



Correction of water interference in gas chromatographic measurements of N₂O and CO₂ of soil origin

Gy. Kampfl (1), K. Kristof (1), A. Balint (1), K. Torkos (2), K. Debreczeni (3) and Gy. Heltai (1)

(1) Szent Istvan University, Pater Karoly utca 1., 2103 Godollo, Hungary, (2) Lorand Eotvos University, Pazmany Peter setany 1/A, 1117 Budapest, Hungary, (3) Pannon University, Deak F. u. 16., 8360 Keszthely, Hungary (Kampfl.Gyorgyi@mkk.szie.hu)

For environmental protection and economic reasons it is important to study the greenhouse gas emission of agricultural origin. Since microbial transformations of carbon and nitrogen cycles of soil result in significant gas emitting processes, it is sensible to perform experiments in which the nitrous oxide (N₂O) and carbon dioxide (CO₂) production of soil origin is investigated. In literature field (Oorts et al., 2007), greenhouse (Notas et al., 2002) and microcosm experiments (Hantschel et al., 1994; Mørkved et al., 2006) are reported and the amount of the released gases is usually measured by gas chromatography using thermal conductivity detector (TCD) for the measurements of CO₂ and electron capture detector (ECD) for the analysis of N₂O. The detection of both gases might be interfered by water vapour which is also present in gas sample. Although in the present research team's micro and mesocosms experiments have been carried out since 2001 in Hungary this interfering effect was generally observed, in literature only a few references can be found dealing with this problem (Kolb, 1999). The water vapour can be removed from gas samples by the application of a cryogenic trap, a backflush or a complex valve and precolumn system (Akimoto et al., 2005) built in the gas chromatograph. However, the application of these tools does not reduce analysis time and may cause additional difficulties e.g. CO₂ might also freeze out. Therefore materials were tested which might have provided suitable solutions for these problems as well. Drierite and Nafion were applied in precolumns in a HP

5890 Series II gas chromatograph coupled to 2 or 4 metres long Porapak-Q columns. "Home made" precolumns filled with Drierite and Perma Pure produced MD-Series Nafion dryer were built in the gas chromatograph in different experiments. According to the results, although Drierite absorbed water vapour, it also adsorbed some of the CO₂ and N₂O from gas samples, so due to this problem and the difficulties of the preparation of the precolumns it was not utilized in further experiments. In the second experiment with Nafion, the water molecules permeated from the gas stream carrying the analysed gas samples to a counter-flowing purge gas stream through the Nafion membrane without significant CO₂ and N₂O loss of the samples, successfully reducing the water content of the samples to such an extent which resulted in a relatively stable baseline at ppm level in the case of ECD and the elimination of interfering water peaks in both cases of ECD and TCD. By the means of Nafion, the measurement processes carried out by manual sampling could be accelerated successfully and the gas chromatograph and the analytical columns could be protected from the disadvantages of the presence of water vapour in the gas samples as well. The improved analytical technique ensures more reliable and effective opportunities for studying the greenhouse gas emission of soil origin. In future experiments the possible limiting factors of the application of Nafion and other type of Nafion dryers are planned to be investigated.

Akimoto, F.; Matsunami, A.; Kamata, Y.; Kodama, I.; Kitagawa, K.; Arai, N.; Higuchi, T.; Itoh, A.; Haraguchi, H. (2005): Cross-correlation analysis of atmospheric trace concentrations of N₂O, CH₄ and CO₂ determined by continuous gas-chromatographic monitoring. *Energy*, 30: 299-311.

Hantschel, R.; Flessa, H.; Beese, F. (1994): An automated microcosm system for studying soil ecological processes. *Soil Science Society of America Journal*, 58: 401-404.

Kolb, B. (1999): Headspace sampling with capillary columns. *Journal of Chromatography A*, 842: 163-205.

Mørkved, P.T.; Dörsch, P.; Henriksen, T.M.; Bakken, L.R. (2006): N₂O emissions and product ratios of nitrification and denitrification as affected by freezing and thawing. *Soil Biology and Biochemistry*, 38: 3411-3420.

Notas, E.; Debreczeni, K.; Fischl, K.; Heltai, Gy. (2002): Transformation of nitrogen fertilizers in greenhouse experiments. *Agrokémia és Talajtan [Agrochemistry and Soil Sciences]*, 51: 147-156.

Oorts, K.; Merckx, R.; Gréhan, E.; Labreuche, J.; Nicolardot, B. (2007): Determinants of annual fluxes of CO₂ and N₂O in long-term no-tillage and conventional tillage

systems in northern France. *Soil and Tillage Research*, 95: 133-148.