Donor-Sigma-Acceptor Molecules Incorporating a Nonadecyl-Swallowtailed Perylene-Bisimide Acceptor

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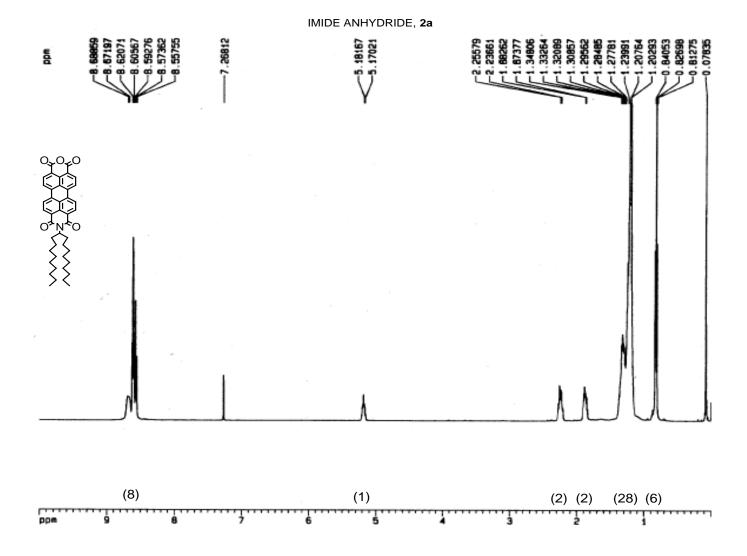
Procedure for preparation of 10-Nonadecanamine.

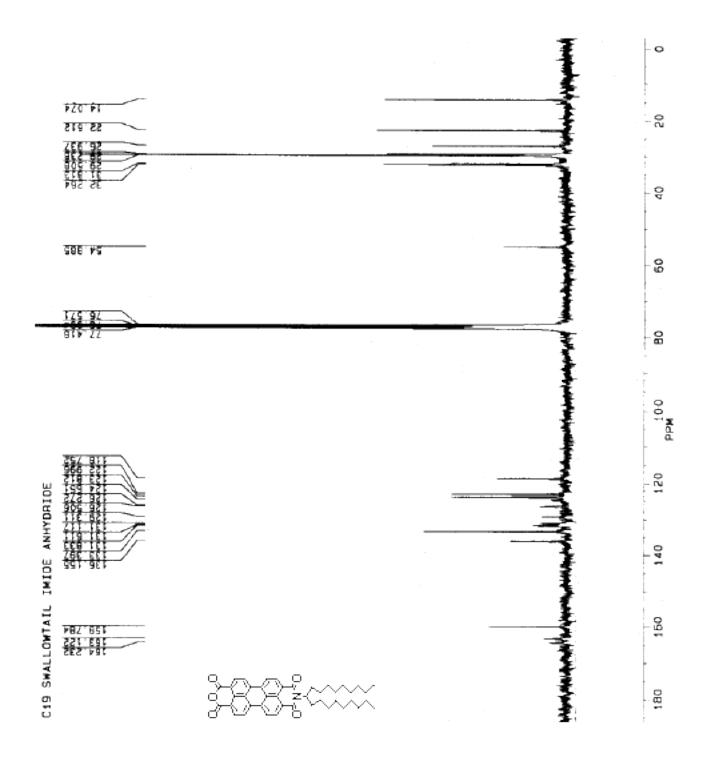
10-Nonadecanamine.¹³ A solution of 1.00 g (3.54 mmol) of 10-nonadecanone in 10 mL of ethanol and 5 mL of pyridine was treated with 0.50 g (7.2 mmol) of hydroxylamine hydrochloride and then heated on a steam bath for 2 h. The resulting mixture was concentrated by rotary evaporation and the residue was partitioned between 5% HCl (aq) and hexanes. The organic layer was dried over MgSO₄ and concentrated by rotary evaporation to yield 1.00 g (3.37 mmol, 95% yield) of 10-nonadecanone oxime as an oil.

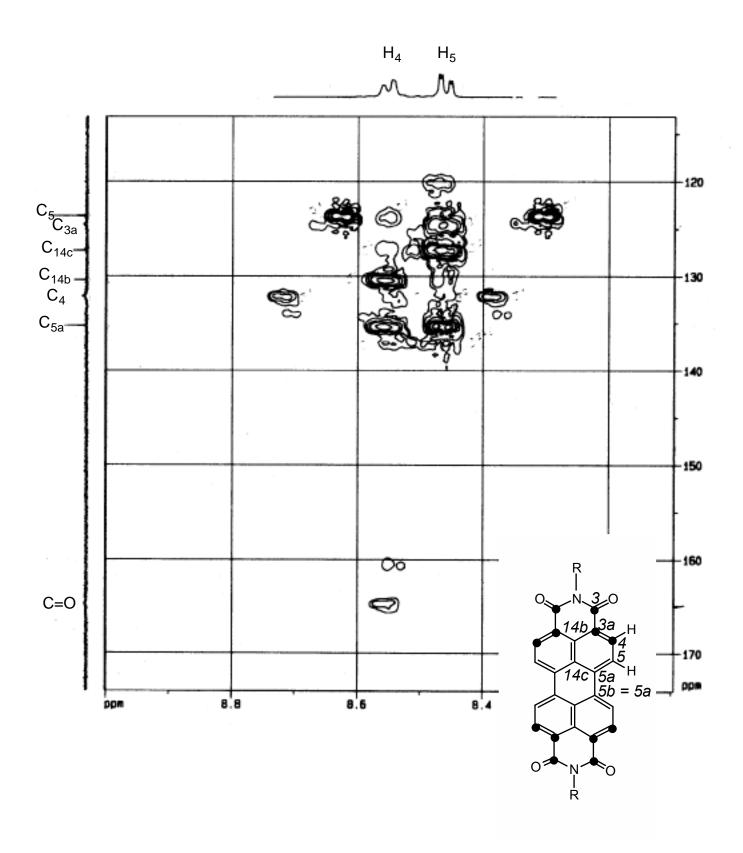
The oxime was dissolved in 25 mL of toluene. About 10 mL was distilled off to dry the system, which was then flushed with N_2 . Four mL of 70% sodium bis(2-methoxyethoxy)aluminum hydride (RedAl) in toluene were added over 5 min. There was immediate gas evolution which subsided as more RedAl was added. The system was refluxed for 2 h. The resulting mixture was carefully added, with stirring, to 20 mL of 5% HCl; then 7 mL of conc HCl was added to dissolve aluminum salts and render the mixture acidic to litmus. The solution was extracted with hexanes and the organic layer was washed with 5% NaOH, dried, and concentrated by rotary evaporation to yield 0.88 g (88% yield) of 10-nonadecanamine. 1 H-NMR (CDCl₃) δ 0.81 (t, 6H), 1.20 (m, 32H), 2.14 (broad, 2H), 2.68 (m, 1H). IR (KBr): 3372 (w), 3256 (w), 2949 (s),1463 (s), 1377 (m), 801 (m), 727 (s) cm⁻¹.

¹³Demmig, S.; Langhals, H. Chem Ber. **1988**, 121, 225-230.

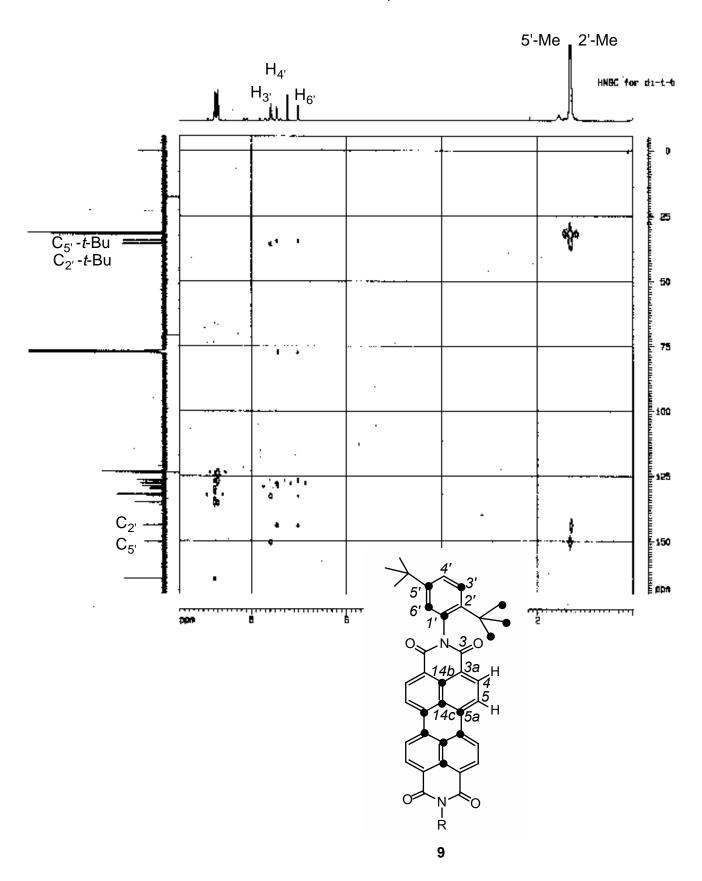
- **N,N'-Di-(10-nonadecyl)perylene-3,4,9,10-bis(dicarboximide)** (**3a**). IR (PFE/PE cards): 2920 (m), 2852 (m), 1694 (m), 1650 (s), 1594 (m), 1575 (w), 1455 (w), 1405 (w), 1338 (s), 1252 (m), 1172 (w), 811 cm⁻¹.
- **N-(10-Nonadecyl)-3,4,9,10-perylenetetracarboxylic acid 3,4-anhydride-9,10-imide (2a).** IR (PE card): 1775 (s), 1732 (m), 1703 (m), 1661 (m), 1594 (s), 1406 (m), 1356 (m), 1319 (s), 1251 (m), 1017 (m), 809 (m) cm⁻¹.
- **N-(10-Nonadecyl)-N'-(1-pyrenylmethyl)perylene-3,4,9,10-bis(dicarboximide) (4).** IR (PE card): 1694 (s), 1658 (s), 1651 (s), 1594 (s), 1436 (w), 1404 (m), 1338 (s), 1260 (s), 1172 (w), 1094 (s), 1021 (s), 846 (m), 809 (s) cm⁻¹.
- **N-(10-Nonadecyl)-N'-(4-[1-pyrenyl]butyl)perylene-3,4,9,10-bis(dicarboximide)** (**5**). IR (PE card) 1691 (s), 1658 (s), 1650 (s), 1596 (m), 1463 (w), 1438 (w), 1405 (m), 1339 (s), 1256 (m), 1165 (w), 840 (m), 808 (m), 745 (m) cm⁻¹.
- **N-(10-Nonadecyl)-N'-(2-ferrocenylethyl)perylene-3,4,9,10-bis(dicarboximide)** (**6**). IR (PE card) 1695 (s), 1658 (s), 1650 (s), 1596 (m), 1405 (m), 1343 (m), 1247 (w), 1164 (w), 1127 (w), 1106 (w), 998 (w), 807 (m), 745 (m) cm⁻¹.
- **N-(10-Nonadecyl)-N'-(2-phenylethyl)perylene-3,4,9,10-bis(dicarboximide)** (**7**). IR (PE card) 1698 (s), 1651 (s), 1594 (m), 1405 (w), 1342 (s), 1259 (m), 1167 (w), 1074 (w), 1021 (w), 848 (m), 809 (s), 745 (m) cm⁻¹.
- **2-Iodo-N,N,N',N'-tetramethyl-1,4-benzenediamine** (**16**). IR (PE card): 2828 (m), 2778 (m), 1600 (m), 1542 (w), 1501 (s), 1443 (m), 1347 (w), 1306 (m), 1223 (w), 1173 (m), 1140 (w), 1044 (w), 1019 (w), 945 (m), 837 (w), 803 (m), 737 (w), 666 (w) cm⁻¹.
- **2-(2-Cyanoethenyl)-N,N,N',N'-tetramethyl-1,4-benzenediamine (19).** IR (PE card): 2828 (m), 2786 (m), 2363 (w), 2214 (m), 1613 (m), 1559 (w), 1505 (s), 1426 (w), 1356 (w), 1314 (w), 1189 (w), 1169 (w), 1144 (w), 1048 (w), 986 (w), 944 (m), 820 (w) cm⁻¹.
- **2-(2-Cyanoethyl)-N,N,N',N'-tetramethyl-1,4-benzenediamine (20).** IR (PE card): 2836 (s), 2247 (w), 1613 (m), 1513 (s), 1451 (w), 1422 (w), 1356 (w), 1297 (w), 1169 (w), 945 (m), 812 (w) cm⁻¹.
- N-(9-Nonadecyl)-N'-(3-[2-(N'',N''',N''',N'''-tetramethyl-1,4-benzenediaminyl)propyl])-perylene-4,5,9,10-bis(dicarboximide) (21). IR (PE card): 1696 (s), 1659 (s), 1596 (s), 1505 (w), 1459 (w), 1405 (m), 1343 (s), 1256 (w), 1165 (w), 853 (w), 812 (m), 745 (m) cm⁻¹.



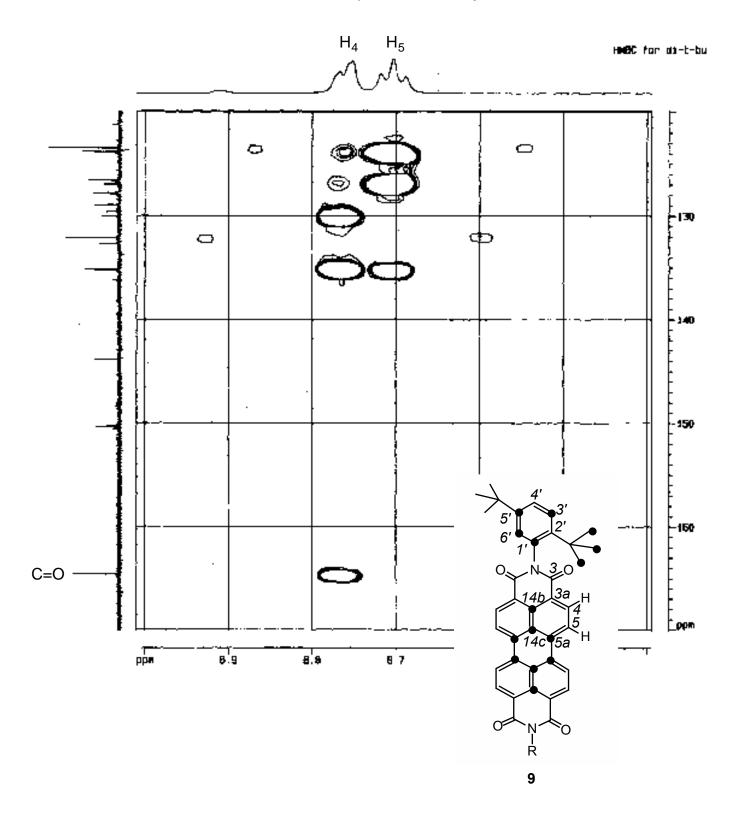




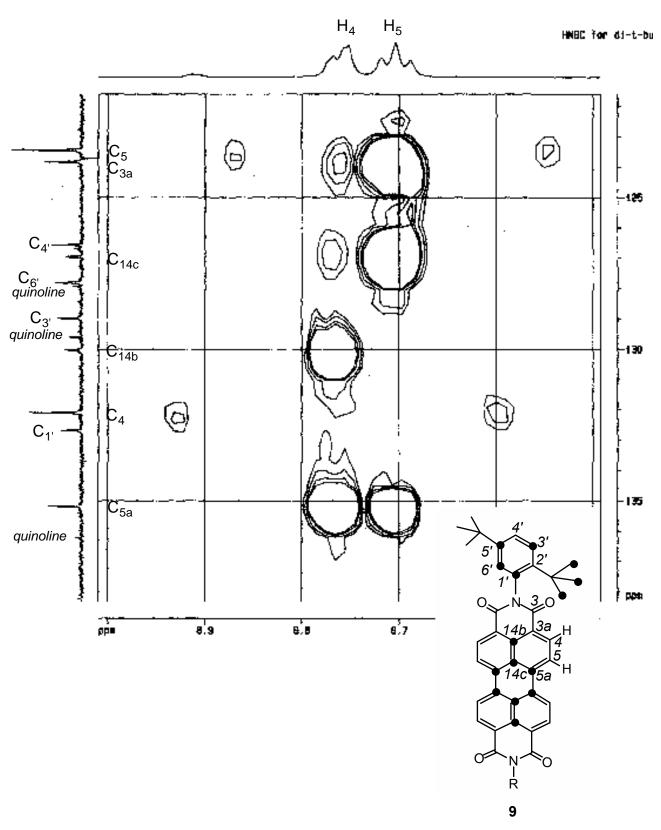
N,N'-Bis-(2,5-di-tert-butylphenyl)perylene-3,4,9,10-bis(dicarboximide), **23** HMBC spectrum



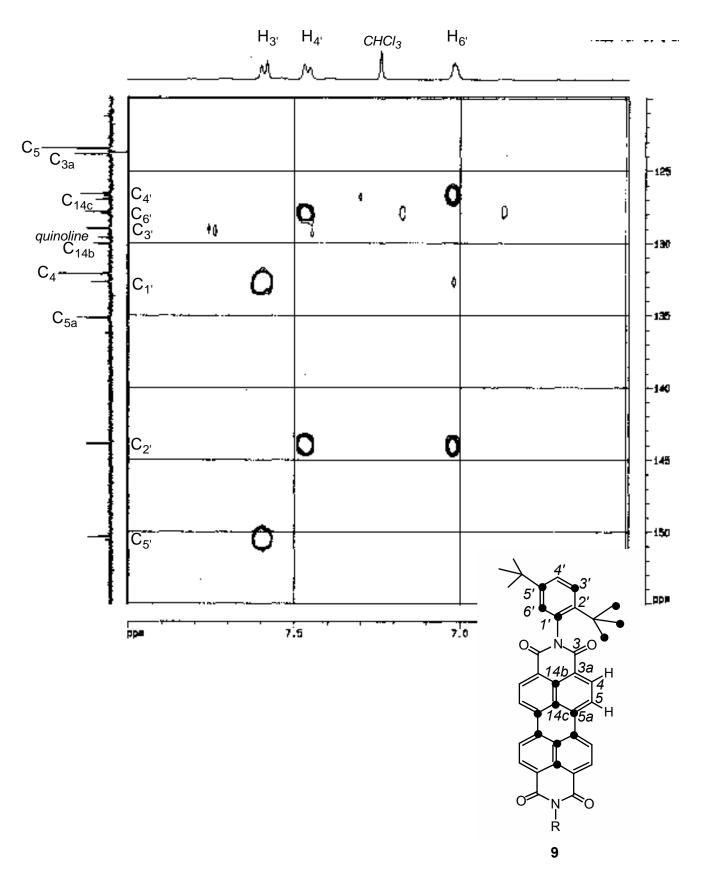
N,N'-Bis-(2,5-di-tert-butylphenyl)perylene-3,4,9,10-bis(dicarboximide), **23** HMBC spectrum, carbonyl



N,N'-Bis-(2,5-di-tert-butylphenyl)perylene-3,4,9,10-bis(dicarboximide), **23** HMBC spectrum, perylene



N,N'-Bis-(2,5-di-tert-butylphenyl)perylene-3,4,9,10-bis(dicarboximide), **23** HMBC spectrum, phenyl



N,N'-Bis-(2,5-di-tert-butylphenyl)perylene-3,4,9,10-bis(dicarboximide), **23** HMBC spectrum, methyl

