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Shock-tube study of the autoignition of *n*-heptane/toluene/air mixtures at intermediate temperatures and high pressures

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Abstract

The ignition delay times of mixtures containing 35% *n*-heptane and 65% toluene by liquid volume at room temperature (i.e., 28% *n*-heptane/72% toluene by mole fraction) were determined in a high-pressure shock tube in the temperature range $620 \leq T \leq 1180$ K at pressures of about 10, 30, and 50 bar and equivalence ratios, ϕ , of 0.3 and 1.0. The equation

$$\tau/\mu\text{s} = 9.8 \times 10^{-3} \exp(15,680 \text{ K}/T)(p/\text{bar})^{-0.883}$$

represents the data for $\phi = 0.3$ in the temperature range between 980 and 1200 K. At lower temperatures no ignition was found at 10 bar within the maximum test time of 15 ms, whereas for 50 bar, a reduced activation energy was observed. A pressure coefficient of -1.06 was found for the data with $\phi = 1.0$. No common equation for the data at $\phi = 1.0$ could be found analogous to that for $\phi = 0.3$ because the ignition delay times show no Arrhenius-like behavior. A comparison with ignition delay times of *n*-heptane/air and toluene/air for $\phi = 1.0$ and 30 bar shows that the values of the mixture of the two components are between the values of the single substances. Furthermore, the results confirm the negative temperature coefficient behavior found for the mixtures at 30 and 50 bar, similar to *n*-heptane/air. A comparison for the other pressure and equivalence ratio values of this study was not possible because of the lack of data for pure toluene. These experimental data have been used in the development of a chemical kinetics model for toluene/*n*-heptane mixtures as described in a companion paper.

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Keywords: *n*-Heptane; Toluene; Autoignition; Shock tube; Reference fuel

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1. Introduction

The focus of development in internal combustion (IC) engines is shifting toward improving fuel economy. Knock, caused by the autoignition of the fuel/air mixture ahead of the advancing flame front, is a major constraint on fuel efficiency of spark-ignition (SI) engines. Meanwhile, there is also a great deal of interest in homogeneous charge compression ignition (HCCI) engines, since they offer the prospect of the high efficiency of the compression-ignition (CI) engine, but with low levels of nitrogen oxides and soot, which are very difficult to control in CI engines. The time of heat release in HCCI engines is determined by the chemical kinetics of the autoignition of the fuel and air mixture, whereas in conventional CI the time of heat release depends on fuel/air mixing as well as on the autoignition properties of the fuel. Thus, autoignition is of critical importance in IC engines and in all cases is governed by the same chemical kinetic mechanism, dominated by decomposition of H_2O_2 [1] and reaction between OH and fuel [2].

Autoignition in an IC engine depends on the fuel as well as on the development of pressure and temperature in the unburnt mixture. Chemical reaction mechanisms applicable to autoignition in IC engines have only been developed for very few pure compounds—mostly paraffins with low carbon numbers [3]. Another difficulty is that in a mixture, the coupling between the chemical kinetics of the components is significant [4]. Thus, even if the autoignition chemistry of two pure fuels is fully understood, it is not a simple matter to develop the autoignition chemistry of mixtures of the two fuels. Chemical kinetic schemes have been developed for mixtures of iso-octane and *n*-heptane, known as primary reference fuels (PRF), but those for other relevant mixtures containing aromatics or olefins and paraffins are only just appearing [5,6]. A parameter often used in autoignition studies is the ignition delay, τ , which is the time required for autoignition to happen once the fuel/air mixture is raised to a given pressure and temperature and held at that condition in a rapid compression machine or a shock tube. τ decreases as either the temperature T or pressure the p increases and can be expressed in general as

$$\tau = f(T)p^{-x}, \quad (1)$$

where x is a constant that has been measured to be different for different types of fuels. If Eq. (1) is known for a given fuel/air mixture, it can be assumed that autoignition occurs under variable p , T conditions when the following condition is satisfied [7]:

$$\int \frac{1}{\tau(p, T)} dt = 1. \quad (2)$$

However, Eq. (1) is known only for a few fuels and over a limited range of p and T .

Practical fuels are complex mixtures of hydrocarbons. Gasoline-like fuels used in SI engines must be much more resistant to autoignition, in order to avoid knock, than Diesel-like fuels used in CI engines. Empirical measures such as the research octane number (RON) and motor octane number (MON) for gasoline-like fuels and the cetane number (CN) for Diesel-like fuels are used to characterize autoignition in IC engines using all such practical fuels, since they cannot be fully described in terms of autoignition chemistry at the moment. However, the RON and MON scale are both based on PRF and the RON and MON of any fuel containing nonparaffinic components describe its autoignition behavior only at the respective RON and MON test conditions. RON is higher than MON for practical fuels but RON is the same as MON for PRF. In fact, for gasoline-like fuels the autoignition quality is best described by the octane index, $\text{OI} = (1 - K) \times \text{RON} + K \times \text{MON}$, where K is a constant depending on the pressure and temperature development in the fuel/air mixture and is a measure of how far away the particular test condition is compared to the RON test condition [8]; OI is the octane number of the equivalent PRF at the operating condition used. The value of K can vary widely, so that the same fuel can behave like different PRF at different operating conditions. Empirically it is found that K depends strongly on $T_{\text{comp}15}$, the compression temperature when the pressure in the mixture is 15 bar [8]. K is negative if $T_{\text{comp}15}$ is less than that found in the RON test condition because, say, at the start of compression, the pressure is higher or the temperature is lower. Thus the OI will be higher than the RON and the fuel will be more resistant to autoignition than a PRF of the same RON.

There has been a recent period when the primary focus of spark-ignition engine development was the control of emissions. However, apart from this period, the main thrust in the development of spark ignition engines has been and once again continues to be to improve efficiency and power density. Strategies used to achieve this, such as increasing the compression ratio, direct injection, and downsizing coupled with turbocharging, lead to a higher pressure of the unburnt mixture at a given temperature. Such strategies also increase the maximum temperature and pressure that the unburned mixture can reach and so make the engine more prone to knock; fuels for such engines need to have greater resistance to autoignition. So the historical trend has been toward a reduction in $T_{\text{comp}15}$ and consequently a decreasing value of K . In fact, in modern engines, the K value is usually negative, so that for a given value of RON, a fuel with lower MON has better resistance to knock; thus fuels that are non-

paraffinic are better at resisting knock [8]. Thus, in such engines, fuels containing aromatics, olefins, and oxygenates will have better anti-knock quality. Such issues are of great practical significance for future fuels and engines, as discussed in [8]. It is important that such empirical knowledge gained about practical fuels is explained in fundamental chemical kinetic terms. This requires development of chemical kinetic schemes for fuel mixtures that are more like practical fuels than PRF. One such fuel is a simple toluene/*n*-heptane mixture, toluene reference fuel (TRF), and an attempt to model the chemical kinetics of such a fuel was described in [5]. A chemical kinetic model for a five-component surrogate fuel for gasoline is discussed in [6]. Refinement of such models depends critically on the availability of experimental data, particularly on ignition delay times [9]. In this work, ignition delay times have been measured for the TRF considered in [5] using a shock tube at variable pressures and temperatures and at two different fuel/air ratios. An accompanying paper describes the refinement of the chemical kinetic model for toluene/*n*-heptane mixtures [10] using these and other data.

2. Experimental

The experiments were carried out in a heated high-pressure shock tube with an internal diameter of 90 mm. It is divided by an aluminum diaphragm into a driver section of 6.1 m and a driven section of 6.4 m in length. The driven section was pumped down to pressures below 10^{-2} mbar. Gas mixtures were prepared by injection of a liquid mixture of the fuel components and subsequent complete evaporation in a stainless steel mixing vessel. The total amount of

fuel and air was controlled manometrically in order to ensure the desired equivalence ratio. The shock tube and the mixing vessel were heated to 130 °C for those experiments at $\phi = 1$. The shock speed was measured over two intervals using three piezoelectric pressure gauges. The data were recorded with a time resolution of 0.1 μ s. The temperature and pressure behind the reflected shock wave were computed from the measured incident shock speed and the speed attenuation using a one-dimensional shock-tube model (shock-tube code of the CHEMKIN package [11]). The estimated uncertainty in reflected shock temperature is less than ± 25 K in the temperature and time range of our measurements. The experiments were carried out in synthetic air containing 79.5% N₂ and 20.5% O₂. ASTM *n*-heptane and ASTM toluene, which are liquid under normal conditions, were injected as liquids into the mixing vessel.

The ignition was observed by measuring pressure profiles with a piezoelectric gauge (PCB HM 112 A03) located 15 mm upstream of the end flange. Also, the CH* emission at 431.5 nm was selected by a narrow-band pass filter and measured with a photomultiplier. All ignition delay times shown in this work were determined by extrapolating the steepest increase of the emission signal to its zero level on the time axis.

The driver gas was mixed in situ by using two high-pressure mass-flow controllers (Bronkhorst Hi-Tec flow meter F-136AI-FZD-55-V and F-123MI-FZD-55-V); see Fig. 1. Helium was used as the main component and argon was added to match the acoustic impedance of the test gas. The required driver gas composition was calculated by a spreadsheet analysis prior to the experiments using equations by Oertel [12] and Palmer and Knox [13]. Concentrations of

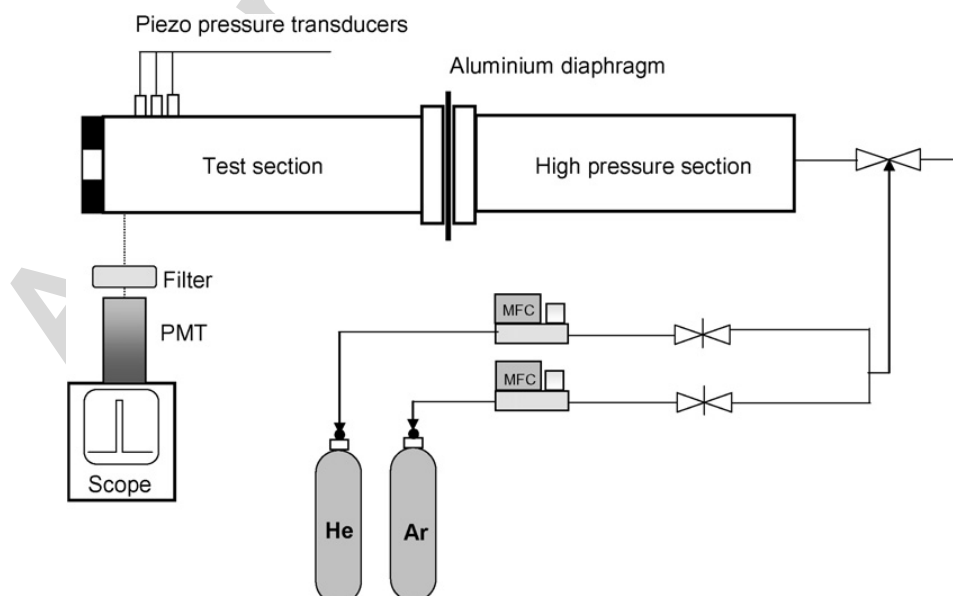


Fig. 1. Experimental setup.

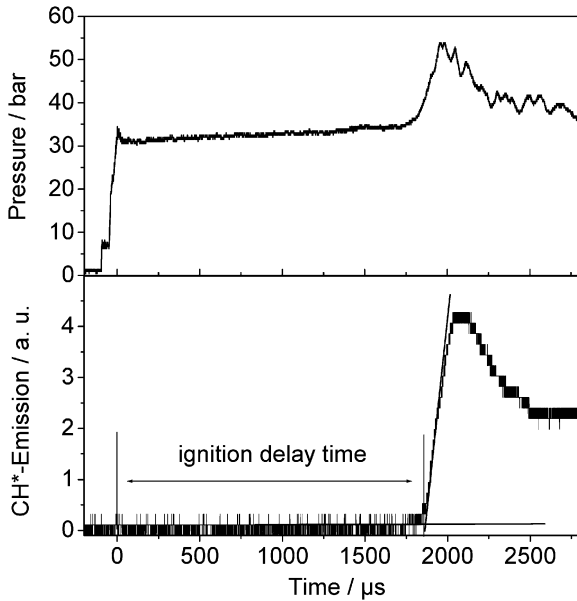


Fig. 2. Typical pressure (upper part) and CH^{*}-emission (lower part) profiles indicating ignition delay in a stoichiometric *n*-heptane/toluene (35/65% by liquid volume)/air mixture. Reaction conditions: $T_5 = 1062$ K, $p_5 = 11.3$ bar.

5 to 20% Ar in He were required to generate tailored shock waves.

3. Results

The temperature range of the present *n*-heptane (35%)/toluene (65% by liquid volume)/air ignition-delay study was $620 \leq T \leq 1180$ K at pressures of about 10, 30, and 50 bar and fuel/air equivalence ratios ϕ , of 0.3 and 1.0. The fuel has a RON of 84 and a MON of 73.2. Typical pressure and CH^{*} emission profiles are shown in Fig. 2. The pressure signal of the experiment at $T = 1062$ K and 11.3 bar with an equivalence ratio $\phi = 1.0$ (upper part of Fig. 2) shows a two-step increase due to the incident and reflected shock wave (time zero) followed by a constant pressure for about 2 ms and then an increase of about 19 bar. The CH^{*} emission (lower part of Fig. 2) remains at zero level for 1.9 ms, followed by a steep rise that indicates ignition.

4. Discussion

The individual ignition delays evaluated from the CH^{*} emission signals are summarized in Figs. 3 and 4 for equivalence ratio $\phi = 0.3$ and 1.0, respectively. The data are also presented in Table 1 together with the experimental conditions, where no ignition was observed during the maximum test time of 15 ms. Because of the nature of shock-tube measurements the pressure scatters slightly around the target pres-

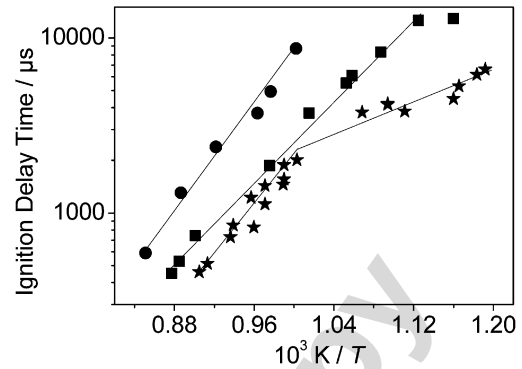


Fig. 3. Measured ignition delay times for the mixture of *n*-heptane and toluene (35/65% by liquid volume) in air ($\phi = 0.3$) at pressures of about 10 (circles), 30 (squares), and 50 bar (stars). The lines connect the data points to illustrate the trends of the data. The actual pressures slightly scatter around the target pressures for each measurement, as shown in Table 1.

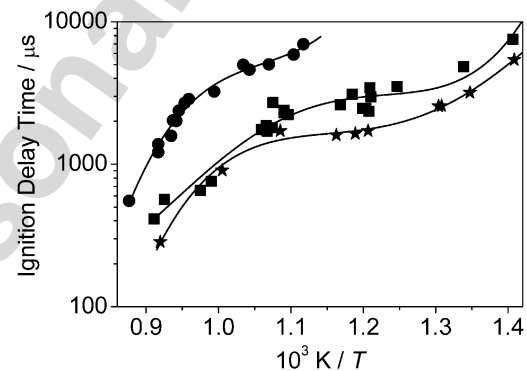


Fig. 4. Measured ignition delay times for the mixture of *n*-heptane and toluene (35/65% by liquid volume) in air ($\phi = 1.0$) at pressures of about 10 (circles), 30 (squares), and 50 bar (stars). The lines connect the data points to illustrate the trends of the data. The actual pressures slightly scatter around the target pressures for each measurement as shown in Table 1.

sures of 10, 30, and 50 bar. The actual pressures are documented in Table 1. The data shown in Figs. 3 and 4 are not corrected for these pressure variations.

We first assume that all the data with equivalence ratio $\phi = 0.3$ can be described by an equation of the form

$$\tau = A \exp(B/T) p^{-x}, \quad (3)$$

where p is the pressure and x is the pressure exponent. Multiple linear regression analysis using $\ln(\tau)$ as the dependent variable and $(1/T)$ and $\ln(p)$ as the independent variables helps identify the value of x as 0.883 using all the data listed in Table 1.

Thus, in the temperature range between 980 and 1200 K, the ignition delay time τ in μ s of the *n*-heptane/toluene/air mixture with $\phi = 0.3$ is well descri-

Table 1

Measured ignition delay times of a mixture of *n*-heptane (35%) and toluene (65%) by liquid volume at room temperature at different equivalence ratios, pressures and temperatures

$\phi = 0.3$			$\phi = 1.0$		
<i>T</i> (K)	<i>p</i> (bar)	τ (μ s)	<i>T</i> (K)	<i>p</i> (bar)	τ (μ s)
1180	11.6	590	1140	11.3	550
1130	10.7	1300	1090	10.3	1380
1090	9.9	2390	1090	11.4	1220
1040	12.3	3720	1070	11.0	1580
1020	12.0	4940	1070	11.4	2030
1000	11.3	8710	1060	11.3	2020
960	10.5	n.i.	1060	11.2	2380
900	11.7	n.i.	1050	10.5	2690
760	12.4	n.i.	1040	10.4	2870
720	10.8	n.i.	1010	9.6	3240
640	12.2	n.i.	970	10.9	4980
			960	13.8	4610
1140	32.7	450	940	11.7	5020
1130	32.2	530	910	10.9	5880
1110	31.0	740	900	10.6	6950
1020	30.4	1860	810	10.6	n.i.
990	33.0	3730	760	10.7	n.i.
950	30.5	5520	710	10.8	n.i.
940	30.1	6090	690	10.1	n.i.
920	30.1	8320	640	9.8	n.i.
890	27.9	12,600			
860	28.2	12,930	1100	31.2	410
850	30.7	n.i.	1080	30.2	570
770	31.9	n.i.	1030	31.5	650
700	30.6	n.i.	1010	30.4	760
630	30.8	n.i.	950	29.6	1750
610	28.0	n.i.	940	30	1870
			940	30.0	1870
1110	51.1	460	940	30.4	1710
1100	50.2	510	930	30.0	1800
1070	47.6	730	930	28.6	2700
1070	47.5	850	920	28.9	2290
1050	45.8	1220	920	28.9	2390
1040	52.4	830	910	28.6	2240
1030	44.5	1430	860	37	2620
1030	60.0	1120	840	29	3100
1010	49.2	1460	830	30	2460
1010	42.5	1880	830	34	2460
1010	57.9	1560	830	30	2350
1000	56.2	2010	830	29	2960
940	49.0	3760	830	30.8	3450
910	46.6	5310	800	32	3510
910	46.6	5440	750	31.5	4840
900	45.0	3810	710	31	7540
860	52.4	4490	670	32	n.i.
860	51.9	5310			
850	50.0	6180	1090	48.3	290
840	49.2	6610	1000	46.6	910
750	50.2	n.i.	920	46.4	1710
680	47.5	n.i.	840	45	1640
680	47.3	n.i.	860	51.5	1600
670	44.8	n.i.	830	49.3	1720

Table 1 (continued)

$\phi = 0.3$			$\phi = 1.0$		
<i>T</i> (K)	<i>p</i> (bar)	τ (μ s)	<i>T</i> (K)	<i>p</i> (bar)	τ (μ s)
			760	46.5	2570
			770	47.3	2560
			740	49.8	3190
			710	52.3	5410
			640	48.7	n.i.

Note. n.i.: no ignition within the maximum observable test time of 15 ms.

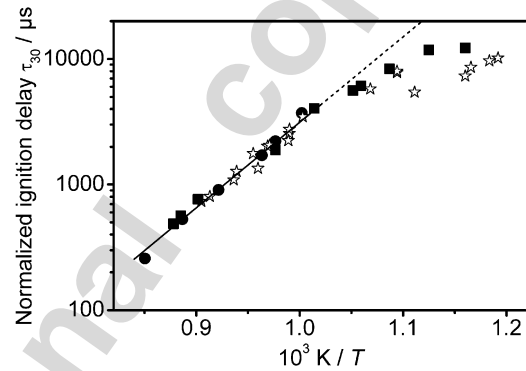


Fig. 5. Ignition delay times for the mixture of *n*-heptane and toluene (35/65% by liquid volume) in air ($\phi = 0.3$) at pressures of about 10 (circles), 30 (squares), and 50 bar (stars) normalized to 30 bar using $(30 \text{ bar}/p)^{-0.883}$. The solid line shows the fit (Eq. (4)) of the normalized data, the dashed line an extrapolation of the fit.

bed by the equation

$$\tau/\mu\text{s} = 9.8 \times 10^{-3} \exp(15,680 \text{ K}/T)(p/\text{bar})^{-0.883}. \quad (4)$$

With the obtained scaling factor $x = 0.883$ the ignition delay times measured for all pressures were normalized to 30 bar and gave

$$\tau_{30}/\mu\text{s} = 4.854 \times 10^{-4} \exp(15,680 \text{ K}/T). \quad (5)$$

In Fig. 5, τ_{30} , the ignition delay time scaled to 30 bar, is plotted against $1000 \text{ K}/T$; $\tau_{30} = \tau(30 \text{ bar}/p)^{-0.883}$. In the temperature range between 980 and 1200 K, a best-fit line given by Eq. (5) fits τ_{30} very well.

For $\phi = 1$, the pressure exponent x is around 1.06. Fig. 6 shows $\tau_{30} = \tau(30 \text{ bar}/p)^{-1.06}$ for this equivalence ratio. As the data do not show Arrhenius-like behavior, they cannot be described by equations such as (4) and (5).

It is worth noting that the value of x is much lower for this fuel than for primary reference fuels reported, for instance, in [9]. In general, compared to paraffins, aromatic and olefinic fuels become more resistant to autoignition, and the value of τ decreases more slowly, when the pressure is increased for a given temperature [8]. In fact, Bradley et al. could explain the

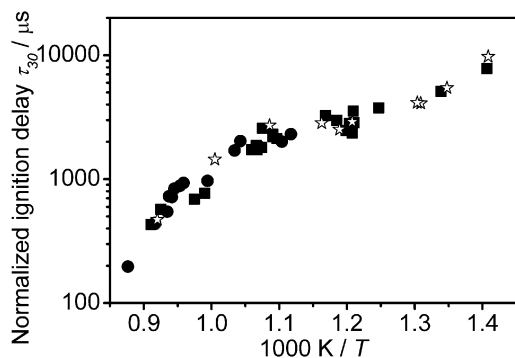


Fig. 6. Ignition delay times for the mixture of *n*-heptane/toluene (35/65% by liquid volume) in air ($\phi = 1.0$) at pressures of about 10 (circles), 30 (squares), and 50 bar (stars) normalized to 30 bar using $(30 \text{ bar}/p)^{-1.059}$.

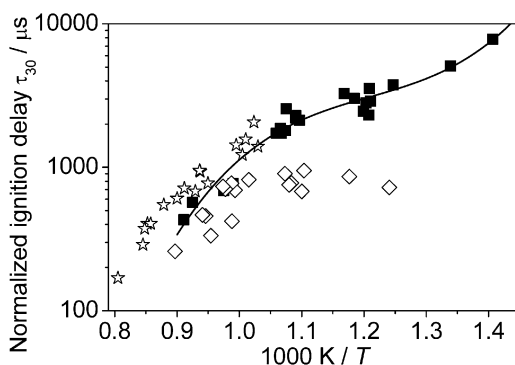


Fig. 7. Comparison of the ignition delay times for the mixture of *n*-heptane and toluene (35/65% by liquid volume) in air ($\phi = 1.0$) (squares) at about 30 bar, and *n*-heptane in air ($\phi = 1.0$) (diamonds [16]), and toluene in air (stars [17]). Data normalized to 30 bar.

behavior of the fuel mixture considered in this paper in an HCCI engine by using Eq. (2) but only by assuming that the value of x was lower for it compared to the PRFs [14]. Similarly Kalghatgi [15] also could explain why a gasoline fuel was more resistant to knock than indicated by its RON, i.e., why the K value was negative, in a SI engine using Eq. (2) but again only by assuming a lower value of x for the gasoline than for the PRFs.

Fig. 7 shows the comparison between the data for the *n*-heptane/toluene mixture and the data for the pure components *n*-heptane and toluene for $\phi = 1$ and 30 bar. The *n*-heptane data at 30 bar are measurements of Gauthier et al. [9] at 15–60 bar normalized to 30 bar [9] using the value of 1.64 for x suggested in that paper; these are in good agreement with the data of Ciezki and Adomeit [16] at pressures of 13 and 42 bar. The toluene experiments were conducted by Davidson et al. [17]. They are also normalized to 30 bar [17]. The ignition delay times of the *n*-heptane/toluene mixture, normalized to 30 bar by assuming the evaluated pressure exponent of 1.06, are in between the values of the pure substances, and the

n-heptane/toluene mixture shows a less pronounced NTC behavior in comparison to *n*-heptane. For the other pressures and equivalence-ratio conditions investigated in this study the comparison to the behavior of the pure substances is not possible because of the lack of data for toluene.

Further studies of toluene/*n*-heptane/isooctane/diisobutylene and isooctane/*n*-heptane/ethanol mixtures are in progress.

5. Conclusions

Ignition delay times of a toluene reference fuel containing 65% toluene and 35% *n*-heptane by liquid volume at room temperature were determined in a high-pressure shock tube at various equivalence ratios, pressures, and temperatures. The current data are, to our knowledge, the first for this mixture. They will help to improve the kinetic model for the toluene reference fuel (TRF) and to gain further understanding of fuel chemistry. This will allow a better understanding of the different autoignition behavior of the TRF and primary reference fuels under different conditions in internal combustion engines [5]. The experimental data from this work have been used to validate an improved kinetic model for the autoignition of *n*-heptane/toluene mixtures as reported in a companion paper [10].

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