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Data Article

LC-MS analysis data of intermediate products used to construct photodegradation pathways for bis(1*H*-tetrazol-5-yl)amine (H_2BTA)

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A R T I C L E I N F O

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A B S T R A C T

The photolysis of bis(1*H*-tetrazol-5-yl)amine (H_2BTA) in water was carried out in SolSim and Rayonet photochemical reactors equipped with solar simulating and ultraviolet lamps, respectively. The intermediary degradation products were monitored and tentatively identified by liquid chromatography – mass spectrometry (LC–MS). A quadrupole time-of-flight mass spectrometer (QTOF) was used to measure the mass-to-charge ratio (*m/z*) of the deprotonated molecular ions ($[M - H]^-$) using electrospray ionization in negative mode (ESI-), thus making it possible to determine the number of C, H, N and O in the molecules. Four major degradation products, namely *N*-(1*H*-tetrazol-5-yl)formamide (T(5yl)FA), 1*H*-tetrazol-5-ylcarbamic acid (T(5yl)CA), *N*-(1*H*-tetrazol-5-yl)carbamohydrazonic acid (T(5yl)CHA) and 1*H*-tetrazol-5-amine (5-AT), have been identified after solar simulated and UV irradiation. This dataset is supplementary to the research paper "Photodegradation of bis(1*H*-tetrazol-5-yl)amine (H_2BTA), a high nitrogen content tetrazole-based energetic compound in water" [1].

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Specifications Table

Subject	Environmental Chemistry
Specific subject area	Defense Industry; nitrogen-rich (N% = 82.34%) bis(1*H*-tetrazol-5-yl) amine is considered a very suitable composite propellant.
Type of data	Tables and Figures
How data were acquired	Data was acquired using liquid chromatography-mass spectrometry (LC-MS) through an Agilent 1200 high-performance liquid chromatography (Agilent Technologies, Santa Clara, CA) coupled to a MicrOTOF-Q mass spectrometer (Bruker Daltonics, Bremen, Germany).
Data format	Raw and Analyzed data
Data source location	National Research Council Canada, 6100 Royalmount Ave, Montreal, Quebec, H4P 2R2, Canada
Data accessibility	Data are within this article
Related research article	A. Halasz, J. Hawari, N.N. Perreault Photodegradation of bis(1*H*-tetrazol-5-yl)amine (H_2BTA), a high nitrogen content tetrazole-based energetic compound in water Chemosphere https://doi.org/10.1016/1.chemosphere.2019.125008

Value of the Data

- The MS data were used to identify degradation products needed to construct the H_2BTA photodegradation pathways.
- The data can be used as a reference for LC-MS analysis of tetrazole-containing chemicals, which have wide industrial applications, particularly in the pharmaceutical, automotive, and, more recently, defense industries.
- The data present in this article provide methods of photolysis and mass spectrometry useful to determine the fate of chemicals in the environment.

1. Data description

The dataset of this article contains information on the degradation products of a tetrazole derivative produced under solar-simulated conditions and 254 nm UV irradiation [1]. Mass spectra of the initial (H_2BTA) and 4 intermediate photodegradation compounds (T(5yl)CHA, T(5yl)CA, T(5yl)FA and 5-AT) showing $[M - H]^-$ and characteristic fragments are presented in Figs. 1–5. Data of H_2BTA and intermediate compounds, including the average of 5 measured *m/z* $[M - H]^-$ values, calculated *m/z* values, average of mass measurement error (ppm), mono-isotopic molecular mass, elemental formula and IUPAC name are presented in Table 1. The average of the data and the error have been processed according to the recommendations of Brenton and Godfrey [2] (Supplementary Tables 1.1 and 1.2). The most probable elemental formula of the intermediates was determined by applying the chemical knowledge of the parent compound, the fragmentation pattern (Supplementary Table 2) and the

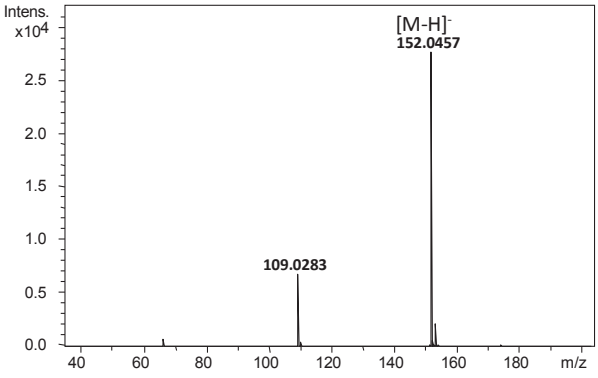

Fig. 1. Mass spectrum of bis(1*H*-tetrazol-5-yl)amine (H_2BTA).

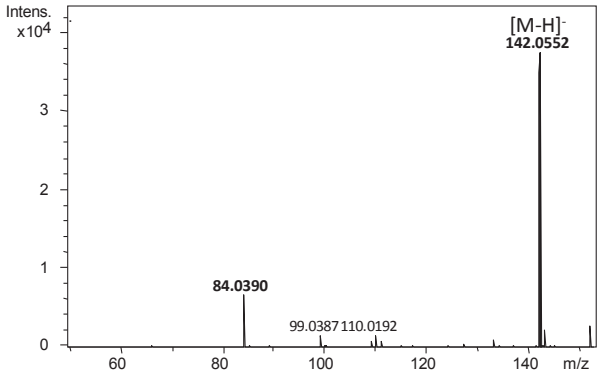

Fig. 2. Mass spectrum of (*Z*)-*N*-(1*H*-tetrazol-5-yl)carbamohydrazonic acid (T(5yl)CHA).

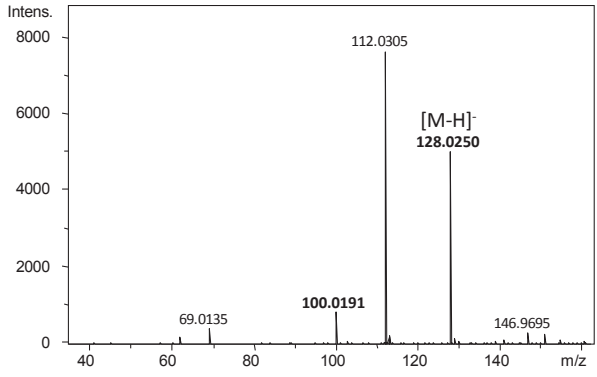

Fig. 3. Mass spectrum of 1*H*-tetrazol-5-ylcarbamic acid (T(5yl)CA).

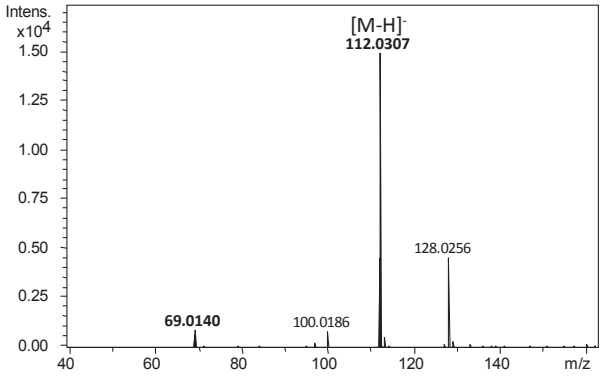

Fig. 4. Mass spectrum of *N*-(1*H*-tetrazol-5-yl)formamide (T(5yl)FA).

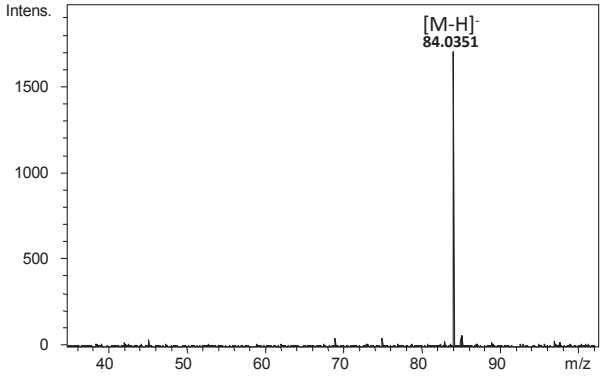

Fig. 5. Mass spectrum of 1*H*-tetrazol-5-amine (5-AT).

Table 1
The average of 5 measured *m/z* $[M-H]^-$ values, calculated *m/z* values, average of mass measurement error (ppm), mono-isotopic molecular mass, elemental formulae and IUPAC names of the initial compound (H_2BTA) and intermediates.

ID	Measured accurate *m/z*	Calculated exact *m/z*	Error (ppm)	Mono-isotopic molecular mass	Elemental formula	IUPAC name
H_2BTA	152.0447	152.0494	12.2	153.0506	$C_2H_3N_9$	Di(1*H*-tetrazol-5-yl)amine
T(5yl)CHA	142.0547	142.0472	53.1	143.0550	$C_2H_5N_7O$	(*Z*)-*N*-(1*H*-tetrazol-5-yl)carbamohydrazonic acid
T(5yl)CA	128.0249	128.0203	36.1	129.0281	$C_2H_3N_5O_2$	1*H*-tetrazol-5-ylcarbamic acid
T(5yl)FA	112.0286	112.0253	29.8	113.0332	$C_2H_3N_5O$	*N*-(1*H*-tetrazol-5-yl)formamide
5-AT	84.0337	84.0327	37.6	85.0383	CH_3N_5	1*H*-tetrazol-5-amine

nitrogen rule [3]. 5-AT was identified by comparison with commercially available reference standard. IUPAC names were generated in ChemDraw Ultra 10.0.

2. Experimental design, materials, and methods

2.1. Chemicals

N,*N*-bis(1*H*-tetrazol-5-yl)amine monohydrate, $H_2BTA{\cdot}H_2O$, was synthesized by Defense Research and Development Canada (DRDC)-Valcartier. 5-Aminotetrazole monohydrate was purchased from Sigma-Aldrich (Canada). Working solutions of H_2BTA (0.35 mM or 0.18 mM) were prepared in deionized water.

2.2. Photolysis experiments

Irradiation experiments were conducted using artificial sunlight generated from a SolSim solar simulating photoreactor (Luzchem Research Inc., Canada). The total power of the solar simulator output spectrum was calibrated to the best approximation of ASTM Air Mass 1.5 Global Tilt Standard in the 280–800 nm regions: total irradiance of 590,000 mW m^{-2}. To accelerate the degradation process, a Rayonet photoreactor (Branford, CT) equipped with 16 ultraviolet lamps (wavelength 254 nm) was also used. In a typical experiment, 5 mL of aqueous solutions of H_2BTA in 20 mL quartz tubes were irradiated in either SolSim or Rayonet photoreactors for 8 days and 65 min, respectively, at room temperature. Samples in SolSim or Rayonet were taken daily or minutes apart, and analyzed without pretreatment

Table 2
QTOF acquision parameter.

Parameter	Value
Source Type	ESI
Ion polarity	Negative
Capillary	4000 V
End Plate Offset	−500 V
Collision Cell RF	50.0 Vpp
Nebulizer	1.6 Bar
Dry Heater	180 °C
Dry Gas flow	8 l/min

by LC-MS/QTOF. Dark controls prepared using H_2BTA solutions protected with aluminum foil were found stable under otherwise similar conditions. The experiments were carried out in duplicates or triplicates.

2.3. LC-MS QTOF analysis

The degradation products were analyzed by liquid chromatography–mass spectrometry (LC-MS) using a MicrOTOF-Q mass analyzer (Bruker Daltonics, Bremen, Germany) attached to an HPLC system (Agilent 1200 Series) equipped with a DAD detector. Aliquots (10 μL) were injected into a 3 micron-pore size Luna Phenyl-Hexyl column (2 μm ID × 150 mm; Phenomenex, USA) at 25 °C. The solvent system was composed of a mixture of MeOH and 0.05% v/v HCOOH in water (10%:90%) at a flow rate of 0.15 mL min^{-1}. For mass analysis, negative electrospray ionization mode (ESI-) was used producing mainly deprotonated molecular mass ions $[M - H]^-$. The mass range was selected from 40 to 1000 *m/z*. The acquision parameters are presented in Table 2. Data analysis was done using Compass DataAnalysis software (Bruker Daltonics, Bremen, Germany).

Acknowledgments

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Conflict of Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.104936.

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