ABSTRACTS *

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* The references given in this section cover articles drawn from 40 primary periodicals and also from N.S.A. and C.A.

A point is made of singling out information about synthetizing and related problems particularly where the main part of the abstracted article deals with applications.

Author and subject indexes will be published for each volume of this journal.

The results obtained from May 1964 until December 1964 have been published in Euratom reports EUR 2212 e and EUR 2212 e suppl.

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1 — SYNTHESIS

1.0 — Deuterium compounds

1.0.1 — *GENERAL*

See also : **66-97,** Labeling by exchange on GLC column

1.0.2 — *ALIPHATIC COMPOUNDS*

66-1

BINSCH G., ROBERTS J. D.

Conformational barriers in medium-sized rings. I. trans-Cyclodecene-1,2,4,4,9,9-d_6

J. Am. Chem. Soc. **87**, 5157 (1965)

Diethyl adipate + $LiAlD_4$, + HBr → 78% 1,6-dibromohexane-1,1,6,6-d_4, + Na diethyl malonate (excess), hydrolysis, -CO_2 → 76% sebacic acid-β,β,β′,β′-d_4, + CH_2N_2, acyloin ring closure, oxidn. to diketone, → corresp. bishydrazone, HgO → 57% cyclodecyne-4,4,9,9-d_4, + B_2D_6, cleavage with deuterioacetic acid → cis-cyclodecene-1,2,4,4,9,9-d_6, benzene photosensitized irradn. in dilute soln. → 17% title compd. (sep. from cis isomer in a 16 step countercurrent distrn., less than 0.5% cis-contamination).

66-2

BOWIE J. H., LAWESSON S.-O., SCHROLL G., WILLIAMS D. H.

Studies in mass spectroscopy. III. Mass spectra of β-keto esters.

J. Am. Chem. Soc. **87**, 5742 (1965)

Ethyl acetoacetate and $D_2{}^{18}O$ applied to a tandem g.l.p.c. column, at 60°, 16 min. → 66% incorporation of ^{18}O, → ethyl 3-oxobutyrate-2,2-d_2-3-^{18}O.

66-3

BROWN L. M., OCCOLOWITZ J. L.

A non-classical carbanion.

Chem. Commun. **1965** (16), 376.
CA **63**, 14660c (1965)

Bicyclo(3.2.1)octa-2,6-diene-4,4-d_2 → bicyclo(3.2.1)oct-2-ene-4,4-d_2 (81% d_2) by redn. with benzenesulfonyl hydrazide in diglyme at 110° (98% yield).

66-4

CASERIO M. C., LEVIN R. D., ROBERTS J. D.

Intermediates in the nitrous acid deamination of 2-(p-(2′-hydroxyethoxy)phenyl) ethylamine.

J. Am. Chem. Soc. **87**, 5651 (1965)

p-Hydroxyphenylacetic acid → amide, + K-tert.-butylate, + ethyl chloroacetate → ethyl p-acetamidophenoxyacetate + $LiAlD_4$ → title compd., D lab. in pos. 1,1,2′,2′.

66-5

DJERASSI C., FENSELAU C.

Mass spectrometry in structural and stereochemical problems. LXXXIV. The nature of the cyclic transition state in hydrogen rearrangements of aliphatic ethers.

J. Am. Chem. Soc. **87**, 5747 (1965)

Ethyl sec-butyl ether, isopropyl n-butyl ether, isopropyl n-pentyl ether with D in various pos. of the butyl- or pentyl moiety and ethyl 4,4-d_2-n-hexyl ether were prepd.

66-6

FISCHER M., PELAH Z., WILLIAMS D. H., DJERASSI C.

Mechanismus der Reduktion von Tosylhydrazonen mit komplexen Metallhydriden.

Chem. Ber. **98**, 3236 (1965)

The title compds. and $LiAlD_4$ (I) or $NaBD_4$ (II) give monodeuterated hydrocarbons. Examining the stereochemical mechanism with steroids, the ratio of $\alpha:\beta$ epimers was 7 : 3 with I and 3 : 7 with II resp.

66-7

HINE J., HOUSTON J. G.,
JENSEN J. H., MULDERS J.

Catalysis of α-hydrogen exchange. I. The reaction of isobutyraldehyde-2-d with tertiary amines and oxygen bases.

J. Am. Chem. Soc. **87**, 5050 (1965)

Isobutyraldehyde-2-d by the reaction of deuterium oxide with isobutenyl acetate in the presence of a little deuterium sulfate, yield 80%.

66-8

JAKLI G., JANCSO G., ILLY J.

Preparation of deutero alcoholates.

Kozlemeny. **13**, 235 (1965)
CA **63**, 17874g (1965)

MeOD and EtOD via the Na alcoholates higher alcohol via Mg and Al alcoholates, 90% D content.

66-9

KING J. F., DURST T.

Sulfenes in the base-induced solvolysis of alkanesulfonyl chlorides.

J. Am. Chem. Soc **87**, 5684 (1965)

Phenylmethanesulfonyl chloride (I) + dioxane/D_2O, + triethylamine, evaporation, + PCl_5, repeating the procedure 5 times → I-1,1-d_2, yield 50%.

66-10

LAMATY G., TAPIERO C., WYLDE R.

Synthèse de t-butyl-4 d-2 cyclohexylamines.

Bull. Soc. Chim. France **1965**, 3085

B_2D_6 (from BF_3. Et_2O + LiD) + 4-tert-butylcyclohexene → corresp. organoborane + p-$MeC_6H_4SO_2NHCl$ → 5- and 4-tert-butyl-cis-p-toluenesulfonamido-cyclohexane-cis-2-d, sepn. by chromatography.

66-11

McLEAN S., HANES P.

Hydrogen migration in cyclopentadienes.

Tetrahedron **21**, 2329 (1965)
CA **63**, 17834f (1965)

Addn. of 0.1 Mole Na cyclopentadienide in 100 ml. diglyme at −10° to 0.225 mole D_2O in 100 ml. diglyme at −10° the mixt. kept 10 min. and distd. at −10° yielded 3.1 g. cyclopentadiene-5-d.

66-12

MEINWALD J., SCHNEIDER R. A.

Photochemical synthesis and reactions of carvonecamphor.

J. Am. Chem. Soc. **87**, 5218 (1965)

Carvonecamphor (I) in ethanol-d + K-t-butoxide → I-α, α-d_2, irradn. in methanol, 12 days → 65% 1-exo-5-dimethyl-syn-2-methoxycarbonylmethyl-d-bicyclo (2. 1. 1)-hexane-5-d.

66-13

MEYERSON S., LEITCH L. C.

Organic ions in the gas phase. XVII. A bicyclic doubly hydrogen-bridged-transition state in decomposition of 6-substituted alkanoic acids and esters.

J. Am. Chem. Soc. **88**, 56 (1966)

6-Phenylhexanoic acid (I)-6-d from 3-phenylprop-2-enol, + $LiAlH_4$, D_2O, HBr, Grignard reagent, + trimethylene oxide-(II)...
I-5,5-d_2 by redn. of phenylacetic acid with $LiAlD_4$, chain extention with II and CO_2,
I-4,4-d_2 from phenylpropionic acid + $LiAlD_4$ and then addn. of II.

66-14

SAMAKHOV A. A., POZDNEV V. F.

Apparatus for catalytic synthesis of deuterio-methyl alcohol in considerable quantities.

Tr. Gos. Inst. Prikl. Khim. **52**, 96 (1964)
CA **63**, 13058e (1965)

A 1 : 2 mixt. of CO and D, 200 atm., reactor packed with 1-2 mm. grains of an $8ZnO.-Cr_2O_3$ catalyst, 380-400°. The product contained 99.5-99.7% alc. and 98-98.5 at. -% D, yield 80-82%, based on D, up to 2 kg. CD_3OD per 24 hrs.

66-15

SHAPIRO S.S., DENNIS D.

Lactic acid racemization in Clostridium butylicum. Evidence for a direct internal hydride shift.

Biochemistry **4**, 2283 (1965)

D-(—)-lactic acid-α-d enzymatically from sodium pyruvate and deuterated $NADH_2$ (from CH_3CD_2-OH).
L-(+)-lactic acid-α-d by coupling the enzymatic reactions of alcohol dehydrogenase from yeast and beef heart L-(+) lactic acid dehydrogenase.

66-16

STREITWIESER A. Jr.,
CALDWELL R. A.

Acidity of hydrocarbons. XIX. Kinetics and mechanism of exchange of benzene and sec-butylbenzene with cesium cyclohexylamide.

J. Am. Chem. Soc. **87**, 5394 (1965)

PhMgBr + 2-butanone → 2-phenyl-2-butanol, → lithium salt, + methyl p-toluenesulfonate → 2-phenyl-2-methoxybutane, cleavage with sodium-potassium alloy and quenching with tritiated D_2O → 2-phenylbutane-2-d,^{3}H.

66-17

WILLHALM B., THOMAS A. F.

Mass spectra and organic analysis. Part VI. The mass spectra of menthone, isomenthone, and carvomenthone.

J. Chem. Soc. **1965** 6478.

Menthone (I)-1-d from piperitone and Li in liquid ND_3. Isotopic purity 82%. In a similar manner I-5-d from menth-3-one-5-one and I-8-d from pulegona, 85 and 75 at. % D resp. Piperitone-9,10-d_6 (II) from isopropyl iodide-1,1,1,3,3,3-d_6 + ethyl sodioacetoacetate, + but-3-en-2-one.

Yield 11,5% based on perdeuterioacetone. Redn. of II → menthone-9, 10-d_6.
Exchange in dioxan-D_2O of piperitone, catal. redn. and back-exchange → menthone-6, 7-d_5 (d_5 : 23%).

1.0.3 — *AROMATIC COMPOUNDS*

66-18

GERSON F., HEILBRONNER E.,
BÖLL W. A., VOGEL E.

Die ESR.-Spektren der Radikal-Anionen 1,6-überbrückter Cyclodecapentaene.

Helv. Chim. Acta **48**, 1494 (1965)

1,6-Methano-cyclodecapentaene (I)-2-d, -2,7-d_2 and -2,10-d_2 resp. from the corresp. bromo deriv. via the Grignard reagent. I-2,5,7,10-d_4 by exchange with deuteriotrifluoroacetic acid. (3 exchanges, 40% yield.) 1,6-Oxidocyclodecapentaene-2,5,7,10-d_4 by exchange with dimethyl sulfoxide-d_6 and K-tert.-butylate. Yield 68%.

66-19

HARPER R. J., KEMBALL C.

The mechanism of exchange and deuteration of aromatic hydrocarbons on metal films. Reactions of p-xylene.

Proc. Intern. Congr. Catalysis. 3rd, Amsterdam, 1964 2, 1145 (Pub. 1965)
CA **63**, 12994a (1965)

The reactions studied include : Exchange of the hydrogen atoms in the methyl groups, on the ring, formation of 1,4-dimethylcyclohexane, replacement of the hydrogen atoms during the formation of the saturated compound.

66-20

HELGSTRAND E.

Primary hydrogen isotope effects in the bromination of 2-bromo-1,3,5-trimethoxybenzene and 2,4-dibromo-1,3,5-trimethoxybenzene. Proximity effects of bromine.

Acta Chem. Scand, **19**, 1583 (1965)

1,3,5-Trimethoxybenzene (I) + D_2O and acetic acid → partially deuterated I (to an extent of 50% for aromatic hydrogens), bromination with N-bromosuccinimide → 2-bromo-I-d and direct bromination → 78% 2,4-dibromo-I (83 atom % D).

66-21

LAMBERT J. B., ROBERTS J. D.

Stereochemical aspects of the Diels-Alder reaction.

Tetrahedron Letters **1965**(20), 1457.
CA **63**, 5501d (1965)

α-Methyl-trans-β-bromostyrene, Li-adduct, + D_2O → α-methyl-trans-β-deuterostyrene. Hydrolysis of the corresp. Grignard reagent afforded a more specifically labeled prod. (78% trans and 2% cis-deuterium).

66-22

MILLER B.

The mechanism of 1,3-migrations of allyl groups in the dienone-phenol rearrangements of 2,6-di-t-butylcyclohexadienones.

J. Am. Chem. Soc. **87**, 5111 (1965)

Tetrabromo-p-cresol, catal. redn. → p-cresol-2,3,5,6-d_4 •(quantitave), refluxing with HCl → 77% p-cresol-3,5-d_2, + isobutylene (H_2 SO_4) → 89% 2,6-di-t-butyl-p-cresol-3, 5-d_2, K-t-butoxide + allyl bromide → 47% 4-allyl-2,6-di-t-butyl-4-methylcyclohexadiene-1-one-3,5-d_2.

66-23

SCHUBERT W. M., LAMM Bo

The acid-catalysed hydration of styrene.

J. Am. Chem. Soc. **88**, 120 (1966)

Styrene-α-d from acetophenone + $LiAlD_4$, H_2O, overall yield 45%, 98.5 atom % D. Styrene-β,β-d_2 from ethyl phenylacetate as above, overall yield 48%, 98.5 atom % D.

66-24

STREITWIESER A. Jr.,
CALDWELL R. A., LAWLER R. G.,
ZIEGLER G. R.

Acidity of hydrocarbons. XX. Comparison of relative proton exchange rates of hydrocarbons with lithium cyclohexylamide and cesium cyclohexylamide.

J. Am. Chem. Soc. **87**, 5399 (1965)

By quenching the Grignard reagents from the corresp. bromides toluene-2-d(^3H), -3-d(^3H) -4-d(^3H), mesitylene-2-^3H were prepd.

66-25

STREITWIESER A. Jr.,
LAWLER R. G.

Acidity of hydrocarbons. XVIII. Exchange reactions of polycyclic aromatic protons with lithium cyclohexylamide.

J. Am. Chem. Soc. **87**, 5388 (1965)

Phenanthrene-9-d, anthracene-9-d, ^3H, (1.6 × 10^6 dpm/mmole), biphenyl-4-d, -2-d, and -3-d by heating the corresp. bromo-derivs. with Bu-Li and quenching with D_2O or T enriched D_2O resp. For anthracene-1-^3H 1-chloroanthraquinone and Zn/NH_3, sp. act. 5.30 × 10^6 d.p.m./mmole.

66-26

STREITWIESER A. Jr.,
LAWLER R. G., PERRIN C.

Acidity of hydrocarbons. XVII. Kinetics and mechanism of proton exchange of benzene and naphthalene with lithium cyclohexylamide in cyclohexylamine.

J. Am. Chem. Soc. **87**, 5383 (1965)

Naphthalene-1-d by quenching the Grignard reagent from 1-bromonaphthalene with D_2O.

Naphthalene-2-d by treating 2-bromonaphthalene in ether for 1 hr. with butyllithium followed by quenching with D_2O, naphthalene-1-^3H in a similar manner, sp. act. 6.4 × 10^6 d.p.m./mmole.
Benzene-1-d and -1-^3H by quenching phenylmagnesium bromide with tritium-enriched D_2O.

66-27

TRAFICANTE D. D., MACIEL G.

Fluorine-19 magnetic resonance study of secondary deuterium isotope effects of the methyl group.

J. Am. Chem. Soc. **87**, 4917 (1965)

m-Fluorobenzyl-α,α-d_2 alcohol (I) from methyl m-fluorobenzoate and $LiAlD_4$; I + $SOCl_2$ → α-chloro-m-fluorotoluene-α, α-d_2 → corresp. Grignard reagent, + D_2O → 55% m-fluorotoluene-α,α,α-d_3, 99.5 trideuterated. In the same way the p-fluoro-deriv. was prepd.

1.0.4 — *HETEROCYCLIC COMPOUNDS*

66-28

KIRBY G. W., SHAH S. W.

Preparation of indole-2-d and -2-t from indoxyl, and the structure of diacetoxymercuriindole.

Chem. Commun. **1965**, 381
CA **63**, 14675e (1965)

Redn. of indoxyl with $LiAlH_4$ and hydrolyzing the complex with D_2O or tritiated water resp. yields the title compds.

66-29

OAE S., KOZUKA S.

Rearrangement of tertiary amine N-oxides. XIV. Mechanism of the reaction of pyridine N-oxide with acetic anhydride.

Tetrahedron **21**, 1971 (1965)
CA **63**, 13011e (1965)

Dipicolinic acid, + $SOCl_2$, + D_2O, decarboxylation, → pyridine-2,6-d_2, + H_2O_2 → pyridine-2,6-d_2 N-oxide contg. 95% D in the α-position.

66-30

RHODES H. J., FANG Sen Maw,
BLAKE M. I.

Synthesis of 2,6-dioxo-3-phthalimidopiperidine-3,4,4,5,5,-d_5 and 2,5-dioxo-3-phthalimidopyrrolidine-3,4,4-d_3 from L-deuterioglutamic acid and L-deuterioaspartic acid.

J. Pharm. Sci. **54**, 1440 (1965)
CA **63**, 16453c (1965)

The perdeuterated amino acids from hydrolyzate of the algae Scenedesmus obliquus; phthaloylation with N-carbethoxyphthalimide, heating with Ac_2O, and heating with urea → title compds.

66-31

THOMAS D. W., BIEMANN K.

Thermal methyl transfer. The mass spectrum of voacamine-d_3.

J. Am. Chem. Soc. **87**, 5447 (1965)

A method was developed for the preparation of trideuteriomethyl esters with high deuterium content.

1.0.5 — *CARBOHYDRATES*

1.0.6 — *PEPTIDES, AMINO ACIDS, PROTEINS*

66-32

PAUL S. D., RAMAMURTHY J.,
CHAWLA A. S.

Labeling of glycine by direct exchange with heavy water.

Indian J. Chem. **3**, 269 (1965)
CA **63**, 17412f (1965)

Glycine-d_2, -d_3, and -d_5 by direct labeling of NH_2CH_2-CO_2H with D_2O alone or in the presence of Pt-black as the catalyst.

In the last case, the extent of deuteration in a single equilibration was 70%.

See also : **66-55**, C-terminal amino acids; D labeling

1.0.7 — *STEROIDS*

66-33

DJERASSI C., SHAPIRO R. H., VANDEWALLE M.

Mass spectrometry in structural and stereochemical problems. LXXXI. Stereospecificity in a hydrogen-transfer reaction characteristic of 6-keto steroids.

J. Am. Chem. Soc. **87**, 4892 (1965)

3α-dl-Cholestan-6-one (**I**) from cholestan-3β-ol-6-one (**II**), the tosylate, redn. with $LiAlD_4$, reoxidn. by the Jones procedure. **II** → ethylene ketal, oxidn. with CrO_3/Py, redn. with $LiAlD_4$ and hydrolysis → 3α-dl-**II**. Transformation of the latter by the reaction sequence applied for the synthesis of **I** to 3,3-d_2-**I** and 3β-d_1-**I** (redn. with $LiAlH_4$) resp.
2β-d_1-Cholestan-6-one from Δ^2-cholesten-6-one via the ethylene ketal (**III**) → 2α, 3α-oxido-deriv., $LiAlD_4$ redn., hydrolysis and elimination of the 3α-hydroxy group (tosylate).
2α-d_1-**I** by deuterioboration of **III** followed by H_2O_2 oxidn. and elimination of the OH group.
Prepn. of 2,2,4,4-d_4-**I**.

See also : **66-6** Deuterated steroids

1.1 — Tritium compounds

1.1.1 — *GENERAL*

66-34

FILATOV E. S., NESMEYANOV An. N. CHIANG, Tsi-wang.

Reactions of hot tritium atoms with alcohols and with some of their binary hydrocarbon mixtures.

pp. 277-99 of STI/PUB/91 (Vol. 1). (IAEA)
N.S.A. **19**, 40622 (1965)

Investigation of the influence exerted by processes of intramolecular and intermolecular energy transfer as a function of concentration of components, and length of the alcohol molecule.

66-35

KUDO Ken-Ichi, ADACHI T., HAZUE M., ENDO M.

Catalytic tritium exchange reactions with tritiated active methylene compound.

Bull. Chem. Soc. Japan, **38**, 2006 (1965)

Ethyl malonate as T source, activity of catalysts : Raney Ni>Pd>Pt, substrates: aliphatic and aromatic hydrocarbons.

66-36

WHITE G. R.

Radioactive hydrogen.

J. New Zealand Inst. Chem., **29**, 14 (1965)
N. S. A. **19**, 40638(1965)

Methods of detecting beta particles and of preparing tritium-labeled compounds are outlined.

See also : **66-58**, Recoil labeling of amino acids

1.1.2 — *ALIPHATIC COMPOUNDS*

66-37

GABRIEL O., ASHWELL G.

Biological mechanisms involved in the formation of deoxysugars. II. Enzymatic conversion of thymidine diphosphoglucose-3-T to thymidine diphospho-4-keto-6-deoxyglucose.

J. Biol. Chem. **240**, 4128 (1965)

Diketobutane + $NaBT_4$ (10 mCi), +$NaBH_4$ for completion → 2,3-butandiol-2, 3-^3H, + periodate → acetaldehyde-1-^3H, sp. act. 2.15 × 10^6 cpm/μmole.

66-38

HILTON B. D., O'BRIEN R. D.

Synthesis of ethyl-^{3}H parathion.

J. Econ. Entomol. **58**, 1025 (1965)
CA **63**, 16240e (1965)

To minimize losses ethyl-^{3}H parathion is synthesized directly from p-nitrophenyl phosphorodichlorodithionate and lab. ethanol in the ethanol ampul. 50% yield of 97-98% radiochem. pure parathion-^{3}H, sp. act. of 4.5 mCi/mmole.

66-39

KOCH G. K., JURRIENS G.

Quantitative chromatographic analysis of organic acids by tritiating methylation.

Nature, **208**, 1312 (1965)

Acid in ether + tritiated water, + diazomethane $\rightarrow$ tritiated methyl esters; which were separated on thin-layer plates.

66-40

SUSAN A. B., REUTOV O. A.

Synthesis of some functional derivatives of the propyl radical labeled with tritium.

Rov. Roumaine Chim. **10**, 625 (1965); Studii Cercetari Chim. **13**, 663 (1965)
CA **64**, 579b (1966)

$CH_3CH:CH_2$ and $TCH_2CH:CH_2$ + TBr and HBr resp., by photochem addn. in the liquid state, addn. of nonlabeled propyl bromide to a product at a sp. activity of 286 µCi/mmole for propylbromide-2-^{3}H and at 200 µCi/mmole for propylbromide-3-^{3}H resp., 98.5% propyl bromide and 1.5% isopropyl bromide; the position of the T was confirmed by successive chem. degradation. Radiochem. yields 86, and 72%. The corresp. amines by reaction of the NH_4 carbamate with the alkyl bromide at the resp. sp. act. 41.5 and 32.5 µCi/mmole. Propylamine-3-^{3}H also by hydrogenolysis of 3-bromopropylamine hydrobromide with T_2 in anhyd. EtOH soln., in the presence of a Pt catalyst.

66-41

TILL A. R., DOWNES A. M.

The preparation of ^{3}H-labeled polyethylene glycol and its use as a soluble rumen marker.

Brit. J. Nutr. **19**, 435 (1965)
CA **63**, 13693e (1965)

Labeling with tritium by direct exposure to $^3H_2$ gas and purifn. by gel filtration.

See also : **66-16**, 2-Phenylbutane-2-d, ^{3}H

1.1.3 — *AROMATIC COMPOUNDS*

66-42

MITCHELL J. S.

On the possibility of the use of radioactive drugs.

Strahlentherapie **127**, 497 (1965)
N. S. A. **19**, 46125 (1965)

Clinical and laboratory studies of the possibility of using tritiated derivatives of Synkayvite, e.g., TRK 219, tetra-sodium-2-methyl-6, 7-ditritio-1, 4-naphthohydroquinone diphosphate as a therapeutic agent for radiation injuries.
Discussion about its behaviour, when tritiated by different methods.

66-43

NEUMANN H. G.,

3-Methyl-1,2-dehydrocholanthrene. Synthesis of 1,2-labeled methylcholanthrene with highly specific activity.

Z. Physiol. Chem. **341**, 298 (1965)
CA **64**, 668g (1966)

3-Methyl-1,2-dehydrocholanthrene was synthesized and its catal. hydrogenation was proposed for the prepn. of spec. tritiated 3-methylcholanthrene.

66-44

OTTO P.

Synthesis of tritiated p-terphenyls, labeled at specific positions.

EUR 2529e (1965)

p-Terphenyl-4-T, -3-T, 2-T and -2′-T by catalytic redn. of the corresp. bromo-p-terphenyls with T_2 under mild condns. (sp. act. 10 mCi/mg). Detn. of the activity distrn. in the different posn. in the molecule by degradn. (nitration to 4,4″-dinitro-p-terphenyl, oxidn. of the latter with CrO_3/Ac_2O to p-nitrobenzoic acid and catal. redn. of the nitro group, bromination in 3, 5-positions).

66-45

STREITWIESER A., Jr.
LAWLER R. G., SCHWAAB D.

On the bromopyrenes.

J. Org. Chem. **30**, 1470 (1965)

2-Aminopyrene + nitrosylsulfuric acid, + $HgBr_2$ + KBr, heating → 2-bromopyrene, + LiBu, quenching with tritiated water → 64% pyrene-2-^3H, sp. act. 3.41×10^7 d.p.m./mmole.

See also : **66-24,** Toluene-2; -3; and 4-^3H
Mesitylene-2-^3H
66-25, Anthracene-1; and -9-^3H
66-26, Naphthalene-1-^3H
Benzene-1-d, ^3H
66-73, Benzene-^3H

1.1.4 — *HETEROCYCLIC COMPOUNDS*

66-46

BARTON D. H. R., HESSE R. H.,
KIRBY G. W.

Phenol oxidation and biosynthesis. Part VIII. Investigations on the biosynthesis of berberine and protopine.

J. Chem. Soc. **1965**, 6379

Tribenzyl ether of 1-(3, 4-dihydroxybenzyl)-7-hydroxy-6-methoxy-2-methyl-1,2,3,4-tetrahydroisoquinoline (I), hydrogenation, heating in dimenthylformamide and tritiated water (triethylamine, N_2) at 100° for 5 days → I-^3H. 2×10^7 dpm/mg. Proof of labeling in o- and p- position to the phenolic hydroxy groups by exchange in D_2O and n. m. r. control.

66-47

RODRIGUEZ PASQUES R. H.

Tritiation of furfural

Arg. Rep. Com. Nacl. Energia At. Inform **153** (1965)

Irradn. of arabinose with recoil tritium, transformation to the title compd. by well known methods, sp. act. 56 μCi/g

66-48

WACKER A., KORNHAUSER A.,
TRÄGER L.

Isotopeneffekte bei der photochemischen Umwandlung von Tritium-markiertem Uracil.

Z. Naturforschg. **20b**, 1043 (1965)

Uracil-5-^3H from 5 mg 5-bromouracil and 100 μCi $LiAlT_4$, purifn. by electrophoresis, yield 8 μCi. Uracil-6-^3H from uracil-5, 6-^3H, iodination to 5-iodo-uracil-6-^3H and subsequent deiodination by irradiation at 300 mμ, yield 35%.

See also : **66-28,** Indole-2-^3H

1.1.5 — *CARBOHYDRATES*

66-49

GABRIEL O., ASHWELL G.

Biological mechanisms involved in the formation of deoxysugars. I. Preparation of thymidine diphosphate glucose labeled specifically in carbon 3.

J. Biol. Chem. **240**, 4123 (1965)

Incubation of Agrobacterium Tumefaciens with sucrose, redn. of the resulting 3-keto-sucrose with $NaBT_4$ → 5% α-D-glucosyl-3T-β-fructoside, selective phosphorylation with sucrose phosphorylase + Pi to α-D-glucose-3T (I) 1-phosphate, enzymic synthesis of thymidine diphosphoglucose-3T. Sp. act. 6,93 × 10^6 cpm/μmole. Decrease in sp. act. of about 10% during 1 year storage in frozen state. Degradation studies with the labeled compds.

See also : **66-83**, Nucleosides-^{3}H

1.1.6 — *PEPTIDES, AMINO ACIDS, PROTEINS*

66-50

BIRKOFER L., HEMPEL K.,
NOUVERTNE W.

Synthese von tritiummarkiertem Ornithin und Arginin hoher spezifischer Aktivität.

Chem. Ber. **98**, 3200 (1965)

Cat. hydrogenation of α-acetamino-β-(2-chloroacetamino-ethyl)-acrylic acid with tritium gas, hydrolysis → DL-ornithine-α, β-^{3}H (I), yield 60%, radiochemical yield 47%, sp. act. 9300 mCi/mmole.
I and O-methyl-pseudourea hydrochloride → arginine-α,β-^{3}H, yield 44%, sp. act. 3000 mCi/mmole.

66-51

FLAVIN M., SLAUGHTER C.

Synthesis of the succinic ester of homoserine, a new intermediate in the bacterial biosynthesis of methionine.

Biochemistry, **4**, 1370 (1965)

O-Succinyl-L-homoserine-4-^{3}H (I) (and by the same way the 2-^{14}C deriv.) via L-homoserine-4-^{3}H (II) (O, 5 mCi, 205 mg), → the N-Cbo deriv. (72%), + succinic anhydride in Py, hydrogenolysis → 39% I from II.

66-52

GOTTLIEB A. A., FUJITA Y.,
UDENFRIEND S., WITKOP B.

Incorporation of cis- and trans-4-fluoro-L-prolines into proteins and hydroxylation of the trans isomer during collagen biosynthesis.

Biochemistry **4**, 2507 (1965)

cis- and trans-4-Fluoro-L-prolines via the tosyloxy-L-proline derivatives by S'N$_2$ displacement with fluoride ion, catalytic tritiation in H_2O-^{3}H/Pt → trans-4-fluoro-L-proline-U-^{3}H, yield 65%, sp. act. 77 mCi/mmole and the corresp. cis-compd. yield 45%, sp. act. 55 mCi/mmole.

66-53

KESSEL D., LUBIN M.

Stability of α-hydrogen of amino acids during active transport.

Biochemistry **4**, 561 (1965)

DL-Alanine, glacial acetic acid and anhydrous sodium acetate, reflux for 1 h. + acetic anhydride, 2 h. at 100° + tritiated water, heating for 1 h, → DL-alanine-α-^{3}H, sp. act. 80.000 cpm/mg.

66-54

MATSUO H., FUJIMOTO Y.,
TATSUNO T.

A novel method for the determination of C-terminal amino acid in polypeptides by selective tritium labeling.

Biochem. Biophys. Res. Comm. **22**, 69 (1966)

The labeling of the C-terminal amino acid is accomplished by formation of the oxazolone by the action of acetic anhydride and subsequent exchange of the α-hydrogen atom.

66-55

MATSUO H., FUJIMOTO Y.,
TATSUNO T.

Novel approach to the C-terminal determination of peptides. Selective ^{2}H- and ^{3}H-labeling reaction of C-terminal amino acids through oxazolone.

Tetrahedron Letters **1965**, 3465.

CA **64**, 806e (1966)

See preceeding abstract.

66-56

PHILLIPS A. T., WOOD W. A.

The mechanism of action of 5′-adenylic acid-activated threonine dehydrase.

J. Biol. Chem. **240**, 4703 (1965)

Threonine-2-^3H by racemization of N-acetyl-DL-allothreonine in the presence of 50 μCi of ^3H_2O, sepn. from the allo isomers by preparative paper chromatography, sp. act. 0.03 μCi/μmole, over-all yield 10%.

66-57

ROCHE J.

Marquage specifique d'hormones thyroidiennes au tritium et étude de leur radiodécomposition.

EUR-405f, 1963.

Catalytic hydrogenolysis with tritium in a new app. Prepn. of α,β-^3H-thyronine, 3′-iodothyronine, 3′,5′-diiodothyronine, 3-iodothyronine, 3,3′-diiodothyronine, and 3,3′,5′-triiodothyronine, purifn. on Dowex 50.

66-58

SIMONOV E. F.,
NESMEYANOV An. N.

Reactions of hot tritium atoms with amino acids.

pp. 411-19 of STI/PUB/91(Vol. 1)., (IAEA).
N. S. A. **19**, 40629 (1965)

Study of the yields from the reaction of hot tritium atoms : with amino acids having lengthened chains, and with amino acids having a carbon chain of constant length, but with various functional substitutes.

1.1.7 — *STEROIDS*

66-59

AKHTAR M., GIBBONS C. J.

A convenient synthesis of (9.19-$^3H_2$)cholecalciferol 3,5-dinitrobenzoate and the mechanism of the precholecalciferol $\rightleftharpoons$ calciferol reaction.

J. Chem. Soc. **1965**, 5964

Prepn. of the title compd.; already preliminarily published, see abstr. 65-262.

See also : **66-93**, Cholest-8-ene-3β,6α-diol-2,4-^3H

1.2 — Carbon-14 compounds

1.2.1 — *GENERAL*

66-60

FIRSOVA L. P., BARAKAT M. F.,
FORYS M., NESMEYANOV An. N.

The interaction of ^{14}C recoil atoms in binary mixtures.

pp. 373-83 of STI/PUB/91(Vol. 1), (IAEA).
N. S. A. **19**, 40627 (1965)

Systems indole-aniline, indole-pyridine, α-picoline-toluene, α-picoline-aniline, etc. See also 66-114.

66-61

MESICH F. G.

Deactivation of reaction complexes in the recoil chemistry of ^{11}C (Thesis).

Ames Lab., Iowa State Univ. of Science and Tech. (1965). IS-T-27.
N. S. A. **19**, 40550 (1965)

The nature and yields of products resulting from the reactions of recoil carbon-11 atoms in liquid hydrocarbons were studied.

66-62

SCHWARZ A.

Durch Gamma-Strahlung induzierte Reaktionen des Tetrachlorkohlenstoffs, Chloroforms und Methylenchlorids mit Cycloalkenen. Strahlenchemische Synthese ^{14}C-markierter Trichlormethylcycloalkane.

KFK-317, (1965)

N. S. A. **19**, 38695 (1965)

Compds. labeled in the methyl group by the use of ^{14}C-labeled carbon tetrachloride were trichloromethylcyclohexene and -cyclopentane and 1-chloro-2-trichloromethylcyclopentane; cyclohexene-chloroform and cyclopentene-chloroform gave dichloromethylcylohexenes and cyclopentanes.

1.2.2 — *ALIPHATIC COMPOUNDS*

66-63

BAKH N. A., KALVAZIN E. P.,
NESMEYANOV A. N.

Tagged aldehydes and ketons.

U. S. S. R. 171, 397 Appl. July 10, 1964.
CA **63**, 16146a (1965)

Prepn. from the action of ionizing radiation on a mixt. of the hydrocarbon with radioactive CO_2. To increase the radiochem, yield ^{14}CO is used as the radioactive oxide and an adsorbent is placed in the reactor zone which is protected from the radiation.

66-64

BONNER W. A., KAKIS F. J.

The Hofmann elimination of trimethyl-1, 2, 2-triphenyl-ethylammonium-1-^{14}C hydroxide.

J. Org. Chem. **30**, 4248 (1965)

Phenyl benzhydryl ketone-carbonyl-^{14}C, (0,7 mCi/mole), (see J. A. C. S. **75**, 5372), Leuckart reductive amination → N-formyl-1, 2, 2-triphenylamine-1-^{14}C, hydrolysis → 94,7% 1,2,2-triphenylethylamine-1-^{14}C, + $CH_3I/Na_2CO_3/H_2O$, heating for 48 hrs. up to 120° → 82,7% quarternary iodide, transformation to the title compd. on an ion exchange column.

66-65

FINCH A. M. T. Jr., VAUGHAN W. R.

Racemization of camphor during n′-sulfonation. Evidence for exclusive exo-methyl migration.

J. Am. Chem. Soc. **87**, 5520 (1965)

Camphor-8-^{14}C; via carbonation of sodio-3-methylnorbornan-2-one, using ^{14}C-labeled carbon dioxide, + methylmagnesium iodide → 2,3-dimethyl-3-hydroxynorbornane-2-carboxylic acid, → 1,7-dimethylnorbornane-7-carbo-2-lactone, + $LiAlH_4$ → 8-hydroxyisoborneol-8-^{14}C, Jones oxidation → 8-hydroxycamphor-8-^{14}C → 8-bromocamphor-8-^{14}C, hydrogenolysis.

66-66

GELLERMAN J. L., SCHLENK H.

Preparation of fatty acids labeled with ^{14}C from Ochromanos danica.

J. Protozool., **12**, 178 (1965)
N. S. A. **19**, 42251 (1965)

Addn. of 10 mCi acetate-1,2-^{14}C to cultures of Ochromonas danica, with a recovery of 21,4% of the radioactivity as mixed methyl esters. Isolation in amounts between 2 and 200 mg by liquid-liquid chromatography followed by gas-liquid chromatography. Linoleate and arachidonate contained not more than 4% isomers. Average of sp. act. about 1 μCi/mg.

66-67

KLIMASHEVSKAYA B. A.,
SEDLETSKAYA I. S., VOLKOVA V. S.

Preparation of octadecane-1-^{14}C and hexadecane-1-^{14}C.

Tr. Gos. Inst. Prikl. Khim. **52**, 53 (1964)
CA **63**, 13053h (1965)

Stearic acid-1-^{14}C and palmitic acid-1-^{14}C resp. (see 65-445) + $LiAlH_4$, + I_2/P, + Zn → title compds. (85-95 and 76% resp.).

66-68

MUNDER P. G., FERBER E.,
FISCHER H.

Untersuchungen über die Abhängigkeit der cytolytischen Wirkung des Lysolecithins von Membranenzymen.

Z. Naturforschg. **206,** 1048 (1965)

Lecithin-2-acyl-^{14}C by incorporation of oleic acid-^{14}C in lysolecithin by acyltransferase from erythrocytes. By incubating slices of mouse liver with acetate-^{14}C lysolecithin-1-acyl-^{14}C of sp. act. of 1.3×10^5 ipm/μmole.

66-69

NISHIMURA J. S., MEISTER A.

Evidence for succicyl phosphate as an enzyme-bound intermediate in the reaction catalyzed by succinyl coenzyme A synthetase.

Biochemistry **4**, 1457 (1965)

Succinyl phosphate-2,3-^{14}C (**I**) via lab. succinic acid (**II**) (sp. act. 9,1 mCi/mmole) → corresp. monobenzyl ester, + phosphoric acid in pyridine/H_2O + N,N'-dicyclohexyl-carbodiimide → benzyl succinyl phosphate, overall yield 30-40% based on **II**, catalytic hydrogenation → **I**.

66-70

VYKHOVANETS V. V., LIPOVICH V. G., KNUTOV V. I., CHENETS V. V., BLYUM O. I., KALECHITS I. V.

Synthesis of methylcyclohexanes tagged with ^{14}C carbon isotope in 1-, 2-, 3-, 4- and 7-positions.

Zh. Vses. Khim. Obshchestva im. D. I. Mendeleeva **10**, 465 (1965)
CA **63**, 14718a, (1965)

Picolines, hydrogenation, Br_2-PBr_3 → 1,5-dibromohexane, 2-methyl- and 3-methyl-1,5-dibromopentane, + $Na^{14}CN$, hydrol. → dicarboxylic acids, Ca salts → cyclohexanones, + N_2H_4-KOH → methylcyclohexane-3(2 and 4)-^{14}C. Methylcyclohexane-1-^{14}C from toluene, the 7-^{14}C isomer from $^{14}CH_3MgI$ and cyclohexanone.

1.2.3 — *AROMATIC COMPOUNDS*

66-71

BLACKBURN D., BURGHARD G.,

Radiosynthesis of dextroamphetamine-^{14}C sulfate.

J. Pharm. Sci. **54**, 1586 (1965)
CA **64**, 620c (1966)

High yield radiosynthesis of dl-amphetamine-7-^{14}C from 0.5 Ci of benzoic-7-^{14}C acid. It was resolved in 84% yields by the successive crystallization of d-amphetamine-^{14}C d-bitartrate and l-amphetamine-^{14}C l-bitartrate salts.

66-72

DALY J. W., BENIGNI J., MINNIS R., KANAOKA Y., WITKOP B.

Synthesis and metabolism of 6-hydroxycatecholamines.

Biochemistry **4**, 2513 (1965)

Enzymatic O-methylation with catechol O-methyltransferase with S-adenosylmethionine-^{14}C as donor of $^{14}CH_3$ (5 μCi, O.25 μmole) → 3-o-methyl-^{14}C-6-hydroxydopamine and 3-o-methyl-^{14}C-6-hydroxynorepinephrine.

66-73

NEFEDOV V. D., SKOROBOGATOV G. A., SMIRNOV V. M., MUSAKIN A. P., VOROB'EV L. N.

Microsynthesis of multiply tagged benzene-^{14}C and isotope effects.

Zh. Organ. Khim. **1**, 1615 (1965)
CA **64**, 617e (1966)

$^{14}C_2H_2$ + Ziegler catalyst from $TiCl_4$ and iso-Bu_3Al in MePh → C_6H_6 in 65-75% yield. Neither C_2H_2 nor C_6H_6 exchange H with MePh or with the Ziegler catalyst. The method may be used to form tritium-tagged C_6H_6.

66-74

PACKTER N. M.,

Studies on the biosynthesis of quinones in fungi. Incorporation of 6-methylsalicylic acid into fumigatin and related compounds in Aspergillus fumigatus I. M. I. 89 353.

Biochem. J. **97**, 321 (1965)

6-Methylsalicylic acid-^{14}C from P. griseofulvum by incubation with Na acetate-1-^{14}C; chromatography of the total ether extract on silicic acid-Celite.

66-75

PATSCHKE L., GRISEBACH H.

Zur Biogenese der Flavonoide X : Ueber den Zeitpunkt der Hydroxylierung im Ring B.

Z. Naturforschg. **20b**, 1039 (1965)

Dibenzylpyrocatechol + $I_2/AgOOCCF_3$ → 4-iodo-deriv., + Cu^{14}CN, catal. redn. in the presence of semicarbazide acetate → 3.4-dibenzyloxybenzaldehyde semicarbazone-carbonyl-^{14}C. The free aldehyde and 2-tetraacetylglucosido-4-benzoyl-phloroacetophenone → 3,4,2′,4′,6′-pentahydroxychalcone 2′-glucoside- (β-^{14}C), over-all yield 12%.

66-76

PETTERSSON G.

The biosynthesis of flavipin. II. Incorporation of aromatic precursors.

Acta Chem. Scand. **19**, 1724 (1965)

Orcinol, Gattermann reaction → orcylaldehyde (I), Clemmensen redn. → 1,3-dihydroxy-4,5-dimethylbenzene (II), + Zn-$(^{14}CN)_2$ (Gattermann reaction), 1 g scale → O,83g 2,4-dihydroxy-5,6-dimethylbenzaldehyde (O.5 mCi) → 2,4-dihydroxy-5,6-dimethylbenzoic acid-carboxyl-^{14}C. The same acid, labeled in the 5-Me group from I-formyl-^{14}C, from which also orsellinic acid-carboxyl-^{14}C (O.4 µCi/mg) was prepd.

66-77

SCHWEITZER J. W.,
FRIEDHOFF A. J.

The distribution of (α-^{14}C) 3,4-dimethoxyphenethylamine among various organs of the rat.

Biochim. Biophys. Acta **111**, 326 (1965)

3,4-Dimethoxybenzyliodide + Na^{14}CN, + $LiAlH_4$ → title compd., sp. act. 0.12 mCi/mmole.

66-78

YEE T. T., McEWEN W. E.,
WOLF A. P.

Tracer study of the acid-catalyzed condensation of 2-benzoyl-1.2-dihydroisoquinaldonitrile with 1.1-diphenylethylene.

Tetrahedron Letters 1965, (35), 3115.
CA **63**, 14659h (1965)

2,3,5-Triphenylpyrrole-5-^{14}C from 1,2,4-triphenyl-1,4-butadione (I) by the method of Smith (J. Chem. Soc. **57**, **643** (1890) 1,1-Diphenylethylene-1-^{14}C from benzophenone + triphenylmethyl-^{14}C phosphonium bromide. I-4-^{14}C from acetophenone-carbonyl-^{14}C + benzoin in presence of KCN.

1.2.4 — *HETEROCYCLIC COMPOUNDS*

66-79

HUNZIKER F., SCHINDLER O.

Zum Stoffwechsel von Noveril : ^{14}C-Markierung und Synthese von Metaboliten.

Helv. Chim. Acta **48**, 1590 (1965)

β-Dimethylamino-ethanol-^{14}C-HBr (I) (5mCi/mmole) + $SOCl_2$ → β-dimethylamino-ethylchloride, + 5-methyl-10,11-dihydro-11-oxo-5H-dibenzo(b, e)-1,4-diazepin → title compd., yield 31,4% based on I.

66-80

PATERSON A. R. P., SIMPSON A. I.

The preparation of uridine-2-^{14}C and inosine-8-^{14}C using exchange reactions catalyzed by intact animal cells.

Can. J. Biochem. **43**, 1996 (1965)

Exchange between uracil-2-^{14}C and uridine in the presence of Ehrlich ascites tumor cells in vitro. Transfer of 43% of the radioactivity, sp. act. 16% of that of uracil. Likely inosine-8-^{14}C from hypoxanthine was prepd.

66-81

SCHLESINGER S., MAGASANIK B.

Imidazolepropionate, a nonmetabolizable inducer for the histidine-degrading enzymes in Aerobacter aerogenes.

J. Biol. Chem. **240**, 4325 (1965)

Incubation of uniformly labeled L-histidine-^{14}C with an extract of A. aerogenes, hydrogenation of the reaction mixture after 3 hrs. → title compd.-^{14}C.

See also : **66-78,** 2,3,5-Triphenylpyrrole-5-^{14}C

1.2.5 — *CARBOHYDRATES*

66-82

BICHUL T. V.,
POCHIKOVSKAYA V. M.,
SAPOZHNIKOVA E. P.,
SMIRNOV V. A., KOZLOVA L. P.

Synthesis of some monosaccharides and their derivatives labeled with isotopic ^{14}C.

Tr. Gos. Inst. Prikl. Khim. **52**. 38 (1964)
CA **63**, 14961e (1965)

66-83

KOHN P., SAMARITANO R. H.,
LERNER L. M.

A new method for the synthesis of furanose derivatives of aldohexoses.

J. Am. Chem. Soc. **87**, 5475 (1965)

By prepn. of tetraacylaldono-γ-lactones via the Fischer-Kiliani cyanohydrin synthesis and redn. with bis-(3-methyl-2-butyl)-borane (I) in almost quant. yields or redn. with I-^{3}H resp. ^{14}C or T can be easely introduced in C-1′ pos. of nucleosides.

66-84

WHITE G. A., ROBINSON J. R.,
UNWIN C. H.

Preparation of specifically labeled D-fructose-^{14}C.

Can. J. Biochem. **43**, 1919 (1965)

D-Fructose-^{14}C in high yield (95%) from specifically labeled D-glucose-^{14}C by redn. with $NaBH_4$ to sorbitol and by the action of purified bacterial sorbitol dehydrogenases (Acetobacter suboxydans). No randomisation of the labeling pattern.

1.2.6 — *AMINO ACIDS PEPTIDES, PROTEINS*

66-85

BUEHLER M. F., MITTA A. E. A.,
de LEZEROVICH J. B.

Sintesis de aminoacidos marcados. II. Sintesis de DL glicine 1C14 Y DL alanina-1C14.

ARCNEA, Inf. No. 142 - (NP-15268), 1965.
N. S. A. **19**, 44047 (1965)

Title compds. via α-bromo-acids; particular emphasis to purifn.

66-86

FISCHER A. G., SCHULZ A. R.,
OLINER L.

Thyroidal biosynthesis of iodothyronines. I. General characteristics and distribution of the bovine enzyme system.

J. Biol. Chem. **240**, 4338 (1965)

U-^{14}C-Labeled 3-Iodotyrosine from U-^{14}C-L-tyrosine (96,8 mCi/mmole), KI-I_2.

U-^{14}C-Labeled 3,5-diiodotyrosine by iodination of L-tyrosine with the use of nitrogen triiodide as the iodinating agent.

66-87

FROST D., MICHEL L.,
SCHWARZ E.

The radio-labeling of organic compounds by $^{12}C(\gamma,n)^{11}C$ reactions by means of bremsstrahlung of a 35-mMev betatron.

Strahlentherapie **127**, 510 (1965)
N. S. A. **19**, 46418 (1965)

Gamma rays can break up chemical bonds as could be shown with urocanic acid in aqueous solution. Free radicals, produced in water, are responsible for the decomposition. Other compounds, such as glycine remain stable and can be used in the investigations.

66-88

HUDSON C. B., ROBERTSON A. V.

Synthesis of (carboxyl-^{14}C)3,4-dehydro-DL-proline.

Aust. J. Chem. **18**, 1677 (1965)

Carbonation of pyrrole Grignard reagent, → ester → amide, redn. with phosphonium iodide in fuming HI → title compd., total radioactivity 29% from $Ba^{14}CO_3$, sp. act. 63 μCi/mmole.

66-89

KLIMASHEYSKAYA B. A.,
SEDLETSKAYA L. S., VOLKOVA V. S.

Synthesis of α-amino acids-2-^{14}C.

Tr. Gos. Inst. Prikl. Khim. **52**, 17 (1964)
CA **63**, 13393h (1965)

Ac $NH^{14}CH(CN)CO_2Et$, Na, + Me_2CHCH_2I, 6 hrs. at 100°, hydrolysis with conc. HCl → dl-leucine-2-^{14}C. Similarly, dl-norvaline-2-^{14}C, dl-valine-2-^{14}C, and dl-alanine-2-^{14}C in yields of 50-65%. Labeling in the 3 or 4 position by starting from the resp. 1- and 2-^{14}C alkyl iodides.

66-90

MUDD S. H., FINKELSTEIN J. D.,
IRREVERRE F., LASTER L.

Transsulfuration in mammals. Microassays and tissue distributions of three enzymes of the pathway.

J. Biol. Chem. **240**, 4382 (1965)

Cystathionine-2-^{14}C (I) from : DL-methionine-2-^{14}C, (0.1 mCi), → DL-homocysteinethiolactone (57% HI + hypophosphorous acid), + KOH → homocysteine, + rat liver cystathionine synthase + L-serine, 50% transformation to I after 135 min.; over-all radiochem. yield 25%.

66-91

SMULSON M. E., NEAL A. L.,

Comparative effects of glutamine analogues on protein metabolism of 6C3H-ED and Ehrlich ascites carcinoma.

Arch. Biochem. Biophys. **112**, 25 (1965)

γ-Glutamylhydrazide-^{14}C from L-glutamic-U-^{14}C acid (0. 144 mg, 0.2 mCi), + thionylchloride in abs. methanol, addn. of hydrazine hydrate to the dry methyl ester.

1.2.7 — *STEROIDS*

66-92

KLIMASHEYSKAYA B. A.,
POLIKARPOVA E. E.

Preparation of cholesterol-4-^{14}C, testosterone-4-^{14}C and progesterone-4-^{14}C.

Tr. Gos. Inst. Prikl. Khim. **52**, 24 (1964)
CA **63**, 13352f (1965)

Oxidn. of cholestenone (I) with H_2O_2, enol lactone, + $^{14}CH_3MgI$ (60% excess), + NaOH → I-4-^{14}C; redn. of the corresp. enol acetate with $NaBH_4$ → cholesterol-4-^{14}C, yield 32%. Ozonisation of testosterone propionate and progesterone resp., the corresp. enol lactones, + $^{14}CH_3MgI$ (20 and 100% excess resp.), + KOH and HCl conc. resp. → testosterone-4-^{14}C (40-3%) and progesterone-4-^{14}C (26-31%) resp.

66-93

SLAYTOR M., BLOCH K.

Metabolic transformations of cholestenediols.

J. Biol. Chem. **240**, 4598 (1965)

Δ^7-Cholestene-3β,6α-diol-4-^{14}C from 3β-acetoxy-Δ^7-cholesten-6-one (I) in 1-butanol, redn. with Na, sp. act. 0.50 mCi/mmole.
Δ^7-Cholestene-3β,6β-diol-4-^{14}C from I (55 mg) and $NaBH_4$ via the diacetyl-deriv., and treatment with $LiAlH_4$, sp. act. O. Oll mCi/mmole, yield 5 mg.
Δ^8-Cholestene-3β,6α-diol-2,4-^{3}H from 6α-acetoxy-Δ^8-cholesten-3-one, proton exchange in $3H_2O/KOH$, + $LiAlH_4$, sp. act. 14,3 mCi/mmole.

1.3 — Halogen labeled compounds

66-94

ROWLAND F. S., WAI C. M.,
TING C. T., MILLER G.

The stereochemistry of the reactions of (n, γ) halogen atoms with alkyl halides in the liquid phase.

pp. 333-48 of STI/PUB/91 (Vol. 1), (IAEA).
N. S. A. **19**, 40625 (1965)

See abstract 64-345.

66-95

VASAROS L.

Reactions of hot ^{38}Cl atoms in mixtures of carbon tetrachloride with aliphatic alcohols.

pp. 301-9 of STI/PUB/91(Vol. 1), (IAEA).
N. S. A. **19**, 40623 (1965)

The yield of reaction products in which the OH radical of aliphatic alcohol was replaced by ^{38}Cl increased with increasing alcohol concentration with a simultaneous decrease in the labeled CCl_4 yield.

66-96

HALPERN A., SOKOLOWSKA A.

Hot atom chemistry of halogens activated in β-decay. II. Reactions of ^{132}I in liquid mixtures of α-propyl halides and benzene or iodobenzene.

J. Inorg. Nucl. Chem. **27**, 1893 (1965)
CA **63**, 12602e (1965)

$^{132}TeCl_4$ + aliphatic-aromatic mixts. The sp. activity of the aliphatic ^{132}I-labeled products increases with the concn. of the aromatic component (benzene).

66-97

HERR W.

Process for the preparation of compounds tagged by radioactive tracers.

British Patent 1,000,760, Aug. 11, 1965.
Priority date Sept. 20, 1960. Germany.
N. S. A. **19**, 44107 (1965)

Compound in a stream of carrier gas, absorbent having absorbed on it a tagged isotope; exchange gas chromatographic column at temperatures not more than 50°C below the boiling point of the compound being tagged. The process is described for tagging halogen-substituted organic compounds, but may also be applied to tritium, deuterium, radiosulfur, and radiophosphorus.

66-98

BIANCHI C., PROTTO C., SOSI S.

Labeling and kinetics of body excretion od ^{131}I-iodothiazide.

Experientia **21**, 619 (1965)

100 μM Iodothiazide + K^{131}I (10 mCi), 5 h, 130-150°C; <3% iodine in the solution after 16 days.

66-99

SIGMAN E. M., ELWOOD Ch.,
REAGAN M. E., MORRIS A. M.,
CATANZARO A.

The renal clearance of ^{131}I-labeled sodium iothalamate in man.

Invest. Urol. **2**, 432 (1965)
N. S. A. **19**, 46079 (1965)

Title compd. from the commercially available product by a method of isotope exchange. Purifn. by precipitation and conversion to the sodium salt; sp. act. of 128μCi/mg, free ^{131}I content of less than 2% up to 3 weeks after labeling.

66-100

FARBWERKE HOECHST A. G.

"Procédé de préparation de chloro-iodo-propyl-inuline marquée par des isotopes radioactifs".

Belg. 661.633 (25. 3. 1965)

Inulin + allyl bromide, + ICl, labeled with iodine isotopes, $\dashrightarrow$ title compd. Sp. activities:20 mCi/g with ^{131}I and 12,3 mCi/g with ^{125}I.

66-101

HARTRODT W.

On radioactive labeled compounds, Part II. A report on the synthesis of ^{131}I-hippuran.

Nucl.-Med., **4**, 423 (1965)
N. S. A. **19**, 42585 (1965)

Sodium orthoiodohippurate achieves the best exchange efficiency (85 to 90%) at pH 5.7 to 6.0 and 120 °C without addition of inactive iodide. A purity control by radiopaper-chromatography did not reveal ^{131}I-iodide or ^{131}I-labeled ortho-iodo-benzoic acid.

66-102

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

Preparation of ^{131}I-labeled insulin and isolation by gel filtration.

Intern. J. Appl. Radiation Isotopes **16**, 668 (1965)

Insulin + nascent iodine -131 (KI + KIO$_3$) subsequent purifn. on a Sephadex G-25 column.

66-103

HALES N.

Radioimmunological methods for hormone determination in blood.

Bull. Schweiz. Akad. Med. Wiss. **21**, 249 (1965)
CA **64**, 968f (1966)

By use ^{131}ICl, ^{131}I incorporation in insulin to the extent of 25% with a sp. act. 5-25 mCi/mg. Prepns. of ^{131}I-labeled insulin were stored at 10° for 3-5 weeks with the loss in radioactivity bound to antiserum entirely accounted for by the half-life of the isotope.

66-104

ELEKES I., KORITSANSZKY D.

Aseptic production of sterile pyrogen-free human albumin, labeled with ^{131}I.

Magy. Radiol., **17**, 166 (1965)
N. S. A. **19**, 40639 (1965)

Improvements on the MCFARLANE method of producing pyrogen-free, ^{131}I-labeled serum albumin. Sp. act. of the resulting solution 1 mCi/ml, albumin-bound ^{131}I activity 45 to 52% of theory, protein content 6 to 7%, inorganic iodine content about O.5% after ion-exchange resin treatment, increase during storage to 5% after 10 days.

66-105

FRANTZ A. G., RABKIN M. T.,
FRIESEN H.

Human placental lactogen in choriocarcinoma of the male. Measurement by radioimmunoassay.

J. Clin. Endocrinol. Metab. **25**, 1136 (1965)
CA **63**, 15185d (1965)

HPL was iodinated and the HPL-^{131}I was purified by vertical starch-gel electrophoresis at pH 8 and 4°, Sp. act. 200-300 μCi/μg without significant immunological alternation.

66-106

GALSKOV A.

Labeling of corticotropin with iodine-125.

Experientia **22**, 63 (1966)

Modification of the method of HUNTER and GREENWOOD. Yield, starting from 5-10 μg corticotropin, about 50-60%, sp. act. 350-600 mCi/mg.

66-107

GILLILAND P. F., PROUT T. E.

Immunologic studies of octapeptides. I. Radioiodination of oxytocin.

Metab. Clin. Exptl. **14**, 912 (1965)
N. S. A. **19**, 44105 (1965)

Synthetic oxytocin to radioiodine solution containing 0.1 to 1.05 mCi of $Na^{131}I$ in a plastic test tube. At one min intervals, addn. of reagents in sequence as follows: Chloramine-T, sodium metabisulfite, potassium iodide. Approx. 20% of the total radioiodine was associated with the peptides, max. sp. act. 20.8 mCi/mg.

66-108

HABER E., PAGE L. B.,
RICHARDS F. F.

Radio immunoassay employing gel filtration.

Anal. Biochem. **12**, 163 (1965)

Ribonuclease-^{125}I by a modification of the HUNTER and GREENWOOD method. Antigenicity was retained. Sp. act. 6-7 × 10^7 d. p. m. By storage at -90° the preparation could be used up to 2 months after iodination.

66-109

SOCOLOW E. L., WOEBER K. A.,
PURDY R. H., HOLLOWAY M. T.,
INGBAR S. H.

Preparation of ^{131}I-labeled human serum prealbumin and its metabolism in normal and sick patients.

J. Clin. Invest. **44**, 1600 (1965)
CA **63**, 18814e (1965)

A prepn. of highly purified thyroxine(T_4)-binding prealbumin of human serum was radioiodinated by a micro-diffusion technique. Neither the electrophoretic mobility nor the T_4-binding capacity of the labeled protein TBPA-^{131}I differed from that of the starting material.

1.4 — Phosphorus-32 compounds

66-110

JACKSON J. E., JACKSON E. M.,
FREEMAN S.

Demonstration, extraction and intracellular distribution of kidney phosphoprotein kinase activity.

Biochim. Biophys. Acta **105**, 483 (1965)

(γ-^{32}P)ATP of high sp. act.(5μCi ^{32}P/μmole, 50-60% incorporation of inorg. ^{32}P) from ADP by utilizing the portion of the glycolytic pathway from fructose 1,6-diphosphate to 3-phosphoglyceric acid.

See also : **66-97,** Labeling by exchange on GLC columns

1.5 — Sulfur-35 Compounds

66-111

DZANTIEV B. G., SHISHKOV A. V.

Development of methods for the hot synthesis of ^{35}S-labeled biologically active substances.

pp. 209-17 of STI/PUB/91 (Vol. I), (IAEA).
N. S. A. **19**, 40619 (1965)

See abstracts 65-490 and 491.

66-112

ZEL'VENSKII Ya. D.,
SHALYGIN V. A., ANDREEVA N. I.

Thiophene-^{35}S.

Zb. Obshch. Khim. **35**, 1369 (1965)
CA **63**, 14796d (1965)

The most satisfactory method is the isotope exchange between thiophene vapor and $Ni^{35}S$ at 600°. The com. Ni on Al_2O_3 catalyst is treated with $H_2{}^{35}S$ at 400°. Sp. act. about 400 mCi/ml.

See also : **66-97**, Labeling by exchange on GLC columns

1.6 — Oxygen labeled compounds

66-113

GREEN M., THORP D. M.

Mechanism of chlorination of phosphorus esters.

Chem. Commun. **1965** (19), 466
CA **63**, 17813f (1965)

The esters benzyl methylphenyl(^{18}O)-phophinate, benzyl neopentyl methyl (^{18}O)-phosphonate and benzyl diphenyl(^{18}O)-phosphate were prepd. by treatment of the corresp. phosphoryl chlorides with benzyl (^{18}O) alc. (0.9 atom-% ^{18}O) and a tertiary base.

66-114

GULICK W. M. Jr., GESKE D. H.

Isotropic oxygen-17 coupling constant in the nitrobenzene anion radical.

J. Am. Chem. Soc. **87**, 4049 (1965)

Nitrobenzene-^{17}O by nitration of benzene in the presence of $AlCl_3$ with NO_2, enriched with ^{17}O by the reaction of NO with $^{17}O_2$. Yield 17,1% calcd. on the basis of mol. oxygen.

66-115

HOTTA H., SUZUKI N.

The effects of metal ions on the autoxidation of benzene in aqueous solutions.

Nippon Kagaku Zasshi, **86**, 651 (1965)
N. S. A. **19**, 44078 (1965)

Oxidation of benzene in aq. solns. of ferrous, ferric, and cupric sulfates → phenol with good yield above 150 °C. Phenol produced in ^{18}O-enriched water contains considerable amounts of ^{18}O.

66-116

LAPIDOT A. SAMUEL D.

On the mechanism of hydrolysis of N-phosphorylcreatine.

Biochim. Biophys. Acta **111**, 537 (1965)

(^{18}O)Sarcosine, by an exchange with 1 N HCl in ^{18}O-enriched water at 50° for 3 h., + cyanamide in ^{18}O-enriched water → (^{18}O)creatine containing 0.34 atom% ^{18}O.

66-117

McCLOSKEY J. A.,
McCLELLAND M. J.

Mass spectra of O-isopropylidene derivatives of unsaturated fatty esters.

J. Am. Chem. Soc. **87**, 5090 (1965)

Unsaturated fatty acids (methyl oleate, elaidate, and nervonate) + $OsO_4/H_2{}^{18}O$ (11.2% excess ^{18}O) → corresp. diols-^{18}O, + acetone → title compd.

See also : **66-2**, Ethyl 3-oxobutyrate-2,2-d_2-3-^{18}O

1.7 — Nitrogen-15 compounds

66-118

BAK B., SÖRENSEN G. O.,
MAHLER L.

Preparation of (^{15}N)- and (4-^{13}C)-pyridine.

Acta Chem. Scand. **19**, 2001 (1965)

Sodium glutacondialdehyde + 33% enriched $^{15}NH_4NO_3$ (I) + anhydrous $MgSO_4$ → pyridine-^{15}N in 5% yield based on **I.** Formaldehyde-^{13}C + ethyl acetoacetate + NH_3 → 3,5-dicarbethoxy-2,6-dimethyl-1,4-dihydropyridine, + $BrCCl_3$, sapon., oxidn.

with $KMnO_4$, → 43% of pyridine-2,3,4,6-tetracarboxylic acid, decarboxylation in quinoline/CuO → pyridine-4-^{13}C, 53% enriched, overall yield 10%.

66-119

LAMBERT J. B., OLIVER W. L.,
ROBERTS J. D.

Nitrogen-15 magnetic resonance spectroscopy. IV. The degenerate bimolecular exchange of protons in ketimines.

J. Am. Chem. Soc. **87**, 5085 (1965)

Benzamide-^{15}N (yield 89,5%) from benzoyl chloride in ether + ammonia, + $NaAlCl_4$ → 73,5% benzonitrile-^{15}N, + PhMgBr → 67,2% diphenylketimine-^{15}N.
n-Butyl chloride + $KC^{15}N$ → butyl cyanide → 41,2% di-n-butylketimine as above.

1.8 — Miscellaneous

66-120

GARZON O. L., PALCOS M. C.,
RADICELLA R.

A technetium-99m labeled colloid.

Intern. J. Appl. Radiation Isotopes **16**, 613 (1965)

Preformed colloidal antimony sulphide, free from hydrogen sulphide, stabilized with polyvinylpyrrolidone + saline solution containing ^{99m}Tc, 30 min. at 120°C. The colloidal particles retain the ^{99m}Tc quantitatively.

66-121

HENNIG K., FRANKE W. -G.,
WOLLER P., KNOLL P.

Existing methods for spleen scintigraphy with bromomercuri-hydroxypropane (BM-HP-^{197}Hg)

Nucl. -Med., **4**, 370 (1965)
N. S. A. **19**, 42584 (1965)

Prepn. of the title compd.

66-122

KOLCHAKOV K., VENKOV L.

Method of producing labeled protein by means of radioactive isotopes of silver.

Comp. Rend. Acad. Bulgare Sci., **18**, 219 (1965)
N. S. A. **19**, 29836 (1965)

Protein marked with ^{110}Ag is characterized by a low content of free and weakly bound silver cations. With electrophoresis in agar-gel, it moves at the same rate as the non-labeled protein.

66-123

KURCBART H., RADICELLA R.

A new ^{32}P-labeled colloid for medical use.

Intern. J. Appl. Radiation Isotopes **16**, 749 (1965)

^{32}P-Labeled colloidal preparation by forming iron hydroxide colloid in a solution containing carrier-free ^{32}P-orthophosphate. This colloid is autoclaved for 30 min. at 120°C and then stabilized with gelatine. More than 97% of the ^{32}P is bound to the colloidal base. The diameter of the particles is about 800 Å.

66-124

MANI R. S., DESAI C. N.,
RAGHAVAN S. V.

Preparation of ^{203}Hg labeled Neaohydrin for medical use.

Indian J. Chem. **3**, 415 (1965)
N. S. A. **19**, 46305 (1965)

Mercuric acetate ^{203}Hg of high sp. act. in methyl alcohol was refluxed with allylurea, + NaCl, evaporation, + dilute NaOH, chromatographic adsorption on alumina. (see 64-372)

66-125

ROOS L., ORCHIN M.

Allylbenzene isomerization catalyzed by deuteriocobalt tetracarbonyl.

J. Am. Chem. Soc. **87**, 5502 (1965)

Title compd. from $Co_2(CO)_8$ *in hexane* and DMF, + DCl at 0°, yield 90% (by titration).

1.9 — Carbon-13 compounds

66-126

MUSSO H., DÖPP D., KUHLS J.

Über den Mechanismus der Kehrmannschen Phenoxazonsynthese. IR-spektroskopische Studien an isotopen Triphendioxazinen.

Chem. Ber. **98**, 3937 (1965)

Hydroxybenzoquinone-1,4-$^{13}C_2$ (**I**) from 2,5-dihydroxy terephthalic acid-2,5,7,8-^{13}C via cyclohexane-1,4-dione (**II**), p-benzoquinone, 1,2,4-triacetoxybenzene, sapon., Ag_2O *oxidn. percentage of C-13 at the* carbonyl-carbon atoms : 33% $^{13}C_2$, 49% $^{13}C_1$.
Transformation to triphenodioxazine (5a, 12a-^{13}C from I, 6a.13a-^{13}C from II)

See also : **66-118** Pyridine-4-^{13}C

2 — RADIODECOMPOSITION, STABILITY, STORAGE

66-127

APELGOT S., EKERT B., FRILLEY M.

Autodecomposition of tritiated thymidine preserved in aqueous solution.

Biochim. Biophys. Acta **103**, 503 (1965)
CA **63**, 13671g (1965)

The rate of autodecompn. was lower at −196° than at 0°, −20°, or −75°, and higher at −20° than at the other 3 temps. Mol. heterogeneity was apparent regardless of the freezing temp., and even macroscopically evident at −20° and was considered to be the cause of autodecompn. Freezing at −196° followed by storage at −20° resulted in slower autodecompn. than freezing and storage at −20° (see 64-1P, and the following abstr.)

66-128

APELGOT S., EKERT B.

Self-decomposition and radiolysis at low temperature of tritiated thymidine in aqueous solution.

J. Chim. Phys., **62**, 845 (1965)
N. S. A. **19**, 40538 (1965)

No decrease of the self-decomposition from 0 to −75 °C. The tritiated thymidine decomposes only under the action of the β rays of its own ^3H atoms (see 64-1P).

3 — PURIFICATION, SEPARATION

66-129

ALEKSANDROV L. V.,
MUSAKIN A. P.

Paper electrophoretic determination of labeled carbohydrates.

Metody Analiza Radioaktivn. Preparatov. Sb. Statei **1965**, 28.
CA **63**, 14044g (1965)

Borate buffer at pH 8 for sepn. of carbohydrates from aldonic acids or corresp. alcs., at pH 9.2 sepn. of glucose and mannose. *It is thus possible to det. the* radiochem. purity of glucose-^{14}C.

66-130

BURIANEK J., CIFKA J.

Rapid determination of the radiochemical purity of orthophosphate-^{32}P.

Z. Anal. Chem., **213**, I (1965)
N. S. A. **20**, 271 (1966)

*Thin-layer chromatography. Factors in*fluencing the R_f values. Sensitivity.

66-131

DENISOV E. N.

Purification of radioactive preparations, labeled with radioactive iodine isotopes.

U. S. S. R. **171.084** (Appl. April 8, 1964).
CA **63**, 12637f, (1965)

A buffer soln. of e.g., Na o-iodohippurate Bengal Pink dye is passed through an ion-exchange resin, e.g. AV-17 in its Cl-form.

66-132

HEISE K. H., KNAPPE T.

Chromatographic purification of higher ^{14}C-labeled fatty acids.

Z. Chem. **5**, 301 (1965)
CA **63**, 16206f (1965)

Carboxylic acids from Grignard reactions, sepn. from by-product hydrocarbons and ketones on a silica gel column (ligroine, CCl_4-benzene, isopropyl ether).

66-133

MELANI F., DITSCHUNEIT H.,
BARTELT K. M., FRIEDRICH H.,
PFEIFFER E. F.

Radioimmunological determination of insulin in blood.

Klin. Wochschr. **43**, 1000 (1965)
CA **64**, 970b (1966)

Human insulin after labeling with ^{131}I by means of chloramine-T and Na^{131}I contains degradation products. In paper electrophoresis, ^{131}I-labeled insulin remained at the origin and the impurities migrated with the α-globulins. Only the fraction remaining at the origin reacted with guinea pig antihuman insulin, and the insulin-antibody complex migrated between the β- and γ -globulins. The labeled insulin could be sepd. from the impurities on Sephadex G75.

66-134

OKAMOTO J., DOBASHI G.

Analysis of labeled organic compounds by radiogaschromatography,

Nuclear Eng. (Tokyo) **11**, 20 (1965)
N. S. A. **19**, 46213 (1965)

The gas chromatographic separation and determination of the radioactivity can be carried out successively. Carrier gas consisting of propane and helium. Examples for the analysis of tritium-labeled radiolytic products of isobutyric acid and the separations of tritiated benzene and pyridine, of tritiated toluene and pyridine, and of tritiated chloroform and pyridine.

66-135

SIMONIS W., GIMMLER H.

Separation of ^{32}P-labeled phosphate esters and ^{14}C-labeled products of photosynthesis by two dimensional thinlayer chromatography.

J. Chromatog. **19**, 440 (1965)
CA **63**, 18627g (1965)

A 2-dimensional paper-chromatographic method for sepn. of phosphate esters and photosynthesis products was modified to be applicable to a thin-layer chromatographic procedure with cellulose powder.

4 — ANALYSIS

4.1 — *DETERMINATION OF ACTIVITY*

66-136

AAKVAAG A., EIK-NES K. B.

Metabolism in vivo of steroids in the canine ovary.

Biochim. Biophys. Acta, **111**, 273 (1965)

A method has been proposed for the measurement of specific radioactivity of androstenedione, testosterone, and estradiol biosynthesized from ^{14}C- and ^{3}H-labeled precursors.

66-137

BAKER N., HUEBOTTER R. J., SCHOTZ M. C.

Analysis of glucose-^{14}C in tissues using thin-layer chromatography.

Anal. Biochem. **10**, 227 (1965)

66-138

BARTLEY J. C., ABRAHAM S.

Improved methods for liquid scintillation assay of (A) ^{14}C compounds on paper chromatograms and (B) ^{14}C-protein.

Atomlight **1965**, (49), 1.

66-139

BLEECKEN S., KAUFMANN G., KUMMER K.

Determination of tritium-labeled compounds on thin-layer chromatograms.

J. Chromatog. **19**, 105 (1965)
CA **63**, 15525f (1965)

Cholesterol-^{3}H on silica gel thin-layers evaluation with an ordinary methane flow counter. Over-all error $\sim$ 10-30%.

66-140

WILSKE K. R., ROSS R.

Autoradiographic localisation of lipid- and water-soluble compounds : a new approach.

J. Histochem. Cytochem, **13**. 38 (1965)
N. S. A. **19**, 43696 (1965)

After administration of estradiol-^{3}H and aspirin-^{3}H tissues were quick frozen, frozen-dried, fixed in the vapor phase with osmium tetroxide or paraformaldehyde, and vacuum embedded in Epon. One-μ sections were coated with emulsion and subsequently developed.

66-141

ZYABKINA E. P.,
MALINOVSKII V. Yu.
MUSAKIN A. P., SOKOLOVA R. I.

Polarographic analysis of some labeled organic substances.

Metody Analisa Radioaktiva. Preparatov. Sb. Statei **1965** 46.
CA **63**, 14046g (1965)

Admixts. of gluconolactone and $H_2C_2O_4$ in samples of glucose-^{14}C were detd., like wise progesterone-4-^{14}C.

66-142

BAGGIOLINI M.

Eine einfache Technik für die Absorption von $^{14}CO_2$ und dessen direkte Messung im Flüssigkeits-Szintillations-zähler.

Experientia **21**, 731 (1965)

Novel and simple kind of $^{14}CO_2$ trap applicable to direct subsequent scint. counting.

66-143

BRENNER-HOLZACH O., LEUTHARDT F.

Methode zur Bestimmung der spezifischen Radioaktivität der C-Atome von Carbonsäuren beim SCHMIDT'schen Abbau.

Helv. Chim. Acta **48**, 1804 (1965)

Absorption of CO_2 in 0.5 N NaOH, manometric detn. in an aliquot part in a Warburg app., absorption in cyclohexylamine of another aliquot part for scintillation counting.

66-144

GEORGI P., LOBER M.

Observations concerning the unstable behavior of ^{14}C-phenylethylamine carbamate in liquid scintillation mixtures.

Acta Biol. Med. Ger., **14**, 91 (1965)
N.S.A. **19**, 38746 (1965)

66-145

KUYPER A. C., CORTESE T. A., AGHDASHI M.

Wet-combustion method for carbon-14 analysis.

Anal Biochem. **8**, 272 (1964)
CA **63**, 18639h (1965)

CO_2 collection in NaOH soln. after combustion with Van Slyke-Folch reagent. In the closed system, the CO_2 was scrubbed in 3 successive traps contg. 30 ml. 5% hydroquinone in 0.01 N H_2SO_4, 30 ml. 1 N NaOH, and 25 ml. 0.01-0.1N NaOH. The aeration tube was washed with H_2O and 5 ml. of 10% $BaCl_2$ and excess NaOH was titrated.

66-146

ROBERTS E., SIMONSEN D. G., SISKEN B.

A convenient method for the determination of metabolically liberated $^{14}CO_2$.

Advan. Tracer Methodol. **2**, **93** (1965)
CA **63**, 16762c (1965)

The method enables small amts. of $^{14}CO_2$ to be trapped in Hyamine base contained in a regulation size counting vial, and to be counted under standard conditions without any addnl. pipetting.

66-147

FODORNE C. P.,

Measurement of soft β-emitting isotopes by means of liquid scintillators.

Atomtech. Tajekoztato; **8**, 80 (1965)
N. S. A. **19**, 39002 (1965)
Review article, 19 references.

66-148

PETROFF C. P., PATT H. H., NAIR P. P.

A rapid method for dissolving tissue for liquid scintillation counting.

Intern. J. Appl. Radiation Isotopes **16**, 599 (1965)

2 N Solution of methanolic KOH as the solubilizing agent, and ethylene glycol monobutyl ether to bring about complete miscibility of the tissue digest with the toluene scintillator solution.
Counting efficiencies for ^{14}C and ^{3}H : 46.5-60.6 and 6.0-8.1% resp.

66-149

KNAELMANN M.

A rapid technique for the determination of soft β-rays from small quantities of plant materials.

Atompraxis **11**, 393 (1965)
CA **63**, 16762g (1965)

Pulverisation and suspension in EtOH, counting of ^{14}C or ^{35}S in a CH_4 flow counter.

66-150

HORESOVSKY O., FRANC Z.

Measurement of soft β-radiation in biological material with the aid of liquid scintillators. III. Determination of ^{35}S after dissolving the sample in nitric acid.

Collection Czech. Chem. Commun. **30**, 3218 (1965)

Decolorisation of colored samples with hydrogen peroxide and dissolution of the evaporation residue in the scintillator itself, in water, and in alcohol.
(See 65-556 and -557.)

66-151

KENNEDY I. R.

Release of nitrogen from amino acids with ninhydrin for ^{15}N analysis.

Anal. Biochem. **11**, 105 (1965)

A quant. method is described, which can be applied to the detn. of ^{15}N enrichment in amino acids.

See also : **66-36**, Tritium detn.
66-39, Organic acids methyl esters-^{3}H, detn.
66-134, GLC for detn. of activity

4.2 — Apparatus

66-152

JORDAN P., KOEBERLE P., LYKOUREZOS A. P.,

Experiences in the determination of ^{3}H and ^{14}C in organic compounds with proportional counters.

Mikrochim. Ichnoanal. Acta **1965**, 660.
CA **63**, 15809b (1965)

For I, the method by Wilzbach without any changes; for ^{14}C, according to Anderson, the following problems and modifications are discussed : prepn. of catalyst, substitution of quartz tubes for glass ones, thermostating of cooled traps for H_2O and CO_2, treatment of the traps before measurement of gas pressure, and influence of O flow rate in the combustion tube. Errors are O. 3% for C and O.2-O.3% for H.
(See also 65-362, and the following abstracts.)

66-153

JORDAN P.

Simultaneous gas-proportional counting of ^{3}H and ^{14}C.

Nucleonics **23**, (11) 46 (1965)

For measuring ^{14}C and tritium in organic compounds, gas-proportional counting is more precise (O.2%), more sensitive and less expensive than the more usual liquid-scintillation counting technique.

66-154

JORDAN P.,
LYKOUREZOS Ph. A. P.

Präzisionsmethode zur Routinebestimmung von ^{14}C und ^{3}H im Proportional-Zählrohr.

Intern. J. Appl. Radiation Isotopes **16**, 631 (1965)

Method for the maintenance of the standard error at the 0.1 per cent level and immediate detection of any abnormal source of error.

66-155

LEVENBOOK L., DINAMARCA M. L.

A simplified ionization chamber procedure for the continuous measurement of respiratory $^{14}CO_2$ of insects.

Anal. Biochem. **11**, 391 (1965)

The experimental insect is placed directly in the ionization chamber of a Nuclear Chicago Corp. Dynacon.

66-156

CHOULES G. L., ZIMM B. H.

An acrylamide gel soluble in scintillation fluid: its application to electrophoresis at neutral and low pH.

Anal. Biochem. **13**, 336 (1965)

By the use of the cross-linking agent, ethylene diacrylate, in place of the usual N,N′-methylenebisacrylamide; the new gel is soluble in Kinard's scintillation-counting fluid after treatment with piperidine for measurement of the radioactivity of labeled protein.

66-157

CHRISTMAN D. R.

Radioisotope techniques in biology-Part I. Measuring radioisotopes in organic compounds.

Nucleonics **23** (12), 39 (1965).

Liquid or solid scintillators, ion chambers or gas counters for measuring the beta particles from ^{3}H, ^{14}C or ^{35}S labeled compounds in biology. Criteria for choosing among them are given.

66-158

HARDIE G., PRESTON C. C.,
EZOP J. J.

A study of the feasibility of using scintillating fibers for low energy beta counting.

IITRI-578P26-8 (1965).
N. S. A. **19**, 44451 (1965)

As a specific example, a medical experiment involving an external blood loop with a-live kidney is discussed. In this particular experiment the liquid scintillation counting technique cannot be used but the fiber beta detector is suitable.

66-159

PANEK K., MUDRA K.

Continuous measurement of the radioactivity in the separation of compounds labeled with ^{35}S by gas-liquid chromatography.

Radiokhimiya **7**, 246 (1965)
CA **64, 257e** (1966)

Proportional counter, satisfactory operation up to 250°, sensitivity at 100° 0.4×10^{-9} Ci ^{35}S.

66-160

SEIMIYA T., SEKINE K., SASAKI T.

Counting of tritium with a thin-windowed Geiger-Mueller counter tube.

J. Sci. Instr. **42,** 90 (1965)
CA **64,** 257a (1966)

Thin film ($10\text{-}40\gamma$/sq. cm.) of polycarbonate treated with poly(vinyl alc.) Counting efficiency 6 to 15% of that for the same source obtained by using the windowless 2π counter.

66-161

THOMAS R. C., JUDY R. W., HARPOOTLIAN H.

Dispenser for addition of internal standard in liquid scintillation counting.

Anal. Biochem. **13,** 358 (1965)

Hamilton repeating dispenser and gastight syringe, designed to deliver one-fiftieth the syringe volume each time its button is depressed. It is thus possible to add 49 aliquots of internal standard before having to refill the syringe. Standard deviation 1.01% for a single addn.

66-162

HORWITZ H., KEREIAKES J. G., GOLDSMITH R. E., POLLACK S. V.

The study and analysis of radiochromatograms following ^{131}I administrations to humans.

Intern. J. Appl. Radiation Isotopes **16,** 721 (1965)

An automated approach to the digitization, analysis and storage of radiochromatographic information is presented.

66-163

BARSDATE R. J., DUGDALE R. C.

Rapid conversion of organic nitrogen to N_2 for mass spectrometry : an automated Dumas procedure.

Anal. Biochem. **13,** *1 (1965)*

The Dumas method as applied to the Coleman Nitrogen Analyser involves oxidation at 700°-1100°C of a sample packed with cupric oxide in a quartz combustion tube. CO_2 of high-purity sweeps the sample through a postheater tube and then into the nitrometer, containing strong KOH soln.

4.3 — Determination of radiochemical pattern

66-164

SRINIVASAN P. R.

The biosynthesis of anthranilate from (3,4-^{14}C)glucose in Escherichia coli.

Biochemistry **4,** 2860 (1965)

Anthranilic acid-^{14}C degradn. via salicylic acid (I), → dinitrosalicylic acid → picric acid, bromopicrin cleavage of these two acids; from I → phenol → trimethylacetic acid.

See also : **66-44** p-Terphenyl-^{3}H degradn.
66-49 α-D-Glucose-3-^{3}H and deriv.

5 — MISCELLANEOUS

66-165

KEGLEVIC D.

Synthesis and application of ^{14}C labeled compounds.

Nukl. Energija No. 2, 14 (1965)
N. S. A. **19**, 42589 (1965)

66-166

PARR R. M.

Measuring radioisotopes in biomedical samples.

Nucleonics **23**; (9), 56 (1965)
N. S. A. **19**, 43659 (1965)

Gamma spectrometry, computer applications, semi-conductor detectors, thin-layer chromatography, liquid-scintillation counting, radioactivity standardization, and activation analysis for measuring radioisotopes in biological and medical samples were discussed at the IAEA symposium, in Vienna, May 24 to 28, 1965. Applications in health physics were also considered.

66-167

PROKOF'EVA G. P., TUPITSYN I. F.

The production of labeled compounds.

Tr. Goz. Inst. Prikl. Khim. No. **52**, 5 (1964)
CA **63**, 12602c (1965)

Compds. intended for nonmedical purposes involved transforming an isotope from its initial chem. form into the required compd., quality control of the product, including the detn. of its chem. and radioactive purity, analysis of the sp. act. of the compd., and the utilization of by-products, (^{14}C, ^{35}S, ^{36}Cl, ^{32}P, and ^{3}H).

66-168

TEOFILOVSKI C., JEZDIC V.

Production of radioisotopes and labeled compounds at the Boris Kidric Institute of Nuclear Sciences.

Nukl. Energija, N° 2, 2 (1965)
N. S. A. **19**, 42587 (1965)