Tritium and Excess Heat Generation during Electrolysis of Aqueous Solutions of Alkali Salts with Nickel Cathode

M. Srinivasan, A. Shyam, T.K. Sankaranarayanan, M.B. Bajpai, H.Ramamurthy, U.K.Mukherjee, M.S.Krishnan, M.G.Nayar and Y.P.Naik

Bhabha Atomic Research Centre
Trombay, Bombay 400 085, India

ABSTRACT

A number of open cell electrolysis experiments of the Mills and Kneizys type using Nickel as cathode, Pt wire as anode and aqueous solutions of carbonates of Potassium, Sodium and Lithium (natural and enriched) as electrolyte have been carried out in three different laboratories at Trombay. The cells were fabricated out of commercial dewar vacuum flasks. The difference in temperature at equilibrium between the operating cells and that of an identical dummy reference flask was measured to deduce excess heat. The cells were calibrated using resistance heaters. In all, studies have been carried out so far in 29 electrolytic cells with various electrolytes. In some cases a mixture of H_2O and D_2O was used. The cells were operated for a few weeks at a time and excess heat up to a maximum of 70% appears to be present in most cells when the input joule power is upto a watt or two. The current density was less than 40 mA/cm^2.

Electrolyte samples before and after electrolysis were analysed for tritium content after microdistillation to eliminate chemiluminiscence effects. Samples from 18 out of 29 experiments analysed have indicated tritium levels varying in the region of 46 Bq/ml to 3390 Bq/ml. One cell with enriched Li_2CO_3 solution in H_2O which was monitored continuously for over a month indicated that tritium generation is continuous. Although the highest amount of tritium produced so far was with a K_2CO_3 in 25% D_2O cell, the generation of tritium in cells containing only H_2O is a new finding.

1. Introduction

Mills and Kneizys were the first to report observation of excess heat in a light water electrolytic cell using a cylindrical cathode made of Nickel sheet, Platinum wire anode and a solution of 0.57 M K_2CO_3 in H_2O as electrolyte. These authors however did not attribute their excess heat results to the occurrence of any nuclear reactions but claimed instead that it was due to the

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formation of "shrunken" hydrogen atoms involving "fractional quantum numbers" (Mills and Kneizys, 1991). The theoretical model of Randell Mills (1989) predicted that excess heat would be obtained with an aqueous solution of K_2CO_3 but not of Na_2CO_3, and they reported (Mills and Kneizys, 1991) that in fact their experiments had confirmed this prediction. They have since reported (Mills and Good, 1992) measuring excess heat margins of several hundred percent, with a scaled up version of their light water cell concept at input joule powers of several tens of watts. Further when intermittent d.c. was applied to the electrodes, the margin of excess heat significantly improved. More importantly they claim that their excess heat observations are much more reproducible than that obtainable with $Pd-D_2O$ cells of the Fleischmann–Pons type (Fleischmann and Pons, 1989).

Shortly after Randell Mills preprint became available, Noninski (1992) announced that he had independently verified their experimental findings. These startling reports of excess heat generation in ordinary water cells appear to have been informally discussed among participants at the Como Cold Fusion meeting of June 1991. Speculating that the source of excess heat in aqueous K_2CO_3 solutions could perhaps be nuclear transmutation reactions of the type

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Robert Bush of California integrated this into his Transmission Resonance Model (TRM) (Bush, 1991) of cold fusion (which he has since begun referring to as Alkali–Hydrogen Fusion) and proceeded to verify his ideas experimentally by setting up Mills type light water cells with Nickel cathodes. The preliminary results of their excess heat and calcium build–up measurements seem to have confirmed his initial conjecture, but with the difference that Bush measured excess heat with solutions of other alkali carbonates also, such as Na_2CO_3 and Rb_2CO_3, besides K_2CO_3 (Bush, 1992). They also reported observing a proportionate increase in calcium concentration in the electrolyte following electrolysis when K_2CO_3 was used and of strontium level when Rb_2CO_3 solution was used, the magnitude of increase being commensurate with the observed excess heat.

It was at this point that we decided to initiate attempts to verify these claims independently in view of its potential importance and significance to the whole field of cold fusion. The first crude cell with a copper constantan thermocouple inserted into the cell and reading to an accuracy of 0.5^{0}C, was set up in January 92 at the Process Instrumentation Systems Division (PISD) of BARC. At the end of January 92 we learnt from Jed Rothwell (Rothwell, 1992) that six other groups in the world had confirmed the generation of excess heat in such $Ni-H_2O$ cells. This communication also contained a recommended protocol for achieving success in such experiments, based on the experience of the successful groups. In March 92 a second group, located at the Purnima Laboratories of BARC began experimenting with cells constructed out of commercial dewar flasks (double walled silvered vacuum jacketed glass dewars). Since preliminary experiments (Shyam, 1992) had indicated that porous Nickel cathodes fabricated by the Desalination Division of BARC appeared to give higher levels of excess heat than rolled Nickel sheets, all experiments since April 92 have mostly used porous Ni as cathode material. In May 92 a third group belonging to the Chemical Engineering Division and located at Hall No. 5 in Trombay joined the search.

2. Description of Cells

All the cells were of the open type with the electrolytic gases escaping through the top plug. Most of the cells were constructed using commercial 300 ml dewar flasks, although some of the early experiments at PISD used a 110 ml flask. The most recent cells set up at Hall–5 employ 500 ml flasks. While the initial experiments used cylindrical geometry for the electrode assembly (1 cm dia x 6 cm long) all subsequent cells have deployed a flat plate geometry (2 cm x 6 cm in size, 0.3 mm thick). The platinum anode which encompassed the Ni cathode was made of 0.15 mm or 0.2 mm dia Pt wire wound over a teflon holder. The lead wires to the electrodes were covered with teflon sleeving upto the top plug so that no portion above the solution level is exposed bare to the electrolytic gases. This is to minimise recombination effects giving rise to errors in calorimetry.

The electrolytic solution was either $\simeq$ 0.57 M K_2CO_3 or 0.57 M Na_2CO_3 or 0.1 M Li_2CO_3. In some experiments carbonates of enriched Li^6 (25% or 54%) were used in place of natural lithium. While most experiments were carried out with H_2O solutions, some runs used a mixture of H_2O and D_2O with the D_2O concentration being 25% or 50% or even 100%. The volumes of the electrolytic solution were 98 ml, 200 ml and 300 ml respectively for the 110 ml, 300 ml and 500 ml flasks.

To speed up data accumulation recent experiments have used banks of five electrolytic cells (flasks) connected in series. A sixth flask served as dummy or reference cell, for the DT (differential temperature between electrolytic cells and the reference cell) measurements. Highly stabilized constant voltage power supplies (0 to 30 V adjustable) with negligible ripple were employed to drive the cells. All cells had a built–in resistor or resistance wire (manganin or nichrome) encased in teflon sleeving or tape besides the Ni–Pt electrode assembly, for calibration purposes.

3. Instrumentation and Technique of Calorimetry

One of the main differences between Pd–D_2O cells and Ni–H_2O cells lies in the current densities involved and hence power inputs dealt with, which in Ni–H_2O cells, are over an order of magnitude lower. This, coupled with the fact that excess heat margins in terms of percentage of joule heat input are much larger here, has a significant bearing on the design of the cells, as well as instrumentation requirements for calorimetry.

The difference in temperature (DT) at steady state between the test electrolytic cell and the dummy cell containing identical volumes of electrolytic solution was taken as a measure of the steady heat generation rate in the test cell. It is well known however that this simple principle of calorimetry is subject to several sources of error which can lead to incorrect results. Some of these are: (a) Role of temperature gradients, if any, within the cell; (b) Effect of fluctuations of ambient temperature; (c) Variation of calibration constant due to changes in solution height in the dewars following evaporation/electrolysis; (d) Errors due to differences, if any, in the heat transfer characteristics between electrolysis runs when bubbles are present and resistance heating runs where no bubbles are present; (e) Inadequate waiting time leading to non attainment of steady state. These and other sources of error arising in calorimetric measurements have been discussed exhaustively in cold fusion literature, ever since the failure of many groups in the world to reproduce the original results of Fleischmann and Pons. However before blindly applying all these arguments to the present experiments, one has to keep in mind the differences between Ni–H_2O cells and Pd–D_2O cells pointed out earlier. In most of the experiments of this paper the input joule power was typically < 2 W.

Amongst the three groups, the best instrumented system for calorimetry at present is that assembled by the PISD group. This has evolved over a period of about 6 months. In the initial series of experiments (designated as X–series) conducted by this group with a 110 ml flask, a 12 point, 0–10 mV range Honeywell recorder (Model Electronik – 15) calibrated for 0–4 mv range (corresponding to 0–100^{0}C for ANSI type T thermocouple) was used to record all parameters, viz. temperature of the electrolytic solution, voltages applied to the cell and heater coil as well as their corresponding currents. The recorder was calibrated using a Keithley 4 $^1/_2$ digit Digital Voltmeter (DVM). For recording currents the voltage drops across two manganin wire resistors were measured. The resistance of these resistors used as standards were in turn determined accurately with the help of a 'decade box'. Manganin wire of 2.2 ohm per meter resistance insulated with teflon was used to make the heaters used for calibration of the cells.

In the X–series of experiments only one thermocouple was used for cell temperature measurement. Room temperature was measured with a calibrated Pt100 RTD (Resistance Temperature Detector). Appropriate correction was applied for the non–linear response of the copper–constantan thermocouples and for cold junction compensation. An accuracy of ± 0.5^{0}C was achieved in differential temperature measurements.

In the second series of experiments with 300 ml flasks currently being conducted by the PISD group (designated as XA–series), a more sophisticated instrumentation system based on a Yokogawa hybrid recorder HR–2500 E (Model 3880), having 30 input channels, 60 channels for computing results and 60 channels for storing constants is being used. In the XA–series each cell has two calibrated ANSI type T thermocouples one immersed upto the bottom of the cell and another dipping just below the surface of the solution, which facilitate monitoring the uniformity of temperature within the solution. It was ensured by this method that no gradient more than ± 0.15^{0}C existed within the solution, inspite of the fact that no stirrer was used. Cold junction compensation for the thermocouples was automatic. The ambient temperature in the perspex enclosure in which the six flasks were housed was measured by means of an RTD. Parameters such as total heat input to cells and DT between average cell temperature and average reference cell temperature (of the two thermocouples) was computed continuously (every 2 seconds) for each cell. Trend recording of the measured parameters such as individual cell/heater voltage as well as the common currents in the electrolysis and heater circuits and computed quantities was carried out at intervals of one minute with a chart speed of 15 mm/hr. The initial and finally stabilised values of all the measured and computed quantities were printed out on the same chart used for trend recording. The accuracies obtained in the various measurements were as follows : For temperatures: ± 0.1^{0}C, for voltages and currents: ± 0.05%.

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PISD (X–series) conducted with the 110 ml flask, reasonable stability appeared to be attained (as evident from the temperature trace on the Honeywell chart recorder) within about 6 to 8 hrs. While these results are still good, there is considerable scatter in these data points and in retrospect we feel it was possibly due to inadequate waiting times. In several experiments both electrolysis and resistance heating was simultaneously carried out, a procedure referred to in literature as "on the fly calibration".

Table I presents a summary of 18 experiments carried out so far wherein the variation of DT with input joule power was studied. The maximum cell current, input power range and other relevant parameters for each experiment are given in the Table. The input joule power is computed as $(V - 1.48) * I$ for H_2O cells and $(V - 1.54) * I$ for D_2O cells and appropriately weighted relations for mixtures of H_2O and D_2O. Figs. 1 to 7 present the calorimetric results in the form of DT vs input joule power (P_j) plots for a few selected experiments of Table I. The least squares fitted quadratic calibration curves pertaining to each of the experiments are also shown plotted. The DT points corresponding to electrolysis runs in all these figures are clearly above the calibration curves indicating the generation of excess heat of 20 to 70% by these cells.

Interestingly, out of all the experiments in which calorimetry has been carried out so far, there is only one cell, namely cell XA–1 (K_2CO_3 in H_2O) which did not show any excess heat at all. This cell was also a Ni–Pt cell similar to others with a flat plate porous Ni cathode but the size of the cathode was slightly smaller being only 2 x 3 cm^2 in area. It was reconstituted from a previously used electrode assembly and not much care appears to have been exercised in handling this electrode assembly. This was the only cell in which the electrolysis and heater calibration points have all fallen on the same curve. In contrast cell XA–2 which was basically a Pt–Pt cell (also K_2CO_3 in H_2O) was expected to yield a single curve showing no excess heat. However surprisingly this cell has also shown some excess heat. Later we learnt that the material of the cathode–mesh used in this cell is not pure Pt but is an alloy of Pt–10% Rh. The anode of XA–2 however was made of standard Pt wire as in all other cells.

The first four rows of Table II summarise the results of some early excess heat measurements carried out at Purnima (Shyam, 1992). Some of these indicated excess heat of 60 to 70% at an input joule power level of $\simeq$ 110 mw.

5. Tritium Level in Electrolytic Solutions

The magnitude of tritium activity in the electrolytic solutions was determined at the end of the electrolysis runs through standard liquid scintillation counting techniques. For this 10 ml samples of the electrolytic solutions were drawn and carefully transferred into clean fresh sample bottles and sent for analysis to the Isotope Division. Microdistillation of every sample was carried out prior to counting to eliminate chemiluminiscence effects. The distillation apparatus was thoroughly washed three times with different reagents and finally rinsed with distilled water and dried to avoid cross contamination. Washed water was counted and the washing process continued until it stopped giving counts. Also the apparatus itself was discarded after use for a few tens of samples. Pipettes, counting vials etc were disposed off after each use. The counting vials were made of glass of low K^{42} content. A digital pipetting system with disposal tips capable of 0.001 ml accuracy was used to measure out exactly 1.000 ml of distilled sample for mixing with 10.0 ml of Dioxane based scintillation cocktail for counting. As a further measure of abundant caution, the vials containing cocktail plus distilled sample were kept in a dark cabinet for several hours for allowing for chemiluminiscence cooling prior to counting.

A Packard Tri–Carb model 3255 liquid scintillation spectrometer was employed and five minute counts were recorded for each sample. A standard tritium sample was also counted with each batch of samples to deduce counting efficiency. Blanks in the form of distilled water, tap water, unelectrolysed alkali solutions etc were also sent to the analysis group in a blind fashion to check for overall reliability of the distillation procedure and counting methodology. It was confirmed that the activity counted is in fact that of tritium and not some other radioactivity, by measuring the pulse height spectrum and comparing it with that of a standard tritium sample. In all over a hundred samples have been distilled and counted for tritium to date. The results of the tritium in electrolyte measurements for the experiments wherein excess heat was investigated systematically are summarised in the last Column of Table I. Table II summarises the tritium data for the other experiments. These data have not been corrected for electrolytic enrichment. This is because the volume reduction of the electrolyte during the whole course of the experiments was less than a factor of 1.5, in most cases. Hence the correction for electrolytic enrichment is quite small.

One point which needs to be commented upon is the apparently high tritium level of the blanks and control samples. This is because of the relatively high background of the counting instrument which is routinely used for counting a variety of samples from different groups in BARC. Even Dioxane based cocktail alone indicated counts equivalent to a few Bq/ml. Hence to be on the conservative side, only values above 10 Bq/ml (twice background value) were regarded as reliable evidence for tritium generation in the present paper.

It is seen that the measured tritium levels in the electrolysed solutions vary from 46 Bq/ml to as much as 3390 Bq/ml. On the whole 15 experiments out of 29 have indicated tritium levels significantly above background values so far. These include K_2CO_3 in 25% D_2O, Li_2CO_3 in 50% D_2O, K_2CO_3 in H_2O as well as Li_2CO_3 in H_2O combinations. Surprisingly most of the natural lithium carbonate in ordinary–water–cells run so far have generated detectable amounts of tritium whereas some enriched Li_2CO_3 in H_2O cells have not. For other combinations also for every successful case there has been atleast one cell with that particular combination which has not yielded tritium. While the maximum amount of tritium generated has been in a K_2CO_3 in 25% D_2O cell (3390 Bq/ml), the second highest (1454 Bq/ml) was with a Li_2CO_3 in H_2O cell. No "magic formula" has emerged for generating high levels of tritium so far. But perhaps the most surprising finding of these studies is that tritium has been generated, possibly for the first time in a light water electrolytic cell.

6. Evidence for Continuous Generation of Tritium

During the series B experiments conducted at Hall 5, samples of electrolyte were collected from the 5 cells of the series and sent for analysis periodically. These results are summarised in Table III. It may be seen that cells B1 and B2 (Li_2CO_3 in H_2O) did not give any tritium throughout. However cells B3, B4 and B5 have indicated tritium in each of the 4 samples taken. Between the 2nd and 3rd sample the cells were operated in reverse electrolysis mode. The cause for the big jump in the tritium level shown by cell B3 in the 4th and final sample is not clear.

In order to study the temporal variation of build–up of tritium during electrolysis systematically, a special cell # OM–3 made of a separating funnel was set up. A porous nickel flat plate electrode assembly, similar to the ones described earlier was used. The evolved electrolytic gases were led into a glass trap containing Pt catalyst for recombination of the gases into H_2O, at room temperature. The re–formed H_2O condensed in the cooler parts. An oil trap isolated this system from the atmosphere. This cell was driven by a pulsed (intermittent d.c) power supply of the type described by Mills and Kneizys (1991). 5 ml samples of electrolyte were drawn once every few days and sent to the Isotope Division for tritium analysis.

In this experiment an attempt was also made to monitor tritium build–up on–line using a system developed by the Environmental Assessment Division of BARC. The on–line monitor comprises of a small scintillation chamber containing solid plastic scintillator fibres in the form of a sponge, with a pair of photomultiplier tubes viewing the chamber and connected to a coincidence circuit. A peristaltic pump was used to continuously circulate the electrolyte through the chamber of the detector assembly. The system was calibrated prior to start–up using Li_2CO_3 solutions containing a known quantity of tritium. An unexpected problem however arose during the investigations with the on–line monitor, in the sense that there was a 'spurious' increase in count rate as soon as the electrolysis was switched on. It appears that some unknown mechanism gives rise to scintillations in the detector assembly when electrolysed alkali carbonate solution is circulated through it. This interference was found to be lesser in the case of 0.1 M Li_2CO_3 electrolyte as compared to 0.57 M K_2CO_3 solutions' presumably because the alkali concentration is lesser in the lithium carbonate case. This was the reason for selecting Li_2CO_3 as electrolyte in cell # OM–3.

Fig.8 shows the continuous build–up of tritium in cell # OM–3 over a period of about 5 weeks. The authors believe that this result presents strong evidence for the continuous occurrence of some nuclear phenomenoa in light water electrolytic cells. The recombined H_2O (about 3 ml) from the electrolyte sample during the first three days of operation of this cell indicated 42.3 Bq/ml of tritium activity .

7. Summary and Conclusions

Measurements of excess heat and tritium production in light water cells of the Mills and Kneizys type have been carried out using Nickel as cathode material, by three different groups at Trombay. The electrolytes used were mostly 0.57 M K_2CO_3 (natural and enriched) dissolved in H_2O or a mixture of H_2O and D_2O. A few experiments were also done with an aqueous solution of 0.57M Na_2CO_3. The current densities employed were < 40 mA/cm2 and input joule powers typically < 2 W. The measured temperature difference (DT) at steady state between a cell undergoing electrolysis and an identical 'reference' cell was compared with the corresponding DT obtained in a calibration run during which a resistance heater immersed in the electrolyte was switched on in place of electrolysis. In all but one of 18 such experiments carried out so far, the DT measured during electrolysis was invariably higher by between 20% and 70% than that measured during corresponding calibration run. Possible differences in heat loss characteristics of a flask with bubbles present, as during electrolysis, and one where no bubbles are present, as in a heater calibration run, cannot be the source of this "excess power". This has been confirmed by measuring the calibration constant several times when electrolysis was underway by simultaneously switching on the resistance heater (so called 'on the fly' calibration). This is also reinforced by the calibration data obtained during reverse electrolysis of several cells.

The other possible source of "excess power" could in principle be partial recombination of H_2 and O_2 in the gas plenum region in the flasks, above the electrolytic solution. For this a few Faraday efficiency measurements were carried out by measuring the total volume of electrolytic gases generated and comparing the same with the expected volume based on ampere–hours or coulombs of charge passed. Such measurements carried out with a simple make shift apparatus indicated a Faraday efficiency of atleast 90%. In this context it is worth noting that if a fraction, f, of the electrolytic gases were to recombine within the flask it could lead to an apparent excess heat of

$$[f * (1.48) / (V - 1.48)] = f/[(V/1.48 - 1)].$$

Hence if the applied voltage is more than 2.96 V, (as in most of our experiments) in order to generate an apparent excess power of say 50% the recombination fraction has to be more than 50%. Thus excess heat margins of 50% or more measured in some of our experiments, particularly in the low input power range, cannot be explained away on the basis of recombination effects.

The quantum of excess power, expressed as a fraction of input power, was in general lower at higher input power levels. The apparent scatter in the electrolysis data points may possibly be due to a "structure" in the excess heat data as observed by Bush (1992) using a flow calorimeter.

The two Na_2CO_3 cells studied by us also appear to have generated some excess heat. This tends to agree with the experimental observations of Bush (1992) but contradicts the results of Mills and Kneizys (1991) as well as Noninski (1992) both of whom have not detected excess heat in Na_2CO_3 experiments.

As for tritium generation only tritium concentrations above a threshold value of 10 Bq/ml have been accepted as reliable in the present paper. In spite of the relatively high background levels involved in the tritium analysis, it can be stated confidently that tritium has been generated in majority (> 50%) of the cells studied by us. Two cells have yielded 4 digit levels of Bq/ml, 8 cells have given 3 digit levels and rest in the 2 digit regime. It is possibly for the first time that significant quantities of tritium is found to have been generated in a light water electrolytic cell.

The results of the B series of experiments with deuterated lithium carbonate solutions (Table III) and the results of the on–line monitor cell # OM–3 (54% enriched $^6Li_2CO_3$ in H_2O) which has operated for more than a month, clearly indicate that tritium generation is continuous in time (see Fig.8). The amount of tritium carried away by/with the electrolytic gases has however not been quantified by us so far. But it is reasonable to assume that it would constitute a non–negligible fraction of the overall quantity of tritium generated.

Since tritium seems to be generated both with K_2CO_3 and Li_2CO_3 with or without the presence of D_2O, we are unable to derive any clue from the results as to the mechanism of tritium generation. There does not appear to be a one–to–one correlation between excess heat and tritium generation. In the experiments where both tritium and excess heat have been measured, the quantity of tritium produced is far below what can be expected, if the same nuclear reactions were to be the cause of excess heat also. Thus, as observed in Pd–D_2O cells of the Fleischmann–Pons type also, tritium generation is obviously a secondary reaction in the context of excess heat production. However the relatively high success rate of detection of tritium in these light water cells clearly signals the occurrence of some nuclear phenomena in them.

The authors agree with other researchers who have experimented with such light water cells using Nickel cathodes, that this type of cells are much less "frustrating" to deal with than Pd–D_2O cells. Also the excess heat results are much more reproducible and success rate fraction higher. Excess heat, if present, is detectable within the first day of electrolysis. However great care needs to be taken in ensuring clean surfaces and avoidance of impurities, to get successful results.

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