WASC 2356
WA1 658

From Touchpaper
Autumn 2014

Article by John Rawlen

'Propellant Stability Work in 2145 and 4110'

Propellant stability work in L145 and H10

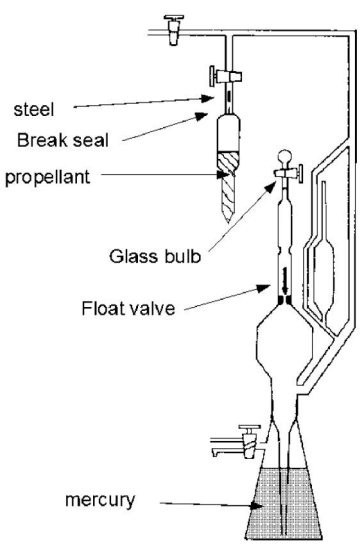
Firstly a bit of background for anyone unfamiliar with stability. All nitrocellulose/nitroglycerine based solid propellants whether for guns or rockets undergo slow decomposition at room temperatures, stabilisers are added to mop up the nitrogen oxides released as these have a catalytic effect on the decomposition (ref 1 which is on the internet). The stabilisers included substances such as methylcentralite or nitrodiphenylamine.

Propellants were heated at various temperatures and times and we had to extract the stabilisers to determine how much was left and extrapolate back to storage temperatures to work out a storage life. When I arrived at ERDE in 1973 fresh from university and started working in L145, methods were antiquated compared to now but were standard methods for the time.

I remember analyses were undertaken using thin layer chromatography. These were about 20cm square glass plates coated with a silica slurry then dried. The propellant extract was spotted onto the bottom of the plate, dipped in a solvent which travelled up the plate by osmosis and in the process separated the stabilisers from other components. The relevant spot was scraped off dissolved up in another solvent and measured using an ultraviolet spectrophotometer, all a very long winded process. Then came high performance liquid chromatography and everything speeded up. Other stabilisers and components were measured by using gas chromatography (as photographed fig 108, ref 1) in L145.

Another product of propellant ageing were gases such as carbon dioxide, nitrogen and oxides of nitrogen, these could induce a pressure inside a large propellant charge and cause it to crack so an understanding of this was important.

A non standard analysis was used for the gases evolved from propellants on storage. This used glass vacuum lines as photographed with me in fig 110, ref 2 and described in ref 3. These were built and maintained by a team of glass blowers, Bert George and Bill Corthine to name a couple. The principal of the method was to take ground propellant and put some in the a “break seal” glass tube. The filling end then had to be “constricted” by heating the glass to red heat and pulling it out without igniting the propellant at the other end of the tube. In the early days, before I arrived, this was done by the glass blowers by hand. One day, as I understand it, when Bill Corthine was doing this some propellant ignited, he escaped with a burned hand. When I arrived a remote glass working lathe was being used to pull the tubes, no doubt “invented” soon after Bill's incident. The tubes were then attached to a vacuum line and all the air pumped out of the tubes and sealed by melting the constriction.



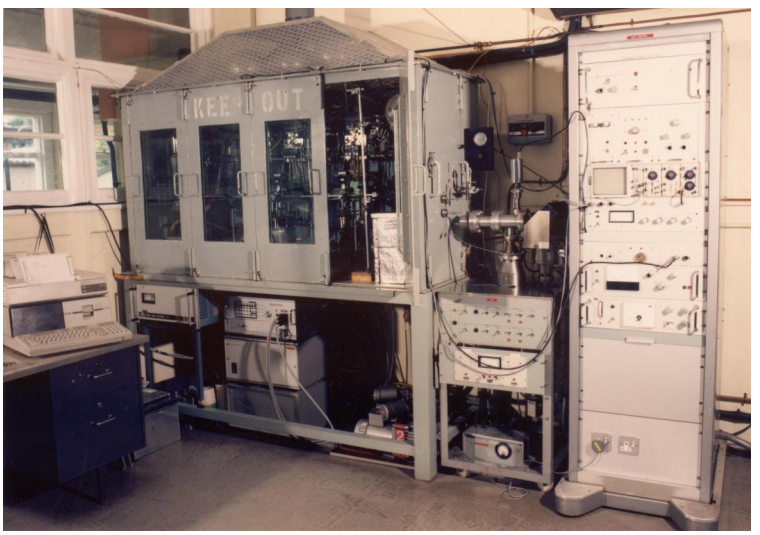
After appropriate storage the tubes were brought back to the vacuum line, as shown in the diagram. A small steel weight was balanced on the thin glass of the break seal and when all the air had been pumped from the vacuum line (using a mercury diffusion pump and checked using a mercury filled McLeod gauge) the steel weight was lifted using a magnet and dropped onto the break seal the break it and release the gases into the vacuum line. By forcing the mercury up and down the Toepler pump the gases could be transferred into the glass bulb. These were small amounts of gas (often only a fraction of a ml at normal pressures). The glass bulb containing the gases was then put on another vacuum line connected to a home made gas

chromatographic device (ref 3) for analysis. The storage tests were at different temperatures and times so that extrapolation to in service conditions could be made.

Sometime in the 1980's we moved from L145 to join General Chemistry in H10 (ref 4)

The list of H10 General Chemistry scientific staff I remember as:- Reg Powell, Mike Farey, Ian Wallace, John Williams, Peter Maher, Paul Bunyan, Sally Westlake, Ray Vaughan, Bernie Downes and myself.

At that time possible gas cracking of the Polaris rocket motors was a concern and we had various samples from motors to measure gas evolution on storage. The method was “modernised” we switched to micro Gas Chromatographic systems with mass spectrometers as detectors (Vacuum Generators Micromass 6 and Micromass 16). The



micromass 6 system and blast cabinet is shown in the photograph.

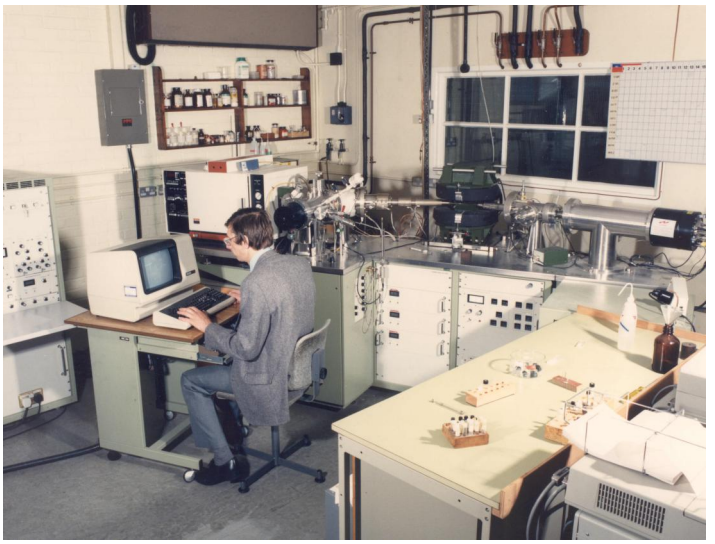
The propellant could not be ground but had to be chopped into tiny pieces, it went to “South Site”, I can't remember whether it was P1 or P2, and came back as thin sheets which we then fed through a hand

cutter John Williams made. This advanced the propellant through a reciprocating guillotine blade, you did this twice at 90 degrees and ended up with lots of little cubes. These were put in steel pots, I suppose about 50 ml capacity, which were sealed to a tiny metal sample loop using gold o-rings and heated in oil baths at appropriate temperatures. John Williams made the o-rings in his office from gold wire using a mini hydrogen oxygen welder (used o-rings were collected up and sent back to Johnson Matthey). To control the valves on the sample loops we used Sinclair ZX spectrum computers running a BASIC programme we had written. Who remembers the days of storing a programme on an audio tape? We also used an early HP desk top computer probably a HP85.

The method was more efficient than the glass tubes as many measurements could be taken from one sample of propellant without the need of a toepler pump. The apparatus including glass vacuum line mercury pumps etc. were housed in blast cabinets in case of accidents. I remember the testing of a steel vessel at New Hill, in spite of a big bang it was not a detonation and the blast cabinets were considered a safe containment.

You will have noticed references to mercury, in a big Toepler pump and associated vacuum line there could be 20 plus pounds of mercury, the lines were built on wooden trays so that mercury spillages, could be sucked up and disposed of but no-one worried about a few globules of mercury on the tray. This is in contrast to recent working life when even mercury thermometers were considered a hazard!

Other chemistry work that went on in H10 included heat evolution from propellants on storage and NOx measurements (Paul Bunyan); mass spectrometry to identify compounds e.g. novel energetic materials (myself) and compatibility testing of initiators, propellants and explosives. We had a top of the range mass spectrometer, a Vacuum Generators 7070EQ, which to those who are technically



minded was a high resolution magnetic mass spectrometer with a quadrupole mass spectrometer behind it to do daughter ion mass spectrometry. There's me in the photo at the controls!

I stayed in H10 until just before the site closed when I transferred to the Fire Research Station and spent my first few years there setting fire to things like aircraft, buses, artificial limbs, and various building structures!

John Rowley

ref 1 ERDE TECHNICAL NOTE No. 47 R Stenson

ref 2 The Listed Buildings and Other Principal Structures at the Royal Gunpowder Mills Waltham Abbey

Author: Les Tucker Publisher: Royal Gunpowder Mills

ref 3 The determination of gases evolved from propellant compositions by Gas Chromatography F I H Tunstall

Chromatographia 3 1970 (note there is also an earlier paper in Chromatographia 1)

ref 4 Touchpaper Jan 2009, Malcolm Bergh