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Energy Dispersive X-ray Fluorescence Spectrometer EDX-7200

Application News

Analysis of Inorganic Elements in Blood Using EDXRF

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User Benefits

- Inorganic elements in blood can be quantified at the µg/mL level without any complicated sample pretreatment.
- Qual-quantitative analysis is possible using the FP (Fundamental Parameter) method, which does not require standards.
- Qual-quantitative results are obtained within 10 minutes per sample.

■ Introduction

Inorganic elements such as potassium, calcium, and iron are essential substances for maintaining normal metabolism and vital activities in the human body. In contrast, heavy metals poisoning can result from accidents or criminal incidents, and the causative elements are diverse. Some of these elements are highly toxic, but because they often lack specific poisoning symptoms, early identification can be difficult. Therefore, rapid analytical methods are needed.

Inorganic elements are generally analyzed by ICP-AES or ICP-MS. Although these techniques allow trace-level quantitation, they require chemical pretreatment such as dilution and deproteinization, and the samples are limited to liquids. In contrast, EDX-7200 enables simple, non-destructive analysis in a short time and has sufficient sensitivity for screening purpose.

This application describes quantitative analysis results for 15 elements in blood using calibration curves, and qualitative and quantitative analysis results using the FP method based on sensitivity coefficients built into the instrument. The accuracy of the quantitative values obtained by the FP method was 84–103%, demonstrating sufficient quantitative performance. Analysis can be completed within 10 minutes per sample.

Fig. 1 EDX-7200

■ Preparation of Calibration Standards

Fifteen elements were selected, including elements frequently involved in poisoning cases. ICP standard solutions (1000 µg/mL) were diluted with ultrapure water to prepare standards with element concentrations of 0, 1, 5, 10, 25, and 50 µg/mL. Then, 2.5 mL of each solution was placed in a sample cell with a 5 µm-thick polypropylene film attached to the bottom. Fig. 2 shows an image of the sample.

Although it is best to match the matrix of the calibration standards to that of the test sample, this application news used aqueous solutions as calibration standards for simplicity.

Fig. 2 Sample Image

■ Linearity of the Calibration Curves

Figs. 3 to 5 show the calibration curves. The integration time for the calibration standards was set to 120 s. In addition, overlap correction (lj) for coexisting elements was applied for the quantitative elements listed in Table 1. For Ni and Sn, the 1 µg/mL calibration samples were excluded because sufficient sensitivity was not obtained with the integration time used in this study.

Table 1 Elements applied with the overlap correction

Quantitative Elements	Overlapping Elements
Cr	V
Cu	Ni
As	Pb
Pb	Se

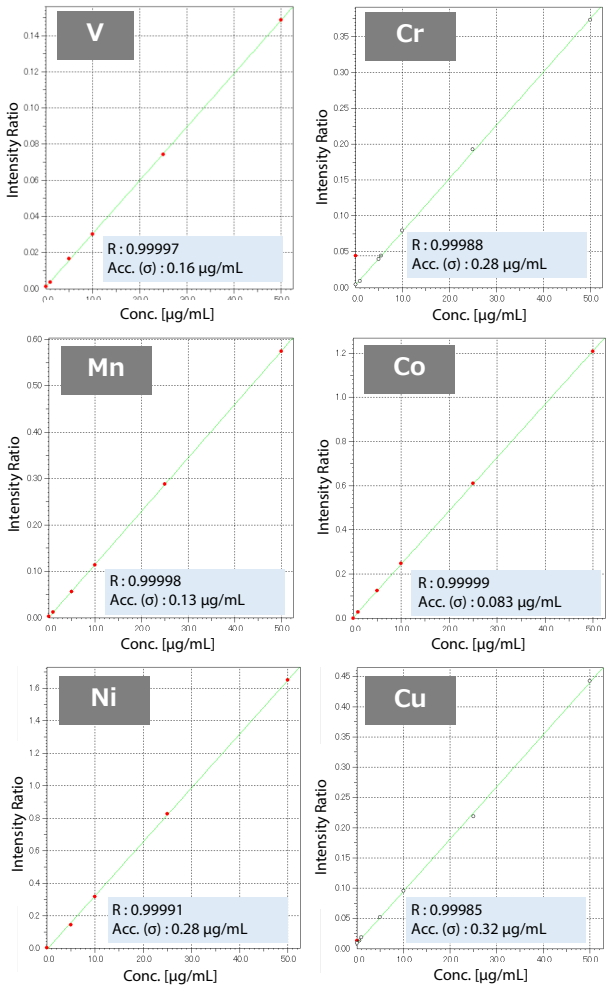

Fig. 3 Calibration Curves Part 1

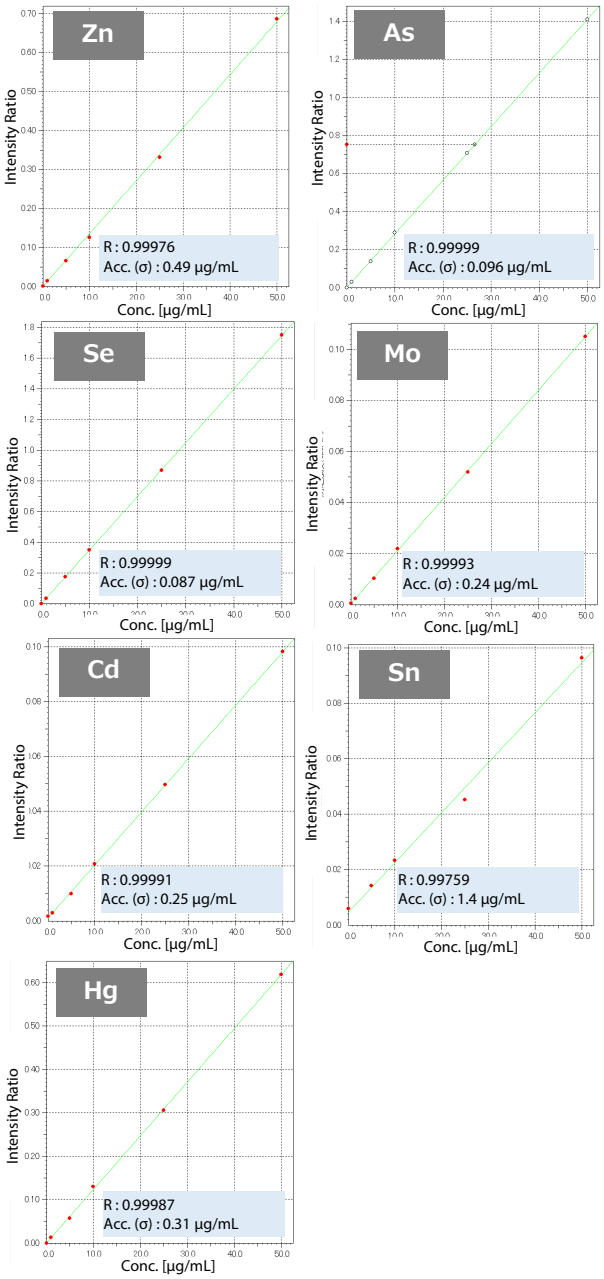

Fig. 4 Calibration Curves Part 2

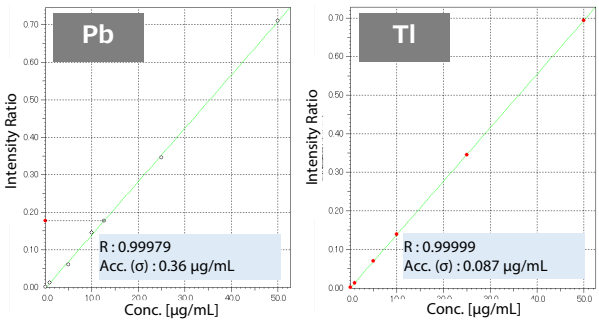

Fig. 5 Calibration Curves Part 3

■ Sample pretreatment of Blood

To evaluate quantitative accuracy, human whole blood purchased from BioIVT was used, and standards were added so that the concentration of each element in blood was 10 µg/mL.

0.25 mL of ICP standard solution (100 µg/mL) was added to 2.25 mL of blood. The mixture was thoroughly mixed using a vortex mixer, and then the entire 2.5 mL was poured into a sample cell with a 5 µm-thick polypropylene film attached to the bottom. Fig. 6 shows an image of the sample. For actual specimens, add 2.5 mL of blood sample to the sample cell.

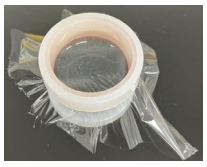

Fig. 6 Sample Image

■ Analysis Results

Fig. 7 shows the qualitative analysis results for the blood blank before standard addition. It was confirmed that the blood blank contained elements such as P, S, Cl, K, Ca, Fe, Cu, Zn, Br, and Rb.

Fig. 8 shows overlaid profiles of the blood sample with each element added to 10 µg/mL and the blood blank. Table 2 shows the quantitative analysis results by the calibration curve method and the qualitative-quantitative analysis results by the FP method. Accuracy was 84 to 118% for the calibration curve method and 84 to 103% for the FP method, indicating that sufficient quantitative accuracy was obtained even with the FP method.

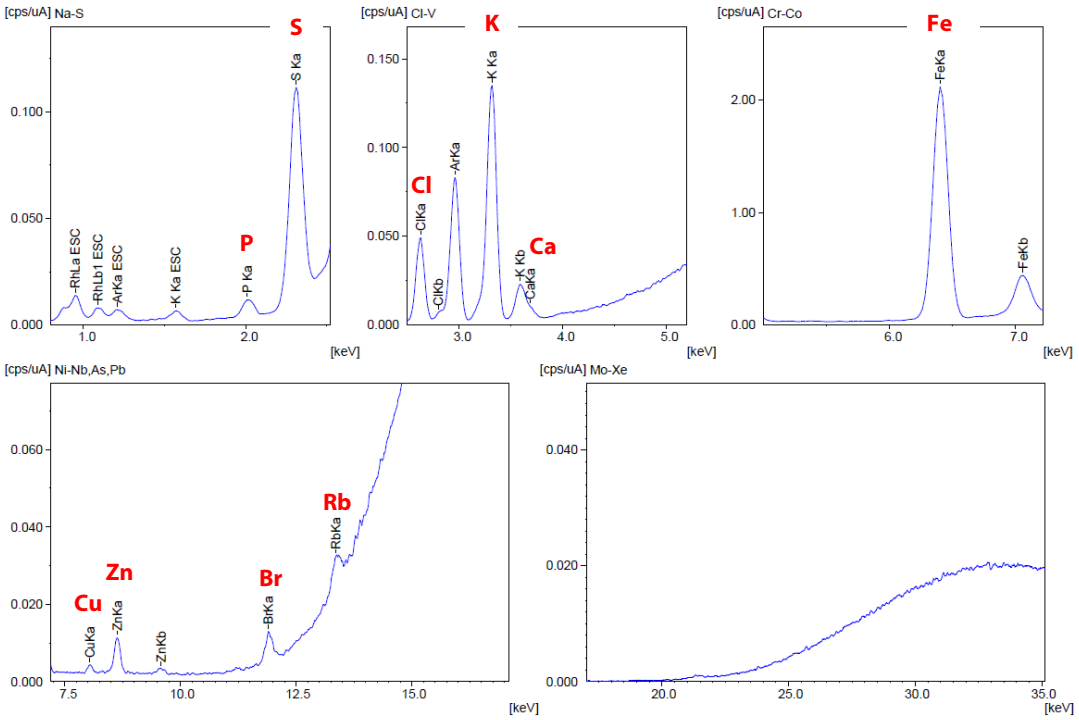

Fig. 7 Qualitative Analysis Results of the Blood Blank

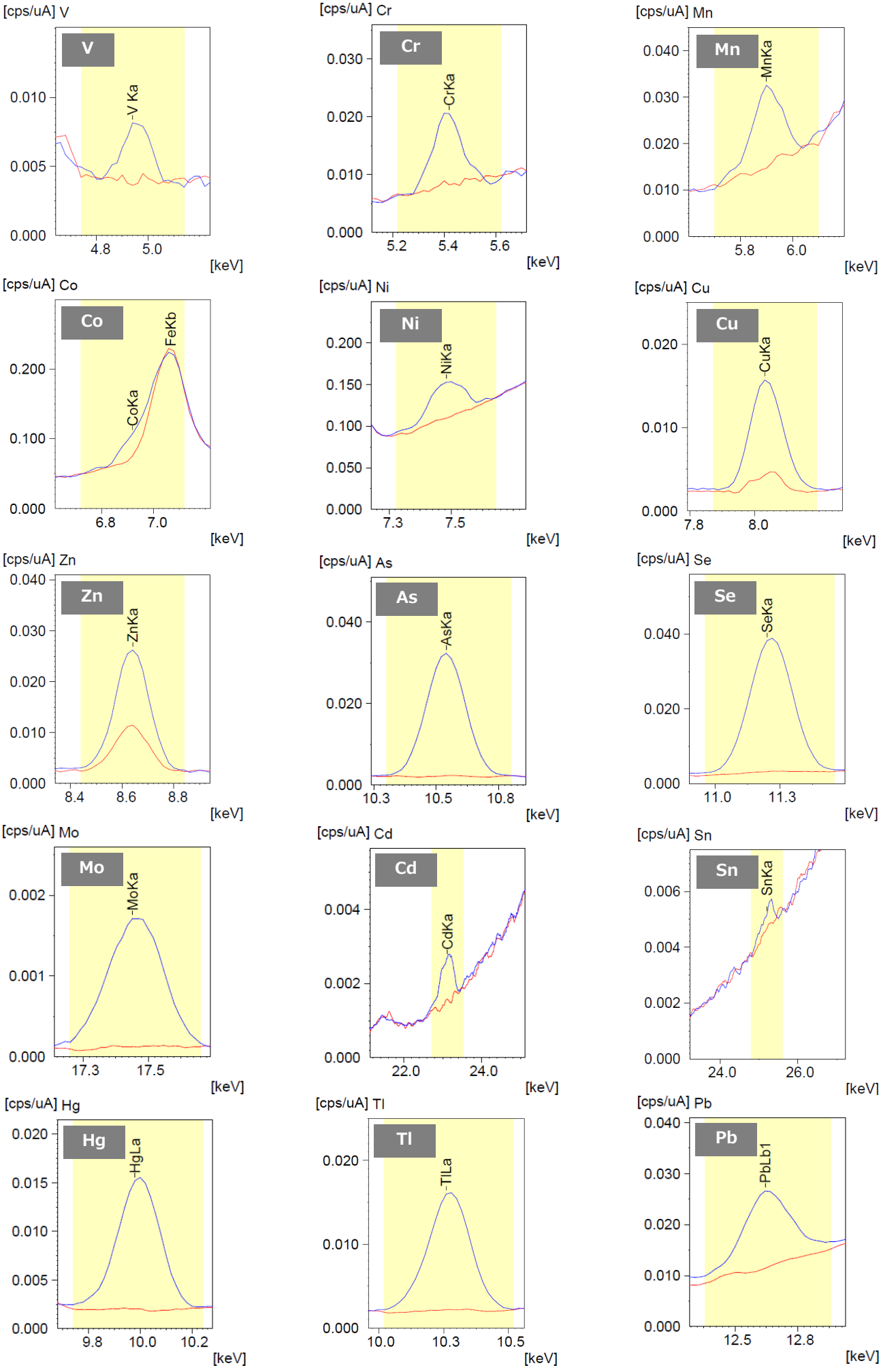

Fig. 8 Overlaid Profiles (Calibration Curve Method, Blue: Each Element 10 µg/mL, Red: Blood Blank)

Table 2 Quantitative Analysis Results [μg/mL]

Quant.	$_{23}$V			$_{24}$Cr			$_{25}$Mn			$_{27}$Co			$_{28}$Ni		
	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy
Calib.	8.8	2.1	88%	10.9	1.5	109%	9.1	1.1	91%	9.5	1.3	95%	9.6	1.0	96%
FP*1	8.7	1.8	87%	9.7	1.8	97%	8.5	1.3	85%	10.0	1.3	100%	9.0	0.8	90%
Quant.	$_{29}$Cu*2			$_{30}$Zn*3			$_{33}$As			$_{34}$Se			$_{42}$Mo		
	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy
Calib.	10.9	0.7	93%	14.3	0.6	84%	9.9	0.6	99%	10.2	0.3	102%	10.0	0.9	100%
FP*1	11.3	0.5	97%	15.3	0.5	94%	9.4	0.3	94%	10.3	0.3	103%	9.0	0.7	90%
Quant.	$_{48}$Cd			$_{50}$Sn			$_{80}$Hg			$_{81}$Tl			$_{82}$Pb		
	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy	Value	3σ	Accuracy
Calib.	11.8	1.7	118%	9.5	2.9	95%	10.3	0.5	103%	9.9	0.5	99%	10.0	0.8	100%
FP*1	9.8	1.7	98%	9.0	2.8	90%	9.6	0.4	96%	9.7	0.4	97%	8.4	0.6	84%

*1 In the FP method, moisture and organic components that cannot be measured were treated as CH_2O balance (remainder) and quantified.
*2 Considering that the blood sample before standard addition contained 1.6 μg/mL of Cu, the accuracy was calculated using the following equation.

$$\text{Accuracy [\%]} = \frac{\text{Value [ppm]} - 1.6}{10.0} \times 100$$

*3 Considering that the blood sample before standard addition contained 5.9 μg/mL of Zn, the accuracy was calculated using the following equation.

$$\text{Accuracy [\%]} = \frac{\text{Value [ppm]} - 5.9}{10.0} \times 100$$

■ Conclusion

The FP method demonstrated high accuracy for quantitation without calibration curves. In addition, blood samples do not require complicated pretreatment such as deproteinization, and the analysis time is only about 10 minutes.

■ Analysis Conditions

The analysis conditions for the calibration curve method are shown in Table 3, and those for the qual-quantitative analysis using the FP method are shown in Table 4.

Table 3 Analysis Conditions (Calibration Curve Method)

Element	V, Cr, Mn, Co, Ni, Cu, Zn, As, Se, Mo, Cd, Sn, Hg, Tl, Pb
Analytical Line	VKα, CrKα, MnKα, CoKα, NiKα, CuKα, ZnKα, AsKα, SeKα, MoKα, CdKα, SnKα,HgLα, TlLα, PbLβ$_1$
Analysis Conditions	Calibration Curve Method (Scattered X-ray Internal Standard Method)
Detector	SDD Detector
X-ray Tube	Rh Target
Tube Voltage	30 kV (V, Cr, Mn, Co, Ni), 50 kV (Cu, Zn, As, Se, Mo, Cd, Sn, Hg, Tl, Pb)
Tube Current	Auto [μA]
Collimator	10 mmφ
Primary Filter	#5 (V), #3 (Cr, Mn, Co, Ni), #4 (Cu, Zn, As, Se, Hg, Tl, Pb), #1 (Mo, Cd, Sn)
Atmosphere	Air
Integration Time	60 s × 4 ch (120 s × 4 ch for Calibration Curve Samples Only)
Dead Time	Max. 30 %

Table 4 Analysis Conditions (Qual-Quantitative Analysis)

Element	Na-U
Analytical Line	Kα, Kβ, Lα, Lβ・・・
Analysis Conditions	Qual-Quantitative Analysis (Thin-Film FP Method *1)
Detector	SDD Detector
X-ray Tube	Rh Target
Tube Voltage	15 kV (Na-S), (Cl-V), 50 kV (Cr-Co), (Ni-Nb, As, Pb), (Mo-Xe)
Tube Current	Auto [μA]
Collimator	10 mmφ
Primary Filter	None (Na-S), #2 (Cl-V), #4 (Ni-Nb, As, Pb), #1 (Mo-Xe)
Atmosphere	Air
Integration Time	60 s × 5 ch
Dead Time	Max. 30 %

*1 For a light-element matrix such as blood, the thin-film FP method was applied and the sample thickness was set, taking into account the greater analysis depth for heavy elements.

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