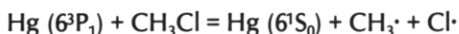


Obituary of C.R. Masson 1922 - 1988

During the decade 1967 - 1976 noteworthy contributions to our knowledge of silicon and sulphur chemistry, to techniques of determining the biosynthesis of natural products and to elucidation of their complex structures were made at the National Research Council's laboratory in Halifax. C.R. Masson, treasurer and long-time member of the Institute, who died on 12 March 1988, played a major role in this extraordinary efflorescence. He was born on 8 September 1922 in Aberdeen and graduated from Aberdeen University in 1943. He pursued graduate studies with H.W. Melville, was awarded a Distiller's graduate scholarship and took up the investigation of molecular weight determinations of polymers by osmometry. This work initially involved modifications of the osmometer designed by Fuoss and Mead (1943), which notably included an effective thermostating device, the use of cellulose membranes biosynthesised by *Acetobacter xylicum* and careful attention to the manipulation of the membranes produced by this organism. The resulting instrument was used to determine the osmotic pressure developed in a number of e.g. toluene solutions of common commercially available polymers and in some cases the values of the Huggins "constant" in these systems. In the cases of samples of polyvinyl acetate in ethyl acetate it was shown that the molecular weights obtained by osmometry agreed well with those calculated from polymerisation kinetics. These results were later elaborated when the molecular weights of a series of vinyl acetate polymers of viscosities in the range 0.5-2.15 were examined by osmometry and by a light scattering method which gave values in general agreement in the range of $1-15 \times 10^5$. The Rayleigh scattering method also provided information on the polymer shapes and this was used to explain discrepancies in the molecular weights obtained by the two methods. Several years later Masson used this experience in studies of the viscosity and degradation kinetics of the naturally occurring sulphated polysaccharides carrageenin, work of considerable contemporary interest in the light of the role these materials play in ulcerative colitis (Marcus, *et al.* 1989).

Free radical chemistry

In 1948 Masson came to Canada to work with E.W.R. Steacie in the National Research Council's laboratory in Ottawa. He became associated with Steacie's long standing interest in the photodecomposition of simple organic compounds in the gas phase with photoexcited ($M(^3P_1)$) mercury, cadmium and zinc species. Steacie and Masson studied the photodecomposition of propane and methyl chloride and in the latter case were able to delineate the stoichiometry of the reaction and to make suggestions for the process at pressures < 100 mm i.e.



and chart the subsequent reactions of the radicals. The discerning reader of these papers will appreciate the amount of work done to produce these results and will note the observation of the sensitivity of $\text{Cl}\cdot$ to the presence of traces of oxygen. Masson's interest in photochemical processes, initiated by lines in the mercury arc spectrum, continued when he moved into Noyes laboratory as the C. & H. Dreyfuss post-doctorate fellow at the University of Rochester, New York. There he studied the photolysis of heptan-3-one in the gas phase and confirmed earlier observations by Bamford and Norrish (1935) that the process $(\text{C}_3\text{H}_7)_2\text{CO} \rightarrow \text{C}_2\text{H}_4 + \text{CH}_3\text{COC}_3\text{H}_7$ occurs. He also demonstrated for the first time that the radical reaction: $(\text{C}_3\text{H}_7)_2\text{CO} = 2\text{C}_3\text{H}_7\cdot + \text{CO}$ is important and he found evidence for 7 other products, mostly from further reactions of $\text{C}_3\text{H}_7\cdot$ many of which he identified. This elucidation of the photochemis-

electrochemical cell was used to follow the kinetics of the dissolution of oxygen into liquid silver, where the rate determining step was shown to be diffusion in the liquid metal. Such kinetic research was taken up by a colleague and continued for many years as an important part of the work of Masson's group.

In his systematic study of gas-metal interactions, Masson first examined the thermodynamic activity of the metal oxide component of MO-SiO_2 melts. One technique for measuring such activities required determining the small (0.001-0.02%) amounts of oxygen that would dissolve in a metal phase in equilibrium with the slag. These techniques involved considerable experimental difficulties and were subject to errors, and Masson carried out a great deal of work to improve the existing methods e.g. vacuum fusion (Sloman, 1959) and isotope dilution (Kirshenbaum and Grosse, 1952) were carefully compared. Analogous investigations into the determination of oxygen in titanium at 1850°C by isotope dilution were reported.

The thermodynamic reference for the activity of FeO was the solubility of oxygen in iron when the latter was in equilibrium with pure FeO . The latter was difficult to obtain — crucibles tend to dissolve in FeO at 1600°C ! Masson developed a levitation method which got rid of the necessity to use a crucible, and then pushed the reference solubility up to the hitherto unapproachable temperature of 1960°C .

Constitution of Metallurgical Slags

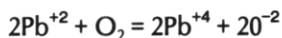
In the 1950's questions were being directed towards the constitution of metallurgical slags in order to rationalize their behaviour as e.g. desulphurizing and dephosphorizing agents towards liquid iron alloys. What was a molten calcium silicate slag like at the molecular level — what were the bonds, what were the aggregations? Masson began to answer some of these questions by applying physical chemical techniques at high temperatures. For example he used the method of depression of freezing point, with molten alkali and alkaline earth fluorides as solvents, to measure the molecular weights of silicate slags. The experiments were difficult, and required ingenious solutions. Masson prepared the purest calcium fluoride then known by the bold expedient of bubbling anhydrous hydrogen fluoride through molten calcium fluoride at 1300°C , the equipment being made of graphite in the hot zone and teflon in the cold.

Another high temperature adaptation of a standard physical chemical method was the use of EMF cells. In the late 1950's Masson used magnesium oxide as an oxygen electrode to measure the oxygen potential of open-hearth slags. This was pioneering work which anticipated by several years the German development of doped zirconium oxide as a thermodynamically acceptable oxygen electrode material. For this work Masson developed an ingenious double-compartment concentration cell slip cast and sintered out of magnesia. This marked the beginning of a line of research in ceramics at the Atlantic Research Laboratory.

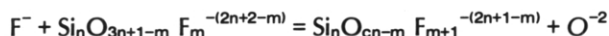
While polymer-ionic theory of slag constitution was being developed, Masson and his collaborators carried out a wide range of experiments to determine the polymerization constants for various systems. These methods depended on finding the thermodynamic activity of the metal oxide component of silicate and silico-phosphate melts. In some experiments slag and metal phases were equilibrated, with the oxygen content of the metal giving a measure of the activity of metal oxide. These experiments were carried out in specially selected and fabricated ceramic crucibles, as well as by levitation.

In other types of experiment an EMF cell was used to measure activities. Both oxygen (doped zirconia) and fluoride (CaF_2) electrolytes were utilized. A great deal of work was done on the lead systems $\text{PbO/SiO}_2/\text{P}_2\text{O}_5$ and $\text{PbO/PbF}_2/\text{SiO}_2$, partly stimulated by the lead-silver development in New Brunswick. This work included activity measurements of plumbous oxide in $\text{PbO/SiO}_2/\text{P}_2\text{O}_5$ solution with up to 12.5

mole % P_2O_5 in the temperature range 1050-1350°C. It was shown that substitution of silica by phosphorus pentoxide reduces the activity of plumbous oxide and leads to increasing the negative partial heats of mixing of the oxide. The solubility and transport of oxygen through and from these melts and evidence that the reaction:



occurred in these slags were also studied in detail. Finally this work was extended to the system $PbO/PbF_2/SiO_2$ and details of the equilibria between the various silicate and fluorosilicate species were delineated to the point that the value of 0.4 ± 0.025 of the equilibrium constant for the reaction series:



was estimated for basic melts. These studies utilized electrochemical methods where the crucible slip-cast from calcium fluoride was used as the solid electrolyte (F^- being solely responsible for conduction) between the $PbO/PbF_2/SiO_2$ and PbO/PbF_2 half cells. In many ways this work paves the way for the preparation of pure compounds of the type $Si_nO_{3n-m}F_{m+1}X_{2n+1-m}$ i.e. a completely novel series of inorganic compounds.

The foregoing sketch of 40 years of scientific effort by Charles Masson does scant justice to his achievement. Belatedly this was recognized by the Alcan award of the Canadian Institute of Mining and Metallurgy and by his election to the Royal Society of Canada. Browsing through his list of publications one is aware of their international flavour. Some of this work was done in laboratories outside the National Research Council and conversely many post-doctorate students of Masson can now be found doing productive work in the United States, Europe and the Far East. Those of us who were his colleagues over a more extended period are aware of our loss and we extend our sympathies to his wife and family.

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