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Operation of a 10-Stage Separation Nozzle Pilot-Plant

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### Operation of a 10-Stage Separation Nozzle Pilot-Plant

by

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presented by R. Schütte

In its present state of development, the separation nozzle process uses a process gas mixture consisting of 5 mole%  $\rm UF_6$  and 95 mole% helium<sup>1)</sup>. The separation factor for the uranium isotopes in one separation nozzle is of the order of 1.01 while the separation factor between  $\rm UF_6$  and helium reaches values of 5 to 10. In a cascade of such separation elements composed of identical stages, a constant  $\rm UF_6$ -helium composition of the feed gas is automatically maintained at the inlet. However, throughout the cascade there is a net up-flow of helium. This helium up-flow has to be withdrawn at the top of the cascade and recycled to the bottom stage.

It is desirable in the operation of the cascade to have equal pressures and equal UF<sub>6</sub>-feed concentrations in all stages. Since the throughputs are determined by the feed pressure and the feed concentration at the nozzle inlets, the pressure and the concentration have to be controlled by adjusting the cut of each stage. The helium withdrawal at the top of the cascade must also be closely controlled. Therefore a detailed knowledge of the gas-dynamic stability and of the general control behaviour of the cascade system is required. To gain this information, as well as general experience on the technology of the process, a small scale pilot-plant was constructed and put into operation.

A computer analysis of the steady state and the transient behaviour of a small separation nozzle cascade was performed previously and had indicated that the system was inherently stable. This stability is due to self-balancing effects of compressor response and separation element operation towards changes of pressures and  $UF_6$ -concentrations. Consequently, the pilot plant was constructed without any automatic control installations<sup>2)</sup>.

In the pilot plant Roots compressors are used for the recompression of the process gas expanded in the separation nozzles. In spite of their low efficiency, these compressors were chosen, because they are best suited to handle the desired comparatively small throughputs and because their compression characteristics are similar to those of turbocompressors, which would be used in a full scale industrial plant. As a result of this choice, a restriction in absolute pressure level and a limitation in the compression ratio had to be accepted.

Fig. 1 shows a cross section of the separation nozzles used in the pilot plant. They are designed for a feed gas pressure,  $p_0 = 48 \text{ mm Hg}$ , on the nozzle inlets. As a consequence, their critical dimensions are larger by a factor of about 15 as compared to the type of nozzles presented in the preceding paper<sup>X)</sup>, which are rated for a feed gas pressure of 600 mm Hg. In the pilot plant, the process gas is expanded from 48 mm Hg to  $p_M = 14 \text{ mm Hg}$  and the expansion ratio of  $p_0/P_M = 3.5$  is somewhat lower than the optimum value of 4. For ease of fabrication of the nozzles the inner wall of the curved Laval nozzle is formed by a solid cylinder, mounted eccentrically to the deflecting wall. In the pilot plant the length of the nozzle slits, perpendicular to the plane shown, is 10 cm and the flow rate of feed gas at the pressure of 48 mm Hg is 60g-moles/h. Under the given conditions, one separation nozzle produces an elementary separation effect  $\boldsymbol{\varepsilon}_A$  for the uranium isotopes of about  $8 \cdot 10^{-3}$  for a UF<sub>6</sub>-cut,  $\boldsymbol{\Psi}_{\rm UF} = 0.5$ 

On the other hand the total stripping factor or total cut is  $\Psi \approx 0.8$ . This is reflected by the difference in molar concentration of UF<sub>6</sub> in the light and in the heavy fraction,  $N_M \approx 0.03$  and  $N_K \approx 0.15$ , respectively. This situation is seen in more detail in the flow diagram of the pilot plant (Fig. 2). It shows the 10 stages of the square cascade with their compressors. The stage interconnections are

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x) E.W. Becker, W. Bier, R. Schütte: Principles and Economic Aspects of the Separation Nozzle Process. Paper presented at the Symposium, Turin 1968

arranged for a  $UF_6$ -cut,  $\vartheta_{UF_7}$  = 0.5. The incoming total flow of 60 g-moles/h of a stage is split into 50 g-moles/h of light fraction containing 1.5 g-moles UF<sub>6</sub>/h and into 10 g-moles/h of heavy fraction also containing 1.5 g-moles UF<sub>6</sub>/h. By mixing the light fraction of one stage with the heavy fraction of the stage, two stages above, the same total inflow of 60 g-moles/h of process gas containing 5% UF<sub>6</sub> is restored at the inlet of the compressor feeding the following stage. In this non-producing cascade, the balance of flows shows a net upflow of 40 g-moles helium/h throughout the cascade, while the  $UF_6$ -flows are balanced. In a producing cascade the relatively small product and waste flows would be superimposed. In either case, the net helium upflow has to be separated from the light fraction of the top stage, stage No. 10, and has to be recycled to the cascade bottom. This task is accomplished by the UF<sub>6</sub>-helium separation plant shown at the top of the cascade, which is fed with the light fraction from stage No. 10 by means of an additional compressor, No. 11.

In the pilot plant, this UF<sub>6</sub>-helium separation station consists of 2 independent low temperature traps cooled with liquid nitrogen. In these traps the UF<sub>6</sub> content of the incoming helium-UF<sub>6</sub>-mixture is frozen out and the UF<sub>6</sub>-free helium is recycled to the cascade bottom. In continuous operation, the traps are used alternatingly and the recovered UF<sub>6</sub> is recycled to the top stage of the cascade. In another type of operation this  $UF_6$  is replaced by fresh  $UF_6$  from storage containers. Owing to the large UF<sub>6</sub>-holdup of the traps compared to the total UF<sub>c</sub> content in the cascade, in either of the two cases the isotope concentration at the top of the cascade remains constant. So, enrichment of the heavy isotope takes place on the way down to the bottom of the cascade. For the total enrichment factor of the cascade, formed with the mole fractions of the light uranium isotope in the light fraction at the cascade top and in the heavy fraction at the cascade bottom, we can expect in the non-producing cascade a value of  $A_{\kappa} \approx 1.083$ .

The Roots compressors of the pilot plant are made of stainless steel and are equipped with an integrated gas cooler on their high pressure side. Vacuum-tight shaft seals are provided in the form of sealing chambers closed by radial-face seals. These seals are cooled by circulation of a UF<sub>6</sub>-resistant fluorocarbon oil, acting as the sealing fluid. In the pilot plant the Roots compressors are arranged in such a manner that the pipe carrying the light fraction of each separation nozzle to the compressor is short. This is shown in Fig. 3. The nozzles are mounted in the cylindrical chambers at the top of each stage. The process gas is fed through the cover of this chamber, on which the separation nozzle is clamped. While the heavy fraction of each separation element is pumped through this cover by a second pipe, the light fraction is collected in the chamber and pumped through the large vertical pipe to the compressor inlet. Into this pipe the heavy fraction coming from stage two stages above is introduced and the total feed stream is compressed and is fed to the next stage nozzle inlet. At each stage three sampling containers are connected in parallel to the process lines. From these containers representative samples can be taken for isotope analysis.

The total cascade has an U-shaped arrangement to minimize the interconnections between top and bottom. All stages can be evacuated through a main vacuum line, which is connected to a central vacuum pump station. Two large buffer volumes can be coupled to the top and to the bottom stages. The UF<sub>6</sub>-helium separation plant is placed below the two buffer tanks. The cascade is constructed completely of stainless steel with Viton O-ring seals. Stainless steel bellow valves are used throughout. Fig. 6 gives the view from the control room of the cascade, showing the row of 5 stages with 6 Roots compressors and one of the buffer tanks in the background. Experiments with UF<sub>6</sub> have been carried out in the pilot plant for about a year and no serious component failure has occurred. The vacuum tightness remains unchanged, the pressure rise by inleaking air being of the order of 10<sup>-4</sup> mm Hg/h.

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For supervision of plant operation the pressures and the feed concentration of each stage are transmitted to the control room, were all data are recorded. Data of interest can be selected for display on a control board.

In assembling the separation elements the mechanical adjustment of the distance between the knife edge and the deflecting wall permits the setting of the desired UF<sub>6</sub>-cut only within a certain limit of error. Therefore, fine adjustment of the UF<sub>6</sub>-cut in the operating elements is achieved by utilizing the influence of the pressure of the heavy fraction on the stripping ratio of the nozzle system. Figure 4 shows the effect of the pressure  $p_K$  of the heavy fraction on the UF<sub>6</sub>-cut,  $\mathcal{O}_{UF_6}$ , and on the elementary separation effect,  $\boldsymbol{\varepsilon}_A$ , of a representative separation nozzle. By increasing the back pressure,  $p_K$ , the UF<sub>6</sub>-cut rises slowly, while the elementary separation effect,  $\boldsymbol{\varepsilon}_A$ , is unaffected over a large range of back pressure.

This procedure permits the precise adjustment of the cut without loss in separation effect within the pressure range shown in the diagram<sup>4)</sup>. Accordingly, all separation nozzles of the pilot plant were mechanically adjusted as to give a  $UF_6$ -cut,  $\mathcal{P}_{UF} \approx 0.45$ , at equal expansion ratios for the heavy and the light<sup>6</sup> fraction, as is the case in the example of Fig. 4. In plant operation of this nozzle, a counter pressure of the heavy fraction of  $p_K \approx 18$  mm Hg would produce the required  $UF_6$ -cut of 1/2. Corresponding calibration curves were recorded for all separation nozzles in a single stage set up<sup>1,3)</sup>. On the basis of these calibration curves the cascade operating conditions could be preset by controlling the pressure of the heavy fractions by means of a throttle valve in the exhaust line of the heavy fraction in each stage.

The plant started operation with  $UF_6$ -helium mixture in November, 1967. The operation proved to be stable from the start. Fig. 5 shows the enrichment factor  $A_s$  along the cascade, measured relative to the isotopic concentration in the heavy fraction of stage 1. D, M, K indicate the sampling points in the feed, in the heavy, and in the light fraction of the various stages. Fig. 5 gives an overall enrichment factor of 1.082. It agrees within the limit of error with the value which was predetermined from the elementary separation effects of the individual nozzles. The test program underway has shown that the fine adjustment

of the UF<sub>6</sub>-cut by the method described above also yields precise adjustment of interstage flow. The plant operation thus can be controlled by means of one valve per stage. Furthermore, the tests have shown that the plant operation is stable, even with irregularly adjusted UF<sub>6</sub>-cuts. Also, a change of 10% in the flow rate of UF<sub>6</sub> recycled from the UF<sub>6</sub>-helium separation plant, which may be considered a large disturbance, gives a detectable change of operating conditions only in the four top stages. The maximum effect of this disturbance is a pressure change of about 3% at stage No. 10.

On the basis of these results it may be expected that no serious problems of control will arise in industrial scale operation of the process.

<sup>1)</sup> E.W. Becker, K. Bier, W. Bier, R. Schütte and D. Seidel; Angew. Chemie, Intern. Edit. <u>6</u>, 507 (1967)

<sup>2)</sup> E.W. Becker, G. Frey, R. Schütte; Bericht KFK 702, Kernforschungszentrum Karlsruhe

<sup>3)</sup> R. Schütte, D. Seidel; Chemie-Ing. Techn. 39, 80 (1967)

<sup>4)</sup> E.W. Becker, G. Frey, R. Schütte and D. Seidel, Atomwirtschaft <u>13</u>, 359 (1968)



Cross section of the separation nozzles used in the pilot plant. Operation conditions given on top.  $p = total pressure; N = mole fraction of UF_6 in the UF_6/He mixture. Subscripts refer to feed gas, light and heavy fraction.$ Fig. 1:

Elementary separation effect

 $\epsilon_{A} = \frac{n_{M}(1-n_{K})}{(1-n_{M})n_{K}} - 1$ 

 $n_{M,n_{K}}$ : Mole fractions of the U<sup>235</sup>-isotope in the light and in the heavy fraction, respectively. Enrichment factor of the cascade  $A_{K} = \frac{n_{M,10}(1-n_{K,1})}{(1-n_{M,10})n_{K,1}}$ 

 $^{n}$ M,10  $^{n}$ K,1 : Mole fractions of the U<sup>235</sup>-isotope in the light fraction of the top stage 10 and in the heavy fraction of the bottom stage 1.



Fig.2: Flow sheet for pilot plant operation without product withdrawl. Figures in front of He and UF<sub>6</sub> indicate flow rates in moles/h.







Fig. 4: The elementary separation effect  $\boldsymbol{\varepsilon}_A$  and the UF<sub>6</sub>-cut  $\boldsymbol{\vartheta}_U$ as function of the heavy fraction counter pressure  $p_K$ . 6 Measured at a representative separation nozzle element.  $\boldsymbol{\varepsilon}_A$  in units of 10<sup>-3</sup>.



isotope in the heavy fraction of stage 1 and in the different fractions at the withdrawl points.



### Fig. 6: View of the cascade from the control room