there was obtained 0.228 g. (47% yield) of pale yellow crystals, m.p. 88–92°. Recrystallization from a small amount of methanol gave small colorless prisms, m.p. 91.5–92.5° (reported[10], 94°), undepressed on admixture with authentic N-acetyl-4-keto-1,2,3,4-tetrahydroquinoline prepared from the free quinolone.[11]

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(10) G. R. Clemo and H. J. Johnson, *J. Chem. Soc.*, 2133 (1930).

(11) W. S. Johnson, E. L. Woroch and B. G. Buell, THIS JOURNAL, **71**, 1901 (1949).

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The Solubility of Lead Chloride in Sodium Chloride, Perchloric Acid and Hydrochloric Acid Solutions[1]

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In connection with an investigation of the crystal growth of lead chloride, measurements were made of the solubility of that compound at 25° in the media listed in Table I.

TABLE I

SOLUBILITIES OF $PbCl_2$ AT 25°

Medium (conc. in molality)	G. $PbCl_2$ per 1000 g. H_2O	Standard deviation	Lit. values (interpolated)
0.4723 *m* NaCl	1.870	0.008	1.87[a]
1.0125 *m* NaCl	1.784	.008	1.74[a]
2.0265 *m* NaCl	2.743	.010	2.72[a]
4.0216 *m* NaCl	8.367	.014	8.55[a]
0.5375 *m* HCl	1.561	.007	(3.68 g. at 0.50 *m*)[g]
0.5487 *m* $HClO_4$	10.88	.04	
1.0368 *m* $HClO_4$	10.56	.05	
2.3239 *m* $HClO_4$	7.421	.025	
3.4565 *m* $HClO_4$	5.183	.013	
6.4730 *m* $HClO_4$	1.781	.005	
Water	....	...	10.87[a], 10.84[b], 10.86[c], 10.75[d], 10.91[e], 10.76[f], 11.03[g], 10.84[h]

[a] G. E. R. Deacon, *J. Chem. Soc.*, 2063 (1927). [b] L. J. Burrage, *ibid.*, 1703 (1926). [c] W. R. Carmody, THIS JOURNAL, **51**, 2909 (1929). [d] T. P. Goulden and L. M. Hill, *J. Chem. Soc.*, 447 (1945). [e] L. Wilkinson, N. O. Bathhurst and H. N. Parton, *Trans. Faraday Soc.*, **33**, 623 (1937). [f] F. Flöttman, *Z. anal. Chem.*, **73**, 1 (1938). [g] H. E. Armstrong and J. V. Eyre, *Proc. Roy. Soc.* (*London*), **A88**, 238 (1913). [h] P. M. Lichty, THIS JOURNAL, **25**, 469 (1903).

Sealed glass tubes containing solution and excess solid $PbCl_2$ were heated to about 100° for several hours, then placed into a thermostat and rotated at 25.00 ± 0.05° for at least 24 hours, after which samples were removed for analysis by suction through a plug of glass wool.

The concentration of acid was determined in each case by titration with standard NaOH to the brom cresol green end-point. Two procedures were used for the determination of lead chloride; (1) triple evaporation with sulfuric acid, followed by dilution with water and the usual gravimetric procedure with filtration on Selas crucibles, and (2) neutralization to the methyl orange end-point with ammonia, precipitation in hot solution with hydrogen sulfide, followed by solution of the precipitate in nitric acid and conversion to lead sulfate as before. Both procedures were followed in the case of the sodium chloride and hydrochloric acid solutions, and gave concordant results; only procedure (2) was used for the solutions containing perchloric acid. The concentration of sodium chloride was found as the difference between total chloride as obtained by the Volhard titration[2] and the amount of chloride as lead chloride calculated from the gravimetric determination of lead.

Demassieux[3] and Deacon[4] have reported that no double salts are formed between NaCl and $PbCl_2$, whereas Kendall and Sloan[5] offered analytical evidence that the solid phase in equilibrium with NaCl solutions of $PbCl_2$ is $NaCl{\cdot}2PbCl_2$ for all concentrations in excess of 0.5 *N* NaCl. The solid phases in equilibrium with the 1, 2 and 4 *m* NaCl solutions of Table I were analyzed for lead according to procedure (1) above. The solid was removed from the liquid phase by filtration, and dried by pressing between filter papers without washing. The analytical results are given in Table II.

TABLE II

ANALYSES OF SOLID SAMPLES

Source of sample	$PbCl_2$, %
Mallinckrodt A.R. $PbCl_2$	100.16, 99.84, 99.96
1 *m* NaCl	98.36, 99.17
2 *m* NaCl	99.16, 98.05
4 *m* NaCl	98.13, 99.21

The analyses of the samples from the NaCl solutions are low as a result of contamination by NaCl, but the data clearly support the conclusion that no double salts form between NaCl and $PbCl_2$ for solutions up to 4 *m* in NaCl under the conditions of our experiments.

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[1] This work was supported in part under Contract No. AT (30-1)-1256 between the Atomic Energy Commission and New York University.

(2) I. M. Kolthoff and E. B. Sandell, "Textbook of Quantitative Inorganic Analysis," 3rd Ed., The Macmillan Co., New York, N. Y., 1952, p. 546, procedure 2.

(3) N. Demassieux, *Ann. chim.*, **20**, 267 (1923).

(4) G. E. R. Deacon, footnote *a* of Table I.

(5) J. Kendall and C. H. Sloan, THIS JOURNAL, **47**, 2306 (1925).

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Anion-exchange Studies. VII.[1,2] Separation of Sulfuric Acid from Metal Sulfates by Anion Exchange

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A strong base anion-exchange resin in the sulfate form (or in combination with other polyvalent anions) might be considered a base which can react with (adsorb) acids according to reactions of the type

$$(SO_4^{=})_r + HX \rightleftharpoons (HSO_4^-)_r + (X^-)_r \qquad (1)$$

where subscript r stands for resin. In the case of the adsorption of sulfuric acid by a sulfate resin equation (1) becomes

$$(SO_4^{=})_r + H_3O^+ + HSO_4^- \rightleftharpoons 2(HSO_4^-)_r + H_2O \qquad (2a)$$

and

$$(SO_4^{=})_r + 2H_3O^+ + SO_4^{=} \rightleftharpoons 2(HSO_4^-)_r + 2H_2O \qquad (2b)$$

The fact that such acid adsorption, possibly according to equations (1) and (2), actually takes place can readily be demonstrated by passing sulfuric acid solutions through sulfate columns, and noting that a considerable volume of effluent is free of acid.

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(1) This document is based on work performed for the Atomic Energy Commission at the Oak Ridge National Laboratory.

(2) Previous paper, K. A. Kraus and G. E. Moore, THIS JOURNAL, **75**, 1460 (1953).

(3) Summer Participant (1951) at Oak Ridge National Laboratory.