Touchpaper
Extract
Spring 2014
Article on
Dinitrogen
Pentoxide
Greville Bagg

Post-war Work at Waltham Abbey

Some time ago whilst taking regular lunchtime refreshment in the Crown pub in Waltham Abbey, Brian Howard told me that he was interested in gathering together information on the work of Waltham Abbey during its post-war era. During my second spell there between 1988 and 1991, one of the pioneering pieces of work being undertaken was on the exploitation of the properties of dinitrogen pentoxide as a nitrating agent. This prompted me to ask Greville Bagg, the leader of the team carrying out the work, whether he had retained any of the several articles written on the subject. The piece below is one such example written in 1990 and published in the ICI house magazine "Nobel Times" on August 7th 1991. It illustrates the ground-breaking work undertaken right up until site closure at Waltham Abbey and in this case successfully transferred to Fort Halstead.

Geoff Hooper

What is Dinitrogen Pentoxide, and Why?

Some ten years ago, a brilliant polymer chemist by the name of Dr Rick Richards, conceived a plan for getting his beloved macro-molecules recognised by his explosive orientated colleagues at Waltham Abbey. He would try and build energetic groups into the molecular chain to produce a thermoset rubbery form of explosive. To do this he would need to find a source of dinitrogen pentoxide which should, because of its nitronium ion, be able to nitrate selective groups on chosen organic compounds without damaging their structure. In 1981, an item of intelligence on possible N_20_5 synthesis was obtained and forwarded to Dr Richards by our man in the US Embassy, a certain Dr Geoff Hooper at that time. This proved to be the tool by which his dream became reality, but unfortunately too late for Dr

Richards died in 1986 before the work was completed. We dedicate this work to his enthusiasm and in memory of the many happy hours spent exchanging N_20_5 chemistry on the back of many beer mats.

Dinitrogen pentoxide, or N_20_5 to give its molecular formula, is a white crystalline solid that will decompose, given half a chance, into lots of brown nitrogen dioxide fumes or pick up moisture to form very concentrated nitric acid. It is also extremely reactive in solution and will rapidly eat its way through gloves, skin, plastics or any other organic material it contacts to give a whole variety of unexpected nitrates. To the chemist, this novel feature provides an exciting mechanism for creating a brand new range of more useful chemicals, especially in the defence field of energetic materials. To the chemical engineer however, this aggressive property is a bit of a nuisance to say the least, since it makes it rather difficult both to manufacture and keep it uncontaminated in the right place long enough for it to be of any commercial use.

And therein lies the nub of the problem, a chemist's dream and a chemical engineer's nightmare. Here we have a brand new chemical reagent tool to help put theoretical synthesis into practice, but only if it can be demonstrated that the N_20_5 can be made and handled in bulk at an economic cost with respect to the predicted end products. Well it has taken us over seven years to do just that and we are now at the point of being the first establishment in the world to drive N_20_5 into commercial production for the manufacture of the next generation of energetic defence materials, the first new range of energetic polymer and crystalline explosives for 50 years.

Given a quantity of N_20_5, it is possible to do everything that you can do with nitric acid and a considerable amount more. Not that it would be necessarily desirable to do so because it will always cost more than the nitric acid from which it can be made.

It is an easy step to deduce that, although its potential as a nitrating agent is as yet largely unexplored, it could also be used to improve upon known reactions involving concentrated nitric acid or mixed acid systems - provided the price is right.

It will perhaps need the initial and urgent drive of high value defence orientated end products to kick it off before lower value civil products can gain a share as the production costs are brought down with increasing scale.

An interesting analogy can be found with the introduction of the ammonia oxidation process for nitric acid circa 1930, for the driving force in this case stemmed from a need to improve explosives production. It was not until the late 1950's and the petroleum boom, did civil industry take over and start exploiting its real value. The potential usage of N_20_5 could account for a significant (albeit small) proportion of the apparent 3,000 million tonnes production capability of nitric acid manufacturing plant in the UK alone. A bit of optimism easily converts this possibility into probable profitable patent royalties sometime during the next decade.

Our researches at Waltham Abbey began in earnest in 1982 and by 1985 we had obtained an estimate of the exciting potential, but as yet unproven, value of end nitrated products as well as determining two possible synthesis routes for the raw reagent. It took a further two years before the production capability for N_20_5 was able to be demonstrated and assessed, and to draw up a short list of the technical problems impeding growth of the overall research and development programme. The key problem turned out to be the perennial one of availability of suitable equipment and materials compatible with this very reactive new reagent. We could certainly make N_20_5 in quantity at a competitive cost but it still leaked through seals on pumps and valves etc to corrode the exterior of plant equipment every few weeks, (most “acid-proof” pumps and valves appear to be held together with corrodible springs and brass nuts!).

At this stage we turned to industry for help in developing and exploiting the processes. We found that the only companies willing to engage in expensive development were those whose businesses centred on marketing plant and processes, and who were therefore in a position to exploit any new technology at the earliest opportunity. Companies who were potential end users of the N_20_5, both in the defence and civil sectors, considered the project too speculative and could not support the long lead times before profitability, i.e. they needed a higher level of demonstration before committing themselves financially.

It took a further three years negotiating with various companies before the overall programme took shape, first signing Confidentiality Agreements which allowed us to control the flow and use of valuable information as well as giving us access to their marketing databases. Finally drawing up License Agreements to cover exploitation. This broadening of our outlook triggered further work and we have since been able to complete most of our patent jigsaw on all the various stages wherein the N_20_5 could be produced, refined and used according to a variety of different techniques.

One process patent on its own is usually fairly weak since the licensee will almost certainly add improvements during the course of development. But a collection can be stronger than the sum of components since each extends the power of negotiation as the licensee is drawn along the path of commitment.

Now, some seven years into the project and three years into commercial liaison, the companies concerned have verified our original research and demonstration work to their satisfaction and have consequentially gone public with their marketing of the technology.

ICI Explosives Ltd, (newly formed to include Nobel's at Ardeer), have announced their intention to complete a new HMX pilot plant by the end of the year which will use N_20_5 in nitric acid as the nitrating agent and incorporate the ICI C&P pilot plant. This will be the world's first demonstrator process and should trigger off wider exploitation. ICI Explosives have also announced their commitment to the RARDE energetic binders programme and they will be open to commercial orders on the first product; polynimmo towards the end of 1990.

It will probably take the full ten years before the project shows the beginning of any financial returns on its investment by 1992, and another five years before this reagent begins to find wider commercial usage in the civil sector. Perhaps one of the arts of financing research lies in predicting what the customer will be needing in the future whilst exploiting past successes to support the present. Another maybe could be in insuring that there is sufficient development lag before actual profit, thereby avoiding the criticism of undertaking 'near market research' with its all too close a connection with privatisation?

Meanwhile our research work has only just begun, there are more associated processes to demonstrate before commercial license and development, and even more defence products to make in quantities sufficient for primary evaluation. And if we don't get there first, someone else will!

Greville Bagg
Chemical Process Research, NP4
7th February 1990

Touring the Fort Halstead laboratories after the signing of the agreement were Richard Lutchford (DRA), Stephen Morris, Gerald Byrne, John Lynch (vice-president and A&A general manager, ICI Explosives), Roger Warren (managing director, military division DRA), Geoff Hooper (DRA), Ian Kirby and Greville Bagg (DRA).