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OBSERVATIONS ON FARADAY AS ORGANIC CHEMIST MANQUÉ

Derek A. Davenport, Purdue University

Faraday's work in pure chemistry has been treated somewhat condescendingly by commentators. Partington dismisses it in less than a page and Williams' rather fuller treatment is consigned to an omnibus chapter titled "The Fallow Years" (1, 2). There is perhaps some justice in this. If we exclude the work on liquefaction of gases as being *sui generis* non-chemical and if we yield that on "Relations of Gold and Other Metals to Light" to the uncoagulated and as yet unnamed colloid chemists (3), we are forced to concede that even those chemical papers Faraday chose to reprint in *Experimental Researches in Chemistry and Physics* (4) are, for the most part, minor work. Faraday's touching footnote to the first of these, "Analysis of Native Caustic Lime", could with equal humility have been appended to several others (5):

I reprint this paper at full length. It was the beginning of my communications to the public, and in its results very important to me. Sir Humphry Davy gave me the analysis to make as a first attempt in chemistry at a time when my fear was greater than my confidence, and both far greater than my knowledge; at a time also when I had no thought of ever writing an original paper on science. The addition of his own comments and the publication of the paper encouraged me to go on making, from time to time, other slight communications, some of which appear in this volume. Their transference from the "Quarterly" into other Journals increased my boldness; and now that forty years have elapsed and I can look back on what the successive communications have led to, I still hope, much as their character has changed, that I have not, either now or forty years ago, been too bold.

There are six papers, however, that taken together provide a striking exception to this generalization: "Two New Compounds of Chlorine and Carbon, etc."; "New Compound of Chlorine and Carbon"; "Hydriodide of Carbon"; "New Compounds of Carbon and Hydrogen"; "Pure Caoutchouc"; and "Mutual Action of Sulphuric Acid and Naphthaline". These reveal that in the years between 1820 and 1826, Faraday had mastered those arts - synthesis, separation, purification, characterization, analysis - necessary to the emerging subdiscipline of organic chemistry. Indeed, it is doubtful that any of his contemporaries could claim greater achievement in that area. Even so he abandoned the subject in 1826, never to return, and if we are to judge by his letters to Liebig and Dumas, among others, he subsequently evinced little interest in the extraordinary efflorescence of organic chemistry that took place during the rest of his life.

"On Two New Compounds of Chlorine and Carbon, and on a New Compound of Iodine, Carbon, and Hydrogen" (6) describes the preparation and characterization of perchloride

of carbon (C_2Cl_6) and protochloride of carbon (C_2Cl_4) while "On a New Compound of Chlorine and Carbon" (7) deals with what the *Merck Index* rather quaintly identifies as hexachlorobenzene or "Anticarie; Bunt-cure; Bunt-no-more; Julin's carbon chloride" (C_6Cl_6) (8). Both early (9) and late (10) Faraday had a particular fondness for the element chlorine, a fondness no doubt devolving from his admiration of Davy. In the published version of the paper on the perchloride and protochloride of carbon he justifies at considerable length the reasons for his investigations. Not so in the *Diary* entry for September 1820, which peremptorily begins (11):

Chlorine and olefiant oil exposed in a retort to sun light soon act; the vessel becomes misty, the colour of the chlorine disappears, a little heat is extricated and the bulk of the gas perhaps from that cause appears increased. The gas contains much M.A. [muriatic acid gas, HCl] and there is a smell as of Phosgene gas. (Query oxygen present?)

Dendritical crystals gradually form; these may be washed in water, dissolved in Alcohol and crystallized.

Three months of intense experimentation (involving at least 20 separate runs and multicomponent analyses) were necessary before Faraday felt confident enough to claim (12):

Other experiments gave very nearly the same results; and I have deduced from them, that one volume of olefiant gas requires five volumes of chlorine for its conversion into muriatic acid and chloride of carbon; that four volumes of muriatic acid gas are formed; that three volumes of chlorine combine with the two volumes of carbon in the



Michael Faraday (Etching by McGuire)

EXPERIMENTAL RESEARCHES

IN

CHEMISTRY AND PHYSICS.

BY

MICHAEL FARADAY, D.C.L., F.R.S.,

FULLEBURN PROFESSOR OF CHEMISTRY IN THE ROYAL INSTITUTION OF GREAT BRITAIN.
 HON. MEM. R.S. ED., CAMB. PHIL., AND MED. CHIRURG. SOCC., F.R.S., ORD. BORGARI "POUR
 LE MÉRITE" EQ. COMMANDER OF THE LEGION OF HONOUR, INSTIT. IMP. (ACAD. SC.)
 PARIS. SOCIUS. ACAD. IMP. SC. VIENNAE. ET PETROP., REG. SC. BEROL.,
 PATRIE, ROMAE, MONACI, BRUXEL., ANSTED., BRUXEL., BOGOS., ITAL.
 HUT. SOCC. REG. GOTTING., ET HAFN., UPSAL., HANLEM.
 ACAD. AMER. BOPT., ET SOC. AMER. PHILAD. SOCIUS. ACAD. PANORM. SOCC. GEORG.
 FLORENT., ET PHILOM. PARIS. INSTIT. WASHINGTON., ET ACAD.
 IMP. MED. PARIS CORRESP., ETC.

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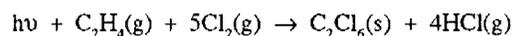
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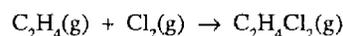
1859.

olefiant gas to form the solid crystalline chloride; and that, when chlorine acts on the fluid compound of chlorine and olefiant gas, for every volume of chlorine that combines, an equal volume of hydrogen is separated.

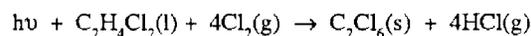
Faraday not only measured in terms of volumes he also reasoned in terms of them. Nonetheless his results translate unambiguously into the modern representation:



He noted that "no muriatic acid gas formed unless chlorine in excess of olefiant gas":



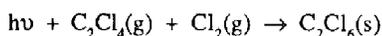
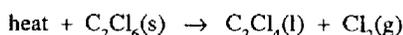
and that this was followed by:



He verified these volumetric arguments by gravimetric determination of the composition of the binary compound: first by converting the carbon to carbon dioxide by heating with

"peroxide of copper" or lime and second by converting the chlorine to silver chloride. In the published paper he cites only "two results from a number of experiments agreeing well with each other". Faraday then gives an indirectly calculated value for the carbon content of the perchloride corresponding to 10.19% (modern value 10.15) and a direct value for the ratio of silver chloride produced to that of perchloride reacted of 3.54 (modern value 3.63). The former agreement may be partly coincidental. Not only did Faraday have to make do with the best available value for the carbon content of his measured volume of carbon dioxide, the *Diary* also reveals much variability with such comments as "Irregular Results do not promise much", "Pretty good but repeat", "Very good expt.", "Too little carbon, too much chloride". The last comment suggests that Faraday, as Kohlrausch was later to remark, "smelt the truth" and thus felt confident in stating that "Three proportions of chlorine 100.5 and Two proportions of carbon 11.4" represented the composition of the perchloride of carbon (13). Allowance must be made for the fact that Faraday's interpretation of his data involved what was tantamount to an atomic weight of approximately six for carbon. It would appear that he rested more confidence in the volumetric than in the gravimetric determination of composition.

The liquid protochloride of carbon was made by passing the perchloride through a hot tube. The reaction was shown to be reversible in sunlight:



Separation was achieved by bulb-to-bulb distillation. Similar experiments to those used to establish the composition of the perchloride enabled Faraday to conclude "the composition of the fluid chloride of carbon to be one proportion of chlorine and one of carbon, or 33.5 of the former, and 5.7 of the latter" (14). Again the *Diary* reveals considerable variability but once again Faraday's "nose" stood him in good stead.

Given the armamentarium of the time, the investigation of chemical and physical properties of the two binary chlorides was wide-ranging: their high temperature interactions with metals, non-metals, and metal oxides; their unreactivity towards acids, bases and, at less than red-heat, oxygen. *Apropos* of the last-mentioned, Faraday notes that when heated with oxygen over mercury "there was no decomposition, or action, until so much mercury had risen in vapour as to aid the oxygen by a kind of double affinity in decomposing the chloride of carbon" (15).

Faraday also reports the addition of iodine to olefiant gas to give a solid, white crystalline body, "having a sweet taste and aromatic smell ... The alcoholic solution is of a very sweet taste, but leaves a peculiarly sharp biting taste on the tongue" (16). The analysis, described in a later note "On Hydriodide of

Carbon" is exemplary (17):

Four grains were passed in vapour over heated copper, in a green glass tube; iodide of copper was formed, and pure olefiant gas evolved, which amounted to 1.37 cubic inch. As 100 cubic inches of olefiant gas weigh about 30.15 grs., so 1.37 cubic inch will weigh 0.413 gr. Now 4 grains minus 0.413 leaves 3.587 iodine, and $3.587 : 0.413 :: 117.75 : 13.55$ nearly. Now 13.55 is so nearly the number of two proportions of olefiant gas, that the substance may be considered as composed of

1 proportion of Iodine 117.75
2 proportions of Olefiant gas . . . 13.4

and is therefore analogous in its constitution to the compound of chlorine and olefiant gas, sometimes called chloric ether.

Faraday found 89.7% of iodine in the compound as compared with the modern value of 90.0% for ethylene di-iodide. In this case exposure of the di-iodide to sunlight caused no further reaction in the presence of excess iodine.

Faraday had established the existence of two chlorocarbons of carbon: the perchloride with three proportions of chlorine and two of carbon and the protochloride with one proportion of chlorine and one of carbon. With impressive pre-homologous logic, he speculated that there should be a third compound containing "two proportions of chlorine and one of carbon" (18). In modern terms, this is carbon tetrachloride, a substance not isolated until 1839 (19).

Chance, however, quickly placed a third perchlorocarbon in Faraday's hands. One "M. Julin, of Abo in Finland", had unaccountably obtained a white solid, seemingly containing only carbon and chlorine, from a process in which "nitric acid is prepared by distilling calcined sulphate of iron with crude nitre in iron retorts". In "On a New Compound of Chlorine and Carbon" (20), Faraday and Phillips establish that this solid contains "one portion of chlorine and two portions of carbon" (21). In 1869 Muller showed it to be hexachlorobenzene.

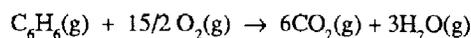
The most celebrated of Faraday's achievements in organic chemistry is his isolation of bicarburet of hydrogen or benzene. This is described, along with the isolation of isobutylene, in "On New Compounds of Carbon and Hydrogen, and On Certain Other Products Obtained During the Decomposition of Oil by Heat" (22). The work started on 26 April 1825 and was pursued with mounting intensity through May and into early June. The "liquor from condensed Oil gas sent to me by Mr. Gordon" was a fiendishly complicated mixture and Faraday spent most of May trying to resolve it into demonstrably pure components. By dint of repeated fractional distillation followed by selective fractional freezing, each stage monitored by analysis, a fairly pure sample of what proved to be bicarburet of hydrogen was obtained. On 25 May, Faraday's *Diary* reports a carbon/hydrogen weight ratio of 11.305. On 4 June,

he finds 11.44, 12.4, and 11.16. Of the second value he notes in the *Diary*: "Must be some mistake here: weight of products surpass original weight." Even so in the published paper he includes this value in calculating a mean value of 11.576. With characteristic intuition Faraday explains away the somewhat low mean value (23):

Now considering that the substance must, according to the manner in which it was prepared, still retain a portion of the body boiling at 186°, but remaining fluid at 0°, and which substance I find, as will be seen hereafter, to contain less carbon than the crystalline compound (only about 8.25 to 1 of hydrogen), it may be admitted, I think, that the constant though small deficit of carbon found in the experiments is due to the portion so retained; and that the crystalline compound would, if pure, yield 12 of carbon for each 1 of hydrogen, or two proportions of the former element and one of the latter.

2 proportionals Carbon 12
1 proportionals Hydrogen 1 } 13 bicarburet of hydrogen

He finds confirmation for his rationale in a vapor density of 40 (H = 1) and in his experimental demonstration of the gaseous volume-ratios corresponding to the modern representation:



It is easy to describe such results but hard to do justice to the experimental skill and perseverance necessary at a time when the classical method of organic analysis was still evolving (24 - 26). In one of the few treatments of "Faraday as a Chemist", William Jackson Pope comes close (27):

He determined the composition of the hydrocarbon by a method so ingenious that it might well tax the skill of the modern worker. He evaporated the hydrocarbon into a known volume of oxygen, noted the increase in gaseous volume, exploded the mixture in the eudiometer and noted the diminution in volume, then treated with caustic potash solution and observed the further diminution in volume due to the removal of the carbon dioxide. The data thus obtained gave the proportion of carbon to hydrogen, and also the density of benzene vapour as compared with hydrogen as the standard; Faraday hence calculated the vapour density as 39, which is the correct value.

Faraday also investigated the chemical properties of "bicarburet of hydrogen" noting its reaction with nitric acid (here Faraday's real nose misled him into suspecting the formation of hydrogen cyanide rather than nitrobenzene). Reaction with sulfuric acid was studied in detail and Faraday was particularly impressed that "no sulphurous acid was formed" as a result of reaction. He was unable to isolate pure benzene sulfonic acid though later he was successful in obtaining barium salts of both the α - and β -isomers of naphthalene sulfonic acid (28). Bicarburet of hydrogen and chlorine gas did not react in the dark but

did so in the presence of sunlight. A solid with "an odour something resembling perchloride of carbon but more resembling artificial camphor" (no doubt *p*-dichlorobenzene) and an unresolved liquid residue (probably largely *o*-dichlorobenzene) resulted (28). A rich and, as it proved, vastly important chemistry was opening up.

From the same oil-gas source Faraday also managed to isolate and largely to purify the most volatile component. Analysis showed that "four volumes or proportionals of hydrogen = 4, are combined with four proportionals of carbon = 24, to form one volume of the vapour, the specific gravity of which would therefore be 28" (29). As Faraday was quick to note "the proportions of the elements in this vapour appear to be the same as in olefiant gas" with its specific gravity of 14 (H = 1). Faraday concluded: "This is a remarkable circumstance, and assists in showing that though the elements are the same, and in the same proportions as in olefiant gas, they are in a very different state of combination" (30). It would be many years before Faraday's discovery could be fully explained. In 1819 Berzelius had called Davy to task for delegating critical analyses to an assistant, in this case the young Michael Faraday (31):

If M. Davy would be so kind as to take the pains of repeating these experiments himself he should be convinced of the fact that when it comes to exact analyses, one should never entrust them into the care of another person; and this is above all a necessary rule to observe when it comes to refuting the works of other chemists who have not shown themselves ignorant of the art of making exact experiments.

Faraday had clearly learned both his lesson and his trade and he must have taken particular pleasure in Berzelius' encomium (32):

One of the most important chemical investigations which has enriched chemistry during 1825 is without doubt that of Faraday on the oily compounds of carbon and hydrogen obtained by compressing the gases obtained by the decomposition of fatty oils.

The paper "On the Mutual Action of Sulphuric Acid and Naphthaline" calls for little further comment (28). From the reaction mixture two new organic acids were isolated in the form of their barium salts. One Faraday dubbed the "flaming salt", the other the "glowing salt". Using "Dr. Prout's newly perfected mercurial trough", Faraday obtained the following remarkable analyses (28):

	<i>Flaming Salt</i>	<i>Glowing Salt</i>
Baryta	27.57 or 78	28.03 or 78
Sulphuric Acid	30.17 or 85.35	29.13 or 81.41
Carbon	41.90 or 118.54	42.40 or 118
Hydrogen	<u>2.877</u> or 8.13	<u>2.66</u> or 7.4
	102.517	102.22

The second and fourth columns "do not differ far from the following theoretical statement" (33):

Baryta	1	proportional	78
Sulphuric Acid	2	proportionals	80
Carbon	20	proportionals	120
Hydrogen	8	proportionals	8

Nowadays we do not write $\text{BaO} \cdot (\text{SO}_3)_2 (\text{C}_{10}\text{H}_8)_2$ for the barium salt of naphthalene sulfonic acid, but once again Faraday's almost unerring nose for the truth is in evidence. Faraday ends his paper with characteristically pragmatic circumspection (34):

As the appropriation of a name to this acid will much facilitate future reference and description, I may perhaps be allowed to suggest that of *sulphonaphthalic acid*, which sufficiently indicates its source and nature without the inconvenience of involving theoretical views.

The last of Faraday's organic researches "On Pure Caoutchouc, and the Substances by which it is Accompanied in the State of Sap or Juice" also had its beginnings in a commercial analysis (35). The fluid provided by a Mr. Hancock was a "pale yellow, thick, creamy looking substance" possessed of a "disagreeable acescent odour, something resembling that of putrescent milk". Bulk analysis yielded (35):

Caoutchouc	317.0
Albuminous precipitate	19.0
Peculiar bitter colouring matter, a highly azotated substance Wax	} 71.3
Substance soluble in water, not in alcohol	29.0
Water, acid, etc.	<u>563.7</u> 1000.0

Of more interest (though not perhaps to Mr. Hancock) was the carbon to hydrogen ratio found for pure caoutchouc. The *Diary* reveals four values: 6.875, 6.582, 7.8(04), and 6.98. The published paper uses the "mean of three best" or 6.812 to 1.000 and concludes that caoutchouc contains "8 proportionals nearly of carbon and 7 of hydrogen". The discarded ratio, 7.8, is in fact closest to that of polyisoprene (7.45). Even Faraday couldn't win them all!

By early 1826 Faraday had completed his researches in organic chemistry and Liebig was in process of setting up shop in Giessen. Faraday's manifest destiny lay elsewhere.

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MICHAEL FARADAY AND THE ART AND SCIENCE OF CHEMICAL MANIPULATION

William B. Jensen, University of Cincinnati

Though a vast secondary literature now exists chronicling the life and achievements of Michael Faraday (figure 1), virtually none of it deals with his only full-length book, *Chemical Manipulation*, first published in 1827 (1). His numerous biographers mention only the fact of its publication, but tell us nothing of its contents and little of the circumstances surrounding its writing. Given the vast amount of important scientific work done by Faraday, this oversight is perhaps understandable. Unlike his famous *Diary* (2), the three volumes of his *Experimental Researches in Electricity* (3), and the companion volume of *Experimental Researches in Chemistry and Physics* (4), *Chemical Manipulation* records no significant scientific discovery. Unlike his famous juvenile lectures on the *Various Forces of Matter* (5) and the *Chemical History of a Candle* (6), or the lesser known *Lectures on the Non-metallic Elements* (7), it lacks accessibility and popular appeal. Yet, as already mentioned, it was the only book explicitly written by Faraday (Table 1) - the volumes of *Experimental Researches* were actually reprints of previously published scientific papers and all three of the juvenile lecture series were transcribed from stenographic notes and edited by others - *Forces of Matter* and the *Chemical History of a Candle* by William Crookes and the *Non-metallic Elements* by John Scoffern.

Nevertheless, it can be argued that *Chemical Manipulation* does merit closer examination, if for no other reason than it gives us valuable insight into the extent of Faraday's training

Table 1. Faraday's books.

<i>Chemical Manipulation</i> , 1827
<i>Six Lectures on the Nonmetallic Elements</i> , 1853
<i>Experimental Researches in Electricity</i> , 3 vols., 1839-1855
<i>Experimental Researches in Chemistry and Physics</i> , 1859
<i>Six Lectures on the Various Forces of Matter</i> , 1860
<i>Six Lectures on the Chemical History of a Candle</i> , 1861



Figure 1. Michael Faraday

as a chemist and the minutiae of the laboratory environment in which he worked on a daily basis. I would like to approach this examination in four stages, starting with an analysis of the origins of the book and the laboratory milieu in which Faraday worked at the Royal Institution, followed by a brief survey of some of the book's predecessors, followed by a survey of its contents, and finally, by a brief look at some of its successors.

Faraday first entered the laboratory of the Royal Institution in the spring of 1813, at age 21, as Humphry Davy's laboratory assistant. After a 19-month leave of absence (October 1813-April 1815) to accompany Davy and his wife on a continental tour, he returned to the Royal Institution as an assistant to William Brande, who had succeeded Davy as Professor of Chemistry after the latter's resignation in 1813. In 1821 Faraday was appointed, at age 29, as "Superintendent of the House and Laboratory" - a promotion which allowed him to marry Sarah Barnard - and in 1825 he became "Director of the Laboratory". It was only in 1834, at age 42, that he was finally appointed Fullerian Professor of Chemistry (8).

The institution in which Faraday found himself had been organized in 1799, largely at the instigation of the American expatriate, Count Rumford, and was located in a remodeled house at 21 Albermarle Street, London (the current front of the building with its stucco pillars was not added until 1838, see page 7 of this issue). As was typical of most laboratory design of the period, the architect in charge of the remodeling placed the chemical laboratory in the basement, where it occupied a position roughly corresponding to that of the original out-