STUDY OF AROCLOR CHEMICAL CLEAN-UP PROCEDURES FOR USE WITH ELECTRON CAPTURE GAS CHROMATOGRAPHY METHODS

INTRODUCTION

The effects of three chemical clean-up procedures on the Aroclor 1200 series have been investigated. The chemical procedures in question; nitration, saponification, and treatment with sulfuric acid were originally proposed by pesticide analysts for the clean-up and identification of pesticides in residue extracts. Later when polychlorinated biphenyls (PCB's) were identified as environmental contaminants these procedures were extended to the clean-up and identification of PCB's in residue extracts.

SUMMARY

1. The quantitative recovery and EC response of Aroclors are not effected by saponification or treatment with sulfuric acid.

2. Nitration can have a significant deleterious effect on the quantitative recovery and EC response of Aroclor 1242.

3. The extent of the effect of the nitration treatment is dependent on the temperature and exposure time employed as well as the Aroclor treated.

4. Analysts employing nitration procedures prior to EC analysis may or may not, depending on the conditions used, see an EC response for Aroclor 1242 if present.

DISCUSSION

We have found that saponification and treatment with sulfuric acid do not effect the quantitative recovery nor the electron capture (EC) response of the Aroclors. However, the nitration procedures have significant deleterious effects on both the quantitative recovery and EC response of the lower chlorinated biphenyls such as Aroclor 1242. The effect of this treatment on the Aroclors are shown graphically in Figures 1-7.

The fact that the lower chlorinated biphenyls are nitrated under these conditions has been verified by GC/Mass identification of the products as nitrochlorobiphenyls.

The nitration procedure employed was referenced by Risebrough* [Erro, F., Bevenue, A., and Beckman, H., Bull. Environ. Cont. Toxicol., 372 (1967)] in his Nature article on PCB residues in the environment.

Additional, we have investigated a similar nitration procedure used by Widmark and co-workers (private communication via R. E. Keller). This treatment differs from Risebrough's in that a shorter exposure time and a lower temperature are employed. The Widmark method greatly diminishes the effects observed with Risebrough's referenced method.



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RESULTS

Verification of the conclusions drawn are shown in Figures 1-7. Extraction and measurement were performed as described in Analytical Method No. 69-13.

*A quote from Risebrough's article [Nature, 220, 1098 (1968)], "The DDT compounds are destroyed by nitration and p,p'-DDT, DDD, and toxaphene are dehydrochlorinated by saponification with alcoholic KOH. PCB is not degraded by either procedure."

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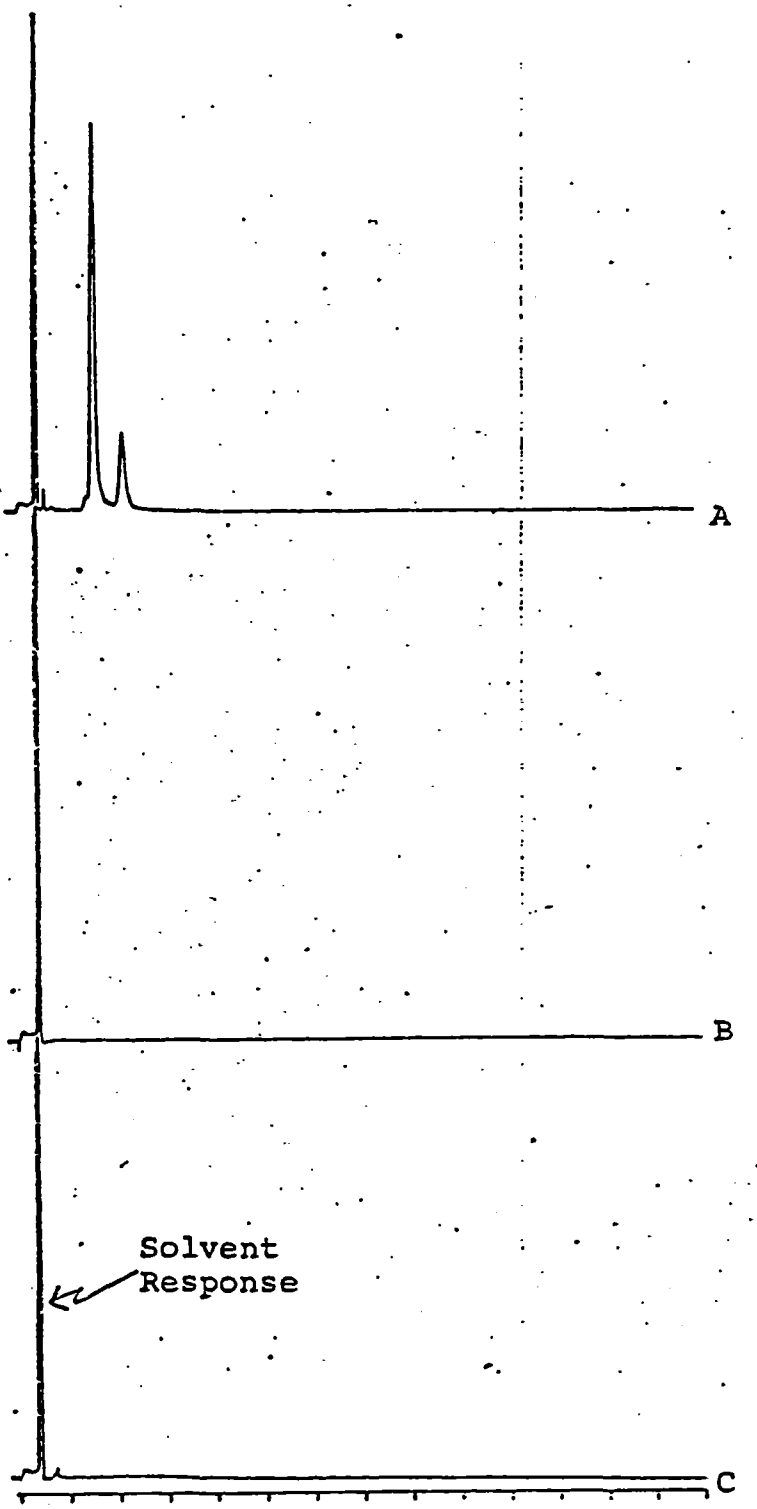

FIGURE 1. Electron capture gas chromatographic trace of (A) biphenyl treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) biphenyl treated with distilled water and extracted into hexane, and (C) untreated biphenyl standard in hexane.

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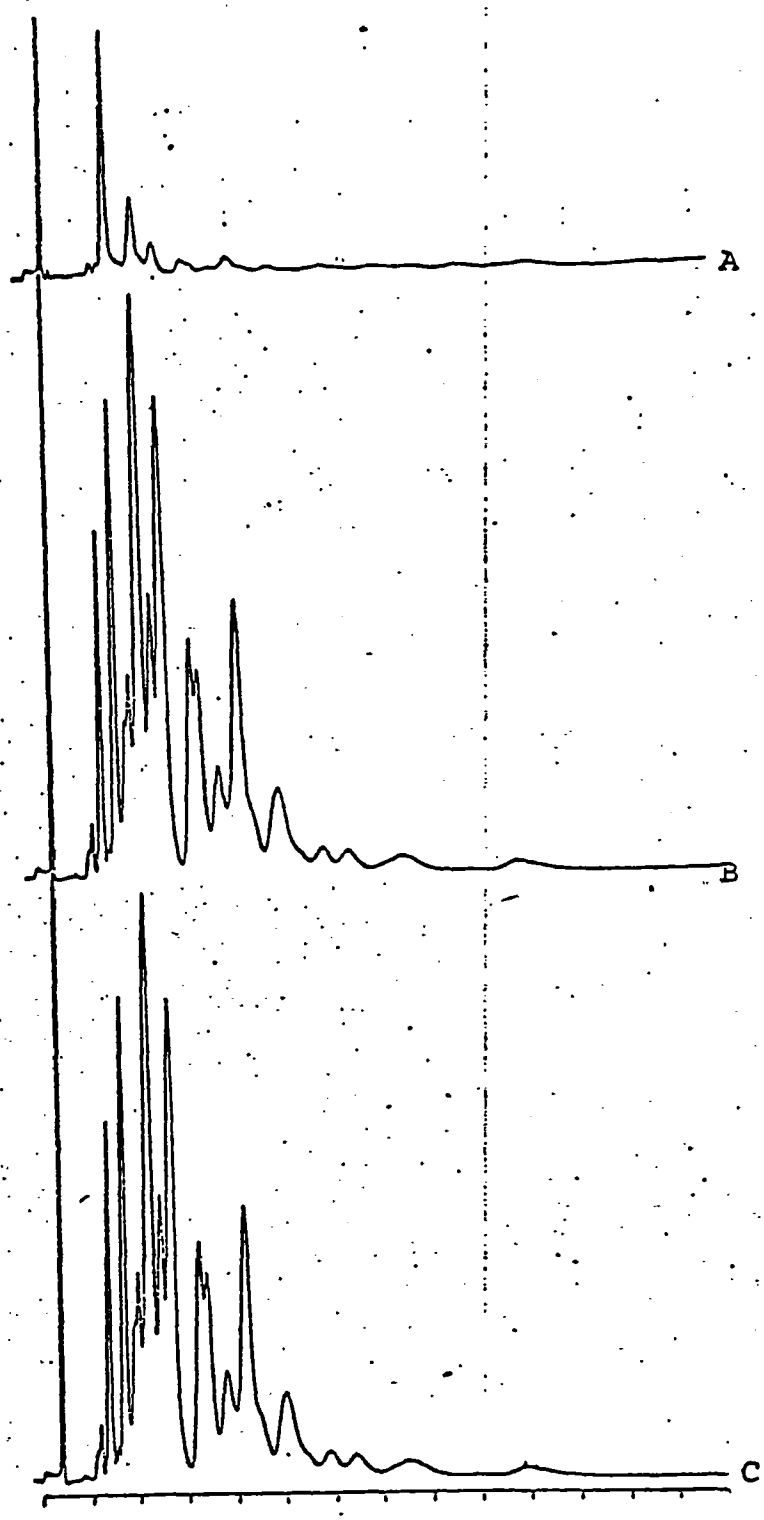

FIGURE 2. Electron capture gas chromatographic trace of (A) Aroclor 1242 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1242 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1242 standard in hexane.

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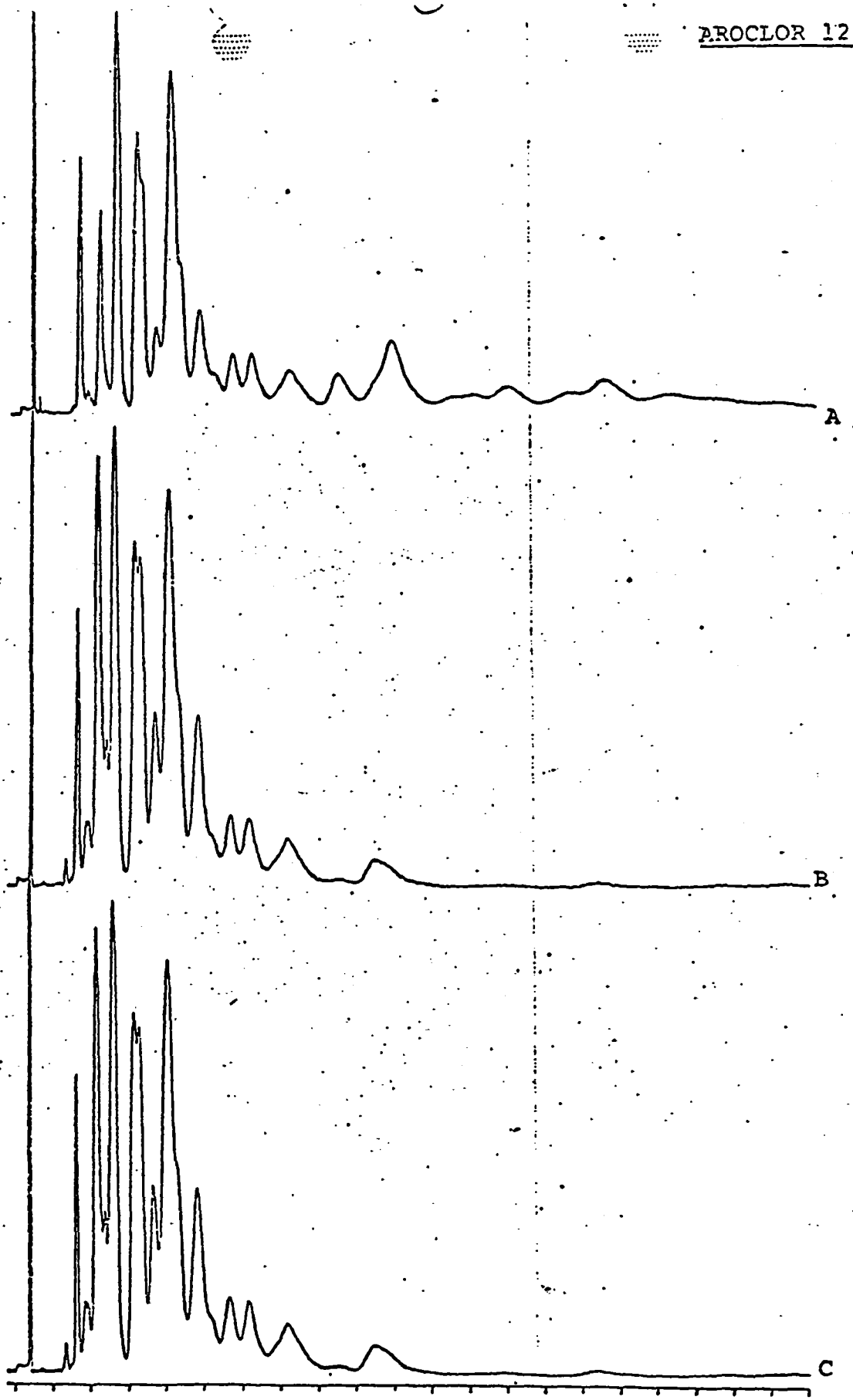

FIGURE 3. Electron capture gas chromatographic trace of (A) Aroclor 1248 treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1248 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1248 standard in hexane.

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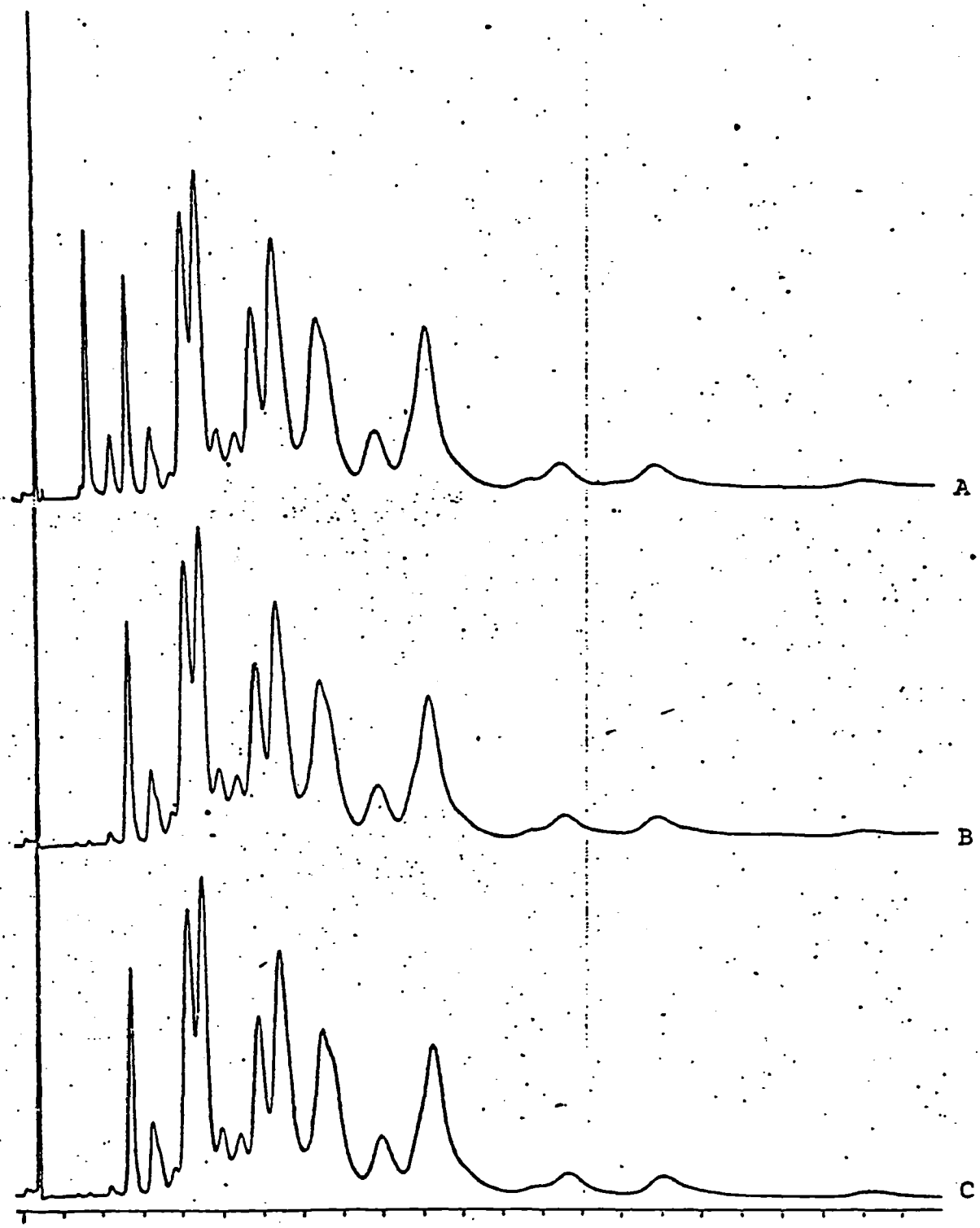

FIGURE 4. Electron capture gas chromatographic trace of (A) Aroclor 1254 treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1254 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1254 standard in hexane.

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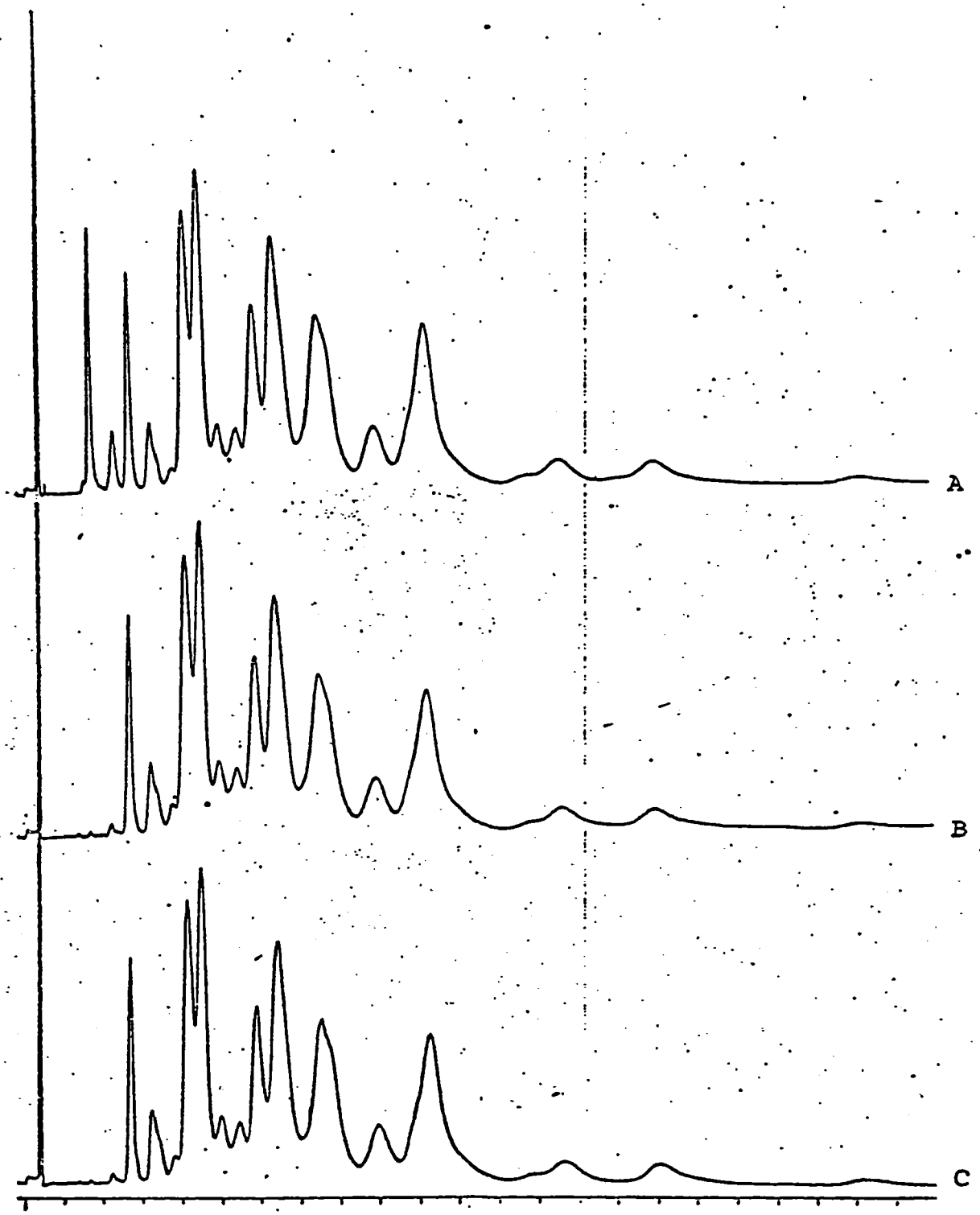

FIGURE 4. Electron capture gas chromatographic trace of (A) Aroclor 1254 treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1254 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1254 standard in hexane.

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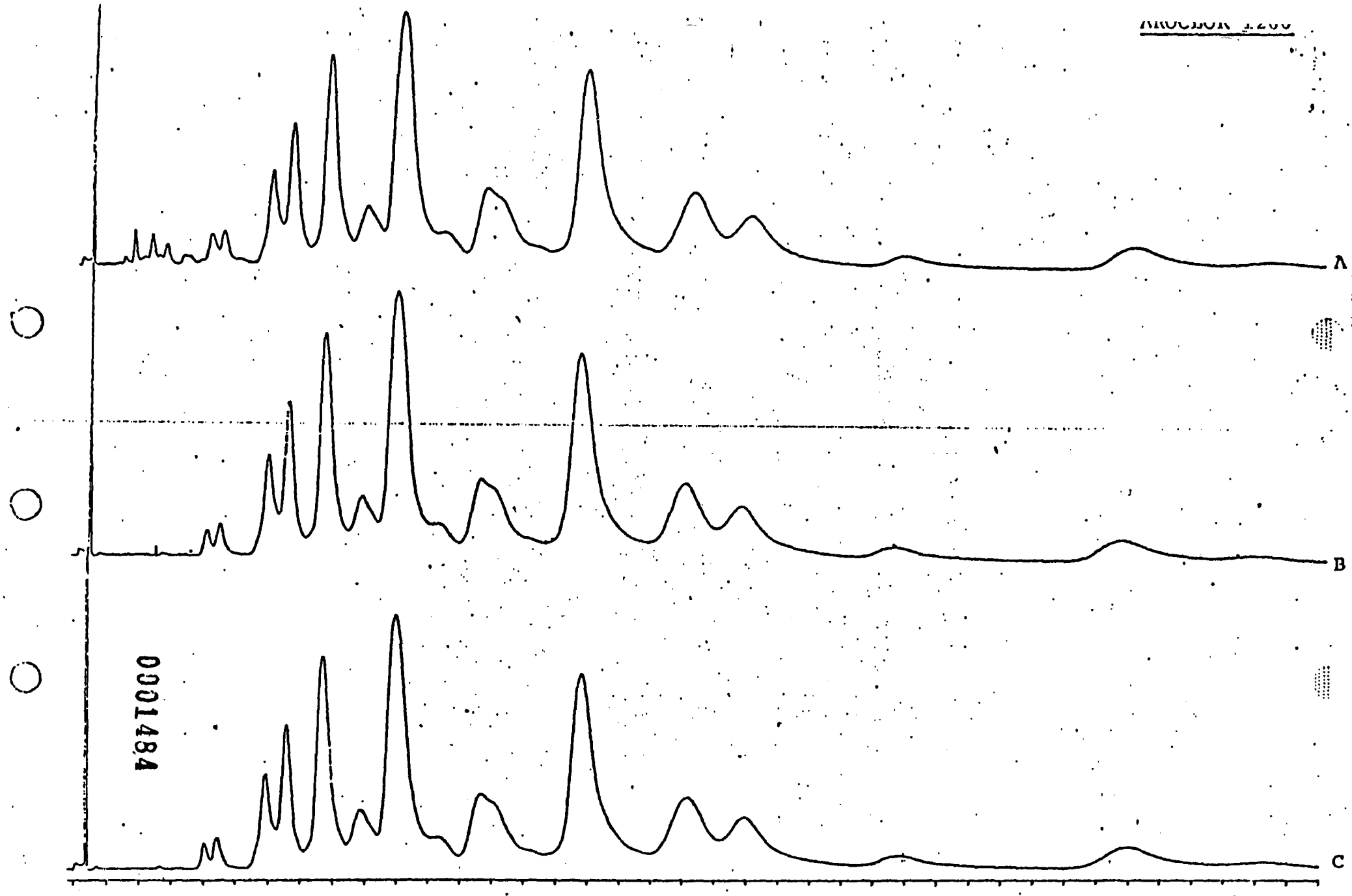

FIGURE 5. Electron capture gas chromatographic trace of (A) Aroclor 1260 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1260 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1260 standard in hexane.

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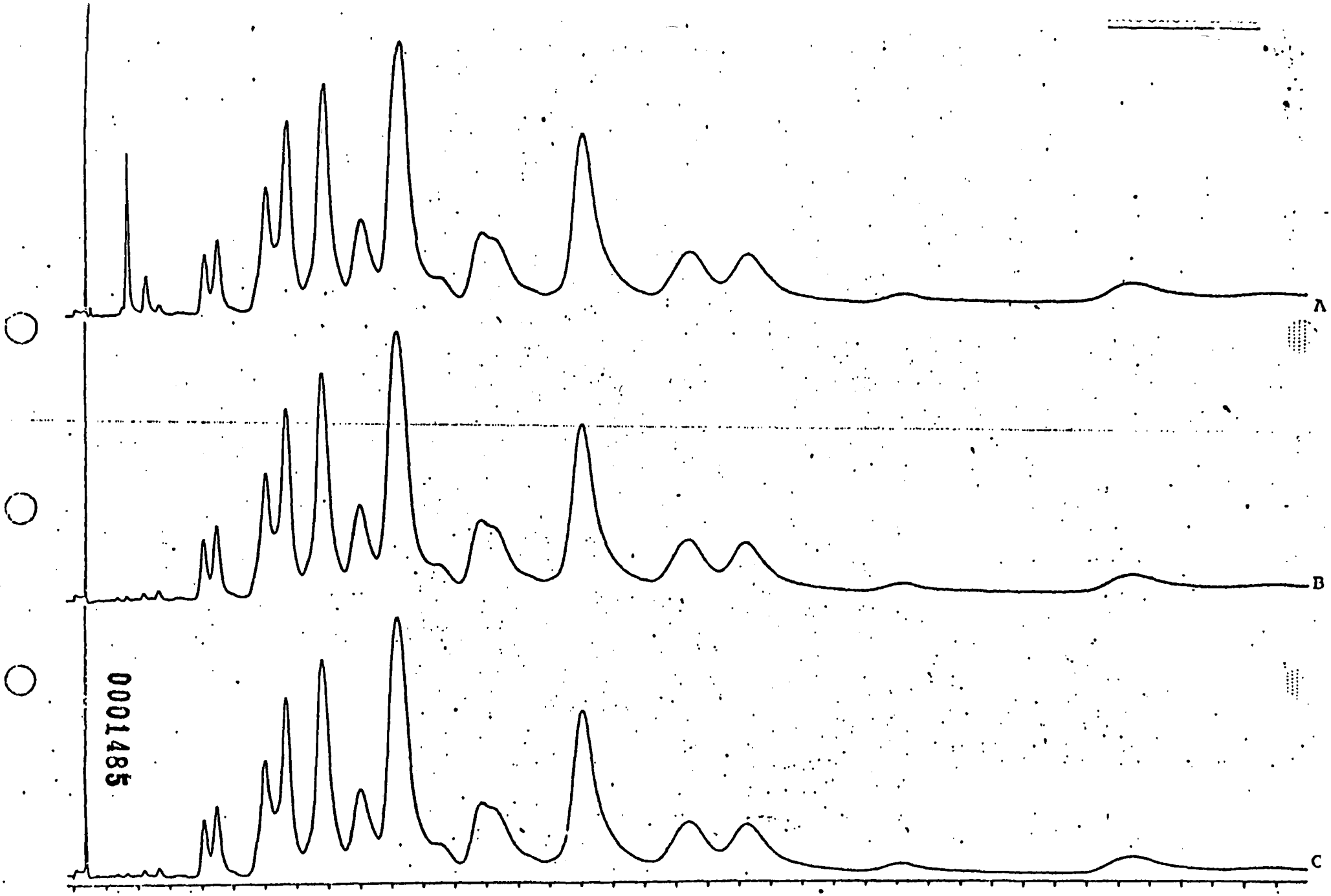

FIGURE 6. Electron capture gas chromatographic trace of (A) Aroclor 1262 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1262 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1262 standard in hexane.

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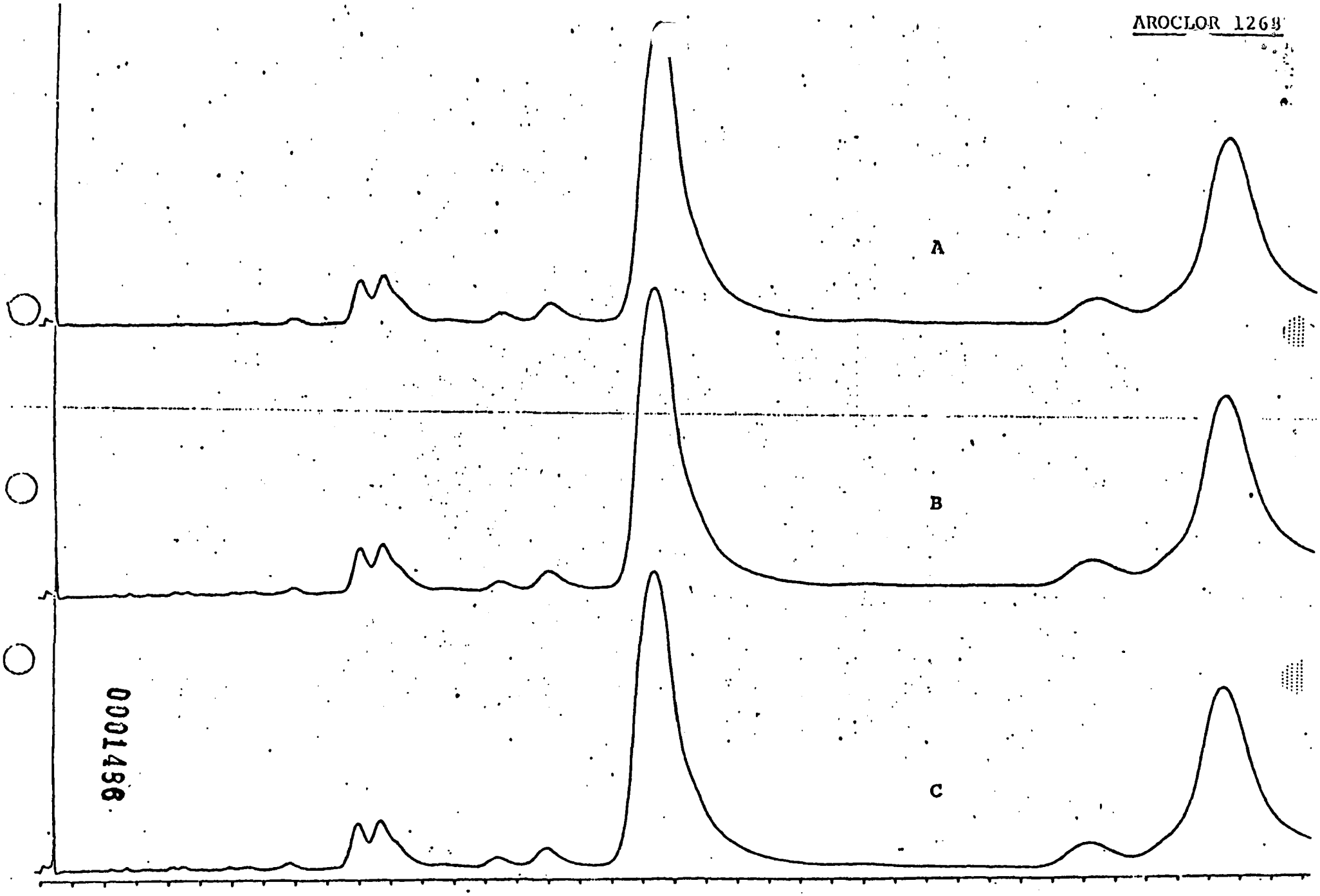

FIGURE 7. Electron capture gas chromatographic trace of (A) Aroclor 1268 and treated with 1:1 mixture of nitric and sulfuric acids and extracted into hexane, (B) Aroclor 1268 treated with distilled water and extracted into hexane, and (C) untreated Aroclor 1268 standard in hexane.

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