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FATE OF HEPTACHLOR AND ITS PRIMARY METABOLITE IN NANUMBA NORTH MUNICIPALITY, GHANA

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Abstract

The aim of this study was to assess the levels of contamination of heptachlor and heptachlor-epoxide in the Nanumba-North Municipality of the Northern Region of Ghana. Gas Chromatography equipped with electron capture detector (GC-ECD) was used to analyse the samples. In all, 148 samples were analysed, consisting ten (10) soil samples, 108 water samples and 30 food crop items. The study revealed the presence of heptachlor and heptachlor-epoxide at varying mean concentrations, with heptachlor-epoxide recording significant concentrations in the samples. The parent compound occupied 40% with a mean value of 0.008 ± 0.005 µg/L whilst heptachlor-epoxide occupied 60% with a mean value of 0.012 ± 0.008 µg/L. The trends of heptachlor and heptachlor-epoxide residues distribution in water samples from the various depths was of the order 30 cm and below > 15-30 cm >0-15 cm. The presence of heptachlor and heptachlor-epoxide in the water samples could be traced to wind drift or atmospheric transport of volatilized pesticides, direct overspray, direct spillage, pesticide misuse by farmers, run-off and leaching from application fields and surrounding areas during and after their applications. The concentrations of heptachlor and heptachlor-epoxide residues in the samples analysed were generally low and below the WHO (2017) MRLs. Therefore, the samples (soils, water and food crops) analysed in the study area at the moment will not pose any significant threat. Besides, heptachlor and heptachlor-epoxide detected showed significant differences at $p < 0.05$. However, just because there are low levels does not mean that consumers will not be exposed to its health risks. Since continuous availability can lead to bioaccumulation and biomagnification through the food chain. The findings therefore, emphasize the need for profound continuous environmental monitoring, enforcement of pesticide regulations, and the promotion of integrated pest management strategies to reduce reliance on persistent organochlorine pesticides and protect soil quality and public health in the Nanumba North Municipality.

Keywords: *Heptachlor; heptachlor-epoxide; Nanumba-North Municipality; GC-ECD*

1.0 Introduction

Heptachlor is a chlorinated dicyclopentadiene insecticide, which is categorized as a Persistent Organic Pollutant due to its pervasive environmental presence and resistance to degradation. Its widespread use and application in agriculture, particularly in areas such as the Nanumba North Municipality of the Northern region of Ghana, contributes to its accumulation across various environmental matrices, posing significant ecological and health risks [1]. Despite bans in many developed nations, organochlorine pesticides such as heptachlor are still used in agriculture in developing countries due to their effectiveness in pest control and ready availability, leading to continuous environmental release [2]. These compounds, characterized by their lipophilicity and resistance to biodegradation, undergo complex transfer mechanisms between soil, water, and food crops item, influencing their distribution and long-term persistence in affected ecosystems. The transformation of heptachlor into more stable and often more toxic metabolites, such as heptachlor epoxide, further exacerbates these environmental concerns by increasing its persistence and bioaccumulation potential within food chains [3]. This pervasive persistence necessitates a comprehensive understanding of their environmental fate, including degradation pathways, transport mechanisms, and metabolic transformations, especially in vulnerable agricultural regions [4:5]. Specifically, the Nanumba North Municipality in Ghana, an area heavily

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reliant on agriculture, likely faces considerable environmental contamination from such persistent organic pollutants, warranting detailed investigation into the concentrations and distribution of heptachlor and its metabolites within its various environmental compartments [6]. Previous research indicates that the improper and illegal application of pesticides in Ghanaian farming communities can lead to their absorption through various routes, including dermal, oral, and respiratory pathways, and it is enough backing to give cause for similar concerns in the Nanumba North Municipality. Such exposure is concerning given documented high levels of pesticides in food and direct exposure among agricultural workers in Ghana [7]. Consequently, understanding the fate of heptachlor and its metabolites is crucial for assessing potential health impacts on local populations and developing effective strategies for environmental remediation in these vulnerable regions. This is particularly pertinent given the widespread detection of various organochlorine pesticides, including heptachlor, in Ghanaian soil and sediment samples, which highlights ongoing environmental contamination. The persistent presence of these compounds underscores the urgent need for a thorough investigation into their environmental distribution and potential for bioaccumulation within the Nanumba North Municipality [8]. This study, therefore, aims to delineate the fate of heptachlor and its primary metabolites within the soil, water, and food crop matrices of the Nanumba North Municipality, thereby providing crucial data for environmental risk assessments and informing targeted mitigation strategies.

2.0 Materials and Methods

2.1 Study area

The study was carried out in the Nanumba North Municipality, which is located in the southeastern part of Northern Region of Ghana. The area lies between latitudes 8.5º N and 9.25º N and longitudes 0.57º E and 0.5ºE (Figure 1). According to the 2010 Population and Housing Census, the total population of Nanumba North Municipality is 141,584. Males constitute 49.4 percent and females represent 50.6 percent in the Municipality. The Municipality has a total land area of 2260.8 sq. Km. The predominant occupation is farming (www.ghanadistricts.com 2011).

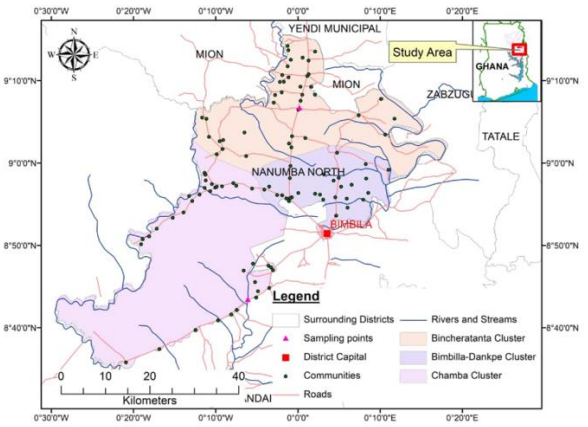

Figure 1: A map showing Nanumba North Municipality in the Northern Region of Ghana

2.2 Collection of samples

Water samples were collected from a River and two Dams. The Kumbo River was used while the Dams were Bincheratanga, and Waanpu. Grab sampling technique was used to collect thirty-six (36) water samples from four zones at three different depths. The depths used were (that is, 0-15cm, 15-30cm, and 30cm and below) using 500 mL pre-cleaned Teflon sample bottles with caps for analysis making a total of one hundred and eight (108) water samples. The Teflon sampling bottles were rinsed well with water to be sampled several times before they were carefully filled to over-flowing, to avoid trapping air bubbles in sealed bottles. Additionally, the Teflon sampling bottles were rinsed with the river and two dams' water before taking the water samples. The samples were labelled and transported to the laboratory within 24–48 hours on ice in clean ice chests and stored in the refrigerator at 4 °C until they were analysed for heptachlor and heptachlor-epoxide. Also, all the selected farms were grouped into three Clusters that is, Bimbilla-Dankpe Cluster (BDS), Bincheratanga Cluster (BS) and Chamba Cluster (CS). Three quadrants of 70 × 70 m were marked out in each Cluster. In each quadrant, three (3) soil samples were collected randomly at depths 0–20 cm with a soil auger. The rectitude of taking this depth was because nutrient uptake by plants is usually reported to be within this horizon [9]. Additionally, one soil sample was taken from a nearby natural forest to act as a control (X1). This gave a total of ten soil samples for the study area. All soil samples were kept in well-labelled plastic polythene containers and transported to the laboratory for analysis. The soil samples were oven-dried at 105 °C to constant weight and sieved using 2 mm nylon mesh. Sampling of yams, maize, and "ayoyo" were done at the three different Clusters in the study area in August 2025, and labelled as the Bimbilla-Dankpe Cluster, Bincheratanta Cluster, and Chamba Cluster. In each Cluster three (3) samples of the selected crops were taken randomly. Additionally, one sample of each crop was taken from farmlands where pesticide application was observed not to be common to act as a control. These gave a total of twenty (30) crop samples for the study area. The samples were packed in black polyethylene bags, labelled accordingly and transported to the laboratory. In the laboratory, the samples were ground into powder. They were then packed in freezer bags and stored in a refrigerator at 4 ^{0}C for analysis.

2.3 Extraction of samples

After filtration of water samples through 0.45 mL fiberglass filters (WHATMAN) to remove debris and suspended material, 1000 mL portions of the filtered water samples were transferred into 2 L capacity separating flasks. A 30 mL of saturated sodium chloride solution (NaCl) was added to each to produce a salt-out effect to adjust the pH to 7. The samples were then thoroughly mixed by inverting the flask three to four times. A 100 mL of dichloromethane as extraction solvent was then added to each sample and vigorously shaken manually for 2–3 min while releasing the pressure

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intermittently. The phases were allowed to separate for 5 min and the dichloromethane extracts (organic layers) were separated from the aqueous layers. The extraction for each water sample was repeated twice with 100 mL of dichloromethane and the organic layers were put together and dried over anhydrous sodium sulfate through filter papers into 50 mL round bottom flasks. The extracts from the water samples were then concentrated on rotary vacuum evaporators (Buchi Ratovapor R-210, USA) to about 1 mL and subjected to silica clean-up. Ten grams (10 g) of the representative soil samples were weighed and quantitatively transferred into 250 mL separating flasks. 10 mL of acetonitrile was added to each of the soil samples in the flasks and ultra-sonicated (Becon FS400b) for 5 min. An additional 10 mL of acetonitrile was added, and the flasks closed tightly. The samples were placed on a horizontal mechanical shaker (Ika-Werke HS 501 Digital) and set to shake continuously for 30 min at 300 mot/ min. The contents were then allowed to stand for 10 min to sufficiently separate the phases or layers. 10 mL of the supernatants were carefully taken by pipette and dried over 2 g anhydrous magnesium sulfate through filter paper into 50 mL round bottom flasks. The concentrates were then adjusted to about 2 mL using the rotary film evaporator (Buchi Ratovapor R-210, USA) at 35 °C, and made ready for the silica clean-up step. All reagents and chemicals were of analytical grade and were used as received. Extraction of heptachlor and heptachlor-epoxide in yam, maize, and "ayoyo" samples were done. Each sample of 5.0 g was placed into a flask and 30 mL of acetone: methanol (1:1 v/v) extraction solvent was added. The content of the flask was shaken continuously on a mechanical flash shaker at 200 rpm for 3 h. The extract was filtered through a Buchner funnel fitted with Whatman filter paper under suction. The filtrate was transferred into a 500 mL separating funnel and 150 mL sodium sulfate solution was added. The mixture was partitioned with 30 mL of dichloromethane and vigorously shaken for 2 min releasing pressure intermittently. The phases were allowed to separate and the lower dichloromethane phase was collected into a flask. The aqueous layer was partitioned twice using 10 mL portions of dichloromethane each time. The dichloromethane extracts were combined and dried on 20 g of anhydrous sodium sulfate in a mini-glass column. The dried extract was concentrated to approximately 2 mL in a rotary evaporator at 37 ^{0}C and stored in a 2 mL sample vial. This was then taken for clean-up.

2.4 Clean-up of samples

Extracts clean-up was done, using polypropylene cartridge columns, packed with one-gram silica gel previously activated for 10 h in an oven at 130 °C, which has a 2 g layer of anhydrous sodium sulfate on top and conditioned with 6 mL dichloromethane. The concentrated extracts were then loaded onto the cartridges, and 100 mL round bottom flasks were placed under the columns to collect the eluates. A 20 mL dichloromethane was then used to elute the columns/cartridges afterward, and the total filtrates (eluents) collected were concentrated just to dryness using the rotary evaporator (Buchi Ratovapor R-210) set at 40 °C. The residues were re-dissolved in 1 mL ethyl acetate by pipetting and transferred into 2 mL standard opening vials before quantitation by gas chromatography (GC) (Varian Association Inc. USA) equipped with electron capture (ECD). Extracts clean up were done, using polypropylene cartridge columns, packed with one-gram silica gel previously activated for 10 h in an oven at 130 °C, which has a 1 cm thick layer of anhydrous magnesium sulfate on top and conditioned with 6 mL acetonitrile. The concentrated extracts were then loaded onto the columns/ cartridges, and 50 mL pear shape flasks were placed under the columns to collect the eluates. A 10 mL acetonitrile was used to elute the columns/cartridges afterward. The total filtrates (eluents) collected were concentrated to dryness using the rotary evaporator (Buchi Ratovapor R-210) set at 40 °C. The residues were re-dissolved in 1 mL ethyl acetate by pipetting and transferred into 2 mL standard opening vials before quantitation by gas chromatography (GC) (Varian Association Inc. USA) equipped with electron capture detector (ECD). All extracts were kept frozen until quantification was achieved. For the clean-up, a 15 g mixture of alumina and activated charcoal (12:1) slurry was packed with dichloromethane in a mini glass column and topped up with a 2 cm layer of anhydrous sodium sulphate. The column was conditioned with 5 mL of dichloromethane and the sample extract was loaded on the column. The sample vial was rinsed two times with 2 mL aliquots of dichloromethane and the rinsed was added to the column. The sample was eluted with 30 mL dichloromethane and elutes concentrated to approximately 2 mL using a rotary evaporator at 37 ^{0}C. The final extracts were refrigerated at 4 ^{0}C until GC analysis. The limit of quantification for heptachlor and heptachlor-epoxide in the samples in this study was 0.001 µg/L.

2.5 Quantitation:

An external method was employed in the determination of the quantities of residues in the sample extracts. A standard mixture of known concentrations of heptachlor was run, and the response of the detector was ascertained. The area of the corresponding peak in the sample was compared with that of the standard. All analyses were carried out in triplicate and the mean concentrations computed accordingly.

2.6 Gas chromatographic determination:

The final extracts were analysed by a gas chromatography (GC)—Varian CP-3800 (Varian Association Inc. USA) equipped with electron capture detector (ECD) CombiPAL Autosampler at the CSIR Pesticide Residues Laboratory, Accra,

Ghana. The GC conditions and the detector response were adjusted so as to match the relative retention times and response as spelt out by the Japanese analytical methods for agricultural chemicals (Syoku-An 2006). The GC conditions used for the analysis were capillary column (fused silica capillary) coated with VF-1701 ms (30 m × 0.25 mm internal diameter, 0.25 µm film thick-ness). The injector and electron capture detector (ECD) temperatures were set at 270 and 280 °C respectively. The oven temperature was programmed as follows: 70 °C held for 2 min, ramp at 25 °C min−1 to 200 °C, held for 1 min, and finally ramp at 25 °C min−1 to 250

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°C. Nitrogen was used as carrier gas at a constant flow rate of 2.0 mL/min and detector make-up gases of 17.0, 14.0 and 10.0 mL/min for air 1, hydrogen and air two respectively. The injection volume of the GC was 2.0 µL in a split less mode. The total run time for a sample was 14 min.

2.7 Data analysis

Statistical package for social sciences (SPSS) was used to generate the means, standard deviation, and standard error for heptachlor and heptachlor-epoxide. One-way analysis of variance (ANOVA) was performed to analyse significant differences in the concentrations of heptachlor and heptachlor-epoxide.

3.0 Results and Discussion

From this study, the trends of and heptachlor and heptachlor-epoxide residues distribution in water samples from the various depths indicated frequent occurrence and higher residue concentrations in water samples at depths 30 cm and below than depths 15-30 cm and 0-15 cm. Heptachlor and heptachlor-epoxide residues measured in the samples analysed were generally low and below their respective World Health Organization MRLs, respectively.

Synopsis of literature revealed that, environmental factors such as low relative humidity, high temperature, wet soil and air movement tend to increase volatilization and this hugely influences the concentration of heptachlor and its metabolites in samples. Heptachlor and its metabolite in the samples analysed decreased with decrease organic carbon and organic matter content. Soil organic carbon enhances adsorption and deposition of heptachlor and its metabolite in the samples, and since the organic carbon and organic matter in the sampled soil was low this could account for the relatively low levels of heptachlor and its metabolite. Perhaps heptachlor and its metabolite levels in the samples were associated with organic matter content of the soil and could be attributed to heptachlor and its metabolite molecules having high tendency to bind to organic matter in soil, similar to fats or lipids of plants and animals. Similarly, a decrease in pH decreases the concentrations of heptachlor and its metabolite. The presence of heptachlor in the water samples could be traced to wind drift or atmospheric transport of volatilized heptachlor, direct overspray, direct spillage, heptachlor misuse by farmers, run-off and leaching from application farms and surrounding areas during and after heptachlor applications. Heptachlor-epoxide was more stable than heptachlor since the half-life of heptachlor is 3.5 years, and that of heptachlor-epoxide is 4 years. This suggested that in the study area, heptachlor breaks down into heptachlor-epoxide which was more stable. Therefore, the mean concentration of heptachlor-epoxide was higher than the parent compound in the soil samples analysed. Perhaps the possible mechanisms for low levels of heptachlor from the samples were characterized by high temperature, 777erosion, volatilisation, uptake by plants and animals, and biodegradation. In water, the predominant fate of heptachlor was hydrolysis, with a half-life of 3.5 days, therefore the estimated period for the transformation of heptachlor to heptachlor-epoxide in the water samples was 3 days (Table 4.b). Similarly, since the half-life of heptachlor is 3.5 years the estimated period for the transformation of heptachlor to heptachlor-epoxide in the agricultural soils was 1.5 years (Table 4.a). Perhaps the low levels of heptachlor and heptachlor-epoxide in the water samples as compared to the soil samples analysed, may be attributed to the low water solubility of heptachlor at 25°C and its high octanol/water partition coefficient (log K_{ow}: 3.87), which allows heptachlor to be preferentially adsorbed in soils rather than released into the water. The molar concentration of heptachlor in the water, soils and food crops was 8.04 picomoles, 24.11 picomoles and 37.50 picomoles, whilst its metabolite molar concentration in water and soils were 28.26 picomoles and 30.83 picomoles. The mean concentrations of heptachlor and heptachlor-epoxide recorded in the soil samples analysed were below the MRL of 0.100 µg/L, and 0.030 µg/L for agricultural soils, (WHO 2017). In general, the mean concentration of heptachlor at the Chamba Cluster was higher than the mean values measured at Bincheratanga and Bimbilla-Dankpe Clusters.

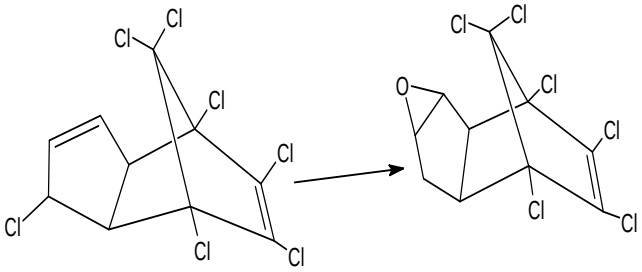

Figure 2: Degradation of heptachlor to heptachlor-epoxide

Table 1: Heptachlor and Heptachlor-epoxide (µg/L) in soil samples in the Nanumba-North Municipality of the Northern Region of Ghana.

CLUSTERS		Heptachlor and its metabolite (µg/L)	
		Heptachlor	Heptachlor-epoxide
0-20 cm	BDCS1	0.003	0.006
	BDCS2	0.005	0.006
	BDCS3	0.008	0.006
	BCS1	0.005	0.007
	BCS2	0.006	0.004
	BCS3	0.006	0.010
	CCS1	0.016	0.021
	CCS2	0.017	0.025
	CCS3	0.013	0.023
	x̄±SD	0.008±0.005	0.012±0.008
	XI	ND	ND
MRLs (WHO,		0.100	0.030

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2017)

BDCS1: soil sample from Bimbilla-Dankpe cluster one; BDCS2: soil sample from Bimbilla-Dankpe cluster two; BDCS3: soil sample from Bimbilla-Dankpe cluster three; BCS1: soil sample from Bincheratanga cluster one; BCS2: soil sample from Bincheratanga cluster two; BCS3: soil sample from Bincheratanga cluster three; CCS1: soil sample from Chamba cluster one; CCS2: soil sample from Chamba cluster two; CCS3: soil sample from Chamba cluster three; Control X1; ND: Not detected.

Table 2.0: (a, b and c) Levels of Heptachlor and Heptachlor-epoxide (µg/L) in water bodies in the Nanumba-North Municipality of the Northern Region of Ghana (ND: Not-detected)

Table 2.0a: Levels of Heptachlor and Heptachlor-epoxide in Waanpu Dam

pesticide residues	Waanpu Dam				
	Depths(µg/L)				
	0-15cm	15-30cm	30cm and below	Mean±SD	WHO MRLs(µg/L)
Heptachlor	ND	ND-0.003	ND-0.009	0.002±0.001	0.100
Heptachlor-epoxide	ND	0.006-0.017	0.010-0.025	0.011±0.010	0.030

Table 2.0b: Levels of Heptachlor and Heptachlor-epoxide in Bincheratanga Dam

pesticide residues	Bincheratanga Dam				
	Depths(µg/L)				
	0-15cm	15-30cm	30cm and below	Mean±SD	WHO MRLs(µg/L)
Heptachlor	ND	0.002-0.004	ND-0.009	0.003±0.002	0.100
Heptachlor-epoxide	ND	0.010-0.017	0.010-0.025	0.012±0.010	0.030

Table 2.0c: Levels of Heptachlor and Heptachlor-epoxide in Kumbo River

pesticide residues	Kumbo River				
	Depths(µg/L)				
	0-15cm	15-30cm	30cm and below	Mean±SD	WHO MRLs(µg/L)
Heptachlor	ND	0.002-0.006	ND-0.009	0.003±0.002	0.100
Heptachlor-epoxide	ND	ND	0.010-0.025	0.012±0.010	0.030

Table 3: Levels of Heptachlor and Heptachlor-epoxide in food crop samples

pesticide residues	Food crops items				
	Selected food crops (µg/L)				
	Yam	Maize	"Ayoyo"	Mean±SD	WHO MRLs(µg/L)
Heptachlor	ND-0.010	ND-0.010	ND-0.010	0.010±0.004	0.100
Heptachlor-epoxide	ND-0.011	ND-0.012	ND-0.021	0.014±0.006	0.030

Table 4.a: Estimated period of decay of Heptachlor and Heptachlor-epoxide in soil samples in the study area

Heptachlor and Heptachlor-epoxide in soil samples in the study area

Name of pesticide	Concentration of pesticide	Name of residue	Concentration of residue	Half-life of pesticide	Estimated period of decay
Heptachlor	0.008	Heptachlor-epoxide	0.012	3.5 years	1.5 years

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Table. 4.b: Some Pesticides and their Metabolites in Water Samples in the Study Area

Name of pesticide	Concentration of pesticide	Name of residue	Concentration of residue	Half-life of pesticide	Estimated period
Heptachlor	0.003	Heptachlor-epoxide	0.011	3.5 days	3 years

4.0 CONCLUSIONS AND RECOMMENDATIONS

4.1 CONCLUSION

Findings from this study established that the trends of heptachlor and heptachlor-epoxide residues distribution in water samples from the various depths were of the order 30 cm and below > 15-30 cm >0-15 cm. Heptachlor and heptachlor-epoxide residues detected in the various samples analysed were generally below their respective Maximum Residual Limits (MRL) WHO, (2017), respectively. The presence of heptachlor and heptachlor-epoxide in the samples could be traced to wind drift or atmospheric transport of volatilized pesticides, direct overspray, direct spillage, pesticide misuse by farmers, run-off and leaching from application fields and surrounding areas during and after their applications. Heptachlor and heptachlor-epoxide detected showed significant differences at $p < 0.05$. However, the mean concentration of heptachlor and heptachlor-epoxide at Chamba Clusters was higher than the mean values measured at Bincheratanga and Bimbilla-Dankpe. Therefore, at the moment heptachlor and heptachlor-epoxide may not pose significant risks to consumers.

4.2 Recommendations

In order to ensure sustainable and desirable environmental conditions in the Nanumba-North Municipality, the environmental protection agency must establish effective and protective measures such as integrated pest management practices and organic farming.

Extension officers should conduct regular monitoring in the municipality to prevent, control, and reduce environmental pollution, thereby minimizing health risks to the population.

DATA AVAILABILITY STATEMENT

Statistical package for social sciences (SPSS) and One-way analysis of variance (ANOVA).

CONFLICT OF INTEREST STATEMENT

Conflict of Interest: The authors declare that they have no conflict of interest.

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