THE Ge(Li) IV PLUTONIUM-239 PACKAGE COUNTER

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ABSTRACT

The Ge(Li) IV is a passive plutonium-239 package counter system capable of detecting and quantitatively analyzing a wide weight range of plutonium-239 in any standard Hanford waste container in compliance with and exceeding waste disposal objectives of less than eight microcuries plutonium-239 per kilogram $\pm 30\%$ at the 95% confidence level. The packages are rotated and oscillated; and the package radiation collimated to compensate for nonhomogeneity. An external radiation source emission is passed through the packages to provide data for radiation self-absorption compensation. Four coaxial Ge(Li) detectors are employed to provide a large area of detecting surface. The 413.7 keV gamma emission of plutonium-239 from within the package and the 355.3 keV gamma emission of an external barium-133 source are pulse-height analyzed and data-reduced under computer program control; the computer reads out grams of plutonium-239 in the package, the computed self-absorption correction, and the precision at the 95% confidence level of both values. The Ge(Li) IV system and its performance are described.

THE Ge(Li) IV PLUTONIUM-239 PACKAGE COUNTER

INTRODUCTION

Passive plutonium package counting systems rely on the gamma emission of plutonium-239 to provide a signal suitable for quantitative analysis. The ^{239}Pu gamma spectrum is complex, the specific activity is low, and the interference from other plutonium isotopic daughters and fission products is ever-present.

Three characteristics are desirable in a package counting system: sensitivity, selectivity, and the ability to correct for self-absorption. Sensitivity has always been the long-suit of Geiger-Mueller tube detectors and NaI(Tl) scintillator detectors. Selectivity is the forte of semiconductor detectors. Since Geiger-Mueller tubes have never had selectivity and the alkaline halide detectors appear to be limited to about 6 percent resolution, Full Width at Half Maximum (FWHM), or 25 keV in the 400 keV range, improvement of the sensitivity of the semiconductor detector appears to be the best alternative for system optimization. Lithium drifted germanium detectors [Ge(Li)] are commercially available with FWHM resolution of less than 3 keV in the 400 keV range; and though more sensitive per unit volume than the other detector types, the available detector volume and area is very limited. Fifty square centimeters of Ge(Li) detection surface area were calculated to be sufficient to achieve minimum measurement goals of eight microcuries plutonium-239 per kilogram of matrix $\pm 30\%$ at the 95% confidence level. The 50 cm² detection surface area is achieved in Ge(Li) IV by mounting four 20 cubic centimeter, closed end detectors with the closed ends in a common plane.

Application of self-absorption correction is relatively simple when the source is of uniform size and distribution in a consistent density medium; however, a waste carton cannot be relied upon to meet these criteria. The best method of determining self-absorption correction is to measure the radiation from a known quantity of one of the emitting substances in the package being analyzed. This, unfortunately, is self-defeating; at best it doubles counting time since such a self-absorption correction requires that the package be counted once with the known source and once without. An alternative, the one which was chosen for application to the Ge(Li) IV ^{239}Pu package counting system, is to measure the transmitted radiation from an external long-lived isotope; this radiation source must be close in energy of emission but not interfering with that of ^{239}Pu , and not normally found associated with ^{239}Pu . Such a source is found in ^{133}Ba .

The Ge(Li) IV Plutonium-239 Package Counter Operating and Servicing Procedures are available as ARH-2021. The procedures are specific to the components incorporated in the Ge(Li) IV system and to the mode of operation in use at Atlantic Richfield Hanford Company. The procedures are made available as a potential aid in specifying units for a like system or more fully understanding specific operational details of Ge(Li) IV.

SUMMARY

The Ge(Li) IV ^{239}Pu package counter developed by the Atlantic Richfield Hanford Company consists of four Ge(Li) detectors in a vertical array having a combined active detection surface area of more than 50 cm². The transmitted 355.3 keV gamma emission of barium-133 is used to determine the self-absorption correction for a rotated package. Gamma emissions from the ^{133}Ba and the ^{239}Pu are pulse-height analyzed and computer-processed to read out grams of plutonium-239 in the

package. Maximum sensitivity is 9.0 mg ^{239}Pu in a 127-liter carton, or 70 μg ^{239}Pu per liter, in a 1024-second counting period.

SYSTEM DESCRIPTION

The Ge(Li) IV ^{239}Pu package counting system consists of the following component blocks:

Figure 1 shows the physical layout of the system in the trial configuration which was used to debug the system and to ascertain its analytical capabilities. The scaffold planking simulated the floor level in the permanent installation site. The ^{133}Ba self-absorption source is located in the stand at the left. Gamma emissions from the ^{133}Ba source pass through the package on the turntable and join with the ^{239}Pu gamma emissions in interaction with the four-barreled Ge(Li) detector assembly. The adjustable blinders limit the amount of ^{239}Pu emission received by the detectors to a wedge-shaped sector which shields out most background and a portion of the signal from the package. The radiation from the ^{133}Ba source that is not attenuated by the package is received at the detectors.

The rack containing the shaping-analyzing-computing components is at the far right of Figure 1, together with the

teletype. The detector signal pulse is shaped in the pre-amplifier and in the amplifier and baseline restorer. The analog pulse is converted to a digital signal and pulse-height analyzed. The stored gamma spectrum is programically data-reduced by the computer under teletype control. System output consists of a printout sheet containing package identification, grams of contained ^{239}Pu , the self-absorption of the package matrix, and the precision of the data.

PERFORMANCE

CAPACITY

Provision has been made in the Ge(Li) IV ^{239}Pu Package Counting System for counting standard Hanford containers from the size of small pasteboard pill boxes to double, stacked 55-gallon drums. Specific adaptors to the turntable have been provided for 10 cm diameter by 15 cm high tins, 127-liter waste cartons, and 55-gallon (208-liter) drums.

Figure 1 is an overall view of the Ge(Li) IV Counter in a temporary setup which was used to perform preliminary mechanical and instrumental tests. The planks on the scaffold simulate the future floor level of the permanent installation. From left to right the component units are the self-absorption source stand, the turntable, the detector cart, the analyzer-computer, and the teletype readout. Both the self-absorption source stand and the detector cart are indexed to a 1" x 2" rail mounted on the floor on opposite sides of the turntable. In general, the high sensitivity (trace to 20 grams ^{239}Pu) counting position is established with the self-absorption source and the greatest projection of the detector blinders within four centimeters of the maximum rotational diameter of the package being counted. In this configuration, the minimum detection capability for the Ge(Li) IV counter is 70 micrograms of ^{239}Pu per liter $\pm 30\%$ at the 95% confidence level (CL) in

large waste containers for a 1024-second counting period. Seven milligrams per liter ^{239}Pu $\pm 5\%$ at the 95% CL are attainable in the same counting configuration with a 1024-second counting period. Longer count times and smaller packages improve both the precision and the lower detection limit.

The range of ^{239}Pu sensitivity in the counting configuration closest to the package is from trace quantities to approximately 20 grams. For greater concentrations of ^{239}Pu , the detector is moved back from the package to a low sensitivity position a distance approximately equal to 1.3 meters from the center of the turntable to the face of the detectors. This is a common position for all package sizes; however, the calibration factors are not the same for each package size. The self-absorption source stand remains in the same position relative to the package for higher weights of plutonium, but the ^{133}Ba source strength is increased. In the low sensitivity position there is no real upper limit to the amount of ^{239}Pu which can be analyzed to a precision of better than $\pm 5\%$ at 95% CL. Criticality control and the advisability of using a less sensitive detector array become overriding considerations before the upper limit of detection is reached with the Ge(Li) IV system.

SIGNAL AVERAGING

The waste containers to be analyzed for ^{239}Pu will not be of uniform density or distribution so they are rotated to minimize the effect of non-uniform distribution of the ^{239}Pu . Rotation averages the radiation centers but radiation intensity varies with the square of the distance between the source and the detector. If the source radiation intensity received by the detectors when the source is at the center of rotation is compared with the amount received when the same source is located at the outer diameter of rotation, the latter radiation intensity will be higher. The relative ratio will

be higher the closer the detector is to the package and the larger the diameter of the package. The blinders shown in Figures 1 and 2 are used to limit the radiation impinging on the detectors to a wedge which does not include the entire rotated diameter of the package. In this way all of the radiation from the center of the package is analyzed, but the radiation from a source near an edge is only intermittently analyzed.

A package taller than the detector array, 32 centimeters, is oscillated vertically while it is rotated. The oscillation also causes a detection gradient which is highest at the center of the rotational axis and lowest at the extremes. The gradient is more varied the closer the detector is to the package and the taller the package, but it is still considerably less varied than the gradient experienced without package oscillation.

Table I is a map of the detection gradient of a 127-liter carton in the high-sensitivity geometry. The 127-liter carton is 45.5 cm x 45.5 cm x 61 cm high and has a rotating diameter of approximately 65 cm. The map represents one quadrant of rotation. Maps of the remaining three quadrants would be mirror images of Table I due to the symmetry of the package. Table II is the quadrant detection gradient map of the same package in the low sensitivity geometry.

SELF-ABSORPTION CORRECTION

There are at least two modes of self-absorption taking place in the package; the absorption occurring in the high-density emitting centers and the absorption occurring in the lower-density, non-emitting bulk contents of the sample package. The ^{133}Ba self-absorption source is only determining an average correction for the latter mode of self-absorption. The combined self-absorption correction applied

TABLE I
A DETECTION GRADIENT MAP OF ONE QUADRANT OF
A 127-LITER CARTON COUNTED IN THE
HIGH SENSITIVITY GEOMETRY OF Ge(Li) IV

		Center ↓	→ 6 cm	→ 6 cm	→ 6 cm	→ 6 cm	→ 6 cm
Midplane	→	100%	98%	102%	104%	114%	100%
	↓						
10 cm		97%	98%	96%	101%	107%	92%
	↓						
10 cm		83%	87%	84%	87%	92%	75%
	↓						
10 cm		68%	69%	68%	71%	67%	55%

TABLE II
A DETECTION GRADIENT MAP OF ONE QUADRANT OF
A 127-LITER CARTON COUNTED IN THE
LOW SENSITIVITY GEOMETRY OF Ge(Li) IV

		Center ↓	→ 6 cm	→ 6 cm	→ 6 cm	→ 6 cm	→ 6 cm
Midplane	→	100%	105%	104%	106%	107%	100%
	↓						
10 cm		97%	99%	100%	102%	104%	106%
	↓						
10 cm		99%	100%	100%	100%	100%	100%
	↓						
10 cm		99%	99%	99%	98%	100%	101%

to the reduction ratio of the ^{133}Ba emission is experimentally determined.

The gamma spectrum shown in Figure 3 illustrates many of the reasons which led to the selection of ^{133}Ba as a self-absorption source and to the selection of the specific peaks of ^{133}Ba and ^{239}Pu used in the computations. The spectrum

points up the following points:

- The peaks of the ^{133}Ba gamma spectrum do not interfere with the major peaks of the ^{239}Pu gamma spectrum.
- The 355.3 keV ^{133}Ba peak and the 413.7 keV ^{239}Pu peak are completely resolved from each other and associated peaks.
- These peaks are close in energy to each other, yet their associated Compton continuums do not cause interference.
- Both of the chosen peaks have the highest specific activity of their isotopic parents in this energy range.

What is not apparent from the spectrum segment is that ^{133}Ba is long-lived with a half-life of 3893 days; and ^{133}Ba is never naturally associated with ^{239}Pu in any Hanford production or waste system.

SYSTEM COMPONENTS AND FUNCTIONS

Specific brands and models of components are cited in this section in fairness to the manufacturers who successfully complied with the specifications for the Ge(Li) IV system. This is not to imply that other manufacturers cannot supply equally acceptable components.

PULSE HEIGHT ANALYSIS AND DATA REDUCTION

The gamma spectral data are pulse height analyzed in a Northern Scientific Model NS-636, 1024 channel analyzer (Figure 4). The pulse height analyzer is interfaced through an NS-440 interface to a Nova 4K by 2^{16} computer. All interactive Input/Output is by means of a Teletype Model ASR33 keyboard.

In addition to the routine programs available from Northern Scientific for the manipulation of data in their analyzer-computer hybrids, there are three custom programs specifically included for data reduction in the Ge(Li) IV system.⁽¹⁾ The program teletype call codes are "S", "F", and "RF". The "S" command initiates a five-point smoothing program on all data stored in pulse height analyzer memory except for the contents of channel zero which contains the time of analysis in seconds. The "F" command initiates a calculation factor loading cycle. A change to any calculation factor requires reloading all the factors.

TABLE III
"F" COMMANDS

Code	Units	Meaning
II	Channels	Integer number of channels on either side of the highest content channel in a region of interest which will be the limit of search for the lowest content channel for use in the determination of noise continuum.
IX	Channels	Integer number of channels on either side of the highest content channel in a region of interest which will be included in the integration of gross peak area.
R	None	Root value used in Absorption Correction (AC) calculations.
BA	Counts/second	Net area of the ^{133}Ba self-absorption source <i>used</i> without absorption.
BN	g $^{239}\text{Pu}/\text{c/s}$	Calibration value for the geometry <i>used</i> .
BB	None	A ratio of the established, time-corrected BA value to the same value determined today.
BK	g ^{239}Pu	Background grams of ^{239}Pu .

The command "RF" initiates the data reduction of the spectral and time data stored in the memory of the pulse height analyzer. Net peak areas are calculated for both the 355.3 keV ^{133}Ba peak and the 413.7 keV ^{239}Pu peak according to the calculation method described in ARH-1877.⁽²⁾ They are labeled CN_1/T and CN_2/T , respectively.

Percent precision at the 95% CL is calculated by the formulae:

$$\% \text{ @ 95\% CL of 355.3 keV peak} = P1 = \frac{196\sqrt{CN_1/T} + 2 \times \text{noise continuum}}{CN_1/T}$$

$$\% \text{ @ 95\% CL of 413.7 keV peak} = P2 = \frac{196\sqrt{CN_2/T} + 2 \times \text{noise continuum}}{CN_2/T}$$

The precision values calculated are representative of counting statistics only.

$$\text{Absorption Correction} = AC = \frac{R/\overline{BA \times BB}}{CN_1/T}$$

Current values of R have been determined experimentally from known standard packages which vary from R values of three for low density material to six for plutonium metal, oxides, fluorides, etc. Provision has been made in the "F" program for R values from 1 to 9.

$$\text{grams } ^{239}\text{Pu} = GP = \left(\frac{CN_2/T \times BN \times AC}{BB} \right) - BK$$

The precision assigned the GP value is the poorer of the P1 or P2 values and is representative of counting statistics only.

The sequence of operations used to obtain data, to reduce it to grams plutonium-239, and to report the results is as follows:

- a. Type K@ [@ = space] to Kill previous data in the pulse height analyzers. Preset time selector should already be set for the live time the package is to be counted.

- b. Remove the old package and load the new package on the turntable.
- c. Type MPA@ to start the Multiple Pulse height Analysis for a preset live time.
- d. After the data accumulation, type S@ to smooth the data.
- e. Type RF, turn on paper tape punch, type @, type any string of alpha numeric characters excluding a space to identify the package, type @.
- f. Turn off paper punch tape.

The operator now has a typed copy of the analytical data and a paper tape containing only reduced data in a report format. The paper tape can be used to generate a report suitable for circulation or to enter the data into a larger, inventory computer file.

Any time the type of package being analyzed is changed, the following items would have to be checked and changed if necessary:

- a. Detector cart position.
- b. Blinder angle.
- c. Barium-133 self-absorption source.
- d. Turntable oscillation period.
- e. "F" factor values.

All of these items can be determined beforehand and performed from a single check list.

DETECTORS

Figure 5 is a view of the Ge(Li) IV detector assembly. There are four 20 cm³ coaxial Ge(Li) detectors in a vertical array to give a combined active detector area of greater than

50 cm². The height of the detector array is 32 cm. Princeton Gamma Tech, Inc., supplied the detector system with each detector separately mounted in a detachable minicryostat to permit repair of a single detector without shutting down the entire counting system. Figures 2 and 5 show the detector manifold minus the number 2 detector; and the data for Table II were obtained with this configuration.

Each minicryostat consists of a detector, detector mount, cryogenic envelope, and threaded cold finger extension. A vacuum valve is recessed into one side and the electrical pass-through is located on the opposite side. The vacuum manifold can also be seen in Figure 5. The four valve handles in line are located over, and connect with, the individual minicryostat valves. The rear valve handle marks the location of the main cryostat vacuum valve. The minicryostat cold finger extension is secured by a nut to the dipstick cold finger in the vertical section of the main cryostat. The small plates at the rear of the main cryostat provide access to the cold finger nuts and provide entry for a two-liter per second Vac-ion pump.

Figure 2 shows the electrical manifold which covers and shields the minicryostat electrical pass-throughs and the signal matching network. All signals are combined and fed to a single preamplifier. Amplification and shaping are provided by a Tennelec TC 137 preamplifier, a TC 202 amplifier, and a TC 611 baseline restorer. The signal is AC coupled to a Northern Scientific NS629 analog to digital converter.

Detector bias of -1550 VDC is supplied through the preamplifier by an AC-powered, NIM, Power Design, Model AEC-1000, DC power supply.

Resolution of the system is rated as better than 4.0 keV FWHM at the 1332 keV peak of ⁶⁰Co. Actual performance is better than 3.0 keV FWHM for the 355.3 keV peak of ¹³³Ba. Full Width at Tenth Maximum (FWTM) is less than 2.0 times the

FWHM value of any peak in the operating range. All performance data have been taken through one-sixteenth inch of lead to reduce the amount of irrelevant low-energy radiation received by the detectors and processed by the preamplifier, amplifier, and input gate of the analog to digital converter. Without the lead, the analog to digital converter dead times are significantly increased with no gain in usable data but with a real time lost in counting.

DETECTOR CART

The entire detector assembly and its supporting hardware are contained on the detector cart shown in Figure 2. The detector assembly is in a standard right angle dipstick configuration. A self-filling collar for liquid nitrogen entry is in the neck of the 35-liter dewar. Between the self-filling collar and the out-of-dewar section of the main cryostat is an aluminum shelf⁽³⁾ which supports the Varian Vacion pump control unit and the adjustable blinders. A lead brick partially counterbalances the weight on the shelf but leaves the blinder end of the shelf heavy regardless of the angular opening of the blinders. The shelf is leveled by means of a "Little Jack" laboratory jack between the dewar handle and the lower surface of the blinder shelf. Thus the shelf is pivoted around the cold finger and lightly supported at one end only. If the detector assembly is moved too close to a rotating package or if it is struck from the side, the detectors are free to rotate out of harm's way.

The dewar rests on an aluminum plate supported by an inch of styrofoam. The styrofoam acts as both a cushion against mechanical vibration and as an electrical insulator isolating the detector ground from any extraneous ground loops. The ground of the detector assembly is carried to the instrument rack only through the preamplifier cable shielding.

The top of the detector cart, which supports the dewar-detector assembly, is hinged at the rear. At the front end, the entire weight of the assembly is supported on a Stratham strain gauge [Stratham Universal Transducing Cell Model UC3 (Gold Cell)] with a 0 to 100 pound range weight reduction adapter [Stratham Model UL4 Load Cell Accessory]. The weight of the assembly is monitored as a measure of liquid nitrogen level in the dewar. An alarm level for the minimum allowable weight of liquid nitrogen is calibrated and set on the API Model 502-L, single high set point meter seen in Figure 2. When the liquid nitrogen level decreases below a safe amount, an audible alarm contained in the base of the cart sounds.

The detector cart is provided with guides at each end so that it may track along a 1" x 2" rail mounted on the floor. The rail is pre-drilled for routine, precalibrated package counting geometries and the cart is indexed to these locations by means of the indexing pin at the rear of the cart.

BLINDERS

Figures 1, 2, and 5 show the detector blinders and their relationships to other components of the Ge(Li) IV counter. Each blinder⁽³⁾ is built up from an eighth-inch aluminum backing plate, one-half inch of lead, 35 mils of cadmium, and 10 mils of copper. The latter two items are used to reduce the amount of re-emitted Pb-K X-ray which reaches the detectors.

The blinder angles are individually adjusted. Care must be exercised not only to maintain symmetry between the blinders themselves, but also between the blinders and the detector heads. Blinder angle settings corresponding to counting geometries are maintained as part of the calibration and are recorded in units of linear measurement between the leading edges of the blinders.

The blinder angles for a particular counting geometry are established by first setting the blinders at an approximation of the final opening. A ^{239}Pu source is placed in the geometric center of a package representative of the geometry being calibrated and a normal analysis is made for ^{239}Pu . The same ^{239}Pu source is moved to a point approximately five centimeters from the corner (or edge) of the package and on the horizontal midplane. Another normal analysis is made. Adjustments are made and recorded on the angle of the blinders and analyses made until the last analyzed value of grams ^{239}Pu equals the first. The blinder opening is then recorded.

TURNTABLE

The turntable is intended to be recessed into the floor. The oscillating piston of the turntable can be fully retracted to rest flush with the floor as an aid to loading heavy packages. A retaining ring anchored to the floor prevents any lateral movement of the turntable during the loading operation.

The turntable, as shown in Figure 6, is a square cross-sectioned piston in a square "cylinder." Both piston and cylinder rotate at a constant four revolutions per minute, supported on ball bearings, and driven by a standard automotive ring gear (see Figure 7). A ball nut is anchored at the base of the piston. Through it and through the base of the cylinder runs a ball screw. The lower end of the ball screw is terminated in a four-inch brake drum. A magnetic brake is concealed below the ball race plate and it is actuated by means of switches mounted on the self-absorption stand. When the brake is closed, gripping the brake drum, the ball screw is not free to rotate with the turntable, causing the piston to climb the screw supported on the ball nut. When the brake is released, the screw is free to rotate and the piston falls on an air column until it reaches: a stop;

the bottom of the cylinder; or until the brake is reactivated. The rate of climb for the cylinder is 10 cm a minute. The Teflon guides and a Teflon "ring" are used to stabilize and center the piston in the cylinder.⁽³⁾

The piston height is 113 cm and the maximum period of safe oscillation with a load is 90 cm. Piston stops and slots have been provided which will permit non-oscillating rotation of the turntable for packages less than 32 cm in height at a piston height of 86 cm. The additional height needed to align small packages with the center of the detector array is provided with customized stages. Figure 6 shows the standard stage which has been provided for the larger packages with the tracking flange for the oscillation limit switch contact levers. Detachable guides have been provided which fit the standard stage and center the 55-gallon drum or the 127-liter carton. The load limit for the turntable is 250 kg.

SELF-ABSORPTION SOURCE STAND

Figures 6 and 8 show the self-absorption source stand. A hinged panel contains the switches that activate and deactivate the magnetic brake, controlling turntable oscillation.⁽³⁾ The upper switch position is fixed to bring the bottom of a package which is to be oscillated level with the lower detector. The lower switch position is adjustable, permitting adjustment of the oscillation of the top of the package to the level of the top detector. For packages too small to require oscillation, the hinged switch panel is swung out of the way.

Barium-133 sources are loaded in the tube receiver at the top of the self-absorption stand as shown in Figure 6. A point source is loaded for small packages and a line source approximately 25 cm long is loaded for oscillated packages. In either case the source is adjusted to a precalibrated position approximately four centimeters from the rotational

diameter of the package. The precalibrated positions are fixed by means of an indexing pin in a 1" x 2" aluminum rail attached to the floor. The front guides for the indexing rail can be seen in Figure 8.

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FIGURE 1
THE Ge(Li) IV ^{239}Pu PACKAGE COUNTER
SITING SIMULATION

FIGURE 2

THE $\text{Ge}(\text{Li})$ IV DETECTOR ARRAY, LIQUID NITROGEN
DEWAR, LIQUID NITROGEN LEVEL MONITORING SYSTEM,
AND DETECTOR CART

FIGURE 3

A SEGMENT OF A BARIUM-133-PLUTONIUM-239 GAMMA SPECTRUM POINT PLOT

FIGURE 4
NORTHERN SCIENTIFIC MODEL NS-636 PULSE HEIGHT
ANALYZER WITH ^{133}Ba - ^{239}Pu GAMMA SPECTRUM
DISPLAYED ON THE OSCILLOSCOPE

FIGURE 5

THE Ge(Li) IV DETECTOR ARRAY.
ONE MINICRYOSTAT WITH ITS DETECTOR
AND ONE BLINDER HAVE BEEN REMOVED

FIGURE 6

Ge(Li) IV PACKAGE TURNTABLE AND
SELF-ABSORPTION SOURCE STAND

FIGURE 7

Ge(Li) IV TURNTABLE DRIVE AND BALL RACE

FIGURE 8

Ge(Li) IV SELF-ABSORPTION SOURCE STAND

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