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Inorganic chemistry laboratory experiment on an energetic nickel (II) coordination compound, aimed at third-year undergraduate students

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Abstract: This inorganic chemistry laboratory experiment, aimed at third-year undergraduate students, focuses on an energetic coordination compound, tris(hydrazine)nickel(II) nitrate $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$. As long as the solid is not subject to mechanical stimuli (impact and friction), it can be safely synthesized and handled. In our laboratory conditions (see supplementary information), the experiment includes synthesis of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ in two different morphologies, visual inspection of the samples, analysis by IR spectroscopy, checking the crystallinity by powder X-ray diffraction, 3D visualization of the crystal structure by means of a free program, and observation by scanning electron microscopy. In that respect, the experiment, as a whole, introduces students to nickel(II) complexes, coordination compounds, energetic materials, sonochemical and morphology-controlled syntheses, and a 3D visualization program for crystal structures and structural models. This laboratory experiment is also proposed in the form of alternate (light) versions so that it can be adopted in another inorganic chemistry laboratory that may have restrictions in terms of equipment, and in another third-year curriculum.

Keywords: coordination compounds; energetic materials; inorganic chemistry; nickel(II) complexes; sonochemical synthesis.

Introduction

Hexaaquanickel(II) $[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$ is a typical octahedral complex where the Ni^{2+} metal center binds to six H_2O ligands. These ligands can be substituted by ammonia NH_3 , a stronger Lewis base, to form hexaamminenickel(II) $[\text{Ni}(\text{NH}_3)_6]^{2+}$. With the ligand ethylenediamine $\text{C}_2\text{H}_4(\text{NH}_2)_2$, tris(ethylenediamine)nickel(II) $[\text{Ni}((\text{NH}_2)_2\text{C}_2\text{H}_4)_3]^{2+}$ forms. These complexes differ in color (green, blue and purple respectively) due to differences in energy between the degenerate $t_{2g}-e_g$ orbitals which is in line with the differences in the Lewis basicity of the ligands (Roberts & Field, 1950; Shakhshiri, Dirreen, & Juergens, 1980; Summerlin, Borgford, & Ealy, 1987). Hydrazine N_2H_4 is another ligand of Ni^{2+} , leading to tris(hydrazine)nickel(II) $[\text{Ni}(\text{N}_2\text{H}_4)_3]^{2+}$. The first coordination compounds of it were discovered over a century ago: the reports on tris(hydrazine)nickel(II) sulfate $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{SO}_4]$ and tris(hydrazine)nickel(II) nitrate $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ were released in 1894 and 1911 respectively (Curtius & Schrader, 1894; Franzen & von Mayer, 1911). It is interesting to note that the latter coordination compound combines an “oxidizer”, NO_3^- , and a “fuel”, N_2H_4 , and this makes it a potential energetic material. In the recent years, $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ was presented as a safer alternative to lead azide $\text{Pb}(\text{N}_3)_2$, a widely used primary explosive (Wojewódka & Bełzowski, 2011; Wojewódka, Bełzowski, Wilk, & Stás, 2009). Indeed, $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ is less sensitive to mechanical stimuli like impact and friction (Hariharanath,

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Chandrabhanu, Rajendran, Ravindran, & Kartha, 2006). Furthermore, it is thermally stable below 120 °C (Chhabra, Talawar, Makashir, Asthana, & Singh, 2003; Zhang, Liu, Song, Liu, & Yang, 2017). No special precaution is required during its synthesis and handling as long as it is not impacted nor is it subject to friction (Patil, Nesamani, & Pai Verneker, 1982; Zhu, Wu, Zhang, & Mu, 1997).

Taking into account all of the safety elements mentioned above, we initiated and developed an inorganic chemistry laboratory experiment focusing on $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$. It is aimed at third-year undergraduate chemistry students who, in accordance with our curriculum, chose the option “science of matter” where inorganic and material chemistries predominate. The experiment is done within a teaching unit entitled “chemistry tutored projects” which main objective is that students work in partial autonomy and on the basis of a research article they found through a rigorous bibliographic study preceding the experiment. The teaching unit consists of 10 sessions of 4 h each, but the laboratory experiment, by itself, consists of six laboratory sessions of 4 h each. Details (about the teaching unit, the inception of the experiment, its development and use by students) are given in SM.

The laboratory experiment includes synthesis of two $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ samples with different morphologies, visual inspection of the samples, analysis by IR spectroscopy, checking on crystallinity by powder X-ray diffraction, 3D visualization of the crystal structure by means of a free computer program, and observation of the particles shape by scanning electron microscopy. Students are accordingly introduced to coordination compounds, energetic materials, sonochemical and morphology-controlled syntheses, preparation of samples for analysis, the aforementioned characterization techniques, and a free 3D visualization program for crystal structures and structural models. With such a laboratory experiment, we also aim at giving students an overview of what is academic research. Students are trained to search the relevant literature, prepare the experiments, analyze and exploit the collected results on the basis of their knowledge and on the open literature, document the work done in relevant way, and present it in a structured and comprehensive way (as a poster).

The laboratory experiment has been designed in line with the equipment available in our chemistry laboratory. In other laboratories, restrictions in terms of equipment and/or differences in the curriculum framework are likely, and that is why, we also propose three alternate versions of the experiment so that it can be more widely adopted. Accordingly, instructor notes and detailed student’s instructions are provided in SM (on pages S18–S14).

Safety and hazards

Impacting and frictioning $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ are to be absolutely avoided. The solid can be easily recovered with a spatula, and this must be done gently. Some solid is likely to be left attached to the vessel walls: do not try to recover it and dispose of it as mentioned below. For the recovered solid, grinding must be avoided. Attenuated total reflectance-Fourier transform IR spectroscopy should be used, to avoid the grinding and pressing steps necessary to prepare a pellet.

Hazardous chemicals are used. Nickel(II) nitrate hexahydrate and hydrazine hydrate combine several health and environmental hazards. The former presents also physical hazards (i.e. explosive properties). All common safety precautions must be taken: wear a labcoat and safety glasses; use protective gloves; and, work under a fume hood. For precaution, it is preferable to target the synthesis of <1 g of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ (Cartwright, 2018).

A way to dispose of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ is to make it react with sodium hydroxide in aqueous solution (pH > 13) under a fume hood (Zhu et al., 1997). The ‘neutralization’ can be accelerated by heating the slurry up to 60–80 °C (Li, Li, Wang, Li, & Qian, 1999); $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ is stable in hot water (pH ~ 7) for at least one week (Zhu et al., 1997).

Experimental procedure

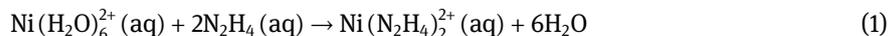
The sonochemical synthesis of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ follows a procedure described in the literature (Wang et al., 2009). Briefly, 0.85 g (2.9 mmol) of nickel(II) nitrate hexahydrate $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (Sigma-Aldrich; $\geq 98.5\%$) is transferred in a beaker and dissolved, under stirring, with 7.8 mL of an aqueous solution of 1 mM (7.8 μmol) polyvinylpyrrolidone ($\text{C}_6\text{H}_9\text{NO}$)_n (denoted PVP) having an average molecular weight of 40000 g/mol (Sigma-Aldrich). Then, 1.05 mL (13.4 mmol N_2H_4) of hydrazine hydrate $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ (Acros Organics; 64%) is added drop by drop, still under stirring. The solution is ultrasonicated for 15 min. The coordination compound precipitates and is separated by centrifugation (6000 rpm, 10 min). It is washed twice with deionized water and once with absolute ethanol, before being dried at 60 °C overnight. The as-obtained sample is denoted **1**. With this procedure, particles with hexagonal morphology are expected to be produced. In parallel, another sample is prepared in a similar way, except for the amount of $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$ that is divided by two (0.525 mL, 6.7 mM). The as-obtained sample is denoted **2**, and particles with sand-rose-like morphology are expected to be obtained.

The molecular structure of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ is verified by attenuated total reflectance-Fourier transform IR spectroscopy (32 scans between 3500 and 500 cm^{-1}). The crystallinity is checked by powder X-ray diffraction (PXRD). The sample preparation is as follows. The solid is gently sieved to collect a homogenous powder and it is placed on sample holder. It is important to mention that $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ is stable under prolonged exposure to the X-ray (Zhu et al., 1997). The data are collected within the 2θ range 10–50°. Afterwards, the experimental pattern is compared to a database containing reference patterns (in our conditions, X-Pert High Score Plus ICC Database).

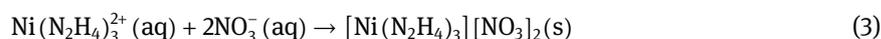
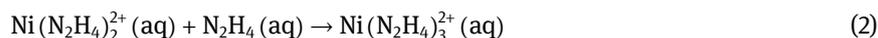
The morphology of the particles of **1** and **2** is verified by scanning electron microscopy (SEM). The samples are prepared as follows. A piece of carbon tape is stuck onto a SEM sample holder, a spatula tip of the sample to be visualized is deposited onto the carbon tape, and the excess of powder is flushed with compressed air.

Results and discussion

Changes of color allow to follow the progress of the synthesis. Aqueous solution of $\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ is green due to $\text{Ni}(\text{H}_2\text{O})_6^{2+}$. It turns blue after addition of few drops of $\text{N}_2\text{H}_4 \cdot \text{H}_2\text{O}$, due to formation of the tetragonal complex $\text{Ni}(\text{N}_2\text{H}_4)_2^{2+}$ (Wang et al., 2009):



Sonication favors formation of the hexagonal complex $[\text{Ni}(\text{N}_2\text{H}_4)_3]^{2+}$ and then precipitation of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$, a purple solid:



$[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ has a very low solubility in water: <4.2 mg in 100 mL, i.e. $<1.5 \times 10^{-5}$ mol/L (Zhu et al., 1997). Sonication allows synthesizing $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ at ambient conditions in a short time period, whereas an alternative approach would be to heat the solution to 65 °C as is the case elsewhere (Cartwright, 2018; Chhabra et al., 2003; Hariharanath et al., 2006; Patil et al., 1982; Wojewódka & Belzowski, 2011; Wojewódka et al., 2009; Zhang et al., 2017; Zhu et al., 1997).

Figure 1 shows the IR spectrum of **1** for which the fingerprint region, albeit dense, is relatively easy to examine. The bands in the 3400 – 3100 cm^{-1} region are due to the N–H stretching vibrations (asymmetric at 3300 cm^{-1} and symmetric at 3228 and 3180 cm^{-1}). The N–H deformation modes are featured by the bands at 1626, 1208, 1185, 647 and 593 cm^{-1} . The band at 977 cm^{-1} is typical of the N–N stretching vibration (Sacconi & Sabatini, 1963). These bands confirm the presence of the ligand N_2H_4 . The signal at 553 cm^{-1} is attributed to Ni–N stretching, which corroborates the coordination of N_2H_4 to Ni(II) (Shawish et al., 2014). The bands of strong intensity between 1450 and 1300 cm^{-1} , as well as the ones with weaker intensity at 1047 and 822 cm^{-1} , are ascribed to the anion NO_3^- (Miller & Wilkins, 1952; Yang & Low, 1973). The spectrum favorably compares with previous results (Tignol & Demirci, 2019; Wang et al., 2009), confirming the successful synthesis of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$.

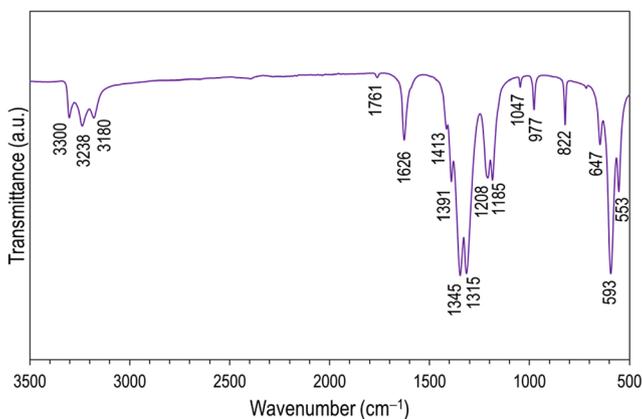


Figure 1: IR spectrum of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$. The adsorption wavenumbers (in cm^{-1}) are given. Except the band at 1761 cm^{-1} (possibly due to water adsorption), they all belong to the title compound as discussed in the text.

Figure 2a shows the PXRD pattern of **1**. It matches the reference pattern n° 96-411-7337 (see SM, Annex 1 on page S15) that belongs to $\text{Ni}_{2.00}\text{N}_{16.00}\text{H}_{24.00}\text{O}_{12.00}$, that is $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$. As the reference pattern is from a previous research article (Bushuyev et al., 2012), the crystallographic information file (CIF) can be freely uploaded (<https://pubs.acs.org/doi/10.1021/ja209640k>), and used to plot the theoretical powder pattern and then visualize the crystal structure. This is an excellent exercise to introduce a 3D visualization program for crystal structures like Mercury from the Cambridge Crystallographic Data Centre (<https://www.ccdc.cam.ac.uk/solutions/csd-system/components/mercury/>). Figure 2b shows the theoretical powder pattern of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$, plotted by means of the CIF and Mercury (see SM, on pages S13–S14). It well compares with the pattern of our $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$, which allows to conclude that its crystal structure is hexagonal with a space group $P\text{-}3\text{c}1$ (n° 165). The Miller indexes of the main peaks are shown. Figure 3 shows a 3D visualization of the crystal structure of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ along the a , b and c crystallographic axes, also obtained by means of the CIF and Mercury. The hexagonal unit cell ($a = b \neq c$, $\alpha = \beta = 90^\circ$, and $\gamma = 120^\circ$) and the octahedral nickel(II) complex (green octahedra) can be clearly seen.

The synthesis protocol of **1** and **2** is based on the use of PVP. The polymer acts as structuring agent. $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ can then present different morphologies provided the experimental conditions are slightly changed (Wang et al., 2009). The SEM micrographs are shown in Figure 4. **1** primarily consists of hexagonal platelets of 1–3 μm . The hexagonal geometry is consistent with the hexagonal crystal structure. This indicates a two-dimensional growth along the a and b crystallographic axes. This is driven by PVP via strong H-bonding between NH_2 of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ and O of the macromolecule. PVP adsorbs onto the lateral surfaces of the growing crystals and limits the growth along the c crystallographic axis. With respect to **2**, the particles show a sand-rose-like morphology with a size of 1–3 μm . The morphology change can be explained as follows. Because of the lower concentration of N_2H_4 , the PVP macromolecules are more prone to adsorption onto the

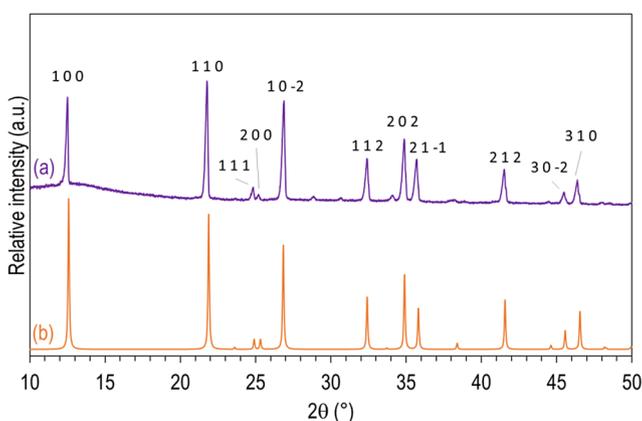


Figure 2: (a) PXRD pattern of the solid $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$, which matches the reference pattern n° 96-411-7337. (b) Theoretical powder pattern of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$, plotted from the CIF available in ref. (Bushuyev et al., 2012). The Miller indexes of the main peaks are given.

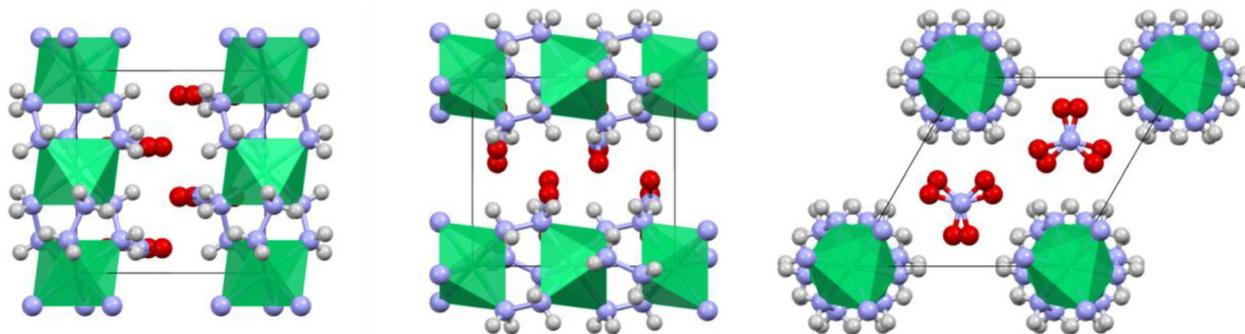


Figure 3: Crystal structure of $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ along the a (left), b (middle) and c (right) crystallographic axes, drawn from the CIF in ref. (Bushuyev et al., 2012) and using the Cambridge Crystallographic Data Centre's Mercury program. Details are given in SM (see on pages S13–S14). The mauve, red and grey spheres show the N, O and H atoms. The Ni atoms are represented by the green octahedra.

side surfaces of the growing crystals and consequently the lateral surfaces are less protected. The crystals can then grow in different orientations, resulting in anisotropic shapes as it is for **2**.

Perspectives of adoption in another laboratory

As mentioned in the introduction and as detailed in SM (see on pages S2–S5), the laboratory experiment, named **V1** (with V as version), has been designed to be realized by two students working in tandem and in six laboratory sessions of 4 h each. In our context, the laboratory sessions are preceded by a rigorous bibliographic study and a preliminary session aiming at introducing the facility and the experiment. Students are expected to work in partial autonomy and have no instructions except the relevant scientific article. **V1** can be adopted in another laboratory. This brings us to propose instructor notes and student's instructions in SM (see on pages S8–S14).

V1 can besides be adapted to be adopted in another laboratory and curriculum. This brings us to propose three alternate versions named **V2**, **V3** and **V4**, **V2** takes into account that access to SEM is restricted to a limited number of laboratories. Instead, an optical microscope can be used provided that magnification up to $1000\times$ is possible (Tignol & Demirci, 2019). The experiment should be realized by two students working in tandem and in six laboratory sessions of 4 h each. **V3** is a lighter version. It takes into account that neither SEM nor optical microscopy. **V3** is structured around synthesis of **1**, analyses by IR and DRX, and introduction to

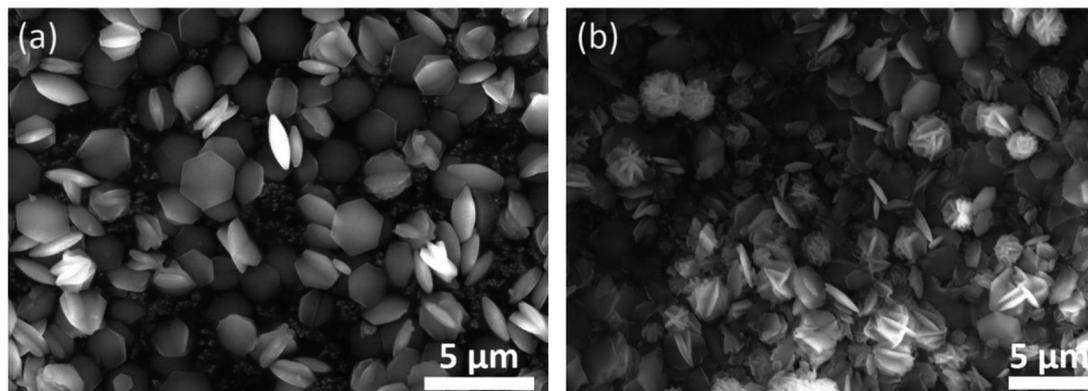


Figure 4: SEM micrographs of the $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$ samples denoted **1** (hexagonal morphology) and **2** (sand-rose-like morphology).

and use of the 3D visualization program. The experiment can be realized by two students working in tandem and in five laboratory sessions of 4 h each. **V4** is a version that is even lighter. It takes into account that neither microscope nor X-ray diffractometer is available. **V4** is structured around synthesis of **1** and analysis by IR. The experiment can be realized by two students working in tandem and in three laboratory sessions of 4 h each. For all of these versions, the experiment should be preceded by a rigorous bibliographic study, which could be done as homework or in class. In any case, this should be done before the laboratory sessions.

Conclusions

In this laboratory experiment, students get to work on tris(hydrazine)nickel(II) nitrate $[\text{Ni}(\text{N}_2\text{H}_4)_3][\text{NO}_3]_2$, an energetic example of nickel(II) coordination compound. The experimental work is preceded by a rigorous bibliographic study that gives the required elements about safety, synthesis and characterization. Students develop the right reflexes and the appropriate behavior for synthesizing and analyzing the energetic material, more broadly a solid chemical, in the safest conditions. The synthetic approach is facile, the collected data are easy to analyze, and thus the experiment can be regarded as simple. Students appreciate this overall ease because they are assured of successfully synthesizing the title compound, and they are able to unambiguously identify the synthesized compound by data analysis. Students are evaluated in two ways. A first mark is given by the instructor to assess the students experimental work as a whole. A second mark is given by a jury of four instructors for an oral examination where students present their main achievements with the help of a poster; the presentation is followed by a session of questions. In the end, students gain a greater experimental independency, a point particularly appreciated.

Supplementary information

Details about the teaching, details about the inception of this experiment, instructor notes, student's instructions, and the reference pattern n° 96-411-7337 are provided in the online supplementary material.

Acknowledgments: UBD thanks Pierre Tignol who did in 2017 the preliminary study that was important for the creation of this laboratory experiment (see SM, on pages S6–S7).

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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