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Control of Biocides 2023

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Sources must be acknowledged

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Preface

In 2023, the Danish Chemical Inspection Service of the Danish Environmental Protection Agency conducted a control of biocide products on the Danish market. This control is in line with the "Danish Political Agreement on biocidal initiatives 2022-2025, Control of Biocides". The purpose was to analyse biocide products available in Denmark to ensure that the active substances in the products comply with the labeled content and specifications provided by manufacturers to the Danish Environmental Protection Agency.

Furthermore, the biocide products collected in the control campaign were tested for selected Per- and Polyfluoroalkyl Substances (PFAS).

Finally, a number of biocide samples were collected specifically for ad hoc rodenticide analysis.

Abbreviations

a.s.: Active substance
CIPAC: Collaborative International Pesticides Analytical Council
DANAK: The Danish Accreditation and Metrology Fund
Danish EPA: The Danish Environmental Protection Agency
DEET: N,N-diethyl-m-toluamide
DTI: Danish Technological Institute
HPLC-DAD: High-performance liquid chromatography - diode array detection
LC-MS: Liquid chromatography - mass spectrometry
PFAS: Per- and Polyfluoroalkyl Substances
RSD: Relative standard deviation
STD: Standard deviation

Resume

Denne rapport beskriver den analytiske kemiske kontrol af biocidprodukter på det danske marked, der er udført af Miljøstyrelsens Kemikalieinspektion i 2023 (Ref. 1).

Den planlagte kontrolkampagne dækkede over tre aktivstoffer, som forefindes i otte udvalgte biocidprodukter. Et overblik over produkter og aktivstoffer er listet i tabellen nedenfor. Produkterne blev analyseret for at verificere, om indholdet af de respektive aktivstoffer var i overensstemmelse med produktspecifikationen og det deklarerede indhold. Grænsen for en accepteret afvigelse i indholdet af aktivstof i forhold til det deklarerede indhold er fastsat i bekendtgørelsen om bekæmpelsesmidler nr. 1278 af 09/06/2021 (gældende bekendtgørelse nr. 961 af 26/06/2023, Ref. 2).

Det målte indhold af aktivstoffer var i overenstemmelse med det deklarerede indhold for 6 ud af 8 produkter, idet resultatet var indenfor toleranceintervallet. For 2 af produkterne var det grundet analysens måleusikkerhed ikke muligt definitivt at sige, at produkterne ikke overholder gældende dansk lovgivning.

Aktiv stof	Antal af analyserede produkter	Antal indenfor toleranceinterval	Antal udenfor toleranceinterval
Bromadiolon	3	2	1*
Difenacoum	1	1	0
DEET	4	3	1*
Total	8	6	2*

Overblik over analyserede produkter og aktivstoffer, som er en del af kontrolkampagnen 2023.

* Grundet måleusikkerheden for analyseresultatet kan det ikke endeligt fastslås, at produktindholdet af aktivstof ikke er i overensstemmelse med dansk lovgivning.

Produkterne, der blev indsamlet, blev også undersøgt for tilstedeværelsen af udvalgte PFAS indholdsstoffer. Resultaterne viste, at der ikke blev påvist nogen PFAS indholdsstoffer i produkterne. Analyserne af PFAS blev udført af Medico Kemiske Laboratorium ApS.

Derudover blev der indsamlet en række prøver til ad hoc-analyse.

 16 prøver blev indsamlet. Alle 16 prøver var mistænkt for uautoriseret anvendelse af rodenticider. Der blev påvist rodenticide aktivstoffer i alle 16 prøver.

Abstract

This report documents the analytical chemical control of selected biocidal products on the Danish market. These tests are part of the Danish Environmental Protection Agency's Chemicals Inspection in 2023 (Ref. 1).

The planned control campaign covered three active substances found in eight selected biocide products. An overview of collected products and active substances are listed in the table below. The products were analysed to verify whether the content of the active substances in the products in question complied with the product specification and the declared content. The tolerance interval of an active substance is specified in the Danish Statutory Order on Pesticides No. 1278 of 06/06/2021 (applicable Danish Statutory Order 961 of 26/06/2023) (Ref. 2).

The measured content of active substances was in accordance with the declared content of 6 out of 8 products, as the results were within the tolerance interval. Due to the measurement uncertainty of the analysis of the last 2 products it was not possible to definitively state that they do not comply with Danish law.

Active substance	No. of analysed	No. within tolerance	No. outside tolerance
	products	interval	interval
Bromadiolone	3	2	1*
Difenacoum	1	1	0
DEET	4	3	1*
Total	8	6	2

Overview of analysed products and active substances in the control campaign 2023.

* Due to the measurement uncertainty of the analysis result it could not be definitively stated that the product dose of the active substance does not comply with Danish law.

The collected products were also examined for the presence of selected PFAS substances. The results showed that no PFAS substances were detected in the products. The analyses of PFAS were conducted by Medico Kemiske Laboratorium ApS.

Additionally, a range of rodenticide samples were collected for ad hoc analysis.

• 16 ad hoc samples were collected. All 16 rodenticide samples that were collected were suspected of being utilised without permit. Rodenticides were identified in all 16 samples.

1. Control of biocides 2023

The control of biocides in 2023 was divided into three main activities:

- The planned control campaign of 2023.
- Analysis of selected PFAS substances in collected products.
- The ad hoc control of potential rodenticides utilised without permit.

1.1 The planned control campaign 2023

The planned control campaign conducted in 2023 covered three active substances (a.s.). The three a.s. were present in eight commercial products found on the Danish market. The a.s. are presented in TABLE 1. The a.s. were selected according to their sales figures in the previous years, availability at retailers, and whether they had been part of previous campaigns. Products were collected by the Chemical Inspection Service of the Danish Environmental Protection Agency (Danish EPA) during the period from April to July 2023. The products were collected from wholesale dealers/importers or at retailers.

Area of application	Active substance	CAS No.	
Dedenticide	Bromadiolone	28772-56-7	
Rodenticide	Difenacoum	56073-07-5	
Insect Repellent	DEET	134-62-3	

TABLE 1. Overview of selected active substances in the 2023 control campaign.

The collected products were stored at the Laboratory for Chemistry and Microbiology, Danish Technological Institute (DTI) (Ref. 4), in their original packaging until the chemical analyses were initiated. The products were stored at ambient temperature and protected from light during the entire storage period. Furthermore, the collected products were sent to Medico Kemiske Laboratorium for testing for the content of selected Per- and Polyfluoroalkyl Substances (PFAS).

1.2 Ad hoc rodenticides

According to Statutory Order no. 2307 of 06/12/2021 (Ref. 3), the application of anticoagulant rodenticides is restricted to authorised exterminators only. Upon suspicion of unauthorised use of rodenticides, samples were collected by the municipalities in Denmark and submitted for analysis of anticoagulant rodenticides.

1.3 Analytical program and responsible party

The analyses of the products and samples were performed by DTI, Laboratory for Chemistry and Microbiology. DTI is a self-owned and not-for-profit Institute (Ref. 4). The Laboratory for Chemistry and Microbiology is accredited by Danish Accreditation and Metrology Fund (DANAK), registration no. 90, according to DS/EN ISO/IEC 17025:2017 (Ref. 5).

2. Control campaign 2023

2.1 Collected products

A total of eight biocide products were collected by the Chemical Inspection Service of the Danish Environmental Protection Agency. Four products were categorised as rodenticides and the other four products were categorised as insect repellent. The rodenticides contained Bromadiolone or Difenacoum as a.s., and the insect repellent contained N,N-diethyl-m-toluamide (DEET) as a.s.

2.2 Tolerances on active substance concentration

The objective of the Danish EPA was to determine the content of a.s. in the products. The results of the quantitative analysis were subsequently compared to the specification of the product and the declared content on the label supplied by the authorization holder.

The Danish Statutory Order on Pesticides No. 1278 of 06/06/2021 (applicable Danish Statutory Order 961 of 26/06/2023) specified the general tolerance of deviation from the declared content of active substances (Ref 2). These tolerances are listed in TABLE 2.

Content of active substances in g/kg or g/L at 20°C	Tolerance of deviation
	± 15% homogeneous formulation
Up to 25	± 25% non-homogeneous formulation
More than 25 up to 100	± 10%
More than 100 up to 250	± 6%
More than 250 up to 500	± 5%
More than 500	± 25 g/kg or ± 25 g/L

TABLE 2. The tolerance of deviation from the declared content of active substance.

2.3 Analysis

2.3.1 Quantification of active substances

The quantitative analyses were performed on a homogenous sub-sample prepared from each product. Five independent quantitative measurements were conducted for each product. If the average result was outside the tolerance interval, then the analysis was repeated with a minimum of five new and freshly prepared samples. The methods used for quantification were validated based on linearity, specificity, accuracy and by using control reference material at two levels.

The validation of implemented methods were performed on at least eight freshly prepared samples of the product. The analyses were performed over at least two days for each product. The mean value of the analyses and the standard deviation (STD) were calculated for each day and for all eight results. The recovery percentage was determined by adding a known amount of the relevant a.s. to a minimum of five samples of each product. The mean recovery and STD were calculated.

The expanded uncertainty (k = 2) for each method was calculated according to the spread of the analysis results, the recovery of fortified samples, and the purity of the reference standard. The expanded uncertainty is used to determine a 95% confidence interval of the result. The expanded uncertainty varies from 3-14% depending on the analytical method, the product formulation, and the purity of available reference material.

TABLE 3. Biocides in the 2023 control campaign.

				Analytic	cal method		
Name	CAS No.	Year selected for control	Principle	DTI method	Accreditation	Adapted from reference method	Molecular structure (Ref 6)
Bromadiolone	28772-56-7	2010, 2005	HPLC-DAD	OA-859	Yes	-	$\begin{array}{c} & & & & \\ & & & \\ & & & \\ & &$
Difenacoum	56073-07-5	2010, 2002, 1994	HPLC-DAD	OA-859	Yes	-	
DEET	134-62-3	-	HPLC-DAD	OA-887	Yes	-	CH ₃

2.3.2 Relative density testing

The density of all liquid products was measured. The results were used to calculate the a.s. in g/L for each product. The density was determined according to DTI's analysis method UA-312. The method is based on CIPAC MT 3 (Ref 8) and is accredited. The density of the product is determined as the average of a triple determination carried out by measuring with a Densito 30 PX densitometer.

2.3.3 Per- and Polyfluoroalkyl Substance testing

The eight products collected in the control campaign were tested for 33 selected Per- and Polyfluoroalkyl Substances. The content of selected PFAS was determined according to the method DS/CEN/TS 15968:2010 at Medico Kemiske Laboratorium ApS. The laboratory is accredited to testing under ISO 17025, but the methods were not accredited. The samples were analysed in duplicates and analysed by LC-MS/MS, however, the substances 8:2 FTAC, 8:2 FTMAC, and 8:2 FTOH were analysed by GC-MS. The expanded uncertainty of the method was 30%.

2.4 Results and conclusions

The results of the quantification analysis of a.s. are presented in TABLE 4 and TABLE 5.

DTI sample no.	Active substance	Label claim g/kg	Tolerance interval g/kg	Analysis result g/kg	RSD %	Comply/Non- comply
174859-1	Bromadiolone	0.05	0.04-0.06	0.06	0.37	Comply
174859-2	Difenacoum	0.05	0.04-0.06	0.05	5.6	Comply
174859-3	Bromadiolone	0.05	0.04-0.06	0.05	1.7	Comply
174859-4	Bromadiolone	0.050	0.038-0.063	0.036	3.1	*

TABLE 4. Analysis results (g/kg) in the rodenticide products.

* Due to the measurement uncertainty of the analysis result it cannot be definitively stated that the product dose of active substance does not comply with Danish law.

DTI sample no.	Active substance	Label claim g/L	Tolerance interval	Analysis result	RSD %	Comply/Non- comply
		-	g/L	g/L		
174859-5	DEET	92	83-101	97	0.89	Comply
174859-6	DEET	340	323-357	350	1.0	Comply
174859-8	DEET	354	336-372	375	0.81	*
174859-9	DEET	340	323-356	348	1.4	Comply

TABLE 5. Analysis result (g/L) in the insect repellent products.

* Due to the measurement uncertainty of the analysis result it cannot be definitively stated that the product dose of active substance does not comply with Danish law.

The results of the quantification analysis of PFAS are presented in TABLE 6. PFAS was not detected in any of the products.

		LOD, LOQ,		Average results (µg/kg)							
Substance	CAS No.	method (µg/L)	product (µg/kg)	174859-1	174859-2	174859-3	174859-4	174859-5	174859-6	174859-8	174859-9
PFBA	375-22-4	0.1*	1.5*	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFPeA	2706-90-3	0.1*	1.5*	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFHxA	307-24-4	0.1*	1.5*	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFHpA	375-85-9	0.1*	1.5*	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFOA	335-67-1	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFNA	375-95-1	0.1*	1.5*	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFDA	335-76-2	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFUnDA	2058-94-8	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFDoDA	307-55-1	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFTrDA	376-06-7	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFOSA	754-91-6	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFBS	375-73-5	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFPeS	2706-91-4	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFHxS	355-46-4	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFHpS	375-92-8	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFOS	1763-23-1	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFNS	68259-12-1	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFDS	335-77-3	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFUnDS	749786-16-1	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFDoDS	79780-39-5	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
PFTrDS	791563-89-8	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
4:2 FTS	757124-72-4	0.5	7.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
6:2 FTS	27619-97-2	0.5	7.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
8:2 FTS	39108-34-4	0.5	7.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
8:2 PAP	57678-03-2	0.5	7.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
8:2 diPAP	678-41-1	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
EtFOSA	4151-50-2	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

TABLE 6. Analysis results of PFAS in the collected samples. N.D. stands for Not Detected.

		LOD,	LOQ, product (µg/kg)	Average results (µg/kg)							
Substance CAS N	CAS No.	method (µg/L)		174859-1	174859-2	174859-3	174859-4	174859-5	174859-6	174859-8	174859-9
EtFOSE	1691-99-2	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
MeFOSA	31506-32-8	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
MeFOSE	24448-09-7	0.1	1.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
8:2 FTAC	27905-45-9	0.5	7.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
8:2 FTMAC	1996-88-9	0.5	7.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.
8:2 FTOH	678-39-7	0.5	7.5	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.	N.D.

* The LOD of the method was 0.2 μ g/L and the LOQ of the products is 3.0 μ g/kg for sample 174859-1.

3. Ad hoc rodenticide samples

3.1 Collected products

A total of 16 samples were submitted for identification and quantification of rodenticides as part of the ad hoc program. The physical condition of the samples varied from pristine to weathered, and the samples were in some cases mixed with other debris, e.g., soil or grain. The collected samples are listed in TABLE 8.

3.2 Analysis

The ad hoc samples were analysed for eight anticoagulant rodenticides according to DTI's method OA-1108 (TABLE 7). The method relies on screening by liquid chromatography with mass spectrometric detection (LC-MS) to determine the a.s. in the product. This analysis is followed by reversed phase liquid chromatography with diode array detection (HPLC-DAD) to both verify and quantify the content of the a.s. The HPLC method is modified from CIPAC method 370 brodifacoum (Ref. 7). The quantitative analysis was performed with a minimum of two replicates.

TABLE 7.	Rodenticides	included i	n the	screening	program.
	Roachtiolaco	molucui	ii uic	Sorcerning	program.

Active substance	CAS No.
Bromadiolone	28772-56-7
Difenacoum	56073-07-5
Difethialone	104653-34-1
Brodifacoum	56073-10-0
Flocoumafen	90035-08-8
Chlorophacinone	3691-35-8
Coumatetralyl	5836-29-3
Warfarin	81-81-2

3.3 Results and conclusions

The results of the analyses of the ad hoc rodenticide samples are listed in TABLE 8. RSD% is the percentage relative reproducibility of the determinations of the product. Rodenticides were identified in all 16 samples.

EPA No.	DTI No.	Sample type	Identified a.s.	Analysis result g/kg	RSD%
2022-86210	168346-1	Red blocks	Chlorophacinon	0.091	0.32
2022-86210	168346-2	Blue/green grains, severely weathered Difenacoum		0.022	136
2022-85126	172571-1	Red grains	Red grains Difethialon		7.3
2022-85126	172571-2	Red grains	Bromadiolone	0.037	1.0
2023-16513	188752-1	Blue grains	Difenacoum	0.047	2.1
2023-16513	188752-2	Blue blocks	Bromadiolone	0.058	0.0
2023-13857	188901-1	Red grains	Difethialon	0.012	6.2
2023-13857	188901-2	Blue foam	Coumatetralyl	3 mg*	-
2023-13857	188901-3	Red grains	Bromadiolone	0.051	0.36
2023-9941	192410-1	Blue pasta	Difethiolon	0.016	0.31
2023-27396	196808-1	Blue grains/pasta	Coumatetralyl	0.99	#
2023-27396	400000.0	Dhua blaaka	Coumatetralyl	0.041	0.67
	196808-2	Blue blocks	Difenacoum	0.044	3.4
2023-27396	106909 2	Blue blocks	Coumatetralyl	0.023	11
	196808-3		Difenacoum	0.038	1.8
2023-23246	197512-1	Unidentifiable, sample severely weathered Brodifacoum		< LOQ	-
2023-23246	197512-2	Red grains with black clumps	Difethialon	0.023	21
2023-23246	197512-3	Blue blocks, severely weathered	Brodifacoum	0.044	2.7

TABLE 8. Analysis results of rodenticides in ad hoc samples.

* Due to the characteristics of the sample, it was necessary to determine the complete concentration of the active substance in the received foam.

[#] Insufficient sample quantity available to perform a duplicate analysis.

4. References

- 1. The Danish Environmental Agency, <u>www.mst.dk</u>, <u>https://mst.dk/kemi/biocider/</u> (in Danish) or <u>https://eng.mst.dk/chemicals/biocides/</u> (in English).
- Statutory Order on Pesticides No. 1278 of 06/06/2021 (applicable Danish Statutory Order 961 of 26/06/2023).
- 3. Statutory Order No. 2307 of 06/12/2021 Bekendtgørelse om forebyggelse og bekæmpelse af rotter (Statutory Order on prevention and control of rodents).
- 4. Danish Technological Institute, Kongsvang Allé 29, DK-8000 Aarhus C, Denmark, <u>www.teknologisk.dk</u> (in Danish), <u>http://www.dti.dk/</u> (in English).
- 5. The Danish Accreditation and Metrology Fund DANAK, <u>www.danak.dk</u> (in Danish), <u>http://english.danak.dk/</u> (In English)
- 6. The e-Pesticide Manual, Nineteenth Edition, online version, 2021. British Crop Protection Council, United Kingdom.
- 7. CIPAC method Brodifacoum 370, http://www.cipac.org/
- 8. CIPAC MT 3 Specific gravity, density, and weight per millilitre. http://www.cipac.org/

Appendix 1

Biocide products collected for the planned control campaign 2023.

TABLE 9. Biocide products.

DTI sample No.	Active substance(s)	Reg. No.	Name of product	Authorization holder
174859-1	Bromadiolone	413-2	Notrac Blox	Bell Laboratories Netherlands B.V.
174859-2	Difenacoum	736-2	Tanaco Difablok	Pelgar International Limited
174859-3	Bromadiolone	736-5	Tanaco Bromablok Pro	Pelgar International Limited
174859-4	Bromadiolone	736-10	Bromakorn Pro	Pelgar International Limited
174859-5	DEET	567-4	Mygfri	Jaico RPD nv
174859-6	DEET	790-3	Insektspray DEET 40%	Primmed B.V.
174859-8	DEET	998-1	Bushman	Juno Europe (CY) Limited
174859-9	DEET	790-3	TravelDeet, DEET 40% Tropical	Primmed B.V.

Control of biocides 2023nglish

This report documents the 2023 control program of biocide products on the Danish market carried out by the Danish Environmental Protection Agency, the Chemical Inspection Service.

As part of the planned control campaign, a total of eight biocide products were collected on the Danish market and analysed to verify whether the content of the active substances in the products complied with the labelled content. Furthermore, the biocide products collected in the control campaign were tested for selected Per- and Polyfluoroalkyl Substances (PFAS).

Additionally, a number of ad hoc samples were collected for control: 16 samples of rodenticides were collected as they had been used without authorisation. Rodenticide active substances were identified in all samples.

Danish

Denne rapport beskriver det kemiske kontrolprogram fra 2023 af biocidprodukter på det danske marked udført af Miljøstyrelsen, Kemikalieinspektionen.

Som en del af den planlagte kontrolkampagne blev otte biocidprodukter udtaget på det danske marked og analyseret for at verificere, om indholdet af de respektive aktivstoffer er i overensstemmelse med det deklarerede indhold. Desuden blev biocidprodukterne testet for udvalgte Per- og Polyfluoralkylstoffer (PFAS).

Derudover blev en række prøver udtaget til ad hoc-kontrol: 16 ad hoc-prøver af potentielle rottemidler, anvendt uden tilladelse, blev indsamlet. Der blev fundet rodenticide aktivstoffer i alle prøver.



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