

APPENDIX I

OPERATIONAL SETTINGS FOR VARIOUS EQUIPMENT

Table I.1. Operational Settings for Carle Analytical Gas
Chromatograph (Model 111).

Columns: one 1/8"x2' stainless steel (28% DC 200/500 on
Chromosorb PAW), 60/80 mesh,
one 1/8"x17' stainless steel (27.5% BIS-EE-A on
Chromosorb PAW), 45/60 mesh,
one 1/8"x14' stainless steel (3% Carbowax 1540
on Porasil C), 80/100 mesh,
one 1/8"x3' stainless steel (0.4% Carbowax 1500
on Carbowax B), 60/80 mesh,
one 1/8"x6' stainless steel (80% Porapak N & 20%
Porapak Q), 50/80 mesh,
one 1/8"x7' stainless steel (Molecular Sieve 13X),
45/60 mesh,
one 1/8"x1.5' stainless steel (Chromosorb PAW),
60/80 mesh,
one 1/8"x3' stainless steel (Molecular Sieve 5A),
80/100 mesh.

Detector: thermal conductivity.

Maximum thermostat temperature: 100°C.

Hydrogen transfer tube temperature: 600°C.

Thermistor current: 25mA.

Carrier flows: 30 mls/min per sample valve at 62 psig
for helium,

Table I.1. (Cont'd.)

Carrier flows: 30 mls/min at 60 psig for nitrogen.

Attenuation: 16 till event #4, then 8 for the rest.

Program: S,

<u>Event</u>	<u>Time (min.)</u>	<u>Slot/Channel</u>	<u>Valve</u>	<u>Position</u>
1	:00	0	V ₂	CW
2	1:33	2	V ₃	CW
3	5:33	8	V ₂	CCW
4	6:13	9	V ₃	CCW
5	12:40	19	V ₁	CW
6	14:53	22	V ₁	CCW
7	38.13	57	--	---
8	38.43	58	--	---

Cycle time: 40 min.

Table 1.2. Operational Settings for Hewlett-Packard Gas Chromatograph (Model 5710A).

Column: one 1/8"x6' stainless steel (3% Dexsil 300 on 100/120 Supelcoport).

Detector: flame ionization.

Sensitivity: 6.

Attenuation: 2.

Detector Temperature: 350 °C. (at 228 mA).

Oven temperature: 8 °C/min. from 99 to 350 °C
(time one = 0 and time two = ∞).

Injection port temperature: 300 °C.

Range voltage: 1.0 mv/cm.

Chart speed: 30 sec/cm.

Integrator attenuation: 64.

Carrier flows: 20 mls/min. at 60 psig for helium,
240 mls/min. at 24 psig for air, and
20 mls/min. at 15 psig for hydrogen.

Sample dilution: 1:4 with carbon disulfide.

Sample size: 1.0 µl.

Table I.3. Operational Settings for Hewlett-Packard Gas Chromatograph (Model 5730A).

Columns: one 1/8"x6' stainless steel (Porapak Q 80/100),
one 1/8"x6' stainless steel (Molecular Sieve 5A
80/100).

Detector: thermal conductivity.

Sensitivity: 6.

Attenuation: 2.

Detector temperature: 250 °C.

Detector current: 228 mA.

Oven temperature: 200 °C.

Injection port temperature: 200 °C.

Recorder range voltage: 0.5 mv/cm.

Recorder chart speed: 30 sec/min.

Integrator attenuation: 4.

Carrier flow: 20 mls/min. at 60 psig for helium.

Sample size: 2.0 μ l.

Table I.4. Operational Settings for Chemisorption
Apparatus.

Vacuum manifold: vacuum valve of reference side - open,
vacuum valves of differential pressure
sensor (both sample and reference)
- open.

Control panel:

Varian ionization gauge: range - 10^{-6} ,
ion gauge - on.

MKS electronics unit: range multiplier - .01,
response - normal,
heater switch - off,
power switch - on.

MKS digital readout: display units - Torr,
head range - 1000,
selector - AUTO.

MKS sensor head selector: head range - 1000,
heater - on,
sensor selection - CH1.

Table I.5. Operational Settings for Philips X-Ray
Diffractometer (Type 42267/0).

Radiation source: $\text{Cu-K}\alpha_1$.

Power supply: 40 KV at 20 mA.

Electronics panel:

Dual HV supply: scint. - 0.8 KV,

fine control - 7.5 KV.

Linear amplifier: gain - 256,

input selector - SCINT.

PHS: baseline - 2.00,

window - 10.00,

mode - Integ,

clock signal - off.

PHS scan: not used.

Linear ratemeter: range - 1 K,

time constant - 2 sec,

% suppression - 0.0,

audible - off.

Table I.6. Operational Settings for Philips X-Ray
Fluorescence Spectrometer (Model PW1410-60K).

Cooling gas flow rate: P-10 gas at 1.0 l/hr.

Generator: 40 KV at 20 mA,
timer hours - ∞ ,
overload - up.

Electronics panel:

Dual HV supply: scint. - 1.0 KV,
fine control - 7.0 KV,
prop. - 1.6 KV,
fine control for prop. - 6.5 KV.

Linear amplifier: gain - 256,
input selector - PROP.

PHS: baseline - 2.00,
window - 6.00,
baseline control - Ext,
mode - Diff,
clock signal - off.

PHS scan: scan control - reset.

Linear ratemeter: range - 5K,
range multiplier - X1,
time constant - 2 sec,
% suppression - 0.0,

Table I.6. (Cont'd)

Linear ratemeter: audible - off.

Scaler/Timer: multiplier - 2,
time constant - 10^1 ,
preset - count,
start, stop, and reset - off.

Teletype transmitter: mode - single,
parity - none,
format - normal,
start, stop, reset, and print -
off.

Teletype: Local.

Spectrometer:

Radiation tube: W.

Crystal: LiF (220),

Collimator: course,

Order: I-F,

Filter: out,

Spinner: off,

Vacuum: on,

Motor drive: out,

Scan: 0

Step scan: off.