

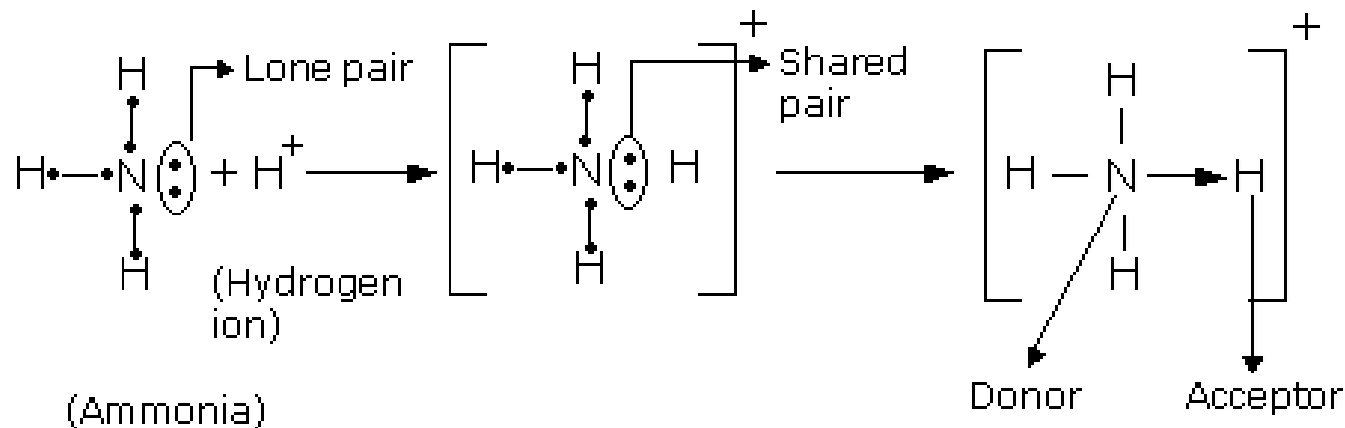
옥살레이트-철 착화합물 합성과 광화학 반응

Purpose

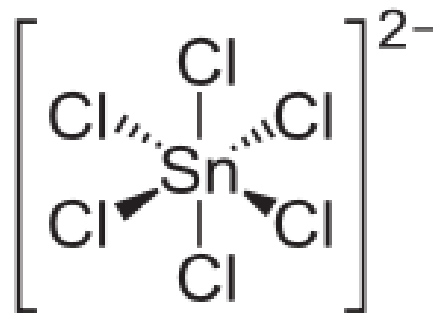
- **목표** : 철 착화합물을 합성하고 이의 광화학 반응을 이용해서 **청사진**을 만든다.
- **핵심개념** : 전이금속 리간드, 착 화합물, 광화학반응, 광량계

Theory

배위결합 (Coordinate Bond)



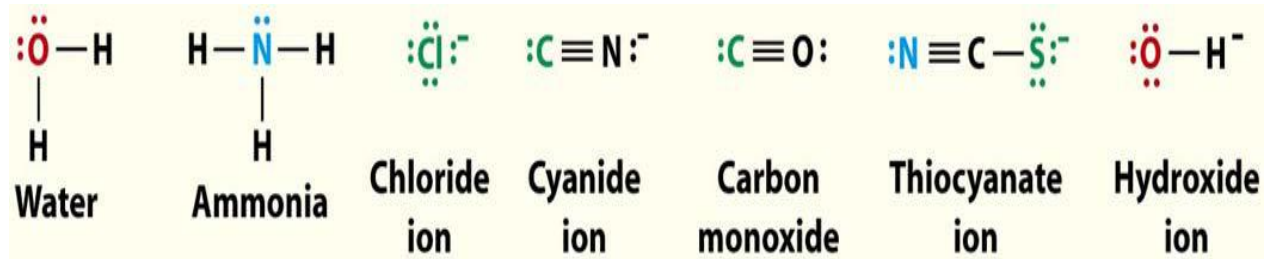
리간드



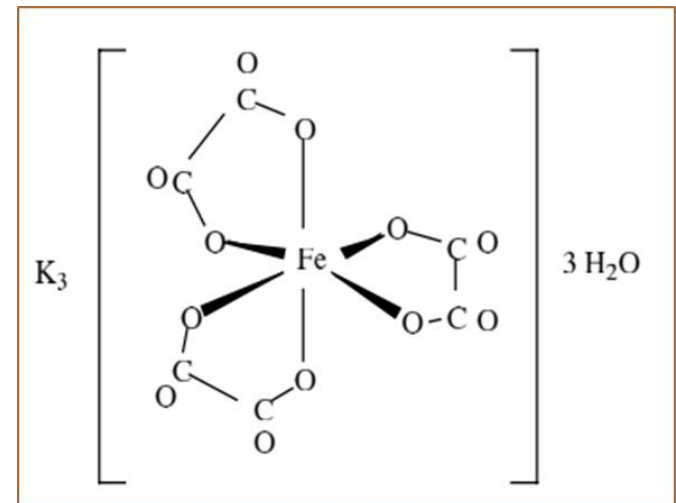
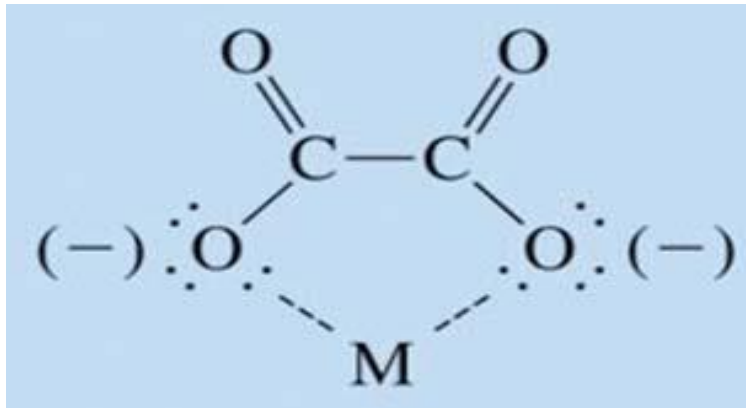
◀ Cl 이 리간드로 작용한 예

Theory

한 자리 리간드



여러 자리 리간드 (킬레이트 리간드)



Theory

전이금속(transition metal)

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LANTHANIDES

58 Ce	59 Pr	60 Nd	61 Pm	62 Sm	63 Eu	64 Gd	65 Tb	66 Dy	67 Ho	68 Er	69 Tm	70 Yb	71 Lu
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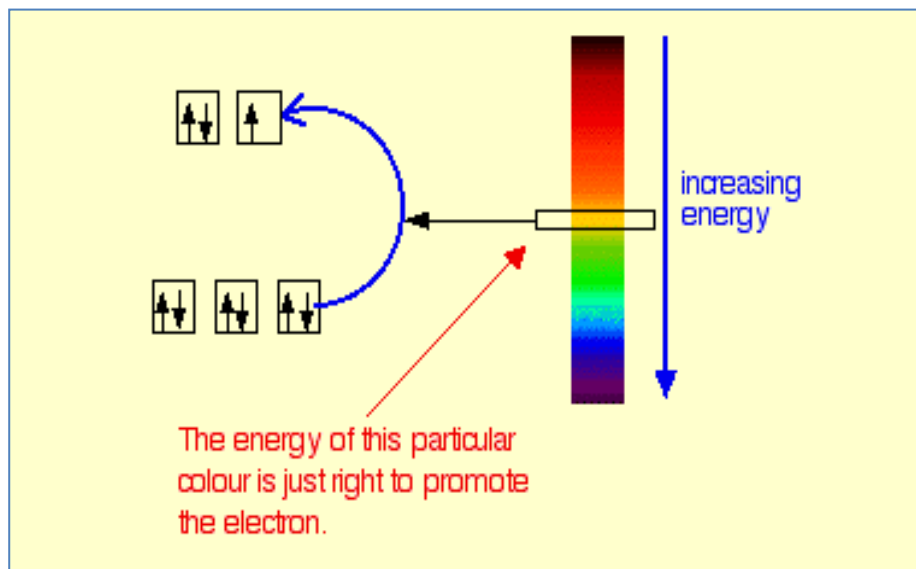
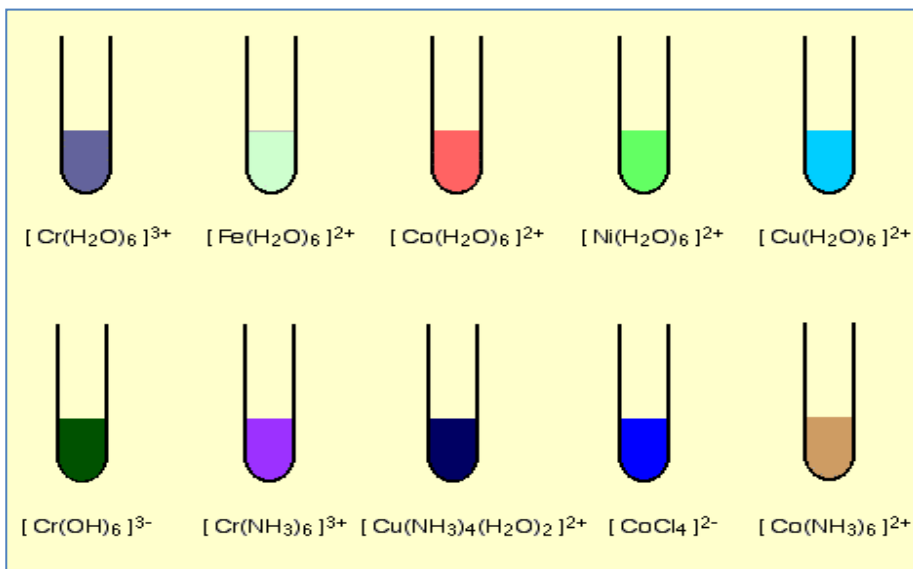
ACTINIDES

90 Th	91 Pa	92 U	93 Np	94 Pu	95 Am	96 Cm	97 Bk	98 Cf	99 Es	100 Fm	101 Md	102 No	103 Lr
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족 →	3	4	5	6	7	8	9	10	11	12
주기 ↓										
4	21 Sc	22 Ti	23 V	24 Cr	25 Mn	26 Fe	27 Co	28 Ni	29 Cu	30 Zn
5	39 Y	40 Zr	41 Nb	42 Mo	43 Tc	44 Ru	45 Rh	46 Pd	47 Ag	48 Cd
6	71 Lu	72 Hf	73 Ta	74 W	75 Re	76 Os	77 Ir	78 Pt	79 Au	80 Hg
7	103 Lr	104 Rf	105 Db	106 Sg	107 Bh	108 Hs	109 Mt	110 Ds	111 Rg	112 Cn

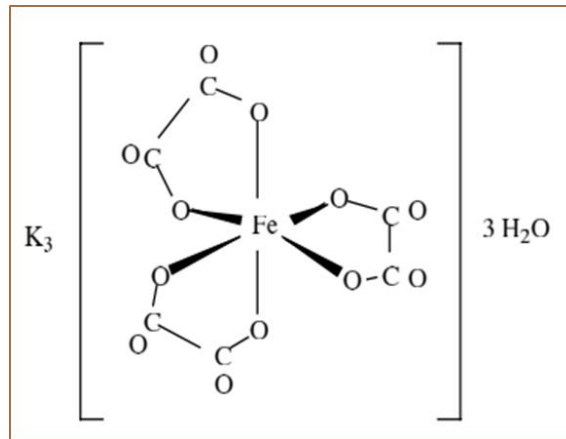
Theory

전이금속 특징

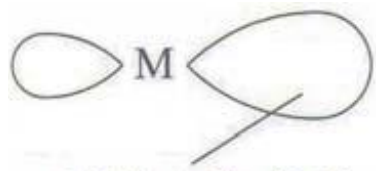


Theory

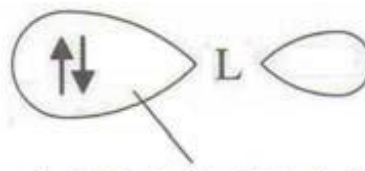
착화합물 (Complex Compound)



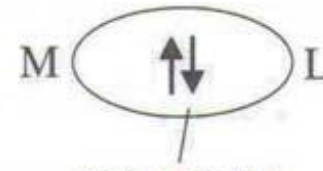
착이온의 구조



비어있는 금속 이온의
혼성 원자 궤도함수



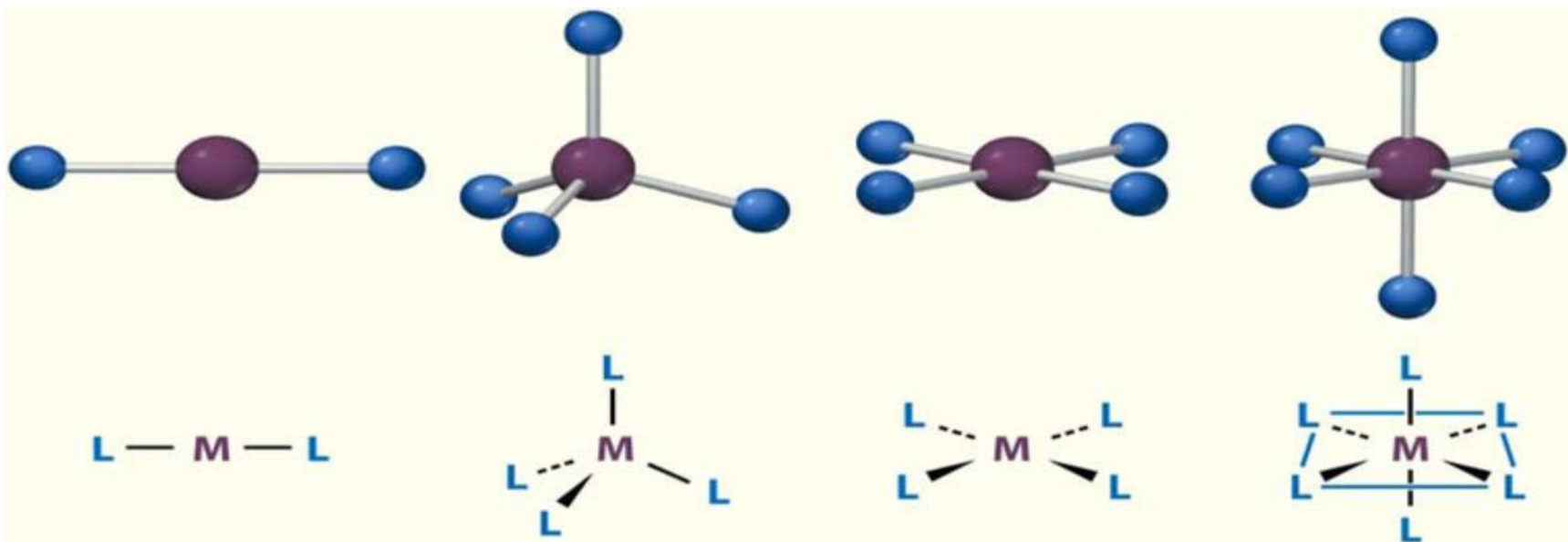
혼성 원자 궤도함수에 있는
리간드의 비공유 전자쌍



배위 공유 결합

Theory

[금속착물의 모양]



선형
(두 개 배위)
 sp

사면체형
(네 개 배위)
 sp^3

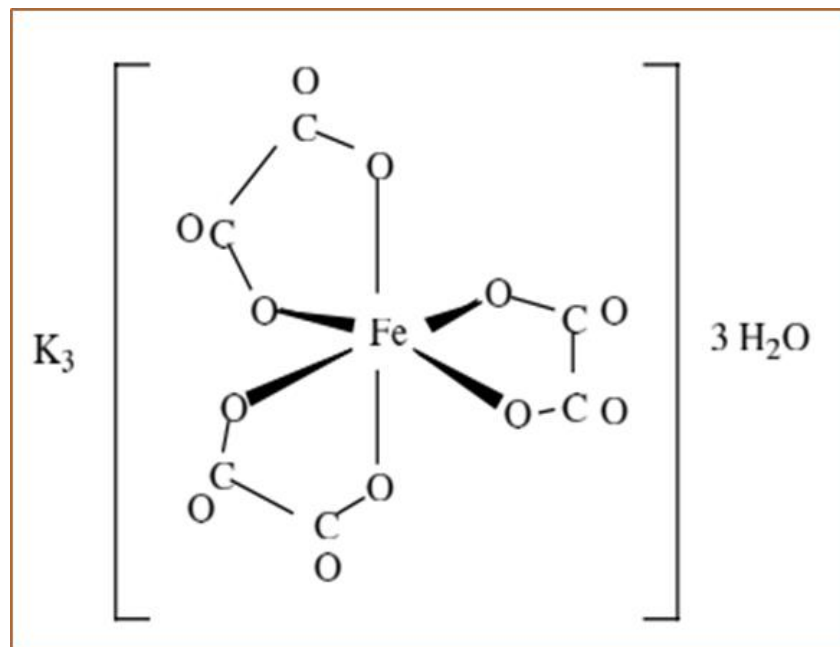
사각평면
(네 개 배위)
 dsp^2

팔면체형
(여섯 개 배위)
 d^2sp^3 or sp^3d^2

Theory

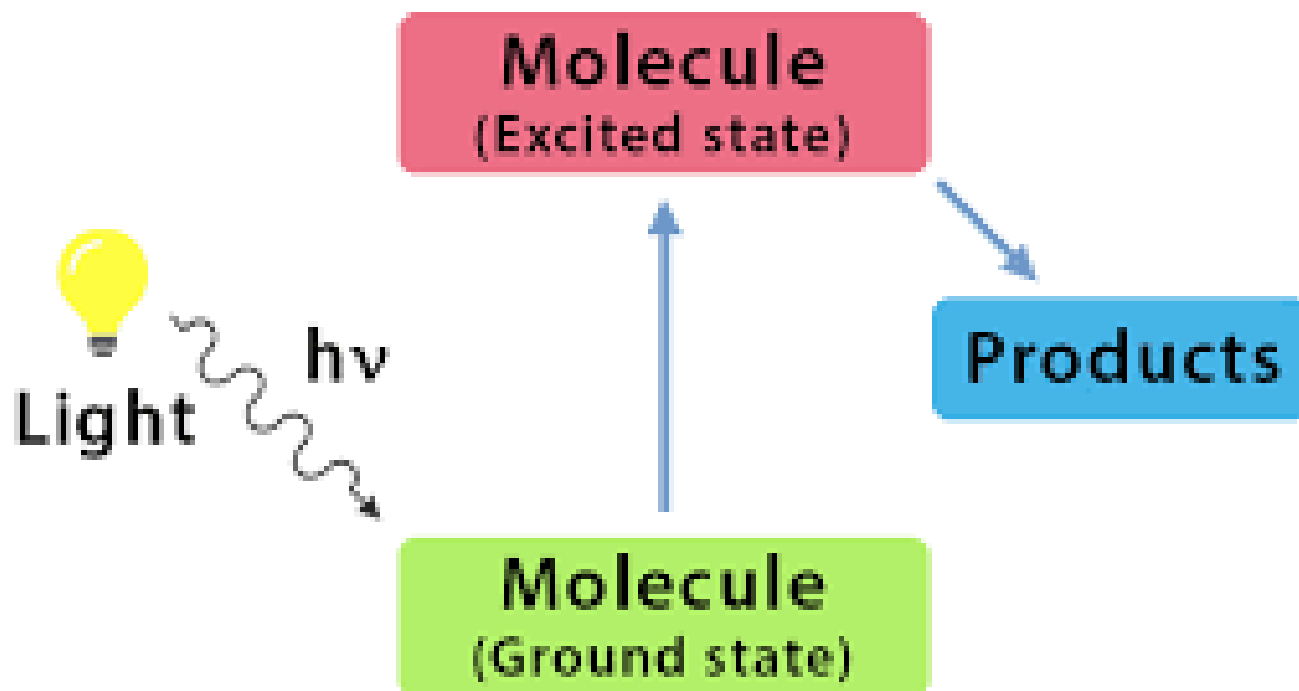


$\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3]$ 착화합물



Theory

광화학반응(photochemical reaction)

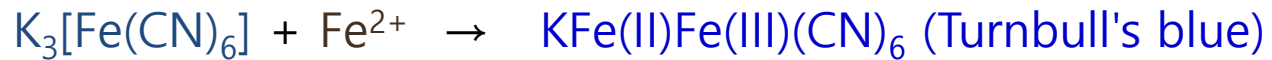


Theory

광화학반응 메커니즘



철 - 옥살레이트 착화합물의 광화학반응 (청사진)



Chemicals & Apparatus

Apparatus

저울, 오븐, Hot plate, 비커, 눈금실린더, 삼각플라스크, Pipette, 시계접시(또는 Petri Dish), 시험관: 3개, Spatula, 유리막대, 뷰흐너 깔때기, aspirator, 여과지, 은박지

Chemicals:

- $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$: Potassium oxalate monohydrate
- $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$: Iron(III) chloride hexahydrate
- 2M H_2SO_4 : sulfuric acid
- 0.1M $\text{K}_3\text{Fe}(\text{CN})_6$: Potassium ferricyanide(III)

Procedure

실험 A $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ 의 합성

실험 B $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ 의 광반응

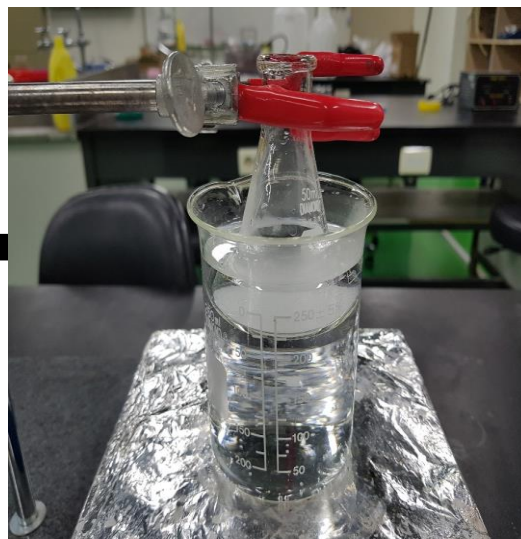
실험 C $\text{K}_3[\text{Fe}(\text{C}_2\text{O}_4)_3] \cdot 3\text{H}_2\text{O}$ 청사진

Procedure

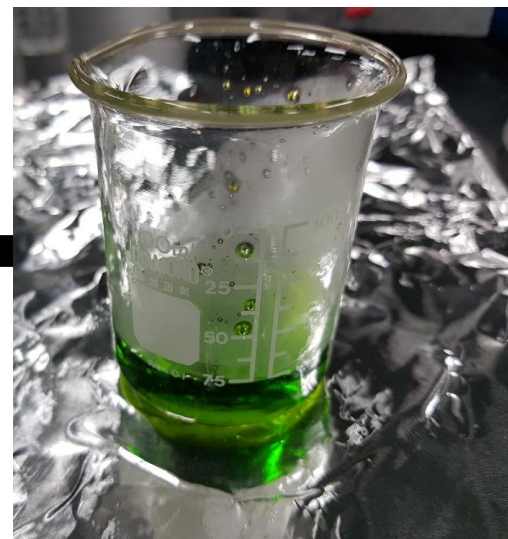
실험 A $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ 의 합성



① (100ml 비커) 4.4 g의 $FeCl_3 \cdot 6H_2O$ 의 무게를 측정해서 넣고 5~6mL의 찬 증류수를 넣어 녹인다.



② (50ml 삼각플라스크) 9g의 $K_2C_2O_4 \cdot H_2O$ 의 무게를 측정해서, 15 mL의 증류수를 넣고 녹을때 까지 물중탕 하면서 가열한다.

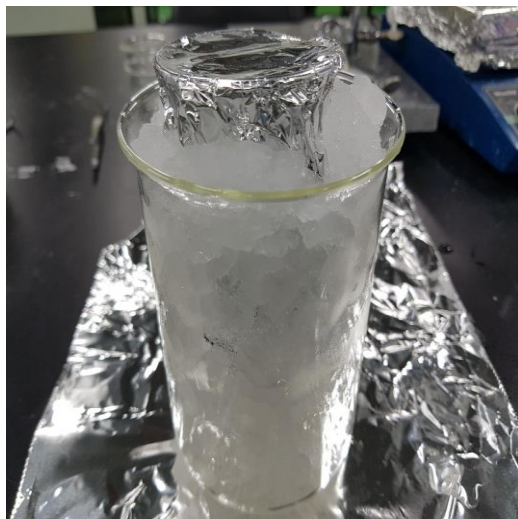


③ $FeCl_3 \cdot 6H_2O$ 의 용액에, 뜨거운 $K_2C_2O_4 \cdot H_2O$ 를 가하고, 녹여준다.
(노란색 -> 초록색)

①, ② : stirring bar 사용하면 잘 녹음.

Procedure

실험 A $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ 의 합성



④ 은박지로 싸서 빛을 차단하고 얼음(또는 찬물)에 비커를 두어 결정이 생길 때까지 기다린다.
(15분 이상)



⑤ 결정화된 생성물을 감압 장치를 이용하여 거르고, 침전물을 **아세톤**으로 씻어서 건조한다. (유리막대로 긁어 모은다)



⑥ 잘 건조 후(오븐), 무게를 측정후 **% 수득률**을 구한다.
바로 다음 실험(실험B)을 수행한다 (**연두색**)
→ 여과지 무게 미리 잴 것!

Procedure

실험 B $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ 의 광반응



① $0.7g$ 의 $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ (실험A제조)을 삼각플라스크 (250ml)에 넣고 100mL 증류수와 3mL의 2M H_2SO_4 를 첨가하고 잘 섞는다. → 은박지로 차단 한 후 황산첨가.

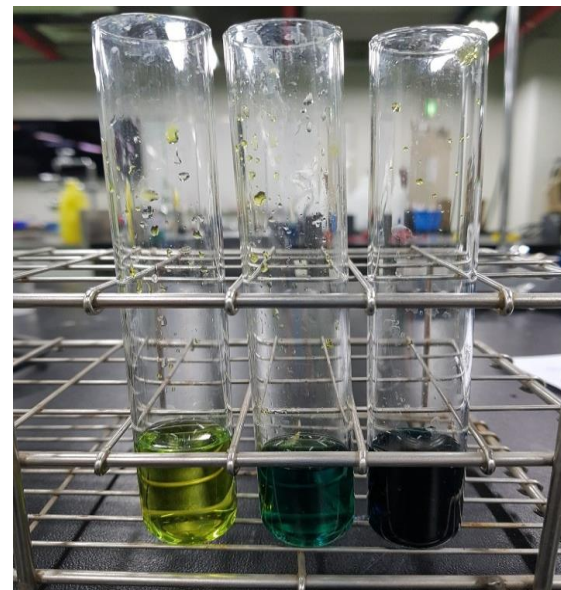


② 가,나,다 3개의 시험관을 준비해서 각각 ①에서 만든 용액 10ml씩을 넣는다.

가 : 은박지로 바로 빛을 차단

나 : 2분간 빛을 쬔 후 은박지로 빛을 차단 (휴대폰 사용)

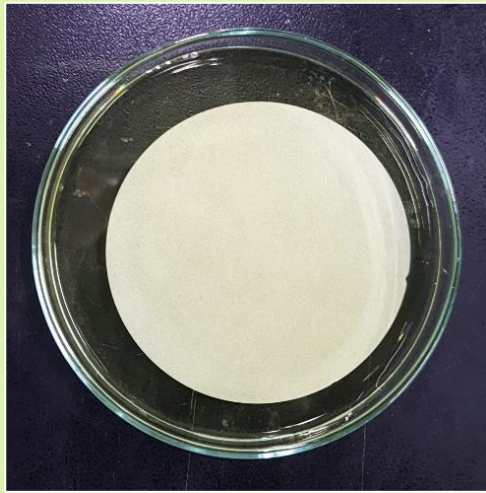
다 : 10분간 빛을 쬔 후 은박지로 빛을 차단 (휴대폰 사용)



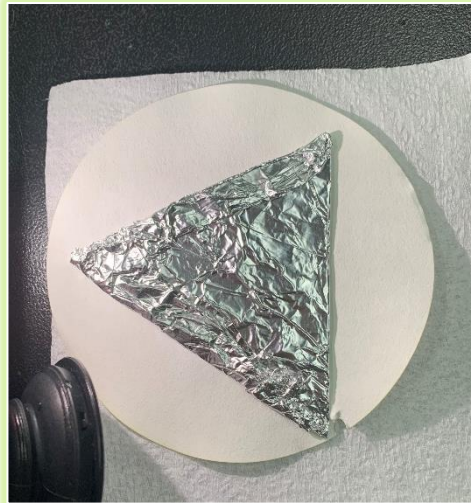
③ 각 시험관에 1ml 썬의 $0.1M K_3Fe(CN)_6$ 용액을 가하고, 변화를 관찰한다. (변화한 3가지 시험관 사진촬영)

Procedure

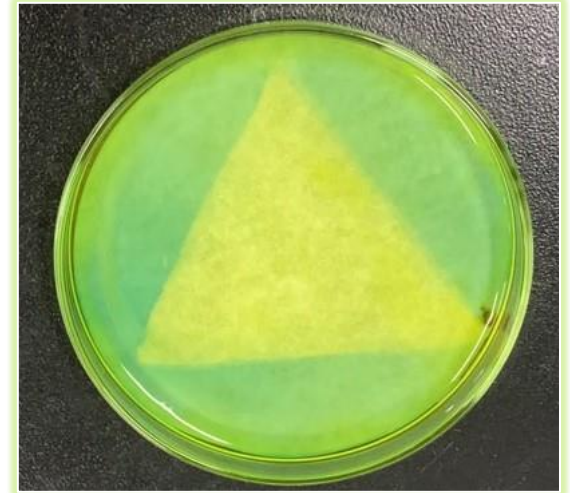
실험 C $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ 청사진



① 실험B 에서 이미 제조한 $K_3[Fe(C_2O_4)_3] \cdot 3H_2O$ 남은 용액을 시계접시(혹은 Petri dish)에 붓고, 이 용액에 여과지를 적신 후 오븐에서 말린다. (잘 마르지 않으면 드라이기 사용)



② 건조된 여과지 위에 원하는 모양으로 만들기 위해서 본인의 열쇠나 은박지를 씌운 물체를 올리고 그 위에서 빛(휴대폰)을 쬐인다. (10분 소요)



③ 건조한 여과지를 0.1M $K_3Fe(CN)_6$ 용액을 담아둔 시계접시 (Petri dish) 위에 놓어 이 용액으로 적신 후, 증류수로 씻는다. (노란색 부분은 씻김)

④ 관찰결과를 사진 촬영한다.