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On Scandium. Part II

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X. *On Scandium.—Part II.*[*]

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Since the publication of the first part of my paper on Scandium,[†] G. Eberhard,[‡] of the Astrophysical Observatory, Potsdam, reasoning from the fact that the strongest lines of the scandium spectrum are observed in the spectra of stars in diverse stages of development, has come to the conclusion that scandium must be universally distributed on the earth. Investigating the arc spectra of 366 minerals and rocks, he obtained the remarkable result that scandium in small quantities is actually one of the most widely distributed earth elements. He shows that it occurs most frequently in zirconium minerals—in beryls, titanates, columbites, and titanocolumbites of the rare earths—also in micas; and, finally, that specimens of wolframite and tinstone from Saxony and Bohemia contain scandium in sufficient quantity to make its extraction advantageous.

These results of Eberhard were quickly verified by Prof. R. J. Meyer,[§] Berlin University, who, in a preliminary paper, has described experiments on extracting scandia from the Zinnwald wolframite. Prof. Meyer concludes that this wolframite contains from 0·14 to 0·16 per cent. of rare earths, and that these rare earths contain 0·30 to 0·33 per cent. of scandia. Thus the original wolframite would contain about $0{\cdot}15/100 \times 0{\cdot}315/100 = 0{\cdot}04725/10000$, or about 0·0005 per cent. of scandia. Prof. Meyer has worked out two methods of separating scandia from the wolframite rare earths:—(*a*) Precipitation with hydrofluoric acid from an intermediate product in which the scandium has been concentrated by an oxalic acid precipitation, and (*b*) precipitation with hydrofluosilicic acid, or sodium silicofluoride in acid solution. The

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[*] The descriptions and analyses of the following salts were communicated to the Society on December 14, 1909, as now printed:—Scandium aurochloride, platinocyanide, iodate, sulphite, malate, malonate, tartrate, racemate, lævotartrate, and mesotartrate.

[†] 'Phil. Trans.,' A, vol. 209, pp. 15–46, March 4, 1908.

[‡] 'Sitzungber. Kgl. Preuss. Akad. Wissensch.,' 1908, xxxviii., 851.

[§] 'Zeit. für Anorg. Chem., lx., 134, November 17, 1908; and 'Chemical News,' xcix., 85, 97, February, 1909.

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raw scandia prepared by the above methods still contains small quantities of yttria and ytterbia, particularly if method (*b*) is used. Prof. MEYER says that his scandia, when freed from cerium and yttrium earths, has the atomic weight 45 to 46, and still contains about 1 per cent. of thorium.*

On reading Prof. MEYER'S paper I examined the felspar and mica which were associated with the parcels of wiikite procured by me from Finland. The felspar contains a small percentage of rare earths, but no trace of scandia could I detect on examining their photographed spectra, the dominant line, wave-length 3613·984, being entirely absent.

The mica was found to contain about 1 per cent. of rare earths, and in the photographed spectrum the dominant lines of scandium are plainly visible. From a comparison of the spectra with those taken with the rare earths from wiikite I should estimate the scandia at about half that in the earths of the best black wiikite.

A specimen of wolframite from Zinnwald was found to contain less than 1 per cent. of rare earths, and in the photographed spectrum of these earths, the dominant lines of scandium were strong, accompanied by only the faintest trace of yttrium or ytterbium lines.

Through the kindness of a friend, Dr. WAHL, of the Mineralogical Institute, University, Helsingfors, I also obtained the following authentic specimens of minerals from the locality whence my wiikite was mined:—

No. 1. Felspar from the pegmatite vein carrying monazite and the scandium-wiikite. Lokasaari.
No. 2. Monazite from Lokasaari.
No. 3. Black wiikite from Lokasaari, Impilaks.
No. 4. Brown wiikite from Lokasaari.
No. 5. Wiikite from Lokasaari, another specimen.
No. 6. Mica from Hunttila, Impilaks. (Probably from the wiikite quarry.)

Of Dr. WAHL'S minerals, No. 1, felspar, contained 1·11 per cent. of rare earths. On photographing the spectrum only a very faint trace of scandium appeared.

No. 2, monazite, contained 17·15 per cent. of rare earths; the photographed spectrum showed no scandium.

No. 3, black wiikite, contained 22·58 per cent. of rare earths; the spectrum showed considerable quantities of scandium.

No. 4, brown wiikite, contained 23·77 per cent. of rare earths; the spectrograph showed only a small amount of scandium.

No. 5, wiikite, contained 24·85 per cent. of rare earths; the photographed spectrum showed considerable quantities of scandium.

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* A mixture of 99·25 parts of scandium with 0·75 part of thorium would have an atomic weight of about 45·5.

No. 6, mica, contained 0·63 per cent. of rare earths, which, on spectrographic examination, contained an amount of scandium about equal to that in the rare earths in Nos. 3 and 5.

I have also to thank Dr. A. Hiorth, of Kristiania, for a typical collection of 23 specimens of rare minerals from Norway; the results of examination of most of these specimens have already been given.

The scandia used in the preparation of some of the salts here described was not absolutely pure. Chemically, no other earth could be detected in it, but the spectrograph revealed traces of yttria and ytterbia. These traces could have been removed by one or more operations, but I thought it advisable to leave them in, for the following reasons: In each operation of purification some loss is unavoidably incurred, and when chemical reactions are insufficient to find the other earths it is not worth diminishing my lessening stock of scandia for the sake of academic purity. The chief reason, however, for leaving these traces in is that they might afford evidence of a difference of behaviour between one earth and another in the presence of some of the acids used. After each quantitative determination the scandia was dissolved in acid and a spectrogram taken to see if yttria or ytterbia were present. The residual earth was then collected from the mother-liquor and a photograph taken of its spectrum. A comparison of the pair of spectra shows at once if any separation has been effected between the earths present. When separation is apparent further experimentation on a larger scale is reserved to a future occasion.

Scandium Iodate,

$Sc(IO_3)_3+18H_2O$.

Scandium iodate is best prepared by the metathesis of a soluble scandium salt with ammonium iodate. It forms a white crystalline powder almost insoluble in water. After being well washed and dried in the air it contains 18 molecules of water. The water is held loosely; long drying in a desiccator, or exposure to a temperature of 100° and slightly higher, drives some of it off; only by repeated trials with slight alterations of temperature have I obtained certain definite hydrates. There are indications of other hydrates, but I have not attempted to prepare them.

Analysis.

(1) 15·483 gr. of 18-hydrate scandium iodate, dried in the air at the temperature of about 15°, were dissolved in hydrochloric acid and heated until chlorine ceased to be evolved. It was then evaporated to dryness with excess of acid and dissolved in water. Ammonia in excess was added, and the whole boiled. The precipitated scandia was filtered, washed, and ignited. It weighed 1·229 gr., = 0·7959 gr. of scandium.

(2) 17·956 gr. of 18-hydrate were heated carefully until the temperature rose to 250°. At this point the white salt became slightly brown. It weighed 11·265 gr., showing a loss of 6·691 gr. of water.

	Theory.		Experiment.	
			(1).	(2).
Sc	44·10	4·94	5·14	62·74
$3(IO_3)$ . . .	524·76	58·75	94·86	
$18H_2O$ . . .	324·29	36·31		37·26
	893·15	100·00	100·00	100·00

Scandium iodate loses three molecules of water when kept for some time in a desiccator, and leaves a hydrate containing 15 molecules of water,

$$Sc(IO_3)_3, + 15H_2O.$$

15·415 gr. of 18-hydrate scandium iodate were dried for some time over sulphuric acid. The 15-hydrate into which it had changed was dissolved in hydrochloric acid and precipitated with ammonia. After ignition the scandia weighed 1·261 gr., = 0·8166 gr. of scandium.

	Theory.		Experiment.
Sc	44·10	5·26	5·30
$3(IO_3)$	524·76	62·54	94·70
$15H_2O$	270·24	32·20	
	839·10	100·00	100·00

When the 18-hydrate scandium iodate is dried for several hours in a vacuum desiccator over sulphuric acid, it loses five molecules of water and leaves a 13-hydrate,

$$Sc(IO_3)_3, + 13H_2O.$$

(1) 7·684 gr. of 13-hydrate scandium iodate, prepared as described, were fused with excess of potassium bisulphate; iodine was evolved, and when the whole was in quiet fusion it was allowed to cool. The melt was boiled out with water, an excess of ammonia added, and the resulting scandia separated. It weighed 0·643 gr., = 0·4164 gr. of scandium.

(2) 5·537 gr. of 13-hydrate scandium iodate were dissolved in acid, precipitated by ammonia as already described, and the resulting scandia collected. It weighed 0·463 gr., = 0·2998 gr. of scandium.

(3) 18·104 gr. of 13-hydrate scandium iodate, dried at 250°, lost 5·205 gr. of water.

	Theory.		Experiment. (1).	Experiment. (2).	Experiment. (3).
Sc	44·10	5·49	5·42	5·41	71·25
$3(IO_3)$ . .	524·76	65·35	94·58	94·59	
$13H_2O$ . .	234·21	29·16			28·75
	803·07	100·00	100·00	100·00	100·00

When the 18-hydrate is heated for some time to 100° it loses eight molecules of water, and leaves a 10-hydrate,

$$Sc(IO_3)_3+10H_2O.$$

20·983 gr. of 10-hydrate scandium iodate, decomposed in the manner already described, yielded 1·907 gr. of scandia, = 1·235 gr. of scandium.

	Theory.		Experiment.
Sc	44·10	5·89	5·88
$3(IO_3)$	524·76	70·06	94·12
$10H_2O$	180·16	24·05	
	749·02	100·00	100·00

When heated to about 250° scandium iodate loses all its water of crystallisation, and becomes anhydrous,

$$Sc(IO_3)_3.$$

15·409 gr. of anhydrous scandium iodate were dissolved in acid, precipitated with ammonia, and the scandia collected. It weighed 1·724 gr., = 1·1116 gr. of scandium.

	Theory.		Experiment.
Sc	44·10	7·75	7·24
$3(IO_3)$	524·76	92·25	92·76
	568·86	100·00	100·00

SCANDIUM SULPHITE,

$$Sc_2(SO_3)_3.$$

Scandium sulphite is formed by mixing solutions of scandium chloride and sodium sulphite. It forms a heavy white precipitate, soluble in excess of sodium sulphite when heated, slightly soluble in hot water and insoluble in cold water. The air dried salt is anhydrous, and has the composition given above.

Analysis.

4·236 gr. of the air-dried salt were dissolved in hydrochloric acid and the solution boiled until sulphurous acid ceased to be driven off. Ammonia in excess was added, and the whole boiled. The scandia was filtered, washed, and ignited. It weighed 1·765 gr. = 1·1430 gr. of scandium.

	Theory.		Experiment.
$2Sc$	88·20	26·86	26·98
$3SO_3$	240·21	73·14	73·02
	328·41	100·00	100·00

SCANDIUM BORATE,

$ScBO_3$.

Scandium hydroxide ground with a solution of boric acid undergoes no apparent change. Slight combination takes place on boiling the mixture, but no definite compound is produced.

Re-crystallised boric acid and scandia were mixed together, the acid being in excess, and the whole heated to bright redness for an hour in a platinum crucible. The mixture fused to a clear liquid, and on cooling the melt was extracted with boiling water, filtered, and washed. The residue on the filter was shaken up with much water, levigated, and poured on to a filter, and thus was separated, until only the larger heavy particles were left behind. The scandium borate thus prepared is in the form of a white powder, having the appearance under the microscope of minute transparent spheres. In transmitted polarised light each spherulite shines like a point of light on the dark field when the analyser is crossed, each point alternately disappearing and reappearing as the stage is rotated.

The analysis of the borate at first presented difficulties. It dissolves easily in dilute acids, and on adding ammonia a precipitate, chiefly of scandium hydroxide, is formed, but the hydroxide carries down with it variable quantities of borate. The plan finally adopted was to dissolve the borate in dilute hydrochloric acid and precipitate hot with ammonia. The precipitate was filtered, washed, and dried. The precipitated hydroxide was then ignited in a platinum crucible, hydrofluoric acid was poured on, and it was kept at a gentle heat for some hours. Strong sulphuric acid was added, and the mixture was gradually heated till dry. The residual scandia was then ignited strongly and weighed.

Analysis.

8·037 gr. of scandium borate, treated in the manner described, gave 5·277 gr. of scandia, = 3·4173 gr. of scandium.

	Theory.		Experiment.
Sc	44·1	42·77	42·52
BO_3	59·0	57·23	57·48
	103·1	100·00	100·00

SCANDIUM AUROCHLORIDE,

$3ScCl_3,2AuCl_3,21H_2O$.

Scandium aurochloride is prepared by mixing strong solutions of the component chlorides, and allowing the mixture to evaporate slowly over sulphuric acid in a vacuum desiccator. The double salt separates out in a felt-like mass of yellow needle-shaped crystals, which are very deliquescent. In this state the salt has the composition given above. The composition is unusual, but the analytical results agree very well with it, and I have not succeeded in preparing a salt with different proportions of the chief constituents. The water of crystallisation can be driven off gradually by careful heating.

For analysis the felt-like mass of aurochloride was made as dry as possible by pressing it several times between warm dry filtering paper until the paper was unsoiled.

Analysis.

26·717 gr. of scandium aurochloride, dried by pressure, were heated in an air-bath to 100°, and kept at that temperature for three hours. The aurochloride melted to a clear orange-coloured liquid, solidifying on cooling to a crystalline mass. The loss of weight after liquefaction was found to be 4·350 gr.

After keeping the salt for several days at 100° the liquid gradually becomes solid and crystalline, and in this state is anhydrous. The further loss of weight was 2·672 gr., equal to a total loss of 7·022 gr.

The residue from which the water had been driven was dissolved in hot water acidulated with hydrochloric acid, and sodium bisulphite was added. Gold commenced to separate at once, and on boiling the metal collected in a coherent sponge. It was filtered, washed with dilute acid, and weighed after ignition.

After separating the metallic gold, the filtrate was mixed with excess of ammonia and boiled. The precipitated scandia was filtered, washed, ignited, and weighed.

The 26·717 gr. of hydrated aurochloride treated in this manner yielded 7·361 gr. of gold, and 3·862 gr. of scandia, = 2·5009 gr. of scandium.

	Theory.			Experiment.	
3Sc	132·30	9·21		9·36	
2Au	394·40	27·45		27·55	
15Cl	531·90	37·01		36·81	
$13H_2O$ . . .	234·21	16·30	26·33	16·28	26·28
$8H_2O$ . . .	144·13	10·03		10·00	
	1436·94	100·00		100·00	

2-Hydrate Scandium Aurochloride,

$3ScCl_3,2AuCl_3,2H_2O$.

During the heating at 100° there were strong indications of an arrest of drying when the salt had been heated for about six days, its weight then being 20·355 gr. The following analysis shows that at this stage the salt was a di-hydrate.

Analysis.

20·355 gr. of di-hydrate scandium aurochloride, when further heated to 100° until it was anhydrous, lost 0·660 gr. of water. The residue, treated as described, gave 7·361 gr. of gold and 3·862 gr. of scandia, = 2·5009 gr. of scandium

	Theory.		Experiment.
3Sc	132·30	12·09	12·29
2Au	394·40	36·03	36·16
15Cl	531·90	48·59	47·62
$2H_2O$	36·03	3·29	3·73
	1094·63	100·00	100·00

8-Hydrate Scandium Aurochloride,

$3ScCl_3,2AuCl_3,8H_2O$.

Scandium aurochloride with eight molecules of water is obtained by drying in a desiccator over sulphuric acid the 21-hydrate. After about a fortnight's drying the weight, which at first rapidly diminishes, becomes constant.

For analysis the hydrate, dried to a constant weight, was dissolved in water and the gold precipitated by the addition of metallic magnesium, the liquid being well boiled. The filtrate from the gold was mixed with an excess of ammonium chloride, ammonia was added, and the whole boiled. The scandia, which came down free from magnesia, was filtered, washed, dried, and ignited.

Analysis.

19·302 gr. treated as described yielded 6·341 gr. of gold and 3·174 gr. of scandia, = 2·0554 gr. of scandium.

	Theory.			Experiment.
$3Sc$	132·30	11·00		10·65
$2Au$	394·40	32·79		32·85
$15Cl$	531·90	44·23	56·21	56·50
$8H_2O$	144·13	11·98		
	1202·73	100·00		100·00

Anhydrous Scandium Aurochloride,

$3ScCl_3,2AuCl_3$.

The weight of the anhydrous salt obtained by continued drying of 26·717 gr. of the 21-hydrate at 100° was 19·695. This amount of anhydrous scandium aurochloride gave 7·361 gr. of gold and 3·862 gr. of scandia, = 2·5009 gr. of scandium.

	Theory.		Experiment.
$3Sc$	132·30	12·50	12·70
$2Au$	394·40	37·26	37·37
$15Cl$	531·90	50·24	49·93
	1058·60	100·00	100·00

Scandium Platinocyanide,

$Sc_2[Pt(CN)_4]_3+21H_2O$.

Scandium platinocyanide is formed by the metathesis in the cold of scandium sulphate and barium platinocyanide.

The barium sulphate filtered off, the clear liquid is evaporated at a moderate temperature. Scandium platinocyanide crystallises out in large monoclinic prisms on a rhombic base, the angles of which are 81° 20′ and 98° 40′. The crystals frequently group themselves in rosettes. They are dichroic, crimson by transmitted light, and a rich metallic green by reflected light. The reflected and transmitted rays are oppositely polarised. The red transmitted ray is extinguished when the short diagonal of the nicol is parallel to the long axis of the crystal, and the reflected green ray is extinguished when the long diagonal of the nicol is parallel to the long axis of the crystal. Light passing along the axis of the crystal is crimson, and not polarised.

[*June 2nd*, 1910.—I have submitted the metallic green crystals of this salt to the low temperature of liquid air with a view to ascertain what change of colour, if any, accompanies the reduction of temperature. After an exposure for ten minutes in liquid air the green colour becomes paler and more of a yellowish hue, while the transmitted light changes from crimson to deep brown. On removing the platinocyanide from the liquid air the normal colours come back as the salt warms up to the temperature of the laboratory.]

The crystals are soluble in water, forming a colourless solution, and are insoluble or nearly so in absolute alcohol. Boiled in alcohol they dehydrate, turning white but not dissolving. The white dehydrated salt dissolves easily in water, and on evaporation separates out in crystals of the normal appearance.

Analysis.

(1) 5·755 gr. of well crystallised and air-dried scandium platinocyanide were dissolved in water and precipitated hot with ammonia. The scandia was filtered off, well washed, dried, ignited, and weighed. It weighed 0·627 gr., = 0·406 gr. of scandium. The filtrate was evaporated to dryness in platinum, and the residual ammonium platinocyanide slowly decomposed by heat and then ignited strongly. The platinum left from the decomposition weighed 2·446 gr.

(2) 9·612 gr. of scandium platinocyanide were fused at a full red heat with potassium bisulphate. Hydrocyanic acid was evolved with much intumescence and separation of platinum, and the mixture finally fused quietly to a clear liquid. The mass was extracted with water containing a little acid, and the platinum filtered off; it weighed 4·028 gr.

Ammonia in excess was added to the filtrate and the precipitated scandia collected; it weighed 1·132 gr., = 0·733 gr. of scandium.

(3) 10·741 gr. of air-dried scandium platinocyanide were kept over sulphuric acid in a desiccator for 80 hours. It lost 2·121 gr. of water. The salt was then put into a hot-air oven and kept at 100° for 17 hours. It became a good canary-yellow, and was found to have lost an additional 0·1343 gr. The temperature was raised to 160° for 10 hours, when an additional 0·644 gr. of water was driven off. The temperature was finally raised to about 180°, when the salt began to decompose. Calculated out, these losses nearly approximate to successive losses of 15, 1, and $4\frac{1}{2}$ molecules of water, with half a molecule still remaining in the salt at 160°, and only driven off as decomposition commences.

	Theory.			Experiment.	
				(1).	(2).
2Sc . .	88·200	6·47	49·37	7·05	49·53
3Pt . .	585·000	42·90		42·50	
12CN .	312·120	22·89	50·63	50·45	50·47
$15H_2O$.	270·240	19·82			
$1H_2O$.	18·016	1·32			
$4\frac{1}{2}H_2O$.	81·072	5·94			
$\frac{1}{2}H_2O$ . .	9·008	0·66			
	1363·656	100·00	100·00	100·00	100·00

	Theory.	Experiment. (3).
$Sc_2[Pt(CN)_4]_3$	72·26	72·26
$15H_2O$	19·82	19·75
$1H_2O$	1·32	1·25
$4\frac{1}{2}H_2O$	5·94	5·99
$\frac{1}{2}H_2O$	0·66	0·75
	100·00	100·00

Normal scandium platinocyanide is not quite stable when its solution is heated and evaporated. During re-crystallisation the solution, at first clear and colourless, deposits a yellow powder and gives an odour of hydrocyanic acid. Analyses of the yellow deposit have not yet given me concordant results; roughly it contains 23·5 per cent. of scandium and 6 per cent. of platinum. The analytical results with the normal platinocyanide given above show an excess of scandium and a deficit of platinum. This may be the result of slight decomposition during re-crystallisation, with production of a trace of the yellow compound.

Scandium Mono-chloroacetate,

$(CH_2ClCOO)_2ScOH+2H_2O$.

Scandium hydroxide, ground and well shaken with a cold aqueous solution of monochloracetic acid, dissolves slowly and almost completely. The clear filtered solution, evaporated in the cold over sulphuric acid, deposits a crystalline crust, appearing under the microscope as flower-like groups of stellate crystals, and showing good colour when examined by polarised light. These crystals are permanent in dry air, and lose no water at 100°. On being heated they decompose quietly, and at a red heat leave a white residue of scandia.

Analysis.

(1) 9·003 gr. of scandium mono-chloroacetate left on ignition 2·193 gr. of scandia, = 1·4202 gr. of scandium.

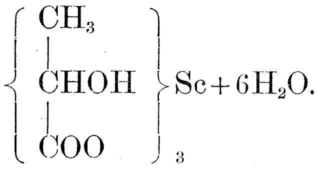
(2) 15·145 gr. of mono-chloroacetate left on ignition 3·634 gr. of scandia, = 2·3530 gr. of scandium.

(3) 14·099 gr. of mono-chloroacetate left on ignition 3·387 gr. of scandia, = 2·1934 gr. of scandium.

	Theory.			Experiment.		
				(1).	(2).	(3).
Sc . .	44·10		15·52	15·77	15·54	15·55
C_4 . .	48·00	239·99	84·48	84·23	84·46	84·45
H_5 . .	5·04					
Cl_2 . .	70·92					
O_5 . .	80·00					
$2H_2O$.	36·03					
	284·09		100·00	100·00	100·00	100·00

The scandia from these analyses showed when spectrographed no trace of extraneous lines. The earth from the filtrate showed a little yttrium and ytterbium.

Scandium Lactate,



$$\left\{\begin{matrix} CH_3 \\ | \\ CHOH \\ | \\ COO \end{matrix}\right\}_3 Sc + 6H_2O.$$

On mixing hot solutions of scandium nitrate and ammonium lactate, the scandium salt on cooling separates in white feathery crystals, slightly soluble in cold, and more soluble in hot water. It is easily soluble in ammonia; on evaporating the ammoniacal solution crystals are deposited which are double refracting, showing colours in polarised light. Dried over sulphuric acid the crystals of scandium lactate contain six molecules of water, four of which are driven off at 110°.

Analysis.

(1) 4·571 gr. of the 6-hydrate scandium lactate left on ignition 0·747 gr. of scandia, = 0·4837 gr. of scandium.

(2) 3·379 gr. of the 6-hydrate scandium lactate left on ignition 0·561 gr. of scandia, = 0·3633 gr. of scandium.

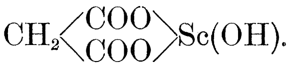
	Theory.		Experiment.	
			(1).	(2).
Sc	44·10	10·52	10·58	10·75
$C_9H_{15}O_9$ . .	267·12	89·48	89·42	89·25
$6H_2O$ . . .	108·10			
	419·32	100·00	100·00	100·00

3·863 gr. of scandium lactate, dried at 120°, left on ignition 0·747 gr. of scandia, = 0·4837 gr. of scandium.

	Theory.		Experiment.
Sc	44·10	12·70	12·52
$C_9H_{15}O_9$	267·12	87·30	87·48
$2H_2O$	36·03		
	347·25	100·00	100·00

The scandium nitrate used in the preparation of the lactate contained traces of yttrium and ytterbium. The scandia resulting from the ignition of the lactate showed no appreciable diminution in the amount of impurity present. The mother-liquors, however, appeared free from both yttrium and ytterbium. The impurities in the lactate could only be detected spectroscopically, and were not present in sufficient quantities to interfere with the properties of the lactate, or to increase perceptibly the atomic weight of the scandium.

Scandium Malonate,



$$CH_2 \begin{matrix} \diagup COO \diagdown \\ \diagdown COO \diagup \end{matrix} Sc(OH).$$

Scandium hydroxide dissolves readily in a cold aqueous solution of malonic acid, and the solution when heated deposits a semi-transparent granular precipitate, having no crystalline appearance under the microscope. This precipitate partially dissolves on cooling.

If this solution is boiled for some time a dense precipitate is formed, which on cooling does not re-dissolve.

When a cold solution of scandium malonate containing excess of malonic acid is slowly heated on a water-bath, it becomes cloudy at first; as the temperature rises the liquid becomes clear, and a thick transparent gummy liquid sinks to the bottom. If now the supernatant liquor is poured off and cold water poured on, the transparent colloid-looking mass is slowly converted into a fine white crystalline powder.

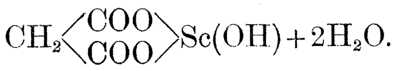
The colloidal gummy mass, smeared on a microscopic slide, dries to a clear non-deliquescent glass-like substance. Exposed to dry air the film cracks and flakes off like a pharmaceutical scale preparation.

A lump of the viscid transparent mass, free from adhering water, dried in a water-oven at 100°, becomes white, hard, and brittle. It is now insoluble in either hot or cold water, although before heating the gummy mass is easily soluble in cold water.

The side of the beaker in which the gummy mass is precipitated by heat from the solution of the malonate is seen to be studded with brilliant colourless transparent crystals, some measuring more than a millimetre across.

According to the mode of preparation, scandium malonate contains either one or two molecules of water.

DI-HYDRATE SCANDIUM MALONATE,



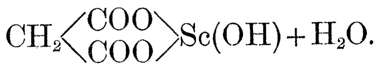
$$CH_2\left\langle\begin{matrix}COO\\COO\end{matrix}\right\rangle Sc(OH)+2H_2O.$$

(1) 35·331 gr. of di-hydrate scandium malonate left after ignition 12·282 gr. of scandia, = 7·9535 gr. of scandium.

(2) 52·299 gr. of scandium malonate, dried at 120°, lost 9·538 gr. of water.

	Theory.		Experiment.	
			(1).	(2).
Sc . . .	44·100	22·14	22·51	81·76
$C_3H_3O_5$ . .	119·024	59·77	77·49	
$2H_2O$ . .	36·032	18·09		18·24
	199·156	100·00	100·00	100·00

MONO-HYDRATE SCANDIUM MALONATE,



$$CH_2\left\langle\begin{matrix}COO\\COO\end{matrix}\right\rangle Sc(OH)+H_2O.$$

Analysis.

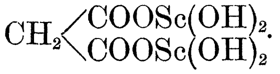
(1) 8·039 gr. of scandium malonate left on ignition 2·984 gr. of scandia, = 1·9324 gr. of scandium.

(2) 7·876 gr. of scandium malonate left on ignition 2·958 gr. of scandia, = 1·9155 gr. of scandium.

(3) 28·281 gr. of scandium malonate left on ignition 10·537 gr. of scandia, = 6·8235 gr. of scandium.

	Theory.		Experiment.		
			(1).	(2).	(3).
Sc	44·100	24·35	24·04	24·32	24·13
$C_3H_3O_5$ . . .	119·024	75·65	75·96	75·68	75·87
H_2O	18·016				
	181·140	100·00	100·00	100·00	100·00

Basic Scandium Malonate,



$$\mathrm{CH_2}\Big\langle\begin{matrix}\mathrm{COOSc(OH)_2}\\ \mathrm{COOSc(OH)_2}\end{matrix}.$$

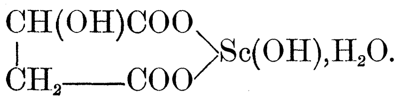
When a large excess of scandium hydroxide is gently warmed with an amount of malonic acid insufficient to dissolve it, and the filtered solution is well boiled, a basic salt is precipitated having the above composition.

Analysis.

16·754 gr. of the salt left on ignition 8·834 gr. of scandia, = 5·7207 gr. of scandium.

	Theory.		Experiment.
Sc_2	88·200	34·15	34·15
$C_3H_6O_8$	170·048	65·85	65·85
	258·248	100·00	100·00

Scandium Malate,



$$\left.\begin{matrix}\mathrm{CH(OH)COO}\\ |\qquad\qquad\\ \mathrm{CH_2{-}\!\!{-}\!\!{-}COO}\end{matrix}\right\rangle\mathrm{Sc(OH),H_2O}.$$

Malic acid and scandium hydroxide rubbed together with a little water react, and the liquid when heated becomes clear. When gradually cooled a precipitate appears, and at the ordinary temperature of the laboratory the solution is opaque and almost solid. The precipitate consists of very fine granules, having no crystalline appearance under the microscope; it re-dissolves on being heated.

Scandium malate formed in this manner is a granular white powder, soluble in hot and difficultly soluble in cold water. It is easily soluble in ammonia, and is not precipitated from the ammoniacal solution by dilute acetic or malic acid; neither is it precipitated by ammonium sulphide. The aqueous solution dries to a transparent gummy mass when evaporated to dryness. It has a strong mousy odour. The ammoniacal solution also dries to a clear glassy mass, entirely soluble in water.

Analysis.

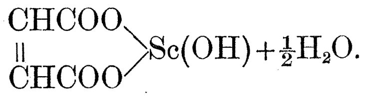
(1) 10·131 gr. of scandium malate dried in a vacuum desiccator lost 0·852 gr. of water after being kept at 110° for twelve hours.

The same 10·131 gr. of scandium malate, after ignition, left 3·324 gr. of scandia, = 2·152 gr. of scandium.

(2) 8·338 gr. of scandium malate, dried in a vacuum desiccator, left on ignition 2·667 gr. of scandia, = 1·727 gr. of scandium.

	Theory.		Experiment.	
			(1).	(2).
Sc	44·100	20·89	21·24	20·71
$C_4H_5O_6$ . . .	149·040	70·58	70·35	79·29
H_2O	18·016	8·53	8·41	
	211·156	100·00	100·00	100·00

Scandium Fumarate,



$$\left.\begin{matrix}\mathrm{CHCOO}\\ \| \\ \mathrm{CHCOO}\end{matrix}\right\rangle\mathrm{Sc(OH)}+\tfrac{1}{2}\mathrm{H_2O}.$$

Fumaric acid is neutralised with dilute ammonia, and to the warm solution is added an equivalent quantity of scandium sulphate. A white precipitate comes down, which partially re-dissolves on cooling. Scandium fumarate, therefore, like so many other scandium salts, is more soluble in cold than in hot water. The liquid is well boiled, kept for some time in a hot-water oven till the precipitate settles, then quickly filtered and washed with boiling water. After drying in a desiccator at the ordinary temperature, it is powdered and again exposed to dry air. Scandium fumarate is a white powder, appearing under the microscope as very minute spheres, slightly soluble in hot, more so in cold water, and easily soluble in ammonia. When heated to redness in the air it decomposes quietly, leaving, after the carbon has burnt off, pure white scandia.

The salt is difficult to get to a constant weight; it had to be heated to 105° in a hot-air bath for many days, and the weight taken at intervals of five or six hours. At the end of a fortnight it was still slightly losing weight. I am therefore inclined to think that the half molecule of water may be due to water of crystallisation not entirely driven off at 105°.

Analysis.

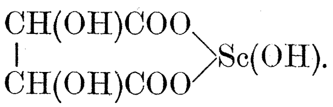
(1) 6·254 gr. of scandium fumarate, heated to redness in the air, left 2·287 gr. of scandia, = 1·4810 gr. of scandium.

(2) 8·245 gr. of scandium fumarate left on ignition 3·055 gr. of scandia, = 1·9783 gr. of scandium.

	Theory.		Experiment.	
			(1).	(2).
Sc	44·10	23·95	23·68	23·99
$C_4H_3O_5$	131·02	76·05	76·32	76·01
$\frac{1}{2}H_2O$	9·01			
	184·13	100·00	100·00	100·00

The scandia obtained in these analyses was tested in the spectrograph, and a minute trace of yttrium and ytterbium lines was just detectable. The earth from the mother-liquor showed the yttrium lines faintly and ytterbium lines strongly. Further experiments are in progress with fumaric acid as a separating agent. METZGER found this acid very effectual in separating thoria from other monazite earths; the present experiments give hopes that it may be useful in separating scandium from ytterbium.

SCANDIUM TARTRATE,



$$\left.\begin{array}{l}\mathrm{CH(OH)COO} \\ | \\ \mathrm{CH(OH)COO}\end{array}\right\rangle \mathrm{Sc(OH)}.$$

Scandium hydroxide is added to a strong solution of tartaric acid with slight warming until the greater part of the scandium hydroxide is dissolved; care must be taken to keep the base in slight excess. The turbid solution is filtered and boiled, when a granular precipitate comes down. This precipitate is well washed with boiling water and dried in a desiccator over sulphuric acid.

In this way scandium tartrate forms a white crystalline powder, insoluble in hot, slightly soluble in cold water, and insoluble in alcohol. Dried in a desiccator it is anhydrous. When heated it emits the empyreumatic odour peculiar to decomposing tartrates and leaves a white residue of scandia.

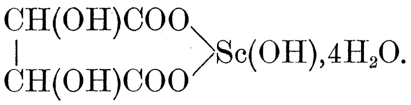
Analysis.

(1) 12·149 gr. of scandium tartrate left on ignition 4·027 gr. of scandia, = 2·6078 gr. of scandium.

(2) 3·900 gr. of scandium tartrate left on ignition 1·267 gr. of scandia, = 0·8205 gr. of scandium.

	Theory.		Experiment. (1).	Experiment. (2).
Sc	44·10	21·09	21·47	21·04
$C_4H_5O_7$ . .	165·04	78·91	78·53	78·96
	209·14	100·00	100·00	100·00

SCANDIUM RACEMATE,



$$\left.\begin{array}{l}\mathrm{CH(OH)COO}\\ |\\ \mathrm{CH(OH)COO}\end{array}\right\rangle \mathrm{Sc(OH),4H_2O.}$$

Scandium racemate is prepared in a similar manner to the tartrate. The two salts, however, are not quite similar. The racemate comes down less plentifully on boiling, and it is not anhydrous as is the tartrate, but contains four molecules of water, one of which is driven off at 115°.

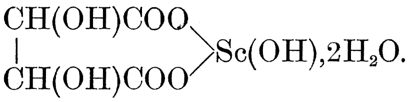
Equal weights of tartaric and racemic acids were dissolved in separate bottles of water, and equal quantities of scandium hydroxide added to each bottle, the amount being chosen so that all dissolved except a small quantity. The two bottles were shaken in the cold for half an hour and filtered. The clear solutions were well boiled. A precipitate was formed in each, but that in the tartrate was double that in the racemate.

Analysis.

4·835 gr. of scandium racemate, heated to a temperature of 115° in a hot-air bath, lost 0·322 gr. of water and left on ignition 1·168 gr. of scandia, = 0·7564 gr. of scandium.

	Theory.		Experiment.
Sc.	44·100	15·68	15·64
$C_4H_5O_7$	165·040	58·69	77·70
$3H_2O$	54·048	19·22	
H_2O	18·016	6·41	6·66
	281·204	100·00	100·00

SCANDIUM LÆVO-TARTRATE.



$$\left.\begin{array}{l}\mathrm{CH(OH)COO}\\ |\\ \mathrm{CH(OH)COO}\end{array}\right\rangle \mathrm{Sc(OH),2H_2O.}$$

Scandium lævo-tartrate is prepared in a similar way to the tartrate. Dried in a desiccator over sulphuric acid it contains one molecule of water. Dried in the air it contains two molecules of water.

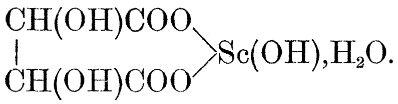
Analysis.

(1) 6·252 gr. of scandium lævo-tartrate, dried in a desiccator for twenty-four hours, left on ignition 1·785 gr. of scandia, = 1·1564 gr. of scandium.

(2) 7·912 gr. of scandium lævo-tartrate, dried in a desiccator, left on ignition 2·250 gr. of scandia, = 1·4570 gr. of scandium.

	Theory.		Experiment.	
			(1).	(2).
Sc.	44·100	17·99	18·50	18·42
$C_4H_5O_7$ . .	165·040	67·31	81·50	81·58
$2H_2O$ . .	36·032	14·70		
	245·172	100·00	100·00	100·00

Scandium Meso-tartrate.



$$\begin{array}{l} CH(OH)COO \\ | \\ CH(OH)COO \end{array} > Sc(OH),H_2O.$$

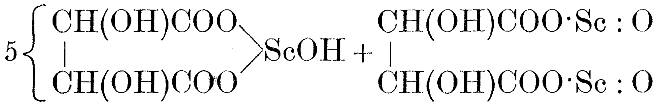
Scandium meso-tartrate is prepared by gently warming a slight excess of scandium hydroxide with aqueous meso-tartaric acid, and then boiling the filtered solution. The meso-tartrate comes down as a white granular precipitate. It is well washed with boiling water and dried over sulphuric acid in a desiccator for twenty-four hours. In this state it contains one molecule of water.

Analysis.

4·899 gr. of scandium meso-tartrate yielded on ignition 1·460 gr. of scandia, = 0·9455 gr. of scandium.

	Theory.		Experiment.
Sc.	44·100	19·41	19·30
$C_4H_5O_7$	165·040	80·59	80·70
H_2O	18·016		
	227·156	100·00	100·00

The analysis of the compounds of scandium with tartaric acid and its isomers has been complicated by the tendency to form basic salts admitting of no simple formulæ. Thus, both the tartrate and lævo-tartrate occasionally form salts containing percentages of scandium ranging near to 23·5, with a deviation of two- or three-tenths of a unit one side or the other. The nearest basic salt which contains such an amount of scandium has the composition



$$5\left\{\begin{array}{l}\mathrm{CH(OH)COO}\\ |\\ \mathrm{CH(OH)COO}\end{array}\right\rangle\mathrm{ScOH} + \begin{array}{l}\mathrm{CH(OH)COO{\cdot}Sc:O}\\ |\\ \mathrm{CH(OH)COO{\cdot}Sc:O}\end{array}$$

and contains 23·49 per cent. of scandium.

Scandium $\alpha\beta$-Dibromopropionate,

$$(\mathrm{CH_2BrCHBrCOO})_3\mathrm{Sc} + (\mathrm{CH_2BrCHBrCOO})_2\mathrm{ScOH}.$$

I have not prepared either the normal or the basic dibromopropionate in a pure state, but I have obtained a salt corresponding to equimolecular proportions of the two salts, having the above composition. The acid was dissolved in water and exactly neutralised with dilute ammonia. To the warm solution was added dilute scandium nitrate until no more precipitate came down on the addition of either reagent to the filtered solution. Scandium dibromopropionate is almost insoluble in hot or cold water and, when dried over sulphuric acid in a desiccator, is a white amorphous powder, so easily electrified that it requires great care in powdering and moving it from one vessel to another or it flies out of the mortar or watch-glasses.

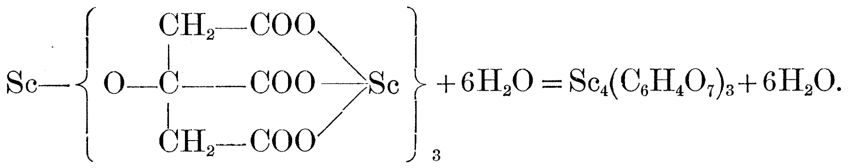
Analysis.

8·117 gr. of scandium dibromopropionate, dried at 110°, yielded on ignition 0·845 gr. of scandia, = 0·5598 gr. of scandium.

	Theory.		Experiment.
Sc_2	88·20	7·00	6·90
$C_{15}H_{16}O_{11}Br_{10}$ . . .	1171·33	93·00	93·10
	1259·53	100·00	100·00

The filtrate and washings from the precipitated salt were concentrated and precipitated with ammonia. The precipitate was dissolved in nitric acid and examined in the spectrograph, whilst a corresponding test was applied to the scandia from the pure analysed salt. On comparing the two spectrograms, nothing but scandium lines could be detected on that of the scandia from the dibromopropionate, while on that of the earth from the filtrate, yttrium could be detected in minute quantity and ytterbium in larger quantities.

Scandium Citrate,



$$\mathrm{Sc}-\left\{\begin{array}{l}\mathrm{CH_2-COO} \\ \mathrm{O-C-COO \rightarrow Sc} \\ \mathrm{CH_2-COO}\end{array}\right\}_3 + 6\mathrm{H_2O} = \mathrm{Sc_4(C_6H_4O_7)_3} + 6\mathrm{H_2O}.$$

Ammonium citrate is added to a solution of scandium nitrate in such proportion that no further precipitation takes place on adding either solution. The result is a dense white precipitate, soluble in excess of ammonium citrate solution.

On boiling the filtrate from the scandium citrate a white precipitate comes down, dissolving again when the liquid cools.

The citrate precipitated in the cold was well washed with hot water, filtered, and dried.

When the citrate is dried in the air it contains six molecules of water, and has the composition given above.

When this salt is dried for some days over sulphuric acid it loses two molecules of water and becomes a 4-hydrate salt.

The 4-hydrate salt, heated in an air-bath for six hours to 100°, loses two more molecules of water, and becomes a 2-hydrate.

Analysis.

(1) 3·541 gr. of scandium citrate, dried in the air, yielded on ignition 1·140 gr. of scandia, = 0·7382 gr. of scandium.

(2) 4·670 gr. of scandium citrate, dried over sulphuric acid for four days, yielded on ignition 1·579 gr. of scandia, = 1·0225 gr. of scandium.

(3) 2·797 gr. of scandium citrate, dried over sulphuric acid for four days, yielded on ignition 0·939 gr. of scandia, = 0·6081 gr. of scandium.

(4) 3·541 gr. of scandium citrate, heated to 110° for 24 hours, lost 0·311 gr. of water.

(5) 4·467 gr. of scandium citrate, dried at 100°, yielded on ignition 1·579 gr. of scandia, = 1·0225 gr. of scandium.

(6) 3·379 gr. of scandium citrate, dried at 110° for two days, lost 0·149 gr. of water.

Analysis of the 6-*Hydrate Citrate.*

	Theory.			Experiment. (1).
$4\mathrm{Sc}$	176·40	20·79		20·85
$3(\mathrm{C_6H_4O_7})$	564·10	66·47	79·21	79·15
$6\mathrm{H_2O}$	108·10	12·74		
	848·60	100·00		100·00

Analysis of the 4-Hydrate Citrate.

	Theory.		Experiment.		
			(2).	(3).	(4).
4Sc	176·40	21·71	21·90	21·74	91·22
$3(C_6H_4O_7)$ . .	564·10	69·42	78·10	78·26	
$4H_2O$ . . .	72·06	8·87			8·78
	812·56	100·00	100·00	100·00	100·00

Analysis of the 2-Hydrate Citrate.

	Theory.		Experiment.	
			(5).	(6).
4Sc . . .	176·40	22·72	22·89	95·59
$3(C_6H_4O_7)$.	564·10	72·64	77·11	
$2H_2O$ . . .	36·03	4·64		4·41
	776·53	100·00	100·00	100·00

The earth resulting from these analyses was dissolved in nitric acid and tested in the spectrograph; no lines of yttrium or ytterbium could be detected. The mother-liquors were evaporated dry with nitric acid and ignited, and the residual earth tested in the spectrograph; traces were seen of yttrium and much more of ytterbium.

SCANDIUM ORTHOCHLOROBENZOATE,

$C_6H_4ClCOOScO+(C_6H_4ClCOO)_2Sc(OH)$.

Orthochlorobenzoic acid is exactly neutralised with ammonia and heated. To the hot solution scandium nitrate is added until no more precipitate comes down on adding a drop either of the ammonia or of the scandium salt. The whole is boiled and allowed to cool. The scandium orthochlorobenzoate forms a white amorphous precipitate, slightly soluble in hot water. It leaves white scandia on ignition in the air.

Analysis.

5·303 gr. of scandium orthochlorobenzoate dried at 105° left on ignition 1·244 gr. of scandia, = 0·8056 gr. of scandium.

	Theory.		Experiment.
Sc_2	88·20	15·01	15·19
$C_{21}H_{13}Cl_3O_8$	499·48	84·99	84·81
	587·68	100·00	100·00

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The scandia used in the preparation of this salt was derived from the quantitative analyses of other organic salts of scandium. Spectrum photographs showed that both the scandia from the precipitated orthochlorobenzoate and that from the mother-liquors were quite free from yttria and ytterbia.

SCANDIUM METANITROBENZOATE,

$$[C_6H_4(NO_2)COO]_3Sc + 4H_2O.$$

Metanitrobenzoic acid is neutralised with ammonia, a hot solution of scandium nitrate added till no more precipitate is formed, and the whole boiled for a few minutes. A white crystalline precipitate falls down. The metanitrobenzoate is very slightly soluble in water, and from a hot solution it separates in the form of flat crystals displaying brilliant colours when examined by polarised light. The salt is well washed, drained on blotting-paper, and dried over sulphuric acid in a desiccator.

Analysis.

(1) 5·624 gr. of scandium metanitrobenzoate left on ignition 0·607 gr. of scandia, = 0·3931 gr. of scandium.

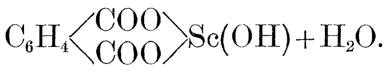
(2) 5·027 gr. of scandium metanitrobenzoate left on ignition 0·543 gr. of scandia, = 0·3516 gr. of scandium.

(3) 7·369 gr. of scandium metanitrobenzoate, dried at 110°, lost 0·864 gr. of water, and left on ignition 0·800 gr. of scandia, = 0·5181 gr. of scandium.

	Theory.			Experiment.		
				(1).	(2).	(3).
Sc	44·10	7·18		6·99	7·00	7·03
$C_{21}H_{12}O_6 3(NO_2)$ . .	498·13	81·09	92·82	93·01	93·00	81·25
$4H_2O$	72·06	11·73				11·72
	614·29	100·00		100·00	100·00	100·00

The scandia used in these experiments was intentionally not quite pure, being vitiated with a little yttria and ytterbia. On testing, the scandia resulting from the decomposition of the metanitrobenzoate showed no trace of the dominant lines of yttrium or ytterbium, while the earth precipitated from the mother-liquors of the scandium metanitrobenzoate on spectroscopic examination showed strong lines of both impurities. It will be remembered that NEISH found that metanitrobenzoic acid was capable of separating thoria from its accompanying earths with quantitative accuracy. Further experiments with this acid are in progress.

SCANDIUM PHTHALATE,



$$C_6H_4{<}\begin{matrix}COO\\COO\end{matrix}{>}Sc(OH)+H_2O.$$

When a solution of scandium nitrate is added to a solution of ammonium phthalate, an amorphous white precipitate of scandium phthalate comes down. It is slightly soluble in excess of scandium nitrate, and insoluble in water or in excess of ammonium phthalate. Washed and dried in the air it contains one molecule of water and has the composition given above; and after being heated for thirty-six hours to 110° it loses half a molecule of water. Probably a little increase of temperature would have rendered the salt anhydrous. I did not pursue the analysis further.

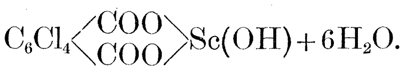
Analysis.

5·251 gr. of air-dried scandium phthalate lost, on drying for thirty-six hours at 110°, 0·192 gr. of water and left on ignition 1·487 gr. of scandia, = 0·9629 gr. of scandium.

	Theory.		Experiment.
Sc	44·10	18·14	18·34
$C_8H_5O_5$	181·04	78·16	78·00
$\frac{1}{2}H_2O$	9·01		
$\frac{1}{2}H_2O$	9·01	3·70	3·66
	243·16	100·00	100·00

The scandia resulting from the above analyses was dissolved in nitric acid and a photograph taken of its spark spectrum. It was seen to be absolutely pure, with no trace of lines belonging to other earths. The scandia remaining in the filtrate after precipitating the phthalate was examined in the same way, and in its photographed spectrum the dominant lines of yttrium and ytterbium could easily be detected, accompanying the strong scandium spectrum. Experiments are now in progress to find if a satisfactory method of separating scandium from yttrium and ytterbium can be based on this reaction.

SCANDIUM TETRACHLOROPHTHALATE,



$$C_6Cl_4{<}\begin{matrix}COO\\COO\end{matrix}{>}Sc(OH)+6H_2O.$$

On mixing together equivalent proportions of scandium nitrate and ammonium tetrachlorophthalate a dense white amorphous precipitate comes down, almost insoluble in hot or cold water. When washed and dried in the air scandium tetrachlorophthalate has the composition given above.

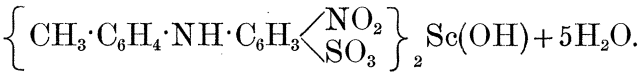
Analysis.

4·830 gr. of scandium tetrachlorophthalate left on ignition 0·692 gr. of scandia, = 0·4481 gr. of scandium.

	Theory.		Experiment.
Sc	44·10	9·36	9·28
$C_8HCl_4O_5$	318·85	90·64	90·72
$6H_2O$	108·10		
	471·05	100·00	100·00

The scandia left on ignition was tested in the spectrograph and found to be quite pure. Yttrium and ytterbium could be detected in the earth obtained from the filtrate left after precipitating the tetrachlorophthalate.

SCANDIUM 2-NITROPHENYL-4′-TOLYLAMINE-4-SULPHONATE.



$$\left\{CH_3 \cdot C_6H_4 \cdot NH \cdot C_6H_3 \begin{matrix} \diagup NO_2 \\ \diagdown SO_3 \end{matrix}\right\}_2 Sc(OH) + 5H_2O.$$

The sodium salt of this acid is dissolved in water and a solution of scandium nitrate added. If the solutions are strong, a red precipitate falls; if weak, the mixture must be concentrated. On cooling, the scandium salt separates in feathery crystals of a fine scarlet colour. It must be purified by re-crystallisation from a hot aqueous solution. The crystals are slightly soluble in cold water.

Heated in an air-bath to 105° for 48 hours, the scarlet crystals assume an orange-yellow colour, losing water and becoming anhydrous.

On being further heated, the salt decomposes rather suddenly, with intumescence, leaving a carbonaceous residue, which at a red heat gradually burns white, leaving scandia.

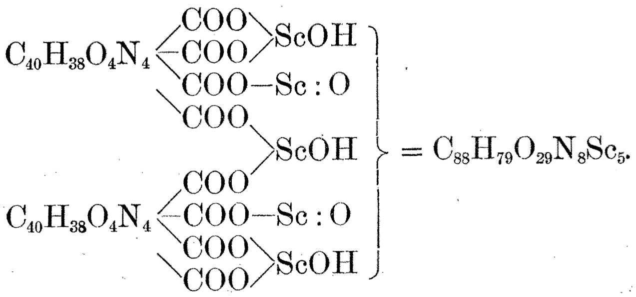
Analysis.

(1) 1·154 gr. of the scandium salt lost on heating 0·136 gr. of water. When heated to redness the same salt left 0·108 gr. of scandia, = 0·0699 gr. of scandium.

(2) 0·863 gr. of the salt, heated to 105°, lost 0·095 gr. of water.

	Theory.			Experiment.	
				(1).	(2).
Sc	44·10		5·76	6·06	88·99
26C	312·00	631·36	82·47	82·15	
23H	23·18				
11O	176·00				
4N	56·04				
2S	64·14				
$5H_2O$	90·08		11·77	11·79	11·01
	765·54		100·00	100·00	100·00

SCANDIUM OCTAMETHYLTETRAMINODIHYDROXYPARADIXANTHYLBENZENETETRA-CARBOXYLATE.



Naturally this is a question of considerable scientific interest. How would scandium behave when combined as a salt with a highly complex acid? In Dr. SILBERRAD's recently published paper on "The Condensation Products of Mellitic Acid with Meta-amino Phenol,"* some highly complex bodies are described; it became interesting to determine precisely what basicity scandia would assume towards polycarboxylic acids of this nature.

By the kindness of Dr. SILBERRAD I have had an opportunity of experimenting with octamethyltetraminodihydroxyparadixanthylbenzenetetracarboxylic acid.

Previous experiments would lead one to expect the scandium salt of this acid to have the composition $C_{44}H_{40}O_{14}N_4Sc_2$. This I could not prepare, the salt produced by metathesis containing more scandium than this formula admits of. The only scandium salt I could form of this acid has the composition shown above.

Octamethyltetraminodihydroxyparadixanthylbenzenetetracarboxylic acid is a dark red powder, insoluble in water and easily soluble in dilute ammonia. The solution in ammonia, very slightly alkaline, is exactly neutralised with acetic acid; to the hot

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* 'Journ. Am. Chem. Soc.,' Feb., 1910, No. 2, p. 189.

solution scandium acetate is added and the whole boiled. On cooling, scandium octamethyltetraminodihydroxyparadixanthylbenzenetetracarboxylate comes down as a red powder, insoluble in cold water.

Analysis.

3·167 gr. of scandium octamethyltetraminodihydroxyparadixanthylbenzenetetracarboxylate left on ignition 0·576 gr. of scandia, = 0·3730 gr. of scandium.

	Theory.		Experiment.
Sc_5	220·50	11·41	11·78
$C_{88}H_{79}O_{29}N_8$ . . .	1711·71	88·59	88·22
	1932·21	100·00	100·00

More than once I have been asked why I chose such very out-of-the-way acids wherewith to prepare scandium salts? I give my chief reason:—Attempts on several occasions have been made to discover a means of separating some of the "rare earths" from their companions by forming compounds with weak organic acids. Thus, in 1897, KOSMANN* employed citric acid in the separation of thoria. URBAIN† used acetylacetonate of sodium for the same purpose. METZGER‡ tried maleic acid, cinnamic acid, picric acid, phthalic acid, and fumaric acid. He found fumaric acid effectual in separating thoria from the other earths in monazite, whilst the other acids also gave partial separations, although not complete.

In 1904, NEISH§ tried many organic acids for the separation of the rare earths, chiefly thoria. Among other acids, he tried gallic, tannic, citric, salicylic, oleic, linoleic, paratoluic, oxyisophthalic, benzoic, meta-, ortho-, and para-nitrobenzoic, and fumaric. Of these the metanitrobenzoic acid proved most effectual, the process being capable of accurate quantitative results. It was found that other rare earths besides thoria behaved differently towards these acids, but the variation was not sufficient to enable the reactions to be used for accurate separations.

Soon after those experimental papers appeared I commenced similar researches hoping to find an organic reagent which would be a precipitant for some of the yttria earths—if not with quantitative accuracy, at all events with sufficient separation to allow a fractionation method to be based on the reaction. My results, not being sufficiently definite, were never published; but as the organic acids were in my laboratory when the scandium research was commenced, I preferred to use these acids,

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* 'Chem. Centralb.,' 1897, Part I., 837; 'Chemical News,' lxxxvi., 218.

† 'Bull. Soc. Chim.,' 1896, xv., 347.

‡ 'Journ. Am. Chem. Soc.,' xxiv., No. 10; 'Chemical News,' lxxxvi., 218, 229, 242.

§ 'Journ. Am. Chem. Soc.,' xxvi., No. 7; 'Chemical News,' xc., 196, 201.

of which the purity and the history were known, rather than start afresh with acids of unknown history.

Many of the acids were prepared and purified by myself; for others I am under obligations to friends, among whom I must specially thank Dr. O. Silberrad.

I wish also to express warm thanks to Dr. von Martius for the munificent gift of 100 gr. of almost pure scandia—a gift which has enabled me to work on a larger scale, and to preserve many specimens of analysed salts for future reference.

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