

## THE $\text{SO}_4^{2-}$ CONCENTRATION AND SULPHUR CONTENT IN LYSIMETRIC WATER AND THROUGHFALL IN BEECH FOREST STANDS IN THE ŠTIAVNICKÉ VRCHY MTS

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### Abstract

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This paper examines results of the sulphate sulphur content and its concentration in soil water from the beech forests situated in the Štiavnické vrchy Mts in Slovakia. The  $\text{S}-\text{SO}_4^{2-}$  content in bulk precipitation, throughfall and soil water for 1989–2007 was studied in these beech stands and open plots. The highest  $\text{SO}_4^{2-}$  values were found in the forest stand plot at 0.25 m depth (42.03  $\text{mg}\cdot\text{l}^{-1}$  in 1988), and the average sulphate sulphur content in soil water increased with soil depth; from 20.2  $\text{kg}\cdot\text{ha}^{-1}\cdot\text{yr}^{-1}$  at the surface to 28.5  $\text{kg}\cdot\text{ha}^{-1}\cdot\text{yr}^{-1}$  at 0.25m depth. The mean  $\text{S}-\text{SO}_4^{2-}$  concentration was 20.7  $\text{mg}\cdot\text{l}^{-1}$  in the surface humus and 30.84  $\text{mg}\cdot\text{l}^{-1}$  at 0.25m. There was an input of 538.5  $\text{kg}\ \text{S}-\text{SO}_4^{2-}\cdot\text{ha}^{-1}$  to the soil during the study period. Regression analysis revealed a statistically significant influence of sulphur content in the atmospheric deposition on the sulphur content in the soil water. A significant correlation between the precipitation amount and the content of sulphur in precipitation and in soil water was observed. The Student's t-test for dependent variables confirmed statistically significant differences between the sulphur content in soil water at 0.10 m and 0.25 m depths in these study areas. Finally, no significant differences were detected at the same plot in different study years.

*Key words:* sulphate, lysimetric water, throughfall, bulk precipitation, Štiavnické vrchy Mts.

### Introduction

The burning of fossil fuels with resultant sulphur content, high consumer demand and energy intensity have caused increasing quantities of pollutants released into the atmosphere.

Oxides of sulphur and nitrogen and particulate matter have been identified as the most toxic pollutants effecting changes in soil sorption complex and soil stability. However, nitrogen, sulphur and their compounds also perform important functions, where sulphur supports atmospheric nitrogen fixation (Fecenko et al., 2010), sulphur oxides dissolve in water and the sulphuric acid product extracts iron from leaf chloroplasts preventing carbon dioxide assimilation from the atmosphere. Sulphur dioxide ( $\text{SO}_2$ ) is the most common form of sulphur in the atmosphere, and it is the most studied emission indicator. According to Lindberg and Lovett (1992), sulphur in the form of  $\text{SO}_2$  represents more than 60% of to-

tal sulphur, and sulphur dioxide enters soil via atmospheric precipitation (Quilchano et al., 2002). Here, it displaces basic ions occasioning acidic conditions over long-term cumulative and dynamic processes (Hruška et al., 2001). Its mainly negative effects are expressed after a long period of time.

This alarming situation of environmental and forest soil degradation led to introduction of deposition limits in forest ecosystems (Mindáš et al., 2001), and these limits were important in the development of the national strategies for decreasing sulphur and nitrogen emissions in Europe and North America (Matzner, Meiwes, 1994). Slovakia is moderately environmentally sensitive according to the assessment of the selected sustainable development indicators. Although the critical sulphur deposition value for Slovakia is established at 10–30 kg.ha<sup>-1</sup>.yr<sup>-1</sup>, this has been exceeded in approximately 25% of forest soil.

Emissions of particle matter released to the atmosphere in 2000 were 83% (58 408 t.yr<sup>-1</sup>) lower than in the 1985–1987 period. A decrease of 77% to 134 376 t.yr<sup>-1</sup> was also observed in SO<sub>2</sub> emissions and 42% to 113 877 t.yr<sup>-1</sup> in NO<sub>x</sub> emissions (Kalúz, 2004). Sulphur dioxide emissions in the Czech Republic and in the western Europe countries of Netherlands, Germany, and Sweden have decreased since the beginning of the 1980's (Zapletal, Chroust, 2005; Fricke, Beilke, 1992; Prechtel et al., 2001).

Despite these optimistic results, according to Alewell et al. (2000) the ecological threat of acidification remains a serious problem. This emanates from two facts: (1) acidification is a long-term process and (2), sulphur's cumulative ability in bottom soil layers.

The aim of this paper is to highlight sulphate sulphur deposition trends in studied areas of the Štiavnické vrchy Mts.

The null hypothesis works on the assumption that the sulphur amounts in lysimetric water in individual soil horizons are mostly influenced by the momentary sulphur amount in atmospheric precipitation and by the precipitation volume, and that the highest sulphur amounts in soil water occur in the deeper layers of soils in areas more affected by emitted pollutants.

## Material and methods

The studied stands and open plots are located in the Štiavnické vrchy Mts in middle Slovakia (48°35' N; 18°51' E). The stands plots are formed by 110 year old beech stands (*Fagus sylvatica* L.) with 0.9 stocking. This area is situated in the southern part at 470 m a.s.l. and it is climatologically classified as a warm district. The mean annual precipitation ranges from 700 to 750 mm, with a maximum of 1005–1020 mm and a minimum of 431–450 mm. The average air temperature ranges from 8.0 to 8.5 °C and the average air temperature range during the growth period is 14.5–15.5 °C (Kellerová, 2005).

The Štiavnické vrchy Mts were strongly affected by pollutants from regional sources of aluminium production, the power industry, transport and a waste dump, with the main contaminants being oxides of fluorine, sulphur and nitrogen, heavy metals such as arsenic and cadmium, and ozone and particulate matter. The long-term increased aerial input of these pollutants to the Štiavnické vrchy Mts' ecosystems caused changes in their ecological conditions. This situation led to the negative quality of the beech stands in the study region. Sulphur dioxide and fluorine had the worst effect on these beech forests (Mihálik, Bublinec, 1995). For many years, the region of the Štiavnické vrchy Mts near the aluminium plant was regarded as the most environmentally polluted area in Slovakia, and was classified in the II.–III degree of exhalation in the 1990's. Since the end of the 1990's, the pollutant level has been decreasing rapidly due to modernization of production processes and stricter legislation, so that the fluorine concentration was reduced to the tolerable level of 1 µg. m<sup>-3</sup> (Urminská et al., 2000).

For this study, the soil solution was sampled by 1000 cm<sup>2</sup> plate plastic lysimeters. The first set was installed in the organic layer (F<sub>00</sub>), while the second and third sets were located at 0.10 m (upper mineral layer) and 0.25 m depths (lower mineral layer) at both study areas (Kukla, 2002). Samples were collected monthly from 1988, and then chemically treated and evaluated.

The samplers for bulk precipitation and throughfall consisted of 660 cm<sup>2</sup> bottles with a funnel inserted in their cap. Ten sampling devices were installed at both the open field and stand sites. The samples were collected monthly following strong precipitation events. The open field and stand samples were separately pooled and representative samples were analyzed.

Sulphate ions were determined by direct titration with lead nitrate using dithizone as the indicator, and results were converted to sulphate sulphur content.

Data was processed by the Statistica 7 statistical programme, and the Student's t-test for dependent variables was used to assess the statistical significance of differences between the study areas. The Statgraphics programme two-sample test was conducted to confirm results. Relationships between precipitation amount, and the sulphate sulphur amount in precipitation and soil water were defined by simple regression analysis.

## Results

The mean annual S—SO<sub>4</sub><sup>2-</sup> deposition to the surface humus in the Štiavnické vrchy Mts was 20.2 kg.ha<sup>-1</sup>.yr<sup>-1</sup> (Table 1). The highest values of sulphate sulphur were recorded in 2001 at an annual deposition of 35.1 kg.ha<sup>-1</sup>.yr<sup>-1</sup>. The maximum sulphur content in precipitation was also registered at 45.3 kg.ha<sup>-1</sup>.yr<sup>-1</sup> in this year. This corresponded to a SO<sub>4</sub><sup>2-</sup> concentration of 21.84 mg.l<sup>-1</sup> (Fig. 1a, b). The lowest annual influx of S—SO<sub>4</sub><sup>2-</sup> to the soil of 10.8 kg.ha<sup>-1</sup>.yr<sup>-1</sup> was recorded in 1989. Variability in results here was over 30% lower than that reported by Dubová and Bublinec (2006) at similar experimental plots located in the Slovak Kremnické vrchy Mts.

Table 1. Descriptive statistics of sulphate in the Štiavnické vrchy Mts in years 1988-2007.

Lysimeter	F <sub>00</sub>	F <sub>10</sub>	F <sub>25</sub>	Open plot wet deposition	Forest throughfall
Štiavnické vrchy Mts					
Valid	19	19	19	13	13
Mean	20.2	21.6	28.3	26.4	17.6
Median	21.4	17.4	27.2	26.5	18.3
Min	10.8	9.6	11.7	12.4	7.2
Max	35.1	37.9	52.6	45.2	25.0
Range	24.3	28.2	40.8	32.8	17.8
V x %	34.1	41.3	40.9	35.2	27.7
Variance	46.9	79.8	134.9	86.6	23.9
Std. Dev.	6.9	8.9	11.6	9.3	4.8
Std. Error	1.6	2.1	2.6	2.5	1.3
Lower Quartile	16.1	14.2	18.1	20.8	14.8
Upper Quartile	22.2	31.2	37.1	30.3	19.8
Percentile 10,00000	11.1	10.8	12.1	13.6	2.9
Percentile 90,00000	33.9	34.5	47.4	38.9	23.9

Note: F<sub>00</sub>—F<sub>25</sub> — lysimeter in the forest.

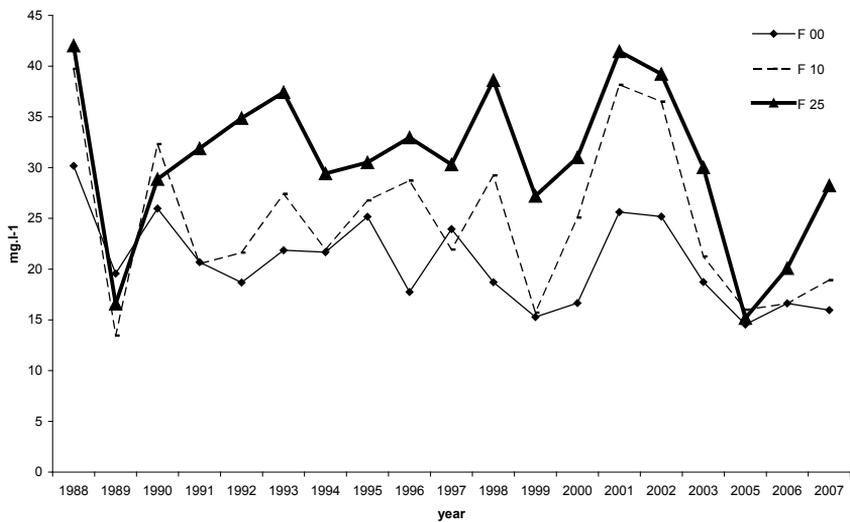


Fig. 1a. The concentrations of  $\text{SO}_4^{2-}$  on the Forest plot of Štiavnické vrchy Mts in years 1988—2007.

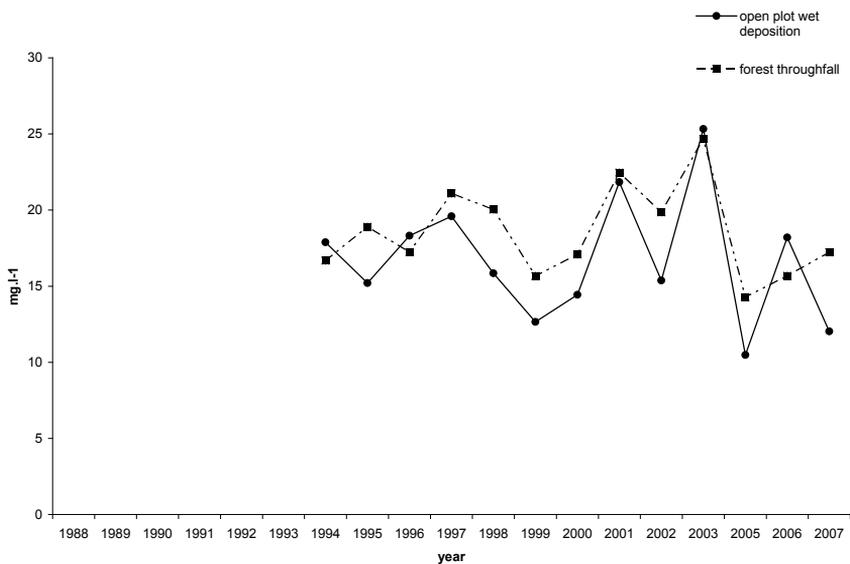


Fig. 1b. The concentrations of  $\text{SO}_4^{2-}$  on the forest throughfall and open plot wet deposition of Štiavnické vrchy Mts in years 1988—2007.

More than 6.75% sulphur was recorded at 0.10 m soil depth in the stand than in the surface layer (mean 21.6 kg.ha<sup>-1</sup>.yr<sup>-1</sup>) during the 1988–1999 period. This was due to the accumulation ability of sulphur in the bottom soil horizons.

Variability in results was approximately 42.30%, and the highest sulphur content was at 0.25 m soil depth. The mean content in this soil horizon was 28.5 kg.ha<sup>-1</sup>.yr<sup>-1</sup>, which is approximately 29.20% higher than that in the F<sub>00</sub> soil layer. The highest values of S—SO<sub>4</sub><sup>2-</sup> were observed at the beginning of research in 1988, with average values of 52.6 kg.ha<sup>-1</sup>.yr<sup>-1</sup>(H<sub>25</sub>) for S—SO<sub>4</sub><sup>2-</sup> content and 42.0 mg.l<sup>-1</sup> for SO<sub>4</sub><sup>2-</sup> concentration (Table 2). The lowest values of sulphate sulphur content of 11.8 kg.ha<sup>-1</sup>.yr<sup>-1</sup> at 0.25 m soil depth were registered in 2005. Although the highest contents of sulphate sulphur were observed in 2001 and 2002, its content at each soil depth declined overall during the research period. The increased 2001–2002 levels are attributed to the active lignite incineration plant in the Slovak Nováky district.

Although the sulphur content reported by Dubová and Bublinc (2006) declined with soil depth at experimental plots in the Slovak Kremnické vrchy Mts, the opposite trend was observed at our study plots in the Štiavnické vrchy Mts. The highest fluxes in sulphur in the soil measured during this research occurred in the autumn months. The values of through-fall deposition and open field deposition decreased during the study period, except for 2001

T a b l e 2. Amount of sulphate ( kg.ha<sup>-1</sup>) in the Štiavnické vrchy Mts in years 1988-2007.

Lysimeter	F <sub>00</sub>	F <sub>10</sub>	F <sub>25</sub>	Open plot wet deposition	Forest throughfall
Štiavnické vrchy Mts					
1988	22.0	29.5	52.6		
1989	10.8	13.5	20.1		
1990	22.2	31.2	33.3		
1991	11.1	17.5	27.3		
1992	11.1	17.1	33.1		
1993	11.7	14.1	15.6		
1994	21.9	32.2	47.4	12.4	7.2
1995	21.4	24.4	33.8	25.0	23.9
1996	26.8	34.5	37.2	32.3	19.8
1997	21.8	15.0	22.0	28.4	19.5
1998	17.3	26.6	29.4	23.6	15.8
1999	21.8	15.9	26.1	26.6	18.4
2000	16.1	16.8	18.2	20.8	12.9
2001	35.1	38.0	37.6	45.3	25.1
2002	33.8	32.3	41.1	27.8	19.5
2003	18.8	17.5	22.7	39.0	22.3
2005	20.4	14.2	11.8	18.1	16.2
2006	22.8	10.8	12.2	30.4	14.9
2007	16.5	9.7	17.2	13.6	13.7
Mean	20.2	21.6	28.5	26.4	17.6
Total	383.1	410.8	538.5	343.2	229.2

Note: F<sub>00</sub>—F<sub>25</sub> — lysimeter in the forest.

when the highest  $\text{SO}_4^{2-}$  contents and concentrations were recorded in both depositions. The  $\text{S}-\text{SO}_4^{2-}$  influx to the humus layer measured  $45.3 \text{ kg}\cdot\text{ha}^{-1}\cdot\text{yr}^{-1}$  in the open field plots.

The  $\text{SO}_4^{2-}$  concentration in soil water increased with depth from  $20.7 \text{ mg}\cdot\text{l}^{-1}$  in the surface humus to  $30.84 \text{ mg}\cdot\text{l}^{-1}$  at  $0.25 \text{ m}$ . This same phenomenon was observed at the plots in the Slovak Kremnické vrchy Mts (Dubová, Bublinec, 2006). The mean  $\text{SO}_4^{2-}$  concentration at the open field was lower at  $16.71 \text{ mg}\cdot\text{l}^{-1}$  than in beech throughfall which recorded  $18.53 \text{ mg}\cdot\text{l}^{-1}$ ), and this is explained by a washing-off effect. The forest canopy enriched the precipitation with captured sulphur compounds. The maximum  $\text{SO}_4^{2-}$  concentrations in soil water was recorded in 1988 at each soil depth, ranging from  $30.18 \text{ mg}\cdot\text{l}^{-1}$  in the surface humus to  $42.03 \text{ mg}\cdot\text{l}^{-1}$  at  $0.25 \text{ m}$  depth, while the lowest were registered in 2005 at  $14.53 \text{ mg}\cdot\text{l}^{-1}$  in the humus layer and  $15.16 \text{ mg}\cdot\text{l}^{-1}$  at  $0.25 \text{ m}$ .

Although these results did not confirm significant differences between the soil horizons, the effect of precipitation sulphur content on soil water sulphur content was quite evident. However, quite significant differences at  $p < 0.001$  were recorded in sulphur content between the  $0.10 \text{ m}$  and  $0.25 \text{ m}$  soil levels.

## Discussion

When results of soil sulphur content are compared between polluted and low pollution areas, determining factors can be identified. The most important factors are the composition of the parent material (Cosby et al., 1986) and the soil depth (Manderscheid et al., 2000). While Katutis et al. (2008) identified the importance of the humus layer composition, Pichler et al., (2006) included forest stand composition in this process. In addition, physical and chemical soil properties can be included in these important factors and Novotný et al. (2008), reported that sulphur concentration in soil water is influenced by atmospheric sulphur deposition. According to Kopeč and Gondek (2002) the influence of altitude must be added to these “natural” determinants.

Anthropogenic factors predominate in influencing the variability in our sulphur and sulphur compound research results. These factors are a major cause of deposition to all forest and aquatic ecosystem areas.

Dubová and Bublinec (2006) measured  $25 \text{ kg S}\cdot\text{ha}^{-1}\cdot\text{yr}^{-1}$  in bulk atmospheric deposition and  $24.9 \text{ kg S}\cdot\text{ha}^{-1}\cdot\text{yr}^{-1}$  in throughfall deposition at similar stands in the Kremnické vrchy Mts. They reported that the highest sulphur content in precipitation occurred there in 1994–1995, and that this was the maximum sulphur concentration in atmospheric deposition and soil water in the whole of Europe during this period. According to results of emission monitoring in Europe in this period  $> 20.0 \text{ kg S}\cdot\text{ha}^{-1}\cdot\text{yr}^{-1}$  was registered in the southeast of Great Britain and in industrial areas of Central Europe. The highest values of sulphate sulphur were measured in the  $\text{H}_0$  horizon in the low-polluted Kremnické vrchy Mts. Similar data was presented by Káňa and Kopaček (2005) in their research in forest soils of the Czech Republic. It is interesting that a higher sulphur content occurred in soil water than in throughfall 4–6 years after cutting to reduce stocking. Škvarenina, (1998) explained that this was due to high sulphur concentrations and sulphur content in the horizontal precipitations of mist and dew.

Tesař et al. (2004) agreed with this explanation when they observed  $\text{SO}_4^{2-}$  concentration in mist and cloud-water horizontal precipitation in Šumava Mts forests in the Czech Republic. These values ranged from 0.31 to 77.6  $\text{mg.l}^{-1}$ , and their beech throughfall levels were from 0.25 to 27.73  $\text{mg.l}^{-1}$ . The annual deposition of sulphur through horizontal precipitation can reach 1,436.88  $\text{kg.km}^{-2}\text{yr}^{-1}$  and it can present 79.6% of total sulphur deposition, while total sulphur deposition in spruce stands can be 49–80  $\text{kg.ha}^{-1}\text{yr}^{-1}$  (Novák et al., 2007).

Meanwhile, the soil at the research plots in the Štiavnické vrchy Mts received 23  $\text{kg S.ha}^{-1}$  via throughfall in a 180–190 years old oak stand at 680 m a. s. l. altitude (Kunca, 2007).

Novotný et al. (2008) measured the  $\text{S—SO}_4$  throughfall flux in beech stands in the Sliezské Beskydy Mts in the Czech Republic at 14.73  $\text{kg.ha}^{-1}\text{yr}^{-1}$  and the bulk deposition at 12.86  $\text{kg.ha}^{-1}\text{yr}^{-1}$ . In addition, they reported 20.52  $\text{kg S—SO}_4^{2-}.\text{ha}^{-1}\text{yr}^{-1}$  in atmospheric precipitation.

This coincided with similar highest sulphate sulfur contents in precipitation and soil water in the autumn and the winter months reported by Dubová and Bublinec in 2006. According to Pichler et al. (2006), maximum  $\text{S—SO}_4^{2-}$  values in precipitation in mixed forests occurred in winter months, while Kaiser and Guggenberger (2005) registered 53% total organic sulphur in the throughfall of 90 years old beech stands during autumn. There were no statistically significant differences recorded in the sulphur content in soil water and precipitation in different years.

According to Dubová et al. (1995), the annual deposition in spruce stands in the Slovak Poľana Mts was 65.4  $\text{kg S.ha}^{-1}\text{yr}^{-1}$ , while, Bublinec and Dubová (1995) reported an input of 44.5  $\text{kg S—SO}_4.\text{ha}^{-1}$  in spruce stand soils compared to 32.7  $\text{kg S.ha}^{-1}$  in open field soils in the Slovak Nízke Tatry Mts.

The sulphur content in spruce throughfall deposition in the Sweden–Italy transect was recorded at 6–42  $\text{kg S.ha}^{-1}$  (Novák et al., 2003). These published data correspond with the throughfall deposition in the spruce stands in southern Poland registered by Małek et al., (2005), while Vogt et al. (2001) observed 2–3  $\text{g S.m}^{-2}\text{yr}^{-1}$  in soil water in western Poland in 1992–1996, with this value corresponding to 20–30  $\text{kg S.ha}^{-1}$ .

## Conclusion

The content and concentration of sulphate ions in each soil horizon in forest stands depend on the total precipitation amount, the sulphur content in precipitation, physical and chemical soil properties, the quality of soil-forming material and the intensity of agricultural activity.

The situation in the polluted Štiavnické vrchy Mts area can be determined by the level of sulphur content and its accumulation in soil horizons. The lowest mean content of 20.2  $\text{kg.ha}^{-1}\text{yr}^{-1}$   $\text{S—SO}_4^{2-}$  was observed in the surface humus and the highest mean content of 28.5  $\text{kg.ha}^{-1}\text{yr}^{-1}$  was at 0.25 m depth. During this research, the total  $\text{S—SO}_4^{2-}$  influx to this soil depth was 538.5  $\text{kg.ha}^{-1}$ . Interesting data was observed in the throughfall deposition, where its sulphur content was lower than that recorded in the soil water. This may well have been due to the filtration effect of the forest canopy or to the accumulated sulphur soil content.

No significant difference was found in precipitation sulphur content at the beech stand and open plot experimental sites. In contrast, the differences between the sulphur content at 0.10 m and 0.25 m soil depths in these study areas were statistically significant. This is explained by the accumulation of sulphur in soil in these areas up to 1990. We proved that the precipitation amount and the sulphur content in atmospheric deposition significantly influenced the sulphur content and concentration in each soil horizon.

Temporal variability in sulphur content was highlighted by the occurrence of maximum  $S-SO_4^{2-}$  values during autumn and the lowest in summer and spring, thus defining that the  $SO_4^{2-}$ -concentrations are linked with these seasons.

Critical limits in soil water at both 0.10 and 0.25 m soil depths were substantially exceeded in 1988, 1994, 1995, 2001 and 2002 in this Štiavnické vrchy Mts study area.

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