Comments on Problems 28 and 29

We, the authors of the practical Preparatory tasks on Analytical chemistry, are very pleased that Problems 28 and 29 provoked real interest of many people involved in IChO. Though the tasks were sufficiently reproducible when we were designing these last autumn, some suggestion which we received within the last two months are really valuable. We are very grateful to everybody who shared with us their ideas how to make the procedures better. We have double checked the possible improvements and today report on the following:

Problem 28

a) HCl in Procedure A is better to be replaced with HNO3 to avoid the possible precipitation of AgCl.

b) Some people observed a difficulty when the Ag+ titration with SCN^- was done under the conditions of insufficient acidity. Normally, the end point should be observed when a slight excess of SCN^- reacts with Fe(III) ions, producing a red color of $Fe(SCN)_n$ complexes. If the H^+ concentration is low, the oxalate ions present in solution react with Fe(III) preventing it from the complexation with SCN^-. In this case the endpoint becomes invisible, as $Fe(SCN)_n$ does not form. To be on a safe side, we propose maintaining a high acid concentration of at least 0.5 M.

c) Some people observed dissolving of lead oxalate in sulfuric acid to be slow and incomplete due to the formation of poorly soluble $PbSO_4$. To facilitate the process, we propose substituting sulfuric acid by 0.5 M nitric acid. Please note that oxalate oxidation will not take place under these conditions.

Problem 29

d) There was a misprint in the posted text. The acetate buffer solution (pH 5.5–6.0) must be not lower than 1.5 M in acetate, which is needed to neutralize a significant amount of HCl added at some steps of the procedure.

e) Some people observed a poor color change in zinc - EDTA titration for the Fe(III)-Zn(II)-Cr(III) mixture. We have investigated the matter and found that the endpoint change becomes sharper if 2 mL of ethanol are added to the flask together with PAN solution.

We summarize all the improvements in the updated version of Problems 28 and 29 which are currently uploaded to the website as well as sent to the teams headmentors.

We wish you every success in preparing for the 47th IChO.

Sincerely,
Team of task authors