The aim of diploma thesis has been development and optimization of method determination of N-nitrososarcosine and N-nitrosoproline in malt by gas chromatography with chemiluminiscence detector. Optimization of extraction method has been performed by response surface method. Quantification has been performed by internal standard method (N-nitrosopipecolic acid, in which matrix effects has been studied. These has been verified in münchen and pilsen malt, therefore matrix-matched calibration has been constructed. The developed method has been aplicated on wheat, münchen and pilsen malt. N-nitrosoproline was detected only in münchen malt and in other cases has been under limit of detection (LOD=4,0 μ g/kg). N-nitrososarcosine was in all cases under limit of detection (LOD=3,7 μ g/kg). The matrix-matched calibration has been constructed for experimental münchen malt with N-nitrosoproline concentration at 13,2 ± 2,9 μ g/kg.