by column chromatography on KSK-grade silica gel (100–200 mkm, Tianjin Sinomed Pharmaceutical, China). Elution was carried out using chloroform and chloroform–methanol mixtures with a stepwise increase in methanol concentration. The obtained eluates were further purified by rechromatography on Sephadex LH-20 (particle size 25–100 mkm, GE Healthcare Bio-Sciences AB, Sweden) and eluted with an ethanol–water mixture (8:2, v/v). As a result, five individual compounds were isolated and identified as kaempferol (1), quercetin (2), isorhamnetin (3), isorhamnetin-3-*O*-glucopyranoside (4), and isoquercitrin (5). The chemical structures of the isolated compounds were established by analysis of their UV, IR, ^{1}H NMR, and ^{13}C NMR spectral data and by comparison with published literature data.

Results and discussion. As a result of the investigation of the aerial parts of *Cicer songaricum*, five flavonoid compounds (1–5) were isolated. The physicochemical characteristics of the isolated flavonoids are presented in Table 1.

Table 1

Physicochemical characteristics of flavonoids isolated from the aerial parts of *Cicer songaricum*

№	Flavonoid	Molecular Formula	Melting Point (°C)
1	Kaempferol (3,4′,5,7-tetrahydroxyflavone) (1)	$C_{15}H_{10}O_6$	265–266
2	Quercetin (3,5,7,3′,4′-pentahydroxyflavone) (2)	$C_{15}H_{10}O_7$	310–312
3	Isorhamnetin (3,5,7,4′-tetrahydroxy-3′-methoxyflavone) (3)	$C_{16}H_{12}O_7$	306–307
4	Isorhamnetin-3-*O*-glucopyranoside (isorhamnetin-3-*O*-β-D-glucopyranoside) (4)	$C_{22}H_{22}O_{12}$	173–175
5	Isoquercitrin (quercetin-3-*O*-β-D-glucopyranoside) (5)	$C_{21}H_{20}O_{11}$	236–238

As a result of the phytochemical investigation of the aerial parts of *Cicer songaricum*, five flavonoid compounds (1–5) were isolated.

Kaempferol (1) was obtained as a yellow powder with a melting point of 305–306 °C. The structure of the compound was elucidated based on its **¹**H NMR and ^{13}C NMR spectral data.

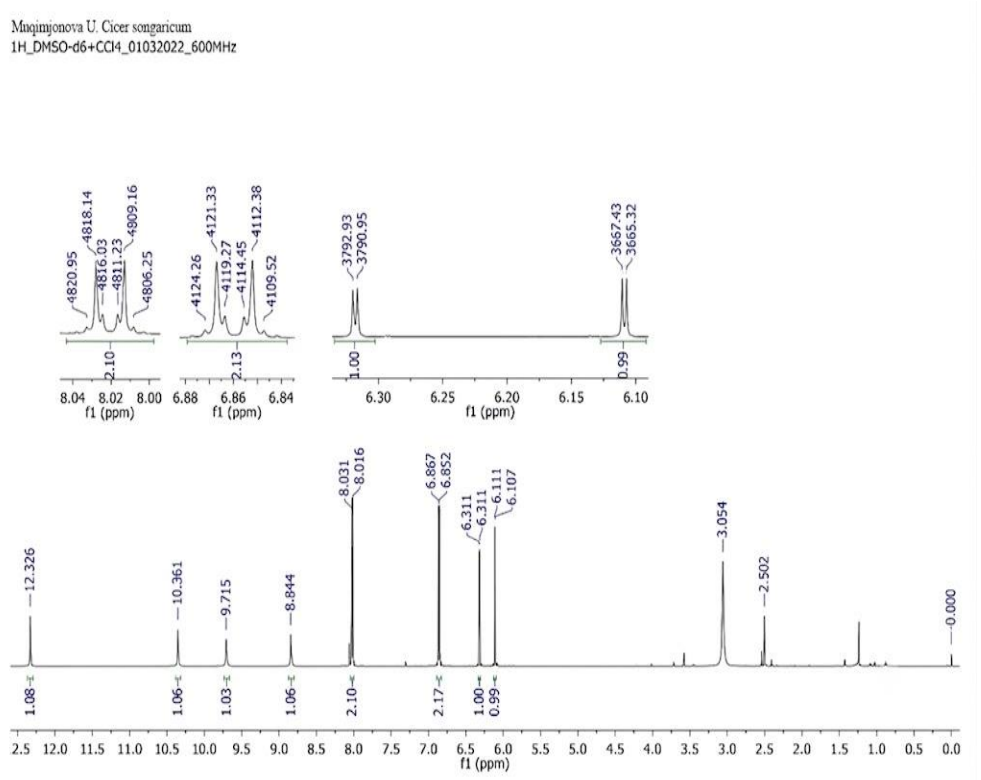


Figure 1. ^{1}H NMR spectrum of kaempferol

^{1}H NMR (600 MHz, DMSO-d_6 + CCl_4, δ, ppm, *J* in Hz): 12.33 (1H, s, 5-OH), 10.36 (1H, s, 7-OH), 9.71 (1H, s, 4′-OH), 8.84 (1H, s, 3-OH), 6.11 (1H, d, *J* = 2.0 Hz, H-6), 6.31 (1H, d, *J* = 2.0 Hz, H-8), 8.03 (2H, d, *J* = 9.0 Hz, H-2′ and H-6′), 6.87 (2H, d, *J* = 9.0 Hz, H-3′ and H-5′).

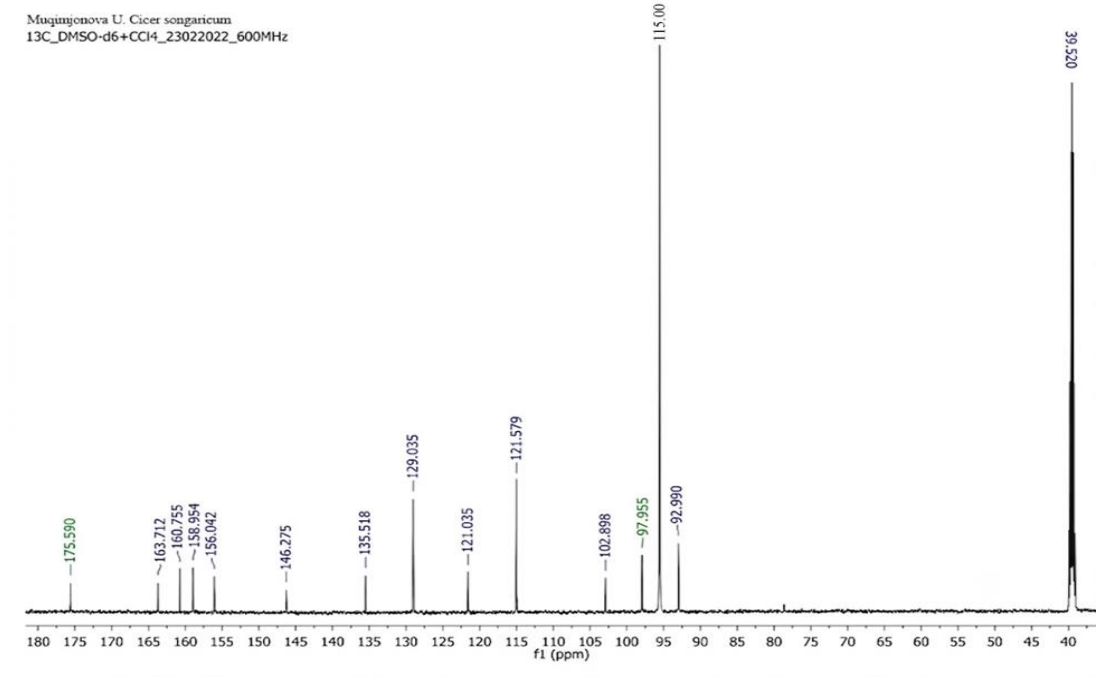
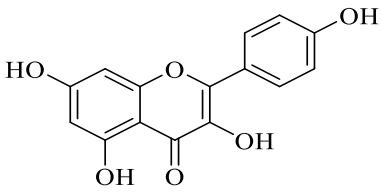


Figure 2. ^{13}C NMR spectrum of kaempferol

^{13}C NMR (150 MHz, DMSO-d_6, δ, ppm): 146.27 (C-2), 135.52 (C-3), 175.59 (C-4), 160.75 (C-5), 97.95 (C-6), 163.71 (C-7), 92.99 (C-8), 156.04 (C-9), 102.90 (C-10), 121.58 (C-1′), 129.03 (C-2′), 115.00 (C-3′), 158.95 (C-4′), 115.00 (C-5′), 129.03 (C-6′).

In the ¹H NMR spectrum, proton signals corresponding to the hydroxyl groups 5-OH, 7-OH, 4′-OH, and 3-OH were observed at δ 12.33, 10.36, 9.71, and 8.84 ppm, respectively. The aromatic protons H-6 and H-8 appeared at δ 6.11 and 6.31 ppm, while the B-ring protons H-2′/H-6′ and H-3′/H-5′ were detected at δ 8.03 and 6.87 ppm, respectively.

In the ¹³C NMR spectrum, signals corresponding to all carbon atoms of the kaempferol molecule were observed and were in good agreement with the literature data [2]. Based on the analysis of the spectral data and comparison with published **¹H** NMR and ¹³C NMR data for kaempferol, compound 1 was identified as kaempferol. To the best of our knowledge, this flavonoid was isolated for the first time from the aerial parts of wild-growing *Cicer songaricum* collected in Uzbekistan, and its chemical structure was established.



1

Quercetin (2) was obtained as a yellow powder with a melting point of 276–278 °C. The structure of the compound was elucidated based on **¹H and ¹³C NMR spectral data**.

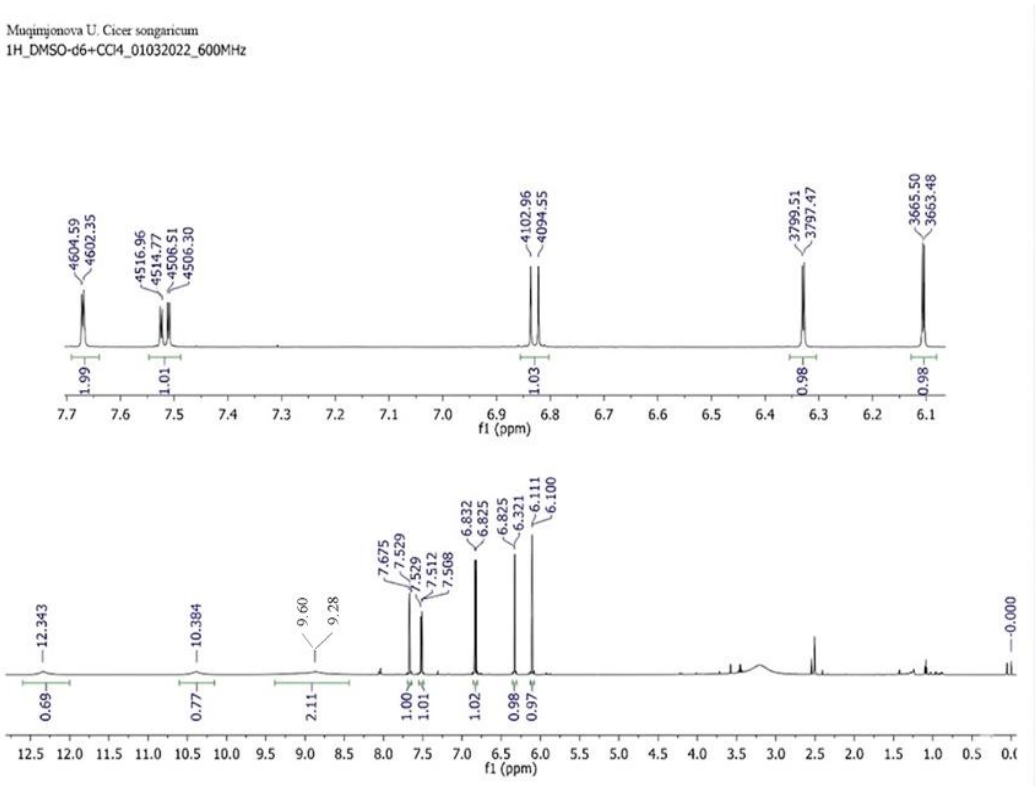


Figure 3. ¹H NMR spectrum of quercetin

¹H NMR (600 MHz, DMSO-d_6 + CCl_4, δ, ppm, *J* in Hz): 12.34 (1H, s, 5-OH), 10.38 (1H, s, 7-OH), 9.60 (1H, s, 4′-OH), 9.28 (1H, s, 3-OH), 6.11 (1H, d, *J* = 2.0 Hz, H-6), 6.32 (1H, d, *J*

= 2.0 Hz, H-8), 7.67 (1H, d, *J* = 2.2 Hz, H-2′), 6.83 (1H, d, *J* = 8.5 Hz, H-5′), 7.53 (1H, dd, *J* = 8.5; 2.2 Hz, H-6′).

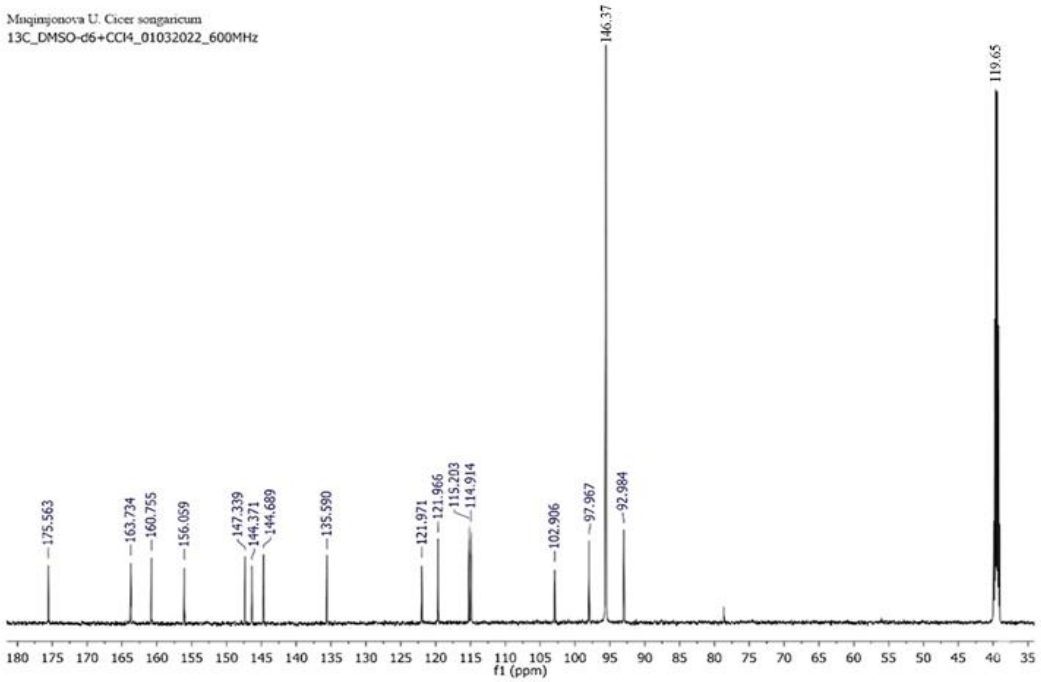
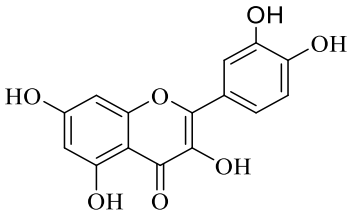


Figure 4. ^{13}C NMR spectrum of quercetin

^{13}C NMR (150 MHz, DMSO-d_6, δ, ppm): 146.37 (C-2), 135.59 (C-3), 175.56 (C-4), 160.75 (C-5), 97.97 (C-6), 163.73 (C-7), 92.99 (C-8), 156.06 (C-9), 102.91 (C-10), 121.97 (C-1′), 114.91 (C-2′), 144.69 (C-3′), 147.34 (C-4′), 115.20 (C-5′), 119.65 (C-6′).

In the ^{1}H NMR spectrum, proton signals corresponding to the hydroxyl groups 5-OH, 7-OH, 4′-OH, and 3-OH were observed at δ 12.34, 10.38, 9.60, and 9.28 ppm, respectively. The aromatic protons H-6 and H-8 appeared as doublets at δ 6.11 and 6.32 ppm. The B-ring protons H-2′, H-5′, and H-6′ were observed at δ 7.67, 6.83, and 7.53 ppm, respectively.

In the ^{13}C NMR spectrum, signals corresponding to all carbon atoms of the quercetin molecule were identified and were in good agreement with the literature data [3]. Based on the analysis of the NMR spectral data of compound 2 isolated from the aerial parts of *Cicer songaricum* collected in various regions of Uzbekistan, the compound was identified as quercetin. Comparison with published ^{1}H and ^{13}C NMR data confirmed that compound 2 is quercetin. This flavonoid was isolated for the first time from *Cicer songaricum*.



2

Isorhamnetin (3) was obtained as a yellow powder with a melting point of 305–306 °C. The structure of the compound was investigated based on ^{1}H and ^{13}C NMR spectral data.

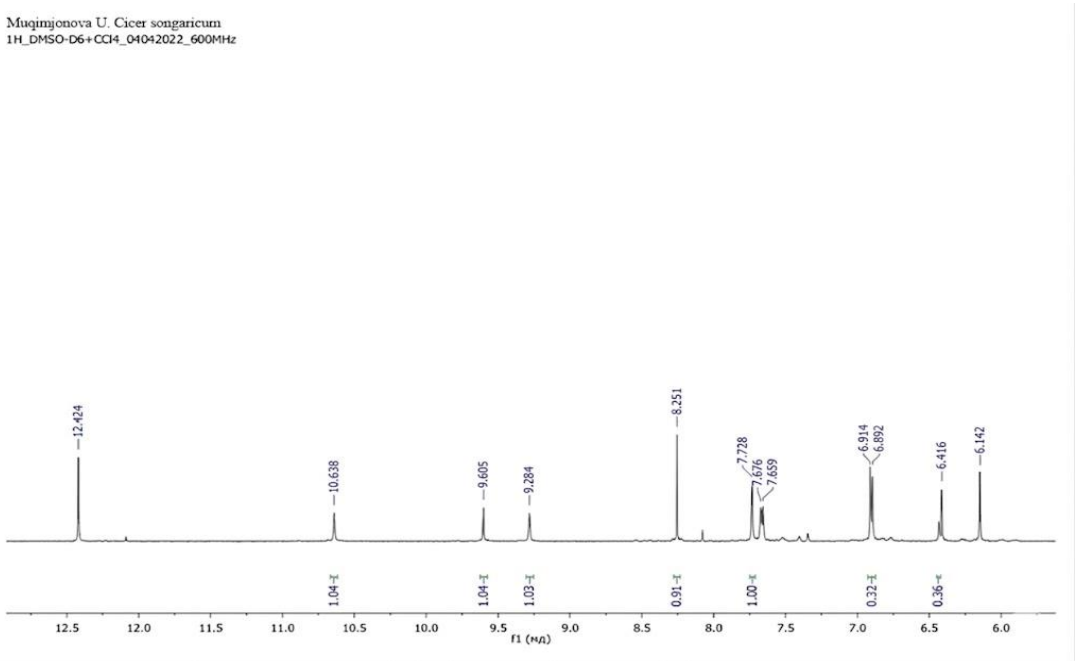


Figure 5. ^{1}H NMR spectrum of isorhamnetin

^{1}H NMR spectrum (600 MHz, DMSO-d_6 + CCl_4, δ, ppm, J/Hz): 12.42 (1H, s, 5-OH), 10.38 (1H, s, 7-OH), 9.60 (1H, s, 4′-OH), 9.28 (1H, s, 3-OH), 6.14 (1H, d, J = 2.0 Hz, H-6), 6.42 (1H, d, J = 2.0 Hz, H-8), 7.73 (1H, d, J = 2.1 Hz, H-2′), 6.90 (1H, d, J = 8.5 Hz, H-5′), 7.66 (1H, dd, J = 8.5, 2.1 Hz, H-6′), 3.85 (3H, s, 3′-OCH_3).

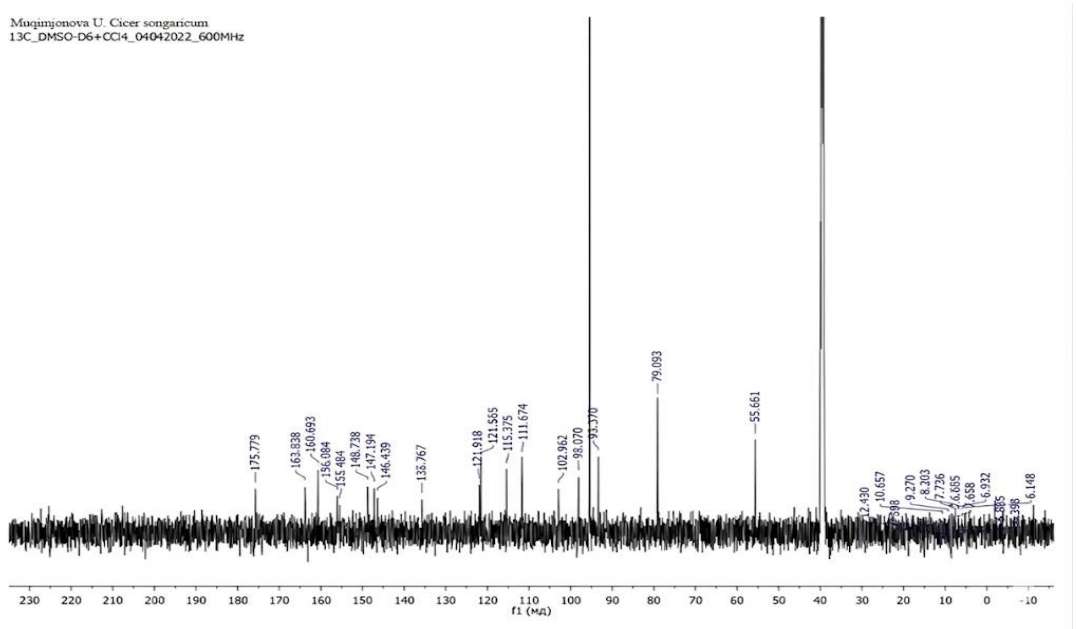


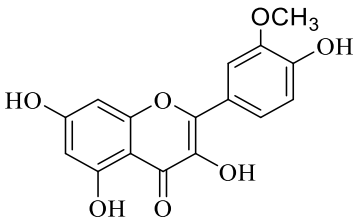
Figure 6. ^{13}C NMR Spectrum of Isorhamnetin

^{13}C NMR spectrum (150 MHz, DMSO-d_6, δ, ppm): 146.44 (C-2), 135.77 (C-3), 175.78 (C-4), 160.69 (C-5), 98.07 (C-6), 163.84 (C-7), 93.37 (C-8), 156.08 (C-9), 102.96 (C-10), 121.92 (C-1′), 111.67 (C-2′), 147.19 (C-3′), 148.74 (C-4′), 115.37 (C-5′), 121.56 (C-6′), 55.66 (3′-OCH_3).

In the ¹H NMR spectrum, proton signals corresponding to the hydroxyl groups 5-OH, 7-OH, 4′-OH, and 3-OH were observed at δ 12.42, 10.38, 9.60, and 9.28 ppm, respectively. The H-6 and H-8 protons of ring A appeared as doublets at δ 6.14 and 6.42 ppm, respectively. The H-2′, H-5′, and H-6′ protons of ring B were observed at δ 7.73, 6.90, and 7.66 ppm, respectively. In addition, the singlet signal at δ 3.85 ppm was assigned to the 3′-OCH_3 methoxy group.

In the ¹³C NMR spectrum, signals corresponding to all carbon atoms of the isorhamnetin molecule, as well as the 3′-OCH_3 methoxy group, were observed. Analysis of the spectral data and comparison with literature values led to the identification of compound 3 as 3,5,7,4′-tetrahydroxy-3′-methoxyflavone (isorhamnetin) [3].

For the first time, 3,5,7,4′-tetrahydroxy-3′-methoxyflavone (isorhamnetin) was isolated from the aerial parts of Cicer songaricum growing in Uzbekistan, and its structure was confirmed by ¹H NMR and ¹³C NMR spectroscopic analyses.



3

Isorhamnetin-3-O-β-D-glucopyranoside (4) was obtained as a yellow-colored compound with a melting point of 173–175 °C**.** The structure of the compound was elucidated using ¹H and ¹³C NMR spectroscopy**.**

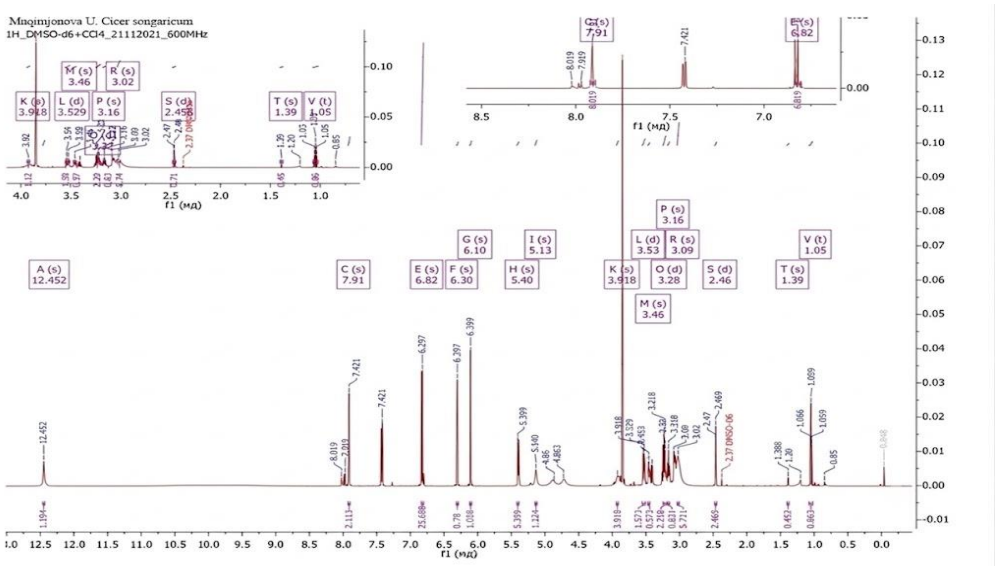


Figure 7. ¹H NMR spectrum of isorhamnetin-3-O-β-D-glucopyranoside

¹H NMR spectrum (600 MHz, DMSO-d_6 + CCl_4, δ, ppm, J/Hz): 6.29 (1H, d, J = 2.1 Hz, H-6), 6.34 (1H, d, J = 2.1 Hz, H-8), 7.92 (1H, d, J = 2.1 Hz, H-2′), 6.89 (1H, d, J = 8.3 Hz, H-5′), 7.42 (1H, dd, J = 8.3, 2.1 Hz, H-6′), 5.40 (1H, d, J = 7.6 Hz, H-1″), 3.21 (1H, m, H-2″), 3.33 (1H, t, J = 8.4 Hz, H-3″), 3.22 (1H, t, J = 8.4 Hz, H-4″), 3.09 (1H, ddd, J = 9.6, 4.6, 2.5 Hz, H-

5″), 3.46 (1H, dd, J = 12.0, 4.8 Hz, H-6″a), 3.54 (1H, m, H-6″b), 3.91 (3H, s, OCH_3), 12.42 (1H, br s, 5-OH), 5.14 (1H, br s, OH), 4.86 (1H, br s, OH), 4.76 (1H, br s, OH).

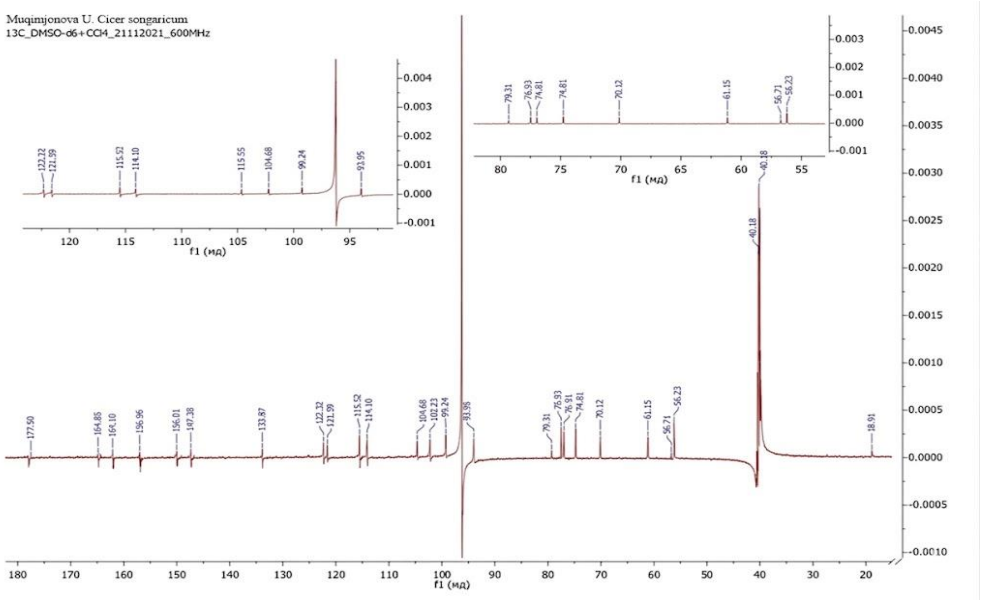


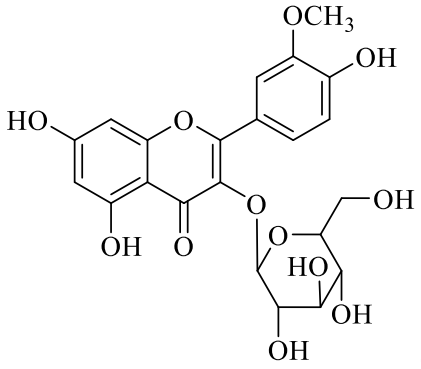
Figure 8. ^{13}C NMR spectrum of isorhamnetin-3-O-β-D-glucopyranoside

^{13}C NMR spectrum (150 MHz, DMSO-d_6 + CCl_4, δ, ppm): 156.01 (C-2), 133.87 (C-3), 177.50 (C-4), 159.98 (C-5), 99.24 (C-6), 164.85 (C-7), 93.96 (C-8), 156.01 (C-9), 104.68 (C-10), 121.97 (C-1′), 114.10 (C-2′), 147.38 (C-3′), 150.01 (C-4′), 114.10 (C-5′), 121.97 (C-6′), 99.24 (C-1″), 74.81 (C-2″), 76.91 (C-3″), 70.12 (C-4″), 76.91 (C-5″), 61.15 (C-6″).

In the ^{1}H NMR spectrum, the H-6 and H-8 protons of the flavonoid aglycone were observed as doublets at δ 6.29 and 6.34 ppm, respectively. The H-2′, H-5′, and H-6′ protons of ring B were recorded at δ 7.92, 6.89, and 7.42 ppm, respectively. The doublet signal of the anomeric proton (H-1″) at δ 5.40 ppm, together with its coupling constant (J = 7.6 Hz), indicated the β-configuration of the glucose moiety. In addition, a singlet signal corresponding to the methoxy group (OCH_3) was observed at δ 3.91 ppm.

In the ^{13}C NMR spectrum, signals corresponding not only to the carbon atoms of the flavonoid skeleton but also to the C-1″–C-6″ carbons of the glucopyranose residue were identified. Comparative analysis of the spectral data with literature values led to the identification of compound 4 as isorhamnetin-3-O-β-D-glucopyranoside [3].

For the first time, isorhamnetin-3-O-β-D-glucopyranoside was isolated from the aerial parts of Cicer songaricum growing in Uzbekistan, and its structure was confirmed by ^{1}H NMR and ^{13}C NMR spectroscopic analyses.



4

Isoquercitrin (quercetin-3-O-β-D-glucopyranoside) (5) was obtained as a yellow powder with a melting point of 236–238 °C**.** The structure of the compound was elucidated on the basis of ^{1}H and ^{13}C NMR spectroscopic data**.**

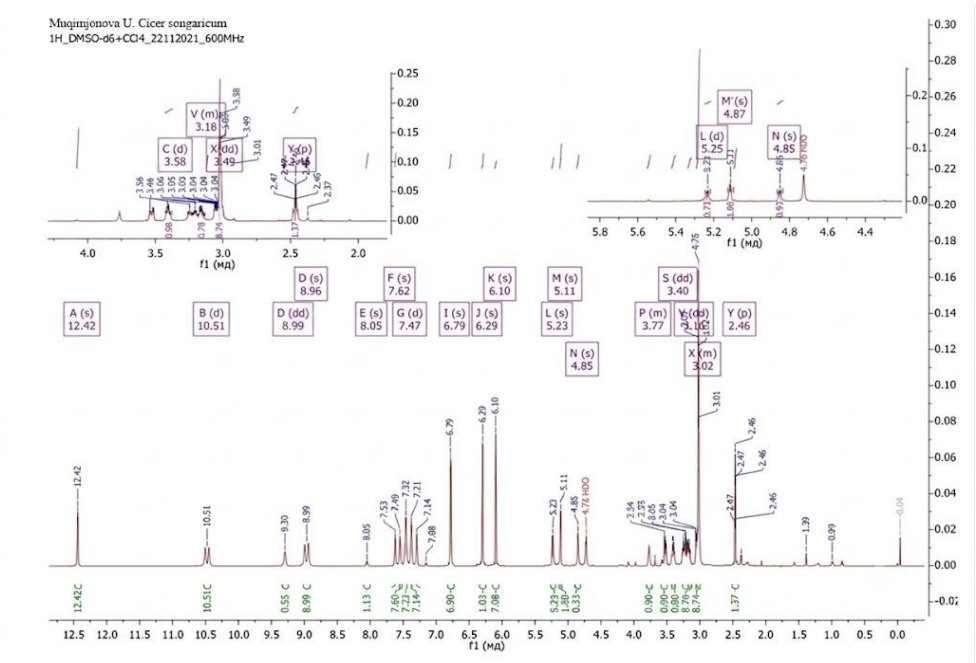


Figure 9. ^{1}H NMR spectrum of isoquercitrin

^{1}H NMR spectrum (600 MHz, DMSO-d_6 + CCl_4, δ, ppm, J/Hz): 6.10 (1H, d, J = 2.1 Hz, H-6), 6.29 (1H, d, J = 2.1 Hz, H-8), 7.53 (1H, d, J = 2.2 Hz, H-2′), 6.79 (1H, d, J = 8.4 Hz, H-5′), 7.49 (1H, dd, J = 8.4, 2.2 Hz, H-6′), 5.23 (1H, d, J = 7.6 Hz, H-1″), 3.33 (1H, ddd, J = 8.3, 7.8, 3.9 Hz, H-2″), 3.34 (1H, td, J = 8.6, 4.1 Hz, H-3″), 3.20 (1H, td, J = 8.6, 4.8 Hz, H-4″), 3.05 (1H, ddd, J = 9.3, 5.0, 2.6 Hz, H-5″), 3.76 (1H, m, H-6″a), 4.85 (1H, ddd, J = 11.6, 5.5, 2.4 Hz, H-6″b), 12.42 (1H, br s, 5-OH).

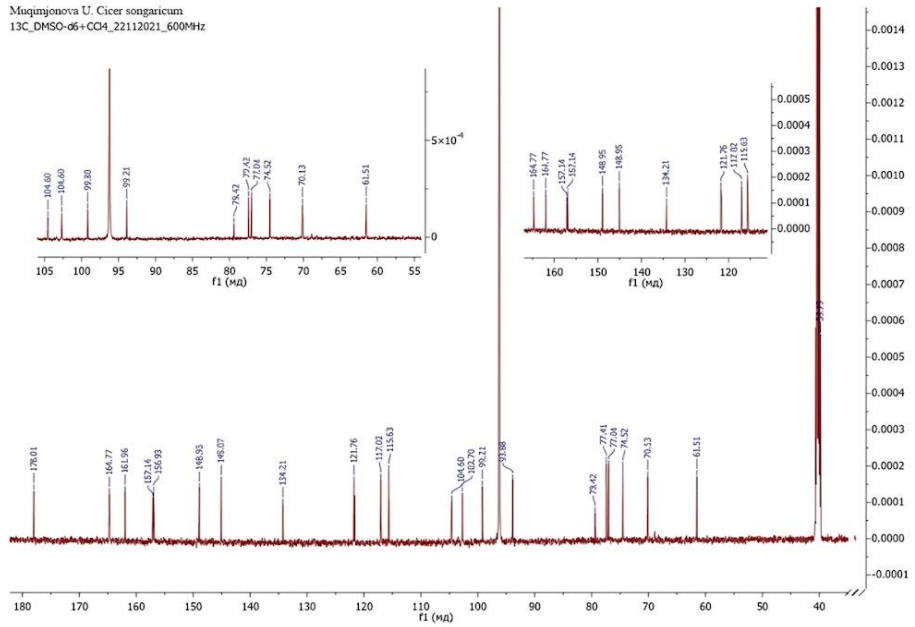
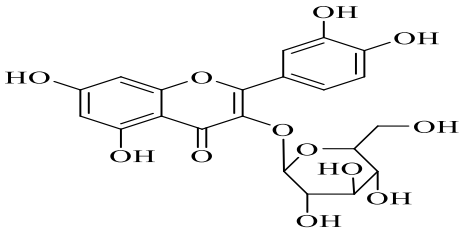


Figure 10. ^{13}C NMR spectrum of isoquercitrin

^{13}C NMR spectrum (150 MHz, DMSO-d_6 + CCl_4, δ, ppm): 157.14 (C-2), 133.56 (C-3), 178.10 (C-4), 161.98 (C-5), 99.71 (C-6), 164.77 (C-7), 93.88 (C-8), 156.93 (C-9), 104.60 (C-10), 120.76 (C-1′), 115.63 (C-2′), 145.07 (C-3′), 148.95 (C-4′), 114.95 (C-5′), 121.08 (C-6′), 102.70 (C-1″), 74.04 (C-2″), 77.41 (C-3″), 69.45 (C-4″), 77.41 (C-5″), 61.05 (C-6″).

In the ^{1}H NMR spectrum, the H-6 and H-8 protons of the flavonoid aglycone were observed as doublets at δ 6.10 and 6.29 ppm, respectively. The H-2′, H-5′, and H-6′ protons of ring B were recorded at δ 7.53, 6.79, and 7.49 ppm, respectively. The doublet signal of the anomeric proton (H-1″) at δ 5.23 ppm, together with its coupling constant (J = 7.6 Hz), indicated the β-configuration of the glucose residue. The remaining protons of the glucopyranose moiety were observed in the δ 3.05–3.76 ppm range. In addition, a broad singlet signal corresponding to the 5-OH hydroxyl group was recorded at δ 12.42 ppm.

In the ^{13}C NMR spectrum, signals corresponding to the carbon atoms of the flavonoid skeleton as well as the C-1″–C-6″ carbons of the glucopyranose residue were identified. Analysis of the spectral data and comparison with literature values led to the identification of compound 5 as quercetin-3-O-β-D-glucopyranoside (isoquercitrin) [4].



5

Conclusion. The results of the conducted studies demonstrated that the aerial parts of Cicer songaricum are a rich source of biologically active phenolic compounds. Compounds 2 and 5 **(**quercetin and isoquercitrin, respectively) were identified and characterized for the first time from this plant in the present study.

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