

The Preparation, Resolution, and Characterization of Tris(1,2-ethanediamine)iridium(III) Complexes

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$[\text{Ir}(\text{en})_3]^{3+}$ (en = 1,2-ethanediamine) is obtained in ca. 70 % yield as the double salt $2[\text{Ir}(\text{en})_3]\text{Cl}_3 \cdot \text{LiCl} \cdot 6\text{H}_2\text{O}$ by reaction of $\text{IrCl}_3 \cdot 4\text{H}_2\text{O}$ with an aqueous solution of en at 170 °C followed by recrystallization from LiCl-solution. The complex ion is resolved via the diastereoisomer $\text{Li}\{(-)[\text{Ir}(\text{en})_3]\}\{(+)\text{-tart}\}_2 \cdot 3\text{H}_2\text{O}$ [(+)-tart = (+)-tartrate anion]. The isomer $(+)[\text{Ir}(\text{en})_3]^{3+}$ is assigned the absolute configuration Δ by the method of active racemates. This assignment agrees with Werner's criterion of least soluble diastereoisomers. The compounds are characterized by their electronic and circular dichroism spectra and their optical rotation. $\text{Li}\{(+)\}_{589}[\text{Co}(\text{en})_3]\}\{(+)\text{-tart}\}_2 \cdot 3\text{H}_2\text{O}$ has also been prepared.

Tris(1,2-ethanediamine)iridium(III) bromide was first reported by Werner *et al.*¹ who prepared this compound in 17 % yield by the reaction of 1,2-ethanediamine(en) with $\text{Na}_3[\text{Ir}(\text{NO}_2)_4\text{Cl}_2] \cdot 2\text{H}_2\text{O}$ (170 °C for 10 h) together with *cis*- $[\text{Ir}(\text{en})_2(\text{NO}_2)_2]^+$ in 30 % yield. The $\text{Na}_3[\text{Ir}(\text{NO}_2)_4\text{Cl}_2] \cdot 2\text{H}_2\text{O}$ was prepared² in ca. 30 % yield from $\text{Na}_2[\text{IrCl}_6] \cdot 6\text{H}_2\text{O}$. Lebedinski³ prepared the iodide from $\text{Na}_3[\text{IrCl}_6] \cdot 12\text{H}_2\text{O}$ and en (140 °C for 15 h, no yield specified). Mathieu⁴ modified the method of Werner but did not specify the yield. Later Lebedinski's method was modified by Watt *et al.*⁵ to give 43 % yield of $[\text{Ir}(\text{en})_3]\text{I}_3$. The extremely soluble chloride has not been prepared directly before. In the present paper we describe a procedure which gives a ca. 70 % yield of the double salt $2[\text{Ir}(\text{en})_3]\text{Cl}_3 \cdot \text{LiCl} \cdot 6\text{H}_2\text{O}$.

Werner¹ resolved the tris(1,2-ethanediamine)iridium(III) ion with sodium-3-nitro-(+)-camphor (Nanic). However, the solubility of the precipitated diastereoisomer, $(-)[\text{Ir}(\text{en})_3](\text{nic})_3$, is so low that purification by recrystallization is impracticable, and furthermore some disagreement exists⁴ about

the amount of resolving agent to be used. By the present method $(-)[\text{Ir}(\text{en})_3]^{3+}$ is precipitated as the tartrate double salt $\text{Li}\{(-)[\text{Ir}(\text{en})_3]\}\{(+)\text{-tart}\}_2 \cdot 3\text{H}_2\text{O}$ which is then purified before conversion to the chloride. This method is analogous to the ones published earlier⁶ for the resolution of $[\text{Rh}(\text{en})_3]\text{Cl}_3$ and $[\text{Cr}(\text{en})_3]\text{Cl}_3$.

EXPERIMENTAL

Materials. Iridium(III) chloride hydrate (ca. 4 mol $\text{H}_2\text{O}/\text{mol Ir}$) was obtained from Johnson, Matthey and Co. and 1,2-ethanediamine (*puriss. p.a.*) from Fluka. All other chemicals were of analytical or reagent grade and were used without further purification.

Instrumentation. Absorption spectra were recorded on a Cary 118C spectrophotometer and circular dichroism on a Roussel-Jouan Dichrographe III. Optical rotation was measured using a Perkin-Elmer polarimeter 141 and X-ray powder diffraction photographs were taken with a Hägg-Guinier focusing camera XDC 700 calibrated with silicon and using $\text{CuK}\alpha$ radiation.

Synthetic Procedures

1. *Bis{tris(1,2-ethanediamine)iridium(III) chloride} lithium chloride, hexahydrate.* 3.5 ml of anhydrous 1,2-ethanediamine (52 mmol, 29 % excess) and 6 ml of water are mixed in a pyrex test tube, and carbon dioxide (which appears to catalyze the reaction) is bubbled through the solution over a period of 1 min. After 5 min, 5.0 g of $\text{IrCl}_3 \cdot 4\text{H}_2\text{O}$ (13.5 mmol) is added in 1 g portions. (It is sometimes necessary to moderate the reaction by cooling the tube under the water tap). The mixture is then heated cautiously to boiling and boiled for a few minutes.

The solution is reheated twice with a few minutes' interval. The test tube is then sealed and placed in an autoclave containing a little water. The autoclave is kept in an oven at 170 °C for 25 h and then allowed to cool to room temperature together with the oven. The ampoule is cooled in icewater and cautiously opened (slight overpressure) by melting off the top. The contents are diluted with 20 ml of water, and 0.5 g of active carbon is added. After standing for 1 h, the mixture is filtered (Whatman No. 50) and washed with water until the filtrate is colourless. The yellow filtrate is acidified with 12 M HCl (ca. 0.7 ml) and evaporated to ca. half volume on a rotating vacuum evaporator (RVE) to expel the carbon dioxide. Then LiOH.H₂O is added until the solution is basic (ca. 0.7 g), and the solution is evaporated to dryness on a RVE (final bath temperature 90 °C). The LiCl, excess ethanediamine and organic by-products are extracted by boiling with 100 ml of absolute ethanol. The resulting precipitate is filtered and washed with three 10 ml portions of absolute ethanol. The residue is dissolved in 20 ml of water by heating, 1 ml of 12 M HCl is added, and the acidic solution is evaporated to dryness on a RVE (final bath temperature 90 °C). LiCl is again removed by extraction with 100 ml of boiling absolute ethanol followed by filtration and washing with absolute ethanol. The light yellow residue (ca. 7 g) is dissolved in the minimum volume* (ca. 13 ml) of boiling half saturated LiCl-solution (23 g of LiCl in 100 ml of water) and allowed to stand overnight. The precipitate is filtered, washed successively with 1 ml portions of icewater, 50 % v/v ethanol, and 96 % ethanol, and dried in air. Yield 4.5 g (60 %) of 2[Ir(en)₃]Cl₃.LiCl.6H₂O. (Found: C 13.01; N 15.12; H 5.58; Cl 22.45. Calc. for LiIr₂C₁₂N₁₂H₆₆Cl₇O: C 13.01; N 15.17; H 5.46; Cl 22.39.)

A second crop of crystals is obtained in the following way: the combined filtrate and washings are evaporated to dryness on a RVE. LiCl is extracted with 100 ml of boiling absolute ethanol followed by filtration and washing with absolute ethanol. The residue is dissolved in the minimum volume (ca. 5 ml) of half saturated LiCl-solution and allowed to stand overnight. The precipitate is filtered, washed successively with 0.5 ml portions of icewater, 50 % v/v ethanol, and 96 % ethanol, and dried in air. Yield 0.7 g (9 %).

2. *Tris(1,2-ethanediamine)iridium(III) chloride, hydrate.* A column of 20 g of Dowex 50W X2 cation exchanger (H⁺-form, 200–400 mesh, ca. 20 cm long, and ca. 1.2 cm diameter) is washed with 50 ml of 4 M HCl followed by 5 ml of water. A solution of 3.0 g of 2[Ir(en)₃]Cl₃.LiCl.6H₂O in 100 ml of water

is applied and the column is washed with 50 ml of water before it is eluted with 80 ml of 0.2 M HCl. All these eluates are discharged. The complex ion is eluted with 80 ml of 4 M HCl followed by 5 ml of water. The eluate is evaporated to dryness on an RVE and the flask and solid residue (2[Ir(en)₃]Cl₃.HCl.6H₂O) are heated to 160 °C for 1 h to expel the hydrogen chloride. The residue is dissolved in 4 ml of water. The solution is filtered and the filter washed with another 1 ml of water. The filtrate is heated and ethanol (ca. 16 ml) is added with reflux until the solution starts to become turbid. The solution is allowed to stand for crystallization and the product is isolated by filtration, washed with two 2 ml portions of a mixture of 3 ml of ethanol and 1 ml of water and air-dried. Yield ca. 2.4 g (83 %) of [Ir(en)₃]Cl₃.aq.* (Found: C 13.53; N 15.62; H 5.42; Cl 19.94 i.e. C:N:H:Cl = 6.00:5.94:28.6:3.00.)

3. *Resolution of the tris(1,2-ethanediamine)iridium(III) ion.* A 2.80 g sample of 2[Ir(en)₃]Cl₃.LiCl.6H₂O (5.05 mmol), 1.15 g of (+)-tartaric acid (7.66 mmol), and 0.64 g of LiOH.H₂O (15.3 mmol) are placed in a 25 ml conical flask and dissolved in 5 ml of boiling water. A clear solution is formed and is allowed to cool by standing for 5 min. 2.5 ml of ethanol is added and the solution is reheated to boiling. The flask is stoppered and the solution is allowed to stand for about one day at room temperature with occasional shaking. The crystal cake is then broken up and allowed to stand for a further 2 h. The precipitate is filtered (the mother liquor is retained for later use), washed with three 1.5 ml portions of 96 % ethanol, and dried in air. Yield 1.85 g of crude Li{(-)[Ir(en)₃]}{(+)tart}₂.3H₂O.

The mother liquor is evaporated to dryness on an RVE (final bath temperature 90 °C). The residue is dissolved in 3 ml of 12 M HCl by heating to boiling, and 20 ml of absolute ethanol is added (a large white crystalline precipitate is formed). The mixture is heated to boiling, and the suspension is allowed to stand for two hours at room temperature before the precipitate is filtered, washed with three 1 ml portions of ethanol, and placed in an oven for 1 h at 160 °C to remove hydrogen chloride. The yield of crude (+)[Ir(en)₃]Cl₃ is 1.1 g. This product is dissolved in 1.1 ml of boiling water and the solution is allowed to stand for crystallization. If a small amount of precipitate (ca. 0.1 g) is not formed within one day, two drops of 96 % ethanol is added. After another 30 min the small precipitate which has formed is filtered off. To the filtrate is added 4 ml of 96 % ethanol, which immediately results in considerable precipitation. The mixture is allowed to stand for two hours before the precipitate is filtered, washed with three 0.5 ml portions

* [Ir(en)₃]Cl₃ is extremely soluble in water.

* The content of water of crystallization is somewhat variable.

of 96 % ethanol, and dried in air. Yield 0.85 g (63 %) of (+)[Ir(en)₃]Cl₃·2H₂O.* (Found: C 13.71; N 16.12; H 5.45; Cl 22.56; Li 0.39 i.e. C:N:H:Cl:Li=6.00:6.05:28.4:3.34:0.30.) λ (nm)/ $\Delta\epsilon_{\text{extr.}}$ (1 mol⁻¹ cm⁻¹): 271/-0.85, 253/-0.34, 232/-0.84.

The crude tartrate double salt is dissolved in 3.6 ml of boiling water and the solution is allowed to stand for five minutes before the addition of 1.8 ml of 96 % ethanol. The mixture is reheated to boiling and allowed to stand overnight for crystallization. After filtration the precipitate is washed twice with 0.5 ml portions of 50 % v/v ethanol, once with 1 ml of 96 % ethanol, and dried in air. Yield 1.6 g (87 %) of Li{(-)[Ir(en)₃]}{(+)-tart}₂·3H₂O (Found: C 22.91; N 11.62; H 5.38. Calc. for Li₂IrC₁₄N₆H₃₈O₁₅: C 23.05; N 11.52; H 5.25.)

The recrystallized tartrate double salt (1.6 g, 2.2 mmol) is dissolved in 1 ml of boiling 12 M HCl and 10 ml of absolute ethanol is added. A large white crystalline precipitate is formed. The mixture is heated to boiling, and the suspension is allowed to stand for 2 h. The precipitate is filtered, washed with three 1 ml portions of 96 % ethanol, and placed in an oven for one hour at 160 °C. The residue (1.0 g) is dissolved in the minimum volume (ca. 1 ml) of boiling water, after which four times this volume of 96 % ethanol is added. After two hours the precipitate is filtered, washed three times with 1 ml portions of ethanol, and dried in air. Yield 0.67 g (57 %) of (-)[Ir(en)₃]Cl₃·2H₂O.* (Found: C 13.70; N 16.16; H 5.40; Cl 21.79; Li 0.35 i.e. C:N:H:Cl:Li = 6.00:6.07:28.2:3.23:0.27.)

4. (+)₅₈₉-Tris(1,2-ethanediamine)cobalt(III) chloride, monohydrate. (+)₅₈₉-Tris(1,2-ethanediamine)cobalt(III) chloride *d*-tartrate, pentahydrate was prepared by the partial asymmetric synthesis of Broomhead *et al.*⁷ and recrystallized twice from hot water. After filtration the crystals are washed with 40 % ethanol and dried in air. λ (nm)/ $\Delta\epsilon_{\text{extr.}}$ (1 mol⁻¹ cm⁻¹): 490/1.82, 432/-0.25, 350/0.26.

A 40.0 g sample of this product (78 mmol) is dissolved in 20 ml of hot 12 M HCl. 250 ml of absolute ethanol is added dropwise to the solution with stirring. The precipitate is filtered, washed with three 30 ml portions of ethanol, and air-dried. The crude product is dissolved in 12 ml of hot water. On cooling to room temperature under stirring some crystals separate. Under continuous stirring 70 ml of absolute ethanol is then added dropwise to the mixture. After filtration the crystals are washed with three 20 ml portions of ethanol and dried in air. Yield 23.6 g (83 %) of (+)₅₈₉[Co(en)₃]Cl₃·H₂O (Found: C 19.67; N 23.17; H 7.36; Cl 29.25. Calc. for CoC₆N₆H₂₆Cl₃O: C 19.82; N 23.11; H

7.21; Cl 29.25.) λ (nm)/ $\Delta\epsilon_{\text{extr.}}$ (1 mol⁻¹ cm⁻¹): 488/1.93, 428/-0.14, 349/0.25.

5. *Active racemate*. A 1.0907 g sample of (+)₅₈₉[Co(en)₃]Cl₃·H₂O (3.00 mmol) and 1.5406 g of (+)[Ir(en)₃]Cl₃·2H₂O (2.93 mmol) are each dissolved in the minimum amount of water (ca. 1 ml) at room temperature. The two solutions are heated to 50 °C, mixed, and allowed to stand for 6 h for crystallization. After filtration the crystals are washed with 0.5 ml of ice-cold water, 0.5 ml of 50 % v/v ethanol, and 1 ml of ethanol and dried in air. Yield 1.2 g (ca. 40 %) of {(+)₅₈₉[Co(en)₃]}{(+)-[Ir(en)₃]}Cl₆·6H₂O.* (Found: C 15.15; N 17.35; H 6.53; Cl 24.43; Co 6.16; Li 0.31 i.e. C:N:H:Cl:Co:Li = 12.00:11.78:61.6:6.56:0.994:0.43.)

6. *Lithium (+)₅₈₉-tris(1,2-ethanediamine)cobalt(III) (+)-tartrate, trihydrate*. [Co(en)₃]Cl₃ was prepared by the method of Work⁸ and recrystallized from water. 10.0 g of the recrystallized product (25 mmol), 5.64 g of (+)-tartaric acid (38 mmol) and 3.16 g of LiOH·H₂O are dissolved in 25 ml of boiling water and the solution is allowed to stand overnight with occasional shaking until the crystals start to separate. The crystal cake is broken up. The crystals are filtered, washed with three 10

* The product is contaminated with LiCl.

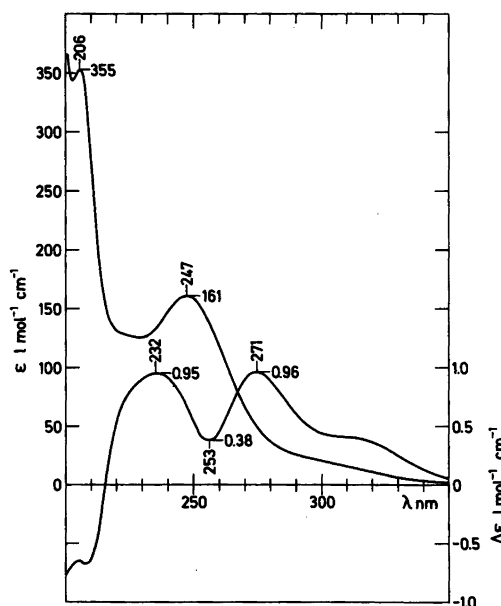


Fig. 1. The absorption spectrum of 2[Ir(en)₃]Cl₃·LiCl·6H₂O and the circular dichroism spectrum of (-)[Ir(en)₃]Cl₃·2H₂O.

* The product is contaminated with LiCl. If necessary it can be purified by the ion exchange procedure described in section 2.

Table 1. Unit cell dimensions determined from X-ray powder photographs. All crystals are indexed as belonging to the hexagonal crystal system.

Compound	<i>a</i> , Å	<i>c</i> , Å	<i>V</i> , Å ³
[Co(en) ₃]Cl ₃ .aq ⁹	11.44 ₃	15.48 ₃	1756
{(+) ₅₈₉ [Co(en) ₃]}{(+)[Ir(en) ₃]}Cl ₆ .aq	11.56 ₂	15.51 ₁	1796
[Ir(en) ₃]Cl ₃ .aq	11.63 ₆	15.42 ₈	1809

ml portions of 40% ethanol and dried in air. The crude product (5.1 g) is dissolved in 30 ml of boiling water. 5 g of LiCl is added and the solution is filtered. To the hot filtrate 20 ml of ethanol is added with stirring. After stirring for a few hours the precipitate is filtered, washed with three 5 ml portions of 40% ethanol, and dried in air. Yield: 3.0 g (40%) of Li{(+)₅₈₉[Co(en)₃]}{(+)-tart}₂.3H₂O (Found: C 27.83; N 14.27; H 6.81; Co 9.89. Calc. for LiCoC₁₄N₆H₃₈O₁₅: C 28.20; N 14.09; H 6.42; Co 9.88.) λ(nm)/Δε_{ext.} (1 mol⁻¹ cm⁻¹): 491/1.80, 434/-0.31, 348/0.27.

RESULTS

The isomer (+)[Ir(en)₃]³⁺ is assigned the absolute configuration Δ, using the method of active racemates,⁹ by correlation with Λ(+)₅₈₉-[Co(en)₃]³⁺ the absolute configuration of which has been determined¹⁰ by X-ray single crystal methods: the racemic compounds [Ir(en)₃]Cl₃.aq and [Co(en)₃]Cl₃.aq and the active racemate {(+)₅₈₉-[Co(en)₃]}{(+)[Ir(en)₃]}Cl₆.aq have almost identical X-ray powder photographs (Table 1), differing from those of the optically active compounds.* This

* (+)[Co(en)₃]Cl₃.aq is tetragonal (*a*=9.67₆ Å, *c*=16.29₅ Å) and (+)[Ir(en)₃]Cl₃.aq is cubic (*a*=21.71₃ Å) like the rhodium analog (*a*=21.70₅ Å, Ref. 11 *a*=21.67₅ Å, space group F4₁32).

assignment agrees with Werner's criterion of least soluble diastereoisomers.¹² The least soluble diastereoisomers Li{act[M(en)₃]}{(+)-tart}₂.3H₂O (M=Cr⁶, Co, Rh⁶, and Ir) all have the absolute configuration Λ. The members of this series are isomorphous (Table 2).* The Co and Ir compounds have not been described before.

The molar rotations are given in Table 3. With the exception of the two lowest wavelengths the results for (-)[Ir(en)₃]Cl₃.2H₂O are in agreement with the values published for the bromide.⁴

The absorption spectrum of 2[Ir(en)₃]Cl₃.LiCl.6H₂O is shown in Fig. 1. The ¹A_{1g}→³T_{1g}(O_h) transition is seen at ca. 310 nm as a "foot" on the low energy side of the ¹A_{1g}→¹T_{1g}(O_h) band and the ¹A_{1g}→¹T_{2g}(O_h) transition appears as a shoulder at ca. 225 nm. The spectrum is in accordance with the results published by Jørgensen¹⁴ and by DeArmond and Hillis.¹⁵ Fig. 1 also shows the circular dichroism (CD) spectrum of Λ(-)[Ir(en)₃]Cl₃.2H₂O. The contour differs from that of the CD spectra of the corresponding Rh and Co complexes in that it is all positive in the *d-d* transition range. A comparison with the CD spectrum¹⁶ of Δ(+)[Ir{(-)chxn}₃]-λλλ³⁺ reveals that the transitions to the E(D₃) and A₂(D₃) excited levels of T_{1g}(O_h) parentage and the

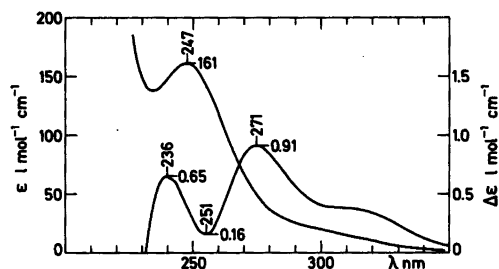
* Kushi *et al.*¹³ have published the crystal structure of the Cr compound (*a*=7.86 Å, *b*=16.99 Å, *c*=9.22 Å, β=100.4°, space group P2₁).

Table 2. Unit cell dimensions determined from X-ray powder photographs. All crystals belong to the monoclinic crystal system and the indexing of all powder photographs is very similar. The chromium compound belongs¹³ to space group P2₁.

Compound	<i>a</i> , Å	<i>b</i> , Å	<i>c</i> , Å	β, °	<i>V</i> , Å ³
Li{(+) ₅₈₉ [Cr(en) ₃]}{(+)-tart} ₂ .3H ₂ O	7.85 ₅	16.98 ₁	9.22 ₂	100.2 ₇	1210
Li{(+) ₅₈₉ [Co(en) ₃]}{(+)-tart} ₂ .3H ₂ O	7.83 ₈	16.89 ₄	9.21 ₆	100.5 ₅	1200
Li{(-) ₅₈₉ [Rh(en) ₃]}{(+)-tart} ₂ .3H ₂ O	7.87 ₁	16.99 ₂	9.21 ₂	100.4 ₆	1212
Li{(-) ₅₈₉ [Ir(en) ₃]}{(+)-tart} ₂ .3H ₂ O	7.87 ₇	17.00 ₇	9.20 ₂	100.5 ₇	1212

Table 3. Molar rotations ($[M] = (100\alpha)/(cl)$ deg. l mol⁻¹ cm⁻¹) in aqueous solution at 25 °C.

	313 nm	364 nm	436 nm	546 nm	578 nm	589 nm
Li{(-)[Ir(en) ₃]}{(+)tart ₂ }.3H ₂ O; $c \approx 2.7 \times 10^{-3}$ M	-672	-383	-350	-220	-197	-182
(-)[Ir(en) ₃]Cl ₃ .2H ₂ O; $c \approx 3.8 \times 10^{-3}$ M	-983	-678	-577	-367	-327	-311
(+)[Ir(en) ₃]Cl ₃ .2H ₂ O; $c \approx 4.2 \times 10^{-3}$ M	907	616	525	333	298	287

Fig. 2. The absorption and circular dichroism spectra of Li{(-)[Ir(en)₃]}{(+)tart₂}.3H₂O.

transition to the $E(D_3)$ excited level of $T_{2g}(O_h)$ parentage are all situated ca. 2000 cm⁻¹ lower in energy.

Fig. 2 shows the absorption and CD spectra of Li{(-)[Ir(en)₃]}{(+)tart₂}.3H₂O. The low wavelength part of the CD spectrum has a lower intensity than that found for the chloride and the band at ca. 235 nm has narrowed considerably. This is due partly to a narrow CD band of the (+)-tartrate ion appearing at 216 nm ($\Delta\epsilon = 2.2$ l mol⁻¹ cm⁻¹) and partly to ion-pairing effects.

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