Calculations. Theoretical calculations were carried out at the restricted Hatree–Fock (RHF) level using AM1 semiempirical SCF-MO method as implemented in a modified version[51] of the MOPAC program.[52]

Acknowledgment. Financial support by the DGICYT (PB87/0019) is gratefully acknowledged. Thanks are also due to Prof. J.D. Martin (U. de La Laguna) and Dr. G. Tojo (U. de Santiago de Compostela) for carrying out our HRMS. Time allocation for calculations, performed with a VAX 8820 computer, was generously provided by the Centre de Càlcul de la Universitat de les Illes Balears.

Note Added in Proof. A total synthesis of racemic podophyllotoxin based on the above grounds (tandem photolysis/intramolecular Diels–Alder) has been recently reported: Kraus, G.; Wu, Y. *J. Org. Chem.* **1992**, *57*, 2922.

Supplementary Material Available: ^{1}H NMR spectra for compounds **5a**, **6a**, **7b**, **8a**, **11**, **13**, **15**, **18a**, **19a**, **18b** + **19b**, **18c** + **19c**, **20a**, and **20c** (16 pages). This material is contained in many libraries on microfiche, immediately follows this article in the microfilm version of the journal, and can be ordered from the ACS; see any current masthead page for ordering information.

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Influence of Microwaves on the Rate of Esterification of 2,4,6-Trimethylbenzoic Acid with 2-Propanol

Kevin D. Raner* and Christopher R. Strauss

CSIRO Division of Chemical and Polymers, Private Bag 10, Clayton, Victoria 3168, Australia

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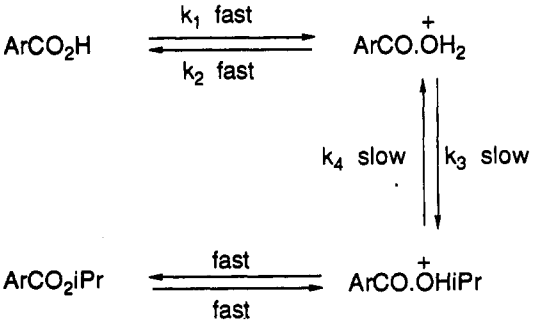
The influence of microwave irradiation on the reaction kinetics of the acid-catalyzed esterification of 2,4,6-trimethylbenzoic acid in *i*-PrOH was investigated. The rate constants for the reaction at various temperatures were measured in experiments conducted in an oil bath and the Arrhenius parameters were calculated. Reactions were carried out under microwave irradiation with different temperature profiles and the final ester concentrations were determined. The measured ester concentrations were in agreement with those calculated by computer modeling of the reaction using the Arrhenius parameters obtained from the oil bath experiments. The rate constant at 150 °C was directly determined in a recently developed microwave reactor and was consistent with the measured Arrhenius parameters. The rate of the esterification was concluded to be the same in both the microwave reactor and in the oil bath experiments.

It is well documented that microwave irradiation can be employed to accelerate chemical reactions[1–3] and rate enhancements of up to a 1000-fold over conventional conditions have been reported.[1]

An explanation for the fact that reaction rates are increased under microwave irradiation could be simply that the radiation leads to an increase in the reaction temperature. There have been suggestions however, of the existence of an additional "microwave effect" which can accelerate a reaction to a rate faster than would be expected on the basis of the measured reaction temperature.[4–6] Others have rejected this proposal.[7,8] Jahngen et al.[4] initially reported an effect in the hydrolysis of ATP. When they were able to take full account of the temperature gradients in the system, however, they concluded that the rate of hydrolysis was not influenced by microwave radiation.[7]

Two main experimental difficulties are largely responsible for the inconsistencies in the above literature. In order to perform kinetics studies the temperature must be known and the reaction solution must be either thermally homogeneous or have thermal gradients that are known or are capable of being modeled.

Scheme I



Domestic microwave ovens operate by alternating from maximum power output to zero power; this arrangement is unsatisfactory for precise control of temperature. For this reason we recently developed a microwave reactor which was more suitable for studying chemical kinetics.[9] The system consisted of a domestic microwave oven which was modified to allow operation at constant power output. The unit also possessed a facility for magnetic stirring. The internal temperature and pressure of the PFA/PTFE reaction vessel could be monitored and the temperature history could be recorded on a computer.

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The relationship between the rates of chemical reactions and temperature is defined by the Arrhenius equation. Therefore determining whether the reaction rate observed under microwave irradiation is the same as that expected from the Arrhenius equation should resolve the debate on the observed rate enhancements. This report examines the influence of microwave radiation on the kinetics of the acid-catalyzed esterification of 2,4,6-trimethylbenzoic acid in *i*-PrOH. The reaction was selected because it proceeded very slowly under reflux at atmospheric pressure but went readily under conditions of elevated temperature and pressure in the microwave unit.[9]

Results and Discussion

The mechanism of acid-catalyzed esterifications is usually of type $A_{Ac}2$, although for hindered carboxylic acids the reaction can also follow the $A_{Ac}1$ mechanism. The kinetics for the hydrolysis of various esters of 2,4,6-trimethylbenzoic acid have been described in the literature under basic[10-12] and acidic conditions.[13-15] Since the kinetics of this particular esterification have not been studied under these conditions, the mechanism operating was uncertain. For the purpose of this work, however, the reaction can be represented in the somewhat simplistic form shown in Scheme I.

Scheme I involves a protonation step followed by a substitution step and finally deprotonation to yield the ester. If the protonated carboxylic acid reacts with *i*-PrOH to form a tetrahedral intermediate, this constitutes the $A_{Ac}2$ mechanism. However, if the protonated acid dehydrates to yield an acyl cation, then the mechanism will be unimolecular ($A_{Ac}1$). If the reverse reaction (hydrolysis) is ignored[16] (i.e., $k_4 = 0$, see Scheme I), then eq 1 can be derived for the case of the $A_{Ac}2$ mechanism and eq 2 for the $A_{Ac}1$ mechanism. In these equations, k_1 and k_2 are the rate constants for the fast initial protonation/deprotonation of the carboxylic acid and k_3 is the rate constant for the substitution step. If the esterification is carried out with a large excess of *i*-PrOH, then [*i*-PrOH] can be regarded as constant and both equations can be represented as shown in eq 3. Since $[H^+]$ is independent of time, eq

$$\frac{d[ArCO_2\text{-}i\text{-}Pr]}{dt} = \frac{k_1k_3}{k_2}[ArCO_2H][i\text{-}PrOH][H^+] \quad (1)$$

$$\frac{d[ArCO_2\text{-}i\text{-}Pr]}{dt} = \frac{k_1k_3}{k_2}[ArCO_2H][H^+] \quad (2)$$

$$\frac{d[ArCO_2\text{-}i\text{-}Pr]}{dt} = k_{obs}[ArCO_2H][H^+] \quad (3)$$

4 can be derived from eq 3, where $[ArCO_2H]_0$ is the initial concentration of the carboxylic acid.

$$[ArCO_2\text{-}i\text{-}Pr]_t = [ArCO_2H]_0(1 - e^{-k_{obs}[H^+]t}) \quad (4)$$

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(16) By subjecting the isopropyl ester and 1 equiv of water to typical reaction conditions, it was found that *ca.* 20% hydrolysis occurred. However, during the initial periods of the esterification reactions, i.e., when the ester and water concentrations are very low, there will be only a negligible amount of ester hydrolysis. Therefore, the esterifications were limited to low conversion. By allowing some reactions to proceed to high conversions it was found that the conversions at equilibrium were in the order of 50–70%.

Table I. Kinetic Data for the Esterification of 2,4,6-Trimethylbenzoic Acid in *i*-PrOH under Catalysis by TsOH with Conventional Heating

temperature (°C)	$[ArCO_2H]_0$ (M)	[TsOH] (M)	[*i*-PrOH] (M)	k_{obs}[a] (M^{-1} s^{-1})
120	0.283	0.190	12.5	7.12×10^{-7}
130	0.281	0.196	12.5	7.81×10^{-6}
140	0.281	0.196	12.5	1.67×10^{-5}
150	0.281	0.196	12.5	3.83×10^{-5}
160	0.281	0.196	12.5	1.27×10^{-4}
170	0.281	0.196	12.5	6.38×10^{-4}

[a] The uncertainty in these measurements is ca. ± 10%.

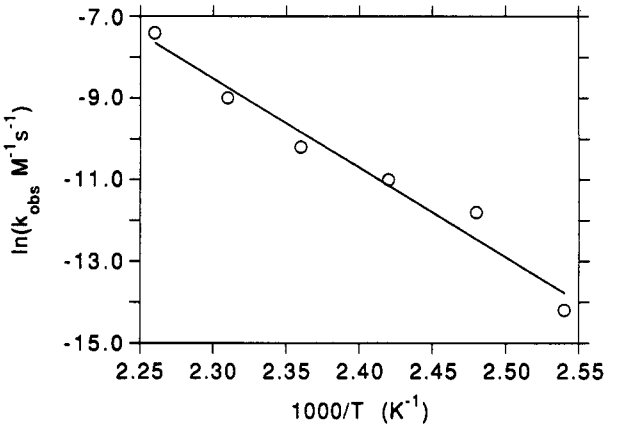

Figure 1. Arrhenius plot for the esterification of 2,4,6-trimethylbenzoic acid in *i*-PrOH under catalysis by TsOH with conventional heating.

This reaction was first investigated under conventional conditions to establish the Arrhenius parameters. Solutions containing 2,4,6-trimethylbenzoic acid and TsOH in *i*-PrOH were prepared. These solutions also contained 2-methylnaphthalene as an internal standard. The solutions were sealed in glass pressure tubes and were then placed in a thermostatted oil bath. Samples were withdrawn at intervals and were analyzed by GC. The overall rate constant, k_{obs}, was obtained by least-squares fitting of eq 4 to a plot of ester concentration versus time. The measured overall rate constants at various temperatures are shown in Table I.

The Arrhenius parameters for this reaction were determined from a plot of ln k_{obs} versus $1/T$. The Arrhenius plot is presented in Figure 1 and the rate constant at temperature T is given by eq 5. The regression errors were calculated at the 95% confidence level.

$$\ln k_{obs}(M^{-1}\ s^{-1}) = (42.3 \pm 4.7) - \frac{183 \pm 16\ (kJ\ mol^{-1})}{RT} \quad (5)$$

A kinetic analysis of this reaction was then made in the microwave reactor.[9] A solution of 2,4,6-trimethylbenzoic acid and TsOH in *i*-PrOH was prepared and added to the PFA Teflon/PTFE reaction vessel. 2-Methylnaphthalene was also added as an internal standard. A magnetic stirrer bar was placed in the vessel which was then positioned in the reactor and fitted with the pressure gauge and fiber optic thermometer. The microwave power was applied and the solution was heated to a suitable reaction temperature. After a certain time, the power was reduced or turned off and the solution was allowed to cool. The recorded temperature–time profile and the Arrhenius parameters (eq 5) were then used to predict the final ester concentration of the reaction. This was accomplished by segmenting the temperature–time profile and fitting polynomial functions to each segment. In this way the temperature profile could be described mathematically. A computer program,

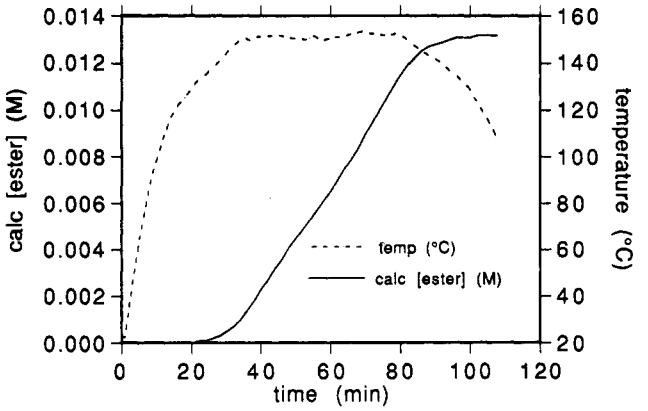

Figure 2. Temperature profile and calculated (LARKIN) [ester] profile of reaction A (see Table II).

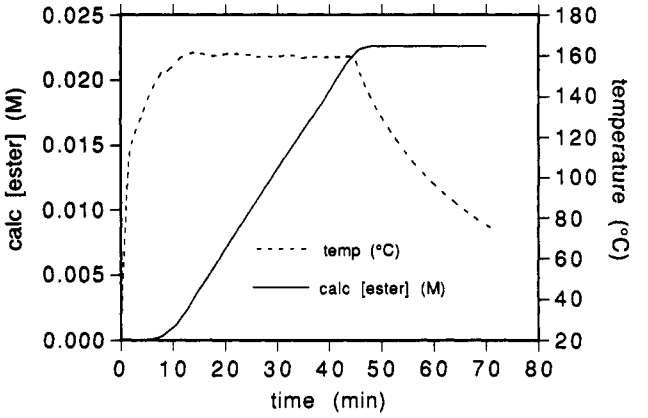

Figure 3. Temperature profile and calculated (LARKIN) [ester] profile of reaction B (see Table II).

Table II. Predicted and Actual Final Ester Concentrations in the Esterification of 2,4,6-Trimethylbenzoic Acid under Microwave Irradiation

reaction[a]	predicted $[ArCO_2\text{-}i\text{-}Pr]_{final}$ (M)	actual $[ArCO_2\text{-}i\text{-}Pr]_{final}$ (M)	% error[b]
A	0.0132	0.0142	7.0
B	0.0226	0.0207	−9.2
C	0.0298	0.0319	6.6

[a] Reaction conditions: $[ArCO_2H]_0$ = 0.280 M, [TsOH] = 0.190 M, [*i*-PrOH] = 12.5 M. Temperature profiles for A, B, and C are shown in Figures 2, 3, and 4, respectively. [b] % error calculated as 100 (actual $[ArCO_2\text{-}i\text{-}Pr]_{final}$ − predicted $[ArCO_2\text{-}i\text{-}Pr]_{final}$)/actual $[ArCO_2\text{-}i\text{-}Pr]_{final}$.

LARKIN,[17] was then used to model the reaction. The mathematical description of the temperature profile was linked to the program as an external subroutine. The reaction mixture was analyzed by GC and the analytically determined final ester concentration was compared to the calculated final ester concentration.

Three reactions were run with three different temperature profiles (see Figures 2–4). The final ester concentrations were predicted and in each case there was good agreement (less than 10% discrepancy, see Table II) with the final ester concentrations as determined by GC analysis. Since the computed final ester concentrations were based on the Arrhenius parameters measured in conventional oil bath experiments, they demonstrate that the reaction under microwave irradiation proceeded to the same degree of conversion as would be expected in an oil bath.

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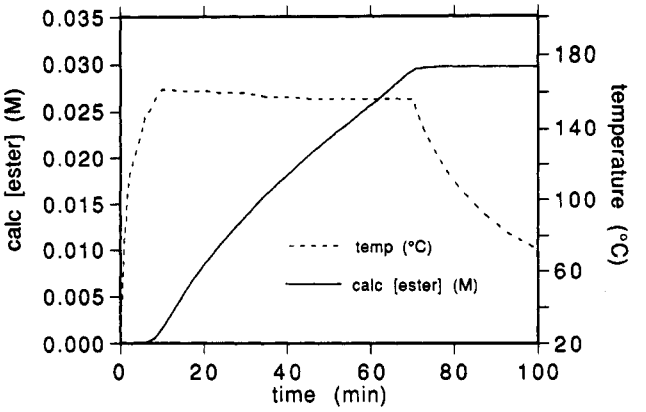

Figure 4. Temperature profile and calculated (LARKIN) [ester] profile of reaction C (see Table II).

Table III. Esterification of 2,4,6-Trimethylbenzoic Acid in *i*-PrOH under Catalysis by TsOH under Microwave Irradiation

time (h)	$[ArCO_2\text{-}i\text{-}Pr]_{final}$ (M)
1.5	0.0203
2.0	0.0258
2.5	0.0317
3.0	0.0354

Figures 2–4 show the temperature profiles for the above reactions and also the calculated ester concentration versus time. Figure 2 shows that a small but significant amount of reaction occurred during the heating up and cooling down periods. However, if the reaction was maintained at a high constant temperature for sufficient time, then the extent of reaction occurring on heating up and cooling down would be insignificant.

Four reactions were conducted in the microwave reactor. These reactions were subjected to a rapid heating up period (<4 min) and were then maintained at 150 ± 0.5 °C for 1.5–3.0 h. When the power was turned off, the initial rate of cooling was fast (the reaction cooled to 120 °C in 5 min). Since the rate of reaction is very slow at temperatures below 120 °C (see Table I), the extent of reaction on heating up and cooling down was considered to be negligible. After cooling, the reaction solution was analyzed by GC and the results are shown in Table III.

Fitting eq 4 to these data gave a rate constant of 6.94×10^{-5} M^{-1} s^{-1}. This rate constant is in reasonable agreement with the rate constant of 3.83×10^{-5} M^{-1} s^{-1}, measured in an oil bath at 150 °C. The experimental error in these measurements is of the order of 10%. Equation 5 yields a more reliable value for the rate constant, derived from conventionally obtained data, as 6.01×10^{-5} M^{-1} s^{-1} at 150 °C. Taking into account the experimental error, this value is in good agreement with the rate constant measured in the microwave reactor.

Conclusions

When the esterification reaction was carried out under microwave irradiation, with variable temperature, the final ester concentration agreed well with that calculated by modeling an analogous oil bath experiment with the same temperature profile. This indicated that the final yield of ester depended only on the nature of the temperature profile and not on the mode of heating. Experiments were also conducted in the microwave reactor such that essentially all of the reaction occurred at constant temperature. The observed rate constant was found to be the same as that calculated from the Arrhenius parameters determined

from experiments conducted in oil baths.

These experiments demonstrate that the reaction rates for the esterification under microwave irradiation are not different from the rates observed under conventional heating and support the conclusions of earlier workers.[7,8]

Experimental Section

NMR spectroscopy was performed at 90 MHz. GC analysis was carried out on an instrument fitted with a QS BP5 capillary column 25 m in length. Helium was used as the carrier gas at a flow rate of 2.0 mL/min. The oven was maintained at 50 °C for 2 min and was then heated at 10 °C/min to a final temperature of 280 °C. The final temperature was held for 10 min.

Esterification of 2,4,6-Trimethylbenzoic Acid with *i*-PrOH under Conventional Conditions. An *i*-PrOH solution containing 2,4,6-trimethylbenzoic acid (0.28 M), TsOH (0.19 M), and 2-methylnaphthalene (0.050 M) was prepared. Samples of this solution (5 mL) were sealed in thick-walled glass tubes and were placed in an oil bath thermostatted at the appropriate temperature. The tubes were withdrawn at intervals and were cooled and opened. Pyridine (0.25 mL) was added to a small sample of the reaction mixture (0.5 mL) and the solution was diluted by addition of dichloromethane (2 mL). This was then analyzed by GC: the t_R of the various peaks were 11.22 (2-methylnaphthalene), 13.06 (2,4,6-trimethylbenzoic acid), and 13.68 min (isopropyl 2,4,6-trimethylbenzoate).

Esterification of 2,4,6-Trimethylbenzoic Acid with *i*-PrOH in the Microwave Reactor. *i*-PrOH solutions (75 mL) containing 2,4,6-trimethylbenzoic acid (0.28 M), TsOH (0.19 M), and 2-methylnaphthalene (0.050 M) were prepared and added to the PFA Teflon/PTFE reaction vessel. The microwave power was applied and the solution temperature was raised to the desired level. The temperature was monitored throughout the experiment by a Luxtron fluoroptic thermometer probe located in the reaction vessel. Thermal homogeneity was maintained by magnetic stirring. After the appropriate time, the microwave power was either reduced or turned off to allow the reaction solution to cool. The vessel was then opened and the contents were analyzed by GC as described above.

Acknowledgment. We thank Kodak (Australasia) Pty Ltd. for financial support and F. Vyskoc for technical assistance.

Registry No. 2,4,6-Trimethylbenzoic acid, 480-63-7; 2-propanol, 67-63-0; isopropyl 2,4,6-trimethylbenzoate, 41589-61-1.

Synthesis of CBI-PDE-I-Dimer, the Benzannelated Analogue of CC-1065

Paul A. Aristoff* and Paul D. Johnson

Medicinal Chemistry Research, The Upjohn Company, Kalamazoo, Michigan 49001

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A practical synthesis of CBI (2), utilizing inexpensive starting materials, was developed and applied to the synthesis of benzannelated analogs of CC-1065, in particular CBI-PDE-I-dimer (13) and CBI-bis-indole (17). While a Sharpless asymmetric dihydroxylation reaction proved effective at providing optically active intermediates, a more classical resolution procedure was used to prepare materials of higher optical purity. A novel cyclization employing a six-membered-ring intermediate (12) was employed to construct the cyclopropyl ring in CBI. Like CC-1065, CBI-PDE-I-dimer appears to cause delayed toxicity in mice.

CC-1065 (1), an extremely potent antitumor antibiotic,[1] exhibits a number of interesting biological effects,[2] including the production of delayed deaths in mice at microgram per kilogram doses.[3] Subsequent investigation of this fascinating natural product revealed that the delayed lethality of the compound resulted when the carbon skeleton of PDE-1-dimer (the right-hand portion of the molecule) was attached to CPI, the left-hand alkylating segment.[4] Structurally simplified CPI derivatives were shown not only to be free of this detrimental toxicity, but also to be much more active than CC-1065, and one such compound has since entered clinical testing.[5] To better understand the structural features of CC-1065 responsible for its biological effects, we have had an interest in preparing compounds containing an altered CPI moiety, including the benzannelated derivative CBI (2). The synthesis of CBI was first reported by Boger who has also reported the preparation of a number of interesting CBI analogues.[6] Cava has also reported the preparation of a protected CBI derivative.[7] Herein we describe an alternative synthesis of CBI and its application to the preparation of CBI-PDE-I-dimer (13). Unlike previous routes

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