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Soil Respiration of Forest Ecosystems in Gradients of Environmental Pollution by Emissions from Copper Smelters

I. A. Smorkalov and E. L. Vorobeichik

Institute of Plant and Animal Ecology, Ural Branch, Russian Academy of Sciences, ul. Vos'mogo Marta 202, Yekaterinburg, 620144 Russia

e-mail: ivan.a.smorkalov@gmail.com

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Abstract—The effect of industrial pollution on soil respiration measured in situ has been studied along two gradients formed by emissions from large copper smelters: the Middle Ural Copper Smelter (spruce–fir forests) and Karabash Copper Smelter (birch forests). Pollution has proved to have a slight effect on soil respiration: its rate drops only in the zone of industrial barren. There is no correlation between soil respiration and distance to the emission source, metal contents in the litter, or litter acidity. A hypothesis is proposed that this may be due to a shift in the ratio of the root and microbial components of respiration in the pollution gradient.

Keywords: soil respiration, in situ measurement, CO_2 emission, forest litter, forest ecosystems, Middle Urals, Southern Urals, copper smelter, heavy metals, sulfur dioxide, acidity, pH.

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Soil respiration is a key component of the carbon cycle in terrestrial ecosystems. It is accounted for by metabolic activities of the soil microflora, plant root systems (including mycorrhizae), and soil fauna (Luo and Zhou, 2006; Naumov, 2009). Soil respiration is determined in situ by measuring the rate of CO_2 emission from the soil surface, which gives an integrated estimate of the intensity of production (respiration of autotrophs) and destruction processes (respiration of heterotrophs) (Hanson et al., 2000; Kuzyakov, 2006). Therefore, soil respiration measurement is widely used for assessing the production of ecosystems as well as for analyzing the activity of soil microbiocenosis (Ryan and Law, 2005). In soil microbiology, respiration is usually estimated ex situ, under laboratory conditions, in samples of soil with the roots removed (in most cases, this is part of the procedure for measuring the biomass of microorganisms by the method of substrate-induced respiration).

Long-term exposure to large doses of heavy metals in experiments usually decreases the rate of soil respiration (Doelman and Haanstra, 1984; Hattori, 1992; Laskowski, Maryanski, and Niklińska,1994; Aoyama and Nagumo, 1997; Frey et al., 2006; Akerblom et al., 2007), although the short-term effect is sometimes positive (Bardgett and Saggar, 1994). Ex situ measurements have revealed a decrease in CO_2 emission from soils affected by polymetal pollution near large metallurgical works (Ebregt and Boldewijn, 1977; Nordgren et al., 1986; Fritze et al., 1989; Vanhala and Ahtiainen, 1994; Zwoliński, 1994; Yao, Xu, and Huang, 2003), highways (Gülser and Erdoğan, 2008), and in the urban environment (Papa et al., 2010). In a number of cases, however, no changes of soil respiration were revealed in industrial pollution gradients (Paton et al., 2006; Stefanowicz, Nikliska, and Laskowski, 2008).

The majority of studies concerning the effects of pollution are based on ex situ estimations of soil respiration, which characterize only the microbial activity. Only a few publications deal with the effect of pollution on respiration in situ (Ramsey et al., 2005a, 2005b; Kozlov, Zvereva, and Zverev, 2009), reporting either a decrease in its rage or the absence of any change. The deficiency and inconsistency of available data make it necessary to accumulate a greater amount of information on the effect of pollution on CO_2 emission in nature. The purpose of this study was to analyze changes in the soil respiration rate measured in situ along two gradients of pollution from major point sources of emissions.

STUDY AREAS

The study was performed near two large plants of the nonferrous metal industry, the Middle Ural Copper Smelter (MUCS) and Karabash Copper Smelter (KCS). The MUCS, in operation since 1940, is located near the town of Revda, 50 km west of Yekaterinburg, in the southern taiga subzone of the Middle Urals. The KCS, in operation since 1910, is near the town of Karabash, 90 km northwest of Chelyabinsk, in the pre-forest–steppe pine–birch forest subzone of Southern Urals. Emissions from both smelters have similar composition. Their basic ingredients are SO_2 and dust parti-

cles with sorbed metals and metalloids (Cu, Pb, Cd, Zn, Fe, As, Hg, etc.). The smelters are also comparable in capacity: each of them annually emitting about 160×10^3 tons of pollutants in the late 1980s and about $30–40 \times 10^3$ tons in the mid-2000s. The MUCS has been in operation continuously, while operation of the KCS was suspended from 1989 to 1997.

Pollution of the territory and transformation of various groups of the biota near these smelters have been described in detail earlier (*Kompleksnaya ekologicheskaya otsenka...*, 1992; Vorobeichik, Sadykov, and Farafontov, 1994; Kaigorodova and Vorobeichik, 1996; Purvis et al., 2004; Bel'skaya and Zinov'ev, 2007; Kozlov, Zvereva, and Zverev, 2009; Trubina, 2009; Zolotarev, 2009; Bel'skii, 2010; Mukhacheva, Davydova, and Kshnyasev, 2010). In both areas, distinct background, buffer, and impact zones can be distinguished: (20–30, 4–15, and 2–5 km from the plant), which represent successive stages of technogenic digression of forest ecosystems. The area in the immediate vicinity of the KCS is a large industrial barren with a specific "moonscape" partly altered by weathering, where higher vegetation and natural soil cover are practically lacking (the upper soil layers are replaced by a technogenic substrate consisting of accumulated erosion products). The fragmentary industrial barren east of the MUCS was not included in analysis.

In the MUCS region, measurements were made in spruce–fir forests, where pollution resulted in a serial replacement of plant associations (from nemoral–wood sorrel, via herb–grass, to moss–horsetail and dead-cover associations). The soil cover was composed of mountain-forest brown, soddy podzolic, and gray forest soils. The litter was 2–3 cm deep in the background zone, 5–7 cm deep in the buffer zone, and 10–15 cm deep in the impact zone.

In the KCS region, this work was performed in derivative birch forests formed in place of pine forests, where the replacement of plant associations under the effect of pollution (from herb, via herb–grass, to dead-cover associations) also took place. The soil cover was composed of brown, soddy podzolic, and dark gray soils. The litter was 1–4 cm deep in the background and buffer zones, 8–10 cm in the impact zone, and totally absent in the barren.

MATERIAL AND METHODS

In each gradient, ten sample sites were selected. In the MUCS region, all of them were aligned in the same direction, west of the smelter; in the KCS region, in two directions (north and east of the smelter). In each of the ten sample sites, three test plots were established at a distance of 50–200 m from each other in localities with similar landscape and soil–vegetation conditions. In each test plot, soil respiration was measured in five randomly chosen points located no less that 5 m apart from each other. Measurements were made between 10 a.m. and 4 p.m. on July 5–8, 2010 along the MUCS gradient and on July 13–16, 2010 along the KCS gradient (a total of 295 measurements in 59 test plots).

The rate of the CO_2 emission from the soil surface was measured with an SR1LP field respirometer (Qubit Systems, Canada) operating by the closed dynamic chamber method (Luo and Zhou, 2006). After the measuring chamber is set on the soil surface, the air circulates within a closed system consisting of the chamber, a pump, a flow velocity detector, and an infrared gas analyzer connected to a portable computer. The CO_2 concentration in the system usually grows linearly (until saturation), which allows calculating the rate of CO_2 emission from the soil. Green parts of vascular plants at the measuring site were cut off, and the chamber (a cylinder 10 cm in diameter and 12.5 cm high) was inserted into the litter to a depth of about 2 cm for 3–4 min; flow velocity was 450–500 ml/min. The intensity of soil respiration (mg CO_2/m^2 per hour) was calculated from the slope of the CO_2 accumulation curve, taking into account the volume of the system, the chamber base area, and the temperature of the soil air. The calculations were based on the most linear segment of the curve (the correlation coefficient for which was over 0.97), starting at least 30 s after the onset of measurement. Litter temperature at a depth of 2–3 cm was measured with a soil thermometer from the respirometer tool kit in the immediate proximity of the chamber.

In each test plot (except in the industrial barren area), five pooled samples of forest litter were taken (each of them composed of five individual samples) to estimate the content of mobile forms of heavy metals (Cu, Pb, Cd) and acidity. Metals were extracted with 5% HNO_3 for 24 h (the litter-to-acid ratio was 1 : 10), and their concentration were measured with an AAS 6 Vario atomic absorption spectrometer (Analytik Jena, Germany). The pH of the litter in deionized water extract (1 : 25) was measured with an inoLab 740 ion meter (WTW, Germany).

The results were processed statistically using one-way and two-way ANOVA; multiple comparisons were made by the Tukey test. The degree of pollution impact was also estimated from the strength of effect on soil respiration (the difference between its average values in the impact and background zones, normalized to the standard deviation; census unit, one test plot). The BCA bootstrap method with 10000 iterations (the RSXL 4.0 program; www.resample.com) was used to calculate 95% confidence intervals (CI). The relationship between respiration and possible predictors was estimated using Spearman's rank correlation coefficient (R).

RESULTS

The background concentrations of metals and patterns of pollution in the two areas were similar (table).

Parameters of forest litter pollution and soil respiration at different distances from two copper smelters

Zone	Plot	Concentration, μg/g			pH_{water}	Soil respiration, mg CO_2/m^2 per hour	Litter temperature, °C
		Cu	Pb	Cd			
MUCS region							
Background	R33W	31.6 (3.3)	73.1 (4.1)	2.9 (0.2)	4.8 (0.1)	759.3 (106.5)	11.9 (0.1)
	R29W	30.3 (0.8)	71.3 (7.0)	2.5 (0.2)	5.1 (0.2)	756.5 (34.1)	14.0 (0.5)
	R20W	39.0 (12.8)	76.0 (14.6)	3.1 (0.3)	5.5 (0.2)	664.6 (19.9)	14.0 (0.7)
Buffer	R10W	231.2 (67.0)	345.2 (20.4)	7.6 (0.7)	4.8 (0.2)	737.6 (8.7)	12.1 (0.1)
	R7W	371.0 (27.0)	527.9 (62.5)	12.3 (0.9)	5.0 (0.2)	736.8 (67.3)	11.5 (0.2)
	R6W	714.8 (199.8)	819.2 (44.7)	15.4 (1.6)	4.6 (0.1)	611.2 (36.3)	11.1 (0.1)
	R4W	1020.8 (46.7)	1135.7 (203.1)	12.0 (2.4)	4.4 (0.1)	605.6 (25.9)	9.8 (0.1)
Impact	R3W	3368.3 (308.6)	2677.8 (210.5)	32.9 (1.2)	4.4 (0.2)	707.2 (46.0)	13.5 (0.3)
	R2W	5530.4 (312.8)	2780.9 (263.8)	28.1 (5.2)	4.0 (0.1)	848.2 (121.8)	13.9 (0.5)
	R1W*	3243.4 (468.8)	3069.0 (281.3)	38.7 (5.6)	4.5 (0.1)	463.4 (102.1)	16.3 (0.7)
KCS region							
Background	K32N	37.4 (0.5)	72.0 (2.6)	1.7 (0.1)	6.4 (0.1)	1083.4 (109.9)	18.9 (0.3)
	K27S	18.6 (1.0)	55.0 (1.5)	1.4 (0.1)	6.6 (0.1)	745.2 (106.2)	14.4 (0.2)
	K26S	24.6 (0.7)	75.7 (3.2)	1.6 (0.1)	6.7 (0.1)	625.0 (39.2)	13.7 (0.1)
Buffer	K18N	89.7 (5.6)	172.9 (6.7)	2.8 (0.1)	6.4 (0.1)	958.0 (117.5)	16.7 (0.2)
	K12S	104.5 (3.1)	275.4 (16.6)	4.3 (0.1)	6.6 (0.1)	673.3 (89.7)	14.0 (0.1)
	K11N	235.0 (57.9)	401.9 (54.2)	5.7 (0.4)	6.4 (0.1)	1047.2 (82.9)	15.8 (0.4)
	K9S	421.1 (60.9)	650.0 (34.3)	8.7 (0.3)	6.6 (0.1)	986.4 (168.6)	14.2 (0.2)
Impact	K5N	4383.2 (66.3)	2260.3 (213.6)	27.3 (1.2)	5.8 (0.1)	737.9 (40.4)	15.1 (0.1)
	K4S	3994.6 (333.4)	1908.5 (252.8)	23.2 (1.8)	5.7 (0.1)	594.1 (16.7)	15.8 (0.3)
	K1S	–	–	–	–	96.2 (27.8)	20.0 (0.7)

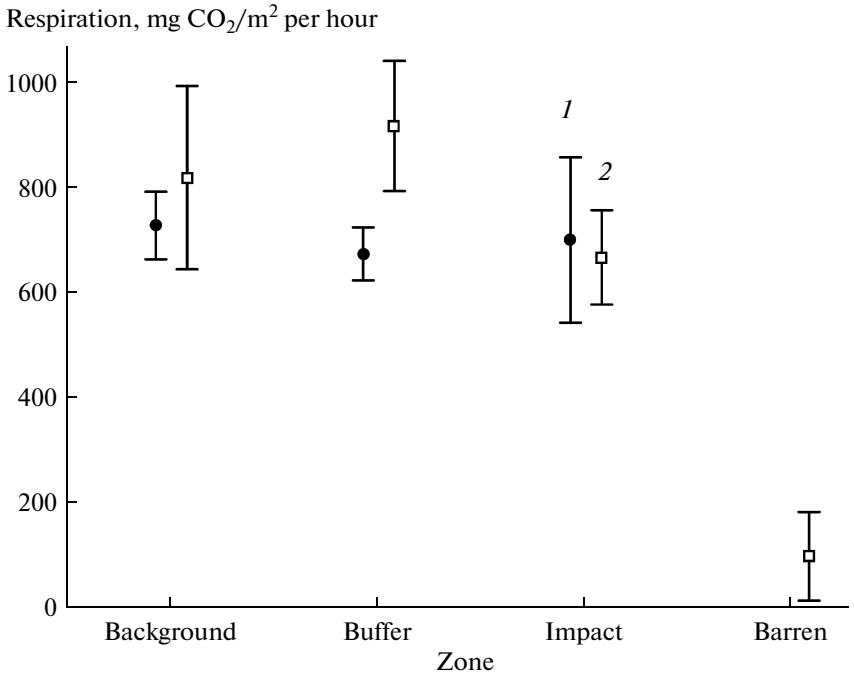
Note: Mean values (standard deviations) are shown ($n = 3$, or $*n = 2$). Figures in the codes of plots indicate the rounded-off distance (km) from the emission source, and the letter at the end indicates direction. Dash indicates cases where measurements were not taken because of the absence of forest litter.

As the smelter was approached, the concentration of Cu in the forest litter increased by a factor of more than 100; of Pb, by a factor of 30–40; and of Cd, by a factor of 10–20. The acidity of the litter in the background zone of MUCS was higher than in the KCS zone, the difference reaching 1.5 pH units; under pollution impact, acidity in both areas increased (pH decreased) by almost 1 unit. The average soil temperature in the MUCS area was approximately 3°C lower than in the KCS area; differences between test plots within each gradient reached 6°C, but there was no significant difference between the background and impact zones (results not shown).

According to the results of one-way ANOVA, differences in respiration between the plots were significant in both areas (MUCS: $F_{9;19} = 4.2$, $p = 0.004$; KCS: $F_{9;20} = 20.2$, $p \ll 0.001$). However, no connection (at the plot level) was revealed either with distance from the emission source (MUCS: $R = 0.43$, $p > 0.2$; KCS: $R = 0.55$, $p = 0.098$; $n = 10$), or with metal concentrations (MUCS: $R = -0.26$ to -0.42, $p > 0.2$, $n = 10$; KCS: $R = -0.15$ to -0.27, $p > 0.5$, $n = 9$), pH (MUCS: $R = 0.10$, $p > 0.8$; KCS: $R = -0.07$, $p > 0.9$), or temperature (MUCS: $R = 0.05$, $p > 0.9$; KCS: $R = 0.06$, $p > 0.9$; $n = 10$). The results of calculated correlations at the level of individual samples (not shown) were similar.

When we combined the results by pollution zones (Figure, two-way ANOVA showed that there were no significant differences between the two regions ($F_{1;53} = 0.6$, $p > 0.5$). At the same time, both the effect of factor "zone" and the effect of interaction "region × zone" were significant ($F_{2;53} = 6.64$, $p = 0.003$; $F_{2;53} = 7.62$, $p = 0.001$, respectively). Multiple comparisons showed that this interaction manifested itself due to higher values of respiration in the KCS buffer zone than in the MUCS buffer zone ($p = 0.030$).

For the MUCS region, the results of one-way ANOVA indicated the absence of any significant effect of pollution zone ($F_{2;26} = 0.5$, $p > 0.6$). The opposite situation was revealed in the KCS region ($F_{2;27} = 9.3$, $p < 0.001$), where the background and buffer zones showed no difference from each other ($p > 0.6$), while the impact zone proved to differ from both the background



Soil respiration rates in the regions of (*1*) MUCS and (*2*) KCS area. Vertical line indicates 95% confidence interval (with test plot taken as a census unit). The industrial barren in the KSC area is excluded from the impact zone.

zone ($p = 0.014$) and the buffer zone ($p < 0.001$). This difference was due to the presence of industrial barren, where soil respiration was reduced by almost an order of magnitude, compared to the background zone. When this territory was excluded from analysis, the conclusion concerning the significant effect of the load zone remained valid ($F_{2;24} = 3.5$, $p = 0.047$), but the difference between the impact and background zones lost statistical significance ($p = 0.300$), although the difference between the impact and buffer zones remained significant ($p = 0.038$).

Comparison of the values of effect between two contrasting pollution zones confirmed the conclusion about different patterns of soil respiration response to pollution in the study regions. In the MUCS region, the effect value was only −0.19, and CI (−1.16 to +0.74) included zero. In the KCS region, the effect was stronger (−1.30), and CI (−1.58 to −0.86) did not pass through zero. When we excluded the industrial barren area, the value of effect decreased to −0.82, but CI (−1.48 to −0.10) did not include zero, although its upper limit was close to it.

Spatial variation in soil respiration was higher in polluted territories: in the MUCS area, variation coefficient (census unit, test plot) was increased from 11.5% (CI 8.7–15.5%) to 27.0% (15.8–46.9%); in the KCS area, from 27.8% (20.8–34.4%) to 61.6% (31.7–106.7%). However, when we excluded the industrial barren, variation in the impact zone became even smaller than in the background zone, with variation coefficient being 12.9% (8.7–14.8%).

DISCUSSION

Our estimations of soil respiration rate in the background zone (625–1100 mg CO_2/m^2 per hour) are close in absolute value to those usually recorded in temperate forests, e.g., 1000–1500 (Mashika, 2006) or 690–1800 mg CO_2/m^2 per hour (Kozlov, Zvereva, and Zverev, 2009). The estimated range of spatial variation in this parameter is also close to those observed in nature (Luo and Zhou, 2006).

Emissions from copper smelters are among the strongest types of pollution. They usually have an adverse effect on all components of terrestrial ecosystems, including the soil microbiocenosis and vegetation (Vorobeichik, Sadykov, and Farafontov, 1994; Kozlov, Zvereva, and Zverev, 2009). It would be logical to assume that this should lead to a decrease in CO_2 emission from the soil. In our case, however, pollution has little effect on soil respiration: indeed, an abrupt drop in its rate is observed only in the industrial barren, while in the rest of the polluted territory its values practically remain within the limits of variation determined by natural causes.

Absolute values of respiration rate in the barren (100 mg CO_2/m^2 per hour) are comparable to those typical for pessimal biotopes of technogenic or natural origin. Thus, CO_2 emission per hour in coal dumps was 140–230 mg/m^2 (Naumov, 2009); in dumps of polymetallic ore processing waste, 50–160 mg/m^2 (Ramsey et al., 2005); and in polar and arid deserts, 50–390 mg/m^2 (Cable et al., 2011). Taking into account the complete absence of vegetation, respiration in the bar-

ren can be attributed almost entirely to the activity of microorganisms resistant to pollution.

Differences in soil respiration between the two regions are due only to the fact that the area of industrial barren near the KCS is much greater (which, in turn, is accounted for by a more hilly topography of the region and longer exposure to pollution). When we exclude the barren from analysis, the patterns of the pollution effect on soil respiration in both regions prove to be very similar, although these regions differ in climate (different geographical subzones), type of vegetation (coniferous and deciduous forests), and soil acidity.

A comparative analysis of in situ soil respiration measurements made in 18 areas polluted by metallurgical works worldwide (five measurements per test plot, ten test plots per plant) (Kozlov, Zvereva, and Zverev, 2009) revealed a general adverse effect of industrial pollution on respiration, and this effect was especially strong in areas affected by the nonferrous metal industry, especially if the soil in these areas was acidified. The copper smelters discussed above have also been studied by those authors, among other sources of emissions. Their conclusion about the MUCS was similar to ours, while in the KCS area they revealed a strong correlation of respiration rate both with distance from the emission source and with the level of pollution. This difference in estimations may be due to bias resulting from insufficient sample size, which can play an important role when soil respiration considerably varies spatially (Luo and Zhou, 2006). Another possible factor is difference in the ratio of samples from the strongly and moderately polluted areas, which in the study by Kozlov et al. was shifted toward the former, compared to our study. The factor of interannual variation also cannot be excluded, as well as the possibility of land regeneration (the above authors made their measurements in 2003).

It is known that soil respiration depends on many abiotic factors, including soil temperature, moisture, texture, and pH (Luo and Zhou, 2006). In a number of cases the connection could be traced between the variation of in situ soil respiration and the contents of metals in the soil (Ramsey et al., 2005b) and soil acidity (Ramsey et al., 2005a). The same predictors can also satisfactorily explain the variation of respiration ex situ (Ebregt and Boldewijn, 1977; Yao, Xu, and Huang, 2003; Stefanowicz, Nikliska, and Laskowski, 2008). However, we have revealed no such connection, which confirms the conclusion that pollution has a weak effect on respiration and indicates that soil respiration is probably regulated by multiple mechanisms, in the absence of a single leading factor.

Soil respiration is an integrated functional characteristic of the intensity of production and destruction processes in terrestrial ecosystems. Thus, the constancy of this parameter in the gradient of pollution indicates either lack of effect on both groups of processes or a shift in the ratio between the contributions of heterotrophs and autotrophs. The task of distinguishing between these contributions is extremely difficult methodologically (Hanson et al., 2000; Kuzyakov, 2006). Their ratio varies under different natural conditions within a very wide range (Hanson et al., 2000; Ryan and Law, 2005; Kuzyakov, 2006), although it is often *a priori* taken to be 1 : 1. We are unaware of attempts to distinguish between the contributions of root and microbial respiration in polluted environments, but indirect evidence suggests that it is this approach that can help us shed some light on factors accounting for the stability of the discussed parameter.

There is ample data that the activity of soil microorganisms is inhibited by high concentrations of metals and increased acidity (Ebregt and Boldewijn, 1977; Doelman and Haanstra, 1984; Nordgren et al., 1986; Fritze et al., 1989; Hattori, 1992; Laskowski, Maryanski, and Niklínska, 1994; Aoyama and Nagumo, 1997; Yao, Xu, and Huang, 2003; Frey et al., 2006; Akerblom et al., 2007). Metal concentrations recorded by us in the impact zones of both smelters are comparable to the concentrations at which mortality of many groups of nonsymbiotic microorganisms is observed (Ebregt and Boldewijn, 1977; Nordgren et al., 1986; Fritze et al., 1989). The inhibition of microbial activity near the studied sources of emissions can be diagnosed from the increased thickness of forest litter and the reduced rate of cellulose destruction (Vorobeichik, Sadykov, and Farafontov, 1994; Kaigorodova and Vorobeichik, 1996; Vorobeichik, 2007). On the other hand, root respiration (including the rhizomicrobial component) appears to be more resistant to pollution. Unfortunately, we have no direct measurements of underground phytomass in the studied areas; there is only evidence that pollution leads to the redistribution of tree roots to lower soil horizons (Veselkin, 2002). However, this is exactly how the indirect data can be interpreted. Thus, the production of the herb–dwarf-shrub and moss layers may remain unchanged in polluted areas due to the compensatory replacement of sensitive by tolerant species (Vorobeichik, Sadykov, and Farafontov, 1994). Furthermore, no strong decrease in the production of trees has been recorded in the MUCS region (Usol'tsev et al., 2010). All this gives us reasons to suggest that the ratio of the contributions of root and microbial respiration under pollution impact should shift toward the former.

Analysis of soil respiration changes in the gradient of pollution is interesting in the context of discussions on the character of relationships between structural and functional parameters of ecosystems. Our results show that soil respiration is a fairly conservative functional characteristic largely invariant to structural changes in ecosystems. The stability of soil respiration in cases where the composition of the biota is obviously undergoing major changes can be regarded as evidence for the hypothesis of the functional redundancy of biotic communities (Ramsey et al., 2005b).

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