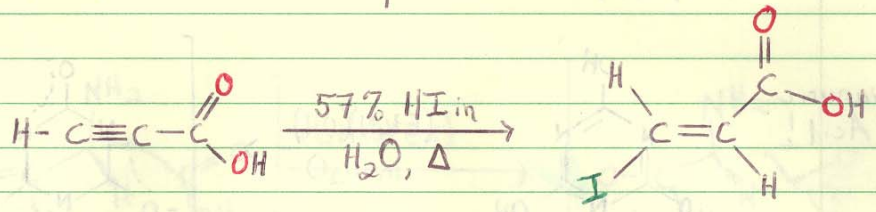
Jim Donahue
4/20/2001

Trans-3-Iodoacrylic Acid-2
Cf. p. 121 in LB V

15



$C_3H_2O_2$	9.0 mL	$C_3H_3IO_2$
70.0477 g/mol	of 57%	197.960 g/mol
2.80 g	HI in H_2O	7.913 g expected
0.040 mol	127.9124 g/mol	for 100% yield
d = 1.138 g/mL	d = 1.701 g/mL	
2.45 mL	15.309 g soln, 8.73 g HI, 0.068 mol	

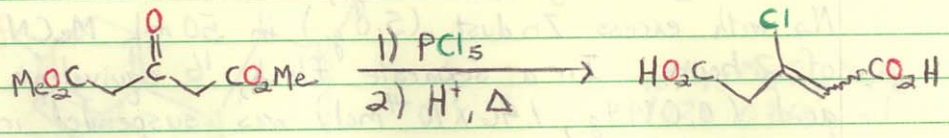
The procedure followed was that described by Abarbri, Mohamed; Parrain, Jean-Luc; Cintrat, Jean-Christophe; Duchêne, Alain Synthesis 1996, 82-86: Propiolic acid (2.80 g, 0.04 mol) and 57% HI in H_2O (9.0 mL, 0.068 mol) were combined in a 25 mL Schlenk tube and degassed with 2 freeze/pump/thaw cycles. The mixture was heated at ~120°C under N_2 for 12 hours. Upon cooling, the mixture solidified.

The crude solid was partitioned between 100 mL each of 0.1 N aqueous HCl and EtOAc. The aqueous phase was extracted with 3 x 20 mL ethyl acetate. The combined EtOAc portions were washed with 50 mL each of saturated aqueous sodium thiosulfate and sodium chloride. These two washings were each back extracted with 20 mL EtOAc. The combined EtOAc portions were stirred over anhydrous Na_2SO_4, filtered, and evaporated to dryness, affording a white solid that was largely pure by ^1H NMR.

The product was recrystallized from ~120 mL of petroleum ether/ether, affording a first crop of 1.8537 g (23.4%) of white needles after standing overnight. A second crop of material (0.2035 g; 51.3% total) was collected a few days later. A third crop (1.4486 g; 69.6% total) was obtained a few days following.

20

Jim Donahue 3-Chloro-2-pentenedioic acid-1
4/25/2001



$C_5H_{10}O_5$	PCl_5	$C_5H_5ClO_4$
150.1314 g/mol	208.23876 g/mol	164.5453 g/mol
d = 1.185 g/mL	40.0 g	
22.7 mL, 26.90 g	0.192 mol	
0.179 mol		

The procedure followed was that described by Ikeda, Izumi; Honda, Kazuhiko; Osawa, Eiji; Shiro, Motoo; Aso, Mariko; Kanematsu, Ken J. Org. Chem. 1996, 61, 2031-2037.

To stirred dimethyl acetone-1,3-dicarboxylate (26.90 g, 0.179 mol) in a 100 mL flask was added PCl_5 (40.0 g, 0.192 mol) in small portions over a period of 1 hour, which produced vigorous bubbling. At this point, the mixture was dark red and was heated to ~50°C for 1/2 hour. After cooling, the mixture was poured onto ice and stirred. To this mixture was added 100 mL CH_2Cl_2, and the organic fraction was then separated in a sepatory funnel. The aqueous phase was extracted with 4x50 mL CH_2Cl_2, the combined CH_2Cl_2 portions being washed with brine and evaporated to a red oil. This oil was suspended in 200 mL of 20% HCl and refluxed for 6 hours. The mixture became a light transparent yellow within a short time of heating. Upon cooling, the mixture was extracted with 4x100 mL Et_2O, dried over Na_2SO_4, filtered, and concentrated to a cream colored solid.

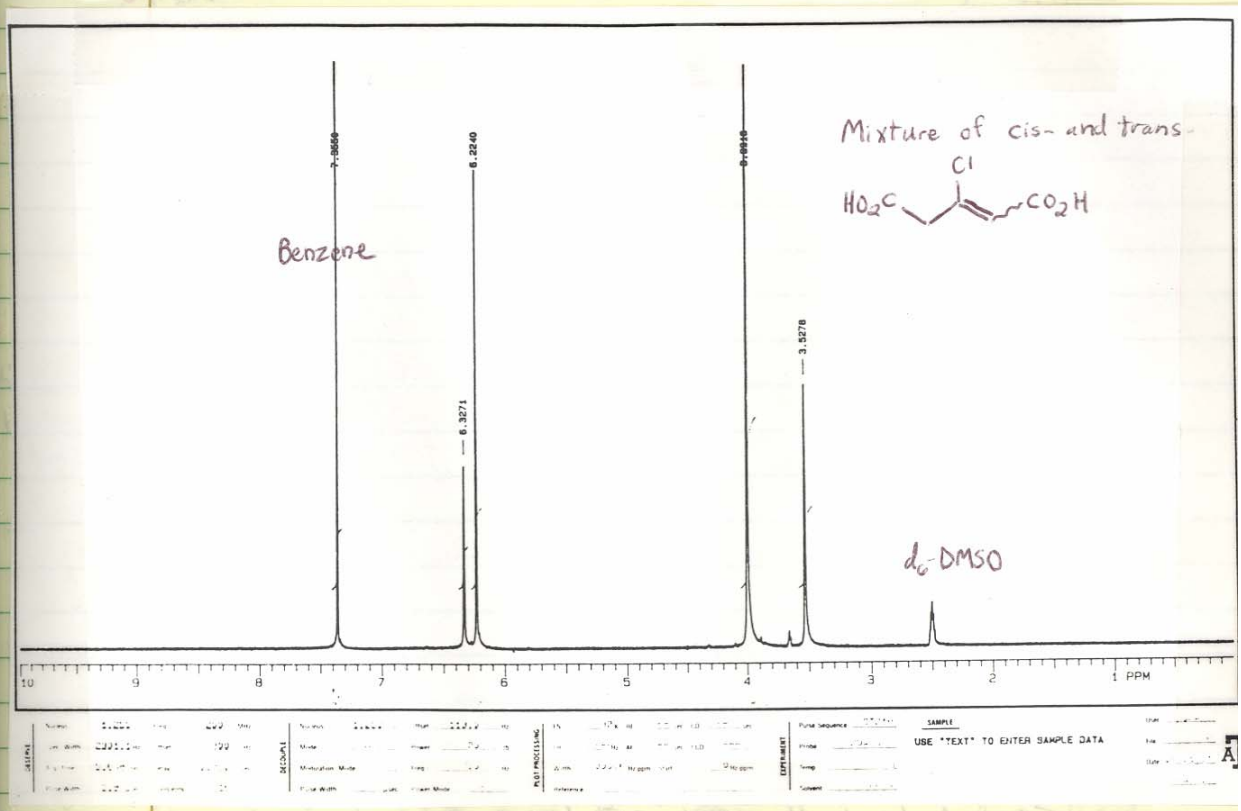
The crude solid was extracted with portions of hot benzene. These extracts were filtered and concentrated to a volume of ~250 mL. Upon standing, this yellowish solution deposited an off-white crystalline solid, some pieces of which might be suitable for X-ray diffraction. After decanting off the benzene

Continued to p. 23

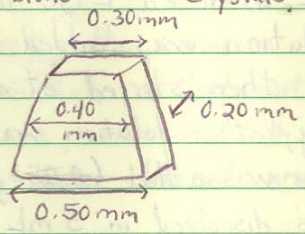
Jim Donahue
5/1/2001

3-Chloro-2-pentenedioic Acid
Continued from p. 23

27



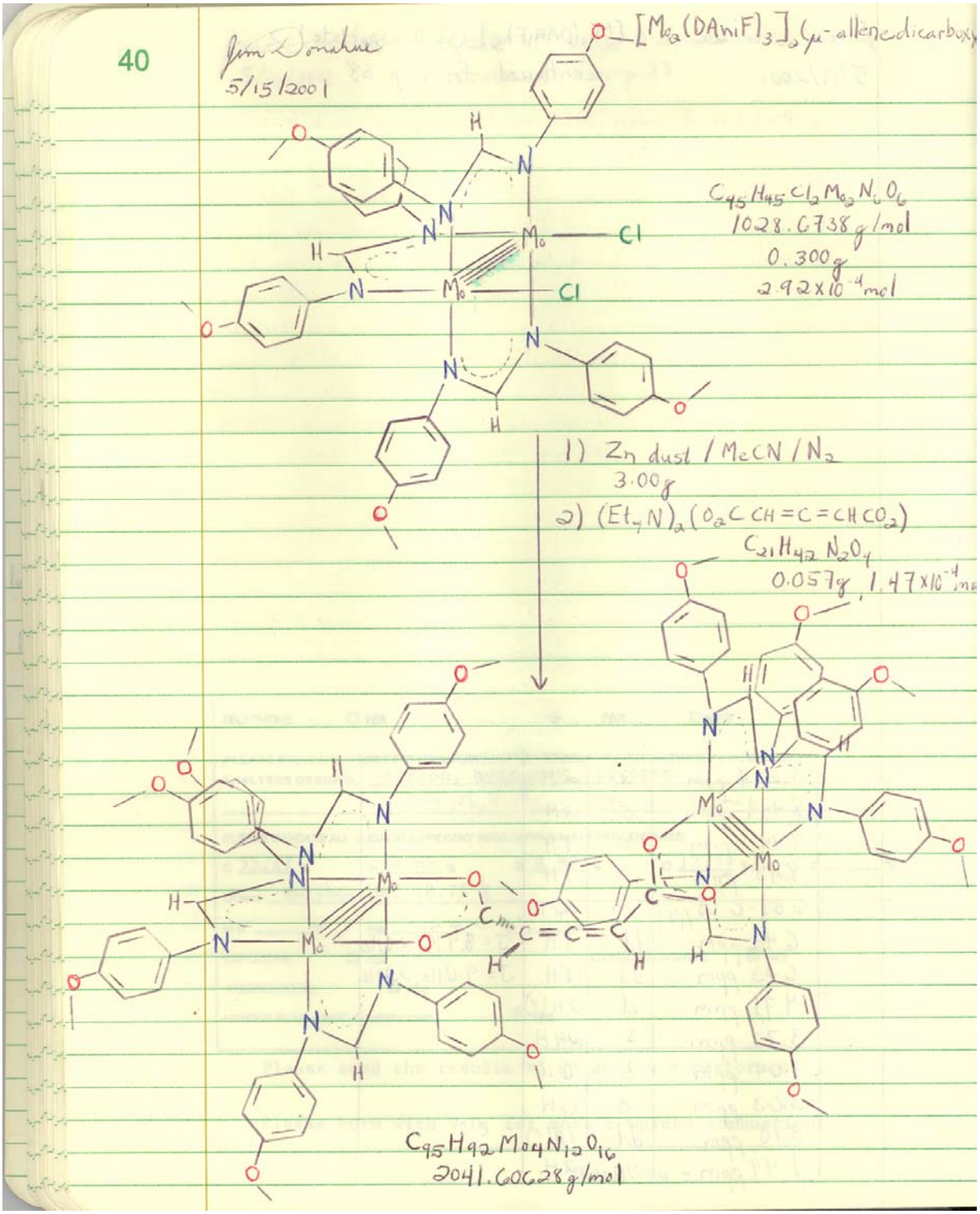
Dimensions of Crystals Used



The colorless block crystals noted on p. 23 were collected and dried, amounting to 1.6732 g altogether.

Continued to p. 30

Sample Notebook Page 4 – Jim Donahue



48

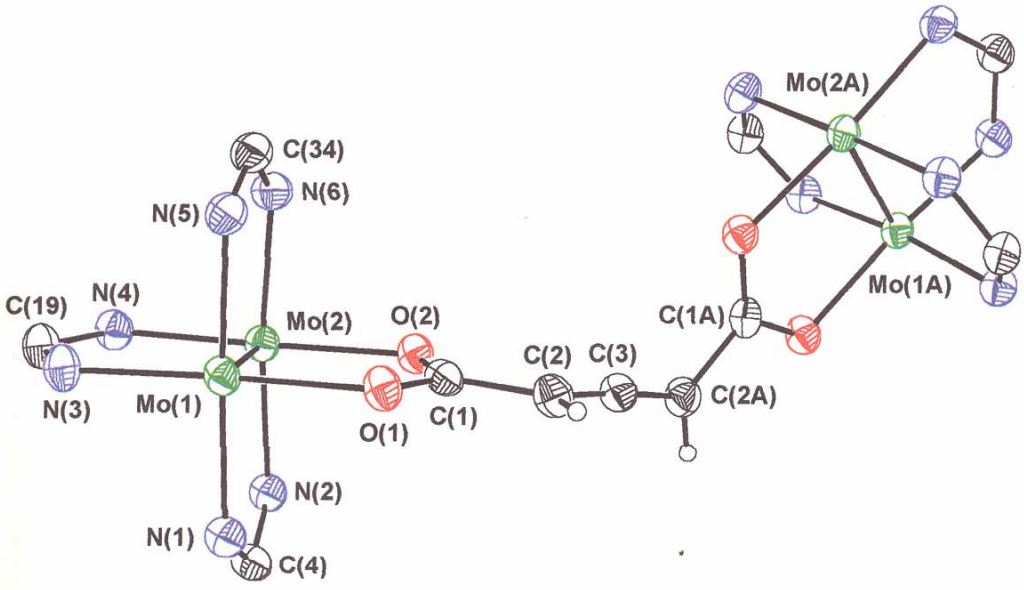
Jim Donahue 5/24/2001

$[Mo_2(DAniF)_3]_2$(μ-allenedicarboxylate)-1,2

Continued from pp. 40-41

The cell recorded on p. 41 was obtained on a cut orange needle crystal from the 1,2-dichloroethane layered diffusion. 3×45 sets of frames at 30 seconds were used.

Core Structure of $[Mo_2(DAniF)_3]_2$(μ-allene-1,3-dicarboxylate)

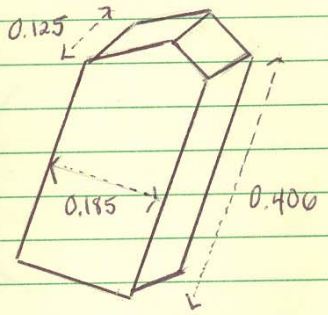


Distance between centroids of Mo_2 units: 9.395 Å
Twist between Mo_2 axes: 62.9°

Jim Donahue $[Mo_2(DAniF)_3]_2$(μ-allenedicarboxylate)-1,2 49

5/26/2001

Dimensions of Crystal



5/26/2001

The crystals from $[Mo_2(DAniF)_3]_2$(μ-allenedicarboxylate)-2 were isolated by decanting away the solvent and drying the crystals under a N_2 stream. These long needle crystals were stored in the N_2 box. 0.132 g were obtained.

Vial and sample for UV-vis	: 3.4861 g
Tare of vial	: 4.4722 g
UV-vis sample	: 0.0139 g

$$\frac{0.0139\ g}{2041.6063\ g/mol} = 6.808 \times 10^{-6}\ mol \text{ in } 25\ mL$$

⇓

A 1.50 mL aliquot of this 25.00 mL solution was taken and further diluted to 25.00 mL

$$\frac{4.085 \times 10^{-7}\ mol}{0.025\ L} = 1.6340 \times 10^{-5}\ M = 0.00001634\ M$$

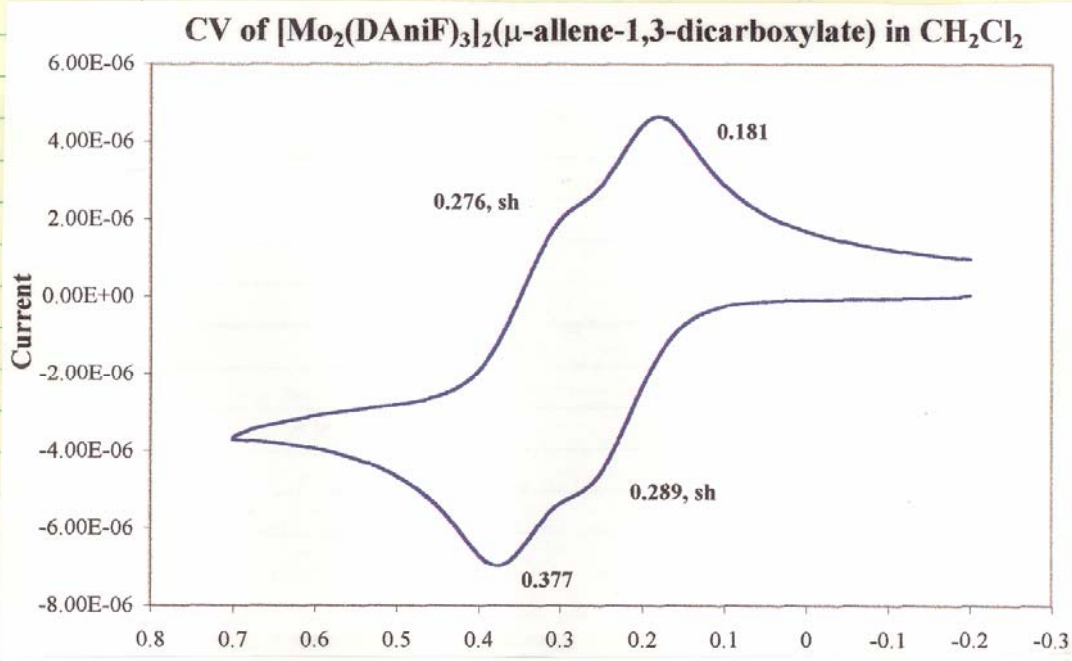
$A = \varepsilon C \ell \qquad \varepsilon = \frac{A}{C\ell} = \frac{A}{C}$

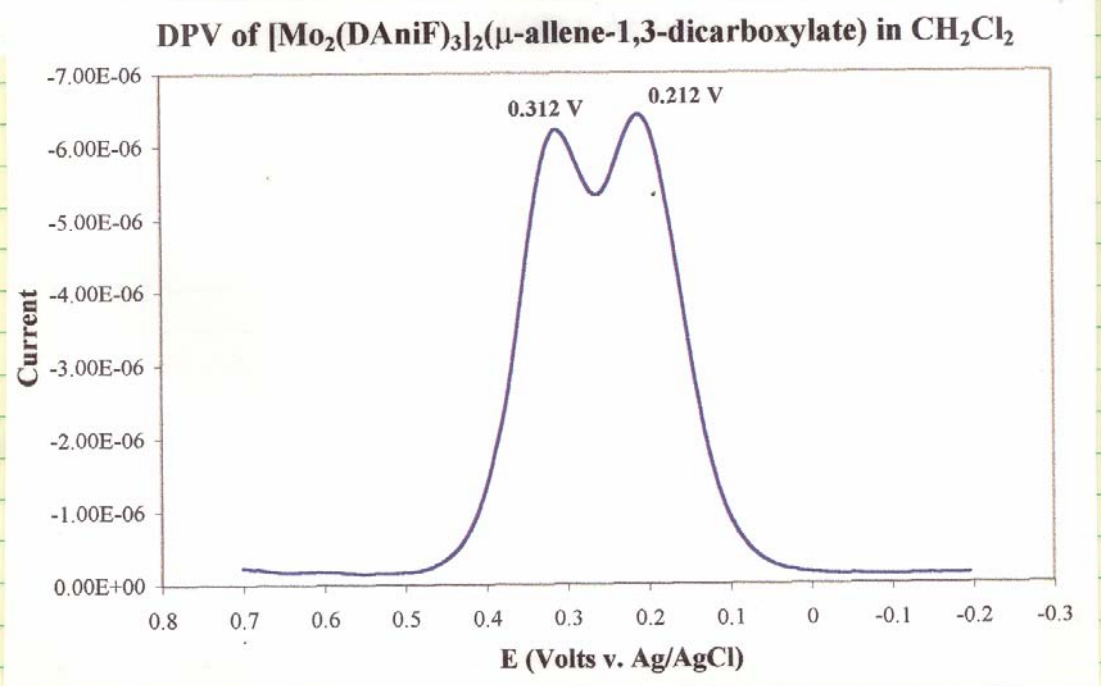
Sample Notebook Page 7 – Jim Donahue

50

Jim Donahue
5/26/2001

$[Mo_2(DAniF)_3]_2$(μ-allene-1,3-dicarboxylate)-1,2
Continued from pp. 48-49

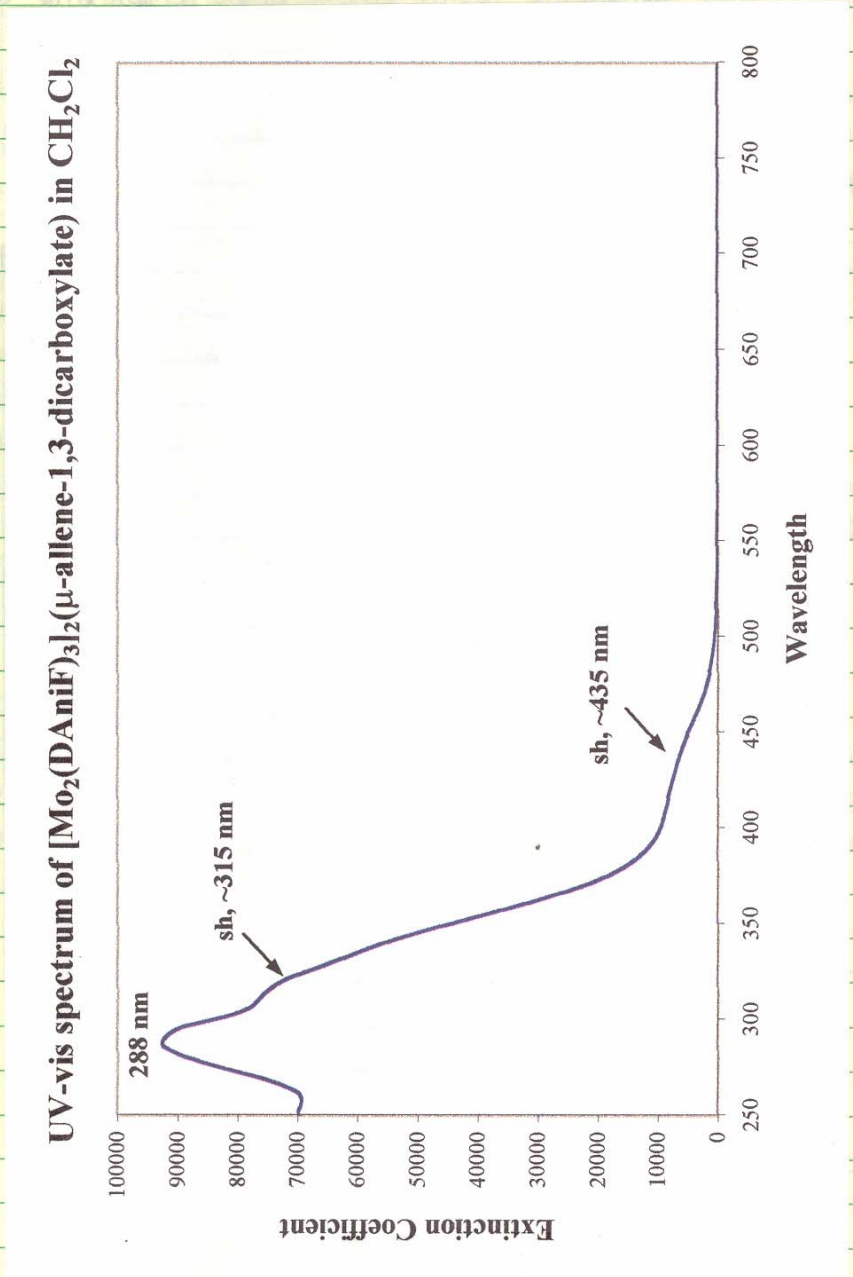




Sample Notebook Page 8 – Jim Donahue

Jim Donahue [$Mo_2(DAniF)_3]_2$(μ-allene-1,3-dicarboxylate)-1,2
5/29/2001 Continued from pp. 48, 49, 50.

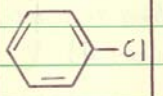
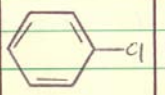
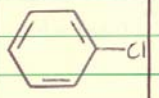
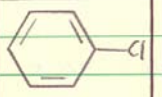
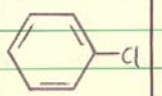
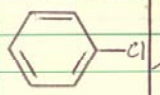
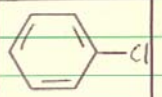
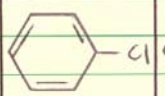
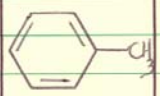
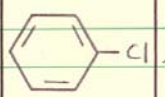
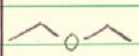
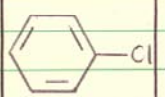
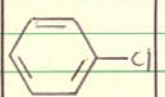
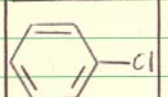
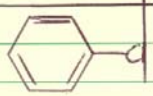
53



Sample Notebook Page 9 – Jim Donahue

Jim Donahue — Crystallization of $Fe_2(S_2C_2(Anisyl)_2)_4$ — 23

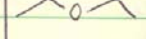
8/7/2005 — Continued from pp. 19-20

Inner	Outer	Observations and Results
	Et_2O	Saturated Sol'n - Filtered through Celite
	Bu^tOMe	Saturated Sol'n; Filtered through Celite Needle crystals! One of these crystals was used in a 60 second data set.
		Saturated Sol'n; Filtered through Celite Very small fine crystals They appear to be thin parallelpipeds  Rough surfaces Very small
		Saturated Sol'n; Filtered through Celite
	Hexanes	Saturated Sol'n; Filtered through Celite Mass of small black needles 
		Saturated Sol'n; Filtered through Celite
		Saturated Solution; Filtered through Celite
		Saturated Solution; Filtered through Celite
		Solution Diluted by factor of 2; Filtered through Celite
	Bu^tOMe	Solution Diluted by factor of 2; Filtered through Celite
		Solution Diluted by factor of 2; Filtered through Celite Ver
		Solution Diluted by factor of 2; Filtered through Celite
ntinued p. 24		
	Hexanes	Solution Diluted by factor of 2; Filtered through Celite

36

Jim Donahue $[Cp_2Co][Fe_2(S_2C_2(Anisyl)_2)_4]$ - 1

8/23/2005 Continued from pp. 34, 35

Inner Solv.	Outer Solv.	Results and Observations
DMF		Filtered through Celite - Solubility in DMF is very high giving brown-black solutions / 10/12/2005 Black plate crystals  that were relatively thick These had pointed tips Cont'd
DMF	Bu^tOMe	Filtered through Celite Black needle crystals up high on sides of vial - One was cut & mounted but showed only modest diffraction
DMF ★		Filtered through Celite These diffracted 10/7/2005 Many black square plate crystals  It was difficult to find a single crystal b/c most had parasites on their large faces.
DMF		Filtered through Celite Grungy yellow-brown solid - No crystals!
DMF		Filtered through Celite Traces of white flocculent solid - Little else No crystals
DMF		Filtered through Celite

A data set was collected on a crystal grown from DMF/Et_2O. See p. 56

These showed Diffraction! See p. 54 bottom for additional comment

10/3/2005

a = 9.4724 Å	b = 14.8249 Å	c = 27.3335 Å	
0.0020	0.0022	0.0063	
0.0037	0.0042	0.0118	
α = 100.877°	β = 89.845°	γ = 108.594°	V = 3565.84
0.027	0.024	0.014	0.71
0.050	0.045	0.026	1.32

This cell was obtained on a crystal from DMF/Bu^tOMe.

Histograms

	0.00	0.05	0.10	0.15	0.20	0.25	0.30	0.35	0.
H	225	0	0	0	0	0	0	0	0
K	225	0	0	0	0	0	0	0	0
L	225	2	0	0	0	0	0	0	0
Ω	139	61	14	7	2	2	0	0	0

Continued to p. 54

GoF = 3.48 This cell was obtained w/ 3 × 60 frames at 60 second/frame. However, ~ 2/3 of the reflections obtained were discarded in order to get the refined cell above

Guidelines for Notebook Recording

1) All laboratory research is to be dutifully recorded in lab notebooks to be supplied by JPD. See JPD if you have filled one up and require a new one.

2) The outside binding of the notebook is to be labeled with the initials of the owner and the notebook number.

3) All notebooks stay in the lab or office. They are not to be removed from 4080 or 4071A ***for any reason***.

4) Notebooks remain the property of the Donahue Group upon departure from the group. A photocopy may be made of notebook pages, but the original notebook must remain with JPD.

5) Each notebook page should be labeled with the user's name, the date, a reaction name and number, and the notebook pages from which the present work is continued. See sample notebook pages. The bottom of the page should refer the reader to continuing pages (if any) later in the notebook or to other relevant pages for comparison.

6) The reaction that is being attempted, even if it is a literature preparation, should be illustrated at the top of the page as a line equation so that it is clear to anyone who should refer to the notebook exactly what has been done.

7) Molecular formulas and weights, boiling points, densities and other useful data are conveniently recorded below the respective compound or reagent in the line equation. See example pages..

8) If a literature preparation is being followed, record that literature citation in the notebook.

9) Record sufficient detail (time, temperature, colors, smells, work-up procedure, potential hazards) to enable another person to readily reproduce your work.

10) Record all physical characterizations of new compounds, such as NMR spectra, UV-vis spectra, electrochemistry, etc., by photoreducing the image and taping it directly and securely into the relevant notebook page. See accompanying notebook pages for examples.

11) For each notebook that is filled, make a table of contents listing on the inside of the front and back covers. See example.