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Determination of Pesticide Residues in Soils of Cotton Farming Areas in Togo

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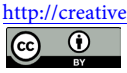

Abstract

The main cash crop in Togo is cotton, with about 70% of agricultural exports. This crop is very dependent on the use of chemical inputs, in particular pesticides. Pesticides used in cotton farming in Togo include organochlorines, triazines, organophosphates and pyrethroids. We conducted a study on the impact of these pesticide use, in particular organochlorines and triazines, on cotton farming soils. We chose the Plateaux region (southern Togo) as the study area because of its high cotton production. Analysis was carried out on crop soil samples at the 0 - 20 cm horizon. Several pesticide residues were found: simazine (0.051 to 0.103 µg/Kg soil); atrazine (0.149 to 0.197 µg/Kg soil); lindane (0.259 to 0.672 µg/Kg soil); β-endosulfan (1.37 to 5.727 µg/Kg soil); dieldrin (0.063 to 1.16 µg/Kg soil); endrin (0.512 µg/Kg soil), Heptachlor (0.489 to 1.243 µg/Kg soil); Heptachlor epoxide (0.928 to 1.633 µg/Kg soil); [2,4'DDT] (0.257 µg/Kg soil); [4,4'DDE] (0.262 µg/Kg soil). These results show pesticide contamination of cotton farming soils.

Keywords

Pesticides, Soils, Residues, Organochlorines, Triazines

1. Introduction

Pesticides are substances; usually chemicals used to preserve crops. Their use has become a significant environmental issue as there is considerable evidence to suggest that they can have harmful effects on soils and natural resources such as natural ground and surface water; the quality of agricultural products; the health of populations related to their uses and the consumption of contaminated food [1]-[6].

The economy of African countries is largely based on agriculture, particularly cash crops. The pesticides use level in Africa is still low compared to other regions in the world, but it is increasing rapidly, especially for cash crops. Several studies have shown pesticide residues in soils, surface waters and in aquatic organisms [7] [8] [9] [10].

In Togo, the economy is largely based on agriculture, where cash crops are increasingly important. Cotton is the main cash crop, with about 70% of agricultural exports [11]. This crop is characterized by the intensive use of chemical inputs, especially pesticides, to limit crop losses due to parasitism, estimated at an average of 60% [12].

The Knowledge of pesticides and their transformation products' fate, some of which may be more toxic than the parent molecules, is an important concern in the context of environmental protection and sustainable development [13] [14].

The determination of pesticides in soils, as in other environmental resources is largely carried out using chromatographic techniques associated with specific detectors [15]. Any pesticide residue analysis technique mainly includes collection or sampling operations, transportation and storage; extraction of the active substances from the matrix by appropriate organic solvents; chemical analysis [16]. Several methods for the extraction of pesticides from soils are described in the literature. These are the soxhlet method; mechanical agitation, ultrasonic extraction, microwave-assisted extraction, supercritical fluid extraction, and Accelerated Solvent Extraction method. The principle of this latter method is based on increasing the quantities extracted by using solvents brought to high temperature (100˚C) and high pressure (100 bar). The temperature modifies the properties of the solvent leading to an increase in its power of solvation and its power of diffusion in solid matrices. The high pressure keeps the solvent in liquid form at high temperatures. The results obtained are comparable to those obtained with the Soxhlet method but with a reduction in the extraction time and the volumes of solvent used [16]. Purification aims to eliminate substances co-extracted with pesticides and which may interfere during instrumental analysis. The process is generally carried out by adsorption chromatography on alumina, silica or florisil or by gel permeation chromatography. Impurities are removed by elution from the column with mixtures of solvents of increasing eluting strength [17].

Several methods can be used to analyze pesticides; they are based on different phenomena that take molecular properties into account [16]. We distinguish spectrometric methods (UV-visible, Infrared (IR), Nuclear Magnetic Resonance (NMR)); methods based on chemical properties (immunoenzymatic methods, mass spectrometry); methods based on the emission of β radiation; methods based on toxicological properties; chromatographic methods, which are the most widely used methods for the analysis of pesticide residues. The chromatographic analysis can be carried out in the liquid phase or in the gaseous phase coupled or not to mass spectroscopy.

Studies on pesticide behavior in different environmental compartments and their fate are insufficient in Togo. In this context, we undertook a study of the impact of pesticide use on soils in cotton farming in Togo.

2. Materials and Methods

2.1. Sampling Area

The present study was conducted in the "Plateaux" region, which is one of the main cotton farming regions in Togo. It covered cotton fields chosen in collaboration with the agents of the "Nouvelle Société Cotonnière du Togo" (NSCT), an institution in charge of cotton production and marketing, mainly according to the production importance. The chosen sites are: Glei; Wahala; Amakpapé (**Figure 1**).

2.2. Sampling Area

Samples were taken with an Endelman auger using a method inspired by Mathieu and Pieltain [18]. The upper layer of soil sampled corresponds to the first 20 centimeters.

Areas of 10 m × 10 m were delimited in the middle of the field. 9 incremental samples were taken within each square (**Figure 2**). The composite samples, resulting from the mixing of the collected primary samples, were carried to the laboratory, pre-treated (homogenization-quartering-air drying-sorting and clod reduction-sieving to 2 mm) and stored at −20˚C for pesticide residue analysis.

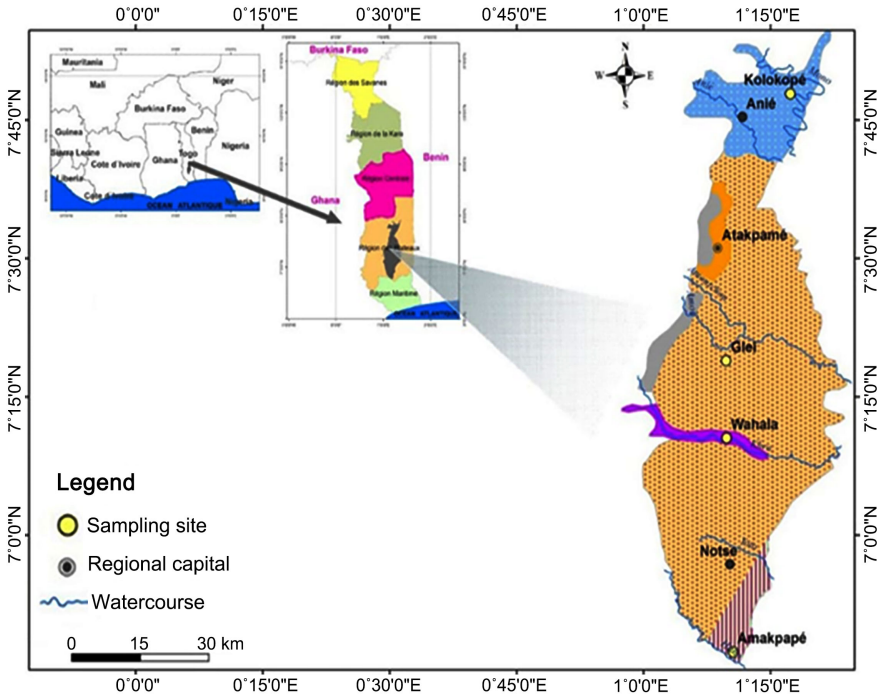

Figure 1. Sampling area.

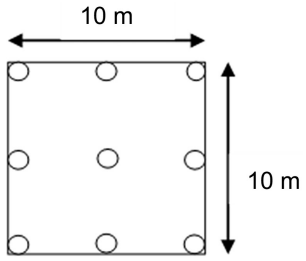

Figure 2. Method of soil sampling.

2.3. Quantification of Pesticide Residues

2.3.1. Extraction and Purification

The Accelerated Solvent Extraction (Dionex, Salt Lake City, Utah) method [19] was used to extract the pesticides. The cells used for extraction are filled with a mixture of the test sample and diatomaceous earth (Varian, Palo Alto, California). The solvent mixture is composed of hexane and acetone. These two solvents are supplied by SDS (Solvent Document Synthèse)/Carlo Erba, F13124 Peypin France. Florisil cartridges, used for extract purification, were supplied by Wasters Corporation (Milford, Massachusetts) and fitted to SGE syringes with standard Luer tips (Scientific Glass Engineering Pty. Ltd, Melbourne, Australia).

2.3.2. Pesticides Determination

The analysis was performed by Gas Chromatography (GC) coupled to a mass spectrometer (GC-Mass). The device used includes a Thermo Fisher Trace GC Ultra chromatograph coupled with a Trace SDQ mass spectrometer. The analysis parameters are summarised in Table 1.

3. Results and Discussion

The pesticides found in the analyzed soils in our study belong to the triazine (therbuthylazine, simazine, atrazine) and organochlorine families (DDT and its metabolites, dieldrin, endrin, lindane, endosulfan, heptachlor and heptachlor epoxide).

Figures 3-5 show the chromatograms obtained during the soil analysis. Different peaks can be seen with retention times that confirm the presence of several compounds.

The different peaks in the chromatogram are identified by mass spectrometry, which allows us to determine the masses of the ions and to assign them to molecules by means of a database search. Several peaks were not identified and are not considered in this study.

The identified molecules concentrations were determined and are presented in the following table (Table 2).

The values of the triazines (therbuthylazine, simazine and atrazine); DDT and its metabolites (DDE and DDD) were compared with the target values of the Netherlands [20].

The values of the other organochlorines (dieldrin, endrin, lindane, endosulfan, heptachlor and heptachlor epoxide) were compared to the soil contamination threshold of the Netherlands [21].

The soil at the Gléi site contains atrazine residues at a value greater than the limit value, which is 0.0002 μg·kg^{-1} (**Table 2**).

Table 1. Parameters for the pesticides determination.

Chromatographic conditions Table	
Injection mode	Splitless
Initial oven temperature	280˚C
Column	Phenomenex Zebron 5 MS
Column length	30 m
Internal diameter	0.25 mm
Film thickness	0.25 μm
Eluting gas	Helium
Elution flow rate	1 ml/min
Detection conditions	
Source temperature	250˚C
Transfer line	300˚C
Electron multiplier	1660 V
Detection mode	SIM

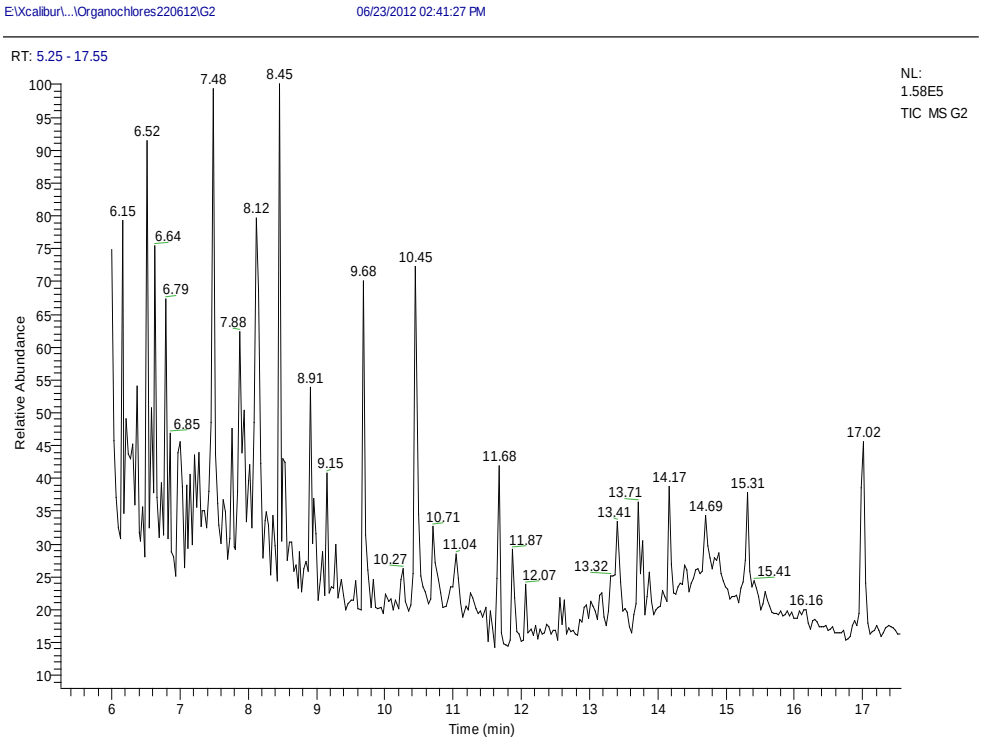

Figure 3. Glei soil Chromatogram.

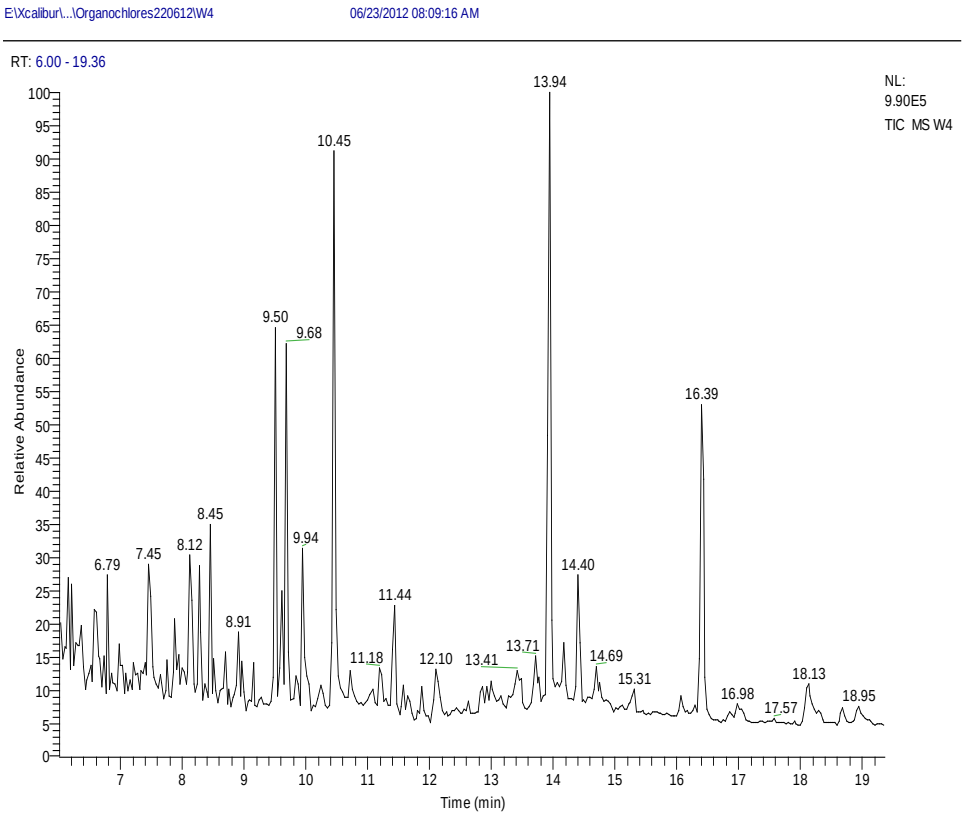

Figure 4. Wahala soil chromatogram.

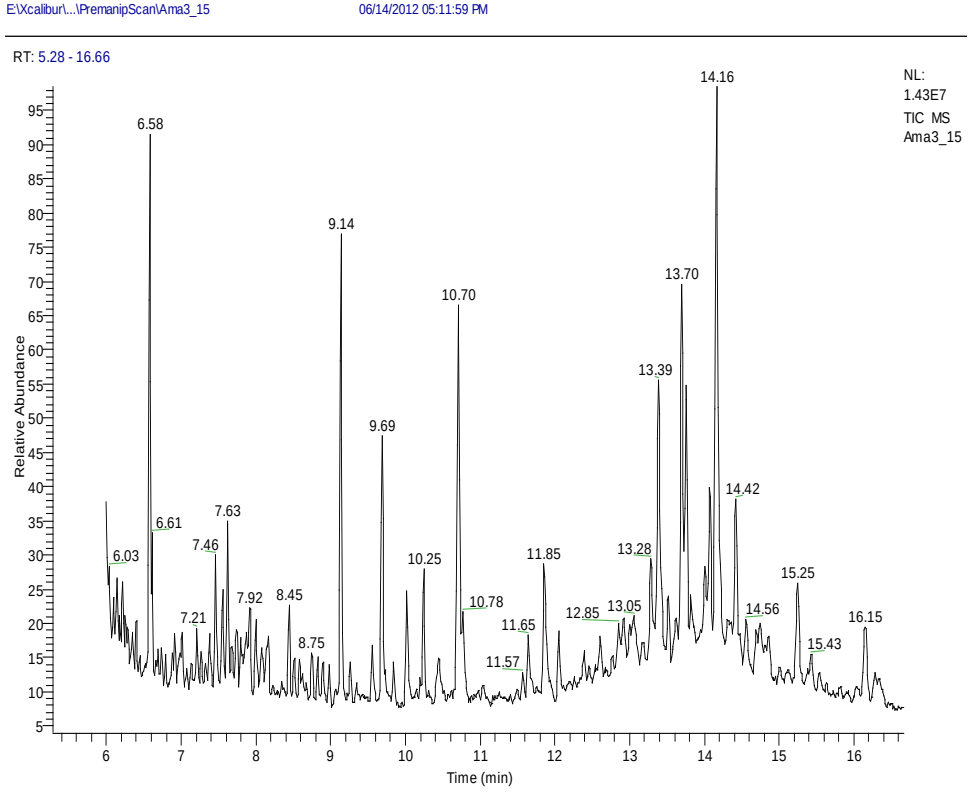

Figure 5. Amakpapé soil chromatogram.

Table 2. Average values of pesticide residues studied in soils from different sites.

Pesticides (µg/Kg soil)		Gléi	Wahala	Amakpapé	Limit values
Herbicides	Atrazine	0.149	ND	0.155	0.0002a
	Simazine	ND	0.051	0.103	-
	Therbuthylazine	ND	ND	ND	-
Insecticides	2,4-DDT	ND	0.257	ND	0.100a
	4,4-DDT	ND	ND	ND	0.100a
	2,4-DDE	ND	ND	ND	0.100a
	4,4-DDE	0.262	ND	ND	0.100a
	Dieldrin	0.669	1.160	0.660	0.500b
	Endrin	ND	0.512	ND	0.040b
	Aldrin	ND	ND	ND	0.060b
	Lindane [γ-HCH]	0.259	0.628	0.672	0.050b
	α-Endosulfan	ND	ND	ND	0.100b
	β-Endosulfan	1.259	5.727	ND	0.100b
	Heptachlor	0.489	0.733	1.243	0.700b
	Heptachlor epoxide	0.991	0.928	1.633	0.0002b

a. Target values in the Netherlands (µg·kg^{-1}) [20]. b. Soil contamination limit in the Netherlands (µg·kg^{-1}) [21].

DDT was not detected in Gléi soil, but its metabolite, 4,4-DDE is present at a level of 0.262 µg·kg^{-1}. This value is greater than the limit value (0.1 µg·kg^{-1}) [20].

Gléi soil does not contain endrin and aldrin. However, dieldrin, a metabolite of aldrin was detected with a content of 0.669 µg·kg^{-1}. This concentration is greater than 0.5 µg·kg^{-1} set as a reference value according to the soil protection directive in the Netherlands [21] (**Table 2**). Our analysis also revealed the presence of traces of lindane in the soil of Gléi at a level well above the contamination limit. The Gléi soil samples contain β-endosulfan with a content above the contamination limit which is 0.1 µg·kg^{-1} (**Table 2**).

We detected heptachlor with a value lower than the limit value (0.7 µg·kg^{-1}) and its stable metabolite which is heptachlor epoxide, obtained by biodegradation, whose content is higher than the limit value which is 0.0002 µg·kg^{-1}.

Wahala soil shows simazine residues. We detected 2.4 DDT in the soil of Wahala at a level of 0.257 µg·kg^{-1}. This concentration is greater than the reference limit (0.1 µg·kg^{-1}) [20] (**Table 2**). However, the DDE was not detected.

Wahala soil also contains endrin and dieldrin with respective concentrations of 1.16 µg·kg^{-1} and 0.512 µg·kg^{-1}. These levels are higher than those set as reference values according to the soil protection directive in the Netherlands [21].

Lindane was detected in the Wahala soil at a level of 0.628 µg·kg^{-1}, higher than the reference value (0.05 µg·kg^{-1}) set by the soil protection directive in the Netherlands [21].

The content of β-endosulfan in the soil of Wahala, 5.727 μg·kg^{-1}, is higher than the limit value (0.1 μg·kg^{-1}) [21] (**Table 2**). This high content may be due to the recent use of this pesticide on the site.

Heptachlor and heptachlor epoxide was detected at respective levels of 0.733 and 0.928 μg·kg^{-1}. The concentrations of these pesticides in the Wahala soil are above the limit values [21].

Amakpapé soil shows residues of atrazine and simazine at respective values of 0.155 μg·kg^{-1} and 0.103 μg·kg^{-1}. These levels are higher than those set as limit values (**Table 2**). DDT and its metabolites, α-endosulfan and β-endosulfan were not detected.

If we did not detect endrin and aldrin in Amakpapé soil, dieldrin is indeed with a value of 0.663 μg·kg^{-1}. This concentration is higher than the reference (0.5 μg·kg^{-1}) of the soil protection directive in the Netherlands [21] (**Table 2**).

Lindane was detected with a content of 0.672 μg·kg^{-1}. This value is greater than 0.05 μg·kg^{-1} set as a reference value according to the soil protection directive in the Netherlands.

Heptachlor and heptachlor epoxide was detected in the soil of Amakpapé at respective levels of 1.243 and 1.633 μg·kg^{-1}. As in the other soils, the concentration of heptachlor is lower than the limit concentration, whereas that of the by-product resulting from its degradation, which is heptachlor epoxide is higher than the limit value (**Table 2**).

According to our study, cotton farming soils have pesticide residue levels. Atrazine is one of the most widely used herbicides in Togo, which may explain its presence in 2 soils. The presence of endrin and dieldrin can be explained by their persistence: biodegradation half-lives in the soil of approximately 4 to 14 years or more have been reported for endrin [22], dieldrin is detectable in the soil after 30 years of application [23]. Lindane is considered to have very low mobility in soils. Due to its lipophilic nature, it is strongly adsorbed by soils rich in organic matter. This may explain its presence in all soils.

The endosulfan, which is still used in cotton growing, is a mixture of two stereoisomers alpha-endosulfan and beta-endosulfan in the proportions α/β = 70/30. According to several studies, β-endosulfan is more persistent than the α isomer in soil. Despite its rapid degradation in water, it can persist for a relatively long period when bound to soil particles [24]. This explains the fact that we detected β-endosulfan during our investigations.

These results are comparable to those obtained in cotton-farming soils in the West African sub-region

According to studies in cotton-farming soils in Burkina-Faso, endosulfan was detected at concentrations of 0.1 to 16.54 μg·kg^{-1} [25].

In Benin, studies have shown endosulfan content of 13.8 μg·kg^{-1} [26]; other studies in the same country revealed the presence of 4,4-DDE (2.0 to 12 μg·kg^{-1}) and 4,4-DDD (0.54 to 1.9 μg·kg^{-1}) [27].

Nevertheless, the levels found during our study are generally lower than the

limit values and those found in 2008 in Togo by Mawussi [28].

The soil of Wahala is the one that contains the largest number (08) of pesticide residues of the three cotton growing soils that were the subject of our study. These results can be explained by the physicochemical properties of this soil which can promote the retention of pesticides. This retention does not allow contamination of groundwater by pesticides. On the other hand, the surface waters of this zone will be vulnerable to contamination.

The soils of Gléï and Amakpapé contain fewer traces of compounds sought, respectively seven (07) for Gléï and six (06) for Amakpapé. This can also be explained by the physicochemical characteristics of these two soils allowing a low retention capacity for organic compounds. Such properties would favor the contamination of groundwater by infiltration of these chemical compounds.

4. Conclusion

Soil analysis of cotton farming areas in the Plateaux region of Togo was carried out by GC/MS. Several pesticide residues were found in the soils studied: simazine, atrazine, DDT and its metabolites, dieldrin, endrin, lindane, endosulfan, heptachlor and heptachlor epoxide. The levels detected are mostly below the reference limits for soil contamination in Canada, the Netherlands and the Environmental Quality Standard Directive 2013/39/EU, except for dieldrin, endrin, lindane, endosulfan, heptachlor and heptachlor epoxide. The results of the present study are lower than those obtained in other countries of the West African sub-region. Nevertheless, risks of groundwater and surface water pollution exist.

Acknowledgements

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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