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# Cyclic Variation of Residual ($CO_2$ + $H_2O$) and Total Pressure in Conifer Stem and Woody Root Tree Rings

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*Tree-ring chronologies of stem discs and core samples have been widely used to reconstruct the climatic conditions of tree growth. However, insufficient attention has been given to the fact that root and stem wood accumulate biogenic gases, whose distribution in annual rings can also be related to the climate-dependent features of tree growth. The study of chronologies of gas samples extracted under vacuum from the wood of tree-ring discs of the Siberian stone pine (Pinus sibirica Du Tour), Scots pine (Pinus sylvestris L.), Siberian larch (Larix sibirica Ledeb.) and Siberian spruce (Picea obovata Ledeb.) suggests that annual distributions of $CO_2$ and $H_2O$ in the rings and pressure variation in extracted samples follow a cyclic pattern. It was found that the sample pressure and the content of $CO_2$ and $H_2O$ in the annual rings in stems and roots of the Siberian stone pine and Scots pine from Tomsk (Russia) area are characterized by varied time cycles, including periods of about 4 and 11 years, the latter corresponding to the period of the solar activity cycle. The four-year cycle in the above chronologies is explained by the presence of similar cycles in temperature and precipitation chronologies, where cyclic variations of $CO_2$ in the rings can be interpreted as a response of the plant to the change in the climatic conditions. The established cyclic variation of the pressure and $CO_2$ content in tree rings in stems and roots indicates that $CO_2$ release into the atmosphere should also follow a cyclic pattern. Therefore, to estimate correctly the release of $CO_2$ by tree stems and large roots, long-term measurements are required.*

*Keywords: ($CO_2$ + $H_2O$), total pressure, stem, woody roots, cyclicity.*

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# Циклические вариации остаточного ($CO_2 + H_2O$) и полного давления в древесине колец ствола и корня хвойных деревьев

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*Древесно-кольцевые хронологии спилов и кернов деревьев широко используются для реконструкций климатических условий роста дерева. Однако остается в стороне тот факт, что древесина корней и стволов сохраняет внутри себя биогенные газы, распределение которых по годичным кольцам также может отражать климатические особенности роста дерева. Исследование хронологий газовых проб, извлекаемых под вакуумом из древесины годичных колец спилов кедра (Pinus sibirica Du Tour), сосны (Pinus sylvestris L.), лиственницы (Larix sibirica Ledeb.), ели (Picea obovata Ledeb), указывает на то, что погодичные распределения сохранившихся в кольцах $CO_2$, $H_2O$ и вариации давления извлекаемой пробы носят циклический характер. Обнаружено, что давление пробы, содержание $CO_2$ и $H_2O$ в годичных кольцах стволов и корней кедра и сосны из района Томска (Россия) испытывают вариации различных временных масштабов, в том числе вариации с периодами около 4 и 11 лет, последний соответствует периоду цикла солнечной активности. Появление цикла с периодом 4 года в найденных хронологиях объясняется существованием подобных циклов в хронологиях температур и осадков, а циклические вариации регистрируемого в кольцах $CO_2$ можно считать реакцией растения на изменение климатических условий. Из обнаруженной циклической вариации давления и $CO_2$ в кольцах спилов стволов и корней деревьев следует, что выделение в атмосферу $CO_2$ должно носить также циклический характер, поэтому для корректной оценки выделения $CO_2$ стволами и крупными корнями следует проводить длительные измерения.*

*Ключевые слова: ($CO_2 + H_2O$), полное давление, ствол, древесные корни, цикличность.*

## Introduction

It is well known that gases fill a considerable part of tree stems. Stem $CO_2$ concentrations were found to vary within < 1–26%, which is much higher than the amounts detected in the atmosphere (0.04%) (Bloemen et al., 2013). Stem $CO_2$ pressure would amount to 22–69 Torr (Levy et al., 1999), exceeding atmospheric $CO_2$ concentration by several orders of magnitude (Teskey et al., 2002; Mcguire et al., 2002).

Stem $CO_2$ occurs both in gaseous and aqueous phases (dissolved $CO_2$). It can be released to the atmosphere by diffusion, transported to tree branches and leaves by transpiration flow, or re-fixed in green stem parts (Saveyn, 2007; Teskey et al., 2008). $CO_2$ is transported by transpiration flow to the parts where its concentration is low, being further transformed from the aqueous to the gaseous phase again (Angert et al., 2012). $CO_2$ stem diffusion (stem respiration) is known to exhibit daily and seasonal dynamics (see, for example, (Etzold et al., 2013)). The main source of stem $CO_2$ is the respiration of living inner bark cells, cambium, and xylem; even though the experiments on $C^{13}$ addition to transpiration flow show that the major part of $CO_2$ arrives from the root system (Bloemen et al., 2013).

A great deal of effort was made to perform extensive long-term investigations into stem tissue respiration, i.e. stem $CO_2$ efflux, as stem respiration varies widely from one tree to the other. This variety is due to the fact that physiological processes involved and physical factors controlling the $CO_2$ efflux and/or influencing radial diffusion of stem $CO_2$ to the atmosphere are very complicated and hard to understand (Saveyn, 2007). That is why it is difficult to estimate $CO_2$ balance for a forest as a whole. It is not surprising, then, that a considerable attention is given to the study of $CO_2$ behavior in tree stems, as carbon dioxide is the main component of the forest carbon balance, and the problem of correct measurements of the stem $CO_2$ efflux is still high on the agenda (Angert et al., 2012).

There is more information published about water content than about tree stem gas concentration (Gartner et al., 2001; Gartner et al., 2004). In natural tree wood, there are only two kinds of water: free water (e.g. in cell lumens), and non-freezing bound water in cell walls. The main stem wood-water interaction occurs by means of hydroxyl groups of wood polymers (Engelund et al., 2013). The interaction appears to be complicated due to non-linearity of bulk flow, capillary condensation of water vapor, gas dissolution and diffusion, migration of bound water through cell walls, etc. (Khazaei, 2008).

Most research on the behavior of $CO_2$ and $H_2O$ in tree wood, however, leaves aside the problems of the residual $CO_2$ and $H_2O$ content in stems and interannual pressure variations in disc tree rings. Using a photoacoustic spectrometer with a tunable $CO_2$ laser it is possible both to trace $CO_2$ in discs, and to characterize $H_2O$ distribution over tree rings. The water content in the discs of living trees has been studied using different methods (e.g. x-ray computer tomography (Fromm et al., 2001)). However, we are not aware of any attempts to describe annual water distribution over rings in dry discs. Laser photoacoustic analysis has been successfully used in many applications for a long time (Meyer et al., 1990; Sigrist, 1994; Harren et al., 2000; Webber et al., 2005; Sigrist et al., 2008; Cernat et al., 2010; Lima et al., 2011; Popa, 2014; Popa et al., 2015), but we were the first to apply this approach to measuring annual $CO_2$ and $H_2O$ distribution in disc wood.

Our findings have shown that a porous wood structure is capable of annual sorption of stem gas components, including water vapor and plant-respired $CO_2$. Thus, for better understanding of the behavior of stem gases, additional chronologies are of special importance. In this paper, we present the main results of our investigations into the chronologies of ($CO_2$ + $H_2O$) vacuum-extracted from stem discs of the Siberian stone pine (*Pinus sibirica* Du Tour), Scots pine (*Pinus sylvestris* L.), Siberian larch (*Larix sibirica* Ledeb.), Siberian spruce (*Picea obovata* Ledeb.) as well as root discs of the Siberian stone pine and Scots pine. An analysis of certain features inherent in the behavior of $CO_2$ chronologies (Sapozhnikova et al., 2013; Ageev et al., 2015) has revealed, among other things: 1) a high correlation between $CO_2$ and $H_2O$ chronologies, 2) a statistically significant correlation between the 4-year pressure variations in vacuum-extracted gas samples and the $CO_2$ content, 3) a clearly pronounced cyclicity in chronologies. We put forward the hypothesis that the stem chronology cyclicity is the result of the root chronology cyclicity and studied the ($CO_2$ + $H_2O$) and the pressure distribution in tree-ring wood of large roots.

The results obtained can be used by dendrochronologists, dendroecologists and experts dealing with the carbon budget and carbon dioxide flux estimations between terrestrial ecosystems and the atmosphere.

## Materials and methods

*Tree-ring samples*

The ($CO_2$ + $H_2O$) content and the total pressure in gas samples vacuum-extracted from the disc stem rings of the Siberian stone pine (*P. sibirica*), Scots pine (*P. sylvestris*), Siberian larch (*L. sibirica*), spruce (*P. obovata*) and disc root rings of the Scots pine and Siberian stone pine that grew at 56°26'N and 85°03'E in Tomsk Oblast (West Siberia, Russia) were studied. All discs were taken from different study sites near Tomsk.

A part of the Scots pine root was separated from a pine sawn in 2013. The root was taken in 2014 from the depth of ~20 cm, its diameter and length were ~8 cm and ~22 cm, respectively; 47 wood rings (of 59) were studied. A sample of the Siberian stone pine root was taken in May 2016 from the root system of the Siberian stone pine sawn in the summer of 2015. The root diameter and length were ~11 cm and ~30 cm, respectively; 21 wood rings (of 28) were studied. Before the measurements, discs and roots were stored under laboratory conditions from 12 weeks to several years, so the wood material can be considered to be room-dried. The method employed allows even very old discs to be used, since tree-ring wood saves bound water with dissolved $CO_2$ regardless to the conditions of disc storage.

*The experimental procedure and data processing techniques*

The analysis of vacuum-extracted gas samples of root-ring wood was performed on a laser photoacoustic (PA) spectrometer with a computer-controlled tunable $CO_2$ laser (Sapozhnikova et al., 2013; Ageev et al., 2015; Ageev et al., 2017). Every root-ring wood sample was planed down with chisels, so that the root material was almost completely utilized. Weighted root-ring wood samples were placed in exposure chambers, and short-term vacuum was created. The pressure of the desorbed gas samples in each exposure chamber was measured with a manometer. The data for gas samples vacuum-extracted from tree-ring wood (desorbed gases) were stored in a file containing recorded PA signals of the gas sample absorption in four laser lines: 10 ***P*** (20, 16, 14) which coincided with the $CO_2$ absorption lines, and 10 ***R*** (20) which coincided with the absorption line of $CO_2$ and water-vapor. The measurements in the ***R*** (20) line allowed to detect the signal from the sum of the gas components ($CO_2$ + $H_2O$). Preliminary calibration of the PA detector allowed the partial pressure of studied gases to be determined.

An isotope analysis of carbon $CO_2$ desorbed from several wood root-rings was performed. $CO_2$ was desorbed from root-ring wood in a stream of gaseous nitrogen at T = 80°C and then was precipitated in the form of $BaCO_3$. Carbon dioxide was produced in the reaction of $BaCO_3$ with orthophosphoric acid and was then frozen and injected into ampoules. In addition, a standard sample was prepared, and carbon isotope composition ($\delta^{13}C$) of the resultant $CO_2$ was measured against the standard sample. The isotope composition was expressed in terms of deviations from internationally accepted standards Vienna Pee Dee Belemnite (VPDB). The composition of stable carbon isotope ($\delta^{13}C$) of wood $CO_2$ was measured to ±0.5 in the Laboratory of Isotope Methods (Tomsk, Russia), using the DELTA V Advantage mass spectrometer with a probability confidence interval of 0.95.

When dealing with the root wood, it was difficult to separate the current year's wood because of either narrow rings (pine roots) or weakly colored latewood (stone pine roots). For this reason, the error in root ring dating was estimated to be 1 year.

To estimate the periodic and temporal variations in the obtained chronologies, we used high-resolution spectral and cross-spectral analyses based on the maximum entropy and wavelet techniques and digital filtration of time series (Jones, 1978; Kay et al., 1981). Long-term trends were eliminated from the data analyzed. Fourier analysis (Fast Fourier Transform, FFT) using the ORIGIN software was also employed for testing periodic signals.

## Results

### *Analysis of the desorbed $CO_2$ carbon isotope composition ($\delta^{13}C$)*

By now the carbon isotope composition of samples desorbed from Siberian stone pine, Scots pine, Siberian larch, and spruce tree ring disc wood has been investigated. The results obtained showed that the gas samples desorbed from several stem discs of the Siberian stone pine, Scots pine, Siberian larch, spruce were enriched in light isotope $^{12}C$ up to ($\delta^{13}C$) = −25.3‰ for spruce, varying between −25‰ (1894) and −36.4‰ (1986) for the Siberian stone pine, from ($\delta^{13}C$) ≈ −25 ‰ up to ($\delta^{13}C$) ≈ −30‰ for the larch, from ($\delta^{13}C$) ≈ −25 ‰ to ($\delta^{13}C$) ≈ −34‰ for the Scots pine (Tomsk Oblast, Russia) (Fig. 1) (Ageev et al., 2016). ($\delta^{13}C$) = − 27‰ for the Siberian stone pine root, and ($\delta^{13}C$ ) = −33.5‰ for the Scots pine root.

It is evident that $CO_2$ is produced by the stem and root itself but not supplied from the atmosphere, as the carbon isotope composition of $CO_2$ in the atmosphere is on average –8.5‰ (Rubino et al., 2013).

### *Special features inherent in the behavior of tree ring ($CO_2 + H_2O$)*

The absorption by two gas components ($CO_2 + H_2O$) in ***R***(20) $CO_2$ laser line was recorded for the Siberian stone pine stem disc, as $CO_2$ and $H_2O$ absorption lines coincide (Fig. 2a). The

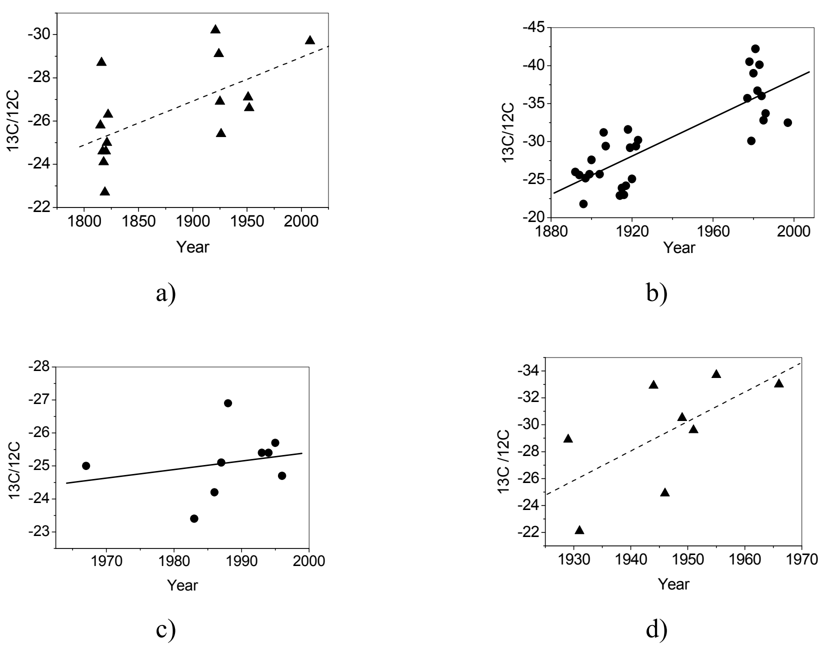



Fig. 1. Annual variations in carbon isotope composition of vacuum-desorbed $CO_2$ ($\delta^{13}C$, ‰) from the tree-ring gas samples of the 300 year old larch (a), Siberian stone pine (b), spruce (c), and Scots pine (d) (Ageev et al., 2016)

chronology of residual ($CO_2$ + $H_2O$) vacuum-desorbed from the tree rings of the Siberian stone pine is characterized by distinct short- and long-term cyclic variations (Fig. 2a). The results of Fourier analysis of the PA signals from ($CO_2$ + $H_2O$) in the Siberian stone pine stem tree rings using the ORIGIN software confirmed the existence of 2- and 4-year cycles (Fig. 2b). Fourier analysis (FFT) also revealed weak spectral maxima with a period approaching that of the 11-year solar cycles (near 10-year cycle in Fig. 2b).

Figure 3 shows the results of FFT analysis of the tree-ring ($CO_2$ + $H_2O$) chronologies for

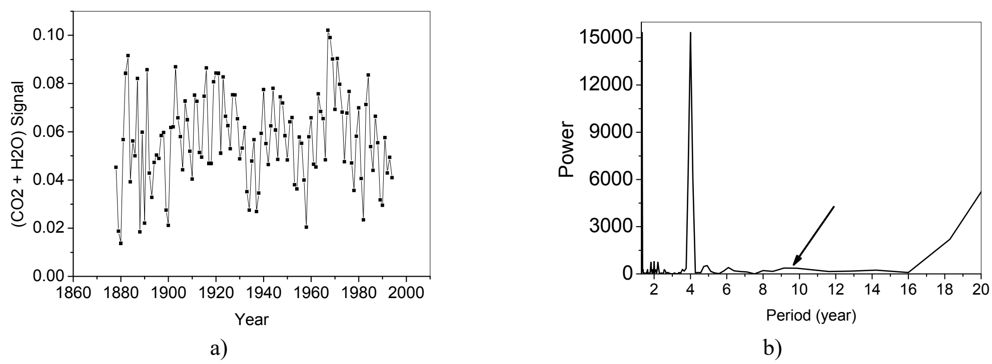



Fig. 2. Interannual variations in PA signals accounting for the behavior of ($CO_2$ + $H_2O$) vacuum-desorbed from stem tree-rings of the Siberian stone pine (N=117, 1878-1994) (a) and results of Fourier analysis of ($CO_2$ + $H_2O$) distribution in annual tree rings (b) (without trend)

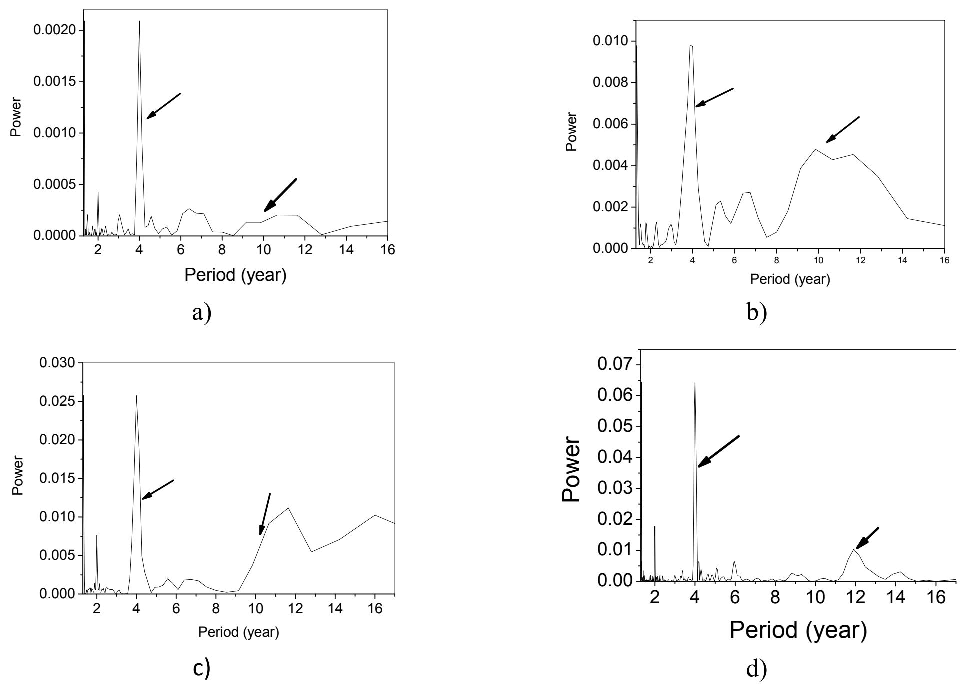



Fig. 3. Results of Fourier analysis of ($CO_2$ + $H_2O$) chronologies for spruce discs: (a) N=96 rings (1919-2004); (b) N=67 rings (1938-2004); for Scots pine discs (c): N=128 rings (1878-2005); and larch disc (d): N=285 rings (1724-2008)

spruce, Scots pine and larch discs the same way it was done for the $CO_2$ chronologies (Ageev et al., 2017).

The disc tree-ring ($CO_2$+$H_2O$) chronologies of conifers which grew in the same region (West Siberia, Russia) exhibited distinct 2-,4- year and approximately 11-year cycles (Figs. 2, 3).

*Total pressure variations in a larch stem disc*

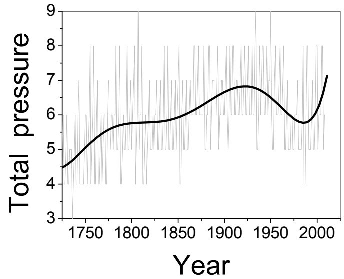
Total pressure variations in a 300-year-old larch stem disc were studied in the exposure chambers. As a result, we obtained ($CO_2$ + $H_2O$) and total pressure variation chronologies, which helped to reveal a 4-year cycle of the ($CO_2$ + $H_2O$) (Fig. 3d) variations associated with a 4-year period of the total pressure variations in gas samples. Figure 4a demonstrates the trend for the variations (polynomial approximation) in the total pressure of the gas samples in the exposure chambers of the 300 year old larch. We analyzed the results obtained for total pressure of the vacuum-extracted gas samples from tree rings to find out whether the measurements showed cyclic variations. The results of Fourier analysis of the variations in tree ring pressure are presented in Fig. 4b: in addition to the 2-year variations, well-defined 4-year cycles can be clearly seen.

*Root characteristics*

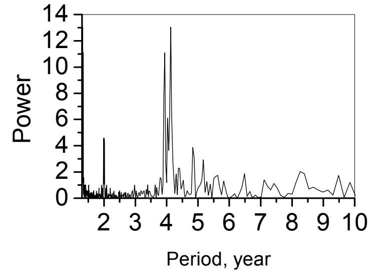
In this section, we present the results of the analysis of ($CO_2$ + $H_2O$) and total pressure variations of the vacuum-desorbed root-ring wood samples of the Scots pine and Siberian stone pine. Figure 5 shows the results of the PA signal measurements for ($CO_2$ + $H_2O$) absorption (a); variation of $CO_2$ concentration (b); total pressure variation in the gas samples (c) and pressure power spectra (d) in Scots pine root-rings. It should be noted that the concentration of the residual $CO_2$ (ppm) varied in pine woody root-rings up to 1200 ppm (atmospheric $CO_2$ now is ~400 ppm).

A linear correlation coefficient between $CO_2$ and ($CO_2$+$H_2O$) chronologies, as we might expect, was found to be quite high: R = 0.79 (N = 49, level of statistical significance $P < 0.0001$), but there was no correlation between these chronologies and the pressure chronology. An examination of $CO_2$ and pressure chronologies of pine root by spectral and cross-spectral analyses (Fig. 5d; Ageev et al., 2017) showed that they have common features – spectral maxima within ~4.5 and 11 year cycles (the latter period correlates with the 11-year solar activity cycle).

Figure 6 shows the PA signal variation for ($CO_2$+$H_2O$) absorption and pressure chronologies of the vacuum-extracted samples of the Siberian stone pine wood. The analysis of the examined chronologies showed that the concentrations of




a)




b)

Fig. 4. Total pressure variations of gas samples in the exposure chambers (a) and results of Fourier analysis of total pressure chronology for wood of larch tree rings (b)

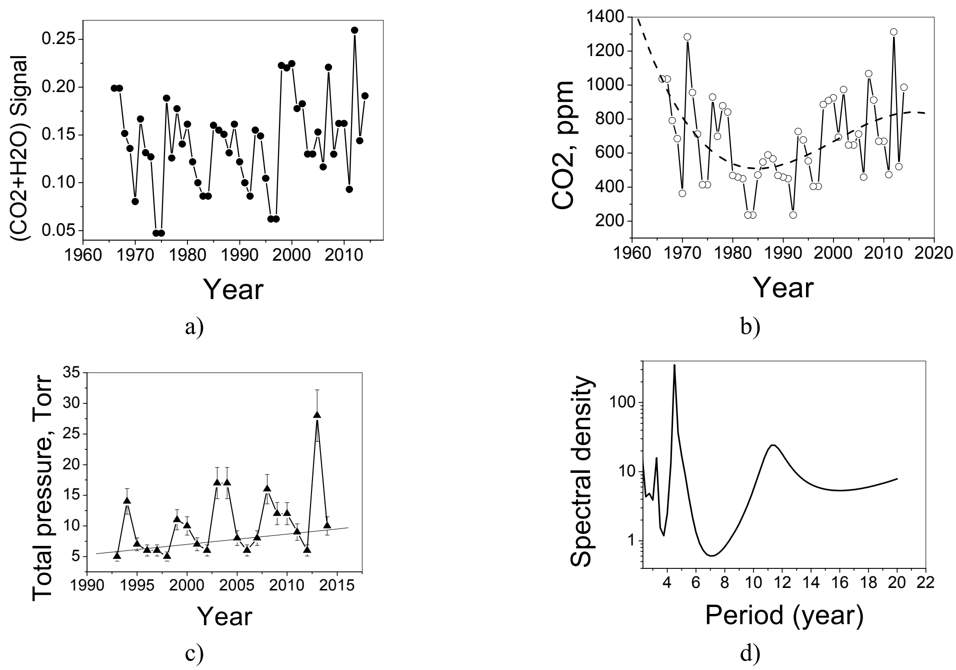



Fig. 5. The PA signal variations for ($CO_2 + H_2O$) absorption (a), the variation of the $CO_2$ concentration (b), the total pressure variation (c), and the power spectra of pressure (d) in the Scots pine root-rings

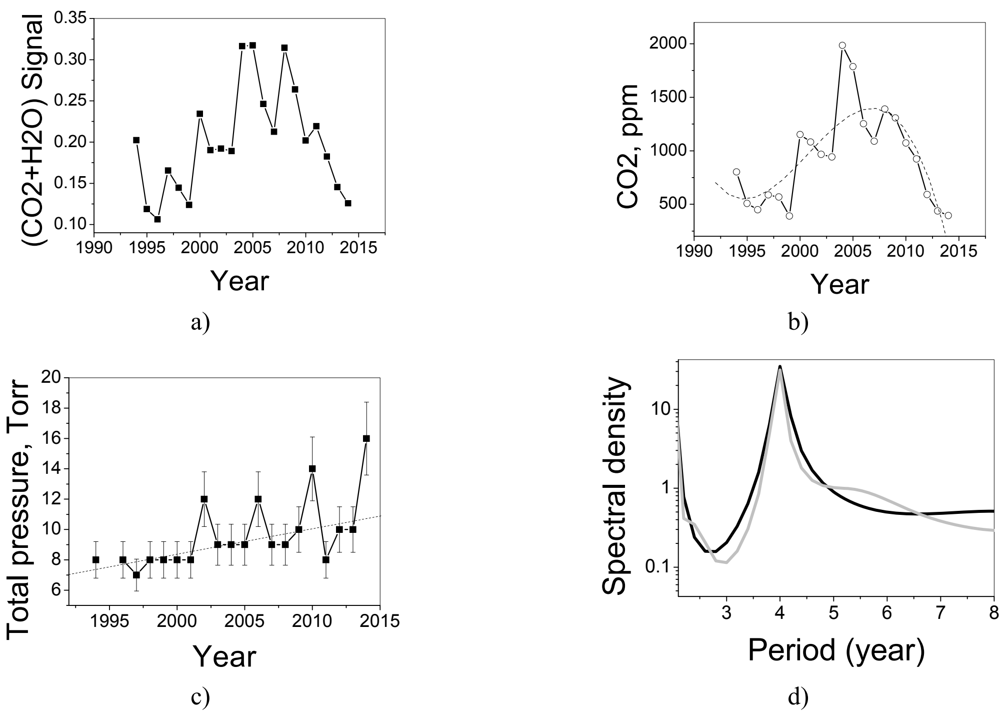



Fig. 6. The PA signal variations for ($CO_2 + H_2O$) absorption (a), the variation of the $CO_2$ concentration (b), the total pressure variation in the vacuum-extracted gas samples (c), and the power spectra of pressure (the black curve – the lower resolution, the gray curve – the higher resolution) (d) in the Siberian stone pine root-rings

the residual $CO_2$ in the wood of both roots are of the same order of magnitude (400-2000 ppm), but the pattern of annual distribution of $CO_2$ concentration is different. As in the previous case, a linear correlation between $CO_2$ and ($CO_2$ + $H_2O$) was found to be high: R = 0.95 (N = 21), P < 0.0001, but there was no correlation between $CO_2$ and pressure chronologies. The chronologies obtained were also examined by spectral and cross-spectral techniques. The spectra of the pressure chronologies for two spectral resolutions are shown in Fig. 6d: the black curve illustrates the lower resolution, and the gray curve illustrates the higher resolution. It can be seen that all parameters undergo variations with a period in the vicinity of 4 years. The cross-spectral analysis shows that the ($CO_2$+$H_2O$) variations with this period are coherent with the pressure variation and are observed approximately in antiphase with them.

*The climatic chronology cyclicity*

The experimental results showed a stable cyclicity for the established pressure chronologies of the vacuum-extracted samples and ($CO_2$ + $H_2O$) for tree-ring discs. To explain this phenomenon, we analyzed the temperature and precipitation chronologies for the region of the tree growth. Earlier we had established a relationship between $CO_2$ in the tree-ring discs and climatic parameters (Sapozhnikova et al., 2013). Figure 7a, b demonstrates the results of Fourier analysis of the temperature and precipitation chronologies (weather station data, Tomsk). The graph shows that the chronologies contain cycles with periods close to 4 years.

**Discussion**

We suggest that a porous wood structure is capable of annual accumulation (sorption) of stem gas components that include $H_2O$ vapor and plant cell-respired $CO_2$. A vacuum extraction and laser photoacoustic analysis provide long ($CO_2$ + $H_2O$) and total pressure chronologies and enable us to reveal a number of features inherent in the time series examined. We have obtained annual total pressure and ($CO_2$ + $H_2O$) distribution in the wood of stem tree rings of the Siberian stone pine (*P. sibirica*), Scots pine (*P. sylvestris*), Siberian larch (*L. sibirica*), spruce (*P. obovata*) and disc root rings of the Scots pine and Siberian stone pine. The stable cyclicity in the obtained results can be explained by the influence of the climatic conditions in which similar cyclicity is observed.

Apparently, a periodic increase in summer precipitation increases root pressure which in its turn affects stem pressure, as it was observed in

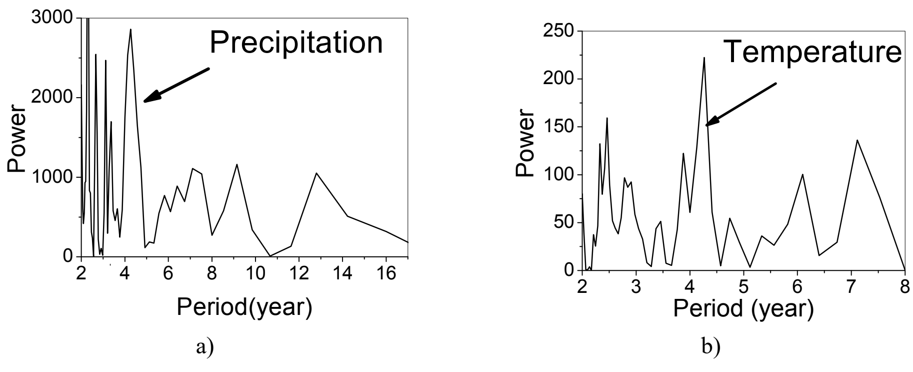



Fig. 7. The result of FFT analysis of the chronologies of summer precipitation (a) and temperatures (b) of the region of tree growth

our experiment. Plant response to this increase may result in the increase of cell respiration, i.e. an increase of $CO_2$ release. A classic example of such response was observed in the Siberian stone pine (see Fig. 1).

A cyclic pressure variation in roots and stems must lead to a cyclic $CO_2$ diffusion into the atmosphere. The data on additional periodic variations of the background $CO_2$ were acquired from the measurements of the $CO_2$ concentrations in the air samples using Fourier spectroscopy (Aref'ev et al., 2014), and it was concluded that the background $CO_2$ was formed for the most part due to equilibrium $CO_2$ exchange between the atmosphere and biosphere and exhibited variations with the outer (generally periodic) heliogeophysical conditions. Apparently, the background $CO_2$ concentrations found in (Aref'ev et al., 2014) to vary within the periods ranging between 2 and 126 months (1.7, 4, 5.2, 6.4, and 10.5 years) account for the cyclic variations of the plant-extracted $CO_2$ in the region under study.

## Conclusions

We have undertaken a study of the ($CO_2$ + $H_2O$) and total pressure variations in stem wood rings of the Siberian stone pine (*P. sibirica*), Scots pine (*P. sylvestris*), Siberian larch (*L. sibirica*), spruce (*P. obovata*) and disc root rings of the Scots pine and Siberian stone pine. The results obtained from this work allow to make the following conclusions:

1. The measured carbon isotope composition of vacuum-extracted stem and root $CO_2$ was $\delta^{13}C \sim (-25) - (-36)$‰. This means that $CO_2$ belongs to the tree itself and is not supplied from the atmosphere.

2. The ($CO_2$ + $H_2O$) and total pressure variations in the gas samples vacuum-extracted from conifer stem and root wood exhibit two- and four-year cycles.

3. Similar cycles in the precipitation and temperatures can explain the observed cyclicity in the chronologies of residual ($CO_2$ + $H_2O$) and in the pressure chronologies in tree rings of the stems and roots of some conifers.

4. The observed cycle that is close to the 11-year period can be related to the 11-year solar activity cycle.

5. The cyclic pressure variation in roots and stems should lead to the cyclic $CO_2$ diffusion into the atmosphere.

To sum up, the results obtained show that tree ring discs contain valuable information for estimating residual $CO_2$ related to respired $CO_2$ and may contribute to better understanding of the role of the gases present in the tree. We believe, ($CO_2$ + $H_2O$) measurements in trees will allow to reveal the mechanism involved in the forest adaptation to environmental and climatic changes. Practical significance of the investigation could be much higher if portable instruments for taking $CO_2$, ($CO_2$ + $H_2O$) cores had been used. An instrument for online tree-ring $CO_2$, ($CO_2$ + $H_2O$) monitoring could be a means to investigate tree adaptation to climate changes and provide useful data about tree health in ecological risk areas.

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