George M. Dawson.
Royal College Chemistry
March 30. 1870.

Filter Ash.

Incinerated 5 filts in platinum crucible.

Crucible with ash = 18·13955
Crucible = 18·12600
Weight of ash of 5 filts = ·01355

·01355 ÷ 5 = ·00271 weight of ash of 1 filt.

Per Centage of Ash.

Dried 2 filts on water bath & weighed in tube

Tube & filts = 13·2130
Tube = 12·2865
Weight of 2 filts = ·9265

·9265 ÷ 2 = ·46325 weight of 1 filt.

·46325 : 100 :: ·00271 : X

X = 0·589 = Per Cent of ash in filter.

I. Estimation of Fe in $\left\{\begin{array}{l} SO_2 \text{ Amo} \\ FeO'' \\ SO_2 \text{ Amo} \end{array}\right. + 6 Aq$

Powdered subst — pressed between filter paper — Weighed out about 1 gm of subst — Dissol in OH_2 — Added about 20 drops of NO_2Ho & then AmHo — Reddish brown ppt — Filt — Washed with Hot OH_2 till free from SO_2Ho_2 — Dried — Ignited & weighed.

Subst & Weigh tube	=	14·4290
Weigh tube back	=	13·3455
Subst emp	=	1·0835

Fe_2O_3 & Crucible	=	18·34900
Crucible alone	=	18·12805
		0·22095
Filt. ash	=	0·00271
Fe_2O_3 found	=	0·21824

$\left\{ \begin{array}{l} SO_2 \text{ Amo} \\ FeO \text{ "} \\ SO_2 \text{ Amo} \end{array} \right.$

				Theory	Experiment
S_2	=	2 X 32	= 64	—	—
Fe	=	56	= 56	14·285	14·099
Am_2	=	2 X 18	= 36	—	—
O_8	=	8 X 16	= 128	—	—
$6OH_2$	=	6 X 18	= 108	—	—
			392		

392 : 56 :: 100 : X

X = 14·285 per cent of Fe by theory.

Comp of Fe_2O_3

Fe_2O_3 160 Fe 112. O 48

160 : 112 :: 0·2184 : X

X = ·152768 = Fe in Fe_2O_3 found.

1·0835 : 100 :: 0·152768 : X

X = 14·099 per cent of Fe found.

II. Estimation of Cl in NaCl.

Ignited NaCl in crucible, weighed about 0.300. Dissolved in OH_2. Acid ~~[illegible]~~ NO_2Ho. added NO_2Ag_o. White ppt. ~~[illegible]~~ washed with Hot OH_2 by decantation. Transferred ppt to filter. Dried, — Fused - weighed

Subst & weigh tube	=	10.7155
Bottle back	=	10.2030
Subst employed	=	.5125

AgCl & Crucible	=	15.345
Crucible	=	14.087
		1.25800
ash	=	.00271
Weight of AgCl	=	1.25529

NaCl

Na	=	23
Cl	=	35.5
		58.5

AgCl

Ag	=	108
Cl	=	35.5
		143.5

Na Cl Cl

58·5 : 35·5 :: 100 : X

X = 60·68 = per cent of Cl in Na Cl by theory

AgCl Cl AgCl obt

143·5 : 35·5 :: 1·2554·29 : X

X = 3105·42 = Cl in AgCl obtained.

Subst emp Cl obt

0·5125 : 3105·42 :: 100 : X

X = 60·593 = per cent of Cl by experiment.

III Estimation of Pb in $\left\{\begin{matrix} NO_2 \\ NO_2 \end{matrix}\right. Pb''$ as $CO\ Pb''$.

Took about ·5 gr g dried substance. Dissolved in OH_2 – Added $CO\ Am\ O_2$ & a little Am Ho. – Heated – filtered – Washed with OH_2 & a little Am Ho. – Dried on filt – Transferred to crucible & ignited till changed to PbO. Incinerated filt on lid. – Treated ash with NO_2Ho & ignited – Weighed.

Bottle & subst	= 12·7360	Cru & subst	= 14·5375
Bottle back	= 12·0655	Cru	= 14·0865
Subst emp	= 0·6705		0·45100
		ash	= 0·00271
		PbO obt	= 0·44829

$\left\{\begin{matrix} NO_2 \\ NO_2 \end{matrix}\right. Pb''$

$N_2 = 28$
$O_6 = 96$
$Pb = 207$
331

Pb O
Pb = 207
O = 16
223

331 : 207 :: 100 : X X = 62·53 per Cent Pb in $\left\{\begin{matrix} NO_2 \\ NO_2 \end{matrix}\right. Pb''$

223 : 207 :: ·44829 : X X = ·416125 Amp Pb obt

·6705 : ·416125 :: 100 : X X = 62·06 per Cent Pb found

IIII.

Estimation of Pb in $\left\{\begin{matrix} NO_2 \\ NO_2 \end{matrix}\right. Pb\text{o}''$ as $CrO_2 Pb\text{o}''$.

Took about 0.5 of dried substance — Dissolved in OH_2 — Acid with $\left\{\begin{matrix} CH_3 \\ COHo \end{matrix}\right.$ — Added $Cr_2O_2KO_2$ — Yellow ppt. Filt — Washed on weighed filter — Dried on water bath & weighed till constant

$\left\{\begin{matrix} NO_2 \\ NO_2 \end{matrix}\right. Pb\text{o}''$

N_2	=	28
O_6	=	96
Pb_1	=	207
		331

$Cr\ O_2\ Pb\text{o}''$

Cr	=	52.5
O_4	=	64
Pb	=	207
		323.5

Weight bottle & subst	=	13.410
Bottle back	=	12.736
Subst emp	=	0.674

Filt & bottle	=	12.7725
Bottle	=	12.2860
Weighed filt	=	0.4865

Bottle filt & ppt	=	13.4310
Bottle	=	12.2860
		1.1450
Filt	=	0.4865
Weight $Cr\ O_2\ Pb\text{o}''$	=	0.6585

331 : 227 :: 100 : X X = 62.53 per cent Pb in $\left\{\begin{matrix} NO_2 \\ NO_2 \end{matrix}\right. Pb\text{o}''$

323.5 : 207 :: 0.6585 : X X = .42136 Pb in $CrO_2 Pb\text{o}$ obt

0.674 : 0.42136 :: 100 : X X = 62.516 per cent Pb found.

V

Estimation of Pb in $\left\{\begin{matrix} NO^2 \\ NO^2 \end{matrix}\right.$ Pbd. as PbO (2nd Ex)

Proceeded as in last experiment.

Bottle & Substance	=	12·065
Bottle back	=	11·455
Substance employed	=	0·610

Crucible & PbO	=	14·440
Crucible	=	14·058
		0·38200
Ash	=	0·00271
PbO obtained	=	0·37829

VI. Estimation of SO_2HO_2 in standard Solution.

Took 20 cc of solution, added $BaCl_2$ – heated nearly to boiling – filt – Washed with OH_2 slightly acid with HCl. till filt gave no ppt with SO_2HO_2. Dried. Transferred to Pt Crucible Ignited – Weighed as SO_2BaO''

SO_2BaO''

Ba	=	137
SO_3	=	80
O	=	16
		233

Crucible + SO_2BaO''	=	20·9572
Crucible	=	18·1265
		2·83070
Filt + ½		0·00407
SO_2BaO'' Obt. =		2·82663

233 : 80 :: 2·82663 : X

X = ·970521 Amt of SO_3 in 20 cc of Sol

9705·21 × 50 = 48·52605 Amt of SO_3 in one Litre of Sol

(By Milford's experiment 48·412)

VII

Estimation of Zn in SO_2ZnO'' 7aq as ZnO.

Dried pulverized salt between filter paper – Took about 1·gm. Dissolved in OH_2 (hot) – Added pure $CONaO_2$ Boiled for some time & filt – clear sol – Washed partly by decantation – Dried – Ignited & weighed as ZnO.

Bottle & Substance	= 14·55000
Bottle Back	= 13·52205
Subst employed	= 1·02795

ZnO & Crucible	= 13·8780
Crucible	= 13·5831
Ash ~~Substance~~ ~~A.Q.AN~~	= ·29490
	·00271
ZnO found	= 0·29219

$SO_2ZnO'' + 7aq$

Zn	=	65
S	=	32
O_4	=	64
$7OH_2$	=	126
		287

ZnO

Zn	=	65
O	=	16
		81

287 : 65 :: 100 : X

X = 22·64 per cent Zn in $SO_2ZnO'' + 7aq$

81 : 65 :: ·29219 : X

X = ·23447

1·02795 : ·23447 : 100 : X

X = 22·80 per Cent Zn found in $SO_2ZnO'' + 7aq$

VIII

Estimation of Cr in $\begin{cases} CrO_2K_2 \\ CrO_2K_2 \end{cases}$ as Cr_2O_3

Powdered substance — heated on water bath — took about 1 g — Dissolved in OH_2 — Added SO_2 till sol became green — boiled — Added Ammo in slightest possible excess, — boiled filt — washed — dried — ignited at red heat — weighed.

Bottle & subst	= 12·34200	Cr_2O_3 & Crucible	= 18·688
Bottle back	= 11·28405	Crucible	= 18·127
Subst empl	= 1·05795	Cr_2O_3	= 0·56100
		Ash	= 0·00271
		Cr_2O_3	= 0·55829

35·59 per cent Cr in $\begin{cases} CrO_2K_2 \\ CrO_2K_2 \end{cases}$ by theory

Cr_2O_3

$Cr_2 = 105$
$O_3 = 48$
153

$153 : 105 :: 55829 : X$ $\quad X = 383140$ amt Cr found

$1·05795 : 383140 :: 100 : X$ $\quad X = 35·61$ per cent Cr found

·02 too high.

Estimation of Pb in galena. (PbS.)

Took about 1 g of subst — Pulverised - dried on water-bath
Treated with HCl & some fragments of Zn till Pb reduced. -
Washed - Dissolved in NO_2Ho — add $AmHo$ & $CO(Am)_2$ -
Filt - Wash - Ignite & weigh as PbO.

Bottle & subst	= ~~14.36200~~	13.9328
Bottle back	= ~~13.28405~~	12.8020
Subst emp	= ~~1.05795~~	1.1308

Subst & Cru =
Cru =
Filter ash =

PbS.

Pb =	207
S =	32
	239

239 : 207 :: 100 : X X = 86.61 per cent Pb in PbS.

Process broke down. Though the HCl & Zn were allowed to act all night, much galena remained undecomposed. On the addition of NO_2Ho — S seperated in clots & much of the Pb came down as SO_2PbO''. Carbon from the Zn also remained, which would have, at any rate necessitated another filtration & washing.

IX

Estimation of Pb in Galena (PbS) (pure)

Took about 1g of subst — Pulverized — dried on water bath — Treated with concentrated NO_2Ho till all the galena decomposed — Added a little SO_2Ho_2 — Evap to dryness — Extracted with alcohol & filt — Washed with alcohol — Ignited — Treated incinerated filt with NO_2Ho & then SO_2Ho_2 — Weighed — Extracted SO_2Pbo'' with $\left\{\begin{matrix}C_2H_3\\ CO\end{matrix}\right\}$ amo Weighed residue & deducted from SO_2Pbo'' found.

Subst obtd	= 12·8031	Crn & SO_2Pbo''	= 14·66000
Bottle back	= 12·2881	Crn & ash	= 14·00751
PbS emp	= ·5150	SO_2Pbo'' obt	= 0·65249

239 : 207 :: 100 : X X = 86·61 per cent Pb in PbS by theory.

303 : 207 :: 65·249 : X X = 44·576 Pb obt.

·515 : 44·576 :: 100 : X X = 86·55 per centage Pb by experiment 0·07 too low.

X Estimation of Al in $\left\{\begin{matrix} SO_2 \; \cancel{Ka}\,\text{Amo} \\ SO_2 \\ SO_2 \\ SO_2 \; \cancel{Ka}\,\text{Amo} \end{matrix}\right. (Al_2O_6) + 24\,OH_2$

Pulverised the crystals & pressed them between filter paper Took about 1g – Dissolved in Hot OH_2 – Added Am Cl & Am Ho in slight excess – Heated – filt – Washed – dried – Ignited carefully – Weighed as Al_2O_3 –

Subst & tube = 14·9690
Tube back = 13·9451
Subst Emp = 1·0239

Crucible & Al_2O_3 = 18.2473
Crucible = 18·1270
0·12030
Ash = ·00271
Al_2O_3 obt = ·11759

Al_2 = 55
S 4 = 128
O 40 = 640
Am ~~X~~ 2 = ~~XXX~~ 36
H 48 = 48
907·~~X~~

Al_2O_3
Al_2 = ~~55~~ 55
O ~~X~~3 = ~~96~~ 48
~~XX~~ 103

907 : 55 : 100 : X X = 6·06 per Cent Al in Ammonia alum.

103 : 55 :: ·11759 : X X = 628013 Amt Al obt

1·0239 : 628013 :: 100 : X X = 6·13 per Cent Al by experiment, 0·07 too high.

XI.

Estimation of Ni in $\begin{cases} SO_2 Ko \\ Ni O'' \\ SO_2 Ko \end{cases} + 6 OH_2$.

Dried powdered salt between blotting paper — Took about 1 g of subst — Dissolved in OH_2 — Added pure Na Ho — Heated to boiling — Washed by decantation on filter with hot water — Dried — Ignited — Weighed as Ni O —

Ni	=	58.5
O_8	=	128
S_2	=	64
K_2	=	78.4
$6OH_2$	=	108
		436.9

Ni O

Ni	=	58.5
O	=	16
		74.5

Bottle & Subst	=	11.1943
Bottle back	=	10.1716
Subst Emp	=	1.0227

Crucible & NiO	=	18.3063
Crucible	=	18.1270
		.17930
ash	=	.00271
Ni O obt	=	.17659

436.9 : 58.8 :: 100 : X X = 13.46 per Cent Ni by theory

74.8 : 58.8 :: .17659 : X X = .138816 Ni obt

1.0227 : .138816 :: 100 : X X = 13.57 per Cent Ni obt 0.11 too high.

XII.

Estimation of Cu in $SO_2CuO'' + 5 \cdot OH_2$

Pulverised – Dried between filter-paper – Took about 19 – Dissolved in OH_2 + in a porcelain basin – Added pure NaHo drop by drop – Washed by decantation on filter – Dried – ignited – weighed as CuO.

Bottle & Subst =	14·1484
Bottle back =	13·0682
Subst Emp =	1·0802

Cu & Subst =	13·9316
Cu =	13·5839
	·34770
2 ash =	·00542
Cu O Found =	0·34228

Per centage of Cu by theory 25·45

79·5 : 63·5 :: 0·34228 : X X = 0·26961 Amt Cu by ex.

1·0802 : ·26961 :: 100 : X X = 24·959 per cent Cu by ex ·491 too little

XIII. Estimation of Pb & Sn in Capsule metal

Took about 1 g of metal. Added dilute NO_2HO — Heated gently till dissolved — Evap to dryness — Extracted with OH_2 & dilute nitric — Washed ppt — Ppt extract & washings with SO_2HO_2 & alcohol as SO_2PbO'' — Washed dried — ignited & weighed as SO_2PbO'' — Weighed tin (washed & ignited as SnO_2 —.

Employed 1·0101 gram of metal.

Crucible & SnO_2 = 13·6960	Crucible & SO_2PbO = 15·3539
Crucible = 13·5832	Crucible = 14·0073
·11280	1·34660
ash = ·00271	ash = ·00271
SnO_2 Obt = ·11009	SO_2PbO Obt = 1·34389.

150 : 118 :: ·11009 : X X = ·086604 amt Sn Obt

1·0101 : ·086604 :: 100 : X X = 8·573 per cent Sn obt

---

303 : 207 :: 1·4389 : X X = ·918103 amt Pb Obt

1·0101 : ·918103 :: 100 : X X = 90·8928 per Cent Pb obt

Pb	90·8928
Sn	8·573
	99·4658

XIV. Estimation of CO_2 in $CONaO_2$

Prepared a CO_2 apparatus introduced OH_2 & weighed - Introduced $CONaO_2$ & weighed again — Allowed the acid to act on the ~~SO~~ $CONaO_2$ ~~& weighed again~~ drew air through apparatus & weighed again.

Apparatus & $CONaO_2$	=	50.5310
Apparatus	=	49.6485
$CONaO_2$ emp	=	0.8825

Before experiment	=	50.5310
After experiment	=	50.1685
Amt CO_2	=	.3625

106 : 44 : 100 : X X = 41 per cent CO_2 in $CONaO_2$ by theory

.8825 : .3625 :: 100 : X X = 41.07 per cent CO_2 by experiment .07 too high

XV

Estimation of CO_2 in Silver Ore (present as $CO\,CaO$)

Proceeded as in last experiment.

1st determination with $SO_2\,HO_2$.

Bottle & ore = 67.3680
Bottle = 66.1654
Ore employed = 1.1926

Apparatus = = 67.368
Apparatus after experiment = 67.031
CO_2 = 0.337

1.1926 : .337 :: 100 : X X = 28.25 per cent CO_2 by experiment.

2 determination with $NO_2\,HO$.

Bottle & Subst = 11.1463
Bottle back = 9.5697
Ore employed = 1.5766

Apparatus before = 69.3645
Apparatus after ex = 68.9115
CO_2 = 0.4530

1.5766 : .4530 : 100 : X X = 28.73 per cent CO_2 found by second experiment

Difference between the two experiments 0.48 per cent.

Estimation of MnO_2 in Manganese ore.

Dried finely pulverised ore on water bath. Introduced a weighed quantity into an apparatus similar to that used in determination of CO_2 — Added about 2 1/2 times the weight of neutral potassic oxalate — ~~Heated gently~~ Allowed SO_2HO_2 to drop in — heated gently when action ceased — Drew out the remaining CO_2 & weighed — Every two combining weights of CO_2 representing one of MnO_2.

1 determination.

Bottle & MnO_2 = 15·5470
Bottle back = 14·4934
Ore emp = 1·0436

Apparatus before = 66·7545
Apparatus after = 66·1165
Amt CO_2 disengaged = 0·6380

2 of CO_2 MnO_2 CO_2

88 : 87 :: ·638 : X X = 0·6307 Amt MnO_2

1·0436 : 0·6307 :: 100 : X X = 60·43 per cent MnO_2 in Ore

2nd experiment

Amt ore emp = 1·1373 Amt CO_2 disengaged = 0·6678

per centage MnO_2 in ore by second ex. = 58·0

3rd experiment

Amount ore emp = 2.98 CO_2 disengaged = 1.7045

per centage by second experiment 56.78

4 experiment

Amount ore emp 2.98

per centage by 4th ex 59.

Four experiments by sol of Cu by Cl disengaged from MnO_2 by HCl. (NOed (p 342)

The two last conducted in an atmosphere of CO_2.

Ex 1 per centage = ~~80.70~~ 67.00

Ex 2 " " = 70.01

Ex 3 (in CO_2) per cent = 55.78

Ex 4 do " " = 56.40

difference between the two last experiments .62.

XVII. Estimation of Ag &c in a silver ore from Thunder Bay, Lake Superior. Qualatative analysis of which showed, Ag, Ca, Small quants of Fe, Mn, Zn, Pb & trace of Al, Acids SiO_2, SH_2, CO_2 & traces of HCl & SO_2HO_2?

CO_2 Est in ex XV = 28.73
Amt $CO\,CaO$" deduced from this quantity of CO_2 = 65.295

Insol gangue. Left after treatment with concent NO_2HO & afterwards with Am Ho (Almost pure SiO_2) = 22.792.

Ag As SAg_2 & Metallic Ag. Dissolved in NO_2HO & pht as Ag Cl 7.374

S Mostly combined with Ag. Determined by oxidising with concent NO_2HO — pht with $BaCl_2$ ignited & weighed as SO_2BaO" = 1.472

Totals of Ag Ore

SiO_2	=	22.792
CO CaO	=	65.295
Ag	=	7.374
S	=	1.472
		96.933
Traces of Fe, Al, Mn, Zn, Pb		3.067
		100.000

100.000
96.933
3.067

~~XVIII~~. Estimation of As &c in a pyrites containing

Took about 2 grm of powdered subst. dissolved in HCl with some $KClO_3$. Oxidised sol with SO_2 – Passed SH_2 – Filter – Treat ppt with Sodic sulphide – Filt – Dissolve in CO HoAms – Filter – Ppt As with HCl – Collect on weighed filt & weighed as As_2S_3 – Re Dissolved ppt in

Bottle & Subst	= 14·5951	Crucible & Insol res	= 12·2086
Bottle back	= 12·5248	Crucible	= 11·5278
pyrites emp	= 2·0703		·6808
		ash	= ·0027
		Insol res	= 0·6781

Subst employed Insol res

2·0703 : 0·6781 :: 100 : X X = 32·75 per cent insol res

Collected As_2S_3 on weighed filt & weighed till constant = ·7276

Took an aliquot part (·332) & determined S by oxidising with NO_2HO & ppting as $BaSO_4$ boo"

S calculated from SO_2 BaO'' = ·0789, for the aliquot part

aliquot pt	whole As_2S_3	S in aliquot		
·332 :	·7276 ::	·0789 :	X	X = ·17291

S in whole quantity of As_2S_3.

·7276 − ·17291 = ·55469

As_2S_3 − S = ·55469

Ore emp	As Abt			
2·0703 :	·55469 ::	100 :	X	X = 26·79

per centage of As in the ore (Traces of Sb & Pb? included)

Totals of As Ore

Insol res = 32·75
Arsenic = 26·79

As=Fe 32·75
{As=Fe = 38·22
70·97

75
28
107 :: 26·79 :: X

18753
26790
2866 53 / 75
225
616
600
165
150
15

38·22

XIX. Estimation of S in Iron pyrites.

By A H Pearsons process, Chemical news, April 29. 1870.

Took about 1 g of pyrites. – Dissolved & oxidised S to SO_2HO_2 by 50 cc of NO_2HO (Specif grav 39 B.) & adding potassic chlorate from time to time – Evap to small bulk – Added concd HCl & evap to dryness to render SiO_2 insol – Treated with HCl & OH_2 – Filt – Added tartaric acid about as much as pyrites employed – Heated nearly to boiling – Add excess $BaCl_2$. Wash by decant first with Hot OH_2 & then sol of $\left\{\begin{matrix}CH_3\\COOH\end{matrix}\right.$ (To dissolve basic nitrate) – Dry – Ignite – Weigh as SO_2BaO" –.

Subst & bottle	= 15·8640
Bottle back	= 14·8032
pyrites employed	= 1·0608

Crn & SO_2BaO"	= 21·855
Crn	= 18·128
	3·7270
Ash	= ·0027
SO_2BaO	= 3·7243

233 : 32 :: 3·7243 : X X = ·5149 amt S

1·0608 : ·5149 :: 100 : X X = 48·208 per cent S in the pyrites

XX. Determination of Specific gravity of Beryls.

(Three small cut stones.)

Weight of Beryls 0·18335.

Accurately counterpoised the spec grav bottle, ascertained the weight of water contained in the bottle, at 15·5 C Added the stones & weighed again at 15·5 C.

Weight of water & stones = 50·0815
Water alone = 49·9775
·1040 = weight of stones − weight of OH_2 displaced.

·18335
·10400
·07935 = weight of OH_2 displaced

$\frac{\cdot 07935}{\cdot 18335}$ = 2·31 Specific gravity found.

Specific gravity by 2nd ex = 2·6

Specif grav of beryl by Dana = 2·67 The last experiment was the best the water having been maintained more constantly at the right temperature.

XXI Estimation of Mg in SO_2 MgO" + 7 Ag.

Took about 1 g subst. Pulverized – pressed between blotting paper. Dissolved in OH_2. Added $AmCl$ & $AmHo$ — Added $POHoNaO_2$ — Agitated allowed to stand some time – Filt – Washed with OH_2 & $AmHo$ — ~~Ignit~~ Dried – Ignited & weighed as $2MgO, PO_5$ —

~~Bottle & Subst = 14.98935~~
~~Bottle back = 13.97630~~
~~Subst Emp = 1.01305~~

~~Cru & Subst = 11.8184~~
~~Cru = 1~~

Bottle & Subst = 11.7245
Bottle back = 10.4704
Subst Emp = 1.2541

Cru & Subst = 18.6920
Cru = 18.1263
Subst obt = .5657
Minus Ash = .5637

222 : 48 :: .5637 : X = .12186 Mg ~~emp~~ obt.

1.2541 : .12186 :: 100 : X X = 9.708 per cent
by experiment, by theory 9.75 .03 too low.

XXII. Estimation of N_2O_5 in NO_2KO_2

I fused a weighed portion with SiO_2 & weighed the crucible before & after fusion, the difference being the N_2O_5 disengaged –

Substance employed = ·7152.

Weight of Cru before ignition = 20·7849
" " after " = 20·4007
N_2O_5 disengaged = ·3842

N	=	14
O_3	=	48
K	=	39.
		101

101 : 40 :: 100 : x x = 53·465 per cent by theory

7152 : 3842 :: 100 : x x = 53·71 per cent by exp.

·25 too high –

XXIII Estimation of N_2O_5 in NO_2Ko by fusion with borax.

Proceeded as in last experiment but used borax instead of SiO_2.

Ex 1

Weight of NO_2Ko emp = 0.377

Loss of weight of crucible on ignition 0.202

377 : 202 :: 100 : X X = 53.58 per cent by experiment. Theory 53.46

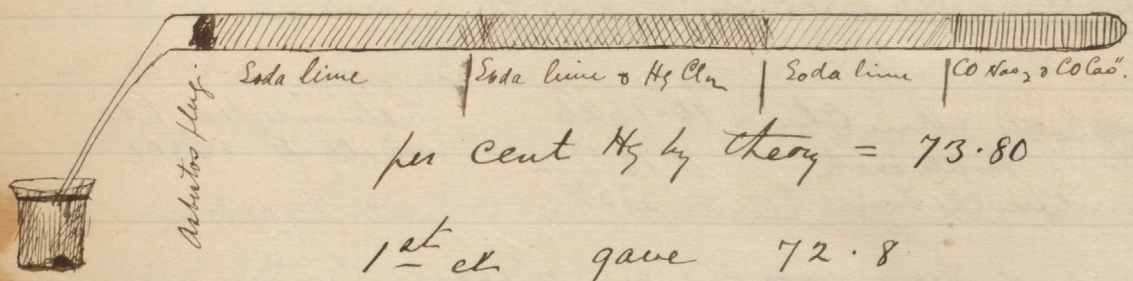
Ex 2. Weight NO_2Ko employed = .8915

Loss of weight of crucible on ignition .46635

.8915 : .46635 :: 100 : X X = 53.51 per cent by 2nd ex. 0.05 too high.

XXIV. Estimation of Hg in $HgCl_2$

Prepared a combustion tube as below, & introduced the $HgCl_2$ mixed with soda lime. Applied heat first at the front end & gradually along the whole tube. Collected the reduced Hg in water. Dried in desicator & weighed



per cent Hg by theory = 73·80

1st ex. gave 72·8

2nd ex gave 72·80

Found that the $HgCl_2$ I had been using was damp

3rd Ex.

Bottle & $HgCl_2$ = 13·5033
Bottle back = 10·4472
$HgCl_2$ Emp = 3·0561

Cu & Hg = 13·7757
Cu = 11·5295
Hg = 2·2462

3·0561 : 2·2462 :: 100 : X X = 73·49 per cent of Hg by ex. 0·31 too low.

XXV. Estimation of ~~Am~~ NH_3 in Am Cl.

Weighed out about 0.3 g of dried salt. dissolved in OH_2, added a few drops of HCl — added $PtCl_4$ in excess — Evap to dryness. — Took up with alcohol & a little ether — washed with alcohol & ether on a weighed filter — Dried at 100° & weighed. ~~Ignited filt in crucible & weighed reduced Pt~~.

Bottle & Am Cl = 10.1626
Bottle back = 9.7871
Am Cl emp = ·3755

Filter weighed till constant ·4301

Bottle filt & $PtCl_4$ 2 Am Cl weighed till constant = 13.48345
Bottle = 11.47950
2.00395
Filter = ·43010
$(PtCl_4)(2\ Am\ Cl)$ obt = 1.57385

Pt = 197
$2NH_3$ = 34
H_2 = 2
Cl_6 = 213
446

446 : 34 :: 1.57385 : X X = ·11997

Amt NH_3 in $PtCl_4$, 2 Am Cl obtained

Amt Am Cl used
0·3755 : 0·11997 :: 100 : X X = 31.94 per centage of ~~Am Cl~~ NH_3 by experiment. per centage by theory = 31.77. By ex 0·17 too high.

XXVI Estimation of Na & K in Rochelle Salt.

$\left\{\begin{matrix} CO\ Ko \\ CH\ Ho \\ CH\ Ho \\ CO\ Nao \end{matrix}\right. + 4OH_2$ (Double tartarate of K & Na.)

Took about 1 gram of substance. Dried between filter papers. — Ignited in Pt crucible untill completely decomposed. Filtered off from charcoal & washed with HCl. Evap to dryness & weighed. — Started the determination of Na & K from the weight of residual chlorides — Added $PtCl_4$ in excess. Evap to dryness on water-bath — Extracted with alcohol & ether — Washed with do — Dried on weighed filt & weighed — Na by difference

Rochelle salt emp = 1·0114.

Weight of Chlorides of Na & K which it should yield, by theory ·61685.

Aliquot part of chlorides taken, to determine Na & K = ·4729

C_4	=	48
H_4	=	4
O_6	=	96
K	=	39
Na	=	23
$4OH_2$	=	72
		282

Rochelle salt emp = 1·0114

Bottle filt & Pt = 12·8346
Bottle = 11·4795
1·3551
Filt = ·4301
Pt of $PtCl_4(KCl)_2$ = ·925

488 : 78 :: ·925 : X X = ·1478 amt of K in Pt.
(atomic weight Pt) (K2)

1·0114 : ·1478 :: 100 : X X = 14·61 per cent K by experiment in Rochelle salt.

per cent by theory = 13·83 by ex ~~[illegible]~~ to high.

XXVII Estimation of $\left\{\begin{matrix}CH_3\\COHo\end{matrix}\right.$ by $\left\{\begin{matrix}CH_3\\COAgO\end{matrix}\right.$.

Precipitated a quantity of NO_2AgO with $CONaO_2$ filt & washed till no longer alkaline. Dissolved the ppt in boiling $\left\{\begin{matrix}CH_3\\COHo\end{matrix}\right.$, — Filt sol & allowed to crystallize — Washed cryst with OH_2 — Dried on water bath — Took a weighed quantity in a weighed crucible — Ignited gently & then strongly, & weighed residual Ag.

1st ex $\left\{\begin{matrix}CH_3\\COAgO\end{matrix}\right.$ emp = ·4253.

Ag after ignition ·27315.

per centage Ag by theory = 64·67

·4253 : ·27315 :: 100 : X X = 64·22 % by ~~the~~ experiment.

2nd ex. $\left\{\begin{matrix}CH_3\\COAgO\end{matrix}\right.$ employed = 1·076

Ag obtained after Ignition = ·69715

1·076 : ·69715 :: 100 : X X = 64·79 per centage by 2nd experiment ·12 too high.

XXVIII Estimation of N in Uric acid ($C_5H_4N_4O_3$)

Weighed out a small quantity of the substance — previously dried on the water bath. Introduced it into a mortar, mixed with Soda lime & passed into a combustion tube. Mixed mortar with fresh soda lime. Heated in a combustion furnace — gradually beginning at the front. Collected the NH_3 in a semi-normal sol of HCl. (1cc = ·014 gN) standardised back with a semi-normal sol of Ammonia —

Bottle & subst = 9·4189
Bottle back = 9·2373
Subst emp = ·1806

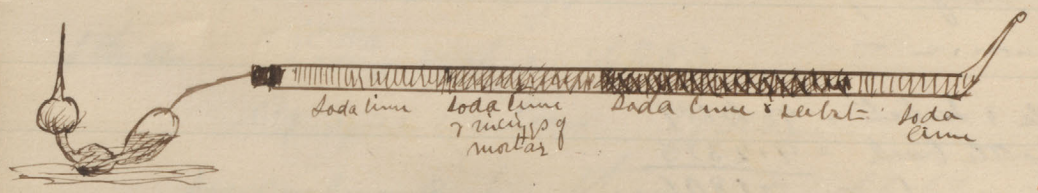
Amt of acid saturated by NH_3 4·2 cc

4·2 cc X ·014 = ·0588 Amt of N found

·1806 : ·0588 :: 100 : X X = 32·6 per cent N in $C_5H_4N_4O_3$ by ex ·7 too low.

~~XXIX~~. Estimation of N in Uric acid $C_5H_4N_4O_3$

Conducted the combustion with soda lime as last time, But lead the NH_3 into ~~[illegible]~~ ~~[illegible]~~ HCl. Added $PtCl_4$ to the acid solution – Evap to dryness – Took up with Alcohol & ether & washed on a weighed filt with Alcohol & ether. Dried till constant on water bath & weighed.



Bottle & uric acid = 9·4762
Bottle back = 9·2408
Uric acid emp = ·2354

Weighed filt = ·434

Bottle filt & ppt of $PtCl_4(AmCl)_2$ = 13·1705
11·4797
1·7008
·434
1·2668

Ammount N. Calculated from weight of ppt = ·0786

·2354 : ·0786 :: 100 : X X = 33·39 per Cent

N by ex by theory = 33·33

by ex 0·06 too high

~~XXX~~. Estimation of C, H & O in Sugar. ($C_{12}O_{11}H_{22}$)

Burnt with CuO in a combustion tube, collected the OH_2 formed in a $CaCl_2$ tube & the CO_2 in Liebig's potash bulbs. Calculated O by difference.

Bottle & Subst = 11·0307
~~Bottle~~ back = 10·7773
Sugar Temp = ·2524

$CaCl_2$ tube after combust = 47·5935
do do after do = 47·4433
OH_2 collected = ·152

Potash bulbs After combust = 38·2635
do do before do = 37·8778
CO_2 found = ·3857

18 : 2 :: ·152 : X X = ·01688 amt H obt

44 : 12 :: ·3857 : X X = ·10519 Amt C obt.

2524 : ·01688 :: 100 : X X = 6·68 per cent H obt

2524 : ·10519 :: 100 : X X = 41·63 per cent C obt

(per cent C + per cent H) ÷ 100 = per cent O = 51·69

	Theory	Experiment
C	42·10	41·63
H	6·43	6·68
O	~~51·69~~ 51·47	51·69

XXX

Preliminary Examination.

In the dry way.

Experiment	Observation	Inference
I Heat a small portion of the finely powdered substance in a dry test tube.	I Substance does not change.	Absence of organic matter, of volatile inorganic matter of substances decompose on ignition of hygroscopic moisture of water of hydration of water of crystallization
	II. Substance changes without decomposition.	
	1st Substance changes colour.	
	Yellow while hot white on cooling	ZnO
	From white to yellowish brown when hot & dirty yellow on cooling	SnO_2
	From white to reddish brown and yellow when cold	PbO*
	From white to orange and reddish brown, dull yellow on cooling.	Bi_2O_3
	2nd Substance fuses	Mostly salts of the alkalies & some salts of the alkaline earths.
	3rd Substance sublimes	Ammonium, Mercury & arsenic compounds, Sulphur, Iodine. Volatile organic acids ex-gr Oxalic, Benzoic & Succinic acids.

Experiment	Observation	Inference.
	The sublimate is white & crystalline	$HgCl_2$, As_2O_3 (octahedral crystals)
	— heavy fumes in the tube	Oxalic acid
	The substance fuses before it sublimes	$HgCl_2$
	or sublimes without fusion; is yellow when hot, white when cold.	Hg_2Cl_2
	Violet vapour, feathery, bluish black crystals	Iodine.
	The sublimate is black, turns red on cooling.	HgS
	The sublimate is yellow, — or it consists of reddish brown drops yellow on cooling.	Free Sulphur.
	Odour of frankincense	Benzoic acid.
	Fumes which cause violent coughing	Succinic acid.
Confirm by heating a little of the dry substance with dry $CONa_2$, or black flux in a bulb tube.	Metallic mirror & globules of metal	Hg compounds.
	Black shining mirror and no globules.	Arsenic compounds.
	NH_3 gas given off.	Ammonium compounds.
	CO_2 evolved	A free acid - e.g. As_2O_3 As_2O_5 - Oxalic, Benzoic Succinic acid.

Experiment	Observation	Inference
Confirm by mixing a little of the substance in a mortar, with soda lime & moistening with a few drops of OH_2.	odour of NH_3	Ammonium compounds, ($CO\,Amo_2$, $PO\,Amo_3$, $BO\,Amo$) give off free NH_3 when heated by themselves.
Confirm by heating with a little concentrated SO_2Ho_2 in a test tube.	CO & CO_2 are evolved.	Oxalic acid
	b With partial or total decomposition.	
	1st Substance fuses & gives off OH_2 of hydration (sometimes without fusion), or	Metallic hydrates.
	gives off OH_2 of crystallization becomming liquid at first, then solid & swelling up considerably.	Salts containing OH_2 of crystallization e.g. Phosphates Borates. Alums.
Confirm by examining the condensed water with litmus paper	Alkaline reaction	Ammonium compounds
	Acid reaction.	Free volatile acids, such as: NO_2Ho, HCl. SO_2Ho_2, $SOHo_2$ &c. or acid salts.
	2nd Substance gives off a gas, or condensable vapor with or without change of colour.	
Confirm by introducing a glowing chip of wood into the test tube.	Oxygen, is given off causing a glowing splinter of wood or charcoal to burst into flame & burn most brilliantly	Metallic peroxides. Chlorates. — Nitrates Bromates — Iodates

Experiment	Observation	Inference
	Oxygen mixed with other gases (e.g. SO_2, N, N_2O_4, Cl, Cl_2O) is given off.	From the decomposition of certain sulphates, nitrates, nitrites, chlorates, hypochlorates - bromates Iodates.
	Cl_2O - from the decomposition of hypochlorite by CO_2 of the atmospheric air.	Hypochlorites.
Confirm by passing gas through an acidulated solution of an alkaline chromate	SO_2 is given off, suffocating odour - acid reaction to bit paper	Acid sulphites, from decomposition of sulphates with or without evolution of O - of earthy sulphites or hyposulphites - from the action of organic matter on sulphates - & from the oxidation of metallic sulphides
Heat also in a bulb of combustion tube open at both ends.		
Pass gas through OH_2 & collect O separately	N_2O_4 & O Reddish brown fumes the O gas supports combustion.	Nitrates of heavy metals. (Pb Bi &c)
Confirm by passing gas through lime or baryta, water.	CO_2 non supporter of combustion, the gas precipitates lime or Baryta water.	Carbonates, (alkaline Carbonates excepted) From decomposition of Oxalates & Cyanates.

Experiment	Observation	Inference.
If CO_2 be given off as well, pass through lime or baryta water before igniting the CO	CO - the gas burns with blue flame	Oxalates & Formates.
The same applies to CO_2 & CN.	CN gas of peculiar odour - burns with peach coloured flame.	Cyanides of the noble metals. also cyanates & sulphocyanates.
	N non supporter of combustion	Ammonic nitrate or some fixed nitrate. in the presence of ammonium salts (Am Cl &c)
	N_2O supporter of combustion.	Ammonic nitrate or some fixed nitrate in presence of ammonium salts.
	SH_2 recognised by its odour & action on lead paper.	Hydrated sulphides, sulphites, & hyposulphites.
	NH_3 characteristic pungent odour & alkaline reaction to test paper	Ammonium salts e.g. $(CO\ Am)_2$ & ammonium salts containing fixed acids from Cyanates in presence of OH_2. & from the decomposition of organic substances containing N.
	CS_2	A product of the decomposition of sulphocyanates.

Experiment	Observation	Inference.
Confirm by passing the gas through a solution of KI & starch paste.	Cl pungent odour – the gas bleaches vegetable colours	Certain Chlorates – Chlorides of the noble metals. e.g. $PtCl_4$ $AuCl_3$
	Br & I. recognised by the odour of their vapour, or sublimate & their action on starch paste.	Certain bromides & Iodides when decomposed in presence of air with formation of acids also some bromates & Iodates.
	S vapour – condensing in upper part of tube in yellow drops.	From metallic per-sulphides e.g. PtS_2, AuS_3, Sb_2S_5, SnS_2 FeS_2. leaving the metal Pt Au or lower sulphides
	3rd Substance becomes carbonised & evolves gaseous products of decomposition.	From decomposition of organic substances
Confirm by treating the residue with dilute HCl.	acetone is given off	Acetates
	A carbonate is left which effervesces with dilute acids, whilst the original substance does not effervesce.	Organic acids combined with alkaline or alkaline earthy bases.
	The carbonate is sol in OH_2 the solution has an alkaline reaction	Organic compound of alkalies
	The carbonate is insol in OH_2	Organic acids combined with alkaline earthy metals.

Experiment	Observation	Inference.
II. Heat a little of the substance under examination on charcoal, before the blowpipe.	1st Substance decrepitates	Sodic chloride &c &c
	2nd Substance deflagrates	Nitrates chlorates &c
	3rd Substance fuses readily & runs into the charcoal, or forms a liquid bead.	Salts of alkalies & some salts of alkaline earths.
	4th An infusable residue is left	
	(a) the residue is white	Salts of Ba, Ca, Sr, Mg, Al, Zn, also silica
	alkaline to litmus paper	Ba, Sr, Ca, Mg,
	highly luminous	SrO, CaO, MgO, ZnO.
Confirm by heating the white infusable residue when cold, with a few drops of $Co(NO_3)_2$ & ignite again strongly.	a fine blue mass is left	Al_2O_3 earthy phosphates silica & many silicates.
	a flesh coloured mass is left	MgO
	a green mass is left	ZnO
	a bluish green mass is left	SnO_2
Confirm by heating the substance in a clear borax bead on a platinum wire, in the inner & outer blowpipe flames.	(b) The residue is coloured	CuO, CoO, NiO, MnO, Fe_2O_3, Cr_2O_3
	oxidising flame green bead white hot blue when cold Reducing flame red bead when cold	CuO.
	Blue bead in ~~oxidising~~ both flames ~~red in reducing~~.	CoO
	Reddish brown bead when hot lighter coloured or colourless when cold	NiO
	Amethyst red bead in oxidising flame colourless in re flame	MnO.

Experiment	Observation	Inference
	brownish-red bead when hot, light yellow, ~~at~~ colourless when cold, in oxidising fl. Red, when hot - orange when cold - lastly greenish, in reducing fl.	Fe_2O_3 or FeO
	green in oxidising fl. Green in reducing fl	Cr_2O_3
III. Heat in the inner blowpipe flame. Confirm by heating a little of the substance on a clean Pt wire (the chlorides of the different metals answer best)	1st Substance colours outer flame	
	golden yellow	Na compounds
	violet	K do
	yellowish green	Ba "
	Crimson	Sr "
	reddish	Ca "
	green	Cu comps. Boric acid
	blue	As_2O_3, Sb, Pb, & $CuCl_2$

Experiment	Observation	Inference.
	2nd Substance volatilizes & yields an incrustation.	
	a white incrustation & characteristic garlic odour	As compounds
	a white incrustation nearer to the substance; also reduced to metallic state; the metallic globules emit white fumes, when withdrawn from the flame, & become surrounded by a net work of fine shining crystals of Sb_2O_3	Sb do
	Incrustation yellow when hot white when cold	Zn do
	Incrustation reddish brown. (readily volatilized)	Cd do
	3rd Substance is reduced to the metallic state.	Au Ag, Cu (also Pt, Fe, Ni, Co)
Confirm by mixing a little of the substance with $CONa_2$ & KCy. & heating on charcoal in inner blowpipe flame.	(a) Without Incrustation	
	Brilliant white white metal	Ag
	yellow metal	Au
	red scales or globules of a red metal	Cu
	Metallic powders, which cannot be fused into globules.	Pt Fe Ni Co
	(b) With incrustation.	
	Metal malleable, incrustation yellow when hot, white when cold (slight)	Sn
	Metal brittle, incrustation dark orange when hot lemon yellow when cold	Bi
	Metal malleable, incrustation brown-yellow when hot, light yellow when cold	Pb

Experiment	Observation	Inference
Heat a little of the substance mixed with dry CO_3Na_2 (free from sulphate) in the reducing flame, & place the fused mass on a silver coin.	Metal brittle, incrustation white. An alkaline sulphide is left. The silver is stained black & the fused mass evolves SH_2 when touched with dilute HCl.	Sb. Sulphur compounds

Note 1. Many of the changes observed, on heating a substance by itself in a tube, occur again when the substance is heated on charcoal, but may be neglected as they are so much more easily studied by heating the substance in a glass tube.

Note 2. A substance under examination may consist of several different bodies & the reactions which they give frequently obscure, or destroy each other. Analytical tables however carefully they may be compiled, however, cannot possibly provide an explanation of every change, that may be produced during the Preliminary Examination of a substance. The student is therefore advised to record faithfully every observation he makes, although he may not be able, at the time to draw any inference from it, & to elicit by further experiments to what class of bodies the substance belongs, which he is called upon to examine in the dry way.

Absolute proof, in many cases, can only be obtained by an examination in the wet way.

Examination of a substance in the wet way.

1st The substance under examination may be liquid — Test by means of litmus paper. It shows acid reaction & may contain a normal salt, possessed of acid reaction, or an acid salt, or lastly a free acid. It shows Alkaline reaction a neutral or normal salt of alkaline reaction, or a free alkali

Evaporate a portion to dryness on a watch glass or the lid of a platinum crucible. It leaves no residue. Indicative of some volatile liquid, such as distilled water, alcohol, ether &c. It leaves a residue which must be subjected to a preliminary examination in the dry way.

2nd The substance under examination consists of a solid body. Dissolve in distilled water, with the application of heat; if the substance be insoluble in water, treat with dilute HCl. (dilute NO_2Ho is but rarely resorted to) & heat to boiling for some time. If dilute acids do not effect the solution of the substance treat with a little concentrated acid. (HCl, or NO_2Ho) and if a residue be still left, with a little aqua regia.

A substance insoluble in both water and acids must be fused with 3 or four times its weight of sodic & potassic carbonates.

Instructions for dissolving solid substances.

1st Boil with distilled water, allow the undissolved portion of the substance to subside before decanting the supernatant clear liquid, & treat the residue again with boiling water, as long as anything is dissolved, which can be ascertained by evaporating a drop or two of the solution on a watch glass.

The Aqueous extract is set aside untile the residue has in like manner been treated with acids. Test with litmus paper.

2nd The Residue insoluable in water, is next treated with dilute HCl & heated to boiling for some time. The undissolved portion is allowed to subside, & the clear fluid decanted. This operation of boiling with acid must be repeated several times, both with dilute & concentrated acid. Boiling with HCl generally dissolves out whatever is soluable, & nitric acid has rarely to be resorted to. Before fusing the residue treat also with small quantities of aqua regia.

3rd The Residue insoluable in water and acids, should be very finely pulverized in an agate mortar, before being mixed with 3 or 4 times its weight of dry Sodic &

potassic carbonates. (Fusion mixture) & fused. The fusion is best performed in a platinum crucible, provided the insoluable residue does not contain any metals capable of forming alloys with platinum. — readily ascertained by an examination in the dry way of the residue.

It may be borne in mind that only Basic, Strontic, Calcic, & Plumbic Sulphate; Plumbic & Argentic chlorides; Silicic anhydride, many silicates; native and ignited Alumina, & aluminates; ignited Chromic oxide Chrome iron ore, Stannic oxide (ignited or as tinstone) a few Metaphosphates & arsenates, Calcic fluoride & a few other fluorides, Sulphur & Carbon. Can possibly be present in the residue. Argentic Ferro & Ferricyanide, Bromide, Iodide, & Cyanide, are decomposed by boiling with aqua-regia into argentic chloride.

Solutions 1 & 2 may be examined seperately, or they may be mixed. A seperate examination of the aqueous & acid extracts, becomes necessary only when the analysis is intended to show how acids & bases are combined in a compound body. The examination of a residue, which requires fusion with alkaline carbonates is invariably conducted seperately. The fused mass is boiled with OH_2 & filtered, & the residue dissolved in HCl or NO_2Ho. The aqueous extract is examined for acids & the residue for bases.

Notes to general table

Note 1 If the original substance had to be dissolved in HCl. SH_2 should be passed at once.

Note 2 In a saturated solution of a Barium salt HCl produces a white ppt soluble in hot OH_2. From an alkaline solution HCl may ppt silicic hydrate (gelatinous), Boric, Benzoic & Uric acids (crystalline) also Antimonic acid (amorphous). Metallic oxides, such as Al_2O_3 Metallic sulphides such as As_2S_3 Sb_2S_3, Sb_2S_5, SnS, & SnS_2, which dissolve in NaHo or SAm_2 may likewise be ppt'ated on the addition of HCl & are best examined seperately.

Note 3 If Arsenic has been detected in the preliminary examination, this filtrate must be boiled with a solution of Sulphurous acid, or hydric ammonic sulphite, then evaporated considerably to expell the SO_2. — Barium Strontium & Lead when present may be precipitated partially or entirely as Sulphates the ppt is best examined seperately.

Note 4 Certain oxychlorides e.g. of Bismuth, Antimony & Tin may be precipitated on the first addition of HCl. But are readily redissolved on the addition of more acid & heating; or the precipitate may be disregarded. Since SH_2 readily converts the finely divided oxychlorides into the corresponding sulphides.

Note 5 SH_2 should be passed once more through the filtrate, to make sure of the complete precipitation of all the metals of Group II.

Note 6 SH_2 often produces merely a precipitation of S, owing to the presence of oxidising agents such as free Cl, Br_2, I, sulphurous, nitrous, hypochlorous, chloric, iodic, bromic, nitric, & chromic acids, or of ferric salts. This precipitate is easily distinguished by its being white & remaining suspended in the solution. It may be neglected. A brick red precipitate of ~~PbSCl~~ PbS $PbCl_3$, sometimes comes down from strongly acid (HCl) solutions, if the solution has not been sufficiently diluted with water.

Note 7 Organic acids e.g. Tartaric Citric &c, also Sugars &c prevent the precipitation of Ferric oxide & alumina in Gr III.

Note 8 It is possible that this SiO_2 may contain other substances e.g. Al_2O_3, Cr_2O_3, Fe_2O_3 (rendered insoluble by strong ignition) SO_2BaO, SO_2SrO & it is necessary to examine it seperately.

Note 9 If the SAm_2 were added at once, Phosphate of Iron Mn, Zn, Ni, & Co, would be decomposed into sulphides with formation of ammonic phosphate, which might produce ~~by acting~~ a precipitate of the alkaline earthy bases, by acting upon salts of these bases other than Phosphates, or act as a solvent for other Phosphates in the presence of Am Ho

Note 10 This filtrate may contain $PO_4H_3O_3$ — a proof that a phosphate of the metals Fe, Mn, Zn, Ni, or Co, was present in the original HCl solution or vice versa, that these metals may be present

Note 11 Small quantities of Borates & fluorides of the alkaline earths, may likewise fall out, together with the alkaline earthy phosphates; but need not be examined further since their bases will be detected in Group IV & their acids, on examining in the usual way for acids.

Note 12 The solution must not be boiled, since the $Am\,Cl$ might become decomposed, & dissolve the alkaline earthy Carbonates, as Chlorides.

General Table for the

To the greater part of the original

ppt may contain
AgCl white
$PbBiCl_2$ "
Hg_2Cl_2 "
(note 2)

Examine by table I for group I.

Filtrate (note 3) largely diluted with OH_2 (note 4) is

ppt may contain
PbS black
HgS "
CuS "
Bi_2S_3 "
CdS yellow
} insol in NaHo or SAm_2

SnS brown
SnS_2 yellow
Sb_2S_3 orange
Sb_2S_5 "
As_2S_3 yellow
Au_2S_3 black
PtS_2 "

Examine by Table II for Group 2

Evaporate filtrate till free from
If oxalates or organic matter
to redness in an open dish;
HCl, add OH_2 & heat, when
of SiO_2 (note 8) Test a small
A. No ppt is obtained – $POHo_3$
of the solution, heat to boiling
B. A ppt. is obtained – $POHo_3$
remaining solution, heat
filtrate add SAm_2 to slight
containing a few drops of S
dish and digest with a

ppt A may consist of
CoS black
NiS "
ZnS white
MnS Flesh coloured
FeS black
Cr_2Ho_6 Bluish green
Al_2Ho_6 yellowish white gelatinous.

Examine by Table III for Group 3 A

ppt B
CoS
NiS
ZnS
MnS
FeS
Cr_2Ho_6
Al_2Ho_6
Together
of Cr &
alkalin
Examin
Group

Examination of Bases.

solution add HCl & heat gently (note 1)

saturated with SH_2 & gently heated (note 5-6)

SH_2, add a few drops of concentrated NO_2Ho & evaporate to dryness (Note 7) be suspected (indicated by the blackening of the residue) heat but not otherwise. Treat the residue with a little concentrated it will either dissolve entirely or leave a white residue of portion of the HCl solution with Ammonic Molybdate.

is absent - Add Am Cl & Am Ho & SAm_2 to the remaining portion and filter quickly. Wash well with hot OH_2 containing a little SAm_2.

is present - Add Am Cl & Am Ho in slight excess to the gently & filter quickly. Wash well with hot OH_2 to the excess, heat to boiling and filter. Wash with hot OH_2 Am_2 (note 9) Transfer ~~with~~ the two precipitates to a porcelain little SAm_2. Filter off and wash well, Neglect filtrate (note 10)

(note 11) may contain
black
"
white
flesh coloured
black
bluish
~~yellowish~~ green
yellowish white
with the phosphates
Al as well as of the
earthy bases
by Table III for
3. B.

Add Am Ho & CO_3Am_2 to filtrate from A or to the united filtrate from B heat gently (note 12) & filter

ppt may contain.
CO BaO white
CO SrO " "
CO CaO " "

Examine by table IV for Group 4.

Solution may contain
K_2O
Na_2O
MgO

Examine by table for group 5.

Tables for the Separation of

ppt. May contain $AgCl$, Hg_2Cl_2, $PbCl_2$ [also $BOHo$, with boiling OH_2 till it is white free from $PbCl_2$

The filtrate may contain $PbCl_2$ On cooling white acicular crystals fall out Confirm by CrO_2 HO_2 Pres of Pb.	The white residue may The solution may. Acidulate with dilu A white curdy ppt Pres

Note 1. Unless HCl has been added in excess of Bi & Sb as well as Sn, also SiO_2, HO_2 & a few group.

the Metals of Group 1

Benzoic or Uric acids (Note 1) Wash the ppt. on the filter

contain $AgCl$, Hg_2Cl_2, — Add excess of dilute $AmHo$.

contain $AgCl$.	The residue is black.
te NO_2Ho.	Confirm by drying residue & heating
of $AgCl$ is obtained	with dry $CONaO_2$ in a bulb tube
	A metallic mirror & globules indicate
of <u>Ag</u>.	<u>Pres of Hg</u> (as Mercurosum)

1 and the solution gently heated certain oxychlorides
other substances may have been precipitated in this.

Table for the Seperation of

The ppt. may contain PbS. HgS, CuS, Bi_2S_3, Cd S, As S. tested for in a seperate portion of the filtrate from group 1) Wash the ppt (note 1)

Residue Wash well. Boil in a little concentrated NO_2Ho until action ceases. Dilute with OH_2. add dilute SO_2Ho_2 as long as a ppt produced, allow to cool & add an equal bulk of alcohol (methylated)

Residue Boil in $\{CH_3 CO$ am o, SO_2Pbo'' dissolves. — allow to cool & filter.		**Solution**, boil off alcohol add excess Am Ho boil & filter.		
Residue consists of HgS & S. or S alone Confirm by reducing the dried HgS in a bulb tube with $CONa_2$ Metallic globules of Hg and mirror **Pres of Hg.** (as ~~[illegible]~~ cinnabar)	**Solution** Add CrO_2KO_2 yellow ppt of CrO_2Pbo'' **Pres of Pb**	ppt. consists of $Bi\,Ho_3$. Dissolve in a few drops of dilute HCl evaporate to dryness & add OH_2. Milkiness from BiOCl **Pres of Bi**	**Solution**, acidulate with & pass through SH_2. Filter wash ppt with weak Boil with dilute SO_2Ho_2	
			Residue dissolve in NO_2Ho add excess of Am Ho & then $\{CH_3 CO Ho$ & $K_4 Fe Cy6$ Brown ppt of $Cu_2 Fe Cy6$. **Pres of Cu**	**Solu** Am Ho SH_2. Yell of Cd **Pres**

(note 1) Cu S is somewhat soluable in SAm_2. HgS in SNa_2. of Cu, with SAm_2, and in the absence of HgS with SNa_2. when a little Cu S will be found in the solution.

the Metals of Group 2.

SnS_2, As_2S_3, Sb_2S_3, Sb_2S_5 - (Au & Pt must be specially untill free from HCl, transfer to a flask, & boil with NaHO or SAm_2 & filter.

all is Filter.

Solution may contain As, Sb, Sn, as sulphosalts. Acidulate with dilute HCl - As_2S_3, Sb_2S_3, & SnS_2 are reprecipitated. Filter & wash, digest with $CO(NH_2)_2$. Filter

es of

h HCl off and SH_2 water & filter.

Residue. Consists of Sb_2S_3 & SnS_2, Dissolve in boiling HCl. Introduce into a Marsh's apparatus in which H is generated by means of Zn & HCl.

SbH_3 comes off. Collect as metallic Sb and identify the deposited metal by means of NaCl. or dry HCl gas

Pres of Sb

The **Residue** in the generating flask consists of Zn & Sn. Detach the precipitated Sn from the strips of Zn, wash and dissolve in a little concentrated HCl by the aid of Pt foil. Dilute with OH_2 & add $HgCl_2$. A white ppt of Hg_2Cl_2 or a grey metallic ppt of Hg is obtained

Pres of Sn.

Solution contains As_2S_3 Precipitate by adding HCl. Confirm pres. of As by reducing with KCy & $CONa_2O_2$ in a bulb tube.

Metallic mirror & garlic odour

Pres of As

tion add & pass

ow ppt S

of Cd

It is therefore advisable to dissolve in the absence If both Cu & HgS are present dissolve in SAm_2

Table for the Seperation of

A. $PO\,H_3$ is absent. Dissolve the ppt. of Group 3 in a little at a gentle heat, as long as any chlorous odour is perceptable. $CO\,Ba''$ and shake well. Allow to subside in a flask kept corked liquid, throw the Ppt. on a filter & wash with hot OH_2.

Solution – Remove $Ba\,Cl_2$ by adding a few drops of dilute $SO_2\,Ho_2$. boil, allow to subside & filter off $SO_4\,Ba''$. To the filtrate add pure $Na\,Ho$. ~~add excess of $\{CH_3$ $\{CO\,Am_2$ and pass a rapid current of SH_2, through the solution for some minutes and filter.~~ till it becomes alkaline boil,

Solution may contain Zn as potassic Zincate. Add SH_2 white ppt. of $Zn\,S$

Pres of Zn

ppt. may contain $Mn\,Ho_2$, $Co\,Ho_2$ & $Ni\,Ho_2$ – wash, in a little dilute HCl, nearly neutralize with $Am\,Ho$, excess of $\{CH_3$ $\{CO\,Am_2$ and pass a rapid current of SH_2 the solution for some minutes & filter.

Solution contains $\{CH_3$ $\{CO\,Mn_2''$ $\{CO$ $\{CH_3$ add $Am\,Cl$, $Am\,Ho$, & SAm_2 Flesh coloured ppt of $Mn\,S$.

Pres of Mn.

Residue. dissolve in HCl & $K\,Cl\,O_3$, neutralize with $CO\,Na_2$, add a weak $K\,Cy$ so as just to redissolve the ppt Boil briskly for some time, allow to cool any slight ppt.) & add a strong solution Allow to stand for some time in a place, as long as a black ppt forms.

ppt $Ni_2\,O_3$ – Confirm by heating a little on a borax bead, before the blowpipe. A cherry red bead

Pres of Ni

Solution con as $K_6\,Co_2\,Cy_{12}$ to dryness, & test the residue. blowpipe on a Blue bead in

Pres of

the Metals of Group 3.

dilute HCl with the addition of a few crystals of ClO_2Ko. Digest. Filter off S if necessary. Neutralize solution with $CO\,Nao_2$, add so as to exclude the air as much as possible. Pour off the clear

& filter.

ppt dissolve in dilute HCl remove $BaCl_2$ by dilute SO_2Ho_2. Filter. To the filtrate add pure NaHo (free from Al_2O_3) in excess & boil for a few minutes.

dissolve add through

ppt. Fuse with fusion mixture of No_2Ko on Platinum. Dissolve in hot OH_2 & filter.

nearly solution of first product (filter off of NaO Cl wash Filter

Residue dissolve in dilute HCl & add $Fe\,Cy_6K_4$

A ppt of Prussian blue

Pres of Fe

Test original HCl sol specially for Fe'' – Fe''' by means of Potassic per-manganate in a highly dilute solution & by $K_4Fe\,Cy_6$

Solution is yellow. Confirm by adding $\left\{\begin{matrix}CH_3\\COHo\end{matrix}\right.$ and $\left(\begin{matrix}CH_3\\CO\end{matrix}\right)_2Pbo''$

Yellow ppt.

Pres of Cr.

Test original sol specially for Cr_2O_3 & CrO_3 by boiling with $CO\,Nao_2$

The filtrate contains the chromate & the residue the chromic oxide, which can be converted into a soluble alkaline Chromate, by fusion $CO\,Nao_2$ & No_2Ko_2

Solution

Acidulate with dilute HCl, & add Am Ho in slight excess

White gelat ppt.

Pres of Al

tains Co – Evaporate a little of before the borax bead bot flames **Co**.

B. PO H_3 present – Dissolve ppt. in dilute HCl with the addition as any chlorous odour is perceptable Filter off S if necessary. produced – Filter hot

Solution, add Fe_2Cl_6 (Note 2) drop by drop, as long as a ppt forms, and till the colour of the supernatant liquid turns red. Digest for some time at a gentle heat, allow to subside & filter hot. Wash with hot OH_2 (See notes).

Solution. Add AmCl, Am HO, & SAm_2. A black ppt is obtained. Filter.

ppt. Consists of P_2O_2, Fe_2O^{III} Neglect

Solution. Add $COAmO_2$

A white ppt

May be $COBaO^{II}$
" " $COSrO^{II}$
" " $COCaO^{II}$

Examine by table IV.

Solution, may contain Mg. Add POH_3NaO_2 white cryst ppt.

Pres of MgO^{II} as a Phosphate

ppt Examine according to Table 3 A for Co, Ni, Mn, Zn, Al, & Cr,

Since $FeCl_6$ has been introduced into the solution, test a portion of the original sol (dissolved in OH_2 or HCl) for Fe^{II} & Fe^{III}

$FeCy_6K_4$ a dark blue ppt.
Cy Am S a blood red colour.

Pres of Fe_2O_3

$Fe_2Cy_{12}K_6$ a blue ppt.

Pres of FeO

Note 1. If no phosphates of Al, Cr, Ba, Sr, Ca, Mg, were which may be readily ascertained by adding a little to Table 3A.

Note 2. If Fe_2Cl_6 produces no further ppt with a portion of the Acetic bases can be present, and no iron salt need be

of a few crystals of $KClO_3$. Digest at a gentle heat, as long (Note 1) Add $\{CH_3 CO\,Amo$ in excess & a little free $\{CH_3 CO\,Ho$ – a ppt is

ppt May consist of $P_2O_2\,Fe_2O''$ (see notes) P_2O_2, Al_2O''; $P_2O_2\,Cr_2O''$. Dry on the filter; fuse in a platinum crucible with SiO_2 & pure fusion mixture together with a little NO_2Ko. Dissolve in hot OH_2 add $CO\,Amo$. Digest, allow to subside, & filter.

Residue $Si_3O_3\,Fe_2O''$, Si_3O_3, Al_2O'' & $Fe\,Ho_6$. Dissolve in HCl evaporate to dryness and ignite gently. Extract with a few drops of concentrated HCl & Hot OH_2. Filter.

Solution. add $\{CH_3 CO\,Amo$ of $\{(CH_3)_2 (CO)_2\,Pbo''$ Yellow ppt Pres of Cr_2O_3 and therefor Pres of Cr_2O_3 as phosphate

A white ppt ~~a white~~ of $P_2O_2\,Pbo''_3$ indicates pres of $PO\,Ho_3$ Neglect.

Solution. Add pure $NaHo$ in excess & heat. Filter

Residue SiO_2 Neglect

Residue Fe_2Ho_6. Confirm by dissolving in HCl & adding $CyAm\,S$. a blood red colour

Pres of Fe_2O_3 as Phosphate

(See explanatory notes)

Solution. Acidulate with dilute HCl, add Am Ho in slight excess. a white gelat ppt.

Pres of Al_2O_3 as phosphate.

originally present, but merely phosphates of Ni, Co, Mn, Fe & Zno Ammonic molybdate to the original sol – Examine at once ~~by~~ according

acid solution, no more phosphates of the alkaline earthy added to the main portion of the solution.

Table for the Seperation of

ppt may contain, $CO\ Bao''$, $CO\ Cao''$, $CO\ Sro''$. Dissolve of HCl. Evaporate to dryness on a waterbath. Pulverize the paste & Digest with absolute Alcohol. Filter off and

Residue $Ba\ Cl_2$ Confirm by dissolving in water and adding $Si\ H_2\ F_6$ or $Cr_2O_2Ko_2$ Pres of Ba.	Solution. add dilute time with a concentrated & filter. Residue $SO_2\ Sro''$. Confirm by blowpipe with concentrated HCl loop of a platinum Pres of —

the Metals of Group 4.

the ppt produced in Group 4, in the least possible quantity residuary chlorides (in the evaporating dish) finely with a small wash with absolute alcohol.

SO_2HO_2 & filter. Digest the precipitated sulphides for some solution of SO_2 AmO_2 & a little Am Ho at a gentle heat

reaction by treating & heating in the wire.	Solution contains SO_2 CaO". Dilute with much OH_2, & add $\left\{\begin{matrix} CO\ Amo \\ CO\ Amo \end{matrix}\right.$ a white ppt.
Sr.	Pres of Ca.

Table for the Seperation of

Solution may contain MgO, K_2O, Na_2O. Evaporate vessel, until no more Ammoniacal fumes come off.

Dissolve a small portion of the residue in OH_2 with the aid of a few drops of HCl and filter if necessary. To the clear filtrate add AmCl, Am Ho & PO Ho Na_2, digest for some time at a gentle heat, stir well with a glass rod, and allow to stand for some time.

A white ppt (crystalline) of PO Am Mgo"

Pres of Mg.

Dissolve the main portion $BaCl_2$ to remove any SO_2 drop by drop as long as a

Residue	Solution. Rem gently and
Mg Ho_2	Residue.
Neglect.	CO Bao"
	Neglect.

the Metals of Group 5.

filtrate from group 4 to dryness & ignite in platinum

of the residue in OH_2 & a little HCl, add a solution of HO_2 present, and filter. To the filtrate add Baryta water ppt. of $MgHO_2$ comes down. Filter.

ove excess of $BaCl_2$ & $BaHO_2$ by the addition of $CO(NH_2)_2$. Heat filter.

<u>Solution</u>. Evaporate to dryness and ignite, test residue on a platinum wire, after moistening with OH_2 in the inner blowpipe flame. — Yellow flame - <u>Pres of Na</u> Reddish violet flame when seen through indigo prism <u>Pres of K</u>. Confirm by $PtCl_4$.

Explanatory Notes to Table 3 B.

Decomposition of alkaline earthy phosphates by means of Fe_2Cl_6 in an acetic acid solution.

1st $AmCl$ & $AmHo$ precipitate Phosphates of Ni, Co, Mn, Zn, Fe, Al, Cr, Ba, Sr, Ca, Mg, as well as Fe_2Ho_6, Al_2Ho_6, Cr_2Ho_6 provided excess of $AmHo$ be avoided.

2nd SAm_2 precipitates from the filtrate salts of Ni, Co, Mn, & Zn, other than phosphates.

On digesting the two ppt's with SAm_2, the phosphates of Ni Co, Mn, Zn, Fe as well as Fe_2Ho_6 are decomposed into sulphides. The Phosphates of Al, Cr, Ba, Sr, Ca, Mg, as well as the hydrates of Al, & Cr, are however not affected.

On dissolving the NiS, CoS, MnS, ZnS, FeS, Al_2Ho_6, $P_2O_2Al_2O^{vi}$, Cr_2Ho_6, $P_2O_2Cr_2O^{vi}$, and alkaline earthy phosphates, in HCl & $KClO_3$. The FeS is converted into Fe_2Cl_6, which in an acid solution, (obtained by adding $\left\{\begin{matrix} CH_3 \\ COamo \end{matrix}\right.$ to the HCl solution) decomposes the phosphate of the alkaline earthy bases, precipitating Ferric phosphate & leaving $BaCl_2$, $SrCl_2$, $CaCl_2$, & $MgCl_2$ in solution.

Now iron may have been absent altogether from the solution of group 3, or it may have been present originally, first as <u>oxide</u>, 2nd - as <u>Phosphate</u>

3rd – both as oxide & phosphate. This distinction is however lost sight of, since by boiling with $SCam_2$ both oxide, & ferric phosphate, are converted into FeS.

1st – If no iron was present in the original solution the ppt. ~~must consist of $P_2O_2Fe_2O^{VI}$ possibly also of~~ produced on adding $\left\{\begin{matrix} CH_3 \\ COAmo \end{matrix}\right.$ in excess, can only consist of Phosphate of Al & Cr, & Fe_2Cl_6 must then be added to the filtrate, till the whole of the $PO\,Ho_3$ has been precipitated.

2nd – If iron was present in the original solution the ppt must consist of $P_2O_2Fe_2O^{VI}$, possibly also of $P_2O_2Al_2O^{VI}$ & $P_2O_2Cr_2O^{VI}$ provided always that Phosphate of the alkaline earthy bases were present in Group 3, because the $PO\,Ho_3$ originally in combination with metallic bases has been removed by filtration. – If not no $P_2O_2Fe_2O^{VI}$ can be precipitated, and iron may be found in the solution,

of B. – No more $Fe\,Cl_6$ need then be added (compare also note 1 group 3)

3rd – If however the ammount of iron present was not sufficient to precipitate the whole of the $PO\,Ho_3$ from the alkaline earthy bases, as $P_2O_2Fe_2O^{VI}$. Some more Fe_2Cl_6 must be added to the acetic acid solution.

Examination for Acids.

1 Before proceeding to the examination for Acids, the Analyst will do well to consider carefully, which acids can possibly be present, in combination with the bases, which were found in the previous examination. A perusal of the "Table of solubility of salts" given in Fresenius' Qualitative Analysis (page 260) will materially assist him, & will probably save much labour & time.

2 Some acids have already been detected in examining for bases, viz As_2O_3, As_2O_5, SiO_2 $PO\,Ho_3$, $CrO_2\,Ho_2$, CO_2, SH_2 $SO\,Ho_2$, $SSO\,Ho_2$, $Cl\,Ho$, $NO\,Ho$, The latter six on dissolving in dilute HCl or else in Group 1.

3 A careful preliminary examination for acids will probably lead to the detection of a further number of acids. e.g. HI ($IO\,Ho$) HBr ($BrO_2\,Ho$), ($ClO_2\,Ho$) $NO_2\,Ho$ ($NO\,Ho$) HCl, $Cl\,Ho$, HF, HCy, H_4FeCy_6, $H_6Fe_2Cy_{12}$, $Cy\,Ho$, & $Cy\,Hs$, $\left\{\begin{matrix}CH_3\\COHo\end{matrix}\right.$, $\left\{\begin{matrix}COHo\\COHo\end{matrix}\right.$. The presence of organic acids will also be indicated. When more than one of these acids is present the detection is somewhat more difficult, & the results obtained by a preliminary examination for acids, require to be carefully confirmed by the subsequent examination of the solutions. Thus a chloride in presence of a nitrate

when treated with concentrated $SO_2 Ho_2$ evolves Cl & red fumes of N_2O_4; in presence of a chromate red fumes of Chlorochromic acid &c.

$HgCl_2$, Hg_2Cl_2, $SnCl_4$, do not evolve HCl when treated with $SO_2 Ho_2$, sulphides when treated with HCl often evolve SH_2 with seperation of S.

SO_2 & SH_2 when evolved simultaneously destroy each other &c.

4. When the preliminary examination ~~gives~~ no distinct indication of the presence of any of the acids just mentioned SO_2Ho_2, $BOHo$, $POHo_3$, $SiOHo_2$ will have to be looked for.

5. The Analyst will have to bear in mind that acids cannot be detected in the same systematic order as bases, & that he ought never to be satisfied, therefore, till he has confirmed the presence of acids by the most characteristic special tests.

Preliminary Examination for Acids.

Experiment	Observation	Inference.
Treat a little of the solid substance in a test tube with dilute HCl & heat gently	Acids given off 1 without decomposition a colourless & inodorous gas is evolved which ppts lime water.	CO_2 (from carbonates, likewise from cyanates & cyanates containing carbonates.)
Confirm by passing SO_2 thro' an acidulated sol of $CrO_2 KO_2$	A gas is evolved having the odour of burning sulphur	SO_2 (from sulphites)
Test with lead paper	A gas is given off possessing a most fœtid odour. the odour of rotten eggs.	SH_2 with metallic sulphides (with the exception of CuS, AgS, HgS, PtS_2, Au_2S_3, & AsS_3.)
	reddish brown fumes.	N_2O_4 (from nitrites) decomposing into N_2O_2 & NO_2 Ho.
	A colourless gas with the odour of bitter almonds	HCy (from cyanides)
	2 With decomposition a yellowish green gas, with suffocating odour bleaching vegetable colours	Cl Ho & Cl (from hypochlorites) Cl alone from decomposition of CrO_2 Ho_2 by HCl.
	SO_2 evolved & yellow S ppt	from hyposulphites
	CO_2 is given off, accompanied by the pungent odour of Cy Ho. Am Cl being formed at the same time & the residue gives off NH_3 when heated with Ca Ho_2.	

Experiment	Observation	Inference.
Heat another portion of the solid substance with moderately concentrated SO_2Ho_2	Acids given off.	
	1 Without decomposition	CO_2, SO_2, SH_2, NO_3, HCy. (already recognised by treating with dilute HCl
	acetic acid is given off.	$\left\{\begin{matrix} CH_3 \\ COHo \end{matrix}\right.$
Confirm by distilling with alcohol & concent SO_2Ho_2	acetic ether $\left\{\begin{matrix} CH_3 \\ COEto \end{matrix}\right.$ is evolved	$\left\{\begin{matrix} CH_3 \\ COHo \end{matrix}\right.$
	Pungent acid fumes	HCl, HF, HCy.
	2 With decomposition	$SSOHo_2$, $ClHo_2$, $CyHo$, (have already been noticed.)
	a gas is evolved possessing the odour of bitter almonds	HCy (from Ferro & Ferri-cyanides)
Confirm by causing the gas to act on SAm_2 & adding F_2Cl_6	blood red coloration	Cyanogen compounds.
Heat a little of the substance with concent SO_2Ho_2, & warm gently (not sufficiently to volatilize the SO_2Ho_2)	1. Acids given off	CO_2 $NOHo$, SO_2 $\left\{\begin{matrix} CH_3 \\ COHo \end{matrix}\right.$
	a. without decomposition	(these acids have already been noticed HCl, HF, NO_2Ho, Benzoic & succinic acids.)
Confirm HCl by heating with MnO_2 & SO_2Ho_2	Cl is evolved, recognised by its odour, its action on vegetable colours & upon KI & starch paste.	HCl.
Confirm by etching upon glass.	Heavy suffocating fumes, which corrode glass.	HF

Experiment	Observation	Inference.
Confirm by adding concent SO_2Ho_2 to a sol of the salt & SO_2FeO" (If a chloride as well as a nitrite is present equite gently previously to heating with SO_2Ho_2 & SO_2FeO".	acid fumes condensing to yellowish liquid.	NO_2Ho
	a brown ring is formed	"
	b. <u>With decomposition</u>	$ClHo$ & $CyHo$ (already mentioned) SH_2, HI, HBr, ClO_2Ho, BrO_2Ho, CrO_2Ho_2. Tartaric & citric acids $\left\{\begin{matrix}COHo\\COHo\end{matrix}\right.$, Cy_4Ho, H_4FeCy_6, H_6FeCy_{12}
	<u>SH_2</u> as well as <u>SO_2</u> are evolved owing to the action of the liberated S upon SO_2Ho_2	Metallic sulphides.
	<u>O is given off</u> & the yellow chromate is changed into the green chromic sulphate.	CrO_2Ho_2
	<u>Cl_2O_4</u>, <u>a greenish-yellow</u> highly explosive gas is given off. Detonation in tube, on heating.	ClO_2Ho
Confirm HI & HBr by distilling with MnO_2 & SO_2Ho_2	<u>Br is evolved</u> (together with HCl & SO_2) condensing to a reddish-brown liquid. colours starch paste yellow.	HBr
	<u>I is given off</u> violet fumes, & sublimate colouring starch paste blue	HI.

Experiment	Observation	Inference
	Cl gas is evolved	HCl as well as $C_2O_2H_2$
	Cl & nitrous fumes are given off	HCl ~~as well~~ together with NO_2Ho is NOHo.
	Substance blackens from seperation of C with evolution of CO, CO_2 & SO_2 & gives off characteristic odour of burnt sugar.	Tartaric & Citric acid
Pass gases through solution of Na Ho. The CO burns with blue flame	CO & CO_2 are evolved without blackening	$\left\{\begin{matrix} COHo \\ COHo \end{matrix}\right.$
	The acid is decomposed into CO & NH_3 the latter remaining with the acid (combined)	Cyanides, Ferro & Ferri Cyanides.
	CO_2 is given off & an ammonium salt is left.	Cyanates.
	CO_2 & SH_2 are evolved with seperation of S which reacts upon the SO_2Ho_2 & forms SO_2. An ammonium salt is left.	Sulphocyanates

Experiment	Observation	Inference.
	II. No gas or vapour is evolved.	SiO_2, B_2O_3, P_2O_5, As_2O_5, SO_3, I_2O_5.
	a gelatinous mass, or a flakey powder is left	$SiOHo_2$ from soluble silica, or silicates decomposed by acids.
	Scaley crystals seperate possessing a pearly lustre	$BOHo$.

Preparation of Solution for Acids.

Great care ought to be bestowed upon the preparation of the solution required for the examination for the acids. On boiling a portion of the substance under examination with CO_3Na_2 nearly all the bases, other than the alkalies, are precipitated in the form of carbonates, basic carbonates, or oxides. The bases have to be separated first, in order to prevent their precipitation by the various reagents employed for the detection of the acids in the wet way, e.g. Ba, Sr, Ca, Pb, on the addition of SO_4Ca'' or SO_4Mg''; Ag, Hg_2'', Pb, by $BaCl_2$ or $CaCl_2$; Fe_2O_3 & others by Am Ho &c. &c.

The precipitate produced by CO_3Na_2 (which ought to be added in the least possible excess) is filtered off & the filtrate tested with a few more drops of CO_3Na_2, if no further ppt. takes place the sol is heated to boiling &

I Dilute HCl added to a portion as long as CO_2 is evolved & till the solution is rendered distinctly acid (on account of small quantities of SO_4Na_2 & NaCl being invariably contained in the commercial CO_3Na_2, it is necessary to test a portion of the HCl solution of the substance prepared, without the intervention of the CO_3Na_2, for SO_4H_2 by means of $BaCl_2$ — A nitric acid solution of the original substance is likewise tested for HCl by adding NO_3Ag.)

II. Another portion of the alkaline filtrate is rendered acid by means of $NO_2 Ho$ & boiled.

III. Another portion by means of dilute $\left\{\begin{matrix} CH_3 \\ CO Ho \end{matrix}\right.$ — & lastly

IV. Another portion of the alkaline solution is carefully rendered <u>neutral</u> by first adding dilute $NO_2 Ho$ drop by drop & boiling as long as CO_2 is evolved & till the sol becomes distinctly acid to test paper, then by adding a few drops of Am Ho, till the solution is neutral to both blue & red litmus paper. It is <u>absolutely necessary</u> that the <u>strictest attention</u>, should be paid to these directions & that ~~there~~ least possible excess of of either acid or alkali should be employed. Since by the formation of an ammonium salt in anything like large quantity, the precipitation of several acids is prevented on account of the formation of a soluable double salt; namely, Ammonic Calcic tartarate, Ammonic Calcic citrate &c. &c. —

If ammonium salts have been formed during the preliminary examination for bases, it is necessary to decompose them by boiling with Na Ho before preparing solution IV.

Ebullition, or fusion with $Co\,Na_2 O_3$ decompose the phosphates of the alkaline earths but very

imperfectly. P_2O_5 in combination with earthy bases however have been already detected, by means of the ammonic molybdate test, during the examination for bases according to Table III, & if P_2O_5 has been found in Group 3.B. without the solution ~~without the~~ giving a precipitate with the group reagents, Am Cl & Am Ho, it proves that some phosphate, other than a metallic, earthy, or alkaline earthy, phosphate is present.

If organic acids have been discovered during the preliminary examination in combination with heavy metals (viz those of the 1st or 2nd groups) SH_2 must be passed through the slightly acidulated (HCl) solution & the ppt filtered off the solution is freed from SH_2 by boiling, then rendered slightly alkaline by means of a few drops of NaHo, & tested by means of CaCl (ammonium salts must be carefully avoided)

No further note need be taken of the following acids, CO_2, SH_2, SO_2, NOHo, ClHo, $\left\{\begin{matrix} CH_3 \\ COHo \end{matrix}\right.$, since they can be detected for certain in the Preliminary Examination, & since they would for the most part be lost by acidulating the solution.

Portion Acidulated with HCl.

SO_2HO_2 on the addition of $BaCl_2$, a white ppt insol on boiling

$SiOHO_2$ on the addition of Am Cl or $CO(AmO)_2$ a gelatinous ppt requires no further confirmation since SiO_2 is left, on evaporating filtrate from Group 2 with NO_2HO

H_4FeCy_6 on the addition of Fe_2Cl_6 a deep blue ppt.

" " " " SO_2Fe" a light blue ppt.

$H_6Fe_2Cy_{12}$ " " " " a deep blue ppt.

" " " " Fe_2Cl_6 a brown coloration only.

If both acids are present, filter off after adding Fe_2Cl_6 & observe the colour of the filtrate. On the addition of $SnCl_2$, or SO_2 to the filtrate a blue ppt is immediately obtained confirmatory of the presence of $H_6Fe_2Cy_{12}$

$CyHS$ On the addition of Fe_2Cl_6 a blood red coloration destroyed by $HgCl_2$ & not by HCl (The red coloration which acetic acid, imparts to a solution of Fe_2Cl_6 is destroyed by HCl.)

H_2SiF_6 On the addition of $BaCl_2$ a gelatinous translucent ppt of Ba"SiF_6 decomposed by strong acids — Confirm by heating the ppt. when SiF_4 & BaF_2 are formed. Am Ho ppts gelatinous $SiOHO_2$ & by Am F + BaF_2 + H_2O are formed.

Portion Acidulated with NO_2HO.

HCl on the addition of NO_2Ago a white curdy ppt soluble in NH_4HO

ClO_2HO gives no ppt with NO_2Ago. From a sol containing HCl & ClO_2HO the former may be removed by means of NO_2Ago. The filtrate containing the chlorate is evaporated to dryness & ignited whereby the chlorate is converted into a chloride which gives a fresh ppt with NO_2Ago.

HBr On the addition of NO_2Ago a dirty white ppt difficultly soluble in $AmHO$. Confirm by adding Cl water to the original solution, & shaking up with ether Br dissolves to a deep red-yellow liquid

BrO_2HO On the addition of NO_2Ago a white amorphous ppt little sol in OH_2 & dilute NO_2HO, easily sol in $AmHO$ $AgBr$ & BrO_2Ago, may be seperated by washing with OH_2. BrO_2Ago being soluble may be detected in the solution, by the addition of SO_2 or SH_2 when a ppt of $AgBr$ is obtained

1. <u>Seperation of HCl from HBr</u>. HCl in presence of HBr is best distinguished by distilling the <u>dry</u> salts with $Cr_2O_3 \cdot KO_2$ & ~~Bot~~ SO_2HO_2

HI. On the addition of NO_2Ago, a yellow ppt scarcely

soluble in Am Ho. Confirm by the blue colour imparted to starch paste, by the iodine liberated by N_2O_3 gas

IO_2Ho On the addition NO_2Ag_2O a white ppt of IO_2Ag_2O insoluble in dilute NO_2Ho; soluble in Am Ho (Ag I insol) whence Ag I is ppted on the addition of SO_2. On adding Acetic acid to a mixture of an Iodide & Iodate the former is instantaniously decomposed. I being set free, which may be confirmed by the delicate Iodide of starch reaction.

2. Seperation of HCl from HI. — HCl in presence of HI cannot be distinguished in the same manner as HCl in the presence of HBr. The two acids are precipitated by NO_2Ag_2O. The ppt is digested with Am Ho & filtered, Yellow Ag I being insoluble in Am Ho. Dilute NO_2Ho ppts white Ag Cl from the filtrate.

3 Seperation of HCl, HBr & HI. — If HCl, HBr & HI are present, ppt HI as CuI_2 by means of SO_2 Cuo & SO_2 Feo or SO_2 filter. Remove Cu & Fe salts by K Ho & examine for HCl & HBr, by evaporating filtrate to dryness & testing one portion for HCl, by Cr_2O_3-KO_2 & SO_2Ho_2 & another for HBr by Cl, water

& ether.

HCy. On the addition of NO_2Ag_2O a white curdy ppt soluble in AmHo & alkaline cyanides. In order to distinguish HCl in the presence of HCy; filter off ppt wash well & ignite in a porcelain crucible at a red heat, then dissolve out the metallic silver left by the decomposition of AgCy with a little hot NO_2Ho_2 & test the clear solution with HCl. — A curdy white ppt indicates HCy. Confirm also by the reaction with Ferroso-ferric salts KHo & HCl, in a fresh portion of the solution.

4. <u>Separation of HCl, HBr, HI, & HCy</u>. If HCl, HBr, HI & HCy are present, test especially for HCy before proceeding to the examination of the other acids, by treating a little of the mixed halogen salts in a small porcelain dish with HCl, causing the evolved HCy to act upon a drop of NO_2Ag_2O or a little SAm2, contained in a second porcelain dish inverted over that containing the mixed salts. A white ppt of AgCy is obtained, or the CyAmo, which is formed gives a blood-red colour with Ferric salts affording equally conclusive proof of the presence of HCy.

CyHS
H_4FeCy_6
$H_6Fe_2Cy_{12}$
} are likewise precipitated on addition of NO_2Ag_2O & are insoluble in dilute NO_2Ho_2 the first & second are insoluble in AmHo

whilst $Ag_6 Fe_2 Cy_{12}$ is readily soluble, these acids are however best detected in the portion acidulated with HCl.

5. No other cyanogen compound but a cyanide gives off HCy when treated with dilute HCl & it is therefore possible also to distinguish HCy in presence of Cy HS (Cy HO) $H_4 Fe Cy_6$ & $H_6 Fe_2 Cy_{12}$.

Portion ~~reduced~~ acidulated with $\left\{\begin{matrix} CH_3 \\ COHo \end{matrix}\right.$

HF On the addition of $CaCl_2$ a white gelatinous ppt. Confirm by ~~acting~~ etching on glass. $SO_2 Ho_2$ $SO_2 Ho_2$ is likewise precipitated by $CaCl_2$, in an acetic acid solution, it is therefore necessary invariably to test specially – by etching on glass – for HF

$\left\{\begin{matrix} COHo \\ COHo \end{matrix}\right.$ On the addition of SO_2 Cao'' a white ppt. $\left\{\begin{matrix} CO \\ CO \end{matrix}\right. Cao''$ is distinguished from CaF_2 by leaving $COCao''$ on ignition, soluble in $\left\{\begin{matrix} CH_3 \\ COHo \end{matrix}\right.$ with effervescence.

Portion rendered Neutral.

$\left\{\begin{matrix} CO\,Ho \\ CH\,Ho \\ CH\,Ho \\ CO\,Ho \end{matrix}\right.$ = $\overline{T}\,Ho_3$

on the addition of $CaCl_2$, a white ppt insoluble in AmCl or cold KHo. — SO_2Ho_2, $\left\{\begin{matrix} CO\,Ho \\ CO\,Ho \end{matrix}\right.$, HJ, $PO\,Ho_3$, $AsO\,Ho_3$, $BO\,Ho$, $SiO\,Ho_2$, CO_2, SO_2 are likewise precipitated by CaCl from a neutral solution of $\overline{T}\,Ho_2$ can obviously be detected only by further special tests, in the presence of any of these acids. The most characteristic reaction consists in warming some $\overline{T}\,Ag_2O_2$ dissolved in a very little dilute AmHo, in a waterbath, until a bright mirror of metallic Ag is obtained.

$\left\{\begin{matrix} CH\,Ho\,(CO\,Ho) \\ CH\,(CO\,Ho) \\ CH_2\,(CO\,Ho) \end{matrix}\right.$ = $\overline{Ci}\,Ho_3$

On the addition of $CaCl_2$, no ppt. till excess of AmHo or $CaHo_2$ has been added & the solution boiled.

The detection of Tartaric acid, & Citric acid indicated in the preliminary Exam, by a strong odour of burnt sugar, requires great care, & strict attention to the few points of distinction between them. The ppt produced by CaCl must be filtered off, & the filtrate tested for Citric acid, by adding a drop of AmHo & boiling. Argentic Citrate dissolved in AmHo forms a mirror only after boiling for ~~some time~~ a long time.

t. Ho On the addition of $CaCl_2$ to a concentrated solution a whitish ppt. soluble in Am Cl. – $BOHo$ is also indicated in the preliminary Exam. by the swelling up of its saline compounds & the precipitation of a concentrated solution of a Borate with concentrated SO_2Ho_2 or HCl. One of the most delicate tests however consists in treating a solution of a Borate, in a watch glass with HCl, till it reacts slightly but distinctly acid, & by immersing a strip of turmeric paper & then drying it at 100° Cent, when the immersed part turns brownish red.

Another most delicate reaction consists in heating a Borate before the blowpipe, with from 3 to 4 times its bulk of a mixture, consisting of 1 part of CaF_2 & 4½ parts of SO_2KoHo. The whole is made into a paste with water, & a little of the moist mass, placed on the loop of a platinum wire & heated in the inner flame. When the outer flame acquires for a moment only a fine green colour; owing to the volatile BF_3 passing through the outer cone of the flame. It is obvious that this reaction may serve equally well for the detection of HF.

$\left\{\begin{matrix} C(C_5H_2)H_2 \\ CO\,HO \end{matrix}\right.$ On the addition of Fe_2Cl_6 a yellowish ppt. Confirm by the reaction given in the preliminary examination. As a ppt of Fe_2Ho_6 is frequently mistaken for ferric succinate, owing to imperfect neutralization.

$\left\{\begin{matrix} CO\,HO \\ C_2H_4 \\ CO\,HO \end{matrix}\right.$ On the addition of Fe_2Cl_6 a reddish brown ppt. Confirm by digesting the ppt with a little Am Ho & filter off. The succinate acid is precipitated from that solution by $BaCl_2$, Alcohol & free Am Ho.

(Distinctive test between $\overline{Bz}\,Ho$ & $\overline{Su}\,Ho_2$)

Gallic & Tannic acid are detected by the addition of Fe_2Cl_6, which produces a black solution. Gelatin precipitates Tannic acid, but not gallic acid.

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