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The Scintillation Efficiency of Benzene*

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The scintillation efficiency of a benzene + p-terphenyl solution, extrapolated to unit energy transfer efficiency, has been determined to be 0.042 (or equivalently $0.0116 \pm .004$ photon/ev). This corresponds to a G value for production of $^1B_{21}$ benzene of $1.55 \pm .05$. It is suggested that the difference between this value and that for other alkyl benzene liquids (1.9-2.5) and for anthracene crystal (2.20) is related to the efficiency with which upper states of these substances make internal conversion to the emitting or transferring state.

1. Introduction

The scintillation efficiency of a crystal is usually defined as either the photon energy or number of photons emitted per ev of absorbed exciting radiation. As such, it can be used together with independently measured emission quantum yields to determine directly the G value for production of the emitting state of the crystal. For "thick" crystals of anthracene ($d > 1.5$ mm) excited by fast electrons, most workers appear to agree on a scintillation efficiency of *ca.* 0.040^{1-7} or, equivalently, 0.015 photons emitted per ev absorbed. Using Birk's⁷ value of 0.68 for the technical fluorescence quantum yield, one obtains a G value of 2.2 for production of the emitting state of anthracene. For diphenylacetylene, stilbene, naphthalene, terphenyl and dibenzyl substantially lower G values of 1.1, 1.5, 1.2, 1.2 and 0.9 have been reported by Chizhikova⁸ for production of the emitting states of these crystals.

Early measurements of Pringle *et al.*⁹ indicated that the scintillation efficiency of a deaerated solution of p-terphenyl in an alkyl benzene solvent was comparable to that of anthracene crystal. In 1963, Hastings and Weber¹⁰ reported an energy efficiency of .052 (.018 photon/ev) for a deaerated solution of PPO and POPOP in toluene containing C^{14} hexadecane. For a similar solution containing H^3 hexadecane an efficiency of

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.044 (.015 photon/ev) can be calculated from their data.¹¹ In the absence of a value either for the efficiency of energy transfer from PPO to POPOP or for the emission quantum yield of POPOP, only lower bounds can be determined for the G value for production of the sensitizing state of toluene. Under the conditions of their experiment (PPO concentration 3 g/liter), it can be estimated that 95% of excited toluene transferred energy to PPO¹². Therefore for C¹⁴ and H³ excitations respectively, we obtain as lower bounds $G = 1.9$ and 1.6 .

It was the purpose of the work herein described to obtain the G value for production of ¹B_{2u} benzene using C¹⁴ as excitation source.

2. Experimental

The determination of S involves two measurements, one of absorbed energy and the other of the total flux of emitted photons. For C¹⁴ beta excitation, the first measurement is a relatively simple one involving merely the determination of sample activity. The average energy of the C¹⁴ beta spectrum is known accurately and corrections for surface to volume effects, if required, can be made with reasonable confidence. The experimental difficulties occur mainly in measurement of the total flux.

The technique that we have employed involves a comparison of the C¹⁴ excited emission from a benzene +p-terphenyl solution with the emission produced by optical excitation of the same solution in the p-terphenyl absorption band. If the emission geometries were the same, then clearly with the additional measurements of absorbed photon intensity and p-terphenyl fluorescence quantum yield, one could determine directly the total emitted quantum flux for C¹⁴ excitation. Unfortunately the emission geometries are never quite the same. The beta excitations are produced uniformly throughout the solution, whereas the density of photon excitations decreases exponentially away from the front window of the cell. Thus appropriate corrections must be made and it is here that most of the uncertainty in the measurement resides.

We introduce the following definitions:

- α —Optical absorption coefficient of p-terphenyl
- J_0 —Number of photons incident on cell/cm² min
- J —Number of photons absorbed/cm³ min

I_0 —Total number of photons absorbed/min

ν —The fraction of emitted photons that are collected by the photomultiplier

I_p —Photomultiplier current when viewing terphenyl emission under optical excitation conditions.

I_β —Photomultiplier current when viewing terphenyl emission under C^{14} excitation conditions

τ —Energy (ev) absorbed from C^{14} disintegration/min-ml

φ —Fluorescence quantum yield of p-terphenyl

S —Scintillation efficiency (photons/ev)

In the above definitions J and ν are to be considered functions of position within the cell. We do not make any correction for reabsorption of the emitted light, since for p-terphenyl there is a sufficiently large Stokes shift to make such corrections negligible for the solution thicknesses employed.

It is clear from the above definitions that

$$J = J_0 \alpha e^{-\alpha z} \quad (1)$$

$$I_0 = \int_V J dv \quad (2)$$

$$\frac{I_p}{I_\beta} = \frac{\varphi}{ST} \frac{\int_V J \nu dv}{\int_V \nu dv} \quad (3)$$

where z is the distance within the solution from the front window of the cell and the integrals extend over the entire cell volume. The cells were all cylindrical with exciting light entering along the axis of the cylinder. The optical path length of the cell was either 1.10 cm or 0.20 cm. In both cases, for the wavelengths and concentrations employed, this length was adequate for complete (i.e., > 99%) absorption of the beam within the solution. The emission was always taken from the back face of the cell. In the following discussion we will use r as the coordinate for the radial distance away from the axis of the cylinder and let R refer to the radius of the exciting beam. Substituting (1) and (2) into (3) gives then

$$S = \frac{S_0 I_0 \alpha}{\pi R^2} \frac{\int_V e^{-\alpha z} \nu(r, z) r dr dz}{\int_V \nu(r, z) r dr dz} \quad (4)$$

where $S_0 = I_p \varphi / I_\beta \tau$ and the integral over V' extends only from $r = 0$ to $r = R$. It is also assumed in the derivation of (4) that the exciting photon

beam is homogeneous over R . The validity of this assumption is examined later.

In the case that α is very large so that absorption is confined essentially to the immediate vicinity of the front window only, Eq. (4) reduces to

$$S = S_0 I_0 I'$$

where

$$I' = \frac{\int_0^R \nu(r, 0) r dr}{\pi R^2 \int_V \nu(r, z) dr dz} \quad (5)$$

For the special case that $\nu(r, z)$ is independent of z for all r from $r = 0$ to $r = R$ and is zero for $r > R$, I' becomes equal merely to $1/\pi R^2 l$, the reciprocal of the illumination volume.

Matheson, Coleman and Bell spectroquality benzene was further purified by three successive recrystallizations from the melt (15% rejection/crystallization) and then distilled in a 50 cm column and the middle fraction collected. Scintillation grade p-terphenyl from Pilot Chemicals was used without additional purification. However, a sample of p-terphenyl that was zone refined in a He atmosphere was found to have a total fluorescence intensity when excited by a 5 mc gamma source identical to that of the unrefined material.

Initially a known volume of benzene was placed in a tube and attached to the vacuum line. On the same manifold were connected vials containing *ca.* 50 μ c of C^{14} benzene vapor (uniformly labeled) with specific activity *ca.* 12.8 mc/millimole. After brief degassing the vials were broken open and their contents condensed into the benzene liquid. The system was then opened and p-terphenyl solutions prepared with this benzene. The solutions were then admitted to degassing tubes to which were attached the fluorescent cells and then degassed by 4 to 5 successive freeze-pump-thaw cycles. Prior to filling with solution, the degassing tubes were baked at 200°C and pumped to 10^{-6} torr.

The source of UV radiation was a Hg medium pressure arc (Hanovia type S-H) used in conjunction with a Bausch and Lomb $f/3.4$ grating monochromator. Excitation was at 3140 Å. The emission was seen from the back face of the cell by a DuMont 6292 photomultiplier usually through a cobalt glass filter to remove the unabsorbed beam. In order to follow drift in the lamp intensity a sealed solution of chromatographed

fluorescein in alcohol was periodically inserted into the cell holder and its emission measured. The cell holder was thermostatted at $27 \pm 1^\circ\text{C}$. Both I_β and I_p were measured with the same solutions, I_β first being measured and then a shutter between lamp and monochromator opened and I_p measured. Since I_β never exceeded *ca.* 0.1% of I_p , correction for the C^{14} induced scintillations during the optical experiments was unnecessary.

The procedure of Hatchard and Parker¹³ was followed for use of potassium ferrioxalate as actinometer. All actinometer solutions were 0.006 M $\text{K}_3\text{Fe}(\text{C}_2\text{O}_4)_3 \cdot 3\text{H}_2\text{O}$ in 0.2 M H_2SO_4 and were photolyzed in cells identical to those used in the emission measurements. Analysis was at 510 m μ with a Beckman DU. The extinction coefficient of Fe^{++} was determined to be 1.147×10^4 l/mole cm at 25°C .

After experiments were completed, the cells were opened and the C^{14} solutions diluted to *ca.* 4×10^4 dpm/ml with 10^{-2} M p-terphenyl in benzene and placed in vials for counting in a Nuclear Chicago Liquid Scintillation Counter. The time for present count of 10^6 was compared with a New England Nuclear standard of 3.83×10^5 dpm/ml diluted by a factor of 10 with 10^{-2} M p-terphenyl in benzene.

3. Results

Initial measurements were made in a 2.6 cm diameter cell with path length $l = 1.10$ cm. Apertures of diameter 1.90 cm were placed immediately in front of and behind the cell. Three solutions were used containing p-terphenyl at 1.43, 2.86 and 5.72×10^{-3} mole/liter. The specific activity was made up to be approximately 6.4 $\mu\text{c/ml}$. Seven actinometer solutions were measured. Illumination times varied from 60 to 117 min. The ratio of photons absorbed/sec to the intensity of fluorescein emission (in amps) was $5.30 \pm 0.04 \times 10^{21}$ photon/min amp. Since the fluorescein emission varied from 4.11×10^{-7} to 4.48×10^{-7} amp, approximately 2.3×10^{15} photon/min were absorbed in the solution. The activity of the sample was ultimately determined to be 1.56×10^7 dpm/ml. Since the average energy of the C^{14} β ray spectrum is 45 kev¹⁴, $\tau = 7.03 \times 10^{11}$ ev/min ml. This, of course, requires some correction for loss of energy at the walls of the cell. For this purpose we follow the treatment of Richards and Rubin¹⁵ who calculate that for a cell with dimensions all larger than the range of the particle, the fraction of energy lost to the walls is

$AL/6V$ where A is the area and V the volume of the cell and L is the range of the particle. The half thickness in Al for C^{14} is 1.9 mg/cm^2 ¹⁶, whereas the range of the maximum and average energy β particle is *ca.* 30 mg/cm^2 and 3 mg/cm^2 respectively.¹⁷ Thus for the cells employed, the fraction of energy absorbed at the walls is calculated to be less than 1-2% and will be ignored in the subsequent discussion.

The results of all measurements are summarized in Table I. The values of S/I presented in column 3 were calculated based on a p-terphenyl fluorescence quantum yield of 0.75.^{18, 19} $(S/I)_{\infty}$ was obtained from the intercept of a linear plot of I/S vs. $1/c$. The ratio of intercept to slope also provides a value for α , the transfer constant. The average value of α from Table I is $1440 \pm 70 \text{ l/mole}$ which is in good agreement with a previously reported value of $1400 \pm 75 \text{ l/mole}$ for the same system excited with the Cs^{137} gamma.²⁰

The extinction coefficient of p-terphenyl at 3140 Å is 2440 l/mole cm . Over the band pass of the monochromator, the average extinction coefficient is about 1600 l/mole cm . Therefore, over our concentration range there was 90% light absorption within 0.07 to 0.4 cm of the front window and consequently there exists some justification for use of Eq. 5. Since the cell was placed *ca.* 5 cm from the photomultiplier it is not unreasonable to invoke the approximation that $\nu(r, z)$ is independent of z within the illumination volume $V = \pi R^2 l$. This will tend to slightly overestimate S . Use of Eq. (5) then results in

$$S = \frac{S_0 I_0}{V} \delta \quad (6)$$

where $\delta = \frac{\int_0^R \nu(r, 0) r dr}{\int_0^{R_0} \nu(r, z) r dr}$ and R_0 is the radius of the cell ($R_0 > R$). The

integral of $\nu(r, 0)$ from R_0 to R is required to take into account the contribution to I_{β} from excitations arising outside of the illumination volume V . Setting $\delta = 1$ provides upper bounds on S . These are listed in column 6 of Table I. As expected, the value for S obtained for the experiment without back aperture is somewhat larger than the other values since δ must be increased on removal of the aperture. It is difficult to estimate lower bounds on δ since $\nu(r, z)$ is expected to be a strong function of z between R and R_0 . However, from the geometry employed a crude

TABLE I Measurements in 1.10 cm Cell

Exptl. Condition	[p-terphenyl] $\times 10^3$ mole/liter	S/I (photon/ev cm ³)	α (l/m)	(S/I) ∞	S(photon/ev)
No filter	5.72	0.0429			
No back aperture	2.86	0.0382	1380	0.0484	0.0154
	1.43	0.0321			
No filter	5.72	0.0390			
1.90 cm back aperture	2.86	0.0338	1400	0.0435	0.0139
	1.43	0.0289			
2 mm thick	5.72	0.0408			
Co filter	2.86	0.0370	1580	0.0454	0.0145
1.90 cm back aperture	1.43	0.0314			
5 mm thick	5.72	0.0405			
C0 filter	2.86	0.0364	1390	0.0457	0.0146
1.90 cm back aperture	1.43	0.0304			

estimate is *ca.* 0.5 and thus the lower bound on *S* is *ca.* 0.01 photon/ev.

To improve the geometry, our final set of measurements were made in cells of path length 0.2 cm. The cell was used with a back aperture (i.e., between cell and photomultiplier) of 1.640 cm and with front apertures of 2.700, 2.170, 1.810, 1.476 and 1.200 cm respectively. The cell diameter was 3.00 cm. Two solutions were used, both containing p-terphenyl at 5.63×10^{-3} M. The specific activity in each cell was initially prepared to be *ca.* 32.8 $\mu\text{C}/\text{ml}$. At the conclusion of the experiment, the measured C^{14} activity was 8.12×10^7 dpm/ml corresponding to $\tau = 3.64 \times 10^{12}$ ev/min ml. Surface to volume corrections were less than 2% and were therefore ignored.

In Table 2 are summarized the more pertinent results of these measurements. If the beam were uniform over the cell, then I_0/R^2 should be constant. From the results presented in column 2 it can be seen that the beam indeed remained reasonably uniform in intensity to $R = 1.085$ cm and fell off by *ca.* 30% beyond this radius.

TABLE 2 Measurements in 0.2 cm Cell

R (cm)	$I_0/R^2 \times 10^{-15}$	$I_p R^2/I_0 \times 10^{21}$	$S/IV \times 10^3$	δ	$S \times 10^3$ (photon/ev)
1.350	2.32	5.50	7.4	1.00	7.4 (10.2) ^a
1.085	3.27	3.92	10.6	1.00	10.6
.905	3.11	3.99	10.7	1.00	10.7
.738	2.92	3.55	12.0	0.89	10.7
.600	3.16	2.35	15.6	0.59	9.2

^a Corrected for non uniformity of the beam (see text)

The integral $\int_0^R \nu(r, o) r dr$ is seen from Eq. (1)–(3) to be proportional to $I_p R^2/I_0$. Thus if $\nu(r, o)$ were independent of r then I_p/I_0 would be constant for all apertures. For the smallest two apertures of 0.600 and 0.738 cm, $I_p/I_0 = 6.55$ and 6.53×10^{-21} amp-min quanta⁻¹. The next aperture of 0.905 cm is larger than the back aperture (0.820 cm). Therefore $\nu(r, o)$ and perforce I_p/I_0 decreases to 4.86×10^{-21} amp-min quanta⁻¹. From the results presented in column 3 of Table 2, it appears that light originating from the region between 0.738 and 0.905 cm contributes only *ca.* 10% to the measured emission signal. Furthermore

light originating from the region $R > .905$ cm does not reach the photomultiplier.

For use in Eq. (6) values for δ were calculated from the results in column 3 of Table 2. With the exception of the results for the largest apertures, the scintillation efficiencies tabulated in column 6 are in good agreement considering the *ca.* 4 fold variation in illumination volume. The low result for the largest aperture is most simply explained in terms of non-uniformity of the beam at large r as has already been discussed. Since scintillations are not collected from this region, S should be calculated using a value of I_0/R^2 averaged over those apertures from 0.600 to 1.085 cm. As indicated in Table 2, this brings S into the range of values obtained for the other apertures.

The average value of S obtained from Table 2 is $0.0103 \pm .0004$ photon/ev at 5.63×10^{-3} M p-terphenyl. The value at infinite terphenyl concentration can be obtained from the equation²⁰

$$S = S_{\infty} \frac{\alpha c}{1 + \alpha c} \quad (6)$$

where $\alpha = 1440$ l/mole as previously discussed. Thus S_{∞} becomes 0.0116 ± 0.004 photon/ev, which is within the bounds established by measurements in the larger cell. Dividing by the p-terphenyl fluorescence quantum yield of $0.75^{18, 19}$ gives finally $G(^1B_{2u}) = 1.55 \pm 0.05$.

4. Discussion

From the G value of 1.55 for benzene and the lower bound of 1.9 obtained from the data of Hastings and Weber¹⁰ for toluene, one calculates that first excited states are produced at least 1.2 times more efficiently in toluene than in benzene. This is in agreement with previously reported relative G values of 1.0, 1.4, 1.5 and 1.6 for benzene, toluene, p-xylene and mesitylene respectively under Cs^{137} gamma excitation conditions.²⁰

The experimental ordering of G values with large gap between benzene and the other alkylbenzenes has been suggested to be related to the varying efficiencies with which upper states of the alkyl benzenes make internal conversion to the first excited singlet state.²⁰ To permit quantitative predictions, it is convenient to divide all energy losses into two classes, those lying below and those above the first ionization potential.

The first class we call "excitations", the second "ionizations". It is then assumed that a) the cross section for the production of an "excitation" lying ϵ *ev* above the ground state is proportional to the optical transition probability to that state,²¹ and that b) an "ionization" does not convert to an "excitation". Within the validity of these assumptions, the relative *G* values were then predicted to be proportional to

$$\langle\beta\rangle = \frac{\int k(\epsilon) \beta(\epsilon) d\ln\epsilon}{\int k(\epsilon) d\ln\epsilon} \quad (7)$$

where $k(\epsilon)$ is the optical extinction coefficient to the state ϵ and $\beta(\epsilon)$ the efficiency of internal conversion from this state to the transferring state. Values of β were originally obtained from emission measurements for excitations between 2700 and 1700 Å.²² Improved values of β have been obtained more recently by Feinleib²³ from measurements extended to 1300 Å. With these values Eq. (7) gives $\langle\beta\rangle = 0.45, 0.73, 0.92$ and 0.92 for benzene, toluene, *p*-xylene and mesitylene, respectively, thus predicting relative *G* values of 1.0, 1.6, 2.0 and 2.0. Although the relative ordering, and the anomolous gap is predicted by this calculation, there remains a tendency to overestimate the *G* values relative to benzene by *ca.* 10–30%. A similar situation obtains for anthracene. A value for $\langle\beta\rangle$ of *ca.* 0.85 has been determined by integration to 1800 Å.²³ Therefore, relative to benzene, *G* for anthracene is predicted to be 1.9, whereas experimentally one obtains 1.4.

We could attempt to improve the agreement by permitting some "ionizations" to convert to "excitations". It has recently been reported that in liquid benzene, the ratio of the yield of triplets produced by processes other than intersystem crossing from the singlet, to the total yield of singlets has bounds of 0.27 and 0.54.²⁴ Assuming that all of these triplets are produced by ion + electron recombinations and that for 1/4 of such recombinations the final state is singlet, we obtain $.14 < G^+ < .28$ where G^+ is the yield of singlets by charge recombination. To explain now the toluene yield of 1.4 relative to benzene requires that at least between 30–50% of all energy losses be "excitations". This, however, seems to be much too large in view of the fact that less than 5% of the total optical oscillator strength lies below the first ionization potential. A possible explanation is that some energy losses lying above the first ionization potential convert to "excitations" without passing through an intermediate state of charge separation.²⁵

It is interesting to note that a similar conclusion can be reached from an alternative calculation based on the absolute G value. Below the first ionization potential of benzene, almost all of the oscillator strength lies within the third electronic absorption system at *ca.* 7 *ev*. Thus assumption (a) leads us to predict that most "excitations" are produced by *ca.* 7 *ev* losses (i.e., $\langle \epsilon \rangle = 7 \text{ ev}$ where $\langle \epsilon \rangle$ is defined through an equation analogous to (7) with β replaced by ϵ .) Since $\langle \beta \rangle = 0.45$, assumption (b) demands that a minimum of 16 *ev* be required to produce one transferring state. Therefore, from $G = 1.55$ we conclude that 24% of the absorbed energy produces "excitations". If we permit "ionizations" to convert to "excitations" within the restrictions previously cited that led to $0.14 < G^+ < 0.28$, we find only little improvement—a reduction from 24% to *ca.* 21%. Thus once again we are led to suspect the possible importance of high energy states that can convert directly to the lower electronic states.

The only important modification required in Eq. (7) by the existence of this process would be to extend the integrals over a much larger region of oscillator strength. However, in view of the reasonable success obtained from the integration to *ca.* 9 *ev*, it is expected that there will not be substantial changes in the relative values of β . Some preliminary results to 11 *ev* tend to confirm this prediction,²³ but measurements to substantially higher energies are clearly required.

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