AUSTRALIAN NATIONAL UNIVERSITY DEPARTMENT OF NUCLEAR PHYSICS 14UD TANK OPENING REPORT No.37

7 January 1983

PROLOGUE:

This report was begun in the middle of October 1982, following a chain break. From then on another chain break led us to undertake a series of measures in order to understand and clear up the problems with our insulating gas. As a result there were several tank openings and significantly long periods when the 14UD was out of commission.

Report No.37 had been written, and typed, up to the start of the second opening when the whole situation developed in complexity and uncertainty. The text up to that point has not been changed, but the information which follows it covers a long time, including extensive periods of gas cleaning. While we have tried to maintain the usual sequential format of these reports we have yielded to the need for clarity and fitted in the information on breakdown products where it makes most sense.

In the middle of October, Trevor Ophel and Alan Cooper began a private crusade against the evil things which affront the purity of insulating gas. Following the chain break of November 1st this crusade was stepped up vigorously. The alchemy we quote is drawn, in most part, from Trevor's rough notes and inserted into the report at relevant places.

Four Openings

5th to 8th October 1982 (4 days open).

1st November 1982 (1 day open).

16th to 18th November 1982 (3 days open).

29th November to 7th December 1982 (8 days open; 6 working days).

REFERENCES: Earlier Tank Opening Reports are referred to by the notation (12/4) etc, meaning Report No. 12, page 4.

REASON FOR THE FIRST TWO TANK OPENINGS

Chain breaks.

PREAMBLE

The 14UD was last closed on 26th August. It ran for much of the time in the region of 14 MV.

There were spasms of lost charge which would appear and disappear accordin to the whim of the machine. Towards the end of September the University held Open Days for the public to visit and so the 14UD was off the air. We used the opportunity to do some conditioning and a few tests relating to lost charge. These tests constantly showed that the phenomenon was inconsistent and therefore was due to movable particulate material on or near the triode mushroom.

On Friday, 1st October, the machine was behaving erratically while running near 14 MV. The current on Chain 2 was varying by plus or minus 2 microamps and so the chain was turned off as a diagnostic. A minute or so later, while we were watching performance, there was a 14 MV tank spark. Upon recovery this was followed by another at 14 MV which caused current to be drawn from both charging and suppressing supplies, a standard indication of a chain break. Tests with the charging cables soon confirmed the diagnosis. We had new chains in positions 1 and 2. The chain in position 3 had only been in for 7 months but this had been a period of virulent breakdown products during which three During this period as well, 7 nylon links were removed from Chain chains died. 3 due to breakdown product induced cracks, (34/1; 35/10; 36/3). The temptation to confirm that the broken chain was the old one and not one of the new ones. was too much for both authors. It was agreed knowingly to commit a sin; but briefly, mind! We ran Motor 3 and it appeared to be running free. Not satisfied we just started Chain 2 motor and there was a horrible scraping noise. We switched off hurriedly and left it at that.

Since it was mid-morning on a Friday preceding a holiday weekend, no-one wanted a tank opening; moreover, some people had already departed on leave. We

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decided to spend the rest of the day pumping the tank down to slightly above atomspheric pressure. The tank was left at this pressure until the following Tuesday, when the rest of the gas was taken out and the doors opened.

OPERATIONAL TIME

During the 36 days since the last closure, the 14UD operated for 628 hours. This was 91% of elapsed time, excluding the days for gas transfer, and 3 open days, plus a day of preparation for them.

THE FIRST TANK OPENING

Exploratory tour.

Neither author was present at the moment of opening the doors and the initial odour in the tank was assessed by trainee sniffers on their first solo. Reeling aghast from the door they declared that the smell was very bad. When the old, more finely tuned noses arrived, and inhaled deeply, it was pronounced by their owners that the smell didn't seem all that bad at all; moreover it cleared much more quickly than of recent times.

Chain 3 was lying in the bottom of the tank. One of its inductors had been thrown past Chain 2 so that the phosphor bronze connection wire was lying between the chain and pulley. The wire had been badly chewed and the pulley rim and shimstock were damaged. We are confident that, if Chain 2 had been running at the time of the Chain 3 break, Chain 2 would have broken also. As it happened, running Chain 2 as a diagnostic after the break did cause real damage. It was the nearest we have come to breaking an innocent chain; Golden Rule number 137 must stand in future: never run any chains after a chain break.

We observed, for the first time, blackening at the cracks in nylon links which had broken. One link had fragmented and when the pieces were collected it appeared that the link had virtually exploded. We believe that the 14 MV spark had in some way "found" a crack caused by past breakdown product attack in the nylon link. The effect was to blow it apart at the crack during a terminal spark.

We discovered that, at the last button-up, a contoured casting cover (14/2) had been put on a wrong casting by mistake; casting 17, which should have had a contoured cover, had one of the flat original ones. When examining the covers we found that several had suffered damage through being dropped, or having others dropped on them. Sharp ridges had been raised on some covers, mostly at corners where pairs butt together. There were distinct spark marks on the equipotential rings near the cover gaps.

These mistakes and damage occurred when all casting covers were taken out of the machine at the last opening in order to rid the column of plating flakes from the failure of the shiny chains. Not enough care was taken with the covers and it became yet again clear just how deleterious sharp protuberances can be; likewise, large and misaligned gaps where covers are not closed properly, can generate sparking.

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The mandatory ritual of meticulous chain examination, link by link, was carried out in the terminal by the usual experts. On this occasion, their search disclosed two instances of bubbles in the nylon links of Chain 1, a new chain put in last opening (photograph). Since these two experts appear to find every new chain fault almost as soon as it develops, we felt that their arrangement of a double fibre optics light pipe, their working position, and, indeed, they themselves, were worth a photograph.

All surfaces in the tank were very dusty. The origin of this loose, household dust-like material is still unknown. The participation of this material in the intermittent lost charge phenomena had been predicted. The patch of breakdown products on the terminal, opposite the corona triode, was much thinner than in the past, encouraging us to believe that the recirculation purification has been improved.

And so to work!

Foils

The terminal foils were changed.

Shaft bearings

All the shaft bearings were listened to and held to be acceptable.

Points

A needle was missing from the H.E. column metering assembly. The assembly was repaired.

Chains

The two links with bubbles were taken out of Chain 1; Chain 2 was shortened by two links. Chain 3, (the one which broke) was very carefully examined and a link with a bubble was found. This suggests that voids in the nylon links have been occurring for some time. Our Chain Gang insist that they have seen bubbles before, but have been told to ignore them. It is regretted that this might be the case. We have no direct evidence that any of our many chain breaks has been precipitated by bubbles in the nylon. Nevertheless, such flaws worry one that past breaks could have been, or future ones might be, caused by such faults in nylon links.

Idlers

One bad bearing was found, and the assembly was replaced.

Breakdown product monitoring

Nylon cable ties, put in the bottom of the tank at the last opening (36/4), were examined. One was wrenched and found to be strong, and not brittle. It was then deliberately broken and removed. A "self-destructed" cable tie was found on the floor. It had been in the tank for a year, having been put in to hold metering leads on 27th October 1981 when it was clamped with only medium tension. It serves as an example of the behaviour of these devices in the tank environment.

We kept an eye on the cable ties we put under high vacuum in a test chamber fitted with a window, (36/4). None had broken in the 10 weeks that they had been there. However, without a mechanical test, we had no way of telling if they were being weakened. We opened the chamber and applied the standard "wrench" to two of the ties to test brittleness. They broke more easily than ties which have been in general use around the lab, or attached in places merely to observe their behaviour. One was a bit tougher than the other. The weaker broke with little or no more effort than had been required to break several in the tank which we then proceeded to report as suffering from exposure to tank atmosphere. While it is clear that nylon can be weakened by a simple drying out process, we are by no means suggesting that the result exonerates breakdown products. It is possible, of course, that vacuum might pump away other substances than water. In addition to these measures we began investigating means of monitoring the condition of the SF₆ insulating gas. A system was devised to detect breakdown products by measuring changes in the electrical conductivity of boric acid after passing contaminated SF₆ through it. Exceptional precautions were taken, i.e. teflon vessels and plumbing, with a good mixing system; controlled flow rate, temperature etc. The performance was tested by running a single corona point in clean SF₆. For 30 kV the corona current was 100 microamps. This produced a change in resistance at the rate of about 70 kilohms/hr (see appendix).

MISCELLANEOUS

SF6 Recirculation and Purification Apparatus.

A preheater was installed on the gas dryer reactivation line; also we fitted a Shaw dewpoint sensor following the activated alumina tower in the recirculator loop. Before the alumina was reactivated with nitrogen the reading was 40 p.p.m. and after recirculation 1 p.p.m.

Lost Charge

The candidates for blame for the intermittent lost charge syndrome were some scaly deposit on the triode mushroom and the dust on all surfaces. There was a stain inside the mushroom, presumably caused by water standing there, having been introduced when the mushroom was previously cleaned in situ. The whole triode apparatus was disassembled, cleaned and polished. New needles were fitted. In the past, thick deposits from the terminal have been sparked through, throwing out flakes which caused fluctuating lost charge. On this occasion, the terminal stain was so light as not even to warrant our usual photograph. We take this to be evidence of an improved breakdown product situation.

Cleaning

Because of the prevalence of loose dust, the entire inside wall of the tank was wiped with tacrags. Judging by the condition of the tacrags, and the students who used them, a lot of dirt was removed from the tank. This effort took 5 students 45 minutes. That these minutes immediately preceded the Friday evening conference seems to enhance their effectiveness.

Button-up

The charging tests went excellently and we closed the machine with only two chains, Nos. 1 and 2.

Initial performance

The conditioning level of the machine quickly reached 13.94 MV and it was

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used at whatever voltage the experimenters required up to this level. A fortnight after button-up the fluctuating lost charge phenomenon recurred. Up to 50 microamps of lost charge coexisted with a triode position producing 40 microamps of triode current at 13.3 MV, 90 p.s.i.a. SF₆. Withdrawal of the triode needles dissipated the lost charge. Turning the machine off and fully withdrawing the triode needles banished the lost charge for a few hours. This behaviour is consistent with loose particulate material. The source of such material is still unknown.

It can be seen from the enclosed plot of terminal voltage that for most of the time the 14UD operated above 13.5 MV, with good spells at 14 MV when required.

Though the instabilities were irritating, performance remained good enough to continue with.

On Friday, 29th October, the breakdown product detector was connected directly to the tank. The first measurement on actual gas in the accelerator during use was concluded just 16 hours before the 1017 hours of service of Chain 2 ended with a bang, not a whimper. We had one measurement only: the resistance had fallen from 94 kilohms to 29.7 kilohms in 5½ hours. To make further runs while the gas was still in the accelerator would have delayed pump-out beyond what we could tolerate.

Performance since the closure on 8th October had been as follows: during the 22 days the 14UD operated for 389 hours. This was 81% of elapsed time, excluding days for gas transfer.

THE SECOND OPENING (1st November).

The tank was pumped out on Sunday by the two chain experts who had the doors opened by 11.30 p.m. They said, as they had said at the previous opening, that the smell from the tank once ventilation was begun, was very pungent and thoroughly unpleasant. They made no attempt to enter the tank that night. Next morning Chain 2 was taken out of the tank and examined in the usual manner About 20 cracks were found in the nylon links of the first half of the chain and we didn't bother with the other half. There was no evidence that the break had occurred from any cause other than the weakening of the links. Chain 1 was then examined in situ, and when about 20 cracks had been found it also was taken out of the machine.

There were minor problems with stabilizing idlers but no evidence that the chain had broken because of mechanical intervention by displaced idlers or inductors.

While the situation had obviously been very serious for a long time it had now become disastrous. Two essentially new chains were riddled with cracks which we could not persuasively attribute to anything other than breakdown products. We had one new chain on hand, but the last place to think of putting a new chain was in the tank. Finally we adopted Robert Rathmell's advice of March 1982. Our intention was to try to purify the SF₆ without chains present.

The decision was made to close the machine exactly as it was, with rings off, terminal lowered, chainless and even untacragged. While the tank was roughed, the new breakdown product detecting system referred to on page 5, was transferred to the gas handling area and gas was sampled from the top of the storage vessel where all the inventory was stored. This tested positive for breakdown products too.

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We are well aware that our breakdown product detecting system is as yet uncalibrated. We are not yet able to relate our measurements quantitatively to SF_6 dissociation by either corona discharge or total discharge of the column by massive tank sparks. We have, nevertheless, demonstrated consistently that the device can tell distinctly the difference between uncontaminated SF_6 and samples from the accelerator or gas storage which obviously contain breakdown products. A description of the conductivity cell is given in the Appendix of this report.

The machine remained closed for two weeks, all of which time was dedicated to improving the condition of the gas by recirculating it through a purifying agent, Vivalyme, a trade name for soda lime supplied by C.I.G. A window was put on top of the dryer tower so that we could study the condition and colour of the Vivalyme. The gas was recirculated continually and monitoring by conductivity measurements was kept up at various locations. In parallel with our measurements, gas samples were taken to the Research School of Chemistry for gas chromatography.

In order to leave no stone unturned, or lid unlifted, all the gas was put into the accelerator and the storage vessel opened to study the condition of the steel samples which we had sandblasted clean and left in the vessel to observe rusting. We found the steel whistle-clean, without a fleck of rust anywhere; a very unexpected attack on the passivation hypothesis.

At the end of two weeks the breakdown product detector indicated that the gas was pure and it was taken out of the tank so that we could put in a chain and observe the effects of running the machine by measurements with the conductivity cell. During the pumpout the H.E. vacuum improved significantly, implying that there was a leak somewhere in the tank.

THE THIRD OPENING (16th - 18th November).

Considerable time was spent trying to pinpoint the leak in the H.E. tube which was traced to the tube section immediately above the second stripper. The responses were such that we concluded the tube section had become porous. Radiation has been attributed to be the cause of porosity in a tube section in the Argonne Dynamitron, and the particular tube section of ours, being adjacent to the second stripper, is in a high radiation region. After application of Glyptal in a variety of spots got us nowhere, the entire ceramic area of the tube section was painted with Glyptal. This expedient closed the leak and led us to believe the porosity theory.

We put a makeshift chain in No.1 position. Most of it was from the chain we had just taken out, the remainder was from the last chain that broke.

After gassing up the machine ran for a full day, and then for several hours on two other days. Gas testing continued. Running the machine, even with only one chain at middle voltages, with SF_6 purified by untreated Vivalyme, was still producing trouble. The lost charge increased to about 20 microamps and the charging efficiency of the chain deteriorated. During this period, the hygrometer indicated a rise in humidity. The calibration of this Shaw hygrometer appeared to be unreliable, probably because the sensor had been exposed to the dryer effluent, rich in nasties.

On November 25th we replaced the full charge of Vivalyme in the dryer with a fresh lot and the machine ran for a number of hours. In the evening there

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were problems with charging and holding voltage. The next day, Friday, the new Vivalyme was removed and replaced with the same alumina taken out earlier in November, but now vacuum reactivated. The problems continued. We looked in the various viewing ports but couldn't see any telltale sparks or lights inside the tank. Whatever was wrong was artful, sly and invisible. No charge was getting to the terminal. We left the machine idle over the weekend and took the gas out on Monday, 29th November.

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THE FOURTH OPENING (29th November to 7th December).

When the doors were opened, we noticed immediately that there was a thin coating of rust coloured material on the floor of the upper doorway where the steel is burnished smooth and clean by the seats of countless trousers. On the platform, the unpainted steel jacks, with which the terminal spinnings are moved, were rusted in appearance too. These jacks, sandblasted clean when they were made two and a half years ago (21/3), have remained in the tank all the time without ever exhibiting signs of rust. (The rust found in the tank should be remembered in terms of the statement on page 7 describing the spotless condition of steel in the storage vessel early in November.) Subsequent tests have shown this material to contain significant iron fluoride and not to be consistent with iron oxide.

To see if the charging problems could be located a charging test was carried out on Chain 1. It performed better than at its pre-buttonup test and we had to look elsewhere for a solution. We had no doubt that our electrical problems had been due to wet gas. The chain was taken out of the machine and examined in the workshop for defective nylon links. Three were found and replaced.

Continuing the examination inside the tank we found a corroded deposit on the tube flanges and also the aluminium "stand-offs" on the tube flange corona point assemblies. Taking a good, hard look at the deposits on the tube and column corona assemblies we decided to remove every assembly and clean them all outside the machine. (We wished to rid the inside of the machine of all unsavoury deposits and also to start afresh with clean corona assemblies so that we could note onset of any new deposits.) The removal of the assemblies enabled us to clean the tube flanges much more thoroughly. At this time, it was noticed that there was a white, crystalline deposit on the tube spark gaps adjacent to the point assemblies (see photograph). All this was cleaned with wipers, merely damp with water. The long weary effort of taking out all the Corona assemblies was performed in the most cheerful manner by our oft-mentioned students. Not only did they remove the assemblies, they were invited to join the older author at table where, for two days, they soaked points in water, scrubbed them with a toothbrush and washed them in alcohol, indulging in merry banter all the while.

The clean points were stuck on foam 'slabs' and set out in the sun to dry like sultanas.

Investigating problems we had experienced with the terminal lens, we found that the 40 megohm series resistors in two of the four supplies were open circuit. Troubles in the past with these helical film resistors led us to replace all four with four 40 megohm Welwyns in series-parallel.

The accelerator tube was let up to atmospheric pressure in order that we could remove and repair the H.E. ball valve. We took the opportunity to take

out the second stripper and check its alignment by the beam spot and renew foils as necessary. The beam spot was barely contained on the foils and so a slight adjustment was necessary.

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Also while the tube was open, we took a look at the sublimers in the L.E. mid-section pump; they were well used, but not wrecked. Both were renewed.

The machine was eventually closed with the same makeshift chain in No.1 position and the other positions were left empty. We had no intention of putting in any of the new chains we already had on hand.

Because of the "rust" found in the machine, all the SF₆ was put into the accelerator on 8th December and the following day the storage vessel was opened. The clean steelwork found "rustfree" at the last opening (3rd November) was now thickly coated with rust. Most interesting of all was the discovery of curious blisters, many already burst, attached to the steel samples like barnacles on a boat (photograph). We observed that the barnacles were most numerous on areas of steel which would have been longest under the liquid in storage. When scraped off with sufficient care to ensure that they did not mix with scale, the barnacles were found to be magnetic. On the floor of the vessel was loose "rust" scale and dust which, when swept up and weighed, amounted to 600 grams.

The storage vessel was swept out, vacuum cleaned and closed again with a new piece of freshly sandblasted steel lying inside.

The machine went into use between 8.5 and 12 MV, during which time the gas was monitored.

On 15th December the alumina was removed from the dryer; the charge with which it was replaced consisted of 50% new Vivalyme on top of which was laid the same quantity of the alumina just removed. The combination was dried by purging with nitrogen heated to 200 deg. C for 4 hours, after which the purifier was evacuated by a rotary pump for 19 hours.

Operating at voltages up to 12.8 MV on the one chain the machine continued in use until Christmas Eve when it was rested until the new year. On 3rd January more tests were made with the conductivity cell before starting up. All was well and normal operation was resumed.

In the near future the new chains, already on hand, will be put in.

Some cheerful news!

We have heard from DSIR in New Zealand that the EN Tandem, which began its life in this department, has entered a new phase. The column was put in during July 1982 in the remarkably short space of five days. Voltage tests were to be carried out on the column before installation of the tube.

The Tandem arrived in Canberra in October 1960 and performed splendidly until its use faded with the availability of the 14UD. We congratulate DSIR and express our pleasure that this accelerator, which we hold in affection, should be thriving again.

D.C. Weisser

T.A. Brinkley

5th January 1983

APPENDIX follows.

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APPENDIX

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Breakdown Product Detector.

In principle, one is concerned about acidic breakdown products of SF_6 as these would be expected to attack nylon. To the naive, HF would seem an obvious possible culprit and indeed, a drop of concentrated HF will eat into a nylon chain link. However conjectural the connection between breakdown products and chain breakages, we chose to try to measure hydrolyzable compounds present in our tank gas.

The work of J.M. Kellor at Oak Ridge alerted us to the problems of devising a simple cheap apparatus which didn't contaminate the sample. This challenge was met by making an atomizer entirely out of teflon which efficiently mixes the sample gas with the detecting liquid, boric acid. The fog produced is condensed on a plug, made of teflon turnings, at the top of the teflon cylinder. The resistivity of the liquid is measured using platinum electrodes and a.c. voltages. See diagrams. When pure, bottled SF_6 is flowed through the device, at 1.4 litres/minute, the resistance remains constant to about 2% at about 100 kilohms for two hours. Tank gas will produce changes of the order of 50 kilohms/hr. The apparatus has been used to monitor the purity of the gas after passing through the recirculation purifier. Our nine months old alumina, after vacuum reactivation at 400F, could not entirely purify the gas presented to it at moderate accelerator conditions, viz. 12 MV, 75 microamps charging current.

Using undried Vivalyme is also unsatisfactory, not necessarily because its purifying power is lessened, but because the SF_6 gets so wet that the accelerator will not work.

Intermingled with the measurements on the machine, offline tests were performed of the purifying power of Vivalyme and activated alumina. The breakdown products were produced in a stainless steel cell at 50 psig SF₆, 30 kV and 100 microamps corona current from a single point. Undried Vivalyme, vacuum dried Vivalyme and virgin alumina removed all the breakdown products produced by the cell over two hours. Our nine months old vacuum activated alumina was inferior. This performance was confirmed with the accelerator experience.

Based on this experience an interim arrangement was chosen using 30 kilograms of Vivalyme loaded on top of about 50 kilograms of used alumina. This layered combination was dried by purging with dry nitrogen preheated to 200 deg. C. for 4 hours. For the next 19 hours, the purifier was evacuated by a rotary pump.

The condition of the gas was monitored frequently while the experimental program continued. The gas from the purifier remained pure while that in the tank showed contamination corresponding to a resistance drop of about 50 kilohms/hr. At the moment we have no way of knowing whether such a rate is normal for our running conditions, or reflects an enhanced production situation. All we can be sure of is that the purifier is working at 100% efficiency for its 45 SCFM.

Duplicate apparatus is being constructed and will be sent to N.E.C. in the hope that they can test the SF_6 in other machines to help us to establish a norm. Perhaps some of the friends on our circulation list will invite N.E.C. to sample their gas using this apparatus. It will have been noted by now that the momentum of our Chain Break Survey has waned substantially since the recent plague of breaks led us to the present investigations. This, we feel, will be excused for the time being.

Enclosures:

Plots of particle masses accelerated, and operating terminal voltages.

NOTE: On the plot of terminal voltages we have drawn a horizontal line at 14 MV for easy reference to performance near the nominal voltage limit of the 14UD.

Photographs:

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- 1. Bubble in a nylon link.
- 2. Another bubble in another nylon link.
- 3. The experts examining Chain 1 in the terminal.
- 4. Rust and "barnacles" on steel from the storage vessel.
- 5. Deposit on tube flanges.
- 6. Photograph of the conductivity cell.
- 7. Diagram of conductivity cell.
- 8. Diagram of breakdown product detection equipment.

