Part I, Chapter 4

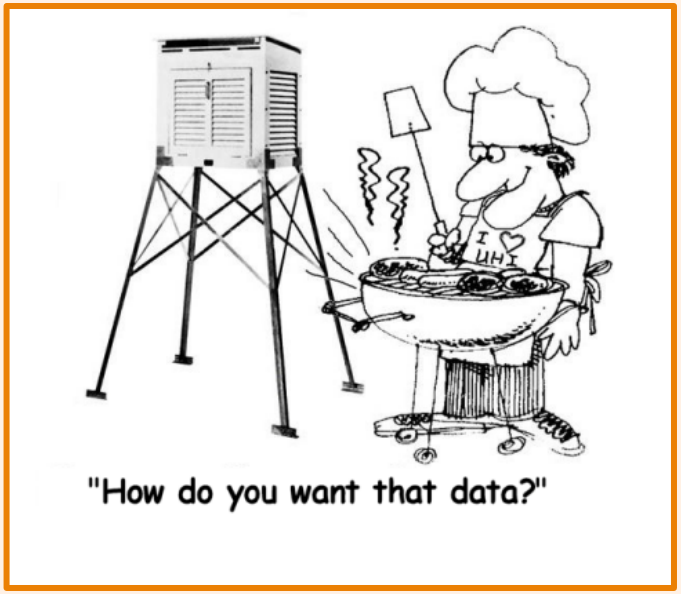
Data Tables and Drawing Schemes

Organizing Data

Standard Sequence of the Science Process

1. Hypothesis
2. Experimental Design
3. Measurements
4. Analysis
5. Hypothesis validated?

Data, Data, Data, Data....



Tables are the starting point for all scientific analyses.

Build your tables at the stage of "experimental design"!

Tables are essential to qualitative and quantitative analysis.

http://www.norcalblogs.com/watts/images/data_grill.gif

Organizing Principle of Tables

Tables are (multi-dimensional) lists.

The ordering parameter employed for listing the experimental variable can be am experimental variable or it can be merely a "count parameter" (i.e., Entry #, Reaction #, ….).

The natural order of the numbers is the obvious choice (Wavelength, Reaction Time, Wavenumber, Percent Reaction, …).

The legitimate data of a scientific paper are the unadjusted, spontaneous results obtained by following a defined procedure.

-- Entry # / Absorption {2-dimensional table}

-- Reaction # / Yield / Purity {3-dimensional table}

-- Wavelength / Absorption {2-dimensional table}

A legitimate experimental variable must have been obtained by following a defined procedure and others must be able to reproduce the data.

General Comments on Tables

Tables have a “Table Header” (a.k.a. “Legend” or “Title”). The Legend starts with “Table X:” and it is completed by a sentence in “Title Format”

Tables must be referred to in the main text. Usually a brief summary of the Table is provided when the table is referred to in the text.

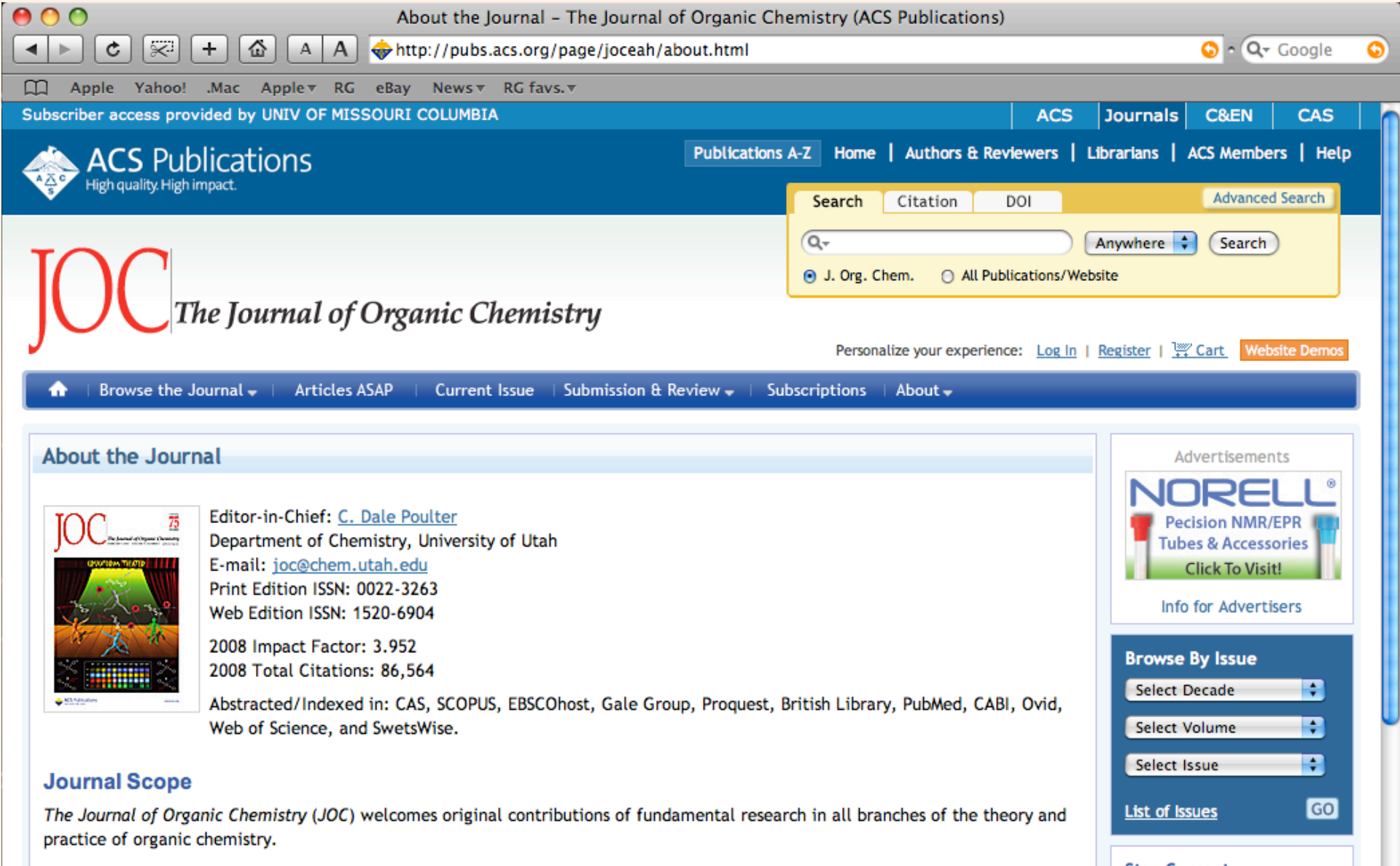
Tables must be self-contained. Tables should be understood without reference to the text.

-- provide “units” of variables and of data

-- use table footnotes to explain units etc.

-- refer to “Guidelines to Authors” about formatting requirements

JOC Setting the Standards



1-Dimensional Tables

1H), 3.99−3.93 (m, 1H), 3.68 (d, J = 3.8 Hz, 2H), 2.24−2.15 (m, 1H), 1.97−1.84 (m, 1H); ^{13}C NMR (MeOH-d_4) δ 141.9, 137.1, 127.8, 92.0, 87.9, 79.5, 73.0, 62.6, 43.6; IR (film) 3333, 2923, 2891, 1682, 1559, 1458, 1066, 997, 815 cm^{-1}; HRMS-FAB (m/z) $[M + NH_4]^+$ calcd for $C_{11}H_{17}NO_3I$, 338.0248, found 338.0248.

Preparation of 9. C-Nucleoside **8** (79 mg, 0.246 mmol) was coevaporated with pyridine three times and dissolved in pyridine (2.0 mL). To the solution was added 4,4-dimethoxytrityl chloride (114 mg, 0.34 mmol). The mixture was stirred at 25 °C for 20 h and concentrated. The residue was loaded onto a silica gel (oven-dried) column and eluted (2:1 hexanes/EtOAc) to give **9** as a colorless foam (102 mg, 67%): ^{1}H NMR (acetone-d_6) δ 7.72 (d, J = 8.1 Hz, 2H), 7.52 (d, J = 7.5 Hz, 2H), 7.42−7.19 (m, 9H), 6.91−6.87 (m, 4H), 5.14 (ddd, J = 9.6, 4.8, 4.8 Hz, 1H), 4.39 (s, 1H), 4.34−4.26 (m, 1H), 4.13−4.05 (m, 1H), 3.80 (s, 6H), 3.28−3.24 (m, 2H), 2.31−2.23 (m, 1H), 1.98−1.88 (m, 1H); ^{13}C NMR (acetone-d_6) δ 158.7, 145.4, 143.1, 137.2, 136.1, 130.1, 128.2, 127.7, 126.6, 113.0, 91.8, 86.9, 85.9, 792, 73.4, 64.6, 54.5, 44.2; IR (film) 3425,2967, 1607, 1508, 1459, 1300, 1250, 1177, 1080, 1034, 1004, 827 cm^{-1}; HRMS-FAB (m/z) $[M+Na]^+$ calcd for $C_{32}H_{31}IO_5Na$ 645.1108, found 645.1099.

Preparation of 10. To a solution of **9** (102 mg, 0.16 mmol) in CH_2Cl_2 (1.6 mL) were added diisopropylethylamine (42 mg,

Fe(II)-EDTA Digestion of Cross-Linked DNA. Fe(II)-EDTA cleavage reactions of ICLs were carried out in 50 μM $(NH_4)_2Fe(SO_4)_2$, 100 μM EDTA, 1 mM sodium ascorbate, 5.0 mM H_2O_2, 100 mM NaCl, and 10 mM potassium phosphate (pH 7.2) for 1 min at 25 °C (total volume of 20 μL each). The reactions were quenched with 100 mM thiourea (10 μL). Samples were lyophilized, resuspended in formamide loading buffer, and subjected to 20% PAGE analysis.

Acknowledgment. We are grateful for support of this research from the National Institute of General Medical Sciences (GM-054996).

Supporting Information Available: Strand damage data for 3′-^{32}P-labeled duplexes. Hydroxyl radical digestion analysis of cross-linked products. Sample autoradiogram of UV-irradiation of 5′- and 3′-^{32}P-**11** showing cleavage pattern and comigration with Maxam−Gilbert sequencing reactions. Spectral data for previously unreported compounds, UV absorption spectra of aryl iodide nucleosides, and ESI-MS for oligonucleotides containing nucleotide analogues. This material is available free of charge via the Internet at http://pubs.acs.org.

"1-dimensional tables" can be ordered lists of experimental variables in experimental sections of papers.

Multi-Dimensional Tables 1

TABLE 2. Summary of Results from Reaction Time Course Experiments[a]

entry	reactants	acid	[acid], mM	time, h[b]	% yield of 5-isocorrole[c]	% yield of self-condensation product[d]
1	**1a + 2a-OH**	$InCl_3$	0.32	2	35	1.4
2	**1a + 2a-OH**	$InCl_3$	1.0	0.5	32	1.9
3	**1a + 2a-OH**	$Sc(OTf)_3$	0.32	0.5	28	4.7
4	**1a + 2a-OH**	$Yb(OTf)_3$	10	1	26	4.5
5	**1a + 2a-OH**	$Dy(OTf)_3$	1.0	8	25	5.1
6	**1b + 2b-OH**	TFA	0.32	0.25	7.8	30
7	**1b + 2b-OH**	$InCl_3$	0.32	0.25	2.7	47
8	**1b + 2b-OH**	$Sc(OTf)_3$	0.32	0.5	6.8	45
9	**1b + 2b-OH**	$Yb(OTf)_3$	0.32	4	6.5	47
10	**1b + 2b-OH**	$Dy(OTf)_3$	1.0	8	7.4	48

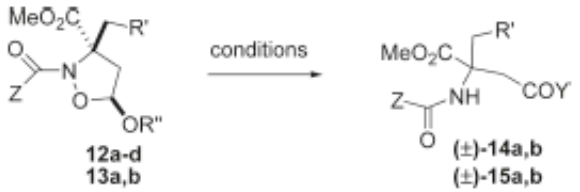
[a]The reactions were performed in CH_2Cl_2 with the indicated reactants (2.5 mM each) on a 20 mL scale at room temperature. The reactions were monitored from 1 min to 24 h. [b]The reaction time that first provided the highest yield of the 5-isocorrole. [c]The highest yield of the 5-isocorrole (HPLC) is reported. [d]The yield of the porphyrin (**1a + 2a-OH**) or porphodimethene (**1b + 2b-OH**) (HPLC) at the time that the highest yield of the 5-isocorrole was first obtained is reported. The yields reported here are generally within ~2% of the highest yield obtained at any time.

Note the formatting of header and footnotes.

Multi-Dimensional Tables 2

One can use graphics in the Table header!

TABLE 5. DAA Compounds *via* Ring-Opening of *N*-Acyl-isoxazolidine 12a–d and 13a,b



12a-d
13a,b

(±)-14a,b
(±)-15a,b

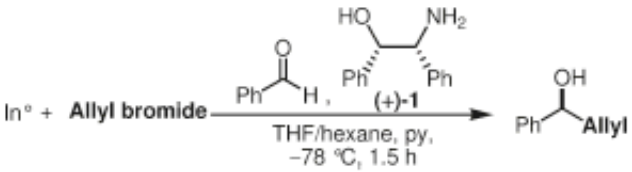
		R′	R″	Z	conditions	product	Y	yield (%)
1	**12a**	CO_2Me	Et	Me	$Mo(CO)_6$ (1 equiv), $MeCN/H_2O$[a], reflux, 2 h	**14a**	H	10
2	**12a**	CO_2Me	Et	Me	$Mo(CO)_6$ (1 equiv), $MeCN/H_2O$, reflux, 16 h	**14a**	H	40
3	**12a**	CO_2Me	Et	Me	$Mo(CO)_6$ (2 equiv), $MeCN/H_2O$, reflux, 72 h	**14a**	H	91
4	**12a**	CO_2Me	Et	Me	SmI_2 (2 equiv), THF, rt, 10 min	**14a**	H	76
5	**12c**	CH_2CO_2Me	Et	Me	$Mo(CO)_6$ (2 equiv), $MeCN/H_2O$, reflux, 42 h[b]	**14b**	H	60
6	**12d**	CH_2CO_2Me	*t*-Bu	Me	$Mo(CO)_6$ (2 equiv), $MeCN/H_2O$, reflux, 96 h[c]	**14b**	H	30
7	**13a**	CO_2Me	*t*-Bu	CF_3	$Mo(CO)_6$ (2 equiv), $MeCN/H_2O$, reflux, 96 h[c]			-[d]
8	**13a**	CO_2Me	*t*-Bu	CF_3	SmI_2 (2.5 equiv), THF, rt, 10 min	**15a**	O*t*-Bu	75
9	**13b**	CH_2CO_2Me	*t*-Bu	CF_3	SmI_2 (2.5 equiv), THF, rt, 10 min	**15b**	O*t*-Bu	81

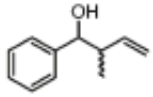
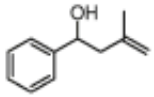
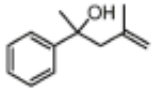
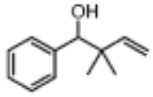
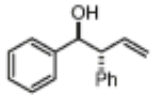
[a]10:3 volume ratio. [b]Complete conversion of the starting material. [c]Incomplete conversion of the starting material. [d]Recovery of starting material.

Multi-Dimensional Tables 3

TABLE 3. The Asymmetric Indium-Mediated Barbier-Type Allylation of Benzaldehyde with Functionalized Allyl Bromides

One can use graphics in the Table header!



Entry	Allyl Bromide	Product	% Yield	% ee (dr *anti/syn*)[a]
1	Crotyl bromide	 **3**	99	72[b] (57:43)
2	Methallyl bromide	 **4a**	70	45[b]
3[c]	Methallyl bromide	 **4b**	55	16[b]
4	Prenyl bromide	 **5**	54	56[b]
5	Cinnamyl bromide	 **6**	50	56[d] (>95:5)

And one can use graphics in Table cells!

[a] *Syn/anti* ratio determined by ^{1}H NMR. [b] Determined by chiral GC analysis. [c] The reaction was conducted with acetophenone and the optimized ketone conditions, in THF at 25 °C for 24 h. [d] Determined by chiral HPLC analysis.

In-Text Reference to Table

Thus, PtOEP-SB sensors increased their pK_a from 5.9 to 7.0 when TCPB content increased from 2.4 to 5.7% but then decreased to 6.1 at 7.6% TCPB. Two other phase transfer reagents, potassium tetrakis (4-*tert*-butylphenyl)borate (TBPB) and sodium tetra(*p*-tolyl)borate (TTB), produced significantly lower pK_a values. Similar values were observed for PdCP-SB sensors showing slightly more basic pK_a than PtOEP-SB. Such dependence of calibration on the nature and concentration of ion transfer reagent can be due to different access of protons to the dye (also seen in other ion-selective membranes.[19,21] Therefore, by selecting the indicator dye and ion transfer additive, pH sensitivity of the sensor can be tuned to cover the range of practical interest (pH 5−8 in this case). On the basis of these results, PtOEP-SB *N2* and *N3* and PdCP-SB *N7* and *N8* sensors (Table 1) were selected for further testing of their O_2 sensitivity and phosphorescent characteristics.

According to the mechanism of protonation (Figure 1), the changes in absorption were accompanied by a marked reduction in phosphorescence intensity signals (Figure 2B). At low pH values in air-saturated buffer at 30 °C, the intensity of the PtOEP-SB sensors decreased by almost 70%. Residual phosphorescence was attributed to incomplete protonation of the dye in polymer membrane.

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Letters to *Analytical Chemistry*

O_2/pH Multisensor Based on One Phosphorescent Dye

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Table 1. Main Characteristics of the PtOEP-SB and PdCP-SB Sensors

reporter dye/sensor no.	absorbance maximum [nm]	ion transfer additive [%] (w/w)	pK^a	emission lifetime[a,b] [μs]
PtOEP-SB	398(pH8.0)			
N1	443(pH2.0)	2.4(TCPB)	5.9	32.8
N2		4.1(TCPB)	6.5	31.0
N3		5.7(TCPB)	7.0	n.m.
N4		7.6(TCPB)	6.1	n.m.
N5		7.6(TBPB)	<4.0	n.m.
N6		7.6(TTB)	<4.0	n.m.
PdCP-SB	398(pH8.0)			
N7	443(pH2.0)	4.1(TCPB)	6.9	60.3
N8		5.7(TCPB)	7.2	n.m.

[a] Phosphorescent measurements in 0.1 M K_2HPO_4, 21 kPa O_2, 30 °C. Standard deviations were ~0.2 μs or 0.1 pH, respectively, ($N = 3$). [b] n.m.: not measured.