nitrate. The cracked end was immersed in a platinum crucible containing the same solution, the acid liquid forming around the cracked end being kept away from the crucible by means of a porous cup. A silver anode was placed in the open end of the bent tube, and a current was sent in series through this cell and through a standard porous cup voltameter. The fall in potential in the total circuit was 14 volts, the resistance being over a thousand ohms, chiefly in the capillary cracks. The electrostenolytic silver formed usually weighed about 5 centigrams and the silver peroxide weighed 5 milligrams. Three trials were made, with ordinary analytical precautions, but no extraordinary care. The weights of cathode silver found in the cell complicated by electrostenolysis were respectively 0.2213, 0.2532, 0.1432, while the standard coulometer yielded 0.2213, 0.2527, 0.1432. Thus the results were identical within the limit of error of the work. This outcome is consistent with Coehn's hypothesis.

Therefore, it was proved that the complication of electrostenolytic deposition does not affect the weight of the true cathode deposit, or the exact application of Faraday's law.

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# ON THE COMPLEXITY OF BERYLLIUM; A DISCUSSION.

BY CHARLES LATHROP PARSONS.

Received February 3, 1905.

KRÜSS AND MORAHT[1] noting the presence of a foreign substance in their ammonium carbonate solution of beryllium hydroxide, which yielded a black precipitate on treatment with ammonium sulphide, implied that a possible new element was under consideration. This claim was not directly made by them, but the fact that they stated it yielded a black sulphide, but a white hydroxide, left no other apparent conclusion. The writer during an extened investigation on beryllium collected a notable quantity of this substance under conditions similar to those which obtained in Krüss and Moraht's work, and showed that it consisted almost if not entirely of a mixture of zinc and iron sulphides, but mainly of zinc[2]. No evidence of the presence of any other substance was obtained. Pollok[3] also showed that this precipitate consisted

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[1] *Ann. Chem.* (Liebig), **262**, 47.

[2] *Ztschr. anorg. Chem.*, **40**, 407; This Journal, **26**, 727.

[3] *J. Chem. Soc.* (London), 1904, p. 604.

mainly of zinc and iron, but he found also some nickel and thought that another unknown substance might be present. His conclusions appeared shortly before my own, but did not come to my attention in time to acknowledge the fact in my paper.

Now another paper has appeared by Pollok[1] in which he reasserts his belief that the Krüss and Moraht precipitate contains a a new rare element and claims to have, himself, proved the presence of another new element accompanying beryllium, evidently in large quantities, for in a single fractional distillation made in a porcelain tube he obtained over 0.4 gram of a chloride which, on analysis, gave an atomic weight as high as 37 for this second new element. I say *second,* for under no possibility could this be the same as that of Krüss and Moraht, which yielded colored compounds and all of which Pollok had first carefullly removed by treatment with ammonium sulphide. These results are, to say the least, startling. Pollok claims by a single recrystallization of the sulphate to have obtained an increase in the equivalent of the metal in the first crop of crystals over the second, in face of the fact that many previous investigators, after many recrystallizations and rejections of the mother-liquors carrying presumably the beryllium of lower atomic weight, have noticed no similar results. Also every sublimation of the chloride gave a more volatile constituent which must, from the analysis, contain much more than the normal equivalent of the metal and although the removal of this metal with a higher atomic weight in such large quantities would, of necessity, much reduce the atomic weight of the true beryllium, no such result is obvious nor are any experiments cited to show the fact. The results obtained by volatilizing the chloride give a basic portion varying in its equivalent from 4.77 to 18.74, and, in the one case given, retreatment reduced the equivalent.

It is certainly to be regretted that Pollok has not given full details of manipulation, for if, by any chance, the gas passed through his distilling apparatus was not perfectly dry, as he thought, his results are easily explained, or if his beryllium chloride was exposed to the air for even an instant in being transferred to the vessel in which it was dissolved, chlorine was almost certainly lost in the form of hydrochloric acid. Pollok speaks of "scraping out" the beryllium chloride, which would indicate that

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[1] *J. Chem Soc.* (London), 1904, p. 1630.

he uses the same method outlined by him in a previous paper[1] in which the chloride was scraped from the sublimation tube into a drying bottle, inevitably coming into contact with the air of the room. In fact he speaks of the great difficulty of transferring and weighing his chloride on account of its great hygroscopicity and in his calculations considers the absorbed moisture, which was admittedly present, but does not take into account the chief fact that this moisture immediately decomposes the chloride with certain loss of chlorine.

It has long been known that beryllium chloride is especially subject to decomposition by the slightest traces of moisture, and it is a peculiarity also of this chloride that the residue left is not effected in solubility by such action nor in any of its ordinary reactions after it is dissolved. In fact, one equivalent of hydrochloric acid will dissolve several equivalents of the basic carbonate. Also by the action of a slight amount of moisture on the perfectly anhydrous chloride, a chloride can be obtained apparently dry, perfectly soluble and still giving a hissing noise as it dissolves, which contains a number of equivalents of beryllium to one of chlorine. This is also true to a varying extent with the acetate, sulphate, etc., as I have elsewhere pointed out.[2]

To properly dry a gas which is to come in contact with any of the beryllium halides and leave them unchanged is no easy problem. Even with magnesium chloride, which is much less affected than the beryllium salt, Richards has shown[3] that to use sulphuric acid as a desiccating agent a number of towers had to be used with fresh acid continually flowing through them. Ordinary gas drying bottles can not safely be used for this purpose with any reagent, and calcium chloride is useless. Morley[4] has described probably the best drying apparatus known, in which phosphoric anhydride removes all but the merest traces of water. It is certain that some such appliance as this must be used if any data are to be obtained as to the beryllium equivalent from its chloride. Awdejews' high results on the chloride were undoubtedly due to the action of water, as I have already pointed out,[5] he having dried his gas by calçium chloride only. Indeed, the thorough drying of the gas itself is not the only problem to be

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[1] *Trans. Roy. Soc.*, Dublin (2), **8**, 150 (1904).

[2] *Ztschr. anorg. Chem.*, **42**, 254; This Journal, **26**, 1437.

[3] *Ztschr. anorg. Chem.*, **13**, 94.

[4] *Am. J. Sci.*, **34**, 199; This Journal, **26**, 1171.

[5] *Ztschr. anorg Chem.*, **40**, 400; This Journal, **26**, 721.

overcome, for Richards has shown that even with magnesium chloride[1] the sublimed salt must be sealed in a perfectly dry atmosphere before being taken from the distillation tube and precautions taken against loss of chlorine in its solution.

It will be noticed in this connection that all duplicates given by Pollok of analyses are evidently of aliquot portions of the solutions after the chloride has been dissolved and not of separate portions of chloride dissolved separately. As none of the separate experiments give results alike, but all, without exception, yield only a chloride with a high equivalent of metal, it would certainly seem as if the trouble must be in the methods used, for otherwise the true beryllium left in his less volatile chloride must decrease in atomic weight. Until such a result is shown and full details of manipulation given, Pollok's conclusions must be accepted with great hesitancy. If, by his single recrystallization of the sulphate, he did not succeed in removing all of the excess of sulphuric acid originally present, it is not at all surprising that his second crop of crystals yielded a lower percentage of beryllium.

It is equally impossible to discuss Pollok's spectroscopic results for there is nothing to indicate which lines grew brighter or fainter by the separation of the new beryllium from the old. The two lines, 3274 and 3247, which he does cite as belonging to some other element, are suspiciously close to two of the prominent lines of the spark spectrum of copper, although there is nothing else to indicate that copper could be present.

I am far from stating or believing that a new element may not be present in beryl in minute quantities, but I have never seen anything to indicate it, although I have obtained results quite similar to Pollok's by the action of water on the chloride of beryllium.

It is true that my work was upon American beryl so far as the identity of my material was known, although I used a large amount of beryllium hydroxide obtained from Kahlbaum, which may perhaps have come from the beryl of Limoges. The latter beryl was, however, the source of supply of many of the European investigators, and it would indeed be strange if this particular beryl contained a new element in quantity sufficiently large to increase the basic constituent 0.6 per cent. by a single fractional crystallization of the sulphate, without previous discovery.

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[1] *Ztschr. anorg. Chem.*, **13**, 84.

Although negative results are seldom published, there can be little doubt that Nilson and Pettersson also obtained results entirely similar to those of Pollok but soon found the true explanation. They claimed that beryllium chloride also attacked glass, but overcame both difficulties sufficiently for vapor density determinations in one of their best researches, "Ueber ein neues mit exacter Temperature Bestimmung verbundênes Verfahren zur Festellung der Dampfdicte fleuchtiger Korper[1], which is, unfortunately, too little quoted. There can be no question that they and other investigators of the atomic weight of beryllium would have used the chloride but for the extreme difficulties of its manipulation which, unconquered, always lead to results tending in the same direction as those of Pollok.

NEW HAMPSHIRE COLLEGE,
DURHAM, N. H., Feb. 1, 1905.

# A CORRECTION.

BY WILLIAM A. NOYES AND HOWARD W. DOUGHTY.[2]

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SOME time ago one of us[3] effected the synthesis of what were supposed to be $\beta,\beta$-dimethyladipic acid and $\alpha,\beta,\beta$-trimethyladipic acid. Recently Blanc[4] has prepared the former acid by a method which seems not likely to involve a molecular rearrangement, but has found the properties of his acid radically different from those of ours. He has been kind enough to call our attention to the difference and to suggest that the formation of our acid may have been accompanied by a molecular rearrangement.

On examination of the literature the close agreement between the melting-point of the acid which one of us has described as $\beta,\beta$-dimethyladipic acid and the melting-point of $\beta$-isopropylglutaric acid,[5] and also the agreement between the melting-point of the acid which was described as $\alpha,\beta,\beta$-trimethyladipic acid and that of cis-$\alpha$-methyl-$\beta$-isopropylglutaric acid[6] was noticed. The $\beta$-isopropylglutaric acid might have been formed in our synthesis as follows: The chlorisocaproic ester,

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[1] *J. prakt. Chem.* (2), **33**, 1.

[2] The work here described was carried out with the aid of the Carnegie Institution.

[3] This Journal, **23**, 392 (1901).

[4] *Compt. Rend.*, **139**, 800.

[5] Howles, Thorpe and Udall: *J. Chem. Soc.*, (London), **77**, 942.

[6] *Ibid.*, **77**, 946.