of compound $2 \cdot 3C_7H_8$ were obtained from toluene at 0 °C. Diffraction data were collected on a IPDS II Stoe image-plate diffractometer with graphite-monochromated $Mo_{K\alpha}$ radiation ($\lambda = 0.71073$ Å). The structure was solved by direct methods (SHELX-97)[17] and refined against F^2 on all data by full-matrix least-squares with SHELX-97.[18] The heavy atoms were refined anisotropically. Hydrogen atoms were included by using the riding model with U_{iso} tied to U_{iso} of the parent atoms. CCDC-173318 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk).

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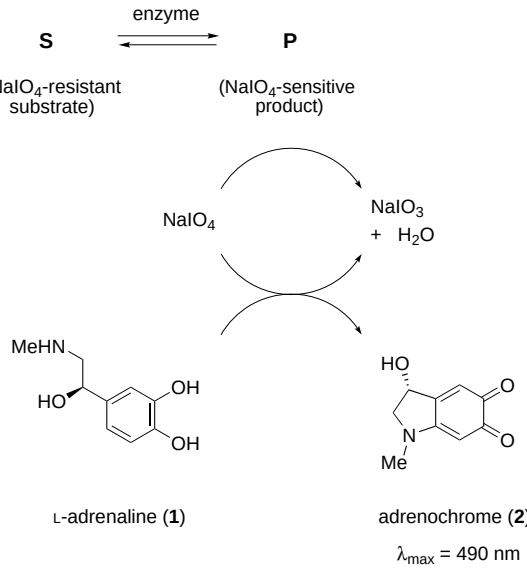
The Adrenaline Test for Enzymes**

Denis Wahler and Jean-Louis Reymond*

Enzyme assays[1] play a key role in the search for novel enzymes,[2] which are in great demand as components of consumer products, industrial processes, diagnostics, and analytical reagents.[3] While many enzyme assays are based on chromogenic or fluorogenic substrates, it is often desirable to have assays that produce a recordable signal indirectly and thus avoid the incorporation of a chromophore into the substrate itself. Such indirect assays are possible with sensors that either record changes in physicochemical parameters such as temperature,[4] pH,[5] or pM[6] upon reaction progress, or that respond by selective recognition of product over substrate through noncovalent interactions.[7, 8] The classical enzyme-coupled enzyme assays achieve the same goal through selective degradation of the reaction product by one or more secondary enzymes, most often by means of a redox chain that leads to the formation of NADH, which is analyzed spectrophotometrically at 340 nm.[9] Enzyme-coupled assays are, however, expensive and incompatible with varying technically important parameters such as cosolvents, pH, and temperature. Herein we report a colorimetric enzyme assay based on the quantification of periodate-sensitive reaction products by back-titration of the oxidant with adrenaline. The assay uses inexpensive, commercially available reagents, and offers a simple solution for assaying a variety of industrially important enzymes with their natural substrates.

We recently reported a series of enzyme substrates that released a colored or fluorescent product by oxidation of the primary enzyme reaction product by alcohol dehydrogenase[10] or sodium periodate.[11] Considering that sodium periodate reacts rapidly with any 1,2-diol or related periodate-sensitive functional groups independent of the presence of a chromophore in the molecule, we reasoned that this oxidant should also be applicable to nonchromogenic and nonfluorogenic substrates if used in a back-titration mode. Thus, any periodate-sensitive reaction product formed from a periodate-resistant substrate should be detectable by adding first a measured amount of sodium periodate, and second a chromogenic or fluorogenic reagent to quantitate the unreacted sodium periodate by optical density (OD) measurements (Scheme 1).



Scheme 1. Principle of the adrenaline test for enzymes.

Adrenaline (**1**) reacts within seconds with sodium periodate to form the red dye adrenochrome (**2**).[12] We have found that this reaction can be used to quantitate sodium periodate independent of the presence of proteins or cosolvents at any pH between 2 and 10 in aqueous buffers.[13] The reaction enabled us to measure various periodate-sensitive functional groups such as 1,2-diols, 1,2-aminoalcohols, 1,2-diamines and α-hydroxyketones by back-titration of sodium periodate (Figure 1).

[*] Prof. Dr. J.-L. Reymond, Dr. D. Wahler
Department of Chemistry & Biochemistry
University of Bern
Freiestrasse 3, 3012 Bern (Switzerland)
Fax: (+41) 31-631-8057
E-mail: jean-louis.reymond@ioc.unibe.ch

[**] We would like to thank Prof. R. Furstoss and Dr. A. Archelas at the Faculté des Sciences de Luminy, Marseille, France, for providing a sample of *Aspergillus niger* EH and Dr. C. Weijers at the Department of Food Technology and Nutrition Sciences, Wageningen University, the Netherlands, for providing a sample of *Rhodotorula glutinis* EH. This work was supported by the University of Bern, the Swiss National Science Foundation, and Protéus SA (Nîmes, France).

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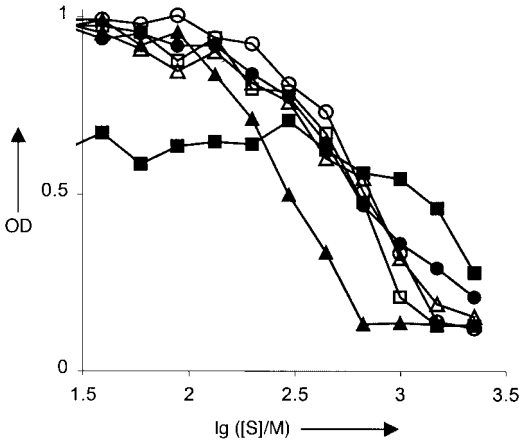
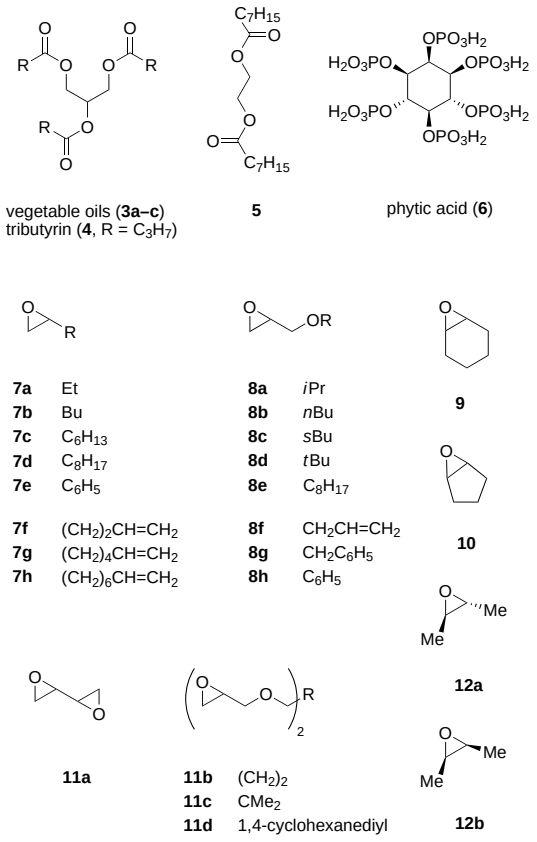


Figure 1. Detection of diols, aminoalcohols, diamines, and hydroxyketones by back-titration of sodium periodate with adrenaline. Observed OD at 490 nm (as given by microtiter-plate reader) is shown as a function of the molar concentration of diol or aminoalcohol (S; substrate) in logarithmic scale, as recorded after oxidation with $NaIO_4$ (1 mM) for 30 min, followed by reaction with **1** (1.5 mM) for 5 min in 20 mM aq phosphate pH 7.2, 26 °C. (●) inositol (EC_{50} = 0.65 mM), (○) benzoine (EC_{50} = 0.71 mM), (▲) glycerol (EC_{50} = 0.31 mM), (□) ethanolamine (EC_{50} = 0.66 mM), (△) ethylene glycol (EC_{50} = 0.76 mM), (■) ethylene diamine in 20 mM aqueous borate at pH 8.8 (EC_{50} = 1.38 mM). EC_{50} is the concentration that inhibits 50 % of the color reaction.

The assay was first applied to measure the activity of lipases and esterases (Figure 3). The back-titration readily detected the interfacial lipolytic activities of the enzymes under biphasic conditions with various vegetable oils (**3a–c**, Scheme 2), which release glycerol upon hydrolysis, as substrates. Interestingly, the pH-indicator method[5] failed to give any signal for these substrates, probably because the products, long-chain fatty acids, remain in the oil phase. The assay was also suitable for detecting esterolytic activities with soluble aliphatic esters such as tributyrin (**4**) and ethylene glycol bisoctanoate (**5**, Figure 2).

We next turned our attention to phytases, a class of acid phosphatases that hydrolyze phytic acid (**6**, Scheme 2) to less phosphorylated *myo*-inositol derivatives. Phytases are used industrially to increase the nutritional value of animal feed.[14] The back-titration with adrenaline could be expected to respond to the hydrolysis of at least two vicinal phosphate groups on phytic acid, which corresponds to the dephosphorylation pattern of most phytases.[14b, 15] The assay was used to record the pH profile of the phytases[16] by taking advantage of the pH-independent formation of adrenochrome (Figure 3). The assay offers a useful alternative to the colorimetric titration of inorganic phosphate for screening phytases with their natural substrate.[17]

The versatility of our assay in terms of substrate structures was demonstrated for epoxide hydrolases, which produce 1,2-diols from epoxides.[18] Twenty-four commercially available aliphatic and aromatic epoxides (**7–12**, Scheme 2) were assayed with epoxide hydrolases from *Aspergillus niger*[19] and *Rhodotorula glutinis*.[20] The reactivity pattern for the substrate series was recorded with both enzymes under their optimal operating conditions (Figure 4). This is the first generally applicable chromogenic assay for epoxide hydrolases.[21]



Scheme 2. Structures of enzyme substrates used in this study.

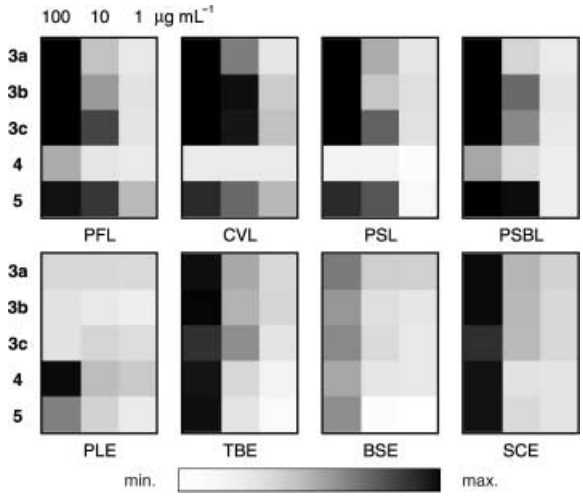


Figure 2. Activity of lipases (upper row of blocks) and esterases (lower row) on vegetable oils (**3a** = olive oil, **3b** = sunflower oil, **3c** = grapeseed oil), tributyrin (**4**), and ethylene glycol bisoctanoate (**5**) as determined by the adrenaline test. For each enzyme (3 × 5 grid), each grayscale square shows the percent reduction of OD_{490} from white (0 %, no activity) to black (100 %, maximum activity) for the indicated substrate (row) at the indicated enzyme concentration (column). Conditions: 1) enzyme in 20 mM aq. phosphate pH 7.2, 1 mM $NaIO_4$, vegetable oils **3a–c** (0.05 mL, 0.4-mL assay in 1.5-mL Eppendorf tube, shaker at 1200 rpm) or soluble substrate **4** and **5** (10 mM), 26 °C, 30 min.; 2) 1.5 mM **1**, 5 min. Key for enzyme samples (Fluka ref.): PFL = *Pseudomonas fluorescens* lipase (F62321); CVL = *Chromobacterium viscosum* lipoprotein lipase (F62333); PSL = *Pseudomonas* sp. lipoprotein lipase (F62335); PSBL = *Pseudomonas* sp. Type B lipoprotein lipase (F62336); PLE = pig liver esterase (F46058); TBE = *Thermoanaerobium brockii* esterase (F46061); BSE = *Bacillus* sp. esterase (F46062); SCE = *Saccharomyces cerevisiae* esterase (F46071).

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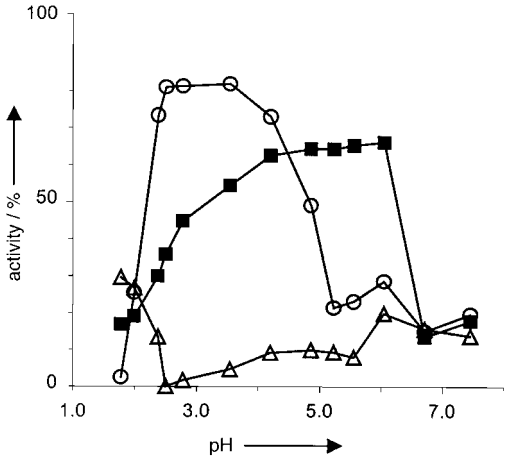

Figure 3. pH Profile of phytases recorded by the adrenaline test. Activity is reported as the percent reduction of OD_{490} relative to reference. Conditions: 1) 10 mM aq potassium phytate (**6**) with 1 mM $CaCl_2$ at the indicated pH, phytase (0.1 mg mL^{-1}), 60 min at 55 °C; 2) 1 mM $NaIO_4$, 30 min, 26 °C; 3) 1.5 mM **1**, 5 min (■) Natuphos® phytase, (△) Novo phytase, (○) *Aspergillus ficuum* phytase. There was no detectable activity in the controls without enzyme.

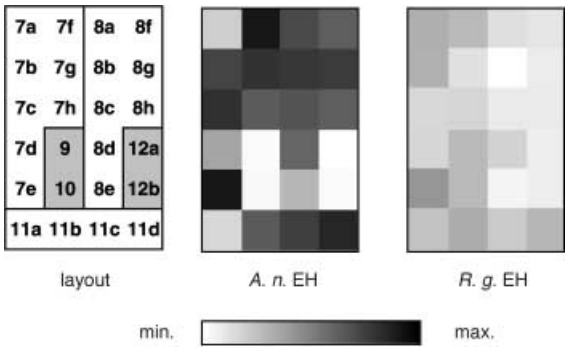

Figure 4. Hydrolytic activities of epoxide hydrolases (EH) towards epoxides **7**–**12** as measured by the adrenaline test. Each grayscale square corresponds to one epoxide according to the layout at left, and shows the percent reduction of OD_{490} from white (0%, no activity) to black (100%, maximum activity). Left: *Aspergillus niger* EH (0.05 mg mL^{-1}, partially purified enzyme preparation), 26 °C. Right: *Rhodotorula glutinis* EH (0.1 mg mL^{-1}, crude enzyme extract), 37 °C. Conditions: 1) 10 mM epoxide in aq. 20 mM phosphate pH 7.2, enzyme, 1 mM $NaIO_4$, 30 min at the indicated temperature; 2) 1.5 mM **1**, 5 min, 26 °C. There was no detectable hydrolysis in control assays without enzyme or with BSA (bovine serum albumin, 2 mg mL^{-1}).

The examples above demonstrate the high-throughput potential and versatility of the adrenaline test for enzymes. The assay quantitates vicinal diols, aminoalcohols, diamines, and α-hydroxyketones (Figure 1), and can be used generally to assay reactions of such functional groups as substrates or as products. As is the case for many catalysis assays such as cat-ELISA (catalytic enzyme-linked immunoabsorbent assay),[8a] and screening methods based on TLC (thin-layer chromatography),[22] MS (mass spectrometry),[23] or capillary electrophoresis CE,[24] the periodate back-titration is only suited for endpoint measurements. Nevertheless, the method is close to ideal since it combines a broad versatility in terms of substrate structure, reaction types, and reaction conditions, with a single, commercially available and inexpensive chromogenic probe that produces a signal directly visible by the eye. This assay is also suited for miniaturization in ultrahigh-throughput screening format.

Experimental Section

Substrates were diluted from 10 mM stock solutions in acetonitrile. Vegetable oils were used pure. Enzymes were diluted from 1 mg mL^{-1} stock solutions in phosphate-buffered saline (160 mM NaCl, 10 mM aq. phosphate, pH 7.4). $NaIO_4$ was added as a freshly prepared 10 mM stock solution in water. Adrenaline (as the HCl salt) was added as a 10 mM stock solution in water. Assays (0.1 mL) were conducted in individual wells of 96-well flat-bottom half-area polystyrene microtiter plates (Costar) as described in the figure legends. The optical density was recorded with a Spectramax 250 Microplate Spectrophotometer (Molecular Devices, $\lambda =$ 490 nm).

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[13] Sodium periodate can also be quantitated with other catechols, including 3-methoxycatechol ($\lambda_{max} = 405$ nm for the quinone) and 2,3-

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dihydroxynaphthalene or 3,4-dihydroxybenzoate (extinction of blue fluorescence at $\lambda_{em} = 440$ nm upon oxidation to the quinone). Noradrenaline reacts similarly to adrenaline down to pH 5. Adrenochrome (**2**) is unstable and polymerizes to brown and finally insoluble black products upon prolonged standing in solution.

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Cubane with a Handle: $[\{In_3As_4Nb\}{-}As]^{7-}$ in $Cs_7NbIn_3As_5$**

Franck Gascoin and Slavi C. Sevov*

Compared to the large number of main group Zintl compounds there are only a few that contain transition metals.[1] This is not surprising since the traditional definition of Zintl phases automatically excludes transition metals. However, many such compounds with transition metals can qualify for Zintl phases when a broader definition is used. The latter includes compounds of transition metals with filled or empty d shells, that is the late transition metals of the Ni, Cu, and Zn groups,[2] and the early transition elements of the Ti, V, and Cr groups at maximum formal oxidation states.[3] There are only two Zintl compounds containing a transition metal with partially filled d shell, both based on manganese, $(AE)_{14}MnPn_{11}$ (AE = alkaline-earth metal, Pn = pnictogen) and $Sr_{21}Mn_4Sb_{18}$.[4] Perhaps only they should be called true "transition metal Zintl phases" although the name is contradictory in itself. All but two of the d^0 compounds contain isolated tetrahedra $[MPn_4]^{n-}$ (M = Nb, Ta, W, Ti).[5] The two exceptions are Na_5HfAs_3 with dimers of edge-sharing tetrahedra of $[Hf_2As_6]^{10-}$ and $Rb_5TaAs_4Tl_2$ with $[TaAs_4]$ tetrahedra, where two Tl atoms bridge opposite edges, $(\mu\text{-Tl})[As_2TaAs_2](\mu\text{-Tl})^{5-}$.[6] Here we report a new d^0 transition metal Zintl phase, $Cs_7NbIn_3As_4$, which contains an unprecedented anion, $[\{In_3As_4Nb\}{-}As]^{7-}$, a cubane made of three indium, four arsenic, and one niobium atom and a "handle" composed of an arsenic atom that is multiply bonded to the niobium corner.

The title compound was initially made in an attempt to synthesize the recently reported $Cs_5In_3As_4$ at temperatures higher than the original 500 °C.[7] The reaction was carried out in niobium containers at 800 °C, at which temperature the arsenic apparently attacked the container and formed the quaternary compound $Cs_7NbIn_3As_4$. Later it was synthesized in high yield using the corresponding elements in stoichiometric ratio at the same temperature.[8]

The overall structure of $Cs_7NbIn_3As_5$ is quite simple and unremarkable,[9] an ionic assembly of isolated anions of $[NbIn_3As_5]^{7-}$ immersed in a "sea" of cesium cations that screen them from each other (inter-anion $d_{min} = 5.009(4)$ Å). What is remarkable is the structure and bonding of the anion (Figure 1). Its geometry can be viewed in a few different ways. The more obvious approach is to recognize the cubane shape made of one Nb, three In, and four As atoms, $[NbIn_3As_4]$, and its "handle" of a fifth arsenic atom attached to the niobium

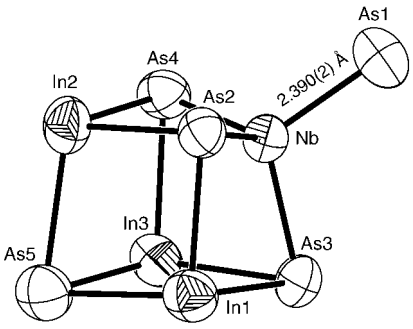

Figure 1. Structure of the cubane with a "handle" $[\{As(InAs)_3\}Nb{=}As]^{7-}$ (ORTEP drawing; thermal ellipsoids at the 95% probability level). The unusually short distance of the handle is shown. The remaining distances [Å] are: Nb-As2 2.484(2), Nb-As3 2.526(2), Nb-As4 2.535(2), In1-As2 2.834(2), In1-As3 2.794(2), In1-As5 2.908(2), In2-As2 2.864(2), In2-As4 2.789(2), In2-As5 2.894(2), In3-As3 2.845(2), In3-As4 2.822(2), In3-As5 2.858(2). The angles [°] at Nb and As5 are in the range 107.26(7)–112.82(7) and 79.32(5)–80.27(5), respectively. The angles around As2, As3, and As4 fall in the range 78.15(6)–82.09(5)°. The angles at the indium atoms that involve As5, 98.34(5)–99.28(6)°, are larger that those that do not, 91.25(5)–92.20(5)°.

[*] Prof. S. C. Sevov, F. Gascoin
Department of Chemistry and Biochemistry
University of Notre Dame
Notre Dame, IN 46556 (USA)
Fax: (+1) 219-631-6652
E-mail: ssevov@nd.edu

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