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Deliverable 4.2

WP4 – TREATMENT: Treatment technology innovation



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ABSTRACT	This deliverable presents the design, implementation, operation and main achievements of the MITO3X® application for chloramination control at the Madrid case study. The work was carried out at the Valmayor Drinking Water Treatment Plant, managed by Canal de Isabel II, where MITO3X® was integrated into the existing pilot plant to support controlled monochloramine formation and reduce the formation of disinfection by-products, with particular attention to trihalomethanes, haloacetic acids and N-nitrosodimethylamine. The Madrid case study focuses on the optimisation of in-situ monochloramine generation by controlling the balance between chlorine and ammonia addition and by monitoring chloramine speciation using absorbance-based measurements. The tests included different chloramination scenarios and water matrices, including raw water, ozone-preoxidised water, advanced-oxidation-treated water and water amended with polyDADMAC as a nitrosamine precursor. The results demonstrate that the MITO3X® system can be successfully implemented for chloramination control and operated under different conditions to evaluate DBP and nitrosamine formation, revealing a clear increase in DBP formation from monochloramine-dominated conditions to mixed and dichloramine-rich conditions, with the highest levels observed under free-chlorine conditions.		

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EXECUTIVE SUMMARY

This work describes the implementation and validation of the MITO3X® application for chloramination control at the Valmayor Drinking Water Treatment Plant (Madrid), carried out within the framework of the intoDBP project. The objective of the work is to optimise in-situ monochloramine generation in order to minimise the formation of disinfection by-products (DBPs) and emerging compounds of concern, including N-nitrosodimethylamine (NDMA).

The MITO3X® pilot system was integrated into the existing pilot facilities at Valmayor and operated under controlled hydraulic and chemical conditions. Experimental campaigns were carried out using different chloramination scenarios, including monochloramine-rich, dichloramine-rich, mixed chloramine and free-chlorine conditions. These scenarios were tested across representative water matrices, including raw water, pre-oxidised water, advanced-oxidation-treated water and matrices amended with polyDADMAC as a nitrosamine precursor.

The results demonstrate that the system enables controlled formation of chloramine species through adjustment of the chlorine-to-ammonia ratio and provides a reliable platform for evaluating DBP formation. A consistent trend was observed in which DBP formation increases progressively from monochloramine-rich to mixed chloramine, dichloramine-rich and free-chlorine conditions across the tested matrices.

Overall, the findings confirm the effectiveness of controlled chloramination as a strategy for DBP mitigation and demonstrate the suitability of the MITO3X® system for optimisation and further implementation in drinking water treatment applications.

1. Introduction

Disinfection is an essential process in drinking water treatment, ensuring microbiological safety and protection against waterborne pathogens (World Health Organization (WHO), 2017). Chlorination remains the most widely used disinfection strategy; however, the reaction of chlorine with natural organic matter and other precursors leads to the formation of disinfection by-products (DBPs) (Richardson et al., 2007). Several classes of DBPs, including trihalomethanes (THMs) and haloacetic acids (HAAs), are regulated due to their potential health impacts (European Union, 2020).

Chloramination, based on the formation of monochloramine through the reaction of chlorine and ammonia, is commonly applied as an alternative disinfection strategy to reduce the formation of regulated DBPs (Deborde & von Gunten, 2008). Compared to free chlorine, monochloramine typically produces lower concentrations of THMs and HAAs. However, chloramination can promote the formation of nitrogenous DBPs, including nitrosamines such as N-nitrosodimethylamine (NDMA), which are of increasing concern (Mitch et al., 2003).

Effective control of chloramination processes therefore requires careful management of operating conditions, particularly the balance between chlorine and ammonia dosing and the resulting chloramine speciation. The ability to generate and maintain monochloramine-rich conditions while limiting dichloramine formation is a key factor for optimising water quality and minimising DBP formation (Schreiber & Mitch, 2005).

Within this context, the intoDBP project aims to develop and validate innovative solutions to control DBP formation in drinking water treatment. The MITO3X® system has been developed to enable controlled in-situ generation of chloramine species, combined with online monitoring tools to support process optimisation.

This deliverable presents the application of the MITO3X® system in the Madrid case study at the Valmayor Drinking Water Treatment Plant. It describes the pilot plant configuration, operational approach and experimental campaigns carried out to evaluate chloramine formation and DBP generation under different conditions and water matrices.

2. Madrid case study and MITO3X® system

The Madrid case study is conducted at the Valmayor Drinking Water Treatment Plant (DWTP), managed by Canal de Isabel II. Valmayor is a large-scale facility where chloramination is applied as part of the final disinfection strategy. This case study is particularly relevant within the intoDBP project because it focuses on the formation and control of nitrogenous DBPs, including N-nitrosodimethylamine (NDMA), under chloramination conditions.

In contrast to other WP4 case studies, which primarily address pre-oxidation and precursor removal, the Madrid application focuses on final disinfection optimisation, specifically on controlling chloramine speciation to minimise DBP formation. **Figure 1** presents a simplified scheme of the Valmayor DWTP, highlighting the treatment stages relevant to the case study.

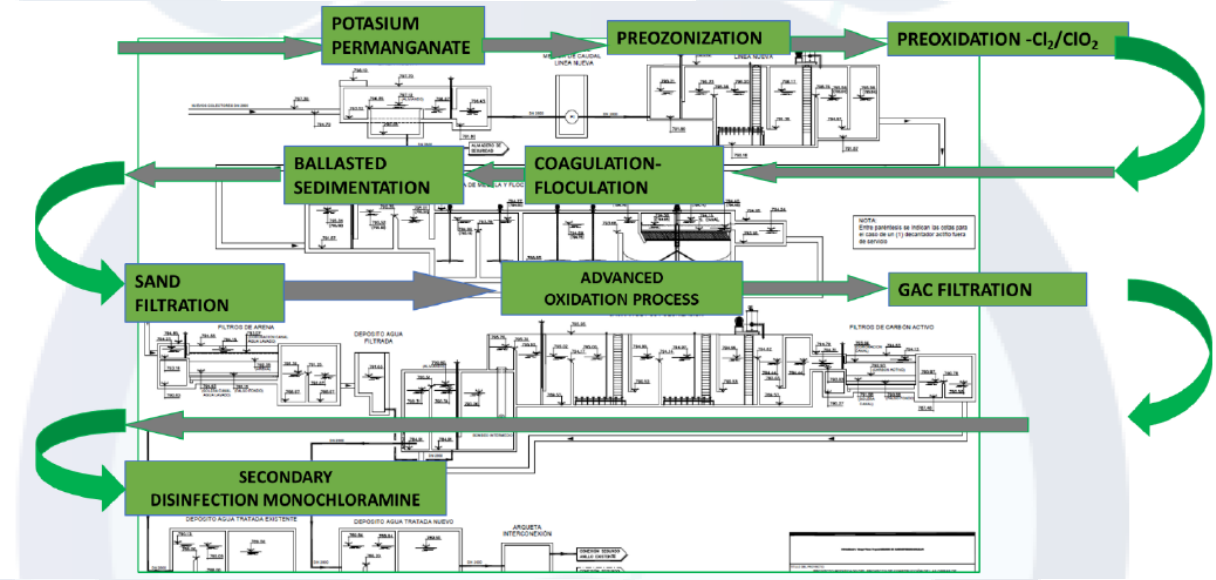


Figure 1. Simplified scheme of the DWTP managed by Canal de Isabel II

The MITO3X® system was integrated into the existing pilot facilities at Valmayor, which allow water from different treatment stages to be tested under controlled conditions. These facilities provide flexibility to evaluate treatment strategies using representative water matrices, including raw and pre-treated waters.

The MITO3X® technology is a compact and modular platform designed for controlled dosing and mixing of reagents in a liquid flow. In the Madrid application, the system is used to dose sodium hypochlorite and ammonium hydroxide as chlorine and ammonia sources, respectively. The objective is to enable in-situ generation of monochloramine under controlled hydraulic and chemical conditions, while avoiding local imbalances that could favour the formation of dichloramine.

The integrated pilot setup includes the MITO3X® reactor, reagent dosing systems, monitoring sensors and control elements installed within the Valmayor pilot line. **Figure 2** shows the MITO3X® pilot system as installed at the case study site. The experimental campaigns were performed using representative water matrices and conditions described in Section 6.



Figure 2. MITO3X® pilot/prototype for in-situ monochloramine generation.

3. Chloramination control concept and monitoring strategy

3.1 Chloramination control concept

Chloramination involves the reaction between chlorine and ammonia to form chloramine species, primarily monochloramine (MCA) and dichloramine (DCA). Among these, monochloramine is generally preferred for drinking water disinfection, as it is associated with lower formation of regulated DBPs such as trihalomethanes (THMs) and haloacetic acids (HAAs). In contrast, dichloramine formation is undesirable due to its association with nitrogenous DBPs, including NDMA (Deborde & von Gunten, 2008; Mitch et al., 2003).

The objective of the Madrid MITO3X® application is therefore to achieve controlled formation of monochloramine, while minimising the formation of dichloramine. This is accomplished by adjusting the relative dosing of sodium hypochlorite and ammonium hydroxide under controlled mixing conditions.

By varying the chlorine-to-ammonia ratio, the system can reproduce different chloramination scenarios, including:

- monochloramine-rich conditions
- mixed monochloramine/dichloramine conditions
- dichloramine-rich conditions
- free-chlorine (hypochlorite-only) conditions

These operating scenarios enable systematic investigation of the relationship between chloramine speciation and DBP formation under controlled experimental conditions.

3.2 Monitoring strategy based on absorbance signals

The MITO3X® system integrates an absorbance-based monitoring approach to support real-time interpretation of chloramine speciation. Chloramination results in the formation of different chloramine species that exhibit distinct absorbance behaviours in the ultraviolet range, leading to characteristic changes in both the intensity and shape of the spectra.

This monitoring strategy provides a practical and rapid method to:

- detect the onset of dichloramine formation

- follow the evolution of chloramine species during dosing adjustments

- support operational control and interpretation of experimental conditions

Error! Reference source not found. presents the UV–Vis absorbance spectra used to interpret chloramine formation under different experimental conditions. The plots show absorbance as a function of wavelength for the different chloramination regimes, including monochloramine-rich, dichloramine-rich and mixed conditions.

The spectra exhibit distinct features depending on chloramine speciation. Monochloramine and dichloramine present different absorbance behaviours in the ultraviolet range, leading to characteristic changes in both the intensity and shape of the spectra. The monitoring approach is based on UV absorbance at wavelengths where monochloramine and dichloramine exhibit distinct molar absorptivities, typically around 245 nm and 295 nm, respectively. Variations in absorbance at these wavelengths can be used to differentiate between monochloramine-dominated and dichloramine-dominated conditions and to identify transitions between these regimes during operation. This approach is consistent with the spectrophotometric chloramine-speciation methodology described by (Schreiber &

Mitch, 2005), in which absorbance measurements at two wavelengths combined with known extinction coefficients can be used to estimate the relative contributions of monochloramine and dichloramine in chloraminated waters.

The three subfigures illustrate (i) the overall absorbance response across the tested conditions, (ii) the comparison of spectral features used to distinguish between chloramine species, and (iii) the specific wavelength regions used to interpret chloramine speciation. These absorbance-based measurements provide a practical tool for monitoring chloramine speciation and support the interpretation and control of monochloramine and dichloramine formation during operation of the MITO3X® system.

Overall, the combination of controlled reagent dosing and absorbance-based monitoring provides a robust framework for real-time chloramination control and experimental evaluation, enabling the assessment of the influence of chloramine speciation on DBP and nitrosamine formation.

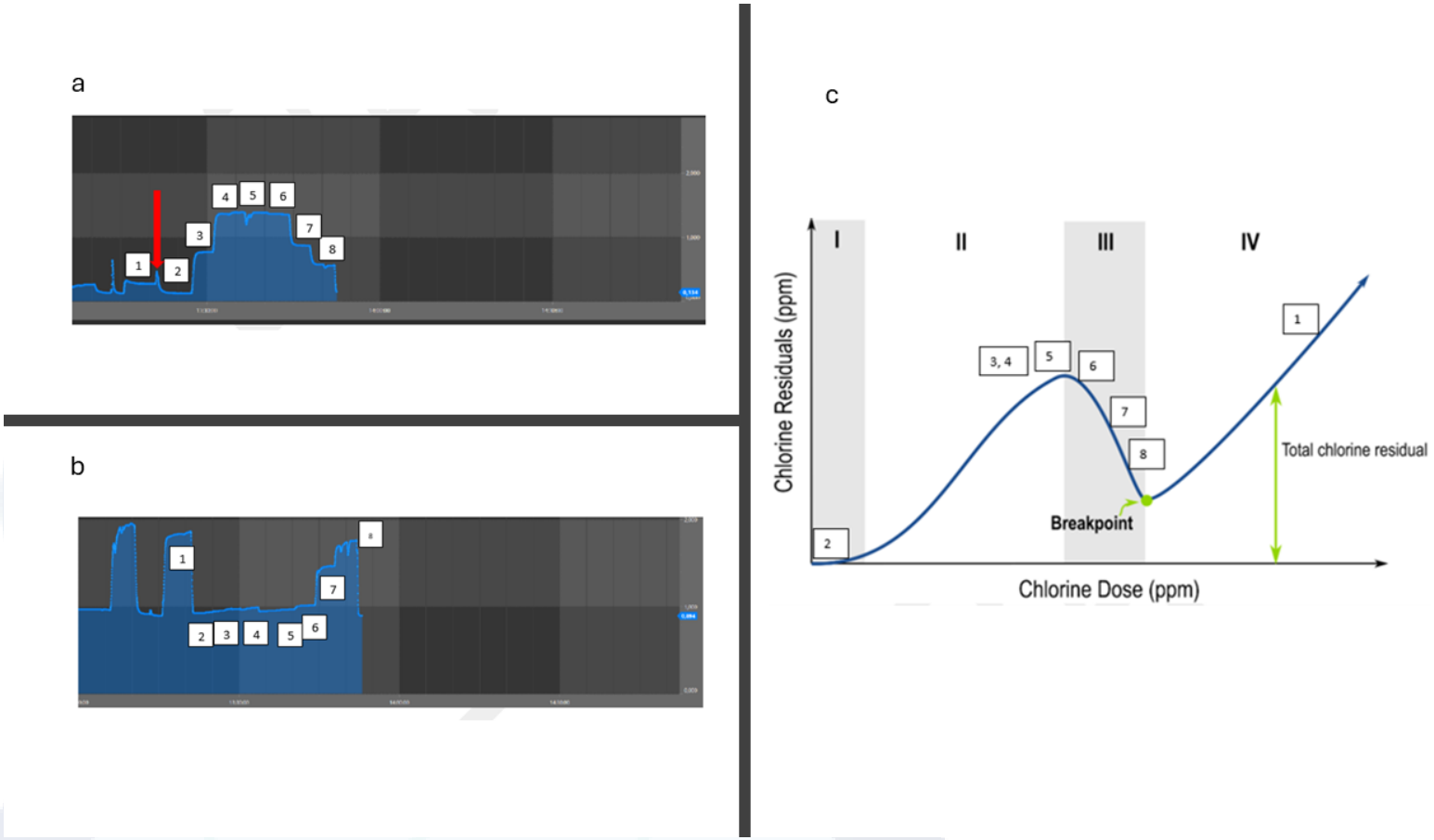


Figure 3. Absorbance-based interpretation of chloramine speciation. a) Absorbance at λ = 245 nm, showing the characteristic monochloramine (MCA) peak. Transitional chloramine formation occurs between points 1 and 2, while MCA decreases at points 7 and 8. b) Absorbance at λ = 295 nm, associated with dichloramine (DCA) formation. Free chlorine conditions are observed at point 1, with increasing DCA contribution at points 7 and 8. c) Interpretation of chloramine speciation based on absorbance signals.

4. Pilot plant configuration and operation

The operation of the components listed in **Table 1** and integrated within the system configuration shown in **Figure A1** is managed by an automation system that can be operated in manual or automatic mode. In manual mode, the operator controls the recirculation valve, centrifugal pumps P01/P02 and peristaltic dosing pumps PD01–PD04. In automatic mode, the start-up sequence is executed stepwise, including opening of the recirculation valve to block outlet flow, starting of the centrifugal pumps after configurable waiting times, activation of the peristaltic dosing pumps, and closure of the recirculation valve to enable outlet flow. The stop sequence opens the recirculation valve, sets the peristaltic pumps to zero dosing, and stops the centrifugal pumps. Alarms can be set to interrupt the automatic sequence when required. Switching between manual and automatic mode stops all active equipment and places the valve in recirculation mode.

The dosing strategy combines flow-based and pH-based control. PD02 and PD03 are regulated either according to the flowrate measured by FT01 or according to a fixed percentage set by the operator. PD01 and PD04 are regulated according to pH or fixed setpoints, with the operator selecting whether pH1, pH2 or the instantaneous average of both pH measurements is used for control. The pH control logic distinguishes between acidic, neutral and basic conditions and regulates the relevant dosing pump according to the pH setpoint and selected pH input.

The automation system also acquires monochloramine sensor data for concentration display and dichloramine sensor data for absorbance interpretation, including a colour-coded indicator for acceptable, limit or unacceptable dichloramine absorbance values. These signals were used for process visualisation and operational interpretation during pilot testing.

Table 1. Summary of MITO3X® pilot components and instrumentation

Category	P&ID tag(s)	Component / function	Role in the Madrid chloramination-control application
Hydraulic pumping	P01–P02	Centrifugal pumps	Provide circulation through the MITO3X® prototype and support operation of the pilot hydraulic line.
Reagent dosing	PD01–PD04	Dosing pumps	Dose reagents required for chloramination and pH/process control.
Reagent storage	TK01	Liquid reagent storage tank, NaOH	Reagent tank for sodium hydroxide / pH adjustment.
Reagent storage	TK02	Liquid reagent storage tank, NH_4OH	Ammonium hydroxide source used for ammonia addition during chloramine generation.
Reagent storage	TK03	Liquid reagent storage tank, NaClO	Sodium hypochlorite source used for chlorine addition during chloramine generation.
Reagent storage	TK04	Liquid reagent storage tank, other	Additional reagent storage, if needed
Mixing / reaction	SM01	Static mixer	Promotes reagent mixing and contact in the water line.
Mixing / reaction	CSP01	Contact section	Provides controlled contact section after reagent addition/mixing.
Process monitoring	FT01	Flow meter	Measures water flowrate during pilot operation and experimental tests.
Process monitoring	PI01–PI02	Pressure meters	Monitor hydraulic pressure at relevant points of the pilot line.
Process monitoring	pH01–pH02	pH sensors	Monitor pH for process interpretation and/or pH-based dosing control.
Process monitoring	FA01–FA02	Absorbance sensors	Support absorbance-based monitoring relevant to chloramine species interpretation.
Dosing flow control	FDM01–FDM03	Dosing manual flow-regulation valves	Manually regulate dosing-line flow conditions.
Process flow control	VR01	Modulating flow-regulation valve	Regulates process flow/recirculation conditions.
Manual valves	VM01–VM04	Manual flow-regulation valves	Enable manual hydraulic control and isolation of parts of the line.
Dosing valves	VMPD01–VMPD04	Dosing manual flow-regulation valves	Enable manual control of dosing lines linked to reagent addition.
Check valves	VC01–VC08	Check valves	Prevent backflow in hydraulic and dosing lines.
Sampling	FApH	Sampling points	Enable collection of samples for laboratory analysis and operational checks.
Control and electrical system	PLC01	Management PLC and electrical panel	Provides automation, control, HMI/electrical management and data acquisition functions.
Discharge / outlet	DG01 / outlet	Discharge tank and outlet	Support discharge/outlet operation after treatment/contact.

5. Pilot commissioning and operating timeline

The initial validation of the MITO3X® concept for chloramination control was carried out during preliminary testing conducted in April 2024 in Puglia (Italy). These tests confirmed the feasibility of in-situ monochloramine generation using sodium hypochlorite and ammonium hydroxide under controlled conditions. During this phase, a limitation associated with absorbance monitoring at 295 nm, relevant for dichloramine detection, was identified and subsequently resolved prior to deployment through sensor replacement and calibration. These preliminary tests were performed at relatively high flowrates, of approximately 6 m^3/h.

Following preliminary validation, the MITO3X® system was installed at the Valmayor DWTP and integrated into the existing pilot facilities. The installation included hydraulic connection to the pilot line, implementation of reagent dosing systems and configuration of monitoring and control components. The commissioning phase involved verification of hydraulic operation, dosing systems, sensor performance and automation sequences under both manual and automatic modes. During commissioning, the system was operated at a reduced flowrate of approximately 1.6 m^3/h to ensure stable start-up and controlled system operation.

Initial commissioning tests were carried out in September 2024, focusing on qualitative chloramine generation and identification of operating conditions suitable for monochloramine formation. These tests confirmed the successful integration of the system under real operating conditions and supported the optimisation of operational parameters. Subsequent testing confirmed stable system performance and consistent monochloramine generation, with initial results documented in October 2024.

The main experimental campaigns were conducted in June 2025 and generated the dataset used for the assessment of DBP and nitrosamine formation under different chloramination scenarios and water matrices. These experiments were conducted under stable operating conditions at flowrates between 2.0 and 2.3 m^3/h.

Based on the successful installation, commissioning and operation of the system, the MITO3X® pilot plant was considered fully operational at the Valmayor facilities as of 10 July 2025. This corresponds to Milestone 15 of the intoDBP project at M44, which was therefore accomplished earlier at M32 (“Pilot plant fully operative at CANAL”). The overall development and operation timeline is summarised in **Table 2.** Further details on the June 2025 experimental campaigns are provided in **Table 3** and further described in Section 6.1.

Table 2. Timeline of MITO3X® pilot development, commissioning and operation at the Madrid case study

Date / period	Location	Activity	Relevance to D4.2
April 2024 (M17)	Puglia, Italy	Preliminary MITO3X® test before Madrid operation	Prototype/pre-commissioning evidence; identified UV-295 nm limitation
September 2024 (M22)	Valmayor, Madrid	Madrid qualitative chloramine-generation tests	First documented Madrid operating tests
October 2024 (M23)	Madrid report	Documentation of Madrid in-situ monochloramine generation tests	Evidence of MCA generation and operational learning
June 2025 (M31)	Valmayor, Madrid	DBP and nitrosamine experimental campaigns	Main experimental evidence for D4.2 results
July 2025 (M32)	CANAL/Valmayor	Analytical results confirmed hypotheses and the pilot is considered fully operative (Milestone 15) and data analysis for D4.2.	Operational-readiness statement based on installation, commissioning and operating tests

6. Experimental methodology and test conditions

Following commissioning and confirmation of stable operation (Section 5), experimental campaigns were carried out to evaluate the performance of the MITO3X® system under controlled chloramination conditions. The methodology was designed to compare different chloramination scenarios and to assess their effect on DBP and nitrosamine formation.

6.1 Experimental design and operating conditions

The full set of experimental conditions, including sample identification, water matrices and tested scenarios is summarised in **Table 3.** The experiments were conducted under stable operating conditions at flowrates between 2.0 and 2.3 m^3/h.

The chloramination tests were performed using commercial sodium hypochlorite as the chlorine source and ammonium hydroxide as the ammonia source. The relative dosing of these reagents was varied to generate controlled chloramine-speciation conditions.

Different chlorine/ammonia dosing configurations were applied to produce hypochlorite-only reference conditions, monochloramine-rich conditions, mixed monochloramine/dichloramine conditions and dichloramine-rich conditions. The target chloramine condition was identified using absorbance at selected wavelengths. Monochloramine formation was followed using the absorbance signal around 245/248 nm, while dichloramine formation was interpreted using the absorbance signal around 295 nm.

The hydraulic design capacity of the MITO3X® Madrid pilot was confirmed as 10 m^3/h. However, the chloramination campaigns were carried out at lower controlled flowrates to ensure sufficiently high concentrations of chloramine species and DBPs/nitrosamines for laboratory analysis and detection.

The experimental conditions described above define the matrix of scenarios analysed in terms of DBP and nitrosamine formation, as described in Section 6.2.

Table 3. Experimental matrices and chloramination scenarios tested during the June 2025 Madrid MITO3X® campaigns.

Date	Water matrix	Sample ID(s)	Scenario tested	Purpose
09/06/2025	Raw water	Sample 1	100% MCA	Baseline monochloramine generation in raw water
09/06/2025	Raw water + polyDADMAC	Sample 2	100% MCA	Test of MCA in the presence of an NDMA precursor
09/06/2025	Ozone-preoxidised water	Sample 3	100% MCA	Effect of MCA after ozone pre-oxidation
09/06/2025	Ozone-preoxidised water	Sample 4	50% MCA + 50% DCA	Effect of mixed chloramine speciation after ozone pre-oxidation
09/06/2025	Ozone-preoxidised water	Sample 5	100% DCA	Effect of DCA-rich conditions after ozone pre-oxidation
09/06/2025	Ozone-preoxidised water	Sample 6	Hypochlorite only	Free-chlorine reference condition
30/06/2025	Ozone-preoxidised water + H_2O_2	Sample 7	100% MCA	Effect of MCA after ozone/H_2O_2 advanced oxidation
30/06/2025	Ozone-preoxidised water + H_2O_2	Sample 8	50% MCA + 50% DCA	Effect of mixed chloramine speciation after ozone/H_2O_2 advanced oxidation
30/06/2025	Ozone-preoxidised water + H_2O_2	Sample 9	100% DCA	Effect of DCA-rich conditions after ozone/H_2O_2 advanced oxidation
30/06/2025	Ozone-preoxidised water + H_2O_2	Sample 10	Hypochlorite only	Free-chlorine reference condition after ozone/H_2O_2 advanced oxidation
30/06/2025	Ozone-preoxidised water + polyDADMAC	Sample 11	100% MCA	Effect of MCA in preoxidised water with NDMA precursor addition
30/06/2025	Ozone-preoxidised water + polyDADMAC	Sample 12	50% MCA + 50% DCA	Effect of mixed chloramine speciation in preoxidised water with NDMA precursor addition
30/06/2025	Ozone-preoxidised water + polyDADMAC	Sample 13	100% DCA	Effect of DCA-rich conditions in preoxidised water with NDMA precursor addition
30/06/2025	Ozone-preoxidised water + polyDADMAC	Sample 14	Hypochlorite only	Free-chlorine reference condition in preoxidised water with NDMA precursor addition

6.2 Water matrices and test scenarios

The experimental campaigns described in Section 6.1 were applied to a range of water matrices representative of the Valmayor treatment system, selected to evaluate the influence of precursor content and upstream treatment processes on chloramine formation and DBP generation.

The tested matrices included:

- raw water
- raw water amended with polyDADMAC as a nitrosamine precursor
- ozone-preoxidised water
- ozone-preoxidised water treated with ozone and hydrogen peroxide (advanced oxidation)
- ozone-preoxidised water amended with polyDADMAC

These matrices were combined with the different chloramination scenarios described above to enable systematic comparison of DBP and nitrosamine formation under varying conditions of precursor content and treatment history. The addition of polyDADMAC in selected tests was used to simulate the presence of nitrogenous precursors and to evaluate the potential formation of N-nitrosodimethylamine (NDMA) under controlled chloramination conditions. The experimental conditions described above define the matrix of scenarios analysed in terms of DBP and nitrosamine formation, as described in section 6.3.

6.3 Analytical parameters and methods

Analytical measurements were carried out by ICRA using the methodologies defined within the intoDBP project framework. The analytical parameters used to assess DBP and nitrosamine formation are summarised in **Table 4**. The analytical assessment focused on regulated and emerging DBPs relevant to chloramination processes.

The main parameter groups included:

- **Trihalomethanes (THMs):**
  chloroform, bromodichloromethane, dibromochloromethane, bromoform and total THMs
- **Haloacetic acids (HAAs):**
  individual haloacetic acids and HAA5
- **Nitrosamines:**
  particular focus on N-nitrosodimethylamine (NDMA), and additional nitrosamines (e.g. NDEA, NDBA)

Supporting parameters included:

- chlorine speciation (free chlorine and combined chlorine);
- absorbance signals at approximately 245–248 nm and 295 nm;
- total organic carbon (TOC) and additional water-quality indicators where available.

These parameters were used to interpret DBP and nitrosamine formation in relation to chloramine speciation, water matrix characteristics and operating conditions.

The analytical methods applied for DBP and nitrosamine determination follow the procedures described in Deliverable D2.1.

Trihalomethanes (THMs):

Samples were subjected to liquid–liquid extraction using methyl tert-butyl ether (MTBE) under acidic conditions (pH ≈ 3.5), with the addition of an internal standard. Quantification was performed using gas chromatography coupled with tandem mass spectrometry (GC-MS/MS).

Haloacetic acids (HAAs)

Samples were acidified and extracted with MTBE, followed by derivatisation through methylation using acidic methanol to increase compound volatility. The derivatised compounds were subsequently analysed by GC-MS/MS.

N-nitrosamines

Nitrosamines were analysed after preconcentration of large-volume samples (approximately 1 L) using solid-phase extraction (SPE) with coconut carbon cartridges. Compounds were eluted with dichloromethane, concentrated to a reduced volume and quantified using GC-MS/MS in selected reaction monitoring (SRM) mode.

Table 4. Summary of analytical parameters used to assess DBP and nitrosamine formation in the Madrid MITO3X® chloramination-control tests.

Parameter group	Compounds / indicators included	Relevance for D4.2
Trihalomethanes	Chloroform, bromodichloromethane, dibromochloromethane, bromoform, total THMs	Main regulated DBP group used to assess the effect of chloramination scenario on THM formation
Haloacetic acids	Individual HAA species and HAA5	Main regulated DBP group used to assess DBP formation under MCA, DCA, mixed chloramine and hypochlorite-only conditions
Halonitromethanes	Trichloronitromethane / chloropicrin, where reported	Additional nitrogenous/halogenated DBP indicator relevant to chlorination/chloramination chemistry
Nitrosamines	NDMA, NDEA, NDBA and other analysed nitrosamines where available	Key emerging DBP group for the Madrid chloramination-control case study, with NDMA as the main target compound
Chlorine speciation	Free chlorine, monochloramine, combined chlorine / dichloramine-related signal	Used to confirm or interpret the intended chloramine-generation scenario
Absorbance signals	Absorbance around 245/248 nm and 295 nm	Used for process interpretation of MCA and DCA formation during MITO3X® operation
General water-quality parameters	TOC and supporting parameters where available	Used to interpret DBP and nitrosamine formation in relation to precursor content and water matrix

7. Results and discussion

7.1 Chloramine generation and process control

The experimental campaigns confirmed that the MITO3X® system enables controlled generation of chloramine species under pilot-scale conditions and across the defined experimental scenarios (**Table 3**) including monochloramine-rich, mixed chloramine and dichloramine-rich conditions. Monochloramine (MCA) formation was successfully achieved through the controlled dosing of sodium hypochlorite and ammonium hydroxide, with stable operation maintained during the experimental campaigns.

The absorbance signal around 245–248 nm provided a consistent indicator of monochloramine presence. A representative example of an absorbance spectrum illustrating this behaviour is provided in Annex D (**Figure D1**). By adjusting the chlorine-to-ammonia dosing ratio, the system allowed controlled transitions between monochloramine-rich, mixed chloramine and dichloramine-rich conditions. The absorbance signal around 295 nm was used as an indicator of conditions associated with dichloramine formation.

Figure 4 illustrates the relationship between chlorine and ammonia dosing conditions and the resulting chloramine speciation, highlighting the transition between MCA-rich and DCA-rich regimes. These results demonstrate that the MITO3X® platform enables reproducible control of chloramine generation under defined operating conditions and provides a suitable experimental tool for investigating the relationship between chloramine speciation and DBP formation.

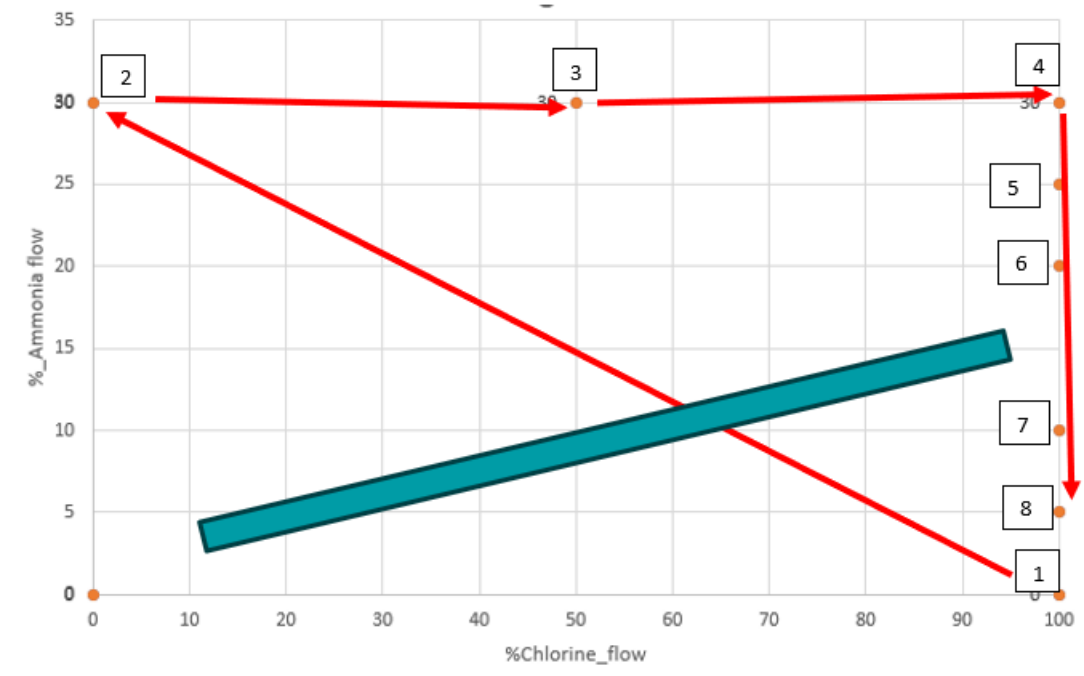


Figure 4. Chlorine and ammonia dosing profile showing the breakpoint position (green bar). Numbered points represent different operating conditions along the dosing curve, ranging from free chlorine (point 1), through monochloramine-dominated conditions (points 2–5), to dichloramine-rich conditions (points 6–8).

7.2 DBP and nitrosamine formation under different conditions

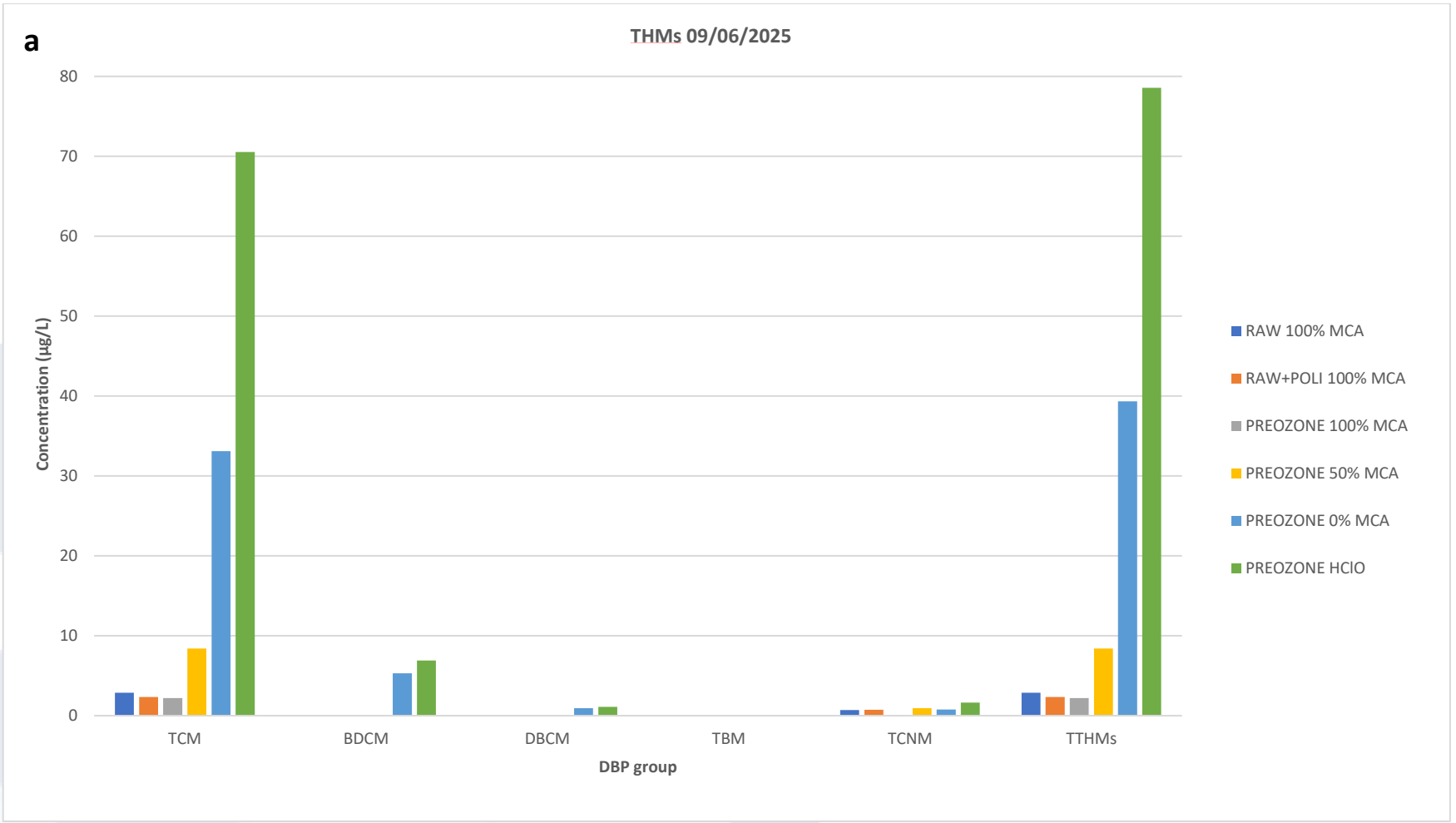
The experimental campaigns generated a dataset on the formation of DBPs and nitrosamines under different chloramination scenarios and water matrices. **Figure 5** and **Figure 6** summarize the formation of trihalomethanes (THMs, expressed as total THMs), haloacetic acids (HAAs, expressed as HAA5), and nitrosamines (primarily NDMA) across the tested conditions, including monochloramine-rich, mixed chloramine, dichloramine-rich and hypochlorite-only scenarios. The values presented in **Figure 5** and **Figure 6** correspond to two separate experimental campaigns conducted on 9 June 2025 and 30 June 2025, respectively, and are derived from the analytical dataset reported in Annex C, **Table C1**. Nitrosamine formation, represented primarily by NDMA, is discussed based on the analytical results in **Table C1**.

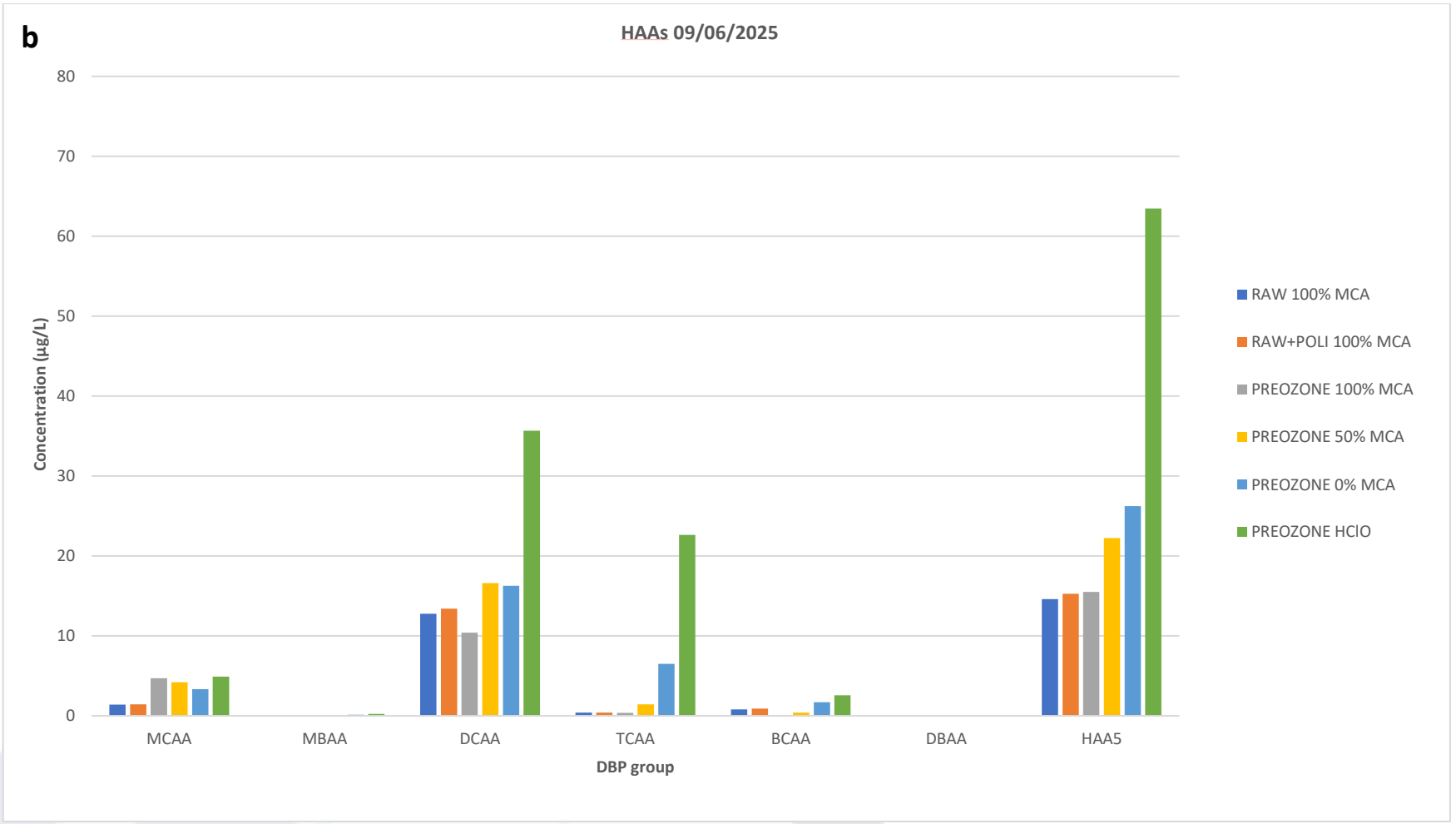
The results show a consistent trend in which hypochlorite-only conditions produce the highest concentrations of THMs, while monochloramine-rich conditions result in significantly lower THM formation. Similar behaviour was observed for HAAs, with lower concentrations generally associated with monochloramine-rich operation compared to dichloramine-rich and free-chlorine conditions.

Nitrosamine formation, particularly NDMA, was also influenced by chloramine speciation and precursor presence. Higher NDMA formation was generally associated with dichloramine-rich and mixed chloramine conditions, especially in matrices amended with polyDADMAC. In contrast, monochloramine-rich conditions were associated with lower NDMA formation across the tested matrices.

The influence of water matrix and upstream treatment processes was clearly evident. Ozone-preoxidised and ozone/H_2O_2-treated waters generally showed lower DBP formation under certain conditions, indicating the role of precursor removal or transformation. However, the observed trends varied depending on the combination of water matrix and chloramination scenario, reflecting the complexity of DBP formation mechanisms.

Overall, the results demonstrate that both chloramine speciation and water matrix characteristics play a key role in determining DBP and nitrosamine formation under chloramination conditions.


a
THMs 09/06/2025
80
70
60
50
40
30
20
10
0
Concentration (µg/L)
TCM
BDCM
DBCM
TBM
TCNM
TTHMs
DBP group
RAW 100% MCA
RAW+POLI 100% MCA
PREOZONE 100% MCA
PREOZONE 50% MCA
PREOZONE 0% MCA
PREOZONE HClO


b
HAAs 09/06/2025
80
70
60
50
40
30
20
10
0
Concentration (µg/L)
MCAA
MBAA
DCAA
TCAA
BCAA
DBAA
HAA5
DBP group
RAW 100% MCA
RAW+POLI 100% MCA
PREOZONE 100% MCA
PREOZONE 50% MCA
PREOZONE 0% MCA
PREOZONE HClO

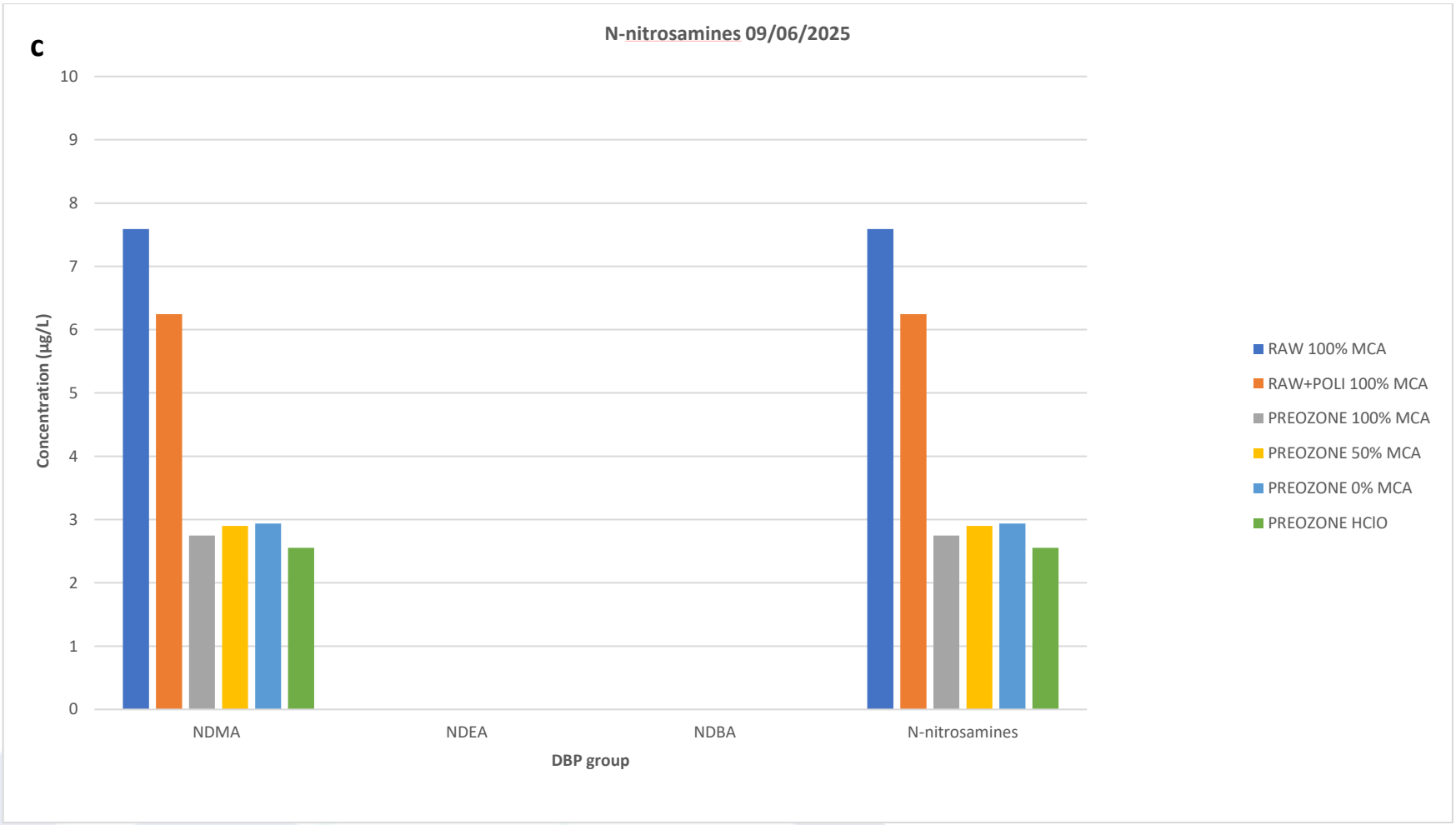
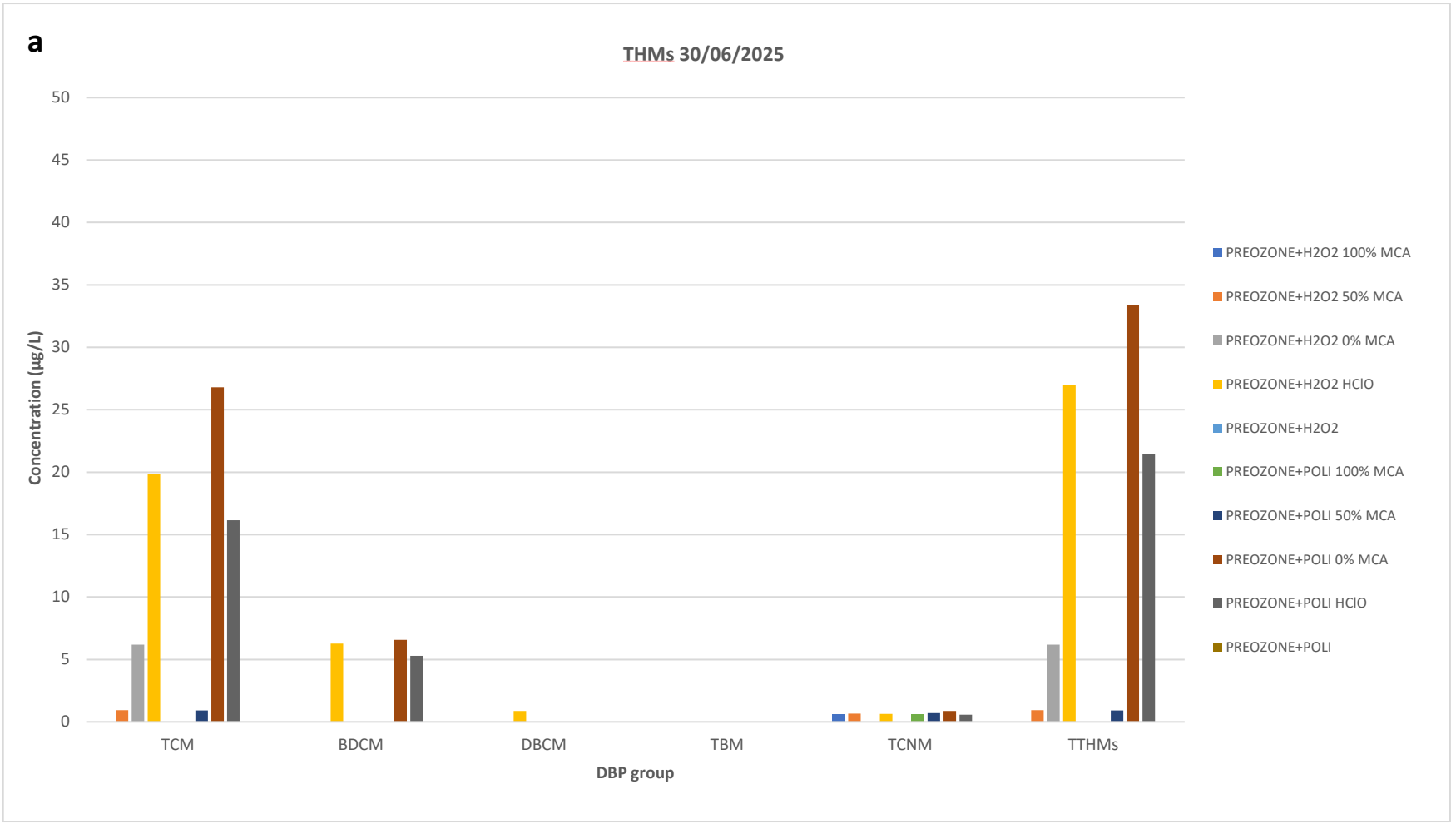
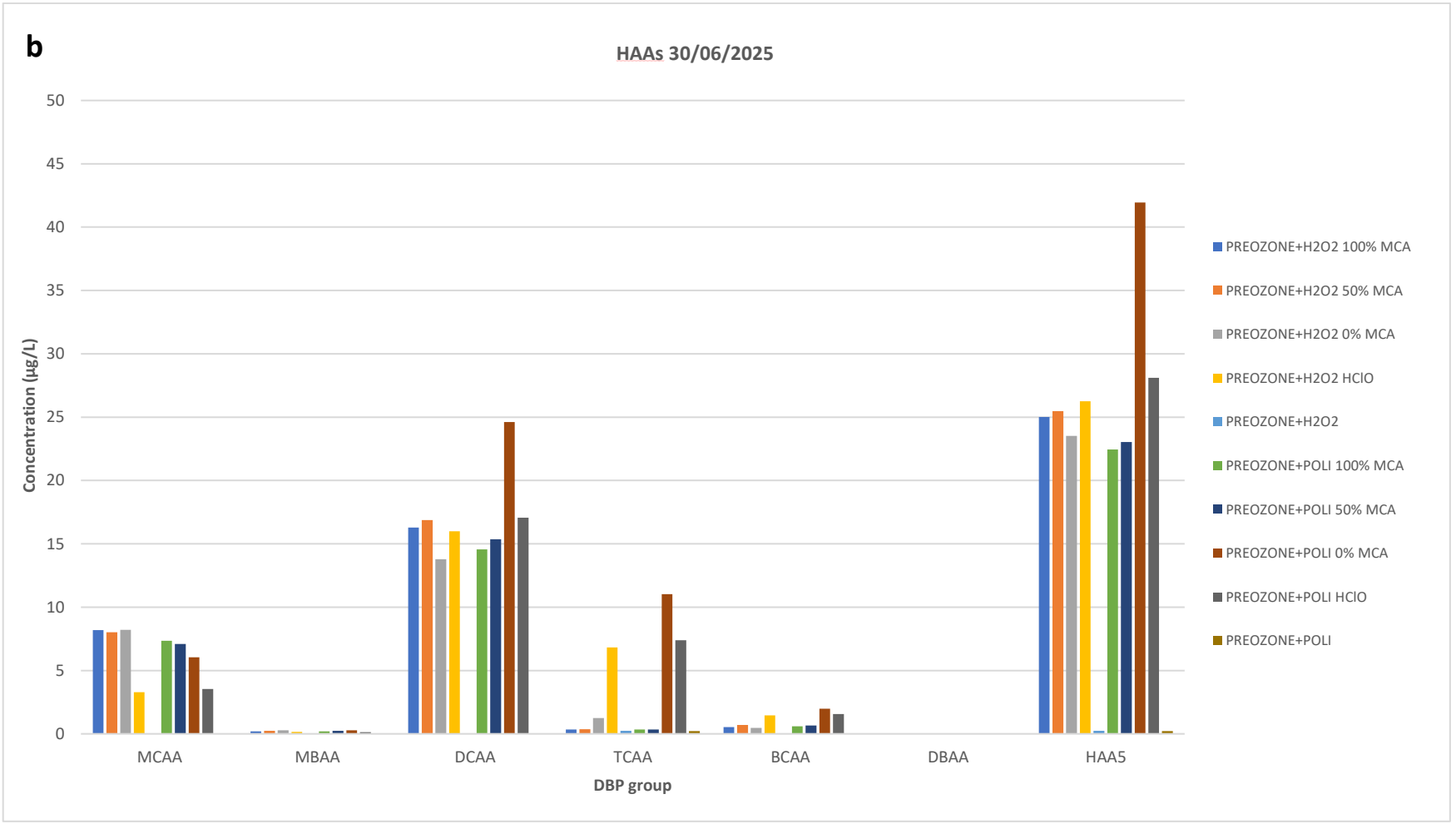


Figure 5. Summary of DBP formation, grouped as THMs (a), HAAs (b) and N-nitrosamines (c), in different water matrices and treatment scenarios, for the 09/06/2025 sample batch. The tested scenarios include MCA-rich (100% MCA), MCA+DCA (50% each), DCA-rich (0% MCA) and hypochlorite-only conditions, as well as raw water conditions as matrix reference.


a
THMs 30/06/2025
Concentration (µg/L)
50
45
40
35
30
25
20
15
10
5
0
TCM
BDCM
DBCM
TBM
TCNM
TTHMs
DBP group
PREOZONE+H2O2 100% MCA
PREOZONE+H2O2 50% MCA
PREOZONE+H2O2 0% MCA
PREOZONE+H2O2 HClO
PREOZONE+H2O2
PREOZONE+POLI 100% MCA
PREOZONE+POLI 50% MCA
PREOZONE+POLI 0% MCA
PREOZONE+POLI HClO
PREOZONE+POLI


b
HAAs 30/06/2025
Concentration (µg/L)
50
45
40
35
30
25
20
15
10
5
0
MCAA
MBAA
DCAA
TCAA
BCAA
DBAA
HAA5
DBP group
PREOZONE+H2O2 100% MCA
PREOZONE+H2O2 50% MCA
PREOZONE+H2O2 0% MCA
PREOZONE+H2O2 HClO
PREOZONE+H2O2
PREOZONE+POLI 100% MCA
PREOZONE+POLI 50% MCA
PREOZONE+POLI 0% MCA
PREOZONE+POLI HClO
PREOZONE+POLI

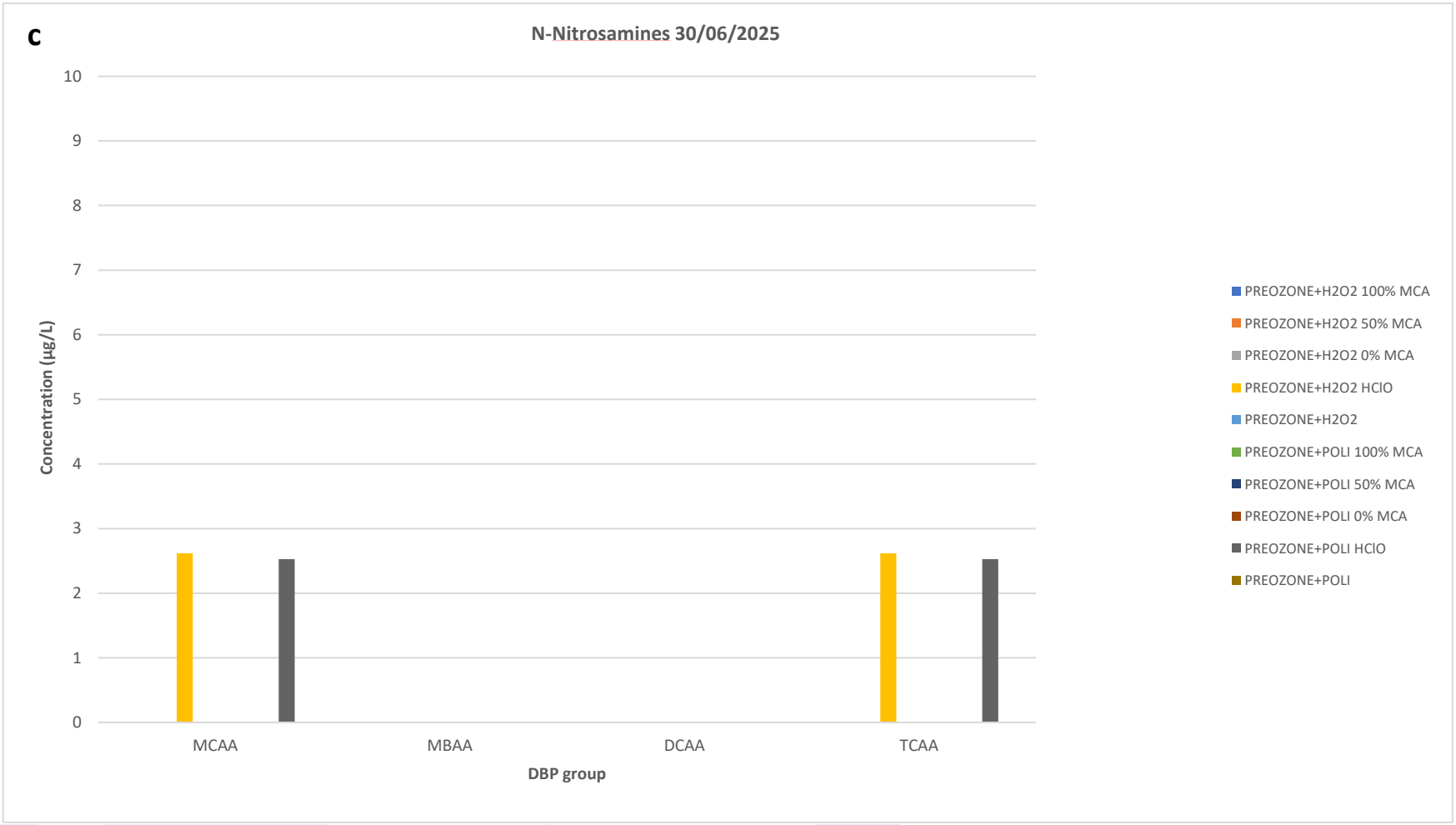


Figure 6. Summary of DBP formation, grouped as THMs (a), HAAs (b) and N-nitrosamines (c), in different water matrices and treatment scenarios, for the 30/06/2025 sample batch. The tested scenarios include MCA-rich (100% MCA), MCA+DCA (50% each), DCA-rich (0% MCA) and hypochlorite-only conditions, as well as raw water conditions as matrix reference.

7.3 Discussion

Across the tested chloramination scenarios, a clear and consistent trend is observed in DBP formation. DBP levels increase progressively from monochloramine-dominated conditions (100% MCA) to mixed chloramine conditions (50% MCA + 50% DCA), and further to dichloramine-rich conditions (0% MCA), reaching the highest levels under hypochlorite-only conditions. This pattern is observed for THMs, HAAs and nitrosamines across all tested water matrices, highlighting chloramine speciation as a primary controlling factor in DBP formation.

The results confirm that monochloramine-rich operation is systematically associated with lower formation of trihalomethanes (THMs), haloacetic acids (HAAs) and N-nitrosodimethylamine (NDMA) compared with dichloramine-rich and hypochlorite-only conditions. This reinforces the importance of maintaining appropriate chlorine-to-ammonia ratios and pH control during chloramination to favour monochloramine formation and minimise dichloramine formation.

At the same time, the results demonstrate that DBP formation is strongly influenced by water matrix characteristics. Variations observed between raw, pre-oxidised and polyDADMAC-amended waters indicate that precursor availability and transformation processes play a key role in determining DBP yields under different operating conditions. In particular, the presence of nitrogenous precursors enhances the potential for NDMA formation, especially under dichloramine-rich conditions. Conversely, pre-oxidation processes such as ozonation and advanced oxidation can modify precursor composition and reduce DBP formation under certain scenarios, although the effect depends on the specific treatment combination.

Taken together, these findings highlight the importance of integrating controlled chloramination with an understanding of upstream treatment processes and water-quality characteristics. The combined influence of chloramine speciation and water matrix should therefore be considered when optimising disinfection strategies aimed at minimising both regulated and emerging DBPs.

8. Conclusions and limitations

This deliverable presents the implementation and validation of the MITO3X® application for chloramination control at the Madrid case study. The results demonstrate that the technology can be successfully integrated into a pilot-scale drinking water treatment environment and operated under controlled conditions.

8.1 Conclusions and main achievements

The experimental results confirm that the MITO3X® system can be used to achieve controlled in-situ chloramine generation and to systematically evaluate the impact of chloramine speciation on DBP formation. Consistent with the results discussed in Section 7, monochloramine-rich operation was consistently associated with lower formation of trihalomethanes (THMs), haloacetic acids (HAAs) and N-nitrosodimethylamine (NDMA) compared with dichloramine-rich and hypochlorite-only conditions. These findings support the relevance of controlled chloramination strategies for DBP minimisation in drinking water treatment.

The results also highlight the importance of considering water matrix characteristics and upstream treatment processes, as these influence the availability and reactivity of DBP precursors.

Based on the implementation and experimental validation carried out at the Madrid case study, the main achievements of the MITO3X® chloramination-control application can be summarised as follows:

- successful integration of the MITO3X® system into the Valmayor pilot plant under real operational conditions
- demonstration of controlled in-situ monochloramine generation using sodium hypochlorite and ammonium hydroxide
- ability to operate under different chloramination scenarios (monochloramine-rich, dichloramine-rich, mixed chloramine and free-chlorine conditions), enabling systematic comparison of treatment performance and detection of spectra associated with proper monochloramine generation
- implementation of absorbance-based monitoring for real-time interpretation of chloramine speciation
- generation of experimental evidence linking chloramine speciation and water matrix to DBP and nitrosamine formation

These findings support the practical relevance of chloramine speciation control as an effective strategy for minimising DBP formation in drinking water treatment.

Based on the installation, commissioning and operation of the system, the MITO3X® pilot plant was considered fully operative at CANAL/Valmayor as of 10 July 2025 (M32), supporting the achievement of Milestone 15 within the intoDBP project, ahead of its planned delivery in M44.

8.2 Limitations

The results presented in this deliverable should be interpreted within the context of the specific experimental conditions tested. The campaigns were designed to demonstrate controlled chloramination and DBP formation trends under representative conditions. While additional experimental scenarios could further refine the understanding, the work fulfils the objective of demonstrating the applicability and performance of the MITO3X® system under pilot-scale conditions.

The experimental flow rate used for the experiments was lower than the pilot plant's nominal design flow rate. This was a purely operational decision to ensure the highest possible concentration of the different species generated, taking into account both the purity of the precursors and the detection and quantification limits of the analytical methods employed. The pilot plant is fully operational and can operate at its nominal design flow rate. In addition, the interaction between chloramine speciation, precursor composition and upstream treatment processes is complex. The trends observed in this study should therefore be interpreted with caution when extrapolating to different water treatment contexts. Each application requires a prior study and needs analysis, based on the precursors available at any given time as well as their purity.

Another self-imposed limitation of scope was to test only the preparation of concentrated monochloramine solutions and to those such preformed monochloramine into the water to be disinfected. Another application would be to direct gaseous reagents (ammonia, chlorine) into the mainstream of the water to be disinfected. This monochloramine formation in situ was however discarded in the planning phase as this would require a pressurization (although perhaps only to 1-2 bars above atmospheric pressure) of the full main water stream, which entails an increased pumping cost and energy.

ANNEXES

Annex A. Pilot design and configuration

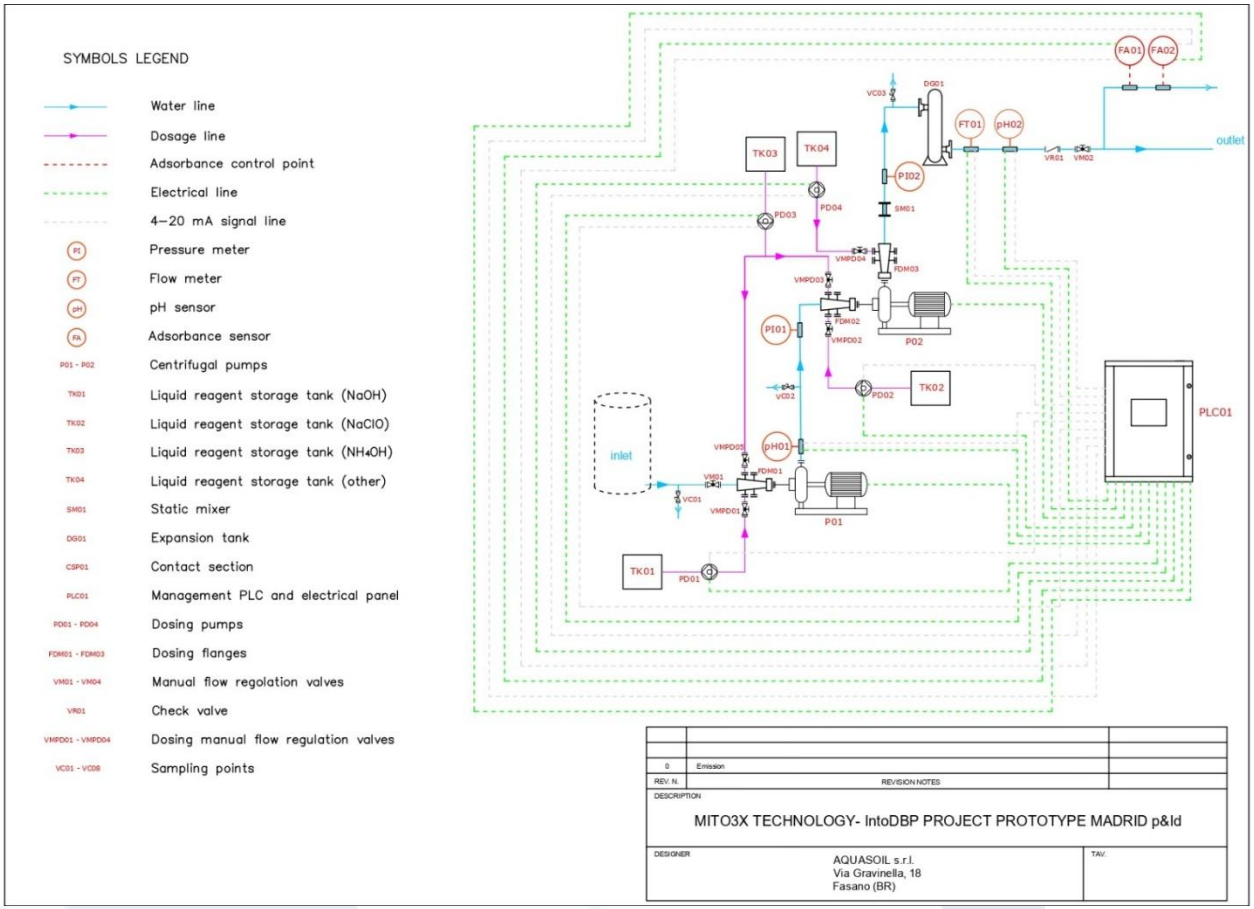


Figure A1. P&ID of the MITO3X® Madrid pilot configuration.

Annex B. Automation and control strategy

The MITO3X® Madrid pilot is equipped with an automation system designed to support controlled operation of the chloramination process under different operating modes and dosing configurations.

Operating modes

The system can operate in two main modes:

- Manual mode, where the operator directly controls the main equipment, including centrifugal pumps (P01–P02), dosing pumps (PD01–PD04) and the recirculation valve.

- Automatic mode, where the system follows a predefined sequence for start-up, operation and shutdown.

Start-up and shutdown sequences

In automatic mode, the system executes a sequential start-up procedure, including opening of the recirculation valve, stepwise activation of centrifugal pumps and dosing pumps, and transition to normal operation through closure of the recirculation valve. The shutdown sequence follows the reverse process, ensuring safe stopping conditions by opening the recirculation valve, setting dosing pumps to zero and stopping centrifugal pumps. Alarm conditions can trigger automatic shutdown.

Dosing control strategy

The dosing strategy combines flow-based and pH-based control approaches:

- Flow-based control is used to regulate sodium hypochlorite and ammonium hydroxide dosing according to the measured flowrate.

- pH-based control is applied to specific dosing pumps, allowing the system to maintain the target pH conditions required for chloramine formation.

The operator can select between different control inputs, including individual pH sensors or averaged values, and adjust setpoints accordingly.

Monitoring and process feedback

The system integrates process monitoring data, including absorbance signals related to monochloramine and dichloramine formation, flowrate and pH measurements. These signals are used for real-time process visualisation and operational interpretation during pilot testing. The automation system supports flexible adjustment of operating parameters, enabling controlled execution of the experimental scenarios described in this deliverable.

Annex C. Analytical results

This annex presents the analytical results obtained during the June 2025 experimental campaigns carried out at the Valmayor pilot facilities. The dataset includes trihalomethanes (THMs), haloacetic acids (HAAs) and nitrosamines measured under the different chloramination scenarios and water matrices tested. These results are summarised in **Table C1**.

Table C1. Analytical results for DBPs and nitrosamines under the tested conditions. All concentrations are reported in µg/L for DBPs and in ng/L for nitrosamines. Values below the limit of detection (LOD) are indicated as <LOD. (1) indicates that only one replicate was analysed. n.a. indicates not available.

		TCM		BDCM		DBCM		TBM		TCNM		MCAA		MBAA		DCAA		TCAA		BCAA		DBAA		NDMA		NDEA		NDBA	
DATE	SAMPLE	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (µg/L)	%RSD	Conc (ng/L)	%RSD	Conc (ng/L)		Conc (ng/L)	Conc (ng/L)
09/06	RAW 100 09/06	2,9	8,9	<LOD	-	<LOD	-	<LOD	-	0,7	4,7	1,4	11,3	<LOD	-	12,8	15,5	0,4	-	0,8	14,2	<LOD	-	7,6	0,5	<LOD	-	<LOD	-
	RAW+POLI 100 09/06	2,3	0,3	<LOD	-	<LOD	-	<LOD	-	0,8	0,1	1,5	16,5	<LOD	-	13,4	19,9	0,4	-	0,9	30,6	<LOD	-	6,2	10,0	<LOD	-	<LOD	-
	PREOZONE 100 09/06	2,2	4,7	<LOD	-	<LOD	-	<LOD	-	<LOD	-	4,7	21,1	<LOD	-	10,4	4,0	0,4	-	<LOD	-	<LOD	-	2,7	6,6	<LOD	-	<LOD	-
	PREOZONE 50 09/06	8,4	0,6	<LOD	-	<LOD	-	<LOD	-	0,9	8,9	4,2	3,9	<LOD	-	16,6	0,9	1,4	-	0,4	1,9	<LOD	-	2,9	5,5	<LOD	-	<LOD	-
	PREOZONE 0 09/06	33,1	n.a. (1)	5,3	n.a. (1)	0,9	n.a. (1)	<LOD	-	0,8	n.a. (1)	3,3	1,2	0,1	16,1	16,3	15,3	6,5	-	1,7	12,8	<LOD	-	2,9	30,7	<LOD	-	<LOD	-
	PREOZONE HClO 09/06	70,5	0,4	6,9	4,0	1,1	0,3	<LOD	-	1,6	1,2	4,9	n.a.	0,2	n.a.	35,7	29,7	22,6	-	2,6	n.a.	<LOD	-	2,6	6,2	<LOD	-	<LOD	-
30/06	PREOZONE+H2O2 100 30/06	<LOD		<LOD	-	<LOD	-	<LOD	-	0,6	3,0	8,2	1,2	0,2	7,0	16,3	0,3	0,3	0,2	0,5	1,3	<LOD	-	<LOD	-	<LOD	-	<LOD	-
	PREOZONE+H2O2 50 30/06	0,9	3,5	<LOD	-	<LOD	-	<LOD	-	0,7	1,5	8,0	0,4	0,2	12,7	16,9	0,4	0,4	1,3	0,7	6,3	<LOD	-	<LOD	-	<LOD	-	<LOD	-
	PREOZONE+H2O2 0 30/06	6,2	0,8	<LOD	-	<LOD	-	<LOD	-	<LOD	-	8,2	2,9	0,3	1,5	13,8	2,3	1,2	7,9	0,5	2,5	<LOD	-	<LOD	-	<LOD	-	<LOD	-
	PREOZONE+H2O2 HClO 30/06	19,9	2,5	6,3	2,8	0,9	5,6	<LOD	-	0,6	4,5	3,3	3,4	0,2	6,4	16,0	0,6	6,8	5,3	1,5	8,9	<LOD	-	2,6	2,5	<LOD	-	<LOD	-
	PREOZONE+H2O2 30/06	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD		0,2	2,0	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-
	PREOZONE+POLI 100 30/06	<LOD		<LOD	-	<LOD	-	<LOD	-	0,6	1,8	7,4	1,8	0,2	1,6	14,6	1,1	0,3	2,5	0,6	0,9	<LOD	-	<LOD	-	<LOD	-	<LOD	-
	PREOZONE+POLI 50 30/06	0,9	5,6	<LOD	-	<LOD	-	<LOD	-	0,7	2,7	7,1	4,1	0,2	9,3	15,4	1,7	0,3	2,9	0,7	0,1	<LOD	-	<LOD	-	<LOD	-	<LOD	-
	PREOZONE+POLI 0 30/06	26,8	3,2	6,6	0,7	<LOD	-	<LOD	-	0,9	1,5	6,0	0,9	0,3	1,9	24,6	0,5	11,0	1,2	2,0	5,6	<LOD	-	<LOD	-	<LOD	-	<LOD	-
	PREOZONE+POLI HClO 30/06	16,2	5,2	5,3	1,1	<LOD	-	<LOD	-	0,6	1,7	3,5	4,7	0,1	7,8	17,1	1,6	7,4	1,0	1,6	1,3	<LOD	-	2,5	2,4	<LOD	-	<LOD	-
	PREOZONE+POLI 30/06	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD		0,2	3,2	<LOD	-	<LOD	-	<LOD	-	<LOD	-	<LOD	-

Annex D. Supporting absorbance spectra

This annex provides a representative example of the absorbance spectra used for the interpretation of chloramine formation during the MITO3X® pilot testing. **Figure D1** presents an example of a UV–Vis absorbance spectrum obtained during the experimental campaigns. The spectrum shows a characteristic absorbance peak in the region around 245–250 nm, which is consistent with conditions associated with monochloramine presence, based on the monitoring approach described in Section 3.2.



Figure D1. Example of UV–Vis absorbance spectrum obtained during MITO3X® operation, showing a characteristic peak in the 245–250 nm region associated with monochloramine-related absorbance signals.

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intoDBP will create innovative tools and strategies to improve water quality management for safe human use and a healthy environment. It focuses on catchment protection and forecasting, transformative drinking water treatment, and real-time monitoring to combat the effects of climate and global change. In particular, intoDBP focuses on pollution and risks related to disinfection byproducts (DBPs). By developing and applying advanced, integrated, and cost-effective sensors and analytical methods, intoDBP will expand knowledge on water quality and DBP precursors to better understand its formation and human exposure in Europe. intoDBP monitoring results will feed into numerical forecasting tools to predict source water changes and formulate climate change adaptation pathways at catchment and treatment scale. intoDBP also develops transformative options for advanced and cost-effective upgrade of water treatment and disinfection.

In the intoDBP consortium researchers, small and large enterprises, communication experts and public services join forces to generate interdisciplinary solutions, that will generate a renewed perspective of drinking water surveillance, support decision-making and governance, and increase system resilience. intoDBP will implement and validate its cross-cutting products in four complementary case studies from three European countries where compliance with DBP regulation currently is an acknowledged challenge. The direct and visible positive impact of intoDBP in the case studies will foster rapid product adoption at a European and global scale, thus strengthening Europe's position and role in the global water market.

Reaching out beyond the water sector itself, intoDBP will directly engage society through surveys to analyze exposure to DBPs, collect data about catchment protection initiatives, create awareness and promote sustainable consumer behavior such as reducing bottled water consumption.



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