Options for the Immobilisation of UK Civil Plutonium -7214

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ABSTRACT

As a result of the reprocessing of spent nuclear fuel the UK expects to accumulate in excess of 100 tonnes of separated plutonium. There is currently no long term strategy for the management of this material. The Nuclear Decommissioning Authority (NDA) has commissioned a programme with Nexia Solutions to examine the alternatives for the disposition of this material and subsequently present the NDA with technically underpinned option(s) for plutonium disposition.

The overall programme comprises two main options, re-use as fuel in reactors and immobilisation in the event of being declared surplus to requirements and is targeted specifically at the requirements of the UK. The disposition programme will take account of the UK reactor portfolio, and the quantity that may require immobilisation will depend on future UK nuclear strategy. Immobilisation options are being reviewed in the light that a proportion of the stockpile could be declared waste.

The options for immobilisation are being assessed in the expectation that any immobilised product would be destined for an as yet undefined repository, preceded by a period of storage in secure facilities. Potential products are being developed to be compatible with a wide range of disposal scenarios.

INTRODUCTION

As a consequence of the termination of the fast reactor programme in the 1990's there is currently no long-term strategy in place for the use or management of separated plutonium. Separated plutonium stocks are held at Sellafield in the form of plutonium dioxide powder, using specially built stores which are subject to international safeguards and inspection by IAEA and Euratom. The plutonium has been stored safely and securely for many decades, and can continue to be so. However storage itself is not a sustainable option for the long term and there remain concerns regarding proliferation resistance.

Based on anticipated arisings from reprocessing fuels from Magnox and AGR reactors the total inventory of separated civil plutonium could rise to about 100 tonnes over the next 10 years.

In April 2005, the UK Nuclear Decommissioning Authority (NDA) took ownership of most of the civil nuclear liabilities and assets in the UK, which include the majority of these stocks. Currently future national policy for disposition remains to be finalised and the feasibility of

future management options needs to be determined in order to allow the NDA to advise government on the ultimate disposition of this material.

NDA has funded Nexia Solutions to develop and carry out a research project which will result in conclusions regarding the technical feasibility of a number of disposition options. These options have been developed from the recommendations of the innovative BNFL Stakeholder Dialogue process [1]. One key recommendation from that process was that plutonium disposition should be underway within 25 years (by 2025) and should be complete within 50 years (by 2050). Whilst these dates do not represent firm commitments, they are indicative of the views of stakeholders.

Two main long term options have been identified for the disposition of separated civil Pu. These are re-use as fuel in existing or future reactors and immobilisation in preparation for disposal in an as yet unspecified repository. It is important to recognise that these two options are not mutually exclusive and it is likely that a fraction of the stockpile could require immobilisation in any event given its level of contamination which could make processing for fuel uneconomic.

This paper will outline the programme aimed at the immobilisation option.

EVALUATION PROCEDURE

The decision as to which option or options should be implemented, will be based on a consideration of a number of factors including technical feasibility, safety and environmental impact, political and stakeholder acceptance and economic viability. These will also be influenced by factors unique to the UK such as the possibility of future new nuclear build and the requirement for medium term storage and the timescales for the implementation of repository disposal.

The overall programme, under which this project sits, is divided into 3 areas of activity:

1. Strategic tasks – which support evaluation of technology options and provide information on safety and environmental impact, political and stakeholder acceptance and economic viability for the decision making process
2. Development of re-use options – which will provide information on the technical feasibility of candidate fuel cycles
3. Development of immobilisation options – which will provide information on the technical feasibility of candidate wasteforms and viability of processing routes to deliver them.

A phased approach has been adopted, with multi-discipline evaluations after each phase for selection of technology options. The initial phase is focussing on a number of immobilisation options which will be reduced on a systematic basis leaving the most credible options for in depth study during the latter phases of the programme.

It is likely that at least a fraction of the feedstock will require immobilisation; typically the material which is heavily contaminated by chlorine as a result of storage in PVC and economics may dictate an immobilisation as opposed to a processing future.

At least one immobilisation option will be presented to the NDA as a result of this programme of work, with the likelihood that a further option or sub-option be proposed as a result of specific circumstances.

FEEDSTOCK

This programme is aimed at providing options for the long-term storage and subsequent repository disposal of the UK stockpile of separated civil plutonium stocks. These include plutonium derived from reprocessing of Magnox reactor fuel, which contains largely Pu-239, as well as AGR derived plutonium which have increasing levels of Pu-241, bringing with it the need for additional shielding measures. This programme excludes material such as plutonium contaminated materials (PCM) or residues containing plutonium which are being addressed under a separate but linked programme of work sponsored by the Sellafield site operator, British Nuclear Group. However there are quantities of plutonium which are heavily contaminated with chlorine and these need to be taken into consideration. The design of any repository has yet to be defined and therefore the wasteform development must be carried out in such a way as to develop a product that will be suitable for disposal over a range of credible scenarios. It is important to note that the plutonium for which these options are being considered is civil in origin and as such will be subject to the necessary safeguards both during processing, storage and disposal.

DEVELOPMENT OF IMMOBILISATION TECHNOLOGY

The BNFL Stakeholder Dialogue process [1] concluded that a significant programme of work would be required to underpin the immobilisation of plutonium stocks, should immobilisation be the strategy chosen for the UK civil plutonium. This conclusion acknowledges that little or no previous work has been carried out in the UK, since hitherto plutonium has been considered an energy asset. However researchers have been active in this area world-wide including a significant level of R&D that has been carried out for the US DoE to support the ex-weapons plutonium disposition programme and it is with this in mind that the current R&D programme has been formulated and targeted specifically at UK requirements.

The highly radiotoxic nature of plutonium warrants a disposition solution which provides a capability to isolate the materials from the biosphere for a period commensurate with the eventual decay of that toxicity. Discussions with stakeholders [1] have highlighted the requirement for robust solutions which can be demonstrated to be fit for purpose through the execution of rigorous R&D in areas such as proliferation resistance, durability in repository situations and criticality control in both storage and disposal. In addition to these imperatives are economic considerations which are affected by such parameters as waste loading. Maximization of waste loading within the limits required for wasteform acceptance may have a large and beneficial impact on storage costs.

In the absence of a qualified repository in the UK for the disposal of large quantities of fissile material, the wasteform design process needs to establish a wasteform which will be suitable for disposal under a number of different scenarios. There are no currently accepted wasteform acceptance criteria in the UK for the immobilisation of high level quantities of plutonium outside the IAEA guidelines. During the development process issues surrounding acceptance are being

discussed with various stakeholders such as Nirex and the Environment Agency with the aim of producing criteria which are achievable and acceptable to future repository operators.

A key measure of the wasteform's ability to deliver the immobilisation of plutonium is its durability or the capability of the wasteform to retain nuclides when leached under repository conditions. Such durability is vital as it is the first line of defence in preventing the plutonium leaching from the wasteform over long timescales and reaching the biosphere. The programme will seek to develop appropriate measures of durability and where possible endeavour to interpret them in the light of extended timescales.

Similarly radiation damage effects on chosen wasteforms will be evaluated to the extent which they affect durability. The majority of this work will take place during the active phase later in the programme but a limited amount of work has already been carried out (see ceramics section) and this has been supported by a programme of work on natural analogues at University of Cambridge.

A strategy to deal with criticality both during storage and disposal is being developed with stakeholders. Wasteform development and inclusion of neutron poisons in the wasteform is central to the approach. This will include the use of hafnium and gadolinium to protect against internal modes of criticality as well as external and extended modes during any future leaching of fissile material.

In the U.S, the ex weapons plutonium immobilisation programme included the presence of a radiation barrier in order to enable the package to meet the spent fuel standard. The value of this approach for the UK is questioned. The environment council report produced on behalf of the BNFL stakeholder dialogue [1] recommended that *"The Company's assessment of the development requirements of immobilisation options focus on those options without an external radiation barrier. The assessment should, however, examine the feasibility and value of other potential "intrinsic" security features."* As such the current wasteform development programme is not considering the addition of an integral radiation barrier.

A broad-based approach is being carried out in phase 1 of the immobilisation development programme. A number of ceramic, glasses and a low spec MOx option are currently being evaluated in terms of both product composition/performance and ease of processing. A review is planned in order to de-select the least appropriate which will allow the more in depth studies to be carried out on the one or more options thought most appropriate to UK needs.

In the initial stages of research, the project is focussing on the use of plutonium surrogates such as cerium to provide data capable of informing choices regarding most suitable immobilisation matrices. In cases where uranium is believed to better replicate the nature of plutonium a facility has been set up in order to enable its use on a small scale. Following down selection of preferred candidates it is felt that data on fabrication and performance will be required from wasteform made with Pu-239 and Pu-238 in the case of radiation damage experiments. A process is underway to investigate the setting up of a fit for purpose glove box facility in the Technology Centre (BTC) at Sellafield in which the behaviour of plutonium containing wasteforms can be validated in order to give a high degree of confidence in the chosen option(s).

The strategy for the storage of the product from any immobilisation plant is to use the current design of Magnox plutonium can which has a capacity of approximately 5 litres and is currently employed for the storage of plutonium oxide powders. In this way the product will be able to be housed in currently designed stores. This strategy may need to be modified should there be a requirement to immobilise the majority of the stockpile when economics may prefer different can designs for an alternatively designed store.

CERAMICS

A review of the literature shows the extent to which ceramics have been considered but not as yet utilised for the immobilisation of actinides such as plutonium. Although initially chosen for the immobilisation of ex weapons plutonium in the US, the immobilisation programme is currently on hold pending review while progress continues on the MOx fuel option.

In addition to the wasteform chosen for the U.S. ex weapons plutonium programme, there exist other potential formulations capable of immobilising plutonium. The choice of pyrochlore phase was informed by feed characteristics and disposal criteria. In the U.K. the feedstock has different characteristics. The plutonium being civil requires safeguards, the isotopic mix and contaminants are different and the final disposal conditions are unknown. For these reasons it was decided that an initial assessment of wasteform candidates should be broad and capable of evaluating the capability of differing host matrices.

To enable an economic assessment of a range of possible ceramic compositions the initial phase of research is being conducted using plutonium surrogates. After initial trials using cerium, the project has progressed to the use of uranium as a plutonium surrogate. This initial programme is being carried out in collaboration with the Immobilisation Science Lab, (ISL) at the University of Sheffield, and benefits from data gained from a closely related programme of work [2] carried out in collaboration with the Australia Nuclear Science and Technology Organisation (ANSTO) which is developing glass ceramic phases for the immobilisation of plutonium waste and residues stored on the Sellafield site.

Wasteform development

Work to date [3] has demonstrated the capability of a range of phases to accommodate plutonium and associated neutron poisons such as gadolinium and hafnium. Results to date are given below. For ease of comparison, waste loadings are quoted as spatial densities of PuO_2 to eliminate ambiguities due to different wasteform densities and wt% values. For benchmarking purposes the wasteform developed by the US DoE comprised 90 vol% of a plutonium host phase based on $CaUTi_2O_7$. The PuO_2 density for this wasteform was 600 kg m^{-3}.

Zirconolite, prototypically $CaZrTi_2O_7$, can accommodate plutonium and neutron poisons by substitution for both calcium and zirconium. A baseline formulation has been designed with a PuO_2 density of 700 kg m^{-3}. This produces a two phase wasteform which will need to be assessed as to its suitability. Alternative plutonium substitution mechanisms could retain a single plutonium host phase to higher plutonium loadings, leading to formulations that better replicate natural zirconolite samples. These are currently being explored.

Zirconate pyrochlores are stoichiometrically $Gd_2Zr_2O_7$ with a target composition of $Gd_2(Zr_{1.6}Pu_{0.2}Hf_{0.2})O_7$. The PuO_2 density is 600 kg m^{-3} but there is currently no evidence that the solid solution mechanism holds. Although the zirconate pyrochlores exhibit good resistance to radiation damage, synthesis temperatures may rule them out as an economic option.

Cubic zirconia, generically $(Zr,Y,Pu)_xO_{2-x}$ has been shown to accommodate PuO_2 at a substitution level of x = 0.05 with a PuO_2 density of 650 kg m^{-3} and it appears that higher plutonium loadings could be achieved. Using this composition neutron poisons are accommodated by substitution of Gd for Y and Hf for Zr and work is now being carried out to verify this.

Britholite is a silicate based apatite structure, $A_{10}(BO_4)_6X_2$, (A = Ca, Pb, REE; B = Si, P, Al; X = O, OH, halide). The basic host phase is $Ca_2Y_8(SiO_4)_6O_2$ with extensive substitution of Gd and Pu possible. High PuO_2 spatial densities of 2000 kg m^{-3} could be possible, however no significant Hf incorporation has been demonstrated and this may count against the candidate in criticality assessments.

The high chemical flexibility of the kosnarite structure has been demonstrated in the literature although the results of the studies carried out under this programme have highlighted potential problems with the synthesis route. Care must be taken when using organic phosphate precursors; fast reaction rates promote the formation of a glass-like product rather than a powder. This may have implications for the feasibility of a single stage reactive sintering fabrication route.

The potential chemical flexibility of murataite make it a promising host phase for mixed waste stream or waste streams containing a high iron or sodium content. Unfortunately attempts to synthesis synthetic murataite via a conventional solid state route proved unsuccessful. In the A_2O_3-MO-TiO_2 (A = Gd, Y and M = Zn, Mn) systems investigated the major phase formed was a titanate pyrochlore and the transition metal components partitioned to a secondary ilmenite structured phase. There was no indication, by XRD, of the formation of any murataite structured phases. Long reaction times or more intimate mixing, afforded by a melting and recrystallising route, may be required to promote the diffusion and cation ordering necessary for the formation of murataite structured phases. On account of these murataite has been disregarded in this instance as a ceramic host phase for plutonium immobilisation.

Achievable PuO_2 waste loadings are likely to exceed the currently accepted levels of around 10%. It is intended to explore the option of higher waste loadings and seek to establish grounds for their acceptance. This has been prompted by the economics of storage which dictate that the costs of stores contribute to a large extent to the overall lifetime costs of storage and disposal.

As such a priority is being placed on the exploration of radiation damage. Wasteforms containing plutonium will experience a level of radiation exposure over prolonged time periods. While different phases may react differently to such exposure, it is important that any change in phase or dimensions must not have a deleterious effect on the waste-form's ability to retain radionuclides. Titanate, silicate and phosphate phases are known to become amorphous (i.e. the internal structure is broken down, which could result in cracking and increased leach rates). Zirconate wasteforms remain crystalline, however the higher temperatures required for their synthesis may rule them out. Work as part of the US ex weapons disposition programme on

titanate ceramics [CaZrTi2O7 and CaUTi2O7] has indicated that radiation induced amorphisation does not lead to a significant increase in leach rates [4]. Further, long term stability of these phases is confirmed by the existence of natural analogues. Zirconolites can contain in excess of 20 wt% U + Th with ages in excess of 2 billion years.

Although a systematic programme of radiation damage experiments will be required to confirm the acceptance of the final wasteform, the opportunity has arisen to examine samples of plutonium containing Synrocs synthesised in the 1980's. Although the SYNROC formulation was originally intended for immobilisation of fission products and the full suite of co existing phases would not be used for an actinide only stream, UKAEA fabricated samples containing Pu-238 to examine the effect of radiation damage on the structure. Although after ca 20 years storage the samples have become amorphous, they have remained mechanically integral giving confidence that the constituent phases, in particular the zirconolite phase would be suitable for actinide immobilisation.

Since cerium is vulnerable to auto reduction from Ce^{4+} to Ce^{3+} it is not an entirely robust simulant for plutonium. Uranium and thorium are more reliably stabilised as tetravalent ions and thus provide an improved surrogate. Findings from the cerium based surrogate work are being confirmed with uranium as a surrogate in the small scale facility in the U active laboratory at the Immobilisation Science Lab. (ISL) University of Sheffield.

All stocks of plutonium particularly those originating from the reprocessing of oxide fuels will be subject to in growth of ^{241}Am from the decay of ^{241}Pu. This will be a function of the age of the plutonium and its isotopic content. Any wasteform used for immobilisation will need to demonstrate the ability to co-immobilise americium which is at its most stable as a trivalent ion. Thus the phases shown to be suitable for immobilising the tetravalent plutonium simulants will be subject to a series of tests which will examine their ability to co-immobilise trivalent simulants such as neodymium or samarium.

Processing

Consolidation processes for the fabrication of preferred ceramic wasteforms are being explored. It is likely that some plutonium will require immobilisation, however the upper limit will be affected by a number of factors, not least the possibility of a new fleet of reactors in the UK which would have the capacity to burn the volumes of MOx fuel that would be required in order to disposition the plutonium stocks . Thus the evaluation must examine how throughput may affect the choice of processing route.

The current baseline envisages the storage of the immobilised product in a Sellafield Product and Residues Store (SPRS) currently under design and construction. In order to comply with that store it is envisaged that the product will be contained within the existing design of Magnox Plutonium can. This can has an approximate volume of 5 litres and is currently used for the storage of plutonium oxide powders.

The two main technologies being evaluated, are Cold Press and Sinter (CPS) and Hot Isostatic pressing (HIP). Cold Press and Sinter is an established technology in the production of fuel pellets and is currently used on the Sellafield site in order to produce MOx pellets.

A similar technology using either cold uniaxial or isostatic pressing followed by sintering is being examined for producing plutonium containing wasteforms. With the given dimensions of a product can, the research is exploring the optimum product size to give best packing densities within the can. Achieving that size can be limited by the effect of firing and cooling temperatures. Larger pucks than those typically produced for fuel might need binders and the burn out of these binders may institute cracking. The current SMP tunnel furnaces may not suitable for making larger pucks due to possible temperature gradients further affecting the mechanical integrity. In addition some phases may require higher consolidation temperatures. e.g.: zirconolite 1450 °C, Zr pyrochlore 1700 °C; cubic ZrO_2 1550 °C and britholite 1600 °C. These limits are being explored.

Hot Isostatic Pressing (HIP) is a technology that has been well established in non-nuclear applications and is under development for the immobilisation of plutonium containing residues on the Sellafield Site. This work is being carried out in collaboration with Australian Nuclear Science and Technology Organisation (ANSTO) and centres on immobilising plutonium containing residues using zirconolite/ pyrochlore ceramics and glass ceramics. [2]. For the residues, the use of HIP conveys a number of advantages, not least that of "zero" emissions from the sealed can. The particular advantage conferred by this technology for plutonium immobilisation is the ability to fabricate products of a size consistent with that of the Magnox plutonium can, thus yielding better overall storage volumes and the accompanying reduction in costs.

Fig 1 Example of HIPped ceramic product capable of being tailored for storage in a Magnox Pu can.
(Illustration courtesy of ANSTO)

In addition to the consolidation technologies it has been shown that preparation of feed powders can have a large effect on the quality of the final product. In this programme it has been demonstrated that attrition milling of a PuO_2 surrogate with the precursor prior to sintering or HIPping leads to a significant improvement in wasteform homogeneity and quality. Further optimisation of powder preparation parameters and consolidation processes is being progressed.

Other technologies such as sol gel and melting routes are being assessed as alternatives to the pressing options, however at this stage it is felt that the level of excessive processing required for sol gel and the coarse microstructure produced by melting techniques would make the processes unsuitable.

VITRIFICATION

Vitrification is an accepted technology for the immobilisation of fission product wastes resulting from the reprocessing of spent fuel. Considerable experience has been built up at Sellafield and elsewhere in the operation of such technology and vitrification is being considered as a possible route for the immobilisation of plutonium. However it is accepted that should a vitrification route be adopted for the immobilisation of plutonium, the processing route would be very different to that chosen for the vitrification of high level waste raffinates (HLW) not least on account of the criticality risk associated with plutonium.

As part of the broad approach, a wide range of glass compositions have been fabricated and assessed for their suitability for the immobilisation of the U.K. separated plutonium stocks [5]. The choice of candidates was based on the body of evidence in the literature. The experimental work to date has been carried out both at Nexia Solutions' Sellafield facility and at the Immobilisation Science Laboratory (ISL) at the University of Sheffield. The first stage experimental programme has allowed the selection of a reduced number of compositions, which are to be assessed at a much greater level of detail in the second stage. The final aim of this programme is to provide relevant technical data in sufficient detail to allow the selection of a single preferred vitrification option to be assessed against other immobilisation options.

Waste loadings of at least 10 wt% for surrogate materials have been demonstrated in a number of matrices. Incorporation rates as high as 20 wt% may also be viable, depending on glass composition and melting temperature, although care should be taken when comparing the behaviour of surrogates with that of plutonium. In all glasses, however, the solubility of plutonium will be improved by increasing the temperature of the melt.

For silicate-based glasses, a number of candidate compositions have been identified by this study; Modified alkali borosilicate (MW), alumino borosilicate (ABS), alkali tin silicate (ATS), and lanthanide borosilicate (LBS). The simple MW, calcium borosilicate (CBS) and lanthanide aluminosilicate (LAS) compositions are considered less good candidates due to a number of factors including poor durability, evidence of crystallisation, and high processing temperatures.

Taking into account ease of melting, potential plutonium waste loading capacity, glass stability, chemical durability and proliferation resistance, the most promising phosphate glasses are the iron phosphate (IP) and sodium aluminium phosphate (NAP) families. Less favourable are lead

iron phosphates (LIP) glasses on the basis of concerns over their behaviour at high melting temperatures, their formation of inhomogeneous materials and poor waste loading capacity.

All of the candidate glasses have chemical durability at least an order of magnitude better than the base MW composition, with the phosphates and LBS in particular demonstrating excellent corrosion resistance. It appears that the silicate compositions have the potential for higher plutonium-incorporation, although this may be slightly misleading due to the use of the less soluble HfO_2 in the phosphate glass plutonium-surrogate. There is also a strong temperature dependence of the waste loading for all the glasses in the study with higher temperatures and reducing conditions increasing the plutonium solubility.

Based upon the results gained during the first phase testing it is planned to select a number of formulations for further study. These will then be optimised for waste loading, chemical durability and criticality control. At this stage surrogates will be further used, although some changes may be made to the stimulant composition in order to better reflect the redox conditions that would prevail in the matrix with plutonium. Further examination of the dependence of criticality on glass composition will be carried out. In particular, an understanding is required of the effect of the various neutron poisons either already present in the matrix or added with the PuO_2.

The first stage experimental programme has included some limited melting of phosphate glasses with UO_3 from the perspective of redox behaviour, density/settling effects, and solubility for criticality control. This work will be expanded to include all of the candidate compositions.

A review of the factors relevant to the development of a plutonium-vitrification process, with particular attention to criticality issues and design of the melter, has been performed. The most significant consideration will be the control of criticality at all stages of the process; feedstock preparation, melter operation, glass pouring and product storage. The required temperature of operation and throughput will also be important parameters in process design, and an understanding of how these affect the criticality potential will be required. A number of melter technologies are being considered and a preferred option will be chosen to assess against the alternative immobilisation technologies.

IMMOBILISATION MOx

The stakeholder dialogue concluded that the use of MOx pellets should also be evaluated as an option for the immobilisation of plutonium. The apparent attraction of this option is the availability of an already established facility capable of manufacturing the "wasteform", viz. the Sellafield MOx Plant (SMP). In this case it is envisaged that the MOX pellets would be manufactured "as normal" with the exception that the dimensional tolerances required of reactor operations could be relaxed. The manufactured pellets could then be introduced into a Magnox Pu can prior to being consigned to storage in the Sellafield plutonium store (SPRS).

However, the performance in disposal scenarios of such a wasteform particularly against the alternatives of glass or ceramic is unknown. In order to establish likely behaviour of this material, samples of MOx pellets fabricated at Sellafield, have been shipped to ITU at Karlsruhe

where they have been prepared for and subjected to leach testing regimes. The leaching mechanism will be studied and figures for release rates will be compared against the preferred glass and ceramic options currently being developed.

Current wisdom suggests that while the leach performance may not be equivalent to that of say a zirconolite ceramic, the possibility of the use of an existing plant as an immobilisation process is superficially attractive. However, dependent on the future orders for the SMP and based on the changes that would need to be made to the process to accommodate the Pu requiring immobilisation, then it may be more economical to design and build a purpose built plant which would then bring into question the use of uranium as a host rather than a more durable ceramic phase manufactured in a similar process.

CONCLUSION

In excess of 100 tonnes of UK civil plutonium require a disposition route. Nexia Solutions are currently developing technically feasible options immobilisation option(s).

A broad initial phase of work is examining the suitability of ceramics, vitrification and immobilisation MOX options. Experiments using surrogates have shown that a number of phases and glass compositions are capable of incorporating economic levels of plutonium. Preferred compositions will have their suitability validated using plutonium.

Initial options will be reduced based on technical criteria taking into account product properties such as durability and ease of processing as discriminators. The reduced number of options will then be subject to greater scrutiny and the final option(s) will be preferred on criteria identified as part of a strategic assessment.

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