

ABSTRACTS SECTION

In this section are given, information on methods of synthesizing labelled compounds and related problems (analysis, assaying, purifying, radiodecomposition, storage. The references cover articles drawn from 40 primary periodicals and also from N.S.A. and C.A.

A point is made of singling out each of the above mentioned aspects in the abstracts particularly where the greater part of the article deals with applications of labelled compounds. This journal will likewise contain author and subject indexes at the end of each volume.

The articles are abstracted by H. Dworschak, CID (Euratom).

The work on this information project was started in May 1964. The results obtained until December 1964 have been published in Euratom reports EUR 2212.e and EUR 2212.e suppl., the latter of which contains author and subject indexes.

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1 — SYNTHESIS

1.1 — Deuterium and tritium compounds

1.1.1 — GENERAL

65-224

CARDINAUD R., BOUHET J.-C.

Influence du support sur le rendement de la tritiation par échange.

Bull. Soc. Chim. France **1965**, 834

Tritiation by exchange shows an evolution of sp. act. proportional to the surface of quartz as inert support. Within certain limits this effect is reversed by active carbon. Example : succinic acid-fumaric acid, which proved to have a common precursor.

65-225

SOKLOWSKA A.

Reactions of tritium recoil atoms in liquid organic mixtures.

(PAN-577/V) (CONF-773-37) Nov. 1964, 19 p.

N.S.A. **19**, 17958 (1965)

The reactions of recoil tritium atoms for H substitution in cyclohexane and benzene molecules in the studied system do not depend on the composition of a mixture.

1.1.2 — ALIPHATIC COMPOUNDS

65-226

ARNOLD D. R., TRECKER D. J., WHIPPLE E. B.

The stereochemistry of the pentacyclo [8.2.1.1^{4,7}.0^{2,5}.0^{3,6}] tetradecanes and -dienes. Norbornene and norbornadiene dimers.

J. Am. Chem. Soc. **87**, 2596 (1965)

Norbornadiene + D-Pd/C yielded 63% of 5,6-exo-exo-d₃-norbornene, when stopped after the uptake of 1,1 mole equiv. of D.

65-227

ATKINSON J. G., FISHER M. H., HORLEY D., MORSE A. T., STUART R. S., SYNNESE E.

Synthesis of isotopically labeled olefins via the Wittig reaction.

Can. J. Chem. **43**, 1614 (1965)

Synthesis of 5-methylene-¹⁴C-bicyclo(2.2.1)-hept-2-ene; propene-1-¹⁴C; 1-butene-1-¹⁴C; propene-1-d₁; 2-methyl-propene-1,1-d₂; 2-methyl-d₃-propene-3,3,3-d₃; 2-methyl-2-butene-4,4,4-d₃; 2-methyl-d₃-2-butene-1,1,1-d₃; methylene-d₂-cyclohexane, using the methylsulfinyl carbanion in dimethyl sulfoxide as the base.

65-228

DEPUY C. H., MORRIS G. F. SMITH J. S., SMAT R. J.

Electronic effects in elimination reactions. V. Bimolecular cis eliminations. 2-aryl-cyclopentyl tosylates.

J. Am. Chem. Soc. **87**, 2421 (1965)

1-Phenylcyclopentene was treated with LiAlD₄ and boron trifluoride etherate. Oxidation with hydrogen-peroxide yielded trans-2-d-2-phenylcyclopentanol (more than 95% D in pos. 2).

65-229

DUFFIELD A. M., BUDZIKIEWICZ H., DJERASSI C.

Mass spectrometry in structural and stereochemical problems. LXX. A study of the fragmentation processes of some five-membered N-alkyllactams and N-alkylsuccinimides.

J. Am. Chem. Soc. **87**, 2913 (1965)

1-Bromopropane-3,3,3-d₃ (I) by redn. of fully deuterated acetic acid with LiAlH₄, treating the alcohol with 48% HBr; the Grignard deriv. of the resulting bromide + CO₂, and subsequent LiAlH₄ redn. and bromination → I (75% d₃, 25% d₀).

65-230

JULLIEN J., PAILLOUS A.

Corrélations entre structure et absorption infrarouge de composés monodéutériés : contribution à l'étude des composés organomagnésiens.

Bull. Soc. Chim. France **1965**, 2236

Redn. of the aldehydes or ketones with LiAlD_4 and transformation of the alcohols to the corresp. bromides (PBr_3/Py), (bromomethane- d_1 , 1-bromoethane- 1-d_1 , 2-bromo-propane- 2-d_1 , 1-bromo-n-hexane- 1-d_1), from which are formed the Grignard compds.

CH_3DONa and n-butylbromide, 60°C , \rightarrow deuteriomethyl-n-butylether.

65-231

LEE C. C., WONG E. W. C.

Secondary α -deuterium kinetic isotope effects in solvolyses of 2-cyclopentylethyl, 2-(2-cyclopenten-1-yl) ethyl, and 2-(3-cyclopenten-1-yl)ethyl p-nitrobenzenesulfonates.

Tetrahedron **21**, 539 (1965)CA **62**, 12996 h (1965)

Redn. of the acetic acids with LiAlD_4 in anhyd. Et_2O gave 2-(2-cyclopentylethanol- $1,1\text{-d}_2$, 2-(2-cyclopenten-1-yl)-ethanol- $1,1\text{-d}_2$, 2-(3-cyclopenten-1-yl)ethanol- $1,1\text{-d}_2$, in yields ranging from 68-97%.

65-232

MORIARTY R. M., RAHMAN M.

The photodecarboxylation of α -azido acids.

J. Am. Chem. Soc. **87**, 2519 (1965)

α -d- α -Azidobutyric acid via ethylbromomalononic acid, \rightarrow corresp. acylchloride, + D_2O , decarboxylation \rightarrow α -d- α -bromobutyric acid; formation of the azide via the ester.

65-233

NEUREITER N. P.

Convenient preparations of α -halo sulfones and their use in the synthesis of deuterated olefins, the desulfurization of alkyl sulfones, and the homologization of olefins.

J. Org. Chem. **30**, 1313 (1965)

Exchange of H in α -halosulfones (I) ($\text{D}_2\text{O}/\text{NaOD}$) and alkaline degradation of I

(Ramberg-Bäcklund reaction) to form the olefines (propene- $1,1,2\text{-d}_3$; 2-butene- $2,3\text{-d}_2$; 3-methyl-1-butene- $1,1,2\text{-d}_3$; 2-methyl-1-butene- $1,1\text{-d}_2$ and 3-methyl-1-pentene- $1,1,2\text{-d}_3$). D-content about 97% in one step of equilibration, total yields between 50 and 82%.

65-234

SAUNDERS Jr. W. H., FINLEY K. T.

The transition state in acetolysis of cyclohexyl tosylate.

J. Am. Chem. Soc. **87**, 1384 (1965)

Prepn. of the 1-d- , $2,2,6,6\text{-d}_4\text{-}$, and trans- 2-d- cyclohexanols and its tosylates by the procedure of Streitwieser et al. (ibid., **80**, 2326) and of the corresp. cis- 2-d deriv. by deuterioboration and oxidn. with $\text{H}_2\text{O}_2/\text{NaOH}$.

65-235

WOLFE S., CAMPBELL P. G. C.

Cyclohexene- $3,3,6,6\text{-d}_4$ a useful compound for the study of mechanism and structure.

Can. J. Chem. **43**, 1184 (1965)

The title compound was prepared by Diels-Alder reaction of butadiene- $1,1,4,4\text{-d}_4$ with ethylene.

65-236

ÄNGGÅRD E., GREEN K., SAMUELSSON B.

Synthesis of tritium-labeled prostaglandin E_2 and studies on its metabolism in guinea pig lung. 37. prostaglandins and related factors.

J. Biol. Chem. **240**, 1932 (1965)

PGE_3 was selectively hydrogenated with tritium gas at 20°C in ethylacetate with Pd-BaSO_4 to PGE_2 . Yield 2-3%, spec. act. : 50mC/mmmole before dilution. Determination of the location of the T-label by : 1. Oxidation, to caproic acid. 2. treatment with NaOH , then ozonolysis to methyl α -acetoxyheptanoate. Practically all activity in pos. 17-18.

65-237

BOHLMANN F., HINZ U.

Polyacetylenverbindungen, LXXII. Ueber biogenetische Umwandlungen des Tridecen-pentains.

Chem. Ber. **98**, 876 (1965)

Propynaldiethylacetal, catalytic redn. with T \rightarrow (2,3-³H)-acroleindiethylacetal \rightarrow corresp. aldehyde + Grignard deriv. of acetaldehyde ethyl-(2-propynyl)-acetal, hydrolysis, + SOCl₂ \rightarrow 3,6-dichloro-1-hexen-4-yne)-1,2-³H, + NaNH₂ \rightarrow 1-hexen-3,5-diyne, + 1-bromo-1,3,5-heptatriyne \rightarrow 1-tridecen-3,5,7,9,11-pentayne-1,2-³H (63,00 \times 10⁸ ipm/mmole).

65-238

KLENBERG D., SAMUELSSON B.

The biosynthesis of prostaglandin E₁ studied with specifically ³H-labeled 8,11,14-eicosatrienoic acids.

Acta Chem. Scand. **19**, 534 (1965)

Keto stearic acid (6-,9-, or 10-keto stearic acid) + NaBT₄ \rightarrow hydroxy-stearic acid \rightarrow tosylate, + LiAlH₄ \rightarrow octadecanol, + chromic acid \rightarrow stearic acid (sp. act. 13.4 μ C/mg), tritium label at C-6, C-9, or C-10. Conversion into 6,9,12-octadeca-triencic acids utilizing Tetrahymena pyriformis. Elongation by a malonic ester synthesis to 8,11,14-eicosatrienoic acids.

65-239

THOMPSON Jr. G. A.

The biosynthesis of ether-containing phospholipids in the slug, Arion ater. I. Incorporation studies in vivo.

J. Biol. Chem. **240**, 1912 (1965)

1-O-Hexadecenylglycerol + tritium gas catal. reduced, removing labile T, \rightarrow chimyl alcohol-³H, which was further exhaustive catal. reduced. Spec. act. : 1.02 \times 10⁸ μ C/mmole.

See also : 65-245, Isopropanol -³H

1.1.3 — AROMATIC COMPOUNDS

65-240

CRAWFORD R. J., WOO Ch.

The conversion of meta- and para-substituted benzaloxime arenesulfonates to nitriles.

Can. J. Chem. **43**, 1534 (1965).

p-Chlorobenzaldehyde-d from N-methyl p-chlorobenzanilide by redn. with LiAlD₄ (yield : 58%).

65-241

SELTZER S., DUNNE F. T.

The mechanism of the thermal decomposition of α -phenylethylazomethane. A two-step reaction.

BNL-8243 and J. Am. Chem. Soc. **87**, 2628 (1965)N.S.A. **19**, 19943 (1965)

Prepn. of α -phenylethyl- α -d-azomethane and α -phenylethylazomethane-d₃.

65-242

WILCOX Jr. C. F., ROBERTS F. D.

Synthesis and some spectral properties of diphenyltritycene.

J. Org. Chem. **30**, 1959 (1963)

9,10-Dibromoanthracene + PhLi + D₂O \rightarrow anthracene-9,10-d₂, + cis-dichloroethylene, + Na \rightarrow 9,10-etheno-9,10-dihydroanthracene-9,10-d₂.

65-243

ZOLTEWICZ J. A., BUNNETT J. F.

Substituent effects on the ratio of proton capture to chloride ion loss by o-chlorophenyl anions in liquid ammonia.

J. Am. Chem. Soc. **87**, 2640 (1965)

Synthesis of o-deuteriochlorobenzene derivatives by treatment of the corresp. arylmagnesium iodide with D₂O. 2-Chloroaniline derivs. (3-, 4-, and 6-chloro-, 4-methyl-) were transformed to the corresp. iodocompds, + Mg, +D₂O \rightarrow chlorobenzene-2-d, 1,2-chlorobenzene-3-d, 1,4-dichlorobenzene-2-d, 4-chlorotoluene-3-d. 1,3-Dichlorobenzene-2-d by alkali cleavage of 2,6-dichlorobenzaldehyde in D₂O.

65-244

BERTINO J. R., JOHNS D. G., ALM-QUIST P., HOLLINGSWORTH J. W.

3,5-Ditritiomethotrexate (I) as a granulocyte label.

Nature **206**, 1053 (1965)

$I\text{-}^3\text{H}$, labeled specifically with tritium in the 3'- and 5'-positions, by catalytic reductive dehalogenation of 3',5'-dichloromethotrexate with tritium gas of high isotopic purity. It was stored at -40°C . Radiation decomposition at the rate of approximately 3 per cent per month.

65-245

GARNETT J. L., LAW S. W., TILL A. R.

Wilzbach tritiation studies.

I. Isotope distribution in tritium-labeled anthranilic acid, isopropanol, and phenyl allyl ether.

Austr. J. Chem. **18**, 297 (1965)

In anthranilic acid (I) positions 3 and 5 have respect. 49.8% and 27.4% of the total act. In isopropanol (II), T in hydroxyl group \gg sec. C-H $>$ prim. C-H. In phenyl allyl ether, polymerization, radiation-induced Claisen rearrangement or saturation of the double bond.

Detn. of T distribn. in I by 5-bromo-I; 3,5-dibromo-I and 2,4,6-tribromoaniline.

65-246

GRISEBACH H., KELLNER S.

Untersuchungen zum Mechanismus der Umwandlung von 4.2'.4'.6'-Tetrahydroxychalkon in Taxifolin in Chamaecyparis obtusa.

Z. Naturforschg. **20b**, 446 (1965)

4.2'.4'.6'-Tetrahydroxychalcone-2'-glucoside-($\beta\text{-}^{14}\text{C}\text{-}\beta\text{-T}$) via the intermediate p-hydroxybenzaldehyde-(carbonyl-T) obtained by reduct. of p-benzyloxybenzoxonitril with T_2/H_2 -Raney-Nickel (70-80% yield of p-benzyloxybenzaldehyde semicarbazone; detn. of T distribn. in this aldehyde, 99% in the CHO-group). The further synthesis by a previously described method.

65-247

HESSELBO T.

The preparation of tritiated dinitrofluorobenzene of high specific activity.

Intern. J. Appl. Radiation Isotopes **16**, 329 (1965)

m-Bromofluorobenzene (I) (0.09 mmole) + tritium gas and Pd/C \rightarrow fluorobenzene-3,5- ^3H \rightarrow 2,4-dinitrofluorobenzene-3,5- ^3H ; yield : 65% based on I, 50% based on T; sp. act. : 19 C/mmole. Less than 1,7% dinitrobenzene as impurity.

1.1.4 — *HETEROCYCLIC COMPOUNDS*

65-248

BROWN N. E., KEYES R. T.

Structure of salts of 4,6-dinitrobenzofuroxan (I).

J. Org. Chem. **30**, 2452 (1965)

5-Deuterio-I from p-d-nitrobenzene \rightarrow aniline \rightarrow 5-d-benzo-furoxan, nitration by the method of Drost (4-nitro-5-d- and 4-nitro-6-d-benzofuroxan, the 2nd nitro-group in pos. 6, hence one half of D is lost).

65-249

COBURN W. C. Jr., THORPE M. C., MONTGOMERY J. A., HEWSON K.

Correlation of the proton magnetic resonance chemical shifts of substituted purines with reactivity parameters. I. 2,6-Disubstituted purines.

J. Org. Chem. **30**, 1110 (1965)

Purine-2-d(I) and purine-6-d(II) from the corresp. chloropurines by hydrogenation with D_2 and Pd/C in D_2O . 55 and 70% yield respect. In I exchange of D to purine-2,6-d₂.

65-250

COBURN W. C. Jr., THORPE M. C., MONTGOMERY J. A., HEWSON K.

Correlation of the proton magnetic resonance chemical shifts of substituted purines

with reactivity parameters. **II. 6-Substituted purines.**

J. Org. Chem. **30**, 1114 (1965)

6-Methylpurine-2-d and 6-methoxypurine-2-d, by deuterolysis of the corresp. 2-chloropurines as described in the preceding abstract; 81 and 68% yields respect. Exchange of the proton in pos. 8.

65-251

PUGMIRE J. R., GRANT D. M.,
ROBINS R. K., RHODES G. W.

Carbon-13 magnetic resonance. III. Purine.

J. Am. Chem. Soc. **87**, 2225 (1965)

8-d-Purine by heating purine in D₂O for 4 hr, and 6,8-d₂-purine by heating the mixture for 72 hr. 80% deuteration in pos. 6. Total yield : 20%.

65-252

DUFFIELD A. M., BUDZIKIEWICZ, H.
DJERASSI C.

Mass spectrometry in structural and stereochemical problems. LXXI. A study of the influence of different heteroatoms on the mass spectrometric fragmentation of five-membered heterocycles.

J. Am. Chem. Soc. **87**, 2920 (1965)

1,4-Butanediol-1,1,4,4-d₄ → corresp. tetrahydrofuran → 1,4-dibromobutane-1,1,4,4-d₄ + sodium sulfide (selenide) → tetrahydrothiophene- $\alpha,\alpha,\alpha',\alpha'$ -d₄ (tetrahydroselelenophene). Succinimide + LiAlD₄ → pyrrolidine- $\alpha,\alpha,\alpha',\alpha'$ -d₄. 1,1-Dichlorosilacyclopentane + LAD → silacyclopentane-1,1-d₂. 1,1-Diiodogermacyclopentane + LAD → germacyclopentane-1,1-d₂.

65-253

DUFFIELD A. M., BUDZIKIEWICZ H.,
DJERASSI C.

Mass spectrometry in structural and stereochemical problems. LXXII. A study of the fragmentation processes of some tobacco alkaloids.

J. Am. Chem. Soc. **87**, 2926 (1965)

Continine (I) (5-oxonicotine) + LiAlD₄ → nicotine-5,5-d₂. I + D₂O heated for 12 days

and subsequent LiAlH₄ redn. → nicotine-4,4-d₂.

Further products : nornicotine-2-d₁; nicotine-2-d₁; anabasin-2-d₁.

65-254

CAYEN M. N., COMMON R. H.

An investigation of urinary conversion products of coumestrol in the fowl.

Biochim. Biophys. Acta **100**, 567 (1965)

Wilzbach tritiation of coumestrol. Equilibration for 24 h in methanol 10 times. Paper chrom. in acetic acid/water/HCl and isopropanol/water. Sp. act. : 0,116 mC/mg.

65-255

SEILER N., WERNER G.

Die Umwandlung von Tropin in Pseudotropin durch Synergismus zweier Bakterienstämme II.

Z. Naturforschg. **20 b.**, 451 (1965)

3 α -Tropanol-3 β -T by hydrogenation of tropinone with tritium-hydrogen and Raney-Nickel; 15% pseudo form. Activity : 12 mC/mmole.

1.1.5 — *ALKALOIDS*

65-256

BARTON D. H. R., KIRBY G. W.,
STEGLICH W., THOMAS G. M.,
BATTERSBY A. R., DOBSON T. A.,
RAMUZ H.

Investigations on the biosynthesis of morphine alkaloids.

J. Chem. Soc. **1965**, 2423

Salutaridine-1-³H from dihydrothebaine- Φ by exchange of hydrogen in C-1 with NaOH-tritiated water and oxidn. with SeO₂ and MnO₂. Redn. with NaBH₄ yielded the epimeric salutaridinols.

See also : **65-290**, Deoxyajmaline -³H

65-291, (+) and (—) Reticuline -1-³H

1.1.6 — CARBOHYDRATES

65-257

ENGLARD S., AVIGAD G.

5-Keto-D-fructose. II. Patterns of formation and of associated dehydrogenase activities in *Gluconobacter cerinus*.

J. Biol. Chem. **240**, 2297 (1965)

5-Keto-D-fructose was reduced by *Gluconobacter cerinus* reductase (T from ^3H -DL-isocitrate \rightarrow NADP ^3H) to fructose-5- ^3H . Spec. act. : 65500 dpm per μmole .

65-258

LEPAGE G. A., JUNGA I. G.

Labeling of nucleosides with tritium.

Can. J. Chem. **43**, 1279 (1965)

A mixture of 2-acetamido-6-chloro-9-(2'-deoxy-3',5'-di-O-p-toluyyl-D-ribofuranosyl)-9H-purine, mercuric chloride and benzene-T (1.2×10^9 c.p.m./ml) were refluxed. After purifn. the nucleoside contained 1.4×10^5 c.p.m./mg. After removal of the blocking groups 9-(β -D-2'-deoxyribofuranosyl)-2-amino-6-mercaptopurine had a activity of 1.4×10^4 c.p.m./mg.

65-259

VON SCHUCHING S., FRYE G. H.

New synthesis of L-xylose-5-t (I).

J. Org. Chem. **30**, 1288 (1965)

Partial hydrolysis of 1,2,3,4-di-O-cyclohexylidene-L-xylofuranose to 1,2-O-cyclohexylidene-L-xylofuranose, catalytic oxidation, and hydrolysis of the calcium salt of 1,2-mono-O-cyclohexylidene-L-xyluronic acid to the acid, \rightarrow methyl ester, $+ \text{NaBT}_4 \rightarrow$ 1,2-mono-O-cyclohexylidene-L-xylofuranose-5- ^3H , \rightarrow I by hydrolysis. 52% radiochem. yield (0,7 mC/mg).

See also : **65-354**, UDP glucose-4- ^3H
UDP galactose-4- ^3H

1.1.7 — PEPTIDES, AMINO ACIDS, PROTEINS

65-260

AHNSTROEM G., EHRENBERG L., ROSEN C.-G.

Tritium labeling of cells in vitro.

Acta Chem. Scand. **19**, 964 (1965)

Labeling of protein molecules in living cells can be performed with isopropyl-1,3- ^3H methanesulfonate under conditions where no harmful effects on the cells accompany the treatment (See also : 64-287).

65-261

LEONIS J.

Studies on the preparation of labeled proteins and peptides.

EUR **1845.f** (1965)

Proteins of the egg white (lysozyme, ovalbumin, conalbumin, ovomucoid) could be labeled biosynthetically by incorporating tritiated tyrosine, tryptophan, arginine or lysine. Spec. act. from 1 to 100 mC/mmmole.

See also : **65-290**, Tryptophan - ^3H

65-296, S-(1,2-dichlorovinyl)-L-cysteine- ^3H

65-356, DL-Lysine-4,5- ^3H

1.1.8 — STEROIDS

65-262

AKHTAR M., GIBBONS C. J.

Synthesis of vitamin D $_3$ -9,19- $^3\text{H}_2$ (I) and the mechanism of the previtamin D $_3$ -vitamin D $_3$ reaction.

Tetrahedron Letters **1965** (9), 509CA **62**, 14766 f (1965)

19-Tolylsulfonyl-cholesteryl acetate \rightarrow 19-iodide, $+ \text{Zn}$ in T_2O , thermally rearranged \rightarrow 7-dehydrocholesterol-19- ^3H , photolysed to previtamin D $_3$ -19- ^3H . Thermal rearrangement to I.

65-263

PAREKH C. K., WASSERMAN R. H.

Preparation of ^3H -vitamin D_3 (I) using column and thin-layer chromatography.J. Chromatog. **17** : 261 (1965)N.S.A. **19**, 22273 (1965)

1) Tritiation of 7-dehydrocholesterol by the Wilzbach procedure with subsequent conversion to I.

2) Direct tritiation of vitamin D_3 at -190°C under nitrogen. A combination of column and thin-layer chromatographic procedures were necessary to purify the product resulting from the first procedure. The product resulting from the second procedure could be purified on thin-layer chromatographic plates alone.

65-264

BERSEUS O., DANIELSON H.
KALLNER A.**Synthesis and metabolism of cholest-4-ene- 7α , 12α -diol-3-one (I) and 5β -cholestane- 7α , 12α -diol-3-one (II). Bile acids and steroids.**J. Biol. Chem. **240**, 2396 (1965)

II from cholic acid- ^3H and isovaleric acid by electrolytic coupling and subsequent oxidn. with aluminum tert. butoxide. Spec. act. $14\ \mu\text{C}/\text{mg}$.

II, treated with SeO_2 gave **I**, spec. act. $8,2\ \mu\text{C}/\text{mg}$.

65-265

CRASTES de PAULET A., BARDOU L.

Synthesis of double labeled cholesteryl linolenate and arachidonate.EUR **2446.f** (1965)

Transesterification of cholesterol acetate + fatty acid methyl ester \rightarrow cholesterol ester + methyl acetate in the microscale (1 to $10\ \mu\text{M}$) : $4\text{-}^{14}\text{C}$ and $7\alpha\text{-}^3\text{H}$ cholesteryl linolenate and arachidonate, cholesteryl linolenate $\text{U-}^{14}\text{C}$ and arachidonate-(5,-6, 8-9, 11-12, 14-15) ^3H of high specific activity.

65-266

KLEIN P. D., KNIGHT J. C.

The exchange labeling of keto steroids with tritium by adsorption chromatography on basic alumina.J. Am. Chem. Soc. **87**, 2657 (1965)

The tritium is not removed by recrystallization in hydroxylic solvents but 95% can be back-exchanged under strongly basic conditions. Reduction of the keto steroids with lithium aluminum hydride occurs without loss of label and provides the corresponding alcohols in which the tritium is no longer exchangeable. The method provides pure, highly labeled products with remarkable economy of tracer. (e.g. cholest-7-en-3-one, $6,6\ \text{mC}/\text{mmole}$ and cholestane-3,6-dione, $9,7\ \text{mC}/\text{mmole}$).

65-267

SHIMIZU K.

Metabolism of cholest-5-ene- 3β , 20α -diol- 7α - ^3H and of cholest-5-ene- 3β , 17 , 20α -triol- 7α - ^3H (I) by human adrenal tissue.J. Biol. Chem. **240**, 1941 (1965)

I from 17α -hydroxypregnenolone- 7α - ^3H and isohexyl magnesium bromide (4.2×10^6 c.p.m. per mg).

1.2 — Carbon-13 and -14 compounds

1.2.1 — GENERAL

1.2.2 — ALIPHATIC COMPOUNDS

65-268

BOHLMANN F., VON KAP-HERR W.,
RYBAK Ch., REPPLINGER J.**Synthese und Biogenese von Anthemis-thioäthern.**Chem. Ber. **98**, 1736 (1965)

Condensation of octa-2,4,6-triyn-1-al and $1\text{-}^{14}\text{C}$ -bromoacetic acid methylester by the Wittig method to cis and trans dehydromatricaric acid methylester- $1\text{-}^{14}\text{C}$. Activity: 5.17×10^4 ipm/mmole.

65-269

BRUNSBURG U., BUNTE O.,
LINDSKOU G. I.

**Synthesis of bromoacetic acid-1,2-¹⁴C (I)
and glycolic acid-1,2-¹⁴C (II).**

Acta Chem. Scand. **19**, 246 (1965)

CA **62**, 14490 c (1965)

B₂CH₂ : CBrOEt-1,2-¹⁴C, hydrolyzed in the presence of 5 μl. 48% HBr → 85% I and 12.5% II, sepd. by paper chromatography or by pasting it with Na₂SO₄ and pentane.

65-270

EHRENFELD R. L.,
MORGENTHAU J. L. Jr.

Synthesis of ¹⁴CF₃CH₂OH.

Intern. J. Appl. Radiation Isotopes, **16**, 331 (1965)

Bromoacetic acid-1-¹⁴C (5 mmole) + SbF₄ → trifluoroethyl bromide (65%, more than 95% pure) + KOOCCH₃ + diethylene glycol → 2,2,2-trifluoroethanol-2-¹⁴C (80%, > 98% pure after destr.).

65-271

GNUCHEV N. V., NEIMAN L. A.,
POZNANSKAYA A. A.

**Synthesis of 5-aminolevulinic-5-¹⁴C acid (II)
and 4,5-dioxovaleric-5-¹⁴C acid (III).**

Biokhimiya **30**, 161 (1965)

CA **62**, 13034 e (1965)

Me 5-bromolevulinic-5-¹⁴C (I) + bromine, sapon. with HBr → 3,5-dibromolevulinic-5-¹⁴C acid, boiled 6 hrs. with H₂O → III.

K phthalimide and I → → 5-aminolevulinic-5-¹⁴C acid hydrochloride.

65-272

HUANG H. S., GOODMAN D. S.

Vitamin A and carotenoids. I. Intestinal absorption and metabolism of ¹⁴C-labeled vitamin A alcohol and β-carotene in the rat.

J. Biol. Chem. **240**, 2839 (1965)

¹⁴C-β-Carotene biosynthetically from 1-¹⁴C-acetate by the fungus *Phycomyces blakes-*

leeanus (strain (—)). TLC together with carrier β-carotene, on silica gel G indicated that 95% of the radioactivity migrated with the narrow band of β-carotene.

65-273

HUGHES C. A., GORDON-GRAY C. G.,
SCHLOSSER F. D., WARREN F. L.

The senecio alkaloids. Part XVII. A study of the formation of the total alkaloid in the plant in relation to the synthesis of the «necine» base and «necic» acid.

J. Chem. Soc. **1965**, 2370

Senecic acid + lead tetraacetate → 5-methyl 6-oxohept-cis-2-ene-3-carboxylic acid + sodium (¹⁴C)cyanide, subsequent hydrolysis → [6-carboxy-¹⁴C]5-methyl-7-hydroxyhept-trans-2-ene-3,6-dicarboxylic acid, integerrineic acid. Spec. act. 6.56 × 10⁸ c.p.m./mmole.

65-274

KOHLHAW G., DEUS B., HOLZER H.

Enzymatic preparation, structure, and properties of thiamine pyrophosphate-activated formaldehyde.

J. Biol. Chem. **240**, 2135 (1965)

2-¹⁴C-Glyoxylic acid from 2,3-¹⁴C-fumaric acid. Isolation by descending chromatography. Yield : 60%.

65-275

MONTGOMERY L. K.,
SCARDIGLIA F., ROBERTS J. D.

Evidence for cyclohexyne and cyclopentyne as intermediates in the coupling reactions of phenyllithium with 1-chlorocyclohexene and 1-chlorocyclopentene.

J. Am. Chem. Soc. **87**, 1917 (1965)

Adipic acid-1,6-¹⁴C (Ba salt) → cyclopentanone-1-¹⁴C (95%) + PCl₅ → 1-chlorocyclopentene-1-¹⁴C (52%). Degradn. by hydrolysis, addn. of Ph-Li to form 1-phenylcyclopentene-1-¹⁴C, and transformation to 5-phenylpentanoic acid by KMnO₄ oxidn., Wolff-Kishner redn. Subsequent Schmidt degradn.

65-276

MORTIMER G. A.

A new high-temperature free-radical source.J. Org. Chem. **30**, 1632 (1965)

Azobisisobutanol diacetate in an overall yield of 36% from azobisisobutyronitrile by methanolysis, redn. of the diester to a diol; and acetylation. Thus, it may be easily tagged with ^{14}C .

65-277

PICHAT L., GUERMONT J.-P.

Synthèses d'acides gras marqués au ^{14}C dans le groupe méthyle; acide caprylique ^{14}C -8; acide palmitique ^{14}C -16; acide stéarique ^{14}C -18.

Bull. Soc. Chim. France **1965** (2), 328

Palmitic acid-16- ^{14}C or stearic acid-18- ^{14}C in a total radioactive yield of 32 and 44% resp. from cyclopentadecanone and cycloheptadecanone resp. by addn. of $^{14}\text{CH}_3\text{MgI}$, dehydration, oxidn. with KMnO_4 and redn. of the semicarbazone by the Wolff-Kishner method. Caprylic acid-8- ^{14}C by addn. of $(^{14}\text{CH}_3)_2\text{Cd}$ to 6-carbomethoxyhexanoyl chloride. Yield: 29% (from $^{14}\text{CH}_3\text{I}$).

See also: **65-227**, Bicyclo(2.2.1)hept-2-ene-5-methylene- ^{14}C
Propene-1- ^{14}C , 1-Butene-1- ^{14}C

1.2.3 — AROMATIC COMPOUNDS

65-278

DIAMOND E. M., LEVY P. R.

Metabolism of hydroxyanthranilic acids. New synthesis of 3-hydroxyanthranilic acid-3- ^{14}C (I).

Rec. Trav. Chim. **84**, 285 (1965)CA **63**, 529 f (1965)

Phenol-1- ^{14}C + NaOH + Et p-toluene-sulfonate \rightarrow phenetole-1- ^{14}C + $\text{BzNO}^3 \rightarrow$ O-nitrophenetole-1- ^{14}C , PtO_2 catalyst/ $\text{H}_2 \rightarrow$ phenetidine-1- ^{14}C , + concd. H_2SO_4 , iodine and glycerol, \rightarrow 45% 8-ethoxyquinoline-

8- ^{14}C , \rightarrow N-benzyl-8-ethoxy-quinolinium-8- ^{14}C bromide, oxidn. with $\text{Ca}(\text{MnO}_4)_2 \cdot 2\text{H}_2\text{O} \rightarrow$ N-benzyl-3-ethoxy-N-formyl-anthranilic acid-3- ^{14}C , + red P, 55% HI, \rightarrow I, activity 28 ± 2 c.p.m./mmole.

65-279

OAE Sh., FURUKAWA N.

The mechanism of the base-promoted rearrangement of N,N-dichlorodiphenylethylamine (I) to desylamine (II).

Bull. Chem. Soc. Japan **38**, 62 (1965)

Phenylacetic acid-1- ^{14}C + $\text{PCl}_3 \rightarrow$ acylchloride, + benzene + $\text{AlCl}_3 \rightarrow$ deoxybenzoin-1- ^{14}C (75%), + NH_4OOCH + formic acid \rightarrow 1,2-diphenylethylamine-1- ^{14}C (85%), + tert-butylhypochlorite \rightarrow I-1- ^{14}C .

II degradn. to benzoic acid + benzonitril with $\text{Pb}(\text{ac})_4$ or to benzoic acid and benzophenone by subsequent treatment with PhMgBr , NaNO_2 , KMnO_4 .

65-280

PACKTER N. M., GLOVER J.

Biosynthesis of ^{14}C fumigatin in *Aspergillus fumigatus*, *fresenius*.

Biochim. Biophys. Acta, **100**, 50 (1965)

13% Fumigatol- ^{14}C , 9560 c.p.m./mg biosynthetically from sodium 2- ^{14}C -acetate (187 μC). A side product if fumigatin- ^{14}C .

65-281

PACKTER N. M., GLOVER J.

Biosynthesis of toluquinones in microorganisms.

Biochim. Biophys. Acta **100**, 57 (1965)

Fumigatin- ^{14}C (see preceding abstr.) + CH_3I (K_2CO_3) \rightarrow ^{14}C -ubiquinone-o (2,3-dimethoxy-5-methyl-p-benzoquinone). 4-Hydroxytoluquinone + metbanol- ^{14}C + $\text{H}_2\text{SO}_4 \rightarrow$ 4-methoxytoluquinone-(OCH_2 - ^{14}C). sp. act. : 28,290 c.p.m./mg.

65-282

PARSON W. W., RUDNEY H.

The biosynthesis of ubiquinone and rhodoquinone from p-hydroxybenzoate and p-hydroxybenzaldehyde in *Rhodospirillum rubrum*.

J. Biol. Chem. **240**, 1855 (1965)

p-Hydroxybenzaldehyde-U-¹⁴C and -carbonyl-¹⁴C from L-tyrosine-U-¹⁴C and -3-¹⁴C by enzymatic transamination, (tyrosine- α -ketoglutarate transaminase) to p-hydroxyphenylpyruvate and decarboxylation. Radiochem. purity 99.5%. The over-all yield was 39 to 40%.

65-283

ZALESSKAYA T. E., REMIZOVA T. B.

The mechanism of isomeric transformations of ketones. IV. The action of perchloric acid on tert-amyl phenyl ketone containing ¹⁴C in the carbonyl.

Zh. Obshch. Khim. **35**, 31 (1965)CA **62**, 13079a (1965)

$\text{EtMe}_2\text{CMgCl} + ^{14}\text{CO}_2 \rightarrow \text{EtMe}_2\text{C}^{14}\text{CO}_2\text{H}$,
 $+ \text{SOCl}_2; + \text{PhMgBr} \rightarrow 62.3\% \text{ EtMe C}^{14}\text{COPh}$.

See also : **65-294**, 3-(3,4-dihydroxyphenyl)-alanine-2-¹⁴C

65-326, Arsanilic acid-1-¹⁴C

1.2.4 — HETEROCYCLIC COMPOUNDS

65-284

BADIELLO R., BRECCIA A.

Synthesis of 3-indolyl-derivatives labeled with ¹⁴C.

Ric. Sci. Rend. Ser. A (2), **6**, 357 (1964)N.S.A. **19**, 24600 (1965)

The compounds are : Gramine, N-carbo-benzoxy-tryptophan, 3-indolylaldehyde, 3-indolylglyoxylic acid, 4-indolyl-acrylic acid, 3-indolylpyruvic acid, and 3-indolylacetamide.

65-285

GOTO M., OKADA T., FORREST H. S.

Synthesis of 2-amino-4-hydroxy-6-(D-erythro-1',2',3'-trihydroxypropyl)pteridine - 3'-phosphate-10-¹⁴C(I) and its metabolism in *Drosophila melanogaster*.

Arch. Biochem. Biophys. **110**, 409 (1965)

2,4,5-Triamino-6-hydroxypyrimidine-5-¹⁴C (1,5 mC/mmole) + D-ribose \rightarrow corresp. 10-¹⁴C-pteridine deriv. (14%), + 2-cyanoethylphosphate \rightarrow I (22%).

65-286

HILL J. A., LEQUESNE W. J.

Isolation of a hitherto undescribed sulfate of cytosine and an improved preparation of the intermediate 1-cyano-2,2-diethoxyethane (I).

J. Chem. Soc. **1965**, 1515CA **62**, 13142 g (1965)

NaCN in $\text{Me}_2\text{SO} + (\text{EtO})_2\text{-CH}_2\text{CH}_2\text{Br} \rightarrow$
 I. Urea-¹⁴C and I \rightarrow cytosine-2-¹⁴C.

65-287

MICETICH R. G., MACDONALD J. C.

Biosynthesis of neospergillic and neo-hydroxyaspergillic acids.

J. Biol. Chem. **240**, 1692 (1965)

L-Leucine-1-¹⁴C methylester + glacial acetic acid \rightarrow cyclo-L-leucyl-L-leucyl (I)
 Degradn. of neospergillic acid-¹⁴C : $\text{Br}_2\text{-H}_2\text{O}$; Zn-acetic acid \rightarrow I, hydrolysis.

65-288

PYNADATH T. I., FINK R. M.

Enzymic preparation of radioactive orotidine 5'-phosphate.

Biochim. Biophys. Acta, **95** : 355 (1965)N.S.A. **19**, 20024 (1965)

Orotidine 5'-phosphate synthesizing system devoid of orotone-5'-phosphate decarboxylase activity from a pyrimidine-requiring mutant of *Neurospora crassa*. 79% yield of (7-¹⁴C)-orotidine-5'-phosphate.

65-289

WHITE E. H., WOERTHER H.,
FIELD G. F., McELROY W. D.

Analogues of firefly luciferin (I).

J. Org. Chem. **30**, 2344 (1965)

I - ^{35}S (D-(—)2-(6'-hydroxy-2'-benzothiazolyl)- Δ^2 -thiazoline-4-carboxylic acid) from ^{35}S -cysteine and 2-cyano-6-hydroxy-benzothiazole (II).

I - ^{14}C from 2-chloro-6-methoxybenzothiazole + K^{14}CN , the methoxyderiv. (40%) was hydrolysed with Py/HCl to II (32%), + D-cysteine $\rightarrow I$ (77%). An alternative synthesis started with 2-chloro-6-hydroxy-benzothiazole.

See also : 65-278, 8-Ethoxyquinoline-8- ^{14}C

1.2.5 — ALKALOIDS

65-290

BARTON D. H. R., KIRBY G. W.
PRAGER R. H., WILSON E. M.

On the origin of the C-1 fragment in indole alkaloids.

J. Chem. Soc. **1965**, 3990

[methyl- ^{14}C]-N-Methyltryptamine (I) from : 3-indolylglyoxalyl chloride + [^{14}C]methylamine \rightarrow N-methyl-3-indolyloxamide, + $\text{LiAlH}_4/\text{AlCl}_3 \rightarrow I$, 40% radiochem. yield from the amide. Tritiation of tryptophan by heating it in tritium-water/ H_2SO_4 . A parallel expt. in D_2O showed, that ca. 80% of the aromatic hydrogens had exchanged. Labeling of deoxyajmaline by the same way (60% exchange of aromatic hydrogens).

65-291

BATTERSBY A. R., FOULKES D. M.,
BINKS R.

Alkaloid biosynthesis. Part VIII. Use of optically active precursors for investigations on the biosynthesis of morphine alkaloids.

J. Chem. Soc. **1965**, 3323

The quadruply-labeled (+)-, and (—)-forms of reticuline have been synthesised. (^{14}C in the skeleton at C-3, in the 4'-methoxyl group, and in the N-methyl group, and with ^3H at C-1).

1.2.6 — AMINO ACIDS,
PEPTIDES, PROTEINS

65-292

ABRAMSKY T., SHEMIN D.

The formation of isoleucine from β -methyl-aspartic acid in *Escherichia coli* W.

J. Biol. Chem. **240**, 2971 (1965)

^{14}C -Methyl-labeled β -methylaspartic acid (0.05 mC/mmol) by condensing ethyl α -bromopropionate-3- ^{14}C and diethyl acetamidomalonate by the method of Dakin (ibid, **141**, 945). The DL-threo and DL-erythro diastereoisomers were separated by fractional crystallization (the erythro racemate contained about 25% of the threo isomers).

65-293

BRAZIL H., SRI RAM J.

Enrichment of proteins with amino acid residues, employing p,p'-difluoro-m,m'-dinitrodiphenyl sulfone (I).

Arch. Biochem. Biophys. **110**, 205 (1965)

To gelatin mixed with ^{14}C -amino acid to give a radioactivity of 0.25 μC , dissolved in 1% sodium carbonate I was added. The reaction mixture was dialyzed. The tyrosyl derivative was characterized by electrophoretic, ultracentrifugal, and spectral studies.

65-294

DUHM B., MAUL W.,
MEDENWALD H., PATZSCHKE K.,
WEGNER L. A.

Tierexperimentelle Untersuchungen mit α -Methyl-Dopa- ^{14}C unter besonderer Berücksichtigung der optischen Isomeren.

I. Mitt. : Synthese, Resorption, Ausscheidung und Metabolismus.

Z. Naturforsch. **20b**, 434 (1965)

Veratric acid-(carboxyl- ^{14}C), SOCl_2 , Rosenmund redn. \rightarrow veratraldehyde, glycid-ester synthesis with 2-chloro-propionic acid ethylester \rightarrow 1-(3',4'-dimethoxyphenyl)-acetone-1- ^{14}C \rightarrow via the hydantoin to D,L- α -methyl-dopa- β - ^{14}C . Separation to the optically pure isomers. Total yield (from BaCO_3) : 28%, sp. act. : 18,5 $\mu\text{C}/\text{mg}$.

65-295

GASPARINI G. M., PICHAT L.,
BARET Ch.

Synthèse de la DL-cystine ^{14}C -3.

Bull. Soc. Chim. France **1965**, 794

Methanol- ^{14}C as Ac-ester, + chlorine \rightarrow formaldehyde- ^{14}C , + acetamido malonate + piperidine \rightarrow Mannich base, quaternisation with CH_3I , + benzylmercaptan \rightarrow s-benzyl-N-acetyl cysteine ethylester, sapon., Na-treatment, oxidn. \rightarrow DL-cystine-3- ^{14}C , sp. act. : 17,5 mC/mmole, total yield : 22% from $\text{Ba}^{14}\text{CO}_3$.

65-296

KLUBES P.

Synthesis of S-(1,2-dichlorovinyl)-D-cysteine-3- ^{14}C and S-(1,2-dichlorovinyl)-L-cysteine-3- ^{14}C and their uses with E. Coli B.

Thesis, University of Minnesota, 1962

Diss. Abstr. **25** (7), 3825 (1965) (order No 63-43006)

Synthesis of the title compds. (DCVD) starting with formaldehyde- ^{14}C and diethyl N-dithiocarbonyloxyaminomalonic acid S-thiolcarbonyloxyaminomalonic acid hydrochloride, DL-cystine, S-benzyl-DL-cysteine, N-acetyl-S-benzyl-DL-cysteine, (resolution with long kidney acylase), S-benzyl-L-cysteine, plus N-acetyl-S-benzyl-D-cysteine, S-benzyl-D-cysteine.

Synthesis of tritiated L-DCVC from tritiated L-cystine gave a product having one-third the sp. act. of the starting material.

65-297

LONG D. A., TRUSCOTT T. G.

Peptide kinetics. Acid catalysed hydrolysis of glycyl-glycylglycine- $^{14}\text{C}_2$ (I).

Trans. Faraday Soc. **61**, 531 (1965)

CA **62**, 14816 h (1965)

Glycine-1,2- $^{14}\text{C}_2$ benzyl ester + carboxyglycylglycine with DCCI \rightarrow I-benzylester, catalytic hydrogenation \rightarrow I.

65-298

MORRIS C. J., THOMPSON J. F.

Conversion of m-carboxyphenylalanine to m-carboxyphenyl-glycine in wedgewood iris leaves.

Arch. Biochem. Biophys. **110**, 506 (1965)

m-Carboxylbenzyl bromide + diethyl-acetamido-malonate-2- ^{14}C (2,54 $\mu\text{C}/\mu\text{mole}$) + Na/EtOH (under strict exclusion of moisture), 18 h refluxed, \rightarrow m-carboxyphenylalanine-2- ^{14}C (30%).

65-299

PICHAT L., ROCHAS G.,
HERBERT M.

Préparation de l'acide DL- α -aminoadipique ^{14}C -6 à partir de cyanure de potassium ^{14}C et α -benzamido δ -valérolactone.

Bull. Soc. Chim. France **1965**, 1384

K^{14}CN and α -benzamido- δ -valerolactone in a sealed tube at 155° C and subsequent hydrolysis gave 34% DL- α -amino-adipic acid-6- ^{14}C , sp. act. : 11,7 mC/mmole after purifn. on Dowex 50 W-12.

65-300

REHBINDER D., GREENBERG D. M.

Synthese von ^{14}C -markierten Phospholipiden.

Ann. Chem. **681**, 182 (1965)

D- α , β -Dipalmitin + POCl_3 \rightarrow dipalmitin phosphatidic acid dichloride + N-Cbo-ethanolamine-1,2- ^{14}C (or N-Cbo-L-serine-U- ^{14}C benzylester) and subsequent hydrolysis and hydrogenation \rightarrow L-1,2-dipalmitin phosphate 2-amino-ethyl-U- ^{14}C ester (or L-serine-U- ^{14}C ester respect.). Yields and sp. act. : 37% (from ethanolamine), 1,8 mC/mmole and 26% (from serine), 1,7 mC/mmole. Radiochem. purity : 88% and 85% respect.

65-301

SCHÖBERL A., PAPE Ch.-V.

Notiz zur Darstellung von [1- ^{14}C]Serin.

Chem. Ber. **98**, 1688 (1965)

N-Chloromethylphthalimide + $\text{KCN-}^{14}\text{C}$ (2,3 mC/mmole) saponification to the

methylester, + methylformate, redn. by NaBH_4 , hydrolysis with $\text{HCl} \rightarrow$ serine- $1\text{-}^{14}\text{C}$.

See also : **65-271**, 5-Aminolevulinic acid- $5\text{-}^{14}\text{C}$.

1.2.7 — CARBOHYDRATES

65-302

CASTANERA E. C., HASSID W. Z.

Properties of uridine diphosphate D-glucuronic acid decarboxylase from wheat germ.

Arch. Biochem. Biophys. **110**, 462 (1965)

Uridine diphosphate-D-glucuronic acid, ^{14}C labeled in the glucuronic moiety by enzymic oxidn. of UDP-D-glucose (2 μmoles , 70 μC , + glycine buffer, pH 8.9, cysteine, DPN, enzyme; purifn. by paper electrophoresis and circular paper chrom.).

65-303

CIARANFI E.

Synthesis of the 3-methyl-glyceraldehyde (I) uniformly labeled with ^{14}C .

EUR 2441.i (1965)

I from acetylene, acetaldehyde and aldol, crotonaldehyde, epoxidation with H_2O_2 , hydrolysis in the presence of formic acid. Spec. act. of 0.485 mC/mmole (92% purity).

65-304

MENDICINO J., PICKEN J. M.

The biosynthesis of the branched chain sugar D-apiose in Lemna and parsley.

J. Biol. Chem. **240**, 2797 (1965)

D-Apiose- ^{14}C by exposing 20 g of Lemna to 20 mC of $^{14}\text{CO}_2$ in a closed system for 10 days; sp. act. : 5×10^5 cpm per μmole .

1.2.8 — STEROIDS

65-305

BELL R. G.

Metabolism of 3,5-cholestadiene- $4\text{-}^{14}\text{C}$ and coprostanol- $4\text{-}^{14}\text{C}$.

Thesis, St. Louis University, 1964.

Diss. Abstr. **25** (8) 4382 (1965)

3,5-Cholestadiene- $4\text{-}^{14}\text{C}$ (sp. act. 3.4×10^6 dpm/mg) by thermal decompn. of K cholesteryl- $4\text{-}^{14}\text{C}$ sulfate (See also 65-142).

65-306

BERSEUS O.

On the stereospecificity of 26-hydroxylation of cholesterol. Bile acids and steroids 155.

Acta Chem. Scand. **19**, 325 (1965)

Cholestérol-1,7,15,22,26- ^{14}C (30 μC) from 2- ^{14}C -mevalonic acid (0.1 mC, 3.9 mg) by incubation with a homogenate of 15 g rat liver.

65-307

KARAVOLAS H. J., ELLIOTT W. H., HSIA S. L., DOISY E. A. Jr., MATSCHINER J. T., THAYER S. A., DOISY E. A.

Bile acids.

XXII. Allocholic acid, a metabolite of 5 α -cholestan-3 β -ol in the rat.

J. Biol. Chem. **240**, 1568 (1965).

Cholesterol- $4\text{-}^{14}\text{C}$ was reduced catalytically to 5 α -cholestan-3 β -ol- $4\text{-}^{14}\text{C}$ by the method of Hershberg et al. (J.A.C.S. **73**, 1144).

Sp. act. : 1.27×10^7 d.p.m./mg

Proof of purity (after chrom. on Florisil) : oxidn. to cholestanone, chrom. with coprostanol.

See also : **65-265**, Cholesterol- $4\text{-}^{14}\text{C}$ esters
Cholesterol linolenate- $\text{U-}^{14}\text{C}$

1.3 — Halogen labeled compounds

65-308

AMIEL S., PAISS Y.

A criterion for selectivity in recoil labeling.

IA-907 Dec. 1964, 15 p.

N.S.A. **19**, 20020 (1965)

Possible applications of selective labeling for the separation of primary nuclear reaction products from the same nuclides produced by radioactive decay, are discussed (Recoil reactions with iodine).

65-309

COHEN E. D., TRUMBORE C. N.

Alkyl iodide-iodine exchange and the Szilard-Chalmers effect.

Science **148**, 1460 (1965)

The rate of isotopic exchange between liquid ethyl iodide and dissolved iodine is

of zero order with respect to iodine concentration. Previously reported values of organic « retention », obtained in studies of (n, γ) recoil in alkyl iodide, are placed in doubt.

65-310

ELIAS H., CHRIST O.,
ROSENBAUM E.

Kinetik des homogenen Isotopenaustausches zwischen stellungsisomeren Hexylhalogeniden und Halogenid-Ionen in Dimethylsulfoxid.

Chem. Ber. **98**, 2725 (1965)

1-, 2-, and 3-Chloro-hexane-³⁶Cl were labeled by exchange with Li³⁶Cl in DMSO, the corresp. bromo-hexanes ⁸³Br-labeled, by exchange with freshly neutron irradiated LiBr.

65-311

GROVES K., ROBBINS A. L.

Preparation of labeled 2,6-dichloro-4-nitroaniline (Botran).

J. Agr. Food Chem. **13**, 118 (1965)

CA **62**, 13068 b (1965)

Chlorination of p-nitroaniline with labeled chlorine gas.

65-312

BUI N. M., GILLET R., DUMONT P.

An improved synthesis of 5-iodo-2'-deoxyuridine-¹³¹I and 5-iodouracil-¹³¹I.

Intern. J. Appl. Radiation Isotopes **16**, 337 (1965)

Iodination of deoxyuridine or uracil with ICl. Yields of 52 to 60% and 65-75% respect. Efficiency of ¹³¹I incorporation : 45-55% and 60-64% respect, no chlorinated prod. and no 5,6 addition.

65-313

ELEKES I.

Description of the substances used in fat absorption examinations and analysis of triolein labeled with ¹³¹I.

Magy. Radiol. **16**, 267 (1964)

N.S.A. **19**, 14992 (1965)

Biochemical and physicochemical characteristics of triolein remained unchanged

during labeling with ¹³¹I if no more than 1% of the olefins are saturated with iodine. 20° C was the optimal temperature. A procedure for determining the amount of iodine addition is described.

65-314

ABDEL-WAHAB M. F.,
EL-KINAWY S. A.

Preparation of radioiodinated serum albumin and radio-iodinated egg albumin by gel filtration.

Intern. J. Appl. Radiation Isotopes **16**, 267 (1965)

Iodination of freshly prepared dried protein at pH 9.3 with K¹³¹I and separation from inorganic compounds with Sephadex G-25. Identical electrophoretic patterns and u.v. spectra before and after labeling.

65-315

HEIDEMAN Jr. M. L., LEVY R. P.,
McGUIRE W. L., SHIPLEY R. A.

¹³¹I-Labeled bovine thyrotropin ; preparation through a new micro technique of iodine distillation and studies of adsorption to glass and polyethylene.

Endocrinology **76** : 828 (1965)

N.S.A. **19**, 24594 (1965)

Bovine TSH preparations in pH 8, 0.2M, sodium borate buffer, were labeled through ¹³¹I₂ distillation, accomplished by controlled bubbling of air and gentle heating. Iodide was removed by dialysis. Good yields with calculated ratios of 0.75-2.4 iodine atoms per TSH molecule. Biol. activity was not significantly less than that of control TSH carried through the procedures except for iodination, but both were up to ca. 50% less active than starting TSH. Investigations on loss during storage under various conditions.

65-316

MERIMEE Th. J., PROUT Th. E.

Purification and study of the principal products contained in preparations of radio-insulin.

Metab. Clin. Exptl. **14**, 115 (1965).

N.S.A. **19**, 17828 (1965)

Separation and recovery of the two principal fractions of radioinsulin preparations: characteristics of degraded insulin-¹³¹I; radioactive contaminants were removed by a procedure based on the difference in affinity of cellulose for the pure insulin-¹³¹I and the radioiodinated peptides. Attempts to use Sephadex G-50 to isolate undegraded insulin-¹³¹I were not successful.

65-317

ROSA U., SCASSELLATI G.,
PENNISI F., AMBROSINO C.,
LIBERATORI J., FEDERIGHI G.,
DONATO L., BIANCHI R.

Protein radioiodination by an electrolytic technique.

Strahlentherapie, Sonderbände **60**, 258 (1965)

CA **62**, 16624 f (1965)

Radioiodine labeling of proteins with iodine, set free at the anode by electrolysis of a KI soln. in which a protein is dissolved. Preliminary expts. on amino acids involved in protein iodination proved that for a proper value of current unwanted oxidative reactions can be minimized (See also : 64-114).

1.4 — Phosphorus-32 compounds

65-318

SIUDA A.

Hot phosphorus atom reactions in liquid organic mixtures.

(PAN-576/V) (CONF-773-36) Nov. 1964, 15 p.

N.S.A. **19**, 17957 (1965)

The yield of ³²P-labeled phenylphosphonic acid formed in 1M benzene solutions of phosphorus trichloride as a result of the ³²P (n, γ)³²P reaction can be affected by surrounding molecules.

65-319

WEHRLI W. E., VERHEYDEN D. L. M.,
MOFFATT J. G.

Dismutation reactions of nucleoside polyphosphates, II. Specific chemical syntheses of α -, β -, and γ -³²P-nucleoside 5'-triphosphates.

J. Am. Chem. Soc. **87**, 2265 (1965)

ADP-morpholidate + ³²P-tributylammonium orthophosphate \rightarrow γ -³²P-ATP. p-Nitrobenzyl phosphoromorpholidate + ³²P-orthophosphate \rightarrow β -³²P-p-nitrobenzyl diphosphate, + AMP-morpholidate \rightarrow γ -p-nitrobenzyl β -³²P-ATP; hydrogenolysis to β -³²P-ATP. 2',3'-O-isopropylideneadenosine + ³²P-2-cyanoethyl phosphate \rightarrow ³²P-AMP \rightarrow phosphoromorpholidate, + pyrophosphate \rightarrow α -³²P-ATP. In a similar way : β , γ -³²P-nucleoside 5'-triphosphates.

1.5 — Sulfur-35 compounds

65-320

HEISE K., MITTAG E.

Organic compounds labeled with ³⁵S. Part I. Synthesis, purification and purity testing of high specific activity methionine-³⁵S. (I)

Kernenergie **8**, 181 (1965)

Methylmercaptan \rightarrow 5-(β -methylmercaptoethyl)-hydantoin-³⁵S saponifying with barite water \rightarrow I, spec. act. 75 mC/mmmole.

65-321

MERZ H., PFLEIDERER G.,
WIELAND T.

Synthesen von ³²S- oder ³⁵S-haltigen Maleinimiden.

Biochem. Z. **342**, 66 (1965)

Preparative methods (in gram- and milligram-scale) are given for the synthesis of N-ethylmercapto-ethyl-maleinimide, N-(4-sulfophenyl)-maleinimide and N-(acetyl-4-sulfamoyl)-maleinimide.

65-322

MOSTI R., DE MARIA V.,
DE MARCO C.

Enzymic preparation of hypotaurine-³⁵S (I).

Giorn. Biochim. **14**, 105 (1965)

CA **63**, 859 d (1965)

Oxidn. of cysteamine-³⁵S (570 μ C) by an enzyme obtained from horse kidney. I contained 70% of the initial radioactivity.

65-323

SARGENT J. R., CAMPBELL P. N.

The sequential synthesis of the polypeptide chain of serum albumin by the microsome fraction of rat liver.

Biochem. J. **96**, 134 (1965)

Methionine-³⁵S from yeast, grown on ³⁵SO₄²⁻. Approximate spec. act. : 430 mC/mmole. During storage in solid state, room temperature, for 2 months no significant radiochemical decomposition.

65-324

THUNEBERG L.

Synthesis of ³⁵S-p-toluene sulphonic anhydride (tosan) (I) of high specific activity.

Intern. J. Appl. Radiation Isotopes **16**, 413 (1965)

Semi-micro scale sulphonation of toluene with coned. ³⁵S-sulphuric acid and thionyl chloride (as water-removing agent) at 110° C; 85% p-and 15% o-isomer. Conversion to I by N,N'-di-(p-tolyl)-carbodiimide. Overall yield of 60-70% based on the sulphuric acid. Sp. act. up to 200 mC/mmole.

65-325

TUDBALL N., NODA Y.,
DODGSON K. S.

The metabolism of L-serylglycine O[³⁵S]-sulphate in the rat.

Biochem. J. **95**, 678 (1965)

Preparation of potassium L-serylglycine O-sulphate-³⁵S (I). Spec. act. 17 μ C/mg. Liberation of inorganic sulfate-³⁵S, when I was stored in solid form. Minimisation of this effect by storing in aq. soln. at -20° C.

See also : **65-289**, Luciferin-³⁵S

1.6 — Oxygen-18 compounds

1.7 — Nitrogen-15 compounds

1.8 — Miscellaneous

65-326

FREDRICKSON R. L.,
BOCCHIERI S. F., GLENN H. J.,
OVERBY L. R.

Synthesis of arsanilic-⁷⁴As acid (I) and arsanilic-¹⁴C acid (II).

J. Assoc. Offic. Agr. Chemists **48**, 10 (1965).

N.S.A. **19**, 15657 (1965).

I and II in 20-30% yield with high radio-purity by the way of the Bart reaction. (p-nitrophenyl diazonium borofluoride and sodium arsenite were coupled and p-nitrophenyl arsonic acid was reduced with iron and hydrochloric acid).

65-327

COHEN Y., WEPIERRE J., PONTY, D.

Fixation of chromium-51 by protein fractions from blood serum.

Strahlentherapie, Sonderbaende **60**, 273 (1965)

CA **63**, 1002a (1965)

⁵¹CrCl₃ was incubated with human and rabbit serum to det. the nature and extent of Cr³⁺ binding by protein. Binding is heterogeneous and it is necessary to det. sp. act. of the Cr³⁺ when labeling proteins.

65-328

DOWNES A. M., McDONALD I. W.

The chromium-51 complex (I) of ethylenediaminetetraacetic acid as a soluble rumen marker.

Brit. J. Nutr. **18** : 153 (1964)

N.S.A. **19**, 20026 (1965)

Aqueous solution of ⁵¹Cr-chromic chloride disodium EDTA, 100° for 1 hr, excess chromic ions precipitation by adding NH₄OH. The filtrate, containing I was used.

65-329

INGRAND J.

Contribution à l'étude des propriétés biologiques des composés marqués au radiochrome ^{51}Cr .

CEA-R-2585; 1964, 116 p.

N.S.A. 19, 14955 (1965).

The preparation and use of ^{51}Cr -labeled erythrocytes, hemoglobin, plasma proteins, and cytochrome C in biological studies are reviewed. (188 references).

65-330

CAMPBELL B. L., ELLIS W. R.

The development of oil-soluble gold-198 compounds for industrial radiotracing.

Intern. J. Appl. Radiation Isotopes 16, 257 (1965)

Gold (III) chloride/HCl solutions extraction by primary, secondary and tertiary amines. Amine-gold salts, stable under certain conditions.

65-331

DRASKOVIC R., CVORIC J., VUKICEVIC S., ILIC R.

On the new procedures for producing radioactive colloidal gold ^{198}Au in the « Boris Kidric » Institute at Vinca.

JPRS-29258 (from Tehnika (Belgrade), 20 : 228 (1965) 16 p.

N.S.A. 19, 20022 (1965)

Constant modification of Henry's method was used to produce colloidal ^{198}Au with an activity of 25 to 50 mC/ml and a dispersal of $200 \pm 50 \text{ \AA}$.

65-332

STERN H. S., ZOLLE I., McAFEE J. G.

Preparation of technetium (^{99m}Tc)-labeled serum albumin (human).

Intern. J. Appl. Radiation Isotopes 16, 283 (1965)

Addition of ^{99m}Tc (from a ^{99}Mo generator) to buffered albumin; separation of free Tc by passing the solution over an Amberlite IRA 400 (Cl^-) column, overlaid with Dowex 1×2 .

See also : 65-339, Colloidal gold-198, purity

2 — RADIODECOMPOSITION, STABILITY, STORAGE

65-333

AMERSHAM, BUCKS, ENG.
THE RADIOCHEMICAL CENTRE

The stability of labeled organic compounds.

R.C.C. Review 3 (1965) 15 p.

N.S.A. 19, 22280 (1965)

Primary internal and external radiation effects and secondary radiation effects, choice of diluent, effects of impurities and of temperature, methods of handling and sampling, use of free radical scavengers, chain reactions, and effects of radiation characteristics; decomposition independent of radiation and decomposition during experiments.

65-334

OSINSKI P.

Influence of molecular structure on the decomposition of steroids in solution by auto-irradiation.

EUR 2435.f (1965)

Increasing number of oxygen atoms favors decomposition, independent from their position in the molecule. The structure of the A-nucleus does not influence the decomposition rate. Various action of the side chain.

65-335

PHILLIPS G. O., DAVIES K. W.

Radiation chemistry of carbohydrates. Part XV. Mechanism of self-decomposition of [¹⁴C] D-glucose.

J. Chem. Soc. **1965**, 2654

Self-decomposition of freeze-dried D-glucose-¹⁴C (5,4 mC/mmmole) : 4% during 48 weeks. Initial-G : 26-29. Moisture increased the decomposition rate.

65-336

ANGHILERI L. J.

Stability of high specific activity iodine-131-labeled insulin.

Intern. J. Appl. Radiation Isotopes **16**, 336 (1965)

The final yield of radioiodination with the described technique was 45-57% and the spec. act. 700-800 mC/mg. A sample was kept at 4° C dissolved in borate buffer pH 8.0, and with 0.2 ml of Cutter human serum albumin in 5 ml.

The damaged insulin, formed as results of radiation breakdown, is very high (after 15 days 66,6%).

65-337

WANG Y.

A study of thyroid uptake of the disassociated I-131 from labeled preparations.

From Society of Nuclear Medicine 11th Annual Meeting, Berkeley, Cal. CONF-640609-4, 9 p.

N.S.A. **19**, 19731 (1965)

The stability of the various ¹³¹I labeled compounds employed in the medicine was evaluated by ¹³¹I thyroid uptake. Iodine-131-Rose Bengal and ¹³¹I-Hippuran are proven to be the most stable compounds.

65-338

PETROVIC S., BECAREVIC, A.,
PETROVIC J.

The isolation and stability of rapidly-labeled ribonucleic acids from rat liver microsomes.

Biochim. Biophys. Acta **95**, 518 (1965)

65-339

BEEN U., HOEYE A.

Determination of auric ions in colloidal gold-198 by paper electrophoresis.

J. Chromatog. **17** : 631 (1965)N.S.A. **19**, 24436 (1965)

Deviation of 30% or less for 1-2% ionic gold. The results also showed that 0,075 M sodium thiosulfate does not change the percentage of gold ions in colloidal ¹⁹⁸Au preparations.

See also : **65-244**, Methotrexate-3',5'-³H**65-315**, Bovine thyrotropin, -¹³¹I**65-323**, Methionine-³⁵S**65-325**, L-Serylglycine O-sulfate-³⁵S**65-371**, Hippuran-¹³¹I

3 — PURIFICATION, SEPARATION

65-340

DRYSDALE J. W., MUNRO H. N.

Small-scale isolation of ferritin for the assay of the incorporation of ¹⁴C-labeled amino acids.

Biochem. J. **95**, 851 (1965)

Removal of the tissue proteins by heat coagulation, chromatography of the ferritin fraction on CM-cellulose and Sephadex G-200.

65-341

MASSAGLIA A., ROSA U., SOSI, S.

Thin-layer chromatography of some ^{131}I and ^{125}I labeled iodopyrimidines and related nucleosides.J. Chromatog. **17**, 316 (1965)N.S.A. **19**, 22131 (1965)

R_F values of iodouracil, iodocytosine, iodouridine, iododeoxyuridine and iodocytidine on layers of silica gel G and cellulose G for a number of solvents.

65-342

PURDY H. R., GOLDMAN N. L., RICHARDSON G. S.

A procedure for the analysis of counter-current distribution data. III. A computer program for the statistical analysis of radiochemical purity.J. Biol. Chem. **240**, 1573 (1965)

Materials : 20α -hydroxypregn-4-en-3-one- 7α - ^3H , DL-leucine-4,5- ^3H , 17β -estradiol-4- ^{14}C .

The program is illustrated for distribn. in which the analysis of radioactivity and weight has been made on the same or alternate tubes.

65-343

TAYLOR K. W., GARDNER G., PARRY D. G., JONES V. E.

The purification of tritium-labeled insulin by precipitation with insulin antibodies.Biochim. Biophys. Acta **100**, 521-529 (1965)

Purification of insulin labeled by incubating tritium-labeled leucine with slices of ox pancreas in vitro on a small scale by precipitation with antibody and further purification by paper chromatography and paper electrophoresis.

65-344

BENTLEY R., SAHA N. C., SWEELEY C. Ch.

Separation of protium and deuterium forms of carbohydrates by gas chromatography.Anal. Chem. **37**, 1118 (1965)

On 15- to 25-meter, narrow-bore packed columns of SE-30 and other liquid phases complete sepn. of trimethylsilyl derivs. of β -glucose from β -glucose- d_6 .

65-345

KIRSCHNER M. A., LIPSETT M. B.

Isotope effects in gas-liquid chromatography of steroids.J. Lipid. Res. **6** : 7 (1965)N.S.A. **19**, 19893 (1965)

Tests were conducted on testosterone-4- ^{14}C and dehydroepiandrosterone-4- ^{14}C acetylated with acetic anhydride-1- ^3H , and with testosterone-1,2- ^3H and dehydroepiandrosterone- 7α - ^3H acetylated with acetic anhydride-1- ^{14}C . $^3\text{H}/^{14}\text{C}$ ratio varied among the fractions collected every 20 sec throughout the steroid peak (columns of 2000 to 3000 theoretical plates).

65-346

VAN HOOK W. A., KELLY M. E.

Gas liquid chromatography at low temperatures. Resolution of some deuterated ethanes.Anal. Chem. **37**, 508 (1965)

Chromatograms for the systems C_2H_6 - C_2D_6 ; C_2H_6 - $\text{C}_2\text{H}_4\text{D}_2$, 1,1d; and C_2H_6 - $\text{C}_2\text{H}_5\text{D}$ between 158° K. and 273° K. Methylcyclopentane on fire brick.

See also : **65-247**, 2,4-Dinitrofluorobenzene-3,5- ^3H

65-263, Vitamin D_3 - ^3H **65-338**, Ribonucleic acids- ^{14}C **65-350**, Ethylene and CO_2 sepn.**65-368**, Thin-layer chromatography for purity checking

4 — ANALYSIS

4.1 — Substances

4.1.1 — *DETERMINATION OF ACTIVITY*

65-347

GEORGI P., LOBER M.,
WANGERMANN G.**Measurement of $^{14}\text{CO}_2$ in the liquid scintillation counter with β -phenylethylamine in a dioxane-naphthalene mixture.**Acta Biol. Med. Ger. **13**, 629 (1964)CA **62**, 16620 d (1965)

Higher count efficiency and better soly. than comparable mixtures.

65-348

LONG C. L., GEIGER J. W.

Liquid scintillation counting of the potassium gluconate derivative of blood glucose.Anal. Biochem. **10**, 253 (1965)N.S.A. **19**, 15354 (1965)

Conditions for the radioactive assay of the water-soluble title compd. are given.

65-349

PICARD M. A., KIRCHMAN R.,
DULCINO J., MASSAUX F.**Measurement of β -rays from carbon-14 using liquid scintillators.**Bull. Inst. Agron. Sta. Rech. Gembloux **31**, 550 (1964)CA **62**, 16621 b (1965)The effects of plant material and gel (Cab-O-sil) on quenching of sucrose- ^{14}C activity were observed. Scintillants PPO and POPOP and solvents toluene or dioxane were used. Dried pulverized apple leaves, hyamine or 70% EtOH exts. of these, and residues from these exts. were tested.

65-350

SHIMOKAWA K., KASAI Z.

Liquid scintillation counting method of ethylene- ^{14}C .Radioisotopes (Tokyo), **14**, 137 (1965)N.S.A. **19**, 24465 (1965)Ethylene- ^{14}C evolved by plant tissue was absorbed in 0,25M mercuric perchlorate, released by addition of 4N lithium chloride, and reabsorbed by 0,1M methanol solution of mercuric acetate. (Contamination of $^{14}\text{CO}_2$ to ethylene- ^{14}C : 0,02 % of total $^{14}\text{CO}_2$.)

Counting in toluene : ethylcellosolve : dioxane (1 : 1 : 1), efficiency : 59%.

65-351

DARUSCHY P.

A fast and simple method for demonstrating tritium in biological material.Atompraxis **11**, 273 (1965)

The water, containing the tritium is removed from the material with the aid of simple xylene distillation, and is then measured in a liquid scintillation counter.

65-352

CARR R. J., GORDON B. E.

Determination of polyacrolein labeled with tritium or carbon-14.J. Polymer Sci. Pt. C **8**, 71 (1965)CA **63**, 1869 d (1965)For T analysis, ethanolic toluene was the absorbent for the water, and for ^{14}C , methanolic benzylamine was used to absorb the CO_2 . Soot and dissolved O caused errors. Advantages : specificity, multiple analysis, convenience, and low cost.

65-353

KUSAMA K.

An improved method for liquid scintillation counting of biological compounds in aqueous solution.

Radioisotopes (Tokyo) **14** : 142 (1965)

N.S.A. **19**, 24466 (1965)

For measuring nucleic acids and nucleotides, 10 ml of toluene scintillator, 0.5 ml of 1M hyamine 10-X chloride in ethanol, and up to 0.3 ml of aqueous sample solution taken. Max. efficiency for ^3H is 18.3% and 53% for ^{14}C .

4.1.2 — DETERMINATION OF RADIOCHEMICAL PATTERN

65-354

BEVILL III R. D., HILL E. A., SMITH F., KIRKWOOD S.

Mechanism of action of UDPgal-4-epimerase : isotope effect studies.

Can. J. Chem. **43**, 1577 (1965)

Synthesis of uridine diphosphate glucose and uridine diphosphate galactose labeled with T in the 4-pos. A degradative procedure that will permit the location and quantitation of the carbon-bound tritium in any hexose or pentose was developed (methyl- α -D-glucopyranoside, periodate oxidn., D'-methoxy-D-hydroxymethyl-diglycolic aldehyd, oxidn. and hydrolysis to D-glyceric acid or redn. to glycerol finally).

65-355

JAMES A. T., HADAWAY H. C., WEBB J. P. W.

The biosynthesis of ricinoleic acid.

Biochem. J. **95**, 448 (1965)

Degradn. of ricinoleic acid from ^{14}C labeled precursors : Redn. (Adams catalyst) \rightarrow 12-hydroxystearic acid + SOCl_2 then KOH \rightarrow octadec-12- and 11-enoic acid, redn. \rightarrow stearic acid, partial oxidn. by KMnO_4 .

65-356

POPENOE E. A., ARONSON R. B., VAN SLYKE D. D.

The formation of collagen hydroxylysine studied with tritiated lysine.

J. Biol. Chem. **240**, 3089 (1965)

DL-Lysine-4,5- ^3H by catalytic hydrogenation of acetamido-(4-amino- Δ^2 -butenyl)-malonic acid diethyl ester hydrochloride. To confirm the location of the T label degradn. with permanganate to glutaric acid and with permanganate, hydrazoic acid and permanganate again to succinic acid. All tritium in pos. 4 and 5.

See also : **65-236**, Prostaglandin - E_2 -17, 18- ^3H

65-245, Anthranilic acid - ^3H

65-275, 1 -Clorocyclopentene -1 - ^{14}C

65-279, Desylamine - ^{14}C

65-287, Neoaspergillic acid - ^{14}C

4.2 — Apparatus

65-357

EDWARDS B., KITCHENER J. A.

A routine method for determination of ^{14}C by liquid scintillation.

Intern. J. Appl. Radiation Isotopes **16**, 445 (1965)

Special flasks, serving both for wet oxidn. of the compd. and subsequently for absorption of CO_2 direct to the liquid scint. counting phials (hyamine hydroxide/methanol). Efficiency about 40%.

65-358

IWAKURA T., KASIDA Y., MORISAKI N.

^{14}C and ^3H measurement by the use of a liquid scintillation spectrometer. IV. Determination of the counting efficiency by the discriminator ratio method.

Radioisotopes (Tokyo), **14**, 132 (1965)

N.S.A. **19**, 24464 (1965)

65-359

SLOT C.

Liquid scintillation counting of nonvolatile water-soluble compounds.

Scand. J. Clin. Lab. Invest. **17**, 182 (1965)

CA **63**, 915a (1965)

Glass fiber as a vehicle for counting ^{14}C -labeled creatine and ^{35}S -labeled thiamine in

serum and urine. Higher counting efficiency than filter paper and excellent reproducibility.

65-360

SPROTT W. E.

A liquid scintillator-based continuous radio chromatogram scanner.

J. Chromatog. **17** : 355 (1965)N.S.A. **19**, 22132 (1965)

Developed paper chromatograms are directly impregnated with a liquid scintillator. Direct quantitative assay of at least 0.1 nC of ^{14}C per spot of 5 cm² or more surface area.

65-361BAGGETT B., PRESSON T. L.,
PRESSON J. B., COFFEY J. C.

Correction for quenching of samples from an oxygen flask combustion method for tritium analysis.

Anal. Biochem. **10** : 367 (1965)N.S.A. **19**, 15356 (1965)

Immediately after the aliquots for counting are pipetted into vials, a stream of compressed air is bubbled through each sample at a rate of about 80 ml/min for 2 min. Samples are recounted after the addition of an internal standard of tritiated palmitic acid in toluene.

A combination of flushing and correction with internal standards gave recoveries of 94-97%.

65-362

JORDAN P., LYKOUREZOS Ph. A. P.

Restlose Ueberführung organischer Verbindungen in gasförmige Produkte zur gleichzeitigen Bestimmung von ^3H und ^{14}C in Proportionalzählrohren.

Helv. Chim. Acta **48**, 581 (1965).

Pyrolysis of organic compds. over NiO-Zn catalysts. Formation of gaseous reactions products only (H_2 , CH_4) in one step.

65-363

PATTERSON M. S., GREENE R. C.

Measurement of low energy beta-emitters in aqueous solution by liquid scintillation counting of emulsions.

Anal. Chem. **37**, 854 (1965)

Toluene : Triton X-100, 2 : 1, can be mixed with 23% water, counting efficiencies of 10% for ^3H and 68% for ^{14}C ; toluene : Triton : ethanol, 8 : 4 : 3, can hold 43% water (58% eff. for ^{14}C) toluene : triton, 7 : 6, forms a fluid emulsion with 43% water (57% efficiency for ^{14}C).

65-364

SCHARPENSEEL H. W. PIETIG F.

Inorganic scintillators for measurement of ^{14}C and ^3H in flow cells of radiochromatographs.

Atompraxis **11**, 98 (1965)CA **63**, 222 g (1965)

Scintillators incorporating ZnS-Ag, CaWO_4 , and Li-Ce glass were evaluated. Detection efficiencies in the range of 1.6-2.6% for ^{14}C and 0.12-0.27% for ^3H .

65-365

POLESKY H. F., SELIGSON D.

Application of liquid scintillation counting to ^{131}I measurement.

Anal. Biochem. **10**, 347 (1965)

Standard liquid scintillation techniques are feasible for counting the beta emission of ^{131}I and are up to seven times more efficient than a standard gamma detection system.

65-366BERGER J.-A., MEYNIEL G.,
PERRIN J.

Comparative study of the measurement of β -activities of thin chromatographic layers; influence of the detector, of the nature of thin layer and of the β -ray energy.

Intern. J. Appl. Radiation Isotopes **15** : 671 (1964)N.S.A. **19**, 19792 (1965)

65-367

BOUCKE, G.

Activity measuring on thin layer chromatograms.

Atompraxis **11**, 263 (1965)

With gas flow counters a counting efficiency of about 5% is obtained for ^{14}C and ^{35}S . For ^3H a windowless gas flow counter gives an efficiency of 1% approximately. Measuring time can be shortened by automatic change of the scanning speed controlled by the count rate. Hints for detecting low activities and for two-dimensional techniques.

65-368

SNYDER F., KIMBLE H.

An automatic zonal scraper and sample collector for radioassay of thin-layer chromatograms.

Anal. Biochem. **11**, 510 (1965)

CA **63**, 2102 d (1965)

1-, 2-, and 5-mm Wide zones of adsorbent are removed. Radioactivity plotted as a continuum of adjacent zones along a chromatostrip provides an extremely sensitive radioindicator of contamination of com. available labeled compds. and of closely related biol. compds. chromatographed by TLC.

65-369

SCHARPENSEEL H. W.

A multipurpose high-vacuum line for gas-phase operations in the radiocarbon and tritium techniques.

Atompraxis **11**, 147 (1965)

CA **63**, 1482 h (1965)

Vacuum system for prepn. of labeled ^3H and ^{14}C compds. and including drying, vacuum distn. fractionation, degradation, synthesis, direct labeling by Wilzbach and Turton (^{14}C) techniques, and measurement of gaseous ^3H and ^{14}C by internal gas counting.

4.3 — Radiochem. methods for analysis of complex compounds

65-370

OVERBY L. R., BOCCHIERI S. F.,
FREDRICKSON R. L.

Chromatographic, electrophoretic, and ion exchange identification of radioactive organic and inorganic arsenicals.

J. Assoc. Agr. Chemists, **48**, 17 (1965)

N.S.A. **19**, 15387 (1965)

Qualitative and quantitative estimation of arsenate- ^{74}As , arsenite- ^{74}As , and arsanilic- ^{74}As acid.

65-371

HOSICK T. A., WATTS F. C.,
MESCHAN I.

Determination of radioiodide contamination of radioiodinated organic salts.

J. Nucl. Med. **6**, 136 (1965)

N.S.A. **19**, 22398 (1965)

The disproportionation of the original radioactivity between the aqueous and I_2 saturated CCl_4 layers after equilibrium is determined.

65-372

LAMBIOTTE M.

Autoradiographic electrophoresis of tritium-labeled compounds in a thin layer of buffered photographic gelatine.

Nature **207**, 516 (1965)

See Abstr. 65-207.

5 — MISCELLANEOUS

65-373

GARFINKEL S. B., MANN W. B.,
MEDLOCK R. W., YURA O.

**The calibration of the National Bureau
of Standards' tritiated-toluene standard of
radioactivity.**

Intern. J. Appl. Radiation Isotopes, **16**,
27 (1965)

65-374

SCHARFFENBERG R. S.
POLLARD Jr. J. K.

**Carbon-14. A comprehensive annual biblio-
graphy of applications in chemistry, biology
and medicine. Volume 2.**

Calbiochem. 1964, 126 p.

N.S.A. **19**, 14947 (1965)

References (2956) are given to publications
from 1962 through 1963. (See also 64-208).

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