R2-D.1: Mechanics of Compounded Explosives for Enhanced Checkpoint Detection

I. PARTICIPANTS

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II. PROJECT DESCRIPTION

A. Project Overview

This project is an important element of the overall ALERT strategy to enhance air travel security. Like project R2-A.1, it is focused on checkpoints by contact sampling of carry-on baggage. Existing methods for contact sampling use traps that are applied manually to extract explosives residue from suspicious bags. These traps are then placed in an ion mobility spectrometer (IMS), where any explosive residue is desorbed from the trap when the temperature is raised to roughly 250°C over a period of approximately 8 seconds. Commercial-off-the-shelf (COTS) traps are optimized to survive repeated exposure to the IMS desorber, but not to extract residue from the surfaces being interrogated.

Considerable effort has been placed on finding ways to improve the sensitivity, accuracy, and response time of IMS tools. However, these efforts have been undertaken without a great deal of consideration of the essential first step in residue detection, which is extraction of the residue from the surface of interest. This project directly addresses this key step by pursuing rational trap design intended to optimize trap properties leading to superior residue harvesting from surfaces. This effort involves several steps, including:

- 1. Investigating the mechanical properties of explosives residues and relating these properties to the effectiveness of residue removal from surfaces. This is the focus of this project.
- 2. Performing rational trap design to optimize the effectiveness of traps at harvesting residue from surfaces. This was the focus of project R2-A.1, from which this current project was derived
- 3. Performing rational trap design to develop traps that retain their chemical and mechanical integrity at current IMS operating temperatures as well as at envisioned operating temperatures up to 400°C, which will be required to detect emerging homemade explosives (HMEs). This was the focus of project R2-A.1, from which this current project was derived.
- 4. Development of methods to assess the topography and adhesivity of substrates that are commonly

screened using traps, to ensure that the new traps are capable of harvesting residues effectively. This was the focus of project R2-A.1, from which this current project was derived.

This project explores the mechanical properties of compounded explosives, such as C4 and Semtex, for several reasons. First, by understanding the mechanics of these compounds, it is possible to identify the controlling processes in contact sampling, which is a critical step in determining effective detection. Second, understanding the mechanics allows optimization of swab design. Third, understanding the mechanics allows optimization of swabbing protocols. Finally, understanding the contact mechanics and the mechanics under load allows us to develop benign surrogates that can be used in place of live explosives during the development of sampling/detection schemes. This last capability is significant. At present, the community that is developing contact sampling methods is limited substantially by difficulties associated with obtaining live explosives to use in their laboratories. A benign surrogate that presents the proper key behaviors would dramatically improve the ability of researchers worldwide to develop best-in-class technology to improve trace explosives detection.



Figure 1: Peak flow stress as a function of increasing compression rates for: 1) simulated C-4 prepared with 40-150 mesh silica; 2) granules created with 40-150 mesh silica and PDMS of varying viscosity; and 3) live C-4. Note that 1, 10, and 100 mm/s correspond to strain rates 0.042 ± 0.004 , 0.431 ± 0.076 , and 4.651 ± 1.752 s-1, respectively.

Figure 1 shows the peak flow stress when granules of live C4, simulated C4, and benign surrogates were subjected to a compressive load. As can be seen, the peak flow stress of the granules changed as a function of the compression rate, with higher rates causing larger peak flow stress values. This is exactly opposite to the behavior that would be expected based on the flow properties of the binder in the granules, in the absence of the particles. The binder is shear thinning. Such a material would exhibit a lower peak flow stress as the compression rate increased. This demonstrates clearly that the presence of the particles within the composite drives the flow behavior. This figure also shows the behavior of composites created with either bimodal particle size distributions (live C4) or unimodal particle size distributions (all other samples) in binders that were either: polydimethlylsiloxane (PDMS, a Newtonian fluid), simulated C4 binder (binder prepared at Purdue using the

same ingredients and method as the binder in live C4), and binder present in live C4. As can be seen, the "simulated C4" and the live C4 do not behave similarly at all. There are two differences between these. The first is the difference in the composition of the solid particles in the granules. The live C4 contains explosives, while the simulated C4 contains silica spheres. As we have shown previously, the composition of the particles has no effect on the mechanical behavior of the granules. The second difference is that the simulated C4 contains particles with a unimodal size distribution, while the live C4 contains particles with a bimodal size distribution. Specifically, the simulated C4 contains particles that are in the "large mode" of the bimodal distribution of the live C4. This difference in particle content (live C4 contains many small particles that are not present in the simulated C4, while both contain large particles) is what makes the difference in the behavior.

To verify that it is possible to make simulated compounded explosives with the same mechanical properties as live C4, simulated C4 was made with the same binder as the live material, but with silica particles having a bimodal size distribution comparable to that of the live material. Several recipes were followed for this purpose, including:

- 1. Simulated C4 with unimodal (large size mode) silica particles,
- 2. Simulated C4 containing 75% large size mode and 25% small size mode silica particles, and
- 3. Simulated C4 containing 50% large size mode and 50% small size mode silica particles.

The mechanical behavior of the resulting composite granules was compared. Figure 2 shows the stressstrain behavior of the granules. As can be seen, at the higher compression rates of 10 and 100 mm/s, the simulated C4 with the bimodal silica particle distributions behaved nearly identically to the live C4. This is a significant result. It means that a benign surrogate to live C4 can be employed in testing of the contact sampling method. This will dramatically reduce the costs associated with contact sampling, as well as increase the ease of experimentation, because live C4 standards will not be needed to support the sampling work.



Figure 2: Peak flow stress as a function of increasing compression rate for simulated C-4 compared with live C-4. For the bimodal distributions, the percentage refers to the mass fraction of 30-40 mesh silica, with the remaining mass fraction comprised of the >230 mesh silica. Note that 1, 10, and 100 mm/s correspond to strain rates 0.042 \pm 0.004, 0.431 \pm 0.076, and 4.651 \pm 1.752 s⁻¹.

To improve the impact of this work on our ability to optimize contact sampling, it is necessary to understand the way that compounded explosives fail when they are wiped from a surface. Three modes are possible:

- 1. The weak link in the adhesion chain is at the residue-baggage interface, and the swab removes the entire residue as it passes over the baggage surface.
- 2. The weak link in the adhesion chain is within the residue, and the residue in some manner (stretching, cracking) fails internally so that a portion of the residue is removed on the wipe.
- 3. The weak link in the adhesion chain is at the residue-swab interface, and the residue is merely smeared across the baggage and none is extracted on the wipe.

To know which of these possible mechanisms is controlling, a detailed theoretical and experimental study is warranted. The theoretical work has begun. It uses a combined computational fluid dynamics discrete element modeling method (CFD-DEM) approach based on a commercial software called Star-CCM, manufactured by CD-adapco. This software uses computational fluid dynamics over a finite element grid (solving the equations of motion over a mesh of appropriate geometry) to describe the motion of the binder within

granules of compounded explosive, and it uses the discrete element method (solving Newton's laws to describe the motion of each individual particle within the binder) to provide a comprehensive description of the way that a residue of compounded explosive will deform and fail when contacted by a swab. The early work in this area has allowed us to use the CFD portion of the package to describe the velocity profile of a model binder (a non-Newtonian fluid) that is sheared between two plates. This is the same sort of load that will be applied during contact sampling of an explosive residue. Figure 3 shows these preliminary results, and compares them to the profile that would be observed in a Newtonian fluid. As can be seen, in the shear thinning non-Newtonian fluid (the model binder), the motion of the top of the fluid causes motion in the underlying fluid that extends only a small distance into the fluid, compared to what is observed in the case of the Newtonian fluid. This reflects the shear thinning nature of the binder. As this work progresses, we will add the particles into the simulation, and will describe the motion of the composite material during contact sampling. What is exciting about this work is that it is completely general in nature, and therefore will be useful for describing the behavior of any compounded explosive, including C4, Semtex, and any of the emerging HMEs that are composite in nature.



Figure 3: Finite element description of a Newtonian (top) and non-Newtonian (bottom) fluid sheared between two plates. The non-Newtonian fluid is a first generation model for the binder in compounded explosives.

B. Biennial Review Results and Related Actions to Address

B.1. Project strengths

- The benign surrogate has considerable value as a training aid for TSA operators to reduce false positives.
- The project seems to have made good progress and is well-positioned to begin transferring results.

B.2. Project weaknesses

• The end users, transition plan, and milestones need better definition.

B.3. How do you plan to address the weaknesses in Year 4?

The weaknesses identified in project R2-D.1 focus on the details of the translation of the work, not on its technical aspects. The end users for this technology include TSA, the Transportation Security Laboratory (TSL), the National Institute of Standards and Technology (NIST), and the companies involved with contact sampling (e.g., Smiths Detection, Morpho, Bruker, etc.). We will provide them with quarterly updates on our progress, which will also provide opportunities for these partners to provide direction to us. The transition plan involves the quarterly reports, presentations at conferences, specifically the annual Trace Explosives Detection (TED) workshop, and the delivery of samples of the surrogates to the government and corporate partners identified above. We will also provide these partners with the standard operating procedure (SOP) for the fabrication of the surrogates and an ingredient list so that they can make the material themselves. Appropriate milestones for this project include: 1) Determining the proper recipes for the surrogates so that their mechanical behavior matches that of the live materials; 2) Validating the mechanism of deformation and failure (removal) from surfaces under different swabbing conditions; 3) Generalizing the understanding developed to allow rapid adjustment of the SOP and recipe in order to mimic the behavior of emerging threats; and 4) Demonstrating that the surrogates perform like the live explosive in a contact sampling environment. The first two of these milestones should be attained during Year 4, but the third, which in many regards may be the most important, will take longer. This general understanding will allow us to be nimble in responding to emerging threats by quickly creating surrogates to enable training.

C. State of the Art and Technical Approach

The state of the art in understanding the behavior of compounded explosives comes from an understanding of the behavior of granulated systems, such as those found in the pharmaceutical and food manufacturing industries. In these industries, analysis such as that shown in Figure 4 is the customary path [1]. Figure 4 shows a classic analysis of the mechanical behavior of granulated solid materials under compressive load. Granulated materials have very high solids loading within a matrix of condensed fluid. Explosives such as C4 and Semtex contain very high loadings of solid RDX and PETN dispersed in a complex fluid mixture (the binder) which contains lubricants, oils, and plasticizers. In this analysis, $Str^* = \frac{\sigma_p d_{sz}}{\gamma cos(\theta)}$ and $Ca = \frac{\mu t_a d_{sz}}{\gamma cos(\theta)}$ where σ_p = the peak flow stress, d_{32} = diameter of particles in granule, γ = the surface tension of the binder liquid, θ = the contact angle of the binder against the particles in the granule, μ = the binder viscosity, and $\dot{\varepsilon}_a$ = the

strain rate of compression. Figure 4 shows two regions of behavior. In Region 1, the dimensionless peak flow stress is independent of the bulk capillary number, while in Region 2 it increases exponentially with changes in this parameter.

Current military specifications for RDX and PETN list ranges of particle diameters from 44 to 2000 μ m and 44 μ m to 800 μ m [2-4]. Due in part to the wide range of particle diameters, variability exists regarding the size of particles found in C4 or Semtex on a surface during swipe sampling [5-9]. Notably, the size of particles deposited by thumbprint on a surface of interest has not been fully evaluated, and the ability to recreate a standard print is lacking greatly [5-9]. Further, some swabs and substrates are woven materials, leading to an entrapment problem that has not been fully evaluated [6-8].



Figure 4: Mechanical analysis of the solid dynamics of granular solids comprised of glass beads in Newtonian fluids (binders) of different viscosity.

Neither has the development in sampling technique been firmly established [5]. For IMS, certain standards apply: a swab must effectively remove solid particulates from a surface, withstand temperatures up to 300°C as employed by the IMS, and be affordable [5-6]. Most current studies consider either cloth or Teflon-coated fiberglass swabs [5-9].

Several parameters are usually not controlled during the development of a successful wiping technique [8]. In particular, variability exists in the applied force of a swipe, the surface area covered, the swab material, the roughness of the swab and substrate materials, and the swipe velocity, among other characteristics [5, 8-9]. The applied load may range from approximately 3 to 60N [5-7]. The Verkouteren group claims that the critical parameters in determining removal efficiency are applied load and the translational force required to overcome the frictional resistance to maintain a constant velocity [5]. The group also claims a direct linear correlation between increased applied force of swiping and particle removal efficiency [5, 7, 8]. The viscosity at the speeds representative of those found in an airport security setting has not been tested. Verkouteren reports a swipe speed of 0.7 cm/s, while methods reported by the Environmental Protection Agency indicate swipe sampling speeds of 10 and 17 cm/s [5, 10]. Note that the speed of a swipe directly correlates to the strain rate, and thus the viscosity of the non-Newtonian binder.

Generally, previous studies have focused on manipulating applied force and attempting to create consistent methods to be employed by swab operators [5, 8, 9]. However, the above studies do not typically analyze the effects of roughness on the adhesion of a particle to either the swab or the substrate. Overall adhesion forces are not fully evaluated, as most tests performed attempt purely to establish a methodology. The deformation and failure of the composite are not evaluated.

Explosives compounds show similar, although slightly different, behavior from the granules in systems using water (a Newtonian fluid) as the binder, for two reasons. First, the binder in Figure 4 is water, which is a Newtonian fluid, while the binder in the compounded explosives is non-Newtonian (shear thinning). Next, the binder in Figure 4 has a very low viscosity (~ 1 Pa s) compared to that of the binder in the compounded explosives (ranging from ~ 10 to ~ 3000 Pa s). Figure 5 (on next page) shows how the various simulants developed in this study behave when evaluated using the approach shown in Figure 4. As can be seen, these surrogate materials (within the blue circle) behave in a manner very similar to that of idealized granular composites (within the blue rectangle). The shift above the idealized curve (solid line) is attributed to the non-ideality of the binder in the surrogates. As the simulants are further refined, through the addition of silica beads with a bimodal size distribution that mimics that in real C4, this analysis will be repeated to compare the behavior of the live material to that of the simulants.



Figure 5: Mechanical analysis of the solid dynamics of granular solids comprised of silica beads in C4 binder and silica beads in PDMS (binders) of different viscosity.

To develop an authentic benign surrogate to live C4 and Semtex, it is necessary to demonstrate that the surrogate demonstrates the same stress response as the live materials, that it has a comparable overall viscosity as the live material, and that it is removed from surfaces in the same manner as the live material. This characterization is performed using a slip-peel tester, which wipes across a surface at a known rate with a known applied downforce. A contaminant of interest is placed on the surface, and its removal during the controlled wiping is documented. A key assumption of this work is that the adhesion between the binder and the explosives particles is immaterial to the explosives removal that will be observed, and that the removal is dominated by the binder adhesion to the substrate on which the residue has been deposited. This will be tested in the future work as outlined below.

D. Major Contributions

Year 3

- 1. Developed CFD-DEM method for evaluating the mechanics of compounded explosives and began evaluation of non-Newtonian fluids representative of binder in compounded explosives.
- 2. Disseminated the results of Year 2 studies in the form of multiple manuscripts in refereed journals.

Year 2

- 1. Determined that the behavior of compounded explosive residue on a surface is controlled by the properties of the binder and the size distribution of explosives within the binder.
- 2. Determined that the adhesion between the binder and the explosive particles is irrelevant to the behavior of the explosive compound.
- 3. Determined that a benign surrogate for a live compounded explosive can be fabricated if the particle

size distribution of the explosive and the mechanical properties of the binder can be matched.

- 4. Developed a theoretical framework for interpreting the behavior of the compounded explosives and for comparing surrogates to live materials.
 - a. These contributions enable the design of improved swabs for contact sampling.
 - b. These contributions enable the design of improved swabbing protocols for contact sampling.
 - c. These contributions enable the design of a benign surrogate for live compounded explosives, which will allow the community to work to develop contact sampling methods in a much more straightforward manner.

E. Milestones

Specific milestones that were achieved in Year 3 include:

- 1. Completion of characterization of mechanical properties of C4 and Semtex.
- 2. Final determination of controlling mechanism(s) in removal of C4 and Semtex from surfaces via contact sampling.
- 3. Completion of fabrication of benign surrogates for C4 and Semtex for use in contact sampling studies.
- 4. Proof of concept that benign surrogates are appropriate replacements for live C4 and Semtex in contact sampling environments.

For Year 4, the following four major milestones are to be achieved: 1) Determining the proper recipes for the surrogates so that their mechanical behavior matches that of the live materials; 2) Validating the mechanism of deformation and failure (removal) from surfaces under different swabbing conditions; 3) Generalizing the understanding developed to allow rapid adjustment of the SOP and recipe in order to mimic the behavior of emerging threats; and 4) Demonstrating that the surrogates perform like the live explosive in a contact sampling environment. Very good progress has been made on the first two milestones, but the third is just underway. It is expected that the first two milestones will be completed during Year 4, while the third and fourth will take up to another year to finish. To bring the work to completion, the recipe for benign surrogates must be finalized and the surrogates must be demonstrated to behave similarly to the live materials during contact sampling. In addition to validating the recipe for benign surrogates and demonstrating that they have similar properties under load, detailed contact sampling studies will be completed to prove the preliminary hypothesis that the behaviors of the bulk materials are good predictors of the contact sampling behavior. These studies will be completed using a recently-purchased Crockmeter.

F. Future Plans

The objectives of this project are:

- 1. To determine the controlling mechanisms in the removal of compounded explosives from surfaces.
- 2. To develop benign surrogates for compounded explosives so that the community can develop sampling protocols without requiring the use of live explosives.

To complete Objective 1, we will finish the studies on the mechanical properties of compounded explosives and surrogates, and will then use a slip-peel tester to study the removal of these materials from surfaces of interest. The mechanical properties and behaviors of the surrogates and live compounds will then be correlated with the removal observed with the slip-peel tester. This correlation will be the basis for determination of the mechanism of explosives removal. If necessary, a high-speed camera will be employed to study the deformation of the residues during the removal process, as indicated in Figure 6 (on the next page). We will use this information when we collaborate closely with Project R2-A.1. In that project, swabs are designed, fabricated and tested. What is necessary to drive that project onward is better understanding of the mechanical behavior of residues, so that appropriate design decisions can be made. When we fully characterize the mechanical behaviors of live C4 and live Semtex, including characterizing their behavior using compression tests and using the slip-peel tester, we will determine the key features in the swab and the swabbing proto-

cols that will allow these residues to be efficiently harvested during trace explosives detection.

In order to complete Objective 2, development of benign surrogates for C4 and Semtex, we will characterize the behavior of the surrogates that we have begun to develop, using both the slip-peel tester and the compression tests described above. Based on the performance of these tests, we will modify the binder and particles used in the surrogates until we match the mechanical and removal properties of the live residues. This will provide the community with "safe" materials with which to work when developing next generation contact sampling protocols.



To complement these experimental studies, we will perform the combined CFD-DEM modeling described above.

Figure 6: Schematic of slip-peel tester modified to allow study of residue removal process.

This work will allow us to predict the removal behavior that is observed with the slip-peel tester, as well as the deformation seen during compaction of explosives granules. With this understanding, we will begin the process of transferring these surrogate materials to the field where they can be used as training aids for screeners, and for the optimization of contact sampling protocols.

III. RELEVANCE AND TRANSITION

A. Relevance of Research to the DHS Enterprise

- 1. Improve detection of trace explosives on luggage and persons through contact sampling/IMS.
 - a. Metrics: Research in the Beaudoin lab has shown that swab-based contact sampling may miss as much as 30% of the residue on a surface. The goal of this research is to substantially improve the detection rate by enabling the design of swabs that effectively interrogate surfaces and harvest residues. To design these swabs, it is essential to understand how the residues adhere to themselves and to surfaces.
- 2. Develop benign surrogate for live compounded explosive that can be used by researchers to develop improved methods for contact sampling.
 - a. Metrics: Only a small fraction of interested labs can perform research to develop improved contact sampling methods, as only a small fraction of such labs can receive and handle explosives. By developing benign surrogates for live compounded explosives, we wish to double the number of labs that are performing research to improve contact sampling.
- 3. Develop training aids for use in preparing screeners for efficient and effective contact sampling for trace explosives detection and transition these to the field.
 - a. Metrics: The aids will require the insertion of a colorant to allow optical assessment of the effectiveness of sampling when training screeners, as well as a method to detect the residual sample on a surface with appropriate resolution. We expect to introduce these at the Purdue airport and at one or two commercial airports.

B. Potential for Transition

The recipe for creating the benign residues will be transferred freely throughout the community through publications and presentations. This will enable improved research on contact sampling of trace residues.

C. Transition Pathway

We will share the recipe for the newly-developed benign surrogates, as well as the critical information regarding the controlling steps in residue removal from surfaces via conference presentations, reports, and journal articles. We will use this information to inform project R2-A.1, which is focused on the development of optimal swabs and swabbing protocols for residue capture by contact sampling. The requisite information will be collected and transferred to the security enterprise. We will work in concert with TSA to develop training protocols using these benign aids.

D. Customer Connections

Stefan Lukow at Morpho Detection is making measurements with the swabs from R2-A.1. As a result, they have an interest in this project, as does Reno DeBono at Smiths, and Cindy Carey at Bruker.

IV. PROJECT ACCOMPLISHMENTS AND DOCUMENTATION

A. Education and Workforce Development Activities

- 1. Course, Seminar, and/or Workshop Development
 - a. Trace Explosives Sampling for Security Applications (TESSA02) workshop organized and led a workshop attended by roughly 70 member of the trace explosive detection community for the purpose of developing a common, well-accepted approach for baselining contact sampling effectiveness.
- 2. Student Internship, Job, and/or Research Opportunities
 - a. Two PhD students, Melissa Sweat and Leo Miroshnik, worked on this project. An undergraduate student, Andrew Parker, and a high school student, Hannah Burnau, also worked on the project.
- 3. Interactions and Outreach to K-12, Community College, and/or Minority Serving Institution Students or Faculty
 - a. One high school student participated in research in my lab during the summer of 2015, and another will join in the summer of 2016.
- 4. Other Outcomes that Relate to Educational Improvement or Workforce Development
 - a. Coordinated transfer of High Tech Tools and Toys program to Purdue First Year Engineering Program.

B. Peer Reviewed Journal Articles

Pending-

- 1. Sweat, M.L., Parker, A.S., Beaudoin, S.P. "Compressive Behaviour of Idealized Granules for the Simulation of Composition C-4." *Propellants, Explosives, and Pyrotechnics*, In Review, 2016.
- 2. Thomas, M., Krenek, E., and Beaudoin, S. "Capillary Forces Described by Effective Contact Angle Distributions via Simulations of the Centrifuge Technique." *MRS Advances*, In Press, 2016.

C. Other Publications

- 1. Chaffee-Cipich, M, Hoss, D., Sweat, M., and Beaudoin, S. "Contact between Traps and Surfaces during Contact Sampling of Explosives in Security Settings." *Forensic Science International*, 260, March 2016, pp. 85-94.
- 2. Thomas, M. and Beaudoin, S. "An Enhanced Centrifuge-Based Approach to Powder Characterization: Particle Size and Hamaker Constant Determination." *Powder Technology*, 286, November 2015, pp. 412-419.
- D. Other Presentations
 - 1. Seminars
 - a. Beaudoin, S. TESSA 02 (2nd Annual Workshop: Trace Explosives Sampling for Security Applications). Hosted by DHS-sponsored ALERT Center of Excellence, Boston, MA, August 2015.
 - b. Beaudoin, S. TESSA 02 (Trace Explosives Sampling for Security Applications), Leadership group planning workshop in preparation for 2nd Annual Workshop. Hosted by DHS-sponsored ALERT Center of Excellence, Boston, MA, April 2016.
 - c. Sweat, M., Parker, A., and Beaudoin, S. "Compressive Behavior of Simulated Explosive-Filled Granular Material." Annual Meeting of the American Institute of Chemical Engineers, Salt Lake City, UT, November 2015.
 - d. Fronczak, S., Dong., J., Thorpe, J., Franses, E., Beaudoin, S., and Corti, D. "A New Method for Determining Hamaker Constants of Solids Using Atomic Force Microscopy." Annual Meeting of the American Institute of Chemical Engineers, Salt Lake City, UT, November 2015.
- E. Student Theses or Dissertations Produced from This Project
 - 1. Sweat, M. "Compressive behavior of simulated explosive-filled composites." PhD Dissertation, Chemical Engineering, Purdue University, January 2016.
 - 2. Thomas, M. "Enhanced centrifuge-based approach to powder characterization." PhD Dissertation, Chemical Engineering, Purdue University, August 2015.
- *F. Requests for Assistance/Advice*
 - 1. From DHS
 - a. Requested to lead the TESSA (Trace Explosive Sampling for Security Applications) activities, including the TESSA02 Workshop on August 5 6, 2015.

V. REFERENCES

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