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Letter

Measurement of the lowest unoccupied molecular orbital energies of molecular organic semiconductors

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ABSTRACT

The lowest unoccupied molecular orbital (LUMO) energies of a variety of molecular organic semiconductors have been evaluated using inverse photoelectron spectroscopy (IPES) data and are compared with data determined from the optical energy gaps, electrochemical reduction potentials, and density functional theory (DFT) calculations. A linear fit to the electrochemical reduction potential (relative to an internal ferrocene reference) vs. the LUMO energy determined by IPES gives a slope and intercept of −1.19 ± 0.08 eV/V and −4.78 ± 0.17 eV, respectively, and 0.92 ± 0.04 and −0.44 ± 0.11 eV, respectively, based on the DFT calculated LUMO energies. From these fits, we estimate the LUMO and exciton binding energies of a wide range of organic semiconductors.

1. Introduction

As the field of organic electronics continues to mature, designing high performance devices requires that the frontier energies of molecular semiconductors be accurately determined. For example, the energy of the highest occupied molecular orbital (HOMO) of molecular thin films can be determined with reasonable accuracy (with an error of typically ±0.2 eV) [1] using ultraviolet photoelectron spectroscopy (UPS). Furthermore, the lowest unoccupied molecular orbital (LUMO) can also be determined from inverse photoelectron spectroscopy (IPES), which allows for a direct measurement of the electron affinity [2,3]. The transport gap (E_t) of these materials is then taken as the difference between the HOMO and the LUMO energy levels. In practice, the LUMO energy is often inaccurately defined as the HOMO energy plus the optical energy gap (E_{opt}), referred to here as the "optical LUMO", where E_{opt} corresponds to the energy of the long wavelength edge of the exciton absorption band. While E_{opt} can be measured to within ±0.05 eV, the value of E_t can exceed this by as much 0.5–1.0 eV due to the exciton binding energy [4]. Additionally, lower energy transitions into intermediate states can lead to an underestimation of both E_{opt} and E_t, and consequently, the LUMO energy. This incomplete picture has led to confusion about charge carrier injection, the role of heterojunction interfaces in determining energy transfer, and charge transport in organic materials. Unfortunately, the consistent use of UPS and IPES can be both time consuming and complex, with differences in measurement conditions resulting in variations in measured energies for the same material [1]. In practice, therefore, indirect methods have been employed to estimate the HOMO and LUMO energies of organic solids, leading to further confusion in the interpretation and comparisons of these various results.

Rather than use data from optical and UPS measurements to locate the LUMO energy, a common alternative is to use results from electrochemical measurements. In principle, the LUMO energy can be determined from the electrochemical reduction potential (E_{red}), a process analo-

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gous to charge injection in organic films. However, E_{red} must be adjusted to account for the differing dielectric media in which the measurements are performed. Nevertheless, E_{red} measured for organic compounds have been shown to depend linearly on their electron affinities in the gas phase [5,6]. The use of E_{red} to determine LUMO energies was originally applied to conjugated oligomers, and has recently been extended to include small molecular weight materials [7,8]. One drawback to this approach is the need to establish an electrochemical reference potential to vacuum. The ferrocenium/ferrocene (Fc^+/Fc) couple with a potential of 4.80 eV relative to vacuum, is commonly used for this purpose [9]. Alternatively, aluminum *tris*-8,hydroxyquinolate (Alq_3) has recently been shown to be a useful electrochemical reference with a potential of −2.30 V with respect to Fc^+/Fc [10]. The LUMO energy of Alq_3 is found to be 2.25 ± 0.25 eV measured by IPES [11]. Implicit in all of these methods is a linear dependence between E_{red} and IPES measured values with a slope equal to unity. However, we recently reported a relationship between the HOMO energy (measured by UPS) and the oxidation potential (E_{ox}) of molecular organic semiconductors [12] that had a linear dependence with a slope of 1.4. The slope was found to be determined, in part, by image forces between the electronic orbital distribution and the metal electrode immersed in a dielectric solution.

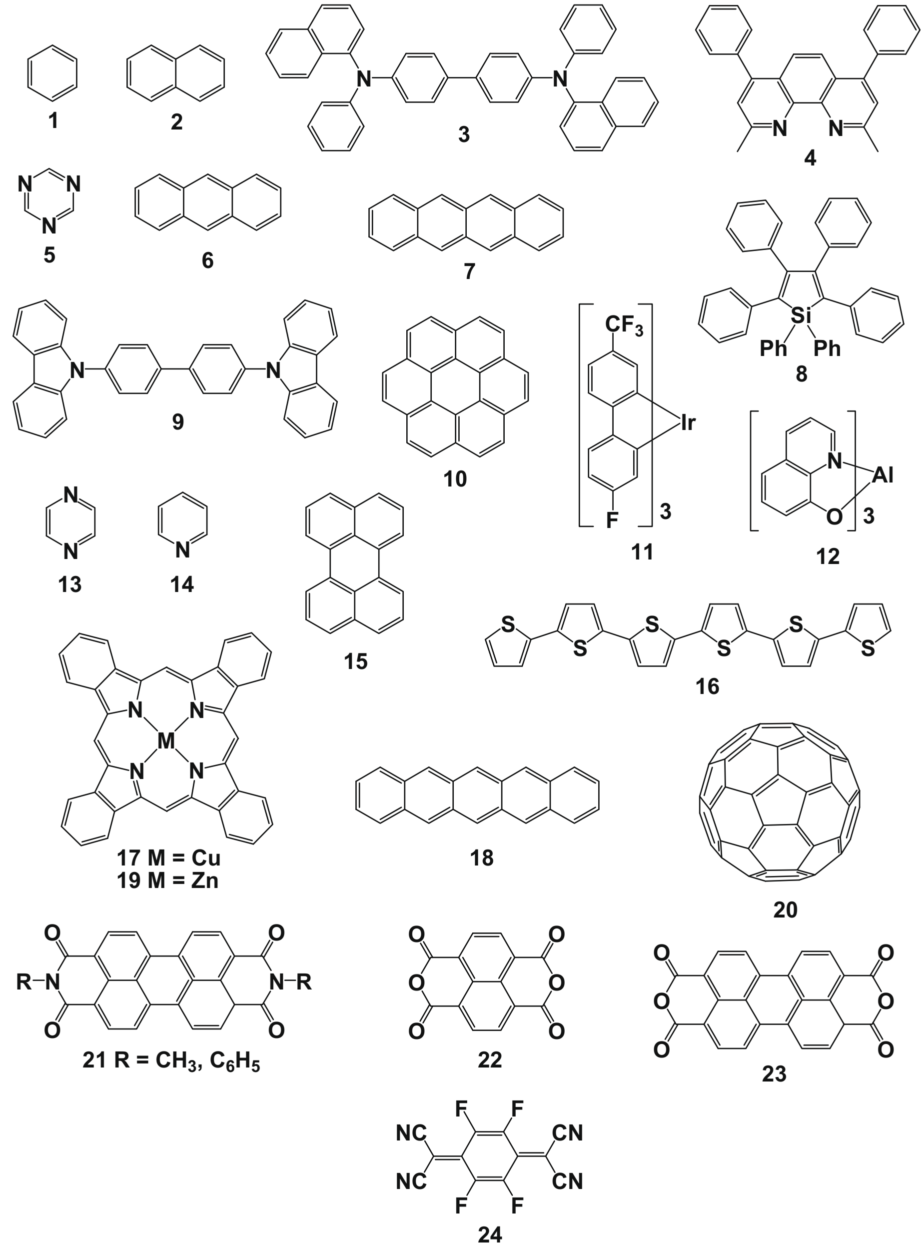

Fig. 1. Molecular structures for the compounds listed in Table 1.

Here, we present the interrelationships between the LUMO energies of a variety of molecular organic semiconductors measured by several techniques. These correlations allow us to evaluate the accuracies and advantages of the various methods, and to extract a relationship between the exciton binding energy and the energy gap.

The electron affinities for 24 compounds shown in Fig. 1 are presented in Table 1. Included in the table are LUMO and HOMO energies obtained from IPES and UPS, the optical energy gap, the optical LUMO (UPS plus E_{opt}), the electrochemical reduction potential, and the LUMO energies obtained from electron density functional theory (DFT) calculations. The LUMO energies obtained by IPES span a range of approximately 5 eV, and include both organic compounds and inorganic complexes. The criteria used to select compounds for Table 1 is that both IPES and reversible reduction energies were available. The errors are ±0.2 eV for UPS, and ±0.35 eV for IPES data. The values do not account for ambiguities in some reports of IPES data taken from the peak maxima, as opposed to others that report the peak onset of the spectra. The use of the peak onset data can lower the IPES values by several 100 meV, an issue that is addressed in detail by Krause et al. [13]. The optical energy gap is estimated from the low energy edge of the absorption band of the lowest energy exciton manifold, preferably where the normalized absorption and fluorescence spectra intersect. This value has an error of ±0.05 eV. To determine the optical LUMO values, data were taken from UPS measurements which give a direct measure of the HOMO energy. Reduction potentials are either taken from literature values, or measured using cyclic voltammetry vs. a Fc^+/Fc reference. A correction was applied to literature values recorded in acetonitrile (0.40 V) or dimethylformamide (0.45 V) vs. a standard calomel electrode (SCE) for comparison purposes [14].

A plot of the transport gap, determined from the difference between the LUMO and HOMO energies measured by photoelectron spectroscopic methods (IPES and UPS, respectively), and the optical gap for the materials in Table 1 is shown in Fig. 2. A linear fit to the data gives a slope and intercept of 1.39 ± 0.15 and -0.46 ± 0.38 eV, respectively, with a correlation coefficient of $r^2 = 0.80822$. The slope indicates that, as the energy gap increases, the exciton binding energy (E_{BE}) increases as well, with a minimum of approximately $E_{BE} = 0.25$ eV for $E_t = 2$ eV, to $E_{BE} = 1.5$ eV at $E_t = 6$ eV (i.e. $E_{BE} - 0.3(E_t - 1\ \text{eV})$). These values fall within the range of independent measurements of the binding energy, determined either from peak-to-peak [4] or peak onset [13] UPS–IPES data, and also correspond to behavior commonly observed in inorganic semiconductors. In that case, the material polarizability (and hence permittivity, ε) decreases with increasing band gap. Then, since $E_{BE} \sim 1/\varepsilon$, we expect to see a linear increase in binding energy with band gap, as observed here for organic semiconductors. We caution, however, that the charge density and distribution, which depend on molecular size, influence the binding

Table 1
Frontier orbital (LUMO and HOMO) energies for a series of molecular organic semiconductor compounds measured by different methods.

	Compound	IPES LUMO (eV)	Ref.	UPS HOMO (eV)	Ref.	E_{opt} (eV)	Ref.	OpticalLUMO (eV)	E_{red} (V)	Ref.	DFT(eV)
1	Benzene	−0.4	[19]	−7.58	[18]	4.68	[20]	−2.90	−3.87	[21]	+0.09
2	Naphthalene	−1.1	[22]	−6.4	[18]	4.00	[20]	−2.4	−2.93	[6]	−0.96
3	NPD	−1.52	[23]	−5.30	[12]	3.12	[a]	−2.18	−2.8	[a]	−1.13
4	BCP	−1.56	[24]	−6.5	[24,25]	3.43	[a]	−3.07	−2.53	[a]	−1.30
5	s-Triazine	−1.58	[26]	–		–		–	−2.47	[27]	−1.57
6	Anthracene	−1.7	[22]	−5.70	[18]	3.25	[20]	−2.45	−2.36	[6]	−1.63
7	Tetracene	−1.8	[22]	−5.10	[18]	2.51	[28]	−2.52	−1.95	[6]	−2.07
8	Silole-Ph_6	−1.85	[11]	−6.19	[11]	2.97	[11]	−3.22	−2.43	[11]	−1.35[b]
9	CBP	−1.9	[29]	−6.1	[29]	3.46	[a]	−2.64	−2.75	[a]	−1.25
10	Coronene	−1.9	[22]	−5.52	[18]	2.90	[30]	−2.62	−2.48	[31]	−1.41
11	Ir(ff-ppy)$_3$	−1.9	[29]	−5.9	[29]	2.56[c]	[32]	−3.34	−2.24	[32]	−1.97
12	Alq_3	−1.96	[23]	−5.65	[12]	2.75	[33]	−2.90	−2.30	[10]	−1.74
13	Pyrazine	−2.08	[26]	–		–		–	−2.56	[27]	−1.42
14	Pyridine[d]	−2.18	[26]	–		–		–	−3.06	[27]	−0.61
15	Perylene	−2.5	[22]	−5.2	[18]	2.83	[30]	−2.37	−2.10	[6]	−1.90
16	a-6 T	−2.57	[4]	−5.3	[34]	2.43	[35]	−2.87	−2.18	[36]	−2.18
17	CuPc	−2.65[e]	[37]	−4.82	[37]	1.80	[38]	−3.02	−1.29	[39]	−2.76
18	Pentacene	−2.8	[40]	−4.85	[18]	2.06	[41]	−2.78	−1.76	[6]	−2.38
19	ZnPc	−3.3	[42]	−5.28	[42]	1.82	[43]	−3.46	−1.31	[44,45]	−2.76
20	C_{60}	−3.5	[46]	−6.17	[47]	1.95	[48]	−4.22	−0.86	[49]	−3.23
21	Me/Ph-PTCDI	−3.95[f]	[37]	−6.6	[37]	2.35	[50]	−4.25	−1.0[g]	[51]	−3.45
22	NTCDA	−4.02	[24]	−7.97	[24]	3.54	[52]	−4.43	−0.82	[53]	−3.99
23	PTCDA	−4.10[h]	[37]	−6.95	[37]	2.20	[54]	−4.75	–		−3.91
24	F_4-TCNQ	−5.24	[42,55]	−8.34	[42,55]	2.94	[a]	−5.40	+0.13	[56]	−5.25

[a] This work.

[b] A value of −1.60 eV is given in Ref. [11].

[c] Estimated from the onset of the emission spectrum.

[d] Pyridine binds strongly to the substrate surface through nitrogen, Ref. [57] making the IPES value unreliable. For this reason, pyridine was not included in the plots shown in Figs. 2–4.

[e] A value of 3.3 eV is given in Ref. [4].

[f] A value of 4.4 eV is given in Ref. [58].

[g] Value is for Ph-PTCDI.

[h] A value of 4.6 eV is given in Ref. [23].

energy and must also be considered in a full analysis of the energy gap.

A plot of the optical LUMO (equal to $|E_{opt} + HOMO|$) vs. LUMO energies measured via IPES is shown in Fig. 3. A linear fit to the data gives a slope and intercept of 1.14 ± 0.16 and 1.19 ± 0.52 eV, respectively, with a correlation coefficient of $r^2 = 0.72019$. The slope is consistent with an increase in the exciton binding energy with increasing electron affinity. For linear acenes, this increase has also been attributed to larger electron-hole Coulombic interactions in smaller π-systems [15]. The relatively poor correlation coefficient is a consequence of two distinct regions apparent in the plot. A linear correlation exists for compounds with optical LUMO energies greater than 4.0 eV, whereas the data show considerable scatter for compounds with higher energies. Some of the variability can be ascribed to ambiguities in assigning the energy of the HOMO–LUMO gap from optical data. For example, we note that compounds such as benzene, BCP, and Ir(ff-ppy)$_3$ (compounds 1, 4, and 11) have weak optical transitions at energies lower than their strong (>1000 M^{-1} cm^{-1}) absorption bands. For these compounds, E_{opt} does not represent a transition to an orbital involved in electron capture, and hence, E_{opt} may underestimate the actual transport gap. This is in contrast to the other materials studied here.

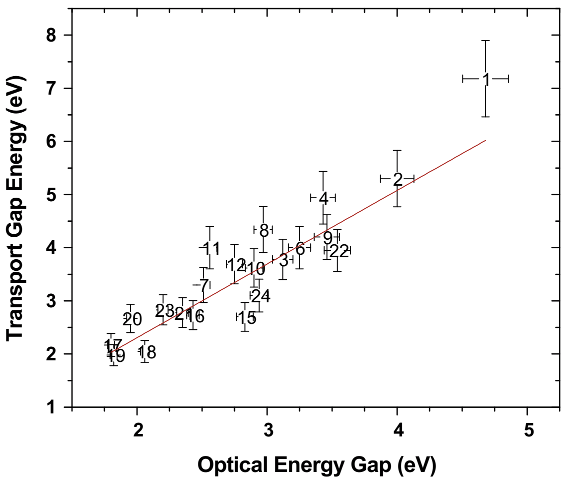

Fig. 2. Optical gap energy vs. transport gap energy. The transport gap is the energy (the lowest unoccupied molecular orbital measured by IPES minus the highest occupied molecular orbital measured by UPS) derived from data given in Table 1. A linear fit to the data is shown.

A plot of LUMO energies determined from IPES data vs. the reduction energy, E_{red}, is given in Fig. 4. A linear fit to the data presented gives a slope and intercept of −1.19 ± 0.08 eV/V and 4.78 ± 0.17 eV, respectively, with a correlation coefficient of $r^2 = 0.92140$. The value of the intercept is in agreement with that commonly assumed for the Fc^+/Fc reference energy relative to vacuum (4.80 eV). The slope implies that compounds with large transport energy gaps are slightly destabilized as neat solids relative to the fluid dielectric medium used to perform the electrochemical measurements. The correlation coefficient is markedly higher than that from Fig. 3, and indicates that reduction potentials are more strongly correlated with the actual LUMO energies, particularly for compounds with large transport gaps.

The dependence of IPES measured LUMOs on LUMO energies obtained from DFT calculations is shown in Fig. 5. A linear fit to the data gives a slope and intercept of 0.92 ± 0.04 and 0.44 ± 0.11 eV, respectively, with a correlation coefficient of $r^2 = 0.95067$. This result is a consequence of the correlations shown by DFT calculations with gas phase electron affinities [16,17]. Since the LUMO energies from DFT calculations are equivalent to values in the gas phase, the near unity value for the slope in Fig. 5 implies that there is relatively constant polarization energy for these materials in the solid state [18]. The quality of the fit indicates that DFT calculations can provide a

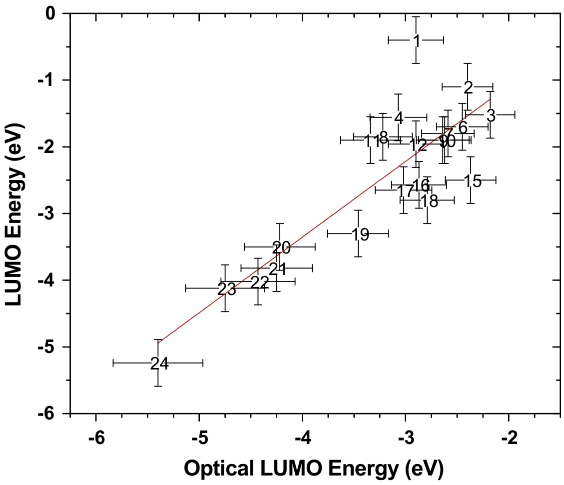

Fig. 3. Optical LUMO energy vs. LUMO energy measured by IPES. The optical LUMO is the difference between the HOMO energy (from UPS) and the optical gap energy.

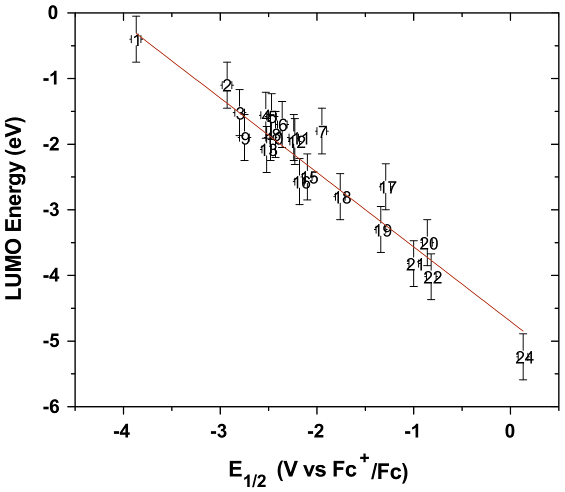

Fig. 4. Plot of the reduction potential vs. LUMO energy determined by IPES. A linear fit to the data is shown.

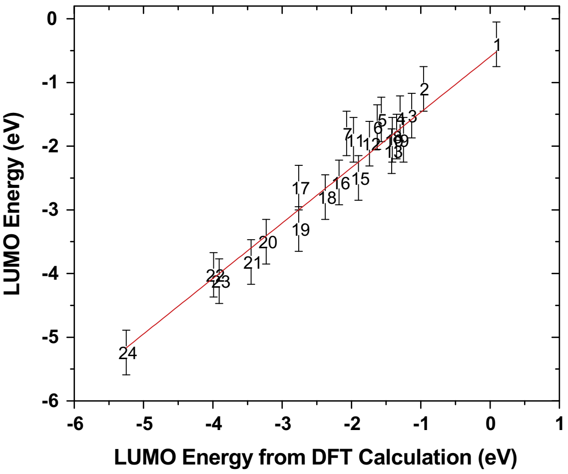

Fig. 5. LUMO energy calculated by DFT methods vs. LUMO energy determined by IPES. A linear fit to the data is shown.

reliable estimate for LUMO energies of organic solids where the needed experimental data are not available.

In summary, the correlations of IPES data with other methods currently used to estimate the LUMO energies of organic solids demonstrate several strengths and weaknesses among the various approaches. While the optical LUMO data can give a reasonable estimate for the LUMO energy for many compounds in the solid state in cases where the exciton binding energy is known, care must be taken to ensure that the lowest energy value of E_{opt} represents the appropriate HOMO–LUMO transition. Uncritical use of this method for some materials can give LUMO energies that can significantly differ from the true LUMO energy. The strong correlation between E_{red} and IPES shows that solution electrochemistry provides a satisfactory means to estimate the LUMO energy in the solid state. For compounds where it is difficult to obtain E_{red} values, use of the LUMO energy derived from DFT calculations also gives an accurate estimate of the transport level.

2. Experimental

The absorption spectra of NPD, BCP, CBP and F_4-TCNQ were recorded in dichloromethane solvent using a Hewlett–Packard 4853 diode array spectrometer. Cyclic voltammetry and differential pulse voltammetry were performed using an EG&G potentiostat/galvanostat model 28. Anhydrous DMF (Aldrich) was used as the solvent under a nitrogen atmosphere, and 0.1 M tetra(*n*-butyl)ammonium hexafluorophosphate was used as the supporting electrolyte. A Pt wire acted as the counter electrode, Ag wire was used as the pseudo-reference electrode, and the working electrode was glassy carbon. The working electrode was 0.2 cm in diameter by 1.5 cm long. The redox potentials are based on values measured from differential pulse voltammetry and are reported relative to an internal ferrocenium/ferrocene reference. Electrochemical reversibility was determined using cyclic voltammetry. The solutions contained only micro-molar concentrations of the sample solutes to prevent shifts in oxidation potential due to concentration effects, and the voltage between the working and counter electrodes was swept at a scan rate of 100 mV/s. DFT calculations were performed structures that were geometry optimized at a B3LYP level using either 6–31 G* (hydrocarbons) or LACVP** (organometallics) basis sets using the Titan software package (Wavefunction, Inc.).

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