catalyst amounts (< 200 μg) identified. Reactions have been observed at temperatures up to 350 °C, indicating that the method can be applied over a wide temperature range. The method has also been applied successfully to the detection and screening of enantioselectivity in liquid-phase reactions (see the following communication),[14] stressing its general usefulness.

Clearly, high-throughput screening of larger libraries could now be achieved with IR imaging of catalyst activity. We are presently engaged in the adjustment of a robotic system for automated library preparation, automated quantification of the heat response of the individual samples on the library with the help of software, and improvement of the signal sensitivity of the camera setup. Detection of catalytic activity in combinatorial libraries reduces the time necessary to find new potential catalysts. The development of the most promising candidates into useful new catalyst materials still requires more conventional laboratory techniques.

Experimental Section

For the sol preparations tetraethoxysilane (TEOS) was used as the silica precursor and tetraisopropoxytitanium as titania percursor. The following precursors were used for the other elements: $PdCl_2$, Na_2PtCl_6, $IrCl_4 \cdot x H_2O$, $RuCl_3 \cdot x H_2O$, $CoCl_2 \cdot 6 H_2O$, $[Fe(acac)_3]$, $[Mn(acac)_3]$, $(i\mathrm{PrO})_3VO$, $CrCl_3 \cdot 6 H_2O$, $[Cu(acac)_2]$, $[Ni(acac)_2]$, $ZnCl_2$, $[Pd(acac)_2]$.

Preparation of sols: The silica sols were prepared according to the standard procedure by Klein et al.[12] The compounds were used in the molar ratios for the sol preparation as indicated by the AMM notation.

The titania sols were prepared by dissolving titanium isopropoxide (3.36 mmol, 1 mL) in dry ethanol (3.33 mL) while stirring. After the mixture had been stirred for 30 min, 8 N HCl (8.33 μL) was added. After 5 min, concentrated HCl (46.7 μmol) was slowly added over about 20 min, then ethanol (833 μL), and finally an ethanolic solution (833 μL) of the metal precursor.

Preparation of the catalyst library: The catalyst library was prepared by pipetting μL amounts of titania or silica sols into the wells (diameter 1.5 mm, depth 0.6 mm) of the slate library substrate. In the case of the silica sols 1.5 μL (equivalent to 192 μg silica catalyst) and in the case of the titania sols 5 μL (equivalent to 182 μg of titania catalyst) were deposited. After evaporation of the solvent the library was heated to 65 °C with a heating rate of 1 °C min^{-1} and kept at that temperature for 30 min. Then the temperature was increased to 250 °C (heating rate of 1 °C min^{-1}) where it was kept for 180 min. The library was then allowed to cool down to room temperature at a rate of about 1 °C min^{-1}.

Correction and calibration procedure of the IR-camera system: The detector response was corrected prior to the catalytic reaction by a two-point correction taking images of the library at temperatures 5 °C above and below the desired reaction temperature (hydrogenation: 100 °C, oxidation: 350 °C). Before the organic reactant was evaporated into the feed flow of hydrogen or synthetic air, an IR image of the library was taken and subtracted as background. To assign a temperature scale to the IR images six calibration measurements were performed for each experiment taking images of the library at six different temperatures. By fitting a quadratic polynome to the pixels of the calibration images temperature values were assigned to the different colors of all corrected images.

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Time-Resolved IR-Thermographic Detection and Screening of Enantioselectivity in Catalytic Reactions

Manfred T. Reetz,* Michael H. Becker, Klaus M. Kühling, and Arnold Holzwarth

Dedicated to Professor Gernot Boche on the occasion of his 60th birthday

The development of chiral homogeneous metal catalysts and biocatalysts for the enantioselective synthesis of optically active compounds is of considerable academic and industrial interest.[1] Traditionally such catalysts have been prepared and studied one by one, which is extremely time-consuming. Following the advent of combinatorial methods in pharmaceutical research,[2] a few attempts have been made to apply certain aspects of combinatorial chemistry to asymmetric catalysis.[3] So far success has been limited, one reason being the lack of efficient methods for rapid screening of enantioselective reactions. Recently we showed how in-vitro evolu-

[*] Prof. M. T. Reetz, Dipl.-Chem. M. H. Becker, Dipl.-Chem. K. M. Kühling, Dr. A. Holzwarth
Max-Planck-Institut für Kohlenforschung
Kaiser-Wilhelm-Platz 1, D-45470 Mülheim an der Ruhr (Germany)
Fax: (+49) 208-306-2985
E-mail: reetz@mpi-muelheim.mpg.de

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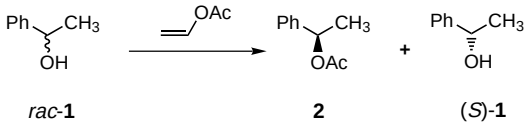
tion can be used to create libraries of mutant lipases for the catalytic enantioselective hydrolysis of chiral esters; the underlying principle is based on the combination of proper methods of random mutagenesis, gene expression, and screening.[4] Accordingly, a rapid photometer test for enantioselectivity was developed, which however is restricted to chiral esters and cannot be adapted to include such substrates as chiral alcohols, diols, amines, amino alcohols, or epoxides.[4, 5] Herein we demonstrate for the first time the use of an infrared camera[6] as a tool in the thermographic screening of enantioselective reactions mediated by biocatalysts or chiral transition metal catalysts.

In all experiments the same instrument[7] and method were employed as described in the preceding publication concerning the detection of the heterogeneous catalysts in gas-phase reactions.[8] In our system we used a modified microtiter plate for carrying out the reactions.[9] As a model reaction we first chose the enantioselective acylation of 1-phenylethanol (**1**) with vinyl acetate, which results in the formation of ester **2**. This transformation is known to occur with 99% enantioselectivity in favor of the (*R*)-ester **2** in the presence of catalytic amounts of immobilized lipase from *Candida antarctica* (Novo SP 435; Scheme 1).[10]



rac-**1** **2** (*S*)-**1**

Scheme 1. Lipase-catalyzed enantioselective synthesis of **2** from *rac*-**1**.

In the present system we prepared separate solutions of vinyl acetate with (*R*)-**1**, (*S*)-**1**, and *rac*-**1** in the wells of a modified microtiter plate.[11] Prior to the initiation of the reaction by addition of the enzyme, the temperature was calibrated in the range of 25–35 °C. Figure 1a shows the respective arrangement of the reaction vessels containing the different solutions at various concentrations at 30 °C. Then the immobilized enzyme (5 mg) was added and the plate shaken (5 s). Temperature changes in the reaction wells were monitored periodically, shaking being interrupted for the measurements. Detection of the reactions was performed for 5 s, resulting in 250 recordings which were averaged and reproduced visually in Figures 1a–c. Although the samples in Figure 1a do not yet contain the biocatalyst, several of them show semicircle-like thermographic emissions of weak intensities. These effects originate from the top part of the walls of the glass vessels and are typical of the setup. The exact position of each vessel changes slightly upon shaking. It is the IR emission stemming from the center of the reaction vessel which is of actual interest and which is not influenced by the effect described above. Figure 1b clearly shows that (*R*)-**1** reacts preferentially with respect to (*S*)-**1**, as seen by the appearance of red spots corresponding to a significant rise in temperature. Under the same conditions the well containing (*S*)-**1** remains cool, whereas *rac*-**1** leads to a moderate rise in temperature. The major part of the reaction is over after 3.5 minutes (Figure 1c). The lowest horizontal row constitutes the control experiment in the absence of enzyme.

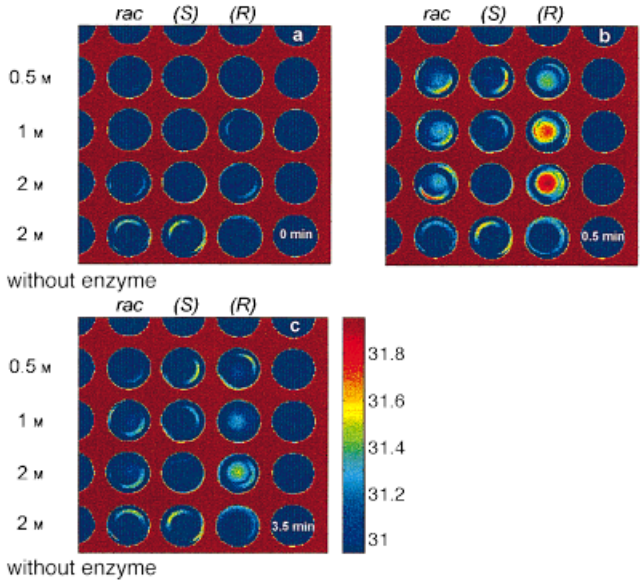
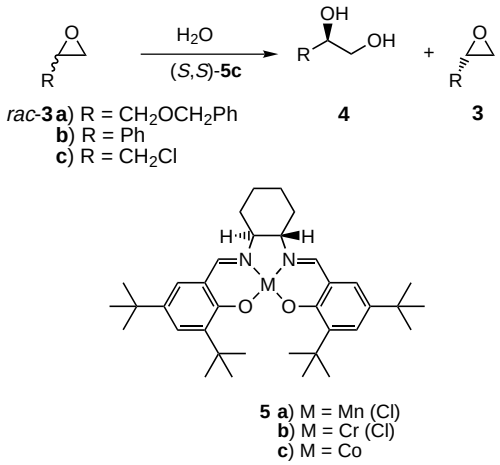


Figure 1. Time-resolved IR-thermographic imaging of the lipase-catalyzed enantioselective acylation of **1** after a) 0, b) 0.5, and c) 3.5 min. The control experiment without enzyme is given in the bottom row in each case. The bar on the far right is the temperature/color key of the temperature window used [°C].

We then turned to enantioselective transition metal catalysis and chose the ring-opening hydrolysis of epoxides **3** with formation of diols **4** as a model reaction under homogeneous conditions (Scheme 2). Jacobsen et al. have described manganese-, chromium-, and cobalt-containing salen catalysts



Scheme 2. Transition metal catalyzed enantioselective synthesis of **4.** See text for more details on the substrates **3** and the catalysts **5**.

5a–c (salen = *N*,*N*′-bis(salicylidene)ethylenediamine dianion), respectively, which induce the kinetic resolution of racemic epoxides by enantioselective ring-opening reactions.[12] In the case of water as the nucleophile, the cobalt catalyst **5c** was reported to be the most active and selective.[13] Accordingly, the (*S*,*S*)-configurated catalyst **5c** reacts completely selectively with (*R*)-**3b** or (*S*)-**3c** in respective racemic mixtures (note change in descriptors due to the CIP-convention), whereas the (*R*,*R*)-catalyst induces the opposite stereoselectivity.

In order to demonstrate the screening capability of the IR camera under these homogeneous conditions, the activity and

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selectivity of the three metal catalysts (*S*,*S*)-**5a**–**c** were tested thermographically in the hydrolysis of epichlorohydrin **3c**.[14] The corresponding time-resolved temperature changes are recorded in Figure 2. The cobalt complex (*S*,*S*)-**5c** is found to be the most active catalyst; it reacts selectively with the epoxide (*S*)-**3c**. The Cr catalyst (*S*,*S*)-**5b** is also selective, although less active, whereas the Mn complex (*S*,*S*)-**5a** displays no significant activity. This is in line with results obtained by Jacobsen et al. using benzoic acid as the nucleophile.[12] It can also be seen that in the case of the Co complex heat generation after seven minutes is so pronounced for the reaction of (*S*)-**3c** and *rac*-**3c** that no significant difference in visualization using the applied temperature window (1 °C range) can be detected. Therefore, the temperature scale covered by the window was increased to a total of 10 °C, resulting in the clear identification of the "hottest" reaction (cf. Figure 2e with 2f).

Finally, relative substrate activity was screened by studying the hydrolysis of three different chiral epoxides **3a**–**c** with the cobalt catalyst (*S*,*S*)-**5c**.[15] Figures 3a–c clearly demonstrate that (*S*)-**3a**[16] is the most reactive substrate followed by (*S*)-**3c** and (*R*)-**3b**. The same relative reactivity of (*R*)-**3b** and (*S*)-**3c** has previously been reported by Jacobsen et al. in laboratory-scale reactions.[13]

In conclusion, we have demonstrated that time-resolved IR-thermographic screening of enantioselectivity in catalytic reactions is feasible. Since spacial resolution is no problem, the screening of large libraries of asymmetric catalysts should be possible. The method should also be amenable to the study of other chemical and biochemical processes in solution such as molecular recognition in host–guest chemistry or antibody–antigen interactions. We are currently attempting to refine our approach to include quantitative analyses of catalyst activity and (enantio)selectivity.

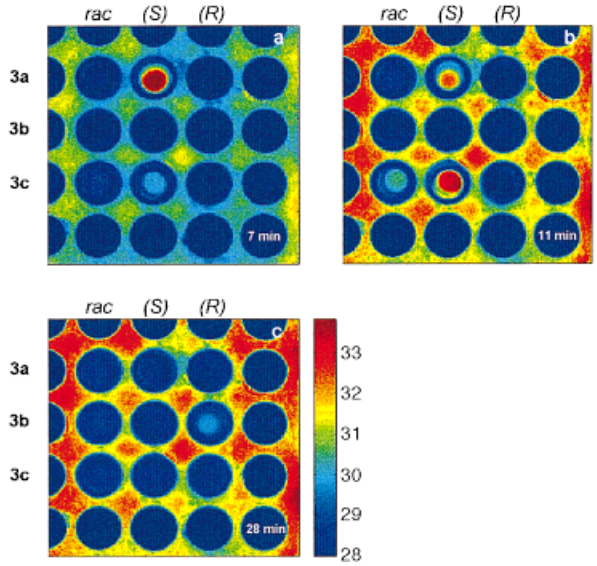

Figure 3. Time-resolved IR-thermographic imaging of the hydrolysis of epoxides **3a**–**c** catalyzed by (*S*,*S*)-**5c** after a) 7, b) 11, and c) 28 min. The bar on the far right is the temperature/color key of the temperature window used [°C].

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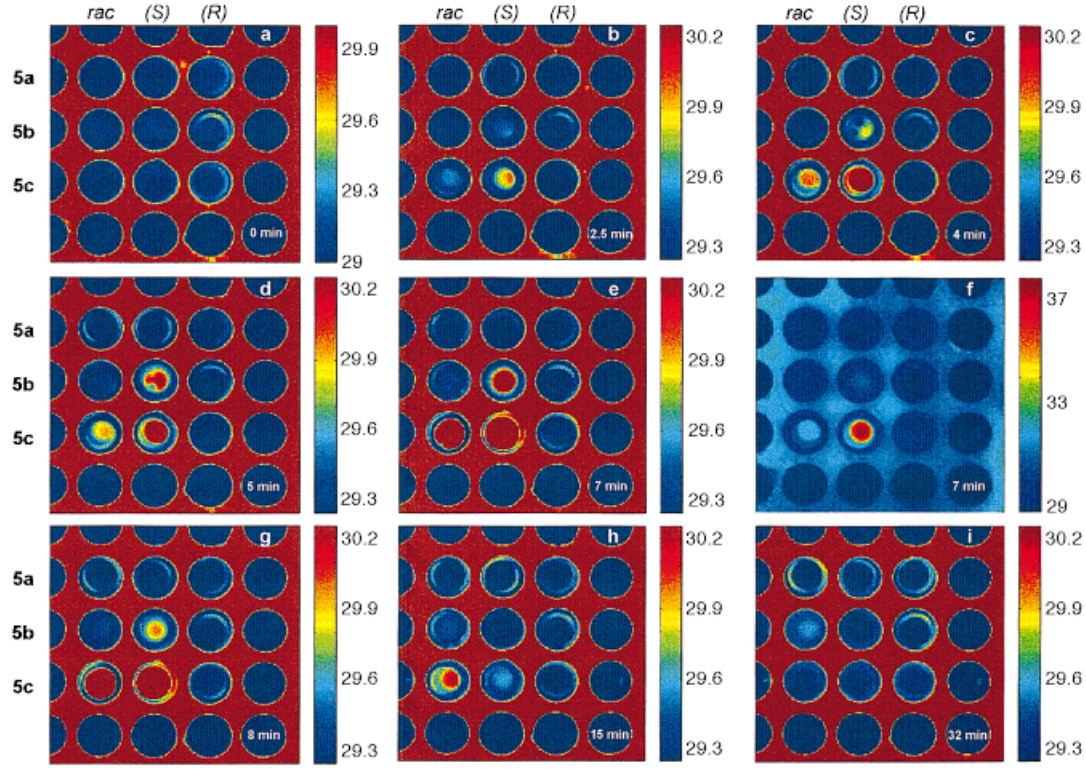

Figure 2. Time-resolved IR-thermographic imaging of the hydrolysis of **3c** catalyzed by metal complexes (*S*,*S*)-**5a**–**c** after a) 0, b) 2.5, c) 4, d) 5, e) 7, g) 8, h) 15, and i) 32 min. In (f) the same figure is shown as in (e), however, the temperature scale ranges over 10 K. The bar on the far right of each figure is the temperature/color key of the temperature window used [°C].

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Keywords: asymmetric catalysis · biocatalysts · combinatorial chemistry · homogeneous catalysis · IR-thermographic screening

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[7] In our system we used an AIM-256[2] IR camera (AIM, Heilbronn, Germany) with a PtSi-FPA detector and a germanium lens. This detection system is sensitive to IR radiation in the wavelength range 3 – 5 μm and to temperature changes of 10 – 100 mK, depending upon the conditions.

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[11] In the experiment equimolar solutions of 100 μL of (*R*)-**1**, (*S*)-**1** and *rac*-**1** in toluene were mixed correspondingly with 100 μL vinyl acetate in toluene. Three different concentrations were used: 0.5 M, 1 M and 2 M.

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[14] For the catalyst screening in the hydrolysis of epichlorohydrin **3c** a mixture of (*S*,*S*)-**5a**, (*S*,*S*)-**5b**, and (*S*,*S*)-**5c** (each 60.0 μmol) in toluene (1.00 mL) and acetic acid (120 μmol, 7.21 mg, 6.86 mL) was stirred in an open flask for 1 h. After removal of the solvent the three residues were dried in vacuum. Each of these activated catalysts (*S*,*S*)-**5a – c** was dissolved in toluene (300 μL) and distributed in three reaction vessels. Epichlorohydrin **3c** (1.00 mmol, 92.5 mg, 78.4 mL) was added. As described before the temperature was calibrated in the range of 24 – 39 °C. The reaction was initiated by the addition of water (0.55 equiv, 0.55 mmol, 9.9 μL) at 27 °C. Screening was performed similar to the first experiments, except that the detection time was prolonged, averaging 500 recordings in 10 s.

[15] For the substrate activity screening the cobalt catalyst (*S*,*S*)-**5c** was activated as described before. Nine aliquots each containing 2.00 μmol of the catalyst in toluene (100 μL) were distributed in the wells of the microtiter plate. The three different epoxides **3a – c** in the (*R*)-, (*S*)-, and *rac*-form were added to the catalyst, resulting in the arrangement shown in Figure 3. The reaction mixtures contained the epoxides at a concentration of 3.95 M in toluene and a final volume of 253 μL. Detection of the reactions was performed for 10 s, resulting in 500 recordings which were averaged.

[16] Substrate **3a** was not included in the studies carried out by Jacobsen et al.[12, 13]

Molecular Model for Aluminophosphates Containing Fluoride as a Structure-Directing and Mineralizing Agent**

Yu Yang, Jiri Pinkas, Martina Schäfer, and Herbert W. Roesky*

Dedicated to Professor Ernst Otto Fischer on the occasion of his 80th birthday

Microporous materials such as zeolites, alumino- and gallophosphates, and their transition metal substituted analogues are currently targets of a very active research effort. Their traditional uses as size- and shape-selective catalysts,[1] molecular sieves, adsorbents, and ion exchangers are complemented by new applications as reaction vessels of molecular dimensions,[2] highly ordered matrices for fabrication of optoelectronic nanodevices[3] and sensors,[4] and molds for the preparation of carbon molecular sieves.[5] Their syntheses rely mainly on hydrothermal methods in aqueous and recently also in nonaqueous media.[6, 7] Important developments in the synthetic methodology were achieved by introduction of HF in the reaction mixture. Fluoride acts as a mineralizer and a structure-directing agent,[8] and its presence promotes growth of large crystals of molecular sieves.[9] Fluoride itself is in many cases not retained in the resulting structure. However, in some frameworks fluoride ions coordinate to the metal center, as has been reported for aluminophosphates (AFI,[8]

[*] Prof. Dr. H. W. Roesky, Y. Yang, Dr. J. Pinkas, Dr. M. Schäfer
Institut für Anorganische Chemie der Universität
Tammannstrasse 4, D-37077 Göttingen (Germany)
Fax: (+49) 551-393373
E-mail: hroesky@gwdg.de

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