7. *Bunsen's Salt.*

By HARRY IRVING and G. W. CHERRY.

The system NH_4Cl–$(NH_4)_4Fe(CN)_6$–H_2O has been studied at 25° and 40° : the only other solid phase is the double salt $2NH_4Cl,(NH_4)_4Fe(CN)_6$—Bunsen's salt. The existence of the trihydrate described by Hölzl could not be confirmed, and his grounds for formulating this salt as $(NH_4)_6[Fe(CN)_6Cl_2],3H_2O$, with 8-covalent iron, are critically examined. A preliminary note of an *X*-ray study of Bunsen's salt is given, and an explanation is proposed for its alleged chromoisomerides.

THE action of ammonium chloride upon Prussian-blue was found by Bunsen (*Pogg. Ann.*, 1835, **36**, 404) to yield fine crystals of a " triple salt " $4NH_4CN,Fe(CN)_2,2NH_4Cl,3H_2O$ [*i.e.*, $2NH_4Cl,(NH_4)_4Fe(CN)_6,3H_2O$] which could be more conveniently prepared from ammonium chloride and ammonium or potassium ferrocyanide. This salt, called Bunsen's salt by subsequent workers, was later prepared by Étard and Bémont (*Compt. rend.*, 1885, **100**, 108), who represented it to be anhydrous although they gave no analyses, and by Freund (*Ber.*, 1888, **21**, 934), by the action on Buff's salt (see below) of alcoholic ammonia. Grailich (" Krystallographisch-optische Untersuchungen," Preisschr., Wien, 1868, 133) studied its optics, and Topsøe (*Arch. Sci. Phys. Nat.*, 1872, **45**, 225) recorded its density as 1·49.

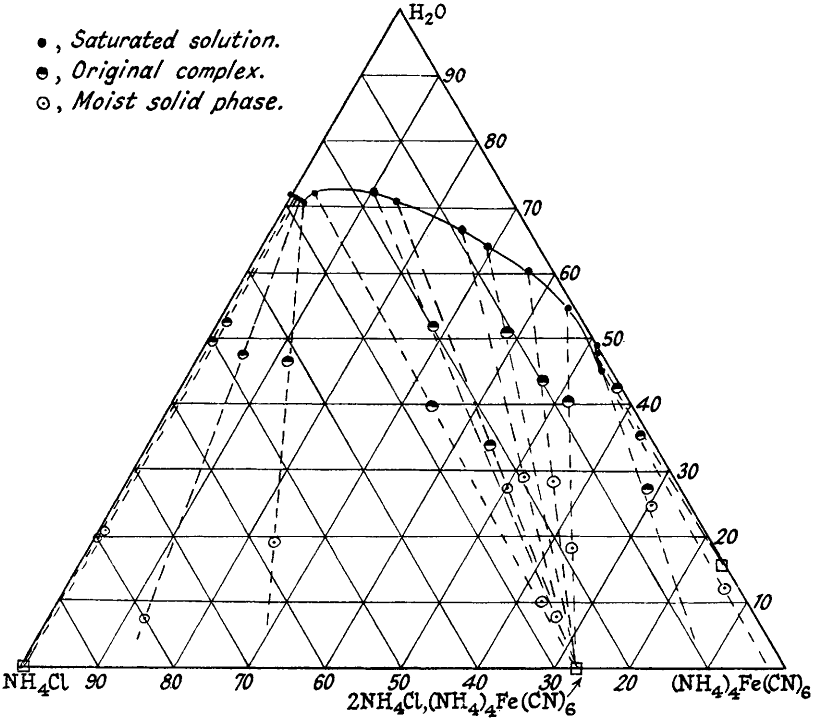
Briggs (J., 1911, **99**, 1027) described two forms of Bunsen's salt : the pale yellow α-form separated from solutions of ammonium chloride and ammonium ferrocyanide containing a little sodium cyanide, whereas the deep amber β-form was obtained from a solution of the two components acidified with dilute acetic acid. The two forms were anhydrous and seemed analytically indistinguishable, yet despite substantially identical solubilities and molecular weights, Briggs formulated them as co-ordination isomers. Both potassium ferrocyanide and ferricyanide were found to give analogous pairs of isomers. When, however, Piutti (*Ber.*, 1912, **45**, 1831) found that the isomer pairs had indistinguishable absorption spectra, and Bennett (J., 1917, **111**, 490) showed that they were crystallographically identical, Briggs (J., 1920, **117**, 1029) suggested that the modified properties of the β-form of potassium ferrocyanide could be explained by assuming that it consisted of the normal, or α-ferrocyanide with about 1% of aquopentacyanide $K_3[Fe(CN)_5(H_2O)]$, *i.e.*, enough to modify the colour and crystal habit, but insufficient to affect the analyses. Presumably a similar explanation was intended for the isomerism of Bunsen's salt.

Recently, in view of the formal analogy which Baeyer and Villiger (*Ber.*, 1902, **35**, 1202) remarked between Bunsen's salt and the oxonium salts $(ROH_2)_4,Fe(CN)_6,2(ROH_2)Cl$ which Buff (*Ann. Chem. Pharm.*, 1854, **91**, 253) obtained by passing hydrogen chloride into an alcoholic solution of hydroferrocyanic acid, Hölzl (*Sitzungsber. Akad. Wien*, 1928, **137**, II*b*, 1110) suggested that these two " double salts " ought to be formulated as $(NH_4)_6[Fe(CN)_6Cl_2]$ and $(ROH_2)_6[Fe(CN)_6Cl_2]$ respectively, both salts containing the same complex anion, admittedly of low stability since their aqueous solutions gave only the analytical reactions of Cl^- and $[Fe(CN)_6]^{4-}$. Subsequently, Hölzl and Stockmair (*ibid.*, 1932, **141**, II*b*, 763) adduced physicochemical evidence for the existence of this complex ion $[Fe(CN)_6Cl_2]^{6-}$ with iron in 8-fold co-ordination. This unorthodox hypothesis, together with the fact that Hölzl's analysis of Bunsen's salt corresponds exactly with that of a trihydrate, induced us to re-examine its composition and constitution.

The phase-rule study of the system NH_4Cl–$(NH_4)_4Fe(CN)_6$–H_2O presented several problems owing to the photosensitivity and ease of oxidation of aqueous solutions of ammonium ferrocyanide, and difficulties inherent in the analysis of mixtures of chloride and ferrocyanide, especially when their molecular proportions are widely dissimilar. Furthermore, the high solubilities of the two salts caused rather viscous solutions which impeded filtration and hindered the isolation of a solid phase even reasonably free from mother-liquor for the " residue method." Known weights of the two salts and air-free water were, therefore, sealed off in a nitrogen atmosphere in glass tubes with specially thin bottoms and brought to equilibrium by shaking in a light-tight zinc box immersed in a thermostat. The phases were separated by filtration through sintered glass in an apparatus immersed in the thermostat, weighed, and analysed volumetrically (see p. 29).

The composition of the original complex being known, analysis of either the resulting saturated solution or the (moist) solid phase would suffice for extrapolation to the composition of the pure solid phase : in many cases both were analysed, and the reliability of a determination could be assessed from the collinearity of the plot of the three sets of concentration data.

The isothermal for 25·1° ± 0·1° is shown in the figure : a substantially similar diagram was obtained at 40·05° ± 0·1°, and throughout this range Bunsen's salt comprises the only new solid phase. A few less accurate measurements at 0° failed to indicate any change in the general character of the phase diagram.



The solid phase appears to be anhydrous $2NH_4Cl,(NH_4)_4Fe(CN)_6$, the solution containing 38·9, 49·7, and 53·4 g. per 100 g. of water at 0°, 25·1°, and 40·05° respectively. Its composition is supported by analyses of the salt prepared in the present work, and of (new) preparations of the α- and β-forms according to Briggs (*loc. cit.*). Data for a preparation from potassium ferrocyanide (Bunsen, *loc. cit.*) and Hölzl's figures are included.

	K, %.	NH_3, %, (a).	Fe, %, (b).	$Fe(CN)_6''''$, %, (c).	Cl, %, (d).
Present authors	0·0	25·8, 25·6	14·2, 14·3	54·2, 54·2	18·2, 18·4
Briggs's α-form	0·0	25·6, 25·8	14·2, 14·25	54·0	18·0
,, β-form	0·0	26·1, 25·8	14·6, 14·4	54·2	18·4
Bunsen's prepn.	4·6	23·5, 23·8	14·0, 14·0	53·7	18·0
Hölzl's ,, ...	0·0	22·9	12·8	—	16·0
Calc. for $2NH_4Cl,(NH_4)_4Fe(CN)_6$	0·0	26·1	14·3	54·2	18·1
,, ,$\frac{1}{2}H_2O$...	0·0	25·4	13·95	53·0	17·7
,, ,$3H_2O$...	0·0	23·0	12·55	—	15·9

(a) Ammonia liberated by alkali. (b) Separated as $Fe(OH)_3$ after ignition with H_2SO_4. (c) Determined by means of ceric sulphate. (d) Determined by Volhard's method.

There is thus no confirmation of Hölzl's trihydrate, which he states was prepared " by Bunsen's method " and " crystallised from water "; unfortunately, the references he quotes are to abstracts of Étard and Bémont's papers (themselves devoid of experimental details), and not to Bunsen's own work.

In preparing Briggs's β-form some Prussian-blue is formed and must be separated by filtration; much more results during Bunsen's method of preparation from potassium ferrocyanide—which also gives a deep amber product. These observations suggested that the " isomerism " of the β-form might be due, not to admixed ammonium aquopentacyanoferroate (see p. 25), but rather to adsorbed or entrained Prussian-blue. Crystallisation of the pale yellow α-form from a colloidal solution of Prussian-blue in air-free water did, in fact, yield amber, almost brown crystals characteristic of the β-form. Unless absolutely dry, all crystals of the β-form turn green on keeping, whereas the α-form can be preserved unchanged. A similar explanation of the " isomerism " of potassium ferricyanide (Locke and Edwards, *Amer. Chem. J.*, 1898, **21**, 193) has been confirmed by Hauser and Biesalski (*Ber.*, 1912, **45**, 3516), who showed that even 0·001 mol. of Prussian-blue per mol. of ferricyanide would modify its colour.

Hölzl's grounds for formulating Bunsen's salt as $(NH_4)_6[Fe(CN)_6Cl_2]$ with 8-co-ordinated iron are derived from conductivity and transport measurements. Although the conductivity in water at 25° of mixtures of ammonium chloride and ammonium ferrocyanide in various proportions was a linear function of their molar fraction in 0·01M-solution, yet Hölzl and Stockmair (*loc. cit.*) found that the conductivities of 0·02, 0·05, and 0·10M-mixtures were invariably less than those calculated by this linear relationship, the defect increasing with increasing concentration. They interpreted these results (some of which, recalculated as equivalent conductances, are reproduced below) as evidence for the formation of " one or more compounds " with the original ions recombined " to higher complexes of lower mobility " : the gradual disappearance of the conductivity defect was taken as evidence for the displacement of the equilibrium $[Fe(CN)_6Cl_2]^{6-} \rightleftharpoons 2Cl^- + [Fe(CN)_6]^{4-}$ to the right with increasing dilution.

Molar ratio $\frac{(NH_4)_4Fe(CN)_6}{NH_4Cl}$.	0·1M.			0·02M.		
	$\Lambda^{25°}_{calc.}$	$\Lambda^{25°}_{obs.}$	Defect.	$\Lambda^{25°}_{calc.}$	$\Lambda^{25°}_{obs.}$	Defect.
10 : 0	312	312	—	405	405	—
9 : 1	295	249	46	385	345	38
8 : 2	278	233	45	360	325	35
7 : 3	261	221	40	338	305	33
6 : 4	244	203	41	315	295	20
5 : 5	228	190	38	293	280	13
4 : 6	211	178	33	270	265	5
3 : 7	194	167	17	255	248	7
2 : 8	177	161	10	230	225	5
1 : 9	160	151	9	203	200	3
0 : 10	143	143	—	180	180	—

Now such conductivity defects are, in point of fact, predicted by theory and realised experimentally even where complex-ion formation can scarcely be involved (*e.g.*, in mixtures of alkali halides, Van Rysselberghe and Nutting, *J. Amer. Chem. Soc.*, 1937, **59**, 333); yet there have been many cases (cf. Van Rysselberghe, Grinnell, and Carlson, *ibid.*, p. 336) where the magnitude of the conductivity defects has led investigators incorrectly to postulate complex-ion formation for, as Davies has pointed out (J., 1938, 448), the conductivity of a mixture of two electrolytes of the same equivalent concentration will differ from that calculated by an additive formula on account of two factors, (*a*) the change in mobility of the ions, and (*b*) the change in ionic concentration when dissociation is incomplete. For example, the deviations which in mixtures of magnesium chloride (completely dissociated) and sodium sulphate amount to as much as 10% of the total conductivity are due to the formation of magnesium sulphate ion-pairs (Davies, *loc. cit.*). Now while 0·1M-ammonium chloride is 87% dissociated, 0·1M-potassium ferrocyanide is only about 50% dissociated, and in view of the substantially identical mobilities of K^+ and NH_4^+ we may take the same figure for 0·1M-ammonium ferrocyanide : the corresponding figures for 0·01M-solutions are 97% and 73%. The " common-ion " effect of adding highly dissociated ammonium chloride to poorly dissociated ammonium ferrocyanide will be still further to reduce the dissociation of the latter and the conductivity of the mixture will fall. On the other hand, addition of ammonium ferrocyanide to ammonium chloride solutions should produce a less significant conductivity decrease. Hölzl's data illustrate both these predictions, and the maximum conductivity defect occurs with less than 10 mols.% of ammonium chloride and then decreases with increasing ferrocyanide content. On the other hand, were the complex $[Fe(CN)_6Cl_2]^{6-}$ to be formed, the maximum defect would occur at the corresponding composition, *i.e.*, about 66 mols.% of ammonium chloride. The gradual appearance of a green colour and increase in the conductivity of ammonium chloride–ferrocyanide mixtures over a period of some 18 hours is not due, as Hölzl inferred, to any decomposition of the hypothecated anion $[Fe(CN)_6Cl_2]^{6-}$, but is an oxidation and photo-change which can be arrested indefinitely in the absence of air and light.

Finally, Hölzl and Stockmair (*loc. cit.*) record a single transport experiment in which the passage of electricity (quantity not given) through a 0·1M-solution of Bunsen's salt caused migration of chloride and ferrocyanide ions in the molecular ratio 2 : 1·03. Since the ionic mobilities are 76·6 and 100·8 respectively, this experiment appears to afford weighty evidence for the real existence of the complex anion $[Fe(CN)_6Cl_2]^{6-}$. We could not, however, confirm this unique experiment, for under all conditions oxidation of ferro- to ferri-cyanide took place. Hölzl did not consider this contingency, and the analytical method which he employed to determine the change in halogen content in the anode compartment (difference between estimate of total $[Fe(CN)_6]^{4-} + [Cl^-]$ by Volhard, and $[Fe(CN)_6]^{4-}$ iodometrically or manganometrically) leads to fictitious high values for $[Cl^-]$ since the argentimetric titre is increased by any ferricyanide formed, whilst oxidation simultaneously decreases $[Fe(CN)_6]^{4-}$. Our experiments showed that oxidation was indeed the principal reaction, and that the concentration of chlorine in the anode compartment, far from increasing as Hölzl recorded, did in fact decrease slightly although chlorate ions could not be detected qualitatively.

A number of experiments on the freezing point of mixtures of calcium ferrocyanide and calcium or barium chloride likewise failed to produce evidence in favour of complex-ion formation of the type postulated by Hölzl.

Isomorphous with Bunsen's salt is the bromine analogue (Bunsen and Himly, *Pogg. Ann.*, 1836, **38**, 208): we have repeated its preparation and confirm the formula as anhydrous $2NH_4Br,(NH_4)_4Fe(CN)_6$. Attempts to prepare double salts from ammonium ferrocyanide and ammonium cyanide, fluoride, or iodide were unsuccessful, and no analogue of Bunsen's salt from potassium ferrocyanide and chloride could be obtained although Étard and Bémont (*loc. cit.*) claim to have prepared $2NH_4Cl,K(NH_4)_3Fe(CN)_6$ and $2NH_4Cl,(NH_4)KH_2Fe(CN)_6$ from potassium ferrocyanide and ammonium chloride. Although Denigès (*Bull. Soc. chim.*, 1916, **19**, 86) has proposed "structural" formulæ for Bunsen's salt and the latter compounds, they are nowadays of no significance and further progress can best be made through a detailed *X*-ray analysis. The preliminary work (below) confirms our formulation and negatives the existence, even in the crystalline state, of the complex anion hypothecated by Hölzl.

X-*Ray Analysis*.—Messrs. G. W. R. Bartindale and H. M. Powell report as follows: The results of the goniometric and optical examination of the crystals agree closely with those of Bennett (*loc. cit.*), except that an additional form {111} was observed. *X*-Ray examination gave for the hexagonal unit cell $a_0 = 9{\cdot}24$, $c_0 = 18{\cdot}92$ A., and $c : a = 2{\cdot}046$; goniometrically $2c : a = 2{\cdot}038$. The density, by flotation, is 1·40 g./c.c., and there are 3 molecules per unit cell. The calculated cell molecular weight agrees closely with the theoretical value and is incompatible with the presence of water of crystallisation. The absent spectra are those characteristic of a rhombohedral lattice. No evidence of the absence of a symmetry centre could be obtained, and the space group is probably $R\bar{3}$. Referred to the hexagonal cell, iron atoms lie at (000), and chlorine atoms at $(00\overset{\pm}{2})$, and at the equivalent points. Preliminary structure determination shows that $z \approx \frac{1}{3}$. This means that the Fe–Cl distance is about 6 A., and at once excludes any possibility of a covalent link between these atoms. A detailed structure determination is in progress.

EXPERIMENTAL.

Materials.—Ammonium ferrocyanide was prepared from "AnalaR" potassium ferrocyanide through the hydroferrocyanic acid–ether complex (Mittasch and Kuss, *Z. Elektrochem.*, 1928, **34**, 159), the latter being freed from potassium and hydrogen chlorides by repeated washing (Wagener and Tollens, *Ber.*, 1906, **39**, 410). The preparation was carried out in a room illuminated by red light, and the product was preserved in a vacuum over calcium chloride.

Qualitative tests (Feigl, " Qualitative Analyse mit Hilfe von Tupfelreaktionen," 2nd Edition, pp. 267, 276) proved the absence of potassium and chloride. The salt precipitated from aqueous solution by alcohol retained about 1½ mols. of water, which could only be removed with difficulty and some decomposition. Analyses of typical preparations established its composition and purity.

	(1).	(2).	(3).	(4).	(5).	(6).	(7).
$[Fe(CN)_6]^{4-}$, %	68·5	69·0	68·4	72·9	72·5	74·6	68·2
$(NH_4)^+$, %	23·35	23·3	23·8	24·6	24·7	25·4	23·2
H_2O, % (by diff.)	8·1	7·65	8·5	2·5	2·8	0·0	8·6
Ratio $\frac{[Fe(CN)_6]^{4-}\%}{(NH_4)^+\%}$ ...	2·93	2·97	2·88	2·97	2·94	2·94	2·94

(1) Original preparation. (2) After 1 week in a vacuum. (3) Preparation dissolved in ice-water and reprecipitated. (4) Benzene added to salt and distilled off rapidly. (5) Benzene added and distilled off below 90°. (6) Calc. for $(NH_4)_4Fe(CN)_6$. (7) Calc. for $(NH_4)_4Fe(CN)_6,1\frac{1}{2}H_2O$.

Each preparation of ammonium ferrocyanide was analysed before use and was employed with " AnalaR " ammonium chloride in making up complexes by weight as described above (p. 25). Preliminary experiments showed that in the absence of light and oxygen the decomposition of N/10-ammonium ferrocyanide solutions did not exceed 0·3% in 10, and 1% in 30 days at 25°. Though equilibrium was reached within 48 hours, each complex was shaken in a thermostat regulated to ± 0·05° for at least 4 days before analysis.

Analysis.—Samples of the phases were diluted with air-free water to convenient known volumes. Ferrocyanide was determined with N/20-ceric sulphate standardised against anhydrous " AnalaR " potassium ferrocyanide (de Beer and Hjort, *Ind. Eng. Chem., Anal.,*

System NH_4Cl–$(NH_4)_4Fe(CN)_6$–H_2O.

[A = NH_4Cl; B = $2NH_4Cl,(NH_4)_4Fe(CN)_6$; F = $(NH_4)_4Fe(CN)_6$.]

Solution.			Original complex.			Moist solid.			Solid phase.
A, %.	F, %.	H_2O, %.	A, %.	F, %.	H_2O, %.	A, %.	F, %.	H_2O, %.	
				At 0·0°.					
13·6	9·7	76·7	15·5	15·8	68·7	24·0	56·0	20·0	B
8·7	18·9	72·4	10·3	22·1	67·6	—	—	—	B
7·0	21·9	71·1	16·5	49·1	34·4	20·4	59·6	20·0	B
				At 25·1°.					
28·3	0·0	71·7		A + H_2O		—	—	—	A
28·0	0·3	71·7	50·1	0·1	49·8	80·1	0·05	19·8	A
27·8	0·4	71·8	46·6	0·2	53·2	79·4	0·2	20·4	A
27·3	1·2	71·5	41·8	10·6	47·6	56·9	23·8	19·3	A, B
27·3	1·0	71·7	47·3	4·9	47·8	80·0	12·8	7·2	A, B
25·3	2·3	72·4	26·1	33·9	40·0	26·0	63·5	10·5	B
17·1	10·3	72·6	20·0	28·0	52·0	25·7	66·4	7·9	B
14·7	14·0	71·3	21·6	44·6	33·8	22·6	50·0	27·4	B
8·5	24·7	66·8		B + H_2O		19·2	51·5	29·3	B
6·2	30·0	63·8	10·6	38·3	51·1	—	—	—	B
3·7	36·7	59·6	9·7	46·6	43·7	16·0	55·7	28·3	B
0·8	44·6	54·6	7·6	51·8	40·6	18·2	63·0	18·8	B
0·6	53·8	45·6	4·0	68·4	27·6	4·7	70·3	25·0	B, F
1·0	53·6	45·5	0·5	63·9	35·6	0·8	87·1	12·1	B, F
0·3	51·7	48·0	0·5	56·8	42·7	0·05	64·9	35·1	F
0·0	51·3	48·7		F + H_2O		—	—	—	F
				At 40·05°.					
31·6	—	68·4		A + H_2O		—	—	—	A
29·9	0·8	69·3	50·1	0·5	49·4	—	—	—	A
29·6	1·3	69·1	49·4	1·0	49·6	—	—	—	A
28·4	1·8	69·8	49·3	10·5	40·2	—	—	—	A, B
28·4	1·6	70·0	29·4	30·7	39·9	—	—	—	A, B
24·3	4·5	71·2	25·0	20·5	54·5	26·7	60·0	13·3	B
23·6	5·2	71·2	24·9	25·1	50·0	—	—	—	B
15·9	13·9	70·2	19·7	29·9	50·4	—	—	—	B
9·5	25·2	65·3		B + H_2O		25·0	67·2	7·8	B
3·7	41·2	55·1	9·4	48·2	42·4	—	—	—	B
1·0	54·6	44·4	5·1	65·1	29·8	—	—	—	B, F
1·1	53·3	45·0	10·0	59·7	30·3	—	—	—	B, F
1·0	54·6	44·4	5·1	65·1	29·8	—	—	—	F
0·6	54·1	45·3	1·0	60·4	38·6	—	—	—	F
0·9	54·9	44·2	0·6	61·9	37·5	0·6	74·1	25·3	F
0·0	54·7	45·3		F + H_2O		—	—	—	F

1935, **7**, 120), *N*-phenylanthranilic acid being used as internal indicator (Syrokomsky and Stiepin, *J. Amer. Chem. Soc.*, 1936, **58**, 928). Silver chloride and ferrocyanide were then precipitated together by a small known excess of N/10-silver nitrate, the excess being determined by the Volhard method. The concentration of chloride ion is then obtained by subtracting four times the cerimetric titre from this argentimetric value. Since this method would clearly prove unreliable for mixtures with small proportions of ammonium chloride, an alternative procedure was adopted wherein ferrocyanide ion was quantitatively precipitated as zinc ammonium ferrocyanide (Cone and Cady, *J. Amer. Chem. Soc.*, 1927, **49**, 356) and removed before the determination of the halogen. Control experiments showed that this process could be used for mixtures containing as little as 1% of ammonium chloride.

Transport Experiments.—These were carried out in a conventional apparatus in series with a silver coulometer. In a preliminary experiment in which a silver anode was employed, the passage of 162·7 coulombs through a M/10-aqueous solution of Bunsen's salt appeared to cause an increase of 0·0150 g.-mol./l. in the concentration of ammonium chloride and a decrease of 0·0074 g.-mol./l. in that of ammonium ferrocyanide. These results were worthless, for qualitative tests (Feigl, *op. cit.*, p. 300) showed the presence of ferricyanide ions. In subsequent experiments a platinum anode and cathode were used, and chloride and ferrocyanide having been determined as above (p. 29), ferricyanide was determined iodometrically in an aliquot portion of the anode solution, the excess of ferrocyanide and that formed during the titration being precipitated as $K_2Zn_3[Fe(CN)_6]_2$ by addition of zinc sulphate (Vogel, "Quantitative Inorganic Analysis," 1939, p. 435). The results of three experiments were:

a.	b.	c.	d.	e.	f.	g.	h.	i.	j.
0·2057	0·2004	0·1164	0·0937	0·0299	0·1236	+0·0072	0·0053	89·0	97·2
0·2190	0·2143	0·1249	0·1007	0·0311	0·1318	+0·0069	0·0047	70·0	102
0·1969	0·1959	0·0998	0·0786	0·0205	0·0991	−0·0007	0·0010	50·9	96·2

a, b = initial and final $[NH_4Cl]$; c, d = initial and final $[(NH_4)_4Fe(CN)_6]$; e = increase in $[(NH_4)_3Fe(CN)_6]$—initially zero; f = $[(NH_4)_4Fe(CN)_6]$ had no oxidation taken place; g = the increase in $[(NH_4)_4Fe(CN)_6]$ in the absence of oxidation; h = *decrease* in $[NH_4Cl]$; i = number of coulombs passed; j = the current efficiency (*q.v.*).

It is obvious that the preponderating change is the electrolytic oxidation of ferro- to ferricyanide which proceeds with approximately theoretical efficiency. The changes in $[NH_4Cl]$ (h), and in $[(NH_4)_3Fe(CN)_6 + (NH_4)_4Fe(CN)_6]$ (f), fall within the limits of accuracy of the experiments.

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