

CW mode-locked dye lasers for ultra fast spectroscopic studies

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Continuously working dye lasers have produced pulses as short as 0.3 ps. The properties of passively and actively mode-locked dye lasers are described and compared. Methods of measuring pulse duration and applications of these lasers to photophysics and photobiology are discussed.

The continuous mode-locked dye laser is beginning to assume a role of considerable importance as a tool for the study of ultra fast photochemical and photophysical processes. The purpose of this article is to describe methods of generation of ultra short pulses from argon or krypton ion laser pumped jet-stream dye lasers, some of the pulses properties, and to give examples of applications that they have so far found in this rapidly expanding field. The attractiveness of the cw mode-locked dye laser stems from three basic properties:

1. Extremely short pulse duration. Pulses of 0.3 ps have been obtained.^{1, 2}
2. Wide wavelength tunability. For 10 ps long pulses a wavelength of 530 nm to 900 nm^{3, 4} for the fundamental and of 265 nm to 450 nm for the second harmonic³ have been reported.
3. Repetition rates from 200 MHz down to Hertz can be obtained.

The combination of high power ion lasers to pump jet-stream dye lasers is common to all current experimental set ups. However, two distinct methods have been used to achieve mode locking in the dye laser, firstly passive mode locking using a saturable absorber^{1, 2} and secondly forced mode locking using an actively mode-locked ion laser as the pump. The latter is called synchronous pumping.^{3, 4, 5} Before describing the two systems in more detail a brief discussion of the principle of mode locking is useful.⁶ In an inhomogeneously broadened laser a number of longitudinal cavity modes will normally oscillate with random phase and amplitudes. If the phase and amplitudes of these longitudinal modes are fixed with respect to each other the laser is said to be 'mode locked' and the laser output consists of a train of pulses separated by the cavity round trip time.⁶ The duration of the pulses depends on the number of modes that can be locked together — the larger the bandwidth for gain the shorter the pulse. The final limit is imposed by the uncertainty principle. Mode locking is normally achieved by modulating the loss (or gain) of the laser at the frequency equal to the inverse of the round trip time for the optical

cavity. This discriminates against all mode combinations except the one in which all modes have the same phase, as only in this case is the optical electric field concentrated in one region of space. In this case the 'pulse' is able to pass through this loss element during the 'open' period, or arrive in the gain medium at the peak of the gain profile.

Passively mode-locked systems

In passive mode locking a continuous argon ion pump laser is used. Mode locking is achieved by means of a saturable absorber inside the dye laser cavity either in a separate jet stream,¹ in contact with the 100% reflector² or even flowing in the same jet stream as the lasing dye.⁷ In rhodamine 6G dye lasers the saturable absorber used is the cyanine dye DODCI. The effect of the saturable absorber is to discriminate in favour of mode combinations where a definite phase and amplitude relationship is maintained because of DODCI's non-linear absorption coefficient at high light intensities (Fig. 1). For sufficiently high incident intensity the absorber becomes bleached (transparent) thus automatically synchronizing the minimum cavity loss with the pulse repetition frequency. Repeated passage of a pulse through the non-linear absorber will also result in pulse shortening since the low intensity wings of the pulse will be clipped off on each traversal of the absorber solution.

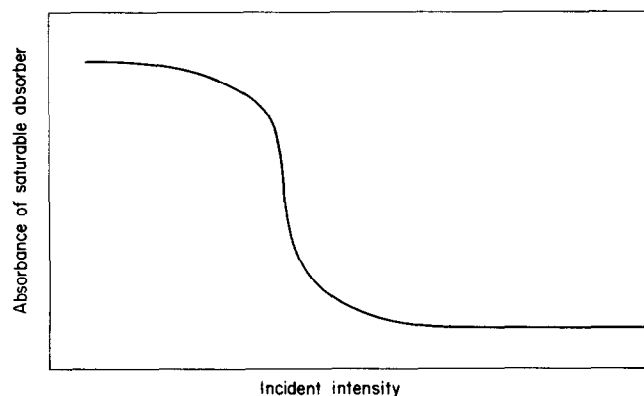


Fig. 1 Schematic diagram of absorbance of a mode-locking dye versus incident light intensity

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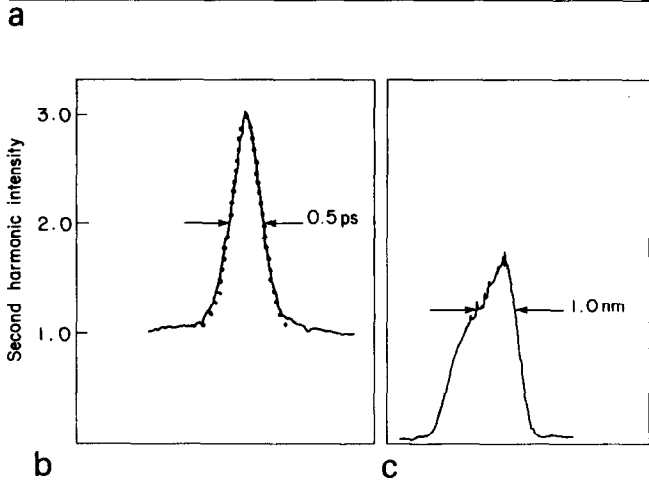
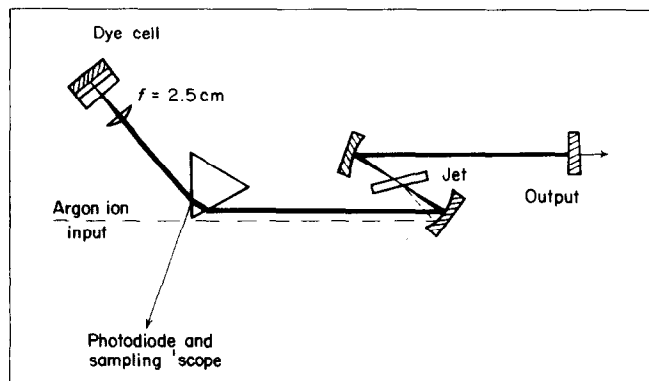


Fig. 2 From reference 9. a — Optical set-up of a passively mode-locked jet stream dye laser; b — temporal properties; c — spectral properties of the pulse from the laser in 2a

Picosecond pulses were first obtained from passively mode-locked cw dye lasers in 1974.⁸ At least two groups have subsequently obtained pulses of 0.3 ps (300 fs) duration, the shortest light pulses so far produced by any laser. Figure 2 shows the optical set up and pulse temporal and spectral properties obtained by Ruddock and Bradley at Imperial College.² Pulses of similar duration had previously been obtained by Shank and Ippen at the Bell Telephone Laboratories¹ using the saturable absorber in a second jet stream. The shortest pulses obtained by both groups are transform limited.

The wavelength tunability of a passively mode-locked laser is limited since accurate matching of dye lasing with the absorption spectrum of the saturable absorber is required. For the rhodamine 6G-DODCI system a tuning range of 598 nm to 615 nm is possible.² By using different combinations of laser and absorber dyes Bradley and coworkers⁹ have obtained subpicosecond pulses from 580 nm to 615 nm and ≤ 5 ps pulses from 580 nm to 630 nm.

Synchronously pumped systems

In the synchronous pumping technique an actively mode-locked argon (or krypton) ion laser producing ≤ 200 ps long pulses is used as an excitation source. The cavity length of a jetstream dye laser is matched with that of the pump laser. Thus the gain of the dye laser is pulsed synchronously with its cavity round trip time. The result is a train of ultra short pulses. Systems operating on this principle are in

operation in a number of laboratories. The experimental set up used in the authors laboratory is shown in Fig. 3.

The mode-locked ion lasers

Reliable operation of a synchronously pumped dye laser depends on the high stability of the mode-locked ion laser in terms of the pulse shape, duration, and repetition frequency. In the ion laser mode locking is achieved by Bragg diffraction of the laser light by an acoustic standing wave in a quartz crystal placed inside the laser cavity. The acoustic wave is generated by a stable rf source (power ≈ 1 W) acting on a transducer. The frequency of the acoustic standing wave is matched to half the round trip frequency of the laser cavity since diffraction occurs in both halves of the acoustic wave cycle. Thus the loss of the cavity is modulated at the round trip frequency and mode locking results.

If good mode locking is to be achieved a stable rf source (better than 1 part in 10^6 stability) operating close to but just below an acoustic resonance of the mode locking crystal must be used. The cavity length of the ion laser must then be adjusted (to within 100 μm) so that the inverse round trip time is equal to the modulation frequency.

The 514.5 nm pulse from our CR12 argon ion laser measured on a sampling oscilloscope is shown in Fig. 4. The pulses were detected by a Telefunken BPW 28A photodiode in a specially designed holder¹⁰ and a Tektronix S6 sampling head. After deconvolution of the photodiode and oscilloscope response functions (200 ps) the average pulse width of the argon laser is 80–100 ps. The response function of the photodiode and oscilloscope was found by monitoring the 10 ps pulses from the dye laser.

The mode-locked dye laser

By simply removing the output mirror and extending the cavity it is simple to adopt a conventional dye laser for synchronous pumping. Although the average power output is rather insensitive to exact matching of the two laser cavities, as Fig. 5 shows the pulse duration of the laser is extremely sensitive to the dye laser cavity length. The results of Fig. 5 were obtained with a CR590 dye laser with a three plate birefringent filter. The traces are the second order autocorrelation traces of the pulses recorded using a zero-background second harmonic generation technique which will be described in detail in the next section. Assuming the pulses to be exponential/ sech^2 ^{12, 13} the shortest pulse in Fig. 5 has a fwhm of 6.4 ps which is close to transform limited for the laser bandwidth. The shortest pulse is obtained just on the short side ($\sim 10 \mu\text{m}$) of the cavity length which gives maximum second harmonic intensity. The pulses have a smooth envelope implying that the laser is well mode-locked. As Fig. 5 demonstrates, correct cavity matching to $\sim 10 \mu\text{m}$ is essential for the shortest pulses and attention must be paid to the thermal stability of the dye laser cavity length particularly if a steel optical table or bench is used. With rhodamine 6G as the lasing dye and 1.1 W average pump power we obtain ~ 400 mW average power from the dye laser at 590 nm. The tuning range is from 570 nm to 645 nm. No adjustment of the dye laser is necessary to obtain good mode locking over this range. The ease of tuning and reliability of operation constitute the major advantages of the synchronously pumped dye laser.

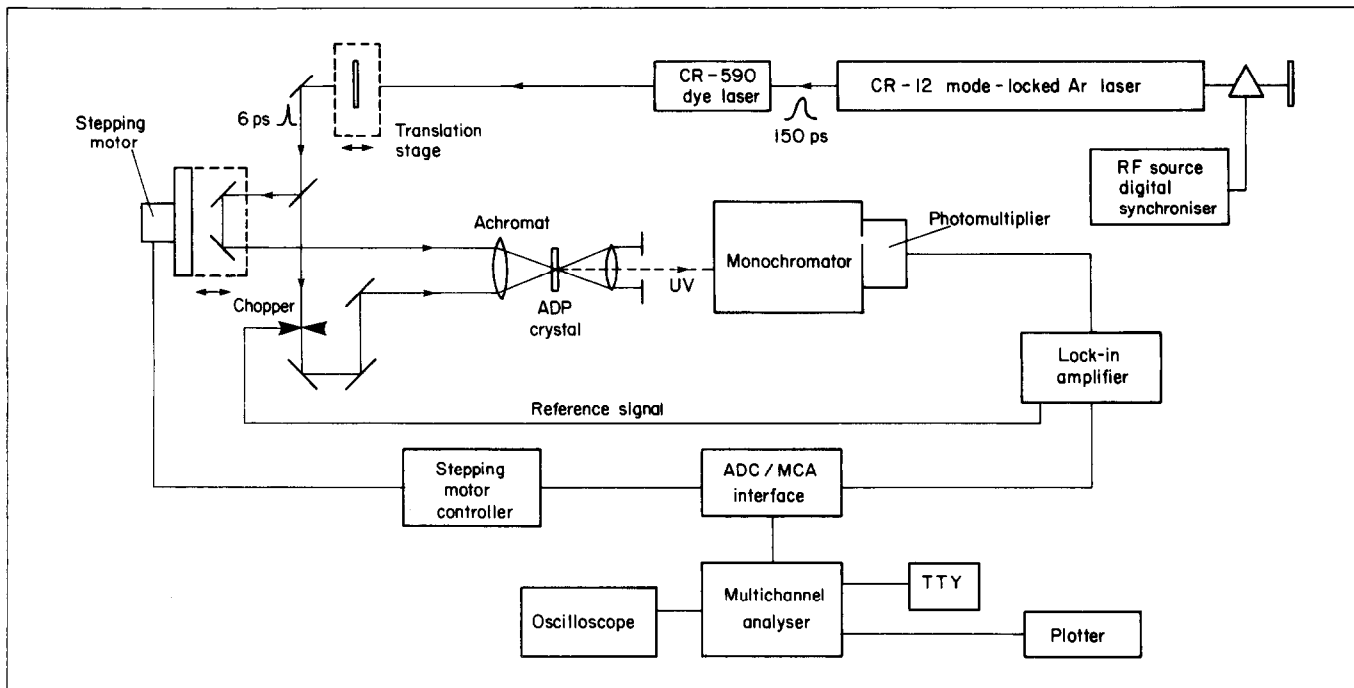


Fig. 3 Optical set-up used for synchronous pumping of a jet-stream dye laser and pulse duration measurements

So far, however, slightly shorter pulses can be obtained from passively mode-locked systems (0.3 ps) but the gap is being rapidly narrowed. The shortest pulses obtained from a simple synchronously pumped system, that are well mode-locked, have a duration of between 1.7 ps and 2.2 ps.^{11-13, 27} Subpicosecond pulses have been obtained by the technique of double synchronous pumping.¹³ Here the output of a synchronously pumped dye laser is used to synchronously pump a second dye laser. The shortest pulse obtained had a duration of 0.7 ps with the autocorrelation best fitted by assuming the pulse shape to be asymmetric with exponential wings. The subpicosecond pulses were obtained over a tuning range of approximately $2 \mu\text{m}$ requiring stability of better than 1 part in 10^6 on the rf source and the final dye laser cavity length.¹³

In principle the range of wavelengths that can be obtained from synchronously pumped systems is enormous. The possibility of mode locking other argon and krypton lines

and synchronously pumping dyes in the blue and near infra-red region has only just begun to be investigated. All the visible argon lines have been mode locked.¹⁰ A mode-locked krypton laser has been used to pump a range of dyes emitting in the 685-900 nm region.⁴ In our opinion the versatility, reliability and comparative ease of operation will enable the synchronously pumped dye laser to become a standard tool for the study of rapid processes in physical science.

Techniques for measurement and their application

Any discussion of picosecond lasers inevitably involves a discussion of the methods used to characterize the laser pulses themselves and to follow events initiated by the laser pulses. Examples of applications of the techniques are given in each section.

Delay line techniques

Laser pulse measurements

The commonest method used to measure the temporal characteristics of the laser pulses described in the previous section is a correlation technique shown schematically in Fig. 6. The pulse is divided into two replicas by means of a beam splitter. The two replica pulses traverse different paths before being recombined in a frequency doubling crystal. The path length of one arm can be varied by driving the two mirrors with a stepping motor. The double reflections in the two arms are designed to prevent optical feedback into the laser cavity. If the two beams enter the frequency doubling crystal colinearly then frequency doubling will be observed for any length of the variable arm. However, when the two pulses are coincident in time a threefold enhancement of the second harmonic intensity will be observed since second harmonic intensity is proportional to the square of the incident intensity. The technique shown in Fig. 6, on the other hand, uses a zero background technique due to Shank and Ippen¹ where the

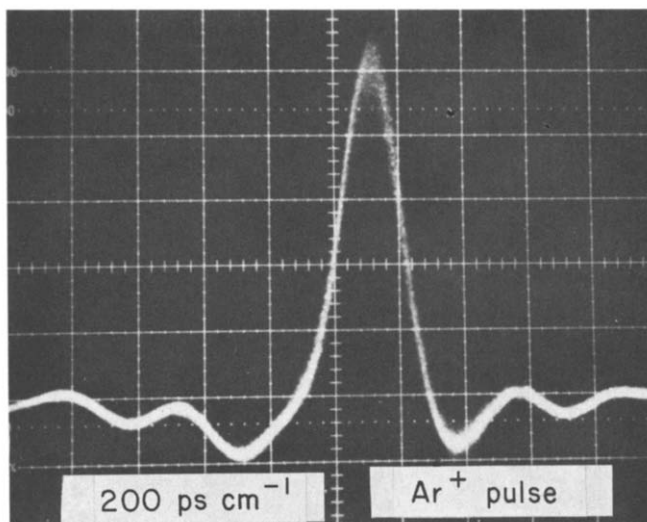


Fig. 4 Sampling oscilloscope trace of mode-locked argon ion laser (200 ps per division)

two pulses enter the crystal non-collinearly and the crystal is set for phase matching at an angle bisecting the two beams. The second harmonic is produced only when both pulses are present in the crystal. The results of Fig. 5 were obtained in this way using a 0.75 mm thick ADP crystal. The function measured by this technique is not the pulse itself

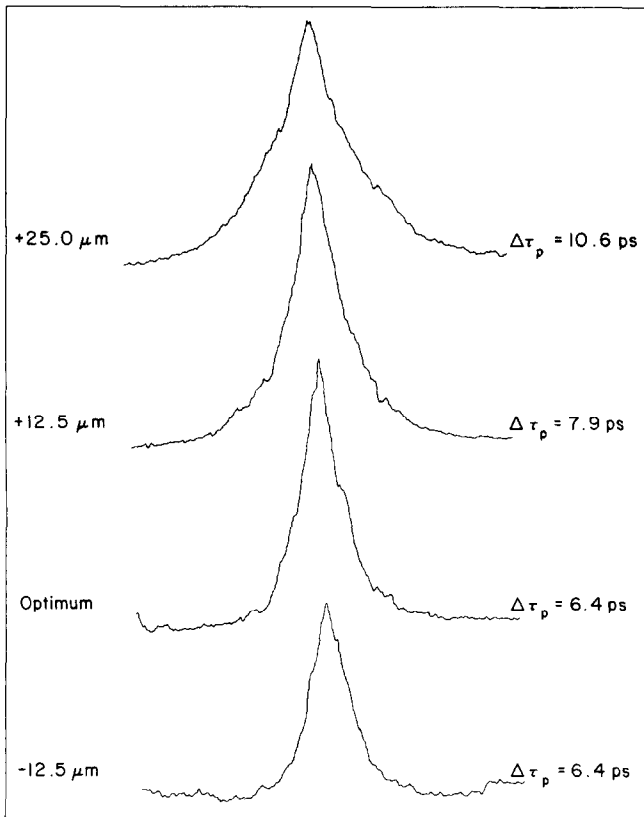


Fig. 5 Sequence of laser pulse autocorrelation traces recorded as a function of dye laser cavity length for a synchronously pumped dye laser. The marked fwhm are those for a Gaussian laser pulse

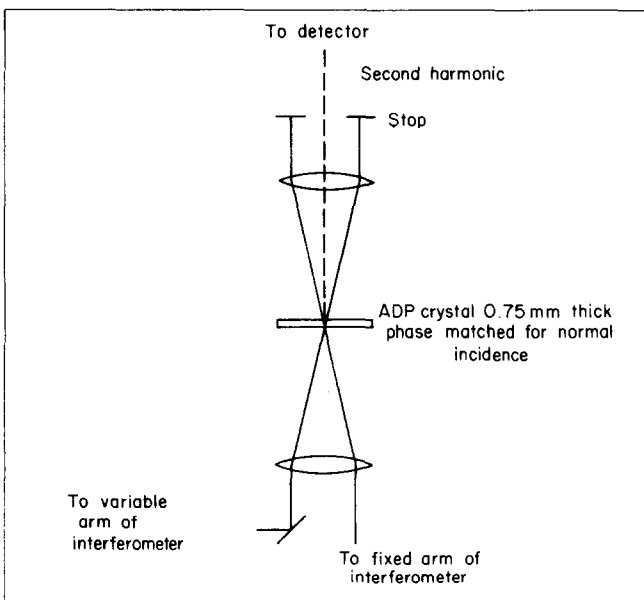


Fig. 6 Schematic of experimental arrangement for zero background second harmonic autocorrelation measurements of laser

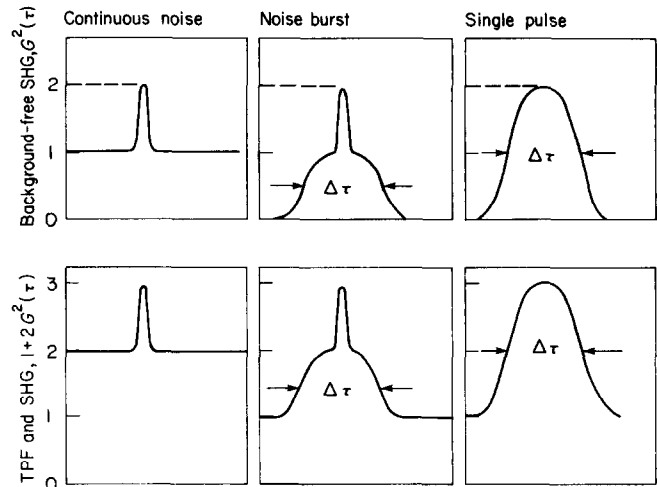


Fig. 7 Forms of autocorrelation trace expected for continuous noise, noise burst (partially mode-locked laser) and single pulse for both normal and zero background SHG measurements

$I(t)$ but its second order autocorrelation function $G^2(\tau)$ where:

$$G^2(\tau) = \langle I(t)I(t+\tau) \rangle / \langle I^2(t) \rangle$$

Here the brackets indicate averaging over a sufficient time period. To obtain the precise pulse duration from the fwhm of $G^2(\tau)$, $(\Delta\tau)$ some form for the shape of $I(t)$ must be assumed. Table 1 gives the ratio $\Delta\tau/\Delta t$ for three different pulse shapes. The durations in Fig. 5 were computed assuming combined exponential /sech² pulseshapes.^{12, 13} ($\Delta\tau/\Delta t = 1.8$) A good deal of care is required in interpretation of autocorrelation traces since even continuous noise will give a spike at zero delay of the interferometer. An excellent discussion has been given by Shank and Ippen¹⁴ and by Bradley and New.¹⁵ Figure 7 gives the form of $G^2(\tau)$ expected for continuous noise, a noise burst (partially mode-locked laser) and a single pulse for both zero background and normal second harmonic generation techniques.¹⁴ The results for normal SHG also apply to two photon fluorescence.

We prefer the zero background technique since intensity in the wings of the pulse is more easily detected, and interference due to the fringes of the interferometer essentially eliminated. For continuously operating lasers the technique of Fig. 6 has the great advantage that the signal can be scanned repeatedly to improve the signal to noise ratio. (Fig. 8 shows digitized traces using 2 plate and 3 plate birefringent filters in the synchronously pumped laser.) The time resolution of the correlation technique is essentially limited by the laser pulse duration, although for very short pulses

Table 1. Pulse duration from autocorrelation width for different pulse shapes. Δt is the full width at half maximum (fwhm) of the pulse $I(t)$ and $\Delta\tau$ is the fwhm of the autocorrelation trace $G(\tau)$ as defined in Fig. 7

$I(t)$	$\Delta\tau/\Delta t$
Gaussian	1.41
Sech ²	1.55
Single sided exponential	2

(≤ 1 ps) care must be taken to use a crystal sufficiently thin that group dispersion between fundamental and harmonic wavelengths is not significant. Figure 9 shows semi-log plots of two autocorrelation traces recorded with the zero background technique. In this experiment the data were recorded digitally by interfacing a voltage to frequency converter between the lock-in amplifier and a multichannel analyser. The second harmonic signal is thus digitized and averaged over the dwell period for any given position of the stepping motor driven delay line. The improvement in dynamic range and signal to noise for Fig. 5 is obvious. Both autocorrelation traces fall off exponentially at first but in the 10%-1% region systematically fall away slightly more rapidly than exponentially. The wider trace corresponds to a cavity $12.5 \mu\text{m}$ longer than the narrower (optimum) trace. The simple function for the actual pulse shape which best fits the measured autocorrelation traces is a single sided exponential.

For this function the fwhm of the laser pulse is $1/2$ that of the $G^2(\tau)$ giving ~ 7 ps for the shorter pulse in Fig. 9.

If instead of two identical pulses, one ultra short pulse, and a second pulse which may be a longer laser pulse or fluorescent light from a sample excited by the laser, are mixed in the nonlinear crystal then the cross correlation of the two pulses gives direct information on the shape of the longer pulse. The intensity of the sum frequency produced is simply proportional to the product of the two incident intensities.⁶ In an elegant example of the technique the frequency sweep in the pulses from a passively mode-locked cw dye laser was studied.^{1, 11} It was found that the laser pulses had a positive chirp, ie lower frequencies are ahead of higher frequencies in the pulse. Such a positive chirp can arise simply from linear dispersion in the laser medium itself (wavelength dependent refractive index) or from

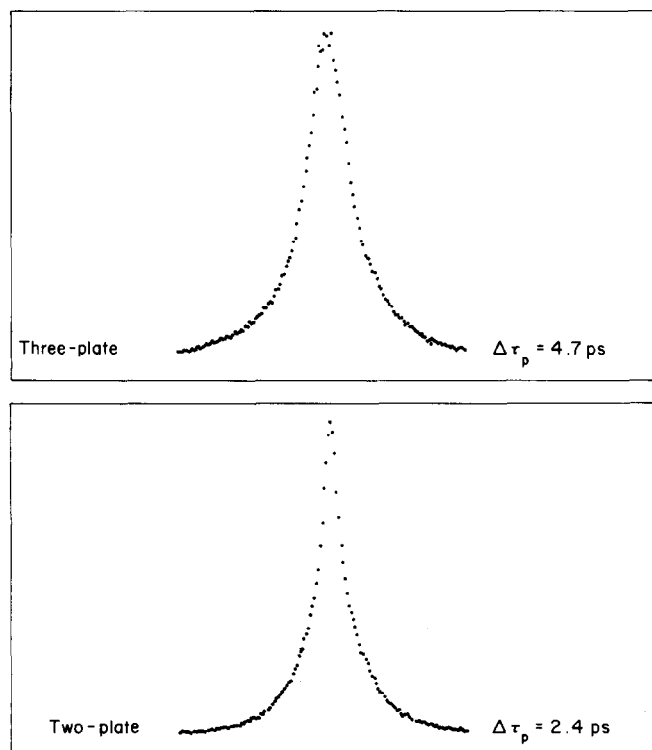


Fig. 8 Digital autocorrelation traces. Upper trace: 3 plate birefringent filter. Lower trace: two plate filter. Resolution $1/3$ ps per channel

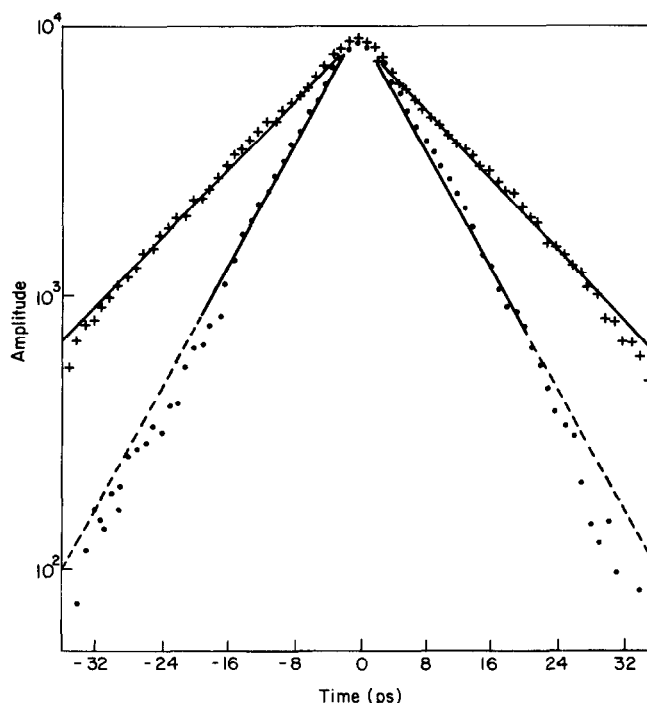


Fig. 9 Semilogarithmic plot of zero background SHG trace for two well mode-locked pulses, one at optimum cavity length and one with a cavity $25 \mu\text{m}$ longer than optimum

nonlinear effects such as self phase modulation.¹⁵ By using a grating pair, devised by Treacy,¹⁶ a positively chirped pulse can be compressed in time. The higher frequencies at the back of the pulse are made to catch up the lower frequencies at the front by virtue of their shorter path length through the device. The results of Shank and Ippen^{1, 14} are shown in Fig. 10. The extra shortening observed in Fig. 10 was obtained by spectral filtering and indicates that the frequency sweep was not entirely linear. The shortest pulses obtained in this way had durations of 0.3 ps. By using the compressed pulse the details of the original uncompressed pulse could be studied. Figure 10 shows the asymmetric envelope of the laser pulse, the temporal behaviour of the longer wavelength components and of the shorter wavelength components clearly demonstrates the frequency sweep.

Fluorescence measurements

The correlation technique is simply extended to measure fluorescence from a sample of chemical or biological interest by mixing a laser pulse with the laser excited fluorescence. As before the time resolution is limited by the laser pulse duration using a synchronously pumped dye laser as an excitation source. Mahr and coworkers at Cornell University excited fluorescence (at 780 nm) in bacteriorhodopsin in the plasma membrane of the bacteria *Halbacterium halobium*.¹⁷ The fluorescence light was mixed with a 590 nm probe pulse in a lithium iodate crystal and the sum frequency at 336 nm was measured as a function of delay between the exciting laser pulse and the probe pulse. The technique proved to be very sensitive since the fluorescence quantum yield was between $1.2-2.5 \times 10^{-4}$ and the fluorescence decay time of the bacteriorhodopsin was 15 ± 3 ps. The signal was enhanced by scanning and computer averaging of the sum frequency intensity over 10^9 laser pulses. The bacteria use a bacteriorhodopsin to pump protons across a membrane and so convert light into

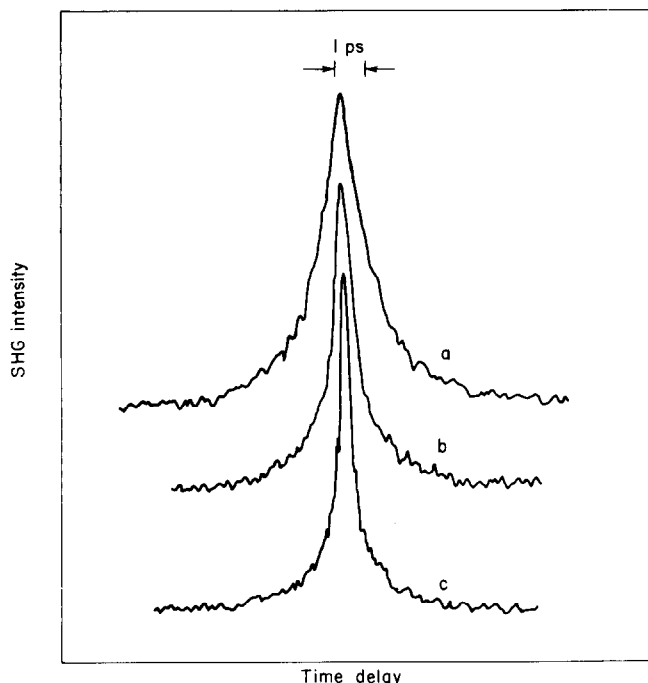


Fig. 10 Grating pair compression of passively mode-locked dye laser pulse.¹⁴ a — Laser output pulse; b — pulse after grating pair compression; c — pulse after compression and spectral filtering

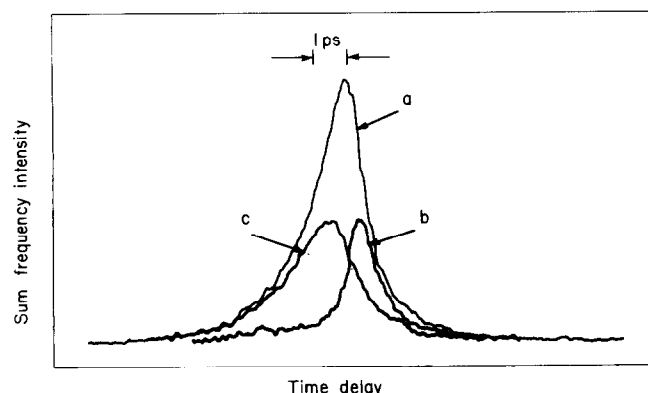


Fig. 11 Laser output pulse probed by compressed pulse of Fig. 10c. a — Pulse envelope; b — shorter wavelength components; c — longer wavelength components

chemical energy, consequently the decay pathways of the initially formed excited states following light absorption are of great interest and relevance for an understanding of the energy conversion process.

Transient absorption measurements

Measurements of transient light absorptions in samples after excitation can also be obtained by using a modification of the delay line technique in Fig. 6. Now the non-linear crystal is replaced by the sample. The absorption (or gain) of a weak probe pulse is measured as a function of delay between excitation and probe pulse. The probe wavelength may be the same wavelength as the excitation, or a harmonic or could originate from a second dye laser synchronously pumped by the same ion laser as the excitation laser but operating at a different wavelength.¹² Recently Jain and Heritage have shown that the pulses from two independent dye lasers synchronously pumped by the same argon laser are temporally synchronized with a jitter of less than 10 ps.¹²

This technique may well prove extremely useful in pico-second transient absorption spectroscopy.

Shank, Ippen and Teschke¹⁹ used the second harmonic of their dye laser to excite very short lived upper singlet states of large organic molecules in solution. The fundamental wavelength of the laser (615 nm) was then used to probe the excited state absorptions produced. In the aromatic hydrocarbon coronene the initially excited state is the third excited singlet state, S_3 . The absorption of the probe pulse (Fig. 12a) is continually modified as the initially formed state relaxes to give the lowest excited singlet state, S_1 . Relaxation is complete in about 4 ps as is shown in Fig. 12b where the absorption due to the relaxed S_1 state (constant at long times — the lifetime of S_1 is several nanoseconds) has been removed from the curve in Fig. 12a. In similar experiments on rhodamine 6G and rhodamine B the same workers found that upper state relaxation was complete within their time resolution of 2×10^{-13} s and so even more rapid techniques will be required to study these processes.

Streak camera detection

Normally the streak camera²⁰ operates on a single shot principle and as such has limited use for applications with continuous lasers. However a repetitively scanning camera (Synchroscan) synchronized to a cw mode-locked laser is being developed by researchers at Imperial College. Currently time resolution is ~ 25 ps and prospects for increases in resolution seem good.²¹ Thus the synchroscan camera is equivalent to a photomultiplier oscilloscope combination with a time resolution of a few tens of picoseconds!

Single photon counting

Time resolved single photon counting is a widely used technique for the measurement of fluorescence lifetimes of excited molecules or atoms.²² Conventionally a weak

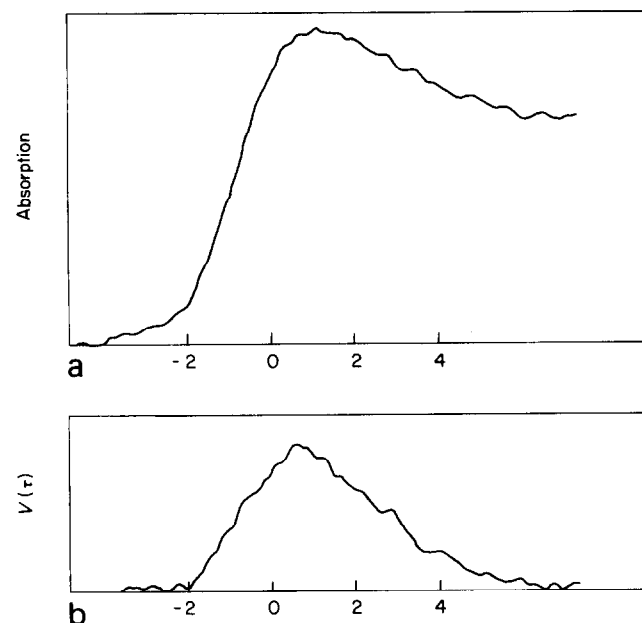


Fig. 12 Excited singlet state absorption in coronene. a — Absorption of probe pulse with time; b — relaxation of upper excited singlet state obtained from a

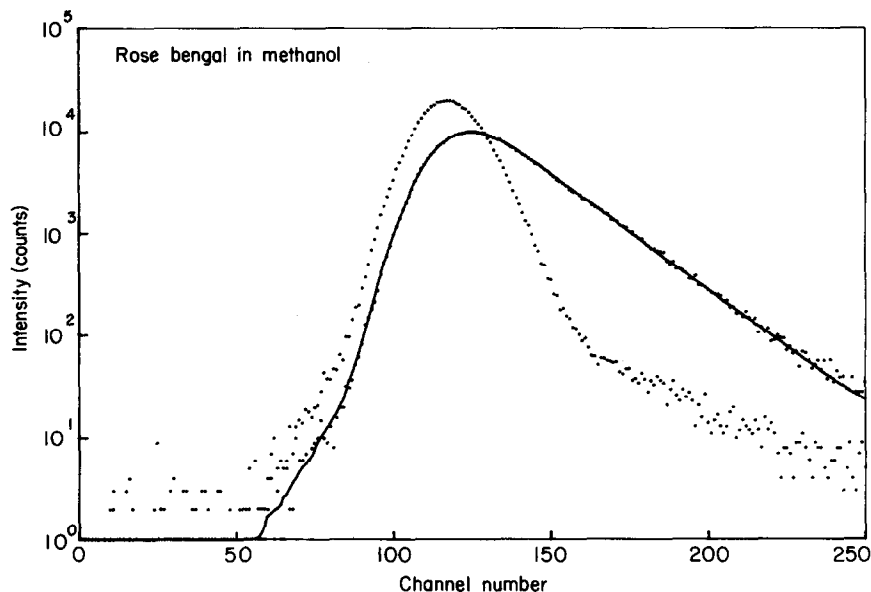


Fig. 13 Time resolved fluorescence decay of rose bengal in methanol

hydrogen discharge lamp with a duration of 2-3 ns is used as an excitation source. By using the short pulse from a cw mode-locked dye laser the technique can be readily extended to measure decay times as short as 100 ps with high precision. Figure 13 shows the fluorescence decay of the dye molecule rose bengal in methanol. The calculated curve is for the convolution of the measured instrument response function and an exponential decay of 597 ps. The agreement with the experimental curve is excellent and the decay time is in good agreement with the previously measured but less precise value of 655 ± 85 ps obtained using a picosecond streak camera.²³

The limiting factor for time resolution of the photon counting technique when a short essentially jitter free excitation pulse is used is the spread of transit times for the photoelectrons in the photomultiplier used to detect the fluorescence.²⁴ Jitter in the electronics (discriminators and time to amplitude converter) is much smaller (< 10 ps) than the photomultiplier transit time spread. In our instrument the measured instrument response function has a halfwidth of 600 ps using a 56 tuvp photomultiplier. With modern crossed field photomultipliers the transit time jitter can be considerably reduced and instrument response functions with widths of ~ 100 ps should be possible in the near future. These tubes should allow reliable deconvolution for fluorescence decay of a few tens of picoseconds and complete the overlap in time ranges of single photon counting and the upconversion technique already described.

The repetition frequency of a cw mode-locked dye laser is often too high to allow complete recovery of the sample between laser pulses. To overcome this problem the repetition rate of the dye laser can be reduced by means of a low voltage Pockels cell between a pair of polarizers. By using count-down logic the frequency of the laser can be reduced by tens to thousands of times with a contrast ratio of $> 1000 : 1$ between selected and rejected pulses. A related technique is that of cavity dumping²⁸ where the output of the dye laser is acousto-optically switched out at the required repetition frequency (up to 2 MHz in a typical system). For low gain lasers cavity dumping can also significantly increase the output energy in the pulse.¹

The ability to detect single photons from a fluorescing sample and the averaging process inherent in the technique means that very low excitation intensities (eg $< 10^{10}$ photons per pulse) may be used. This is important in, for example, photosynthetic systems and in molecular crystals where lifetime shortening caused by excitation annihilation have been observed in experiments with mode-locked solid state lasers where the data must be collected in a single laser shot.^{25, 26} Reducing the intensity to a sufficiently low level usually results in a poor signal to noise ratio and small dynamic range in single shot experiments. No annihilation effects are observed using the cw mode-locked dye laser as the excitation source and using photon counting one can routinely obtain four decades of decay information. A typical result with dark adapted chloroplasts²⁹ is shown in Fig. 14.

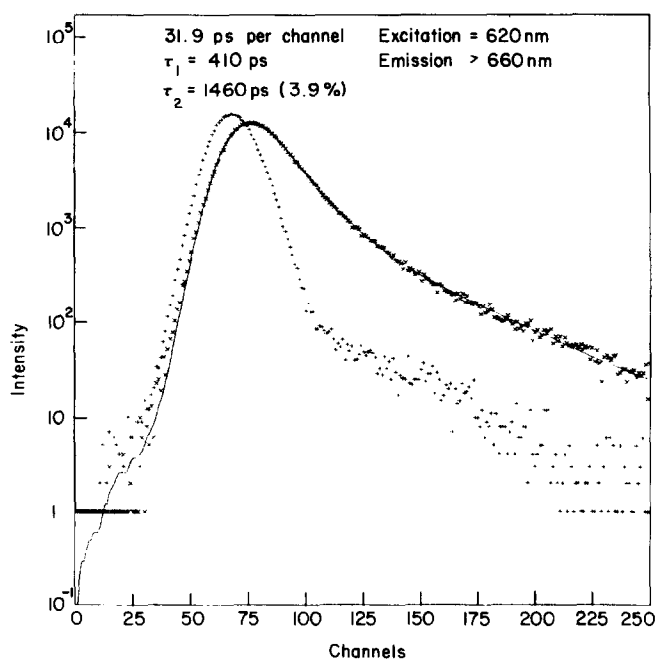


Fig. 14 Time resolved fluorescence decay of dark adapted chloroplasts

Conclusions

The cw mode-locked dye laser is a very attractive light source for time resolved spectroscopic studies in the 10^{-9} - 10^{-13} s time region. Because of its simplicity and reliability the synchronous pumping method is the best technique for most applications where time resolutions of < 1 ps is not required. Techniques are improving rapidly and 0.1 ps pulses can be expected in the near future. A wavelength tuning range of 260-1000 nm is possible with current techniques.

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