



# Quantum yields of I(2 P 1/2) for CF3I, C2F5I, iC3F7I, nC3F7I, nC6F13I, and 1,2C2F4I2 at 308 and 248 nm

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# Quantum yields of $I({}^{2}P_{1/2})$ for $CF_{3}I$ , $C_{2}F_{5}I$ , *i*- $C_{3}F_{7}I$ , *n*- $C_{3}F_{7}I$ , *n*- $C_{6}F_{13}I$ , and 1,2- $C_{2}F_{4}I_{2}$ at 308 and 248 nm

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A new method is applied to measure the wavelength-resolved quantum yields of  $I({}^{2}P_{1/2})$  for six molecules relevant to the iodine  $I({}^{2}P_{1/2})$  atoms and accounts for the strong exciplex emission, also at 1.3  $\mu$  m, from the  $I({}^{2}P_{1/2})$ -iodide molecules. In addition, digital processing is used to correct for noise, quenching, and the detector rise time. These features avoided the shortcomings of previous measurements. The following molecules were investigated: CF<sub>3</sub>I, C<sub>2</sub>F<sub>5</sub>I, *i*-C<sub>3</sub>F<sub>7</sub>I, *n*-C<sub>3</sub>F<sub>7</sub>I, *n*-C<sub>6</sub>F<sub>13</sub>I, and 1,2-C<sub>2</sub>F<sub>4</sub>I<sub>2</sub>. All six iodides present high yields of  $I({}^{2}P_{1/2})$  at the pumping wavelengths of 308 and 248 nm used. The high  $I({}^{2}P_{1/2})$  yields for the molecules *n*-C<sub>6</sub>F<sub>13</sub>I and 1,2-C<sub>2</sub>F<sub>4</sub>I<sub>2</sub> are reported for the first time.

### I. INTRODUCTION

The high-power photodissociation atomic iodine laser at 1.315  $\mu$ m is an important tool for laser-plasma experiments.<sup>1</sup> The laser transition occurs between the metastable level I( ${}^{2}P_{1/2}$ ), hereafter I\*, and the ground state level I( ${}^{2}P_{3/2}$ ), hereafter I. Both levels are produced in the photodissociation act

$$RI + hv(UV) \begin{pmatrix} R + I^* \\ R + I \end{pmatrix}, \qquad (1)$$

where RI is an iodine bearing molecule, usually  $i-C_3F_7I$ . The relative amount of I\* atoms produced in Eq. (1), i.e., the *quantum yield*, depends on the pumping wavelength. The quantum yield is an important parameter in the evaluation of iodine laser media since it determines the upper bounds for the laser output power and efficiency.<sup>2</sup>

This work reports on accurate and wavelength-resolved measurements of the quantum yields of I\* for the molecules  $CF_3I$ ,  $C_2F_5I$ ,  $i-C_3F_7I$ ,  $n-C_3F_7I$ ,  $n-C_6F_{13}I$ , and 1,  $2-C_2F_4I_2$ . The last two molecules are new high-yield parent molecules.<sup>2</sup> The experimental method used was based on the measurement of the I\* concentration through the time-resolved infrared emission of the I\* atoms at 1.3  $\mu$ m and involved three special features. First, the data were corrected for the collisional enhancing of the 1.3  $\mu$ m emission caused by the exciplex emission of the I\*-iodide molecule.<sup>3</sup> Second, the fluorescence signal was digitally processed to account for noise, quenching and the detector rise time. Third, an excimer laser was used to provide short UV pulses (20 ns) at 308 or 248 nm, in order to photolyze the iodides under welldefined conditions. These three features avoided the shortcomings of previous measurements (see, e.g., Refs. 4, 5, and 6 and the critical review in Ref. 7). A complete list of earlier work on the alkyl iodides is given in Ref. 7.

#### **II. EXPERIMENTAL**

The experimental arrangement used to measure the fluorescence intensity as a function of the iodide pres-

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sure is shown in Fig. 1, without the gas handling system. Its three main parts are: the UV excimer laser, the RI cell with the 1.3  $\mu$ m detector, and the digital electronics.

The homemade excimer laser provided approximately 100 mJ of UV energy in 20 ns.<sup>2</sup> It was very well shielded to avoid EMI down to the microvolt level. The shot-to-shot energy reproducibility was 10%. The laser was employed to provide pump photons at 248 nm (KrF excimer) or 308 nm (XeCl excimer). These two wave-lengths correspond to good measurement points in the absorption spectra of the six RI, as can be seen in Figs. 2, 3, and in Ref. 7.

The construction of the fluorescence cell needed special care. First, the UV photons were observed to produce strong IR fluorescence from the first type of window material used, of Suprasil quartz. This problem was avoided with the use of  $CaF_2$  windows. Second, the UV radiation from the excimer laser also contained some superfluorescent atomic IR lines around 1.3  $\mu$ m that shadowed the weak fluorescence from the I\*. With the use of a scattering-free geometry this IR noise source was eliminated. Third, to prevent visible radia-



FIG. 1. Experimental arrangement to measure the I\* quantum yield at various pump wavelengths, without the gas-handling system.



FIG. 2. Absorption spectrum of  $n-C_6F_{13}I$ .

tion from reaching the Ge detector and to place it as close as possible to the observation volume (in order to avoid the use of a lens system), a Si plate (transmitting above 1.2  $\mu$ m) and the detector itself were placed in a small niche in the wall of the cell, perpendicular to the laser beam. Last, the cell was electrically insulated from the bench and from the gas-handling system. Before going to the digital oscilloscope, a ground-shielded amplifier (Tektronix 7A22 in a 7000 main frame) raised the signal level by a factor of 10 K. The amplifier was adjusted to cut-off frequencies below 0.1 Hz and above 0.5 MHz. The total rise time of the detector-amplifier system was limited by the amplifier at 2.5  $\mu$ s. This rise time was much faster than the quenching time of the I\* species, under the experimental conditions used.

The UV pulse energy (in the 100 mJ level) was measured by a calorimeter (Gentec PRJ-D) with a threedigit display. The RI pressure (in the 1 to 100 mbar range) was measured by a capacitive gauge (MKS Baratron PDR-C-2) with digital output, with an absolute error smaller than  $\pm 2\%$ . The amplified signal from the Ge detector was sampled by a digital oscilloscope (Tektronix 468) with a useful bandwidth of 10 MHz. The oscilloscope provided for enough voltage and time resolution. The digitized signal was transferred to a Commodore 3032 computer and processed. The computer program calculated the best fit of a simulated signal (see Sec. II) to the noisy signal.

The gas-handling apparatus included a turbo-pump (oil free) and two glass reservoirs to allow vacuum distillation of the iodide before each measurement. This procedure eliminated absorbed gases, such as oxygen, that could quench the I\*. Some small pieces of indium were placed inside the reservoirs to react with any free  $I_2$ , since  $I_2$  is a strong quencher of I\*. The whole gas system was pumped down to  $10^{-5}$  mbar before each measurement.

# II. QUANTUM YIELD OF I\*

When only dissociative states are involved, the quantum yield to produce  $I^*$  in Eq. (1) is given by

$$\phi(\lambda) = \frac{[\mathbf{I}^*]}{[\text{absorbed } h_{\mathcal{U}}]} = \frac{[\mathbf{I}^*]}{[\mathbf{I}^*] + [\mathbf{I}]} , \qquad (2)$$

where hv means the pump photon and [X] is the concentration of X in particles/cm<sup>3</sup>. The threshold quantum yield  $\phi_{th}$  to have population inversion depends on the degeneracies of the I\* and I levels and is  $\phi_{th} = 0.33$ . If the iodide medium is optically thin, the branching ratio to produce I\* in the photodissociation act is

$$b(\lambda) = \frac{\sigma^*(\lambda)}{\sigma_T(\lambda)} , \qquad (3)$$

where  $\sigma^*(\lambda)$  is the absorption cross section to produce I\*, and  $\sigma_T(\lambda)$  is the total absorption cross section. If the parent molecule loses only one iodine atom, the branching ratio of I\* is equal to the quantum yield defined in Eq. (2). Equation (3) is the operational definition of  $\phi(\lambda)$  to be used in this work.

#### A. The method

If the medium RI is optically thin and has a low degree of photodissociation, the following system of differential equations

$$\frac{d[\mathbf{I}^*]}{dt} = \sigma^* N P(t) \lambda / hc - [\mathbf{I}^*] / Q , \qquad (4)$$

$$\frac{dV}{dt} = -V/\tau + DA[I^*], \qquad (5)$$

gives the relation between V(t), the fluorescence signal measured by the detection system but without noise, and [I\*], the I\* concentration. The parameters above are: N, the RI concentration; P(t), the pump power flux at  $\lambda$ ; Q, the total quenching time constant of the I\* species;  $\tau$ , the rise time of the detection system; D, the detection efficiency of the 1.3  $\mu$ m radiation including electrical and optical factors, and A, the radiative transition rate at 1.3  $\mu$ m. Among the eight parameters in Eqs. (4) and (5), the parameters D and  $\tau$  are apparatus constants. D is unknown but  $\tau$  can easily be measured. In this work,  $\tau = 2.5 \ \mu$ s.

If  $Q \gg \tau$ , i.e., if the detection system is much faster than the quenching reactions, the above equations have



FIG. 3. Absorption spectrum of  $1, 2-C_2F_4I_2$ .

the approximate solution: [since P(t) is a temporal  $\delta$  function]

$$V(t) = \tau DA[I^*]_0 \exp(-t/Q) [1 - \exp(-t/\tau)], \qquad (6)$$

where  $[I^*]_0$  is the initial I\* concentration produced at the end of the UV pulse. The expression for  $[I^*]_0$  is

$$[I^*]_0 = \sigma^* N E \lambda / hc , \qquad (7)$$

where  $E = \int P(t) dt$  is the UV pump energy fluence. The detected signal also includes electrical and digitalization noise. Let W(t) = V(t) + X(t) represent the detected signal, where X(t) is the (small) noise part. In order to average out noise and to account for  $\tau$  and Q, W(t) can be written as

$$\ln\left[\frac{W(t)}{1-\exp(-t/\tau)}\right] = \ln(\tau DA [I^*]_0) - t/Q + \tilde{X}(t) \qquad (8)$$

from Eq. (6) where  $\tilde{X}(t)$  is the noise part transformed. The left-hand side includes only directly measured quantities. If X(t) = 0, Eq. (8) can be seen as a straight line with intercept  $\ln(\tau DA[I_{\delta}^{*}])$  and slope  $-Q^{-1}$ . In calculating the best fit of a straight line through the experimental time points as given by Eq. (8), the effect of noise can be reduced and the two quantities  $DA[I^{*}]_{0}$  and Q can be obtained. The energy-normalized number

$$S(N) = DA[I^*]_0 \tau hc/E , \qquad (9)$$

which is a function of the RI concentration, is the main experimental result to be used and will hereafter be designated simply by S. This number represents an estimate of the fluorescence signal at t = 20 ns (i.e., after the UV pulse), for unit UV energy. With Eq. (9), if the quantum yield  $\phi_1$  is known then  $\phi_2$  can be expressed by

$$\phi_2 = \frac{S_2/N_2}{S_1/N_1} \quad \frac{A_1\sigma_{T1}}{A_2\sigma_{T2}} \phi_1 , \qquad (10)$$

as a function of well-defined parameters.

#### B. Correction for the exciplex emission

It is a common assumption to make  $A_1 = A_2$  [see Eq. (10)]. This, however, is not correct. As recently reported,<sup>3</sup> the radiative transition rate A at 1.3  $\mu$ m depends both on the type of the RI molecule and on its pressure through the equation

$$A = (8 + kN)s^{-1}, (11)$$

where k is called the exciplex emission rate. Equation (11) describes the increase of the radiative transition rate of the I\* atom (equal to 8 s<sup>-1</sup>) by the exciplex emission of the RI · I\* molecule. Since the exciplex emission lies very close to the atomic emission line (within 2 nm), both emissions cannot be spectrally separated with the interference filters available (the minimum half-width is 10 nm). Furthermore, for  $k = 6 \times 10^{-18}$  molecule<sup>-1</sup> cm<sup>3</sup> s<sup>-1</sup> (the rate for  $i-C_3F_7I^3$ ), a RI pressure of 10 mbar is already enough to increase A by 18% over the atomic value of 8 s<sup>-1</sup>. This means that the exciplex contribution to the signal measured at 1.3  $\mu$ m is considerable even at low pressures. As explained in, <sup>3</sup> the reason for this strong contribution is that, although the exciplex concentration is small, the exciplex transition is allowed whereas the



FIG. 4. Plot of S/N from Eq. (10), in arbitrary units, for two iodides  $RI_1$  and  $RI_2$  that have the same quantum yield of I\* but have different exciplex emission rates.

atomic iodine transition is not. The Einstein A coefficient of the exciplex transition is approximately  $10^6$  times larger<sup>8</sup> than the A coefficient of the atomic transition.

Since different RI have different exciplex emission rates<sup>3</sup> the relative exciplex effect must be considered when comparing  $S_1$  and  $S_2$  for two iodides, as in Eq. (10). To illustrate the point, Fig. 3 shows a plot of S(N)/N, from Eq. (9), for two iodides RI<sub>1</sub> and RI<sub>2</sub> that have the same quantum yield of I\*, but different exciplex rates. As is clear from Fig. 3, the fluorescence signal (proportional to S) can be stronger for RI<sub>1</sub> than for RI<sub>2</sub> because  $A_1 > A_2$  rather than  $\phi_1 > \phi_2$ .

How can the relative exciplex effect be accounted for in Eq. (10)? The answer can be motivated by Fig. 4. From the experimental points S(N) of Eq. (9) one can calculate the intercept of S(N) with the N = 0 axis by fitting a straight line through the points [S(N)/N, N]. This intercept is free from the exciplex contribution. Denoting this intercept by F, Eq. (10) changes to the following expression:

$$\phi_2 = \frac{F_2}{F_1} \frac{\sigma_{T1}}{\sigma_{T2}} \phi_1 .$$
 (12)

The quantum yield can be normalized<sup>9</sup> relative to  $\phi_R = 1$ , for any reference high-yield iodide, giving from Eq. (12)

$$\phi = \frac{\sigma_{TR}}{F_R} \quad \frac{F}{\sigma_T} \quad . \tag{13}$$

Equation (13) allows the measurement of low-error quantum yields.

#### IV. RESULTS

The compound  $n-C_3F_{1}I$  is known to have approximately unit quantum yield of I\*, anywhere in the first absorption band.<sup>10</sup> It was therefore chosen to be the reference RI with  $\phi_{R} = 1$ . Note that this is only a convenient normalization procedure for the wavelength-dependent data. Following the procedure of the former section, the S(N) data for the six iodides were measured, at 308 and 248 nm. Figure 5 shows the S(N) data at 308 nm for  $n-C_3F_{1}I$ . The numbers over the points signify the number of plotted points. The energy density of the UV laser beam was approximately 30 mJ/cm<sup>2</sup>. This is not expected to cause any pyrolysis effects since the iodide was always in the optically thin regime and with a low



FIG. 5. Graph of the experimental data S(N)/N for  $n-C_3F_7I$  at 308 nm. The number over the points signifies the number of plotted points. The vertical axis is in arbitrary units.

degree of photodissociation.

The S data were used to calculate the intercept F, as in Sec. II. The experimental results for the six RI are summarized in Table I for  $\lambda = 308$  nm and in Table II for  $\lambda = 248$  nm. The quantum yields are calculated by means of Eq. (13).

For the sake of comparison, Table III presents the broadband data of<sup>4</sup> and the data at 266 nm of<sup>5</sup> (but uncorrected for the exciplex emission), for four iodides. The data of Refs. 4 and 5 were normalized relative to  $n-C_3F_7I$ , i.e.,  $\phi_R = 1$ .

The quenching rates of I\* by  $n-C_6F_{13}I$  and  $1, 2-C_2F_4I_4$ were unknown. Although the iodides were not completely free of all quenching impurities, good estimates of the rates could be obtained by the present method. The quenching rates were derived from the Stern-Volmer plot of the decay time 1/Q, from Eq. (9) (i.e., from the slope of the 1/Q points vs N). The rates are  $10^{-17}$  molecule<sup>-1</sup> cm<sup>3</sup> s<sup>-1</sup> for  $n-C_6F_{13}I$  and  $10^{-16}$ molecule<sup>-1</sup> cm<sup>3</sup> s<sup>-1</sup> for  $1, 2-C_2F_4I_2$ . These rates are low enough to allow the iodides to be used as iodine laser media. The exciplex emission rates k of Eq. (13) were also measured. They were obtained from the slope of the (S/N, N) plot. The rates for CF<sub>3</sub>I, C<sub>2</sub>F<sub>5</sub>I,  $i-C_3F_7I$  and  $n-C_3F_7I$  have already been reported.<sup>3</sup> The rates for

TABLE I. Experimental results for the six RI at 308 nm. The number F is the limit of S/N, from Eq. (19), for zero RI pressure. The quantum yield  $\phi$  was calculated by means of Eq. (15).

RI	$\sigma_T$ $10^{19} \text{ cm}^2$	$F/\sigma_T$	φ
	(±5%)	(±5%)	
$n-C_3F_7I$	1.20	1.92	= 1
$1, 2 - C_2 F_4 I_2$	1.01	$1.7 \pm 0.2$	$0.9 \pm 0.1$
CF <sub>3</sub> I	0.344	1.60	$0.83 \pm 0.05$
$i-C_3F_7I$	1.53	1,93	$1.01 \pm 0.05$
$C_2F_5I$	0.584	1.99	$1.04 \pm 0.05$
$n-C_6F_{13}I$	1.76	1.85	$0.96 \pm 0.05$

TABLE II. The same as in Table I, but at 248 nm.

RI	$\sigma_T$	$F/\sigma_T$	$\phi$
	(± 5%)	(±5%)	
$n-C_3F_7I$	3.16	1.34	= 1
$1, 2-C_2F_4I_2$	9,66	$0.9\pm0.1$	$0.6 \pm 0.1$
CF <sub>3</sub> I	2,60	1.01	$0.75 \pm 0.05$
$i-C_3F_7I$	1.93	1.24	$0.93 \pm 0.05$
$C_2F_5I$	3.29	1.35	$1.01 \pm 0.05$
$n-C_6F_{13}I$	3.47	1.38	$1.03 \pm 0.05$

 $n-C_6F_{13}I$  and  $1, 2-C_2F_4I_2$  are  $(8 \pm 2) \times 10^{-18}$  and  $(1 \pm 0.2) \times 10^{-17}$  molecule<sup>-1</sup> cm<sup>3</sup> s<sup>-1</sup>, respectively. Since the exciplex emission represents a quenching channel for the I\* atoms, <sup>3</sup> the exciplex emission rate is actually part of the total quenching rate. For the six iodides studied, the exciplex emission rate accounts for ~ 10% of the total quenching rate.

The measurements reported here show that the  $C_n F_{2n+1}I$  iodides have almost identical high quantum yields at different points within the first absorption band, contrary to Ref. 11.

The di-iodide 1,  $2-C_2F_4I_2$  does not belong to the same group as the other iodides studied here. As stated in,<sup>2</sup> the di-iodide was expected to present a high yield of I\* because of its structural formula

$$\begin{array}{cccccc}
F & F \\
| & | \\
I - C - C - I \\
| & | \\
F & F
\end{array}$$
(16)

in which the  $F_2C-CF_2$  group acts as a buffer chain and isolates both I atoms. This means that, regarding photodissociation along a R-I bond, the di-iodide should behave almost like two independent  $CF_3I$  molecules. Indeed, we observe its quantum yields to be similar to the yields for  $CF_3I$ .

#### V. SUMMARY

This paper reports on wavelength-resolved measurements of the quantum yields of I\* for six molecules relevant to the iodine photodissociation laser, using IR fluorescence. The method employed accounted for the strong exciplex emission of the I\*-iodide molecules.

TABLE III. Quantum yields of I\* from Ref. 4 (broadband) and Ref. 5 (at 266 nm but uncorrected for the exciplex emission). The numbers are normalized relative to  $\phi = 1$  for  $n-C_3F_7I$ .

RI	$\phi$ (broadband)	φ (266 nm)
CF <sub>3</sub> I	$0.92 \pm 0.03$	$0.93 \pm 0.005$
C <sub>2</sub> F <sub>5</sub> I	>0.99	•••
$i-C_3F_7I$	$0.91 \pm 0.02$	• • •
$n-C_3F_7I$	= 1	= 1

#### J. Chem. Phys., Vol. 79, No. 1, 1 July 1983

In addition, digital detection and processing was used to correct for noise, quenching and the rise time of the detection system. The use of a short pulse UV excimer laser also simplified the data analysis. The data were measured at 308 and 248 nm, which correspond to good measurement points in the first absorption band of the iodides. The compounds  $CF_3I$ ,  $C_2F_5I$ ,  $i-C_3F_7I$ ,  $n-C_3F_7I$ , and  $n-C_6F_{13}I$  belong to the same  $C_nF_{2m+1}I$  group and show similar absorption features in the first band. Their I\* quantum yields, within this band, are of the order of unity. The di-iodide  $1, 2-C_2F_4I_2$  also shows high yields within its first absorption band. The compounds  $n-C_6F_{13}I$  and  $1, 2-C_2F_4I_2$  are new high yield molecules for the iodine laser.

The present results avoided the shortcomings of previous measurements and provide a general method to measure the quantum yields of other iodides, regardless of quenching and exciplex emission of the I\* atom with the parent molecule.

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- <sup>2</sup>E. Gerck, MPQ-Report No. 58. Garching, Federal Republic of Germany (1982).
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- <sup>4</sup>Since the quantum yield of I\* depends on the pumping wave-

length, broadband data are not enough to characterize the pump bands of different iodides. See T. Donohue and J. R. Wiesenfeld, J. Chem. Phys. 53, 3130 (1975), for an example of broadband data.

- <sup>5</sup>A buffer gas or the parent molecule itself can considerably enhance the infrared fluorescence at 1.3  $\mu$ m by exciplex emission (Ref. 3). Previous measurements did not take this emission into account, as in L. S. Ershov, V. Yu. Zalesskii, and V. N. Sokolov, Sov. J. Quantum Electron. 8, 494 (1978).
- <sup>6</sup>If flashlamps are used to photolyze the medium, the long duration of the pump pulse ( $\mu$ s) makes the generation (pumping) and disappearance (quenching and hot recombination) of the I\* atoms occur simultaneously. The respective positive and negative contributions to the I\* concentration cannot be separated from each other. The use of a cw lamp to dissociate the iodide causes also the same problems but on a worse scale. On the other hand, the use of a short pulse (ns) to photolyze the medium makes the time evolution of the I\* to be well defined. For long pumping-time data see Ref. 4 and A. M. Pravilov, A. S. Kozlov, and F. I. Vilesov, Sov. J. Quantum Electron. 8, 666 (1978).
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- <sup>9</sup>This is a standard procedure to obtain absolute quantum yields from relative data. For an example with I\*, see H. Hofmann and S. R. Leone, J. Chem. Phys. **69**, 3819 (1978). The authors, however, did not take into account the exciplex emission and the (probable) reaction  $\text{HgI}_2 + hv(270 \text{ nm}) \rightarrow \text{Hg}$ + I\* + I. This reaction reduces the quantum yield by a factor of 2 (because  $\phi$  is defined as the ratio of [I\*] to [I\*] + [I]). For a discussion on the quantum yield of I\* for  $\text{HgI}_2$  see Ref. 2, p. 41.
- <sup>10</sup>The broadband data of Donohue and Wiesenfeld (Ref. 4) support  $\phi \cong 1$  for  $n-C_3F_7I$ , for any wavelength within its first absorption band.
- <sup>11</sup>A. M. Pravilov, Sov. J. Quantum Electron. 11, 847 (1981).