



Taynara Carvalho Silva^a, Diego Gomes da Rocha Voris^a,
Monique Cardozo^a, Letivan Gonçalves de Mendonça Filho^a,
Alexandre Martins Castilho^b, Armando Morado Ferreira^c

<http://dx.medra.org/10.17374/CI.2023.105.3.16>

^aInstituto de Defesa Química, Biológica, Radiológica e Nuclear (IDQBRN), Exército Brasileiro,
Avenida das Américas, 28705, Área 4, 23020-470, Rio de Janeiro-RJ, Brazil

^bCentro Tecnológico do Exército (CTEx), Exército Brasileiro, Avenida das Américas, 28705, 23020-470,
Rio de Janeiro-RJ, Brazil

^cChefia de Ensino, Pesquisa, Desenvolvimento e Inovação (EPDI) do Departamento de Ciência e Tecnologia
(DCT), Exército Brasileiro, Praça Duque de Caxias, 25, 7° andar, 20221-260, Rio de Janeiro-RJ, Brazil
monique.cardozo@eb.mil.br

OPCW DESIGNATION: A BRAZILIAN ENDEAVOUR

Created in 2015, the Brazilian Army's CBRN Defence Institute (IDQBRN) now has the first OPCW-designated lab in GRULAC (Group of Latin America and the Caribbean) to analyse environmental samples. The process and history of Brazil's chemical defence activities are outlined to aid aspiring organisations. This achievement emphasises the role of science and technology in fulfilling CWC obligations and promoting a world free of chemical weapons.

Introduction

In the second half of 2021, the Chemical Analysis Laboratory of the Brazilian Army's CBRN Defence Institute (LAQ/IDQBRN) became the first designated laboratory by the Organisation for the Prohibition of Chemical Weapons (OPCW) in the Latin America and Caribbean (GRULAC) region for the analysis of authentic environmental samples.

With the ability to detect evidence of chemical warfare agents (CWAs) both as intact compounds and through their precursors and degradation products, LAQ's unprecedented feat highlights Brazil in the international community as one of the only 21 countries (a total of 26 laboratories) with this status in April 2023 [1], to have laboratories presenting technical competence consistent with the international standards of OPCW to receive actual environmental samples of chemical warfare agents collected during off-site inspections [2].

Brazil is a peaceful nation that has never developed or possessed weapons of mass destruction. Still, it has one of the largest chemical industries in the world [3]. As some substances with legitimate peaceful uses can also be used to make chemical weapons, the so-called dual-use chemicals [2], industry in States Parties that work with CWC-related compounds in their processes are subject to inspections to ensure that there is no deviation from the original purpose [4]. For example, triethanolamine,

a chemical product used to balance the pH in cosmetic preparations, personal hygiene products, and cleaning products, is listed in the CWC Annex on Chemicals [5] as it was a precursor in the production of nitrogen mustard, used in the first world war [6]. Given the relevance of the Brazilian chemical industry, Brazil played a crucial role during CWC negotiations, especially within GRULAC, with the Brazilian Chemical Industry Association (ABIQUIM) advising on workable solutions for the industry as part of the Brazilian delegation [4]. In this regard, Brazil has stood out in its actions to implement the provisions of the CWC, especially regarding annual declarations, control of transfers, and protection activities [7]. In Brazil, the national authority for implementing the CWC is exercised by the Ministry of Science, Technology, and Innovation (MCTI), whose executive secretariat is the General Coordination of Sensitive Goods (CGBE), which chairs, by delegation, the meetings of the Interministerial Commission for Application of the CWC Devices (CIAD) [7]. In addition, Brazil also cooperates closely with OPCW through permanent representation on the Executive Board [2]. Despite all the rigorous control imposed by the Convention and inspection bodies, there is always a risk of accidents or criminal use of these products. In this context, on-site and off-site analyses are the only way to gather factual evidence on the presence (or absence) of chemicals relevant to the CWC [8],



Fig. 1 - SISDQBRNEx organizations and strategic lines of action

being part of the CWC's hallmark verification mechanism. In addition, Brazil was on the scene of several events with international repercussions between 2011 and 2016. As ensuring the safety of the authorities and civilians involved was imperative, structuring reference laboratories to provide an unequivocal identification of threats was a legacy of this period. In line with the National Defense Strategy of Brazil [9] and to improve the response to chemical, biological, radiological and nuclear (CBRN) threats, it was structured the Chemical, Biological, Radiological and Nuclear Defense System of the Brazilian Army (SisDQBRNEx), which brings together capabilities related to the CBRN defence area distributed in different organisations with different functions. Coordination of SISDQBREx is carried out by the Land Operations Command (COTER), which, as the central body of the system, is responsible for preparing and deploying CBRN defence troops, coordinating CBRN defence activities within the scope of the Brazilian Army and improving doctrine [10].

SisDQBRNEx comprises three lines of action (Fig. 1): operational, scientific and pedagogical. The operational one has two troops to be employed: a CBRN Defense battalion (1° Btl DQBRN) and a CBRN Defense Company (Cia DQBRN), whose specific training of professionals in the CBRN area is carried out by the Specialized Instruction School (EsIE). Scientific assistance is provided by the CBRN Defence Institute (IDQBRN), the Military Institute of Engineering (IME) and the Army Institute of Biology (IBEx). Additionally, the Army Logistics Command (COLOG) provides administrative and logistical support, such as equipment maintenance, while the Directorate of Health (DSAU) is responsible for medical care. This entire network comprising the SISDQBRNEx is crucial to discouraging and preventing threats and enhancing national resilience [10].

CBRN in the Brazilian Army

The origins of CBRN defence in Brazil date back to the Interwar Period (1919-1939) [11]. However, it is commonly attributed to the creation of the Specialised Instruction Centre (today, EsIE) in 1943 (Fig. 2). to prepare specialists from the Brazilian Expeditionary Force for its participation in World War II [12]. With this, the Brazilian Army assumed the role of a precursor of military capacity in CBRN defence in the country, taking the responsibility of inducing the area's development and strengthening the military expression of national power [11]. Operational CBRN defence activities date back to 1953 when the Chemical War School Company (Cia Es GQ) was created. This organisation was later replaced in 1987 by the Chemical, Biological, and Nuclear Defense Company (Cia DQBN). When restructuring the Army's CBRN

defence system in 2012, it was decided to expand Cia DQBN into the 1° Btl DQBRN, which stands out as the primary operating unit of the Brazilian Army's DQBRN System. Its members are trained in actions focused on restoring the combat ability of troops impacted by exposure to CBRN agents of war. Specifically, their training involves carrying out reconnaissance of affected areas and identifying and decontaminating materials, sites, and personnel. Scientific research in CBRN Defense in the Army began in 1994 at the Army Technological Center (CTEx) in the for-

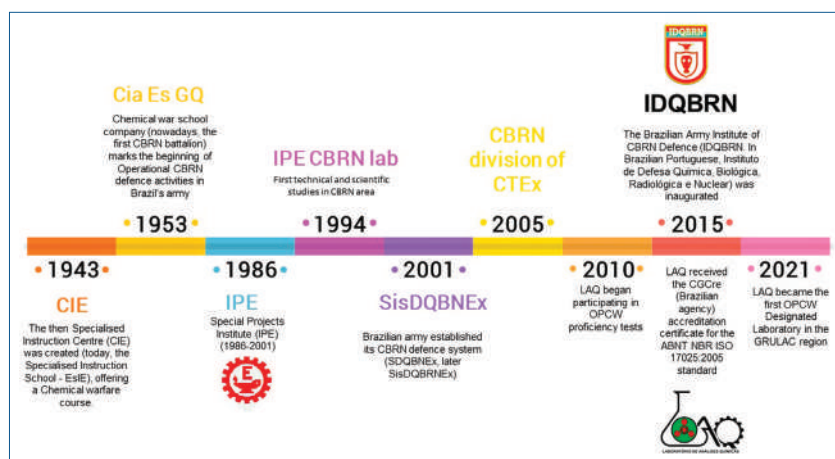


Fig. 2 - Brazilian Army CBRN technical activities milestones

mer Special Projects Institute (IPE). In 2005, CTE's DQBN Division took over IPE's attributions, which led to the creation of the Technical Sections: Chemical Defense Section (SDQ), Biological Defense Section (SDB) and Radiological and Nuclear Defense Section (SDRN). The primary function of the DQBN Division was to provide scientific advice to the Brazilian Army on detection, identification, decontamination, protection, and response against CBRN agents.

The restructuring of SISDQBRNEx in 2012 also transformed the DQBN Division, leading to the creation of IDQBRN in 2015, which consolidated research activities in several areas of CBRN defence. These included: analytical chemistry projects related to compliance with the provisions of the Chemical Weapons Convention, research into new antidotes and potential antimicrobial drugs, analysis of environmental samples to identify pathogenic strains and qualification of equipment and radiological technologies, as well as specific training to meet the demands of the Brazilian CBRN troop and society in recognising threats of this type.

IDQBRN has a highly qualified workforce acting in multidisciplinary lines. Its team comprises specialists in various fields, including physics, engineering (chemistry, mechanics, nuclear and materials), chemistry, pharmacy, veterinary medicine, and microbiology. Technical support and maintenance personnel also assist.

The primary focus of IDQBRN revolves around three critical missions: researching and developing products related to CBRN defence, providing expert scientific advice in the CBRN field, and serving as a reference point for matters related to CBRN. The main activity of IDQBRN remains identification, and all other activities are linked to it, as depicted in Fig. 3. The organisation has 11 specialised laboratories that conduct technical activities, including the LAQ/IDQBRN.

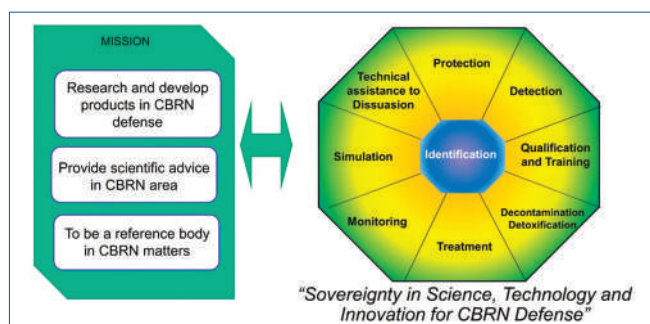


Fig. 3 - IDQBRN Missions and Activities

LAQ work towards the designation

The international network of OPCW-designated laboratories is essential for conducting the off-site analysis. OPCW guarantees these labs are well prepared for this critical task by requiring them to maintain an accredited quality system per the standards ISO/IEC 17025:2017 or equivalent and to perform successfully in its last three consecutive official OPCW proficiency tests [13].

OPCW's proficiency tests are qualitative, *i.e.* based on determining whether relevant chemicals are present or absent in samples of various matrices (such as soil, water, organic waste, wipe, and others) [8]. The relevant chemicals can be any scheduled chemicals, their precursors, and degradation products spiked at 1-10 ppm level, challenging the world's best chemical analysis laboratories in technical skills, analytical methodologies, safety protocols, chain-of-custody procedures, etc. [14].

Each participant is given two sets of three samples, namely a test sample, a control sample, and a blank sample, all belonging to the same matrix. No indications are provided about which is which, and the participants are not limited in their choice of sample preparation or analytical methods. Typically, sample preparation protocols involve several steps, such as clean-up, extraction, derivatisation, and concentration [15]. At least two distinct analytical techniques must be utilised, one being a spectrometric method to confirm identifications. Additionally, before introducing the sample, laboratories prepare a solvent blank to verify the absence of any reported chemicals. Moreover, participants must carry out all work promptly since they only have 15 calendar days from receiving the samples to perform the analysis and submit their report [16]. The report should include names, structures, and available CAS numbers of any identified chemicals, a description of the sample preparation methods used, a description of the analytical techniques employed, and the analytical data obtained, which should include both chromatographic and spectrometric data along with the corresponding analytical conditions [15]. Throughout the entire document, it is crucial to maintain an impeccable chain of evidence, connecting each test sample to every reported chemical in the final result delivered [16]. The laboratories must achieve either A, A, A or A, A, B grades in their most recent three tests [17]. A status of A is given to laboratories that correctly identify



all spiking chemicals in the test without reporting errors. At the same time, a grade of B indicates either a missed spiking chemical or a reporting error resulting in a non-scoring chemical. Any laboratory receiving more than one B grade or a lower rating is suspended and ineligible to receive authentic samples for analysis [8].

The high degree of quality and technical capacity of the laboratories are demonstrated by the rigorous performance requirements, the infinite number of possible reportable chemicals and the fact that certain spiking chemicals used in proficiency tests may not be present in any spectral databases [15]. In this case, it is necessary to predict possible structures of probable compounds, obtain analytical patterns of the same through organic synthesis and compare the spectral data obtained to have the corresponding identification.

Since 2010, LAQ has been taking part in OPCW Proficiency Tests (Fig. 4), using gas chromatography (GC)-mass spectrometry (MS) as the principal analytical method in various permissible configurations involving different columns and sources, along with other GC-specific detectors: flame photometric and nitrogen-phosphorus. In recent times, the lab has also incorporated liquid chromatography into its analytical techniques. Furthermore, to fulfil the quality assurance requisite, since November 2015, LAQ/IDQBRN has been accredited, for analysis of CWC-related chemicals, by the General Coordination for Ac-

Participation Number	Year	Proficiency Test	Compounds Present	Identified	Accepted	OPCW Grade
Trial	2010	28	7	5	0	Trial
Trial	2011	29	8	6	1	Trial
Trial	2011	30	8	7	0	Trial
1	2012	31	7	7	0	F
2	2012	32	8	4	1	F
3	2013	33	8	6	2	D
4	2013	34	7	4	3	C
5	2014	35	9	7	6	C
6	2014	36	7	6	6	B
7	2015	37	7	4	4	C
8	2015	38	6	6	3	C
-	2016	39	8	-	-	-
9	2016	40	7	7	6	B
10	2017	41	7	7	6	B
11	2017	42	7	4	4	C
12	2018	43	6	5	4	C
-	2018	44	8	-	-	-
13	2019	45	7	7	7	A
-	2019	46	9	-	-	-
-	2020	47	8	-	-	-
14	2020	48	6	6	6	A
15	2021	49	6	5	5	B
-	2021	50	6	-	-	-
-	2022	51	7	-	-	-
17	2022	52	7	7	7	A

Fig. 4 - LAQ results in OPCW Proficiency tests

creditation of Inmetro (Cgcre), the only accreditation body recognised by the Brazilian Government to accredit conformity to the requirements of the standard ISO/IEC 17025.

Currently, the laboratory staff functions in the following manner: a team dedicated to preparing each sample set, another responsible for performing chromatographic tests, a team analysing the experimental results, and a team involved in preparing the report. All team members participate in reviewing the final document. Organic synthesis provides additional support, enabling the definite identification of chemical products that may not be present in existing databases.

In the past, our lab faced challenges not achieving good results on OPCW proficiency tests due to a high number of non-conformities, most related to the reporting process. However, through diligent efforts and proactive measures, we have successfully addressed these non-conformities and implemented corrective actions to prevent their recurrence. These improvements have enhanced our quality management system, ensuring the reliability and accuracy of results. As a result, we have experienced a positive trend with improved grades and finally achieved designation status in 2021.

Recognition of the long journey LAQ/IDQBRN has travelled to OPCW designation underscores the importance of continued efforts to improve quality. This includes regular reviews of the quality management system, internal audits, training programs, and other measures to comply with ISO/IEC 17025 standards. By maintaining a proactive approach and continuously monitoring and addressing any non-conformities, LAQ/IDQBRN seeks to maintain the quality and accuracy of its present and future results, in addition to continuous improvement in all activities performed (Fig. 5).



Fig. 5 - Chemical defence activities carried out at IDQBRN

Conclusion

Establishing a comprehensive response system for a vast country like Brazil is increasingly challenging due to emergencies, scientific advancements, and external actors not affiliated with the state. Therefore, implementing, integrating, and optimising national response systems, such as the Brazilian Army Sis-DQBRNEx, is crucial. Moreover, the chemical industry's significance in Brazil means that the country has substantial responsibilities.

Although Brazil has taken notable actions to comply with the provisions of the CWC, including annual declarations, transfer controls, and protective measures, maintaining an OPCW-designated laboratory is crucial for a country's national security and international standing. It demonstrates a commitment to safety and security measures against dangerous chemical agents and provides access to the expertise and resources necessary to respond effectively to any potential threat, guaranteeing the Brazilian nation a deterrent power against chemical weapons. Therefore, to maintain its unique position in terms of assistance and protection capacity within the GRULAC region and aligned with its regional role of Brazil in the OPCW, it is sensible to have an OPCW-designated laboratory as a contribution to CWC verification activities. Therefore, the role of LAQ/IDQBRN as the only designated laboratory in the GRULAC region is critical in deterring and preventing the use of chemical weapons, ultimately reducing the global risk of such threats.

REFERENCES

- [1] OPCW Technical Secretariat, Note by the Director-General: Status of Laboratories Designated for Authentic Environmental Sample Analysis, Apr 2023.
- [2] Organisation for the Prohibition of Chemical Weapons, <https://www.opcw.org/>
- [3] R.M.C. Dunlop, Statement by the Deputy Permanent Representative of Brazil to the United Nations at the OPCW High Level Meeting "Fifteen Years of the Chemical Weapons Convention: Celebrating Success. Committing to the Future", 2012.
- [4] A. Üzümcü, Statement by the Director-General of the OPCW to the Brazilian Chemical Industry Association (ABIQUIM), 2014.
- [5] Chemical Weapons Convention, 1997, <https://www.opcw.org/chemical-weapons-convention/download-convention>
- [6] "Diretor da OPCW visita o Brasil", Intertox, 2014, <https://intertox.com.br/diretor-da-opcw-visita-o-brasil/>
- [7] P.A.M. Cabral, C.E.G. Ilha *et al.*, *Revista Virtual de Química*, 2014, **6**.
- [8] M.-M. Blum, R.V.S.M. Mamidanna, *Anal. Bioanal. Chem.*, 2014, **406**, 5067.
- [9] Brasil, Ministério da Defesa, Estratégia Nacional de Defesa, 2016.
- [10] I. de M. Berard, A contribuição do SISDQBRNEX do Exército Brasileiro para a manutenção dos acordos internacionais nas áreas química, biológica, radiológica e nuclear, ESAO, 2020.
- [11] L.R. dos Santos Junior, Observatório Militar da Praia Vermelha, 2022.
- [12] P.A. de M. Cabral, S.F. de A. Cavalcante, *CBRNe World*, 2022, Aug, 470.
- [13] E.W.J. Hooijschuur, A.G. Hulst *et al.*, *Trends in Analytical Chemistry*, 2002, **21**, 15.
- [14] The Case for Modern Forensic Science, *Science & Technology Review*, 2018, Jul/Aug, 4.
- [15] V. Dubey, S. Velikeloth *et al.*, *Accred. Qual. Assur.*, 2009, **14**, 431.
- [16] OPCW, Quality System Document of the OPCW: Work Instruction for the Reporting of the Results of OPCW Proficiency Tests QDOC/LAB/ WI/PT04, 2022.
- [17] OPCW, Guidelines on the Designation of Laboratories for the Analysis of Authentic Samples EC-XX/DEC.3, 2000.

Accreditamento OPCW: un impegno brasiliano

Creato nel 2015, il CBRN Defense Institute (IDQ-BRN) dell'esercito brasiliano è il primo laboratorio accreditato dall'OPCW nel GRULAC (Gruppo America Latina e Caraibi) per analizzare campioni ambientali. Nell'articolo vengono ripercorsi il processo e la storia delle attività di difesa chimica del Brasile per aiutare le altre organizzazioni aspiranti a questo ruolo. Questo risultato sottolinea il ruolo della scienza e della tecnologia nell'adempimento degli obblighi CWC e nella promozione di un mondo libero dalle armi chimiche.