Anal. Calcd for $C_{27}H_{31}ClN_2O_2$: C, 71.90; H, 6.93; Cl, 7.86; N, 6.21; mol wt, 451. Found: C, 71.82; H, 7.08; Cl, 10.24; N, 6.25; mol wt, 345.

12(13)-Bromo-3,3,8,8-tetramethyl[10]paracyclophan-5-one (Vb).—A mixture of 25 ml of carbon tetrachloride, 0.286 g (1 mmole) of the ketone Va, and a crystal of iodine was placed in a black flask and stirred for 15 min. A solution of 0.160 g (1 mmole) of bromine in 15 ml of carbon tetrachloride was added over a 5-min period and the entire mixture was stirred for 22 hr. The solution was poured into 100 ml of 1 *M* sodium bisulfite solution and stirred until colorless. The organic layer was washed with cold water, dried, and the solvent was evaporated, leaving a white solid, mp 150–155°. Three recrystallizations (ethanol–water) gave 0.300 g (84%) of the bromo ketone as white needles, mp 169–168°.

Anal. Calcd for $C_{20}H_{29}BrO$: C, 65.75; H, 8.00; Br, 21.87; mol wt, 365. Found: C, 65.97; H, 8.16; Br, 22.14; mol wt, 367.

The usual tests[25] supported the fact that the halogen was in the aromatic nucleus.

3,3,8,8-Tetramethyl-5-oxo[10]paracyclophane-12(13)-sulfonamide (Vc).—A solution of 0.50 g (1.7 mmoles) of the ketone Va in 2 ml of anhydrous chloroform was cooled to 0° and 10 ml of chlorosulfonic acid added all at once. The reaction mixture was allowed to warm to room temperature over the course of 1 hr, then poured over ice, and finally extracted with chloroform. The chloroform solution was placed in an erlenmeyer flask, 20 ml of concentrated ammonium hydroxide was added, and the entire mixture was stirred at 50° for 4 hr. The dried chloroform layer was evaporated leaving a tan solid, mp 165–175°. Three recrystallizations [petroleum ether (bp 90–100°)–benzene] gave a light tan solid, mp 174.5–175.5°.

Anal. Calcd for $C_{20}H_{31}NO_3S$: C, 65.71; H, 8.55; N, 3.83; S, 8.77. Found: C, 65.96; H, 8.55; N, 3.70; S, 8.75.

Attempts to form the **semioxamazone derivative** by the method Leonard and Boyer[11] using *d*-5-(α-phenylethyl)semioxamazide failed to yield a derivative.

Registry No.—Va, 10197-52-1; 2,4-dinitrophenylhydrazone of Va, 10197-53-2; Vb 11 isomer, 10235-62-8; Vb 12 isomer, 10197-54-3; Vc 12 isomer, 10197-55-4; Vc 11 isomer, 10239-63-1; VIa, 10197-56-5; VIb, 10197-57-6; VIc, 10197-58-7; VIIa, 10197-59-8; VIIIa, 10197-60-1; VIIIc 12 isomer, 10197-61-2; VIIIc 11 isomer, 10197-62-3; 3,3,8,8-tetramethyl[10]parcyclophan-5-one azine, 10197-63-4.

Nucleotides. VII.[1] Preparation and Optical Rotatory Dispersion of Some 9β-D-Ribofuranosyl-3,5′-purine Cyclonucleosides[2]

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Cyclization of purine and pyrimidine ribonucleosides to 3,5′- and 2,2′-cyclonucleosides, respectively, has until recently been restricted to intramolecular alkylation involving 5′- or 2′-tosyl or -iodo derivatives of the nucleosides.[4] However, Fox, *et al.*,[5] have prepared 2,2′-anhydro-1-β-D-arabinofuranosyluracil directly by treatment of uridine with thiocarbonyldiimidazole. A similar reaction between uridine and diphenyl carbonate is also useful for production of the same 2,2′-cyclonucleoside.[6] Diphenyl carbonate converts adenosine and inosine to the 2′,3′ cyclic carbonates[6] and under appropriate conditions can also convert inosine to bis(inosine 5′-)carbonate.[1] The present communication describes the reactions of this reagent with other nucleosides. An interesting finding was that xanthosine can be smoothly converted, *via* a 2′,3′-carbonate, to 3,5′-cycloxanthosine.

The preparation of 2,2′-anhydro-1-β-D-arabinofuranosylcytosine by treatment of cytidine with polyphosphoric acid has been described.[7] Treatment of $N^4,O^{3'},O^{5'}$-triacetylcytidine with *p*-toluenesulfonyl chloride yields $N^4,O^{3'},O^{5'}$-triacetyl-1-β-D-arabinofuranosylcytosine, presumably through the corresponding 2,2′-cyclonucleoside.[8] Attempts to prepare a 2,2′-cyclonucleoside by reaction of cytidine, 5′-O-tritylcytidine[9] or N^4-benzoylcytidine[10] with diphenyl carbonate yielded mixtures of at least six products.[11] Similar results were obtained when the above compounds were treated with *p*-nitrophenoxycarbonyl chloride in pyridine.

Reaction of xanthosine (**1**) with diphenyl carbonate yielded a product which analyzed as the 2′,3′-carbonate of 3,5′-cycloxanthosine (**2**). In accord with the proposed structure the compound did not react with periodate in the manner of a *cis*-α-glycol, but showed carbonyl absorption in the region 1830 cm^{-1} typical of organic five-membered cyclic carbonates.[12] On mild alkaline hydrolysis a compound was obtained which showed properties identical with 3,5′-cycloxanthosine (**3**) recently prepared[13] from 2′,3′-O-isopropylidene-3,5′-cycloguanosine by successive alkaline and acidic treatments. Diazotization of 2′,3′-O-isopropylidene-3,5′-cycloguanosine (**4**) followed by deblocking produced the same cycloxanthosine. The conversion of **4** to **3** occurred in low over-all yield,[14] but served to add to the already substantial evidence[13] that cyclization is at the 3 position. (See Scheme I.) As expected, the pK_a (9.4) of **3** was similar to that (10.1)[15] of 3,9-dimethylxanthine. The conversion of **1** to **2** probably involves attack by N-3 on a 5′-carbonate derivative of xanthosine 2′,3′-carbonate because under similar conditions diphenyl carbonate converts inosine to bis(inosine 2′,3′-carbonate 5′-)carbonate *via* inosine 2′,3′-carbonate[1] and it also appears to convert isopropylideneguanosine to a 5′-carbonate derivative (see below).

Attempts to prepare 3,5′-cycloguanosine by reac-

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(10) D. H. Rammler and H. G. Khorana, *J. Am. Chem. Soc.*, **84**, 3112 (1962).

(11) The conditions used were those of ref 6, with $NaHCO_3$ as catalyst. With cytidine, 1.2 molar equiv of phenol was also used as catalyst (*cf.* the conversion of adenosine to adenosine 2′,3′-carbonate[6]).

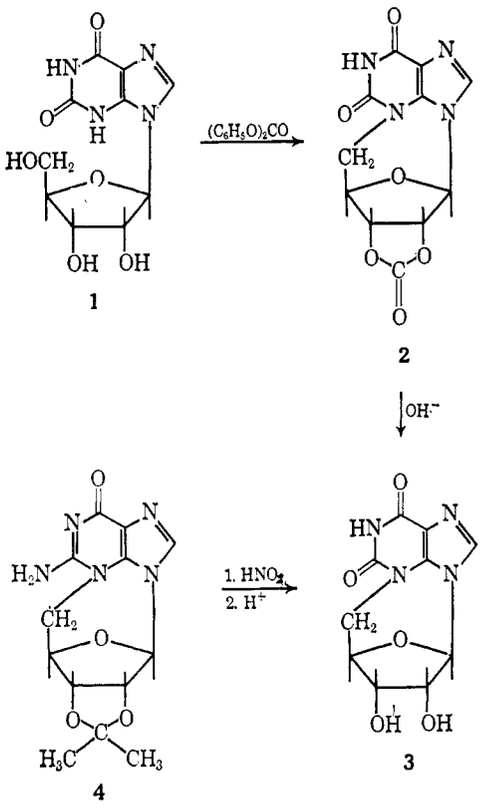
(12) L. Hough, J. E. Priddle, and R. S. Theobald, *J. Chem. Soc.*, 1934 (1962).

(13) R. E. Holmes and R. K. Robins, *J. Org. Chem.*, **28**, 3483 (1963).

(14) 3-Methylguanine is also resistant to diazotization: G. B. Elion, *ibid.*, **27**, 2478 (1962); L. B. Townsend and R. K. Robins, *J. Am. Chem. Soc.*, **84**, 3008 (1962).

(15) W. Pfleiderer and G. Nübel, *Ann.*, **647**, 155 (1961).

SCHEME I



tion between guanosine and diphenyl carbonate were unsuccessful and yielded numerous products. Reaction between diphenyl carbonate and 2′,3′-O-isopropylideneguanosine resulted in the initial formation of a highly insoluble product which showed carbonyl absorption at 1760 cm^{-1} indicative of an acyclic carbonate[12] and was probably bis(2′,3′-O-isopropylideneguanosine 5′-)carbonate. This product dissolved on further heating to yield a mixture similar to that obtained with guanosine itself.

The optical rotatory dispersion curves of β-purine nucleosides and nucleotides give negative Cotton effects,[16-18] whereas the α-purine nucleosides give positive Cotton effects;[18] further, evidence has been presented[19] that β-purine cyclonucleosides in the *syn* conformation, *e.g.*, 3,5′-cyclonucleosides, show positive Cotton effects while those possessing the *anti* conformation, *e.g.*, 8,5′-cyclonucleosides, show negative Cotton effects. Such correlations, if confirmed, are of considerable interest inasmuch as they would assist assignment of the configuration of purine nucleosides and nucleotides in aqueous solution.

We have examined the optical rotatory dispersion curves of 3,5′-cycloxanthosine, 2′,3′-O-isopropylidene-3,5′-cycloguanosine, and 2′,3′-O-isopropylidene-3,5′-cycloadenosine iodide at various pH values. The pK_a values of the cyclonucleosides were determined and the pH values were selected so that one ionic species would predominate for each ORD determination. It is known[17] that the ORD curves of nucleosides and nucleotides are significantly influenced by the presence on the base of a positive or a negative charge; furthermore, 3,5′-cycloxanthosine and 3,5′-cycloguanosine, in the form of their neutral molecules, appear to possess considerable zwitterionic character.[13] An ORD curve for 2′,3′-O-isopropylidene-3,5′-cycloadenosine iodide could not be determined at alkaline pH values since under these conditions the compound eliminates C-2 to form an imidazole derivative.[20]

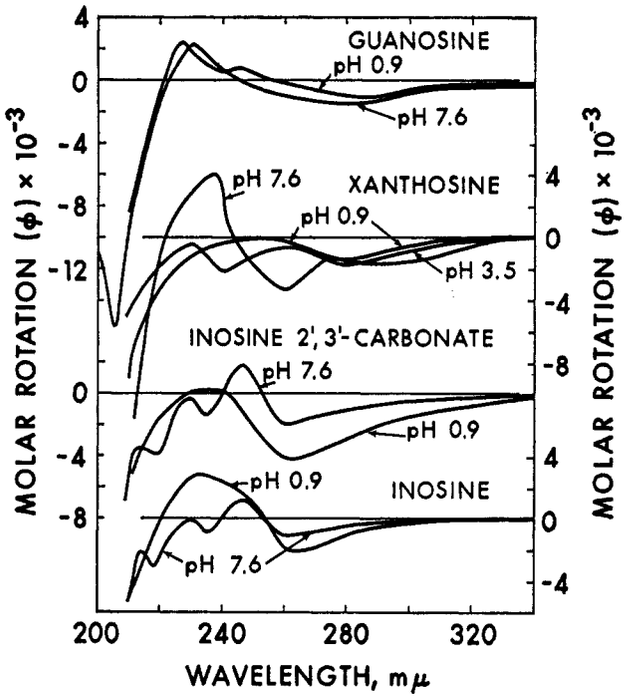


Figure 1.—Ultraviolet rotatory dispersion of purine ribonucleosides.

Figure 1 shows the ultraviolet rotatory dispersion characteristics of ribonucleosides which are related to the cyclonucleosides of the present study. The pK_a values of these nucleosides[21] are such that at pH 7.6 (Figure 1) inosine and guanosine exist as the uncharged species and xanthosine exists as the monoanion, and that at pH 0.9 (Figure 1) the nucleosides (except for xanthosine, pK_a 0.8) are present principally as the monocations. At pH 3.5 xanthosine has no net charge. The ORD of guanosine at pH 7.6 agrees with that reported previously;[17] the remaining ORD profiles of Figure 1 are hitherto unreported. The 2′,3′-carbonate moiety is seen to exert little effect on the rotatory properties of inosine; the 2′,3′-O-isopropylidene blocking group is reported[19] to behave similarly.

The ORD curves of the cyclonucleosides are given in Figure 2. The magnitudes of their rotations are considerably higher than those of the uncyclized counterparts in Figure 1. Although pH has a considerable effect on the position and amplitude of the Cotton effect, it does not affect the sign of the Cotton effect except in the case of 3,5′-cycloxanthosine at pH 7.6 which shows a small positive Cotton effect. All the other ORD curves of Figures 1 and 2 show negative Cotton effects in accord with the general rule for purine β-nucleosides but contrary to the hypothesis[19] that

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(21) G. H. Beaven, E. R. Holiday, and E. A. Johnson in "The Nucleic Acids," Vol. 1, E. Chargaff and J. N. Davidson, Ed., Academic Press Inc., New York, N. Y., 1955, p 508.

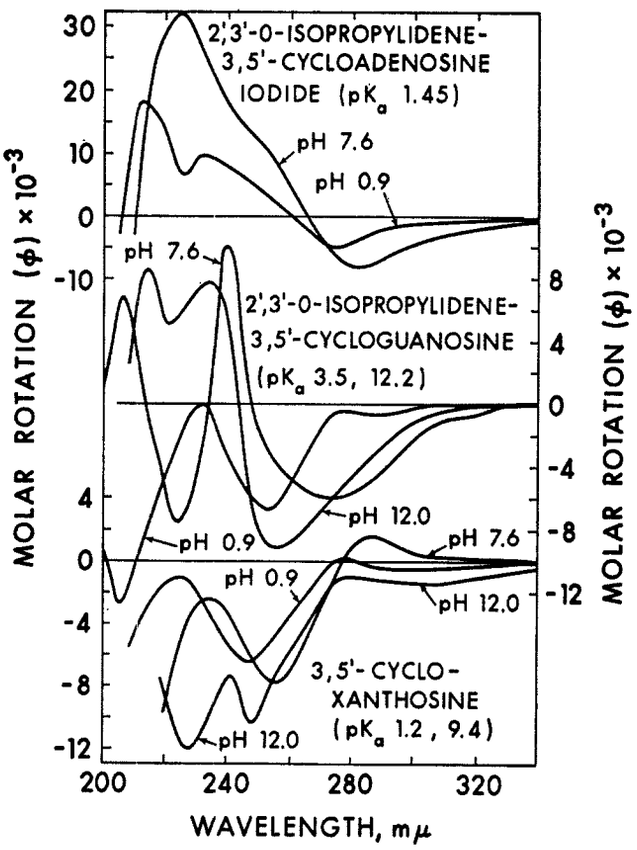

Figure 2.—Ultraviolet rotatory dispersion of 3,5′-purine cyclonucleosides.

purine β-nucleosides in the *syn* conformation possess positive Cotton effects. It has been reported[19] that aqueous solutions of 2′,3′-O-isopropylidene-3,5′-cycloadenosine iodide exhibit a positive Cotton effect. This compound was prepared for the present work by the method of Todd, *et al.*,[22] and showed properties consistent with the proposed structure; the ultraviolet absorption maximum was identical with that published by Bernhauer and Irion.[23]

Clearly more observations must be made on the ORD of the cyclonucleosides before a correlation can be made between this data and the configuration of the compounds.

Experimental Section

Melting points (corrected) were determined on a Kofler micro hot stage. Analyses were by Dr. A. Bernhardt, Mülheim, Germany, on samples dried at 100° *in vacuo* for 8 hr over P_2O_5. Ultraviolet absorption spectra were determined on a Cary Model 15 spectrophotometer. Infrared spectra were determined in KBr disks in a Perkin-Elmer 137B spectrophotometer. Optical rotatory dispersion curves were run on a Cary Model 60 spectropolarimeter. Concentrations were 0.02% in a 1-cm cell. ORD curves were determined in HCl (pH 0.9), 0.01 *M* potassium phosphate buffer (pH 7.6), and in 0.01 *N* NaOH (pH 12.0). pK_a values were determined at 22° by titration of a 0.05 *M* aqueous solution with 0.3 *M* HCl or NaOH using a Corning Model 12 pH meter. N,N-Dimethylformamide was purified by treatment with Drierite and distillation. Paper chromatography was carried out on Whatman No. 1 paper using n-butyl alcohol–acetic acid–water (5:2:3) as solvent.

3,5′-Cycloxanthosine 2′,3′-Carbonate.—Xanthosine (0.5 g, 1.76 mmoles) was suspended in dry N,N-dimethylformamide (2 ml) and sodium hydrogen carbonate (0.02 g) was added. The mixture was refluxed in an oil bath (bath temperature 150°) and diphenyl carbonate (0.45 g, 2.10 mmoles) was added. Refluxing was continued for 45 min and a further batch of diphenyl carbonate (0.1 g, 0.49 mmole) was added. After refluxing for an additional 10 min all suspended material had dissolved and the mixture was poured while still hot into ether. The white precipitate was filtered off and washed with ether. It consisted of a major component of R_f 0.30 which reacted negatively when sprayed[24] for *cis*-α-glycol systems and a minor component of R_f 0.16 which reacted positively with the same reagent. The material of lower R_f corresponded to xanthosine. The precipitate was crystallized from methanol–acetic acid to yield 3,5′-cycloxanthosine 2′,3′-carbonate (0.26 g, 51%) as colorless needles, mp 258–260° dec. A sample for analysis was crystallized from aqueous acetic acid and had mp 260–264°; λ_{max} (0.01 *M* HCl) was 265 mμ (ε 10,740) 234 mμ (ε 9560), λ_{min} (0.01 *M* HCl) 249 mμ (ε 7240) and 217 mμ (ε 4600), λ_{max} (0.01 M NaOH) 268 mμ (ε 11,440), λ_{min} (0.01 *M* NaOH) 251 mμ (ε 7260); carbonyl absorption was at 1840, 1825, 1730, and 1710 cm^{-1}.

Anal. Calcd for $C_{11}H_8N_4O_6 \cdot CH_3COOH$: C, 44.33; H, 3.64; N, 16.02. Found: C, 44.25; H, 3.41; N, 15.91.

3,5′-Cycloxanthosine.—3,5′-Cycloxanthosine 2′,3′-carbonate (0.10 g, 0.28 mmole) was dissolved in 2 *N* ammonium hydroxide (10 ml) and warmed on the boiling water bath for 30 min. The solution was cooled and evaporated to small volume (2 ml) whereupon 3,5′-cycloxanthosine (0.06 g, 81%) separated as colorless needles which when recrystallized from water had mp 310–314°. Its R_f (0.16) was identical with that of xanthosine and gave a positive reaction to the *cis*-glycol spray.[24] Admixture with xanthosine depressed the melting point to 290–300°; λ_{max} (0.01 *N* HCl) was 265 mμ (ε 10,500) and 236 mμ (ε 8950), λ_{min} (0.01 *N* HCl) 249 mμ (ε 7150) and 218 mμ (ε 4720), λ_{max} (0.01 *N* NaOH) 267 mμ (ε 11,220), and λ_{min} (0.01 *N* NaOH) 249 mμ (ε 7110); carbonyl absorption was at 1730 and 1710 cm^{-1}; pK_a = 9.37 and 1.2.

Anal. Calcd for $C_{10}H_{10}N_4O_5$: C, 45.13; H, 3.76; N, 21.04. Found: C, 45.32; H, 3.88; N, 20.93.

2′,3′-*O*-Isopropylidene-3,5′-cycloguanosine.—This compound was prepared by the methods of Holmes and Robins[13] and Baker, *et al.*[25] Before isolation of the free base according to the method of Holmes and Robins, the iodide in 0.01 *M* HCl showed λ_{max} 229 mμ (ε 17,100) with a shoulder at 255–260 mμ and λ_{min} 215 (ε 11,670). In 0.01 *M* NaOH λ_{max} was 262–267 mμ (ε 7240) and λ_{min} was 253 mμ (ε 6310). These figures are in agreement with those of Baker, *et al.*[25] The free base, isolated from this material, in 0.01 *M* HCl had λ_{max} 247 mμ (ε 8680) and λ_{min} 220 mμ (ε 2530). In 0.01 *M* NaOH λ_{max} was 266 mμ (ε 7810) and λ_{min} was 220 mμ (ε 2530). These figures agree with those of Holmes and Robins.[13] The difference in the spectra is probably due to removal of contaminating *p*-toluene sulfonate anion which has a maximum at 222 mμ (ε 9940).

The product gave a spot of R_f 0.61 which reacted negatively when sprayed for *cis*-α-glycol systems. The pK_a values were 12.22 and 3.50.

Deamination of 2′,3′-O-Isopropylidene-3,5′-cycloguanosine.—2′,3′-O-Isopropylidene-3,5′-cycloguanosine (0.2 g) was dissolved in 2 *N* acetic acid (62 ml). Barium nitrite (4 g) was added and the solution was allowed to stand overnight. Sulfuric acid (1.7 g) was added and the precipitated barium sulfate was centrifuged off. The supernatant solution was shaken with 2-octanol-deactivated Norit (1 g)[26] which was filtered off and washed with water, and the products were eluted with 5% ammonia in 50% aqueous ethanol (100 ml). The eluate was evaporated to dryness and the residue was dissolved in 50% aqueous acetic acid (5 ml). The solution was warmed on the water bath for 2 hr and evaporated to dryness. The residue was crystallized from aqueous acetone to yield 3,5′-cycloxanthosine (0.001 g) mp 306–308°, not depressed on admixture with 3,5′-cycloxanthosine prepared from xanthosine. The two preparations had identical R_f values and ultraviolet and infrared absorption spectra.

2′,3′-O-Isopropylidene-3,5′-cycloadenosine Iodide.—This compound was prepared by the method of Todd, *et al* [22] In 0.01 *M* potassium phosphate buffer pH 7.6 and in 0.01 *M* HCl this compound showed λ_{max} 273 mμ (ε 13,560) and λ_{min} 241 mμ (ε

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(26) C. J. Threlfall, *Biochem. J.*, **65**, 694 (1957).

4310). In 0.01 *M* NaOH λ_{max} was 268 mμ and λ_{min} was 252 mμ On acidification of the alkaline solution λ_{max} was 282 mμ and λ_{min} was 251 mμ. The pK_a was 1.46. On titration of the compound with alkali drifting to lower pH was observed, and upon back-titration of this solution a pK_a of 2.45 was found.

Registry No.—**2**, 10299-76-0; **3**, 10380-93-5; **4**, 10299-77-1; 2′,3′-O-isopropylidene-3,5′-cycloadenosine iodide, 10299-78-2.

Acknowledgments.—The authors are pleased to thank Dr. C. M. Kay for the use of the spectropolarimeter and Mr. K. Okawa for determination of ORD curves.

Relative Stabilities of Copper Chelates of 2-Acetylcyclanones[1]

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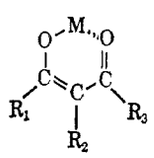
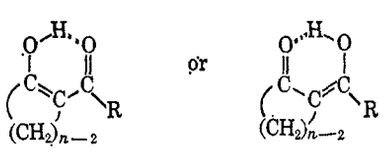
Received October 26, 1966

The stabilities of metal chelates of 1,3-diketones **1**, as expressed by the strengths of the oxygen–metal bonds as well as by stability constants,[3,4] are noticeably affected by electronic effects of the substituents R_1, R_2, and R_3. At the same time, the enolic character of **2**



1

and many other properties of cyclic systems are distinctly altered by strain (bond angle, torsional, and transannular) within the alicyclic ring.[5] It was of interest, therefore, to see if the stabilities of the copper chelates of the enols **2** are also dependent upon the sizes of the alicyclic rings.



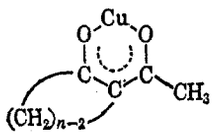
2

The C=C and Cu–O bond infrared absorption frequencies of chelates **5**–**15**, Table I appears in Table II. If the Cu–O bond frequencies are taken as a measure of chelate stability,[3] there is observed an alternation with ring size, with the odd-membered rings being generally more stable than the even-membered rings. An alternation with ring size also occurs for enol contents of cyclanones and their 2-acyl derivatives[6] but, in that case, the even-membered rings are more enolic than the odd-membered rings.

As an alternative check on their relative stabilities,[7] the conductivities of the chelates were measured. It is reasoned that the more stable is the chelate, the smaller is the concentration of copper ion in solution and the smaller should be the conductivity. The data are given in Table III. The relative stabilities of the chelates, determined by the two methods, follow (in

TABLE I

ANALYSES OF COPPER CHELATES OF 2-ACETYLCYCLANONES



Compd	*n*	Solvent,[a] mp (°C)	Calcd, % C	Calcd, % H	Found, %[b] C	Found, %[b] H
5	5	Et_2O–THF (238)	53.58	5.78	53.71	5.78
6	6	Et_2O (168–169)	56.20	6.49	56.31	6.74
7	7	THF (222.5–223.5)	58.44	7.08	58.14	7.06
8	8	THF (265 dec)	60.35	7.60	60.17	7.02
9	9	THF (199)	62.01	8.04	61.57	7.34
10	10	THF (184–187)	63.48	8.44	63.57	8.45
12	12	THF–$CHCl_3$ (208 dec)	65.91	9.09	65.75	9.11
15	15	THF (199 dec)	68.70	9.84	68.58	9.75

[a] Recrystallization solvent: Et_2O = ether; THF = tetrahydrofuran. [b] Analyses were performed by the Schwarzkopf Microanalytical Laboratory.

TABLE II

SOME INFRARED ABSORPTION PEAKS (RECIPROCAL CENTIMETERS) OF CHELATES **5**–**15**

Chelate	$\nu_{C=C}$	ν_{Cu-O}
5	1597	495
6	1573	452
7	1577	474
8	1569	452
9	1569	467
10	1564	460
12	1570	469
15	1574	471
Copper acetylacetone		455

TABLE III

CONDUCTIVITY DATA FOR COPPER CHELATES OF 2-ACETYLCYCLANONES IN 75% AQUEOUS PYRIDINE

Chelate	*M* concn × 10^3	Conductance, *L* × 10^5 ohm^{-1} cm^{-1}	Vol. *V* × 10^{-4} ml	Equiv conductance, Λ ohm^{-1} cm^2 equiv^{-1}
5	4.93	1.948	10.136	1.974
6	4.64	8.004	10.077	8.618
7	4.83	6.199	10.340	6.412
8	4.61	7.628	10.840	8.267
9	4.64	9.168	10.780	9.883
10	5.10	9.853	9.803	9.659
12	4.99	8.828	11.248	11.248
15	4.66	7.151	10.739	7.679

(1) Abstracted from the Ph.D. dissertation of N. J. King presented to Howard University, June 1966.

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(7) Polarographic measurements with a dropping mercury electrode were also made on the chelates. Each chelate has two waves; however, the first wave blends in with the anodic curve from dissolution of Hg and the second wave [probably for Cu(I)] occurs at the same potential for all of the chelates. Since the potential for the first wave is not clearly discernable, the relative stabilities were not determinable by this method.