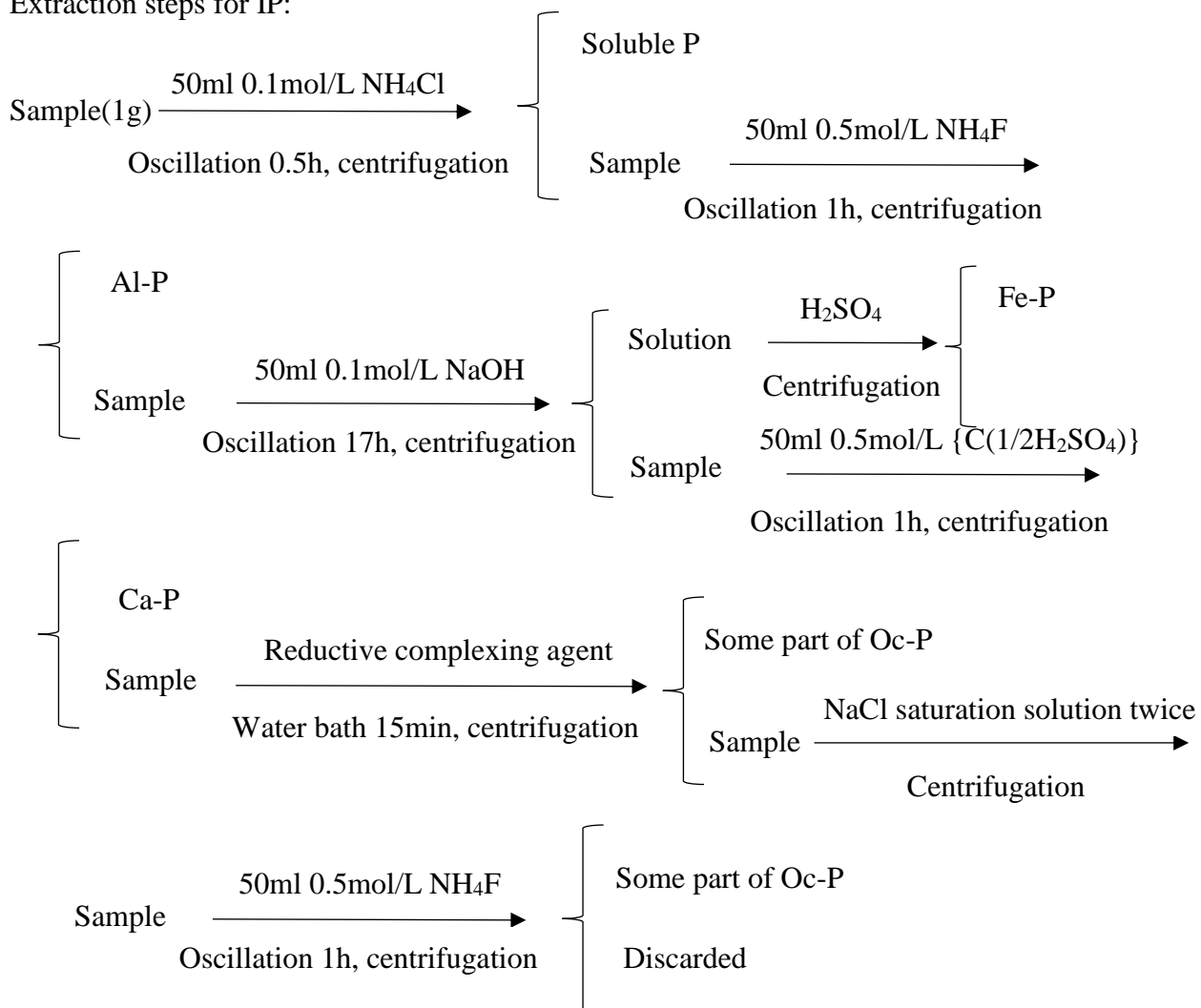


Supplementary file for testing method:

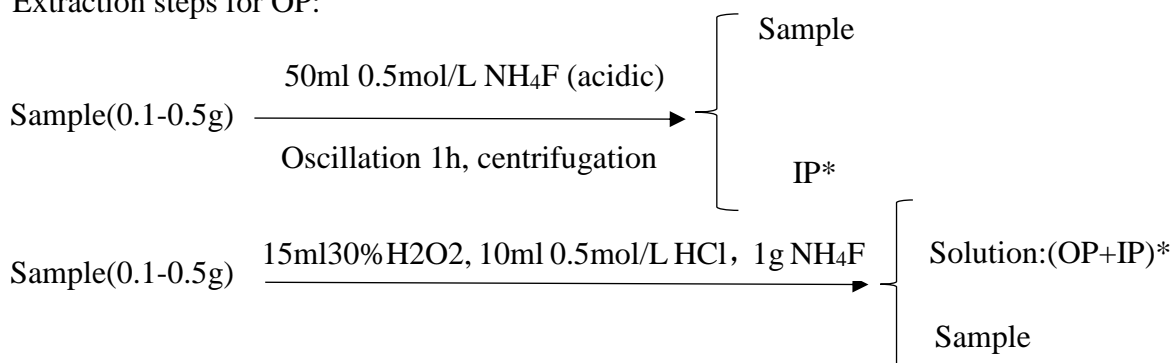
The testing method used in this research refers to the reference [5], which is “Jin Xiangcan, Tu Qingying, et al. The standard methods in Lake Eutrophication investigation (second edition). Beijing: China Environmental Science Press, 1990. (in Chinese)”. The method used in this research is described in the Page 226-229 of the reference.

1. Flow chart:

Extraction steps for IP:



Extraction steps for OP:



$$OP = (OP + IP)^* - IP^*$$

2. Reagents:

- (1) 1mol/L NH_4Cl .
- (2) 0.5mol/L NH_4F (neutral).
- (3) $\text{C}(1/2\text{H}_2\text{SO}_4) = 2\text{mol/L}$, $\text{C}(1/2\text{H}_2\text{SO}_4) = 1\text{mol/L}$, $\text{C}(1/2\text{H}_2\text{SO}_4) = 0.5\text{mol/L}$, $\text{C}(1/2\text{H}_2\text{SO}_4) = 0.1\text{mol/L}$.
- (4) Saturated sodium chloride solution.
- (5) 0.1mol/L NaOH and 2mol/L NaOH.
- (6) Dinitrophenol indicator.
- (7) 0.3mol/L Sodium citrate solution.
- (8) 1mol/L NaHCO_3 solution.
- (9) Solid $\text{Na}_2\text{S}_2\text{O}_4$.
- (10) 30% H_2O_2 .
- (11) 6mol/L HCl, 0.5mol/L HCl, 0.1mol/L HCl.
- (12) 0.8mol/L H_3BO_3 .

3. Procedures:

(1) Separation and determination of soluble phosphorus (SP)

Weigh 1g of dry sediment sample in 100ml centrifugal tube, add 50ml 1mol/L NH_4Cl solution. Cover the tube and then oscillate it on the oscillator for 0.5h. Centrifuge it and carefully pour the supernatant out as completely as possible to determine soluble phosphorus. The residual samples in the centrifugal tube were retained for separation of Al-P.

(2) Separation and determination of aluminium phosphorus (Al-P).

Add 50ml neutral 0.5 mol/L NH_4F solution was added to the sample treated with NH_4Cl , oscillate it for 1 h. Centrifuge it and supernatant was carefully poured out to determine Al-P. The residual in the centrifugal tube were retained for the separation of Fe-P.

Absorb 20ml supernatant in 50ml volumetric bottle, adjusting pH to 3 (dinitrophenol as indicator, pale yellow) by H_2SO_4 , which $\text{C}(1/2\text{H}_2\text{SO}_4)$ equals to 1 mol/L. Phosphorus in solution was determined by molybdenum antimony resistance method, which is Al-P.

(3) Separation and determination of iron-bound phosphorus (Fe-P).

Add 50ml 0.1 mol/L NaOH solution, oscillate it for 17h. After centrifugation, the supernatant is carefully poured out. The treated sample is reserved for the determination of Ca-P.

The supernatant was poured into another centrifugal tube. Two drops of $\text{C}(1/2\text{H}_2\text{SO}_4)$ sulfuric acid of 2mol/L were added first, and then another drop was added until the organic colloid coagulated. Then the supernatant was centrifuged and retained.

Take 5ml supernatant in 50ml volumetric bottle, add 35ml of water, use dinitrophenol as indicator, adjust pH to 3. Add 5ml molybdenum antimony reagent, then Fe-P was determined.

(4) Separation and determination of calcium phosphorus (Ca-P).

50ml of sulfuric acid which $C(1/2H_2SO_4)$ equals to 0.5mol/L was added to the sample after separation of Fe-P. The sample was shaken for 1 h and centrifuged. The supernatant was used to determine Ca-P, and the residual sample was used to determine the occluded phosphorus.

Take 2ml supernatant in 50ml volumetric bottle, add 35ml of water, adjust pH to 3, add 5 ml molybdenum antimony reagent, to determine Ca-P.

(5) Separation and determination of occluded phosphorus (Oc-P).

40ml 0.3mol/L sodium citrate solution and 5ml 1mol/L $NaHCO_3$ solution were added to the sample after sulfuric acid treatment. The sample was heated to 80°C in water bath, then 1 g $Na_2S_2O_4$ was added, stirred and centrifuged for 15 minutes in 80°C water bath. The supernatant was collected in a 100 ml capacity bottle. The residual samples were washed twice with saturated sodium chloride solution, 25 ml each time. The two eluents were incorporated into the 100 ml volumetric flask mentioned above, and the volume was fixed to the scale.

Determination of phosphorus in Solution: Put 5ml of the solution into 150 ml conical bottle, add 10 ml of water and 10 ml of 30% H_2O_2 , and heat it with gentle fire, so that the effect is not too strong. When bubbles stop occurring, boiling solution should not be steam-dried before oxidation is completed, otherwise it will be carbonized and affect colorimetric determination. After the oxidation is completed, the conical bottle is moved to the water bath, the solution is steamed and dried, 10 ml 2mol/L NaOH is added, boiled in the water bath for 5 minutes, and the turbid liquid is poured into the 15 ml centrifugal tube for centrifugation. The supernatant is poured into a 50 ml capacity bottle. The original 150 ml conical bottle was washed with 10ml of water, poured into the centrifugal tube, centrifuged, and the clear liquid was merged into the above 50 ml capacity bottle, repeated twice in a row, and finally volume was set.

Absorb the above constant volume solution of 20ml, adjust the pH to 3, add 5 ml molybdenum antimony reagent, constant volume to 50 ml, the determination of phosphorus, that is Oc-P.

(6) Separation and determination of organic phosphorus (OP).

0.1-0.5g of sediment were weighed and put it into 50ml calibration tube, mix with 15 ml 30% H_2O_2 , boil in boiling water bath for 0.5h, then add 15ml water and 10ml 0.5mol/L HCl, and finally add water to 50 ml. After plugging the tube, shake it for 30 minutes, add 1 g NH_4F , shake it for 1 hour, and then filter it with a dry flat ceramic funnel. Drain 10 ml of filtrate, put it into 250ml beaker, and add 15ml 0.8mol/L H_3BO_3 solution, heat it until it become dry. Add 10ml 0.1mol HCl and heat it until it become dry again. The residue was dissolved in $C(1/2H_2SO_4)$ 0.1mol/L sulfuric acid solution, adjust the pH to 3, added 30ml of water. Then add 5ml of molybdenum antimony reagent, fix volume to 50ml, colorimetric determination. The difference between the phosphorus measured here and the phosphorus measured only by acid- NH_4F is organic phosphorus.