Cover Sheet for

ENVIRONMENTAL CHEMISTRY METHOD

Pestcide Name: Mecoprop (MCPP)

MRID #: 400535-02

Matrix: Soil

Analysis: HPLC/UV

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400535-02

LX179-01 (MCPP) Field Dissipation-Terrestrial
For Crop Use (FIFRA 164-1)

APPENDIX B

Analysis of Soil Samples

Pages 25-27

APPENDIX B

I. EXTRACTION OF MCPP FROM SOIL

A. Weigh 50.0 grams of processed soil into 250 ml centrifuge bottle.

B. Add stir bar and 200 ml of a 50/50 ethanol/deionized water solution to centrifuge bottle. Cap centrifuge bottle and stir for 1/2 hour.

C. Centrifuge for 10 minutes at 2000 rpm.

D. Decant supernatant into labelled 500 ml separatory funnel.

E. Prepare 100 ml of 50/50 ethanol-deionized water solution and add approximately 50 ml to centrifuge bottle. Using glass stir rod, loosen soil. Add remaining portion of ethanol-deionized water solution rinsing stir rod thoroughly.

F. Stir for approximately 30 seconds.

G. Centrifuge for 10 minutes at 2000 rpm.

H. Decant supernatant into separatory funnel.

I. Add 1.0 ml of concentrated hydrochloric acid and 100 ml of chloroform to the separatory funnel.

J. Swirl separatory funnel for approximately 1 minute, releasing pressure occasionally.

K. Allow interface to form. Swirl occasionally to break large bubbles.

L. Weigh 25 grams of anhydrous sodium sulfate and place on coarse fritted glass filter. Place filter on top of a 500 ml evaporating flask.

M. Remove chloroform portion from separatory funnel allowing it to filter through the sodium sulfate layer and the course fritted filter.

N. Add 50 ml chloroform to separatory funnel. Swirl for approximately 1 minute releasing pressure occasionally.

O. Allow interface to form. Swirl occasionally to break large bubbles.

P. Remove chloroform portion allowing it to filter through the sodium sulfate layer and the coarse fritted filter.

Q. Using a pasteur pipet, rinse the coarse fritted filter with approximately 50 ml chloroform.

R. Place evaporating flask on the rotovac at 40C and evaporate to dryness under vacuum.

S. Add 5.0 ml mobile phase to evaporating flask to dissolve residue. Transfer to a 5 ml volumetric flask using a pasteur pipet. Do not bring volumetric flask to volume.

II. INSTRUMENTS AND EQUIPMENT

A. Tracor 995 Chromatographic Pump

B. Tracor 970A Variable Wavelength Detector

C. Varian 9176 Recorder

D. Z Module with C-18 cartridge, Waters #85721

E. 100 µl Injection Loop

III. REAGENTS

A. Deionized Water

B. Ethanol, Dehydrated, 200 Proof, U.S. Industrial Chemicals Co.

C. Chloroform (HPLC), Mallinckrodt

D. Hydrochloric Acid, J.T. Baker Chemical Co.

E. Sodium Sulfate, Granular Anhydrous, Mallinckrodt

F. Acetonitrile (HPLC), American Burdick and Jackson

G. Glacial Acetic Acid, J.T. Baker Chemical Co.

IV. LIQUID CHROMATOGRAPHIC UNIT

A. Parameters

1. Flow: 2.0 ml/min
2. Wavelength: 280 nm
3. Absorbance: 0.04 to 0.005 AUFS
4. Injection Volume: 100 µl

B. Mobile Phase - Make Fresh Daily

1. Measure 370 ml of acetonitrile in a 500 ml graduated cylinder and transfer to a 1000 ml Erlenmeyer flask.
2. Add deionized water to 1000 ml.
3. Add 10 ml of glacial acetic acid to the above solution.
4. Add stir bar and thoroughly mix.
5. Degas solution using a medium fritted glass filter.

6. With the conditions listed above, the MCPP peak was eluted at a retention time of 10 to 12 minutes.

V. PREPARATION OF STANDARDS

A. Make a primary standard solution by weighing out approximately 12 mg MECOPROP into a 10 ml volumetric flask. Dilute to volume with mobile phase.

B. Make a secondary standard by pipetting 1.0 ml of the primary standard solution into a 25 ml volumetric flask using the positive displacement pipet. Dilute to volume with mobile phase. Approximate Concentration: 50 µg/ml.

C. Make working standards by diluting appropriate aliquots of the secondary standard with mobile phase. Approximate concentrations: 1, 5 and 10 µg/ml.

VI. CALCULATIONS

The concentration of MCPP was calculated for each sub-sample using the following equation:

$$\text{conc in ppm} = \frac{(\text{h-unk}/\text{h-std}) \times (\text{conc std in } \mu\text{g/ml}) \times D}{(C) \times (\text{dry weight of soil in grams})}$$

h-unk = peak height of unknown
h-std = average peak height of standard
D = dilution factor
C = correction factor determined by fortified control soil sample

The dry weight of soil will be calculated using a factor obtained by moisture determination.

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APPENDIX C

Representative Chromatograms from Analyses

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Representative chromatograms from the analysis for MCPP are presented on the following pages:

1) A standard solution of MCPP (CH-686)

2) An analysis of a soil layer 1 from the Georgia site (CH-562)

3) An analysis of a soil layer 2 from the Georgia site (CH-577)

4) An analysis of a soil layer 3 from the Georgia site (CH-825)

5) An analysis of a soil layer 4 from the Georgia site (CH-521)

6) An analysis of a soil sample fortified with MCPP at a level of 1.0 ppm (CH-287)

7) An analysis of a soil sample fortified with MCPP at a level of 0.2 ppm (CH-305)

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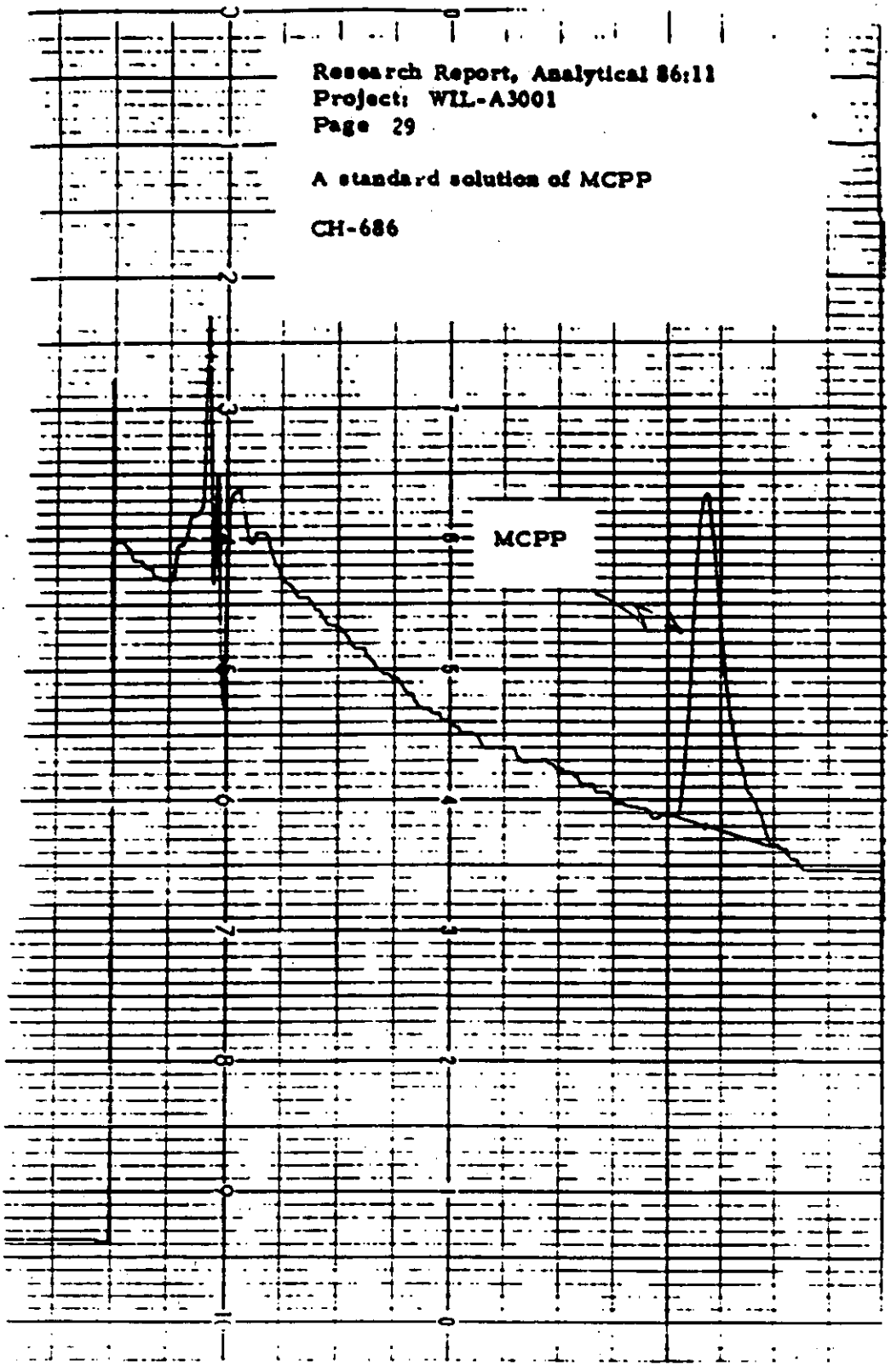
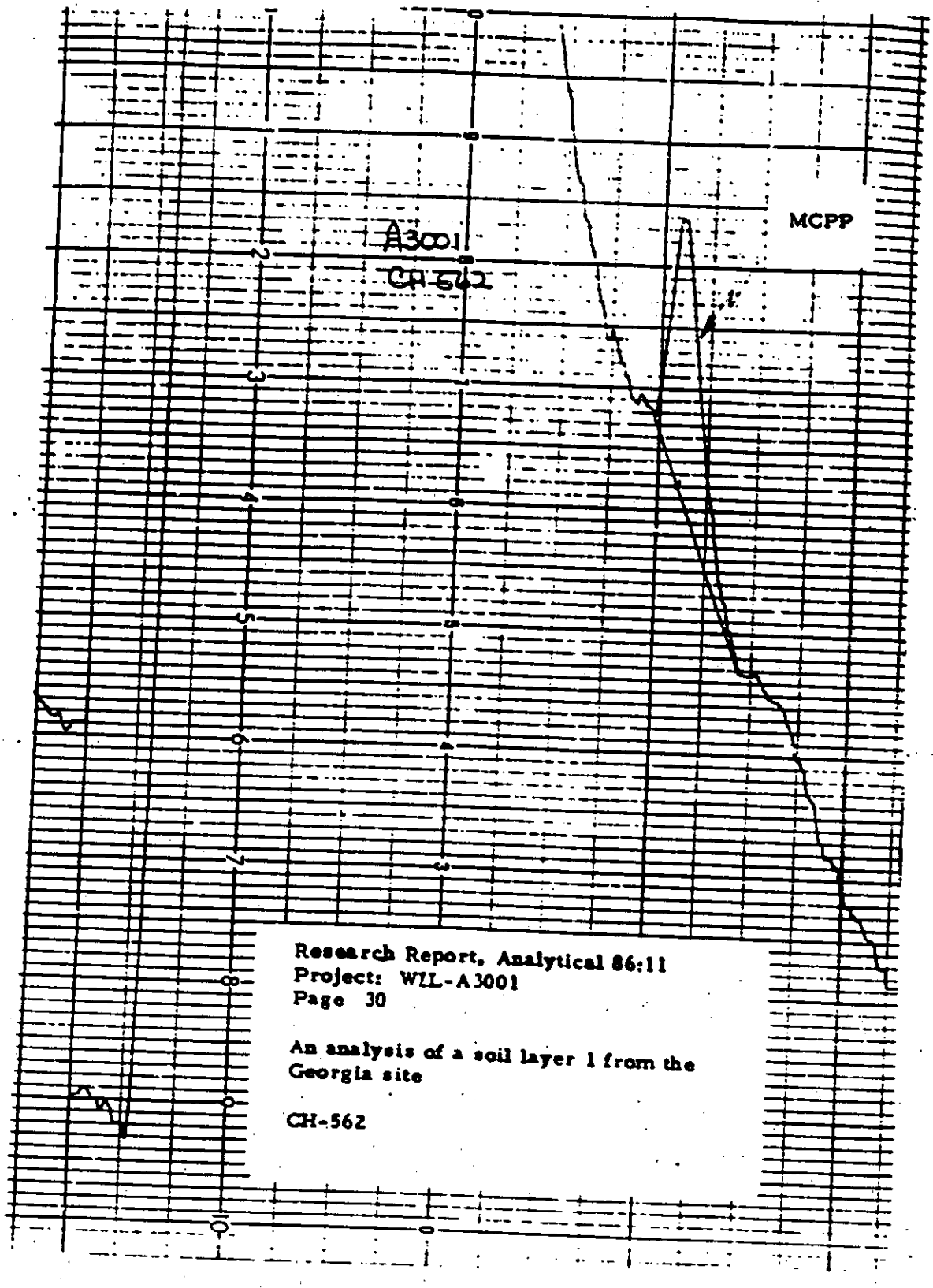
A standard solution of MCPP

CH-686





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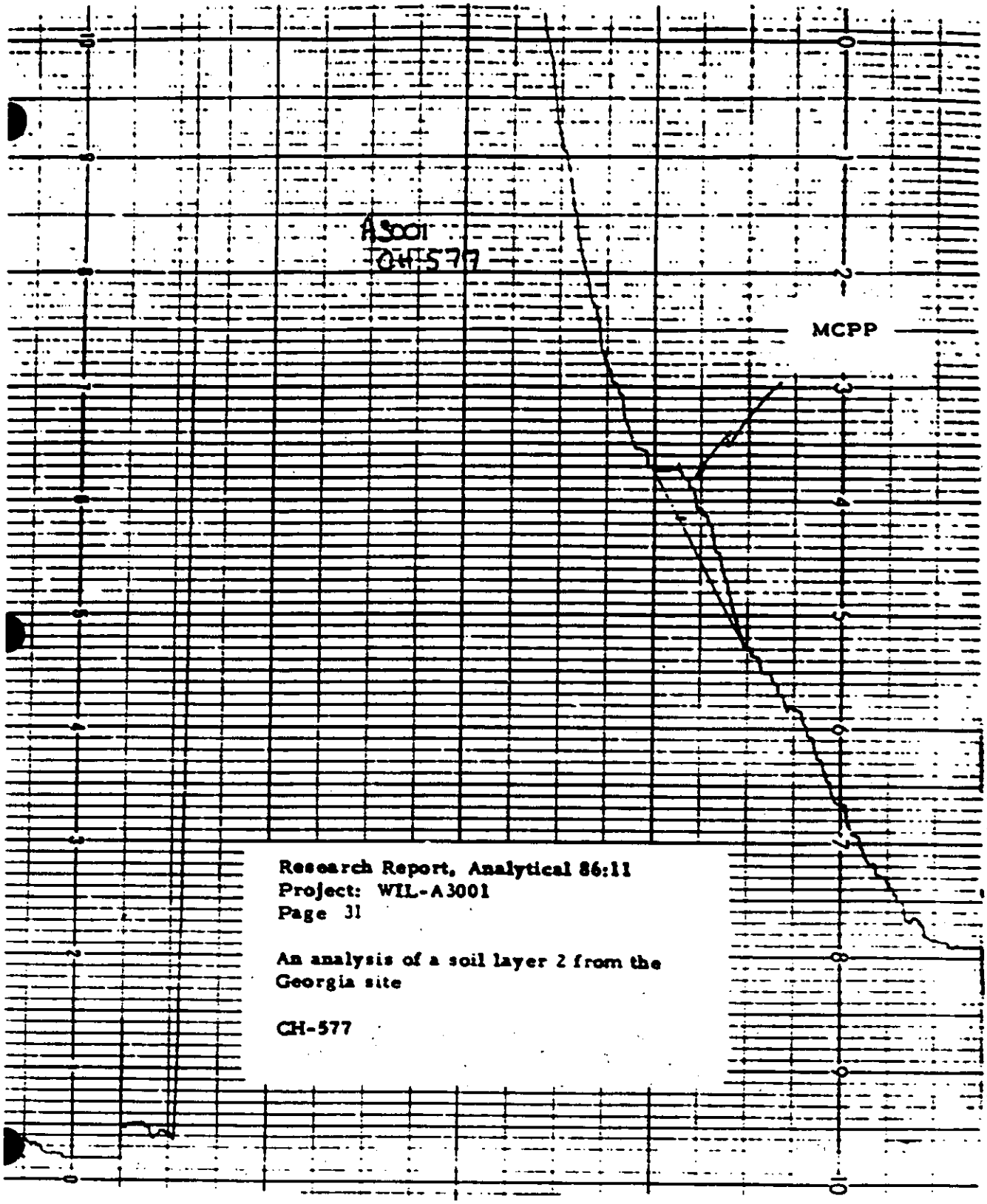
An analysis of a soil layer 1 from the Georgia site

CH-562



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An analysis of a soil layer 2 from the Georgia site

CH-577

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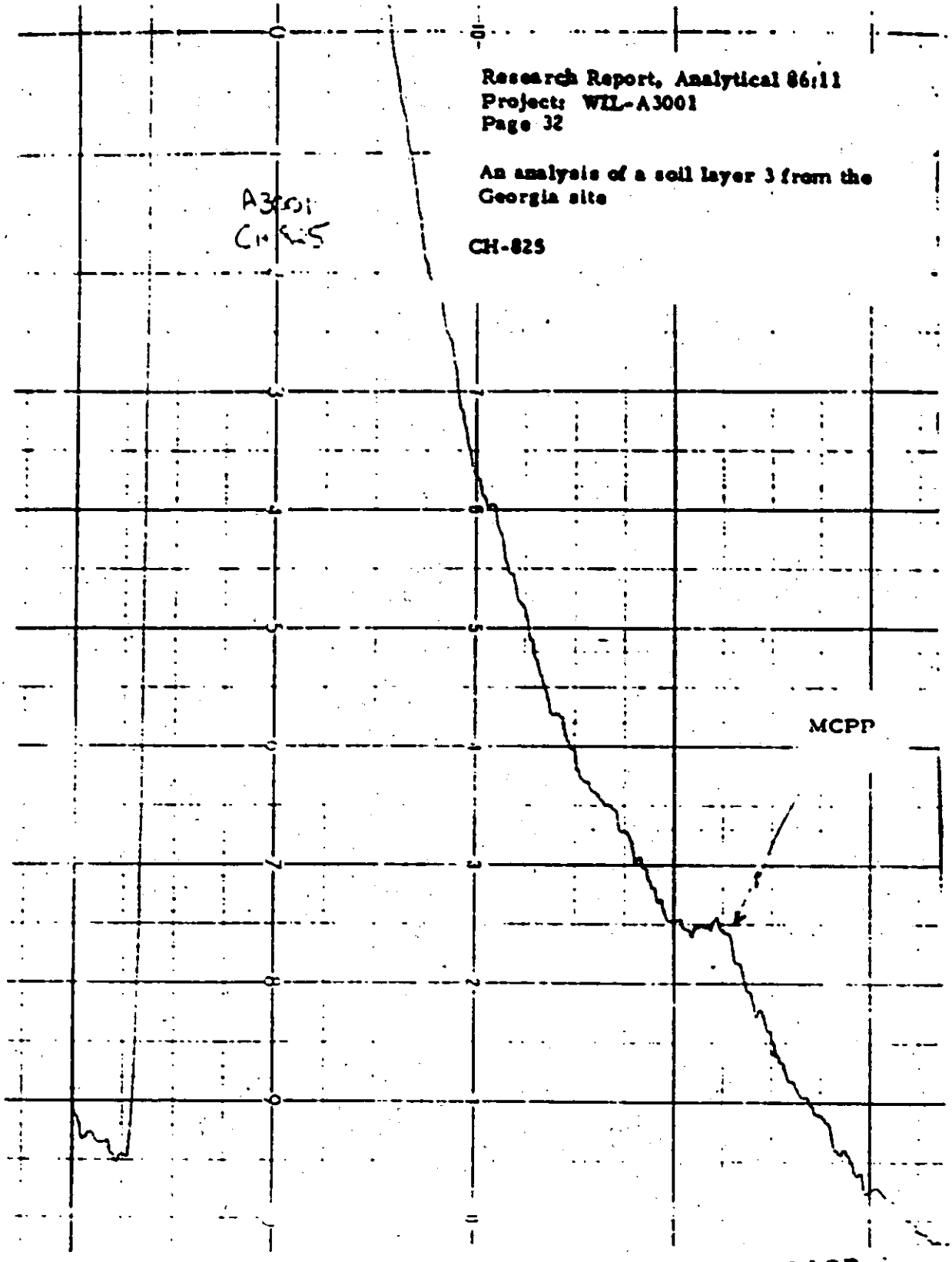
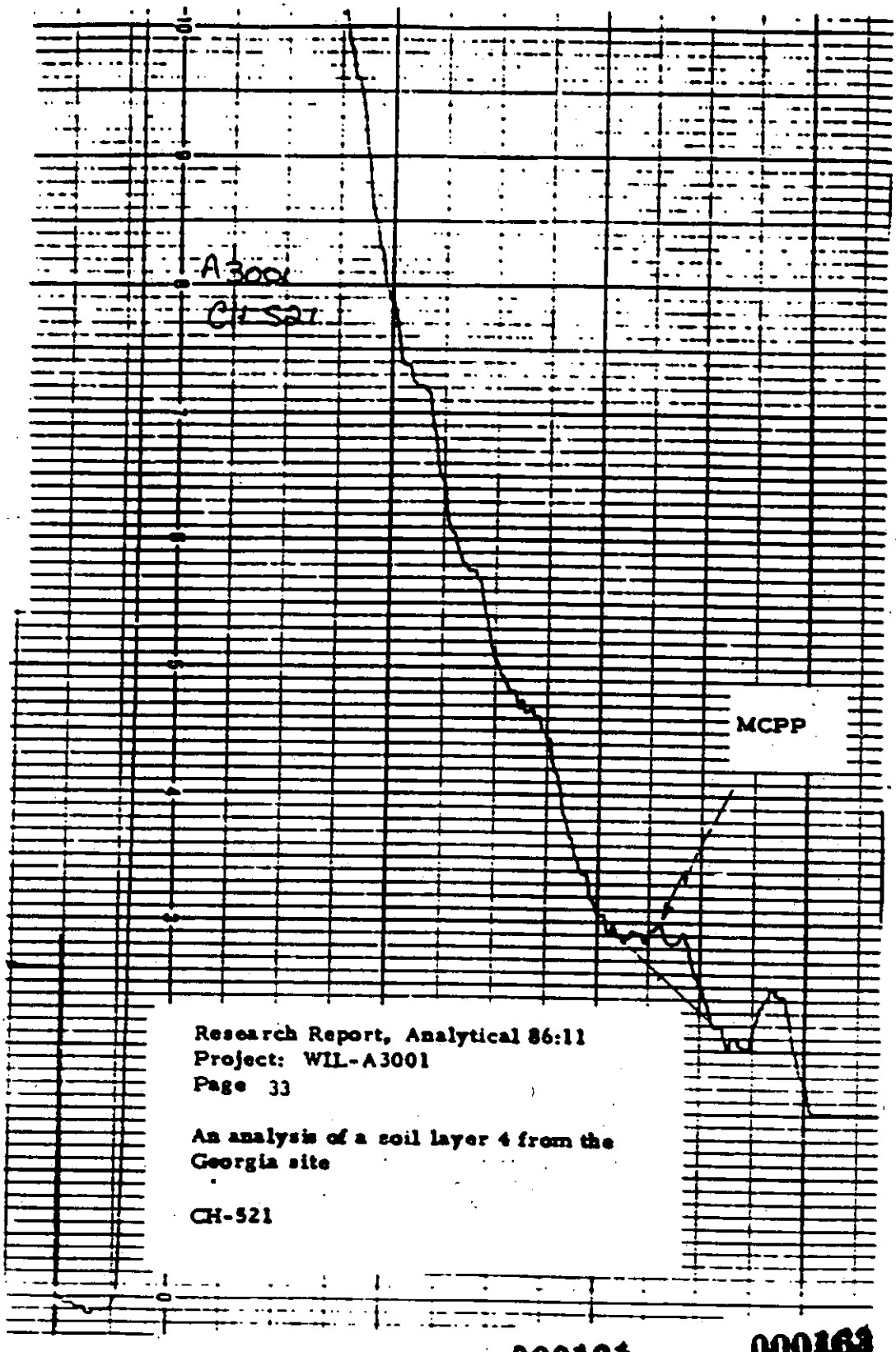
An analysis of a soil layer 3 from the Georgia site

CH-825

A3001
CH-5





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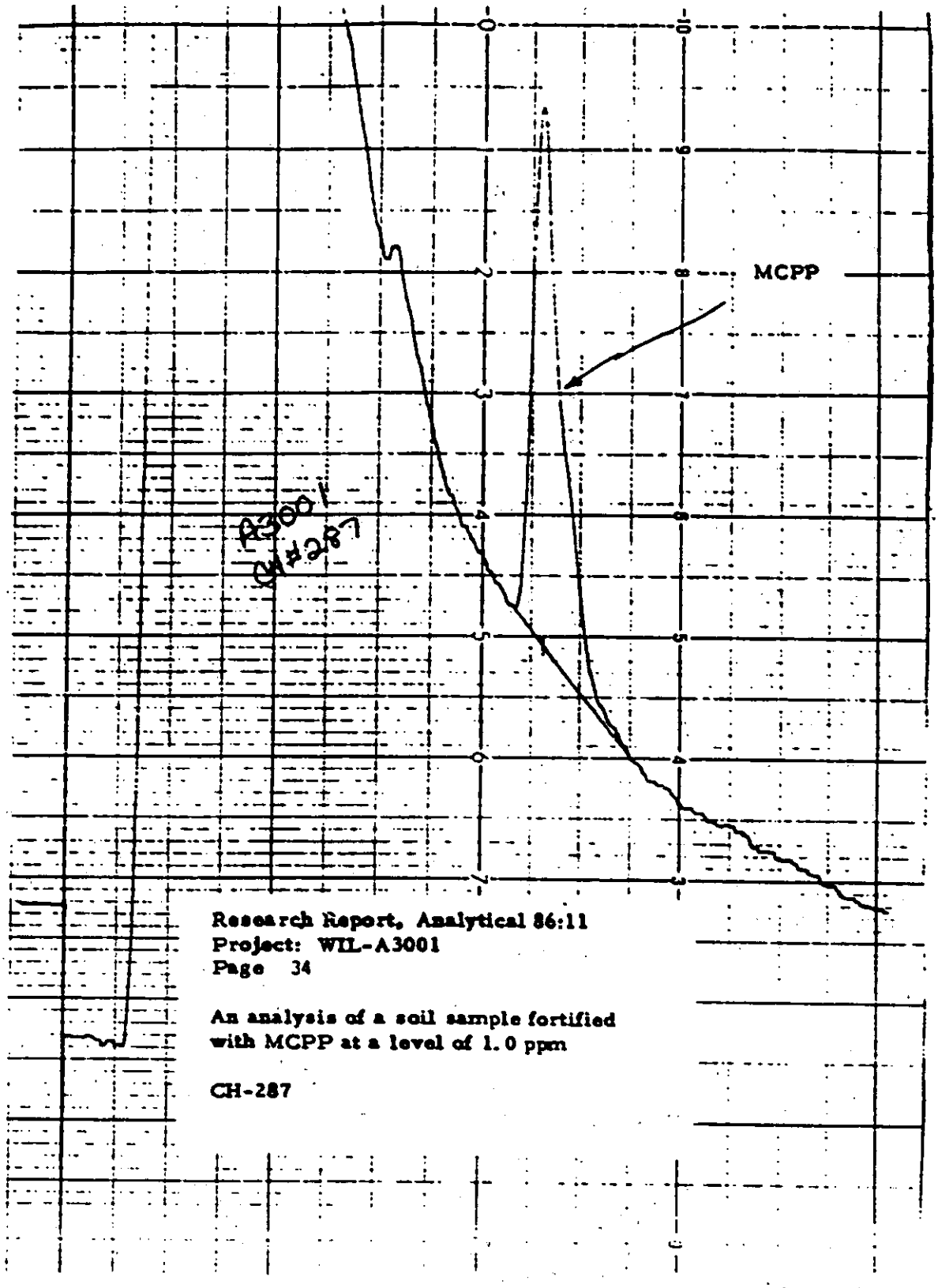
An analysis of a soil layer 4 from the Georgia site

CH-521



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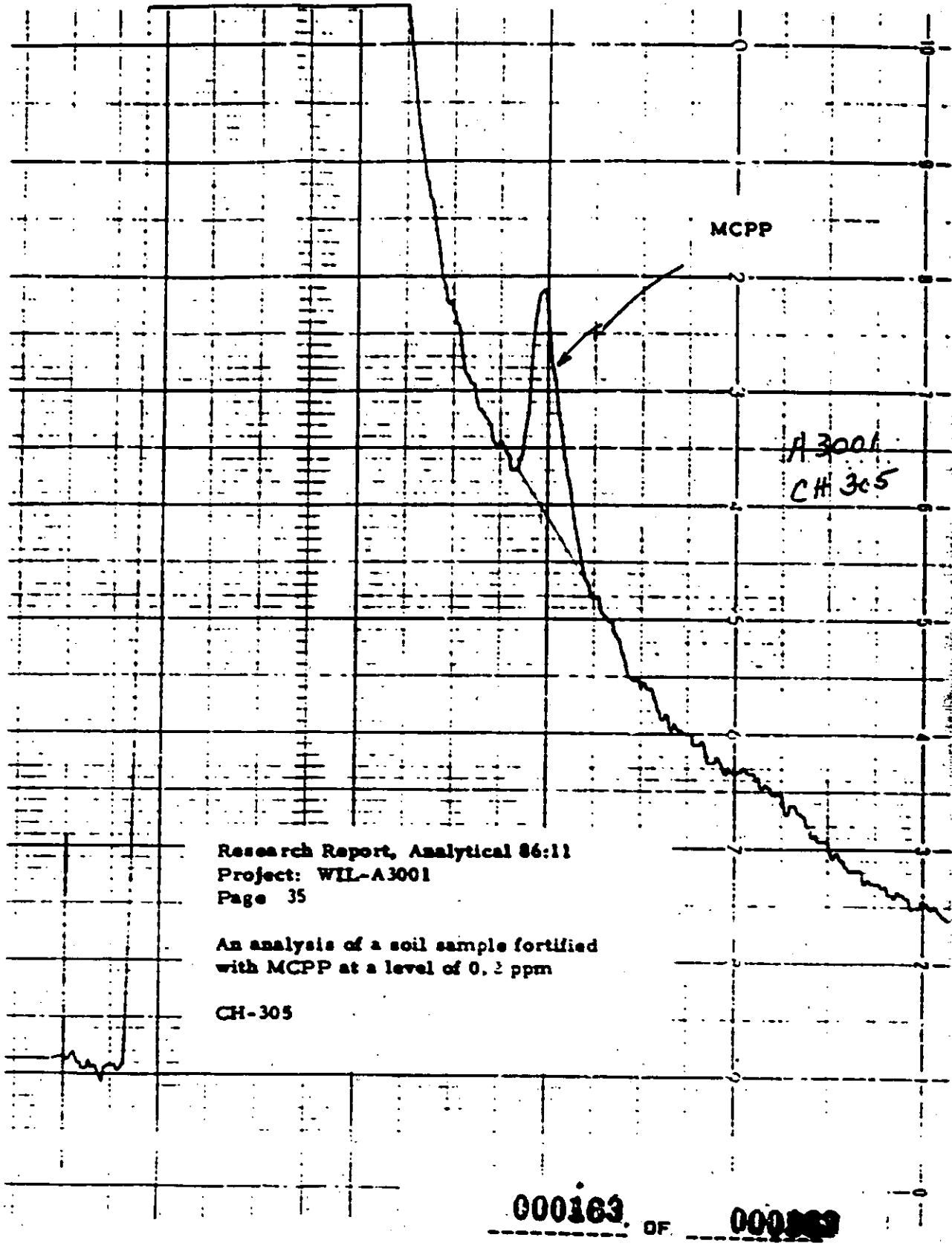
An analysis of a soil sample fortified with MCPP at a level of 1.0 ppm

CH-287



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An analysis of a soil sample fortified with MCPP at a level of 0.2 ppm

CH-305