Research of 5 extracts of wild Amur grape *Vitis amurensis* Rupr. and identification of its polyphenolic composition by tandem mass spectrometry (HPLC-MS / MS)

Mayya P. Razgonova^{1,2,*}, Alexander M. Zakharenko^{1,2}, Irina V. Derbush¹, Andrey S. Sabitov¹, and Kirill S. Golokhvast^{1,2,3,4}

¹N.I. Vavilov All-Russian Institute of Plant Genetic Resources, B. Morskaya, 42-44, 190000 Saint-Petersburg, Russian Federation

²Far Eastern Federal University, Sukhanova, 8, 690950 Vladivostok, Russian Federation

³Pacific Geographical Institute, Far Eastern Branch of the Russian Academy of Sciences, Radio, 7, 690041 Vladivostok, Russian Federation

⁴Siberian Federal Scientific Centre of Agrobiotechnology, Centralnaya, Presidium, 633501 Krasnoobsk

Abstract. *Vitis amurensis* Ruprecht contains a large number of polyphenolic compounds which are biologically active components. For the most efficient and safe extraction supercritical carbon dioxide was used. In this work, for the first time, a comparative metabolomic study of biologically active substances of wild grapes collected from five different places of the Primorsky and Khabarovsk territories is carried out. To identify target analytes in ethanol extracts of grape berries, high performance liquid chromatography (HPLC) was used in combination with an amaZon SL ion trap (manufactured by BRUKER DALTONIKS, Germany) equipped with an ESI electrospray ionization source in negative and positive ion modes. The mass spectrometer was used in the scan range m/z 100 - 1.700 for MS and MS / MS. Used fragmentation of the 4th order. Primary mass spectrometric results showed the presence of 89 biologically active compounds corresponding to the species *V. amurensis*, moreover, salvianolic acids F, D and G, oleanolic, ursolic, myristoleic acids, berbericin, mearnssetin, esculin, nevadensin, stigmaterol, fucosterol, phlorizin, L-tryptophan identified for the first time in *V. amurensis*.

1 Introduction

Researchers attribute the appearance of the first representatives of the *Vitaceae* family, belonging to the genus *Vitis*, to the Upper Cretaceous period, when there were already types of plants that were very similar in leaves to vines. The absence of seeds does not allow, however, to have complete confidence in their belonging to the genus *Vitis* [1, 2]. To these types, researchers include the *Vitis dakotana* Berry vine found in the Upper Cretaceous

* Correspondence: razgonova.mp@dvfu.ru

sediments in Harding County in South Dakota, which is very similar in appearance to modern vines [3, 4].

The evolution of grape plants approaching the cultivated vine, judging by the fossil finds, took place especially intensively in Central and Southern Europe during the second half of the Tertiary period and then especially in the Quaternary period. On the territory of Russia, quite a lot of finds of fossils are also known belonging to the genera *Cissites*, *Ampelopsis*, *Parthenocissus* and especially to the genus *Vitis*: *V. sachalinensis* Krysh. and *V. crenata* Heer on Sakhalin, *V. teutonica* A. Br. – near Taganrog and on the Irtysh River, as well as *V. praevinifera* Sap. - on the Krynka river. All these data show that the evolution of the vine on the territory of Russia proceeded from ancient times. And now in Russia in many areas wild grapes *V. sylvestris* Gmel grow [5, 6, 7].

Very little information is available about the culture of East Asian grape varieties. *V. lanata* Roxb is cultivated in eastern India. and *V. tomentosa* Heyne, in Japan and in Korea – *V. Thunbergii* Sieb. et Zucc. called *V. Seiboldii* hort [8]. More complete information is available regarding *V. amurensis* Rupr., which was first introduced into the culture by I.V. Michurin. In his work "Results of half a century of work" I.V. Michurin describes four forms of *V. amurensis* Rupr., which were isolated in the Far East [9, 10].

For the isolation of biologically active substances, ripe fruits, fruit skins, combs, leaves, seeds, vine bark, red grape wine are used. Fruits contain 65-85% water, 10-33% sugar (glucose and fructose), phlobaphene, gallic acid, quercetin, enin, glycosides - monodelphinidin and didelphinidin, acids (malic, silicic, salicylic, phosphoric, tartaric, citric, etc.) pectin and tannins, potassium, magnesium, calcium, manganese, cobalt, iron and vitamins: B₁, B₂, B₆, B₁₂, A, C, P, PP, folic acid, and enzymes.

The dominant class of biologically active compounds of fruits, and especially grape ridges, are bioflavonoids and, in particular, the so-called complexes of oligomeric proanthocyanidins or condensed tannins, which are polymeric forms of flavonoids from the group of catechins [11].

In European medicine, until recently, grapes were widely used as a means of therapy and rehabilitation for a wide range of diseases: chronic recurrent inflammatory processes, tuberculosis, kidney disease, arterial hypertension, etc.

The aim of this work was a comparative metabolomic study of biologically active substances of wild grapes harvested in five different places in the Russian Far Eastern taiga in the Primorsky and Khabarovsk territories. High performance liquid chromatography (HPLC) in combination with a BRUKER DALTONIKS ion trap (tandem mass spectrometry) was used to identify target analytes in extracts. This paper presents a detailed study of the metabolomic composition of grape juice from fruits taken from five habitats of *V. amurensis* in the Far East: Pakhtusov Islands and Rikord Island (Peter the Great Bay, Sea of Japan), the vicinity of Artem (Primorsky Territory), the vicinity of the river Arsenyevka (Primorsky Territory), environs of Vyazemsky (Khabarovsk Territory).

2 Materials and methods

2.1 Materials

The object of the study was the berries of the wild grape *V. amurensis*, collected in the floodplain of the Arsenyevka River, Primorsky Territory (N. 44 ° 52'18 ", E 133 ° 35'12"; brown-gley bleached soils) in the vicinity of Vyazemsky, Khabarovsk Territory (N 47 ° 32'15 ", E 134 ° 45'20"; podzolic-brown forest heavy loamy soils), in the vicinity of Artem, Primorsky Territory (St. lat. 43 ° 21'34 ", E 132 ° 11'19"; yellow-brown earth soils), on Rikord Island, Peter the Great Bay (N 42 ° 52'54 ", E 131 ° 40'06 "; yellow-brown earth

soils), on Pakhtusov Islands, Peter the Great Bay (N 42 ° 53'57", E 131 ° 38'45 "; yellow-brown soil). The grapes were harvested at the end of August and September 2020. All samples morphologically corresponded to the pharmacopoeial standards of the State Pharmacopoeia of the Russian Federation [12].

2.2 Methods

2.2.1 Fractional maceration

To obtain highly concentrated extracts, fractional maceration was applied. In this case, the total amount of the extractant (ethyl alcohol of reagent grade) is divided into 3 parts and is consistently infused with grapes with the first part, then with the second and third. The infusion time of each part of the extractant was 7 days.

2.2.2 High performance liquid chromatography

To perform the separation of multicomponent mixtures, a Shimadzu LC-20 Prominence HPLC high pressure liquid chromatograph (Shimadzu, Japan) was used, equipped with a UV detector and a Shodex ODP-40 4E reverse phase column. The gradient elution program is as follows: 0.0 – 4 min, 100% CH₃CN; 4 - 60 min, 100% – 25% CH₃CN; 60 - 75 min, 25% – 0% CH₃CN; control wash 75 - 120 min 0% CH₃CN. All HPLC analysis was done with an SPD-20A UV-VIS detector (Kanda-Nishikicho 1-chrome, Shimadzu, Chiyoda-ku, Tokyo, Japan) at 230 nm and 330 nm; temperature 17 ° C. The injection volume was 1 ml.

2.2.3 Tandem mass spectrometry

Mass spectrometric data were obtained using an amaZon SL ion trap (manufactured by BRUKER DALTONIKS, Germany) equipped with an ESI electrospray ionization source in negative and positive ion modes. The optimized parameters are obtained as follows: ionization source temperature: 70 ° C, gas flow: 4 L / min, nebulizer gas (nebulizer): 7.3 psi, capillary voltage: 4500 V, end plate bend voltage: 1500V, fragmentary: 280V, collision energy: 60 eV. The mass spectrometer was used in the scan range m/z 100 – 1.700 for MS and MS / MS. Used fragmentation of the 4th order.

3 Research results

Clarification of the metabolomic composition is an extremely important result in the system of biochemical analysis. In this work, the HPLC-MS / MS method was used with additional ionization and analysis of fragmented ions. High-precision mass spectrometric data were recorded on an AMAZON SL BRUKER DALTONIKS ion trap equipped with an ESI source in the negative / positive ion mode. A total of 300 peaks of the isolated target analytes were found on the ion chromatogram (**Fig. 1**).

Fig. 1. Distributed graph of tandem mass spectrometry of the analyzed target analytes of the EtOH extract of *V. amurensis* (Vyazemsky, Khabarovsk Territory), represented by an ion chromatogram (Brown line – graph of the intensity of the signal of positive ions; blue line – graph of the intensity of the signal of negative ions; black line – total intensity of positive ions; blue line – total intensity of negative ions).

Based on the measurement results, a unified system table of molecular masses and fragmented ions of target analytes isolated in extracts of *V. amurensis* was compiled. (**Table 1**).

Table 1. Metabolome analysis of biologically active substances isolated from extracts of *V. amurensis*

№	Identification	Formula	Observed mass [M-H] ⁻	Observed mass [M+H] ⁺	MS/MS Stage 1 fragmentation	MS/MS Stage 2 fragmentation	MS/MS Stage 2 fragmentation	References
1	Malic acid	C ₄ H ₆ O ₅	133		115			[22, 32, 57, 66]
2	Tartaric acid	C ₄ H ₆ O ₆	149		131			[32, 66]
3	Umbelliferone	C ₉ H ₆ O ₃	161		115			[29, 36]
4	<i>p</i> -Coumaric acid	C ₉ H ₈ O ₃		165	146	119		[13, 21, 22, 26, 29, 46]
5	Gallic acid	C ₇ H ₆ O ₅		171	126			[13, 20, 29, 36, 53]
6	Indole-3-carboxylic acid	C ₁₀ H ₉ NO ₂		176	130			[50]
7	Esculetin	C ₉ H ₆ O ₄		179	133	115		[29, 71]
8	Caffeic acid	C ₉ H ₈ O ₄	179		133			[13, 15, 29, 46, 55]
9	Citric acid	C ₆ H ₈ O ₇	191		111; 173; 143; 127			[42, 57, 59, 66]
10	Quinic acid	C ₇ H ₁₂ O ₆	191		111; 173	111		[22, 29, 30, 42, 53, 57, 61]

11	Dihydroferulic acid	C₁₀H₁₂O₄	195		159; 129; 113	122		[27, 30, 38]
12	Ethyl gallate	C₉H₁₀O₅	197		169	125		[31]
13	L-Tryptophan	C₁₁H₁₂N₂O₂	205		188	146; 170; 118	144; 118	[43, 59]
14	Sinapic acid	C₁₁H₁₂O₅	225		179; 153; 115	133; 115	115	[13, 19, 29, 56, 57]
15	Myristoleic acid	C₁₄H₂₆O₂	227		209; 181; 155	199; 181; 127		[30]
16	Resveratrol	C₁₄H₁₂O₃	229		142; 184	114		[30, 60]
17	Apigenin	C₁₅H₁₀O₅	271		253; 181	137		[13, 34, 43, 58, 67]
18	Naringenin	C₁₅H₁₂O₅	273		227; 155	209	139	[29, 53, 60, 62]
19	Linolenic acid	C₁₈H₃₀O₂	279		260; 176; 120			[19, 71]
20	Kaempferol	C₁₅H₁₀O₆	287		269; 227; 153	153		[29, 42, 45, 68]
21	Luteolin	C₁₅H₁₀O₆	287		271; 225; 175	158		[34, 43, 60, 67]
22	Dihydrokaempferol	C₁₅H₁₂O₆	289		271; 199; 127	243; 189; 118		[24, 41, 54]
23	Catechin	C₁₅H₁₄O₆	289		245; 205	203	188	[13, 25, 29, 53, 57, 60]
24	Epicatechin	C₁₅H₁₄O₆	291		272; 175; 130	157	140	[13, 29, 46, 53]
25	9-Oxo-10E,12Z-octadecanoic acid	C₁₈H₃₀O₃	295		249; 165	220; 125		[30, 71]
26	Caffeoylmalic acid	C₁₃H₁₂O₈	295		133;179; 148; 119	115		[57]
27	Coutaric acid	C₁₃H₁₂O₈	295		163	119	119	[29]
28	Kaempferide	C₁₆H₁₂O₆	301		283	265; 239; 211; 185; 133	211; 151	[45, 70, 74]
29	Ellagic acid	C₁₄H₆O₈	303		172;158	144; 127; 116		[29, 46, 52, 59]
30	Quercetin	C₁₅H₁₀O₇	303		285; 163	267; 159	239	[13, 25, 29, 51, 56, 60, 62, 68]
31	Hesperitin	C₁₆H₁₄O₆	301		257; 151	228; 189		[16, 29, 60]

32	Dihydroquercetin	C₁₅H₁₂O₇		305	259; 149	199; 241; 159	171	[29, 60, 64]
33	Caftaric acid	C₁₃H₁₂O₉	311		149; 221	131		[17, 22, 29, 55]
34	Salvianolic acid F	C₁₇H₁₄O₆		315	269; 243; 213; 185; 144	207; 181; 153	179; 161; 133	[22]
35	Protocatechuic acid-O-hexoside	C₁₃H₁₆O₉	315		153; 298	151		[22, 57, 62]
36	Dihydroxybenzoyl-hexoside	C₁₃H₁₆O₉	315		153; 253	151; 184		[44]
37	Myricetin	C₁₅H₁₀O₈	317		273; 191	255; 229; 205; 187; 163; 125	227	[29, 30, 36, 51, 60]
38	Fertaric acid [Fertarate]	C₁₄H₁₄O₉	325		193; 149	134		[29]
39	<i>p</i>-Coumaric acid-O-hexoside	C₁₅H₁₈O₈	325		193; 163	119		[30, 50, 51, 57, 62]
40	Galloyl glucose	C₁₃H₁₆O₁₀	331		313; 195	166		[59]
41	Gallic acid hexoside	C₁₃H₁₆O₁₀	331		271; 169; 125	125		[48]
42	Mearnsetin	C₁₆H₁₂O₈		333	318; 301; 273; 245; 193; 165; 139	301; 289; 271; 219; 192; 153; 136		[52]
43	Esculin	C₁₅H₁₆O₉	339		177; 293	131		[29, 30, 34, 71]
44	Salvianolic acid G	C₁₈H₁₂O₇		341	323; 295; 255; 195; 159	305		[35, 69]
45	Nevadensin	C₁₈H₁₆O₇	343		328; 259	313	269	[45, 69]
46	Palmatine	C₂₁H₂₂NO₄		353	335; 235	317; 235; 137		[18, 71]
47	Hexose-hexose-N-acetyl	C₁₄H₂₅NO₁₀	366		186; 142	142		[40]
48	Fraxin	C₁₆H₁₈O₁₀		371	208; 352	135		[29]
49	Fraxetin-7-O-beta-glucuronide	C₁₆H₁₆O₁₁		385	367; 272; 209; 175; 143	158		[73]
50	Polydatin	C₂₀H₂₂O₈	389		227; 343	184	143	[46, 60]

51	Fucosterol	C₂₉H₄₈O		413	395; 355; 271; 194; 119	297; 199	268; 187	[30]
52	Stigmasterol	C₂₉H₄₈O		413	301; 259; 189	171		[14, 20, 30]
53	Salvianolic acid D	C₂₀H₁₈O₁₀	417		373	329	287; 209	[21, 22]
54	Apigenin-7-O-glucoside	C₂₁H₂₀O₁₀		433	414; 287; 186	241; 158		[15, 17, 29, 34, 42, 43, 58]
55	Pelargonidin-3-O-glucoside (callistephin)	C₂₁H₂₁O₁₀		433	414; 271; 172	172; 226	116	[23, 59, 65]
56	Phlorizin	C₂₁H₂₄O₁₀		437	397; 217	377		[26, 29, 30, 46, 53, 57]
57	Catechin gallate	C₂₂H₁₈O₁₀	441		289; 169	245; 205	203	[29, 33]
58	Kaempferol-3-O-galactoside	C₂₁H₂₀O₁₁		449	287	269; 217		[25, 29]
59	Eriodictyol-7-O-glucoside	C₂₁H₂₂O₁₁	449		269	207; 251	165	[16, 29, 42, 63]
60	Dihydrokaempferol glucoside	C₂₁H₂₂O₁₁	449		287	227; 269	225; 149	[46]
61	Oleanoic acid	C₃₀H₄₈O₃		457	439; 411; 365; 337; 293; 248; 205	364; 337; 309; 219	337; 319; 301; 279; 247; 232; 219	[45, 61]
62	Ursolic acid	C₃₀H₄₈O₃		457	411; 393; 365; 337; 279; 247	365; 337; 292; 279; 247; 219; 205		[20, 44, 61, 69]
63	Isorhamnetin 3-O-rhamonoside	C₂₂H₂₂O₁₁	461		315; 152; 219			[52, 53]
64	Peonidin-3-O-glucoside	C₂₂H₂₃O₁₁ +		463	301	286; 258	268; 258; 230; 202; 174; 121	[28, 29, 37, 59, 65]
65	Hyperoside	C₂₁H₂₀O₁₂	463		301;179	257; 179	255;147	[13, 25, 26, 29, 51, 60, 63]
66	Quercetin 3-O-glucoside	C₂₁H₂₀O₁₂		465	303	285; 257; 229; 201; 150	229; 201; 155	[15, 25, 29, 34, 43, 46]

67	Taxifolin-3-<i>O</i>-glucoside	C₂₁H₂₂O₁₂		467	449; 303; 188	287; 132	260	[29]
68	Quercetin-3-<i>O</i>-glucuronide	C₂₁H₁₈O₁₃	477		301; 179	273; 179	151	[25, 29, 57, 65]
69	Isorhamnetin 3-<i>O</i>-glucoside	C₂₂H₂₂O₁₂		479	317; 287	301; 257	274; 228; 149	[15, 25, 29, 52]
70	Myricetin-3-<i>O</i>-galactoside	C₂₁H₂₀O₁₃	479		299; 153	271	243; 171	[13, 25, 29, 51, 63]
71	Dimethylellagic acid hexose	C₂₂H₂₀O₁₃		493	331	299; 270; 242; 179;150	270; 225	[59]
72	Malvidin 3-<i>O</i>-glucoside	C₂₃H₂₅O₁₂		493	331	315; 179	315; 179	[29, 37, 65]
73	5-<i>O</i>-(4'-<i>O</i>-<i>p</i>-coumaroyl glucosyl)quinic acid	C₂₂H₂₈O₁₃		501	339; 277; 203	277		[34]
74	<i>p</i>-Coumaroylcaffeoylquinic acid	C₂₅H₂₄O₁₁		501	355; 483; 181	225; 281; 193; 120	133	[61]
75	Coumaric acid derivative	C₃₀H₃₀O₇		503	457; 411; 382; 339; 293	409; 391; 367; 339; 293	323; 293; 233; 205	[57]
76	Malvidin acetyl hexoside	C₂₅H₂₈O₁₃		537	331; 299; 261	243; 211; 154; 111		[30]
77	Procyanidin A-type dimer	C₃₀H₂₄O₁₂		577	425; 397; 373; 287; 245; 181	245; 218; 189; 123		[13, 29, 51, 61]
78	Isovitexin 6''-<i>O</i>-deoxyhexoside	C₂₇H₃₀O₁₄		579	415; 297; 177	397; 344	362	[44]
79	Vitexin 2''-<i>O</i>-glucoside	C₂₇H₃₀O₁₅		595	415; 353; 283; 265	176		[44]
80	Kaempferol-3,7-Di-<i>O</i>-glucoside	C₂₇H₃₀O₁₆		611	449; 287	229; 165	213; 111	[39, 47]
81	Cyanidin 3,5-<i>O</i>-dihexoside	C₂₇H₃₁O₁₆		611	287; 449	287		[23, 49]
82	Cyanidin 3,5-<i>O</i>-diglucoside	C₂₇H₃₁O₁₆		611	287; 449	287; 269; 231; 199; 161	231; 213; 189; 175; 147	[37, 65]
83	Apigenin 6-<i>C</i>-[6''-acetyl-2''-<i>O</i>-deoxyhexoside]-glucoside	C₂₉H₃₂O₁₅		621	561; 547; 461	533;461; 433	433	[44]

84	Peonidin-3,5-diglucoside	C₂₈H₃₃O₁₆		625	301; 463	286	258	[37, 65]
85	Malvidin 3-(6-O-coumaroyl)glucoside	C₃₂H₃₁O₁₄		639	331	315; 299; 270; 242; 179; 150	287; 270; 213; 179	[29, 37, 65]
86	Petunidin 3-O-glucoside-5-O-glucoside	C₂₈H₃₃O₁₇		641	317; 479; 420; 257	302	274; 228	[37, 65]
87	Malvidin 3-(6'-p-caffeoyl)glucoside)	C₃₂H₃₁O₁₅		655	493; 331	315; 179	313; 179	[28, 29]
88	Malvidin 3,5-diglucoside	C₃₂H₃₁O₁₅		655	331; 493	299; 179	281	[37, 65]
89	Cyclopassifloic acid glucoside	C₃₇H₆₂O₁₂		699	537; 347; 271	375; 259; 185		[44]

Research carried out using tandem mass spectrometry showed the presence of 89 biologically active compounds corresponding to the *V. amurensis* species. Salvianolic acids F, D and G, oleanolic, ursolic, myristoleic acids, berbericin, mearnsin, esculin, nevadensin, stigmasterol, fucosterol, Phlorizin, L-Tryptophan are identified for the first time in *V. amurensis*.

The identification of compounds (*m/z* values and fragmented ions) was carried out by comparing the obtained experimental data with known scientific results or mass spectrometric libraries. Anthocyanins have been identified in the extracts: Malvidin-3-*O*-glucoside, Pelargonidin-3-*O*-glucoside (callistephin), Peonidin-3-*O*-glucoside, Cyanidin-3,5-dihexoside, Cyanidin-3,5-diglucoside, Peonidin-3,5-diglucoside, Malvidin 3-(6-*O*-coumaroyl) glucoside, Petunidin-3-*O*-glucoside-5-*O*-glucoside, Malvidin 3-(6'-*p*-caffeoyl glucoside), Malvidin 3,5-diglucoside. Obtained mass spectrometry data correlate with scientific sources [13, 23, 28, 29, 37, 49, 51, 59, 61, 65]. A large group of flavonoids identified; flavonols Kaempferol, Aromadendrin, Kaempferide, Quercetin, Dihydroquercetin, Kaempferol-3-*O*-galactoside, Quercetin 3-*O*-galactoside, Taxifolin-3-*O*-glucoside, Quercetin-3-*O*-glucuronide, Isorhamnetin-3-*O*-rhamnoside, Isorhamnetin-3-*O*-glucoside, Myricetin-3-*O*-galactoside, Kaempferol-3,7-Di-*O*-glucoside [13, 24, 25, 29, 41, 42, 45, 51, 54, 56, 57, 60, 62, 64, 65, 68, 70, 74]; flavones: Apigenin, Luteolin, Nevadensin, Apigenin-7-*O*-glucoside, Isovitexin 6"-*O*-deoxyhexoside, Vitexin 2"-*O*-glucoside, Apigenin 6-C-[6"-acetyl-2"-*O*-deoxyhexoside]-glucoside [13, 15, 17, 34, 43, 44, 45, 58, 60, 67, 69]; flavanones: Naringenin, Hesperitin, Eriodictyol-7-*O*-glucoside [16, 29, 42, 53, 60, 62, 63]; Flavan-3-ols: Catechin, Epicatechin [13, 25, 29, 46, 53, 57, 60].

Glycosylated coumarins have also been identified: Umbelliferone, Esculin, Fraxin, Fraxetin-7-*O*-beta-glucuronide [29, 30, 34, 36, 71, 73], berberine Palmatine [18, 71], stilbenes Polydatin and trans-Resveratrol, [30, 46, 60], sterols: Fucosterol, Stigmasterol [14, 20, 30], dihydrochalcone Phlorizin [26, 29, 30, 46, 53, 57].

It should be noted that compounds such as coumarins Umbelliferone, Fraxin and Esculin, flavone Nevadensin, flavan-3-ol Epicatechin, sterol Fucosterol, flavanol Taxifolin-3-*O*-glucoside were identified by mass spectrometry only in island samples of wild grapes. *V. amurensis* (Pakhtusov Islands and Rikord Island, Peter the Great Bay, Sea of Japan).

4 Conclusions

Amur grape *V. amurensis* Ruprecht contains a large number of polyphenolic complexes, which are biologically active compounds. In this work, we have tried for the first time to conduct a comparative metabolic study of biologically active substances of wild grapes obtained from five different places in the Primorsky and Khabarovsk territories. HPLC in combination with a BRUKER DALTONIKS ion trap (tandem mass spectrometry) was used to identify target analytes in extracts. The results showed the presence of 89 biologically active compounds corresponding to the species *V. amurensis*, and Salvianolic acids F, D and G, Oleanolic, Ursolic, Myristoleic acids, Berbericinin, Mearnsetin, Esculin, Nevadensin, Stigmasterol, Fucosterol, Phlorizin, L-Tryptophan were identified for the first time in *V. amurensis*.

The findings may support future research into the production of various pharmaceutical and dietary supplements containing *V. amurensis* extracts. A wide variety of biologically active polyphenolic compounds opens up rich opportunities for the creation of new drugs and biologically active additives based on extracts from this family of grapes (*Vitaceae*).

References

1. Ampelography USSR, 494 (1946)
2. N.I. Vavilov, *Origin and geography of cultivated plants*, 440 (1987)
3. E.W. Berry, *Lower Cretaceous. Baltimore, Maryland Geological Survey* (1911)
4. E.W. Berry, *Maryland Geological Survey* (1917)
5. A. N. Krishtofovich, Paleontological history of grapes, **23(5-6)**, 365 (1938)
6. Krishtofovich A. N. Sarmatian flora of the Krynka river, **98**, 5 (1931)
7. A. N. Krishtofovich, M. I. Borsuk, Miocene plants from the Irtysh River near the city Tara in Western Siberia, **5**, 375 (1939)
8. G. Hegi Vitaceae, Rebengewachse. Illustrierte Flora von Mittel-Europa. Munchen, **5(1)**, 350 (1925)
9. I.V. Michurin, The results of half a century of work on the development of new varieties of fruit plants, **I-II** (1929)
10. I.V. Michurin, Bulletin of gardening, fruit growing and horticulture, **4-5** (1907)
11. V.G. Sprygin, N.F. Kushnerova, S.E. Fomenko, T.N. Gordeychuk, E.E. Solodova, Respiratory Physiology and Pathology Bulletin, **11**, 1 (2002)
12. State Pharmacopeia XIV (2018) <https://pharmacopoeia.ru>
13. G. Abeywickrama, S.C. Debnath, P. Ambigaipalan, F. Shahidi, *Phenolics of selected cranberry genotypes and their antioxidant efficacy* (2016)
14. D. Bakir, M. Akdeniz, A. Ertas, M.A. Yilmaz, I. Yener, M. Firat, U. Kolak, Food Biochem (2020)
15. L. Barros, M. Duenas, A.M. Carvalho, Food and Chem. Toxicol, **50(5)**, 1576 (2012)
16. A. Bodalska, A. Kowalczyk, M. Wlodarczyk, I. Feska, Analysis of Polyphenolic Composition of a Herbal Medicinal Product—Peppermint, **25**, 69 (2020)
17. C. Carazzone, D. Mascherpa, G. Gazzani, A. Papetti, Food Chem, **138**, 1062 (2013)
18. D.S.A. Cassiano, I.M.A. Reis, I. de Oliveira Estrela, H.F. de Freitas, S.S. da Rocha Pita, J.M. David, A. Branco, Comp. Biol. Chem, **83**, 107129 (2019)

19. W. Chen, L. Gong, Z. Guo, W. Wang, H. Zhang, X. Liu, S. Yu, L. Xiong, J. Luo, *Molecular Plant*, **6(6)**, 1769 (2013)
20. X. Chen, P. Zhu, B. Liu, D. Ge, L. Wei, Y. Xu, *J. Pharm. Biomed. Anal*, **159**, 490 (2018)
21. X. Chen, S. Zhang, Z. Xuan, D. Ge, X. Chen, J. Zhang, Q. Wang, Y. Wu, B. Liu, *The Cells. Molecules*, **22**, 811 (2017)
22. M. Cirlini, P. Mena, M. Tassotti, K.A. Herrlinger, K. Nieman, C. Dall'Asta, D. Del Rio, *Molecules*, **21**, 1007 (2016)
23. L.P. da Silva, E. Pereira, T.C.S.P. Pires, M.J. Alves, O.R. Pereira, L. Barros, I.C.F.R. Ferreira, *Food Res. Int*, **119**, 34 (2019)
24. Daikonya, A.; Kitanaka, S. Constituents isolated from the roots of *Rhodiola sacra* S. H. Fu. Japan. *J Food Chem Safety*, **18(3)**, 183 (2011)
25. M. De Rosso, L. Tonidandel, R. Larcher, G. Nicolini, A. Dalla Vedova, F. De Marchi, M. Gardiman, M. Giust, R. Flamini, *Food Chem*, **163**, 244 (2014)
26. Z. Fan, Y. Wang, M. Yang, J. Cao, A. Khan, G. Cheng, *Food Chem*, **318**, 126512 (2020)
27. T. Farrell, L. Poquet, F. Dionisi, D. Barron, G. Williamson, *J. Pharm. Biomed. Anal*, **55**, 1245 (2011)
28. M. Garg, M. Chawla, V. Chunduri, R. Kumar, S. Sharma, A. Sharma, N.K. Kaur, N. Kumar, J.K. Munday, M.K. Saini, S.P. Singh, *J. Cereal Sci*, **71**, 138e144 (2016)
29. P. Goufo, R.K. Singh, I. Cortez, *Antioxidants*, **9**, 398 (2020)
30. A.R. Hamed, S.S. El-Hawary, R.M. Ibrahim, U.R. Abdelmohsen, A.M. El-Halawany, *Bioassay Guided Approach. J. Chrom. Sci*, **1** (2020)
31. F. Han, Y. Li, L. Ma, T. Liu, Y. Wu, R. Hu, A. Song, R. Yin, *Talanta*, **160**, 183 (2016)
32. V. Ivanova-Petropulos, Z. Naceva, V. Sandor, L. Makszin, L. Deutsch-Nagy, B. Berkics, T. Stafilov, F. Kilar, *Electrophoresis*, **39**, 1597 (2018)
33. R. Jaiswal, L. Jayasinghe, N. Kuhnert, *J. Mass Spectrom*, **47**, 502 (2012)
34. R. Jaiswal, H. Muller, A. Muller, M.G.E. Karar, N. Kuhnert, *Phytochem*, **108**, 252 (2014)
35. R.-W. Jiang, K.-M. Lau, P.-M. Hon, T.C.W. Mak, K.-S. Woo, K.-P. Fung, *Current Med. Chem*, **12**, 237 (2005)
36. S. Kim, S. Oh, H.B. Noh, S. Ji, S.H. Lee, J.M. Koo, C.W. Choi, H.P. Jhun, L. Roots. *Molecules*, **23**, 3001 (2018)
37. E.S. Lago-Vanzela, R. Da-Silva, E. Gomes, E. Garcia-Romero, E. Hermosin-Gutierrez, *Agric. Food Chem.*, **59**, 13136 (2011)
38. R. Lang, N. Dieminger, A. Beusch, Y.-M. Lee, A. Dunkel, B. Suess, T. Skurk, A. Wahl, H. Hauner, T. Hofmann, *Anal. Bioanal. Chem. Sci.*, **8(08)**, 116 (2018)
39. G. Le Gall, M.S. DuPont, F.A. Mellon, A.L. Davis, G.J. Collins, M.E. Verhoeven, I.J. Colquhoun, *J. Agricult. Food Chem*, **51**, 2438 (2003)
40. T. Levandi, T. Pussa, M. Vaher, A. Ingver, R. Koppel, M. Kaljurand, *Food Chem*, **63(1)**, 86 (2014)
41. T.H. Lee, C.C. Hsu, G. Hsiao, J.Y. Fang, W.M. Liu, C.K. Lee, *Planta Med.*, **82(8)**, 698 (2016)
42. X. Li, T. Tian, *Frontiers in Pharm*, **9**, 1067 (2018)
43. M.M. Marzouk, S.R. Hussein, A. Elkhateeb, M. El-shabrawy, E.-S. S. Abdel-Hameed, S.A. Kawashty, *J. Applied Pharm. Sci.*, **8(08)**, 116 (2018)

44. M. Ozarowski, A. Piasecka, A. Paszel-Jaworska, A. D. Siqueira de Chaves, A. Romaniuk, M. Rybczynska, A. Gryszczynska, A. Sawikowska, P. Kachlicki, P.L. Mikolajczak, A. Seremak-Mrozikiewicz, *Braz. J. Pharmacol*, **28**, 179 (2018)
45. R. Pandey, B. Kumar, *J. Liquid Chromatogr. & Related Technol*, **39**, 225 (2016)
46. L. Paudel, F.J. Wyzgovski, J.C. Scheerens, A.M. Chanon, R.N. Reese, D. Smiljanic, C.; C. Wesdemiotis, J.J. Blakeslee, K.M. Riedl, P.L. Rinaldi, *J. Agricult. Food. Chem*, **61**, 12032 (2013)
47. A. Petsalo, J. Jalonen, A. Tolonen, *Chromatogr*, **1112(1-2)**, 224 (2006)
48. S. Piccolella, G. Crescente, M.G. Volpe, M. Paolucci, S. Pacifico, *Molecules. Chem*, **24**, 3630 (2019)
49. P.C. Pradhan, S. Saha, *J Food Sci. Technol*, **53(2)**, 1205 (2016)
50. P. Quifer-Rada, A. Vallverdu-Queralt, M. Martinez-Huelamo, G. Chiva-Blanch, O. Jauregui, R. Estruch, R. Lamuela-Raventos, *Food Chem*, **169**, 336 (2015)
51. N. Rafsanjany, J. Senker, S. Brandt, U. Dobrindt, A. Hensel, *J. Agric. Food Chem*, **63**, 8804 (2015)
52. S.A.O. Santos, C.S.R. Freire, M.R.M. Domingues, A.J.D. Silvestre, C.P. Neto, *J. Agric. Food Chem*, **59**, 9386 (2011)
53. S.A.O. Santos, C. Vilela, C.S.R. Freire, C.P. Neto, A.J.D. Silvestre, *J. Chromatogr. B*, **938**, 65 (2013)
54. A.J. Seukep, Y.-L. Zhang, Y.-B. Hu, M.-Q. Guo, *Pharmaceut*, **13**, 59 (2020)
55. K. Schoedl, A. Forneck, M. Sulyok, R. Schuhmacher, *J. Agric. Food. Chem*, **59**, 10787 (2011)
56. M. Sharma, R. Sandhir, A. Singh, P. Kumar, A. Mishra, S. Jachak, S.P. Singh, J. Singh, J. Roy, *Front. Plant. Sci.*, **7**, 1870 (2016)
57. V. Spinola, J. Pinto, P.C. Castilho, *Food Chemistry*, **173**, 14 (2015)
58. M. Suarez, A. Macia, M.-P. Romero, M.-J. Motiva, *J. Chromatogr. A.*, **1214**, 90 (2008)
59. J. Sun, X. Liu, T. Yang, J. Slovin, P. Chen, *Food Chem*, **146**, 289 (2014)
60. J. Sun, F. Liang, Y. Bin, P. Li, C. Duan, *Molecules*, **12**, 679 (2007)
61. L. Sun, S. Tao, *S. Molecules*, **24**, 159 (2019)
62. A. Vallverdu-Queralt, O. Jauregui, A. Medina-Rejon, R.M. Lamuela-Raventos, *Food Chem.*, **60**, 3373 (2012)
63. M.N. Viera, P. Winterhalter, G. Jerz, *Phytochem. Anal.*, **27**, 116 (2016)
64. I.V. Voskoboinikova, N.A. Tjukavkina, S.V. Geodakyan, Y.A. Kolesnik, V.K. Kolhir, V.A. Zjuzin, S.J. Sokolov, *Phytotherapy Res.*, **7**, 208 (1993)
65. H. Wang, E.J. Race, A.J. Shrikhande, *Agric. Food Chem.*, **51**, 1839 (2003)
66. S. Wang, C.Q. Fan, P. Wang, *J. Chromatogr. B.*, **981**, 1 (2015)
67. A. Wojakowska, J. Perkowski, T. Goral, M. Stobiecki, *J. Mass. Spectrom*, **48**, 329 (2013)
68. J. Xiao, T. Wang, P. Li, R. Q. Liu, Li, K. Bi, *J. Chromatogr. B.*, **1028**, 33 (2016)
69. L. L. Xu, J. J. Xu, K. R. Zhong, Z. P. Shang, F. Wang, R. F. Wang, B. Liu, *Molecules*, **22**, 1756 (2017)
70. X. Xu, B. Yang, D. Wang, Y. Zhu, X. Miao, W. Yang, *Molecules*, **25**, 4612 (2020)
71. L. Yang, X. Meng, X. Yu, H. Kuang, *J. Pharm. Biomed. Analys*, **134**, 43 (2017)
72. S.T. Yang, X. Wu, W. Rui, J. Guo, Y.F. Feng, *Acta Chromatographica*, **27**, 711 (2015)

73. T. Yasuda, M. Fukui, T. Nakazawa, A. Hoshikawa, K. Ohsawa, J. Nat. Prod., **69**, 755 (2006)
74. W.-H. Zhang, I.-Ch. Chao, D.-J. Hu, F. Shakerian, J. AOAC Int., **102(3)** (2019)