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MODIFICATION OF A THERMOGRAVIMETRIC BALANCE FOR THE ANALYSIS OF ALPHA- AND LOW ACTIVITY BETA/GAMMA SPECIMENS

G. BART, F. PETRIK, E. SPRUNGER and P. GRITSCH

Eidgenössisches Institut für Reaktorforschung, Ch-5303 Würenlingen, (Switzerland)

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ABSTRACT

The thermochemical analysis of radioactive material requires extensive modification of equipment depending on the toxicity and radioactivity of the radionuclides analyzed. This paper describes the modification and recommissioning of a thermogravimeter at the Swiss Federal Institute for Reactor Research (EIR).

I INTRODUCTION

The study of thermochemical reactions of radioactive material requires special precautions depending on the type and amount of radiation emitted. The strongly penetrating gamma (γ) radiation of many radionuclides needs to be minimized with heavy biological shieldings. However standard sample containers are adequate to exclude damage through external irradiation of beta (β) and mainly alpha (α) particle emitting species with high ionisation densities. Nevertheless there exists a risk of incorporation of the latter nuclides which necessitates a high standard of containment.

In the EIR Hotlaboratory (HL), studies are carried out concerning the thermal treatment of plutonium (Pu) containing fast breeder fuels. In the future it is also intended to analyze the thermal behaviour of bitumen matrix Pu contaminated wastes.

A commercial Mettler [1] thermoanalyzer TA-1 has been installed for use in these projects. The original apparatus is shown in Fig. 1.

Both of these projects give rise to an alpha (Pu) handling problem. The risk of external γ-irradiation is minimal due to the small sample size and can be controlled by measuring the local dose rate. The commercial thermoanalyzer has therefore been modified to handle α and low activity β/γ specimens.

2. MODIFICATION CONCEPT

To exclude the risk of α incorporation, Pu is handled exclusively in closed boxes maintained at a negative pressure of 2–5 mbar. This condition also applied to the

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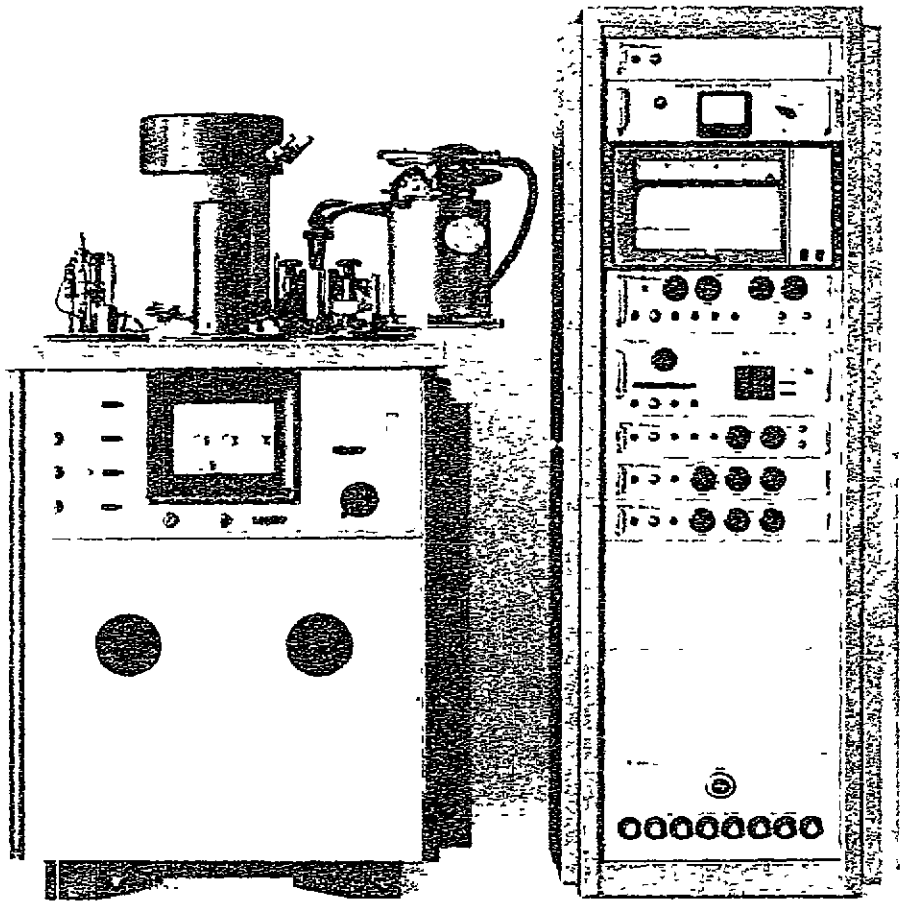

Fig. 1. Front view of the commercial thermogravimeter Mettler TA-1

handling of the α active specimens for the thermoanalyzer. Since the sample chamber, balance and pumping systems can all be contaminated, they all had to be built within the alphabox. The following conditions had therefore to be satisfied

(a) The equipment maintenance had to be optimized
(b) The furnace selection (HTF or SHTF) had to be simplified
(c) The heat produced by the HT-furnace had to be extracted.
(d) All balance manipulations had to be carried out with thick rubber gloves
(e) The balance maintenance had to be accomplished in situ

3. MODIFICATION DETAILS

The modified equipment is shown in Figs. 2 and 3. To ease maintenance, as many as possible of the electrical and mechanical components were removed and installed

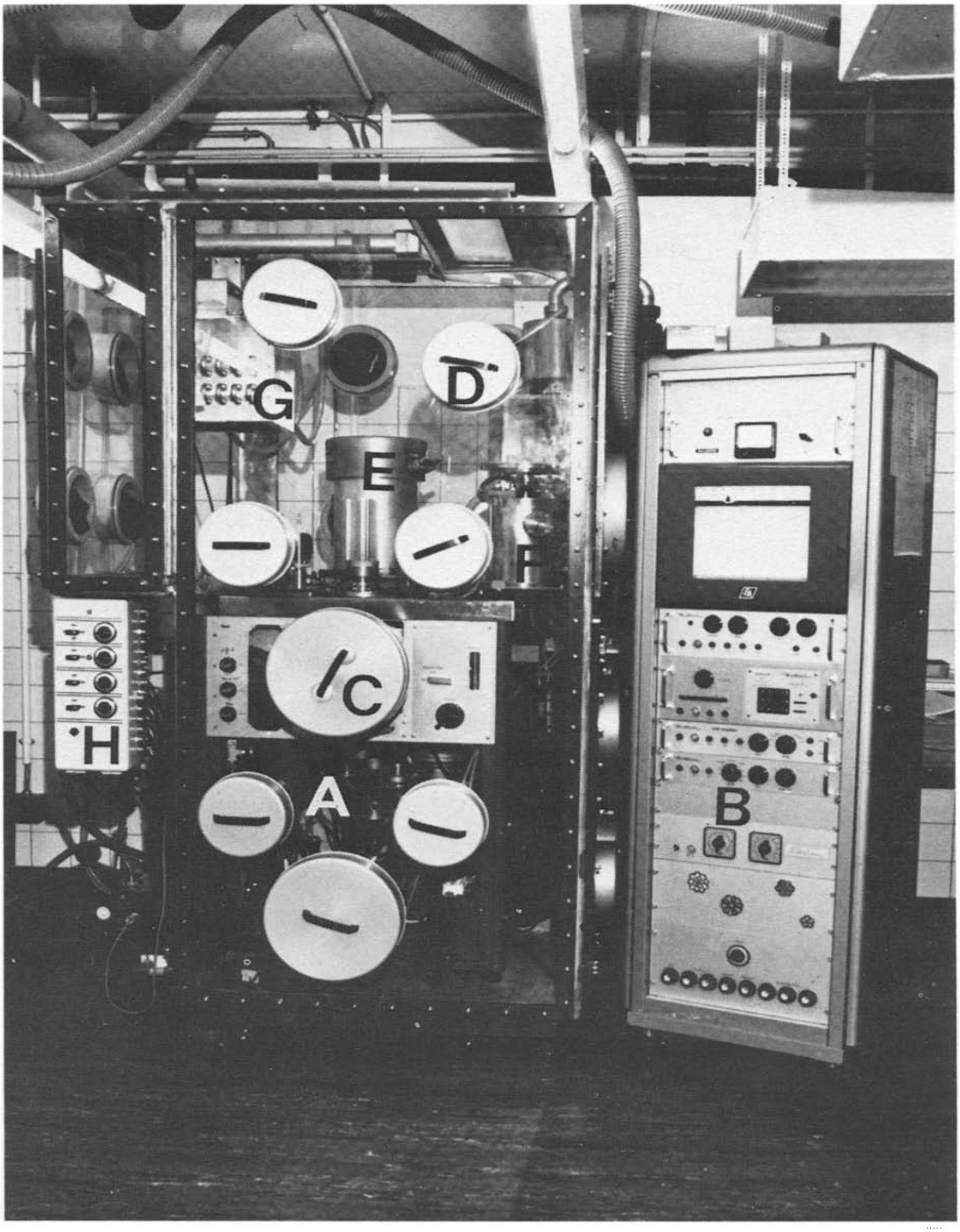

Fig 2 Front view of the modified thermoanalyzer A. Balance body (with balance and pumping systems), B. balance control cabinet, C. balance access door D, glove port, E high temperature furnace (HTF), F. super high temperature furnace (SHTF) G. cooling system for HTF gases (pneumatically movable) H. balance switch board

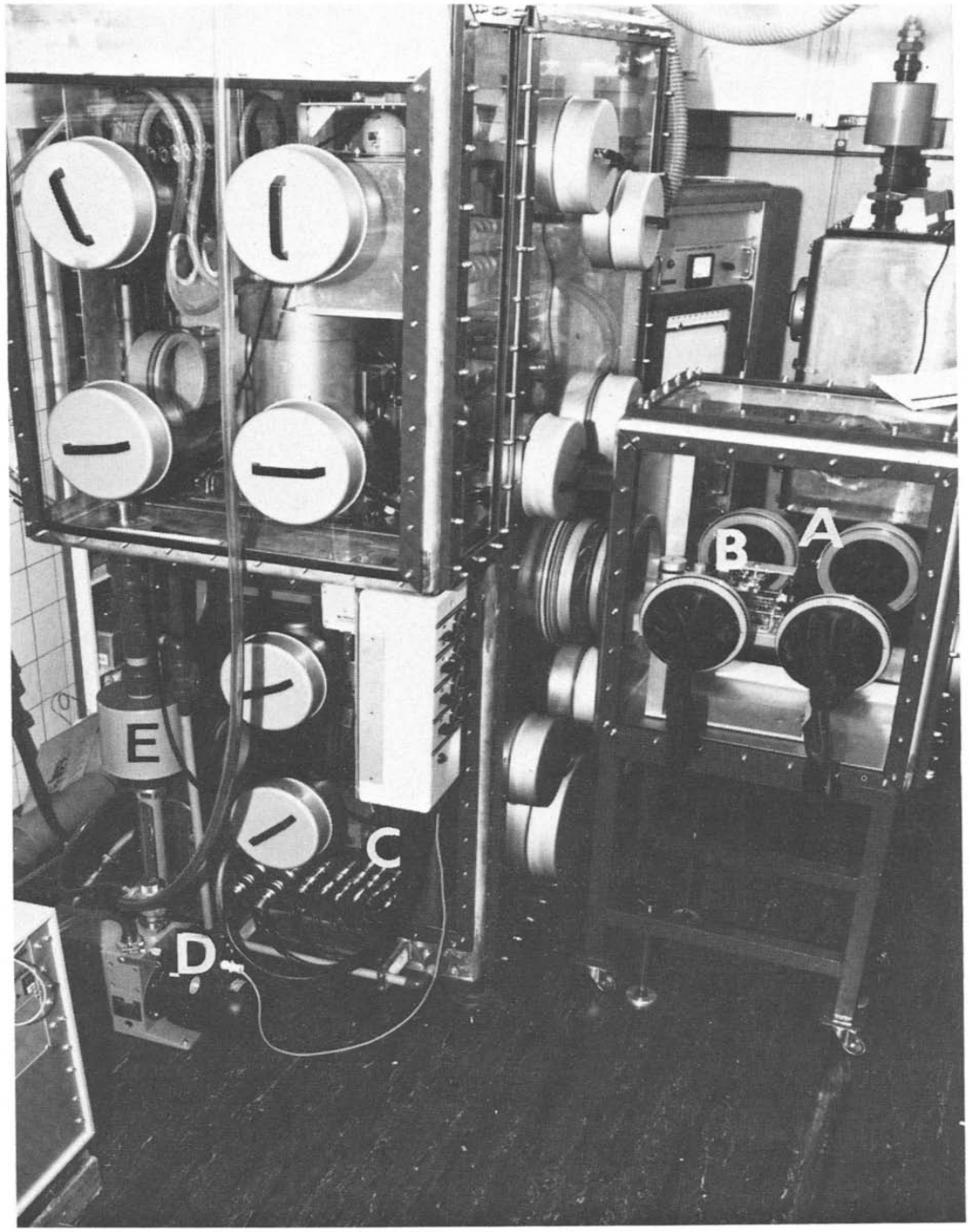

Fig 3 Side view of thermogravimeter- and balance maintenance box A Maintenance box attached to the balance box B balance body on transfer rails C electrical leads plugged to the box wall D roughing pump E box inlet air filter

outside the α box, e g the rotary pump (*D*, Fig 3), the water flow meter of the diffusion pumps and one of the electrical distributor cabinets. All components can be reached through glove ports All electrical containment penetrations were made

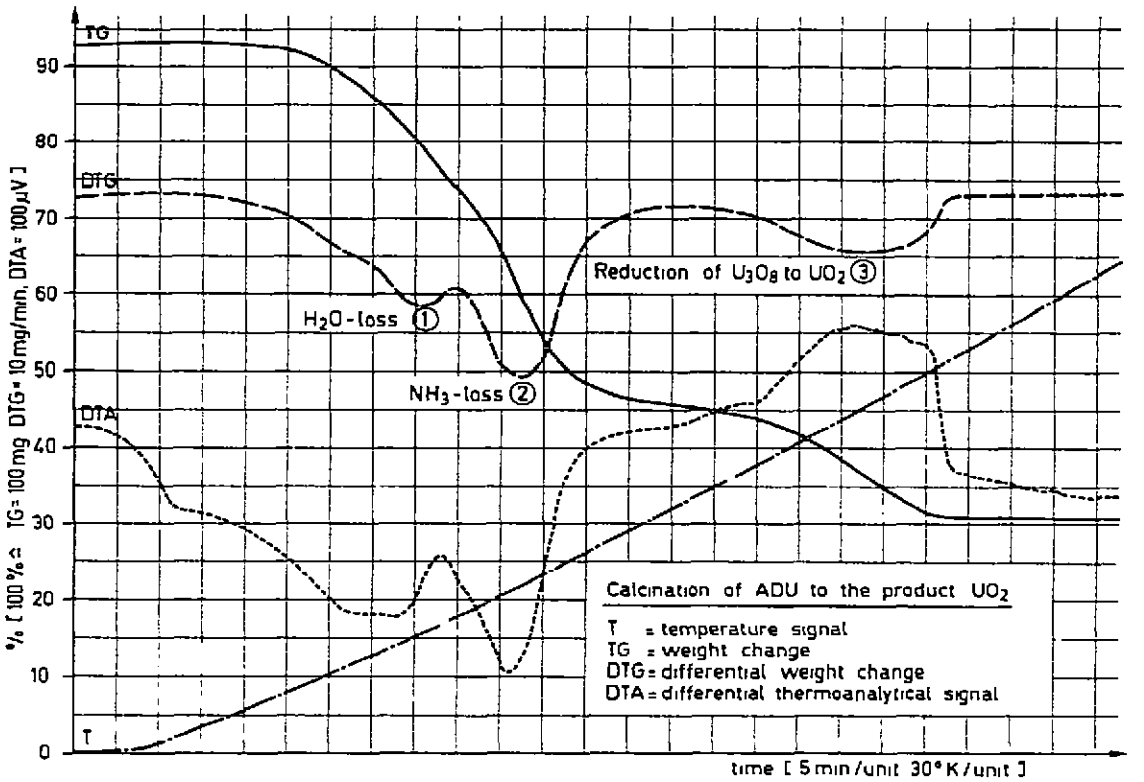

Fig 4 Thermogram of ammonium diuranate calcined to uranium dioxide with the rebuilt equipment

using "Lemo" couplings (C, Fig. 3) [2].

In the commercial apparatus the SHT and HT furnaces are lifted by means of a counterweight system. This was replaced by a pneumatic lift Centering of the crucible carrier over the balance body is assisted by using a prismatic viewing system The balance switch board was installed outside the α box (H, Fig 2) Heat extraction from the 4.5 kW HT-furnace is accomplished by means of a pneumatically transportable heat exchanger, mounted at the top end of the box (G, Fig 2). In operation, the hot air leaving the furnace is sucked through the cooler lamellate. The α box is flushed with dry air or nitrogen. A pressure regulation system controls the negative pressure of the box Particle filters in the air lines (E, Fig. 3) complete the containment system. The gases purging the sample and balance chambers are removed together with the box atmosphere to the common laboratory extraction system. The gases purging the sample chamber can be used to identify volatile components by passing through a leak valve to a quadrupole mass spectrometer. The laboratory α activity is continuously monitored by a semiconductor analyzer

Balance maintenance will be accomplished in a separate box (A, Fig. 3) which can be sealed to the balance box as required. The balance is transferred to the maintenance box via a rail system mounted in the service box (B, fig 3). To check for in box water leakage, a survey monitor was installed on the floor of the box The device cuts off the water supply to the box in the event of leakage

4 RECOMMISSIONING

Since modifications were completed, the thermoanalyzer has been used to produce a uranium carbide model fuel. Figure 4 shows a typical thermogram of the calcining stage of the heat treatment. Starting with carbon containing dried ammonium diuranate $[(NH_3)_x(UO_3)_y(H_2O)_z]$, UO_2 was produced as an intermediate product, ready for sintering to form UC. The volatile components during the calcining process were identified using the quadrupole mass spectrometer. The modified equipment will now be used to optimize the heat treatment of Pu containing fuel samples.

5 REFERENCES

1 Mettler Instrumente AG, CH 8606 Greifensee-Zürich, Switzerland
2 Lemo SA Electronique, CH 1100 Morges, Switzerland