NR 1290(ESR/ENG)

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Date: January 27, 1992

Study Title

Metabolism of NTN 33893 in eggplant by planting hole application

Data Requirement

EPA Guidelines Subdivision O Section 171-4(a)2

Author

[redacted]

Laboratory Project ID

No. 89054/ESR

Performing Laboratory

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Project ID : No. 89054/ESR
Test substance : NTN 33893 (imidacloprid)
Study initiation date : 23 Jun. 1989
Experimental start date : 20 Jul. 1989
Experimental termination date: 08 May 1990

Signatures:

[redacted]

Date: 05 Feb. 1991
(Study completion date)

Date: 08 Feb. 1991

Date: 13 Feb. 1991

Date: 05 Feb. 1991

Date: 13 Feb. 1991

Archives: GLP Archives (Room No. 2306) in the ESR building

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Quality Assurance Statement

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SUMMARY

Metabolism of NTN 33893 (pyridylmethy-^{14}C-label) in eggplant was done under the GLP regulations. The objective of this study was to clear absorption, translocation and degradation of NTN 33893 in the plant after 1 % granule (0.94 % a.i.) was applied to soil at a maximum commercial rate of 2 g per a young plant (8 leaves stage) by planting hole application. The radioactivities were accounted for 2.72% of the applied radioactivity in aerial parts (stem and leaves) at 14 days after the application and an average of 1.64% in aerial parts (foliage and fruit) at 69 days. Since more than 88 % of the radioactivities were found in the leaves, the absorbed radioactivities seemed to be translocated acropetally to leaves. In the edible parts sampled at 49, 53 and 67 days after the application, 0.01 to 0.02 % of the applied radioactivity (0.032 to 0.053 mg/kg, an average of 0.043 mg/kg in NTN 33893 equivalent) were found.

Similar metabolites were found both in the leaves and edible parts. WAK 4103, NTN 35884, NTN 33519, WAK 3839, NTN 38014, RBN 1114 and CNA were found as metabolite in the foliage at 69 days after the application. Major metabolites in the foliage were NTN 38014 (an average of 24.6 % of the radioactivity found in the foliage), RBN 1114 (5.6%) and WAK 4103 (3.6 %). Unchanged parent compound and nonextracts were accounted for 10.2 % and 9.3 % of the found radioactivity, respectively.

In the edible parts, NTN 33893 (an average of 18.9 % of the radioactivity found, an average of 0.0081 mg/kg), NTN 38014 (14.0 %, 0.0049 mg/kg), RBN 1114 (13.0 %, 0.0066 mg/kg), CNA (13.4 %, 0.0035 mg/kg), WAK 4103 (3.2 %, 0.0015 mg/kg), NTN 35884(0.2 %, <0.0005 mg/kg) and WAK 3839 (0.1 %, <0.0005 mg/kg) were found. Major metabolites in the edible parts were NTN 38014, RBN 1114 and CNA. The amount of nonextracts was accounted for 6.5% (0.0028 mg/kg in NTN 33893 equivalent) and unknown metabolites accounted for greater than 10 % of the radioactivity were not present in the edible part.

The results of this study showed major metabolic pathways of NTN 33893 in eggplant are elimination of nitro moiety, hydroxylation of imidazolidine ring and cleavage of C-N bond between pyridylmethyl moiety and imidazolidine ring.

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INTRODUCTION

NTN 33893 (imidacloprid), a systemic insecticide having a new type of structure and a wide spectrum, has been developed by BAYER AG and Nitokuno. Since the chemical will be used widely to control pests in paddy and upland fields, metabolism studies in plants were carried out with some crops. Environmental science research (ESR) in Yuki research center contracted under cooperative studies with BAYER AG to investigate metabolism of NTN 33893 in eggplant to partially fulfill the registration requirements set forth by U.S.A, European countries and Japan. The study was done according to EPA Guidelines Subdivision O Section 171-4(a)2 in compliance with the GLP regulations. The purposes of this study are to clear absorption, translocation, material balance, major metabolic pathways and terminal residue of NTN 33893 in eggplant after planting hole application. All raw data containing floppy disks and original report were archived in the ESR building (room No. 2306).

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MATERIALS

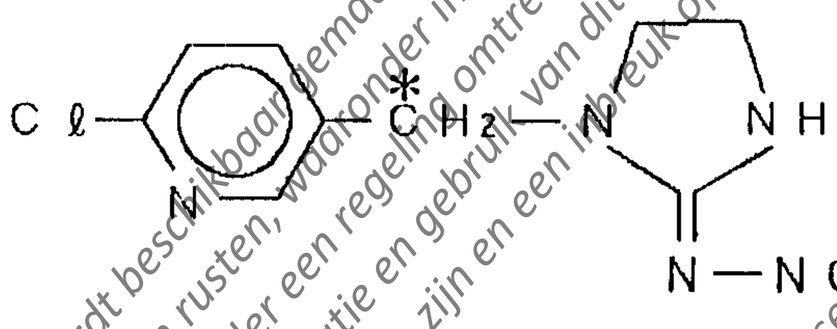
1. Test substances

1-1 Non-labeled NTN 33893

Lot No.	:	ESR No. 494
Purity	:	>99%(HPLC)
Chemical name	:	1-(6-chloro-3-pyridylmethyl)-N-nitro(imidazolidin-2-ylidene)amine (IUPAC) 1-[(6-chloro-3-pyridinyl)methyl]-4,5-dihydro-N-nitro-1H-imidazol-2-amine (CAS)
Common name	:	imidacloprid
CAS No.	:	105827-78-9
Molecular formula	:	$C_9H_{10}ClN_5O_2$
Molecular weight	:	255.66
Solubility (at 20 °C)	:	water 0.51g/l n-hexane <0.1g/l dichloromethane 50-100g/l 2-propanol 1-2g/l

1-2 ^{14}C-labeled NTN 33893

Structure



*: ^{14}C labeled position

Lot No.	:	KML 1705 (46-nit-4/ESR)
Radiochemical purity	:	>99% (HPLC, TLC)
Specific radioactivity	:	5.74MBq/mg (155μCi/mg)
	:	1467MBq/mmol (39.6mCi/mmol)

The labeled NTN 33893 was supplied by [redacted] (Isotope laboratory, BAYER AG) in July,1989.

2. Reference substances

The reference substances were supplied by synthesis divisions of Nitokuno and BAYER AG. The structures of references were summarized in Appendices I and II.

3. Test plant

Plant name : Eggplant (Solanum melongena L.)
Race : Senryou No.2
Stage : Eight leaves

Senryou No. 2 is a popular race in Japan. A young plant (2-3 leaves stage) was obtained from Daiyatopy Co. and cultivated in a greenhouse until 8 leaves stage.

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4. Test soil

Texture : Light clay(LiC, international), Clay(CL, USDA)
Origin : Volcanic ash
Sampling area: Tama test field of Nitokuno, Yuki-shi Ibaraki, JAPAN

Characteristics of the soil were shown in Appendix III.

5. Planting container

Container : Wagner pot (plastics)
Surface area : 1/2000a (500cm^2)
Height : 30cm
Soil height in the container : 29cm

The test plants were transplanted into the containers at the same time of chemical application.

6. Greenhouse

The cultivation of test plants was done in a greenhouse (No.2) located in 4th floor of the ESR building. The greenhouse was build up with Pyrex-glasses. Temperature and humidity in the greenhouse were controlled and monitored automatically during the cultivation.

7. Reagents

Water : High purity water obtained from Milli Q SP (Waters) equipped with a sterile filter (0.22μm)

Organic solvents : HPLC grade (Wako, Japan)

Cocktail for liquid scintillation counting : Ready-gel (Beckman), Permafluor V and Carbo-sorb (Packard)

Reagent for paired ion chromatography : PIC B-8 Low UV (Waters)

Ion exchange resin : XAD-2 (Nakarai, Japan), CM-Sepharose CL-6B and DEAE-Sepharose CL 6B (Pharmacia)

TLC plate : Glass plate coated with silica gel (silica gel F_{254}, 0.25mm thickness, Merck)

8. Equipments

High performance liquid chromatography (HPLC) : The system described below was used.

Column : RP-Select B (5μm, 4mm X 250mm, Merck)
: RP-8(e) (5μm, 4mm X 250mm, Merck)
Pump : Waters, model 600E
UV detector : Waters, model 490
Radioactivity detector : Raytest, Ramona-5-LS
Sample injector : Gilson, model 231

TLC liner analyzer : Raytest, Rita-3200

Liquid scintillation counter (LSC) : Beckman, model LS 5801 and LS 3801

Oxidizer : Packard, model 306 and Aloca, model ASC-113

Freeze dryer : Labconco, model 5

Centrifuge machine : Hitachi, model SCR20BA

Rocking mixer : Aichidenki, model RM-30

Homogenizer: Kinematica, Polytron

Mass spectrometer : JEOL, model Dx 303 equipped with computer (JEOL, model DA 5100)

METHODS

1. Formulation

One % NTN 33893 granule was prepared and then used for the study.

a.i.	a.i. %	type	specific radioactivi.
^{14}C-NTN 33893	0.94	granule	97147.2dpm/a.i.μg
non-labeled NTN 33893	0.95	granule	

1-1 Preparation of 1% ^{14}C NTN 33893 granule

A portion (72.7 ml) of ^{14}C-NTN 33893 methanol solution (0.364mg/ml, 56.4μCi/ml) was pipetted into a brown vessel and evaporated solvents under reduced pressure (26.5mg a.i., 4.1mCi). A portion (7.0ml) of dichloromethane and 67.2mg of non-labeled NTN 33893 were added into the vessel. After mixing the vessel, 7.0042mg of carrier (size distribution:0.6 to 1.1mm, contents:bentonite 35%, talc 64.5%, sodium lignin sulfonic acid 0.5%) were also added into the vessel and the solvent was evaporated absolutely by using a rotary evaporator. The granule prepared was transferred into a mixing bottle from the vessel. A portion (3ml) of dichloromethane and 2.907g of the carrier were added again into the vessel and handled with same manner described above. The combined granule was mixed sufficiently in the bottle and about 10g of 1% ^{14}C-NTN 33893 granule were obtained. After the preparation, a portion of the granule was checked a.i. contents and stability by TLC and HPLC analyses prior to the application.

1-2 Preparation of 1% non-labeled NTN 33893 granule

About 100g of 1% non-labeled NTN 33893 granule were also prepared by the same manner. Stability and contents of a.i. in the granule were checked prior to application.

2. Application of the chemicals to the plant

Recommended application amounts of 1% NTN 33893 granule are 1 to 2g per plant for side dressing and/or 30kg/ha (0.3kg a.i./ha)

for incorporation. In this study, 2g of 1% NTN 33893 granule (maximum recommended amount) were applied to the young plant (8 leaves stage) by planting hole application at the same time of transplantation (Appendix IV). Four types of experiments (No. 1, 2, 3 and 4) were provided according to the purpose of study as described below (Table I).

<u>Experiment No. 1</u>

Purpose : Balance studies of radioactivities and metabolites
Application : 2g of the ^{14}C-formulation per plant by planting hole application (20/Jul./1989)
Plant ID No. : #1, 2, 3, 4, 5, 6, 7 and 8 (#7 and 8 were control)

<u>Experiment No. 2</u>

Purpose : Identification of metabolites
Application : 2g of the non-labeled formulation per plant by planting hole application (20/Jul./1989)

Stem injections [total 0.777MBq, 0.135mg a.i. per plant by DMSO/H_2O (20/80,v/v) solution]
0.168MBq/10μl (03/Aug./1989)
0.113MBq/10μl (10/Aug./1989)
0.254MBq/20μl (17/Aug./1989)
0.242MBq/20μl (24/Aug./1989)

Plant ID No. : #9 and 10

<u>Experiment No. 3</u>

Purpose : Identification of metabolites by application of an excessive amount
Application : 10g of the ^{14}C-formulation per plant by planting hole application (20/Jul./1989)
Plant ID No. : #11 and 12

<u>Experiment No. 4</u>

Purpose : Identification of metabolites by application of an excessive amount
Application : 10g of the non-labeled formulation per plant by planting hole application (20/Jul./1989)
Plant ID No. : #13, 14, 15 and 16

3. Sampling

Sampling was done according to the purpose of the experiment (Table II).

Experiment No. 1

The aerial part of plant was sampled at 14 days (plant ID : #2) and 35 days (plant ID : #3 and 4) after the application. The fruits were sampled successively in a term of harvest (49 to 67 days) and then the aerial part of the plant was sampled at 69 days (plant ID : #5, 6, 7 and 8) after the application. The soils in planting containers were also sampled at 14, 35 and 69 days. The samples were stored in the freezer (No. 213, -20°C)

until analysis.

Experiments No. 2, 3 and 4
After sampling of the fruit in the term of harvest, the aerial part and soil were sampled at 69 days after the application. The samples were stored in the freezer (No.213, -20°C) until analysis.

4. Analysis

4-1 Preparation of samples
The aerial part of plant was separated to stem, leaves and others (flowers and flower clusters). The fruit was separated to edible part and calyx. The root was removed from soil and then the soil was mixed sufficiently by the rocking mixer for 30 min. The sample was measured fresh weight prior to the preparation.

4-2 Extraction and fractionation
4-2-1 Plant samples
The samples prepared were homogenized with 100 to 250ml of 80 % methanol (methanol/water=80/20, v/v) for 5 min by using Polytron (1g of sample per about 2.5ml of the solvent). After centrifuge separation and filtration, residue was extracted two times more by the same manner. The residue was homogenized again with 100 to 250ml of 80% acetonitrile (acetonitrile/water=80/20, v/v) for 5 min. After removing the solvents of the combined extracts, 0.1 to 0.2g of NaCl were added into the extract. The extract was partitioned two times with 100 to 200ml of n-hexane and then partitioned successively three times with 100 to 300ml of dichloromethane. The dichloromethane phase was washed two times with 50ml of water and the water layers were combined with the aqueous phase. The n-hexane Fr., dichloromethane Fr. and aqueous Fr. were obtained from the extract. A portion of aqueous Fr. (equivalent to 50g of sample) was concentrated and subjected to XAD-2 column chromatography (125ml of resin, a 2.5cm i.d. X 30cm glass column equipped with low pressure pump and UV-254nm detector). The sample solution was injected into bottom of the column and 400ml of water (XAD-2 H_2O Fr. Aq 1) and 500ml of methanol (XAD-2 methanol Fr. Aq 2) were eluted successively from the column bottom to top at a rate of 4 to 5ml/min. When necessary for identification and quantification of metabolites, the eluate of XAD-2 column chromatography was evaporated and dissolved in 10ml of water, and the samples were subjected to CM-Sepharose chromatography and/or DEAE-Sepharose column chromatography (125ml of the resin, a 2.5cm i.d. X 30cm glass column equipped with low pressure pump and UV-254nm detector). The metabolites were eluted successively with 400ml of H_2O and 500ml of ammonic methanol (25% ammonia water/methanol=1/9, v/v) in the CM-Sepharose chromatography. In case of the DEAE-Sepharose chromatography, 250ml of H_2O and 500ml methanol containing phosphoric acid (0.02% phosphoric acid in methanol) were used successively as eluent (Fig. 1).

4-2-2 Soil

After measurement of water contents of the soil, 20g of the soil (dry weight basis) was extracted with 100ml of 80 % acetonitrile (acetonitrile/water=80/20, v/v) for 30 min at room temperature by using an ultrasonic generator. After filtration, the soil was extracted again by the same manner and then the extracts were combined.

4-3 Analysis of the radioactivity

4-3-1 Soluble Radioactivity

4-3-1-1 Determination by liquid scintillation counter (LSC)

An aliquot (20 to 50μl) of each fraction was transferred into a 20ml standard polyethylene vial and 7ml of scintillation cocktail (Ready-gel) was added into the vial. After mixing of them, the sample was subjected to LSC determination.

4-3-1-2 Analysis by thin layer chromatography (TLC)

An aliquot of each concentrated fraction was spotted onto silica gel thin layer plate and developed with the solvent mixture (solvent system A, B and E) described bellow.

Solvent system A : $CH_3COOC_2H_5/CH_3CHOHCH_3/H_2O$(65/23/12, v/v)
B : $CHCl_3/CH_3OH/CH_3COOH/H_2O$(65/23/3.5/3.5, v/v)
E : $CH_3CN/CHCl_3$(50/50, v/v)

The developed plate was subjected to TLC liner analyzer and autoradiography. The reference substances were cochromatographed with the sample and/or developed after mixing with the sample. Behaviors of the reference substances on the TLC plate were summarized in Appendix V.

4-3-1-3 Analysis by high performance liquid chromatography (HPLC)

An aliquot (50μl) of each concentrated fraction was subjected to HPLC analysis. Behaviors of the reference substances in the HPLC analysis were summarized in Appendix VI.

Conditions of HPLC analysis
Column : RP-Select B and/or RP-8(e)
Eluent : 10% A (5 min.)-->100% A (gradient of 40 min)
A + B = 100 %
A = CH_3CN/H_2O(50/50, v/v) + 0.005M PIC B-8
B = H_2O + 0.005M PIC B-8
Flow rate : 1ml/min
Detector : UV and radioactivity

4-3-2 Insoluble radioactivity (non-extractable radioactivity)

After extraction, residual materials of plant and soil samples were dried up by using the freeze dryer. A portion of dried material was subjected to combustion analysis and carbon dioxide was trapped with 6ml of Carbo-sorb. After adding 12ml of Permafluor V, the solution was subjected to LSC analysis.

4-4 Identification and quantification of radioactive substances

Radioactive substances in the TLC and HPLC analysis were identified by comparison with the behavior of the reference substances. Each radioactive substance on the TLC plate was scraped away from the plate and transferred into the vial. After adding 1ml of 50% methanol (CH_3OH/H_2O=50/50, v/v), the vial was subjected to ultrasonic treatment for more than 30 min. Seven ml of Ready-gel was added into the vial and then the radioactivity was determined by LSC analysis. The metabolites obtained from the experiments No.3 and 4 (application of an excessive amount) were cleaned up and purified repeatedly by column chromatography and TLC. The isolated metabolites were subjected to MS analysis.

Conditions of MS

Ionization method	: CI(positive) and FAB(positive)
Ionization voltage	: 250eV(CI)
Reaction gas	: isobutane(CI)
Collision gas	: xenon(FAB)
1st accelerating voltage	: 3KeV(FAB)
Sample induction	: glass capillary (CI)
	: matrix (FAB)
	glycerol/thioglycerol(50/50, v/v)

RESULTS AND DISCUSSION

1. Cultivating and sampling of the test plant (Experiments No. 1, 2, 3 and 4)

The NTN 33893 granule was applied conventionally to young eggplants (8 leaves stage) by planting hole application at the same time of transplanting (20/Jul.1989). The plants were cultivated in the greenhouse until harvest time (27/Sep.1989). These samples were sampled according to the schedule described in Table II, however one of the sample (Plant ID:#1) could not be used for experiment because of root rot. The other plants except Plant ID:#1 grew up normally and no differences were observed between treated samples and controls. Sprinkling was done suitably at a rate of 500 to 1000ml/plant. Osadan® (fenbutatin oxide, 25% wettable powder, X1000) was applied leaves to control tick on 29/Aug. and 04/Sep. The temperature and humidity in the greenhouse were recorded automatically during the cultivation and these values were 23 to 30°C and 45 to 83%, respectively.

2. Absorption, translocation and distribution of applied radioactivity in the test plant (Experiment No. 1)

When ^{14}C-NTN 33893 granule was applied to a young eggplant by planting hole application, the radioactivities in the plant (foliage and fruits) were accounted for 2.72% of the applied radioactivity at 14 days, an average of 2.66% at 35 days and an average of 1.64% at 69 days after the application (Tables III, IV, V, VI and VII). The amounts of radioactivity found in the plant seemed to decrease outwardly with time. Those results show

the test plants uptake rapidly the chemicals surrounding in roots just after the application, but the rate of uptake may decrease with time. Therefore the amounts of radioactivities exhausted outside as metabolites may be greater than the amounts of radioactivities being uptaken. The NTN 33893 absorbed to the plant seemed to be translocated acropetally, and 98.8% of the radioactivity absorbed (2.69% of the applied radioactivity) was found in the leaves of plant (ID:#2) at 14 days after the application. Similar results were obtained from the plants sampled at 69 days (ID:#5 and #6). However 67.7% of the radioactivity was found in the stem of plant sampled at 35 days (ID:#3), this phenomena seemed to be a differential case depending on something about plant physiology. The fruits were harvested at 49 days (ID:#5), 53 days (ID:#6) and 67 days (ID:#6) after the application, and the radioactivity found in the fruit was accounted for 1.74% of the radioactivity found in the aerial part (0.03% of the applied radioactivity), 1.39% (0.02%) and 1.47% (0.03%), respectively. When the fruits were separated to edible part and calyx, 26.2 to 49.7% of the radioactivity was found in the calyx. The amounts of radioactivities in the edible parts were accounted for 0.01 to 0.02% of the applied radioactivity, namely, residual concentrations in NTN 33893 equivalent were calculated to be 0.032 to 0.053 mg/kg in the edible part (Table VIII).

3. Metabolism of NTN 33893 in eggplant (Experiment No. 1)

3-1 Metabolism in foliage

The samples were separated to each part (stem, leaves, edible part, calyx and others) and extracted total four times with aqueous acetonitrile and aqueous methanol. The extracts (soluble radioactivity fraction) were partitioned with successively n-hexane and dichloromethane, that is, n-hexane, dichloromethane and aqueous fractions were obtained. The aqueous fraction was fractionated furthermore by XAD-2 column chromatography, since the fraction contained too much impurities for TLC and HPLC analyses, then water eluate fraction (XAD-2:H_2O, Aq 1) and methanol eluate fraction (XAD-2:MeOH, Aq 2) were obtained (Fig. 1). In the leaves sampled at 14 days (Plant ID:#2), 98.8% of the radioactivity in aerial part was found and fractionated to n-hexane Fr. (4.5%), dichloromethane Fr. (40.3%), aqueous Fr. (48.6%) and non-extracts (5.4%) (Table. IX). About 50% of absorbed NTN 33893 was changed rapidly to water soluble substances. Each fraction was subjected to TLC and HPLC analyses. NTN 33893, WAK 4103, NTN 35884, NTN 33519, WAK 3839 and NTN 38014 were found in the dichloromethane Fr. of leaves extract. These substances were identified by TLC analyses with the reference substances (Fig. 2). NTN 33893 was accounted for 32.2 % in the fraction. In aqueous fraction of leaves extracts, NTN 33893, NTN 38014, WAK 4103, NTN 35884, WAK 3839, RBN 1114 and CNA were found by TLC and HPLC analyses. The major metabolites in aqueous fraction were NTN 38014 (20.1%), WAK 4103 (7.1%) and RBN 1114 (4.2%). In the samples at 35 days (ID:#3 and #4), 52.0 to 65.0% of the radioactivity was found in the aqueous fraction (Table X and XI). NTN 33893 was accounted for 8.6 to

9.0%. NTN 38014 (30.7 to 37.0%) and RBN 1114 (4.5 to 5.6%) were found as major metabolites (Fig. 4, 5, 6, 7, 8 and 9). In the samples at 69 days (ID:#5 and #6), NTN 33893 was accounted for 8.8 to 11.3% and major metabolites were NTN 38014 (23.7 to 25.6%) and RBN 1114 (5.2 to 6.1%)(Tables XII and XIII, Fig. 10, 11 and 12). The residual concentrations of NTN 33893 and its major metabolites (NTN 38014, WAK 4103 and RBN 1114) in the foliage were calculated to be an average of 0.15mg/kg, 0.29mg/kg, 0.05mg/kg and 0.10mg/kg, respectively (Table XIV). The results show NTN 33893 translocated to foliage was changed to polar metabolites after elimination of nitro moiety, hydroxylation of imidazolidine ring and conjugation. Non-extractable radioactivities were accounted for 5.5 to 9.8% in foliage and unknown metabolites accounting for greater than 10% of the radioactivity were not present.

3-2 Metabolism of NTN 33893 in fruit (edible part)

The fruits were sampled at 49 days (ID:#5), 53 days (ID:#6) and 67 days (ID:#5), then were separated to edible part and calyx. In analysis of the edible parts, the radioactivities were accounted for 18.0 to 24.9% in dichloromethane Fr., 69.7 to 74.7% in aqueous Fr. and 5.4 to 7.3% in non-extracts. The distribution of radioactivities in fractions and metabolites found were similar to the results obtained from analysis of foliage (Tables XV, XVI and XVII). Unchanged parent compound was accounted for 15.9 to 25.0% of the radioactivity found in the edible part and was a major part of radioactivity in dichloromethane Fr. (Fig. 13). Small amounts (0.3 to 3.4%) of WAK 4103, NTN 35884, WAK 3839, CNA and NTN 38014 were also found in the fraction. When the aqueous Fr. was subjected to XAD-2 column chromatography, the radioactivities were fractionated to 4.0 to 26.0% in the water eluate Fr. (Aq 1) and 48.7 to 69.0% in the methanol eluate Fr. (Aq 2). In the fruit (ID:#5 49 DAT), 26.0% of the radioactivity was found in the water eluate Fr.(Aq 1). This phenomenon was not observed in the fractions of foliage analysis. When the water eluate Fr. (Aq 1) was subjected to DEAE-Sepharose column chromatography (anion exchange), 21.9% of the radioactivity was eluted in acidic methanol Fr. (Aq 1-2). The major metabolite in the fraction was identified to be CNA (19.5%) by TLC and HPLC analyses (Fig. 14). When the methanol eluate Fr. (48.7% of the radioactivity found in the edible part, Aq 2) of XAD-2 column chromatography was subjected to CM-Sepharose column chromatography (cation exchange), the radioactivity was fractionated to 21.2% in water eluate Fr. (Aq 2-1) and 27.5 % in ammonic methanol Fr.(Aq 2-2). NTN 38014 was accounted for 23.3% in the ammonic methanol Fr. by TLC and HPLC analyses (Fig. 15). When the water eluate Fr. (Aq 2-1) of CM-Sepharose chromatography was subjected again to DEAE-Sepharose column chromatography, the radioactivity was fractionated to 8.8% in water eluate Fr. (Aq 2-1-1) and 12.4% in acidic methanol Fr. (Aq 2-1-2). NTN 38014 (2.5%) and WAK 4103 (0.2%) were found in the water eluate Fr. (Aq 2-1-1) and CNA (6.5%) and NTN 33893 (0.4%) were found in the acidic methanol Fr. (Aq 2-1-2) by TLC and HPLC analyses (Fig. 16 and 17). These results were

summarized that NTN 38014, CNA and NTN 33893 were found in the edible part at 49 days, and were accounted for 26.5%, 26.4% and 15.9% of the radioactivity found in the part, respectively. However CNA was accounted for only 4.4 to 7.6% in the edible parts sampled at 53 days and 67 days (ID:#5 and #6), RBN 1114 that is one of a major metabolite in foliage was accounted for 17.9 to 26.7% in the parts (Tables XVI and XVII, Fig. 18). Since similar metabolites were found both in foliage and edible part, these metabolites found in edible part seemed to be produced in foliage and then translocated to edible part. The results also suggested metabolic systems of hydroxylation and oxidation that may change RBN 1114 to CNA via PAC as intermediate exist in edible part. The concentrations of radioactivities in NTN 33893 equivalent were accounted for 0.0315 to 0.0534mg/kg in the edible part and 0.0020 to 0.0036mg/kg in non-extracts of the part (Table XVIII). The concentrations of NTN 33893 were 0.0058 to 0.0134mg/kg in the edible part. NTN 38014, RBN 1114 and CNA were found as major metabolites and were accounted for 0.0018 to 0.0107mg/kg (conversion factor:210/255), <0.0005 to 0.0171mg/kg (conversion factor:305/255) and 0.0009 to 0.0080mg/kg (conversion factor: 157/255), respectively. Small amounts of WAK 4103, NTN 35884 and WAK 3839 were also found and were accounted for <0.0005 to 0.0027mg/kg (conversion factor: 271/255), <0.0005mg/kg (conversion factor:253/255) and <0.0005 mg/kg (conversion factor :239/255), respectively. Unknown metabolites accounted for greater than 10% of the found radioactivity were not present in the edible part.

4. Isolation and identification of metabolites in eggplant (Experiments No. 2, 3 and 4)

The samples treated with excessive amounts of NTN 33893 by planting hole application and/or stem injection were extracted and fractionated in the experiments 2, 3 and 4. Each fraction was subjected to MS analysis following purification and isolation by using TLC and column chromatography.

4-1 Experiment of stem injection (Experiment No. 2)

The aerial parts (ID:#9 and #10, planting hole application of excessive amounts of non-labeled granule and four times of stem injections of ^{14}C-NTN 33893) were sampled at 69 days and then extracted and fractionated by the same manner. The total radioactivities detected were 0.513MBq in ID:#9 and 0.460MBq in ID:#10, namely, these radioactivities corresponded to 66.0% and 59.2% of the injected radioactivity, respectively. NTN 33893, WAK 4103, NTN 35884, NTN 33519, WAK 3839, NTN 38014 and RBN 1114 were found in dichloromethane and aqueous fractions by TLC analysis (Fig. 19). The patterns of chromatograms were almost same as the results obtained from the experiment No. 1, therefore NTN 33893 applied by soil application itself may be absorbed directly from the roots and degraded in the plant, that is, it may be little chance to occur a phenomenon that metabolites produced in soil is absorbed and found in the plant in the experiment No. 1. The radioactive compounds found in the

aerial parts were 80.1 to 89.4μg in NTN 33893 equivalent. These radioactivities were not enough to isolate and identify each metabolite.

4-2 Experiments of an excessive amount application (Experiments No. 3 and 4)

The extracts of aerial part sampled at 69 days (ID:#11 to #16) were combined and fractionated by the same manner. A portion of the dichloromethane fraction was subjected to TLC analysis (Fig. 20). The remaining of the fraction was subjected to silica gel column chromatography (Wako gel C200, 50g) and eluted with a series of 200ml of ethyl acetate/n-hexane (20/80, v/v), 100ml of ethyl acetate/n-hexane (50/50, v/v), 100ml of ethyl acetate, 200ml of methanol/ethyl acetate (20/80, v/v), 100ml of methanol/ethyl acetate (50/50, v/v), 200ml of methanol and 200ml of acetic acid/methanol (5/95, v/v). In fractions of Fr.-5 and Fr.-6 (methanol/ethyl acetate, 20/80, 100ml), 85.9% of the radioactivities subjected was found (Fig. 21 and 22). WAK 3772, PAC, WAK 3839, and WAK 4103 were purified and isolated in the fractions by TLC analysis. WAK 3772 was identified by TLC analysis with reference substance (Fig. 23). PAC was identified by TLC and HPLC analyses with reference substance (Fig.24). WAK 3839 was identified by TLC, HPLC and MS analyses with reference substance (Fig.25, 26 and 27). WAK 4103 was identified by TLC and HPLC analyses with reference substance (Fig. 28).

The radioactivities in aqueous fraction were purified successively with XAD-2 column chromatography and CM-sepharose column chromatography, and then were subjected repeatedly to TLC analysis. WAK 4103, NTN 35884, NTN 38014 and RBN 1114 were found in the aqueous fraction. WAK 4103 was identified by TLC, HPLC and MS analyses (Fig. 29, 30 and 31). NTN 35884, NTN 38014 and RBN 1114 were also identified by the same manner (Fig. 32, 33, 34, 35, 36, 37 and 38).

5. The radioactivities in soil (Experiment No. 1)

The soil was extracted two times with 80% aqueous acetonitrile (acetonitrile/H_2O=80/20, v/v) by using an ultrasonic generator after mixing the soil sampled. The radioactivities were accounted for 1.666mg/kg in NTN 33893 equivalent at 14 days, 1.431 mg/kg and 1.601mg/kg at 69 days after the application. When the extracts were subjected to TLC and HPLC analyses, 62.1 to 81.7% of the radioactivity was found as the unchanged parent compound (Tables XIX and XX, Fig. 39, 40 and 41). WAK 4103, WAK 3839 and CNA were also found as metabolites, but no metabolites accounted for greater than 2% of the found radioactivity were present. The concentration of NTN 33893 was accounted for 1.361mg/kg at 14 days and decreased an average of 0.991mg/kg at 69 days. Since the radioactivities in non-extracts increased with time, a portion of NTN 33893 seemed to be changed to bound residues.

CONCLUSION

The absorption, translocation and metabolism of NTN 33893 in eggplant were investigated after 2g of 1% NTN 33893 granule was applied conventionally to a young eggplant by planting hole application. The radioactivities were accounted for 2.72% (ID:#2) of the applied radioactivity in aerial parts (leaves and stem) at 14 days, 1.79% (ID:#5) and 1.48%(ID:#6) in aerial parts (leaves, stem and fruits) at 69 days (Tables III, VI and VII). Since a greater than 88% of found radioactivities was located in leaves, the radioactivity absorbed seemed to be moved acropetally to leaves. In the foliage (ID:#5 and #6) sampled at 69 days, unchanged NTN 33893, NTN 38014, RBN 1114, WAK 4103, NTN 35884, WAK 3839, NTN 33519 and CNA were found and then were accounted for an average of 10.2% of the radioactivity found in the foliage, 24.6%, 5.6%, 3.6%, 1.3%, 0.3%, 0.1% and 0.9%, respectively. The radioactivity in the non-extract was 9.3% (Table XIV). No other radioactive substances accounted for greater than 10% of the found radioactivity were present in the aerial part except unchanged parent compound and metabolites identified. These results suggested the major metabolites in foliage were NTN 38014, RBN 1114 and WAK 4103.

The fruits were sampled at 49, 53 and 67 days and then were separated to edible part and calyx prior to analysis. The radioactivities were accounted for 0.032 to 0.053mg/kg in NTN 33893 equivalent in the edible parts. The radioactivities corresponded to 0.01 to 0.02% of the applied radioactivity. In calyx, the concentrations of radioactivity were 0.104 to 0.483 mg/Kg. NTN 33893, NTN 38014, RBN 1114, CNA, WAK 4103, NTN 35884 and WAK 3839 were found in the edible parts and were accounted for an average of 0.0081mg/kg (18.9% of the radioactivities found), 0.0049mg/kg (14.0%), 0.0066mg/kg (13.0%), 0.0035mg/kg (13.4%), 0.0015mg/kg (3.2%), <0.0005mg/kg (0.2%) and <0.0005mg/kg (0.1%), respectively. The radioactivity of non-extract in the edible part was an average of 0.0028mg/kg in NTN 33893 equivalent. No radioactive substances accounted for greater than 10 % of the found radioactivities were present in the edible parts except parent compound and metabolites identified. These results showed the major metabolites in edible part of eggplant were NTN 38014, RBN 1114 and CNA.

When the samples applied with ^{14}C-NTN 33893 (DMSO/H_2O solution) by stem injections were analyzed, the metabolites found were almost same metabolites obtained from the samples applied by planting hole application. These results showed the identified metabolites in the samples applied by planting hole application were produced in eggplant after absorbing NTN 33893 itself from the roots.

PAC, WAK 3772, WAK 4103, NTN 35884, NTN 38014, WAK 3839 and RBN 1114 were isolated and identified in the aerial parts of samples applied with excessive amounts of ^{14}C-NTN 33893 by planting hole application. These results obtained from the experiments using excessive amounts and recommended amounts of NTN 33893 showed major metabolic pathways of NTN 33893 in eggplant were :

1. Elimination of nitro moiety

2. Hydroxylation of imidazolidine ring
3. Cleavage of C-N bond between pyridylmethyl moiety and imidazolidine ring.

The proposed metabolic pathways of NTN 33893 in eggplant were showed in Fig. 42.

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11. Sample processing (SOP M 5105)
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15. Identification of metabolites (SOP M 0330)
16. Procedures for archival storage (SOP G 5003)

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Table I Application amounts of the NTN 33893 to the plant by planting hole application

Plant ID	Exper. No.	^{14}C-Granule(mg)[1]	^{12}C-Granule(mg)[2]	a.i.(mg)	Radioactiv.(MBq)
# 1	1	392.4	1603.8	18.79	5.973
# 2	1	397.4	1602.4	18.96	6.049
# 3	1	395.4	1602.4	18.94	6.018
# 4	1	410.6	1603.4	19.09	6.230
# 5	1	1996.8	0	18.77	30.392
# 6	1	1995.9	0	18.76	30.379
# 7	1(Cont.)	0	0	0	0
# 8	1(Cont.)	0	0	0	0
# 9*	2	0	2003.0	19.03	0
# 10*	2	0	2003.3	19.03	0
# 11	3	1995.2	8004.1	94.79	30.368
# 12	3	2015.3	7999.7	94.94	30.674
# 13	4	0	10002.4	95.02	0
# 14	4	0	10001.3	95.01	0
# 15	4	0	10011.0	95.10	0
# 16	4	0	9994.3	94.95	0

1) 0.94 % a.i. ^{14}C-NTN 33893 granule
2) 0.95 % a.i. ^{12}C-NTN 33893 granule
* A total amount of 0.777 MBq (0.135 mg a.i.) was additionally injected into the stem at 14, 21, 28 and 35 days after the planting hole application

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Table II Sampling date of the plant

Plant ID	Sampling date	DAT[1)]
# 1	Not used in the experiment due to root rot	
# 2	03/Aug./1989 (aerial part and soil)	14
# 3	24/Aug./1989 (aerial part and soil)	35
# 4	24/Aug./1989 (aerial part and soil)	35
# 5	07/Sep./1989 (fruit)	49
	25/Sep./1989 (fruit)	67
	27/Sep./1989 (aerial part and soil)	69
# 6	11/Sep./1989 (fruit)	53
	27/Sep./1989 (aerial part and soil)	69
# 7	27/Sep./1989 (aerial part and soil)	69
# 8	13/Sep./1989 (fruit)	55
	27/Sep./1989 (aerial part and soil)	69
# 9	27/Sep./1989 (aerial part and soil)	69
# 10	27/Sep./1989 (aerial part and soil)	69
# 11	27/Sep./1989 (aerial part and soil)	69
# 12	07/Sep./1989 (fruit)	49
	11/Sep./1989 (fruit)	53
	13/Sep./1989 (fruit)	55
	27/Sep./1989 (aerial part and soil)	69
# 13	27/Sep./1989 (aerial part and soil)	69
# 14	27/Sep./1989 (aerial part and soil)	69
# 15	27/Sep./1989 (aerial part and soil)	69
# 16	27/Sep./1989 (aerial part and soil)	69

1) The days after treatment

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Table III The balance of radioactivity in the plant and soil (Plant ID : # 2, DAT : 14)

Sample	Weight (g)	Radioactiv.(MBq)	a.i.(μg)[1)]	mg a.i./kg[1)]	Distribution (%) of radioactiv.	
Stem	31.719	1.925×10^{-3}	6.03	0.190	1.17	(0.03)
Leaves	55.971	1.630×10^{-1}	510.31	9.117	98.83	(2.69)
					subtotal 100	(2.72)
Soil	8905[2)]	4.738	14836.03	1.666		(78.25)
		total 15352.37			total	(80.97)

The values in parenthesis are calculated with the applied radioactivity
1) Equivalent to the NTN 33893
2) Dry weight

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Table IV The balance of radioactivity in the plant and soil (Plant ID : # 3, DAT : 35)

Sample	Weight (g)	Radioactiv.(MBq)	a.i.(μg)[1]	mg a.i./kg[1]	Distribution (%) of radioactiv.	
Stem	65.706	1.144×10^{-1}	359.67	5.474	67.68	(1.90)
Leaves	76.216	5.291×10^{-2}	166.37	2.183	31.31	(0.88)
Others[2]	4.735	1.714×10^{-3}	5.39	1.139	1.01	(0.03)
					subtotal 100	(2.81)
Soil	8914[3]	4.290	13491.43	1.514		(71.29)
					total	(74.10)
			total 14022.86			

The values in parenthesis are calculated with the applied radioactivity
1) Equivalent to the NTN 33893
2) Flowers and flower clusters
3) Dry weight

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Table V The balance of radioactivity in the plant and soil (Plant ID : # 4, DAT : 35)

Sample	Weight (g)	Radioactiv.(MBq)	a.i.(μg)[1]	mg a.i./kg[1]	Distribution (%) of radioactiv.	
Stem	63.075	4.001×10^{-3}	12.22	0.194	2.55	(0.06)
Leaves	70.740	1.517×10^{-1}	463.23	6.548	96.75	(2.43)
Others[2]	10.433	1.098×10^{-3}	3.35	0.322	0.70	(0.02)
					subtotal 100	(2.51)
Soil	9036[3]	4.716	14403.96	1.594		(75.70)
		total	14882.76		total	(78.21)

The values in parenthesis are calculated with the applied radioactivity
1) Equivalent to the NTN 33893
2) Flowers and flower clusters
3) Dry weight

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Table VI The balance of radioactivity in the plant and soil (Plant ID : # 5, DAT : 69)

Sample	Weight (g)	Radioactiv. (MBq)	a.i. (μg)[1]	mg a.i./kg[1]	Distribution (%) of radioactiv.	
Stem (DAT:69)	111.955	3.141×10^{-2}	19.40	0.173	5.75	(0.10)
Leaves (DAT:69)	110.459	4.808×10^{-1}	296.96	2.688	87.99	(1.58)
Fruit (DAT:49)	97.511	9.511×10^{-3}	5.87	0.060	1.74	(0.03)
Edible part			[4.13]	[0.049]	[1.22]	
Calyx			[1.74]	[0.129]	[0.52]	
Fruit (DAT:67)	51.700	8.009×10^{-3}	4.95	0.096	1.47	(0.03)
Edible part			[2.49]	[0.053]	[0.74]	
Calyx			[2.46]	[0.483]	[0.73]	
Others[2] (DAT:69)	14.302	1.670×10^{-2}	10.32	0.721	3.05	(0.05)
					subtotal 100	(1.79)
Soil (DAT:69)	9645[3]	2.500×10	15438.46	1.601		(82.26)
		total	15775.96		total	(84.05)

The values in parenthesis are calculated with the applied radioactivity
1) Equivalent to the NTN 33893 2) Flowers and immature fruits 3) Dry weight

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Table VII The balance of radioactivity in the plant and soil (Plant ID : # 6, DAT : 69)

Sample	Weight (g)	Radioactiv.(MBq)	a.i.(μg)[1)]	mg a.i./kg[1)]	Distribution (%) of radioactiv.	
Stem (DAT:69)	96.543	2.051 X 10^{-2}	12.67	0.131	4.55	(0.07)
Leaves (DAT:69)	80.750	4.141 X 10^{-1}	255.75	3.167	91.79	(1.36)
Fruit (DAT:53)	100.815	6.292 X 10^{-3}	3.89	0.039	1.39	(0.02)
Edible part			[2.87]	[0.032]	[1.02]	
Calyx			[1.02]	[0.104]	[0.37]	
Others[2)] (DAT:69)	8.290	1.022 X 10^{-2}	6.31	0.762	2.27	(0.03)
					subtotal 100	(1.48)
Soil (DAT:69)	9523[3)]	2.207 X 10	13627.93	1.431		(72.65)
					total	(74.13)
		total 13906.55				

The values in parenthesis are calculated with the applied radioactivity
1) Equivalent to the NTN 33893
2) Flowers and immature fruits
3) Dry weight

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Table VIII Residual amounts of radioactivities in the plant and soil

Sample	Residual amount (mg a.i./kg)[1]				
	ID:# 2, DAT:14	ID:# 3, DAT:35	ID:# 4, DAT:35	ID:# 5	ID:# 6
Stem	0.190	5.474	0.194	0.173 (DAT:69)	0.131 (DAT:69)
Leaves	9.117	2.183	6.548	2.688 (DAT:69)	3.167 (DAT:69)
Fruit [Edible part] {Calyx}				0.060 [0.049] {0.129} (DAT:49)	0.039 [0.032] {0.104} (DAT:53)
				0.096 [0.053] {0.483} (DAT:67)	
Others		1.139	0.322	0.721 (DAT:69)	0.762 (DAT:69)
Soil	1.666	1.514	1.594	1.601 (DAT:69)	1.431 (DAT:69)

1) Equivalent to the NTN 33893

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Table IX The distribution of radioactivities and metabolites in the plant (Plant ID : # 2, DAT : 14)

Sample	Radioactivities (%)			
	Fraction		Substances	
Stem	n-Hexane	0		
	CH_2Cl_2	0.544		
	Aqueous	0.542		
	Residue	0.081		
	subtotal	1.167		
Leaves	n-Hexane	4.497		
	CH_2Cl_2	40.338	NTN 33893	32.161
			NTN 38014	1.291
			WAK 4103	1.686
			NTN 35884	0.726
			NTN 33519	0.161
			WAK 3839	1.452
	subtotal	37.477		
	Aqueous	48.568		
	(Aq 1)	(0)		
	(Aq 2)	(48.568)	NTN 33893	0.461
			NTN 38014	20.102
			WAK 4103	7.096
			NTN 35884	0.811
			WAK 3839	0.398
			RBN 1114	4.245
			CNA	1.219
	subtotal	34.332		
	Residue	5.430		
	subtotal	98.833		
	total	100	total	71.809

Aq 1 : H_2O fraction of XAD-2 C.C.
Aq 2 : CH_3OH fraction of XAD-2 C.C.

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Table X The distribution of radioactivities and metabolites in the plant (Plant ID : # 3, DAT : 35)

Sample	Radioactivities (%)			
	Fraction		Substances	
Stem	n-Hexane	0.333		
	CH_2Cl_2	32.392	NTN 33893	7.006
			NTN 38014	16.831
			WAK 4103	0.444
			NTN 35884	0.667
			NTN 33519	0.168
			WAK 3839	0.845
			subtotal	25.961
	Aqueous	34.619		
	(Aq 1)	(0.826)		
	(Aq 2)	(33.793)	NTN 38014	12.564
			RBN 1114	3.261
			WAK 4103	1.203
			subtotal	17.028
	Residue	0.335		
	subtotal	67.679		
Leaves	n-Hexane	0.757		
	CH_2Cl_2	4.801	NTN 33893	1.562
			NTN 38014	1.998
			WAK 4103	0.113
			NTN 35884	0.153
			NTN 33519	0.158
			WAK 3839	0.098
			subtotal	4.082
	Aqueous	17.424		
	(Aq 1)	(0.633)		
	(Aq 2)	(16.791)	NTN 38014	5.605
			WAK 4103	0.349
			RBN 1114	1.229
			subtotal	7.183
	Residues	8.325		
	subtotal	31.307		
Others	n-Hexane	0		
	CH_2Cl_2	0.060		
	Aqueous	0.940		
	Residue	0.014		
	subtotal	1.014		
	total	100	total	54.254

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Table XI The distribution of radioactivities and metabolites in the plant (Plant ID : # 4, DAT : 35)

Sample	Radioactivities (%)			
	Fraction		Substances	
Stem	n-Hexane	0		
	CH_2Cl_2	0.618		
	Aqueous	1.704		
	Residue	0.231		
	subtotal	2.553		
Leaves	n-Hexane	3.359		
	CH_2Cl_2	21.572		
			NTN 33893	8.950
			NTN 38014	5.956
			WAK 4103	1.752
			NTN 35884	0.671
			NTN 33519	0.386
			WAK 3839	0.738
			subtotal	18.453
	Aqueous	63.320		
	(Aq 1)	(1.603)		
	(Aq 2)	(61.717)		
			NTN 38014	24.792
			WAK 4103	0.944
			NTN 35884	0.531
			RBN 1114	5.555
			CNA	1.000
			subtotal	31.822
	Residue	8.495		
	subtotal	96.746		
Others	n-Hexane	0		
	CH_2Cl_2	0.231		
	Aqueous	0.439		
	Residue	0.031		
	subtotal	0.701		
	total	100	total	50.275

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Table XII The distribution of radioactivities and metabolites in the plant (Plant ID : # 5, DAT : 69)

Sample	Radioactivities (%)			
	Fraction		Substances	
Stem	n-Hexane	0		
	CH_2Cl_2	1.255		
			NTN 33893	1.134
	Aqueous	3.826		
	Residue	0.857		
	subtotal	5.938		
Leaves	n-Hexane	2.172		
	CH_2Cl_2	16.746		
			NTN 33893	9.366
			NTN 38014	3.234
			WAK 4103	0.474
			NTN 35884	0.899
			NTN 33519	0.137
			WAK 3839	0.303
	subtotal	14.413		
	Aqueous	63.000		
	(Aq 1)	(1.327)		
	(Aq 2)	(61.673)		
			NTN 33893	0.833
			NTN 38014	20.512
			WAK 4103	3.707
			NTN 35884	0.506
			RBN 1114	5.236
			CNA	0.882
	subtotal	31.676		
	Residue	8.986		
	subtotal	90.904		
Others	n-Hexane	0		
	CH_2Cl_2	1.449		
	Aqueous	1.492		
	Residue	0.217		
	subtotal	3.158		
	total	100	total	47.223

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Table XIII The distribution of radioactivities and metabolites in the plant (Plant ID : # 6, DAT : 69)

Sample	Radioactivities (%)			
	Fraction		Substances	
Stem	n-Hexane	0		
	CH_2Cl_2	1.520		
			NTN 33893	1.398
	Aqueous	2.428		
	Residue	0.663		
	subtotal	4.611		
Leaves	n-Hexane	1.792		
	CH_2Cl_2	10.852		
			NTN 33893	6.610
			NTN 38014	1.409
			WAK 4103	0.355
			NTN 35884	0.583
			NTN 33519	0.086
			WAK 3839	0.188
			subtotal	9.231
	Aqueous	72.923		
	(Aq 1)	(1.622)		
	(Aq 2)	(71.301)		
			NTN 33893	0.806
			NTN 38014	24.192
			WAK 4103	2.460
			NTN 35884	0.613
			RBN 1114	6.139
			CNA	0.891
			subtotal	35.101
	Residue	7.524		
	subtotal	93.091		
Others	n-Hexane	0		
	CH_2Cl_2	1.107		
	Aqueous	1.033		
	Residue	0.158		
	subtotal	2.298		
	total	100	total	45.730

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Table XIV The residual amounts of NTN 33893 and its metabolites in the aerial parts (stem, leaves and others)

Radioactive. and substances	Residual amount (mg/kg)		
	ID : # 2 (DAT 14)	Mean of ID : # 3 and 4 (DAT 35)	Mean of ID : # 5 and 6 (DAT 69)
Total radioactiv.[1)]	5.8882	3.4727	1.4241
Residue[1)]	0.3245 (5.511)	0.3026 (8.715)	0.1325 (9.306)
NTN 33893	1.9208 (32.622)	0.3041 (8.757)	0.1456 (10.226)
NTN 38014	1.0374 (21.393)	0.9695 (33.899)	0.2880 (24.561)
WAK 4103	0.5495 (8.782)	0.0886 (2.400)	0.0542 (3.581)
NTN 35884	0.0898 (1.537)	0.0348 (1.009)	0.0186 (1.313)
NTN 33519	0.0078 (0.161)	0.0102 (0.356)	0.0014 (0.115)
WAK 3839	0.1021 (1.850)	0.0274 (0.841)	0.0034 (0.252)
RBN 1114	0.2990 (4.245)	0.2084 (5.018)	0.0960 (5.633)
CNA	0.0442 (1.219)	0.0106 (0.496)	0.0078 (0.886)

The values in parenthesis are radioactivities found (%) in the aerial parts.
1) Equivalent to the NTN 33893

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Table XV The distribution of radioactivities and metabolites in the fruit (Plant ID : # 5, DAT : 49)

Sample	Radioactivities (%) Fraction		Substances	
Edible part	n-Hexane	0		
	CH_2Cl_2	17.984		
			NTN 33893	15.505
			NTN 38014	0.749
			WAK 4103	0.273
			NTN 35884	0.452
			WAK 3839	0.246
			CNA	0.369
			subtotal	17.594
	Aqueous	74.746		
	XAD:H_2O (Aq 1)	(26.002)		
			CNA	19.548
	XAD:CH_3OH (Aq 2)	(48.744)		
			NTN 33893	0.376
			NTN 38014	25.734
			WAK 4103	0.233
			CNA	6.473
			subtotal	52.364
	Residue	7.270		
	total	100	total	69.958
Calyx	n-Hexane	0		
	CH_2Cl_2	29.297		
	Aqueous	67.401		
	Residue	3.302		
	total	100		

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Table XVI The distribution of radioactivities and metabolites in the fruit (Plant ID : # 5, DAT : 67)

Sample	Radioactivities (%) Fraction		Substances	
Edible part	n-Hexane	0		
	CH_2Cl_2	24.901		
			NTN 33893	23.858
			WAK 4103	0.359
			subtotal	24.217
	Aqueous	69.654		
	XAD:H_2O (Aq 1)	(5.579)		
	XAD:CH_3OH (Aq 2)	(64.075)		
			NTN 33893	1.179
			NTN 38014	4.870
			WAK 4103	4.450
			RBN 1114	26.713
			CNA	7.574
			subtotal	44.786
	Residue	5.445		
	total	100	total	69.003
Calyx	n-Hexane	0		
	CH_2Cl_2	47.817		
	Aqueous	46.722		
	Residue	5.461		
	total	100		

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Table XVII The distribution of radioactivities and metabolites in the fruit (Plant ID : # 6, DAT : 53)

Sample	Radioactivities (%)		
	Fraction		Substances
Edible part	n-Hexane	0	
	CH_2Cl_2	20.793	
			NTN 33893 17.104
			WAK 4103 3.373
			subtotal 20.477
Aqueous	72.973		
	XAD:H_2O (Aq 1)	(3.977)	
	XAD:CH_3OH (Aq 2)	(68.996)	
			NTN 33893 1.380
			NTN 38014 7.079
			WAK 4103 1.504
			RBN 1114 17.939
			CNA 4.423
			subtotal 32.325
Residue	6.234		
total	100		total 52.802
Calyx	n-Hexane	0	
	CH_2Cl_2	40.338	
	Aqueous	54.200	
	Residue	5.462	
	total	100	

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Table XVIII The residual amounts of NTN 33893 and its metabolites in the edible part (Plant ID : # 5 and 6)

Radioactiv. and substances	Residual amount (mg/kg)			
	Edible part (84.012 g) (# 5, DAT 49)	Edible part (46.610 g) (# 5, DAT 67)	Edible part (91.013 g) (# 6, DAT 53)	Mean[1] (# 5 and # 6)
Total radioactiv.[2]	0.0492	0.0534	0.0315	0.0428
Residue[2]	0.0036 (7.270)	0.0029 (5.445)	0.0020 (6.234)	0.0028 (6.461)
NTN 33893	0.0078 (15.881)	0.0134 (25.037)	0.0058 (18.484)	0.0081 (18.875)
NTN 38014	0.0107 (26.483)	0.0021 (4.870)	0.0018 (7.079)	0.0049 (13.970)
WAK 4103	<0.0005 (0.506)	0.0027 (4.809)	0.0016 (4.877)	0.0015 (3.206)
NTN 35884	<0.0005 (0.452)	<0.0005 (n.d)	<0.0005 (n.d)	<0.0005 (0.171)
NTN 33519	<0.0005 (n.d)	<0.0005 (n.d)	<0.0005 (n.d)	<0.0005 (n.d)
WAK 3839	<0.0005 (0.246)	<0.0005 (n.d)	<0.0005 (n.d)	<0.0005 (0.093)
RBN 1114	<0.0005 (n.d)	0.0171 (26.713)	0.0068 (17.939)	0.0066 (12.984)
CNA	0.0080 (26.390)	0.0025 (7.574)	0.0009 (4.423)	0.0035 (13.412)

The values in parenthesis are radioactivities found (%) in the edible part. n.d : not detected
1) Average of the edible part (# 5 and # 6)
2) Equivalent to the NTN 33893

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Table XIX The distribution of radioactivities and metabolites in the soil

Sample	Radioactivities (%)			
	Fraction		Substances	
# 2, DAT:14	Extracts	84.3		
			NTN 33893	81.72
	Residue	15.7		
# 3, DAT:35	Extracts	75.4		
			NTN 33893	70.50
			WAK 4103	1.44
			WAK 3839	0.98
			CNA	0.65
			total	73.57
	Residue	24.6		
# 4, DAT:35	Extracts	72.0		
			NTN 33893	67.37
			WAK 4103	0.94
			WAK 3839	0.86
			CNA	0.64
			total	69.81
	Residue	28.0		
# 5, DAT:69	Extracts	67.2		
			NTN 33893	62.13
			WAK 4103	0.96
			WAK 3839	0.89
			CNA	0.59
			total	64.57
	Residue	32.8		
# 6, DAT:69	Extracts	74.1		
			NTN 33893	68.87
			WAK 4103	0.93
			WAK 3839	0.51
			CNA	0.41
			total	70.72
	Residue	25.9		

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Table XX The residual amounts of NTN 33893 and its metabolites in the soils

Radioactiv. and Substances	Residual amount (mg/kg) # 2, DAT:14	# 3, DAT:35	# 4, DAT:35	# 5, DAT:69	# 6, DAT:69
Total radioactiv.[1)]	1.666	1.514	1.594	1.601	1.431
Residue[1)]	0.262	0.372	0.446	0.525	0.371
NTN 33893	1.361	1.067	1.074	0.995	0.986
WAK 4103	<0.005	0.023	0.016	0.016	0.014
WAK 3839	<0.005	0.014	0.013	0.013	0.007
CNA	<0.005	0.006	0.006	0.006	<0.005

1) Equivalent to the NTN 33893

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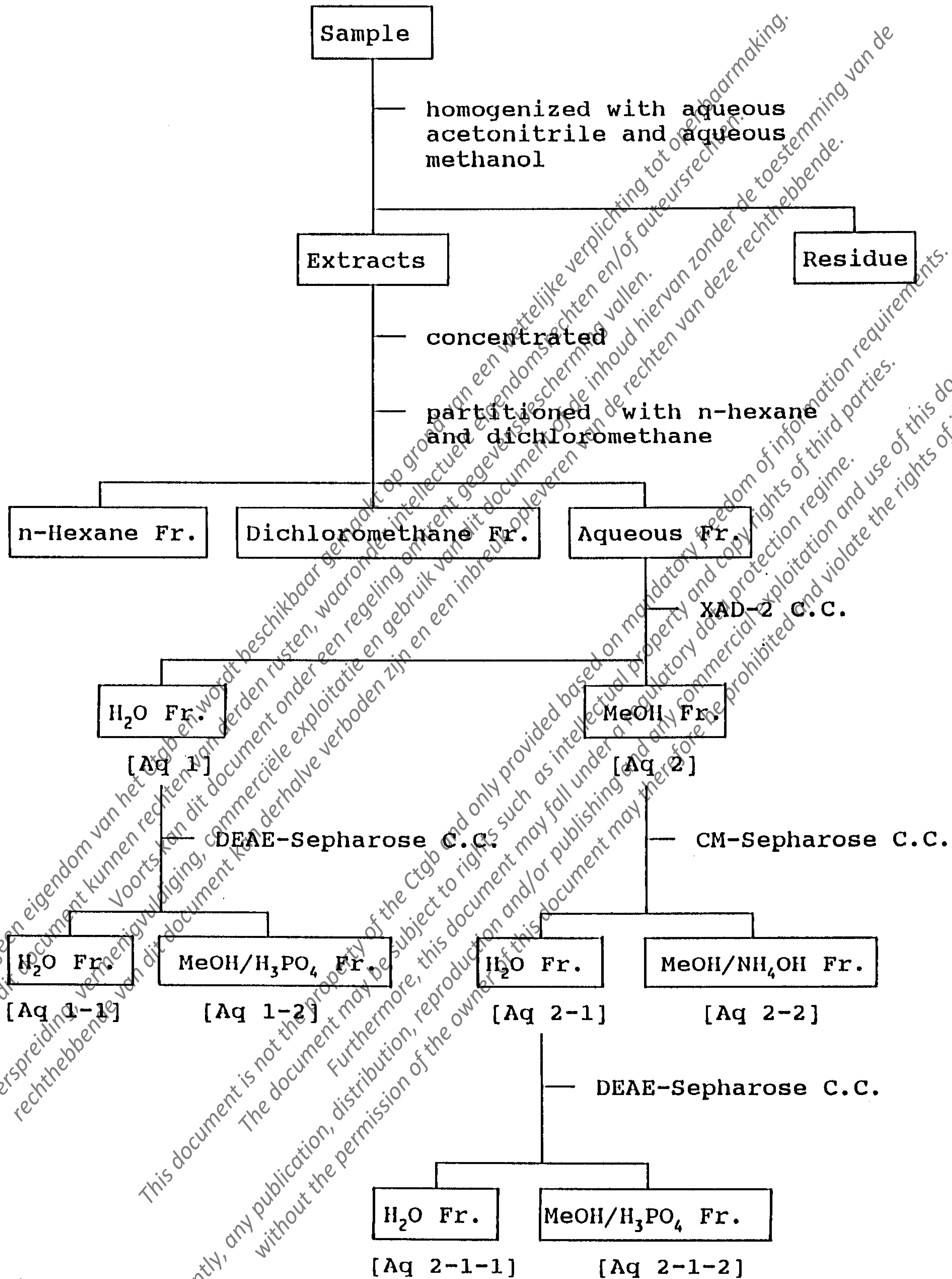

Fig. 1 Fundamental method of extraction and fractionation of the plant samples

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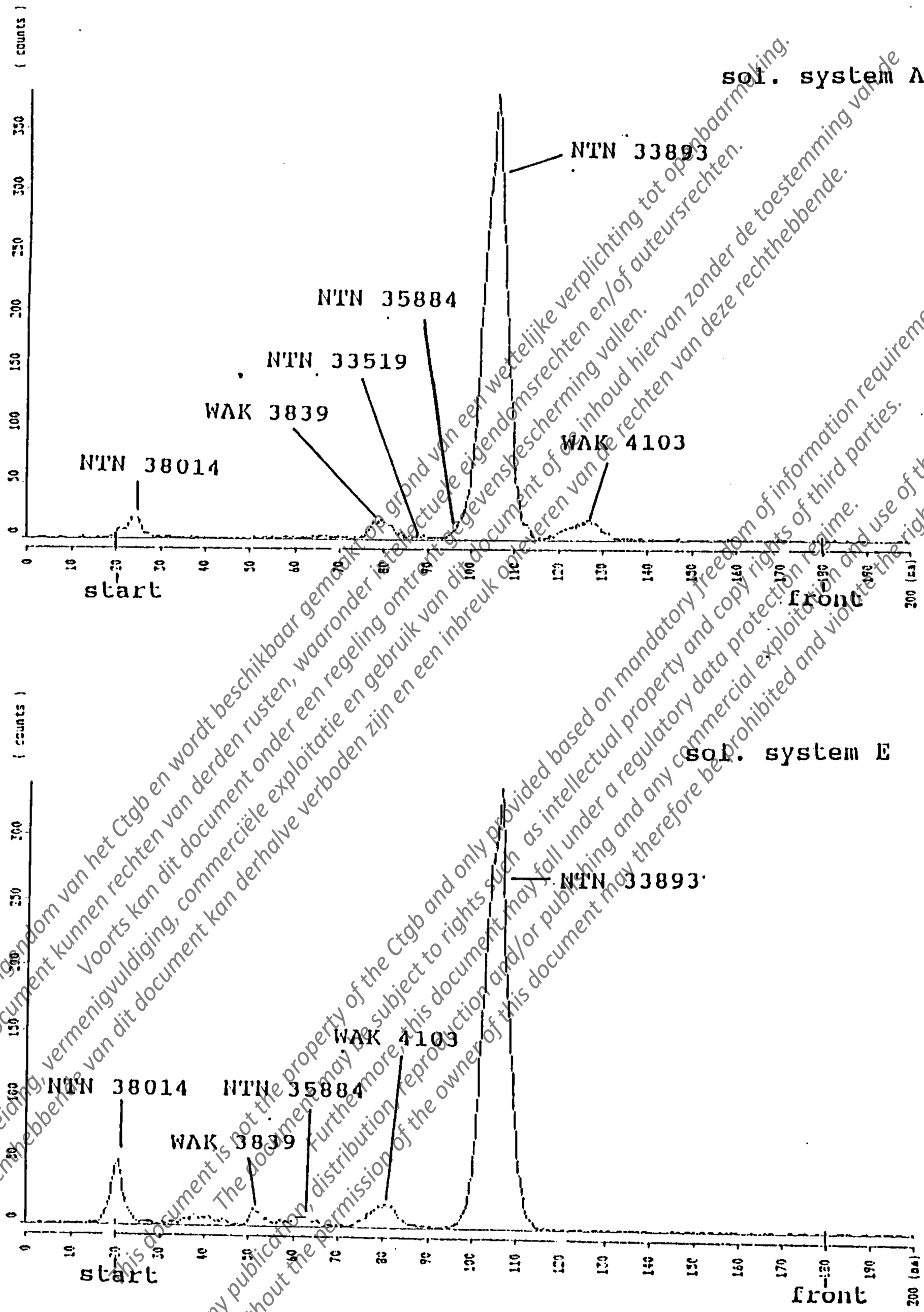

Fig. 2 TLC chromatograms of dichloromethane fraction (Plant ID : # 2, leaves, DAT : 14)

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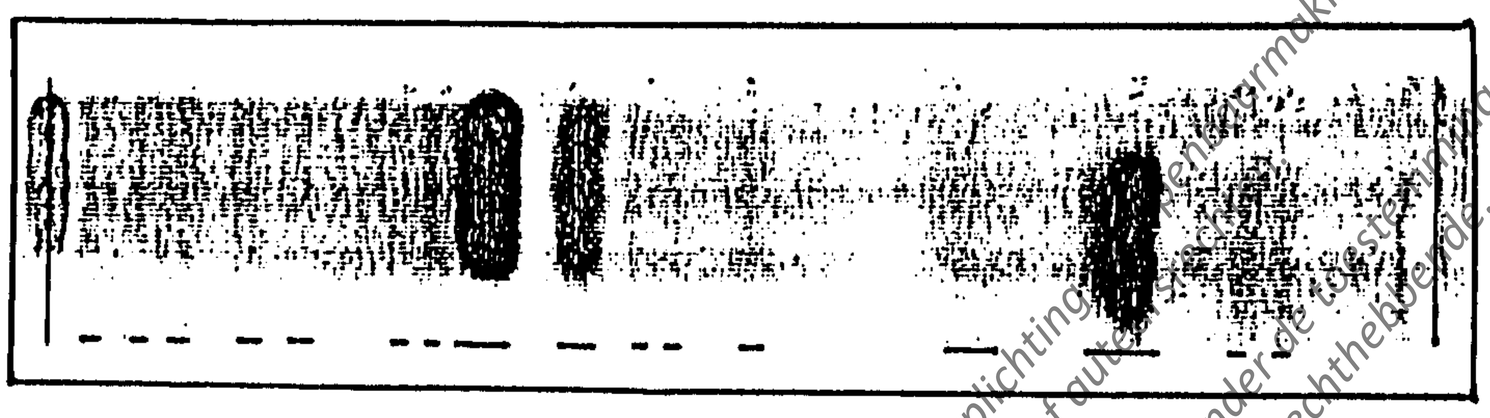

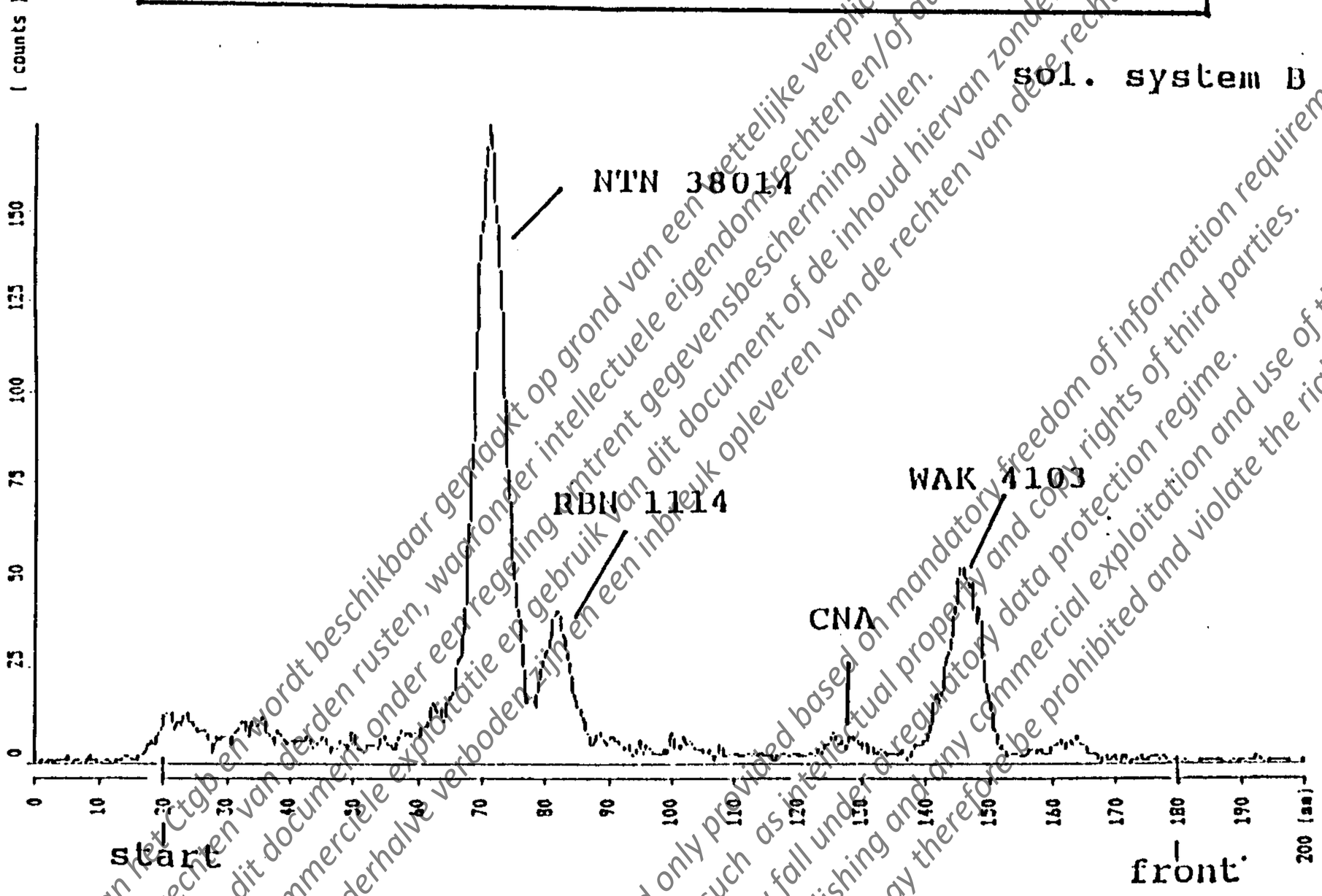

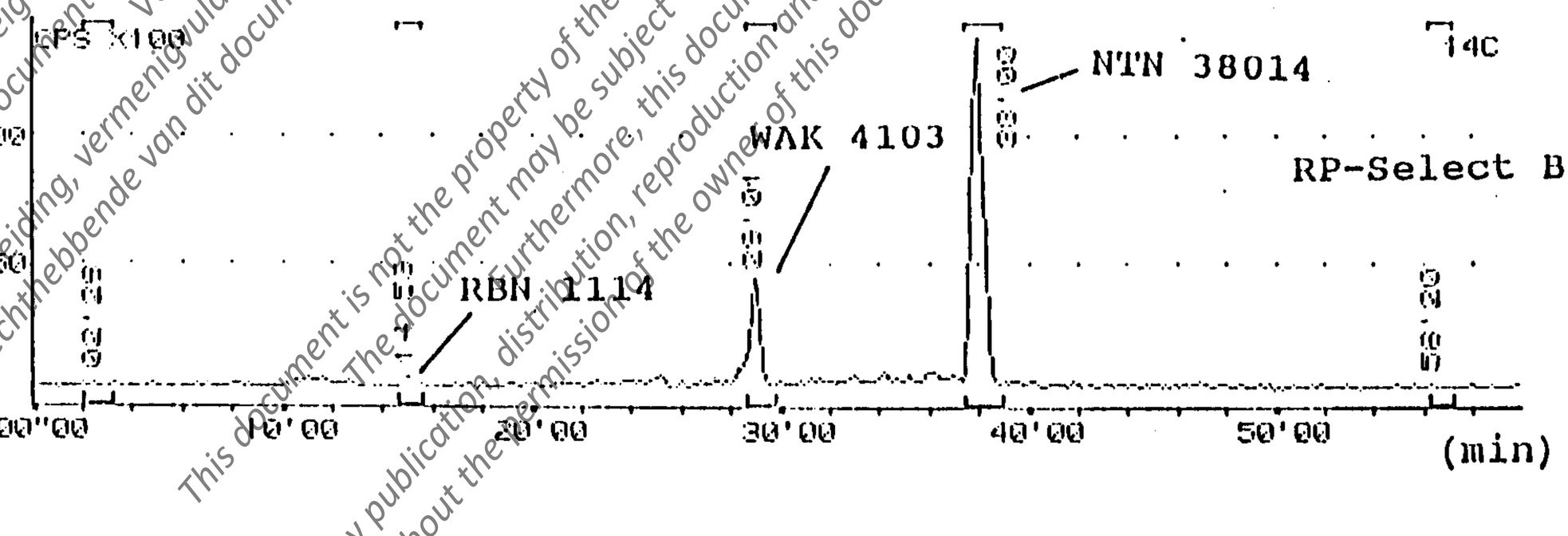

Fig. 3 TLC and HPLC chromatograms of aqueous fraction (Aq 2)
(Plant ID : # 2, Leaves, DAT : 14)

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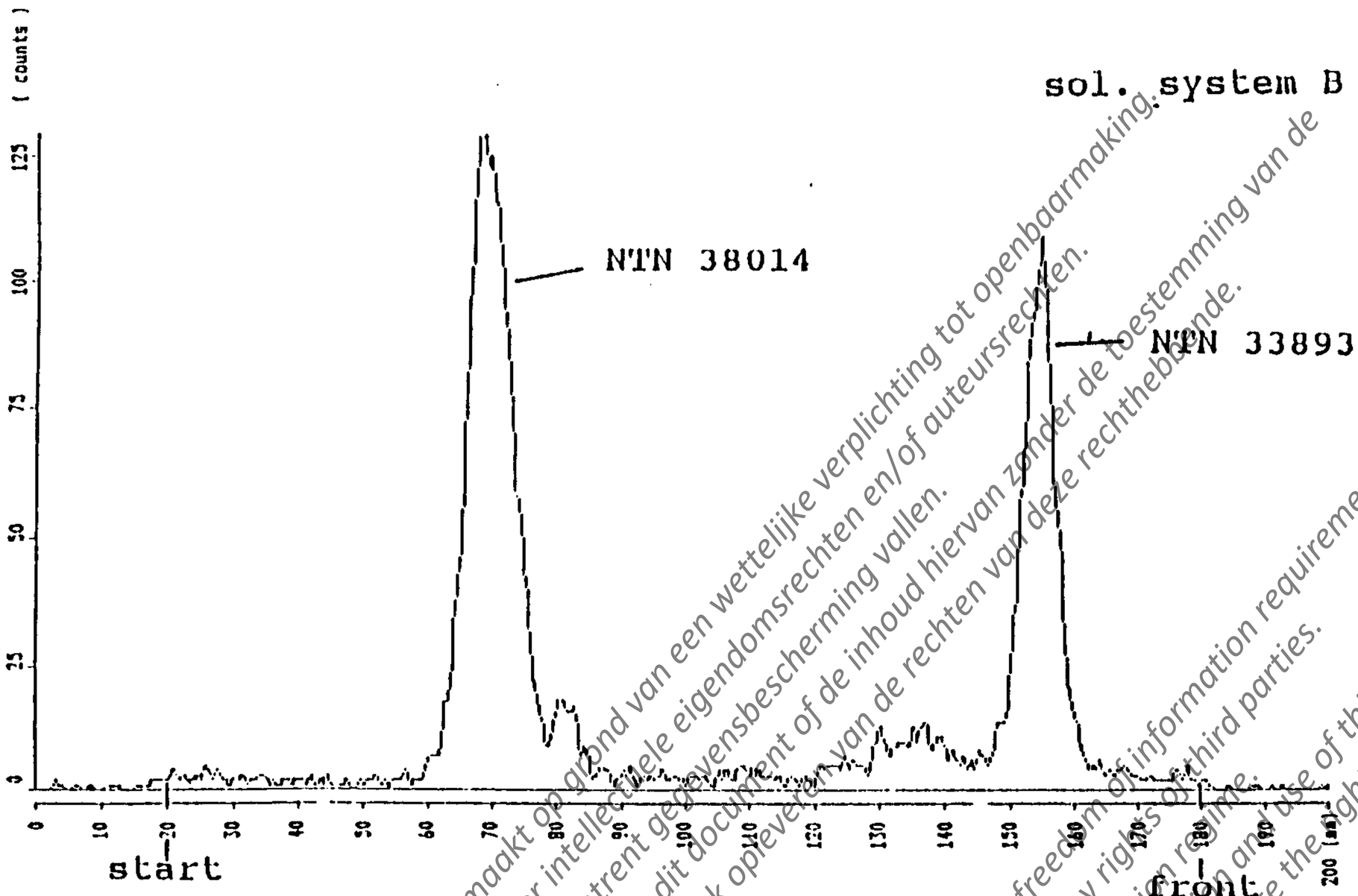

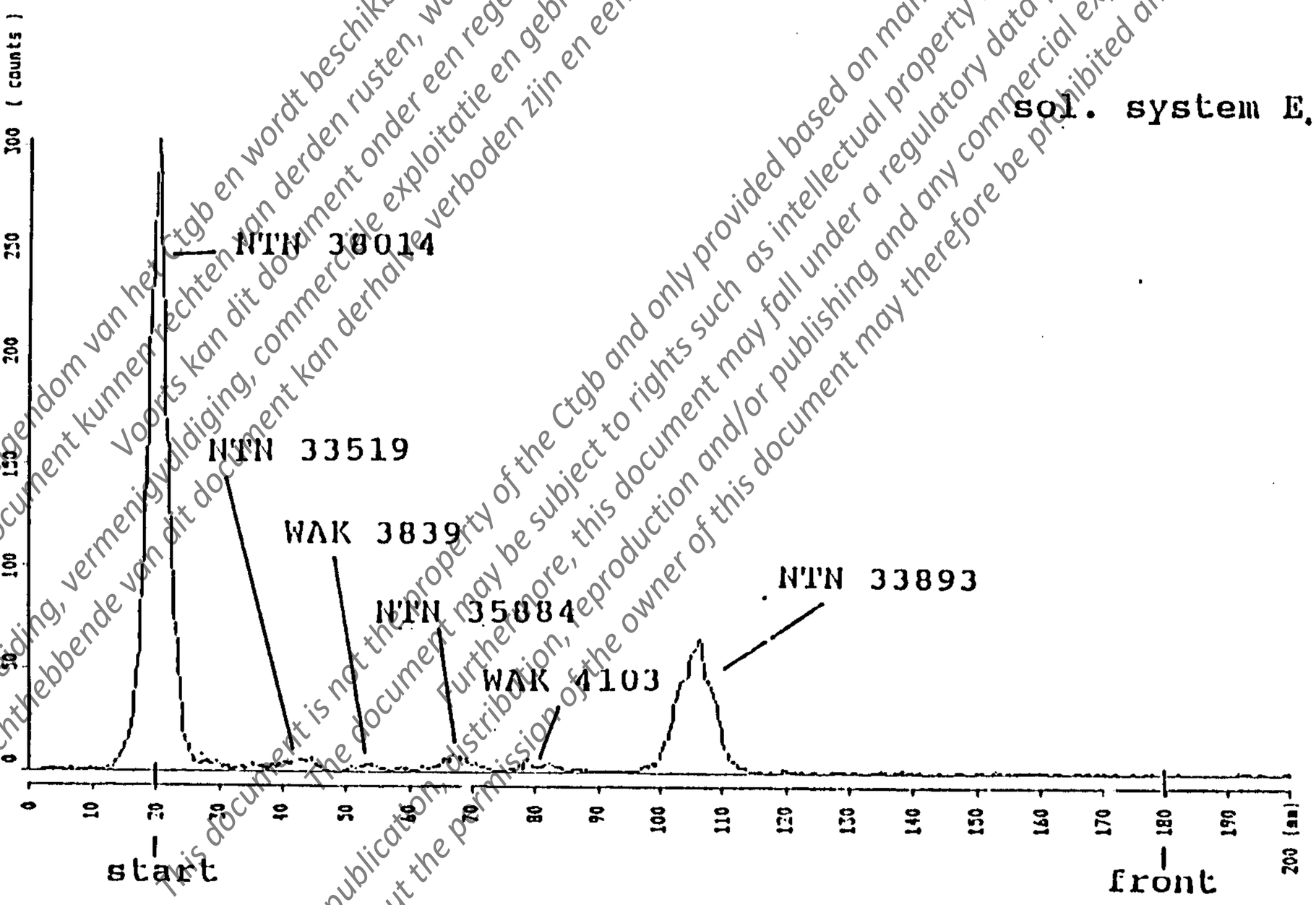

Fig. 4 TLC chromatograms of dichloromethane fraction (Plant ID : # 3, stem, DAT : 35)

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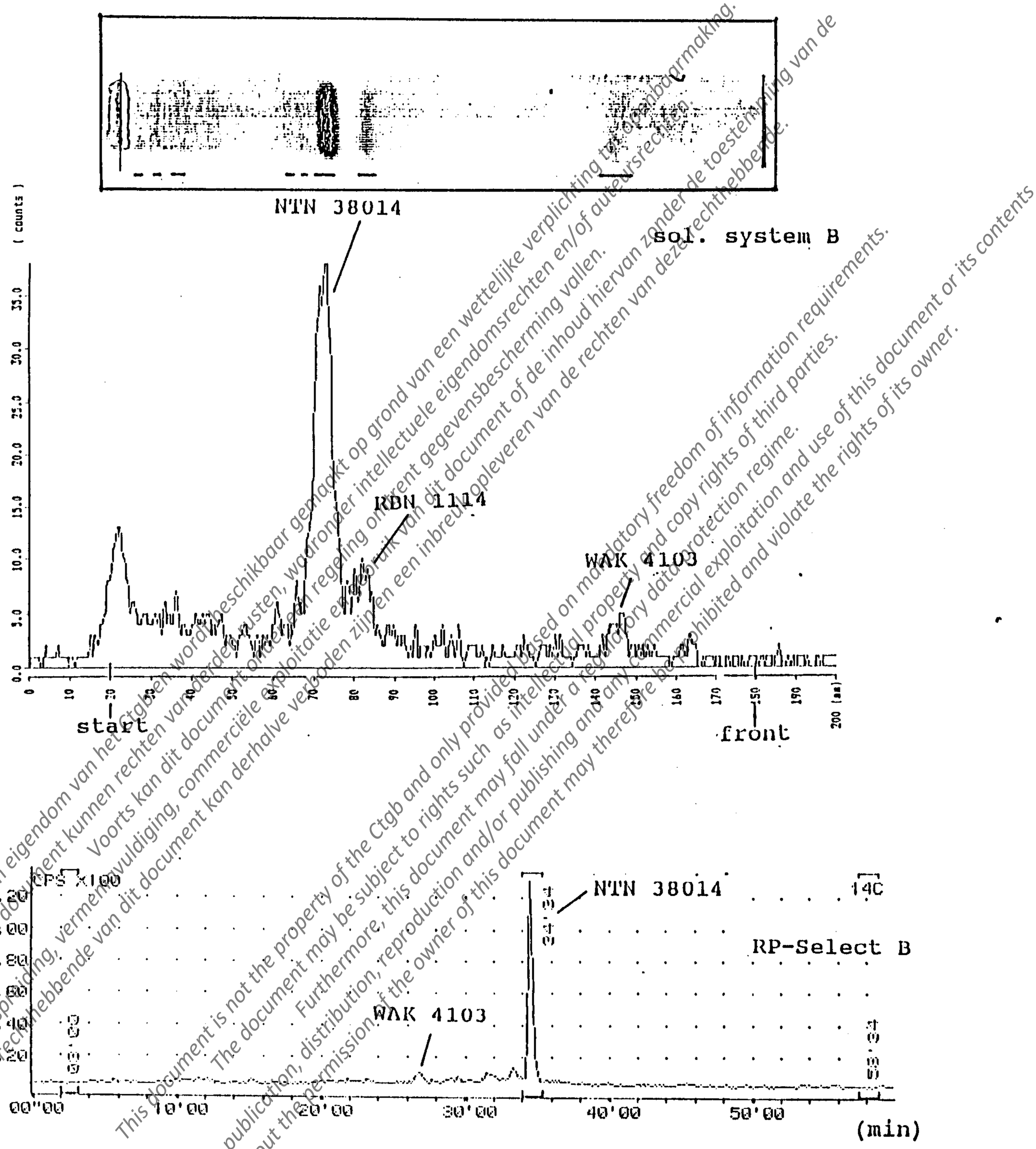

Fig. 5 TLC and HPLC chromatograms of aqueous fraction (Aq 2) (Plant ID : # 3, stem, DAT : 35)

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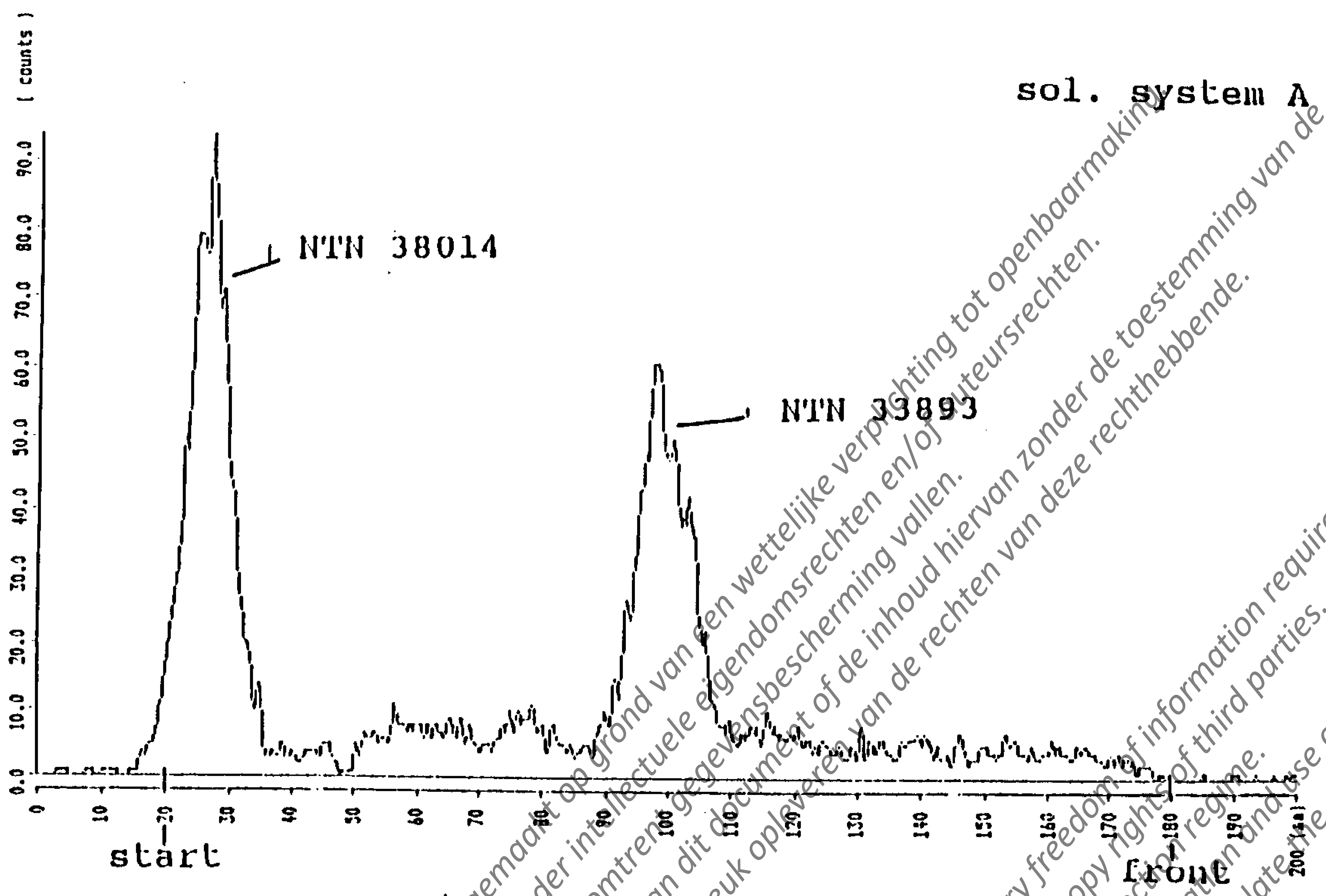

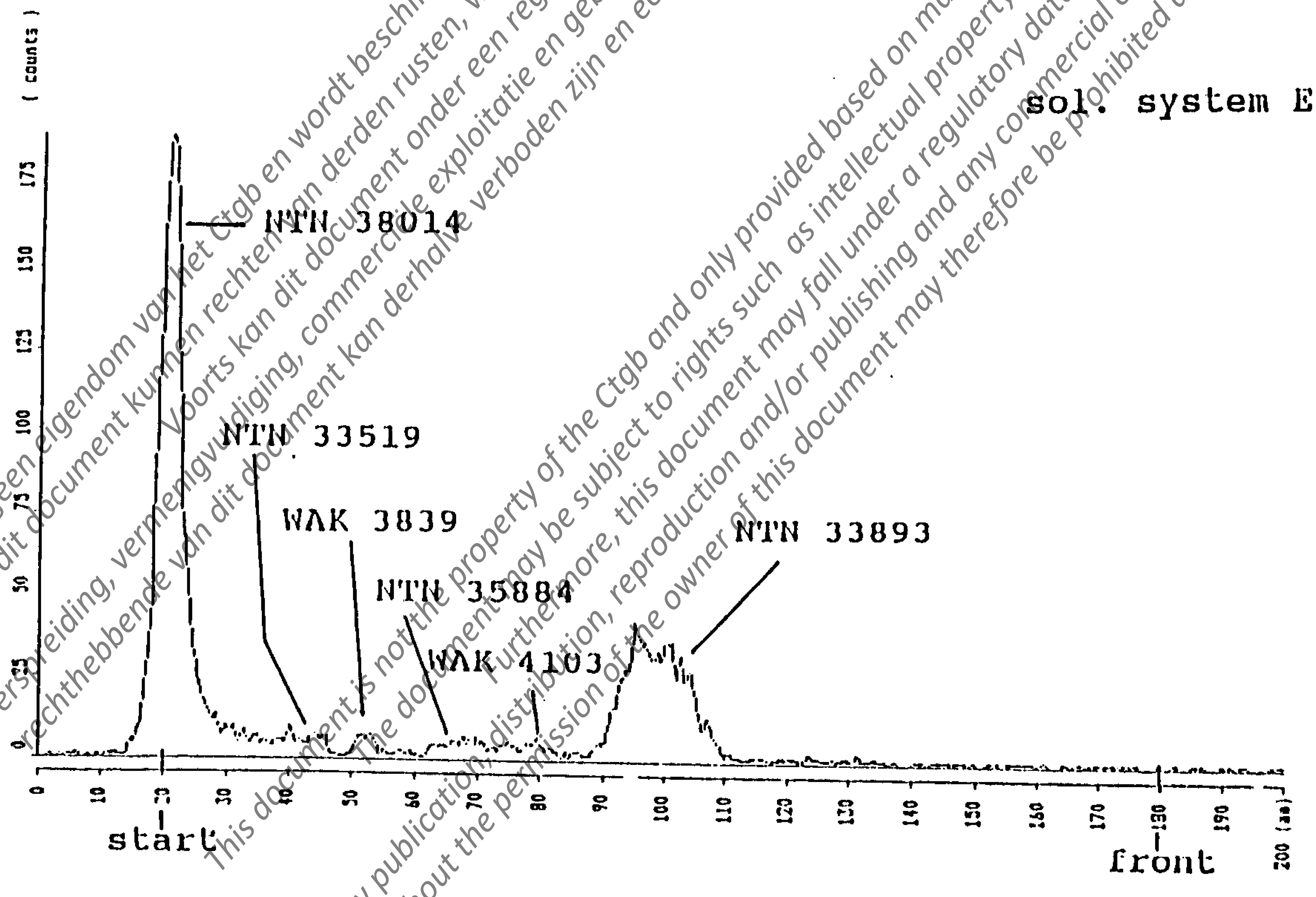

Fig. 6 TLC chromatograms of dichloromethane fraction (Plant ID : # 3, leaves, DAT : 35)

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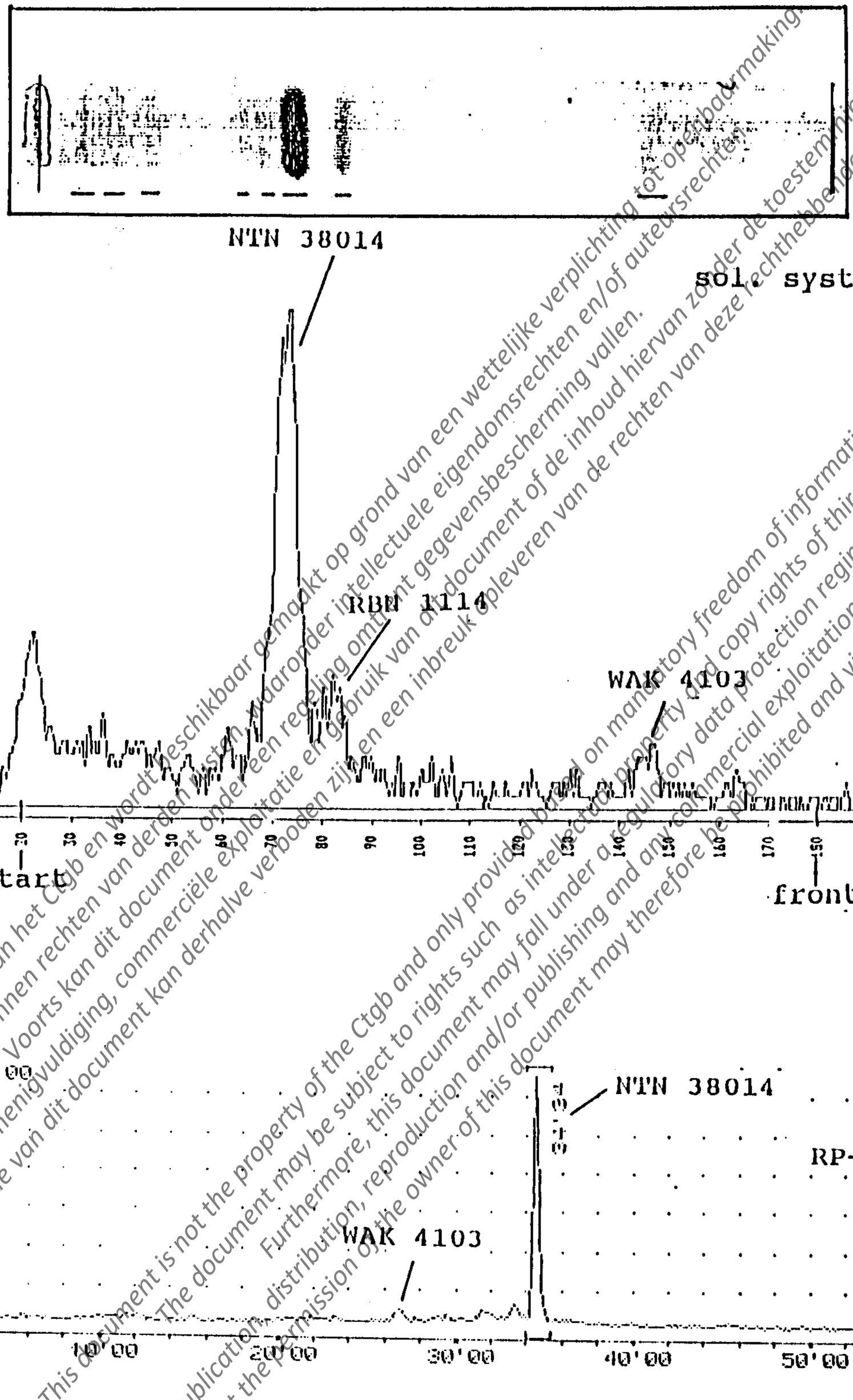

Fig. 7 TLC and HPLC chromatograms of aqueous fraction (Aq 2) (Plant ID : # 3, leaves, DAT : 35)

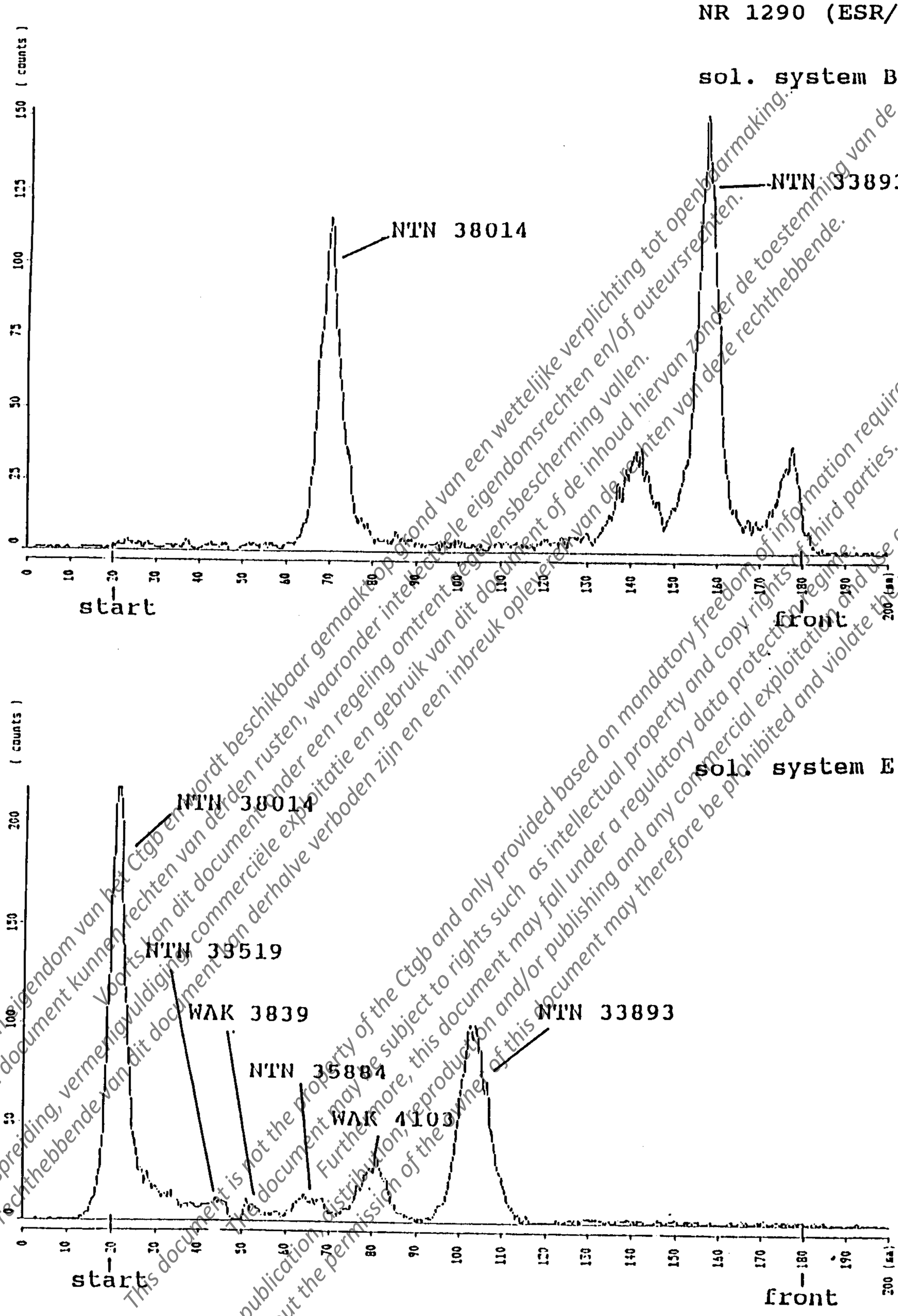

Fig. 8 TLC chromatograms of dichloromethane fraction (Plant ID : # 4, leaves, DAT : 35)

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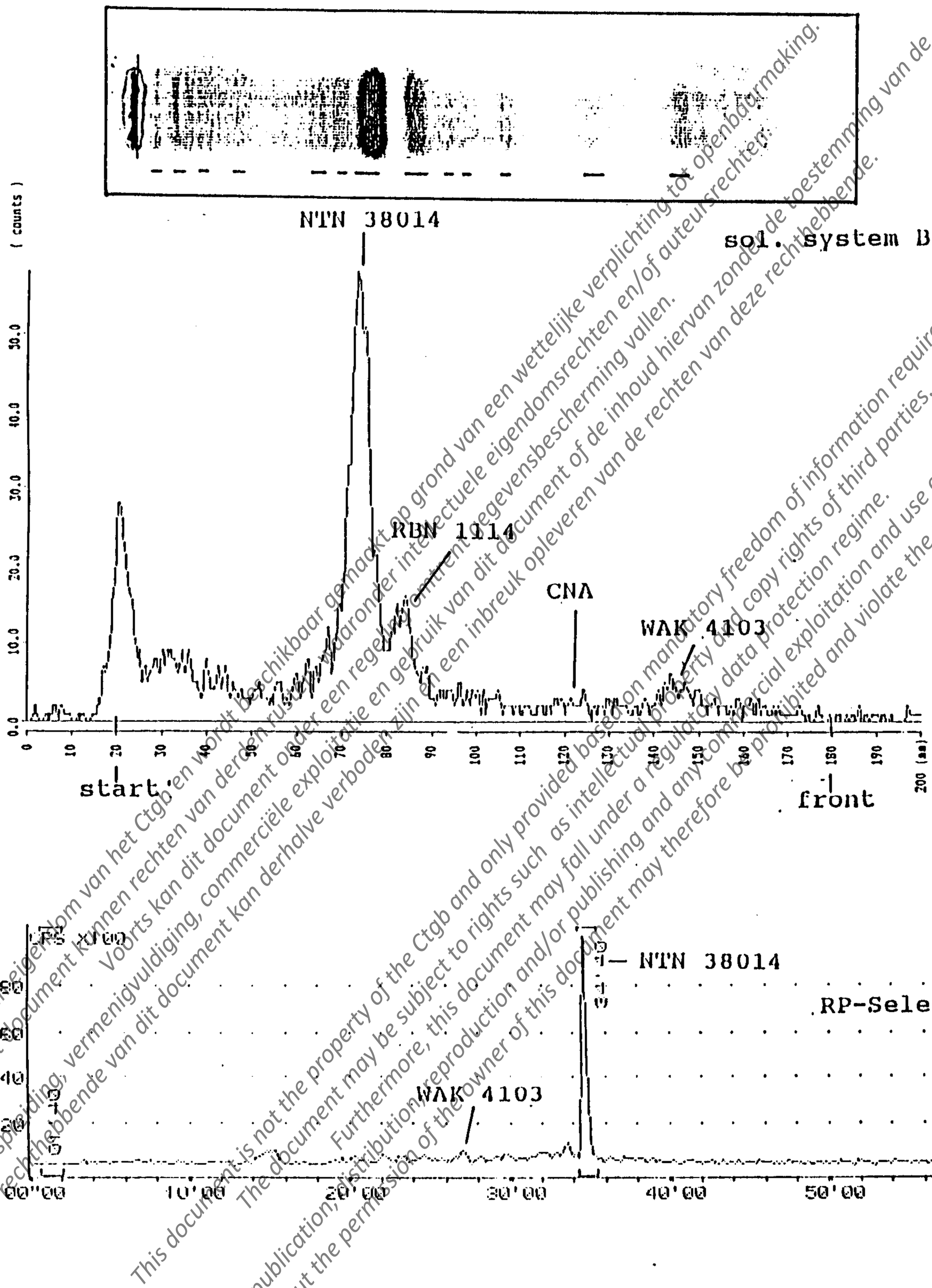

Fig. 9 TLC and HPLC chromatograms of aqueous fraction (Aq 2) (Plant ID : # 4, leaves, DAT : 35)

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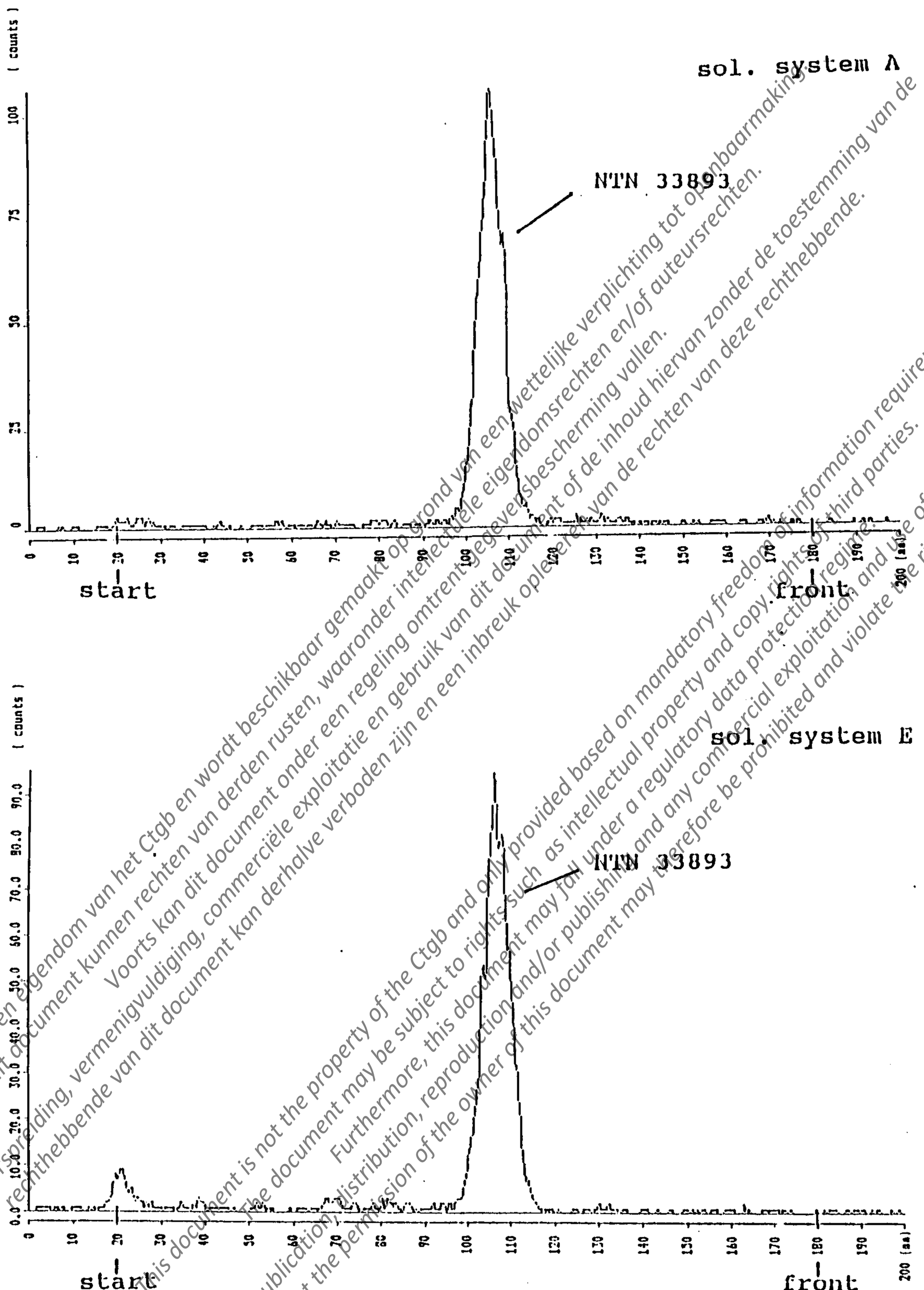

Fig. 10 TLC chromatograms of dichloromethane fraction (Plant ID : # 5, stem, DAT : 69)

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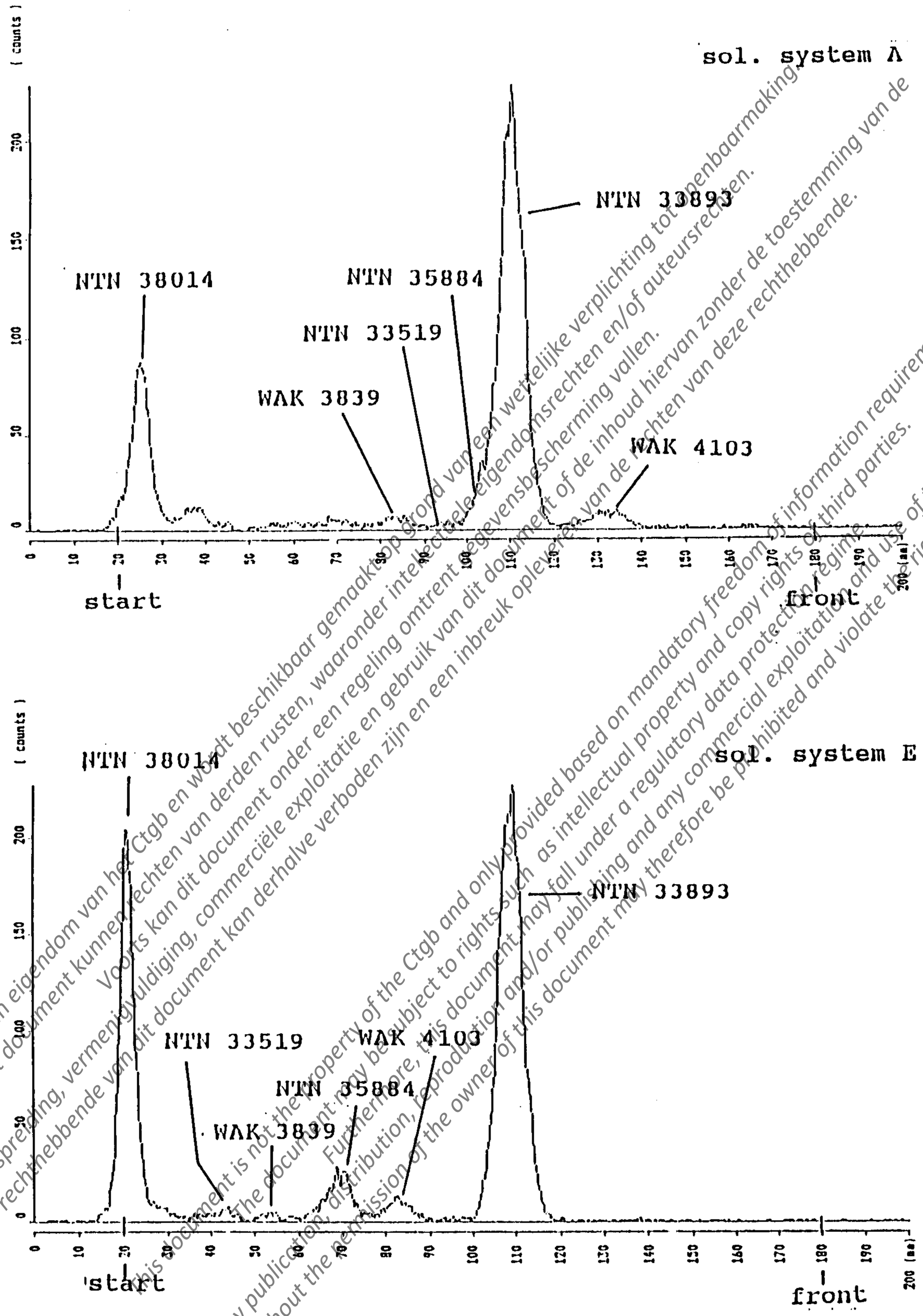

Fig. 11 TLC chromatograms of dichloromethane fraction (Plant ID : # 5, leaves, DAT : 69)

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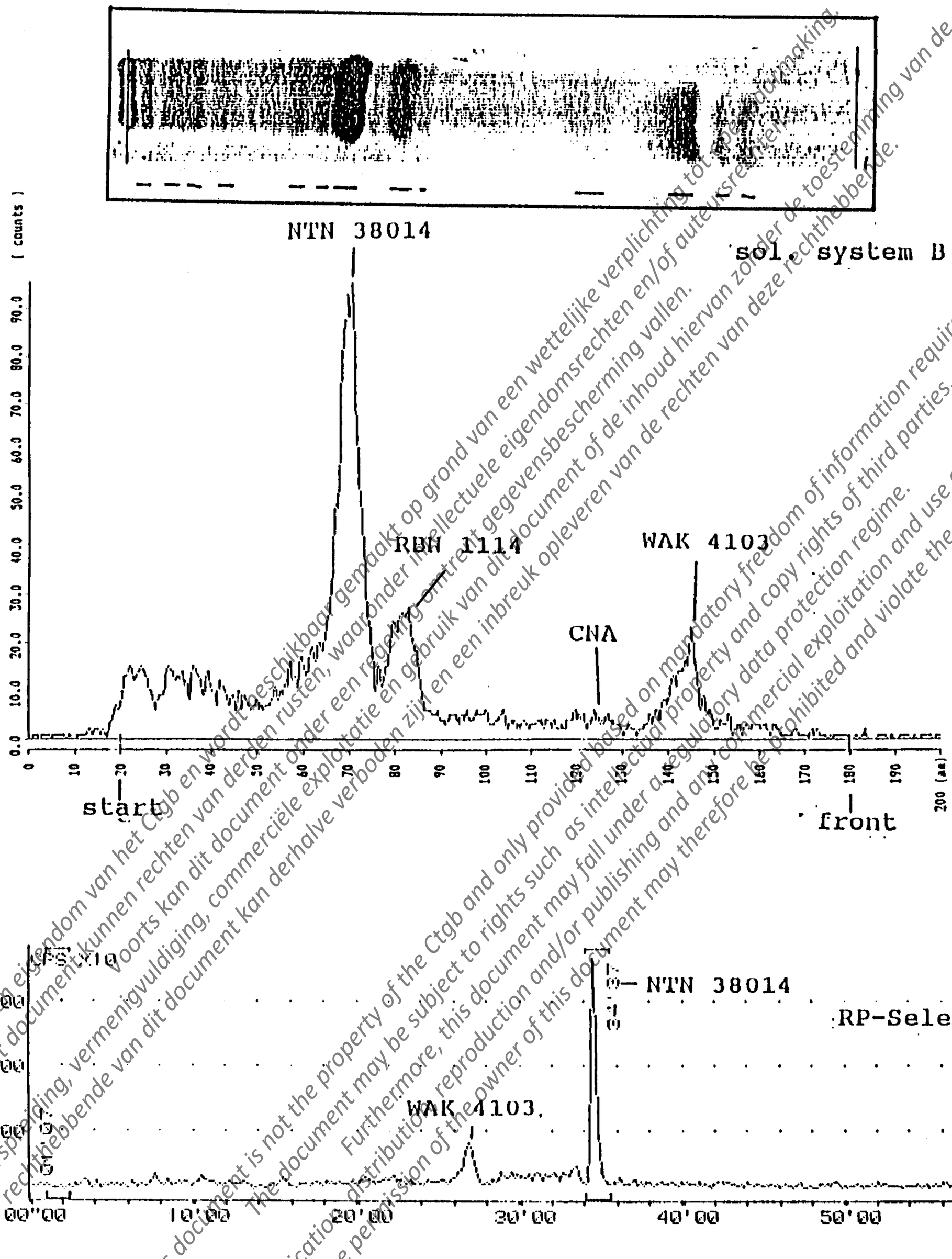

Fig. 12 TLC and HPLC chromatograms of aqueous fraction (Aq 2)
(Plant ID : # 5, leaves, DAT : 69)

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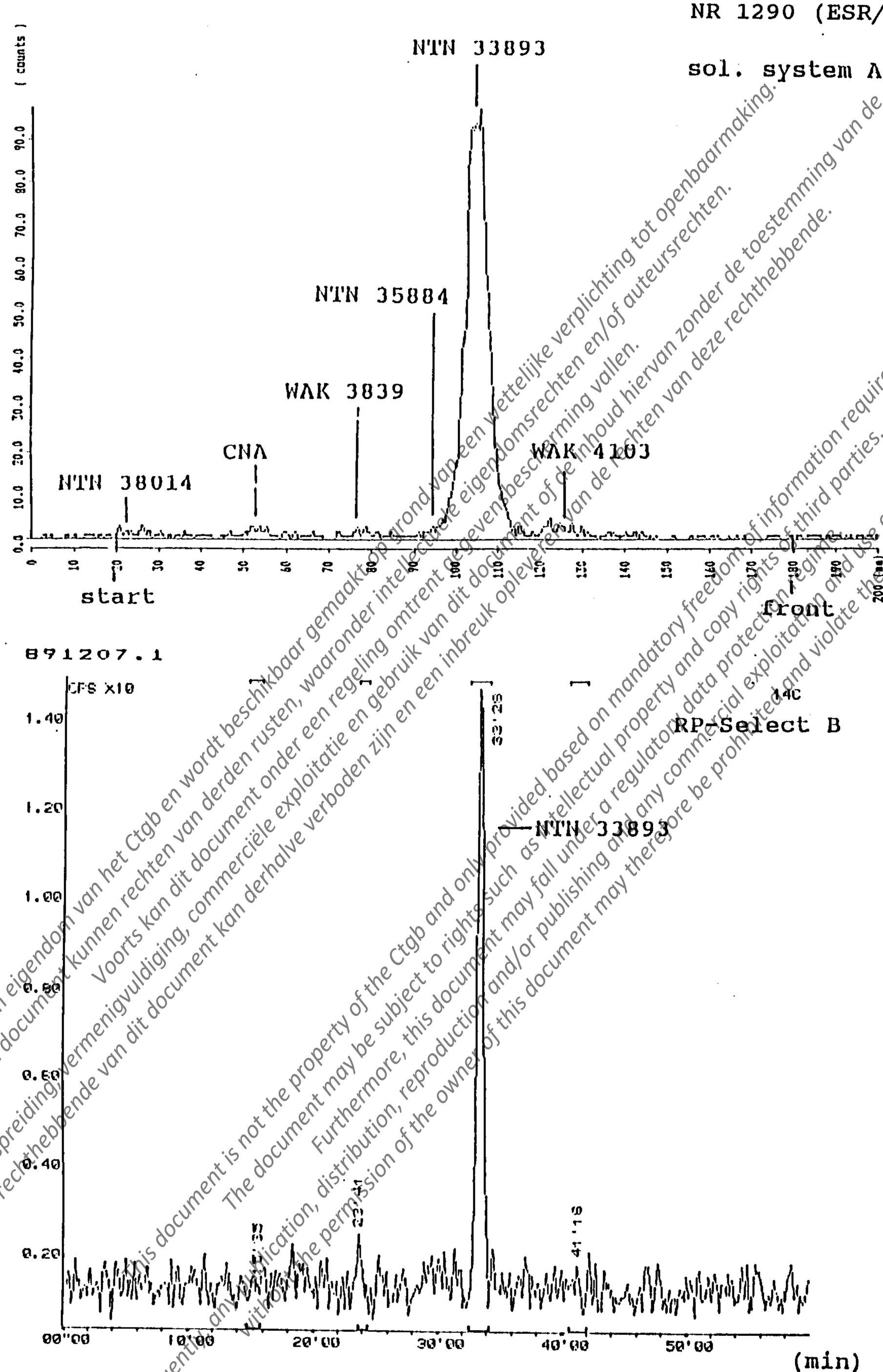

Fig. 13 TLC and HPLC chromatograms of dichloromethane fraction (Plant ID : # 5, edible part, DAT : 49)

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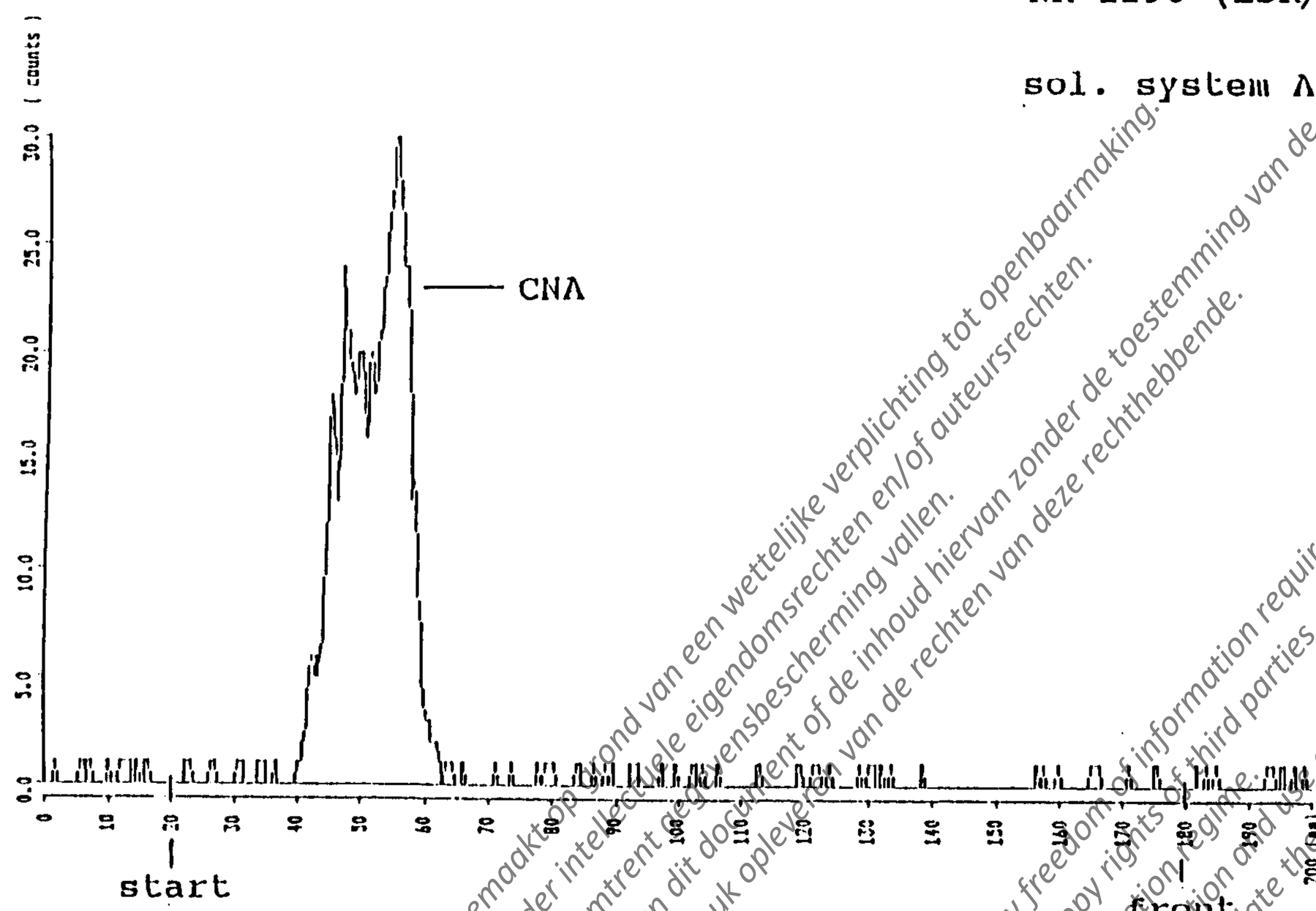

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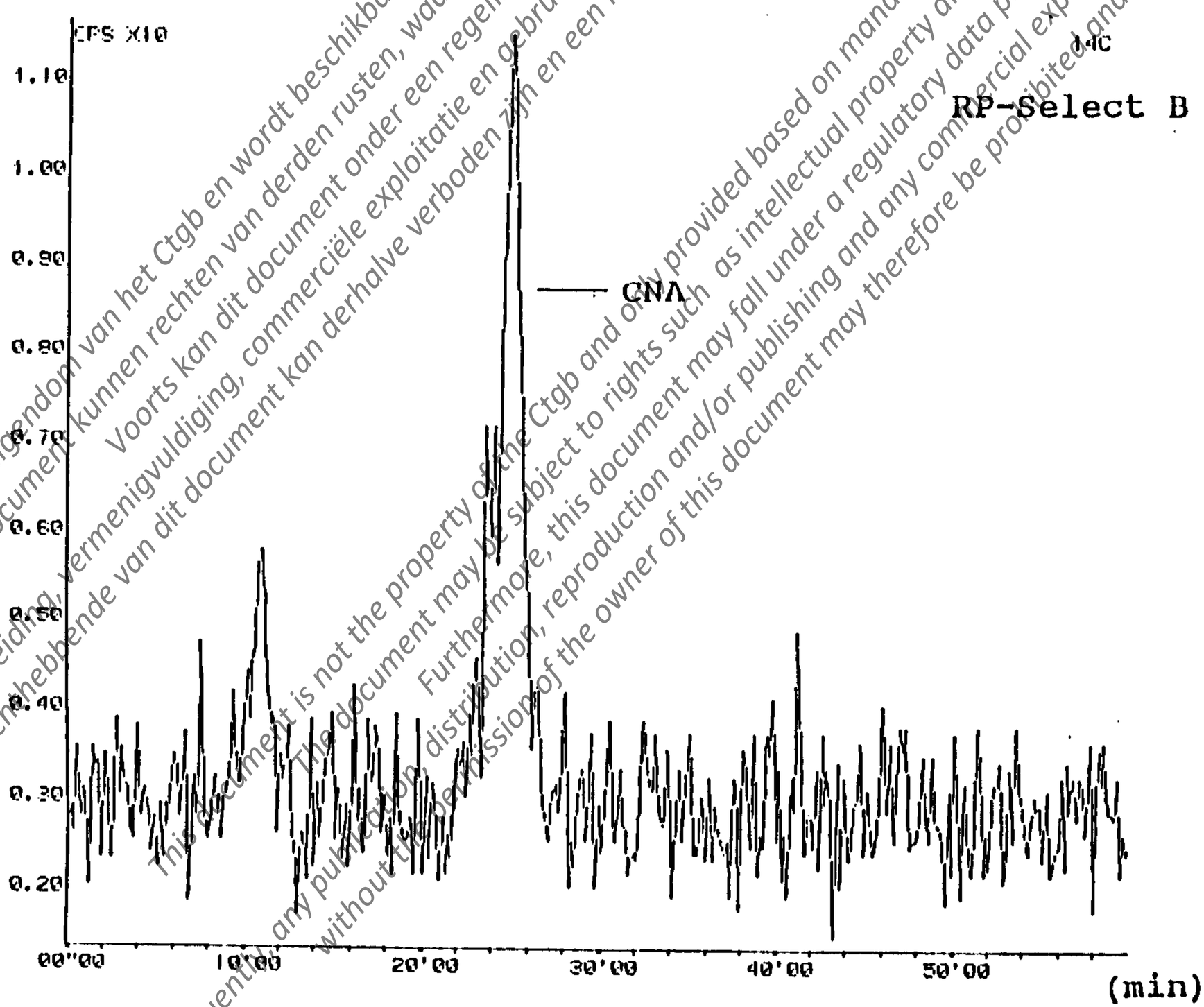

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Fig. 14 TLC and HPLC chromatograms of aqueous fraction (Aq 1-2)
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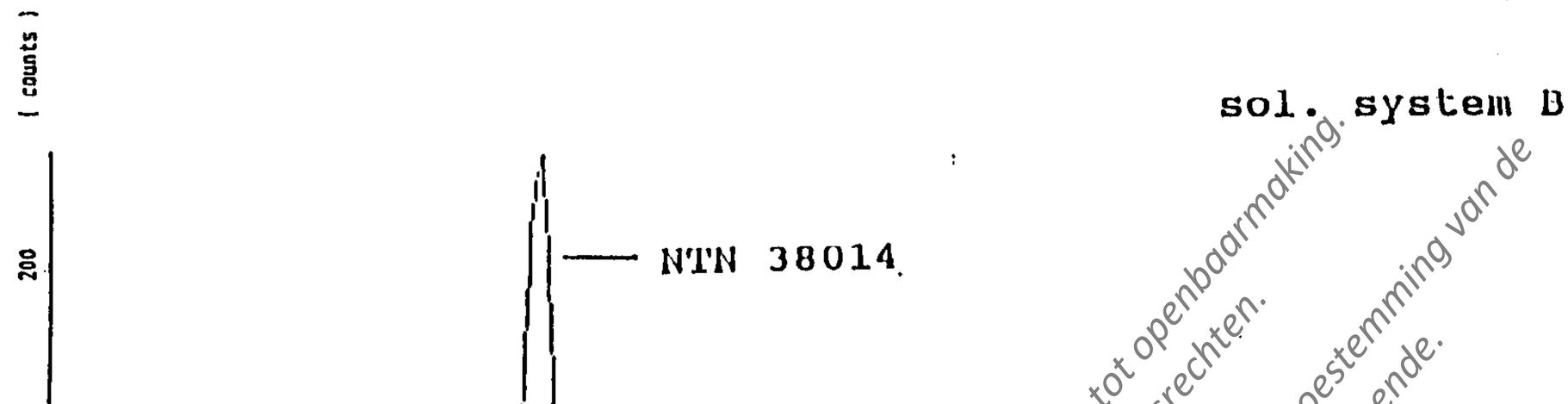

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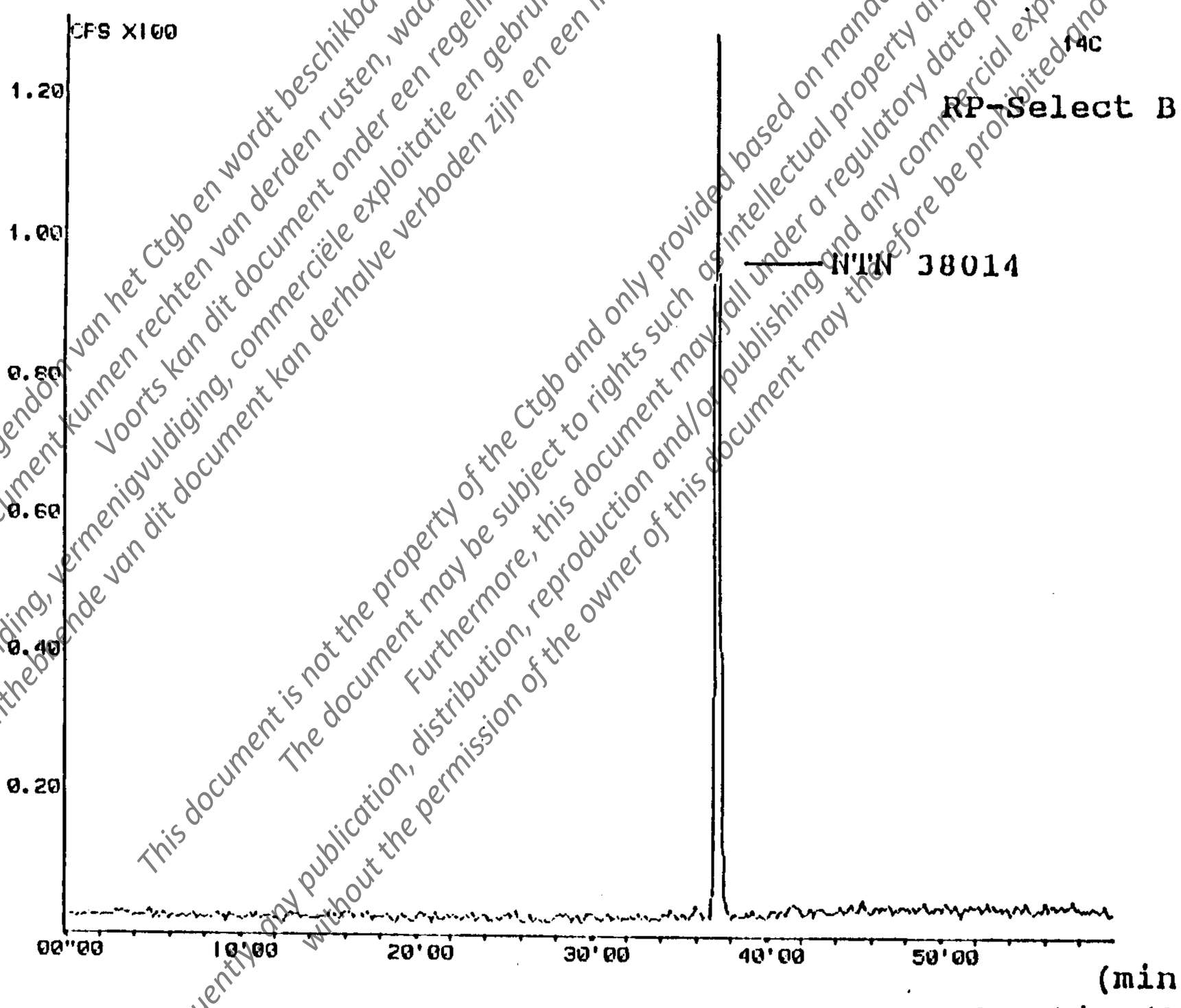

Fig. 15 TLC and HPLC chromatograms of aqueous fraction(Aq 2-2)
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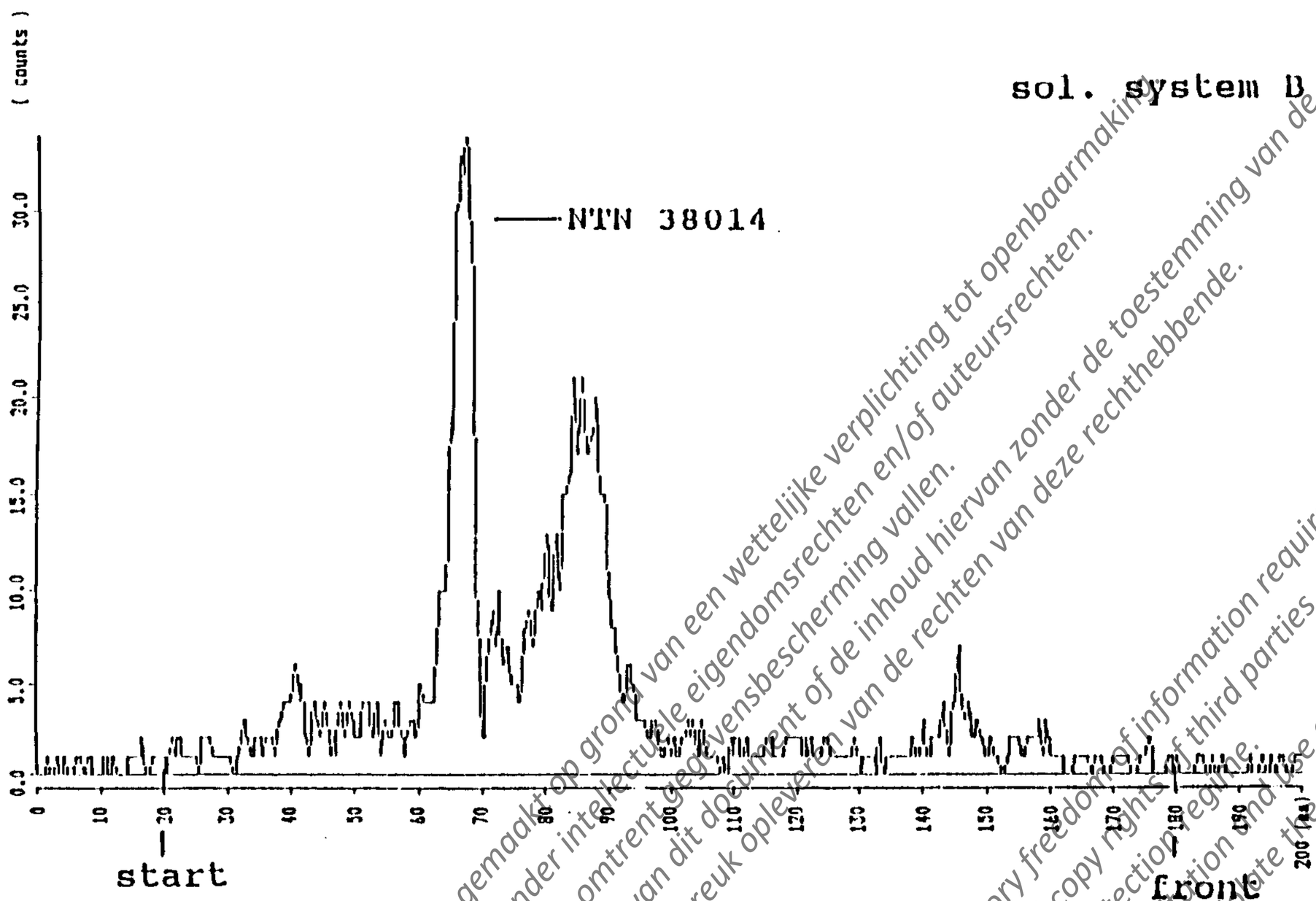

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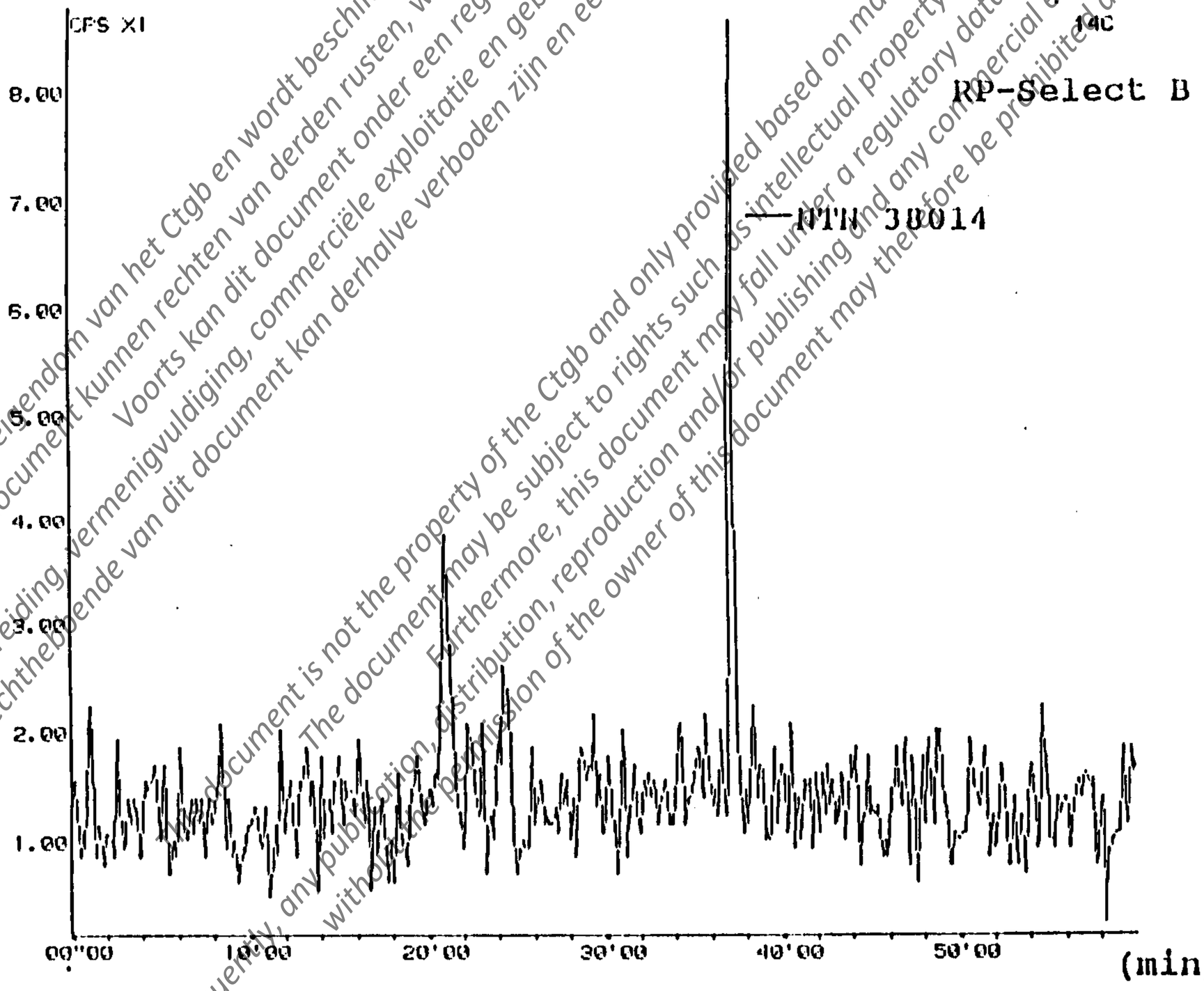

Fig. 16 TLC and HPLC chromatograms of aqueous fraction(Aq 2-1-1)
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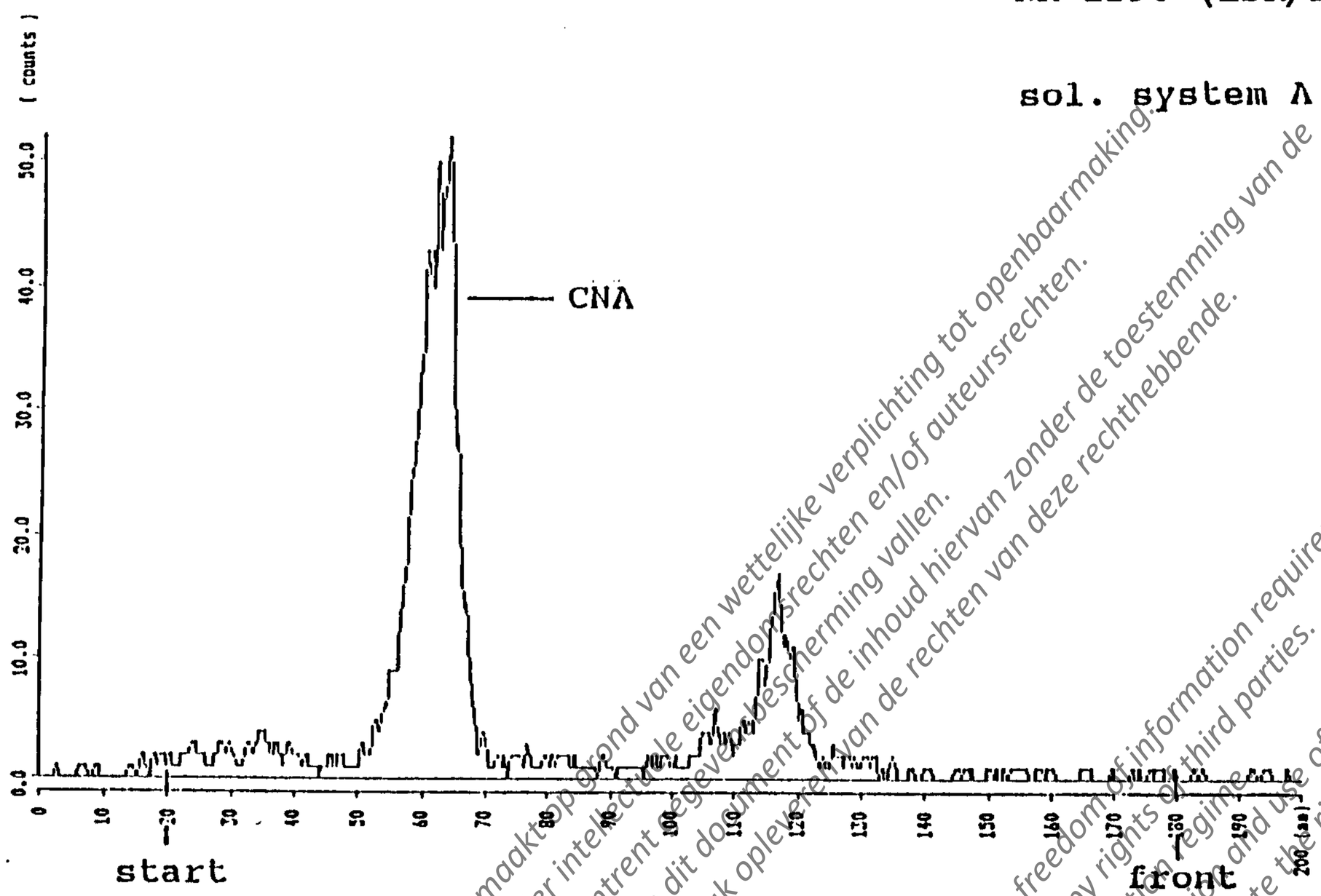

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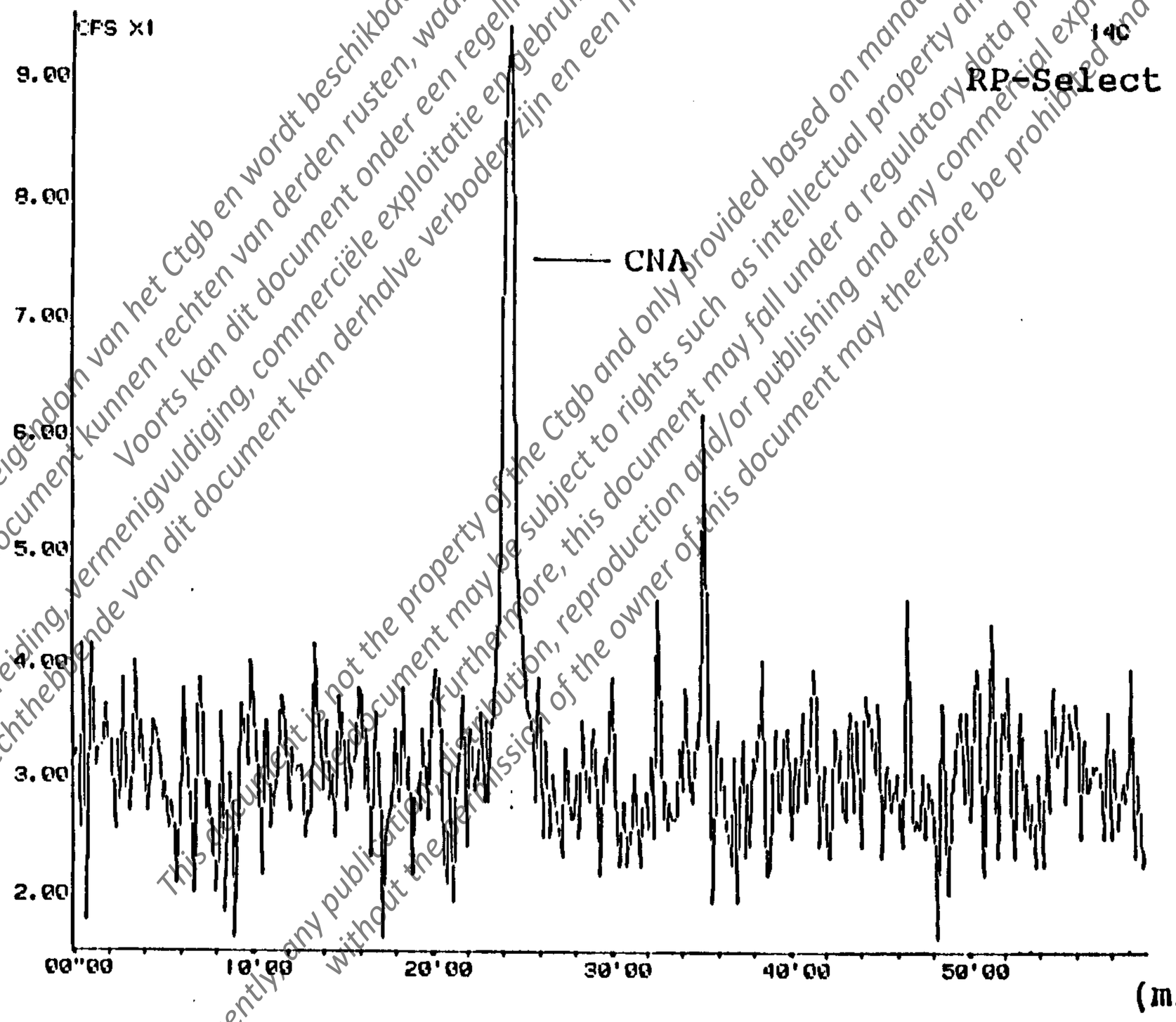

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Fig. 17 TLC and HPLC chromatograms of aqueous fraction(Aq 2-1-2)
(Plant ID : # 5, edible part, DAT : 49)

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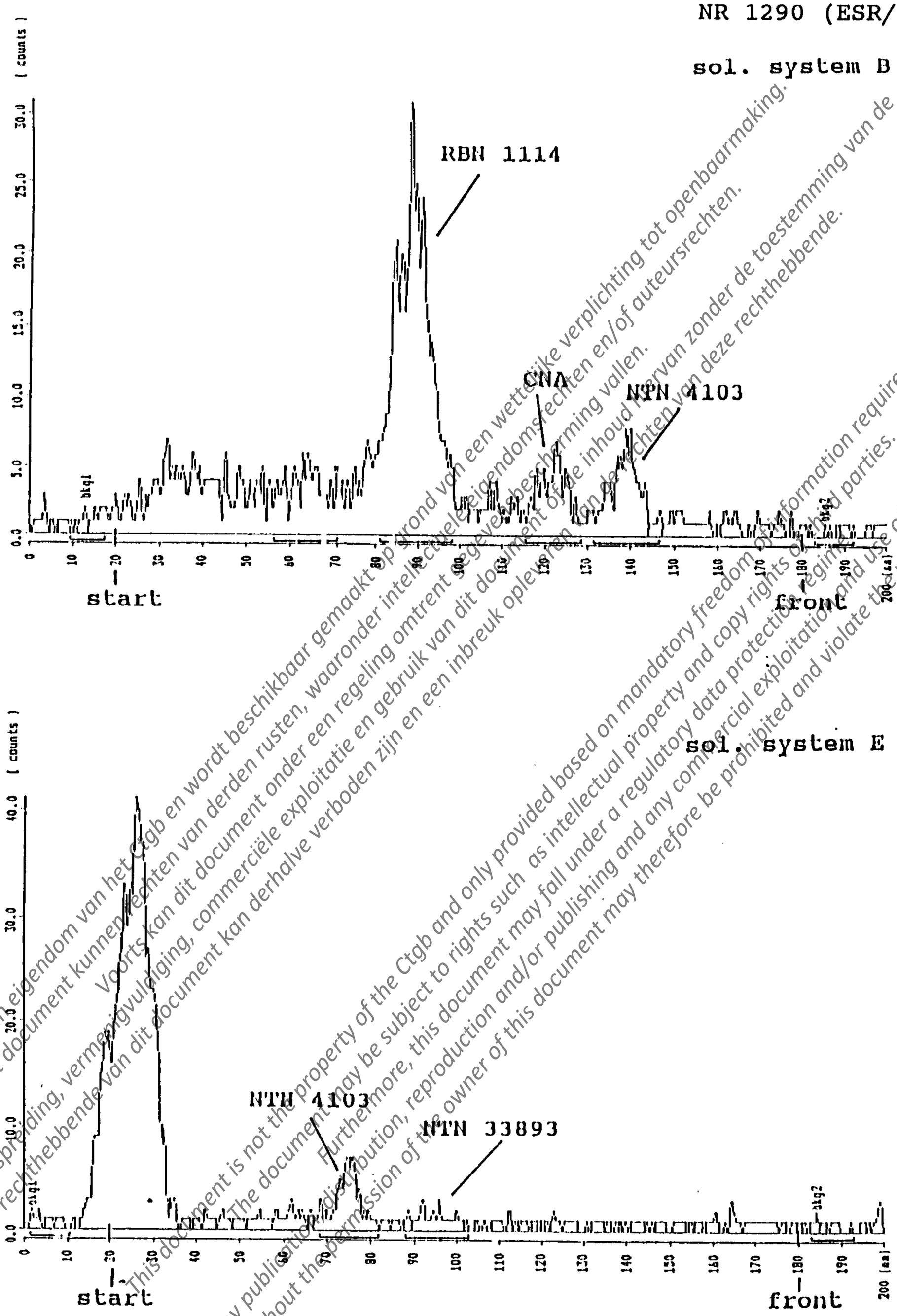

Fig. 18 TLC chromatograms of aqueous fraction
(Plant ID : # 5, edible part, DAT : 67)

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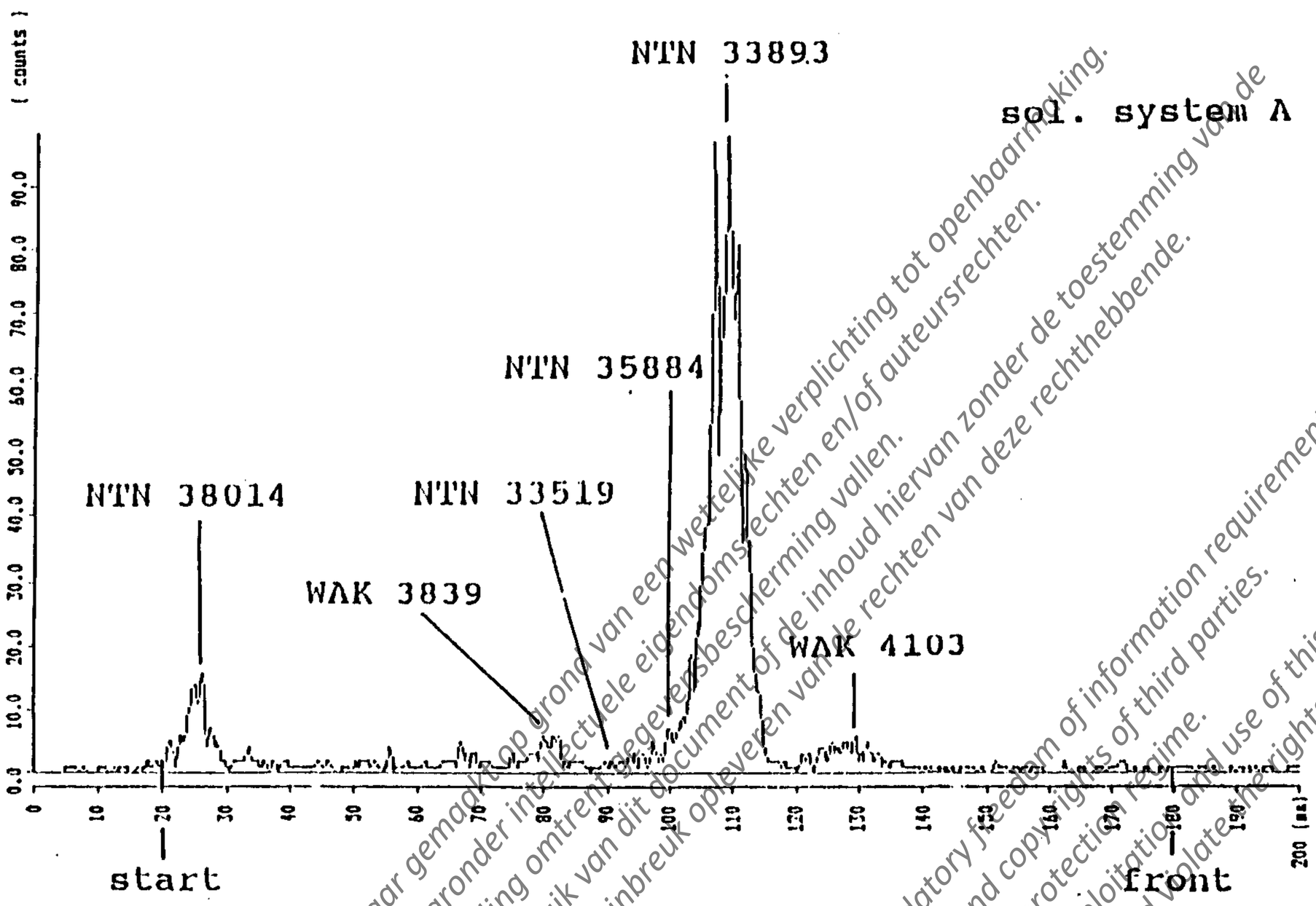

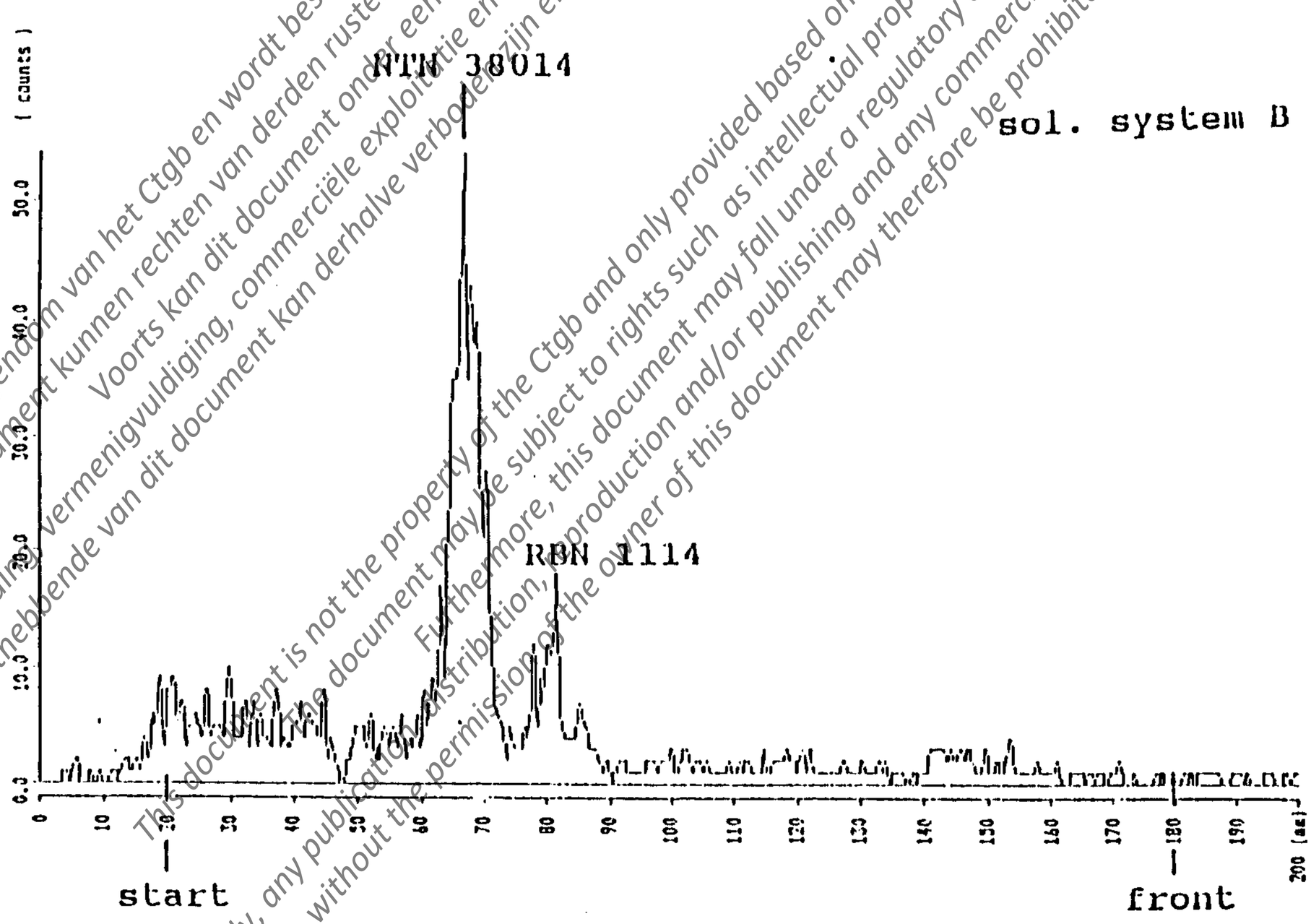

Fig. 19 TLC chromatograms of dichloromethane fraction (upper) and aqueous fraction (lower)
(Plant ID : # 10, leaves, stem injections)

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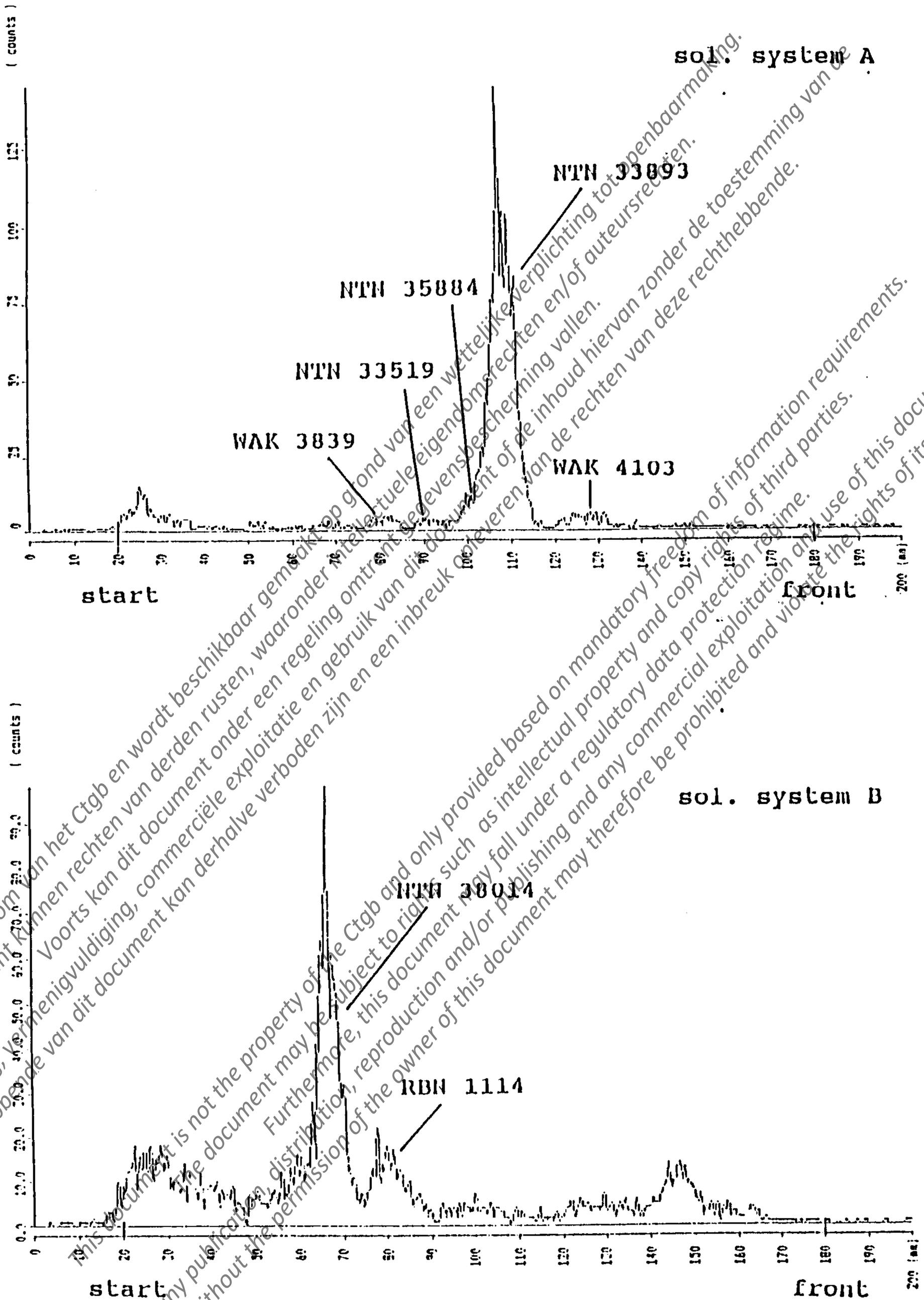

Fig. 20 TLC chromatograms of dichloromethane fraction (upper) and aqueous fraction (lower)
(Plant ID : # 11, leaves, application of exaggerate amount)

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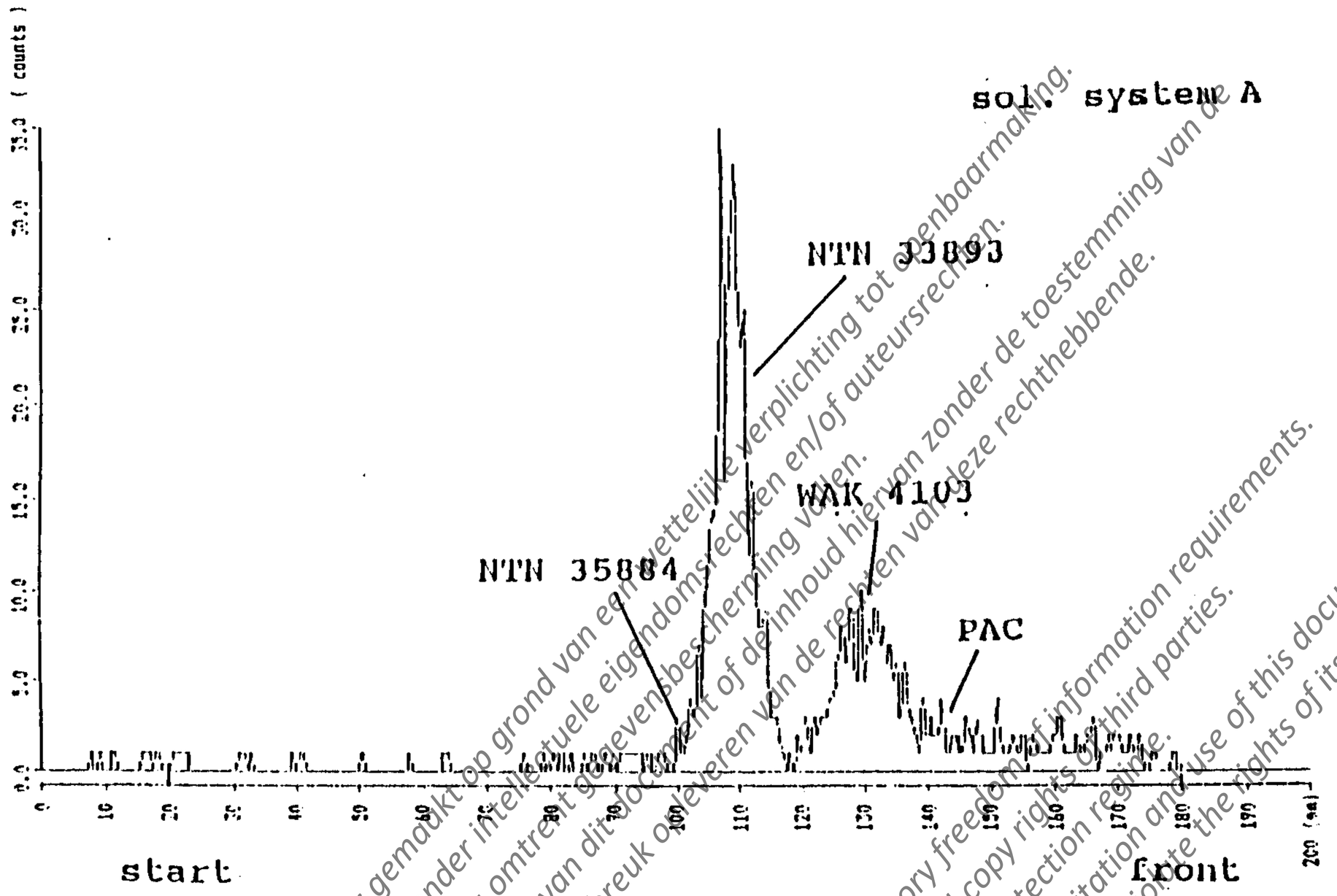

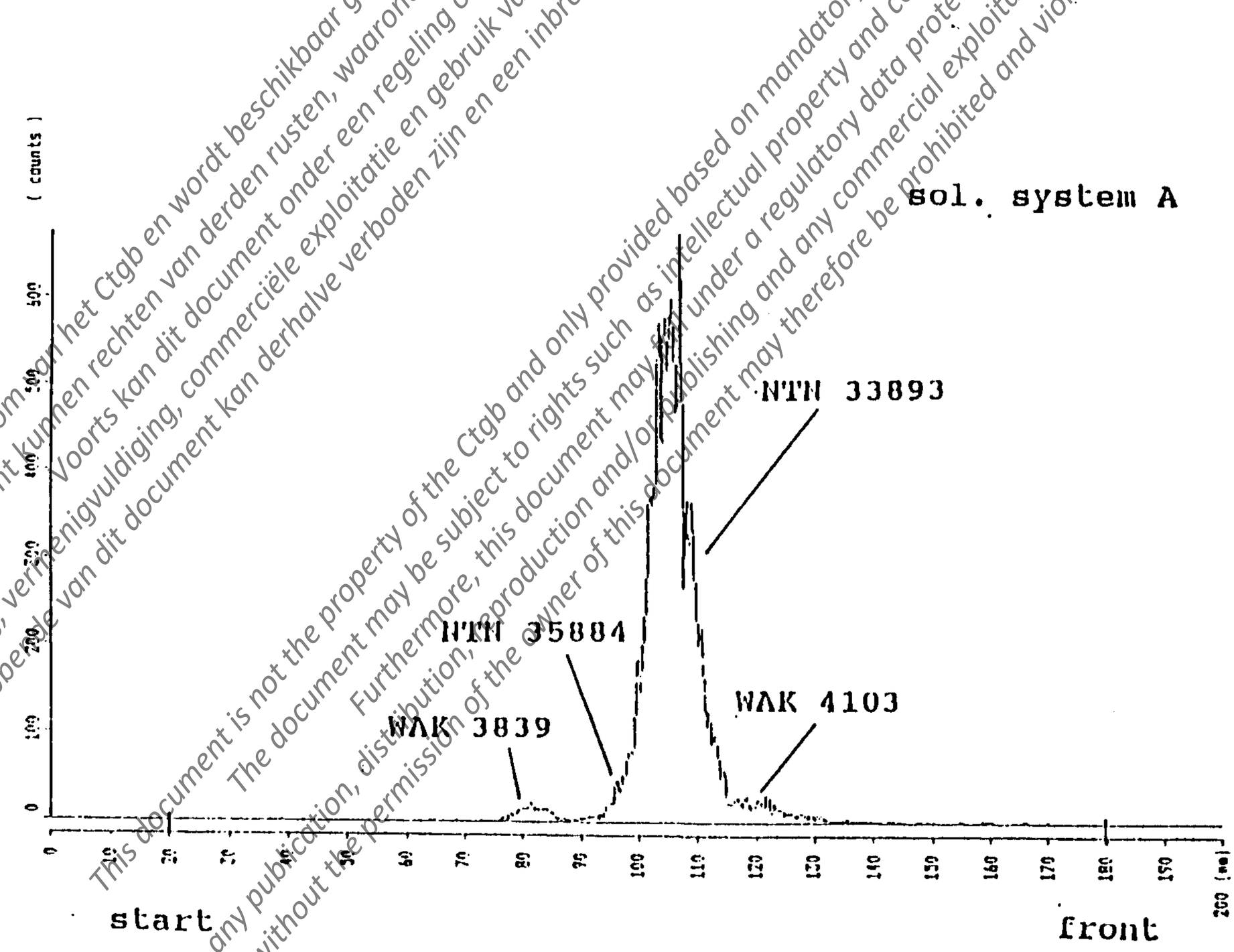

Fig. 21 TLC chromatograms of Fr.-5 (upper) and Fr.-6 (lower) (Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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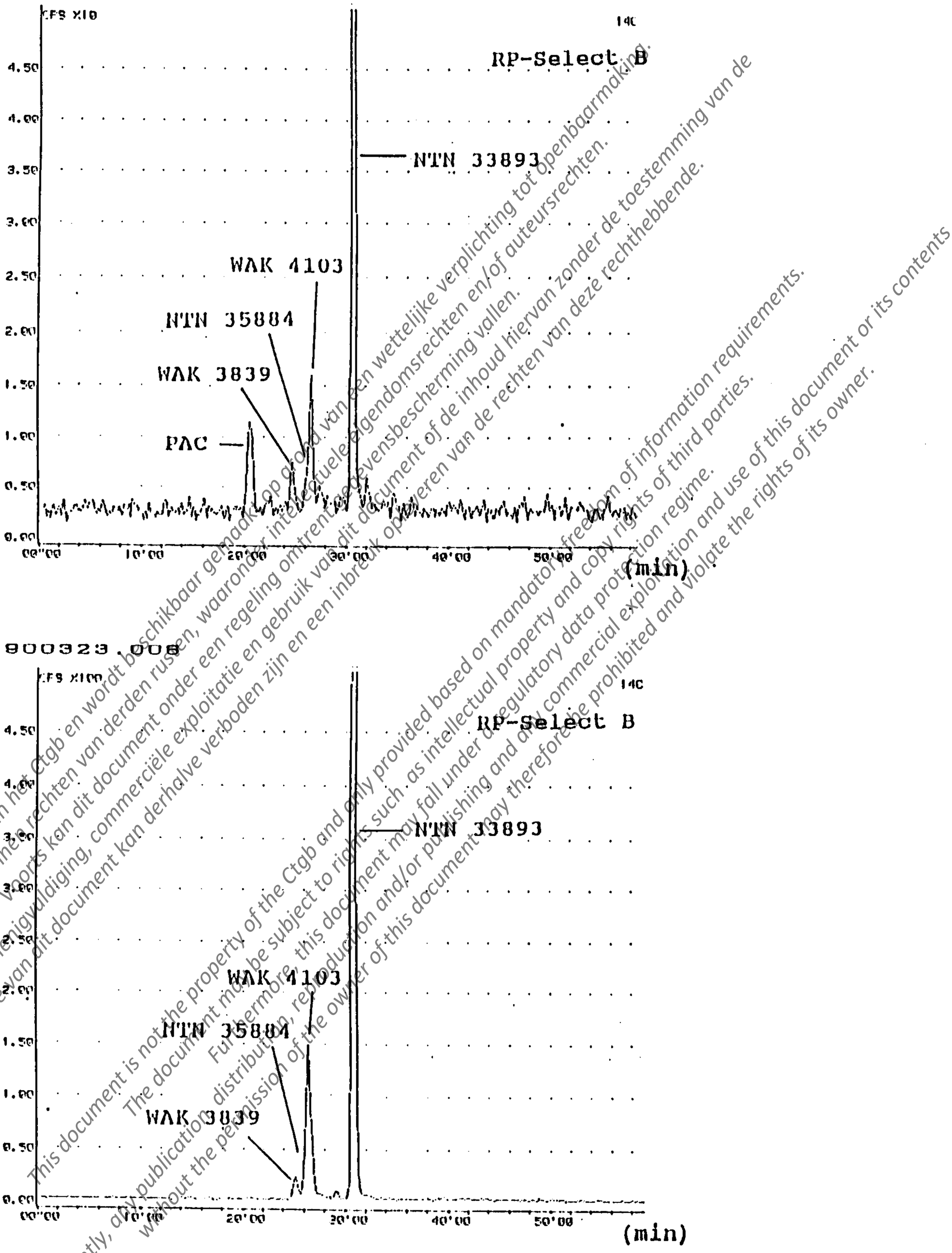

Fig. 22 HPLC chromatograms of Fr.-5 (upper) and Fr.-6 (lower) (Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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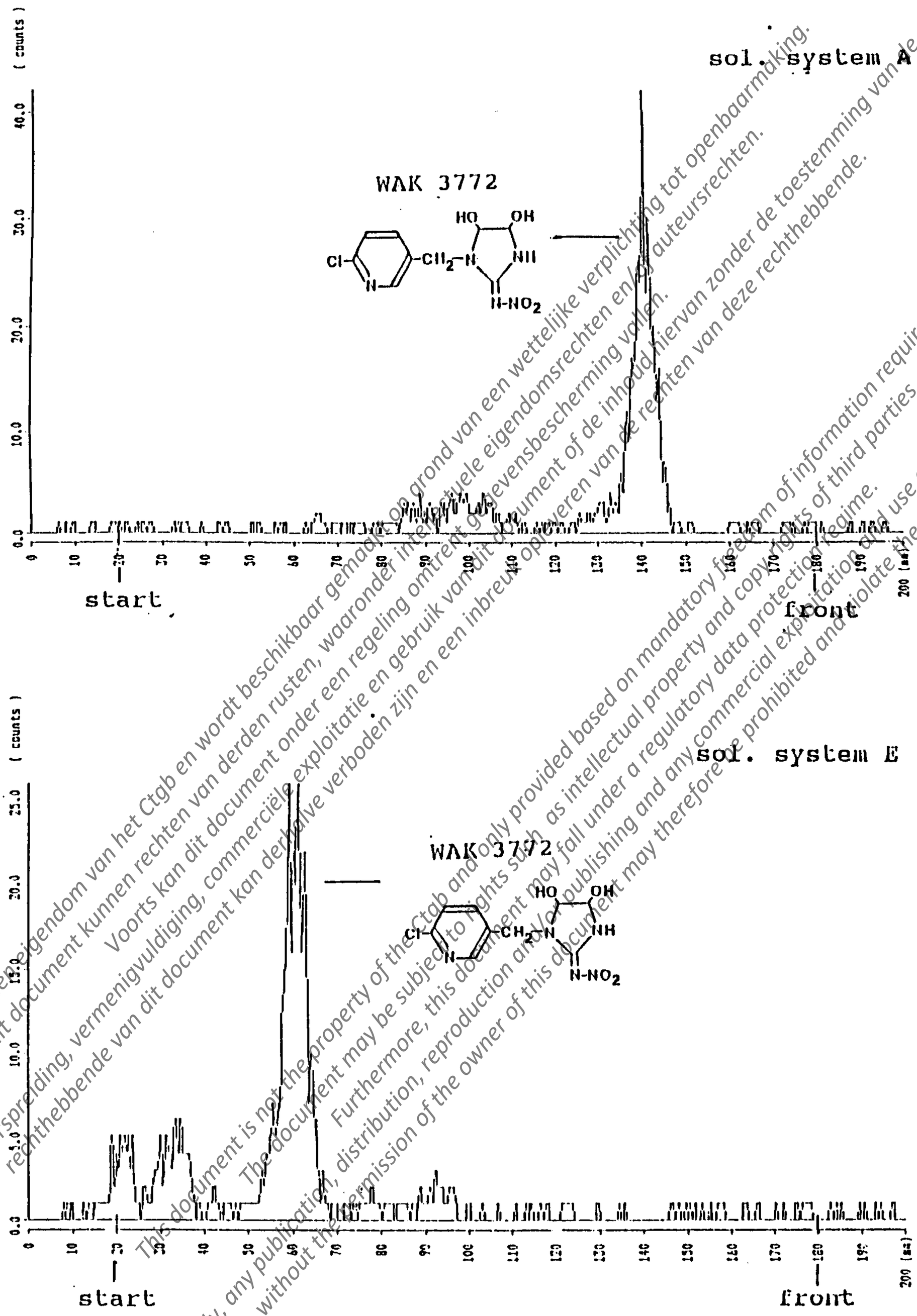

Fig. 23 TLC chromatograms of isolated metabolite (WAK 3772) (Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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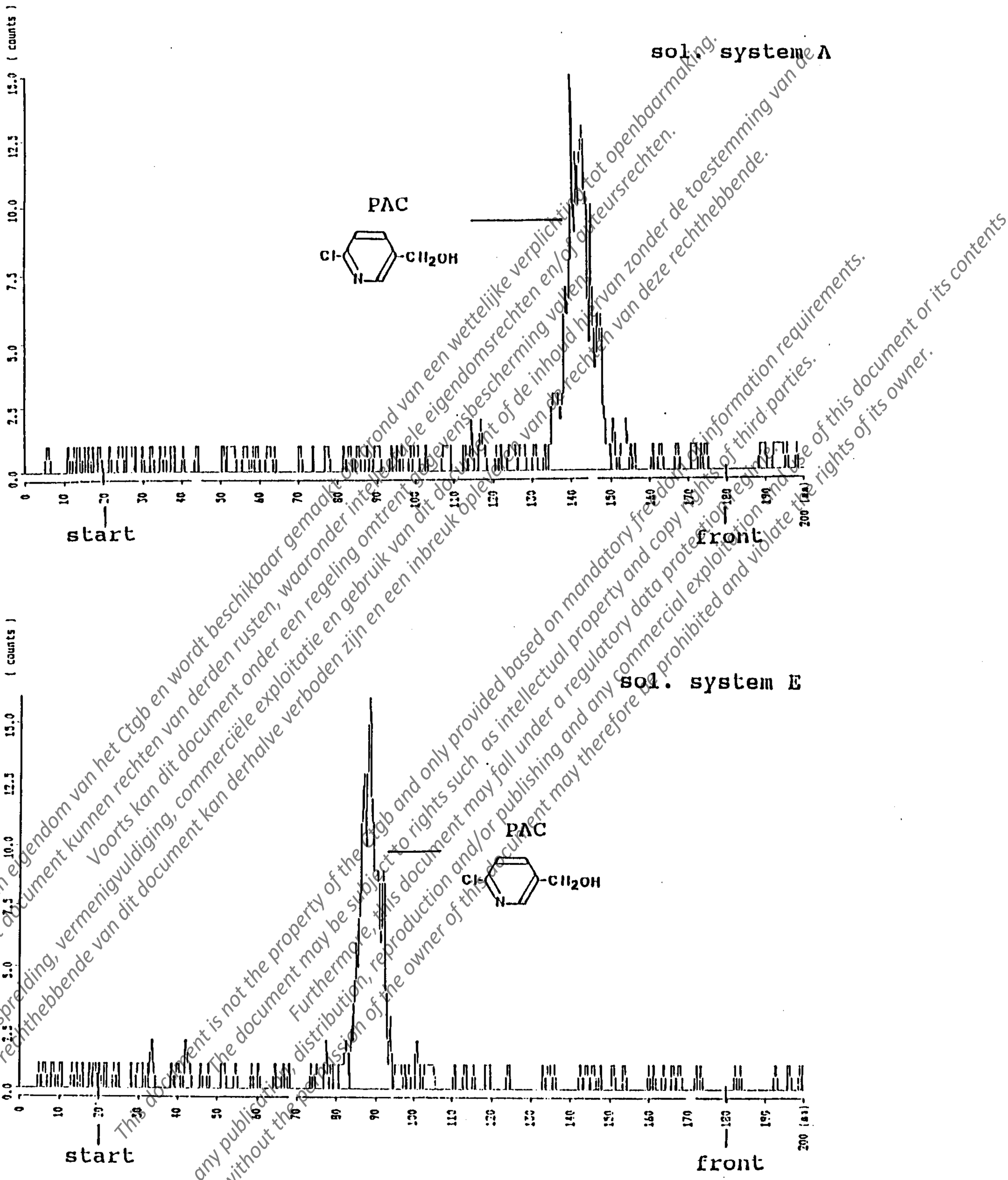

Fig. 24 TLC chromatograms of isolated metabolite (PAC) (Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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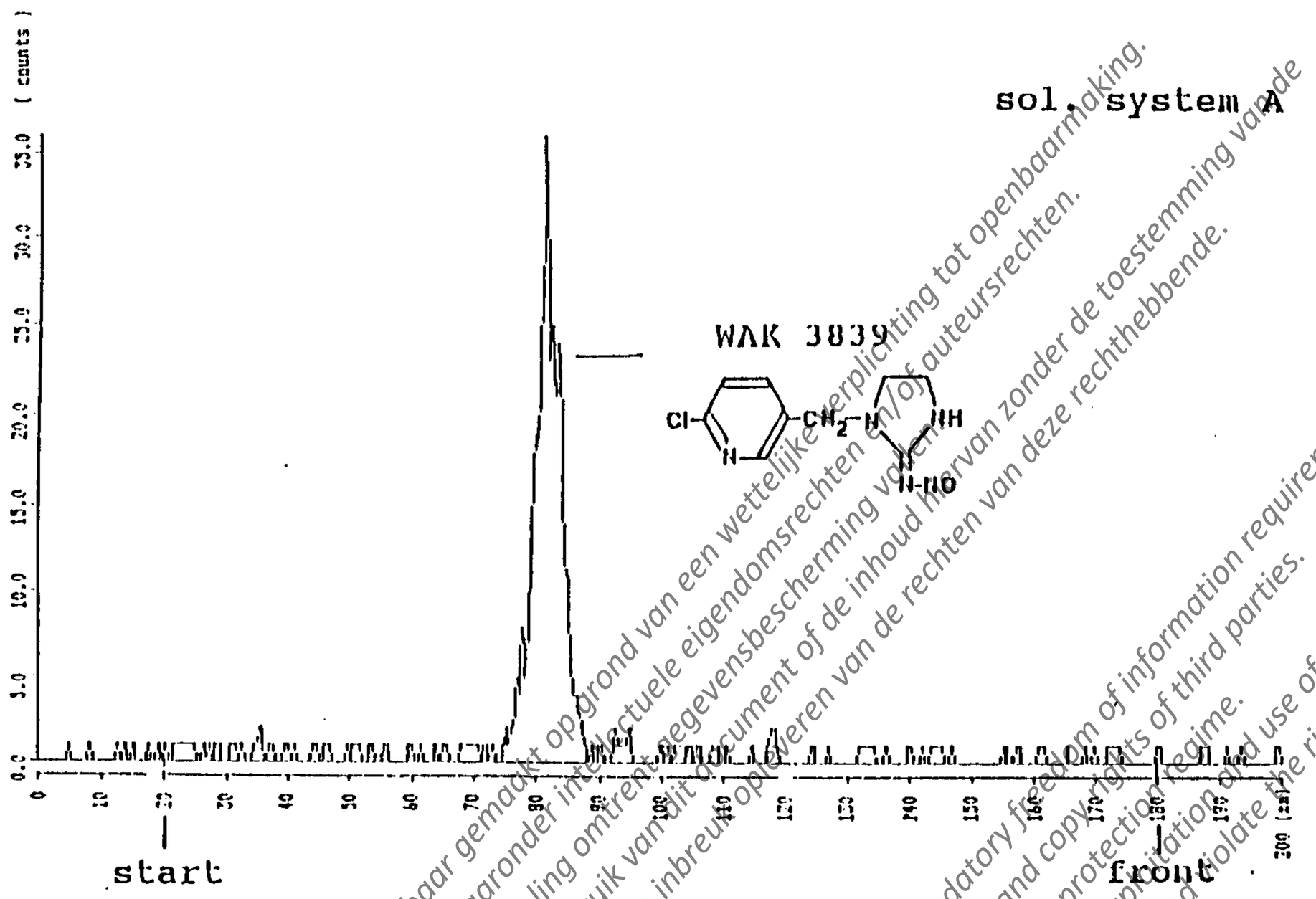

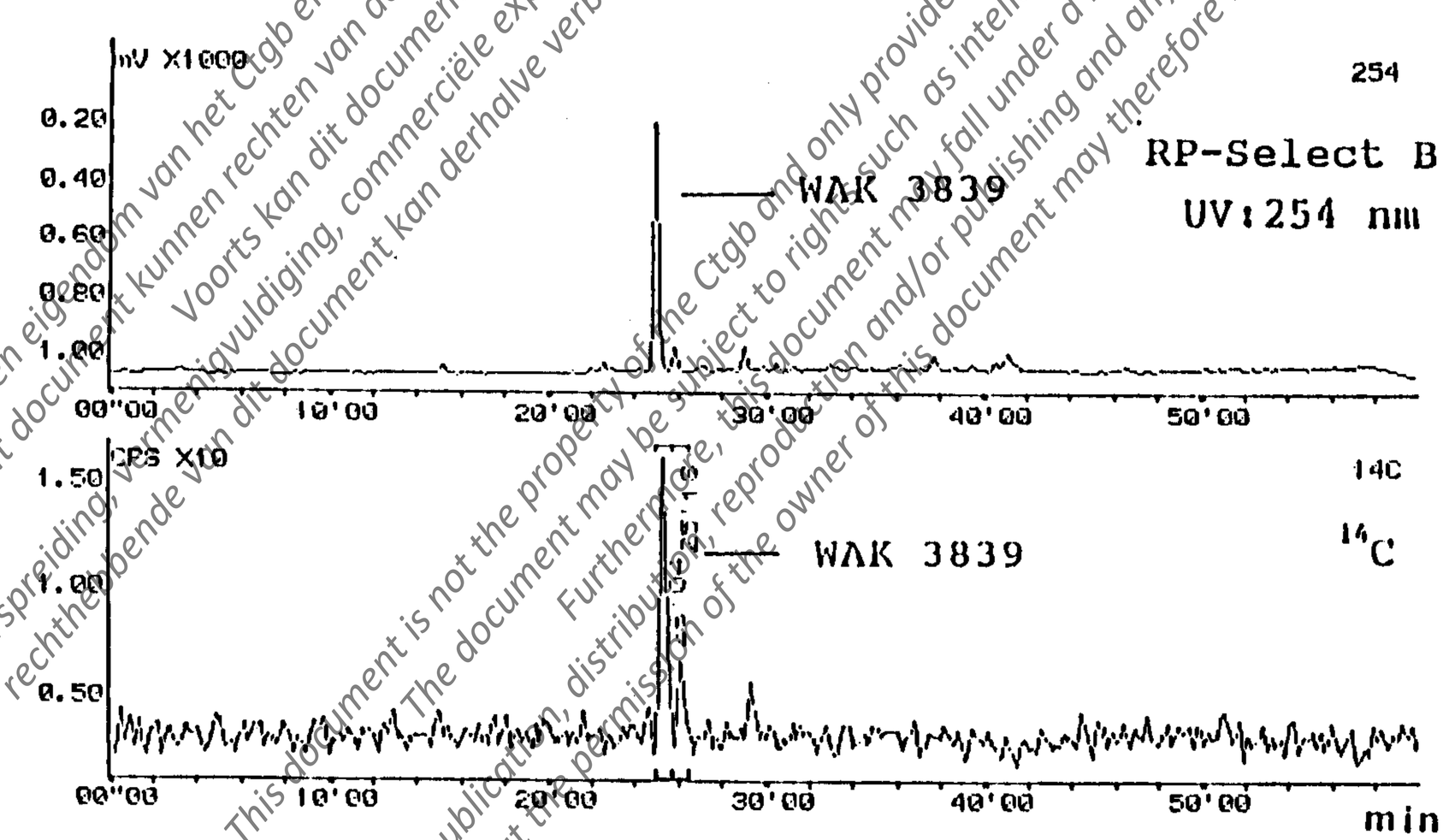

Fig. 25 TLC and HPLC chromatograms of isolated metabolite (WAK 3839)
(Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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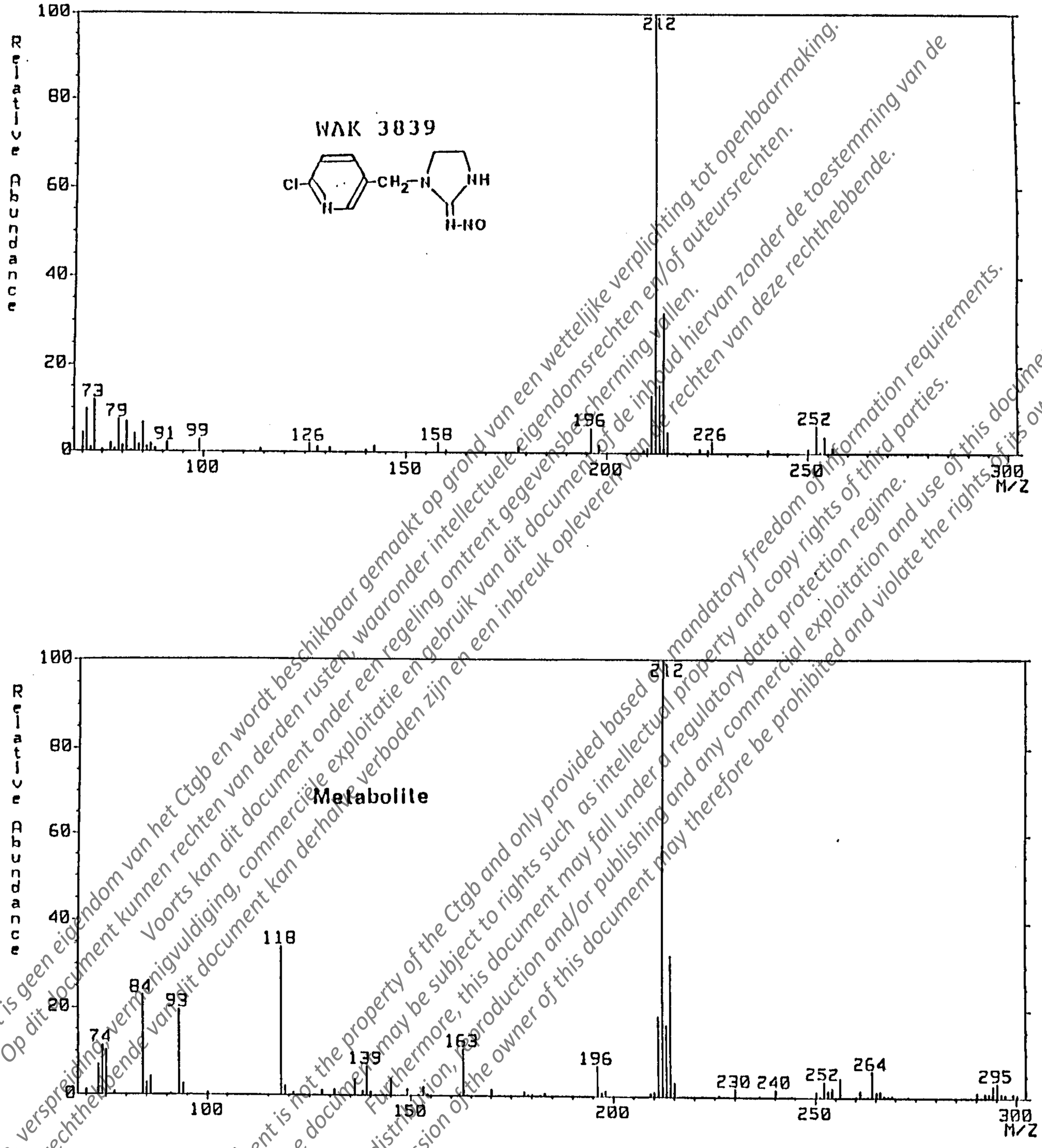

Fig. 26 CI(+)-Mass spectra of the reference WAK 3839 (upper) and isolated metabolite (lower) (Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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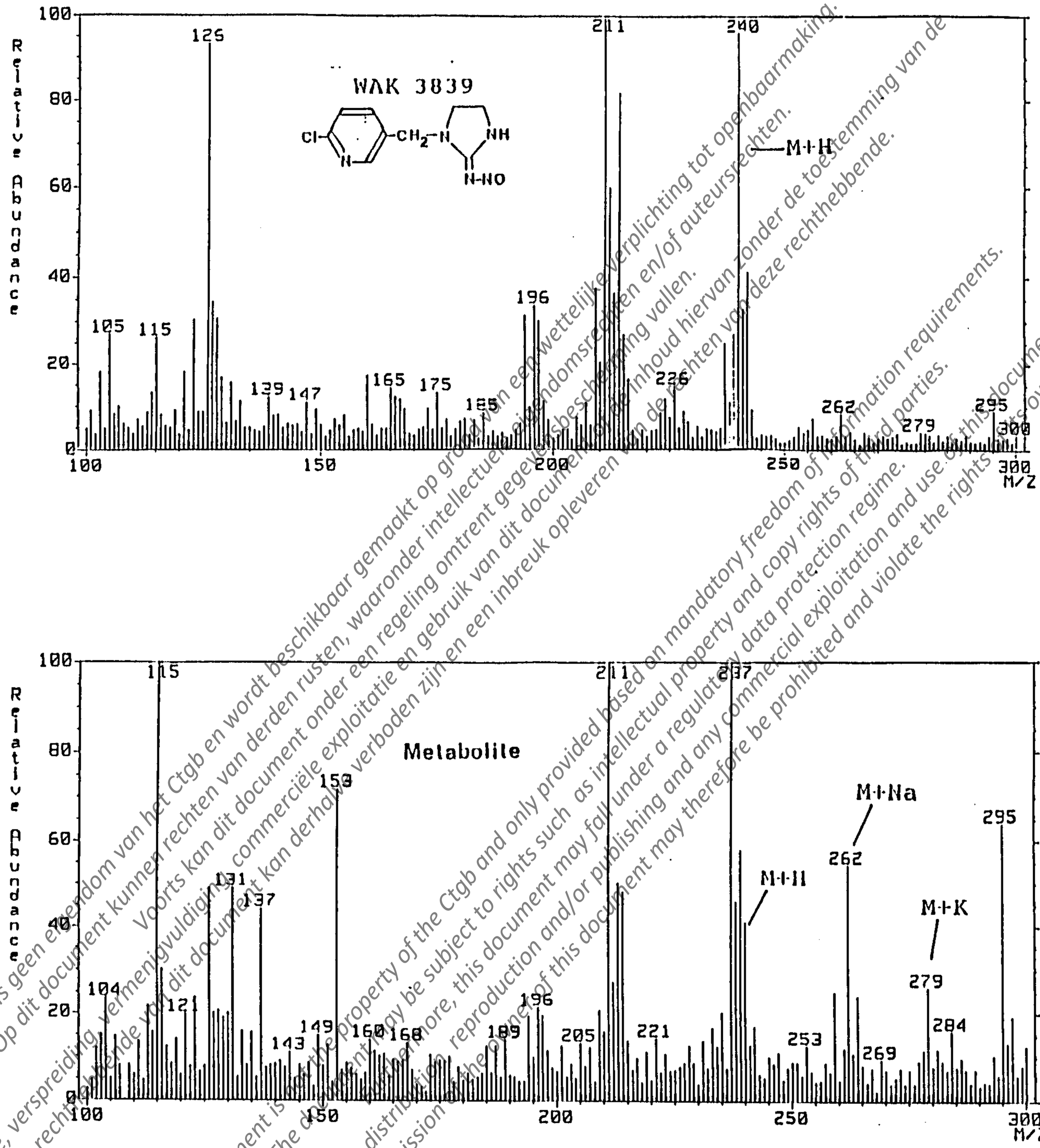

Fig. 27 FAB(+)-Mass spectra of the reference WAK 3839 (upper) and isolated metabolite (lower) (Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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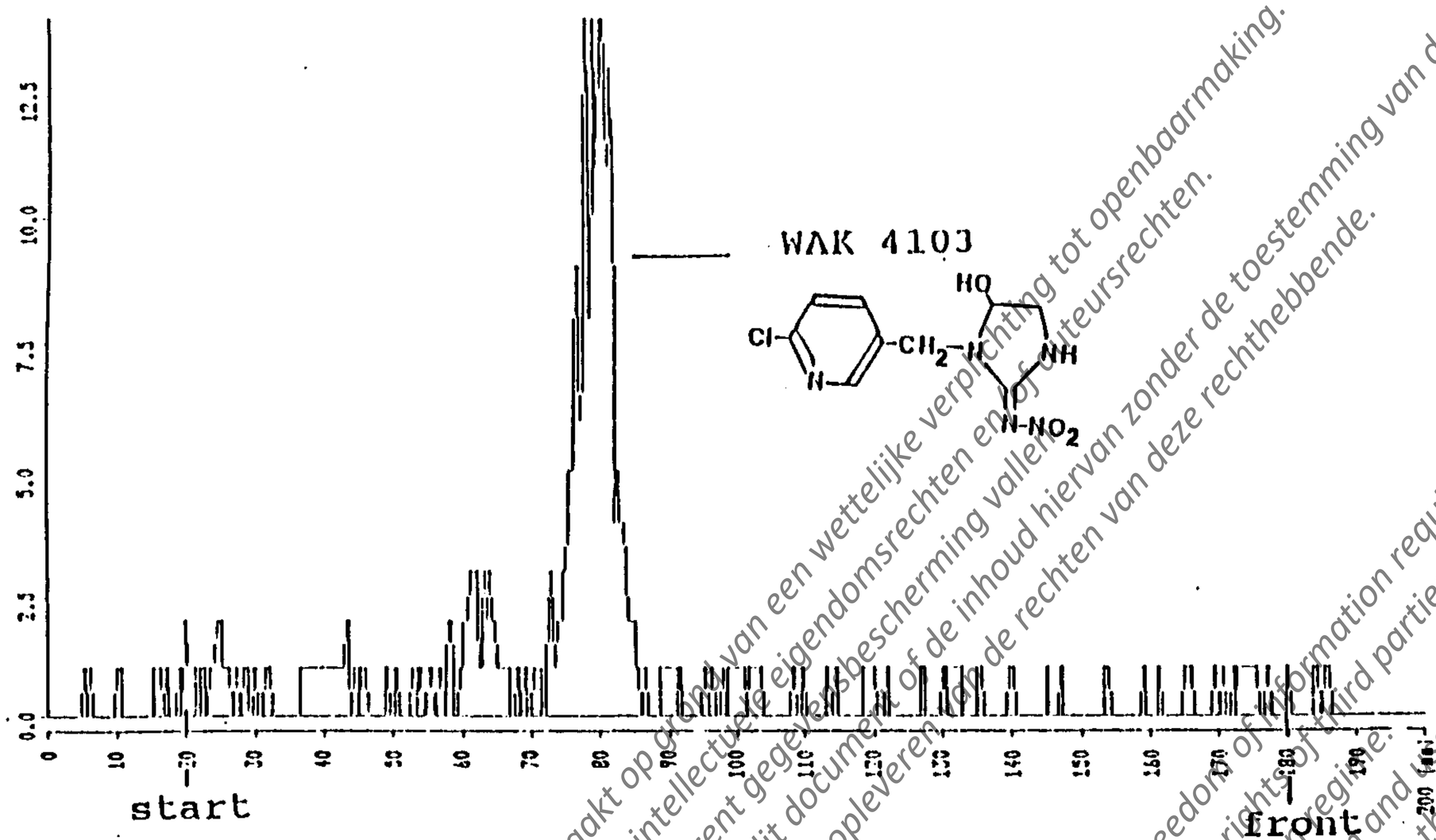

Fig. 28 TLC and HPLC chromatograms of isolated metabolite (WAK 4103)
(Plant ID : # 11 to 16, dichloromethane fraction of aerial part, DAT : 69)

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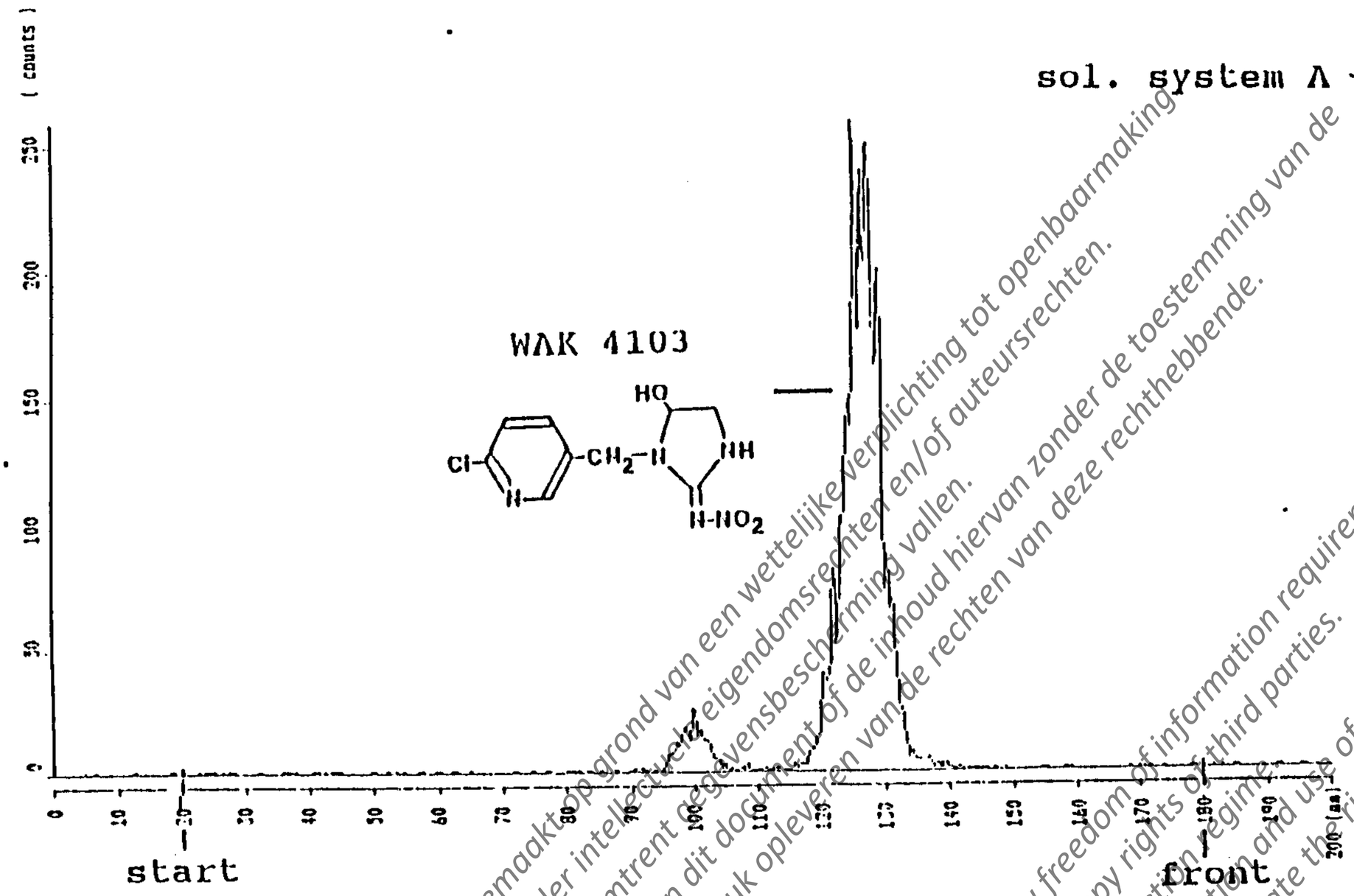

Fig. 29 TLC and HPLC chromatograms of isolated metabolite (WAK 4103)
(Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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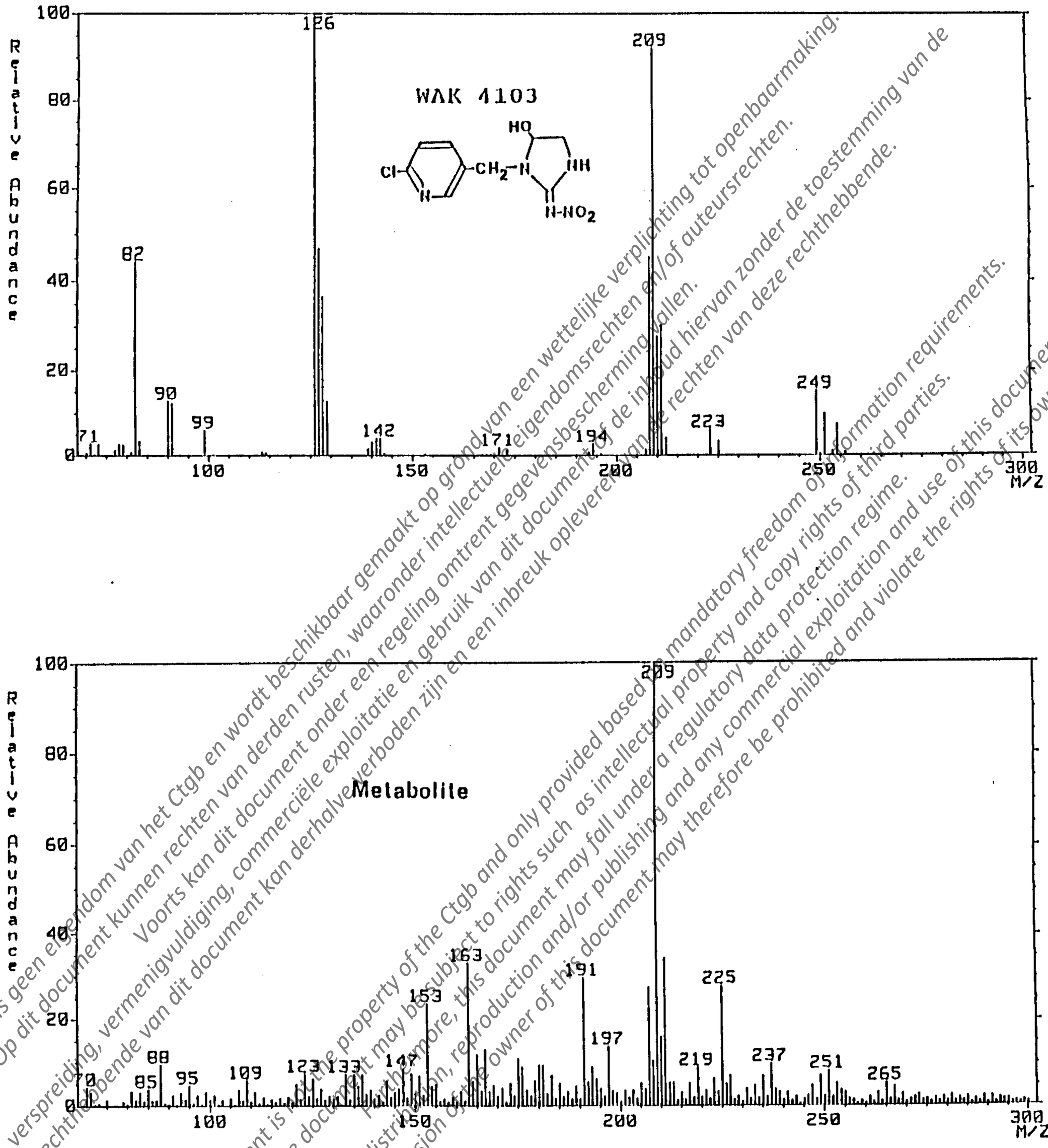

Fig. 30 CI(+)-Mass spectra of the reference WAK 4103 (upper) and isolated metabolite (lower) (Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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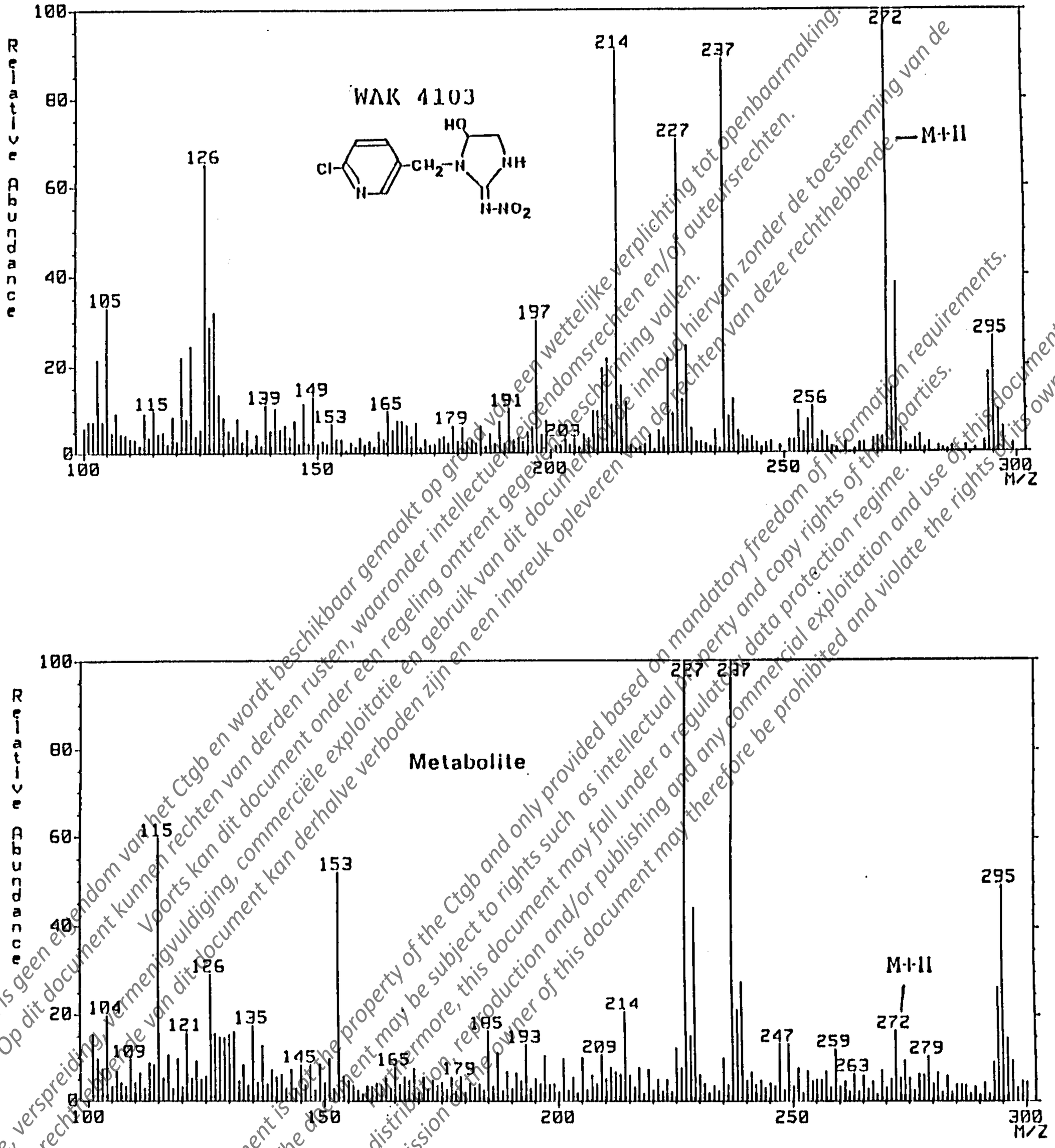

Fig. 31 FAB(+)-Mass spectra of the reference WAK 4103 (upper) and isolated metabolite (lower)
(Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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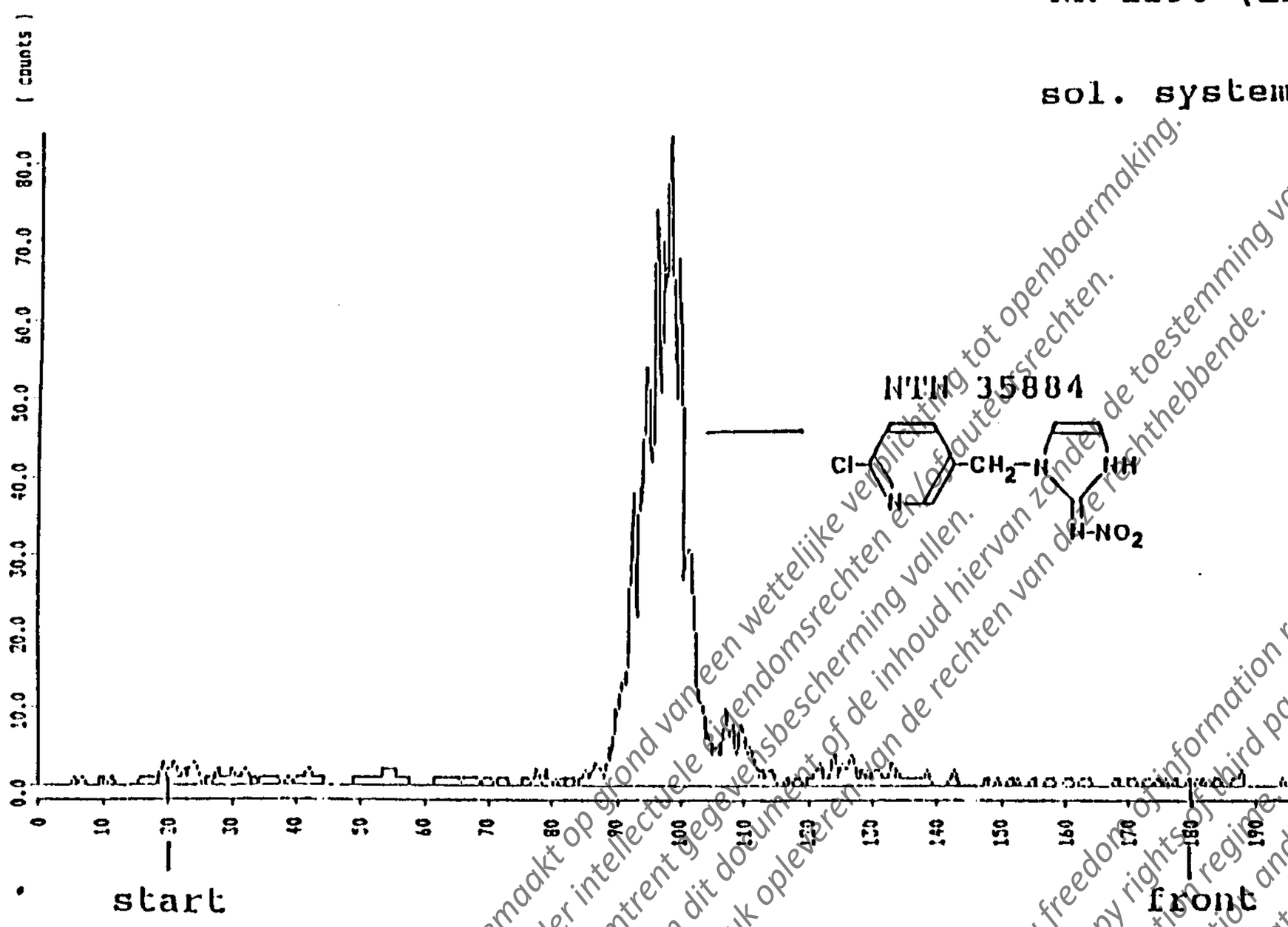

Fig. 32 TLC and HPLC chromatograms of isolated metabolite (NTN 35884)
(Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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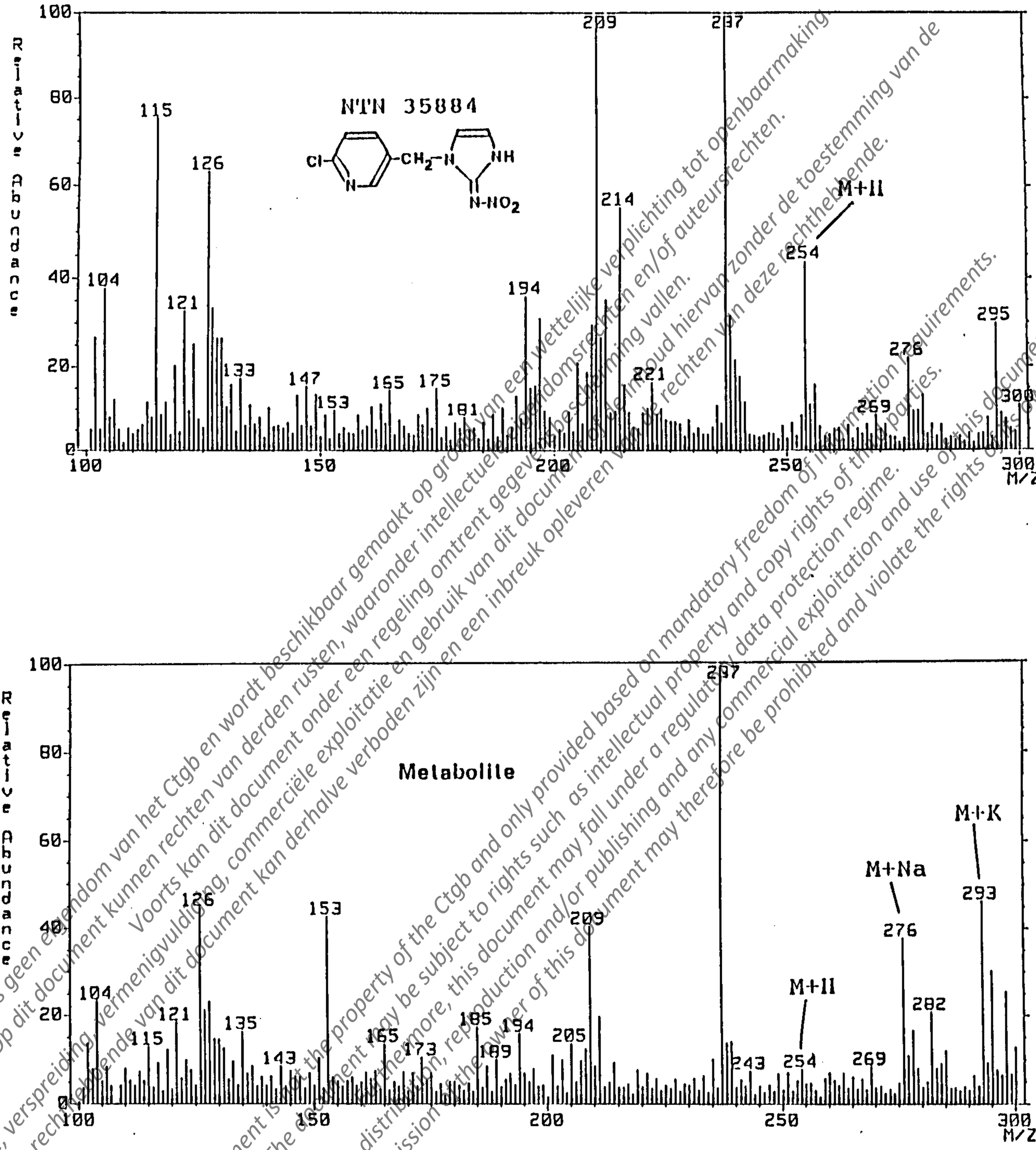

Fig. 33 FAB(+)-Mass spectra of the reference NTN 35884 (upper) and isolated metabolite (lower)
(Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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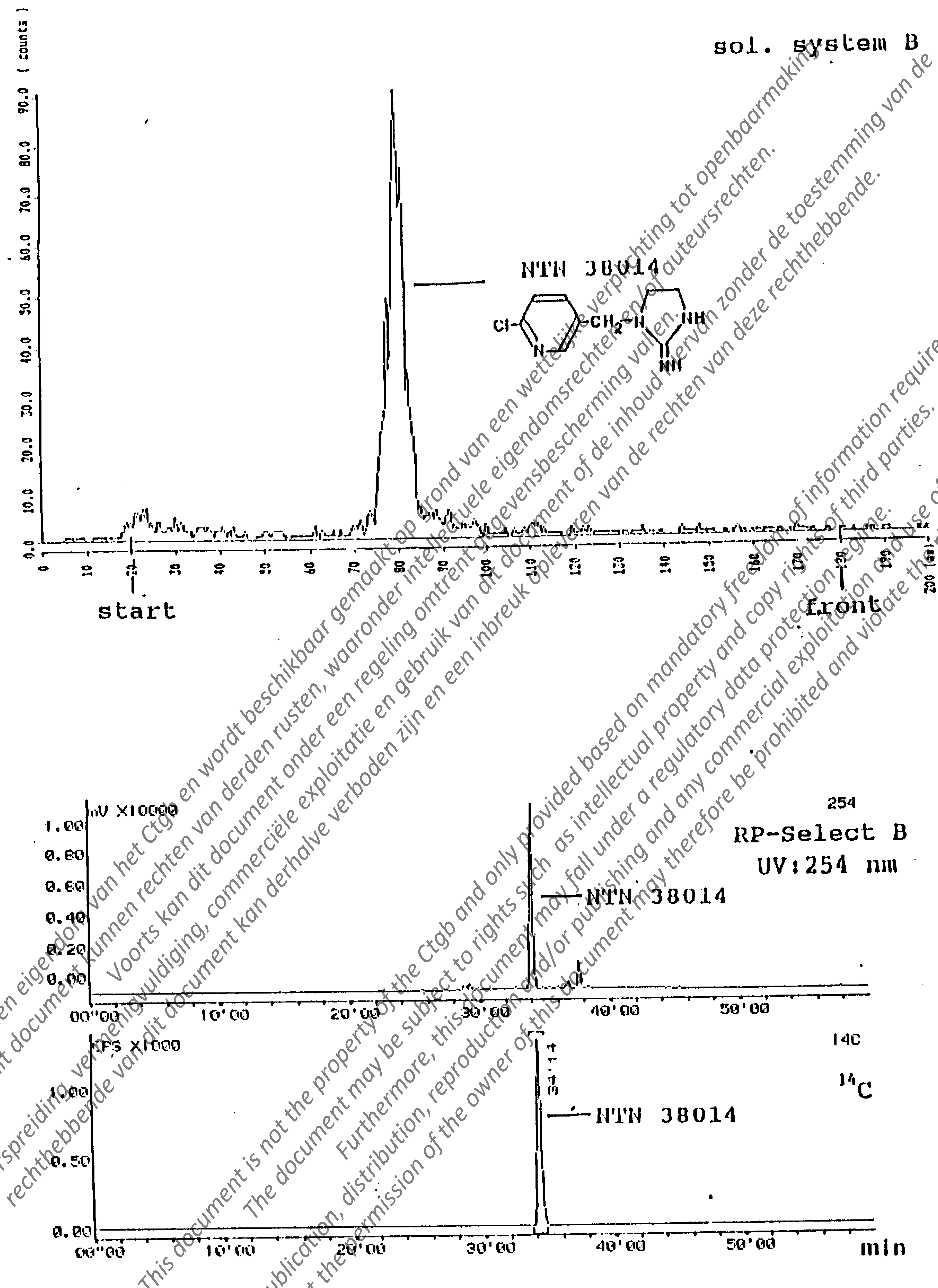

Fig. 34 TLC and HPLC chromatograms of isolated metabolite (NTN 38014)
(Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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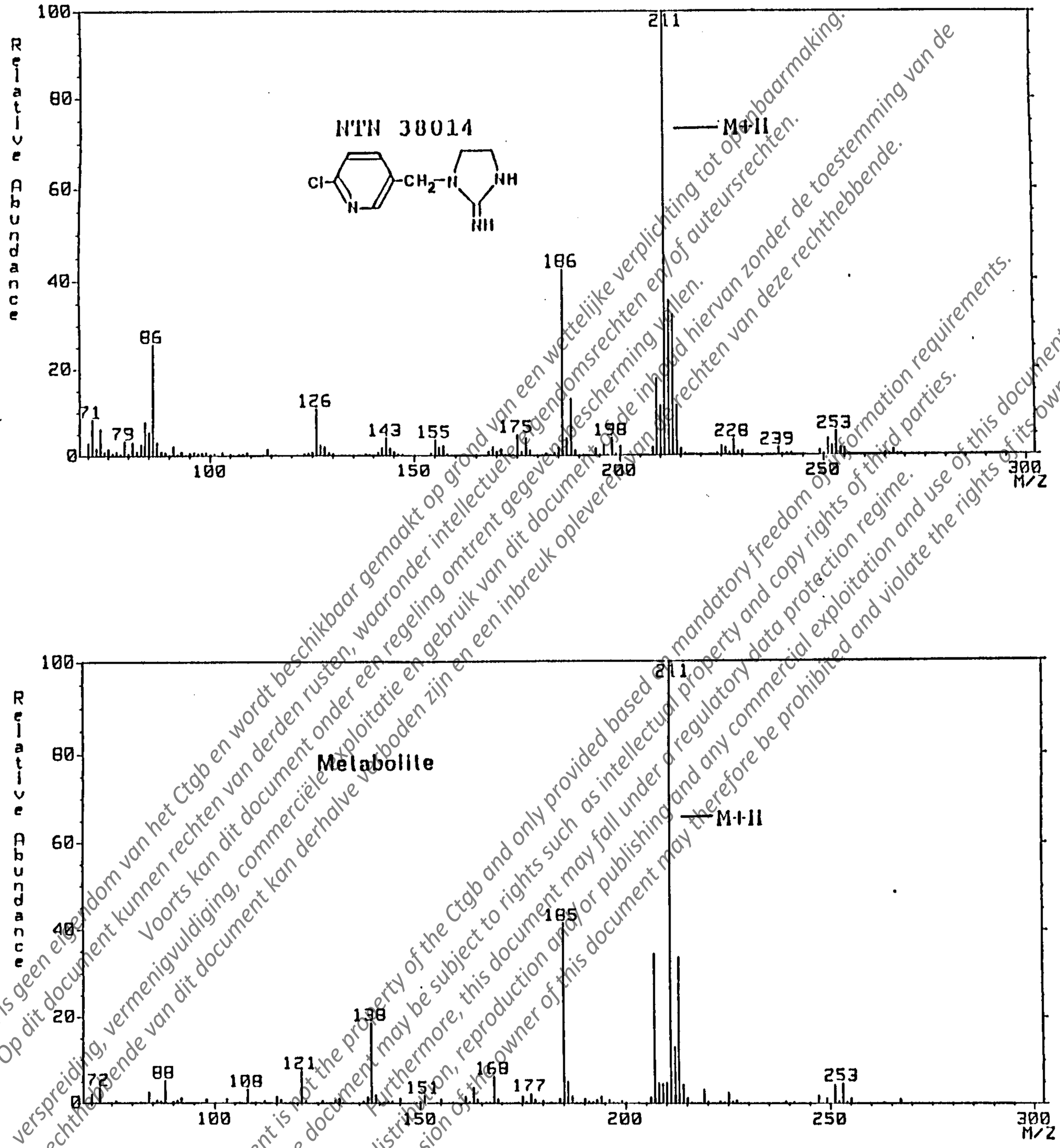

Fig. 35 CI(+)-Mass spectra of the reference NTN 38014 (upper) and isolated metabolite (lower) (Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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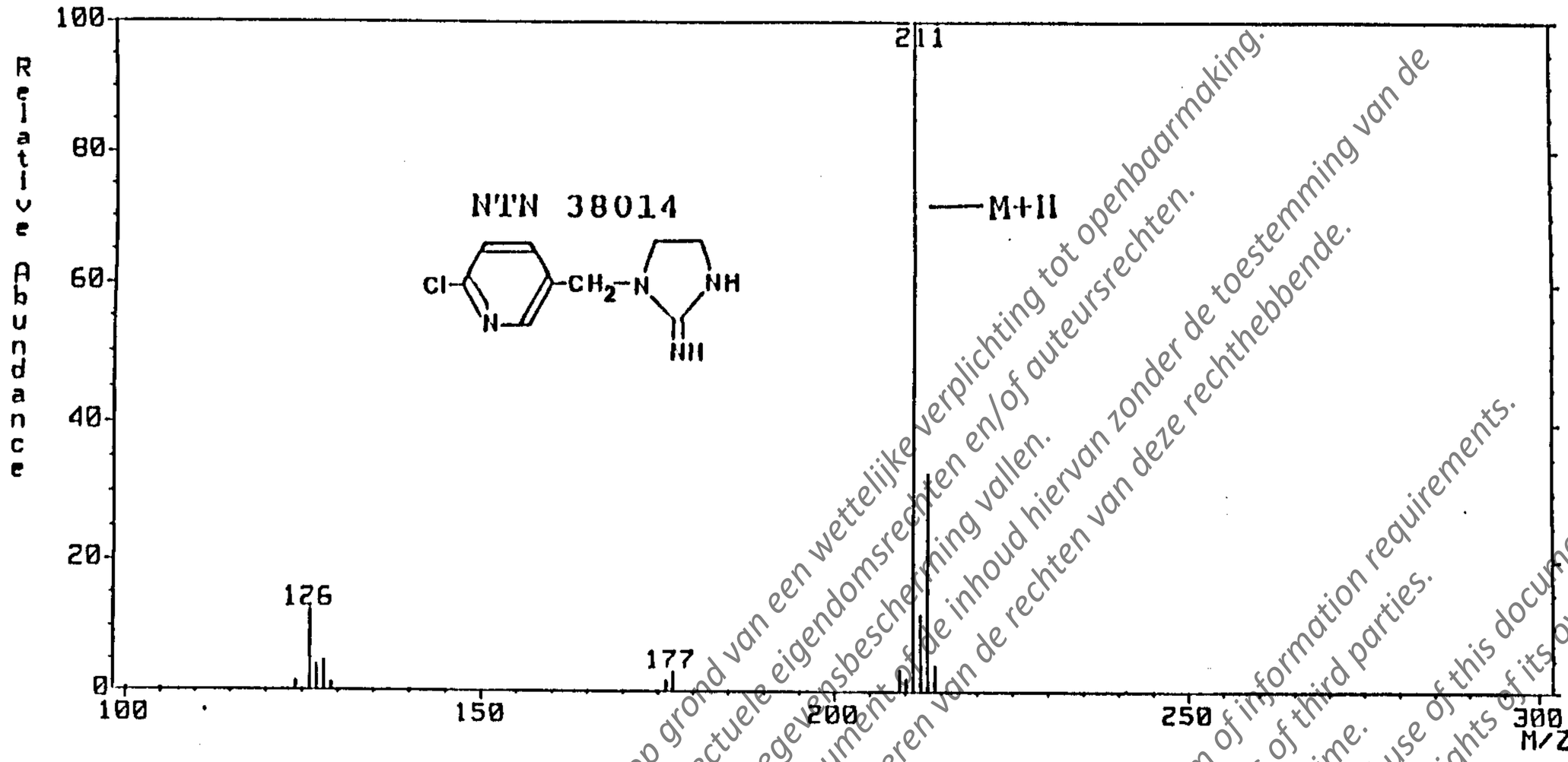

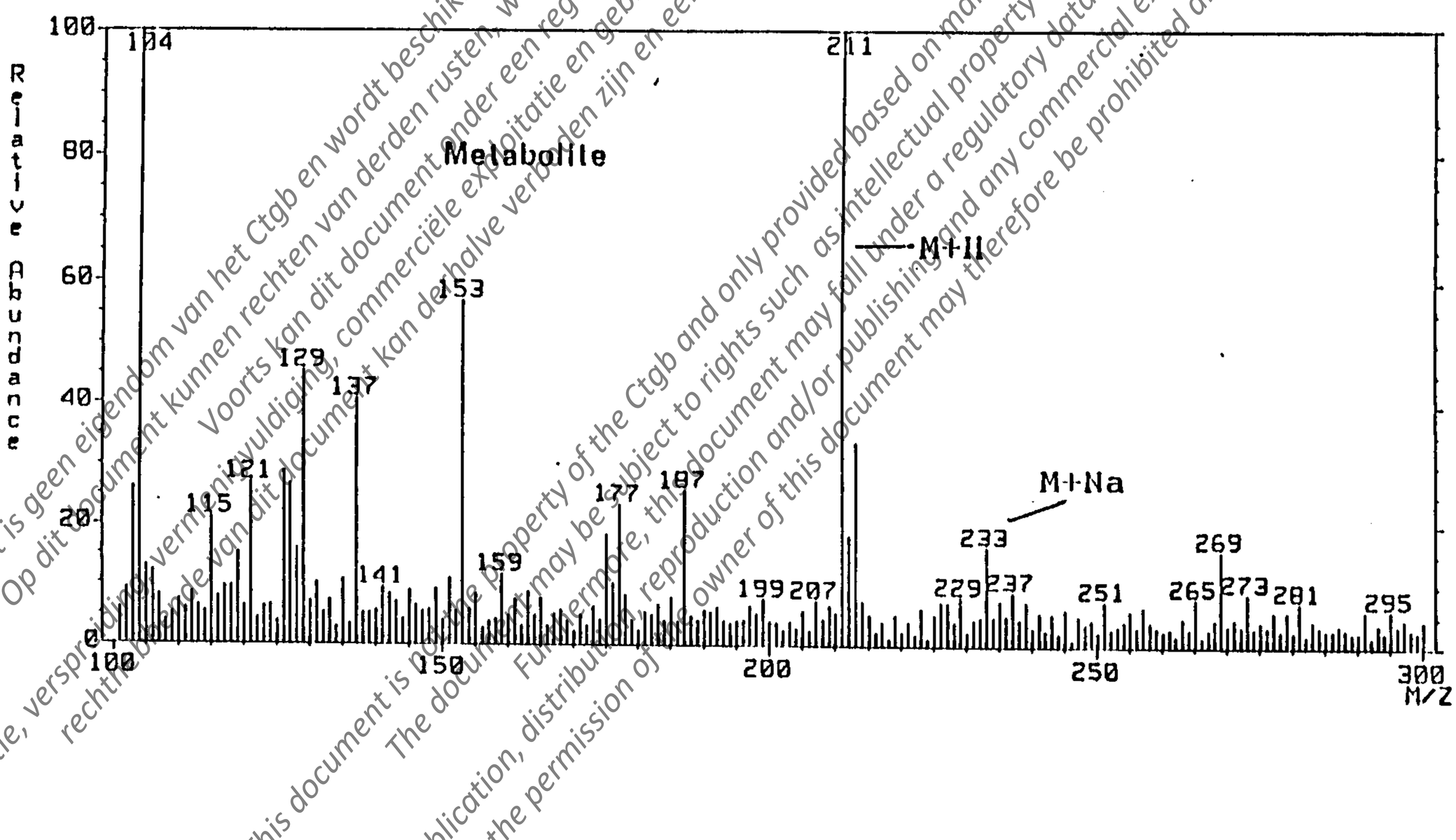

Fig. 36 FAB(+)-Mass spectra of the reference NTN 38014 (upper) and isolated metabolite (lower)
(Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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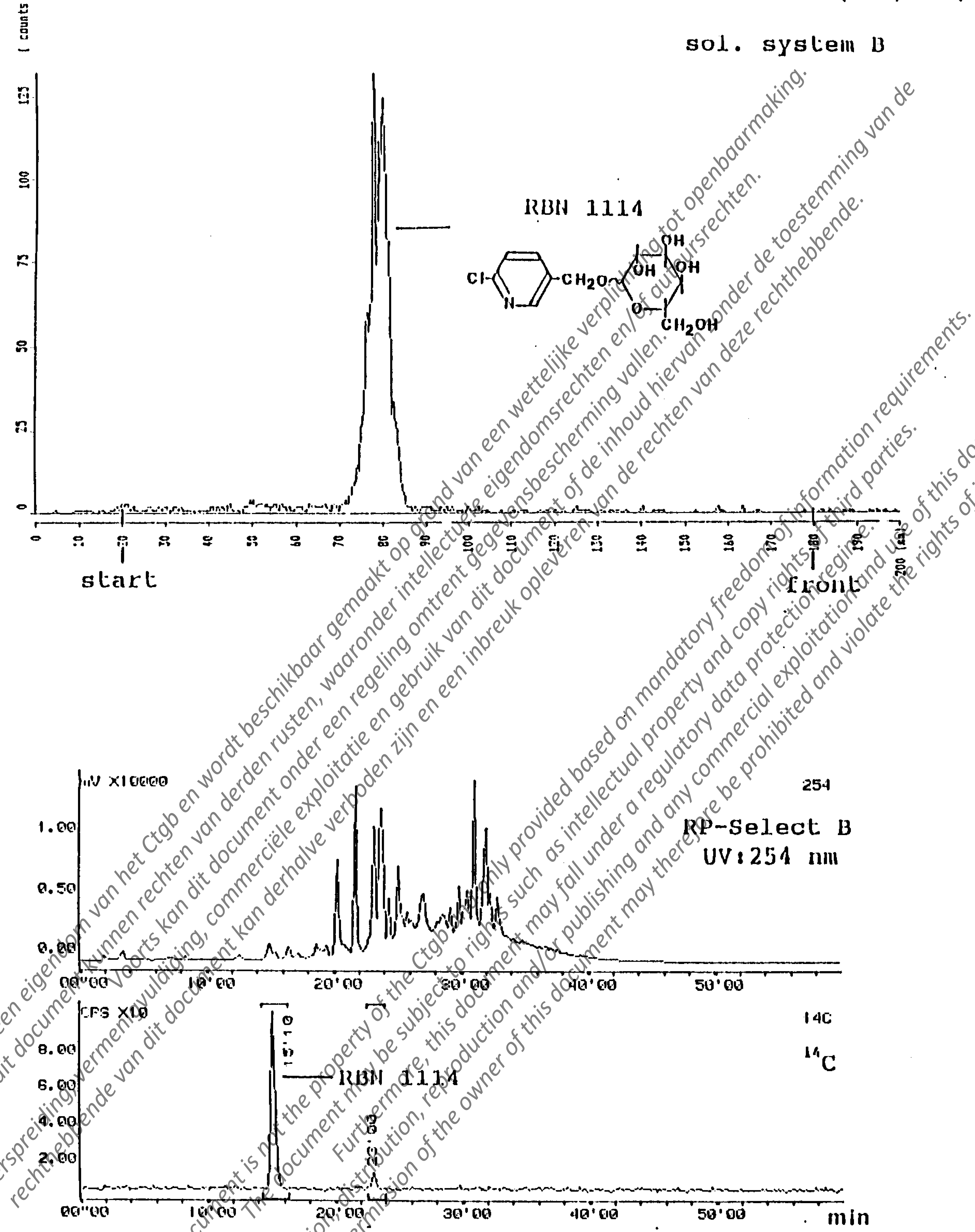

Fig. 37 TLC and HPLC chromatograms of isolated metabolite (RBN 1114)
(Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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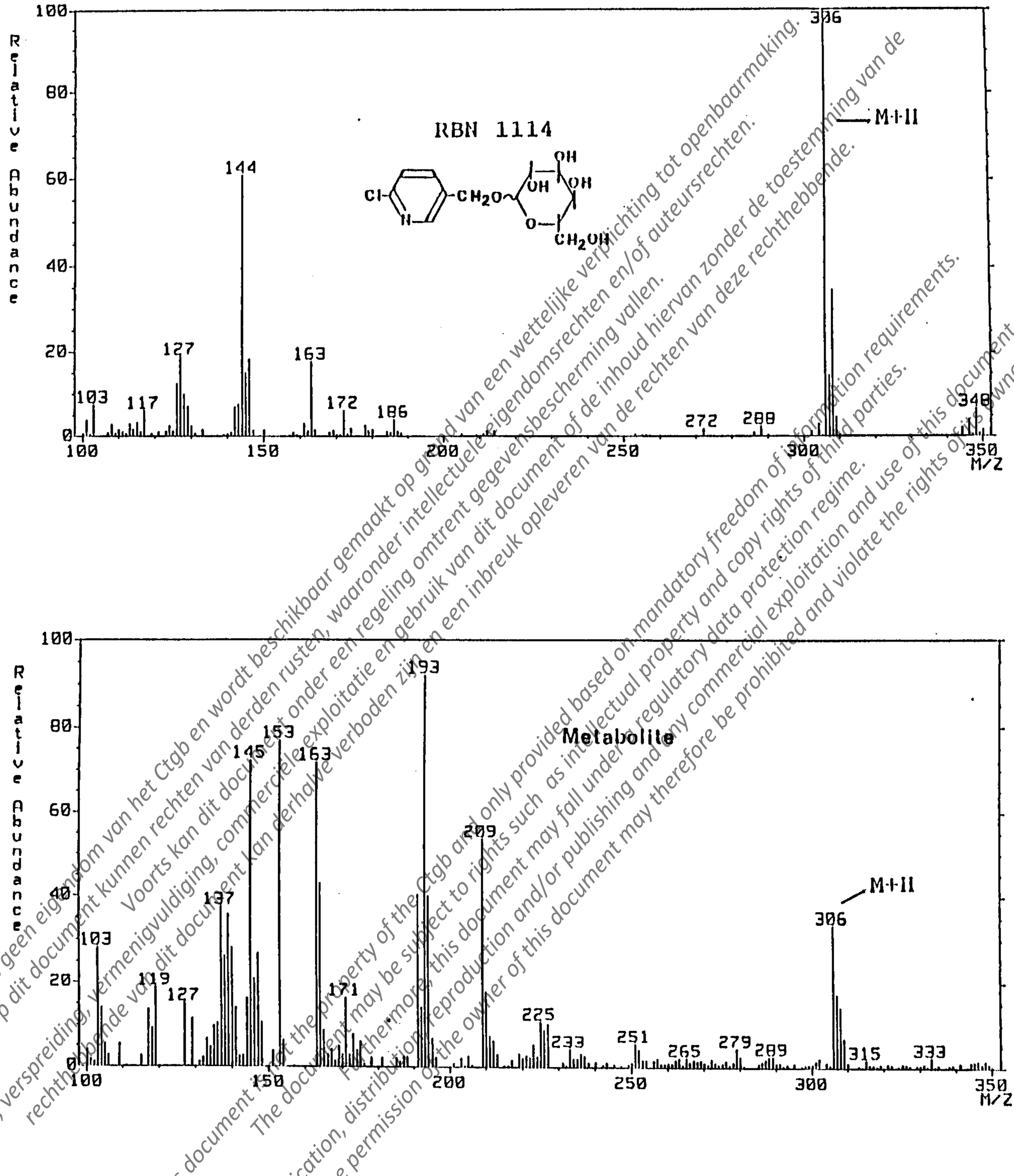

Fig. 38 CI(+)-Mass spectra of the reference RBN 1114 (upper) and isolated metabolite (lower) (Plant ID : # 11 to 16, aqueous fraction of aerial part, DAT : 69)

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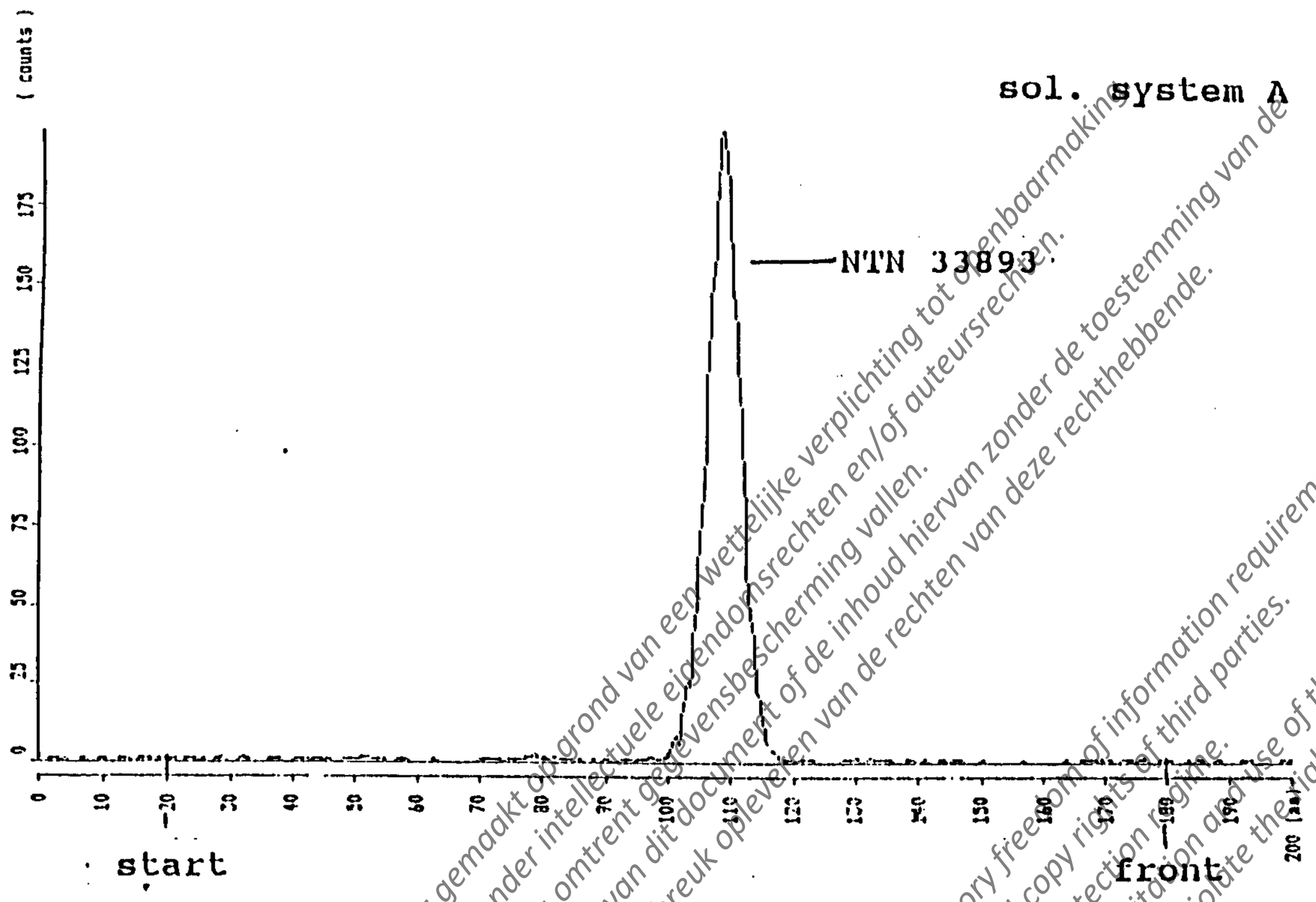

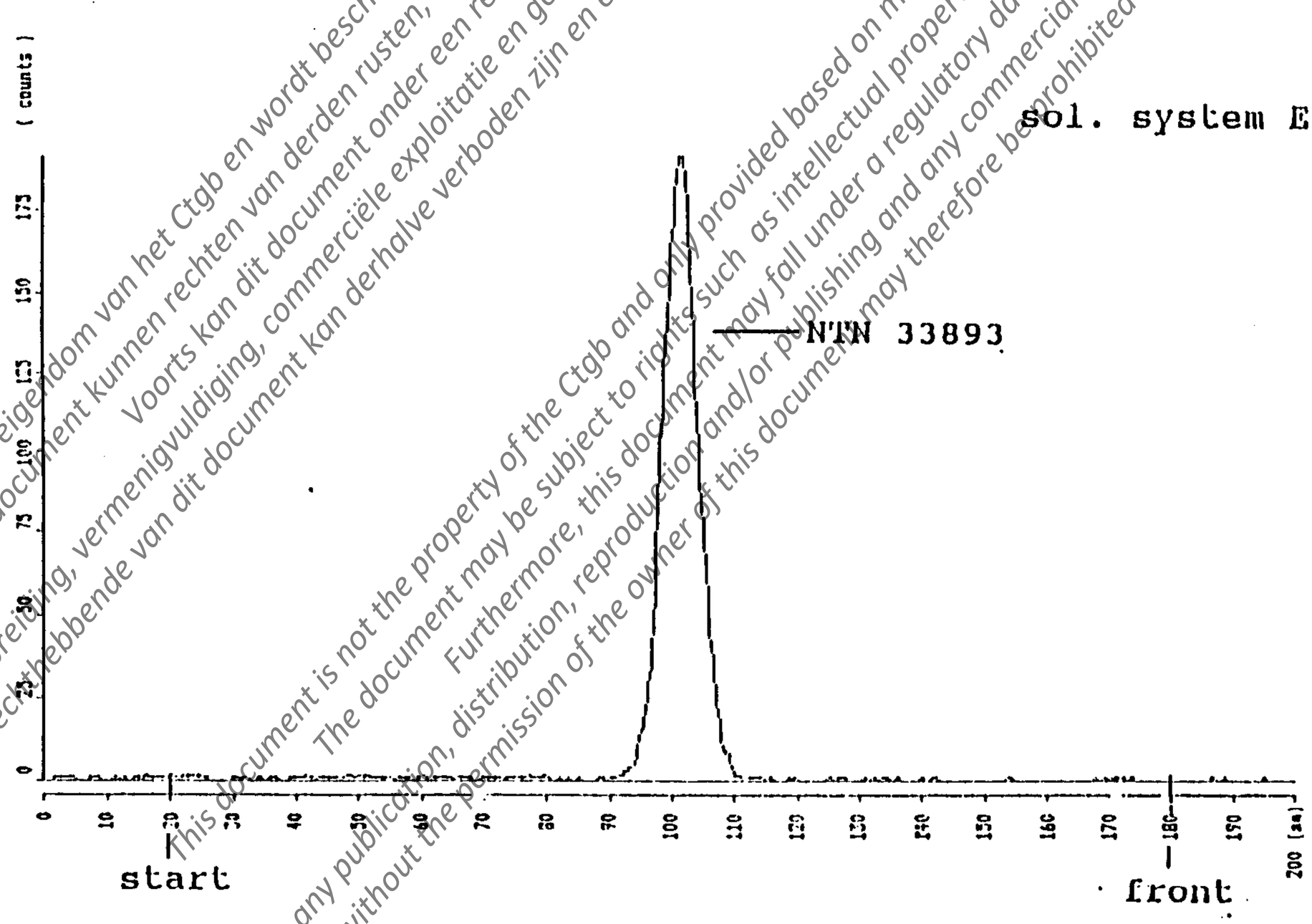

Fig. 39 TLC chromatograms of soil extracts
(Plant ID : # 2, soil, DAT : 14)

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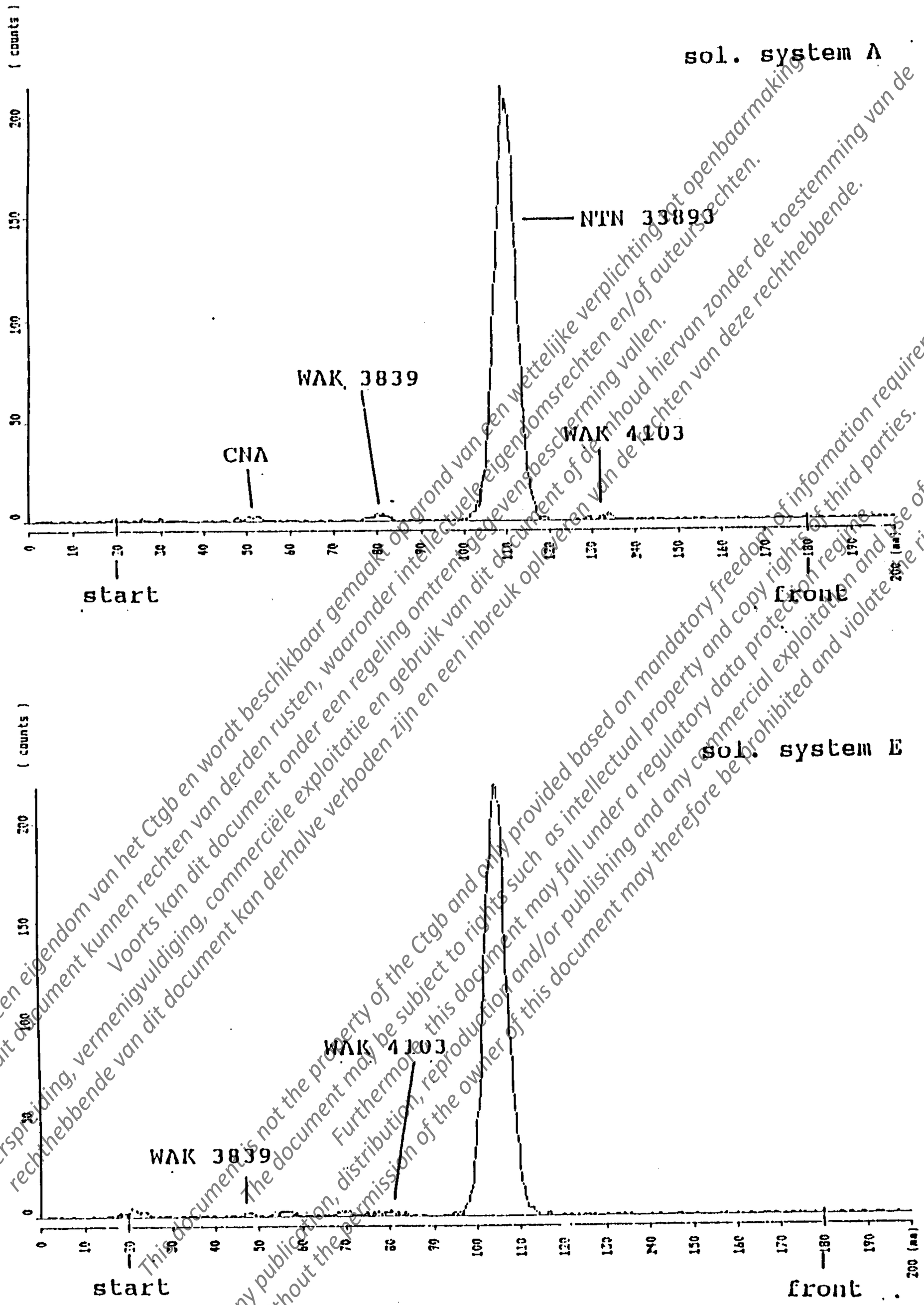

Fig. 40 TLC chromatograms of soil extracts
(Plant ID : # 4, soil, DAT : 35)

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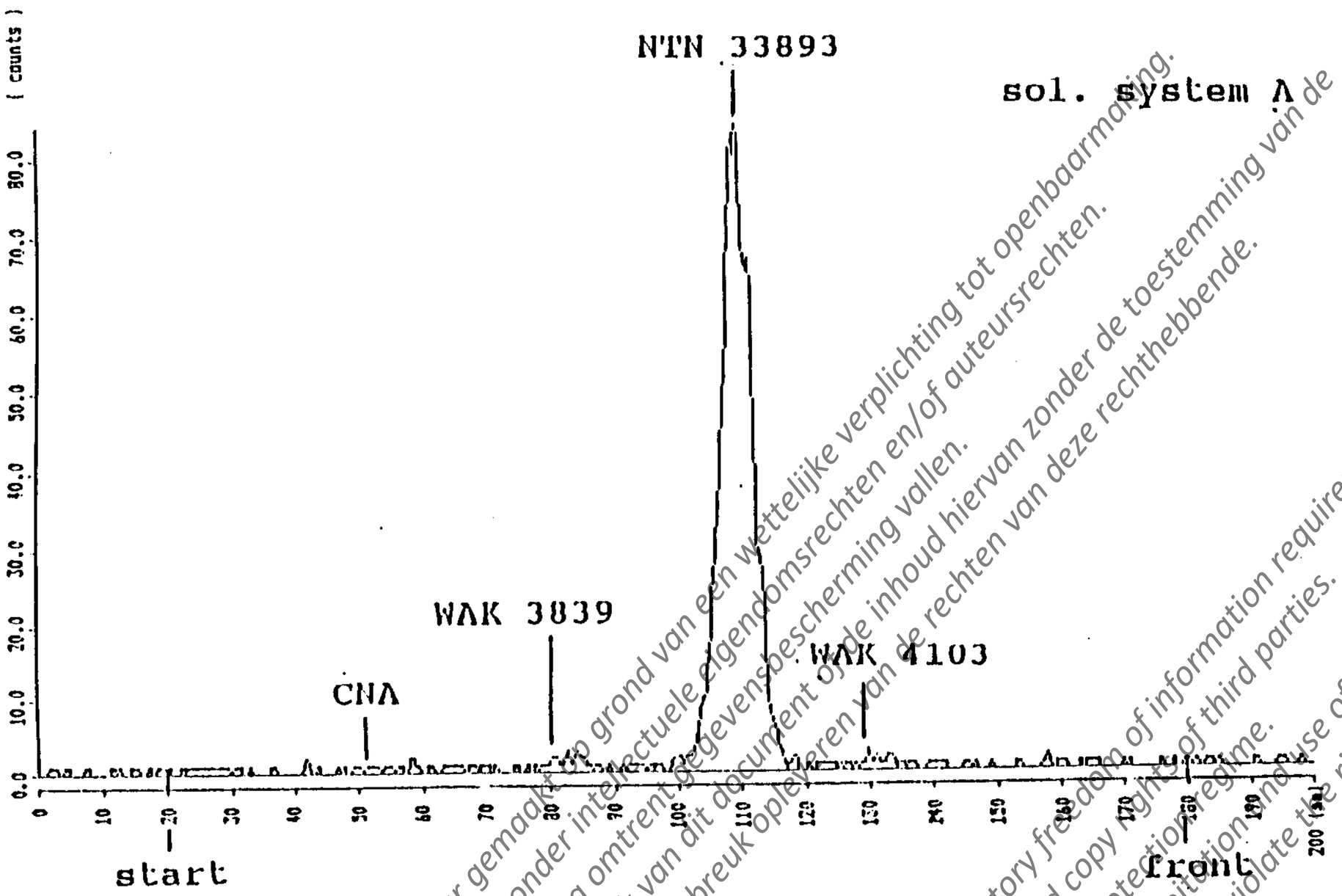

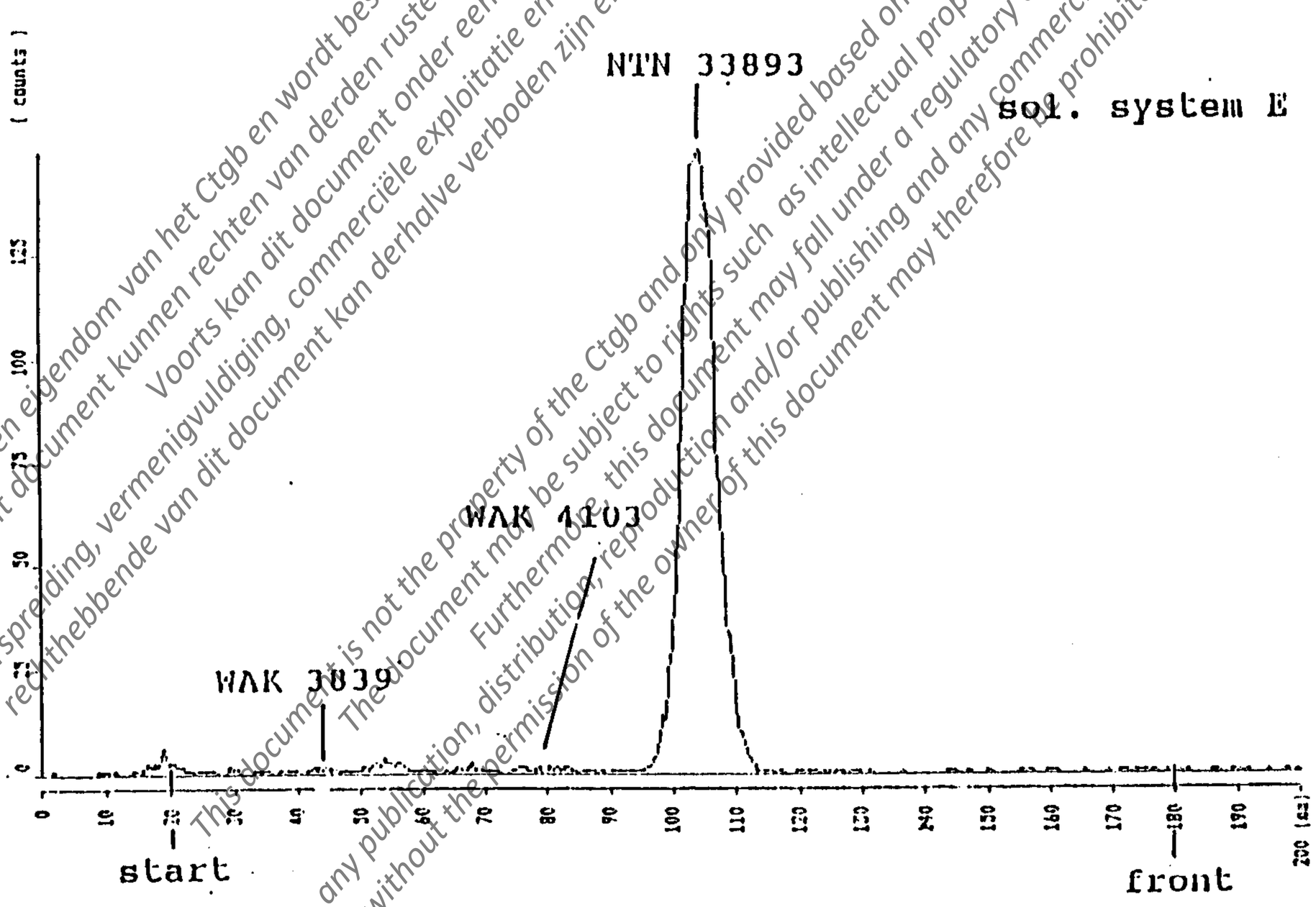

Fig. 41 TLC chromatograms of soil extracts
(Plant ID : # 6, soil, DAT : 69)

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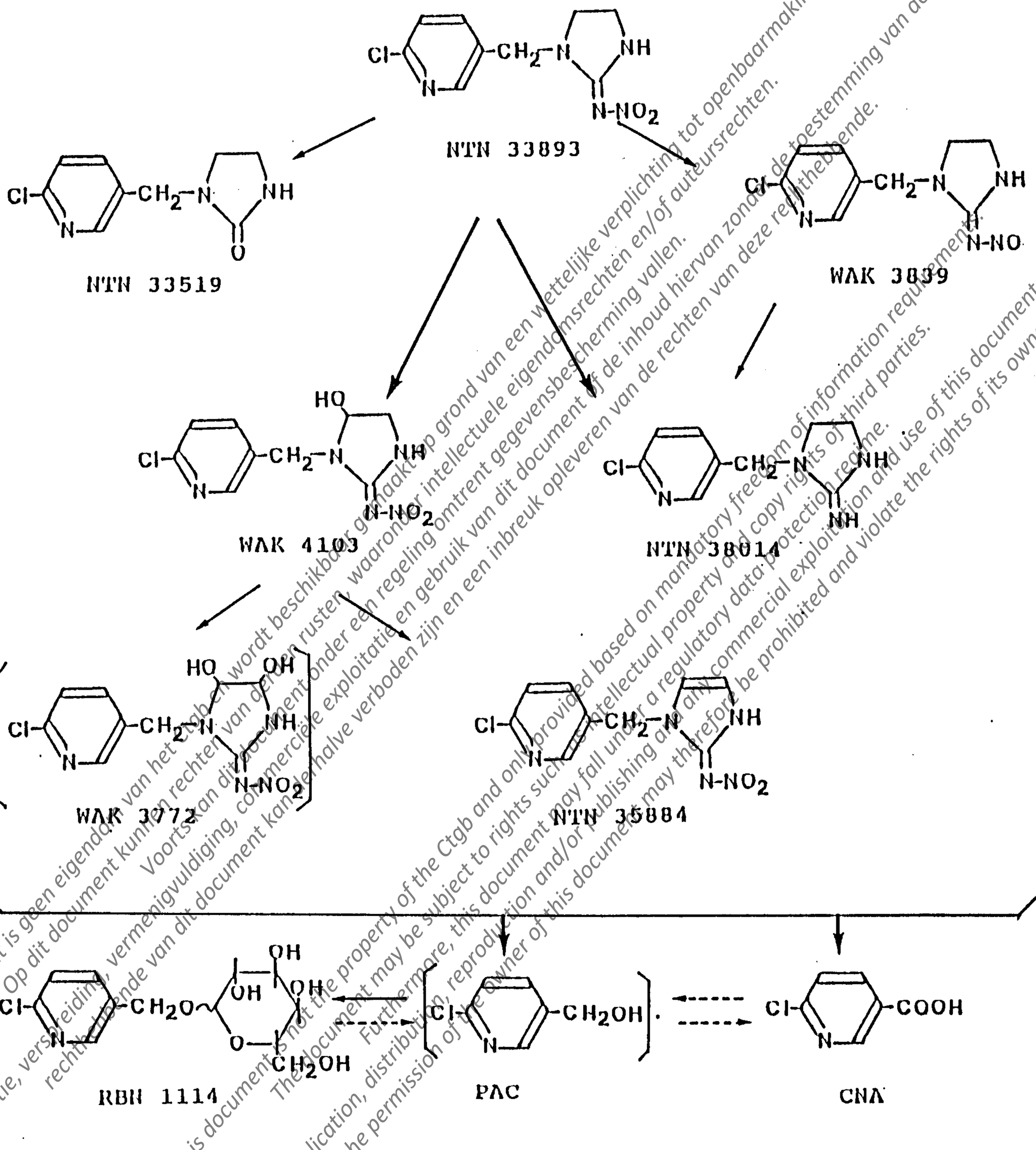

Fig. 42 Proposed metabolic pathways of the NTN 33893 in eggplant

[ ] : WAK 3772 and PAC were found only in the samples applied excessive amount of the NTN 33893

----> : Tentative pathway

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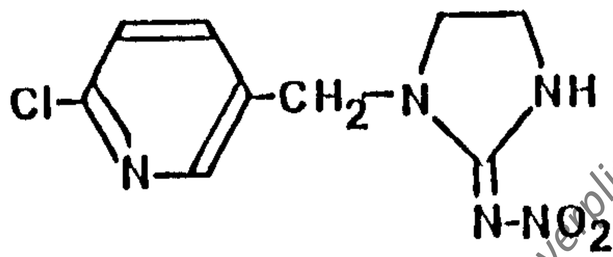
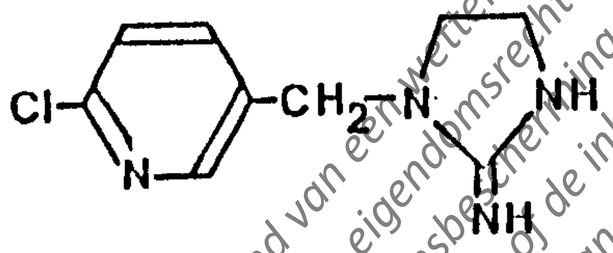
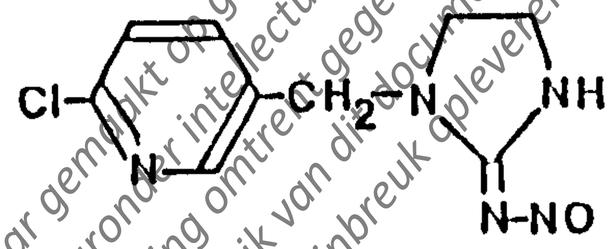
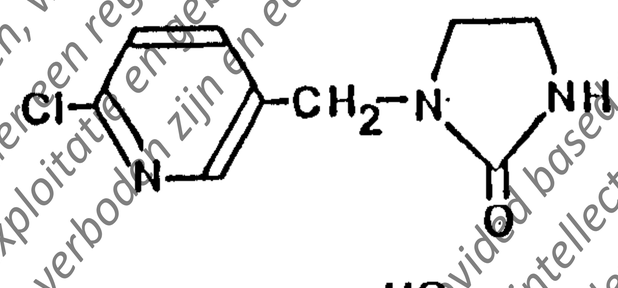
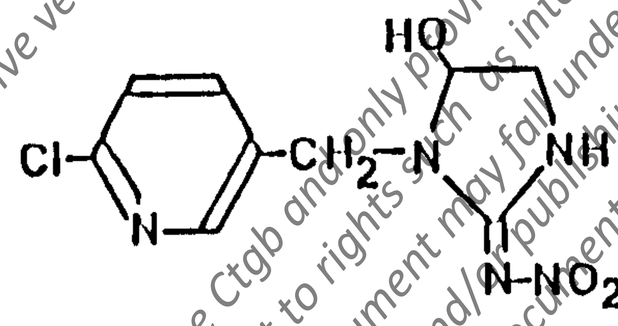
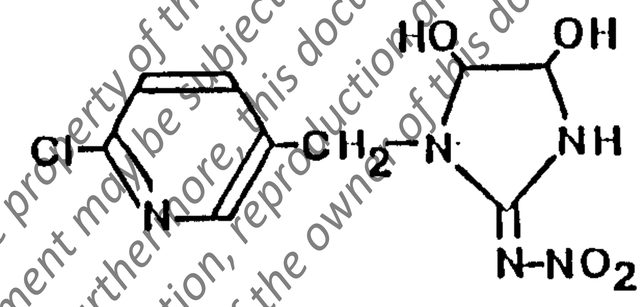
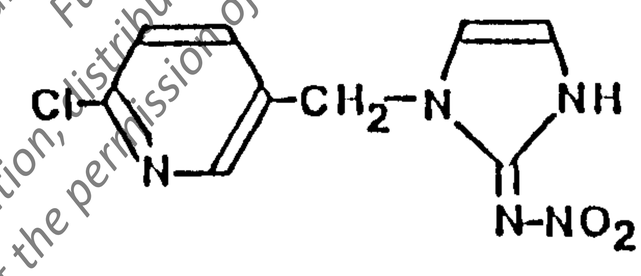
Appendix I Structural information of the NTN 33893 and reference substances (1)

Substances	Structure	Mol. weight
NTN 33893		255
NTN 38014		210
WAK 3839		239
NTN 33519		211
WAK 4103		271
WAK 3772		287
NTN 35884		253

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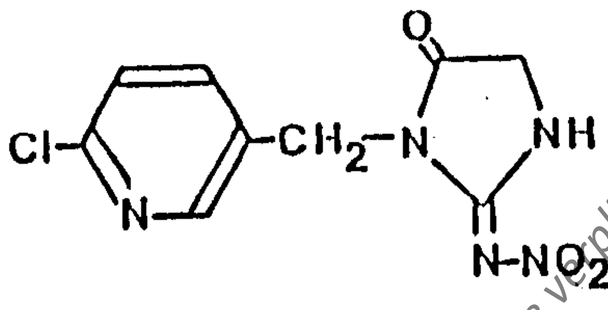
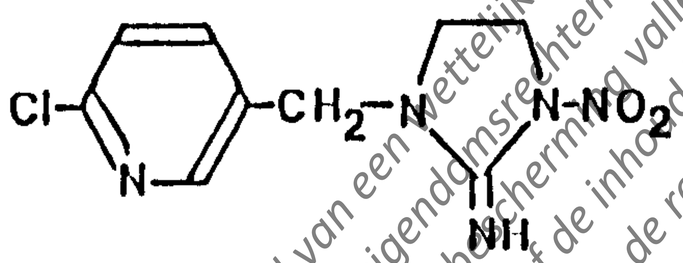
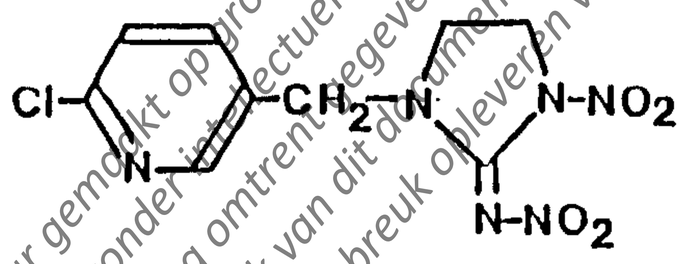
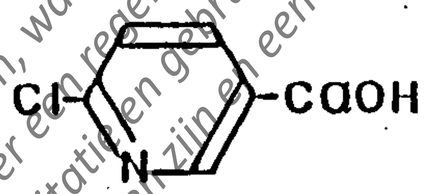
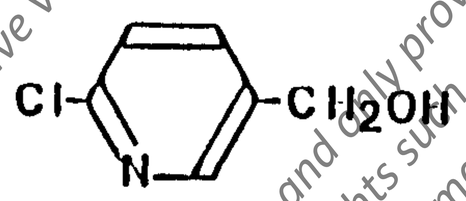
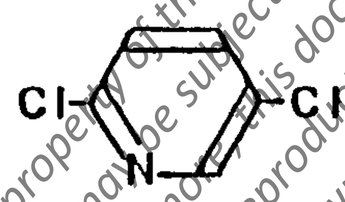
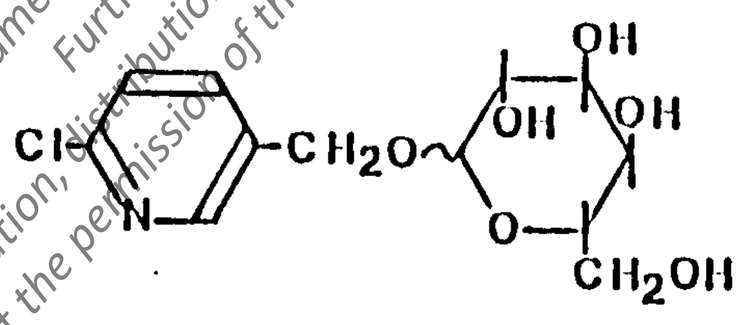
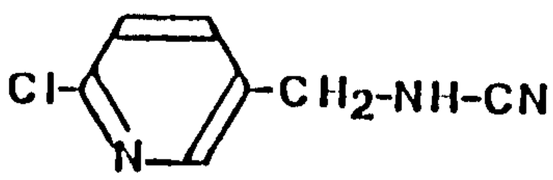
Appendix II Structural information of the NTN 33893 reference substances (2)

Substances	Structure	Mol. weight
WAK 3738		269
DIJ 10533		255
DIJ 9979		300
CNA		157
PAC		143
PAD		141
RBN 1114		305
WAK 4613		167

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Appendix III Soil characteristics

Origin : volcanic ash soil obtained from Tama test field of Nitokuno, Ibaraki, Japan

Soil texture

International : light clay (LiC)

coarse sand	2.0 - 0.2 mm	10.6 %
fine sand	0.2 - 0.02 mm	26.8 %
silt	0.02 - 0.002 mm	31.7 %
clay	< 0.002 mm	30.9 %

USDA : Clay (CL)

very coarse sand	2.0 -1.0 mm	0.2 %
coarse sand	1.0 - 0.5 mm	0.9 %
medium sand	0.5 - 0.25 mm	6.5 %
fine sand	0.25 - 0.10 mm	14.9 %
very fine sand	0.10 - 0.05 mm	7.3 %
silt	0.05 - 0.002 mm	41.9 %
clay	< 0.002 mm	28.3 %

pH : 5.7 at 26·C (H_2O), 4.6 at 26·C (KCl)

Organic carbon : 4.05 % (dried weight basis)

Cation exchange capacity (CEC) : 16.4 me/100 g (dried weight basis)

Phosphate adsorption coefficient : 1560 (dried weight basis)

True density : 2.51 (dried weight basis)

The data described above were supplied by soil analysis laboratory of Palyno Survey Company Ltd.,Tokyo, Japan

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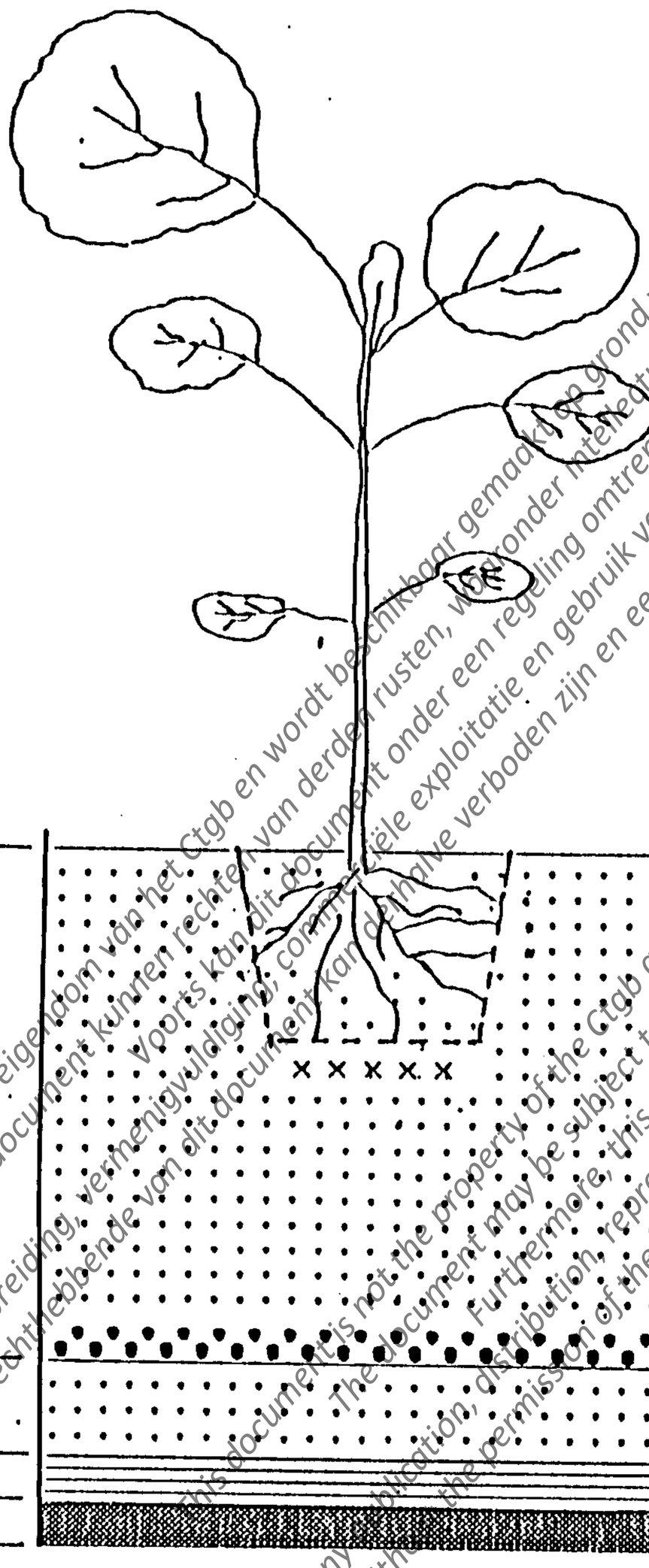
Appendix IV Preparation of the planting container and planting hole application



planting hole area

× × × 1 % granule

soil containing 5 g of artificial fertilizer and 5 g of magnesia lime

compost

sand

pumice stone

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Appendix V Behavior of the NTN 33893 and reference substances on the silica gel TLC plate

Substances	Rf value		
	Solvent system A	B	E
NTN 33893	0.56 (1.00)	0.83 (1.00)	0.49 (1.00)
NTN 38014	0.01 (0.02)	0.30 (0.36)	0 (0)
WAK 3839	0.39 (0.70)	0.79 (0.95)	0.19 (0.39)
NTN 33519	0.46 (0.82)	0.84 (1.01)	0.13 (0.27)
WAK 4103	0.68 (1.21)	0.78 (0.94)	0.34 (0.69)
WAK 3772	0.76 (1.36)	0.66 (0.80)	0.24 (0.49)
NTN 35884	0.50 (0.89)	0.75 (0.90)	0.28 (0.57)
WAK 3738	0.74 (1.35)	0.83 (1.00)	0.50 (1.03)
DIJ 10533	0.62 (1.11)	0.86 (1.04)	0.28 (0.57)
DIJ 9979*	0.28 0.67	0.91 0.69	0.28 (0.57)
CNA	0.22 (0.39)	0.66 (0.80)	0 (0)
PAC	0.77 (1.38)	0.93 (1.12)	0.38 (0.78)
PAD	0.70 (1.25)	0.84 (1.01)	0.40 (0.82)
RBN 1114	0.40 (0.71)	0.41 (0.49)	0.01 (0.02)
WAK 4613*	0.73 0.59	0.96 0.93	0.42 0.14

The values in parenthesis are relative value based on NTN 33893
Solvent system A : EtOAc/i-PrOH/H_2O (65/23/12, v/v)
B : $CHCl_3/CH_3OH/CH_3COOH/H_2O$ (65/25/3.5/3.5, v/v)
E : $CH_3CN/CHCl_3$ (50/50, v/v)
* Insufficient purity or decomposition in the TLC analysis

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Appendix VI Behavior of the NTN 33893 and reference substances in the HPLC analysis

Compound	R.t. (min)			
	RP-Select B column		RP-18 (e) column	
NTN 33893	30.54	(1.00)	29.38	(1.00)
NTN 38014	34.20	(1.12)	34.67	(1.18)
WAK 3839	25.35	(0.83)	23.50	(0.80)
NTN 33519	27.49	(0.90)	25.85	(0.88)
WAK 4103	26.57	(0.87)	25.56	(0.87)
WAK 3772	24.74	(0.81)	24.68	(0.84)
NTN 35884	26.26	(0.86)	25.27	(0.86)
WAK 3738	32.37	(1.06)	31.73	(1.08)
CNA	21.38	(0.70)	20.57	(0.70)
PAC	20.46	(0.67)	19.39	(0.66)
PAD	24.73	(0.81)	23.50	(0.80)
RBN 1114	14.66	(0.48)	16.45	(0.56)
WAK 4613*	39.40	45.50	39.66	45.54

The values in parenthesis are relative value based on NTN 33893
Solvent sequence (HPLC)
1ml/min 10% A (5 min)------>100% A (40 min) A + B = 100%
A: CH_3CN/H_2O + PIC B-8 (50/50, v/v + 0.005 M PIC)
B: H_2O + PIC B-8 (0.005 M PIC)
* : insufficient purity or decomposition in the HPLC analysis

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