A SCHEME FOR THE SEPARATION AND DETECTION OF AL, CR, FE, CO, NI, MN, ZN, BA, CA, SR, MG, IN THE PRESENCE OF PHOSPHORIC, ARSENIC, OXALIC, BORIC, SILICIC, HYDROFLUORIC, ACETIC, TARTRIC ACIDS AND ORGANIC MATTER.

By J. S. C. WELLS, PH.D., AND H. T. VULTE, PH.D.*

It is a well-known fact that the usual separation with ammonium hydrate, chloride and sulphide fail in the presence of any of the above-mentioned acids. The various methods in use are long, and in most cases inaccurate. In presenting the following, we have tried to cover every point of importance, and wherever possible have used quantitative methods for the sake of accuracy.

The scheme has been given a careful trial, and seems to meet all requirements.

If a solution containing the before-mentioned acids and bases is made alkaline with ammonium hydrate, the following is likely to occur:—

H ₃ PO ₄	ppts.	Al	Cr	Fe(ic)	Co	Ni	Mn	Zn	Ba	Sr	Ca	Mg
H ₃ BO ₃	"	"	"	"	"	"	"	"	"	"	"	"
H ₂ SiO ₃	"	"	"	"	"	"	"	"	"	"	"	"
H F	"	"	"	"	"	"	"	"	"	"	"	"
H ₂ C ₂ O ₄	"	chiefly										
H ₃ AsO ₄	"	Fe(ic) and Mg							"	"	"	"
H ₂ C ₄ H ₄ O ₆	}	prevent precipitation of Al, Cr, Fe, if boiling and neutral, HC ₂ H ₃ O ₂										
HC ₂ H ₃ O ₂												
Organic Matter	}	throws down Al, Cr and Fe as Basic Acetates.										

To test for the presence of these compounds three small portions of the main solution are taken as follows:—

Part I. Add HNO₃ and (NH₄)₂ Mo O₄ a yellow cryst. ppt. indicates phosphates, if heated gently a yellow colour or ppt. indicates silicates and arsenates.

Part II. Add H₂C₄H₄O₆ and NH₄O H in excess a gelatinous ppt. indicates phosphates, borates, etc.

Part III. Add conc. H₂SO₄, and evaporate to dryness; a carbonaceous residue indicates organic acids or matter.

All the acids mentioned, except phosphoric, boric and arsenic, may be removed by evaporation with HNO₃ and ignition. Borates are soluble in a considerable excess of ammonium chloride, and give no trouble.

Arsenates are removed by reduction with SO₂ and precipitation as As₂S₃ by H₂S. This should be done before adding HNO₃.

After making these tests, return to main solution.

Boil out H₂S. Add a few drops HNO₃ (Note 1). If H₂C₂O₄ or organic matter is present evaporate to dryness and ignite gently. If not, evaporate, but do not ignite. Treat residue with HCl (conc.), dilute with H₂O and boil; it dissolves wholly or leaves a white residue of SiO₂. Filter. To filtrate add NH₄Cl and NH₄OH in slight excess, heat gently and filter quickly, wash ppt. A with hot water. To filtrate add (NH₄)₂S slight xs heat, filter and wash ppt. B. Examine filtrate for the alkalies and alkaline earths. Transfer ppts. A and B to a porcelain dish and digest with a little (NH₄)₂S. (Note 2), filter and wash with H₂O and (NH₄)₂S.

* *School of Mines Quarterly*, X., 3.

Filt. (NH ₄) ₃ PO ₄ (NH ₄) ₂ S Reject	Residue Al ₂ (OH) ₆ Cr ₂ (OH) ₆ FeS, Al ₂ (PO ₄) ₂ Cr ₂ (PO ₄) ₂ Ba ₃ (PO ₄) ₂ Sr ₃ (PO ₄) ₂ Ca ₃ (PO ₄) ₂ NH ₄ Mg(PO ₄), MnS, ZnS, CoS, NiS, S. Treat with cold very dilute HCl sp. gr. 1.05, filter and wash.					
	Residue NiS + CoS + S Test a portion in Borax Bead Blue = Co Brown = Ni If blue, diss. in Cl evap. to ex- pel xs acids and Cl, dil. + H ₂ O nearly neutralise + KOH add KCY until ppt. formed is diss., boil for 4 or 5 minutes, cool, add large xs KOH + Br water a black ppt. Ni ₂ (OH) ₆ wash with very dilute NH ₄ OH to re- move last trace of Co test in Bo- rax bead. Brown Bead Ni	Filtrate Contains Fe, Al, Cr, Zn + Mn as chlorides, and Al, Cr, Ba, Sr, Ca, Mg as Phosphates. Boil out H ₂ S, dilute and make alkaline with Na ₂ CO ₃ , add Bromine water and boil until Cr is all oxidised and Br. removed. Filter.				
		Filtrate Na ₂ CrO ₄ Acidify with H ₂ A. Test for Cr with BaCl ₂ yellow ppt. BaCrO ₄ = Cr. (Note 3.)	Ppt. Al ₂ (OH) ₆ Al ₂ (PO ₄) ₂ Fe ₂ (OH) ₆ Fe ₂ (PO ₄) ₂ Ba, Sr, Ca, Mg Phos. Zn + Mn Basic Carbs. Diss. in least possible quantity of dil. HCl and add Fe ₂ Cl ₆ until a few drops of the sol. give a yellow or red ppt. with NH ₄ OH. (Note 4.) Nearly neutralise with Na ₂ CO ₃ , dilute and add xs of Na ₂ A. Boil and filter hot. Wash ppt. with boiling H ₂ O.			
			Ppt. Fe and Al as Phos- phates and Basic Ace- tates add KOH and boil.	Filtrate . Zn Mn Ba Sr Ca Mg as acetates or chlorides add NH ₄ Cl NH ₄ OH + (NH ₄) ₂ S. Filter and wash.		
			Ppt. Fe ₂ (OH) ₆ Fe ₂ (PO ₄) ₂	Filt. Add HCl to acid re- action and then make alk. with (NH ₄) ₂ CO ₃ white ppt. Al ₂ (OH) ₆ and Al ₂ (PO ₄) ₂ .	Ppt. ZnS + MnS.	Filtrate Ba, Ca, Sr, Mg acetates or chlorides. Examine in usual manner.
			Treat with H ₂ A.			
			Res ZnS Treat on charcoal with Co(NO ₃) ₂ green mass Zn.	Solution Mn ₂ A ₂ add KOH white ppt. turning brown in the air, fuse with Na ₂ CO ₃ . Green mass Na ₂ MnO ₄ indi- cates Mn		

NOTES.

I. HNO_3 is added to oxidize Fe and destroy $\text{H}_2\text{C}_2\text{O}_4$, organic acids and matter.

II. NH_4OH ppts. Fe, Al, Cr as hydrates and phosphates, and the other metals of the group as phosphates, if they are in combination with that acid, it also ppts. all of the phosphates of the alkaline earths that may be present.

Ppt. A is treated with $(\text{NH}_4)_2\text{S}$ to convert the phosphates (except those of Al, Cr, Ba, Sr, Ca and Mg) into sulphides, the H_3PO_4 is taken up by the NH_4OH in a soluble form and filtered off, thereby making a partial separation of the phosphoric acid.

III. A pink or green colouration may be caused by the oxidation of Mn to permanganate or manganate, neither interferes with the BaCl_2 test.

IV. Ferric salt is added to carry down the phosphoric acid, as $\text{Fe}_2(\text{PO}_4)_3$, white solution must be alkaline with NH_4OH , when all the H_3PO_4 is pptd. the NH_4OH brings down the yellow or brown $\text{Fe}_2(\text{OH})_6$.

V. Instead of scheme as above the following may be used :—

Filt. containing Mn, Zn, Ba, Sr, Ca, Mg as acetates or chlorides add Br and boil as long as a black ppt. is formed and colour of Br remains ; filter.

Ppt. $\text{MnO}_2 \cdot \text{H}_2\text{O}$ test in Na_2CO_3 bead.	Filt. Zn, Ba, Sr, Ca, Mg acetates or chlorides.	
	Warm, pass a rapid current of H_2S , filter.	
	ZnS white, if dark coloured test on char- coal with $\text{Co}(\text{NO}_3)_2$.	Filt. Boil out H_2S , and proceed as usual.